FINAL
REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN

PROJECT OPERATIONS PLAN

VOLUME III-B
QUALITY ASSURANCE PROJECT PLAN

OLIN CHEMICAL SUPERFUND SITE
51 EAMES STREET
WILMINGTON, MASSACHUSETTS

Submitted to:
United States Environmental Protection Agency
Region I – New England
One Congress Street
Boston, Massachusetts 02114

Submitted by:
Olin Corporation
3855 North Ocoee Street, Suite 200
Cleveland, Tennessee 37312

Prepared by:

MACTEC

MACTEC Engineering and Consulting, Inc.
107 Audubon Road
Wakefield, Massachusetts 01880

Project No. 6107090016/01

August 14, 2009

SDMS DocID 458071

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MACTEC Electronic Signature
Peter Thompson
Project Manager

MACTEC Electronic Signature
Michael Murphy
Project Principal

MACTEC Electronic Signature
Christian Ricardi
Project Quality Assurance Officer

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1.0 INTRODUCTION

This document is the Quality Assurance Project Plan (QAPP) for remedial investigations and actions completed at the Olin Chemical Superfund Site located in Wilmington, Massachusetts. This QAPP and the associated the Field Sampling Plan (FSP) comprise the Sampling and Analysis Plan (SAP) for the RI/FS. Together with the Work Plan these documents define investigation objectives, procedures, and quality assurance and quality control (QA/QC) for this program. This project will be completed in accordance with the Statement of Work, Remedial Investigation , and Feasibility Study (RI/FS) prepared by the United States Protection Agency (USEPA) and dated June 2007. QA/QC objectives identified in USEPA New England (Region 1) and the Massachusetts Contingency Plan (MCP) have been incorporated into this QAPP. The format of this document is based on Region I, EPA-New England guidance (USEPA, 1999). Discussions of historical data and previous investigations, proposed remedial investigation project objectives, data quality objectives (DQOs), sampling design and rationale, and schedule are detailed in the FSP and in the Draft FRI (MACTEC, 2007).

EPA-NE QAPP Worksheet #1

1.1 TITLE AND APPROVAL PAGE

Quality Assurance Project Plan for Remedial Investigation/Feasibility Study, Olin Wilmington Site, Wilmington, Massachusetts
Document Title

US EPA
Lead Organization (Agency, State, Tribe, Federal Facility, PRP, or Grantee)

Tige Cunningham and Chris Ricardi, MACTEC Engineering and Consulting, Inc.
Preparer's Name and Organizational Affiliation

511 Congress Street, Portland, ME 04102
Preparer's Address and Telephone Number

August 14, 2009
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: **MACTEC Electronic Signature**
Peter Thompson/MACTEC Engineering and Consulting, Inc.
August 13, 2009

Investigative Organization's Project QA Officer: **MACTEC Electronic Signature**
Chris Ricardi/MACTEC Engineering and Consulting, Inc.
August 13, 2009

Lead Organization's Project Manager: **Olin Electronic Signature**
Signature/Date
Steve Morrow/Olin Corporation

Approval Signatures: _____
Signature/Date
James M. Dilorenzo/ Remedial Project Manager, EPA

Other Approval Signatures: **MACTEC Electronic Signature**
Michael Murphy/Project Principal, MACTEC Engineering and Consulting, Inc.
August 13, 2009

EPA-NE QAPP Worksheet #2

2.0 DOCUMENT OVERVIEW

Site Name/Project Name: Olin Chemical Superfund Site
Site Location: Wilmington, Massachusetts
Site Number/Code: EPA ID # MAD001403104
Operable Unit: OU1, OU2, and OU3
Contractor Name: MACTEC Engineering and Consulting, Inc.
Contract Number: GCA – N/A
Contract Title: N/A
Work Assignment Number: 6107090016/01
Anticipated date of QAPP Implementation: Summer/Fall 2009

1. Identify Guidance used to prepare QAPP:

Region I, EPA-New England Compendium of Quality Assurance Project Plan Guidance, Final, October 1999.

2. Identify EPA Program: **CERCLA/Superfund**

3. Identify approval entity: **EPA-NE**

4. Indicate whether the QAPP is a generic program QAPP or a project specific QAPP. (underline one)

5. List dates of scoping meetings that were held:
January 17, 2008/March 18, 2008/July 17, 2008/March 17, 2009

6. List title of QAPP documents and approval dates written for previous site work, if applicable:

Title	Approval Date
"Final Quality Assurance Project Plan, Olin Wilmington Property, 51 Eames Street, Wilmington, MA", LAW Engineering and Environmental Services, Kennesaw, GA 30144.	August 1999

7. List organizational partners (stakeholders) and connection with EPA and/or State:

United States Environmental Protection Agency Region 1
Olin Corporation
Nobis Engineering
Wilmington Environmental Restoration Committee (WERC)

MADEP/USEPA – Primary Regulatory Agencies

8. List data users:

US EPA, MADEP, MACTEC, Olin Corporation, other subcontractors.

9. If any required QAPP Elements (1-20), Worksheets and/or Required Information are not applicable the project, then underline the omitted QAPP Elements, Worksheets and Required Information on the attached Table. The following worksheets were omitted from the QAPP:

Worksheet #8b (Problem Definition/Site History and Background) – Information on the Site history and background information is presented in the Draft Focused RI Report (MACTEC, 2007).

Worksheet #12a (Logic to determine sample locations) – Information detailing the logic used to determine sample location, analytical parameters, numbers and frequency is presented in the FSP.

Worksheet #14 (Field Equipment Calibration) - Field equipment intended for investigation at the Olin Site include submersible and peristaltic pumps for ground water sample collection. Calibration of these pumps is not required.

Worksheet #22b – Field duplicates and QC blank criteria are established for all analytes in each method on Worksheet #22a. Criteria for individual analytes is not needed.

Worksheet #24b (Fixed Laboratory Method/SOP Precision and Accuracy Table) – Laboratory sensitivity and quantitation limits for analytical parameters with multiple analytes is presented in Worksheet 9b. Analytical precision and accuracy acceptance criteria for the analytes and methods are presented in Worksheet #11 and Worksheet #24a.

Worksheet #25 (Non-Direct Measurements Criteria and Limitations Table) – Information detailing data collected outside of the current data collection activity that will be used to make environmental decisions is presented in the Draft Focused RI Report (MACTEC, 2007).

Worksheet #27a (Assessment and Response Actions) – Information detailing procedures for indentifying the correcting problems during the project is detailed in Section 16 of the QAPP.

Worksheet #30 (Data Usability Assessment) – Information detailing procedures for determining data usability assessment is detailed in Section 20 of the QAPP.

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA NEQAPP Worksheet #	REQUIRED INFORMATION
Project Management and Objectives			
A1	1.0 Title and Approval Page	1	- Title and Approval Page
A2	2.0 Table of Contents and Document Format 2.1 Table of Contents 2.2 Document Control Format 2.3 Document Control Numbering System 2.4 EPA-NE QAPP Worksheet #2	2	- Table of Contents - EPA-NE QAPP Worksheet
A3	3.0 Distribution List and Project Personnel Sign-off Sheet	3 4	- Distribution List - Project Personnel Sign-off Sheet
A4, A8	4.0 Project Organization 4.1 Project Organizational Chart 4.2 Communication Pathways 4.2.1 Modifications to Approved QAPP 4.3 Personnel Responsibilities and Qualifications 4.4 Special Training Requirements/ Certification	5a 5b 6 7	- Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
A5	5.0 Project Planning/Project Definition 5.1 Project Planning Meetings 5.2 Problem Definition/Site History and Background	8a <u>8b</u>	- Project Scoping Meeting Attendance Sheet with Agenda and other Project Planning Meeting Documentation - Problem Definition/Site History and Background - EPA-NE DQO Summary Form - Site Maps (historical and present)
A6	6.0 Project Description and Schedule 6.1 Project Overview 6.2 Project Schedule	9a 9b 9c 9d 10	- Project Description - Contaminants of Concern and Other Target Analytes Table - Field and Quality Control Sample Summary Table - Analytical Services Table - System Designs - Project Schedule Timeline Table
A7	7.0 Project Quality Objectives and Measurement Performance Criteria 7.1 Project Quality Objectives 7.2 Measurement Performance Criteria	11	- Project Quality Objectives/Decision Statements - Measurement Performance Criteria Table

Note: Underlined worksheet numbers are not included in QAPP, see page 2-2.

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA NEQAPP Worksheet #	REQUIRED INFORMATION
Measurement/Data Acquisition			
B1	8.0 Sampling Process Design 8.1 Sampling Design Rationale	12a 12b	- Sampling Design and Rationale - Sampling Locations, Sampling and Analysis Method/SOP Requirements Table - Sample Location Map
B2, B6, B7, B8	9.0 Sampling Procedures and Requirements 9.1 Sampling Procedures 9.2 Sampling SOP Modifications 9.3 Cleaning and Decontamination of Equipment/Sample Containers 9.4 Field Equipment Calibration 9.5 Field Equipment Maintenance, Testing and Inspection Requirements 9.6 Inspection and Acceptance Requirements for Supplies/Sample Containers	13 12b 14 15	- Sampling SOPs - Project Sampling SOP Reference Table - Sampling Container, Volumes and Preservation Table - Field Sampling Equipment Calibration Table - Cleaning and Decontamination SOPs - Field Equipment Maintenance, Testing and Inspection Table
B3	10.0 Sample Handling, Tracking and Custody Requirements 10.1 Sample Collection Documentation 10.1.1 Field Notes 10.1.2 Field Documentation Management System 10.2 Sample Handling and Tracking System 10.3 Sample Custody	16	- Sample Handling, Tracking and Custody SOPs - Sample Handling Flow Diagram - Sample Container Label (Sample Tag) - Chain-of-Custody Form and Seal
B4, B6, B7, B8	11.0 Field Analytical Method Requirements 11.1 Field Analytical Methods and SOPs 11.2 Field Analytical Method/SOP Modifications 11.3 Field Analytical Instrument Calibration 11.4 Field Analytical Instrument/ Equipment Maintenance, Testing and Inspection Requirements 11.5 Field Analytical Inspection and Acceptance Requirements for Supplies	17 18 19	- Field Analytical Methods/SOPs - Field Analytical Method/SOP Reference Table - Field Analytical Instrument Calibration Table - Field Analytical Instrument/Equipment Maintenance, Testing and Inspection Table

Note: Underlined worksheet numbers are not included in QAPP, see page 2-2.

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA NEQAPP Worksheet #	REQUIRED INFORMATION
B4, B6, B7, B8	12.0 Fixed Laboratory Analytical Method Requirements 12.1 Fixed Laboratory Analytical Methods and SOPs 12.2 Fixed Laboratory Analytical Method/SOP Modifications 12.3 Fixed Laboratory Instrument Calibration 12.4 Fixed Laboratory Instrument/ Equipment Maintenance, Testing and Inspection Requirements 12.5 Fixed Laboratory Inspection and Acceptance Requirements for Supplies	20 21	- Fixed Laboratory Analytical Methods/SOPs - Fixed Laboratory Analytical Method/SOP Reference Table - Fixed Laboratory Instrument Maintenance and Calibration Table
B5	13.0 Quality Control Requirements 13.1 Sampling Quality Control 13.2 Analytical Quality Control 13.2.1 Field Analytical QC 13.2.2 Fixed Laboratory QC	22a 22b 23a 23b 24a 24b	**Sampling** - Field Sampling QC Table - Field Sampling QC Table cont. **Analytical** - Field Analytical QC Sample Table - Field Analytical QC Sample Table cont. - Field Screening/Confirmatory Analysis Decision Tree - Fixed Laboratory Analytical QC Sample Table - Fixed Laboratory Analytical QC Sample Table cont.
B9	14.0 Data Acquisition Requirements	25	- Non-Direct Measurements Criteria and Limitations Table
A9, B10	15.0 Documentation, Records and Data Management 15.1 Project Documentation and Records 15.2 Field Analysis Data Package Deliverables 15.3 Fixed Laboratory Data Package Deliverables 15.4 Data Reporting Formats 15.5 Data Handling and Management 15.6 Data Tracking and Control	26	- Project Documentation and Records Table - Data Management SOPs

Note: Underlined worksheet numbers are not included in QAPP, see page 3-2.

REQUIRED EPA QA/R-5 QAPP ELEMENTS	REQUIRED EPA-NE QAPP ELEMENTS and CORRESPONDING EPA-NE QAPP SECTIONS	EPA NEQAPP Worksheet #	REQUIRED INFORMATION
Assessment/Oversight			
C1	16.0 Assessments and Response Actions 16.1 Planned Assessments 16.2 Assessment Findings and Corrective Action Responses 16.3 Additional QAPP Non-Conformances	27a 27b	- Assessment and Response Actions - Project Assessment Table - Project Assessment Plan - Audit Checklists
C2	17.0 QA Management Reports	28	- QA Management Reports Table
Data Validation and Usability			
D1	18.0 Verification and Validation Requirements		- Validation Criteria Documents
D2	19.0 Verification and Validation Procedures	29a 29b 29c	- Data Evaluation Process - Data Validation Summary Table - Data Validation Modifications
D3	20.0 Data Usability/Reconciliation with Project Quality Objectives	30	- Data Usability Assessment

Note: Underlined worksheet numbers are not included in QAPP, see page 2-2.

3.0 DISTRIBUTION LIST AND PROJECT PERSONNEL SIGN-OFF SHEET

3.1 DISTRIBUTION LIST

The Distribution List (Worksheet #3) documents who will receive copies of the approved QAPP and any subsequent revisions or amendments to the QAPP. A complete copy of the QAPP and any subsequent revisions will be maintained on file at the Olin Corporation.

3.2 PROJECT PERSONNEL SIGN-OFF SHEET

Project personnel performing field related RI work on the Olin Wilmington Site will read this QAPP and perform tasks as described in this QAPP. The project personnel sign-off sheet (Worksheet #4) will be used to document that the appropriate personnel have read the QAPP. This requirement extends to MACTEC personnel and other subcontractors involved with data collection and analysis.

Copies of signed project personnel sign-off sheets will be returned to MACTEC and maintained in the central project file at MACTEC. These sheets will be made available to Olin, Massachusetts DEP, and USEPA-NE upon request.

EPA-NE QAPP Worksheet #3

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Number of Copies
James M. DiLorenzo	Remedial Project Manager	USEPA	617-918-1247	7
Steve Morrow	Olin Site Manager	Olin	423-336-4511	1
Peter Thompson	Project Manager	MACTEC, Portland	207-775-5401	1
Michael Murphy	Project Principal	MACTEC, Wakefield	781-245-6606	1
Joe Coyne	MADEP Project Manager	MADEP	617-348-4066	1

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #4

Project Personnel Sign-Off Sheet

Organization: MACTEC Engineering & Consulting, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Peter Thompson	Project Manager	207-775-5401			
Michael Murphy	Project Principal	781-245-6606			
Chris Ricardi	Project QA Officer and Lead Chemist	207-775-5401			
Tige Cunningham	Project Chemist	207-775-5401			
Chris Mazzolini	Field Operations Leader	781-245-6606			

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #4

Project Personnel Sign-Off Sheet

Organization: Olin Corporation

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Steve Morrow	Olin Site Manager	423-336-4511			

Organization: GeoMega

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Chris Dacey	Senior Hydrogeologist	720-266-5426			

Prepared by: TC
Checked by: CR
10/14/08

Organization: TestAmerica – Westfield Massachusetts

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Steven Hartman	Lab Manager	413-572-4000			
Jamie Wickham	Lab QA Manager	413-572-4000			
Becky Mason	Lab Project Manager for Olin	413-572-4000			

4.0 PROJECT ORGANIZATION

This section identifies the organizations and key personnel participating in the Olin Chemical Superfund Site project. The specific roles and responsibilities of the key personnel are included in this section. An explanation of the lines of authority, reporting relationships and communication pathways are provided. Qualifications of key personnel are included in the resumes included in Appendix A.

4.1 PROJECT ORGANIZATION CHART

Organizations involved in the Olin Chemical Superfund Site project are identified in the project organization chart (Worksheet #5a/5b). The responsibilities of key personnel are described in Section 4.3.

4.2 COMMUNICATION PATHWAYS

The lines of authority and communication specific to this study are also presented in the organization chart (Worksheet #5a/5b). The MACTEC Project Manager will serve as the communication link between Olin Corporation and MACTEC. The MACTEC Project Manager will be kept verbally apprised of the program's status by the MACTEC Field Operations Leader and the MACTEC Project Quality Assurance (QA) Officer. These individuals will notify the MACTEC Project Manager in a timely manner of any internal or subcontractor issues that potentially affect budget, schedule, and/or achievement of the project objectives. The MACTEC Project Manager will in turn communicate these issues to the Olin Corporation Project Manager. Laboratories will communicate issues to the MACTEC Project Chemist who will in turn communicate these issues to the MACTEC Project Manager. The MACTEC Project Manager will in turn notify the Olin Corporation PM of these issues.

4.2.1 Modifications to Approved QAPP

Any predetermined changes to the scope or procedures stated in this QAPP will be documented as QAPP revisions and must go through the same review and approval process as the original QAPP. The control block in the upper right corner of each changed page will be updated to reflect the date of the change and the revision number.

For changes requiring immediate resolution and implementation, approval by phone will be secured from Olin Corporation and USEPA. This verbal approval will be documented in phone logs and will be followed by formal revision of the QAPP as described above.

If modifications to the QAPP are required, the MACTEC Project QA Officer will schedule a meeting with the appropriate Olin Chemical Superfund Site team members to discuss the changes, make the necessary modifications to the QAPP, and submit them to the Project Manager for review. The revised QAPP, or QAPP sections, will be presented to Olin Corporation, MADEP, and USEPA for review and approval. After the QAPP has been approved, the revised QAPP, or QAPP sections, will be provided to the Olin Chemical Superfund Site team members, according to the original QAPP Distribution List.

The project personnel sign-off sheet will be resubmitted to the appropriate personnel and will ensure that all appropriate personnel have read the revised QAPP. This sheet will be returned to MACTEC, stored in the project files, and will be available upon request.

4.3 PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS

The responsibilities of management, QA, field, and laboratory personnel are outlined below. Project personnel in responsible roles are identified in Worksheet #6, by name, title and affiliation. The resumes of these key personnel are included in Appendix A.

4.3.1 Management Responsibilities

Site Team Leader

Garland Hilliard is the Olin Site Team Leader and is responsible for the overall management of Olin's responsibilities under the Agreement on Consent (AOC). He will hold the following roles and responsibilities:

- Responsible for leading activities designed to meet Olin's obligations under the AOC and SOW;
- Responsible for providing review and approval of deliverables prepared for submission to USEPA in fulfillment of the Olin's obligations under the AOC and SOW; and
- Responsible for communication of Olin project commitments to Olin Management.

Olin Corporation Project Manager

The Site Manager (SM) for the Olin Chemical Superfund Site is Steve Morrow. His primary responsibilities include administration of Olin Corporation responsibilities, oversight of the day-to-day activities, and receipt of all required written matter. Mr. Morrow is also responsible for providing technical oversight and guidance and reviewing all technical deliverables, including plans and reports.

MACTEC Project Manager

The MACTEC Project Manager, Mr. Peter Thompson, has responsibility for technical, financial, and scheduling matters and will serve as the main contact with the Olin Corporation PM. Other duties, as necessary, include:

- assuring adherence to project plans and obtaining approvals for any changes to these plans;
- assuring that approved procedures meet project objectives;
- reviewing and approving all sampling procedures;
- assigning duties to project staff and orienting the staff to the specific needs and requirements of the project;
- serving as the focus for coordination of field task activities, communications, reports, and technical reviews, and other support functions, and facilitating activities with the technical requirements of the project;
- coordinating field and office activities with the Project QA Officer, Project Chemist, and Field Operations Leader;
- implementing recommendations made by the Project QA Officer
- initiating corrective actions;
- monitoring schedules for field, analytical, and data validation activities associated with the field sampling program; and
- Maintaining the project file.

MACTEC Project Principal (Technical Leader)

The MACTEC Project Principal, Michael Murphy, has responsibility for providing technical guidance during field sampling activities, analysis of data and production of reports. Other duties, as necessary, include

- provide senior technical guidance and direction to the project;

- technical oversight of project deliverables during their preparation;
- responsible for review and approval of MACTEC deliverables in conjunction with the Project Manager;
- responsible for technical direction of project staff under his direct supervision;
- responsible for the technical quality of MACTEC project work and deliverables;
- direct technical staff during preparation of technical deliverables and oversee review of other professionals to verify work is being completed by professionals with appropriate training and experience to competently complete work efforts;
- direct technical scope of data collection activities to verify that data intended to be collected is sufficient, suitable and appropriate for completion of risk assessment activities for the project; and
- provide senior technical support at meetings requested by USEPA.

4.3.2 Quality Assurance Responsibilities

MACTEC Project QA Officer

The MACTEC Project QA Officer, Mr. Chris Ricardi, has overall responsibility for quality assurance oversight. The MACTEC Project QA Officer communicates directly to the MACTEC Project Manager. Specific responsibilities include:

- preparing the QAPP;
- reviewing and approving QA procedures, including any modifications to existing approved procedures;
- ensuring that QA audits of the various phases of the project are conducted as required;
- providing QA technical assistance to project staff;
- following up on corrective action;
- ensuring that data validation/data assessment is conducted in accordance with the QAPP; and
- reporting on the adequacy, status, and effectiveness of the QA program to the MACTEC Project Manager.

4.3.3 Field Responsibilities

MACTEC Field Operations Leader

The MACTEC Field Operations Leader, Mr. Mazzolini, has overall responsibility for completion of field activities in accordance with the QAPP and is the communication link between the field team, subcontractors, and MACTEC project management. Specific responsibilities include:

- understanding and implementing the Field Sampling Plan and QAPP;
- coordinating activities in the field;
- assigning specific duties to field team members;
- ensuring site security and access;
- training field staff;
- overseeing and coordinating field data collection;
- creating and maintaining the Site logbook;
- creation, distribution, and tracking of field logbooks;
- ensure use, review, and filing of Field Data Records (FDRs);
- mobilizing and demobilizing of the field team and subcontractors to and from the site,
- resolving any logistical problems that could potentially hinder field activities, such as equipment malfunctions or availability, personnel conflicts, or weather-dependent working conditions;
- implementing field quality control (QC) including issuance and tracking of measurement and test equipment; supervision of the proper labeling, handling, storage, and shipping of samples including chain-of-custody procedures and control of field documentation; and
- assisting with report preparation.

Field Staff

The field staff reports directly to the MACTEC Field Operations Leader. The responsibilities of the field team include:

- understanding and implementing the FSP and the QAPP requirements as they relate to their duties;
- collecting samples, conducting field measurements, and decontaminating equipment according to documented procedures stated in the QAPP;
- ensuring that field instruments are properly operated, calibrated, and maintained, and that adequate documentation is kept for all instruments;

- collecting the required QC samples and thoroughly documenting QC sample collection;
- ensuring that field documentation procedures are followed and data are complete and accurate;
- complete field logbook entries documenting daily activities;
- complete all FDRs applicable to tasks assigned; and
- communicating any nonconformance or potential data quality issues to the MACTEC Field Operations Leader.

MACTEC Project Chemist

The MACTEC Project Chemist, Mr. Tige Cunningham, will be the primary line of communication with the analytical laboratories and will be responsible for coordinating delivery of laboratory reports to MACTEC. He will organize lab data and maintain documentation of data review and data validation activities for the project.

4.3.4 Laboratory Responsibilities

Analyses will be performed by various laboratories under a subcontract to MACTEC. The primary laboratory will be TestAmerica (TAL) in Westfield, Massachusetts. The shipping address of TAL Westfield is:

TestAmerica
53 Southampton Road
Westfield, MA 01085
Phone: 413.572.4000, FAX: 413-572-3707

Laboratory Manager

The Laboratory Manager is ultimately responsible for the data produced by the laboratory. The current TestAmerica-Westfield Lab Manager is Steve Hartman. His email address is: Steve.Hartmann@testamericainc.com. Specific responsibilities of the lab manager include:

- implementing and adhering to the QA and corporate policies and procedures within the laboratory
- approving Standard Operating Procedures (SOPs)
- maintaining adequate staffing
- implementing internal/external audit findings and corrective actions

Laboratory QA Manager

The Laboratory QA Manager reports directly to the Laboratory Manager. The current TestAmerica-Westfield Lab QA Manager is Christine Furcinite-Reynolds. Her email address is: chris.reynolds@testamericainc.com. Specific responsibilities include:

- approving the laboratory SOPs
- ensuring and improving quality within the laboratory
- supervising and providing guidance and training to laboratory staff
- addressing all client inquiries involving data quality issues
- performing QA audits and assessments
- tracking external and internal findings of QA audits
- coordinating laboratory certification and accreditation programs

Laboratory Project Manager

The Laboratory Project Manager is the primary point of contact between the laboratory and MACTEC. The current TestAmerica-Westfield Project Manager for Olin is Becky Mason. Her email address is: Becky.Mason@testamericainc.com. Specific responsibilities of the Laboratory Project Manager include:

- keeping the laboratory and client informed of project status;
- monitoring, reviewing, and evaluating the progress and performance of projects;
- reporting client inquiries involving data quality issues or data acceptability to the Laboratory QA Manager and to the operations staff; and
- reviewing project data packages for completeness and compliance to client needs.

Laboratory Section Leaders

Specific responsibilities include:

- supervising daily activities within the group
- supervising QC activities
- supervising the preparation and maintenance of laboratory records

- evaluating instrument performance and supervising the calibration, preventive maintenance, and scheduling of repairs
- overseeing or performing review and approval of all data

Laboratory Analyst/Technicians

Each analyst or technician is responsible for:

- performing technical procedures and data recording in accordance with documented procedures
- performing and documenting calibration and preventive maintenance
- performing data processing and data review procedures
- reporting nonconformances to the appropriate personnel
- ensuring sample and data integrity by adhering to internal chain-of-custody procedures

Laboratory Sample Custodians

The Sample Custodian ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody. Other specific responsibilities include:

- notifying the Laboratory Project Manager of any discrepancies or anomalies with incoming samples
- logging samples into the laboratory tracking system
- ensuring that all samples are stored in the proper environment
- overseeing sample disposal

4.3.5 Additional Laboratories Performing Analyses

Lancaster Labs, Inc.
2425 New Holland Pike Lancaster, PA 17601.
Tel. (717) 656-2300
(Nicole Maljovek)

- Organics Lab Manager : Duane Luckenbill
- QA Manager: Kathy Loewen
- Olin Project Manager: Nicole Maljovek (email: nmaljovec@lancasterlabs.com) (phone [717] 656- 2308 ext. 1537)

Katahdin Analytical Services, Inc.
600 Technology Way, Scarborough, ME 04074,
Telephone: (207) 874-2400, Fax: (207) 775-4029

- Lab Manager : Deborah Nadeau
- QA Manager: Leslie Diamond
- Olin Project Manager: Andrea Colby (email: acolby@katahdinlab.com)

Analytics Lab, LLC.
195 Commerce Way, Suite E, Portsmouth, New Hampshire 03801.
Tel. (603) 436-5111 or (800) 929-9906, Fax. (603) 430-2151

- Lab Manager : Steve Knollmeyer (email address:sknollmeyer@analyticslab.com)
- QA Manager: Melissa Gulli (email address: mgulli@analyticslab.com)

4.3.6 Additional Subcontractors

Geomega Inc.

Geomega has provided consulting services to Olin on this project over the past decade and has principally been involved in hydrogeological investigations, groundwater modeling, and investigation of the geochemistry of DAPL interactions in subsurface and surface water systems. Geomega will continue to support Olin in providing these technical services for the project.

Geomega Senior Geochemist

Andy Davis is a principal at Geomega, will serve as Geomega's senior geochemist for the RI/FS project, and will contribute his technical expertise and oversight to interpretation of geochemical effects of potential remedial efforts at the Site related to DAPL constituents in the subsurface and in surface water. His input will assist evaluating effects of remedial alternatives and activities on mobility and fate of Site contaminants.

Geomega Senior Hydrogeologist

Christopher Dacey will serve as Geomega's senior hydrogeologist for the RI/FS project and will contribute his technical expertise and oversight to induction logging programs, other aspects of hydrogeologic characterization, statistics, and groundwater modeling efforts at the Site.

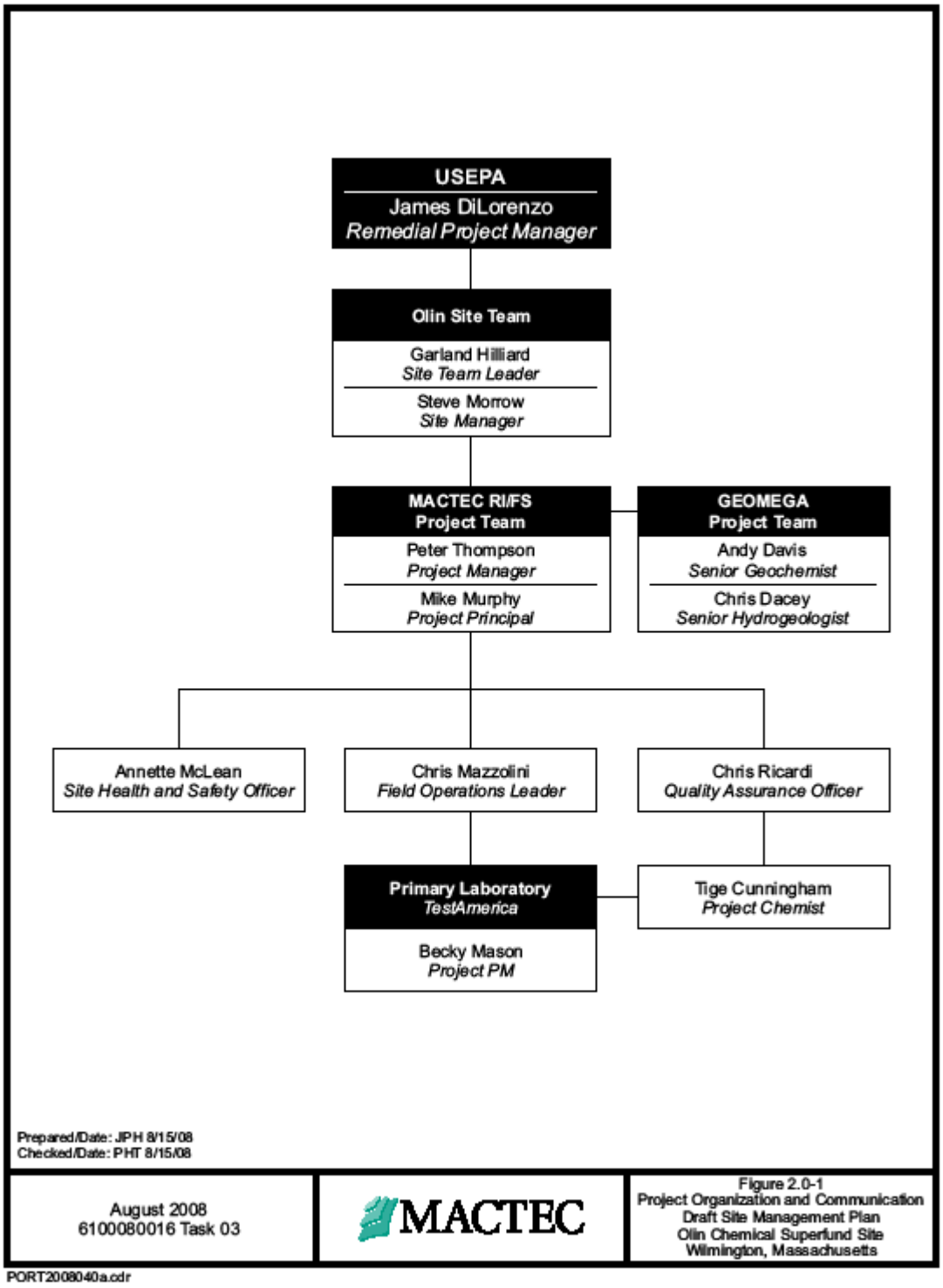
4.3.7 Special Training Requirements/Certification

Most of the activities included in this investigation include routine sampling and analyses with no special training requirements and certifications needed. Staff working on-site will have completed the OSHA/HAZWOPER 40-hour health and safety training and will also have currently (within the past year) completed the OSHA/HAZWOPER 8-hour annual refresher health and safety training. Health and safety training records are maintained by MACTEC. Worksheet #7 summarizes this information. Prior to the start of the investigation, all field personnel will be given instruction specific to the project, covering the following areas:

- organization and lines of communication and authority
- overview of the FSP and QAPP, including sample collection, handling, and labeling procedures
- QA/QC requirements
- documentation requirements
- health and safety requirements

Instructions will be provided by the MACTEC Field Operations Leader and MACTEC Project QA Officer. A Health and Safety Officer (HSO) will be designated by the Field Operations Leader during field activities. The HSO will complete the Health and Safety Log as described in Section 10.0.

EPA-NE QAPP Worksheet #5a/5b
Organization Chart/Communication Pathways



EPA-NE QAPP Worksheet #6
Personnel Responsibilities and Qualifications Table

Name	Organizational Affiliation	Responsibilities	Location of Personnel Resumes, if not included	Education and Experience Qualifications
Peter Thompson	MACTEC	Project Manager	Appendix A	Appendix A
Michael Murphy	MACTEC	Project Principal	Appendix A	Appendix A
Chris Ricardi	MACTEC	Project QA Officer and Lead Chemist	Appendix A	Appendix A
Nadia Glucksberg	MACTEC	Principal Hydrogeologist	Appendix A	Appendix A
Tony Rodalakis	MACTEC	Senior Scientist	Appendix A	Appendix A
Chris Mazzolini	MACTEC	Field Operations Leader	Appendix A	Appendix A
Tige Cunningham	MACTEC	Project Chemist	Appendix A	Appendix A

EPA-NE QAPP Worksheet #7

Special Personnel Training Requirements Table

Project Function	Specialized Training Title of Course or Description	Training Provided By	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Activities on Superfund Site	OSHA/HAZWOPER 40 hour training	Various trainers	Various	All staff to be used on-site	All	MACTEC
Field Activities on Superfund Site	OSHA/HAZWOPER 8 hour annual refresher training	Various trainers	Within last year, annually	All staff to be used on-site	All	MACTEC

Prepared by: TC
Checked by: CR
10/14/08

5.0 PROJECT PLANNING/PROJECT DEFINITION

This section documents project planning, identifies the environmental problem, defines the environmental questions that need to be answered, and provides background information.

5.1 PROJECT PLANNING MEETINGS

Project planning meetings were first held with EPA on February 1, 2006 regarding historical activities at the Olin Property (Property) and the transition to CERCLA. An Administrative Order of Consent (AOC) and Statement of Work (SOW) became effective in June 2007. A draft Focused Remedial Investigation (FRI) Report was submitted to EPA on October 2, 2007. On January 17, 2008 a meeting was held with EPA to discuss preliminary comments on that document. On March 18, 2008 a meeting was held with EPA to discuss additional comments on the Draft FRI Report and requested additional information to be included in the RI Work Plan. On July 17, 2008 another meeting was held to review progress included in the RI Work Plan and to discuss the RI Analyte list for the RI Work Plan. Worksheet #8a provides details on the meetings.

5.2 PROBLEM DEFINITION/SITE HISTORY AND BACKGROUND

This section presents an overview of historic information, current site condition descriptions, and other existing data applicable to the Olin Chemical Superfund Site project. A more detailed discussion with figures is presented in the Draft Focused Remedial Investigation Report (MACTEC, 2007) and companion FSP.

5.2.1 Site Description

The Site is comprised of the Olin Property, an approximately 50-acre parcel, at 51 Eames Street in Wilmington, Massachusetts and adjoining off-Property areas impacted by manufacturing and waste disposal activities at the Property. The northern part of this property was formerly the site of a chemical manufacturing facility.

The Site was listed on the National Priorities List (NPL) pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Section 105, 42 U.S.C. ' 9605 on April 19, 2006 (71 FR 20,016). In September 2005, when the USEPA identified the Site as a Proposed Site for the NPL, the primary substance used by USEPA to score the Site was NDMA (USEPA, 2005). The primary exposure pathway evaluated by USEPA was groundwater. Other principal contaminants noted by EPA included chloride, sodium, sulfate and ammonia. Environmental contamination has migrated to surface water at on-Property locations by groundwater pathways. Prior to the NPL listing, the Site has been the subject of many years of investigations and response activities carried out by Olin Corporation and supervised by Massachusetts Department of Environmental Protection (MADEP) under Chapter 21E of the General Laws of Massachusetts and the MCP. The Site has been a Priority site under the MCP since 1993, and a Tier I site since 1994.

5.2.2 History of Operations and Site Background

The Site includes the approximate 50-acre Olin Property and adjacent off-Property areas that have been impacted by manufacturing and waste disposal activities at the Property. The Property is located in a heavily industrialized area within the southern portion of the Town of Wilmington and is the location of a former manufacturing facility that produced specialty chemicals for the rubber and plastics industry from 1953 to 1986. From 1953 to 1980, the facility was owned by several companies whose operations resulted in environmental contamination to on-Property soil, groundwater, sediment and surface water. Supplemental information with respect to the manufacturing operations of the facility is provided in Appendix A of the FSP. That appendix summarizes manufacturing operations, including raw materials, waste materials, products, facility buildings, storage tanks, septic tanks, leach fields, and major waste disposal features. Figure 2.1-1 in the FSP shows historical features of the Property such as building locations, waste disposal features, and tank farms.

Olin purchased the Property in 1980 and ceased operations in 1986. After Olin initiated closure of the site in 1986, the chemical manufacturing buildings were demolished and removed along with the closure of the lined lagoons. Since 1987, Olin has conducted numerous environmental investigations, risk assessments and remedial actions to develop an understanding of the nature of environmental impacts and to protect human health and the environment. These investigations and cleanups were carried out by Olin and supervised by Massachusetts Department of Environmental Protection (MassDEP) under Chapter 21E of the General Laws of Massachusetts and the MCP. The investigations and assessments were based on data collected and analyzed in accordance with work plans approved by the MassDEP. Data generated

are of known data quality and usability determined by data validation procedures that met the MCP requirements.

These investigations and subsequent remedial actions have resulted in the excavation and off-site disposal of soil from Lake Poly, two Drum Disposal Areas, a Buried Debris Area and sediment from the on-Property West Ditch (on-PWD), the on-PWD Wetland, the South Ditch, and the Central Pond. All removal actions were conducted in accordance with work plans approved by the MassDEP and soils and sediments were manifested, transported and disposed of at facilities licensed and approved for such materials. An area of trimethylpentenes (TMP) in soil and shallow groundwater known as the EPH/VPH area (referring to Extractable Petroleum Hydrocarbons and Volatile Petroleum Hydrocarbon analyses) and located near Plant B was identified and successfully remediated using an air sparge/soil vapor extraction system (AS/SVE). This AS/SVE system was closed and removed subsequent to approval by the MassDEP.

Since 1997, Olin has operated the Plant B groundwater recovery/treatment system as an Immediate Response Action (IRA) under the MCP. The system was installed in response to the seepage of a light non aqueous phase liquid (LNAPL) into the East Ditch that is located at the eastern perimeter of the former facility property. The LNAPL is a process oil that also contains bis(2-ethylhexyl)phthalate (BEHP), NDPA, and TMPs. The system was designed to create a groundwater cone of depression to prevent migration of the LNAPL and to allow for mechanical removal of the material. Groundwater extracted during operation of the system is treated to remove iron and ammonia as well as dissolved organic compounds. The treated groundwater is discharged to surface water on the former Facility property in compliance with a Remediation General Permit (RGP).

In 2000/2001, Olin constructed a Slurry Wall/Cap Containment Area (the Containment Area) around the on-Property portion of the Upper Dense Aqueous Phase Liquid (DAPL) Pool as a Release Abatement Measure (RAM) consistent with the MCP. The intent of this source control action was to eliminate, to the extent feasible, the on-Property DAPL source material as a source of dissolved constituents to groundwater. The Containment Area is comprised of a perimeter slurry wall keyed into bedrock and a temporary cap to minimize infiltration of precipitation into the Containment Area. Construction of the slurry wall was preceded by a pre-design boring program to determine the depth to bedrock, and completion of chemical compatibility testing of the DAPL and the design slurry mixture. The slurry wall working with the geometry of the bedrock depression contains the on-Property DAPL and overlying impacted groundwater within the Containment Area. The compatibility testing indicated that the

hydraulic conductivity of the design slurry mix decreased over a six month period in contact with DAPL material. These results suggest that hydraulic performance of the design slurry mixture improves when in contact with DAPL material.

Currently, the Property is not in active use and contains a vacant office and laboratory building, a general purpose building with pilot lab, two warehouses, a building which houses a groundwater recovery and treatment facility (Plant B) and a closed landfill (Calcium Sulfate Landfill). In 2006, Olin installed a 40-foot office trailer near Plant B for use by the Olin employees who operate Plant B and maintain the Property. Property access is controlled along the entire Property perimeter by an eight foot high fence and all access gates are locked when Olin employees are not on the Property.

The Olin Property is situated over glacial outwash deposits that overlie glacial till and igneous and metamorphic bedrock. The saturated thickness of these glacial deposits increases within a buried bedrock valley to the northwest of the Property forming an aquifer underlying Maple Meadow Brook and Saw Mill Brook known as the MMBA. The Town of Wilmington operated a municipal water supply well field located over one-half mile northwest of the Property within the MMBA. During 2002, the Town wells were in use intermittently, with certain wells being taken off-line by the Town during different time periods in response to concerns of potentially exceeding nitrite standards in the distribution system. In October 2002, the Town ceased use of all the wells in the MMBA except the Town Park Well. In April 2003, the use of this well field was suspended by the Town of Wilmington Water Department due to the detection of N-nitrosodimethylamine (NDMA) in four of the five MMBA municipal water supply wells (MWSWs). NDMA was not detected in the Town Park Well, and NDMA was not detected within the water supply distribution system. Olin worked with the Town of Wilmington to develop a supplemental water source through a water purchase agreement from adjacent communities after the Town suspended use of the MWSWs. This supplemental water source remains in service. In order to provide a permanent supply, the town has constructed a new connection to the Massachusetts Water Resources Authority (MWRA) system that is funded by Olin and the other respondents.

In September 2005, USEPA identified the Site as a Proposed Site for the National Priorities List (NPL). The primary hazardous substance used by USEPA to score the Site was NDMA (USEPA, 2005). The primary exposure pathway evaluated by USEPA was groundwater. Other principal contaminants noted in the NPL listing included chloride, sodium, sulfate and ammonia.

EPA-NE QAPP Worksheet #8a
Project Scoping Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program:	**Site Location**: 51 Eames, Wilmington, MA
Projected Dates of Sampling: TBD 2009	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
	Other Site Number/Code: N/A
	Phase: ERA SA/SI Pre-RI RI (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:

Date of Meeting: February 1, 2006
Meeting Location: Olin Wilmington Superfund Site, Eames Street, Wilmington, MA.

Name	Project Role	Affiliation	Phone #	e-Mail Address
Peter Thompson	Project Manager	MACTEC	207-775-5401	
Mike Murphy	Principal Scientist	MACTEC	781-213-5600	
Chris Ricardi	QA Officer	MACTEC	207-775-5401	
Garland Hillard	Olin Site Team Leader	Olin Corporation	423-336-4479	
Steve Morrow	Olin Site Manger	Olin Corporation	423-336-4511	
Wes Kelman		EPA	617-918-1540	
Tom Murphy		NETransrail	781-844-2795	
Richard Sugatt		USEPA	617-918-1415	
Daryl Luce		USEPA	617-918-1336	
Diane Silverman		M&E	781-224-6233	
Martha Zinbel		M&E	781-224-6168	
Lourie O'Connor		M&E	781-224-6431	
Warren Diesl		M&E	871-224-6182	

Jim DiLorenzo	Remediation Project Manager	EPA	617-918-1247	
Meeting Purpose: Discuss site history and the transition to CERCLA				
Comments and Action Items: Discussed historic investigative activities. Olin presented a Site description overview, contamination assessment information and historic remedial actions, and a summary of the historic analytical chemistry programs and validation processes.				

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #8a
Project Scoping Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program:	**Site Location**: 51 Eames, Wilmington, MA
Projected Dates of Sampling: TBD 2009	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
	Other Site Number/Code: N/A
	Phase: ERA SA/SI Pre-RI RI (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:

Date of Meeting: January 17, 2008
Meeting Location: Olin Wilmington Superfund Site, Eames Street, Wilmington, MA.

Name	Project Role	Affiliation	Phone #	e-Mail Address
Peter Thompson	Project Manager	MACTEC	207-775-5401	
Mike Murphy	Principal Scientist	MACTEC	781-213-5600	
Mirette Habib		MACTEC	781-213-5605	
Garland Hillard	Olin Site Team Leader	Olin Corporation	423-336-4479	
Steve Morrow	Olin Site Manager	Olin Corporation	423-336-4511	
James Cashwell		Olin Corporation	423-336-4012	
Richard Willey		USEPA	617-918-1266	
Heather Ford		Nobis	978-722-1013	
Boyd Alloy		Nobis	978-722-1006	
Joe Coyne		MassDEP	617-348-4066	
Marc Bouvier		Nobis	978-722-1018	
Jim DiLorenzo	Remediation Project Manager	EPA	617-918-1247	

Meeting Purpose: EPA presentation of comments on Draft Focused RI Report.

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #8a
Project Scoping Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program:	**Site Location**: 51 Eames, Wilmington, MA
Projected Dates of Sampling: TBD 2009	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
	Other Site Number/Code: N/A
	Phase: ERA SA/SI Pre-RI RI (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:

Date of Meeting: March 18, 2008
Meeting Location: Olin Wilmington Superfund Site, Eames Street, Wilmington, MA.

Name	Project Role	Affiliation	Phone #	e-Mail Address
Peter Thompson	Project Manager	MACTEC	207-775-5401	
Mike Murphy	Principal Scientist	MACTEC	781-213-5600	
Richard Sugatt		USEPA	617-918-1415	
Garland Hillard	Olin Site Team Leader	Olin Corporation	423-336-4479	
Steve Morrow	Olin Site Manager	Olin Corporation	423-336-4511	
James Cashwell		Olin Corporation	423-336-4012	
Richard Willey		USEPA	617-918-1266	
Heather Ford		Nobis	978-722-1013	
Cynthia Woods		Avatar	978-722-1349	
Joe Coyne		MassDEP	617-348-4066	
Marc Bouvier		Nobis	978-722-1018	
Jim DiLorenzo	Remediation Project Manager	EPA	617-918-1247	

Meeting Purpose: RI scoping meeting #1; EPA data requests for OU1, OU2, and OU3

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #8a
Project Scoping Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program:	**Site Location**: 51 Eames, Wilmington, MA
Projected Dates of Sampling: TBD 2009	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
	Other Site Number/Code: N/A
	Phase: ERA SA/SI Pre-RI RI (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:

Date of Meeting: July 17, 2008
Meeting Location: Olin Wilmington Superfund Site, Eames Street, Wilmington, MA.

Name	Project Role	Affiliation	Phone #	e-Mail Address
Peter Thompson	Project Manager	MACTEC	207-775-5401	
Mike Murphy	Principal Scientist	MACTEC	781-213-5600	
Richard Sugatt		USEPA	617-918-1415	
Garland Hillard	Olin Site Team Leader	Olin Corporation	423-336-4479	
Boyd Allen		Nobis	978-722-1018	
Gail DeRuzzo		Nobis	978-722-1331	
Heather Ford		Nobis	978-722-1013	
Cynthia Woods		Avatar	978-722-1349	
Marc Bouvier		Nobis	978-722-1018	
Jim DiLorenzo	Remediation Project Manager	EPA	617-918-1247	

Meeting Purpose: RI scoping meeting #2

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #8a
Project Scoping Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program: N/A	**Site Location**: 51 Eames, Wilmington, MA
Project Date(s) of Sampling: N/A	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
Meetings held from July 2007 through November 2007 are documented the Semi-Annual Status Report No 1. (SASR) submitted to USEPA Region 1 on January 2, 2008.	**Other Site Number/Code**: N/A
	Phase: ERA SA/SI Pre-RI ~~RI~~ (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:
Date of Meetings: July 2007 through November 7, 2007	
Meeting Purpose: Various	
Comments and Action Items: Olin has been involved in several meetings since July 2007. On August 20, 2007, and October 11, 2007, Olin participated in the Technical Assistance Grant (TAG) meetings with USEPA. Representatives of Olin also participated in the public meeting, held on September 5, 2007, which updated residents on the status of the remedial investigation at the site. On October 3, 2007, Olin met with USEPA to discuss preliminary comments of the IRSWP. On October 10, 2007, Olin hosted a stakeholders meeting to review the organization and scope of the recently submitted Draft Focused RI Report. Then on November 7, 2007, Olin met with EPA to present a web-based tool for USEPA to use for accessing data from the Olin Wilmington database.	

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #8a
Project Scoping Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program: N/A	**Site Location**: 51 Eames, Wilmington, MA
Project Date(s) of Sampling: N/A	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
Meetings held from December 2007 through May 2008 are documented the Semi-Annual Status Report No 2. (SASR) submitted to USEPA Region 1 on June 27, 2008.	**Other Site Number/Code**: N/A
	Phase: ERA SA/SI Pre-RI ~~RI~~ (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:
Date of Meetings: December 2007 through May 2008	
Meeting Purpose: Various	
Comments and Action Items: Olin, USEPA and their consultants met to review USEPA comments and questions concerning the Draft Focused RI Report. At conclusion of this meeting, Olin and USEPA had a discussion pertaining to the DAPL Pilot Study design.	

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #8b
Draft RI/FS Work Plan Review Meeting Attendance Sheet

EPA Regulation Program: RCRA FIFRA TSCA CERCLA DW CWA CAA (underline one)	**Site Name**: Olin Wilmington Superfund Site
Program: N/A	**Site Location**: 51 Eames, Wilmington, MA
Project Date(s) of Sampling:N/A	**CERCLA Site/Spill Identifier No.**: MAD001403104
Project Manager: Peter Thompson	**Operable Unit**: N/A
Meetings held from December 2007 through March 2009 are documented the Semi-Annual Status Reports No 2, 3(SASR) submitted to USEPA Region 1 in June, 2008, and .January 2009. Next SASR due in July 2009.	**Other Site Number/Code**: N/A
	Phase: ERA SA/SI Pre-RI ~~RI~~ (phase I, etc.) FS RD RA post-RA (underline one)
	Other phase:
Date of Meetings: March 17, 2009 Attended by Olin Corporation, USEPA, Nobis Engineering, and MACTEC Consulting and Engineering, Inc.	
Meeting Purpose: Review EPA Comments on Draft RI/FS Work Plan	
Comments and Action Items: Olin, USEPA and their consultants met to review USEPA comments and questions concerning the Draft RI/FS Work Plan. The Meeting was followed by an April 15, 2009 conference call with the same parties to discuss proposed approach by Olin to satisfy Work Plan deficiencies identified by USEPA..	

6.0 PROJECT DESCRIPTION AND SCHEDULE

This section provides a general overview of the activities that will be performed and how and when they will be performed based on site background information, site background data, preplanning site visits, and scoping meetings. A summary of these activities is presented on Worksheet #9a, which follows. Specific details for individual project activities will be discussed in later sections of the QAPP. Additional details are presented in the companion Field Sampling Plan for this project.

6.1 PROJECT OVERVIEW

The primary objectives of this investigation are to provide current data to confirm prior results and support human health and ecological risk assessments. The data will be used to support a feasibility study to identify remedies to address risks the Site may pose to human health and the environment. The objective of the RI/FS is to develop a well-supported Proposed Plan (PP) and Record of Decision (ROD) for the Site. These objectives will be satisfied by the sampling and analysis program outlined in Worksheets #9b and #9c.

6.1.1 Sampling Tasks

The sampling tasks are described in detail in the companion FSP. The FSP includes sampling of surface water, surface soil, subsurface soil, sediment, and ground water. Sampling is expected to occur beginning in 2009.

MACTEC and Geomega will conduct the field work in accordance with relevant guidance used by EPA in conducting RI/FS activities. The specific objectives of the project are listed below.

- Confirm prior environmental data;
- Confirm the sources, nature, extent, and distribution of contaminants released;
- Determine and quantify potential exposure pathways;
- Provide sufficient information to assess the current and future potential risks to human health and the environment; and
- Provide sufficient information to evaluate the remedial alternatives, design remedial actions, select a remedy and issue a ROD for the Site.

6.1.2 Analytical Tasks

The contaminants of potential concern (COPCs) and other target analytes have been identified based on the historical data. Worksheet #9b summarizes the COPCs, other target analytes, method detection limits (MDLs), quantitation limits (QLs), and project action limits (PALs) for the program. Worksheet #9c summarizes the analytical parameters associated with each analysis, the concentration level and field QC samples required. A summary of analytical methods and media is presented on Table 6.1.

The goal of this investigation will be to obtain high quality data for groundwater, surface water, soil, and sediment matrices and to obtain MDLs/QLs that are routinely used for CERCLA investigations and support comparisons to guidance values and standards PALs that are identified as applicable site investigation objectives to the extent that is analytically feasible. For a subset of parameters and analytes, PALs concentrations are lower than MDLs/QLs that can be achieved using available analytical methods. These analytes are identified on Worksheet #9b. The analytical approach proposed in this QAPP is interpreted to be the best technical approach to obtain data on a wide variety of parameters identified for the RI while providing low detection limits for use in contamination and risk assessments.

This QAPP includes proposed testing for a variety of parameters using analytical methodologies that are not routinely included in CERCLA investigations. Analytical methods for a subset of parameters have been developed as performance base procedures by subcontracted analytical laboratories. These methods are described in detail in the QAPP worksheets and SOPs included as Appendix B.

Analytical procedures for indoor air sampling to generate data used to evaluate vapor intrusion may be added to the RI investigation pending results of soil and groundwater testing as described in the FSP. If vapor intrusion sampling is included in this investigation, analytical procedures for air will be added to this QAPP as an addendum. Analytical procedures will be determined based on compound lists identified during the review of soil and groundwater data. Procedures for air sampling and analytical testing will be developed based on project data needs and a review of methods that are commercially available at subcontract laboratories.

6.2 PROJECT SCHEDULE

An overview of the proposed schedule is shown in Worksheet #10. The expanded project schedule is attached to the Project Operation Plan cover letter.

Table 6.1 - Summary of Analytical Methods and Media

Analytical Parameter	Analytical Method	Groundwater	Surface Water	Soil	Sediment
VOCs	SW-846 8260B	X	X	X	X
SVOC	SW-846 8270C – Low Level	X	X	X	X
Pesticides	SW-846 8081	X	X		
PCBs	SW-846 8082	X	X	X	
Herbicides	SW-846 8151A	X	X		
EPH	MADEP EPH-98-1	X	X	X	X
VPH	MADEP VPH-98-1	X	X	X	X
TAL Metals	SW-846 6010B (ICP) / 6020A (ICP-MS)/ Hg by 7470A/7471	X	X	X	X
Hexavalent Chromium	SW-846 7199 (soil/sediment prep method 3060A)	X	X	X	X
Alkalinity	SM 2320B	X	X		
Ammonia	QuikChem: LACHAT 10-107-06-1-A	X	X	X	X
Chemical Oxygen Demand	EPA 410.4	X	X		
Bromide	EPA 300	X	X		
Chloride	EPA 300	X	X	X	X
Fluoride	EPA 300	X	X		
Nitrate and Nitrite	EPA 300	X	X		
Sulfate	EPA 300	X	X	X	X
Hardness	SM 2340B	X	X		
pH	SM 4500	X	X		
Specific Conductance	SM 2510B	X	X		
Total Dissolved Solids (TDS)	SM 2540C	X	X		
Total Organic Carbon	SM 5310B (aqueous) / Lloyd Kahn (sediment, soil)	X	X	X	X
Total Suspended Solids (TSS)	SM 2540D	X	X		
Formaldehyde, Acetaldehyde	SW-846 8315	X	X	X	X
Hydrazine, MMH and UDMH	Modified 8315 LC/MS/MS	X	X	X	X
phthalic anhydride (acid)	Modified Method 8000 - LC	X	X	X	X
N,N-dimethylformamide (DMF)	Modified 8033 - GC/NPD	X	X	X	X
NDMA	Modified 8270C low level	X	X	X	X
NDMA / NDPA	Modified EPA 521	X	X		
Nonylphenol (alkylphenols)	Modified 8270 SIM	X	X		
Opex and Kempore	Lab Specific HPLC method and SW-846 8000B	X	X		
Perchlorate	SW-846 6850	X			

EPA-NE QAPP Worksheet #9a

Project Description

Sampling Tasks: See the Field Sampling Plan and Section 6.1.1. For a discussion of field procedures, refer to Section 9.0.

Analysis Tasks: See Section 6.1.2. For a discussion of analytical procedures, refer to Sections 11 and 12.

Quality Control Tasks: For a detailed discussion, refer to Section 13.

Secondary Data: For a detailed discussion, refer to Section 14.

Data Management Tasks: For a detailed discussion, refer to Section 15.

Documentation and Records: For a detailed discussion, refer to Section 15.

Data Packages: For a detailed discussion, refer to Section 15.

Assessment/Audit Tasks: A minimum of one field audit is scheduled for this investigation. An audit of the analytical lab is not scheduled. The primary laboratory, TAL - Westfield, is accredited through the National Environmental Laboratory Accreditation Program (NELAP) which included QA Program and procedure reviews as discussed in Section 12. A project-specific laboratory audit will be completed only if corrective action measures are identified that are determined to be critical to the project analytical program. Audits will be performed as described in Section 16.

Data Verification and Validation Tasks: Data for VOCs, SVOCs, pesticides, PCBs, and metals will undergo a 90 Tier II, 10 percent Tier III validation in accordance with the EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, December 1996. Additional data validation procedures are described for other project methods. For a detailed discussion, refer to Sections 18 and 19.

Data Usability Assessment Tasks: Each of the Project Quality Objectives listed in Worksheet 11 will be examined to determine if the objective was met. The examination will include a review of both laboratory and field data. Each analysis will be evaluated separately in terms of the major impacts observed from the Data Validation, Data Quality Indicators (PARCCS), and measurement performance criteria assessments. For a detailed discussion, refer to Section 20.

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater *(GW)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
71-55-6	1,1,1-Trichloroethane	200	20000	1	0.7	1	NA	NA
79-34-5	1,1,2,2-Tetrachloroethane	0.067	50000	0.5	0.21	0.5	NA	NA
630-20-6	1,1,1,2-Tetrachloroethane	0.52	50000	0.5	0.21	0.5	NA	NA
76-13-1	1,1,2-Trichloro-1,2,2-Trifluoroethane	1500	NS	1	0.63	1	NA	NA
79-00-5	1,1,2-Trichloroethane	0.24	50000	1	0.72	1	NA	NA
75-34-3	1,1-Dichloroethane	2.4	20000	1	0.67	1	NA	NA
75-35-4	1,1-Dichloroethene	7	30000	1	0.64	1	NA	NA
563-58-6	1,1-Dichloropropene*	NS	NS	1	0.68	1	NA	NA
87-61-6	1,2,3-Trichlorobenzene	NS	NS	1	0.42	1	NA	NA
96-18-4	1,2,3-Trichloropropane	0.0096	NS	1	0.65	1	NA	NA
95-63-6	1,2,4-Trimethylbenzene	15	NS	1	0.099	1	NA	NA
120-82-1	1,2,4-Trichlorobenzene	8.2	50000	1	0.34	1	NA	NA
96-12-8	1,2-Dibromo-3-chloropropane*	0.00032	NS	5	0.58	5	NA	NA
106-93-4	1,2-Dibromoethane	0.0065	50000	1	0.54	1	NA	NA
95-50-1	1,2-Dichlorobenzene	370	2000	1	0.62	1	NA	NA
107-06-2	1,2-Dichloroethane	0.15	20000	1	0.62	1	NA	NA
78-87-5	1,2-Dichloropropane	0.39	50000	1	0.51	1	NA	NA
108-67-8	1,3,5-Trimethylbenzene*	12	NS	1	0.66	1	NA	NA
541-73-1	1,3-Dichlorobenzene	40	50000	1	0.64	1	NA	NA
142-28-9	1,3-Dichloropropane*	730	NS	1	0.57	1	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater *(GW)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
106-46-7	1,4-Dichlorobenzene	0.43	8000	1	0.62	1	NA	NA
123-91-1	1,4-Dioxane*	3	NS	50	7.7	50	NA	NA
594-20-7	2,2-Dichloropropane*	NS	NS	1	0.6	1	NA	NA
107-39-1	2,4,4-Trimethyl-1-pentene	NS	NS	1	0.81	1	NA	NA
107-40-4	2,4,4-Trimethyl-2-Pentene	NS	NS	1	0.5	1	NA	NA
95-49-8	2-Chlorotoluene*	730	NS	1	0.65	1	NA	NA
591-78-6	2-Hexanone*	NS	NS	10	7.29	10	NA	NA
106-43-4	4-Chlorotoluene*	2600	NS	1	0.62	1	NA	NA
67-64-1	Acetone	3000	50000	50	20	50	NA	NA
71-43-2	Benzene	0.41	10000	1	0.74	1	NA	NA
108-86-1	Bromobenzene*	20	NS	1	0.64	1	NA	NA
74-97-5	Bromochloromethane	NS	NS	1	0.78	1	NA	NA
75-27-4	Bromodichloromethane	1.2	50000	1	0.63	1	NA	NA
75-25-2	Bromoform	0.0083	50000	1	0.6	1	NA	NA
74-83-9	Bromomethane	2	50000	2	0.28	2	NA	NA
104-51-8	Butylbenzene*	260	NS	1	0.59	1	NA	NA
75-15-0	Carbon Disulfide	560	NS	10	0.62	10	NA	NA
56-23-5	Carbon Tetrachloride	0.13	5000	1	0.71	1	NA	NA
108-90-7	Chlorobenzene	91	1000	1	0.65	1	NA	NA
75-00-3	Chloroethane	2100	NS	2	0.405	2	NA	NA
67-66-3	Chloroform	0.19	10000	1	0.66	1	NA	NA
74-87-3	Chloromethane	6.7	NS	2	0.30	2	NA	NA
156-59-2	Cis-1,2-Dichloroethene	70	50000	1	0.65	1	NA	NA
10061-01-5	cis-1,3-Dichloropropene	NS	NS	0.4	0.4	0.4	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater *(GW)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
110-82-7	Cyclohexane	13000	NS	10	TBD	10	NA	NA
124-48-1	Dibromochloromethane	0.15	50000	0.5	0.5	0.5	NA	NA
74-95-3	Dibromomethane*	370	NS	1	0.65	1	NA	NA
75-71-8	Dichlorodifluoromethane	14	NS	1	0.24	1	NA	NA
60-29-7	Diethyl ether*	520	NS	10	0.63	10	NA	NA
108-20-3	Diisopropyl Ether*	830	NS	10	0.62	10	NA	NA
637-92-3	Ethyl Tertiary Butyl Ether*	NS	NS	5	0.67	5	NA	NA
100-41-4	Ethylbenzene	1.5	4000	1	0.67	1	NA	NA
87-68-3	Hexachlorobutadiene*	0.6	3000	0.4	0.4	0.4	NA	NA
98-82-8	Isopropylbenzene	8.4	NS	1	0.57	1	NA	NA
79-20-9	Methyl acetate	37000	NS	10	TBD	10	NA	NA
78-93-3	Methyl Ethyl Ketone*	350	50000	10	10	10	NA	NA
108-10-1	Methyl Isobutyl Ketone*	350	50000	10	8.47	10	NA	NA
1634-04-4	Methyl tert-butylether	12	50000	1	0.63	1	NA	NA
108-87-2	Methylcyclohexane	710	NS	10	TBD	10	NA	NA
75-09-2	Methylene Chloride	4.8	50000	2	1	2	NA	NA
108-38-3	m-Xylene*	1400	500	2	1.35	2	NA	NA
91-20-3	Naphthalene*	0.14	20000	5	2	5	NA	NA
103-65-1	n-propylbenzene*	320	NS	1	0.7	1	NA	NA
95-47-6	o-Xylene*	1400	500	1	0.54	1	NA	NA
99-87-6	p-Isopropyltoluene*	NS	NS	1	0.59	1	NA	NA
106-42-3	p-Xylene*	1500	500	2	1.35	2	NA	NA
135-98-8	Sec-butylbenzene*	250	NS	1	0.67	1	NA	NA
100-42-5	Styrene	100	6000	1	0.6	1	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater *(GW)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
994-05-8	Tert-amyl Methyl Ether*	90	NS	5	0.61	5	NA	NA
98-06-6	Tert-butylbenzene*	290	NS	1	0.61	1	NA	NA
127-18-4	Tetrachloroethene	0.11	30000	1	0.48	1	NA	NA
109-99-9	Tetrahydrofuran*	1300	NS	10	8.03	10	NA	NA
108-88-3	Toluene	1000	4000	1	0.56	1	NA	NA
156-60-5	Trans-1,2-Dichloroethene	90	50000	1	0.6	1	NA	NA
10061-02-6	trans-1,3-Dichloropropene	NS	NS	0.4	0.4	0.4	NA	NA
79-01-6	Trichloroethene	1.7	5000	1	0.59	1	NA	NA
75-69-4	Trichlorofluoromethane	180	NS	1	0.31	1	NA	NA
75-01-4	Vinyl Chloride	0.016	50000	0.5	0.46	0.5	NA	NA
1330-20-7	Xylenes (total)	200	500	2	1.35	2	NA	NA

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8260B list. Compound is on Method 8260B list for the MCP.
Tentatively Identified Compouns (TICs) will be reported for 8260B analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.
USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Secondary Maximum contaminant Levels. Spring.

Prepared by: BR
Checked by: TC
8/7/09

MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low/medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit (PAL)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
90-12-0	1-Methylnaphthalene	2.3	NS	TBD	TBD	TBD	NA	NA
92-52-4	1,1-Biphenyl	0.9	50000	5.0	TBD	5.0	NA	NA
95-94-3	1,2,4,5-Tetrechlorobenzene	11	NS	5.0	0.16	5.0	NA	NA
120-82-1	1,2,4-Trichlorobenzene*■	8.2	50000	5.0	0.307	5.0	NA	NA
95-50-1	1,2-Dichlorobenzene*■	370	2000	5.0	0.291	5.0	NA	NA
541-73-1	1,3-Dichlorobenzene*■	40	50000	5.0	0.252	5.0	NA	NA
106-46-7	1,4-Dichlorobenzene*■	0.43	8000	5.0	0.286	5.0	NA	NA
58-90-2	2,3,4,5-Tetrachlorophenol	1100	NS	20	TBD	20	NA	NA
95-95-4	2,4,5-Trichlorophenol	200	3000	5.0	0.261	5.0	NA	NA
88-06-2	2,4,6-Trichlorophenol	6.1	500	5.0	0.311	5.0	NA	NA
120-83-2	2,4-Dichlorophenol	10	2000	5.0	0.299	5.0	NA	NA
105-67-9	2,4-Dimethylphenol	60	50000	5.0	0.395	5.0	NA	NA
51-28-5	2,4-Dinitrophenol	73	20000	5.0	0.083	5.0	NA	NA
121-14-2	2,4-Dinitrotoluene	0.22	50000	5.0	0.238	5.0	NA	NA
606-20-2	2,6-Dinitrotoluene	37	NS	5.0	0.280	5.0	NA	NA
91-58-7	2-Chloronaphthalene	2900	NS	5.0	0.331	5.0	NA	NA
95-57-8	2-Chlorophenol	10	40000	5.0	0.234	5.0	NA	NA
91-57-6	2-Methylnaphthalene	10	3000	1.0	0.328	1.0	NA	NA
95-48-7	2-Methylphenol	1800	NS	5.0	0.195	5.0	NA	NA
88-74-4	2-Nitroaniline	110	NS	5.0	0.267	5.0	NA	NA
88-75-5	2-Nitrophenol	NS	NS	5.0	0.224	5.0	NA	NA
91-94-1	3,3'-Dichlorobenzidine	0.15	2000	5.0	0.174	5.0	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low/medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit (PAL)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
15831-10-4	3&4-Methylphenol*	NS	NS	5.0	0.152	5.0	NA	NA
99-09-2	3-Nitroaniline	NS	NS	5.0	0.169	5.0	NA	NA
534-52-1	4,6-Dinitro-2-Methylphenol	3.7	NS	5.0	0.250	5.0	NA	NA
101-55-3	4-Bromophenyl-phenylether	NS	NS	5.0	0.348	5.0	NA	NA
59-50-7	4-Chloro-3-Methylphenol	NS	NS	5.0	0.410	5.0	NA	NA
106-47-8	4-Chloroaniline	0.34	300	5.0	0.463	5.0	NA	NA
7005-72-3	4-Chlorophenyl-phenylether	NS	NS	5.0	0.363	5.0	NA	NA
100-01-6	4-Nitroaniline	3.4	NS	5.0	0.223	5.0	NA	NA
100-02-7	4-Nitrophenol	NS	NS	5.0	0.054	5.0	NA	NA
83-32-9	Acenaphthene	20	5000	1.0	0.332	1.0	NA	NA
208-96-8	Acenaphthylene	300	3000	0.3	0.284	0.3	NA	NA
98-86-2	Acetophenone	3700	NS	5.0	0.362	5.0	NA	NA
62-53-3	Aniline*	12	NS	50	0.191	50	NA	NA
120-12-7	Anthracene	2000	3000	1.0	0.357	1.0	NA	NA
1912-24-9	Atrazine	0.29	NS	5.0	TBD	5.0	NA	NA
103-33-3	Azobenzene*	0.12	NS	5.0	0.18	5.0	NA	NA
100-52-7	Benzaldehyde	3700	NS	5.0	TBD	5.0	NA	NA
56-55-3	Benzo(a)anthracene	0.029	1000	0.30	0.3	0.30	NA	NA
50-32-8	Benzo(a)pyrene	0.0029	500	0.20	0.2	0.20	NA	NA
205-99-2	Benzo(b)fluoranthene	0.029	400	0.30	0.253	0.30	NA	NA
191-24-2	Benzo(g,h,i)perylene	300	3000	0.5	0.215	0.5	NA	NA
207-08-9	Benzo(k)fluoranthene	0.29	100	0.3	0.128	.30	NA	NA
65-85-0	Benzoic acid	150000	NS	5.0	0.136	5.0	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low/medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit (PAL)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
119-61-9	Benzophenone	NS	NS	TBD	TBD	TBD	NA	NA
100-51-6	Benzyl alcohol	18000	NS	10	0.119	10	NA	NA
39638-32-9	Bis (2-Chloroisopropyl) ether*	30	50000	5.0	0.26	5.0	NA	NA
111-91-1	bis(2-Chloroethoxy)methane	110	NS	5.0	0.331	5.0	NA	NA
111-44-4	bis(2-Chloroethyl) ether	0.012	50000	5.0	0.225	5.0	NA	NA
117-81-7	Bis(2-Ethylhexyl) phthalate	4.8	30	2.0	0.414	2.0	NA	NA
85-68-7	Butylbenzylphthalate	35	NS	5.0	0.530	5.0	NA	NA
105-60-2	Caprolactam	18000	NS	5.0	TBD	5.0	NA	NA
86-74-8	Carbazole	NS	NS	5.0	TBD	5.0	NA	NA
218-01-9	Chrysene	2	3000	1.0	0.349	1.0	NA	NA
53-70-3	Dibenz(a,h)anthracene	0.0029	40	0.5	0.169	0.5	NA	NA
132-64-9	Dibenzofuran	NS	NS	5.0	0.341	5.0	NA	NA
84-66-2	Diethylphthalate	2000	9000	5.0	0.386	5.0	NA	NA
131-11-3	Dimethylphthalate	30000	50000	5.0	0.370	5.0	NA	NA
84-74-2	Di-n-butylphthalate	3700	NS	5.0	0.711	5.0	NA	NA
101-84-8	Diphenyl oxide	NS	NS	TBD	TBD	TBD	NA	NA
117-84-0	Di-n-octylphthalate	NS	NS	5.0	0.22	5.0	NA	NA
206-44-0	Fluoranthene	90	200	1.0	0.416	1.0	NA	NA
86-73-7	Fluorene	300	3000	1.0	0.348	1.0	NA	NA
118-74-1	Hexachlorobenzene	0.042	6000	1.0	0.330	1.0	NA	NA
87-68-3	Hexachlorobutadiene**■	0.33	3000	0.60	0.358	0.60	NA	NA
77-47-4	Hexachlorocyclopentadiene	0.18	NS	5.0	0.513	5.0	NA	NA
67-72-1	Hexachloroethane	3.8	50000	3.0	0.269	3.0	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low/medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit (PAL)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
193-39-5	Indeno(1,2,3-cd)pyrene	0.029	100	0.5	0.206	0.5	NA	NA
79-59-1	Isophorone	NS	NS	5.0	0.351	5.0	NA	NA
91-20-3	Naphthalene■	0.14	20000	1.0	0.340	1.0	NA	NA
98-95-3	Nitrobenzene	0.12	NS	5.0	0.362	5.0	NA	NA
621-64-7	N-Nitroso-di-n-propylamine	0.0096	NS	5.0	0.351	5.0	NA	NA
122-39-4	Diphenylamine∆	910	NS	TBD	TBD	TBD	NA	NA
86-30-6	N-nitrosodiphenylamine	14	NS	5.0	0.385	5.0	NA	NA
87-86-5	Pentachlorophenol	0.56	200	1.0	0.172	1.0	NA	NA
85-01-8	Phenanthrene	300	50	0.20	0.2	0.20	NA	NA
108-95-2	Phenol	900	2000	5.0	0.075	5.0	NA	NA
129-00-0	Pyrene	80	20	5.0	0.580	5.0	NA	NA

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8270C list. Compound is on Method 8270C list for the MCP.
■ - Compound results will be reported from 8260B VOC analysis if available.
∆ An additional Florisil clean-up step (Method 3620B) to separate N-nitrosodiphenylamine from Diphenylamine will be performed by the laboratory on a sub-set of samples to provide information of compound identification.
Tentatively Identified Compouns (TICs) will be reported for 8270C analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.

Prepared by: BR
Checked by: TC
8/7/09

USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Secondary Maximum Contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

EPA-NE QAPP Worksheet #9b- Method 8081
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: *Pesticides*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8081, L-29

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
72-54-8	4,4`-DDD	0.2	50	0.1	0.015	0.1	NA	NA
72-55-9	4,4`-DDE	0.09	400	0.05	0.013	0.05	NA	NA
50-29-3	4,4`-DDT	0.2	1	0.1	0.013	0.1	NA	NA
309-00-2	Aldrin	0.004 (**)	20	0.1	0.015	0.1	NA	NA
319-84-6	Alpha-BHC	0.011 (**)	NS	0.1	0.011	0.1	NA	NA
5103-71-9	Alpha-Chlordane	NS	NS	0.1	0.015	0.1	NA	NA
319-85-7	Beta-BHC	0.037 (**)	NS	0.1	0.014	0.1	NA	NA
319-86-8	Delta-BHC	NS	NS	0.1	0.013	0.1	NA	NA
60-57-1	Dieldrin	0.0042 (**)	0.5	0.1	0.021	0.1	NA	NA
959-98-8	Endosulfan I	NS	NS	0.1	0.015	0.1	NA	NA
33213-65-9	Endosulfan II	NS	NS	0.1	0.024	0.1	NA	NA
1031-07-8	Endosulfan sulfate	NS	NS	0.1	0.01	0.1	NA	NA
72-20-8	Endrin	2	5	0.1	0.023	0.1	NA	NA
7421-93-4	Endrin aldehyde	NS	NS	0.1	0.02	0.1	NA	NA
53494-70-5	Endrin ketone	NS	NS	0.1	0.012	0.1	NA	NA
58-89-9	Gamma-BHC/Lindane	0.061 (**)	4	0.1	0.011	0.1	NA	NA
5103-74-2	Gamma-Chlordane	NS	NS	0.1	0.033	0.1	NA	NA
76-44-8	Heptachlor	0.015 (**)	1	0.1	0.011	0.1	NA	NA
1024-57-3	Heptachlor epoxide	0.0074 (**)	2	0.1	0.011	0.1	NA	NA
72-43-5	Methoxychlor	40	10	0.1	0.018	0.1	NA	NA
8001-35-2	Toxaphene	0.061 (**)	NS	0.5	0.177	0.5	NA	NA

Notes:

Prepared by: BR
Checked by: TC
10/14/08

Compound list includes CLP Target Compound List
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.
USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Secondary Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

EPA-NE QAPP Worksheet #9b- Method 8082
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: *PCBs*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8082, L-28

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
12674-11-2	Aroclor 1016	0.5	10	0.3	0.11	0.3	NA	NA
11104-28-2	Aroclor 1221	0.0068 (**)	10	0.3	0.3	0.3	NA	NA
11141-16-5	Aroclor 1232	0.0068 (**)	10	0.3	0.1	0.3	NA	NA
53469-21-9	Aroclor 1242	0.034 (**)	10	0.3	0.14	0.3	NA	NA
12672-29-6	Aroclor 1248	0.034 (**)	10	0.3	0.11	0.3	NA	NA
11097-69-1	Aroclor 1254	0.034 (**)	10	0.3	0.08	0.3	NA	NA
11096-82-5	Aroclor 1260	0.034 (**)	10	0.3	0.09	0.3	NA	NA
37324-23-5	Aroclor 1262	0.5	10	0.3	0.1	0.3	NA	NA
11100-14-4	Aroclor 1268	0.5	10	0.3	0.05	0.3	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards*. Office of Water. EPA 816-F-03-016. June.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Secondary Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b – Method 8151A
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: Herbicides
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Method 8151A, L-33

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L) [1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
93-76-5	2,4,5-T	370	NS	1	0.09	1	NA	NA
93-72-1	2,4,5-TP (Silvex)	50	NS	1	0.087	1	NA	NA
94-75-7	2,4-D	70	NS	1	0.088	1	NA	NA
94-82-6	2,4-DB	290	NS	1	0.291	1	NA	NA
75-99-0	Dalapon	200	NS	4	0.228	4	NA	NA
1918-00-9	Dicamba	110	NS	1	0.096	1	NA	NA
120-36-5	Dichlorprop	NS	NS	8	0.147	8	NA	NA
88-85-7	Dinoseb	7	NS	1	0.2	1	NA	NA
94-74-6	MCPA	18	NS	100	7.65	100	NA	NA
93-65-2	MCPP	37	NS	100	18.8	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method MADEP EPH
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Extractable Petroleum Hydrocarbons (*EPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP EPH, L-25

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
NA	C11-C22 Aromatics	200	30000	100	100	100	NA	NA
NA	C11-C22 Aromatics Unadjusted	200	30000	100	NA	100	NA	NA
NA	C19-C36 Aliphatics	5000	20000	100	100	100	NA	NA
NA	C9-C18 Aliphatics	700	20000	100	100	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method MADEP VPH
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Volatile Petroleum Hydrocarbons (*VPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP VPH, L-26

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
NA	C5 – C8 Aliphatics Unadjusted	300	4000	100	NA	100	NA	NA
NA	C5 – C8 Aliphatics	300	4000	100	NA	100	NA	NA
NA	C9 – C10 Aromatics	200	4000	100	NA	100	NA	NA
NA	C9 – C12 Aliphatics Unadjusted	700	20000	100	NA	100	NA	NA
NA	C9 – C12 Aliphatics	700	20000	100	NA	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: *Metals*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7470A, L-1

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
7429-90-5	Aluminum	50	NS	100	2.2	100	NA	NA
7440-36-0	Antimony	6	8000	6	2.3	6	NA	NA
7440-38-2	Arsenic*	0.045	900	10	1.4	10	NA	NA
7440-39-3	Barium	2000	50000	10	0.25	10	NA	NA
7440-41-7	Beryllium	4	50	1	0.029	1	NA	NA
7440-43-9	Cadmium	5	4	1	0.18	1	NA	NA
7440-70-2	Calcium	NS	NS	400	27	400	NA	NA
7440-47-3	Chromium	100	300	5	0.17	5	NA	NA
7440-48-4	Cobalt	11	NS	10	0.59	10	NA	NA
7440-50-8	Copper	1000	NS	10	1.6	10	NA	NA
7439-89-6	Iron	300	NS	100	16	100	NA	NA
7439-92-1	Lead	15	10	5	1.06	5	NA	NA
7439-95-4	Magnesium	NS	NS	400	8.3	400	NA	NA
7439-96-5	Manganese	50	NS	10	0.33	10	NA	NA
7439-97-6	Mercury	0.57	20	0.2	0.129	0.2	NA	NA
7440-02-0	Nickel	100	200	10	1.6	10	NA	NA
7440-09-7	Potassium	NS	NS	4000	812	4000	NA	NA
7782-49-2	Selenium	50	100	10	2.2	10	NA	NA
7440-22-4	Silver	100	7	5	0.86	5	NA	NA
7440-23-5	Sodium	20000	NS	2000	65	2000	NA	NA
7440-28-0	Thallium*	2	3000	10	1.9	10	NA	NA
7440-31-5	Tin	22000	NS	50	3.9	50	NA	NA

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: *Metals*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7470A, L-1

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
7440-62-2	Vanadium	30	4000	10	0.29	10	NA	NA
7440-66-6	Zinc	5000	900	50	2.9	50	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
* - Results for arsenic and thallium will be reported using method 6020A.
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.
USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Secondary Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

Prepared by: BR
Checked by: TC
8/7/098

EPA-NE QAPP Worksheet #9b- Metals by Method 6020A
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: *Metals*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 6020A, L-1

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
7440-38-2	Arsenic (**)	0.045	900	1	0.397	1	NA	NA
7440-28-0	Thallium	2	3000	1	0.331	1	NA	NA

Notes:
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.
USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Secondary Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Hexavalent Chromium
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: *Hexavalent Chromium*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 7199, L-38

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
18540-29-9	Hexavalent Chromium	100	300	1.0	0.020	1.0	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.
USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Secondary Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water.* Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.
2 - Ecological PALs are from the following:
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Nitrate, Nitrite, Sulfate, Fluoride, Bromide, Chloride, Alkalinity, Hardness, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Ammonia, pH, Specific Conductance, Chemical Oxygen Demand (COD), Total Organic Carbon (TOC).

Medium/Matrix: Water

Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)

Analytical Parameter(s): Nitrate, Nitrite, Sulfate, Fluoride, Bromide, Chloride, Alkalinity, Hardness, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Ammonia, pH, Specific Conductance, Chemical Oxygen Demand (COD), Total Organic Carbon (TOC).

Concentration Level: *Low*

Field Analytical or Fixed Laboratory Method/SOP: Methods listed below. Refer to Worksheet #20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Analysis Method	Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte		Human Health PAL (mg/L)	Eco PAL (mg/L)	Project Quantitation Limit (mg/L)	MDL (mg/L)	QL (mg/L)	MDL (mg/L)	QL (mg/L)
16984-48-8	Fluoride	EPA 300	2000	NS	0.1	0.025	0.1	NA	NA
24959-67-9	Bromide	EPA 300	NS	NS	0.01	0.025	0.01	NA	NA
14797-55-8	Nitrate	EPA 300	10	NS	0.05	0.031	0.05	NA	NA
14797-65-0	Nitrite	EPA 300	1	NS	0.01	0.004	0.01	NA	NA
14808-79-8	Sulfate	EPA 300	250	NS	2.0	0.26	2.0	NA	NA
16887-00-6	Chloride	EPA 300	250	NS	1.0	0.44	1.0	NA	NA
E-11778	Hardness (as Calcium Carbonate)	SM 2340B	NS	NS	2.6	0.152	2.64	NA	NA
11-43-8	Alkalinity	SM 2320B	NS	NS	1.0	0.220	1.0	NA	NA
E-10173	Total Suspended Solids (TSS)	SM 2540D	NS	NS	5	3.82	5	NA	NA
E-10173	Total Dissolved Solids (TDS)	SM 2540C	500	NS	10	6.72	10	NA	NA
7664-41-7	Ammonia	QuikChem: LACHAT 10-107-06-1-A	NS	NS	0.10	0.073	0.10	NA	NA
NA	pH (standard pH units)	SM 4500	NS	NS	0.1	NA	0.1	NA	NA
NA	Specific Conductance (umhos/cm)	SM 2510B	NS	NS	1.0	0.89	1.0	NA	NA
E-10107	Chemical Oxygen Demand	EPA 410.4	NS	NS	20	2.6	20	NA	NA
E-10195	Total Organic Carbon	SM 5310B	NS	NS	1.0	0.79	1.0	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/L – milligrams per liter (parts per billion)
1 - Human Health PALs are the lowest from the following:
USEPA, 2003. *National Primary Drinking Water Standards.* Office of Water. EPA 816-F-03-016. June.

Prepared by: BR
Checked by: TC
8/7/09

MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Secondary Maximum contaminant Levels. Spring.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.

EPA-NE QAPP Worksheet #9b- Formaldehyde and Acetaldehyde
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: Formaldehyde and Acetaldehyde
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: SW-846 Method 8315, L-32

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
50-00-0	Formaldehyde	7300	NS	50	5	50	NA	NA
75-07-0	Acetaldehyde	2.2	NS	100	10	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEAP, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Hydrazine, MMH, UDMH
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: Hydrazine, Mono-methylhydrazine (MMH), Unsymmetrical dimethylhydrazine (UDMH)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Lancaster Laboratory Modified 8315, L-24

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
302-01-2	Hydrazine	0.022	NS	0.2	0.05	0.2	NA	NA
60-34-4	MMH	NS	NS	0.5	0.25	0.5	NA	NA
57-14-7	UDMH	NS	NS	0.5	0.25	0.5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Phthalic anhydride (acid)
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: Phthalic anhydride (acid)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified HPLC Method 8000, L-36

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
85-44-9	phthalic anhydride (acid)	73000	NS	10	1.4	10	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PAL is from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N,N-dimethylformamide (DMF)
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: N,N-dimethylformamide
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified Method 8033-GC/NPD, L-35

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
68-12-2	DMF	3700	NS	20	TBD	20	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PAL is from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N-nitrosodimethylamine (NDMA) and N-nitroso-di-n-propylamine (NDPA)
Medium/Matrix: water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: N-nitrosodymethylamine (NDMA)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified EPA 521 – GC/CI/MS/MS, L-5

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
62-75-9	NDMA	0.00042	NS	0.002	0.000741	0.002	NA	NA
621-64-7	NDPA	0.0096	NS	0.01	0.00066	0.01	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 = Human Health PAL is the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N-nitrosodimethylamine (NDMA)
Medium/Matrix: water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: N-nitrosodymethylamine (NDMA)
Concentration Level: Low/Medium
Field Analytical or Fixed Laboratory Method/SOP: Modified 8270C Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)*	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
62-75-9	NDMA	0.00042	NS	1	0.04	1	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
* - Modified Method 8270C Low Level analysis used for well locations with historical concentrations of NDMA greater than 1 µg/L.
1 = Human Health PAL is the lowest from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Standards and Guidelines for Contaminants in Massachusetts Drinking Water*. Massachusetts Drinking Water Guidelines. Spring.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Nonylphenols (alkylphenols)
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: Nonylphenol tech mix, Nonylphenol Diethoxylate tech mix, p-tert-Octyl Phenol, Bisphenol-A
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: STL West Sacramento, CA – SOP Alkylphenols by GC/MS-SIM, L-34

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
84852-15-3	Nonylphenol tech mix	NS	NS	5	1.6	5	NA	NA
NA	Nonylphenol Diethoxylate tech mix	NS	NS	20	1.9	20	NA	NA
140-66-9	p-tert-Octyl Phenol	NS	NS	2	TBD	2	NA	NA
80-05-7	Bisphenol-A	1800	NS	2	TBD	2	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PALs are from the following:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Opex and Kempore
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Groundwater (GW)
Analytical Parameter: Dinitrosopentamethylenetetramine (Opex) and Azodicarbonamide (Kempore)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: SW-846 Method 8000B (HPLC), L-11

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
101-25-7	Opex	NS	NS	100	20	100	NA	NA
123-77-3	Kempore	NS	NS	1000	230	1000	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- SW-846 Method 6850
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Groundwater (GW)*
Analytical Parameter: Perchlorate
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: SW-846 6850, L-37

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
CAS Number	Analyte	Project Action Limit (PAL)		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
14797-73-0	Perchlorate	2	NS	1	0.20	1	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PAL is from the lowest of the following sources:
USEPA, 2009, *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Tap Water, http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. Standards and Guidelines for Contaminants in Massachusetts Drinking Water. Massachusetts Maximum contaminant Levels. Spring.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method 8260B

Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (*SW)*
Analytical Parameter: *Volatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
71-55-6	1,1,1-Trichloroethane	NS	11	1	0.7	1	NA	NA
79-34-5	1,1,2,2-Tetrachloroethane	NS	380	0.5	0.21	0.5	NA	NA
630-20-6	1,1,1,2-Tetrachloroethane	NS	2000	1	0.21	0.5	NA	NA
76-13-1	1,1,2-Trichloro-1,2,2-Trifluoroethane	NS	NS	1	0.63	1	NA	NA
79-00-5	1,1,2-Trichloroethane	NS	500	1	0.72	1	NA	NA
75-34-3	1,1-Dichloroethane	NS	47	1	0.67	1	NA	NA
75-35-4	1,1-Dichloroethene	NS	25	1	0.64	1	NA	NA
563-58-6	1,1-Dichloropropene*	NS	NS	1	0.68	1	NA	NA
87-61-6	1,2,3-Trichlorobenzene	NS	NS	1	0.42	1	NA	NA
96-18-4	1,2,3-Trichloropropane	NS	NS	1	0.65	1	NA	NA
120-82-1	1,2,4-Trichlorobenzene	NS	30	1	0.34	1	NA	NA
95-63-6	1,2,4-Trimethylbenzene	NS	NS	1	0.099	1	NA	NA
96-12-8	1,2-Dibromo-3-chloropropane*	NS	NS	5	0.58	5	NA	NA
106-93-4	1,2-Dibromoethane	NS	9600	1	0.54	1	NA	NA
95-50-1	1,2-Dichlorobenzene	NS	14	1	0.62	1	NA	NA
107-06-2	1,2-Dichloroethane	NS	910	1	0.62	1	NA	NA
78-87-5	1,2-Dichloropropane	NS	360	1	0.51	1	NA	NA
108-67-8	1,3,5-Trimethylbenzene*	NS	NS	1	0.66	1	NA	NA
541-73-1	1,3-Dichlorobenzene	NS	38	1	0.64	1	NA	NA
142-28-9	1,3-Dichloropropane*	NS	NS	1	0.57	1	NA	NA
106-46-7	1,4-Dichlorobenzene	NS	9.4	1	0.62	1	NA	NA
123-91-1	1,4-Dioxane*	NS	22000	50	7.7	50	NA	NA
594-20-7	2,2-Dichloropropane*	NS	NS	1	0.6	1	NA	NA
107-39-1	2,4,4-Trimethyl-1-pentene	NS	NS	1	0.81	1	NA	NA
107-40-4	2,4,4-Trimethyl-2-Pentene	NS	NS	1	0.5	1	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B

Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (*SW)*
Analytical Parameter: *Volatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
95-49-8	2-Chlorotoluene*	NS	NS	1	0.65	1	NA	NA
591-78-6	2-Hexanone*	NS	99	10	7.29	10	NA	NA
106-43-4	4-Chlorotoluene*	NS	NS	1	0.62	1	NA	NA
67-64-1	Acetone	NS	1500	50	20	50	NA	NA
71-43-2	Benzene	NS	114	1	0.74	1	NA	NA
108-86-1	Bromobenzene*	NS	NS	1	0.64	1	NA	NA
74-97-5	Bromochloromethane	NS	NS	1	0.78	1	NA	NA
75-27-4	Bromodichloromethane	NS	20000	1	0.63	1	NA	NA
75-25-2	Bromoform	NS	230	1	0.6	1	NA	NA
74-83-9	Bromomethane	NS	16	2	0.275	2	NA	NA
104-51-8	Butylbenzene*	NS	NS	1	0.59	1	NA	NA
75-15-0	Carbon Disulfide	NS	0.92	10	0.62	10	NA	NA
56-23-5	Carbon Tetrachloride	NS	9.8	1	0.71	1	NA	NA
108-90-7	Chlorobenzene	NS	38	1	0.65	1	NA	NA
75-00-3	Chloroethane	NS	NS	2	0.405	2	NA	NA
67-66-3	Chloroform	NS	28	1	0.66	1	NA	NA
74-87-3	Chloromethane	NS	NS	2	0.304	2	NA	NA
156-59-2	Cis-1,2-Dichloroethene	NS	590	1	0.65	1	NA	NA
10061-01-5	cis-1,3-Dichloropropene	NS	NS	0.4	0.4	0.4	NA	NA
110-82-7	Cyclohexane	NS	NS	10 (estimated)	TBD	10 (estimated)	NA	NA
124-48-1	Dibromochloromethane	NS	3400	0.5	0.5	0.5	NA	NA
74-95-3	Dibromomethane*	NS	NS	1	0.65	1	NA	NA
75-71-8	Dichlorodifluoromethane	NS	NS	1	0.239	1	NA	NA
60-29-7	Diethyl ether*	NS	NS	10	0.63	10	NA	NA
108-20-3	Diisopropyl Ether*	NS	NS	10	0.62	10	NA	NA
637-92-3	Ethyl Tertiary Butyl Ether*	NS	NS	5	0.67	5	NA	NA
100-41-4	Ethylbenzene	NS	7.3	1	0.67	1	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B

Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (*SW)*
Analytical Parameter: *Volatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
87-68-3	Hexachlorobutadiene* (**)	NS	0.053	0.4	0.4	0.4	NA	NA
98-82-8	Isopropylbenzene	NS	NS	1	0.57	1	NA	NA
79-20-9	Methyl acetate	NS	NS	10 (estimated)	TBD	10 (estimated)	NA	NA
78-93-3	Methyl Ethyl Ketone*	NS	2200	10	10	10	NA	NA
108-10-1	Methyl Isobutyl Ketone*	NS	170	10	8.47	10	NA	NA
1634-04-4	Methyl tert-butylether	NS	100000	1	0.63	1	NA	NA
108-87-2	Methylcyclohexane	NS	NS	10 (estimated)	TBD	10 (estimated)	NA	NA
75-09-2	Methylene Chloride	NS	940	2	1	2	NA	NA
108-38-3	m-Xylene*	NS	1.8	2	1.35	2	NA	NA
91-20-3	Naphthalene*	NS	12	5	2	5	NA	NA
103-65-1	n-propylbenzene*	NS	NS	1	0.7	1	NA	NA
95-47-6	o-Xylene*	NS	NS	1	0.54	1	NA	NA
99-87-6	p-Isopropyltoluene*	NS	NS	1	0.59	1	NA	NA
106-42-3	p-Xylene*	NS	NS	2	1.35	2	NA	NA
135-98-8	Sec-butylbenzene*	NS	NS	1	0.67	1	NA	NA
100-42-5	Styrene	NS	32	1	0.6	1	NA	NA
994-05-8	Tert-amyl Methyl Ether*	NS	NS	5	0.61	5	NA	NA
98-06-6	Tert-butylbenzene*	NS	NS	1	0.61	1	NA	NA
127-18-4	Tetrachloroethene	NS	45	1	0.48	1	NA	NA
109-99-9	Tetrahydrofuran*	NS	NS	10	8.03	10	NA	NA
108-88-3	Toluene	NS	9.8	1	0.56	1	NA	NA
156-60-5	Trans-1,2-Dichloroethene	NS	970	1	0.6	1	NA	NA
10061-02-6	trans-1,3-Dichloropropene	NS	NS	0.4	0.4	0.4	NA	NA
79-01-6	Trichloroethene	NS	47	1	0.59	1	NA	NA
75-69-4	Trichlorofluoromethane	NS	NS	1	0.307	1	NA	NA
75-01-4	Vinyl Chloride	NS	930	0.5	0.462	0.5	NA	NA
1330-20-7	Xylenes (total)	NS	13	2	NA	2	NA	NA

Prepared by: BR
Checked by: TC
10/14/08

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8260B list. Compound is on Method 8260B list for the MCP.
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
Tentatively Identified Compouns (TICs) will be reported for 8260B analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: Semiv*olatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
92-52-4	1,1-Biphenyl	NS	14	5.0	TBD	5.0	NA	NA
90-12-0	1-Methylnaphthalene	NS	2.1	TBD	TBD	TBD	NA	NA
95-94-3	1,2,4,5-Tetrechlorobenzene*	NS	3	5.0	0.158	5.0	NA	NA
120-82-1	1,2,4-Trichlorobenzene*■	NS	30	5.0	0.307	5.0	NA	NA
95-50-1	1,2-Dichlorobenzene*■	NS	14	5.0	0.291	5.0	NA	NA
541-73-1	1,3-Dichlorobenzene*■	NS	38	5.0	0.252	5.0	NA	NA
106-46-7	1,4-Dichlorobenzene*■	NS	9.4	5.0	0.286	5.0	NA	NA
58-90-2	2,3,4,6-Tetrachlorophenol	NS	1.2	20	TBD	20	NA	NA
95-95-4	2,4,5-Trichlorophenol	NS	130	5.0	0.261	5.0	NA	NA
88-06-2	2,4,6-Trichlorophenol	NS	4.9	5.0	0.311	5.0	NA	NA
120-83-2	2,4-Dichlorophenol	NS	11	5.0	0.299	5.0	NA	NA
105-67-9	2,4-Dimethylphenol	NS	100	5.0	0.395	5.0	NA	NA
51-28-5	2,4-Dinitrophenol	NS	19	5.0	0.083	5.0	NA	NA
121-14-2	2,4-Dinitrotoluene	NS	44	5.0	0.238	5.0	NA	NA
606-20-2	2,6-Dinitrotoluene	NS	81	5.0	0.280	5.0	NA	NA
91-58-7	2-Chloronaphthalene	NS	0.396	5.0	0.331	5.0	NA	NA
95-57-8	2-Chlorophenol	NS	24	5.0	0.234	5.0	NA	NA
91-57-6	2-Methylnaphthalene	NS	70	1.0	0.328	1.0	NA	NA
95-48-7	2-Methylphenol	NS	13	5.0	0.195	5.0	NA	NA
88-74-4	2-Nitroaniline	NS	NS	5.0	0.267	5.0	NA	NA
88-75-5	2-Nitrophenol	NS	NS	5.0	0.224	5.0	NA	NA
91-94-1	3,3'-Dichlorobenzidine	NS	4.5	5.0	0.174	5.0	NA	NA
108-39-4	3-Methylphenol (reported as 3&4-Methylphenol)*	NS	62	5.0	0.152	5.0	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: Semiv*olatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
99-09-2	3-Nitroaniline	NS	NS	5.0	0.169	5.0	NA	NA
534-52-1	4,6-Dinitro-2-Methylphenol	NS	23	5.0	0.250	5.0	NA	NA
101-55-3	4-Bromophenyl-phenylether	NS	1.5	5.0	0.348	5.0	NA	NA
59-50-7	4-Chloro-3-Methylphenol	NS	34.8	5.0	0.410	5.0	NA	NA
106-47-8	4-Chloroaniline	NS	10	5.0	0.463	5.0	NA	NA
7005-72-3	4-Chlorophenyl-phenylether	NS	NS	5.0	0.363	5.0	NA	NA
106-44-5	4-Methylphenol (reported as 3&4-Methylphenol)	NS	25	5.0	0.152	5.0	NA	NA
100-01-6	4-Nitroaniline	NS	NS	5.0	0.223	5.0	NA	NA
100-02-7	4-Nitrophenol	NS	60	5.0	0.054	5.0	NA	NA
83-32-9	Acenaphthene	NS	23	1.0	0.332	1.0	NA	NA
208-96-8	Acenaphthylene**	NS	0.14	0.30	0.284	0.30	NA	NA
98-86-2	Acetophenone	NS	NS	5.0	0.362	5.0	NA	NA
62-53-3	Aniline*	NS	4.1	5.0	0.191	5.0	NA	NA
120-12-7	Anthracene**	NS	0.035	1.0	0.357	1.0	NA	NA
1912-24-9	Atrazine	NS	NS	5.0	TBD	5.0	NA	NA
103-33-3	Azobenzene*	NS	NS	5.0	0.18	5.0	NA	NA
100-52-7	Benzaldehyde	NS	NS	5.0	TBD	5.0	NA	NA
56-55-3	Benzo(a)anthracene**	NS	0.025	0.30	0.3	0.30	NA	NA
50-32-8	Benzo(a)pyrene**	NS	0.014	0.20	0.2	0.20	NA	NA
205-99-2	Benzo(b)fluoranthene	NS	0.42	0.30	0.253	0.30	NA	NA
191-24-2	Benzo(g,h,i)perylene**	NS	0.02	0.5	0.215	0.5	NA	NA
207-08-9	Benzo(k)fluoranthene	NS	0.14	0.30	0.128	.30	NA	NA
65-85-0	Benzoic acid	NS	42	5.0	0.136	5.0	NA	NA
119-61-9	Benzophenone	NS	NS	TBD	TBD	TBD	NA	NA
100-51-6	Benzyl alcohol	NS	8.6	10	0.119	5.0	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: Semiv*olatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
108-60-1	Bis (2-Chloroisopropyl) ether*	NS	24000	5.0	0.261	5.0	NA	NA
111-91-1	bis(2-Chloroethoxy)methane	NS	NS	5.0	0.331	5.0	NA	NA
111-44-4	bis(2-Chloroethyl) ether	NS	19000	5.0	0.225	5.0	NA	NA
117-81-7	Bis(2-Ethylhexyl) phthalate	NS	3.0	2.0	0.414	2.0	NA	NA
85-68-7	Butylbenzylphthalate	NS	19	5.0	0.530	5.0	NA	NA
105-60-2	Caprolactam	NS	NS	5.0	TBD	5.0	NA	NA
86-74-8	Carbazole	NS	NS	5.0	TBD	5.0	NA	NA
218-01-9	Chrysene**	NS	0.07	1.0	0.349	1.0	NA	NA
53-70-3	Dibenz(a,h)anthracene**	NS	0.04	0.5	0.169	0.5	NA	NA
132-64-9	Dibenzofuran	NS	3.7	5.0	0.341	5.0	NA	NA
84-66-2	Diethylphthalate	NS	110	5.0	0.386	5.0	NA	NA
131-11-3	Dimethylphthalate	NS	23000	5.0	0.370	5.0	NA	NA
84-74-2	Di-n-butylphthalate	NS	9.7	5.0	0.711	5.0	NA	NA
101-84-8	Diphenyl oxide	NS	NS	TBD	TBD	TBD	NA	NA
117-84-0	Di-n-octylphthalate	NS	30	5.0	0.224	5.0	NA	NA
206-44-0	Fluoranthene	NS	0.9	1.0	0.416	1.0	NA	NA
86-73-7	Fluorene**	NS	0.14	1.0	0.348	1.0	NA	NA
118-74-1	Hexachlorobenzene**	NS	0.0003	1.0	0.330	1.0	NA	NA
87-68-3	Hexachlorobutadiene**■	NS	0.053	0.60	0.358	0.60	NA	NA
77-47-4	Hexachlorocyclopentadiene	NS	77	5.0	0.513	5.0	NA	NA
67-72-1	Hexachloroethane	NS	8	3.0	0.269	3.0	NA	NA
193-39-5	Indeno(1,2,3-cd)pyrene**	NS	0.14	0.5	0.206	0.5	NA	NA
78-59-1	Isophorone	NS	920	5.0	0.351	5.0	NA	NA
91-20-3	Naphthalene■	NS	12	1.0	0.340	1.0	NA	NA
98-95-3	Nitrobenzene	NS	220	5.0	0.362	5.0	NA	NA
621-64-7	N-Nitroso-di-n-propylamine	NS	NS	5.0	0.351	5.0	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: Semiv*olatile Organics*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8270C Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
122-39-4	Diphenylamine Δ	NS	412	TBD	TBD	TBD	NA	NA
86-30-6	N-nitrosodiphenylamine	NS	210	5.0	0.385	5.0	NA	NA
87-86-5	Pentachlorophenol	15	4	1.0	0.172	1.0	NA	NA
85-01-8	Phenanthrene	NS	3.6	0.20	0.2	0.20	NA	NA
108-95-2	Phenol	NS	62	5.0	0.075	5.0	NA	NA
129-00-0	Pyrene**	NS	0.089	5.0	0.580	5.0	NA	NA

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8270C list. Compound is on Method 8270C list for the MCP.
** - PAL is less than the MDL/QL. No additional analysis is proposed for these analytes. See discussion in Section 7.1
■ - Compound results will be reported from 8260B VOC analysis if available.

Prepared by: BR
Checked by: TC
10/14/08

Δ An additional Florisil clean-up step (Method 3620B) to separate N-nitrosodiphenylamine from Diphenylamine will be performed by the laboratory on a sub-set of samples to provide information of compound identification.
Tentatively Identified Compouns (TICs) will be reported for 8270C analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method 8081
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: *Pesticides*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8081, L-29

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
72-54-8	4,4`-DDD**	NS	0.011	0.1	0.015	0.1	NA	NA
72-55-9	4,4`-DDE**	NS	0.00000000451	0.05	0.013	0.05	NA	NA
50-29-3	4,4`-DDT**	0.001	0.000011	0.1	0.013	0.1	NA	NA
309-00-2	Aldrin	NS	0.017	0.1	0.015	0.1	NA	NA
319-84-6	Alpha-BHC	NS	2.2	0.1	0.011	0.1	NA	NA
5103-71-9	Alpha-Chlordane	NS	NS	0.1	0.015	0.1	NA	NA
319-85-7	Beta-BHC	NS	0.495	0.1	0.014	0.1	NA	NA
319-86-8	Delta-BHC	NS	2.2	0.1	0.013	0.1	NA	NA
60-57-1	Dieldrin**	0.056	0.000071	0.1	0.021	0.1	NA	NA
959-98-8	Endosulfan I	0.056	0.051	0.1	0.015	0.1	NA	NA
33213-65-9	Endosulfan II	0.056	0.051	0.1	0.024	0.1	NA	NA
1031-07-8	Endosulfan sulfate	NS	0.051	0.1	0.01	0.1	NA	NA
72-20-8	Endrin**	0.036	0.0023	0.1	0.023	0.1	NA	NA
7421-93-4	Endrin aldehyde	NS	0.15	0.1	0.02	0.1	NA	NA
53494-70-5	Endrin ketone	NS	NS	0.1	0.012	0.1	NA	NA
58-89-9	Gamma-BHC/Lindane	NS	0.016	0.1	0.011	0.1	NA	NA
5103-74-2	Gamma-Chlordane	NS	NS	0.1	0.033	0.1	NA	NA
76-44-8	Heptachlor**	0.0038	0.0036	0.1	0.011	0.1	NA	NA
1024-57-3	Heptachlor epoxide**	0.0038	0.0036	0.1	0.011	0.1	NA	NA
72-43-5	Methoxychlor	0.03	0.019	0.1	0.018	0.1	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8081
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: *Pesticides*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8081, L-29

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
8001-35-2	Toxaphene**	0.0002	0.00014	0.5	0.177	0.5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method 8082
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: *Polychlorinated Biphenyls (PCBs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8082, L-27

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
12674-11-2	Aroclor 1016	NP	NP	0.3	0.11	0.3	NA	NA
11104-28-2	Aroclor 1221	NP	NP	0.3	0.3	0.3	NA	NA
11141-16-5	Aroclor 1232	NP	NP	0.3	0.1	0.3	NA	NA
53469-21-9	Aroclor 1242	NP	NP	0.3	0.14	0.3	NA	NA
12672-29-6	Aroclor 1248	NP	NP	0.3	0.11	0.3	NA	NA
11097-69-1	Aroclor 1254	NP	NP	0.3	0.08	0.3	NA	NA
11096-82-5	Aroclor 1260	NP	NP	0.3	0.09	0.3	NA	NA
37324-23-5	Aroclor 1262	NP	NP	0.3	0.1	0.3	NA	NA
11100-14-4	Aroclor 1268	NP	NP	0.3	0.05	0.3	NA	NA

Notes:
NP – Not Proposed NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

EPA-NE QAPP Worksheet #9b – Method 8151A
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface water (SW)
Analytical Parameter: Herbicides
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Method 8151A, L-33

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L) [1]	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
93-76-5	2,4,5-T	NS	686	1	0.09	1	NA	NA
93-72-1	2,4,5-TP (Silvex)	NS	30	1	0.087	1	NA	NA
94-75-7	2,4-D	NS	220	1	0.088	1	NA	NA
94-82-6	2,4-DB	NS	NS	1	0.291	1	NA	NA
75-99-0	Dalapon	NS	NS	4	0.228	4	NA	NA
1918-00-9	Dicamba	NS	NS	1	0.096	1	NA	NA
120-36-5	Dichlorprop	NS	NS	8	0.147	8	NA	NA
88-85-7	Dinoseb	NS	0.48	1	0.2	1	NA	NA
94-74-6	MCPA	NS	NS	100	7.65	100	NA	NA
93-65-2	MCPP	NS	NS	100	18.8	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Ecological PALs are from the following:
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method MADEP EPH
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: Extractable Petroleum Hydrocarbons (*EPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP EPH, L-25

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
NA	C11-C22 Aromatics Unadjusted	NS	5	100	NA	100	NA	NA
NA	C11-C22 Aromatics	NS	5	100	NA	100	NA	NA
NA	C19-C36 Aliphatics	NS	2100	100	NA	100	NA	NA
NA	C9-C18 Aliphatics	NS	1800	100	NA	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Method specified target compounds will be reported using SVOC method 8270C
Highlighted cells indicate PAL is less than Project Quantitation Limit.
2 - Ecological PALs are the lowest from the following:
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method MADEP VPH
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: Volatile Petroleum Hydrocarbons (*VPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP VPH, L-26

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
NA	C5 – C8 Aliphatics Unadjusted	NS	250	100	NA	100	NA	NA
NA	C5 – C8 Aliphatics	NS	250	100	NA	100	NA	NA
NA	C9 – C10 Aromatics	NS	540	100	NA	100	NA	NA
NA	C9 – C12 Aliphatics Unadjusted	NS	1800	100	NA	100	NA	NA
NA	C9 – C12 Aliphatics	NS	1800	100	NA	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit

Prepared by: BR
Checked by: TC
10/14/08

µg/L – micrograms per liter (parts per billion)
Method specific target compounds will be reported using VOC Method 8260B.
2 - Ecological PALs are from the following:
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: *Metals*
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7470A, L-1

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
7429-90-5	Aluminum	87	87	100	2.2	100	NA	NA
7440-36-0	Antimony	NS	80	6	2.3	6	NA	NA
7440-38-2	Arsenic	150	36	10	1.4	10	NA	NA
7440-39-3	Barium	NS	220	10	0.25	10	NA	NA
7440-41-7	Beryllium	NS	3.6	1	0.029	1	NA	NA
7440-43-9	Cadmium(**)	0.25	0.091	1	0.18	1	NA	NA
7440-70-2	Calcium	NS	NS	400	27	400	NA	NA
7440-47-3	Chromium	NS	11	5	0.17	5	NA	NA
7440-48-4	Cobalt	NS	24	10	0.59	10	NA	NA
7440-50-8	Copper*	9	1.58	10	1.6	10	NA	NA
7439-89-6	Iron	1000	1000	100	16	100	NA	NA
7439-92-1	Lead*	2.5	0.5	5	1.06	5	NA	NA
7439-95-4	Magnesium	NS	NS	400	8.3	400	NA	NA
7439-96-5	Manganese	NS	120	10	0.33	10	NA	NA
7439-97-6	Mercury(**)	0.77	0.0013	0.2	0.129	0.2	NA	NA
7440-02-0	Nickel	52	8.2	10	1.6	10	NA	NA
7440-09-7	Potassium	NS	NS	4000	812	4000	NA	NA
7782-49-2	Selenium	5	5	10	2.2	10	NA	NA
7440-22-4	Silver* (**)	NS	0.03	5	0.86	5	NA	NA
7440-23-5	Sodium	NS	NS	2000	65	2000	NA	NA
7440-28-0	Thallium	NS	10	10	1.9	10	NA	NA
7440-31-5	Tin	NS	NS	50	3.9	50	NA	NA

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: *Metals*
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7470A, L-1

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
7440-62-2	Vanadium	NS	12	10	0.29	10	NA	NA
7440-66-6	Zinc	120	36	50	2.9	50	NA	NA

Notes:
* - Results for copper, lead, and silver will be reported using method 6020A.
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Metals by Method 6020A
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: *Metals*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 6020A, L-1

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
7440-50-8	Copper	9	1.58	1	0.85	1	NA	NA
7439-92-1	Lead	2.5	0.5	1	0.29	1	NA	NA
7440-22-4	Silver (**)	NS	0.03	1	0.44	1	NA	NA

Notes:
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Hexavalent Chromium
Medium/Matrix: *Water*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Surface Water (SW)*
Analytical Parameter: Hexavalent Chromium
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 7199, L-38

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/L)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/L)[1]	Eco PAL (µg/L)[2]		MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
18540-29-9	Hexavalent Chromium	11	11	1.0	0.02	1.0	NA	NA

Notes:
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for this analyte. See discussion in Section 7.1
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Nitrate, Nitrite, Sulfate, Fluoride, Bromide, Chloride, Alkalinity, Hardness, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Ammonia, pH, Specific Conductance, Chemical Oxygen Demand (COD), Total Organic Carbon (TOC).
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface water (SW)
Analytical Parameter(s): Nitrate, Nitrite, Sulfate, Fluoride, Bromide, Chloride, Alkalinity, Hardness, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Ammonia, pH, Specific Conductance, Chemical Oxygen Demand (COD), Total Organic Carbon (TOC).
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Methods listed below. Refer to Worksheet #20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Analysis Method	Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte		Human Health PAL (mg/L)[1]	Eco PAL (mg/L)[2]	Project Quantitation Limit (mg/L)	MDL (mg/L)	QL (mg/L)	MDL (mg/L)	QL (mg/L)
16984-48-8	Fluoride	EPA 300	NS	NS	0.1	0.025	0.1	NA	NA
24959-67-9	Bromide	EPA 300	NS	NS	0.01	0.025	0.01	NA	NA
14797-55-8	Nitrate	EPA 300	NS	NS	0.05	0.031	0.05	NA	NA
14797-65-0	Nitrite	EPA 300	NS	NS	0.01	0.0037	0.01	NA	NA
14808-79-8	Sulfate	EPA 300	NS	NS	2.0	0.26	2.0	NA	NA
16887-00-6	Chloride	EPA 300	230	230	1.0	0.44	1.0	NA	NA
E-11778	Hardness (as Calcium Carbonate)	SM 2340B	NS	NS	2.64	0.152	2.64	NA	NA
11-43-8	Alkalinity	SM 2320B	NS	NS	1.0	0.220	1.0	NA	NA
E-10173	Total Suspended Solids (TSS)	SM 2540D	NS	NS	5	4.23	5	NA	NA
E-10173	Total Dissolved Solids (TDS)	SM 2540C	NS	NS	10	6.72	10	NA	NA
7664-41-7	Ammonia	QuikChem: LACHAT 10-107-06-1-A	3	3	0.10	0.084	0.10	NA	NA
NA	pH (standard pH units)	SM 4500	NS	NS	0.1	NA	0.1	NA	NA
NA	Specific Conductance (umhos/cm)	SM 2510B	NS	NS	1.0	0.8883	1.0	NA	NA
E-10107	Chemical Oxygen Demand	EPA 410.4	NS	NS	20	2.6	20	NA	NA
E-10195	Total Organic Carbon	SM 5310B	NS	NS	1.0	0.7875	1.0	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/L – milligrams per liter (parts per billion) umhos/cm – micromhos per centimeter

Prepared by: BR
Checked by: TC
8/7/09

1 - Human Health PALs are from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
2 - Ecological PALs are the lowest from the following:
USEPA. 2006. *National Recommended Water Quality Criteria.* Freshwater CCC. Office of Water and Office of Science Technology.
MassDEP. 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards.
Suter, G.W., and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Formaldehyde and Acetaldehyde
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface water (SW)
Analytical Parameter: Formaldehyde and Acetaldehyde
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: SW-846 Method 8315, L-32

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
50-00-0	Formaldehyde	NS	NS	50	5	50	NA	NA
75-07-0	Acetaldehyde	NS	NS	100	10	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Hydrazine, MMH, UDMH
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: Hydrazine, Mono-methylhydrazine (MMH), Unsymmetrical dimethylhydrazine (UDMH)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Lancaster Laboratory Modified 8315, L-24

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
302-01-2	Hydrazine	NS	NS	0.2	0.05	0.2	NA	NA
60-34-4	MMH	NS	NS	0.5	0.25	0.5	NA	NA
57-14-7	UDMH	NS	NS	0.5	0.25	0.5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Phthalic anhydride (acid)
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: Phthalic anhydride (acid)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified HPLC Method 8000, L-36

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
85-44-9	phthalic anhydride (acid)	NS	NS	10	1.4	10	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N,N-dimethylformamide (DMF)
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: N,N-dimethylformamide (DMF)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified Method 8033-GC/NPD, L-35

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
68-12-2	DMF	NS	NS	20	TBD	20	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N-nitrosodimethylamine (NDMA) and N-nitros-di-n-propylamine (NDPA)
Medium/Matrix: water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: N-nitrosodymethylamine (NDMA)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified EPA 521 – GC/CI/MS/MS, L-5

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
62-75-9	NDMA	NS	10000	0.005	0.000741	0.002	NA	NA
621-64-7	NDPA	NS	NS	0.01	0.00066	0.01	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Nonylphenols (alkylphenols)
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface Water (SW)
Analytical Parameter: Nonylphenol tech mix, Nonylphenol Diethoxylate tech mix, p-tert-Octyl Phenol, Bisphenol-A
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: STL West Sacramento, CA – SOP Alkylphenols by GC/MS-SIM, L-34

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)[1]	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
84852-15-3	Nonylphenol tech mix	NS	NS	5	1.6	5	NA	NA
NA	Nonylphenol Diethoxylate tech mix	NS	NS	20	1.9	20	NA	NA
140-66-9	p-tert-Octyl Phenol	NS	NS	2	TBD	2	NA	NA
80-05-7	Bisphenol-A	NS	NS	2	TBD	2	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Opex and Kempore
Medium/Matrix: Water
Region I Matrix Code (from EPA-NE DQO Summary Form): Surface water (SW)
Analytical Parameter: Dinitrosopentamethylenetetramine (Opex) and Azodicarbonamide (Kempore)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: SW-846 Method 8000B (HPLC), L-11

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/L)	Eco PAL (µg/L)	Project Quantitation Limit (µg/L)	MDL (µg/L)	QL (µg/L)	MDL (µg/L)	QL (µg/L)
101-25-7	Opex	NS	NS	100	20	100	NA	NA
123-77-3	Kempore	NS	NS	1000	230	1000	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
630-20-6	1,1,1,2-Tetrachloroethane	2000	225000	2.5	0.625	2.5	NA	NA
95-63-6	1,2,4-Trimethylbenzene	67000	NS	2.5	0.38	2.5	NA	NA
71-55-6	1,1,1-Trichloroethane	500000	29800	2.5	0.52	2.5	NA	NA
79-34-5	1,1,2,2-Tetrachloroethane	590	127	2.5	0.63	2.5	NA	NA
76-13-1	1,1,2-Trichloro-1,2,2-Trifluoroethane	43000000	NS	12.5	0.70	12.5	NA	NA
79-00-5	1,1,2-Trichloroethane	1100	28600	2.5	0.30	2.5	NA	NA
75-34-3	1,1-Dichloroethane	3400	20100	2.5	0.26	2.5	NA	NA
75-35-4	1,1-Dichloroethene	250000	8280	2.5	0.49	2.5	NA	NA
563-58-6	1,1-Dichloropropene*	NS	NS	2.5	0.42	2.5	NA	NA
87-61-6	1,2,3-Trichlorobenzene	NS	20000	2.5	0.55	2.5	NA	NA
96-18-4	1,2,3-Trichloropropane	91	3360	2.5	0.65	2.5	NA	NA
120-82-1	1,2,4-Trichlorobenzene	87000	11100	2.5	0.36	2.5	NA	NA
96-12-8	1,2-Dibromo-3-chloropropane*	5.6	35.2	5	1.37	5	NA	NA
106-93-4	1,2-Dibromoethane	34	1230	2.5	0.65	2.5	NA	NA
95-50-1	1,2-Dichlorobenzene	1000000	2960	2.5	0.20	2.5	NA	NA
107-06-2	1,2-Dichloroethane	450	14200	2.5	0.26	2.5	NA	NA
78-87-5	1,2-Dichloropropane	930	32700	2.5	0.58	2.5	NA	NA
108-67-8	1,3,5-Trimethylbenzene*	47000	NS	2.5	0.23	2.5	NA	NA
541-73-1	1,3-Dichlorobenzene	100000	37700	2.5	0.44	2.5	NA	NA
142-28-9	1,3-Dichloropropane*	1600000	NS	2.5	0.59	2.5	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
106-46-7	1,4-Dichlorobenzene	2600	546	2.5	0.22	2.5	NA	NA
123-91-1	1,4-Dioxane*	44000	1830	250	27.7	250	NA	NA
594-20-7	2,2-Dichloropropane*	NS	NS	2.5	0.73	2.5	NA	NA
107-39-1	2,4,4-Trimethyl-1-pentene	NS	NS	5	0.52	5	NA	NA
107-40-4	2,4,4-Trimethyl-2-Pentene	NS	NS	5	0.30	5	NA	NA
78-93-3	2-Butanone*	500000	89600	25	10.95	25	NA	NA
95-49-8	2-Chlorotoluene*	1600000	NS	2.5	0.31	2.5	NA	NA
591-78-6	2-Hexanone*	NS	NS	25	9.2	25	NA	NA
106-43-4	4-Chlorotoluene*	5500000	NS	2.5	0.45	2.5	NA	NA
108-10-1	4-Methyl-2-pentanone	500000	4430	25	7.51	25	NA	NA
67-64-1	Acetone	500000	2500	250	32.2	250	NA	NA
71-43-2	Benzene	1100	255	2.5	0.22	2.5	NA	NA
108-86-1	Bromobenzene*	94000	NS	2.5	0.34	2.5	NA	NA
74-97-5	Bromochloromethane	NS	NS	2.5	0.66	2.5	NA	NA
75-27-4	Bromodichloromethane	280	540	2.5	0.37	2.5	NA	NA
75-25-2	Bromoform	61000	15900	2.5	0.45	2.5	NA	NA
74-83-9	Bromomethane	7900	235	5	0.75	5	NA	NA
104-51-8	Butylbenzene*	NS	NS	2.5	0.26	2.5	NA	NA
75-15-0	Carbon Disulfide	670000	94.1	2.5	0.58	2.5	NA	NA
56-23-5	Carbon Tetrachloride	250	2980	2.5	0.68	2.5	NA	NA
108-90-7	Chlorobenzene	310000	13100	2.5	0.27	2.5	NA	NA
75-00-3	Chloroethane	15000000	NS	5	1.05	5	NA	NA
67-66-3	Chloroform	300	1190	2.5	0.31	2.5	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
74-87-3	Chloromethane	120000	10400	5	0.62	5	NA	NA
156-59-2	Cis-1,2-Dichloroethene	100000	NS	2.5	0.18	2.5	NA	NA
10061-01-5	cis-1,3-Dichloropropene	NS	398	2.5	0.45	2.5	NA	NA
110-82-7	Cyclohexane	7200000	NS	2.5	TBD	2.5	NA	NA
124-48-1	Dibromochloromethane	700	2050	2.5	0.70	2.5	NA	NA
74-95-3	Dibromomethane*	780000	65000	2.5	0.74	2.5	NA	NA
75-71-8	Dichlorodifluoromethane	190000	39500	5	0.33	5	NA	NA
60-29-7	Diethyl ether*	16000000	NS	2.5	0.91	2.5	NA	NA
108-20-3	Diisopropyl Ether*	1200000	NS	2.5	0.35	2.5	NA	NA
637-92-3	Ethyl Tertiary Butyl Ether*	NS	NS	2.5	0.60	2.5	NA	NA
100-41-4	Ethylbenzene	5700	5160	2.5	0.20	2.5	NA	NA
87-68-3	Hexachlorobutadiene*	6000	39.8	2.5	0.52	2.5	NA	NA
98-82-8	Isopropylbenzene	2200000	NS	2.5	0.38	2.5	NA	NA
79-20-9	Methyl acetate	78000000	NS	2.5	TBD	2.5	NA	NA
1634-04-4	Methyl tert-butylether	39000	NS	2.5	0.50	2.5	NA	NA
108-87-2	Methylcyclohexane	3400000	NS	2.5	TBD	2.5	NA	NA
75-09-2	Methylene Chloride	11000	4050	5	0.48	5	NA	NA
108-38-3	m-Xylene (reported as m&p Xylene)*	500000	NS	5	0.69	5	NA	NA
91-20-3	Naphthalene*	3900	99.4	25	0.50	25	NA	NA
103-65-1	n-propylbenzene*	NS	NS	2.5	0.29	2.5	NA	NA
95-47-6	o-Xylene*	500000	NS	2.5	0.26	2.5	NA	NA
99-87-6	p-Isopropyltoluene*	NS	NS	2.5	0.36	2.5	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
106-42-3	p-Xylene (reported as m&p Xylene)*	500000	NS	5	0.69	5	NA	NA
135-98-8	Sec-butylbenzene*	NS	NS	2.5	0.28	2.5	NA	NA
100-42-5	Styrene	30000	4690	2.5	0.50	2.5	NA	NA
994-05-8	Tert-amyl Methyl Ether*	NS	NS	2.5	0.60	2.5	NA	NA
98-06-6	Tert-butylbenzene*	NS	NS	2.5	0.22	2.5	NA	NA
127-18-4	Tetrachloroethene	570	9920	2.5	0.57	2.5	NA	NA
109-99-9	Tetrahydrofuran*	NS	NS	25	10.8	25	NA	NA
108-88-3	Toluene	500000	5450	2.5	0.23	2.5	NA	NA
156-60-5	Trans-1,2-Dichloroethene	110000	784	2.5	0.37	2.5	NA	NA
10061-02-6	trans-1,3-Dichloropropene	NS	398	2.5	0.44	2.5	NA	NA
79-01-6	Trichloroethene	2800	1387	2.5	0.37	2.5	NA	NA
75-69-4	Trichlorofluoromethane	800000	16400	5	0.72	5	NA	NA
75-01-4	Vinyl Chloride	60	623	2.5	0.56	2.5	NA	NA
1330-20-7	Xylenes (total)	500000	4162	10	0.95	10	NA	NA

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8260B list. Compound is on Method 8260B list for the MCP.
Tentatively Identified Compouns (TICs) will be reported for 8260B analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil.

Prepared by: BR
Checked by: TC
10/14/08

http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites.* Industrial Soil.
http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method 8270C Low Level
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
90-12-0	1-Methylnaphthalene	22000	NS	TBD	TBD	TBD	NA	NA
92-52-4	1,1-Biphenyl	1000000	60000	33.33	TBD	33.33	NA	NA
95-94-3	1,2,4,5-Tetrachlorobenzene	18000	2020	33.33	2.24	33.33	NA	NA
120-82-1	1,2,4-Trichlorobenzene*■	87000	11100	33.33	6.48	33.33	NA	NA
95-50-1	1,2-Dichlorobenzene*■	1000000	2960	33.33	3.61	33.33	NA	NA
541-73-1	1,3-Dichlorobenzene*■	100000	37700	33.33	9.66	33.33	NA	NA
106-46-7	1,4-Dichlorobenzene*■	2600	546	33.33	6.01	33.33	NA	NA
58-90-2	2,3,4,6-Tetrachlorophenol	NS	NS	333	333	333	NA	NA
95-95-4	2,4,5-Trichlorophenol	1000000	4000	33.33	3.77	33.33	NA	NA
88-06-2	2,4,6-Trichlorophenol	44000	9940	33.33	6.99	33.33	NA	NA
120-83-2	2,4-Dichlorophenol	60000	87500	33.33	6.42	33.33	NA	NA
105-67-9	2,4-Dimethylphenol	500000	10	33.33	5.80	33.33	NA	NA
51-28-5	2,4-Dinitrophenol	50000	60.9	33.33	4.89	33.33	NA	NA
121-14-2	2,4-Dinitrotoluene	1600	1280	33.33	4.70	33.33	NA	NA
606-20-2	2,6-Dinitrotoluene	61000	32.8	33.33	4.22	33.33	NA	NA
91-58-7	2-Chloronaphthalene	6300000	12.2	33.33	5.12	33.33	NA	NA
95-57-8	2-Chlorophenol	100000	243	33.33	3.25	33.33	NA	NA
91-57-6	2-Methylnaphthalene	310000	3240	33.33	25.44	33.33	NA	NA
95-48-7	2-Methylphenol	3100000	40400	33.33	4.76	33.33	NA	NA
88-74-4	2-Nitroaniline	180000	74100	166.7	21.48	166.7	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C Low Level
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
88-75-5	2-Nitrophenol	NS	1600	33.33	3.86	33.33	NA	NA
91-94-1	3,3'-Dichlorobenzidine	1000	646	33.33	24.36	33.33	NA	NA
108-39-4	3-Methylphenol (reported as 3&4 Methylphenol)*	3100000	3490	33.33	6.33	33.33	NA	NA
99-09-2	3-Nitroaniline	NS	3160	166.7	9.51	166.7	NA	NA
534-52-1	4,6-Dinitro-2-Methylphenol	6100	144	166.7	8.52	166.7	NA	NA
101-55-3	4-Bromophenyl-phenylether	NS	NS	33.33	6.93	33.33	NA	NA
59-50-7	4-Chloro-3-Methylphenol	NS	7950	66.7	9.20	66.7	NA	NA
106-47-8	4-Chloroaniline	2400	1100	66.7	13.52	66.7	NA	NA
7005-72-3	4-Chlorophenyl-phenylether	NS	NS	33.33	5.30	33.33	NA	NA
106-44-5	4-Methylphenol (reported as 3&4 Methylphenol)	310000	163000	33.33	6.33	33.33	NA	NA
100-01-6	4-Nitroaniline	24000	21900	166.7	8.89	166.7	NA	NA
100-02-7	4-Nitrophenol	NS	5120	166.7	6.49	166.7	NA	NA
83-32-9	Acenaphthene	1000000	20000	33.33	4.89	33.33	NA	NA
208-96-8	Acenaphthylene	100000	682000	33.33	5.89	33.33	NA	NA
98-86-2	Acetophenone	7800000	300000	33.33	3.6	33.33	NA	NA
62-53-3	Aniline*	85000	56.8	33.33	17.29	33.33	NA	NA
120-12-7	Anthracene	1000000	1480000	33.33	10.50	33.33	NA	NA
1912-24-9	Atrazine	2100	NS	33.33	TBD	33.33	NA	NA
103-33-3	Azobenzene*	4900	NS	33.33	4.14	33.33	NA	NA
100-52-7	Benzaldehyde	7800000	NS	33.33	TBD	33.33	NA	NA
56-55-3	Benzo(a)anthracene	150	5210	33.33	6.39	33.33	NA	NA
50-32-8	Benzo(a)pyrene	15	1520	33.33	6.10	33.33	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C Low Level
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
205-99-2	Benzo(b)fluoranthene	150	59800	33.33	7.64	33.33	NA	NA
191-24-2	Benzo(g,h,i)perylene	1000000	119000	33.33	6.48	33.33	NA	NA
207-08-9	Benzo(k)fluoranthene	1500	148000	33.33	9.24	33.33	NA	NA
65-85-0	Benzoic acid	240000000	NS	TBD	TBD	TBD	NA	NA
119-61-9	Benzophenone	NS	NS	TBD	TBD	TBD	NA	NA
100-51-6	Benzyl alcohol	31000000	65800	33.33	7.69	66.7	NA	NA
108-60-1	Bis (2-Chloroisopropyl) ether*	2000	19900	33.33	3.37	33.33	NA	NA
111-91-1	bis(2-Chloroethoxy)methane	180000	302	33.33	5.99	33.33	NA	NA
111-44-4	bis(2-Chloroethyl) ether	190	23700	33.33	8.10	33.33	NA	NA
117-81-7	Bis(2-Ethylhexyl) phthalate	35000	910	33.33	10.57	33.33	NA	NA
85-68-7	Butylbenzylphthalate	260000	239	33.33	7.28	33.33	NA	NA
105-60-2	Caprolactam	31000000	NS	33.33	TBD	33.33	NA	NA
86-74-8	Carbazole	NS	NS	33.33	TBD	33.33	NA	NA
218-01-9	Chrysene	7000	4730	33.33	7.19	33.33	NA	NA
53-70-3	Dibenz(a,h)anthracene	15	18400	33.33	6.73	33.33	NA	NA
132-64-9	Dibenzofuran	NS	NS	33.33	19.27	33.33	NA	NA
84-66-2	Diethylphthalate	1000000	24800	33.33	4.70	33.33	NA	NA
131-11-3	Dimethylphthalate	1000000	200000	33.33	4.90	33.33	NA	NA
84-74-2	Di-n-butylphthalate	6100000	90	33.33	10.04	33.33	NA	NA
101-84-8	Diphenyl oxide	NS	NS	TBD	TBD	TBD	NA	NA
117-84-0	Di-n-octylphthalate	NS	709000	33.33	9.44	33.33	NA	NA
206-44-0	Fluoranthene	1000000	122000	33.33	21.09	33.33	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C Low Level
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
86-73-7	Fluorene	1000000	30000	33.33	12.80	33.33	NA	NA
118-74-1	Hexachlorobenzene	300	199	33.33	7.91	33.33	NA	NA
87-68-3	Hexachlorobutadiene■	6000	39.8	33.33	6.64	33.33	NA	NA
77-47-4	Hexachlorocyclopentadiene	370000	755	66.7	9.10	66.7	NA	NA
67-72-1	Hexachloroethane	9000	596	33.33	6.36	33.33	NA	NA
193-39-5	Indeno(1,2,3-cd)pyrene	150	109000	33.33	10.17	33.33	NA	NA
78-59-1	Isophorone	510000	139000	33.33	7.14	33.33	NA	NA
91-20-3	Naphthalene■	3900	99.4	80	73.93	80	NA	NA
98-95-3	Nitrobenzene	4400	1310	33.33	4.54	33.33	NA	NA
62-75-9	N-nitrosodimethylamine	2.3	0.0321	33.33	3.77	33.33	NA	NA
621-64-7	N-Nitroso-di-n-propylamine	69	544	33.33	15.6	33.33	NA	NA
86-30-6	N-nitrosodiphenylamine	99000	545	33.33	6.67	33.33	NA	NA
122-39-4	Diphenylamine∆	1500000	1010	TBD	TBD	TBD	NA	NA
87-86-5	Pentachlorophenol	3000	119	33.33	6.89	33.33	NA	NA
85-01-8	Phenanthrene	1000000	45700	40	38.36	40	NA	NA
108-95-2	Phenol	500000	30000	33.33	4.17	33.33	NA	NA
129-00-0	Pyrene	1000000	78500	33.33	8.83	33.33	NA	NA

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8270C list. Compound is on Method 8270C list for the MCP.
■ - Compound results will be reported from 8260B VOC analysis if available.

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Δ An additional Florisil clean-up step (Method 3620B) to separate N-nitrosodiphenylamine from Diphenylamine will be performed by the laboratory on a sub-set of samples to provide information of compound identification.
Tentatively Identified Compouns (TICs) will be reported for 8270C analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm.May 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method 8082
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): *Soil (SO)*
Analytical Parameter: *PCBs*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8082, L-28

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
12674-11-2	Aroclor 1016	3900	6520	100	13.61	100	NA	NA
11104-28-2	Aroclor 1221	170	332	100	15.15	100	NA	NA
11141-16-5	Aroclor 1232	170	332	100	23.49	100	NA	NA
53469-21-9	Aroclor 1242	220	329	100	64.27	100	NA	NA
12672-29-6	Aroclor 1248	220	72	100	22.3	100	NA	NA
11097-69-1	Aroclor 1254	220	111	100	26.21	100	NA	NA
11096-82-5	Aroclor 1260	220	332	100	27.71	100	NA	NA
37324-23-5	Aroclor 1262	3000	332	100	13.72	100	NA	NA
11100-14-4	Aroclor 1268	3000	332	100	21.42	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg – micrograms per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.

Prepared by: BR
Checked by: TC
10/14/08

1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm.May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.

Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method MADEP EPH
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: Extractable Petroleum Hydrocarbons (*EPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP EPH, L-7

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
NA	C11-C22 Aromatics	800000	NS	3300	NA	3300	NA	NA
NA	C11-C22 Aromatics Unadjusted	800000	NS	3300	NA	3300	NA	NA
NA	C19-C36 Aliphatics	2500000	NS	3300	NA	3300	NA	NA
NA	C9-C18 Aliphatics	1000000	NS	3300	NA	3300	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
Method specific target compounds will be reported using VOC Method 8270C
1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.

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Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method MADEP VPH
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: Volatile Petroleum Hydrocarbons (*VPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP VPH, L-8

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
NA	C5 – C8 Aliphatics Unadjusted	100000	NS	5000	NA	5000	NA	NA
NA	C5 – C8 Aliphatics	100000	NS	5000	NA	5000	NA	NA
NA	C9 – C10 Aromatics	100000	NS	5000	NA	5000	NA	NA
NA	C9 – C12 Aliphatics Unadjusted	1000000	NS	5000	NA	5000	NA	NA
NA	C9 – C12 Aliphatics	1000000	NS	5000	NA	5000	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
Method specific target compounds will be reported using VOC Method 8260B.
1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.

Prepared by: BR
Checked by: TC
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Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: *Metals*
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7471A, L-2

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (mg/kg)[1]	Eco PAL (mg/kg)[2]		MDL (mg/kg)	QL (mg/kg)	MDL (mg/kg)	QL (mg/kg)
7429-90-5	Aluminum	77000	3.83	2.5	0.194	2.5	NA	NA
7440-36-0	Antimony	20	0.142	0.5	0.137	0.5	NA	NA
7440-38-2	Arsenic	0.39	0.25	1	0.081	1	NA	NA
7440-39-3	Barium	1000	1.04	0.5	0.017	0.5	NA	NA
7440-41-7	Beryllium	0.7	1.06	0.2	0.002	0.2	NA	NA
7440-43-9	Cadmium	2	0.00222	0.2	0.0043	0.2	NA	NA
7440-70-2	Calcium	NS	NS	10	1.056	10	NA	NA
7440-47-3	Chromium	30	0.4	0.5	0.073	0.5	NA	NA
7440-48-4	Cobalt	23	0.14	0.5	0.016	0.5	NA	NA
7440-50-8	Copper	3100	5.4	1	0.078	1	NA	NA
7439-89-6	Iron	55000	NS	5	0.643	5	NA	NA
7439-92-1	Lead	300	0.0537	0.5	0.044	0.5	NA	NA
7439-95-4	Magnesium	NS	NS	10	0.548	10	NA	NA
7439-96-5	Manganese	1800	220	1	0.096	1	NA	NA
7439-97-6	Mercury	4.3	0.1	0.1	0.045	0.1	NA	NA
7440-02-0	Nickel	20	13.6	1	0.014	1	NA	NA
7440-09-7	Potassium	NS	NS	200	53.3	200	NA	NA
7782-49-2	Selenium	390	0.0276	0.5	0.272	0.5	NA	NA
7440-22-4	Silver	100	2	0.5	0.045	0.5	NA	NA
7440-23-5	Sodium	NS	NS	100	5.89	100	NA	NA
7440-28-0	Thallium	5.1	0.027	1	0.191	1	NA	NA

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (*SO)*
Analytical Parameter: *Metals*
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7471A, L-2

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (mg/kg)[1]	Eco PAL (mg/kg)[2]		MDL (mg/kg)	QL (mg/kg)	MDL (mg/kg)	QL (mg/kg)
7440-31-5	Tin	NS	NS	0.09	5	0.094	NA	NA
7440-62-2	Vanadium	550	0.714	1	0.015	1	NA	NA
7440-66-6	Zinc	2500	6.62	2.5	0.134	2.5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/kg – micrograms per kilogram (parts per million)
Highlighted cells indicate PAL is less than Project Quantitation Limit.

Prepared by: BR
Checked by: TC
10/14/08

1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Hexavalent Chromium (Cr+6)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter(s): Hexavalent Chromium
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3060A/7199, L-37

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (mg/kg)	Eco PAL (mg/kg)	Project Quantitation Limit (mg/kg)	MDL (mg/kg)	QL (mg/kg)	MDL (mg/kg)	QL (mg/kg)
18540-29-9	Hexavalent Chromium	30	12.01	2	0.111	2	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/kg – micrograms per kilogram (parts per million)
1 - Human Health PALs are the lowest from the following:
ORNL. 2008. *Screening Levels for Chemical Contaminants.* Residential Soil. July.
ORNL. 2008. *Screening Levels for Chemical Contaminants.* Industrial Soil. July.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Ammonia, Total Organic Carbon (TOC), Nitrate, Nitrite, Sulfate, and Chloride.
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter(s): Ammonia, Total Organic Carbon (TOC), Nitrate, Nitrite, Sulfate, and Chloride.
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Methods listed below. Refer to Worksheet #20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)									
		Analysis Method	Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte		Human Health PAL (mg/Kg)	Eco PAL (mg/Kg)	Project Quantitation Limit (mg/Kg)	MDL (mg/Kg)	QL (mg/Kg)	MDL (mg/Kg)	QL (mg/Kg)
7664-41-7	Ammonia	QuikChem: LACHAT 10-107-06-1-A	NS	NS	5	TBD	5	NA	NA
E-10195	Total Organic Carbon	Lloyd Kahn	NS	NS	1000	TBD	1000	NA	NA
14797-55-8	Nitrate	EPA 300	130000	NS	5	TBD	5	NA	NA
14797-65-0	Nitrite	EPA 300	7800	NS	1	TBD	1	NA	NA
14808-79-8	Sulfate	EPA 300	NS	NS	40	TBD	40	NA	NA
16887-00-6	Chloride	EPA 300	NS	NS	20	TBD	20	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/kg – micrograms per kilogram (parts per million)
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.

EPA-NE QAPP Worksheet #9b- Formaldehyde and Acetaldehyde
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: Formaldehyde and Acetaldehyde
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: SW-846 Method 8315, L-32

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
50-00-0	Formaldehyde	12000000	83300	100	78	100	NA	NA
75-07-0	Acetaldehyde	11000	NS	200	29	200	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.

Prepared by: BR
Checked by: TC
10/14/08

1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Hydrazine, MMH, UDMH
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: Hydrazine, Mono-methylhydrazine (MMH), Unsymmetrical dimethylhydrazine (UDMH)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Lancaster Laboratory Modified 8315, L-24

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
302-01-2	Hydrazine	NS	NS	2	0.5	2	NA	NA
60-34-4	MMH	NS	NS	5	2	5	NA	NA
57-14-7	UDMH	NS	NS	5	2	5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Phthalic anhydride (acid)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: Phthalic anhydride (acid)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified HPLC Method 8000, L-36

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
85-44-9	phthalic anhydride (acid)	120000000	NS	100	18	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PAL is the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N,N-dimethylformamide (DMF)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: N,N-dimethylformamide
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified Method 8033-GC/NPD, L-35

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
68-12-2	DMF	6100000	NS	200	TBD	200	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N-nitrosodimethylamine (NDMA)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Soil (SO)
Analytical Parameter: N-nitrosodimethylamine (NDMA)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Method 3550/3546 / Modified EPA 8270 Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
62-75-9	NDMA	2.3	0.0321	5	1	5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.

Prepared by: BR
Checked by: TC
10/14/08

1 - Human Health PALs are the lowest from the following:
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Residential Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
USEPA, 2009. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*. Industrial Soil. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. May, 19.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-1 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-2 Standards. February.
MassDEP, 2008. *Massachusetts Contingency Plan.* 310 CMR 40.0974(2) Table 5, S-3 Standards. February.
2 - Ecological PALs are the lowest from the following:
USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.
Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-85/R2. November 1997.
Efroymson, R.A., M.E. Will, G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997.
Sample, B.E., and D.M. Opresko, G.W. Sutter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
630-20-6	1,1,1,2-Tetrachloroethane	NS	NS	2.5	0.625	2.5	NA	NA
95-63-6	1,2,4-Trimethylbenzene	NS	NS	2.5	0.38	2.5	NA	NA
71-55-6	1,1,1-Trichloroethane	NS	30	2.5	0.52	2.5	NA	NA
79-34-5	1,1,2,2-Tetrachloroethane	NS	850	2.5	0.78	2.5	NA	NA
76-13-1	1,1,2-Trichloro-1,2,2-Trifluoroethane	NS	NS	12.5	0.70	12.5	NA	NA
79-00-5	1,1,2-Trichloroethane	NS	518	2.5	0.30	2.5	NA	NA
75-34-3	1,1-Dichloroethane (**)	NS	0.575	2.5	0.26	2.5	NA	NA
75-35-4	1,1-Dichloroethene	NS	19.4	2.5	0.49	2.5	NA	NA
563-58-6	1,1-Dichloropropene*	NS	NS	2.5	0.42	2.5	NA	NA
87-61-6	1,2,3-Trichlorobenzene	NS	NS	2.5	0.55	2.5	NA	NA
96-18-4	1,2,3-Trichloropropane	NS	NS	2.5	0.65	2.5	NA	NA
120-82-1	1,2,4-Trichlorobenzene	NS	5062	2.5	0.36	2.5	NA	NA
96-12-8	1,2-Dibromo-3-chloropropane*	NS	NS	5	1.37	5	NA	NA
106-93-4	1,2-Dibromoethane	NS	NS	2.5	0.65	2.5	NA	NA
95-50-1	1,2-Dichlorobenzene	NS	294	2.5	0.20	2.5	NA	NA
107-06-2	1,2-Dichloroethane	NS	250	2.5	0.26	2.5	NA	NA
78-87-5	1,2-Dichloropropane	NS	333	2.5	0.58	2.5	NA	NA
108-67-8	1,3,5-Trimethylbenzene*	NS	NS	2.5	0.23	2.5	NA	NA
541-73-1	1,3-Dichlorobenzene	NS	1315	2.5	0.44	2.5	NA	NA
142-28-9	1,3-Dichloropropane*	NS	NS	2.5	0.59	2.5	NA	NA
106-46-7	1,4-Dichlorobenzene	NS	318	2.5	0.22	2.5	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
123-91-1	1,4-Dioxane*	NS	119	250	27.7	250	NA	NA
594-20-7	2,2-Dichloropropane*	NS	NS	2.5	0.73	2.5	NA	NA
107-39-1	2,4,4-Trimethyl-1-pentene	NS	NS	5	0.52	5	NA	NA
107-40-4	2,4,4-Trimethyl-2-Pentene	NS	NS	5	0.30	5	NA	NA
78-93-3	2-Butanone*	NS	42.4	25	10.95	25	NA	NA
95-49-8	2-Chlorotoluene*	NS	NS	2.5	0.31	2.5	NA	NA
591-78-6	2-Hexanone*	NS	22	25	9.2	25	NA	NA
106-43-4	4-Chlorotoluene*	NS	NS	2.5	0.45	2.5	NA	NA
108-10-1	4-Methyl-2-pentanone	NS	25.1	25	7.51	25	NA	NA
67-64-1	Acetone (**)	NS	8.7	250	32.2	250	NA	NA
71-43-2	Benzene	NS	142	2.5	0.22	2.5	NA	NA
108-86-1	Bromobenzene*	NS	NS	2.5	0.34	2.5	NA	NA
74-97-5	Bromochloromethane	NS	NS	2.5	0.66	2.5	NA	NA
75-27-4	Bromodichloromethane	NS	NS	2.5	0.37	2.5	NA	NA
75-25-2	Bromoform	NS	492	2.5	0.45	2.5	NA	NA
74-83-9	Bromomethane	NS	1.37	5	0.75	5	NA	NA
104-51-8	Butylbenzene*	NS	NS	2.5	0.26	2.5	NA	NA
75-15-0	Carbon Disulfide(**)	NS	0.85	2.5	0.58	2.5	NA	NA
56-23-5	Carbon Tetrachloride	NS	47	2.5	0.68	2.5	NA	NA
108-90-7	Chlorobenzene	NS	291	2.5	0.27	2.5	NA	NA
75-00-3	Chloroethane	NS	NS	5	1.05	5	NA	NA
67-66-3	Chloroform	NS	22	2.5	0.31	2.5	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
74-87-3	Chloromethane	NS	NS	5	0.62	5	NA	NA
156-59-2	Cis-1,2-Dichloroethene	NS	NS	2.5	0.18	2.5	NA	NA
10061-01-5	cis-1,3-Dichloropropene	NS	NS	2.5	0.45	2.5	NA	NA
110-82-7	Cyclohexane	NS	NS	2.5	TBD	2.5	NA	NA
124-48-1	Dibromochloromethane	NS	NS	2.5	0.70	2.5	NA	NA
74-95-3	Dibromomethane*	NS	NS	2.5	0.74	2.5	NA	NA
75-71-8	Dichlorodifluoromethane	NS	NS	5	0.33	5	NA	NA
60-29-7	Diethyl ether*	NS	NS	2.5	0.91	2.5	NA	NA
108-20-3	Diisopropyl Ether*	NS	NS	2.5	0.35	2.5	NA	NA
637-92-3	Ethyl Tertiary Butyl Ether*	NS	NS	2.5	0.60	2.5	NA	NA
100-41-4	Ethylbenzene	NS	89	2.5	0.20	2.5	NA	NA
87-68-3	Hexachlorobutadiene*	NS	26.5	2.5	0.52	2.5	NA	NA
98-82-8	Isopropylbenzene	NS	NS	2.5	0.38	2.5	NA	NA
79-20-9	Methyl acetate	NS	NS	2.5	TBD	2.5	NA	NA
1634-04-4	Methyl tert-butylether	NS	NS	2.5	0.50	2.5	NA	NA
108-87-2	Methylcyclohexane	NS	NS	2.5	TBD	2.5	NA	NA
75-09-2	Methylene Chloride	NS	159	5	0.48	5	NA	NA
108-38-3	m-Xylene (reported as m&p Xylene)*	NS	25	5	0.69	5	NA	NA
91-20-3	Naphthalene*	180	176	25	0.50	25	NA	NA
103-65-1	n-propylbenzene*	NS	NS	2.5	0.29	2.5	NA	NA
95-47-6	o-Xylene*	NS	NS	2.5	0.26	2.5	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8260B
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: *Volatile Organic Compounds (VOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 8260B, L-3, Low level (DI water) collection method

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
99-87-6	p-Isopropyltoluene*	NS	NS	2.5	0.36	2.5	NA	NA
106-42-3	p-Xylene (reported as m&p Xylene)*	NS	NS	5	0.69	5	NA	NA
135-98-8	Sec-butylbenzene*	NS	NS	2.5	0.28	2.5	NA	NA
100-42-5	Styrene	NS	254	2.5	0.50	2.5	NA	NA
994-05-8	Tert-amyl Methyl Ether*	NS	NS	2.5	0.60	2.5	NA	NA
98-06-6	Tert-butylbenzene*	NS	NS	2.5	0.22	2.5	NA	NA
127-18-4	Tetrachloroethene	NS	410	2.5	0.57	2.5	NA	NA
109-99-9	Tetrahydrofuran*	NS	NS	25	10.8	25	NA	NA
108-88-3	Toluene	NS	50	2.5	0.23	2.5	NA	NA
156-60-5	Trans-1,2-Dichloroethene	NS	400	2.5	0.37	2.5	NA	NA
10061-02-6	trans-1,3-Dichloropropene	NS	NS	2.5	0.44	2.5	NA	NA
79-01-6	Trichloroethene	NS	112	2.5	0.37	2.5	NA	NA
75-69-4	Trichlorofluoromethane	NS	NS	5	0.72	5	NA	NA
75-01-4	Vinyl Chloride	NS	202	2.5	0.56	2.5	NA	NA
1330-20-7	Xylenes (total)	NS	160	10	0.95	10	NA	NA

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8260B list. Compound is on Method 8260B list for the MCP.
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for these analytes. See discussion in Section 7.1..
Tentatively Identified Compouns (TICs) will be reported for 8260B analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit

Prepared by: BR
Checked by: TC
10/14/08

µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
MassDEP. 2006. Technical Update Revised Sediment Screening Values.
2 - Ecological PALs are the lowest from the following:
MacDonald, D.D., C.G. Ingersoll, T.A. Berger. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater systems. Archives of Environmental Contamination and Toxicology 39, 20-31. January 13, 2000.
Jones, D.S., G.W. Suter II, and R.N. Hull, 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Oak Ridge National Laboratories (ES/ER/TM-95/R4) November 1997.
Persaud, D., R. Jaagumagi, and A. Hayton. 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Ontario Ministry of Environment and Energy. ISBN 0-7778-9248-7. August 1993.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
90-12-0	1-Methylnaphthalene	NS	130	TBD	TBD	TBD	NA	NA
92-52-4	1,1-Biphenyl	NS	1100	33.33	TBD	33.33	NA	NA
95-94-3	1,2,4,5-Tetrachlorobenzene	NS	1252	33.33	2.24	33.33	NA	NA
120-82-1	1,2,4-Trichlorobenzene*■	NS	5062	33.33	6.48	33.33	NA	NA
95-50-1	1,2-Dichlorobenzene*■	NS	294	33.33	3.61	33.33	NA	NA
541-73-1	1,3-Dichlorobenzene*■	NS	1315	33.33	9.66	33.33	NA	NA
106-46-7	1,4-Dichlorobenzene*■	NS	318	33.33	6.01	33.33	NA	NA
58-90-2	2,3,4,6-Tetrachlorophenol	NS	NS	333	TBD	333	NA	NA
95-95-4	2,4,5-Trichlorophenol	NS	NS	33.33	3.77	33.33	NA	NA
88-06-2	2,4,6-Trichlorophenol	NS	208	33.33	6.99	33.33	NA	NA
120-83-2	2,4-Dichlorophenol	NS	81.7	33.33	6.42	33.33	NA	NA
105-67-9	2,4-Dimethylphenol	NS	304	33.33	5.80	33.33	NA	NA
51-28-5	2,4-Dinitrophenol	NS	6.21	33.33	4.89	33.33	NA	NA
121-14-2	2,4-Dinitrotoluene	NS	14.4	33.33	4.70	33.33	NA	NA
606-20-2	2,6-Dinitrotoluene	NS	39.8	33.33	4.22	33.33	NA	NA
91-58-7	2-Chloronaphthalene	NS	417	33.33	5.12	33.33	NA	NA
95-57-8	2-Chlorophenol	NS	31.9	33.33	3.25	33.33	NA	NA
91-57-6	2-Methylnaphthalene**	NS	20.2	33.33	25.44	33.33	NA	NA
95-48-7	2-Methylphenol	NS	12	33.33	4.76	33.33	NA	NA
88-74-4	2-Nitroaniline	NS	NS	166.7	21.48	166.7	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
88-75-5	2-Nitrophenol	NS	NS	33.33	3.86	33.33	NA	NA
91-94-1	3,3'-Dichlorobenzidine	NS	127	33.33	24.36	33.33	NA	NA
108-39-4	3-Methylphenol (reported as 3&4 Methylphenol)*	NS	52.4	33.33	6.33	33.33	NA	NA
99-09-2	3-Nitroaniline	NS	NS	166.7	9.51	166.7	NA	NA
534-52-1	4,6-Dinitro-2-Methylphenol	NS	104	166.7	8.52	166.7	NA	NA
101-55-3	4-Bromophenyl-phenylether	NS	1200	33.33	6.93	33.33	NA	NA
59-50-7	4-Chloro-3-Methylphenol	NS	388	66.7	9.20	66.7	NA	NA
106-47-8	4-Chloroaniline	NS	146	66.7	13.52	66.7	NA	NA
7005-72-3	4-Chlorophenyl-phenylether	NS	NS	33.33	5.30	33.33	NA	NA
106-44-5	4-Methylphenol (reported as 3&4 Methylphenol)	NS	20.2	33.33	6.33	33.33	NA	NA
100-01-6	4-Nitroaniline	NS	NS	166.7	8.89	166.7	NA	NA
100-02-7	4-Nitrophenol	NS	13.3	166.7	6.49	166.7	NA	NA
83-32-9	Acenaphthene	NS	6.71	33.33	4.89	33.33	NA	NA
208-96-8	Acenaphthylene (**)	NS	5.87	33.33	5.89	33.33	NA	NA
98-86-2	Acetophenone	NS	NS	33.33	3.6	33.33	NA	NA
62-53-3	Aniline* (**)	NS	0.31	33.33	17.29	33.33	NA	NA
120-12-7	Anthracene	57	57.2	33.33	10.50	33.33	NA	NA
1912-24-9	Atrazine	NS	NS	33.33	TBD	33.33	NA	NA
103-33-3	Azobenzene*	NS	NS	33.33	4.14	33.33	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment *(SD)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
100-52-7	Benzaldehyde	NS	NS	33.33	TBD	33.33	NA	NA
56-55-3	Benzo(a)anthracene	100	108	33.33	6.39	33.33	NA	NA
50-32-8	Benzo(a)pyrene	150	140	33.33	6.10	33.33	NA	NA
205-99-2	Benzo(b)fluoranthene	NS	10400	33.33	7.64	33.33	NA	NA
191-24-2	Benzo(g,h,i)perylene	NS	170	33.33	6.48	33.33	NA	NA
207-08-9	Benzo(k)fluoranthene	NS	240	33.33	9.24	33.33	NA	NA
65-85-0	Benzoic acid	NS	130	167	14.4	167	NA	NA
119-61-9	Benzophenone	NS	NS	TBD	TBD	TBD	NA	NA
100-51-6	Benzyl alcohol	NS	1	66.7	7.69	66.7	NA	NA
108-60-1	Bis (2-Chloroisopropyl) ether*	NS	NS	33.33	3.37	33.33	NA	NA
111-91-1	bis(2-Chloroethoxy)methane	NS	NS	33.33	5.99	33.33	NA	NA
111-44-4	bis(2-Chloroethyl) ether	NS	3520	33.33	8.10	33.33	NA	NA
117-81-7	Bis(2-Ethylhexyl) phthalate	NS	182	33.33	10.57	33.33	NA	NA
85-68-7	Butylbenzylphthalate	NS	1970	33.33	7.28	33.33	NA	NA
105-60-2	Caprolactam	NS	NS	33.33	TBD	33.33	NA	NA
86-74-8	Carbazole	NS	NS	33.33	TBD	33.33	NA	NA
218-01-9	Chrysene	170	166	33.33	7.19	33.33	NA	NA
53-70-3	Dibenz(a,h)anthracene	33	33	33.33	6.73	33.33	NA	NA
132-64-9	Dibenzofuran	NS	420	33.33	19.27	33.33	NA	NA
84-66-2	Diethylphthalate	NS	295	33.33	4.70	33.33	NA	NA
131-11-3	Dimethylphthalate	NS	NS	33.33	4.90	33.33	NA	NA
84-74-2	Di-n-butylphthalate	NS	1114	33.33	10.04	33.33	NA	NA

EPA-NE QAPP Worksheet #9b- Method 8270C
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment *(SD)*
Analytical Parameter: Semiv*olatile Organic Compounds (SVOCs)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3546/8270C Low Level Method, L-4

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)[1]	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
101-84-8	Diphenyl oxide	NS	NS	TBD	TBD	TBD	NA	NA
117-84-0	Di-n-octylphthalate	NS	40600	33.33	9.44	33.33	NA	NA
206-44-0	Fluoranthene	420	423	33.33	21.09	33.33	NA	NA
86-73-7	Fluorene	77	77.4	33.33	12.80	33.33	NA	NA
118-74-1	Hexachlorobenzene	NS	20	33.33	7.91	33.33	NA	NA
87-68-3	Hexachlorobutadiene■	NS	26.5	33.33	6.64	33.33	NA	NA
77-47-4	Hexachlorocyclopentadiene	NS	901	66.7	9.10	66.7	NA	NA
67-72-1	Hexachloroethane	NS	584	33.33	6.36	33.33	NA	NA
193-39-5	Indeno(1,2,3-cd)pyrene	NS	200	33.33	10.17	33.33	NA	NA
78-59-1	Isophorone	NS	432	33.33	7.14	33.33	NA	NA
91-20-3	Naphthalene■	180	176	80	73.93	80	NA	NA
98-95-3	Nitrobenzene	NS	145	33.33	4.54	33.33	NA	NA
621-64-7	N-Nitroso-di-n-propylamine	NS	NS	33.33	3.77	33.33	NA	NA
62-75-9	N-nitrosodimethylamine	NS	NS	33.33	15.6	33.33	NA	NA
86-30-6	N-nitrosodiphenylamine	NS	NS	33.33	6.67	33.33	NA	NA
122-39-4	Diphenylamine▲	NS	34.6	TBD	TBD	TBD	NA	NA
87-86-5	Pentachlorophenol	NS	23000	33.33	6.89	33.33	NA	NA
85-01-8	Phenanthrene	200	204	40	38.36	40	NA	NA
108-95-2	Phenol	NS	49.1	33.33	4.17	33.33	NA	NA
129-00-0	Pyrene	200	195	33.33	8.83	33.33	NA	NA

Prepared by: BR
Checked by: TC
10/14/08

Notes:
Analytes present are a combination of the USEPA Target Compound List (TCL) and the Massachusetts Contingency Plan (MCP) compound list.
* - Additional compound added to the TCL 8270C list. Compound is on Method 8270C list for the MCP.
(**) - PAL is less than the MDL/QL. No additional analysis is proposed for these analytes. See discussion in Section 7.1.
■ - Compound results will be reported from 8260B VOC analysis if available.
Δ - An additional Florisil clean-up step (Method 3620B) to separate N-nitrosodiphenylamine from Diphenylamine will be performed by the laboratory on a sub-set of samples to provide information of compound identification.
Tentatively Identified Compouns (TICs) will be reported for 8270C analysis.
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
MassDEP. 2006. Technical Update Revised Sediment Screening Values.
2 - Ecological PALs are the lowest from the following:
MacDonald, D.D., C.G. Ingersoll, T.A. Berger. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater systems. Archives of Environmental Contamination and Toxicology 39, 20-31. January 13, 2000.
Jones, D.S., G.W. Suter II, and R.N. Hull, 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Oak Ridge National Laboratories (ES/ER/TM-95/R4) November 1997.
Persaud, D., R. Jaagumagi, and A. Hayton. 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Ontario Ministry of Environment and Energy. ISBN 0-7778-9248-7. August 1993.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

EPA-NE QAPP Worksheet #9b- Method MADEP EPH
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: Extractable Petroleum Hydrocarbons (*EPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP EPH, L-7

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)	Eco PAL (µg/kg)[2]		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
NA	C11-C22 Aromatics	NS	90	3300	NA	3300	NA	NA
NA	C11-C22 Aromatics Unadjusted	NS	90	3300	NA	3300	NA	NA
NA	C19-C36 Aliphatics	NS	9880	3300	NA	3300	NA	NA
NA	C9-C18 Aliphatics	NS	3170	3300	NA	3300	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
Method specific target compounds will be reported using SVOC Method 8270C
2 - Ecological PALs are from the following:
Battelle, 2007, "Sediment Toxicity of Petroleum Hydrocarbon Fractions," Prepared for Massachusetts Department of Environmental Protection, Office of Research and Standards, Boston, Massachusetts.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Method MADEP VPH
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: Volatile Petroleum Hydrocarbons (*VPH)*
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method MADEP VPH, L-8

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (µg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (µg/kg)	Eco PAL (µg/kg)		MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
NA	C5 – C8 Aliphatics Unadjusted	NS	1590	5000	NA	5000	NA	NA
NA	C5 – C8 Aliphatics	NS	1590	5000	NA	5000	NA	NA
NA	C9 – C10 Aromatics	NS	240	5000	NA	5000	NA	NA
NA	C9 – C12 Aliphatics Unadjusted	NS	NS	5000	NA	5000	NA	NA
NA	C9 – C12 Aliphatics	NS	NS	5000	NA	5000	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
Method specific target compounds will be reported using VOC Method 8260B.
2 - Ecological PALs are from the following:
Battelle, 2007, "Sediment Toxicity of Petroleum Hydrocarbon Fractions," Prepared for Massachusetts Department of Environmental Protection, Office of Research and Standards, Boston, Massachusetts.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: *Metals*
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7471A, L-2

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (mg/kg)[1]	Eco PAL (mg/kg)[2]		MDL (mg/kg)	QL (mg/kg)	MDL (mg/kg)	QL (mg/kg)
7429-90-5	Aluminum	NS	NS	2.5	0.194	2.5	NA	NA
7440-36-0	Antimony	NS	NS	0.5	0.137	0.5	NA	NA
7440-38-2	Arsenic	33	6	1	0.081	1	NA	NA
7440-39-3	Barium	NS	NS	0.5	0.017	0.5	NA	NA
7440-41-7	Beryllium	NS	NS	0.2	0.002	0.2	NA	NA
7440-43-9	Cadmium	5	0.6	0.2	0.0043	0.2	NA	NA
7440-70-2	Calcium	NS	NS	10	1.056	10	NA	NA
7440-47-3	Chromium	110	26	0.5	0.073	0.5	NA	NA
7440-48-4	Cobalt	NS	50	0.5	0.016	0.5	NA	NA
7440-50-8	Copper	150	16	1	0.078	1	NA	NA
7439-89-6	Iron	NS	20000	5	0.643	5	NA	NA
7439-92-1	Lead	130	31	0.5	0.044	0.5	NA	NA
7439-95-4	Magnesium	NS	NS	10	0.548	10	NA	NA
7439-96-5	Manganese	NS	460	1	0.096	1	NA	NA
7439-97-6	Mercury	0.18	0.174	0.1	0.045	0.1	NA	NA
7440-02-0	Nickel	49	16	1	0.014	1	NA	NA
7440-09-7	Potassium	NS	NS	200	53.3	200	NA	NA
7782-49-2	Selenium	NS	NS	0.5	0.272	0.5	NA	NA
7440-22-4	Silver	NS	0.5	0.5	0.045	0.5	NA	NA
7440-23-5	Sodium	NS	NS	100	5.89	100	NA	NA
7440-28-0	Thallium	NS	NS	1	0.191	1	NA	NA

EPA-NE QAPP Worksheet #9b- TAL Metals
Medium/Matrix: *Solid*
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (*SD)*
Analytical Parameter: *Metals*
Concentration Level: *Low/Medium*
Field Analytical or Fixed Laboratory Method/SOP: Method 6010B, 7471A, L-2

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

CAS Number	Analyte	Project Action Limit		Project Quantitation Limit (mg/kg)	Achievable Laboratory Limits		Analytical Method Limits	
		Human Health PAL (mg/kg)[1]	Eco PAL (mg/kg)[2]		MDL (mg/kg)	QL (mg/kg)	MDL (mg/kg)	QL (mg/kg)
7440-31-5	Tin	NS	NS	5	0.094	5	NA	NA
7440-62-2	Vanadium	NS	NS	1	0.015	1	NA	NA
7440-66-6	Zinc	460	120	2.5	0.134	2.5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/kg – micrograms per kilogram (parts per million)
Highlighted cells indicate PAL is less than Project Quantitation Limit.
1 - Human Health PALs are from the following:
MassDEP. 2006. Technical Update Revised Sediment Screening Values.
2 - Ecological PALs are the lowest from the following:
MacDonald, D.D., C.G. Ingersoll, T.A. Berger. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater systems. Archives of Environmental Contamination and Toxicology 39, 20-31. January 13, 2000.
Jones, D.S., G.W. Suter II, and R.N. Hull, 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Oak Ridge National Laboratories (ES/ER/TM-95/R4) November 1997.
Persaud, D., R. Jaagumagi, and A. Hayton. 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Ontario Ministry of Environment and Energy. ISBN 0-7778-9248-7. August 1993.
USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Hexavalent Chromium (Cr+6)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (SD)
Analytical Parameter(s): Hexavalent Chromium
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Method 3060A/7199, L-38

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (mg/kg)	Eco PAL (mg/kg)	Project Quantitation Limit (mg/kg)	MDL (mg/kg)	QL (mg/kg)	MDL (mg/kg)	QL (mg/kg)
18540-29-9	Hexavalent Chromium	NS	NS	2	0.111	2	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/kg – micrograms per kilogram (parts per million)

Prepared by: TC
Checked by: CR
4/24/09

EPA-NE QAPP Worksheet #9b- Ammonia, Chloride, Nitrate, Nitrite, Sulfate, and Total Organic Carbon (TOC)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (SD)
Analytical Parameter(s): Ammonia, Chloride, Nitrate, Nitrite, Sulfate, and Total Organic Carbon (TOC)
Concentration Level: *Low*
Field Analytical or Fixed Laboratory Method/SOP: Methods listed below. Refer to Worksheet #20

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Analysis Method	Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte		Human Health PAL (mg/Kg)	Eco PAL (mg/Kg)	Project Quantitation Limit (mg/Kg)	MDL (mg/Kg)	QL (mg/Kg)	MDL (mg/Kg)	QL (mg/Kg)
7664-41-7	Ammonia	QuikChem: LACHAT 10-107-06-1-A	NS	NS	5	TBD	5	NA	NA
E-10195	Total Organic Carbon	Lloyd Kahn	NS	NS	1000	TBD	1000	NA	NA
14797-55-8	Nitrate	EPA 300	NS	NS	5	TBD	5	NA	NA
14797-65-0	Nitrite	EPA 300	NS	NS	1	TBD	1	NA	NA
14808-79-8	Sulfate	EPA 300	NS	NS	40	TBD	40	NA	NA
16887-00-6	Chloride	EPA 300	NS	NS	20	TBD	20	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
mg/kg – micrograms per kilogram (parts per million)

Prepared by: TC
Checked by: CR
8/7/09

EPA-NE QAPP Worksheet #9b- Formaldehyde and Acetaldehyde
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (SD)
Analytical Parameter: Formaldehyde and Acetaldehyde
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: SW-846 Method 8315, L-32

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
50-00-0	Formaldehyde	NS	NS	100	78	100	NA	NA
75-07-0	Acetaldehyde	NS	NS	200	29	200	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)
Highlighted cells indicate PAL is less than Project Quantitation Limit.

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Hydrazine, MMH, UDMH
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment(SD)
Analytical Parameter: Hydrazine, Mono-methylhydrazine (MMH), Unsymmetrical dimethylhydrazine (UDMH)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Lancaster Laboratory Modified 8315, L-24

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
302-01-2	Hydrazine	NS	NS	2	0.5	2	NA	NA
60-34-4	MMH	NS	NS	5	2	5	NA	NA
57-14-7	UDMH	NS	NS	5	2	5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- Phthalic anhydride (acid)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (SD)
Analytical Parameter: Phthalic anhydride (acid)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified HPLC Method 8000, L-36

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
85-44-9	phthalic anhydride (acid)	NS	NS	100	18	100	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)
1 - Human Health PAL is from the following:

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N,N-dimethylformamide (DMF)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment(SD)
Analytical Parameter: N,N-dimethylformamide (DMF)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Modified Method 8033-GC/NPD, L-35

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)

		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
68-12-2	DMF	NS	NS	200	TBD	200	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/L – micrograms per liter (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9b- N-nitrosodimethylamine (NDMA)
Medium/Matrix: Solid
Region I Matrix Code (from EPA-NE DQO Summary Form): Sediment (SD)
Analytical Parameter: N-nitrosodimethylamine (NDMA)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: Method 3550/3546 / Modified EPA 8270 Low Level, L-6

Contaminants of Concern and Other Target Analytes Table (Reference Limit and Evaluation Table)								
		Project Action Limit			Achievable Laboratory Limits		Analytical Method Limits	
CAS Number	Analyte	Human Health PAL (µg/kg)	Eco PAL (µg/kg)	Project Quantitation Limit (µg/kg)	MDL (µg/kg)	QL (µg/kg)	MDL (µg/kg)	QL (µg/kg)
62-75-9	NDMA	NS	NS	5	1	5	NA	NA

Notes:
NS - None Specified, NA - Not Applicable, TBD – To be determined, MDL - Method Detection Limit, QL - Quantitation Limit
µg/kg– microgram per kilogram (parts per billion)

Prepared by: BR
Checked by: TC
10/14/08

EPA-NE QAPP Worksheet #9c:
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	MS/MSD		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks[1]	No. of PE Samples[2]	Total No. of Samples to Lab
						No. of MS	No. of MSD					
SW, GW	VOCs	Low/Medium	SW-846 8260B	TBD	5% of field samples	5% of field samples	5% of field samples	1 per cooler containing VOC samples	NA	5% of field samples	1 per event	TBD
SW, GW	SVOC	Low/Medium	SW-846 8270C	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	1 per event	TBD
SW, GW	Pesticides	Low/Medium	SW-846 8081	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	1 per event	TBD
SW, GW	PCBs	Low/Medium	SW-846 8082	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	1 per event	TBD
SW, GW	Herbicides	Low	SW-846 8151A	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	1 per event	TBD
SW, GW	EPH	Low	MADEP EPH-98-1	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SW, GW	VPH	Low	MADEP VPH-98-1	TBD	5% of field samples	5% of field samples	5% of field samples	1 per cooler containing VPH samples	NA	5% of field samples	NA	TBD
SW, GW	TAL Metals	Low/Medium	SW-846 6010B (ICP) / 6020A (ICP-MS)/ Hg by 7470A	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	1 per event	TBD
SW, GW	Hexavalent Chromium	Low/Medium	SW-846 7199	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD

EPA-NE QAPP Worksheet #9c:
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	MS/MSD		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks[1]	No. of PE Samples[2]	Total No. of Samples to Lab
						No. of MS	No. of MSD					
SW, GW	Alkalinity	Low	SM 2320B	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Ammonia	Low	QuikChem: LACHAT 10-107-06-1-A	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Chemical Oxygen Demand	Low	EPA 410.4	TBD	5% of field samples	NA	NA	NA	NA	NA	NA	TBD
SW, GW	Chloride	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Hardness	Low	SM 2340B	TBD	5% of field samples	NA	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Bromide	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Fluoride	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Nitrate	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Nitrite	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	pH	Low	SM 4500	TBD	5% of field samples	NA	NA	NA	NA	NA	NA	TBD

EPA-NE QAPP Worksheet #9c:
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	MS/MSD		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks[1]	No. of PE Samples[2]	Total No. of Samples to Lab
						No. of MS	No. of MSD					
SW, GW	Specific Conductance	Low	SM 2510B	TBD	5% of field samples	NA	NA	NA	NA	NA	NA	TBD
SW, GW	Sulfate	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Total Dissolved Solids (TDS)	Low	SM 2540C	TBD	5% of field samples	NA	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Total Organic Carbon	Low	SM 5310B	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Total Suspended Solids (TSS)	Low	SM 2540D	TBD	5% of field samples	NA	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Formaldehyde, Acetaldehyde	Low	SW-846 8315	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	Hydrazine, MMH and UDMH	Low	SW-846 Modified 8315	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SW, GW	phthalic anhydride (acid)	Low	Modified SW-846 8000 (Internal SOP - LC65)	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SW, GW	N,N-dimethylformamide (DMF)	Low	Modified 8033 - GC/NPD	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SW, GW	NDMA/NDPA	Low	Modified EPA 521	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD

EPA-NE QAPP Worksheet #9c:
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	MS/MSD		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks[1]	No. of PE Samples[2]	Total No. of Samples to Lab
						No. of MS	No. of MSD					
SW, GW	Nonylphenols	Low	Modified 8270 SIM	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SW, GW	Opex and Kempore	Low	Lab Specific HPLC method and SW-846 8000B	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
GW	Perchlorate	Low	SW-846 Method 6850	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SO,SD	VOCs	Low/Medium	SW-846 8260B	TBD	5% of field samples	5% of field samples	5% of field samples	1 per cooler containing VOC samples	NA	5% of field samples	1 per event	TBD
SO,SD	SVOC	Low/Medium	SW-846 8270C	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	1 per event	TBD
SO	PCBs	Low/Medium	SW-846 8082	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	1 per event	TBD
SO,SD	EPH	Low	MADEP EPH-98-1	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SO,SD	VPH	Low	MADEP VPH-98-1	TBD	5% of field samples	5% of field samples	5% of field samples	1 per cooler containing VPH samples	NA	5% of field samples	NA	TBD
SO,SD	TAL Metals	Low/Medium	SW-846 6010B (ICP) / 6020A (ICP-MS)/ Hg by 7471B	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	1 per event	TBD

EPA-NE QAPP Worksheet #9c:
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	MS/MSD		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks[1]	No. of PE Samples[2]	Total No. of Samples to Lab
						No. of MS	No. of MSD					
SO,SD	Hexavalent Chromium	Low/Medium	SW-846 7199	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SO, SD	Ammonia	Low	QuikChem: LACHAT 10-107-06-1-A	TBD	5% of field samples	5% of field samples	NA	NA	NA	NA	NA	TBD
SO, SD	Chloride	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	NA	NA	TBD
SO, SD	Sulfate	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	NA	NA	TBD
SO, SD	Nitrate	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	NA	NA	TBD
SO, SD	Nitrite	Low	EPA 300	TBD	5% of field samples	5% of field samples	NA	NA	NA	NA	NA	TBD
SO, SD	Total Organic Carbon	Low	Lloyd Kahn	TBD	5% of field samples	5% of field samples	NA	NA	NA	NA	NA	TBD
SO, SD	Formaldehyde, Acetaldehyde	Low	SW-846 8315	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SO, SD	Hydrazine, MMH and UDMH	Low	SW-846 Modified 8315	TBD	5% of field samples	5% of field samples	NA	NA	NA	5% of field samples	NA	TBD
SO, SD	phthalic anhydride (acid)	Low	Modified SW-846 8000 (Internal SOP - LC65)	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD

EPA-NE QAPP Worksheet #9c:
Field and Quality Control Sample Summary Table

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	MS/MSD		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks[1]	No. of PE Samples[2]	Total No. of Samples to Lab
						No. of MS	No. of MSD					
SO, SD	N,N-dimethylformamide (DMF)	Low	Modified 8033 - GC/NPD	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD
SO,SD	NDMA	Low	Modified 8270C – Low Level method	TBD	5% of field samples	5% of field samples	5% of field samples	NA	NA	5% of field samples	NA	TBD

1= Equipment blanks are not collected for grab samples, from tap locations, from surface water obtained directly from stream using only the sample bottle or from locations using dedicated sampling equipment.
2= PE Samples will be submitted with aqueous and soil samples at the beginning of the RI sampling program.
SW = Surface Water GW = Groundwater SD = Sediment SO = Soil MS = Matrix Spike MSD = Matrix Spike Duplicate TBD = To Be Determined

Prepared by: TC
Checked by: CR
10/14/08

Modified EPA-NE QAPP Worksheet #9d

Analytical Services Summary

Medium/Matrix	Analytical Parameter	Concentration Level	Analytical Method/ LAB SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address: Contact Person and Telephone Number)
SW, GW	VOC	Low/Medium	5030B/8260B / L-3	28 Days	Test America Laboratories (TAL)* – Westfield 53 Southampton Road Westfield, MA 01085 Becky Mason (413) 572-4000	Not Applicable
SW, GW	SVOC	Low/Medium	3510C/8270C/ L-4	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Pesticides	Low	3510 / 8081/ L-29	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	PCBs	Low	3510C/8082 / L-27	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Herbicides	Low	3535 / 8151A/ L-33	28 Days	Analytics Laboratory, LLC 195 Commerce Way Portsmouth, NH 1-800-929-9906 Steve Knollmeyer	Not Applicable
SW, GW	EPH	Low	MADEP EPH / L-25	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	VPH	Low	MADEP VPH / L-26	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Metals	Low/Medium	6010B (ICP) / 6020A (ICP-MS)/ Hg by 7470A/ L-1	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Hexavalent Chromium	Low/Medium	7199 / L-38	28 Days	Test America Laboratories – Edison, NJ 777 New Durham Rd # 7 Edison, NJ 08817 (732) 549-3900	Not Applicable
SW, GW	Alkalinity	Low/Medium	SM 2320B/ L-15	28 Days	Test America Laboratories – Westfield	Not Applicable

Modified EPA-NE QAPP Worksheet #9d

Analytical Services Summary

Medium/Matrix	Analytical Parameter	Concentration Level	Analytical Method/ LAB SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address: Contact Person and Telephone Number)
SW, GW	Ammonia	Low/Medium	QuikChem: LACHAT 10-107-06-1-A/ L-20	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Chemical Oxygen Demand (COD)	Low/Medium	410.4/ L-21	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Hardness	Low/Medium	SM 2340B/ L-16	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Nitrate	Low/Medium	300.0/ L-17	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Nitrite	Low/Medium	300.0/ L-18	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Fluoride	Low/Medium	300.0/ L-18	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Bromide	Low/Medium	300.0/ L-18	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	pH	Low/Medium	SM 4500/ L-15	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Specific Conductance	Low/Medium	SM 2510B/ L-15	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Sulfate	Low/Medium	300.0/ L-12	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Chloride	Low/Medium	300.0/ L-13	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Total Dissolved Solids (TDS)	Low/Medium	SM 2540C/ L-19	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Total Suspended Solids (TSS)	Low/Medium	SM 2540D/ L-23	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Total Organic Carbon (TOC)	Low/Medium	SM 5310B/ L-22	28 Days	Test America Laboratories – Westfield	Not Applicable
SW, GW	Formaldehyde / Acetaldehyde	Low	8315 / L-32	28 Days	Test America Laboratories – Tallahassee	Not Applicable

Modified EPA-NE QAPP Worksheet #9d

Analytical Services Summary

Medium/Matrix	Analytical Parameter	Concentration Level	Analytical Method/ LAB SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address: Contact Person and Telephone Number)
SW, GW	Hydrazine / MMH / UDMH	Low	SW-846 Modified 8315 / L-24	28 Days	Lancaster Labs, Inc. 2425 New Holland Pike Lancaster, PA (717) 656-2300	Not Applicable
SW, GW	Phthalic anhydride (acid)	Low	Modified 8000 – Internal SOP – Analysis by HPLC / L-36	28 Days	Test America Laboratories – Tallahassee, Florida	Not Applicable
SW, GW	N,N-dimethylformamide (DMF)	Low	Modified 8033 – GC/NPD / L-35	28 Days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, ME 04074 Tel: (207) 874-2400	Not Applicable
SW, GW	NDMA / NDPA	Low	Modified EPA 521/ L-5	28 Days	Test America Laboratories Sacramento - 880 Riverside Parkway, West Sacramento, CA 95605. Tel. (916) 373-5600	Not Applicable
SW,GW	Nonylphenol (technical mix), Nonylphenol Diethoxylate (technical mix), 4-nonlyphenyl, Nonphenol isomers	Low	Modified 8270 SIM / L-34	28 Days	Test America Laboratories – West Sacramento, California	Not Applicable
SW, GW	Opex / Kempore	Low	Method 8000B HPLC / L-11	28 Days	Lancaster Labs, Inc. 2425 New Holland Pike Lancaster, PA (717) 656-2300	Not Applicable
GW	Perchlorate	Low	SW-846 Method 6850 / L-37	28 Days	Lancaster Labs, Inc.	Not Applicable
SO, SD	VOC	Low/Medium	5035/8260B / L-3	28 Days	Test America Laboratories – Westfield	Not Applicable

Modified EPA-NE QAPP Worksheet #9d

Analytical Services Summary

Medium/Matrix	Analytical Parameter	Concentration Level	Analytical Method/ LAB SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address: Contact Person and Telephone Number)
SO, SD	SVOC	Low/Medium	3550/8270C/ L-4	28 Days	Test America Laboratories – Westfield	Not Applicable
SO	PCBs	Low	3550B / 8082/ L-28	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	EPH	Low	MADEP EPH / L-7	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	VPH	Low	MADEP VPH / L-8	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Metals	Low/Medium	6010B (ICP) / Hg by 7471B/ L-2	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Hexavalent Chromium	Low/Medium	3060A/7199 / L-38	28 Days	Test America Laboratories – Edison, NJ	Not Applicable
SO, SD	Ammonia	Low/Medium	QuikChem: LACHAT 10-107-06-1-A/ L-20	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Nitrate	Low/Medium	300.0/ L-17	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Nitrite	Low/Medium	300.0/ L-18	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Chloride	Low/Medium	300.0/ L-13	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Sulfate	Low/Medium	300.0/ L-12	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Total Organic Carbon (TOC)	Low/Medium	Lloyd Kahn/ L-39	28 Days	Test America Laboratories – Westfield	Not Applicable
SO, SD	Formaldehyde / Acetaldehyde	Low	8315 / L-32	28 Days	Test America Laboratories – Tallahassee	Not Applicable
SO, SD	Hydrazine / MMH / UDMH	Low	SW-846 Modified 8315 / L-24	28 Days	Lancaster Labs, Inc.	28 Days

Modified EPA-NE QAPP Worksheet #9d

Analytical Services Summary

Medium/Matrix	Analytical Parameter	Concentration Level	Analytical Method/ LAB SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address: Contact Person and Telephone Number)
SO, SD	Phthalic anhydride (acid)	Low	Modified 8000 – Internal SOP – Analysis by HPLC / L-36	28 Days	Test America Laboratories – Tallahassee, Florida	Not Applicable
SO, SD	N,N-dimethylformamide (DMF)	Low	Modified 8033 – GC/NPD / L-35	28 Days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, ME 04074 Tel: (207) 874-2400	Not Applicable
SO, SD	NDMA	Low	Modified 8270C Low Level Method / L-6	28 Days	Test America Laboratories – Westfield	Not Applicable

NOTES:
* = Test America Laboratories was formerly Severn Trent Laboratories (STL).
SW = Surface Water, GW= Groundwater, SO=Soil, SD= Sediment
VOC = Volatile Organic Compounds, MMH = Mono-methylhydrazine,UDMH = Unsymmetrical dimethylhydrazine
SVOC = Semivolatile Organic Compounds, ETPH = Extractable Total Petroleum Hydrocarbons, PCB = Polychlorinated Biphenyls

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #10
Project Schedule Timeline Table

Activities	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
	Anticipated Date(s) of Initiation	Anticipated Date of Completion		
RI Work Plan (Draft) Preparation	March 2008	October 13, 2008	RI Work Plan - Draft	October 13, 2008
RI Work Plan (Final) Preparation	January 1, 2009	April 30, 2009	RI Work Plan - Final	April 30, 2009
2009 RI Field Work	06/30/2009	12/2009	Not Applicable	NA
RI/FS ** Report Preparation	TBD	TBD	RI/FS Report	TBD

* Includes Screening Level Ecological Risk Assessment
** Includes Baseline Human Health and Ecological Risk Assessments
TBD – To Be Determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
04/23/09

7.0 PROJECT QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITERIA

This section provides an overview of the environmental decisions that need to be made and the level of data quality needed to ensure that these decisions are based on sound scientific data. Project quality objectives and measurement performance criteria presented in this section will cover work performed at the Olin Chemical Superfund Site. Additional details of site specific activities will be presented in Field Sampling and Analysis Plan or Task-specific work plans.

7.1 PROJECT QUALITY OBJECTIVES

As discussed in Section 6.1, the primary objectives of the investigation are to complete a RI/FS, to further define the nature, extent, and distribution of site-related contaminants, to determine if these contaminants present a human health or ecological risk, and to fill in any data gaps which may be present based on a review of historical data presented in the Draft Focused Remedial Investigation Report (MACTEC, 2007). The sampling and analysis program outlined in the FSP and QAPP Worksheets #9b, #9c, and #9d, will provide analytical chemistry data to support these objectives.

Project quality objectives (PQOs) include planning the collection of chemical data on a variety of parameters, and the identification of target analytes, concentration levels, media to be sampled, analysis methods, QA/QC needs, and appropriate sampling techniques. These are detailed on Worksheets #9a, #9b, and #9c, and in Section 9.0. The quantity of data needed to meet the PQOs, including the number of samples for each analytical parameter of each media is detailed in the FSP. To ensure sample integrity, sample handling protocols are established on Worksheet #12b. The quality of data needed to achieve the PQOs is described using data quality indicator goals (precision, accuracy, representativeness, comparability, completeness, selectivity, and sensitivity) required of each analytical parameter used for each media sampled. The limits set on each of these items are referred to as measurement performance criteria and define the quality of data generated. Measurement performance criteria presented in Section 7.2 have been established for each parameter in order to ensure the data are sound, highly defensible, and with low enough quantitation limits to support human health or ecological risk assessments.

The target analytes are listed on Worksheet #9b for each analytical parameter. Worksheet #9b includes MDLs, project QLs and associated PALs for each target analyte. The worksheet has been completed for each matrix and each parameter. The PAL values were obtained from the following sources:

Groundwater PALs

The groundwater PAL for each chemical is the minimum value from the following sources:

Human

- USEPA. 2009. Regional Screening Levels for Chemical Contaminants. Tap Water. May 19;
- Federal Maximum Contaminant Level. (USEPA, 2003. National Primary Drinking Water Standards. Office of Water. EPA 816-F-03-016. June.);
- Target Groundwater Concentration Corresponding to Target Indoor Air Concentration. (USEPA, 2002. Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). EPA530-D-02-004. Table 2C. November.);
- Massachusetts Maximum Contaminant Level. (MassDEP, 2008. Standards and Guidelines for Contaminants in Massachusetts Drinking Water. Massachusetts Maximum Contaminant Levels. Spring.);
- Massachusetts Secondary Maximum Contaminant Level. (MassDEP, 2008. Standards and Guidelines for Contaminants in Massachusetts Drinking Water. Secondary Maximum Contaminant Levels. Spring.);
- Massachusetts Drinking Water Guidelines. (MassDEP, 2008. Standards and Guidelines for Contaminants in Massachusetts Drinking Water. Massachusetts Drinking Water Guidelines. Spring.);
- Massachusetts Contingency Plan GW-1 Standards. (MassDEP, 2008. Massachusetts Contingency Plan. 310 CMR 40.0974(2) Table 1, GW-1 Standards. February.); and
- Massachusetts Contingency Plan GW-2 Standards. (MassDEP, 2008. Massachusetts Contingency Plan. 310 CMR 40.0974(2) Table 1, GW-2 Standards. February.).

Ecological

- Massachusetts Contingency Plan GW-3 Standards. (MassDEP, 2008. Massachusetts Contingency Plan. 310 CMR 40.0974(2) Table 1, GW-3 Standards. February.)

Surface Water PALs

The surface water PAL for each chemical is the minimum value from the following sources:

Human

- USEPA. 2006. National Recommended Water Quality Criteria. Office of Water and Office of Science Technology. Freshwater CCC. 2006.

Ecological

- USEPA. 2006. National Recommended Water Quality Criteria. Office of Water and Office of Science Technology. Freshwater CCC. 2006;

- Massachusetts Department of Environmental Protection (MassDEP) 2006. Development of GW-3 Standards Using AQUIRE Data, 2006 Update. Update to Background Documentation for the Development of the MCP Numerical Standards. Bureau of Waste Site Cleanup and Office of Research and Standards;

- Suter, G.W, and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects of Aquatic Biota: 1996 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-96/R2. June 1996; and

- USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

Soil PALs

The soil PAL for each chemical is the minimum value from the following sources:

Human

- USEPA. 2009. Regional Screening Levels for Chemical Contaminants. Residential Soil. May 19;

- USEPA. 2009. Regional Screening Levels for Chemical Contaminants. Industrial Soil. May 19;

- Massachusetts Contingency Plan S-1 Standards. (MassDEP, 2008. Massachusetts Contingency Plan. 310 CMR 40.0974(2) Table 5, S-1 Standards. February.);

- Massachusetts Contingency Plan S-2 Standards. (MassDEP, 2008. Massachusetts Contingency Plan. 310 CMR 40.0974(2) Table 5, S-2 Standards. February.); and

- Massachusetts Contingency Plan S-3 Standards. (MassDEP, 2008. Massachusetts Contingency Plan. 310 CMR 40.0974(2) Table 5, S-3 Standards. February.).

Ecological

- USEPA ECO-SSL – Ecological Soil Screening Levels. (USEPA. 2005. ECO-SSL - Ecological Soil Screening Levels. Interim Final. Office of Emergency and Remedial Response, Washington, DC: November, 2003. Revised February, 2005.);

- Efroymson, R.A, M. E. Will, G. W. Suter II, and A. C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Prepared for the U.S. Department of Energy. ES/ER/TM-85/R3. November 1997;

- Efroymson, R., M.E. Will, and G.W. Suter. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-126/R2. November 1997;

- Sample, B.E., and D.M. Opresko, G.W. Suter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Prepared for U.S. Department of Energy. ES/ER/TM-86/R3. June 1996; and

- USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

<u>Sediment PALs</u>

The sediment PAL for each chemical is the minimum value from the following sources:

- MassDEP, 2006. Technical Update Revised Sediment Screening Values;

- MacDonald, D. D., C. G. Ingersoll, T. A. Berger. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater systems. Archives of Environmental Contamination and Toxicology 39, 20-31. January 13, 2000;

- Jones, D.S., G.W. Suter II, and R.N. Hull, 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision; Oak Ridge National Laboratories (ES/ER/TM-95/R4); November 1997;

- Persaud, D., R. Jaagumagi, and A. Hayton, 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario; Ontario Ministry of Environment and Energy; ISBN 0-7778-9248-7; August, 1993; and

- USEPA. 2003. USEPA, Region V, RCRA Ecological Screening Levels. August 22.

For analysis of VOCs, SVOCs, PAHs, pesticides, PCBs, metals, herbicides, formaldehyde and wet chemistry parameters, routine SW-846 methods have been chosen. For VOCs, SVOCs, and pesticides the USEPA Contract Laboratory Program (CLP) OLM4.3 Target Compound List (TCL) with additional compounds from the MCP (MEDEP, 2004) will be reported by laboratories performing the analyses. MCP compounds added to the TCL list have been marked with an asterisk on Worksheet #9b. Special laboratory methodologies have been identified for the analysis of hydrazine, Opex, Kempore,

nonylphenols, phthalic anhydride, dimethylformamide (DMF), and NDMA/NDPA. The objective for the analysis for these compounds is to determine the presence or absence in media (surface water, groundwater, soil, sediment) and determine if they are COPCs.

In general, the proposed analytical methodologies are equivalent to CLP Methods and SW-846 Methods that are routinely used at CERCLA sites. These methods provide trace level analysis (low part per billion and part per trillion) for the majority of analytes and achieve QLs that are equal to, or less than, the PAL for most target analytes. An additional low detection limit method has been added for NDMA and NDPA because they are important site contaminants. Based on historical information, additional non-routine analytical methods have been added to the analytical scope to cover an expanded list of target analytes.

For some analytes, PAL concentrations are less than QLs provided by the analytical method. PALs which are less than QLs are highlighted in Worksheet #9b and summarized on Table 7.1. Laboratories will report QLs as low as technically possible and will report detected analytes as estimated values between the MDL and QL. Those analytes that have MDLs that are less than PQLs are identified on Table 7.1. The primary COPCs at the site are VOCs, SVOCs, metals and inorganics. The proposed analytical methods for VOCs, SVOCs, and metals provide low concentration detection limits for target analytes. In many cases where the QL is greater than the PAL, the analytical MDL is below the PAL, and the laboratory will report detections at or below PQLs. For some target analytes, it is not technically possible to obtain QLs/MDLs that are less than PALs using the VOC, SVOC, or metals routine SW-846 methods. These analytes are discussed in the following sections for soil, groundwater, surface water, and sediment media.

Some of the PALs are based on very conservative guidance values which many not be values actually used in the baseline human health or ecological risk assessments. The PALs have been identified from the universe of potentially applicable or relevant human health and ecological screening values and regulatory standards and guidance values. During the human health and ecological risk assessments, site-specific decisions will be made concerning the appropriate screening levels, and the actual remedial requirements may be based on risk calculations, ARARs, or a mixture of those. Therefore, the screening values, regulatory standards, and guidance values used to identify PALs in Worksheet 9b do not necessarily represent preliminary remediation goals or remedial requirements for the Site. They will be used in the data screening and evaluation process during preliminary risk assessment to identify COPCs and the most appropriate values will be based on a site-specific evaluation.

Groundwater

VOCs

The following compounds have project action limits that are lower than the laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- 1,1,2,2-tetrachloroethane
- 1,1,2-trichloroethane
- 1,2,3-trichloropropane
- 1,2-dibromo-3-chloropropane
- 1,2-dibromoethane
- 1,2-dichloroethane
- 1,2-dichloropropane
- 1,4-dichlorobenzene
- Benzene
- Carbon tetrachloride
- Chloroform
- Dibromochloromethane
- Naphthalene
- Tetrachloroethene
- 1,4-dioxane
- Vinyl Chloride

The compounds 1,2-dibromo-3-chloropropane and 1,2-dibromoethane are added to the VOC list because they are listed as MADEP MCP analytes (MADEP, 2004). They are not on the EPA CLP TCL. The MCP indicates that detection limits for Method 8260B may not achieve regulatory compliance. These compounds have not been identified as contaminants of concern based on historical data. No additional analyses that obtain lower detection limits are planned for these compounds. Results will be reported to the best attainable detection limits using Method 8260B.

The compound 1,4-dioxane is on both the MADEP MCP Method 8260B list (MADEP, 2004) and the EPA CLP TCL. The MCP method identifies a groundwater standard of 50 µg/L. A human health PAL of 3 µg/L is identified in Worksheet 9b. This compound has not been identified as a contaminant of

potential concern based on historical data. No additional analyses are planned for this compound. Results will be reported to the best attainable detection limits using Method 8270C.

For the remaining VOCs listed above, the detection limits provided by the Method 8260B in the QAPP are equivalent what is routinely reported using EPA CERCLA low detection limits procedures. VOCs will be reported to the lowest achievable detection limits.

SVOCs

The following SVOC compounds have PALs that are lower than the laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- 1,1-biphenyl
- 1-methylnaphthalene
- 2,4-Dinitrotluene
- 3,3-dichlorobenzidine
- 4-Chloroaniline
- Atrazine
- Azobenzene
- Hexachlorobenzene
- Hexachlorocyclopentadiene
- Nitrobenzene
- PAH (benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, dibenz(a,h)anthracene, indeno(1,2,3-cd)pyrene, naphthalene)

The human health PAL for 1,1-biphenyl (0.9 µg/L) is less than the quantitation limit of 5 µg/L provided by the laboratory. This compound is added to the project list because it is on the CLP TAL. The laboratory does not routinely include 1,1-biphenyl on the Method 8270 target list, and MDLs have not been established. Results for 1,1-biphenyl will be reported to the best attainable detection limits using Method 8270C.

The low concentration Method 8270C analysis has been developed by TAL that allows the reporting of PAH compounds at sub-ppb concentrations. Some of the groundwater quality PALs are lower than the detection limits in the TAL method. PAH compounds are not identified a primary site contaminants and

PAH compounds will be reported to the best attainable detection limits using the low concentration Method 8270C procedure.

The remaining compounds have been added to the SVOC target compounds list to cover the full list of target analytes identified for Method 8270. Results will be reported to the best attainable detection limits.

Pesticides and PCBs

The following pesticide compounds have PALs that are lower than the laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- Aldrin
- Dieldrin
- Heptachlor epoxide
- Toxaphene
- PCBs

Routine Method 8081 and Method 8082 analytical procedures have been included in the QAPP because pesticide and PCB parameters are included in the full suite CLP methods that are routinely employed at CERCLA sites. Detection limits in the Method 8081 and Method 8082 analytical procedures are at a similar concentration range as those reported the CLP. Pesticide and PCB analytes will be reported to the lowest achievable detection limits.

Metals

Arsenic has project action limit (human health) of 0.045 µg/L, which is below the RL of 1 µg/L and MDL of 0.397 µg/L (by Method 6020A). Results for arsenic will be reported to the best attainable detection limits using Method 6020A. No additional analyses that obtain lower detection limits are planned for arsenic.

Acetaldehyde

Acetaldehyde will be analyzed using HPLC Method 8315. MDLs will be determined prior to sample analysis. It is anticipated that the MDL will be greater than the PAL and that results will be reported to the best achievable detection limits.

Hydrazine

Hydrazine will be analyzed using a modified Method 8315 LC/MS/MS procedure that obtains very low detection limits with an MDL (0.05 µg/L) that is slightly higher than the PAL of 0.022 µg/L. This method offers a good approach for obtaining low detection limits with highly reliable results.

NDMA/NDPA

USEPA modified Method 521 will be used to analyze groundwater samples for NDMA and NDPA. This method is a dual quadrapole mass spectrometry (GC/MS/MS) procedure that obtains the lowest possible detection limits for these parameters. The method modification includes substituting solid phase extraction with USEPA Method 3510C. A project detection limit goal of 0.002 µg/L for NDMA has been used during previous investigations for this project. The PAL (0.00042 µg/L) in this QAPP is based on new regional screening values from the USEPA Region 1 guidelines (USEPA, 2008). The NDMA MDL (0.00074 µg/L) is slightly higher than the PAL. NDMA will be reported to the lowest achievable detection limit using Modified EPA Method 521. The PAL for NDPA (0.0096 µg/L) in this QAPP is based on new regional screening values from the USEPA Region 1 guidelines (USEPA, 2008). NDPA will be reported to the MDL (0.00066 µg/L) as indicated in Table 7.1.

Surface Water

VOCS

The following compounds have PALs that are lower than the laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- hexachlorobutadiene

This compound will be reported to the lowest achievable detection limit.

SVOCs

The following compounds have PALs that are lower than the laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- 1-methylnaphthalene
- hexachlorobenzene

- hexachlorobutadiene
- PAH - (acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, chrysene, dibenz(a,h)anthracene, fluorene, indeno(1,2,3-cd)pyrene, and pyrene)

The compounds hexachlorobenzene and hexachlorobutadiene are not identified as site contaminants in groundwater or soils for this investigation based on the Draft FRI Report (MACTEC, 2007), and results for these compounds will be reported to the best attainable detection limits using the low concentration Method 8270C described in the QAPP.

The low concentration Method 8270C analysis has been developed by TAL that allows the reporting of PAH compounds at sub-ppb concentrations. Some of the surface water quality PALs are lower than the detection limits in the TAL method. PAH compounds are not identified a primary site contaminants and PAH compounds will be reported to the best attainable detection limits using the low concentration Method 8270C procedure.

Pesticides

Very low human health and ecological benchmarks limits were identified for a subset of pesticides and PCBs in surface water. The following compounds have PAL concentrations less that method detection limits:

- 4,4-DDT
- 4,4-DDD
- 4,4-DDE
- Dieldrin
- Endrin
- Heptachlor
- Heptachlor epoxide
- Toxaphene

Surface water sampling is not currently planned for pesticides or PCBs during the RI program because these parameters have not been identified as significant site contaminants during past investigations. Routine Method 8081 and Method 8082 analytical procedures have been included in the QAPP because pesticide and PCB parameters are included in the full suite CLP methods that are routinely employed at CERCLA sites. Detection limits in the Method 8081 and Method 8082 analytical procedures are at a

similar concentration range as those reported the CLP. These pesticide and PCB methods are included in this QAPP for future reference in the event the pesticide or PCB is considered in the future.

EPH

The PAL for aromatic fractions (5 µg/L) is less than the reporting limits of the MADEP EPA method. The published MADEP method identifies reporting limits of 100 µg/L for each fraction. The method quantitation limit identified by TAL is also 100 µg/L. The PAL is based on MCP GW-3 criteria. Results for the aromatic fractions will reported to the detection limits identified in the MADEP EPH method.

Metals

The following metals have project action limits that are lower than the Method 6010B laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- cadmium
- copper
- silver
- mercury

Copper and silver will be analyzed using Method 6020 ICP-MS to obtain the lowest achievable detection limits for these parameters. Detection limits for copper and lead are less than PALs using the Method 6020 procedure. For silver and cadmium, neither method provides detection limits less than the PAL. Results for silver will be reported to the best attainable detection limits using Method 6020. The detection limits reported for cadmium are lower for Method 6010B ICP-AES procedure and results for cadmium will be reported to the best attainable detection limits using Method 6010B.

Analysis for mercury will be completed using Method 7470A. This method is designed to evaluate sub-ppb concentrations and is the routine method used for aqueous analysis. This method provides detection limits below the PAL identified for human health, but does not obtain detection limits down to the ecological PAL. Mercury has not been identified as a site contaminant for soil or groundwater, and results will be reported to the lowest detection limit possible using Method 7470A.

Soil

NDMA

A human health PAL of 2.3 µg/kg and an ecological PAL of 0.0321 µg/kg are identified for NDMA. The method for NDMA in soil is a modified low concentration Method 8270 procedure developed by TAL. MDLs are not currently available and will be determined prior to the RI program. The method achieves a QL of 5 µg/kg. The MDL reported by TAL when they developed the method was 0.79 µg/kg and an estimated MDL of 1 µg/kg is included in the QAPP. NDMA will be reported to the lowest attainable detection limits using the modified Method 8270 procedure.

Metals

The following metals have PALs that are lower than the Method 6010B laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- cadmium
- selenium
- thallium

For cadmium, selenium, and thallium Method 6010B will be used to provide the lowest possible detection limits. Because of matrix interference that often occurs with Method 6020 in soil, this method has not been included for these analytes. Results will be reported to the best attainable detection limits using Method 6010B.

Sediment

VOCs

The following compounds have PALs that are lower than the laboratory QLs and MDLs attained by the laboratory for Method 8260B:

- acetone

The ecological PAL listed for acetone (8.7 µg/kg) is less than the detection limits provided in the TAL method. Acetone is a common field and laboratory contaminant (USEPA, 1996) and evaluation of this compound in the low ppb range is difficult due to possible field and laboratory contamination. Because the lab MDL is only slightly higher than the PAL, this compound will be reported to the best attainable detection limits using Method 8260B.

SVOCs

The following compounds have ecological PALs that are lower than the laboratory QLs and MDLs attained by the laboratory and summarized in Worksheet 9b:

- aniline
- Benzyl alcohol
- PAH - acenaphthylene, 2-methylnaphthalene

Aniline is added to the project SVOC list because it is listed as an MADEP MCP analyte (MADEP, 2004). The MCP method does not identify standards or detection limit requirements for this compound. Results for aniline will be reported to the best attainable detection limits using Method 8270C.

Benzyl alchol was added to the project SVOC list by USEPA. Results for benzyl alcohol will be reported to the best attainable detection limits using Method 8270C.

The low concentration Method 8270C analysis has been developed by TAL that allows the reporting of PAH compounds to low ppb concentrations. For acenaphthylene and 2-methylnaphthalene the sediment ecological PALs are slightly lower than the MDLs provided in the TAL method. PAH compounds were not identified in previous investigations as primary site contaminants and these PAH compounds will be reported to the best attainable detection limits using the low concentration Method 8270C procedure.

VPH and EPH

The PAL for VPH C5-C9 aliphatic and C9-C10 aromatic fractions are less than the reporting limits of the MADEP method. The published MADEP method and the TAL procedure described in Worksheet 9b identify reporting limits of 5000 µg/kg for each fraction. Ecological PALs for C5-C9 aliphatic (1590 µg/kg) and C9-C10 aromatic fractions (240 µg/kg) are listed based on MADEP guidelines. Results for the aromatic fractions will reported to the detection limits identified in the MADEP VPH method.

The PAL for EPH C9-C18 aliphatic and C11-C22 aromatic fractions are less than the reporting limits of the MADEP method. The published MADEP method identifies reporting limits of 20,000 µg/kg for each fraction. The TAL procedure described in Worksheet 9b lists a quantitation limit of 3300 µg/kg. Ecological PALs for C9-C18 aliphatic (3170 µg/kg) and C11-C22 aromatic fractions (90 µg/kg) are listed based on MADEP guidelines. Results for the aliphatic and aromatic fractions will reported to the detection limits identified in the MADEP VPH method.

7.2 MEASUREMENT PERFORMANCE CRITERIA

The Olin Wilmington Site Remedial Investigation QA/QC program is designed to produce data of the quality necessary to achieve PQOs and meet or exceed the minimum standard requirements for field and analytical methods. The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting which will provide results that are scientifically valid, and the levels of which are sufficient to meet PQOs. Specific procedures for sampling, chain of custody, laboratory and field instruments calibration, laboratory analysis, reporting of data, internal quality control, preventative maintenance of field and laboratory equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to state the specific, required QA objectives for accuracy, precision, representativeness, completeness, sensitivity, selectivity, and comparability for analytical chemistry methods.

Measurement performance criteria for precision, accuracy/bias, representativeness, completeness, sensitivity, quantitation limits, selectivity, and comparability have been established for each matrix and parameter and are summarized in Worksheet #11. A summary of analytical method QC limits for accuracy and precision are also summarized on Table 7.2 and Table 7.3. These measures of performance are also referred to as Data Quality Indicators (DQIs). DQIs are used to evaluate data usability during data validation activities described in Section 18.

7.2.1 Precision

Precision is described as the agreement among a set of duplicate or replicate measurements. Precision is measured using relative percent difference (RPD) for two data points.

7.2.2 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates (one extra sample in addition to the original field sample). Field duplicates will be collected at a frequency of one per 20 investigative samples per matrix/media per analytical parameter. Precision will be measured through the calculation of RPD. The resulting information will be used to assess sample homogeneity, spatial variability at the site, sample collection reproducibility, and analytical variability. Field duplicate goals

are RPDs ≤30 for aqueous samples and ≤50 for solid samples, as per the EPA-NE Data Validation guidelines.

7.2.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of RPD for laboratory duplicate samples (two samples from the same container) and/or matrix spike (MS), matrix spike duplicate (MSD) samples. Laboratory precision measures both sample preparation and analysis reproducibility. Precision control limits are provided in Worksheet #11.

For the organic analyses of soil, sediment, ground water, and surface water, laboratory precision will be assessed through the analysis of MS/MSD samples. MS/MSD samples will be performed at a frequency of one per twenty investigative samples for each media. For the inorganic analyses of soil, sediment, surface water, groundwater, laboratory precision will be assessed through the analysis of laboratory duplicate samples. Laboratory duplicate samples will be performed at a frequency of one per twenty investigative samples.

7.2.4 Accuracy

Accuracy is a measure of agreement between an observed value and an accepted reference value. Accuracy of chemistry data is evaluated using laboratory control samples (LCS), matrix spikes, surrogate spikes, and performance evaluation (PE) samples.

7.2.5 Field Accuracy Objectives

Accuracy in the field is achieved through the adherence to established field instrument calibration and sample collection procedures, sample handling, preservation, and holding time requirements. Accuracy will also be evaluated through the use of field blanks, trip blanks, equipment blanks, and cooler temperature blanks as described in Section 13.

7.2.6 Laboratory Accuracy Objectives

Laboratories assess the overall accuracy of their instruments and analytical methods (independent of sample or matrix effects) through the measurement of QC samples including calibration standards,

method blanks, instrument blanks, PE samples, and LCS. The percent recovery of surrogate compounds in organic analyses, and percent recovery of spiked compounds in MS analyses are also used to evaluate effects of sample matrix on analytical accuracy. Accuracy control limits are provided in Worksheet #11. The laboratory method blanks are also analyzed to evaluate possible adverse effects of sample contamination from an outside source (i.e., sample preparation or sample analysis) and could result in a positive or negative bias.

7.2.7 Representativeness

Representativeness is a qualitative parameter which expresses the degree to which the data and sampling design accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and the laboratory quality control program. Sampling design is described in the FSP. Formal procedures for sample collection and analysis are established with the goal of obtaining data that represent physical and chemical conditions.

7.2.8 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP and sampling SOPs are followed and that proper sampling, sample handling, and sample preservation techniques are used. Representativeness may also be assessed by the use of field duplicate samples. By definition, field duplicate samples are collected so they are equally representative of a given point in space and time. In this way, they provide both precision and representativeness information.

In general, representativeness in the field will be maximized by following SOPs, proper sample homogenization procedures, proper sample preservation procedures, utilizing experienced/trained sampling crews, and conducting field audits.

7.2.9 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, appropriate methods, and meeting sample holding times. Following the detailed requirements outlined in the EPA

methods, SW-846 methods and/or lab developed analytical methods/SOPs will maximize the representativeness of the laboratory data.

The program has also been designed to ensure the proper handling of sediment and soil samples with potentially high moisture. The MACTEC field sample manager will allow all sediment samples to settle for at least 2 hrs prior to shipment. If a clear layer of water forms above the soil matrix, it will be decanted. The off-site laboratory will also be instructed to decant any water layer that is observed prior to sample preparation. The aliquot used for percent moisture determination will be collected after the sample is decanted and homogenized in the sample container. If a moisture content $\geq$70% is determined, the laboratory will take corrective actions (centrifuge sample) and the decanting and homogenization process will be done again. The decant procedure will not be used for sediment or soil samples submitted for VOC analysis. Sediment and/or soil collected for VOCs will be preserved in the field with DI water or methanol depending on the analysis level (i.e. low or medium). A separate container will be collected for VOC soil and sediment percent solids determination. The aliquot used for percent moisture determination for VOC sediment/soil samples will be collected prior to decanting of water from the sample.

7.2.10 Comparability

Comparability is a qualitative parameter that expresses the confidence with which one data set can be compared to another.

7.2.11 Measures to Ensure Field Comparability

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP, QAPP, and SOPs are followed, and that proper sampling and preservation techniques are used.

7.2.12 Measures to Ensure Laboratory Comparability

Comparability is dependent on the use of EPA methods, SW-846 methods and/or lab developed analytical methods/SOPs, the reporting of data in standardized units, and acceptable PE sample results. PES samples will be submitted to laboratories performing VOC, SVOC, PCB, pesticide, herbicide, and metals

analyses on Olin samples. The PES results will be used to evaluate lab data comparability to know concentrations in soil and water media.

7.2.13 Sensitivity

Sensitivity is the ability of the instrument or method to detect the contaminants of concern at the level of interest. Method and instrument sensitivity will be evaluated through the use of MDL studies for all analyses. QLs will be established for each analyte. QL and MDLs are used to establish method sensitivity. Worksheet 9b presents QL and MDL data, and provides a comparison to USEPA and MADEP water and soil standards and guidelines that will be used to evaluate contamination during the RI.

7.2.14 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. "Normal conditions" are defined as the conditions expected if the sampling plan was implemented as planned.

7.2.15 Field Completeness Objectives

Field completeness is a measure of the amount of (1) valid measurements obtained from all the measurements taken in the project and (2) valid samples collected. The field completeness objective is greater than 90 percent. This allows for the potential loss of samples due to sampling problems or bottle breakage during transport.

7.2.16 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all valid samples submitted to the laboratory. The laboratory completeness objective is greater than 95 percent. This allows for the potential loss of samples impossible to analyze due to unforeseen interferences and rejected data following data validation.

TABLE 7.1
ANALYTE DETECTION LIMIT REVIEW

Media	Parameter	Analyte	PAL-HH	PAL-eco	Project QL	MDL	RL	Report MDL
GW	VOC	1,1,2,2-Tetrachloroethane	0.067	50000	0.5	0.21	0.5	
		1,1,2-Trichloroethane	0.24	50000	1	0.72	1	
		1,2,3-Trichloropropane	0.0096	NS	1	0.65	1	
		1,2-Dibromo-3-chloropropane	0.00032	NS	5	0.58	5	
		1,2-Dibromoethane	0.0065	50000	1	0.54	1	
		1,2-Dichloroethane	0.15	20000	1	0.62	1	
		1,2-Dichloropropane	0.39	50000	1	0.51	1	
		1,4-Dichlorobenzene	0.43	8000	1	0.62	1	
		1,4-Dioxane	3	NS	50	7.7	50	
		Benzene	0.41	10000	1	0.74	1	
		Carbon Tetrachloride	0.13	5000	1	0.71	1	
		Chloroform	0.19	10000	1	0.66	1	
		Dibromochloromethane	0.15	50000	0.5	0.5	0.5	
		Naphthalene	0.14	20000	5	2	5	
		Tetrachloroethene	0.11	30000	1	0.48	1	
		Vinyl Chloride	0.016	50000	0.5	0.46	0.5	
	SVOC	1-Methylnaphthalene	2.3	NS	TBD	TBD	TBD	
	SVOC	1,1-Biphenyl	0.9	50000	5	TBD	5	
		1,4-Dichlorobenzene	0.43	8000	5	0.286	5	x
		3,3'-Dichlorobenzidine	0.15	2000	5	0.174	5	
		2,4-Dinitrotoluene	0.22	50000	5	0.238	5	
		4,6-Dinitro-2-Methylphenol	3.7	NS	5	0.25	5	x
		4-Chloroaniline	0.34	300	5	0.463	5	x
		4-Nitroaniline	3.4	NS	5	0.223	5	x
		Atrazine	0.29	NS	5	TBD	5	
		Azobenzene	0.12	NS	5	0.18	5	
		Benzo(a)anthracene	0.029	1000	0.3	0.3	0.3	
		Benzo(a)pyrene	0.0029	500	0.2	0.2	0.2	
		Benzo(b)fluoranthene	0.029	400	0.3	0.253	0.3	
		Benzo(k)fluoranthene	0.29	100	0.3	0.128	0.3	
		Dibenz(a,h)anthracene	0.0029	40	0.5	0.169	0.5	
		Hexachlorobenzene	0.042	6000	1	0.33	1	
		Hexachlorocyclopentadiene	0.18	NS	5	0.513	5	
		Indeno(1,2,3-cd)pyrene	0.029	100	0.5	0.206	0.5	
		Naphthalene	0.14	20000	1	0.34	1	
		Nitrobenzene	0.12	NS	5	0.362	5	

TABLE 7.1
ANALYTE DETECTION LIMIT REVIEW

Media	Parameter	Analyte	PAL-HH	PAL-eco	Project QL	MDL	RL	Report MDL
		Pentachlorophenol	0.56	200	1	0.172	1	x
	Pesticides	Aldrin	0.004	20	0.1	0.015	0.1	
		Alpha-BHC	0.011	NS	0.1	0.011	0.1	x
		Beta-BHC	0.037	NS	0.1	0.014	0.1	x
		Dieldrin	0.0042	0.5	0.1	0.021	0.1	
		Gamma-BHC/Lindane	0.061	4	0.1	0.011	0.1	x
		Heptachlor	0.015	1	0.1	0.011	0.1	x
		Heptachlor epoxide	0.0074	2	0.1	0.011	0.1	
		Toxaphene	0.061	NS	0.5	0.177	0.5	
	PCBs	Aroclor 1221	0.0068	10	0.3	0.3	0.3	
		Aroclor 1232	0.0068	10	0.3	0.1	0.3	
		Aroclor 1242	0.034	10	0.3	0.14	0.3	
		Aroclor 1248	0.034	10	0.3	0.11	0.3	
		Aroclor 1254	0.034	10	0.3	0.08	0.3	
		Aroclor 1260	0.034	10	0.3	0.09	0.3	
	Herbicides	MCPA	18	NS	100	7.65	100	x
		MCPP	37	NS	100	18.8	100	x
	Metals	Arsenic	0.045	900	1	0.397	1	
	Aldehydes	Acetaldehyde	2.2	NS	100	TBD	100	
	Hydrazines	Hydrazine	0.022	NS	0.2	0.05	0.2	
	Nitrosamines	NDMA	0.00042	NS	0.002	0.000741	0.002	
		NDPA	0.0096	NS	0.01	0.00066	0.01	x
SW	VOC	Carbon Disulfide	NS	0.92	10	0.62	10	x
		Hexachlorobutadiene	NS	0.053	0.4	0.4	0.4	
		m-Xylene	NS	1.8	2	1.35	2	x
	SVOC	1-Methylnaphthalene	NS	2.1	TBD	TBD	TBD	
		1,2,4,5-Tetrechlorobenzene	NS	3	5	0.158	5	x
		2,4,6-Trichlorophenol	NS	4.9	5	0.311	5	x
		2-Chloronaphthalene	NS	0.396	5	0.331	5	x
		3,3'-Dichlorobenzidine	NS	4.5	5	0.174	5	x
		4-Bromophenyl-phenylether	NS	1.5	5	0.348	5	x
		Acenaphthylene	NS	0.14	0.3	0.284	0.3	
		Aniline	NS	4.1	5	0.191	5	x
		Anthracene	NS	0.035	1	0.357	1	
		Benzo(a)anthracene	NS	0.025	0.3	0.3	0.3	
		Benzo(a)pyrene	NS	0.014	0.2	0.2	0.2	
		Benzo(g,h,i)perylene	NS	0.02	0.5	0.215	0.5	
		Benzo(k)fluoranthene	NS	0.14	0.3	0.128	0.3	x

TABLE 7.1
ANALYTE DETECTION LIMIT REVIEW

Media	Parameter	Analyte	PAL-HH	PAL-eco	Project QL	MDL	RL	Report MDL
		Benzyl alcohol	NS	8.6	10	0.12	10	x
		Chrysene	NS	0.07	1	0.349	1	
		Dibenz(a,h)anthracene	NS	0.04	0.5	0.169	0.5	
		Dibenzofuran	NS	3.7	5	0.341	5	x
		Fluoranthene	NS	0.9	1	0.416	1	x
		Fluorene	NS	0.14	1	0.348	1	
		Hexachlorobenzene	NS	0.0003	1	0.33	1	
		Hexachlorobutadiene	NS	0.053	0.6	0.358	0.6	
		Indeno(1,2,3-cd)pyrene	NS	0.14	0.5	0.206	0.5	
		Pyrene	NS	0.089	5	0.58	5	
	Pesticides	4,4`-DDD	NS	0.011	0.1	0.015	0.1	
		4,4`-DDE	NS	4.51E-09	0.05	0.013	0.05	
		4,4`-DDT	0.001	0.000011	0.1	0.013	0.1	
		Aldrin	NS	0.017	0.1	0.015	0.1	x
		Dieldrin	0.056	0.000071	0.1	0.021	0.1	
		Endosulfan I	0.056	0.051	0.1	0.015	0.1	x
		Endosulfan II	0.056	0.051	0.1	0.024	0.1	x
		Endosulfan sulfate	NS	0.051	0.1	0.01	0.1	x
		Endrin	0.036	0.0023	0.1	0.023	0.1	
		Gamma-BHC/Lindane	NS	0.016	0.1	0.011	0.1	x
		Heptachlor	0.0038	0.0036	0.1	0.011	0.1	
		Heptachlor epoxide	0.0038	0.0036	0.1	0.011	0.1	
		Methoxychlor	0.03	0.019	0.1	0.018	0.1	x
		Toxaphene	0.0002	0.00014	0.5	0.177	0.5	
	Herbicides	Dinoseb	NS	0.48	1	0.2	1	x
	EPH	C11-C22 Aromatics Unadjusted	NS	5	100	NA	100	
		C11-C22 Aromatics	NS	5	100	NA	100	
	Metals	Aluminum	87	87	100	2.2	100	x
		Cadmium	0.25	0.091	1	0.18	1	
		Copper	9	1.58	10	1.6	10	
		Mercury	0.77	0.0013	0.2	0.129	0.2	
		Nickel	52	8.2	10	1.6	10	x
		Selenium	5	5	10	2.2	10	x
		Zinc	120	36	50	2.9	50	x
		Lead	2.5	0.5	1	0.29	1	x
		Silver	NS	0.03	1	0.44	1	
Soil	SVOC	2,4-Dimethylphenol	500000	10	33.33	5.8	33.33	x
		2-Chloronaphthalene	6300000	12.2	33.33	5.12	33.33	x

TABLE 7.1
ANALYTE DETECTION LIMIT REVIEW

Media	Parameter	Analyte	PAL-HH	PAL-eco	Project QL	MDL	RL	Report MDL
		Benzo(a)pyrene	15	1520	33.33	6.1	33.33	x
		Dibenz(a,h)anthracene	15	18400	33.33	6.73	33.33	x
		N-nitrosodimethylamine	2.3	0.0321	33.33	3.77	33.33	
	PCBs	Aroclor 1248	220	72	100	22.3	100	x
	NDMA	NDMA	2.3	0.0321	5	1	5	
	Metals	Antimony	20	0.142	0.5	0.137	0.5	x
		Arsenic	0.39	0.25	1	0.081	1	x
		Cadmium	2	0.00222	0.2	0.0043	0.2	
		Chromium	30	0.4	0.5	0.073	0.5	x
		Cobalt	23	0.14	0.5	0.016	0.5	x
		Lead	300	0.0537	0.5	0.044	0.5	x
		Selenium	390	0.0276	0.5	0.272	0.5	
		Thallium	5.1	0.027	1	0.191	1	
		Vanadium	550	0.714	1	0.015	1	x
SED	VOC	1,1-Dichloroethane	NS	0.575	2.5	0.26	2.5	x
		2-Hexanone	NS	22	25	9.2	25	x
		Acetone	NS	8.7	250	32.2	250	
		Bromomethane	NS	1.37	5	0.75	5	x
		Carbon Disulfide	NS	0.85	2.5	0.58	2.5	x
	SVOC	2,4-Dinitrophenol	NS	6.21	33.33	4.89	33.33	x
		2,4-Dinitrotoluene	NS	14.4	33.33	4.7	33.33	x
		2-Chlorophenol	NS	31.9	33.33	3.25	33.33	x
		2-Methylnaphthalene	NS	20.2	33.33	25.44	33.33	
		2-Methylphenol	NS	12	33.33	4.76	33.33	x
		4-Methylphenol (reported as 3&4 Methylphenol)	NS	20.2	33.33	6.33	33.33	x
		4-Nitrophenol	NS	13.3	166.7	6.49	166.7	x
		Acenaphthene	NS	6.71	33.33	4.89	33.33	x
		Acenaphthylene	NS	5.87	33.33	5.89	33.33	
		Aniline	NS	0.31	33.33	17.29	33.33	
		Benzyl alcohol	NS	1	66.7	7.6	66.7	
		Dibenz(a,h)anthracene	33	33	33.33	6.73	33.33	x
		Hexachlorobenzene	NS	20	33.33	7.91	33.33	x
		Hexachlorobutadiene	NS	26.5	33.33	6.64	33.33	x
	EPH	C11-C22 Aromatics	NS	90	3300	NA	3300	
		C11-C22 Aromatics Unadjusted	NS	90	3300	NA	3300	
		C9-C18 Aliphatics	NS	3170	3300	NA	3300	
	VPH	C5 – C8 Aliphatics	NS	1590	5000	NA	5000	

TABLE 7.1
ANALYTE DETECTION LIMIT REVIEW

Media	Parameter	Analyte	PAL-HH	PAL-eco	Project QL	MDL	RL	Report MDL
		Unadjusted						
		C5 – C8 Aliphatics	NS	1590	5000	NA	5000	
		C9 – C10 Aromatics	NS	240	5000	NA	5000	

Notes:

Aqueous units = µg/L

Organic Analytes Solid units = µg/Kg

Metal Solid units = mg/Kg

Prepared by: CR
Checked by: TC
04/28/09

TABLE 7.2
SUMMARY OF QC LIMITS FOR SURROGATES AND SPIKES

PARAMETER	QC TEST	ANALYTE	WATER (%R)	SOIL (%R)
Volatiles	**Surrogate**	All Surrogates	70 - 130	70 - 130
	LCS	All Target Compounds	70 - 130	70 - 130
	MS/MSD	All Target Compounds	70 - 130	70 - 130
Semivolatiles	**Surrogate**	Phenol-d5	15 -110	30 - 130
		2-Fluorophenol	15 -110	30 - 130
		2,4,6-tribromophenol	15 -110	30 - 130
		nitrobenzene-d5	30 - 130	30 - 130
		2-fluorobiphenyl	30 - 130	30 - 130
		terphenyl-d14	30 - 130	30 - 130
	LCS	Base-Neutral Compounds	40-140	40-140
		Acid Compounds	30-130	30-130
	MS/MSD	Base-Neutral Compounds	40-140	40-140
		Acid Compounds	30-130	30-130
NDMA / NDPA	**Internal Standard**	NDMA-d6 and NDPA-d14	25-140	50-200
	LCS	NDMA/NDPA	60-140	40-140
	MS/MSD	NDMA/NDPA	60-140	40-140
	Surrogate	Nitrobenzene-d5	NA	40-140
PCBs	**Surrogate**	Decachlorobiphenyl	30 - 150	30 - 150
		Tetrachloro-m-xylene	30 - 150	30 - 150
	LCS	All Target Compounds	40 - 140	40 - 140
	MS/MSD	All Target Compounds	40 - 140	40 - 140
Pesticides	**Surrogate**	Decachlorobiphenyl	30 - 150	30 - 150
		Tetrachloro-m-xylene	30 - 150	30 - 150
	LCS	All Target Compounds	40 - 140	40 - 140
	MS/MSD	All Target Compounds	30 - 150	30 - 150
Inorganics	**LCS**	All Target Compounds	80 - 120	80 - 120
	MS/MSD	All Target Compounds	75 -125	75 - 125
VPH	**Surrogate**	All Surrogates	70 -130	70 -130
	LCS	All Target Compounds	70 -130	70 -130
	MS/MSD	All Target Compounds	70 -130	70 -130

TABLE 7.2
SUMMARY OF QC LIMITS FOR SURROGATES AND SPIKES

PARAMETER	QC TEST	ANALYTE	WATER (%R)	SOIL (%R)
Extractable Petroleum Hydrocarbons (EPH)	**Surrogate**	All Surrogates	40 -140	40 -140
	Fractionation Surr.	2-bromonaphthalene	40 -140	40 -140
	LCS	All Target Compounds	40 -140	40 -140
	MS/MSD	All Target Compounds	40 -140	40 -140
Alkylphenols (i.e. nonylphenols)	**Surrogate**	All Surrogates	40 -140	N/A
	LCS	All Alkylphenols	40 -140	N/A
	MS/MSD	All Alkylphenols	40 -140	N/A
N,N-dimethylformamide	**Surrogate**	Dimethylformamide	70 -130	70 -130
	LCS	N,N-dimethylformamide	70 -130	70 -130
	MS/MSD	N,N-dimethylformamide	70 -130	70 -130
Phthalic Anhydride (acid)	**LCS**	All Target Compounds	50 -150	60 -140
	MS/MSD	All Target Compounds	50 -150	60 -140
Opex and Kempore	**LCS/LCSD**	All Target Compounds	70 -130	N/A
	MS/MSD	All Target Compounds	75/125	N/A
Hydrazine, MMH, UDMA	**LCS/LCSD**	All Target Compounds	70-130	70 -130
	MS/MSD	All Target Compounds	70-130	40 -140
Formaldehyde and Acetaldehyde	**LCS**	All Target Compounds	80-120	80-120
	MS/MSD	All Target Compounds	75-125	75-125
Perchlorate	**LCS**	All Target Compounds	80 - 120	N/A
	MS/MSD	All Target Compounds	80 - 120	N/A

Prepared by: TC
Checked by: CR
04/23/09

Notes:
LCS = Laboratory Control Sample
MS/MSD = Matrix spike/ Matrix Spike Duplicate
%R = Percent Recovery
QC = Quality Control
N/A = Not Applicable

TABLE 7.3

SUMMARY OF PRECISION LIMITS FOR FIELD DUPLICATES, LAB DUPLICATES AND MS/MSD

PARAMETER	QC TEST	RPD Water	RPD Soil
Volatiles	**Field Duplicates**	30	50
	MS/MSD	30	30
Semivolatiles	**Field Duplicates**	30	50
	MS/MSD	20	30
NDMA / NDPA	**Field Duplicates**	30	50
	MS/MSD	20	50
PCBs	**Field Duplicates**	30	50
	MS/MSD	50	50
Pesticides	**Field Duplicates**	30	50
	MS/MSD	30	30
Inorganics	**Lab Duplicates**	20	35
	Field Duplicates	30	50
	MS/MSD	20	35
VPH	**Field Duplicates**	30	50
	MS/MSD	50	50
Extractable Petroleum Hydrocarbons (EPH)	**Field Duplicates**	30	50
	MS/MSD	50	50
Alkylphenols (i.e. nonylphenols)	**Field Duplicates**	30	N/A
	MS/MSD	50	N/A
N,N-dimethylformamide	**Field Duplicates**	30	50
	MS/MSD	50	50
Phthalic Anhydride (acid)	**Field Duplicates**	30	50
	MS/MSD	50	50
Opex and Kempore	**Field Duplicates**	30	N/A
	MS/MSD	20	N/A
	LCS/LCSD	20	N/A
Hydrazine, MMH, UDMA	**Field Duplicates**	30	50
	MS/MSD	20	50
	LCS/LCSD	20	35
Formaldehyde and Acetaldehyde	**Field Duplicates**	30	50
	MS/MSD	25	30
Perchlorate	**Lab Duplicates**	20	N/A
	Field Duplicates	30	N/A
	MS/MSD	20	N/A

Notes:
LCS = Laboratory Control Sample
MS/MSD = Matrix spike/ Matrix Spike Duplicate
QC = Quality Control
RPD = Relative Percent Difference
N/A = Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	GW/SW				
Analytical Parameter	VOCs				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 8260B / L-3	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≥ 30	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 70-130%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 70-130%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be < 10 x QL and acetone and 2-butanone must be < 2x QL)	Method Blanks, Storage Blanks, Equipment Blanks, Trip Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Instrument Blanks	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Accuracy/Bias	Percent recoveries 70-130%	Matrix Spike/ Matrix Spike Duplicates	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SW, GW				
Analytical Parameter	SVOCs				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 8270C / L-4	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 20	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries for Acid fraction compounds: 30 - 130% Percent recoveries for Base Neutral fraction 40 – 140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries for Acid fraction compounds: 30 - 130% Percent recoveries for Base Neutral fraction 40 – 140%	Laboratory Control Sample	A
		Accuracy/Bias	Phenol-d5, 2-Fluorophenol and 2,4,6-tribromophenol are 15-110%. Nitrobenzene-d5, 2-fluorobiphenyl and terphenyl-d14 are 30-130%.	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL (except phthalates must be < 5 x QL)	Equipment Blanks and Method Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Pesticides				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 8081/ L-29	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 30	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 30-150%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 40-140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 30-150%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SW, GW	
Analytical Parameter	PCBs	

Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW 846 8082/ L-27	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40-140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 40-140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 30-150%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Herbicides				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 8151A/ L-33	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 30	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 30-150%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 40-140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 40-140%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW	
Analytical Parameter	EPH	

Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	MADEP EPH-98-1/ L-25	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40-140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40-140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 40-140%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	VPH				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	MADEP VPH-98-1/ L-26	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 70-130%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 70-130%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 70-130%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	TAL Metals				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 6010B(ICP), 6020A (ICP-MS), 7470A (Hg) / L-1	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL *No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20 if results are ≥ 5x QL	Laboratory Duplicates	A
		Accuracy/Bias	Percent recoveries 75 – 125%, RPD < 20	Matrix Spikes	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A
		Accuracy/Bias	± 15% of original result (original concentrations must be > 50 times the MDL to be evaluated)	Serial Dilution Analysis	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 50 – 150%	Quantitation Limit Check Standard	A
		Accuracy/Bias	Percent recoveries 80 – 120%. Professional judgment for evaluation of positive bias.	Interference Check Sample	A
		Accuracy/Bias - Contamination	Absolute value of target metals must be < QL	Initial Calibration Blanks, Continuing Calibration Blanks, Preparation Blanks, and Equipment Blanks	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	TAL Metals				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
		Accuracy/Bias (ICP/MS only)	60 – 125% of IS in blank calibration standard	Internal Standards	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

IS – Internal Standard

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Hexavalent Chromium				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 7199 / L-38	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL *No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20 if results are ≥ 5x QL	Laboratory Duplicates	A
		Accuracy/Bias	Percent recoveries 75 – 125%, RPD < 20	Matrix Spikes	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A
		Accuracy/Bias - Contamination	Absolute value of target metals must be < QL	Initial Calibration Blanks, Continuing Calibration Blanks, Preparation Blanks, and Equipment Blanks	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Alkalinity/Chloride/ Conductivity/Ammonia/Nitrate/Nitrite/Bromide/Fluoride/COD/ Sulfate/Hardness/TOC				
Concentration Level	Low/Medium				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	Alkalinity by SM 2320B/ L-15 Chloride by EPA 300.0/ L-13 Conductivity by SM 2510B / L-15 Ammonia by QuikChem Method 10-107-06-1-A / L-20 COD by EPA 410.4 / L-21 Sulfate by EPA 300.0 / L-12 Nitrate by EPA 300.0 / L-17 Nitrite by EPA 300.0 / L-18 Fluoride by EPA 300.0 / L-18 Bromide by EPA 300.0 / L-18 Hardness by SM 2340B / L-16 TOC by SM 5310B / L-22	Precision – Overall	* RPD ≤ 30 when positive results for both samples are ≥ 5x QL * Absolute difference < 2x QL when positive results for both samples are < 5x QL * RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL * No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	Percent recoveries 75 – 125%/RPD ≤ 20, N/A for Conductivity, Alkalinity, Hardness and TOC	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	Low level Standard	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Alkalinity/Chloride/ Conductivity/Ammonia/Nitrate/Nitrite/Bromide/Fluoride/COD/ Sulfate/Hardness/TOC				
Concentration Level	Low/Medium				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Total Dissolved Solids/Total Suspended Solids/pH				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	TDS by SM 2540C / L-19 TSS by SM 2540D / L-23 pH by SM 4500 / L-15	Precision – Overall	* RPD ≤ 30 when positive results for both samples are ≥ 5x QL * Absolute difference < 2x QL when positive results for both samples are < 5x QL * RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL * No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	N/A	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	Low level Standard	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Formaldehyde and Acetaldehyde				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 8315 / L-32	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	85 – 115% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 75 – 125%/RPD ≤ 20	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Hydrazine, MMH, UDMH				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	SW-846 Modified 8315 / L-24	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	LCS/LCSD	A
		Accuracy/Bias	70 – 130% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 70 – 130%/RPD ≤ 30	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 70 – 130%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Phthalic anhydride (acid)				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	HPLC / L-36	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	80 – 120% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries within 50-150; RPD ≤ 50	Matrix Spikes	A
		Accuracy/Bias	N/A	Surrogates	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries within 50-150	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	N,N-dimethylformamide (DMF)				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	Modified 8033 GC/NPD/ L-35	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	75 - 125% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 70-130 - RPD ≤ 20	Matrix Spikes	A
		Accuracy/Bias	Surrogate is Diethylformamide – 70-130%	Surrogates	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 70-130%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

Medium/Matrix	SW, GW				
Analytical Parameter	NDMA				
Concentration Level	Low/medium				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	Modified 8270C – Low Level / L-6	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 20	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries for NDMA 60 – 140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 60 – 140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent Recovery 60-140%	Surrogate	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks and Method Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits. Signal/noise ratio >3 for positive detections and low point initial calibration standard	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SW, GW				
Analytical Parameter	NDMA/NDPA				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	Modified EPA 521 / L-5	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 20	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 60 – 140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 60 – 140%	Laboratory Control Sample	A
		Accuracy/Bias	Internal Standard Surrogate 25-140%	Isotope Dilution IS	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks and Method Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits. Signal/noise ratio >3 for positive detections and low point initial calibration standard	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Alkylphenols – Nonylphenol, Nonylphenol diethoxylate, 4-nonylphenyl, Nonylphenol isomers				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	Modified 8270 SIM / L-34	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected..	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	85 – 115% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 40-140%/RPD ≤ 20	Matrix Spikes	A
		Accuracy/Bias	Internal Standard is Acenaphthene-d10 50-200%	Internal Standard	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 40 – 140%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SW/GW				
Analytical Parameter	Opex and Kempore				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1, S-2	Lab Specific HPLC method and SW-846 8000B / L-11	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	LCS/LCSD	A
		Accuracy/Bias	85 – 115% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 75 – 125%/RPD ≤ 20	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 70 – 130%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	GW				
Analytical Parameter	Perchlorate				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-1	SW-846 Method 6850 / L-37	Precision – Overall	* RPD ≤ 30 when positive results for both samples are ≥ 5x QL * Absolute difference < 2x QL when positive results for both samples are < 5x QL * RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL * No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 20	Laboratory Duplicates	A
		Accuracy/Bias	Percent recoveries 80 – 120%/RPD ≤ 30	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A

Prepared by: TC
Checked by: CR
10/14/08

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Measurement Performance Criteria Table

Medium/Matrix	SO, SD				
Analytical Parameter	VOCs				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	SW-846 8260B / L-3	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≥ 30	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 70-130%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 70-130%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be < 10 x QL and acetone and 2-butanone must be < 2x QL)	Method Blanks, Storage Blanks, Equipment Blanks, Trip Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Instrument Blanks	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Accuracy/Bias	Percent recoveries 70-130%	Matrix Spike/ Matrix Spike Duplicates	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SO, SD				
Analytical Parameter	SVOCs				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	SW-846 8270C / L-4	Precision – Overall	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 20	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries for Acid fraction compounds: 30 - 130% Percent recoveries for Base Neutral fraction 40 – 140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries for Acid fraction compounds: 30 - 130% Percent recoveries for Base Neutral fraction 40 – 140%	Laboratory Control Sample	A
		Accuracy/Bias	Phenol-d5, 2-Fluorophenol and 2,4,6-tribromophenol are 30-130%. Nitrobenzene-d5, 2-fluorobiphenyl and terphenyl-d14 are 30-130%.	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL (except phthalates must be < 5 x QL)	Equipment Blanks and Method Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SO				
Analytical Parameter	PCBs				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-3, S-15, S-16	SW 846 8082/ L-28	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40-140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 40-140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 30-150%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

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Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	EPH				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	MADEP EPH-98-1/ L-7	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40-140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40-140%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 40-140%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	VPH				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	MADEP VPH-98-1/ L-8	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 70-130%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 70-130%	Laboratory Control Sample	A
		Accuracy/Bias	Percent recoveries 70-130%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks, Storage Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	TAL Metals				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	SW-846 6010B(ICP), 6020A (ICP-MS), 7471B (Hg) / L-2	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 50 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL *No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35 if results are ≥ 5x QL	Laboratory Duplicates	A
		Accuracy/Bias	Percent recoveries 75 – 125%, RPD < 35	Matrix Spikes	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A
		Accuracy/Bias	± 15% of original result (original concentrations must be > 50 times the MDL to be evaluated)	Serial Dilution Analysis	A
		Accuracy/Bias	No false negatives, no false positives, quantitation within warning limits	Single Blind PES	A
		Accuracy/Bias	Percent recoveries 50 – 150%	Quantitation Limit Check Standard	A
		Accuracy/Bias	Percent recoveries 80 – 120%. Professional judgment used to evaluate positive bias.	Interference Check Sample	A
		Accuracy/Bias - Contamination	Absolute value of target metals must be < QL	Initial Calibration Blanks, Continuing Calibration Blanks, Preparation Blanks, and Equipment Blanks	S & A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	TAL Metals				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
		Accuracy/Bias (ICP/MS only)	60 – 125% of IS in blank calibration standard	Internal Standards	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

IS – Internal Standard

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	Hexavalent Chromium				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	SW-846 3060A/7199 / L-38	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 50 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL *No situations where one result is detected at ≥ 5x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35 if results are ≥ 5x QL	Laboratory Duplicates	A
		Accuracy/Bias	Percent recoveries 75 – 125%, RPD < 35 REDOX dependent	Matrix Spikes	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A
		Accuracy/Bias - Contamination	Absolute value of target metals must be < QL	Initial Calibration Blanks, Continuing Calibration Blanks, Preparation Blanks, and Equipment Blanks	S & A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	Ammonia / TOC / Chloride / Sulfate / Nitrate / Nitrite				
Concentration Level	Low/Medium				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	Ammonia by QuikChem Method 10-107-06-1-A / L-20 TOC by Lloyd Kahn / L-39 Chloride by EPA 300.0/ L-13 Sulfate by EPA 300.0 / L-12 Nitrate by EPA 300.0 / L-17 Nitrite by EPA 300.0 / L-18	Precision – Overall	* *RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35	Laboratory Duplicates	A
		Accuracy/Bias	80 – 120% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries within 75-125, RPD ≤ 35	Matrix Spikes	A
		Accuracy/Bias	N/A	Surrogates	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Accuracy/Bias	Percent recoveries within 80-120	Laboratory Control Sample	A

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Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	Formaldehyde and Acetaldehyde				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	SW-846 8315 / L-32	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35	Laboratory Duplicates	A
		Accuracy/Bias	85 – 115% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 75 – 125%/RPD ≤ 20	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 80 – 120%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	Hydrazine, MMH, UDMH				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	SW-846 Modified Method 8315 / L-24	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35	LCS/LCSD	A
		Accuracy/Bias	70 – 130% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 40 – 140%/RPD ≤ 30	Matrix Spikes	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 70 – 130%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	Phthalic anhydride (acid)				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	HPLC / L-36	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected..	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35	Laboratory Duplicates	A
		Accuracy/Bias	80 – 120% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries within 60-140, RPD ≤ 50	Matrix Spikes	A
		Accuracy/Bias	N/A	Surrogates	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries within 60-140	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

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Measurement Performance Criteria Table

Medium/Matrix	SO/SD				
Analytical Parameter	N,N-dimethylformamide (DMF)				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	Modified 8033 GC/NPD/ L-35	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPD ≤ 35	Laboratory Duplicates	A
		Accuracy/Bias	75 – 125% of true value	Calibration Check	A
		Accuracy/Bias	Percent recoveries 70-130% RPD ≤ 35	Matrix Spikes	A
		Accuracy/Bias	Surrogate is Diethylformamide – 70-130%	Surrogates	A
		Accuracy/Bias – Contamination	Target analytes must be ≤ ½ QL	Method Blanks	A
		Accuracy/Bias	Percent recoveries 70-130%	Laboratory Control Sample	A
		Sensitivity	MDL/QL evaluated versus project action limits	MDL study	A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A

EPA-NE QAPP Worksheet #11

Measurement Performance Criteria Table

Medium/Matrix	SO, SD				
Analytical Parameter	NDMA				
Concentration Level	Low				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
S-2, S-3, S-15, S-16	Modified 8270C – Low Level / L-6	Precision – Overall	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL *No situations where one result is detected at ≥ 2x QL and other result is not detected.	Field Duplicates	S & A
		Precision – Laboratory	RPDs ≤ 50	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries for NDMA 40 – 140%	Matrix Spike/ Matrix Spike Duplicates	A
		Accuracy/Bias	Percent recoveries 40 – 140%	Laboratory Control Sample	A
		Accuracy/Bias	Surrogate is Nitrobenzene-d5 40-140%	Surrogates	A
		Accuracy/Bias – Contamination	No target compounds ≥ QL	Equipment Blanks and Method Blanks	S & A
		Data Completeness	Field 90%, Laboratory 95%	Data Completeness Check	S & A
		Sensitivity	MDL/QL evaluated versus project action limits. Signal/noise ratio >3 for positive detections and low point initial calibration standard	MDL study	A

8.0 SAMPLING PROCESS DESIGN

The details of the sampling rationale and locations are documented in the FSP accompanying this QAPP. Worksheet #12b provides details on the sample matrices, parameters, applicable SOPs, containers and preservation requirements, and holding times.

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples (Identify field duplicates and replicates)[2]	Sampling SOP	Analytical Method/ SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	VOCs	Low	TBD	S-1, S-2	SW-846 8260B / L-3	3 x 40 ml	3 x 40 ml VOA vials	pH < 2 w/HCl: No headspace; Cool, 4ºC	14 days to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	VOCs	Low/High	TBD	S-3, S-15, S-2, S-16	SW-846 5035A / 8260B / L-3	3 x 40ml and (1) 2oz jar for % solids	3 x 40ml (2 with DI water, 1 with methanol)	Low-Conc: 5 ml DI water with stir bar, 4ºC. High-Conc: 10 ml methanol, 4ºC.	Low-Conc (DI water): 48-hours to freeze vials and 14 days to analysis. High-Conc (methanol): 14 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	SVOC	Low	TBD	S-1, S-2	SW-846 8270C / L-4	2 x 1L	2 x 1L Amber Glass	Cool, 4ºC	7 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	SVOCs	Low	TBD	S-3, S-15, S-2, S-16	SW-846 8270C / L-4	1 x 4 oz.	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	14 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Pesticides	Low	TBD	S-1, S-2	SW-846 8081 / L-29	2 x 1L	2 x 1L Amber Glass	Cool, 4ºC	7 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	PCBs	Low	TBD	S-1, S-2	SW-846 8082 / L-28	2 x 1L	2 x 1L Amber Glass	Cool, 4ºC	7 days to extraction; 40 days from extraction to analysis

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples (Identify field duplicates and replicates)[2]	Sampling SOP	Analytical Method/ SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
See FSP for Sample Location	See FSP for Location ID	SO	TBD	PCBs	Low	TBD	S-3, S-15, S-2, S-16	SW-846 8082 / L-27	1 x 8 oz	1 x 8 oz. clear wide mouth glass	Cool, 4ºC	14 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Herbicides	Low	TBD	S-1, S-2	SW-846 8151A / L-33	2 x 1L	2 x 1L Amber Glass	Cool, 4ºC	7 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	EPH	Low	TBD	S-1, S-2	MADEP EPH-2004 / L-7	2 x 1L	2 x 1L Amber Glass	Cool, 4ºC, pH < 2 w/ 1:1 HCL	14 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	EPH	Low	TBD	S-3, S-15, S-2, S-16	MADEP EPH-2004 / L-7	1 x 4 oz	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	14 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	VPH	Low	TBD	S-1, S-2	MADEP VPH-2004 / L-8	3 x 40 ml	3 x 40 ml VOA vials	pH < 2 w/HCl: No headspace; Cool, 4ºC	14 days to analysis for preserved sample
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	VPH	Low	TBD	S-3, S-15, S-2, S-16	MADEP VPH-2004 / L-8	1 x 40mL and (1) 2oz jar for % solids	1 x 10g in 10ml of methanol.	10g in 10ml of methanol.	28 days to analysis for preserved sample

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	**Location ID Number**	**Medium/ Matrix**	**Depth (Units)**	**Analytical Parameter**	**Conc. Level**	**No. of Samples (Identify field duplicates and replicates)[2]**	**Sampling SOP**	**Analytical Method/ SOP**	**Sample Volume**	**Containers (Number, size and type)**	**Preservation Requirements (chemical, temperature, light protected)**	**Maximum Holding Time (preparation/ analysis)**
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	TAL Metals	Low	TBD	S-1, S-2	SW-846 6010B/6020A/7470A / L-1	1 x 500mL	1 x 500mL polyethylene and a separate container if collecting a dissolved fraction	pH < 2 w/ HNO_3; Cool, 4ºC	Hg: 28 days to analysis Other Metals: 180 days to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	TAL Metals	Low	TBD	S-3, S-15, S-2, S-16	SW-846 6010B/ 6020A/7471B / L-2	1 x 4 oz.	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	Hg: 28 days to analysis Other metals: 180 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Hexavalent Chromium	Low	TBD	S-1, S-2	SW-846 7199 / L-38	1 x 250mL	1 x 250mL polyethylene	Cool, 4ºC No Headspace	**24-Hours to analysis**
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Hexavalent Chromium	Low	TBD	S-3, S-15, S-2, S-16	SW-846 3060A / 7199 / L-38	1 x 4 oz.	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	30 days to digestion, 7 days to analyze digestate
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Sulfate[1]	Low	TBD	S-1, S-2	USEPA 300 / L-12	1 x 500 mL	1 x 500 mL polyethylene	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Sulfate	Low	TBD	S-3, S-15, S-2, S-16	USEPA 300 / L-12	1x 4oz	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Chloride	Low	TBD	S-3, S-15, S-2, S-16	USEPA 300 / L-13	1x 4oz	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Chloride[1]	Low	TBD	S-1, S-2	USEPA 300 / L-13	1 x 500 mL	1 x 500 mL polyethylene	Cool, 4ºC	28 days to analysis

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples (Identify field duplicates and replicates)[2]	Sampling SOP	Analytical Method/ SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Alkalinity[1]	Low	TBD	S-1, S-2	SM 2320B / L-15	1 x 500 mL	1 x 500 mL polyethylene	Cool, 4ºC	14 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Nitrate	Low	TBD	S-1, S-2	USEPA 300.0 / L-17	1 x 500 mL	1 x 500 mL polyethylene (can be collected with nitrite)	Cool, 4ºC	48 hours to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Nitrate	Low	TBD	S-3, S-15, S-2, S-16	USEPA 300.0 / L-17	1x 4oz	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Nitrite	Low	TBD	S-1, S-2	USEPA 300.0 / L-18	1 x 500 mL	1 x 500 mL polyethylene (can be collected with nitrate)	Cool, 4ºC	48 hours to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Nitrite	Low	TBD	S-3, S-15, S-2, S-16	USEPA 300.0 / L-18	1x 4oz	1 x 4 oz. clear wide mouth glass	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Flouride	Low	TBD	S-1, S-2	USEPA 300.0 / L-18	1 x 500 mL	1 x 500 mL polyethylene	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Bromide	Low	TBD	S-1, S-2	USEPA 300.0 / L-18	1 x 500 mL	1 x 500 mL polyethylene	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Hardness	Low	TBD	S-1, S-2	SM 2340B / L-16	1 x 250 mL	1 x 250 mL polyethylene	pH < 2 w/ HNO_3	180 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Total Dissolved Solids (TDS)	NA	TBD	S-1, S-2	SM 2540C / L-19	1 x 500 mL	1 x 500 mL polyethylene	none	7 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Total Suspended Solids (TSS)	NA	TBD	S-1, S-2	SM 2540D / L-19	1 x 500 mL	1 x 500 mL polyethylene	none	7 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Ammonia	Low	TBD	S-1, S-2	Quick Chem Method 10-107-06-1-A / L-20	1 x 250 mL	1 x 250 mL polyethylene	H_2SO_4 to pH<2, Cool, 4ºC	28 days to analysis

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples (Identify field duplicates and replicates)[2]	Sampling SOP	Analytical Method/ SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Ammonia	Low	TBD	S-3, S-15, S-2, S-16	Quick Chem Method 10-107-06-1-A / L-20	1x 4oz	1 x 4 oz. amber wide mouth glass	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	pH	Low	TBD	S-1, S-2	SM 4500 / L-15	1 x 250	1 x 250 mL polyethylene	Cool, 4ºC	Immediately upon arrival at the lab
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Specific Conductance	Low	TBD	S-1, S-2	SM 2510B / L-15	1 x 250 mL	1 x 250 mL polyethylene	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	COD	Low	TBD	S-1, S-2	USEPA 410.4 / L-21	1 x 250	1 x 250 mL polyethylene	H_2SO_4 to pH<2, Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	TOC	Low	TBD	S-1, S-2	SM 5310B / L-22	3 x 40 ml	3 x 40 ml VOA vials	H_2SO_4 to pH<2 or HCL to pH<2 and Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	TOC	Low	TBD	S-3, S-15, S-2, S-16	Lloyd Kahn / L-39	1x 4oz	1 x 4 oz. amber wide mouth glass	Cool, 4ºC	28 days to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Formaldehyde and Acetaldehyde	Low	TBD	S-1, S-2	SW-846 8315 / L-32	2 x 250mL	2 x 250mL Amber Glass	Cool, 4ºC	3 day hold time to extraction, then 3 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Formaldehyde and Acetaldehyde	Low	TBD	S-3, S-15, S-2, S-16	SW-846 8315 / L-32	1x 4oz	1 x 4oz. clear wide mouth glass	Cool, 4ºC	3 day hold time to extraction, then 3 days from extraction to analysis

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples (Identify field duplicates and replicates)[2]	Sampling SOP	Analytical Method/ SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Hydrazine, MMH and UDMH	Low	TBD	S-1, S-2	Modified Method 8315 / L-24	2 x 40mL	2 x 40mL amber VOC vials	Zero headspace, Acetate buffer, Cool, 4ºC	10-days to be derivatized / analyze extracts within 5 days of derivatization
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Hydrazine, MMH and UDMH	Low	TBD	S-3, S-15, S-2, S-16	Modified Method 8315 / L-24	1x 2oz	1 x 2 oz. amber glass	Cool, 4ºC	14-days to be extracted and derivatized / extracts must be analyzed within 72 hours of derivatization
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Phthalic Anhydride (acid)	Low	TBD	S-1, S-2	Lab Specific HPLC Method / L-36	3 x 40mL	3 x 40mL VOC vials	Cool, 4ºC out of direct sunlight	7 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	Phthalic Anhydride (acid)	Low	TBD	S-3, S-15, S-2, S-16	Lab Specific HPLC Method / L-36	1 x 8 oz.	1 x 8 oz. clear wide mouth glass	Cool, 4ºC out of direct sunlight	14 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	N,N-dimethylformamide (DMF)	Low	TBD	S-1, S-2	Modified 8033 – GC/NPD / L-35	2 x 40mL	2 x 40mL VOC vials	Cool, 4ºC	14 days to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	N,N-dimethylformamide (DMF)	Low	TBD	S-3, S-15, S-2, S-16	Modified 8033 – GC/NPD / L-35	1x 2oz	1 x 2 oz. clear wide mouth glass	Cool, 4ºC	14 days to extraction; 40 days from extraction to analysis

EPA-NE QAPP Worksheet #12b –Surface Water/Groundwater/ Sediment/ Soil

Sampling Locations, Sampling Matrices, Parameters, SOPs, and Container Requirements

Sampling Location	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc. Level	No. of Samples (Identify field duplicates and replicates)[2]	Sampling SOP	Analytical Method/ SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	NDMA / NDPA	Low	TBD	S-1, S-2	Modified EPA 521 / L-5	2 x 1L	2 x 1L Amber Glass	Cool, 4ºC	7 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SO/SD	TBD	NDMA	Low	TBD	S-3, S-15, S-2, S-16	Modified 8270C – Low Level Method / L-6	1 x 8 oz.	1 x 8 oz. clear wide mouth glass	Cool, 4ºC	14 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	Alkylphenols – Nonylphenol, Nonylphenol diethoxylate, 4-nonylphenyl, Nonylphenol isomers	Low	TBD	S-1, S-2	SW-846 8270C SIM / L-34	2 x 1L	2 x 1L Amber Glass	H_2SO_4 to pH<2 and Cool, 4ºC	7 days to extraction; 40 days from extraction to analysis
See FSP for Sample Location	See FSP for Location ID	SW/GW	TBD	OPEX and Kempore	Low	TBD	S-1, S-2	Lab Specific HPLC Method, and SW-846 8000B / L-11	2 x 40mL	2 x 40mL VOC vials	Cool, 4ºC	7-days to analysis
See FSP for Sample Location	See FSP for Location ID	GW	TBD	Perchlorate	Low	TBD	S-1	SW-846 6850 / L-37	2 x 40mL	2 x 40mL VOC vials	none	28 days to analysis

[1] Sulfate, chloride, and alkalinity can be collected in a single 500 ml bottle. TBD = To Be Determined

[2]Field duplicate samples will be collected at frequency outlined in EPA-NE QAPP Worksheet #9c; location of field duplicates not yet determined.
Holding times are calculated from date of sample collection.

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9.0 SAMPLING PROCEDURES AND REQUIREMENTS

This section provides an overview of how samples will be collected. Specific sampling procedures are presented as SOPs in Appendix C of the FSP. The selected sampling procedures will ensure that representative samples are collected in a consistent manner, that contamination is not introduced during collection, and all required sample media/matrices, locations, and properly preserved volumes are collected in order to meet PQOs.

9.1 SAMPLING PROCEDURES

SOPs which will be utilized during sampling are listed below:

- MACTEC SOP No. S-1, Low Flow / Low Stress Groundwater Sampling
- MACTEC SOP No. S-2, Surface Water and Sediment Sampling
- MACTEC SOP No. S-3, Surface Soil Sampling
- MACTEC SOP No. S-4, Calibration of Field Instruments for Water Quality Parameters
- MACTEC SOP No. S-5, Decontamination of Field Equipment
- MACTEC SOP No. S-6, Monitoring Well Development
- MACTEC SOP No. S-7, Procedures for Measuring Groundwater Levels
- MACTEC SOP No. S-8, Sample Packaging and Shipment
- MACTEC SOP No. S-9, Sample Chain of Custody Procedure
- MACTEC SOP No. S-10, Use of Field Logbooks
- MACTEC SOP No. S-11, Procedure for Description and Identification of Soils
- MACTEC SOP No. S-12, Monitoring Well and Piezometer Installation Procedures
- MACTEC SOP No. S-13, Field Preservation of VOA and VPH Soil Samples
- MACTEC SOP No. S-14, Soil Headspace Screening Procedure
- MACTEC SOP No. S-15, Split-Spoon/Split-Barrel Subsurface Soil and Standard Penetration Test Procedure
- MACTEC SOP No. S-16, Geoprobe® Direct Push Sampling
- MACTEC SOP No. S-17, Calibration Procedure for PID
- MACTEC SOP No. S-18, Procedure for Sonic Drilling
- MACTEC SOP No. S-19, Field Sample Tracking System

Worksheet #13 summarizes the procedures that will be used during this investigation. The appropriate number, size, and type of sample containers to be used for collection of all field samples and field QC samples are detailed on Worksheet #12b.

It is anticipated that ground water sampling will be performed using submersible pumps at all monitoring wells. However, the use of peristaltic pumps may be necessary at wells where submersible pumps are not feasible (i.e., narrow diameter wells).

9.2 SAMPLING SOP MODIFICATIONS

It is anticipated that no modifications will be made to the sampling SOPs presented in this QAPP or in the Field Sampling Plan; however, if sampling procedures are modified, documentations of the changes will be recorded in the SOP and in Worksheet #13. Changes to sampling procedures will be communicated to the MACTEC project manger and MACTEC project QA officer and reported in the RI report.

9.3 CLEANING AND DECONTAMINATION OF EQUIPMENT/SAMPLE CONTAINERS

This section describes the procedures for the initial cleaning of sample equipment and subsequent decontamination procedures that will be followed during sampling activities. Cleaning/decontamination procedures apply to all equipment that will come in contact with a sample. The SOP for equipment cleaning and decontamination is provided in the Appendix C of the FSP (MACTEC SOP No. S-5, Decontamination of Field Equipment). Pre-cleaned bottles will be used for all sampling procedures. These bottles will be supplied by an external vendor. The certificates of cleanliness will be kept in project files located at MACTEC in Wakefield, Massachusetts.

9.4 FIELD EQUIPMENT CALIBRATION

Field equipment intended for investigation include submersible and peristaltic pumps for ground water sample collection. Calibration of these pumps is not required.

9.5 FIELD EQUIPMENT, MAINTENANCE, TESTING, AND INSPECTION REQUIREMENTS

The field equipment preventative maintenance program is designed to ensure the effective completion of the sampling effort and to minimize equipment down time. Worksheet #15 summarizes the field equipment maintenance, testing and inspection requirements for ground water sampling pumps. The maintenance responsibilities for field equipment will be assigned to the MACTEC Field Operations Leader. Field personnel will be responsible for daily field checks and calibrations and for reporting any problems with the equipment. The maintenance schedule will follow the manufacturer's recommendations. Field personnel will also be responsible for ensuring that critical parts are included with the field equipment. Critical spare parts will be immediately available to reduce potential downtime. The inventory will primarily contain parts that are subject to frequent failure, have limited useful lifetimes, and/or cannot be obtained in a timely manner. Backup instruments and equipment will be available within 1-day shipment to avoid delays in the field schedule.

9.6 INSPECTION AND ACCEPTANCE REQUIREMENTS FOR SUPPLIES/SAMPLE CONTAINERS

Critical supplies and sample containers will be inspected in the following manner.

Critical Supplies and Consumables	Inspection Requirements and Acceptance Criteria	Responsible Individual
Sample bottles	Visually inspected upon receipt for cracks, breakage, and cleanliness. Must be accompanied by certificate of analysis.	Field Operations Leader
Chemicals and reagents	Visually inspected for proper labeling, expiration dates, appropriate grade	Field Operations Leader

Supplies and consumables not meeting acceptance criteria will initiate the appropriate corrective action. Corrective measures may include notification of vendor and subsequent replacement of defective or inappropriate materials. Actions taken will be documented in the project files.

EPA-NE QAPP Worksheet #13:
Project Sampling SOP Reference Table

Reference Number	Title and Number	Originating Organization	Equipment Identification	Modified for Project Work Yes (Y) or No (N)	Comments
S-1	SOP No. S-1, Low Flow / Low Stress Groundwater Sampling.	MACTEC	Primarily: adjustable rate, peristaltic pump with Polyethylene and silicone tubing; Where necessary: adjustable rate, submersible (bladder) pump with Polyethylene tubing;	N	None
S-2	SOP No. S-2, Surface Water and Sediment Sampling.	MACTEC	Hand auger, shovel, trowel, stainless steel spoons and bowls.	N	None
S-3	SOP No. S-3, Surface Soil Sampling.	MACTEC	Hand auger, Stainless steel trowel/shovel, stainless steel spoons and bowls	N	None.
S-4	SOP No. S-4, Calibration of Field Instruments for Water Quality Parameters.	MACTEC	Water quality parameter meter, turbidity meter	N	None
S-5	SOP No. S-5, Decontamination of Field Equipment.	MACTEC	Liquinox, deionized water, methanol, 10% nitric acid, scrub brushes, wash basins, aluminum foil, polyethylene sheeting	N	None
S-6	SOP No. S-6, Monitoring Well Development.	MACTEC	Portable submersible pump, adjustable rate, peristaltic pump and tubing	N	None
S-7	SOP No. S-7, Procedures for Measuring Groundwater Levels	MACTEC	Electronic water level indicator	N	None

EPA-NE QAPP Worksheet #13:
Project Sampling SOP Reference Table

Reference Number	Title and Number	Originating Organization	Equipment Identification	Modified for Project Work Yes (Y) or No (N)	Comments
S-8	SOP No. S-8, Sample Packaging and Shipment	MACTEC	Coolers, plastic bags, duct tape, vermiculite, bubble wrap, ice, chains of custody	N	None
S-9	SOP No. S-9, Sample Chain of Custody Procedure	MACTEC	Chains of custody, custody seals, sample labels	N	None
S-10	SOP No. S-10, Use of Field Logbooks	MACTEC	Field Logbooks	N	None
S-11	SOP No. S-11, Procedure for Description and Identification of Soils	MACTEC	Sand grading chart, field logbook, folding ruler, color chart, field data record	N	None
S-12	SOP No. S-12, Water Monitoring Well and Piezometer Installation	MACTEC	Well screens, riser and end caps, filter pack, seal, ground surface protection casing	N	None
S-13	SOP No. S-13, Field Preservation of VOA and VPH Soil Samples	MACTEC	Pre-preserved VOA vials (DI water and methanol for VOC Method 5035a and methanol for VPH), 20 ml sampling syringe, utility knife.	N	None
S-14	SOP No. S-14, Soil Headspace Screening Procedure	MACTEC	Plastic bags or jars, PID meter, soil sampling equipment, sample jars, aluminum foil	N	None
S-15	SOP No. S-15, Split-Spoon/Split-Barrel Subsurface Soil and Standard Penetration Test Procedure	MACTEC	Drill equipment; Drill rig, Split spoon samplers and drill rods, Field logbook, Stakes, pin flags, or other	N	None

EPA-NE QAPP Worksheet #13:
Project Sampling SOP Reference Table

Reference Number	Title and Number	Originating Organization	Equipment Identification	Modified for Project Work Yes (Y) or No (N)	Comments
			marker Folding engineer's rule, Labeled sample jars (for classification), Indelible marker, Decontamination equipment, Camera		
S-16	SOP No. S-16, Geoprobe® Direct Push Sampling	MACTEC	Geoprobe, sampling equipment, Direct push rods, non-petroleum based lubricants, Geo-Pump peristaltic pump, Decontamination equipment, IDW Drums, PPE	N	None
S-17	SOP No. S-17, Calibration Procedure for PID	MACTEC	Photo ionization detector, Zero gas cylinder, Span gas cylinder, Field Instrument Calibration Record (See FSP Appendix B), Field logbook	N	None
S-18	SOP No. S-18, Procedure for Sonic Drilling	MACTEC	Drill rig, Auger bits, Down-hole sampling equipment, Drill rods, non-petroleum based lubricants, Pump and tremie piping, Decontamination equipment, IDW Drums, PPE	N	None

EPA-NE QAPP Worksheet #13:
Project Sampling SOP Reference Table

Reference Number	Title and Number	Originating Organization	Equipment Identification	Modified for Project Work Yes (Y) or No (N)	Comments
S-19	SOP No. S-19, Field Sample Tracking System	MACTEC	PC computer with Windows, MS Access, copy of MACTEC field sample tracking program, printer, labels	N	None

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EPA-NE QAPP Worksheet #15

Field Equipment Maintenance, Testing and Inspection Table

Sampling Equipment/ Instrument	Maintenance Activity	Testing Activity	Inspection Activity	Responsible Person	Frequency	Acceptance Criteria	Corrective Action	SOP Reference
Peristaltic Pump	NA	Operation	Visual inspection for defective parts	FOS Lead, FOL, Field Technician	Each pump prior to use	No visually defective parts, Pump is operable, Conformance to manufacturer standards	Repair, replace parts; use backup pump	S-1
Peristaltic Pump	Cleaning	NA	NA	FOS Lead, FOL, Field Technician	Each pump prior to use	No visually dirty parts	Re-clean	S-1
Peristaltic Pump	NA	Equipment Blank (EB)	NA	FOS Lead, FOL, Field Technician	Every 20 samples	No target analytes ≥ QL and no interferences detected	If EB levels impact data usability, re-clean, retest and resample and/or qualify data during data validation	S-1
Portable Submersible Pump	NA	Operation	Visual inspection for defective parts	FOS Lead, FOL, Field Technician	Each pump prior to use	No visually defective parts Pump is operable	Replace parts; Repair if not operable or use backup pump	S-1, S-6
Portable Submersible Pump	Cleaning	NA	NA	FOS Lead, FOL, Field Technician	Each pump prior to use	No visually dirty parts	Re-clean	S-1, S-6
Portable Submersible Pump	NA	Equipment Blank (EB)	NA	FOS Lead, FOL, Field Technician	Every 20 samples	No target analytes ≥ QL and no interferences detected	If EB levels impact data usability, re-clean, retest and resample and/or qualify data during data validation	S-1, S-6

NA – Not Applicable

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10.0 SAMPLE HANDLING, TRACKING AND CUSTODY REQUIREMENTS

10.1 SAMPLE COLLECTION DOCUMENTATION

This section of the QAPP describes field documentation procedures that will be followed for this project. Records of field activities will be prepared throughout the project.

A variety of records are used to capture information and record field data. Field data will be recorded on the following logs, forms, and/or notebooks:

- Site Logbook
- Field Notebooks
- Field Logbook
- Field Data Records (FSP Appendix B)
- Photographs
- Equipment Calibration Logs
- Health and Safety Logs
- On-site Field Chemistry Log Book (if needed)
- Field QC Sample Record

The MACTEC Field Operations Leader has the responsibility to maintain the various logs, forms, and notebooks that document daily field activities as discussed below. Individual responsibilities will be delegated to other field staff as appropriate. Special emphasis will be placed on the completeness and accuracy of all information recorded in the field, and will contain statements that are legible, accurate, and inclusive documentation of project activities. Because the logbooks, field data forms, and chain-of-custody forms provide the basis for future reports, they must contain accurate facts and observations. The language used in recording field data will be objective, factual, and free of personal interpretations or other terminology that may prove inappropriate. At the completion of field activities or field programs the field logbooks and field data records will be scanned to an Adobe (.pdf) document to be archived as an electronic copy.

10.2 LOGBOOKS AND FIELD RECORD DOCUMENTATION

Site and field Logbooks will be used to document procedures performed by field personnel. The site logbook and field logbooks provide a daily hand written account of all field activities. Logbooks are hardcover books that are permanently bound. All entries are made in permanent black or blue ink, and corrections are made with a single line with the author initials and date. Each page of the logbook will be dated and signed by the person completing the log. Partially completed pages will have a line drawn through the unused portion at the end of each day.

The cover of each logbook will be entitled with the facility and project name "Olin Chemical Superfund Site", the name of the subcontractor or agency completing the logbook, the logbook type (i.e., Site Logbook or sequentially numbered Field Logbook), and the date the logbook was started. The Site Logbook will contain a comprehensive listing of all field logbooks created for the project.

10.2.1 Site Logbook

The site logbook is a record of all site activities completed for each day or operation. Entries are made daily to document the important activities of that day. The Field Operations Leader, or designee, will complete the site logbook. At a minimum the site logbook will contain the following information:

- a list of all field logbooks created for the project;
- names, titles, and affiliations of all project related personnel present at the site during each day of operation;
- a brief summary of all activities completed for each day of operation;
- a listing of any changes made to established Work Plan, FSP, or QAPP procedures;
- a summary of any problems encountered during the day including a description of corrective actions and impacts on the project; and
- record of health and safety issues.

10.2.2 Field Logbooks

The MACTEC field team will follow MACTEC SOP S-10, Use of Field Logbooks. This SOP is included in Appendix C of the FSP.

Field logbooks will provide the means of recording the chronology of data collection activities performed during the investigation. As such, entries will be described in as much detail as possible so that a particular situation could be reconstructed without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be stored in the project files when not in use. Each logbook will be identified by the project-specific document number. All logbooks will be water resistant and have sequentially numbered pages.

The title page of each logbook will contain the following:

- person to whom the logbook is assigned
- the logbook number
- project name and number
- site name and location
- project start date
- end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, and names of all sampling team members present will be entered. Each page of the logbook will be signed and dated by the person making the entry. All entries will be made in permanent ink, signed, and dated and no erasures or obliterations will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is signed and dated by the sampler. The correction shall be written adjacent to the error.

Field activities will be fully documented. Upon receipt of the field logbook for a particular activity, the designated person recording the notes will begin recording notes on a new page. The person recording the notes will sign the top of the new page and indicate the date, time, and weather conditions, prior to recording information about the field activity. The field logbook will indicate whether any Field Data Record forms are used. When the designated person recording the notes either relinquishes the field logbook to another team member or turns the book in at the end of the day, the person relinquishing the field logbook will affix a signature and date to the bottom of the last page used. If the page is not complete, a diagonal line will be struck across the blank portion of the page. Information included in the logbook or associated field data record forms will include, but may not be limited to:

- chronology of activities, including entry and exit times
- names of all people involved in sampling activities and organizational affiliations
- level of personal protection used
- any changes made to planned protocol
- names of visitors to the site during sampling and reason for their visit
- sample location and identification
- dates (month/day/year) and times (military) of sample collection
- measurement equipment identification (model/manufacturer) and calibration information
- field monitoring instrument results (if not recorded on a field data record)
- site observations
- sample collection methods and equipment
- sample collection date and time
- sample depths
- whether grab or composite sample collected
- how sample composited, if applicable
- sample description (color, odor, texture, etc.)
- sample identification code
- tests or analyses to be performed
- sample preservation and storage conditions
- equipment decontamination procedures
- QC sample collection
- unusual observations
- record of photographs
- sketches or diagrams
- signature of person recording the information

Field logbooks will be reviewed on a daily basis by the MACTEC Field Operations Leader.

10.2.3 Field Data Record Forms

Field data records will be used to record sample collection information during field activities. A complete set of Field Data Records is provided in Appendix B of the FSP. These forms are designed to capture

data from each type of field activity that is completed during the RI. Field personnel are instructed to utilize these forms during the field activities for which each form was designed.

As with the field logbooks, all documentation will be recorded in permanent ink. Corrections to errors in documentation or recorded calculations will be made by first striking out the error with a single line so as not to obliterate the original entry. Then the replacement entry or value will be inserted where appropriate. The person originating the change will initial and date each separate change. All revisions, deletions, and changes will be made in indelible ink.

10.2.4 Photographs

Field personnel will be instructed to photo-document field activities where possible. Examples of items that may require photographic documentation include:

- general site topography
- sampling locations
- existing monitoring locations
- physical appearance of environmental samples
- physical appearance of ground water, surface water, sediment, and soil

A field logbook entry or Photograph Log will be used to record the date, time, and description (caption) of photographs taken at the site. Digital photographs will be downloaded from the camera and photographic files saved on the MACTEC/Olin project drive.

10.2.5 Equipment Calibration Log

A field data record form will be used to record which instruments were calibrated each day (identified by manufacturer, model number and serial number), the individual who performed the calibration, and any notes regarding the maintenance of the instrument.

10.2.6 Health and Safety Log

A Site Logbook entry will be used to record any Health and Safety issues that arise during field activities. Any injuries, illnesses, use of first aid supplies, use of personal protective equipment (for levels A, B or C

only, if needed), or possible work-related symptoms will be recorded in the log together with the date, the name(s) of the affected individual(s), and a description of the incident. The designated HSO and FOL will be responsible for these entries.

10.2.7 Field QC Sample Record

During field sampling investigations the FOL will maintain a record of all field QC samples that are generated. Field QC samples include QC blanks (field blanks, trip blanks, and equipment blanks), field duplicates, and MS/MSD samples. An example of a Field QC Sample record is included in Figure 10-4. This record will be provided to the project chemist for use during data validation.

10.3 FIELD DOCUMENTATION MANAGEMENT SYSTEM

The MACTEC Field Operations Leader will maintain an inventory of all logbooks used during the program and will be responsible for ensuring that they are archived in the project files following the completion of the investigation.

Completed field data records will be maintained by the MACTEC Field Operations Leader during the duration of the program and will be archived in the project files following completion of the sampling effort.

10.3.1 Sample Handling and Tracking System

This section documents the procedures that will be followed to identify and track samples collected in the field, samples delivered or shipped to a fixed laboratory for analysis, and sample transfer throughout the laboratory.

A computerized sample tracking program will used to ensure that all relevant sample information is recorded accurately and completely at each stage of the sample handling process. The sample tracking program will be the primary method used to record sample collection information and print individual bottle labels. This program can also be used to generate a COC. An example of the computer generated COC is presented in Field Sample Tracking System SOP. This SOP (S-19) is included in Appendix C of the FSP. Sample collection information is entered into the sample tracking data base by the field sampler or designated sample manager at the time of sample generation. Information from the sample including

sample ID, location ID, date collected, analytical methods, containers and preservatives, and sampler name is captured in the field and downloaded directly into an Access database. An electronic COC can be generated directly from the data base and sent to the laboratory. Sample Delivery Groups (SDGs) may be identified in the sample tracking process and information on QC samples, QC blanks, matrix spikes, field duplicates, and performance evaluation samples will also be tracked. Electronic sample collection information can be exported from the sample tracking program to Excel for reporting purposes.

The goal of each COC record is the same: to document the identification, source, contents, condition, date/time and parties involved in each sample's collection and transfer. Labels are created for every bottle needed for a sample. Bottles are then checked out to the sample team that collects the sample. When the team returns with the collected sample(s), the samples are recorded as "checked in" to the sample tracking program by the sample administrators. When the sample administrator ships the samples to the lab, the samples are recorded as "shipped" in the same sample tracking program and a hardcopy COC is produced for signature. Date and time data are recorded at every key step. An SDG report is also available to check the progress of the SDG and associated QC samples.

The sample tracking data is directly loaded into the MACTEC Technical Environmental Database (TED) to provide a summary of samples, analytical parameters, and sample collection dates. This summary is used to track the project schedule and sample analysis and reporting status. The data base is also used to track sample data reporting by off-site laboratories and verify completeness of the data deliverables.

10.3.1.1 Off-site Laboratory Sample Tracking

The computerized sample tracking program will be established at the field office to store sample collection information for samples collected for off-site laboratory analysis. Sample tracking procedures are described in the Field Sample Tracking System SOP located in the FSP.

Sample tracking starts with information entered from the FSP sample collection specifications and is used to print labels for sample bottles. As samples are collected, further data on sample collection date, time, depth and sample collector are entered and the status is updated to "collected". When samples are ready to be shipped, data on the SDG and shipping date are entered and the status is updated to "shipped". When the lab reports sample results, the sample tracking database system is used to build the final data base containing sample chemical, survey, and geological data. Finally, when the lab sends a data package the data are checked against the sample tracking file for completeness and data on lot, lab ID, preparation

data and time and analysis date and time are collected. At this point, the tracking cycle is complete. The following data are captured by Sample Tracking System:

STUDY	Area of concern or investigation
SITE_ID	Sample location ID
SITE_TYPE	Type of location (well, surface soil sample, sediment, soil boring, Seep Sample)
SAMP_ID	Unique sample ID – (see table 10.1)
QC_TYPE	Indicates whether sample is normal, duplicate, matrix spike or quality control
MATRIX	Sample matrix (Aqueous, Non-Aqueous)
DEPTH	Depth of sample collection (if applicable)
SDATE	Sample collection date
STIME	Sample collection time
COLLECTOR	Initials of person collecting sample
SDG	Sample Delivery Group for sample
TRIP	Trip blank associated with sample
RINSATE	Rinse blank associated with sample
STATUS	Where sample is in collection process - (labels printed, sample collected, sample prepared for shipping, sample shipped to lab, lab received, data received from lab)
ARFDATE	Date sample was shipped to lab
ANALYSIS	Analysis requested from lab

10.3.2 Sample Identification and Labeling

Samples collected during Site activities shall be assigned unique sample identification (ID) numbers. These numbers are necessary to identify and track each of the samples collected for analysis during

completion of the project. In addition, the sample ID numbers shall be used to identify and retrieve the analytical results received from the laboratory, as well as other data related to the sample.

Sample IDs for previously collected samples will be included in the database as they were originally identified. No changes will be made to sample IDs for previously collected samples. The following text describes the sample designations for future sampling. A summary of sample ID formats is presented in Table 10.1. It should be noted that both environmental samples and QA/QC samples will be collected and submitted for laboratory analysis. The QA/QC samples will include field duplicates, matrix spikes and matrix spike duplicates, and field QC blank samples (field blanks, equipment rinsate blanks, and trip blanks). Blank samples will have sample IDs that identify the sample as a specific type of blank (rinsate, trip, etc.) and it will include a numerical identifier as well. Blank samples will not contain any location ID.

In general, sample IDs will identify, in the following order, the Site (Olin Chemical Superfund Site), the medium sampled, the sample location, and a QA/QC designation (for samples submitted as field duplicates, for matrix spike analysis). In addition, for soil and sediment samples, the depth interval for the sample will also be included in the sample ID. Multiple samples (groundwater samples collected over time, for example) at a given location will all have the same sample ID, but they will be identified uniquely by the combination of the sample ID and sample date. With the exception of blank samples, each sample ID will contain the sample location. Future samples collected at previously sampled locations will be identified using the previously identified sampling location.

The sample ID code is not limited to a specific number of digits, except for practical limitations in listing the sample ID in report tables. Sample IDs will be assigned as described below:

In all sample IDs, the first two digits, OC, indicate the Site name (Olin Chemical).

Groundwater:

For groundwater samples, no explicit medium code will be included in the sample ID. For groundwater samples from conventional monitoring wells, piezometers, and temporary groundwater sampling locations, the sample ID will contain the location ID (shaded in the example following) and a QA/QC designation: **OC-GW-32D-XXX,** where XXX may be:

DUP = field duplicate sample

MS = matrix spike sample

MSD = matrix spike duplicate

For regular field samples, the QA/QC code will be left blank.

For groundwater samples from multilevel wells the sample ID will contain the location ID followed by the sample port number (shaded in following example) and a QA/QC designation: **OC-MP-1-17-XXX,** where XXX may be:

DUP = field duplicate sample

MS = matrix spike sample

MSD = matrix spike duplicate

For regular field samples, the QA/QC code will be left blank

Groundwater sample IDs will not contain a medium sampled code because the location IDs will contain either "MW", "GW", or "PZ", all of which indicate a groundwater sample. If filtered surface water samples are collected, the filtered fractions will be identified on the sample container label and in the comment section of the COC.

Surface Water and Sediment:

Surface water and sediment sample IDs will identify the medium sampled (SW for surface water, and SD for sediment), location ID (shaded in following example), upper and lower depth of sample (in feet, with one decimal point), and a QA/QC designation: **OC-SW-ISCO2-XXX , or OC-SD-ISCO2--0.0/2.0-XXX** where XXX may be:

DUP = field duplicate sample

MS = matrix spike sample

MSD = matrix spike duplicate

For regular field samples, these digits will be left blank

If filtered surface water samples are collected, the filtered fractions will be identified on the sample container label and in the comment section of the COC.

Surface Soil Samples Collected by Hand Methods

Soil samples will be collected by hand methods (only surface soils collected) and by drill rigs or direct push methods (both surface soil and subsurface soil samples will be collected via these methods). At locations where soil samples will be collected **only by hand methods**, the sample locations will be considered surface soil locations. The location ID for surface soil locations will be designated as SS-XXX. The surface soil location IDs will be sequential staring with the number 400.

The sample ID for soil samples from surface soil locations will contain the location (e.g. SS-401), upper and lower depth of sample (in feet, with one decimal point), and QA/QC designation **OC-SS-401-0.0/2.0-XXX ,** where XXX may be :

DUP = field duplicate sample
MS = matrix spike sample
MSD = matrix spike duplicate
For regular field samples, these digits will be left blank

Soil sample IDs will not contain an explicit medium sampled code, since soil sample location IDs will contain either "SS" or "SB", both of which indicate a soil sample.

Soil Samples Collected from Soil Borings

Machine-driven soil borings will be used to collect both surface soil samples and subsurface soil samples at numerous locations. These locations will be considered soil boring locations. The soil boring locations will be designated as SB-XXX. The soil boring location IDs will be sequential staring with the number 400.

The sample ID for soil samples from soil boring locations will contain the location (e.g. SB-401), upper and lower depth of sample (in feet, with one decimal point), and QA/QC designation: **OC-SB-401-3.0/9.0-XXX ,** where XXX may be:

DUP = field duplicate sample
MS = matrix spike sample
MSD = matrix spike duplicate
For regular field samples, these digits will be left blank

For example, the sample OC-SB-401-3.0/10-DUP would be a duplicate sample collected from the 3 to 10 foot interval from SB-401. OC-SB-401-0.0/2.0 would be a field sample collected from the 0 to 2 foot interval from the same boring.

Sample depths equal to, or greater than, 10 feet will be rounded to the nearest whole number.

Field QC Blanks

Field QC Blanks (field blanks, trip blanks, and equipment blanks) will be identified with a sample ID code indicated in Table 10.1. For example, **OC-EB-01** will be the first equipment rinse blank collected during the field event. Blank numbers will be assigned sequentially during the field program by the FOL or field sample manager.

TABLE 10.1
SAMPLE NUMBERING SCHEME

Media	Sample Type	Numbering Scheme
AQUEOUS		
Monitoring Wells	Groundwater	OC-GW-XX-XXX
Monitoring Wells	Groundwater – Multi level well	OC-MP-XX-XXX
Surface Water	Surface Water	OC-SW-XX-XXX
SOLID		
Sediment	Sediment	OC-SD-XX-XXX_XXX
Soil	Surface Soil	OC-SS-XX-XXX-XXX
Soil	Soil Boring	OC-SB-XX-XXX-XXX
MISCELLANOUS		
Blank	Field Blank	OC-FBK-XXX
Blank	Trip Blank	OC-TBK-XXX
Blank	Equipment Blank	OC-EBK-XXX
Drum	Drum	OC-D-XXX
QUALITY CONTROL SAMPLE CODES		
Field Sample = FS		
Trip Blanks = TB		
Field Blank = FB		
Equipment Blanks = EB		
Performance Evaluation Samples = PE		
Field Duplicates = FD		
Matrix Spike = MS		
Matrix Spike Duplicate = SD		

Notes: X = denotes place holder

Prepared by: TC
Checked by: CR
10/14/08

10.4 SAMPLE PRESERVATION, CONTAINERIZATION, AND SHIPPING

Summaries of sample containers, required sample volumes, preservation, and holding time requirements for all samples are presented in Worksheet #12b.

Samples will be shipped to the laboratory via Federal Express or couriered to the lab within twenty-four to forty-eight hours of sample collection using the overnight delivery service with coolers under custody seal. If analytical holding times are 24 hours-48 hours from time of sample collection or if samples need to be preserved at the laboratory, samples will be shipped within 24 hours of sample collection using the overnight service. An attempt will be made to collect these samples at the latter part of the day in order to provide the laboratory with adequate time for analysis prior to exceeding the holding time.

10.5 SAMPLE CUSTODY

A program has been established for sample chain-of-custody that will be followed during sample handling activities in both field and laboratory operations. The primary purpose of chain-of-custody procedures is to document the possession of the samples from collection through shipping, storage and analysis to data reporting and disposal.

A sample is considered to be under a person's custody if:

- the item is in the actual possession of a person
- the item is in the view of the person after being in actual possession of the person
- the item was in the actual physical possession of the person but is locked up to prevent tampering
- the item is in a designated and identified secure area

10.5.1 Field Sample Custody

Sample handling is an important part of the field investigation program since samples that are incorrectly handled can affect the quality of data. Sample handling begins at the collection of the samples and continues until the sample has been analyzed. An over-riding consideration essential for the validation of environmental measurement data is the necessity to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. FDRs, field logbook entries, and chain of custody forms provide evidence of sample tracking from collection to shipment, laboratory receipt, and laboratory custody.

A chain of custody form will be generated in the field at the time that samples are prepared for shipment to the laboratory. The COC form may be hand written or generated by the Field Sample Tracking System as described in SOP S-9 Sample Chain of Custody Procedure.

A computerized tracking system will be established for all samples to maintain a record of all samples collected for chemical analysis. Labels will be attached to sample containers prior to the time of collection and will be used to identify the sample type and the vertical and horizontal location where the sample originated. The label will contain a sample identifier, sampler initials and date, time sampled, analytical method, and sample preservation. Computer based spreadsheet and database files will be created with all the sample information needed to document the sampling and analytical activities, and to verify that all requested sample results are reported by the on-site and off-site laboratories.

Sample chain-of-custody and packaging procedures are summarized below. These procedures will ensure that the samples will arrive at the laboratory with the chain-of-custody intact. The MACTEC Field Operations Leader (or designee) is responsible for overseeing and supervising the implementation of proper sample custody procedures in the field and up until the samples have been transferred to a courier. The chain-of-custody procedures are initiated in the field immediately following sample collection. The procedures consist of: (1) preparing and attaching a unique sample label to each sample collected, (2) completing the chain-of-custody form, and (3) preparing and packing the samples for shipment.

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or dispatched properly. Field procedures have been designed such that as few people as possible will handle the samples. For samples that are collected that will be analyzed in the field (i.e. ammonia by HACH method 8038), a Field Screening Sample COC will be completed. An example of the Field Screening COC is included in Figure 10-1.
- All bottles will be identified by the use of pre-printed adhesive sample labels with site name and location, sample number, sample locations, date/time of collection, type of preservation, type of analysis, and sampler's initials. The sample numbering system is presented in Section 10.3.2 of this QAPP and Section 7 of the FSP. In most cases, sample labels will be generated using the Sample Tracking System prior to the sampling event.
- Sample labels will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample label because the pen would not function in wet weather. In addition, with the exception of VOC soil vials, sample labels will be covered with clear tape to minimize water damage during transit.
- Samples will be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents the transfer of custody of samples from the sampler to

another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage location.

- MACTEC will follow MACTEC SOP S-9, Sample Chain of Custody Procedure in Appendix C of the FSP. This SOP is included in the companion Field Sampling Plan.
- All shipments will be accompanied by the chain-of-custody record identifying the contents of the cooler or shipment container. The original record will accompany the shipment, and copies will be retained by the sampler and placed in the project files. Examples of chain-of-custody generated using the Sample Tracking Program, and a hand written COC, are included in Figure 10-2.
- Samples will be properly packaged for shipment and dispatched to the laboratory for analysis, with a separate signed custody record enclosed in and secured to the inside top of each sample box or cooler. MACTEC SOP S-8, Sample Packaging and Shipment is provided in Appendix C of the FSP and will be followed during this investigation. Shipping containers will be secured for shipment to the laboratory. If an authorized laboratory courier does not pickup the samples from the project site, custody seals will be attached to the front right and back left of the cooler and covered with clear plastic tape after being signed by field personnel. An example of a cooler custody seal is provided in Figure 10-3. Subsequently, the cooler will be strapped shut with strapping tape in at least two locations.
- Samples remain in the custody of the sampler until transfer of custody is completed. This consists of delivery of samples to the laboratory sample custodian, and signature of the laboratory sample custodian on the chain-of-custody or traffic report document as receiving the samples and signature of sampler as relinquishing samples.

10.5.2 Laboratory Sample Custody

Samples will be received and logged in by a designated sample custodian or his/her designee. Upon sample receipt, the sample custodian will

- examine the shipping containers to verify that the custody tape is intact;
- examine all sample containers for damage;
- determine if the temperature required for the requested testing program has been maintained during shipment and document the temperature on the chain-of-custody or sample login records;
- compare samples received against those listed on the chain-of-custody or traffic report;
- verify that sample holding times have not been exceeded;
- examine all shipping records for accuracy and completeness;
- determine sample pH (if applicable) and record on chain-of-custody or sample login forms;
- with the exception of VOC and VPH samples, aliquots which require acidification will be checked with pH paper and recorded on the chain-of-custody or sample login forms. VOC and VPH samples will be checked after the water in the vial has been analyzed. VOC and VPH pH results will be recorded on the instrument run-log;

- sign and date the chain-of-custody or traffic report immediately (if shipment is accepted) and attach the air bill;
- note any problems associated with the coolers and/or samples on the cooler receipt form and notify the Laboratory Project Manager, who will be responsible for contacting the MACTEC Lead Chemist or MACTEC Project Manager;
- attach laboratory sample container labels with unique laboratory identification and test; and
- place the samples in the proper laboratory storage.

Following receipt, samples will be logged in according to the following procedure:

- The samples will be entered into the laboratory tracking system. At a minimum, the following information will be entered: project name or identification, unique sample numbers (both client and internal laboratory), type of sample, required tests, date and time of laboratory receipt of samples, and field identification provided by field personnel.
- The Laboratory Project Manager will be notified of sample arrival.
- The completed chain-of-custody or traffic report, air bills, and any additional documentation will be placed in the final evidence file.

FIGURE 10-1
FIELD SCREENING SAMPLE COC

Sample Location	Sample ID	Matrix ***	Date Collected	Time Collected	Sampler Initials	Off-site Split**	Sampler Comments

Notes:

* All soil samples must have a container ID number

** Check box if a split sample for off-site laboratory confirmation is collected from this location

*** Matrix = Use matrix Codes from Table 10.1 (i.e. SD = sediment)

FIGURE 10-2

Computer Generated Chain Of Custody

Olin Chemical Superfund Site

MACTEC E&C

Lab:

Sample #	Sample Date	Sample Time	Field Sample ID	Qty Total	Qty Each	Bottle Size and Material		Preservative	Media	Method	Fraction
556	10/25/2207	16:20	ATMW012XXX03XX	5							
					2	1 Liter	Amber Glass	4 deg.C	GW	PCBs -8082	T
					1	500 mL	Poly	HNO3,4 deg.C	GW	TAL Metals + Mo -6010B / 7470A	T
					2	40 mL	Glass Vial	HCL, 4 deg.C	GW	VOCs -8260B + TICs	T
558	10/25/2207	16:20	ATMW012XXX03XX	4							
					2	1 Liter	Amber Glass	4 deg.C	GW	SVOCs -8270C + TICs	T
					2	40 mL	Glass Vial	HCL, 4 deg.C	GW	VOCs -8260B + TICs	T

Relinquished:_____ Date: ___/___/____ Time:_____ Received:_____ Date: ___/___/_____ Time:____

Relinquished:_____ Date: ___/___/____ Time:_____ Received:_____ Date: ___/___/_____ Time:____

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Figure 10-2 (cont)
Hand Written Laboratory Chain of Custody Form

TestAmerica, Inc.
Chain of Custody Form

•53 Southampton Road
Westfield, MA 01085
(P) 413-572-4000
(F) 413-572-3707
Westfield

•148 Rangeway Road
N. Billerica, MA 01862
(P) 978-667-1400
(F) 978-667-7871
Boston - Service Center

Client: _____ Client Project #: _____ Job# Quote# PO#

Address: _____ Site ID & State: _____ Shaded areas for office use

Reports Sent To: _____ Invoice same as Report to? ☐

Phone: Fax: Email: Email Rpt: ☐

If invoice contact or address different, note in Comments

Requested Turnaround Time (PLEASE SPECIFY)

STANDARD _____ RUSH _____ *(Lab Approval Required)*

Regulatory Programs/Presumptive Certainty/QC Forms

MADEP MCP ☐ GW1/S1 ☐ PWS DEP Forms ☐
CTDEP RCP ☐ CT RSR ☐ EDD Required ☐
QA Rpt: No QC ☐ Std QC Rpt ☐ CLP QC Rpt: ☐

500-series for drinking water
600-series for waste water, NPDES
6000-series for groundwater, soil, waste
8000-series for groundwater, soil, waste
Use comments section to further define.

Comments
(Special Instructions)

Please print legibility. If the analytical requests are not clearly defined on the chain-of-custody, the turnaround time will begin after all questions have been satisfactorily answered.

Sample Type Codes: WW-Wastewater, DW-Drinking Water, SW-Surface Water, GW-Groundwater, LW Lab Water, A-Air, "Z"-Other (please specify)

Sample I.D.	Sample Type	Sampler's Initials	Date / Time Collected	Grab	Comp.	# Containers	Plastic(P) or Glass(G)	Preservative: NaHSO4/MeOH	HNO3 to pH <2	H2SO4 to pH <2	HCl to pH <2	NaOH to pH >12	Na2S2O3	None / 4° C	524 / 624 / 8260	525 / 625 / 8270	PCB / Pest / Herbicide	EPH / VPH	DRO / GRO / ETPH	Metals (Please Specify)	Mercury	General Chemistry	Bacteriological	Toxicity			

Comments:

Sampled by (print): Signature:

Cooler ? Y / N

MADEP Requirement
Samples Iced? Y / N

Relinquished by:	Date:	Time:	Received by:	Date:	Time:
Relinquished by:	Date:	Time:	Received by:	Date:	Time:
Relinquished by:	Date:	Time:	Received by:	Date:	Time:

Temp @ receipt: _____ °C

Preservation / pH checked? Y / N

By:_____ Date:_____

TestAmerica WESTFIELD

Page_____ of _____

White = Lab file Yellow = Report copy Pink = Customer copy

WI-QA-010 Rev 3a

STL-8245 (1000)

Figure 10-3

Custody Seal

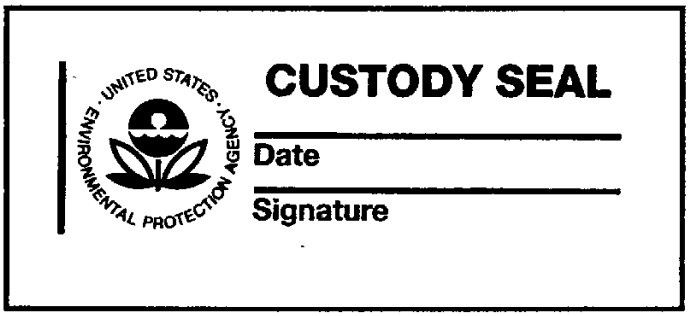

Figure 10-4
Field QC Sample Record

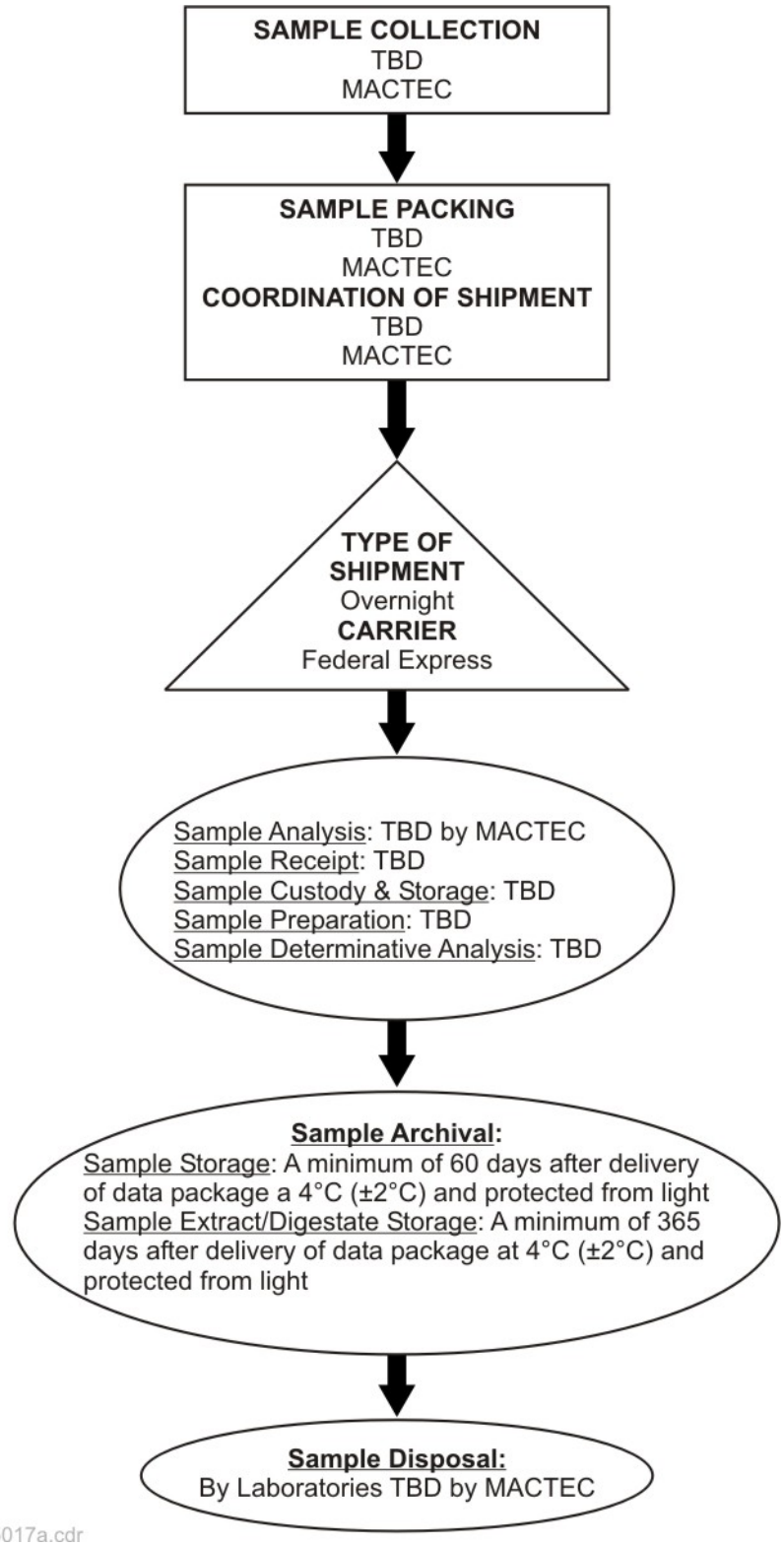

Figure 10-5
Field QC Sample Log

Blank Sample ID	Date Collect	Media	Equip type	Analytical Methods																	Comment
				VOC	SVOC	M-T	M-F	PCB	NDMA	VPH	EPH	F	Hy	PA	DMF	NP	pest	herb	K-O	G-chem	

Notes:
DMF= N-N-dimethylformamide M=F = filtered dissolved metal G-chem = general chemistry (list analytes in comment) PA= phthalic anhydride M-T = unfiltered total metal
F = Fromaldehyde NP = nonylphenol Hy = hydrazine K-O = Kempore/Opex Media = groundwater (GW), soil (S), surface water (SW), sediment (SED)

11.0 FIELD ANALYTICAL METHOD REQUIREMENTS

This section describes the analytical techniques that will be used in the field to generate monitoring and field screening data .

11.1 FIELD ANALYTICAL METHODS AND SOPS

SOPs or methods applicable to field analyses for this investigation are summarized in Worksheet #17. MACTEC SOP No. S-1 will be used for the analysis of water quality parameters associated with ground water and surface water samples. The Health and Safety Plan covers the procedures used to monitor VOCs with the Flame Ionization Detector (FID) and Photo ionization Detector (PID). The manufacturer's procedure for use of the water quality meter, turbidity meter, PID and Spectrophotometer are included in Appendix C. The manufacturer's procedures for use of the water level meter are included in Appendix C

11.2 FIELD ANALYTICAL INSTRUMENT CALIBRATION

MACTEC SOP S-4 will be used in the calibration of equipment related to water quality parameters. Worksheet #18 provides information relative to the calibration of all field instruments.

All materials, including standards or standard solutions, will be dated upon receipt, and will be identified by material name, lot number, purity or concentration, supplier, recipient's name, and expiration date.

11.2.1 Organic Vapor Detection Instrument

An FID or PID will be used to monitor air quality during sampling procedures. Prior to daily field operations and at the end of the day, the instrument will be checked for electronic calibration and adjusted as necessary. A 100 ppmV methane standard (for the FID) and a 100 ppmV isobutylene standard (for the PID) will be used for establishing instrument settings. Calibration will be documented on the field instrument calibration FDR (FSP Appendix B). If non-compliant instrument performance is noted, the instrument will be checked following the manufacturer's troubleshooting procedures. Instrument-specific calibration and maintenance (if needed) and records will be stored by the MACTEC Field Operations Leader.

11.2.2 pH/Conductivity/Temperature/DO/ORP/Salinity/Turbidity Measurements

A YSI 600XLM meter, or equivalent, equipped with an in-line flow-through cell for continuous monitoring, will be utilized to determine pH, temperature, conductivity, dissolved oxygen (DO), salinity, and/or oxidation-reduction potential (ORP) measurements in conjunction with water quality sample collection. The Lamotte 2020 Turbidity meter, or equivalent, will be used to determine turbidity in conjunction with water quality sample collection.

The instruments will be calibrated prior to each day's operation (FSP Appendix B). Calibration data including reference material and dates of reference material preparation and expiration, and the true value observed will be recorded in the field logbook.

If calibration verification standard recovery is determined to be outside the acceptance criteria, the specific probe will be reconditioned and recalibrated or replaced.

11.2.3 Electronic Water Level Indicator

A Solinst Model 101 electronic water level meter, or equivalent, will be used to measure water levels. The instrument will be calibrated prior to use with a measuring tape.

11.2.4 HACH DR 2800 Spectrophotometer

The HACH DR 2800 Spectrophotometer will be used to measure ammonia in aqueous samples. The HACH DR 2800 Spectrophotometer is calibrated by the manufacture prior to operation of the instrument in the field. HACH Method 8038 will be used to analyze for ammonia in association with the monitoring RGP program and Plant B. Prior to analyzing samples; an ammonium accuracy check will be performed on the HACH DR 2800 Spectrophotometer to insure proper instrument calibration. Worksheet #23b presents the estimated level of detection of ammonium as analyzed by the HACH DR 2800 Spectrophotometer. The HACH DR 2800 Spectrophotometer instrument manual, method 8038 and, and procedure for analyzing accuracy checks and samples is presented in Appendix C of this QAPP.

11.3 FIELD ANALYTICAL INSTRUMENT/EQUIPMENT MAINTENANCE, TESTING AND INSPECTION REQUIREMENTS

This section describes the procedures and documentation activities that will be performed to ensure that all field analytical instrumentation and equipment are available and in working order when needed. Worksheet #19 summarizes the field analytical instrument maintenance, testing, and inspection requirements. The field instrument preventative maintenance program is designed to ensure the effective completion of the sampling effort and to minimize instrument down time. The maintenance responsibilities for field instruments will be assigned to the MACTEC Field Operations Leader. Field personnel will be responsible for daily field checks and calibrations and for reporting any problems with the instruments. The maintenance schedule will follow the manufacturer's recommendations. Field personnel will also be responsible for ensuring that critical parts are included with the field instruments. Critical spare parts will be immediately available to reduce potential downtime. The inventory will primarily contain parts that are subject to frequent failure, have limited useful lifetimes, and/or cannot be obtained in a timely manner. Backup instruments and equipment will be available within 1-day shipment to avoid delays in the field schedule.

11.4 FIELD ANALYTICAL INSPECTION AND ACCEPTANCE REQUIREMENTS FOR SUPPLIES/SAMPLE CONTAINERS

Critical supplies and sample containers will be inspected in the following manner.

Critical Supplies and Consumables	Inspection Requirements and Acceptance Criteria	Responsible Individual
Sample bottles	Visually inspected upon receipt for cracks, breakage, cleanliness. Must be accompanied by certificate of analysis.	Field Operations Leader
Chemicals and reagents	Visually inspected for proper labeling, expiration dates, appropriate grade Record lot numbers of reagents used for calibration.	Field Operations Leader

Supplies and consumables not meeting acceptance criteria will initiate the appropriate corrective action. Corrective measures may include notification of vendor and subsequent replacement of defective or inappropriate materials. All actions will be documented in the project files.

EPA-NE QAPP Worksheet #17

Field Analytical Method/SOP Reference Table

Reference Number	Title, and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Originating Organization	Analytical Parameter	Instrument	Organization Performing Field Analysis	Modified for Project Work Yes (Y) or No (N)
F-1	MACTEC SOP No. S-1, Low Flow / Low Stress Groundwater Sampling.	Screening	NA	MACTEC	pH	YSI 600 XLM (or equivalent) pH probe	MACTEC	N
F-2	MACTEC SOP No. S-4, Calibration of Field Instruments for Water Quality Parameters.				DO	YSI 600 XLM (or equivalent) DO probe	MACTEC	N
					Specific Conductivity	YSI 600 XLM (or equivalent) Specific Conductance Electrode	MACTEC	N
					Temperature	YSI 600 XLM (or equivalent) Temperature Sensor	MACTEC	N
					ORP	YSI 600 XLM (or equivalent) ORP/Eh electrode	MACTEC	N
					Turbidity	LaMotte 2020, HACH 2100P (or equivalent) Turbidity meter	MACTEC	N
F-3	MACTEC SOP No. S-7, Procedures for Measuring Groundwater Levels.	Screening	NA	MACTEC	Water Level	Electronic Water-Level Indicator	MACTEC	N
F-4	See Health and Safety Plan. MACTEC SOP No. S-17, Calibration Procedure for PID.	Screening	NA	MACTEC	Total VOCs	FID or PID	MACTEC	N

EPA-NE QAPP Worksheet #17

Field Analytical Method/SOP Reference Table

Reference Number	Title, and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Originating Organization	Analytical Parameter	Instrument	Organization Performing Field Analysis	Modified for Project Work Yes (Y) or No (N)
F-7	HACH Method 8038. Appendix C of QAPP.	Screening	NA	MACTEC	Ammonia	HACH DR-2800 (spectrophotometer)	MACTEC	N

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #18

Field Analytical Instrument Calibration Table

Instrument	Activity	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	Field Method Reference / SOP Reference
pH Probe	Calibrate probe with temperature-equilibrated standards to bracket expected pH values	Daily-before use Calibration check – at end of day, or if instrument gives erratic results	Stable readings ± 10% pH standard.	If probe reading fails to stabilize, do not use. Check/replace probe and recalibrate or service as necessary. Repeat analysis of affected samples or qualify data if analysis cannot be repeated.	TBD	F-1, F-2 / S-1, S-4
DO Probe	Calibrate with 2 standards – saturated DO standard and 0.0 mg/L DO standard	Daily-before use Calibration check - at end of day, or if instrument gives erratic results	±10% of saturation concentration.	If DO reading exceeds criterion, then prepare new 0.0 mg/L DO standard, clean probe and/or change membrane. Recalibrate or service as necessary. Repeat analysis of affected samples or qualify data if analysis cannot be repeated.	TBD	F-1, F-2 / S-1, S-4
Specific Conductance Electrode	Calibrate electrode with a standard solution close to expected sample values.	Daily-before use Calibration check - every four hours during operation and at end of day, or if instrument gives erratic results	± 10% of standard value.	If sp. conductance electrode reading exceeds criterion, then clean probe or service as necessary and recalibrate. Repeat analysis of affected samples or qualify data if analysis cannot be repeated.	TBD	F-1, F-2 / S-1, S-4
Thermistor-Temperature Sensor	Calibrate against NIST-certified thermometer.	Calibration check –prior to onset of program	± 2.0 °C of NIST certified thermometer.	If temperature sensor reading exceeds criterion, then clean probe, or service as necessary and recalibrate. Repeat analysis of affected samples or qualify data if analysis cannot be repeated.	TBD	F-1, F-2 / S-1, S-4
Turbidity meter	HACH-calibrate with <0.01, 20, 100, and 800 NTU standards. LaMotte-calibrate with 0.1 and 10 NTU standards.	Daily-before use Calibration check-at end of day, or if instrument gives erratic results	± 10% per scale.	If turbidity reading exceeds criterion, then re-calibrate or service as necessary. Repeat analysis of affected samples or qualify data if analysis cannot be repeated.	TBD	F-1, F-2 / S-1, S-4

EPA-NE QAPP Worksheet #18

Field Analytical Instrument Calibration Table

Instrument	Activity	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	Field Method Reference / SOP Reference
ORP/ Eh Probe	Calibrate against a solution.	Daily-before use Calibration check -at end of day, or if instrument gives erratic results	Meter reading of 240mv standard between 200-275 mv	If ORP/Eh reading exceeds criterion, then have manufacture recalibrate. Repeat analysis of affected samples or qualify data if analysis cannot be repeated.	TBD	F-1, F-2 / S-1, S-4
Electronic Water-Level Indicator	Verify scale with measuring tape.	Beginning of field program.	± 0.1 feet	Replace	TBD	F-3 / S-7
FID	Calibrate with 100 ppmV methane standard. Blank: zero air check	Daily-before use	± 10% of true value	Recalibrate or service; rerun affected sample.	TBD	F-4 / S-17
PID	Calibrate with 100 ppmV isobutylene standard. Blank: zero air check	Daily-before use	± 10% of true value	Recalibrate or service; rerun affected sample.	TBD	F-4 / S-17
HACH DR-2800 (spectrophotometer)	Check accuracy with 1.0 mg/L Ammonia standard. Standard is made according to method.	Prior to starting analytical program. HACH DR-2800 is calibrated by the manufacturer. Check accuracy of instrument daily before use.	± 10% of standard value	Prepare new 1.0 mg/L standards and re-analyze. Document nonconformance. Return unit to HACH for re-calibration if unit fails.	TBD	F-7
	Instrument Blank/ Method blank with de-ionized water	One per day or after 20 samples have been analyzed which is more frequent.	No concentrations above method detection limits.	Recalibrate instrument is or service as per manufacturer instructions or analyze different lot of de-ionized water.		

TBD – To Be Determined

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #19
Field Analytical Instrument/Equipment Maintenance, Testing and Inspection Table

Instrument	Maintenance Activity	Testing Activity	Inspection Activity	Responsible Person	Frequency	Acceptance Criteria	Corrective Action	Field Method Reference / SOP Reference
pH Probe	Clean probe			TBD	When unstable readings occur	Stable after 3 minutes	Clear probe; and/or replace or service	F-1, F-2 / S-1, S-4
		QC Check		TBD	See Worksheet #18	See Worksheet #18		
			Visual Inspection	TBD	Daily before use	No defective parts noted		
DO Probe	Clean probe, change KCI and Teflon® membrane			TBD	• When bubbles are visible under membrane • When significant deposits of dried electrolyte are visible on membrane or o-ring • When probe gives unstable readings	NA	Clear probe; and/or replace or service	F-1, F-2 / S-1, S-4
		QC Check		TBD	See Worksheet #18	See Worksheet #18		
			Visual Inspection	TBD	Daily before use	No defective parts noted		
Specific Conductance Electrode	Clear opening to conductivity probe			TBD	Prior to initial use	No dirty parts	Clear probe and/or replace or service	F-1, F-2 / S-1, S-4
		QC Check		TBD	See Worksheet #18	See Worksheet #18		
			Visual Inspection	TBD	Daily before use	No defective parts noted		
Thermistor-Temperature Sensor		QC Check		TBD	See Worksheet #18	See Worksheet #18	Replace or Service	F-1, F-2 / S-1, S-4
			Visual Inspection	TBD	Daily before use	No defective parts noted		
Turbidity meter		QC Check		TBD	See Worksheet #18	See Worksheet #18	Replace or Service	F-1, F-2 / S-1, S-4
			Visual Inspection	TBD	Daily before use	No defective parts noted		
ORP/Eh Electrode		QC Check		TBD	See Worksheet #18	See Worksheet #18	Manufacturer must recalibrate	F-1, F-2 / S-1, S-4

EPA-NE QAPP Worksheet #19
Field Analytical Instrument/Equipment Maintenance, Testing and Inspection Table

Instrument	Maintenance Activity	Testing Activity	Inspection Activity	Responsible Person	Frequency	Acceptance Criteria	Corrective Action	Field Method Reference / SOP Reference
			Visual Inspection	TBD	Daily before use	No defective parts noted		
FID and PID	Clean Detector			TBD	When unstable readings occur	± 10% of true value of standard	Clean and/or replace detector	F-4 / S-17
		QC Check		TBD	See Worksheet #18	See Worksheet #18		
			Visual Inspection	TBD	Daily before use	No defective parts noted		
Electronic Water Level Indicator	Clean Indicator			TBD	Daily before use	No dirty parts	Clean and/or Replace	F-3 / S-17
			Visual Inspection	TBD	Daily before use	No defective parts noted		
HACH DR-2800 (spectrophotometer)		QC Check		TBD	See Worksheet #18	See Worksheet #18	Replace or Service	F-7
			Visual Inspection	TBD	Daily before use	No defective parts noted		

TBD – To Be Determined

Prepared by: TC
Checked by: CR
10/14/08

12.0 FIXED LABORATORY ANALYTICAL METHOD REQUIREMENTS

This section of the QAPP describes the analytical techniques that will be used by the fixed laboratories to generate definitive data for the project. It documents the fixed laboratory analytical methods that will be used to meet measurement performance criteria and achieve the project-required quantitation limits for all contaminants of concern and other target compounds in the specific matrices as identified on Worksheet #9b.

Subcontract laboratories selected for the RI are accredited through the NELAP. The primary laboratory for the RI, TAL-Westfield, also holds Massachusetts certification for the analysis of potable and non-potable water. Certification documents are included in Appendix E. These certificates ensure that laboratories meet national and state quality standards for environmental laboratory testing.

12.1 FIXED LABORATORY ANALYTICAL METHODS AND SOPS

Laboratories performing the analyses in support of this program are presented in Worksheet #20. Worksheet #20 also describes the USEPA SW-846 and EPA analytical methods that will be used by laboratories during this investigation. The SW-846, EPA, and lab specific analytical methods are included for each parameter, matrix, and concentration level that will be investigated. Laboratory analytical methods and SOPs to be used during this project are presented in Appendix B of this QAPP.

Laboratories will be contracted by Olin to perform analyses using methods in Worksheet #20. Test America Laboratories (TAL) is the primary laboratory for this investigation. A copy of the TAL laboratorys QA manual is presented in Appendix B of this QAPP.

The following fixed laboratories will be available to provide analytical analysis:

- Test America Laboratories - Westfield (formerly Severn Trent Laboratories or STL) – Westfield 53 Southhampton Road, Westfield, MA 01085. Tel. (413) 572-4000
- Test America Laboratories – Edison - 777 New Durham Rd # 7, Edison, NJ 08817 Tel (732) 549-3900
- Test America Laboratories - Tallahassee - 2846 Industrial Plaza Drive , Tallahassee, FL 32301. Tel (850) 878-3994, Fax (850) 878-9054
- Test America Laboratories – Denver - 1746 Cole Blvd, Bldg. 21 Suite 225, Lakewood, CO 80401. Tel.(866) 465-6653

- Test America Laboratories – West Sacramento - 880 Riverside Parkway, West Sacramento, CA 95605. Tel. (916) 373-5600 Fax: 916.372.1059
- Katahdin Analytical Services, Inc. - 600 Technology Way, Scarborough, ME 04074, Telephone: (207) 874-2400, Fax: (207) 775-4029
- Analytics Lab, LLC. 195 Commerce Way, Suite E, Portsmouth, New Hampshire 03801. Tel. (603) 436-5111 or (800) 929-9906, Fax. (603) 430-2151
- Lancaster Labs, Inc. 2425 New Holland Pike Lancaster, PA 17601. Tel. (717) 656-2300

12.2 FIXED LABORATORY ANALYTICAL METHOD/SOP MODIFICATIONS

USEPA SW-846, EPA and lab specific analytical methods will be followed as written. However, laboratories may deviate from the method specifications if the deviations are approved by MACTEC prior to the onset of the program. Upon selection of the laboratory(s), any deviations from method specifications, as written, will be provided in an addendum to Olin Corporation and EPA for review.

12.2.1 Analysis of NDMA in Water Samples

Analysis of NDMA in surface water and groundwater will be completed by Test America Laboratories in Sacramento California and in Westfield, Massachusetts. Analysis of NDMA will be completed using Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (GC/CI/MS/MS) by Modified EPA Method 521. EPA method 3510C, Liquid-Liquid Extraction will be used to extract the NDMA in the aqueous samples. Prior to extraction of the sample a know amount of NDMA-d6 (an isotope of NDMA) is spiked into the sample. The NDMA-d6 and NDMA (target compound) have similar recoveries during extraction, concentration and chromatographic analysis. The relative response value of the isotope is used in conjunction with calibration standards of NDMA to determine sample concentrations of NDMA directly. The reporting limit of NDMA in groundwater for this project using Modified EPA Method 521 is currently 2 nanograms per Liter (ng/L) (parts per trillion). The reporting limit of NDMA in surface water for this project using Modified Method 521 is currently 5 ng/L (parts per trillion) in surface water. N-nitros-di-n-propylamine (NDPA) has been included as an additional target compound for the Modified EPA Method 521 analysis. The reporting limit for NDPA is 10 ng/L.

Groundwater samples will be collected in areas of the Site that have historically had concentrations of NDMA greater than 1 ug/L (parts per billion). Samples collected with NDMA concentrations that are suspected to be above 1 ug/L will be analyzed by a Modified 8270C Low Level Mass Spec method. Test

America Laboratories in Westfield, Massachusetts will be performing the Modified 8270C Low Level Mass Spec method. The reporting limit for this method is 1 ug/L.

12.2.1.1 Elevated NDMA Reporting Limits

Test America Laboratories will take necessary precautions to reduce elevated NDMA reporting limits. The reporting limit of NDMA is usually dependent on the level of interference I sample matrices rather than instrumental limitations. Interferences from solvents, reagents, glassware and other sample processing hardware which yield artifacts and/or elevated baselines will be examined through the analysis of method blanks. Method blanks will be analyzed by the laboratory with every batch of samples. Interferences from solvents, reagents, glassware will be reduced by the lab through purification of solvents and oven baking of solvent rinsed glassware at 450 ^{o}C.

Elevated reporting limits may be due to the presence of interfering compounds at high concentrations in samples (>1000µg/L). The lab may analyze samples at a dilution to reduce the interference effects on NDMA. If the lab suspects that interfering compounds are present (dark extract color during extraction/concentration), communication will be made to MACTEC prior to dilution analysis.

12.2.2 Analysis of NDMA in Soil and Sediment Samples

Analysis of NDMA in soil and sediment will be completed by Test America Laboratories in Westfield Massachusetts. Analysis of NDMA will be completed using full scan GC/MS by modified method SW-846 8270C. EPA method 3550B, ultrasonic extraction will be used to extract the NDMA in the solid samples. Nitrobenzene-d5 will be used for the surrogate standard and 1,4-dichlorobenzene-d4 will be used for the internal standard. The reporting limit of NDMA by Test America is 5 µg/kg (parts per billion).

12.3 FIXED LABORATORY INSTRUMENT CALIBRATION

Worksheet #21 details the calibration procedures associated with analytical methods and associated instruments. These calibration procedures ensure that the analytical methods and selected instrumentation meet project requirements for selectivity, sensitivity, accuracy and precision of quantitation. These calibration procedures are also discussed in the USEPA SW-846, EPA, and lab specific analytical methods.

12.4 FIXED LABORATORY INSTRUMENT/EQUIPMENT MAINTENANCE, TESTING AND INSPECTION REQUIREMENTS

This section describes the procedures and documentation activities that will be performed to ensure that all fixed laboratory instrumentation and equipment are available and in good working order when needed. Worksheet #21 also details the fixed laboratory instrument maintenance, testing, and inspection requirements.

The maintenance responsibilities for fixed laboratory instruments will be assigned to the Laboratory Section Managers. Laboratory analysts will be responsible for daily checks and calibrations and for reporting any problems with the instruments. The maintenance schedule will follow the manufacturer's recommendations. Laboratory personnel will also be responsible for ensuring that critical parts are kept with the fixed laboratory instruments. Critical spare parts will be immediately available to reduce potential downtime. The inventory will primarily contain parts that are subject to frequent failure, have limited useful lifetimes, and/or cannot be obtained in a timely manner.

Annual preventative maintenance service visits will involve cleaning, adjusting, inspecting, and testing procedures designed to minimize product failure and/or extend the product's life. Between visits, laboratory analysts will be responsible for performing routine operator maintenance and cleaning in accordance with the manufacturer's specifications.

12.5 FIXED ANALYTICAL INSPECTION AND ACCEPTANCE REQUIREMENTS FOR SUPPLIES/SAMPLE CONTAINERS

Critical supplies and sample containers will be inspected in the following manner.

Critical Supplies and Consumables	Inspection Requirements and Acceptance Criteria	Responsible Individual
Sample bottles	Visually inspected upon receipt for cracks, breakage, cleanliness. Must be accompanied by certificate of analysis.	Sample Custodian
Chemicals and reagents	Visually inspected for proper labeling, expiration dates, appropriate grade Record lot numbers of reagents used for standard	Laboratory Analyst

Critical Supplies and Consumables	Inspection Requirements and Acceptance Criteria	Responsible Individual
	preparation.	

Supplies and consumables not meeting acceptance criteria will initiate the appropriate corrective action. Corrective measures may include notification of vendor and subsequent replacement of defective or inappropriate materials. All actions will be documented in the project files.

The use of materials of known purity and quality will be utilized for the calibration of all instruments as part of this project. The laboratories will carefully monitor the use of all laboratory materials including solutions, standards and reagents through well documented procedures.

All solid chemicals and acids/bases used by the laboratories will be reagent grade or better. All gases will be high purity or better. All standards or standard solutions will be obtained from U.S. Environmental Protection Agency certified commercial sources.

All materials including standards or standard solutions will be dated upon receipt, and will be identified by material name, lot number, purity or concentration, supplier, receipt/preparation date, recipient/preparer's name, and expiration date.

Standards or standard solution concentrations will be validated prior to use. Standards and standard materials will be checked for signs of deterioration including unusual volume changes (solvent loss), discoloration, formation of precipitates or changes in analyte response. All standards and standard solutions will be properly stored and handled and will be labeled with all appropriate information including compound/solution name, concentration, solvent, expiration date, preparation date, and the initials of the preparer.

All solvent materials or materials used as part of a given procedure will also be checked. Each new lot of solvent will be analyzed to ensure the absence of interference.

EPA-NE QAPP Worksheet #20:

Fixed Laboratory Analytical Method/SOP Reference Table

Reference Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrument	Modified for Project Work Y or N
L-1	Test America Laboratories, Inc.	Lab SOP ID: MES00200.MA (ICP), MES02800.MA (ICP-MS), MES00400.MA (Hg). Analysis of Low Concentration TAL Metals in Aqueous Samples by USEPA SW-846 6010B / 6020A (ICP-MS) / Hg by 7470A, USEPA 1996.	Definitive	6010B / 6020A /7470A	Metals	ICP, ICP-MS, CVAA	N
L-2	Test America Laboratories, Inc.	Lab SOP ID: MES00200.MA (ICP), MES02800.MA (ICP-MS), MES00400.MA (Hg). Analysis of Low Concentration Metals in Solid Samples (soil, sediment) by USEPA SW-846 6010B (ICP) / 6020A (ICP-MS) / Hg by 7471B, USEPA 1996.	Definitive	6010B / 6020A /7471B	Metals	ICP, ICP/MS, CVAA	N
L-3	Test America Laboratories, Inc.	Lab SOP ID: MSS00104.MA. Analysis of Volatile Organic Compounds in Aqueous and Solid matrix by USEPA SW-846 8260B, USEPA 1996.	Definitive	8260B	VOCs	GC/MS	N
L-4	Test America Laboratories, Inc.	Lab SOP ID: MSS00300.MA. Analysis of Semi-Volatile Organic Compounds in Aqueous and Solid matrix by USEPA SW-846 8270C and Low level 8270C, USEPA 1996.	Definitive	8270C	SVOCs	GC/MS	N
L-5	Test America Laboratories, Inc. (West Sacramento, CA)	Lab SOP ID: WS-MS-0012. Determination of Nitrosamines by Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (GC/CI/MS/MS), Rev 1. Modified EPA Method 521.	Definitive	Modified EPA 521	NDMA / NDPA	GC/CI/MS/MS	Y
L-6	Test America Laboratories, Inc.	Lab SOP ID: MSS00300.MA (addendum). Analysis of Nitroso-dimethylamine (NDMA) in Solid and Aqueous matrix by Modified by USEPA SW-846 8270C Low Level Method (addendum), USEPA 1996.	Definitive	8270C	NDMA	GC/MS	Y
L-7	Test America Laboratories, Inc.	Lab SOP ID: GCS00300.MA. Analysis of Extractable Petroleum Hydrocarbons in Solid matrix by MADEP EPH-98-1, May 2004.	Definitive	MADEP EPH-98-1	EPH	GC/FID	N

EPA-NE QAPP Worksheet #20:

Fixed Laboratory Analytical Method/SOP Reference Table

Reference Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrument	Modified for Project Work Y or N
L-8	Test America Laboratories, Inc.	Lab SOP ID: GCS00500.MA. Analysis of Volatile Petroleum Hydrocarbons in Solid matrix by MADEP VPH-98-1, May 2004.	Definitive	MADEP VPH-98-1	VPH	GC/FID/PID	N
L-11	Lancaster Labs, Inc.	Lab SOP ID: #2726 (Opex) and #2727 (Kempore). Analysis of Opex (Dinitrosopentamethylenetetramine) and Kempore (Azodicarbonamide) in Water by Method 8000B HPLC.	Definitive	Method 8000B	Opex and Kempore	HPLC	Y
L-12	Test America Laboratories, Inc	Lab SOP ID: INS05600.MA. Analysis of Sulfate by Ion Chromatography in Aqueous matrix by USEPA 300.0, Part A, EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	300.0	Sulfate	Lachat IC5000	N
L-13	Test America Laboratories, Inc	Lab SOP ID: INS05600.MA. Analysis of Chloride by Ion Chromatography in Aqueous and solid matrix by USEPA 300.0, Part A, EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	300.0	Chloride	Lachat IC5000	N
L-15	Test America Laboratories, Inc	Lab SOP ID: INS07300.MA. Analysis of Specific Conductivity, Alkalinity and/or pH using the ManSci MANTECH Autotitrator in Aqueous matrix by SM 2510B, SM 2320B, SM 4500, Standard Methods.	Definitive	Standard Methods 2510B, 2320B and 4500.	Specific Conductivity, Alkalinity and/or pH	ManSci MANTECH Autotitrator	N
L-16	Test America Laboratories, Inc	Analysis of Hardness in Aqueous matrix by SM 2340B, Standard Methods for the Determination of Water and Wastes. 18th Edition, 1992.	Definitive	SM 2340B	Hardness	NA	N
L-17	Test America Laboratories, Inc	Lab SOP ID: INS05600.MA. Analysis of Nitrate by Ion Chromatography in Aqueous and solid matrix by USEPA 300.0, Part A, EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	300	Nitrate	Lachat IC5000	N

EPA-NE QAPP Worksheet #20:

Fixed Laboratory Analytical Method/SOP Reference Table

Reference Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrument	Modified for Project Work Y or N
L-18	Test America Laboratories, Inc	Lab SOP ID: INS05600.MA. Analysis of Nitrite by Ion Chromatography in Aqueous and solid matrix by USEPA 300 EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	300	Nitrite, Fluoride, Bromide	Lachat IC5000	N
L-19	Test America Laboratories, Inc	Lab SOP ID: INS00900.MA. Analysis of Total Dissolved Solids (TDS) by USEPA 160.1 in Aqueous matrix, EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	160.1	TDS	NA	N
L-20	Test America Laboratories, Inc	Lab SOP ID: INS02700.MA. Analysis of Ammonia in Aqueous and solid matrix by QuikChem Method 10-107-06-1-A	Definitive	NA	Ammonia	NA	N
L-21	Test America Laboratories, Inc	Lab SOP ID: INS04300MA. Analysis of Chemical Oxygen Demand in Aqueous matrix by USEPA 410.4, EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	410.1	Chemical Oxygen Demand	NA	N
L-22	Test America Laboratories, Inc	Lab SOP ID: INS06900.MA. Analysis of Total Organic Carbon in Aqueous matrix by USEPA 415.1, EPA Methods for the Chemical Analysis of Waters and Wastes, 1983.	Definitive	415.1	Total Organic Carbon	NA	N
L-23	Test America Laboratories, Inc	Lab SOP ID: INS00200.MA. Analysis of Total Suspended Solids (TSS) by Standard Methods 2540D and EPA Method 160.2	Definitive	SM 2540D / 160.2	TSS	NA	N
L-24	Lancaster Labs, Inc	Lab SOP #10342 (water) and 10346 (soil). Determination of Hydrazine, Monomethylhydrazine, and 1,1-Dimethylhydrazine in Aqueous Samples by LC/MS/MS (SOP #10342, October 29, 2008) and Determination of Hydrazine, Monomethylhydrazine, and 1,1-Dimethylhydrazine in Soil Samples by LC/MS/MS (SOP #10346, February 10, 2009). SOPs are combined in L-24.	Definitive	Modified 8315	Hydrazine, MMH and UDMH	LC/MS/MS	Y

EPA-NE QAPP Worksheet #20:

Fixed Laboratory Analytical Method/SOP Reference Table

Reference Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrument	Modified for Project Work Y or N
L-25	Test America Laboratories, Inc.	Lab SOP ID: GCS00300.MA. Analysis of Extractable Petroleum Hydrocarbons in Aqueous matrix by MADEP EPH-98-1, May 2004.	Definitive	NA	EPH	GC/FID	N
L-26	Test America Laboratories, Inc.	Lab SOP ID: GCS00500.MA. Analysis of Volatile Petroleum Hydrocarbons in Aqueous matrix by MADEP VPH-98-1, May 2004.	Definitive	NA	VPH	GC/FID/PID	N
L-27	Test America Laboratories, Inc	Lab SOP ID: GCS00202.MA. Analysis of Poly Chlorinated Biphenyls in Aqueous matrix by method USEPA SW-846 8082A, USEPA 1996.	Definitive	8082A	PCBs	GC/ECD	N
L-28	Test America Laboratories, Inc	Lab SOP ID: GCS00202.MA. Analysis of Poly Chlorinated Biphenyls in Solid matrix by method USEPA SW-846 8082A, USEPA 1996.	Definitive	8082A	PCBs	GC/ECD	N
L-29	Test America Laboratories, Inc	Lab SOP ID: INS00200.MA. Analysis of Pesticides in Aqueous matrix by method by USEPA SW-846 8081, USEPA 1996.	Definitive	8081	Pesticides	GC/ECD	N
L-32	Test America Laboratories, Inc. (Tallahassee, Florida Branch)	Lab SOP ID: LC10:12.07.06:5. Analysis of Formaldehyde and Acetaldehyde in Aqueous and Solid matrix by USEPA SW-846 8315, USEPA 1996.	Definitive	NA	Formaldehyde and Acetaldehyde	HPLC	N
L-33	Analytics Laboratory, LLC.	Lab SOP ID: QA-H005. Analysis of Herbicides in Aqueous matrix by USEPA SW-846 8151, USEPA 1996.	Definitive	NA	Herbicides	GC/MS	N
L-34	Test America Laboratories, Inc. (Sacramento, California Branch)	Analysis of Alkylphenols (nonylphenol, nonylphenol diethoxylate, 4-nonylphenyl, nonylphenol isomers) by GCMS-SIM.	Definitive	NA	Nonylphenol, Nonylphenol diethoxylate, 4-nonylphenyl, Nonylphenol isomers	GCMS-SIM	N
L-35	Katahdin Analytical Services.	Lab SOP ID: CA-309. Direct Injection Method for the Determination of Dimethylformamide Using GC/NPD – EPA SW-846 Method 8033 Modified	Definitive	Modified 8033	N,N-dimethylformamide (DMF)	GC/NPD	Y

EPA-NE QAPP Worksheet #20:

Fixed Laboratory Analytical Method/SOP Reference Table

Reference Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrument	Modified for Project Work Y or N
L-36	Test America Laboratories, Inc. (Tallahassee, Florida)	Lab SOP ID: TL-LC-066, Rev 5 Analysis of Phthalic Acid and Maleic Acid in Water and Soil by HPLC, October 10, 2007.	Definitive	Modified 8000	Phthalic anhydride (acid)	LC	Y
L-37	Lancaster Labs, Inc.	Lab SOP # 6386, 6557. Rev 03. Determination of Perchlorate in Aqueous Samples and Soil Samples by LC/MS/MS	Definitive	6850	Perchlorate	LC/MS/MS	N
L-38	Test America Laboratories, Inc. (Edison, NJ)	Lab SOP ID: ED-WET-078. The Analysis of Hexavalent Chromium in Aqueous Samples and Digestates from Solid Samples Via Ion Chromatography by SW-846 Method 7199, April 22, 2008, Rev 5.	Definitive	7199	Hexavalent Chromium	IC	N
L-39	Test America Laboratories, Inc	Lab SOP ID: WE-WET-034. Analytical Procedures for the Total Carbon, Non-purgeable Organic Carbon (Total Organic Carbon) and Total Iorganic Carbon by Lloyd Kahn, November 5, 2008. Rev. 0	Definitive	Lloyd Kahn	Total Organic Carbon	NA	N

Notes:
GC/MS – Gas Chromatograph/Mass Spectrometer
ICP – Inductively Coupled Plasma LC – Liquid Chromatography
CVAA – Cold Vapor Atomic Absorption
HR – High Resolution MS - Mass Spectrometer
TBD – To Be Determined
NA – Not Applicable
GC/MS-SIM – Gas Chromatograph/Mass Spectrometer - Single Ion Monitoring

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
GC/MS	SVOCs	Cut column, change liner, and replace septa as needed. Manual tune if DFTPP not in criteria.	IC – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCC is out of criteria or when manual tune performed	IC – minimum RF of ≥ 0.050 for each SPCC, % RSD of ≤ 30% for each CCC. If RSD for an analyte is > 15% apply linear or quadratic method for quantitation	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	TBD	L-4, L-6, L-33
			CV – at the beginning of each 12 hour shift immediately after DFTPP tune.	CV – minimum RF of each SPCC ≥ 0.050, % RSD ≤ 20% for each CCC.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.		
GC/MS-SIM	Alkylphenols	Cut column, change liner, and replace septa as needed. Manual tune if DFTPP not in criteria.	IC – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCC is out of criteria or when manual tune performed	IC – minimum RF of ≥ 0.050 for each SPCC, % RSD of ≤ 35% for each CCC.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	TBD	L-34
			CV – at the beginning of each 12 hour shift immediately after DFTPP tune.	CV – minimum RF of each SPCC ≥ 0.050, % RSD ≤ 30% for each CCC.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
GC/MS	VOC Analysis	Bake out trap and column, manual tune if BFB tune not in criteria, change septa as needed, cut column as needed, change trap as needed	IC – instrument receipt, instrument change (new trap, column, etc.), when CCC does not meet criteria or when manual tune performed	IC – RSD for each CCC ≤ 30%, minimum mean RF for each SPCC as noted in 7.3.5.4 of method 8260B. If RSD for an analyte is > 15% apply linear or quadratic method for quantitation	Perform necessary equipment maintenance and check calibration standards	TBD	L-3
			CV – at beginning of each 12 hour shift immediately after BFB tune.	CV - %D for each CCC ≤ 20%, minimum RF for each SPCC as noted in 7.3.5.4 of method 8260B.	Perform necessary equipment maintenance and check calibration standards		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
GC/CI/MS/MS	NDMA / NDPA analysis in water	Cut column, change liner, and replace septa as needed.	IC – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCC is out of criteria or when manual tune performed	IC – minimum RF of ≥ 0.050, % RSD of ≤ 20% for NDMA and NDPA. If RSD for NDMA/NDPA is > 20% re-analyze the initial calibration	Perform necessary equipment maintenance and check calibration standards. Filament replacement may solve this issue, otherwise, replacing the ion source with a clean one will probably be necessary	TBD	L-5
			CV – at the beginning of each 12 hour shift.	CV - % Recovery of NDMA/NDPA 80-120, minimum RF of ≥ 0.050.	Perform necessary equipment maintenance and check calibration standards. Re-calibrate instrument.		
GC/FID	EPH	Cut column, change liner, and replace septa as needed. Clean FID per manufactures specifications.	IC – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCC is out of criteria.	IC- min correlation of 0.990. The %RSD must be ≤ 25.	Perform necessary equipment maintenance and check calibration standards	TBD	L-7, L-25

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
			CV – at the beginning of each day or after 10 samples.	CV - %D for each CCC ≤ 25%	Perform necessary equipment maintenance and check calibration standards. Re-calibrate instrument.		
GC/PID and FID	VPH	Cut column as needed, change trap as needed. Clean FID per manufactures specifications.	IC – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCC is out of criteria.	IC- min correlation of 0.990. The %RSD must be ≤ 25.	Perform necessary equipment maintenance and check calibration standards	TBD	L-8, L-26
			CV – at the beginning of each day or after 20 samples.	CV - %D for each CCC ≤ 25%	Perform necessary equipment maintenance and check calibration standards		
GC/NPD	N,N-Dimethylformamide (DMF)	Cut column as needed, change trap as needed. Clean NPD per manufactures specifications.	IC – Instrument receipt, instrument change (new column), when CV is out of criteria.	IC- min correlation of 0.995.	Perform necessary equipment maintenance and check calibration standards	TBD	L-35

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
			CV – at the beginning of each day or after 10 samples.	CV - %D $\leq$ 25%	Perform necessary equipment maintenance and check calibration standards		
ICP, CVAA	Metals Analysis	ICP: Check argon tank pressure, check tubing, nebulizer, clean plasma torch assembly, clean filters, check o-rings CVAA: Inspect pump windings, rotate and replace if necessary, check gases.	Initial Calibration: Daily, once every 24 hours or every time instrument set up	r $\geq$ 0.995 for multi-point calibration, ICP uses single point calibration	Perform necessary equipment maintenance and check calibration standards	TBD	L-1, L-2
			Initial Calibration Verification: Immediately after instrument calibration	90-110% of true value for ICP; 80-120% of true value for CVAA	Perform necessary equipment maintenance and check calibration standards		
			Continuing Calibration Verification: After every 10 samples or every 2 hours, whichever is more frequent, and at end of analytical sequence	90-110% of true value for ICP; 80-120% of true value for CVAA	Perform necessary equipment maintenance and check calibration standards		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
ICP/MS	Metals Analysis	Clean nebulizer, check pump tubing, replace disposables, check torch alignment.	Initial: Daily, every 24 hours or every time instrument is set up	r ≥ 0.995	Perform necessary equipment maintenance and check calibration standards	TBD	L-1, L-2
			Initial Calibration Verification: immediately after initial calibration	90-110% of true value	Perform necessary equipment maintenance and check calibration standards		
			Continuing Calibration Verification: after every 10 samples or every 2 hours, whichever is more frequent, and at end of analytical sequence	90-110% of true value	Perform necessary equipment maintenance and check calibration standards		
			Continuing Calibration Verification: after every 10 samples.	90-110% of true value	Perform necessary equipment maintenance and check calibration standards		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
HPLC	Opex and Kempore	Replace HPLC column, check pump tubing, replace disposables (tubing)	Initial Calibration: Daily or every time instrument set up	IC- min correlation of 0.990 if linear regression is used. The %RSD must be ≤ 20 if an RF is used.	Perform necessary equipment maintenance and check calibration standards	TBD	L-11
			Continuing Calibration Verification: after every 10 samples.	85-115% of true value	Perform necessary equipment maintenance and check calibration standards		
HPLC	Formaldehyde and Acetaldehyde	Replace HPLC column, check pump tubing, replace disposables (tubing)	Initial Calibration: Daily or every time instrument set up	IC- min correlation of 0.99.	Perform necessary equipment maintenance and check calibration standards	TBD	L-32
			Continuing Calibration Verification: after every 20 samples or 12-hours.	85-115% of true value	Perform necessary equipment maintenance and check calibration standards		
HPLC	Phthalic Anhydride (acid)	Replace HPLC column, check pump tubing, replace disposables (tubing)	Initial Calibration: Daily or every time instrument set up	IC- min correlation of 0.990 if linear regression is used. The %RSD must be ≤ 10 if an RF is used.	Perform necessary equipment maintenance and check calibration standards	TBD	L-36

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
			Continuing Calibration Verification: after every 10 samples at the beginning and end of the analytical sequence.	80-120% of true value	Perform necessary equipment maintenance and check calibration standards		
Elemental Analyzer	TOC Analysis	Clean system, replace disposables, check oxygen flow	Initial: Daily prior to sample analysis	$r \geq 0.995$	Perform necessary equipment maintenance and check calibration standards	TBD	L-22
			Continuing: Every 10 samples and at the end of the sequence	$\pm$ 10% of true value	Perform necessary equipment maintenance and check calibration standards		
Lachat IC5000	Chloride, nitrate, nitrite, sulfate, bromide and fluoride	Clean system, replace disposables. Inspect outer and inner chamber for cleanliness, check tubing, check flow of reagents, calibrate by outside vendor	Initial: Daily prior to sample analysis	Linear Regression = $r \geq 0.995$	Perform necessary equipment maintenance and check calibration standards	TBD	L-13, L-12, L-17, L-18, L-24
			Continuing: After every 10 samples.	90 – 110% of true value	Perform necessary equipment maintenance and check calibration standards		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
GC-ECD	PCB/Pesticide/Herbicides Analysis	Initial Calibration	*Prior to analysis of initial project samples and as needed afterward*	RSD ≤20% or linear correlation coefficient >0.99 for each target compound	*Check system. Document corrective action Repeat as needed to meet criteria prior to analysis of any samples*	TBD	L-27, L-28, L-29, L-33
		Initial Calibration Verification	*Prior to analysis each 12 hour shift*	Relative difference or drift ≤15% from initial calibration	*Check system. Document corrective action. Repeat ICV analysis. Repeat initial calibration*		
		Continuing Calibration Check	Every 10 samples	Relative difference or drift ≤15% from initial calibration or mean %difference or drift for all targets <15%	Check system. Document corrective action. Repeat calibration check		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
LC/MS/MS	Perchlorate	Initial Calibration	*Prior to analysis of initial project samples and as needed afterward*	RSD ≤15% or linear correlation coefficient >0.995	*Check system. Document corrective action Repeat as needed to meet criteria prior to analysis of any samples*	TBD	L-37
		Initial Calibration Verification	*After every IC*	Relative difference or drift ≤15% from initial calibration	*Check system. Document corrective action. Repeat ICV analysis. Repeat initial calibration*		
		Continuing Calibration Check	*Every 10 samples*	Relative difference or drift ≤15% from initial calibration for mid-level standard and ≤50%	*Check system. Document corrective action. Repeat calibration check*		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
Metrohm MIC (IC)	Hexavalent Chromium	Initial Calibration	*Prior to analysis of initial project samples and as needed afterward*	Linear correlation coefficient ≥0.999	*Check system. Document corrective action Repeat as needed to meet criteria prior to analysis of any samples*	TBD	L-38
		Initial Calibration Verification	*After every IC*	Within 10% of true value	*Check system. Document corrective action. Repeat ICV analysis. Repeat initial calibration*		
		Continuing Calibration Check	*Every 10 samples*	Within 10% of true value	*Check system. Document corrective action. Repeat calibration check*		

EPA-NE QAPP Worksheet #21

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	Method/SOP Reference
LC/MS/MS	Hydrazine, MMH and UDMH	Initial Calibration	*Prior to analysis of initial project samples and as needed afterward*	Quadratic or linear correlation coefficient >0.99	*Check system. Document corrective action Repeat as needed to meet criteria prior to analysis of any samples*	TBD	L-24
		Initial Calibration Verification	*After every IC*	Relative difference or drift ≤30% from initial calibration	*Check system. Document corrective action. Repeat ICV analysis. Repeat initial calibration*		
		Continuing Calibration Check	*Every 10 samples*	Relative difference or drift ≤30% from initial calibration	*Check system. Document corrective action. Repeat calibration check*		

NOTES:
TBD = To Be Determined
NA = Not Applicable
IC = Ion Chromatography
LC = Liquid Chromatography
ICP-MS = Inductively Coupled Plasma-Mass Spectrometry
GC/MS = Gas Chromatography- Mass Spectrometry
GC-ECD = Gas Chromatography-electron capture detector
CVAA = Cold Vapor Atomic Absorption

Prepared by: TC
Checked by: CR
10/14/08

13.0 QUALITY CONTROL REQUIREMENTS

13.1 SAMPLING QUALITY CONTROL

This section of the QAPP identifies the QC procedures, checks, samples, and their respective acceptance limits, that will be used to monitor the quality of various aspects of the sampling event. Their required analysis frequency, acceptance limits and corrective actions are also documented in this section of the QAPP. Worksheet #22a summarizes this information for each matrix, analytical parameter and sampling method. It should be noted that Bottle Blanks are not being submitted for analysis during this investigation. All bottles will be certified clean from the manufacturer and the certifications will be stored in the project file. Potential contaminants due to the sample container will also be detected in the equipment blanks described below.

13.1.1 Equipment Blanks

Internal quality control checks will include analysis of equipment blanks to check for procedural contamination at the site that may cause sample contamination. Equipment blanks will be prepared by routing deionized water through sampling equipment after equipment decontamination and before field sample collection. Equipment blanks will be submitted at a frequency of one per twenty samples, per matrix and per parameter. It should be noted that equipment blanks will not be collected for any parameters associated with residential well samples collected directly from taps, groundwater samples collected using dedicated equipment, surface water samples that are collected as direct grabs, and TOC analyses of soil and/or sediment samples.

13.1.2 Cooler Temperature Blanks

Cooler temperature blanks consist of a sample container filled with non-preserved water (potable or distilled) and are included in all coolers which contain samples which require temperature preservation. The laboratory uses these temperature blanks to measure temperature of the samples during sample shipment. The temperature goal for these blanks is 4 ºC ±2º. The laboratory records the results of the temperature blanks on the chain-of-custody or sample login form immediately upon receipt of the samples at the laboratory, prior to inventory and refrigeration. If temperature blanks are outside this range, the laboratory will contact the MACTEC QA Officer to determine if sample analysis will continue. It is

possible that sample temperatures may exceed temperature goals if shipped the same day as collection and equilibrium with cooler ice has not been reached. The QA Officer will examine field collection information and use professional judgment.

13.1.3 Field Duplicates

Field duplicates can be divided into two categories: duplicate subsamples and collocated samples. Duplicate subsamples are an additional aliquot of the same sample submitted for the same parameters as the original sample and will be used for aqueous and solid matrices. Duplicate subsamples will be collected by alternately filling sample bottles from the source being sampled. Field duplicates will be used to assess the sampling and analytical precision. Field duplicates will be submitted at a frequency of one per twenty investigative samples, per matrix and analytical parameter. Collocated samples are two samples collected next to each other in the same vertical position. Collocated samples require two separate sample collections at the same location. The collection of collocated samples is not anticipated during sample activities at the Site.

13.1.4 Field Blanks

Field blanks will be used to evaluate the potential for contamination as a result of the water used to decontaminate equipment during sampling activities. Field blanks will be collected for all parameters associated with sampling methods that utilized equipment that is decontaminated with DI water. A water field blank will be collected from the source of DI water used during field investigation programs. One field blank will be collected per sampling event per method.

13.2 ANALYTICAL QUALITY CONTROL

This section identifies the QC procedures, checks, and samples, and their respective acceptance limits, that will be used during the project to monitor the quality of various preparatory and analytical steps. Worksheet #23a summarizes this information for each matrix and field analytical parameter where QC procedures are required. Worksheet #24a summarizes this information for each matrix and fixed laboratory analytical parameter.

13.2.1 Field Analytical QC

Instrument blanks are required to be analyzed with the FID or PID to evaluate potential contamination resulting from the instrumentation.

13.2.2 HACH DR-2800 Spectrophotometer Analytical QC

Field analytical QC samples collected for Ammonia analysis by the HACH DR-2800 Spectrophotometer are presented in Worksheet #23a and Worksheet #23b.

13.2.3 Fixed Laboratory QC

All required QC checks and QC samples and the associated QC acceptance limits are detailed in the associated USEPA SW-846, or associated EPA methods.

13.2.3.1 Method Blanks

Method blanks will be performed as part of each analytical batch for each methodology performed. Method blanks are used to evaluate contamination introduced during sample preparation and/or analysis by the laboratory.

13.2.3.2 Instrument Blanks

Instrument blanks are used to evaluate contamination resulting from the analytical reagents and the instrumentation. In addition, instrument blanks are sometimes used to assess potential carryover after the analysis of a highly contaminated sample. Instrument blanks are only required for select analytical parameters.

13.2.3.4 Matrix Spike Samples

The matrix spike samples are used to determine laboratory preparation and analysis bias for specific compounds in specific matrices (i.e., sample specific QC). Matrix spikes are typically performed at a frequency of one per twenty investigative samples.

13.2.3.5 Surrogate Spikes

Surrogate spikes are used to evaluate extraction efficiency or analytical bias on a sample by sample basis for organic parameters. Surrogate spikes are added to all samples for organic parameters. Surrogate spikes are another measure of sample-specific QC.

13.2.3.6 Laboratory Control Samples

LCSs are used to evaluate the ability of the laboratory to accurately identify and quantify target compounds in a clean laboratory. The LCS are prepared from a secondary source standard. LCSs are performed as part of each analytical batch for each methodology.

13.2.3.7 Performance Evaluation Samples

Performance evaluation samples (PESs) are used to evaluate sample handling procedures from the field to the laboratory and to evaluate the laboratory's ability to accurately identify and quantify target compounds in a reference matrix. PESs for VOCs, PCBs, SVOCs, and metals will be provided by EPA Region I and will be submitted at the beginning of the sampling program. If the laboratory is unable to produce passing score results, then PESs will be re-submitted to the laboratory. It should be noted that PESs are not always available for all parameters and matrices. If necessary, water PESs will be submitted with soil samples. PESs for this program will be single blind PES.

13.2.3.8 Laboratory Duplicate

Laboratory duplicates are used to evaluate laboratory preparation and analysis precision. These analyses are typically performed for inorganic parameters only. Laboratory duplicates are performed at a frequency of one per twenty samples.

13.2.3.9 Matrix Spike Duplicate Samples

MSDs are used to evaluate laboratory preparation and analysis bias and precision for specific compounds in specific sample matrices (i.e., sample specific QC). MSDs are performed for organic parameters only.

13.2.3.10 Internal Standards

Internal standards are used to qualify target analytes and measure instrument stability. Internal standards are typically only used for GC/MS organic analyses and ICP/MS analyses. Internal standards are spiked into all samples and are considered a sample-specific QC measure.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2
Medium/Matrix	GW/SW
Analytical Parameter	VOCs
Concentration Level	Low
Analytical Method/SOP Reference	L-3, SW-846 8260B
Sampler's Name	TBD
Field Sampling Organization	MACTEC
No. of Sample Locations	TBD

Field QC:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be < 10x QL and acetone and 2-butonone must be < 2x QL
Trip Blanks	1 per shipment	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be < 10x QL and acetone and 2-butonone must be < 2x QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous) at the beginning of sample program.	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	SVOCs, NDMA					
Concentration Level	Low					
Analytical Method/SOP Reference	L-4, L-5 SW-846 8270C (SVOCs), Modified EPA 521 (NDMA/NDPA), Modified 8270C Low Level					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL (except phthalates must be ≤ 5x QL)
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous) for SVOCs at the beginning of sample program. No PES for NDMA/NDPA.	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2
Medium/Matrix	SW, GW,
Analytical Parameter	Pesticides, PCBs, Herbicides
Concentration Level	Low
Analytical Method/SOP Reference	L-27, L-29 SW-846-8081 (Pesticides), 8082 (PCBs), 8151A (Herbicides)
Sampler's Name	TBD
Field Sampling Organization	MACTEC
No. of Sample Locations	TBD

Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning of sample program.	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW,					
Analytical Parameter	Extractable Petroleum Hydrocarbons (EPH), Volatile Petroleum Hydrocarbons (VPH)					
Concentration Level	Low					
Analytical Method/SOP Reference	L-25, L-26, MADEP EPH-2004 (EPH), MADEP VPH-2004 (VPH)					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	1 per shipment-VPH	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW/GW					
Analytical Parameter	TAL Metals					
Concentration Level	Low					
Analytical Method/SOP Reference	L-1, L-2, SW-846 6010B/ 6020A/Hg by 7470A					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4^oC, ± 2^oC	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4^oC, ± 2^oC
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning of sample program.	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample. *Applies only to dissolved metals; not applicable to total metals.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW/GW					
Analytical Parameter	Hexavalent Chromium					
Concentration Level	Low					
Analytical Method/SOP Reference	L-38, SW-846 7199					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	Sulfate, Chloride, Nitrate, Nitrite, Fluoride, Bromide					
Concentration Level	Low					
Analytical Method/SOP Reference	L-12, L-13,L-17, L-18, USEPA 300 (Sulfate, Chloride, Nitrate, Nitrite, Fluoride, Bromide)					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW/GW					
Analytical Parameter	Alkalinity, TSS, TDS, Hardness, Ammonia, pH, Chemical Oxygen Demand (COD), Total Organic Carbon (TOC), Specific Conductance.					
Concentration Level	Low					
Analytical Method/SOP Reference	L-15, L-16, L-19, L-20, L-21, L-22, L-23 SM 2320B (Alkalinity), SM 2340B (Hardness), Quick Chem Method 10-107-06-1-A (Ammonia), SM 2540C (TDS), SM 2540D (TSS), SM 4500 (pH), 410.4 (COD), SM 5310B (TOC), SM 2510B (Specific Conductance)					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinseate Blanks*	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	Formaldehyde and Acetaldehyde					
Concentration Level	Low					
Analytical Method/SOP Reference	L-32, Analysis of Formaldehyde and Acetaldehyde by USEPA SW-846 8315					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	Hydrazine, MMH, UDMH					
Concentration Level	Low					
Analytical Method/SOP Reference	L-24, Analysis of Hydrazine, MMH, and UDMH by Modified Method 8315					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	Phthalic anhydride (acid)					
Concentration Level	Low					
Analytical Method/SOP Reference	Modified 8000 HPLC/ L-36					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target compounds ≥ QL	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	N,N-Dimethylformamide (DMF)					
Concentration Level	Low					
Analytical Method/SOP Reference	Modified 8033 GC/NPD / L-35					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target compounds ≥ QL	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	Nonylphenols (alkylphenols)					
Concentration Level	Low					
Analytical Method/SOP Reference	Modified 8270 GC/MS-SIM / L-34					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1, S-2					
Medium/Matrix	SW, GW					
Analytical Parameter	Opex and Kempore					
Concentration Level	Low					
Analytical Method/SOP Reference	L-11, Analysis of Opex and Kempore, Lancaster Lab Specific Method					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-1					
Medium/Matrix	GW					
Analytical Parameter	Perchlorate					
Concentration Level	Low					
Analytical Method/SOP Reference	SW-846 Method 6850 / L-37					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks*	5%	Not Applicable	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 2x QL *RPD ≤ 30 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Notes: * = Rinse blanks / Equipment blanks are not collected when dedicated sampling equipment is used to collect sample.

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16, S-13					
Medium/Matrix	SO/SD					
Analytical Parameter	VOCs					
Concentration Level	Low/medium					
Analytical Method/SOP Reference	L-8, SW-846 5035/8260B					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be < 2.5x QL and acetone and 2-butanone must be < 5x QL)
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	1 per cooler	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be < 2.5x QL and acetone and 2-butanone must be < 5x QL)
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning of sample program	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	SVOCs					
Concentration Level	Low					
Analytical Method/SOP Reference	L-4, SW-846 8270C					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL (except phthalates must be ≤ 5x QL)
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning of sample program.	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO					
Analytical Parameter	PCBs					
Concentration Level	Low					
Analytical Method/SOP Reference	L-28, SW-846 8082					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds $\geq$ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, $\pm$ 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, $\pm$ 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD $\leq$ 50 when positive results for both samples are $\geq$ 2x QL *RPD $\leq$ 50 when positive result for one sample is $\geq$ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning of sample program	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	Extractable Petroleum Hydrocarbons (EPH), Volatile Petroleum Hydrocarbons (VPH)					
Concentration Level	Low					
Analytical Method/SOP Reference	L-7, L-8, Extractable Petroleum Hydrocarbons (EPH), Volatile Petroleum Hydrocarbons (VPH)					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds $\geq$ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	$4^{o}C, \pm 2^{o}C$	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	$4^{o}C, \pm 2^{o}C$
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD $\leq$ 50 when positive results for both samples are $\geq$ 2x QL *RPD $\leq$ 50 when positive result for one sample is $\geq$ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning of sample program	No false negatives, no false positives, quantitation within warning limits	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	TAL Metals					
Concentration Level	Low					
Analytical Method/SOP Reference	L-2, SW-846 6010B / 6020A (TAL Metals)/Hg by7471					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target analytes $\geq$ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	$4^{o}C, \pm 2^{o}C$	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	$4^{o}C, \pm 2^{o}C$
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD $\leq$ 50 when positive results for both samples are $\geq$ 2x QL *RPD $\leq$ 50 when positive result for one sample is $\geq$ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	One per matrix (aqueous, solid) at the beginning, middle, and end of sample program.	No false negatives, no false positives, quantitation within warning limits.	Qualify data and direct laboratory to investigate problem	Data Validator and Laboratory QA/QC Manager	Accuracy / bias	No false negatives, no false positives, quantitation within warning limits
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	Haxavalent Chromium					
Concentration Level	Low					
Analytical Method/SOP Reference	L-38, SW-846 3060A/7199					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds $\geq$ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, $\pm$ 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, $\pm$ 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD $\leq$ 50 when positive results for both samples are $\geq$ 2x QL *RPD $\leq$ 50 when positive result for one sample is $\geq$ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	Ammonia, Total Organic Carbon (TOC), Chloride, Nitrate, Nitrite, Sulfate					
Concentration Level	Low					
Analytical Method/SOP Reference	L-20, L-39, L-13, L-17, L-18, L-12 Quick Chem Method 10-107-06-1-A (Ammonia), Lloyd Kahn (TOC), Chloride, Nitrate, Nitrite, Sulfate					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinseate Blanks	5%	No target analytes ≥ QL	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	$4^{o}C$, ± $2^{o}C$	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	$4^{o}C$, ± $2^{o}C$
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL *Absolute difference < 2x QL when positive results for both samples are < 5x QL *RPD ≤ 30 when positive result for one sample is ≥ 5x QL and positive result for other sample is < 5x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	Formaldehyde and Acetaldehyde					
Concentration Level	Low					
Analytical Method/SOP Reference	L-32, Analysis of Formaldehyde and Acetaldehyde by USEPA SW-846 8315					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	Hydrazine, MMH, UDMH					
Concentration Level	Low					
Analytical Method/SOP Reference	L-24, Analysis of Hydrazine, MMH, and UDMH by Modified Method 8315					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or quality data.	Field Sampler and Data Validator	Accuracy/bias-contamination	No target analytes $\geq$ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1per cooler	$4^{o}C, \pm 2^{o}C$	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	$4^{o}C, \pm 2^{o}C$
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD $\leq$ 50 when positive results for both samples are $\geq$ 2x QL *RPD $\leq$ 50 when positive result for one sample is $\geq$ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	Phthalic anhydride (acid)					
Concentration Level	Low					
Analytical Method/SOP Reference	Modified 8000 HPLC/ L-36					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4ºC, ± 2ºC	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4ºC, ± 2ºC
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	N,N-dimethylformamide (DMF)					
Concentration Level	Low					
Analytical Method/SOP Reference	Modified 8033 GC/NPD / L-35					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds $\geq$ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	$4^{o}C, \pm 2^{o}C$	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	$4^{o}C, \pm 2^{o}C$
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD $\leq$ 50 when positive results for both samples are $\geq$ 2x QL *RPD $\leq$ 50 when positive result for one sample is $\geq$ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

EPA-NE QAPP Worksheet #22a

Field Sampling QC Table

Sampling SOP	S-3, S-15, S-16					
Medium/Matrix	SO/SD					
Analytical Parameter	NDMA					
Concentration Level	Low					
Analytical Method/SOP Reference	L-6, SW-846 Modified 8270C low level					
Sampler's Name	TBD					
Field Sampling Organization	MACTEC					
No. of Sample Locations	TBD					
Field QC:	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	5%	Not Applicable	Reclean, retest, resample, and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Contamination	No target compounds ≥ QL
Bottle Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Trip Blanks	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Cooler Temperature Blanks	1 per cooler	4°C, ± 2°C	Resample and/or qualify data	Field Sampler and Data Validator	Accuracy / bias-Preservation	4°C, ± 2°C
Field Duplicate Pairs	5%	Not Applicable	Resample and/or qualify data	Field Sampler and Data Validator	Assess sampling and laboratory precision	*RPD ≤ 50 when positive results for both samples are ≥ 2x QL *RPD ≤ 50 when positive result for one sample is ≥ 2x QL and positive result for other sample is < 2x QL
Collocated Samples	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Field Splits	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
PES sent to Laboratory	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:_____						

EPA-NE QAPP Worksheet #23a
Field Analytical QC Sample Table

Medium/Matrix	SW					
Sampling SOP	S-2					
Analytical Parameter	Ammonia analysis using the HACH DR 2800					
Concentration Level	Medium					
Analytical Method/ SOP Reference	F-7					
Field Analytical Organization	MACTEC					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	Once Daily	No detects > detection limit for Ammonia	If detects > QL then re-analyze associated samples with new method blank	Analyst	Accuracy/bias-contamination	No detects > detection limit for specific metal
Instrument Blank	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Storage Blank	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Lab Duplicate	Daily	*RPD ≤ 30 when positive results for both samples are ≥ 5x QL	Assess laboratory precision and resample and/or qualify data	Analyst	Precision	*RPD ≤ 50 when positive results for both samples are ≥ 5x QL
Calibration Verification Check	Beginning of each day and end of each day.	±10% of target	If outside of 10%, re-analyze check standard, if still out then re-calibrate instrument or estimate field result	Analyst	Accuracy	±10% of target
Laboratory Matrix Spike	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Matrix Spike Duplicates	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
LCS	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
LFB	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Surrogates	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Internal Standards (ISs)	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Other:						

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #23b – On-site analysis of ammonia using the HACH DR-2800 spectrophotometer

Sampling SOP: S-2
Analytical Method/SOP: Aqueous Analysis for Ammonia by HACH Method 8038, F-7.

Field Analytical QC Sample Table cont.

Analyte	Achievable Sensitivity/ Quantitation Limits (mg/L) [1]	Field Analytical Precision	Field Analytical Accuracy/Bias
Ammonia	0.02	RPD ≤ 30%	90-110%

1 – Method Range for Ammonia by HACH 8038 is 0.02 – 2.5 mg/L

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	GW/SW
Sampling SOP	S-1, S-2
Analytical Parameter	VOCs
Concentration Level	Low
Analytical Method/ SOP Reference	SW-8260B / L-3
Laboratory Name	Test America Laboratories, Inc.
No. of Sample Locations	TBD

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every 12 hours prior to sample analysis	No target compounds ≥ QL (except methylene chloride and cyclohexane must be <10x QL and acetone and 2-butanone must be < 2 x QL)	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be <10x QL and acetone and 2-butanone must be < 2 x QL)
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 70-130% RPD ≤ 30% with MSD
Matrix Spike Duplicates	One per 20	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ precision//bias	Percent recovery 70-130% RPD ≤ 30% with MS
LCS	One per analysis day	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy	Percent recoveries 70-130%
Surrogates	3 per sample	NS	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 70-130%
Internal Standards (ISs)	6 per sample	Area counts: - ± 50% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	Area counts: - ± 50% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard
Other: Continuing Calibration Check compounds (CCC)	Every 12 hours	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	GW/SW					
Sampling SOP	S-1, S-2					
Analytical Parameter	VOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-8260B / L-3					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Instrument Tune with DFTPP	Beginning of the analytical run or every 12hrs	Acceptance limits specified in method	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	SVOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8270C / L-4					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every extraction batch	No target compounds ≥ QL (except phthalates must be ≤ 5x QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL (except phthalates must be ≤ 5x QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL (except phthalates must be ≤ 5x QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL (except phthalates must be ≤ 5x QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One per 20	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery
Matrix Spike Duplicates	One per 20	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ Precision/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery and RPDs ≤20
LCS	One per batch	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery
Surrogates	6 per sample	Acid Compounds: 15-110% Recovery Base-Neutral Compounds: 30-130% Recovery	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 15-110% Recovery Base-Neutral Compounds: 30-130% Recovery

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	SVOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8270C / L-4					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Internal Standards (ISs)	6 per sample	Area counts: -50% ±100% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	Area counts: -50% ±100% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard
Other: Continuing Calibration Check compounds (CCC)	Every 12 hours	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	Beginning of the analytical run or every 12hrs	Acceptance limits specified in method	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	PCB/Pesticides					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8082 (PCB) and 8081 (Pesticides) / L-27, L-29					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	70-130% or lab specified limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 30-150% for Pesticides and a RPD ≤ 30% with MS. Percent recovery 40-140% for PCBs and a RPD ≤ 50% with MS.
Matrix Spike Duplicates	One per 20	70-130% or lab specified limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 30-150% for Pesticides and a RPD ≤ 30% with MSD. Percent recovery 40-140% for PCBs and a RPD ≤ 50% with MSD.
LCS	One per batch	70-130% or lab specified limits	Reanalyze and qualify data	Analyst and Data Validator	Precision	Percent recovery 40-140%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	PCB/Pesticides					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8082 (PCB) and 8081 (Pesticides) / L-27, L-29					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Surrogates	3 per sample	NS	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries Percent recovery 30-150%
Continuing Calibration Verification (CCV)	Every 12 hours or 10 samples and at the beginning and end of analytical sequence	Calibration factor (CF) ≤ 15 %D from the mean initial calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	CF ≤ 25 %D from the initial calibration standard

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
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10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	GW/SW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Herbicides					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8151A / L-33					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	Percent recovery 30-150%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 30-150% RPD ≤ 30% with MSD
Matrix Spike Duplicates	One per 20	Percent recovery 30-150%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ precision/bias	Percent recovery 30-150% RPD ≤ 30% with MS
LCS	One per batch	Percent recovery 30-150%	Reanalyze and qualify data	Analyst and Data Validator	Precision	Percent recoveries 40-140%
Surrogates	1 per sample	Percent recovery 30-150%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 30-150%
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 12 hours or 10 samples	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	GW/SW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Extractable Petroleum Hydrocarbon (EPH)					
Concentration Level	Low					
Analytical Method/ SOP Reference	MADEP EPH-2004 / L-25					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20 samples	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 40-140% RPD ≤ 50% with MSD
Matrix Spike Duplicates	One per 20 samples	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 40-140% RPD ≤ 50% with MS
LCS	One per batch	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Precision	Percent recoveries 40-140%
Surrogates	1 per sample	Percent recovery 40-140%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 40-140%
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

NS = Not Specified by method

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	GW/SW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Volatile Petroleum Hydrocarbon (VPH)					
Concentration Level	Low					
Analytical Method/ SOP Reference	MADEP VPH-2004 / L-26					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	One per 20	RPD ≤ 50	qualify data	Data Validator	Precision	RPD ≤ 50
Matrix Spike	One per SDG	Percent recoveries 70-130%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 70-130% RPD ≤ 50% with MSD
Matrix Spike Duplicates	One per SDG	Percent recoveries 70-130%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 70-130% RPD ≤ 50% with MS
LCS	One per analysis day or SDG	Percent recoveries 70-130%	Reanalyze and qualify data	Analyst and Data Validator	Precision	Percent recoveries 70-130%
Surrogates	1 per sample	Percent recoveries 70-130%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 70-130%
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	%D≤ 25% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	%D between the initial calibration ≤ 25%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	TAL Metals					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 6010B (ICP)/6020A (ICP/MS)/Hg by 7470A / L-1					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One per preparation batch	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Reagent Blank**	ICB: immediately after ICV CCB: every 10 samples immediately after CCV	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD < 20 if results ≥ 5x QL	Qualify data	Analyst and Data Validator	Precision	RPD < 20 if results ≥ 5x QL
Laboratory Matrix Spike	One per 20	Percent recoveries 75-125%	Qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75-125%
Matrix Spike Duplicates	NA	NA	NA	NA	NA	NA
LCS	One per batch	Percent recoveries 80-120%	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80-120%
ICV/CCV	Every ten samples, beginning and end of each run.	±10% of true value for all metals, except Hg is ±20%	Reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	90-110% except Hg is 80-120%
Surrogates	NA	NA	NA	NA	NA	NA

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	TAL Metals					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 6010B (ICP)/6020A (ICP/MS)/Hg by 7470A / L-1					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Internal Standards (ISs) (ICP/MS Only)	Every sample	30-120% of IS in calibration blank	Dilute sample 5x, add IS and reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	30-120% of IS in calibration blank
Other: Serial Dilution	One per SDG	Within 10% of original determination	Qualify data	Analyst and Data Validator	Accuracy/bias	Within 5% of original determ
Other: Quantitation Limit Check Standard	Beginning and end of curve	Percent recoveries 70-130%	Recalibrate and reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 50-150%
Other: Interference Check Sample	Beginning and end of run not to exceed 8 hours	Percent recoveries 80-120%.	Recalibrate and reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80-120%, Profession judgment used for evaluation of positive or negative interference if Al, Ca, Fe, Mg are at concentrations in the sample that are >50% of the ICS concentration.

Prepared by: TC
Checked by: CR
10/14/08

** Also referred to as initial and continuing calibration blanks.
TBD = To Be Determined
NA = Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Hexavalent Chromium					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 7199, L-38					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One per preparation batch	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Reagent Blank**	ICB: immediately after ICV CCB: every 10 samples immediately after CCV	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD < 20 if results ≥ 5x QL	Qualify data	Analyst and Data Validator	Precision	RPD < 20 if results ≥ 5x QL
Predigestion Laboratory Matrix Spike	One per 20	Percent recoveries 85-115%	Qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75-125%
Matrix Spike Duplicates	NA	NA	NA	NA	NA	NA
Post Verification Spike (PVS)	One per batch or one per 20	Percent recoveries 85-115%	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 85-115%
LCS (soluble)	One per batch or one per 20	Within certified accepted range	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75-125%
LCS (insoluble)	One per batch or one per 20	Percent recoveries 80-120%	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80-120%
ICV/CCV	Every ten samples, beginning and end of each run.	±10% of true value	Reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	90-110%

** Also referred to as initial and continuing calibration blanks. NA = Not Applicable

Prepared by: TC
Checked by: CR
04/24/09

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Chloride, Sulfate, Nitrate, Nitrite, Fluoride, Bromide					
Concentration Level	Low					
Analytical Method/ SOP Reference	USEPA 300 (Chloride, Sulfate, Nitrate, Nitrite, Fluoride, Bromide) / L-13, L-17, L-18					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< QL for each anion	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch prior to samples and after last calibration standard	No target analytes ≥ ½ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One per batch	Percent recoveries 75 – 125%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75 – 125%
Matrix Spike Duplicates	NA	RPD ≤ 20	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	RPD ≤ 20
LCS	One every analytical batch prior to samples	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Chloride, Sulfate, Nitrate, Nitrite, Fluoride, Bromide					
Concentration Level	Low					
Analytical Method/ SOP Reference	USEPA 300 (Chloride, Sulfate, Nitrate, Nitrite, Fluoride, Bromide) / L-13, L-17, L-18					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
	and after last calibration standard					
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Alkalinity, Hardness					
Concentration Level	Low					
Analytical Method/ SOP Reference	SM 2340B (Hardness), SM 2320B (Alkalinity) / L-16, L-16					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 1.0uS/cm	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch prior to samples and after last calibration standard	No target analytes ≥ ½ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One per batch	Percent recoveries 75 – 125%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75 – 125%
Matrix Spike Duplicates	NA	NA	NA	NA	NA	NA
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Alkalinity, Hardness					
Concentration Level	Low					
Analytical Method/ SOP Reference	SM 2340B (Hardness), SM 2320B (Alkalinity) / L-16, L-16					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined NA – Not Applicable

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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	pH, Specific Conductance					
Concentration Level	Low					
Analytical Method/ SOP Reference	SM 4500 (pH), SM 2510B (Specific Conductance) / L-15					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 1.0uS/cm for Conductivity	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	NA	NA	NA	NA	NA	NA
Matrix Spike Duplicates	NA	NA	NA	NA	NA	NA
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Total Dissolved Solids (TDS), Total Suspended Solids (TSS)					
Concentration Level	Low					
Analytical Method/ SOP Reference	SM 2540C (TDS), SM 2540D (TSS) / L-19, L-23					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 10 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	NA	NA	NA	NA	NA	NA
Matrix Spike Duplicates	NA	NA	NA	NA	NA	NA
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Ammonia					
Concentration Level	Low					
Analytical Method/ SOP Reference	Quick Chem Method 10-107-06-1-A / L-20					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 0.1 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch prior to samples and after last calibration standard	< 0.1 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One per distillation batch	Percent recoveries 75 – 125%	Reanalyze and/or qualify data	Analyst and Data Validator	Accuracy	Percent recoveries 75 – 125%
Matrix Spike Duplicates	One per distillation batch	Percent recoveries 75 – 125%	Reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/precision	Percent recoveries 75 – 125%
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Ammonia					
Concentration Level	Low					
Analytical Method/ SOP Reference	Quick Chem Method 10-107-06-1-A / L-20					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Chemical Oxygen Demand (COD), Total Organic Carbon (TOC)					
Concentration Level	Low					
Analytical Method/ SOP Reference	USEPA 410.4 (COD), SM 5310B (TOC) / L-21, L-22					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 20 mg/L (QL)	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	< 20 mg/L (QL)	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One every analytical batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One every analytical batch	Percent recoveries 75 – 125%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%
Matrix Spike Duplicates	One every analytical batch	Percent recoveries 75 – 125%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/precision/bias	Percent recoveries 80– 120%
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Chemical Oxygen Demand (COD), Total Organic Carbon (TOC)					
Concentration Level	Low					
Analytical Method/ SOP Reference	USEPA 410.4 (COD), SM 5310B (TOC) / L-21, L-22					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Formaldehyde, Acetaldehyde					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8315 / L-32					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Reagent/Method Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike Sample	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	75-125%
LCS	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%
Surrogates	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RSD≤ 15% for each analyte

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
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10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Hydrazine, MMH, UDMH					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified Method 8315 / L-24					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Reagent/Method Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike Sample	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	75-125%
LCS/LCSD	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%
Surrogates	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RSD≤ 15% for each analyte

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Phthalic Anhydride (acid)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified 8000 - HPLC / L-36					
Laboratory Name	Test America Laboratories (Tallahassee)					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every prep batch	No target compounds ≥ QL	Re-clean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Lab Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One per 20 samples	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 50-150
Matrix Spike Duplicates	One per 20 samples	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery is 50-150 and RPDs ≤50
LCS	One per analytical batch	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 50-150
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Verification (CCV)	Every 10 samples shots and at the end of the sequence	%D ≤ 20%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	CCV %D ≤ 20%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	N,N-dimethylformamide (DMF)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified 8033 – GC/NPD / L-35					
Laboratory Name	Katahdin Analytical Services					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every prep batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Lab Duplicate	One per ten	RPD ≤ 20%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RPD ≤ 30%
Laboratory Matrix Spike	One per analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 70-130
Matrix Spike Duplicates	One per analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery is 70-130 and RPDs ≤50
LCS	One per analytical batch	Percent Recovery: 70-130	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 70-130
Surrogates	With every sample	Percent Recovery: 70-130	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 70-130
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Verification (CCV)	Start of sample analysis and after every 10 samples	%D ≤ 25%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	CCV %D ≤ 25%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	NDMA / NDPA					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified EPA 521 / L-5					
Laboratory Name	Test America Laboratories, Inc. (Sacramento, CA)					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every extraction batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One per SDG	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 60-140
Matrix Spike Duplicates	One per SDG	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery is 60-140 and RPDs ≤20
LCS	One per batch	Percent Recovery 70-130%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 60-140
Isotope IS	With every sample	Percent Recovery 25-150%	Professional Judgment	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 25-150
Other: Continuing Calibration Check compounds (CCC)	Daily and every 10 samples	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	Beginning of the analytical run or every 12hrs	Acceptance limits specified in method	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined NA = Not Applicable NS = Not Specified by method

Prepared by: TC
Checked by: CR
04/23/09

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Nonylphenol (alkylphenols)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified GCMS-SIM / L-34					
Laboratory Name	Test America Laboratories, Inc. (Sacramento, CA)					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every extraction batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One per analytical batch	Percent Recovery: 40-140	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 40-140
Matrix Spike Duplicates	One per analytical batch	Percent Recovery: 40-140	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery is 40-140 and RPDs ≤50
LCS	One per analytical batch	Percent Recovery: 40-140	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 40-140
Surrogates	With every sample	Percent Recovery: 40-140	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 40-140

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Nonylphenol (alkylphenols)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified GCMS-SIM / L-34					
Laboratory Name	Test America Laboratories, Inc. (Sacramento, CA)					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Internal Standards (ISs)	With every sample	Percent Recovery: 50-200%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	Area counts: -50% ±100% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard
Other: Continuing Calibration Verification (CCV)	Start of every 12 hour shift	%D ≤ 25%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	CCV %D ≤ 25%
Instrument Tune with DFTPP	Prior to sample analysis	Masses within 0.45 amu of target masses	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method
amu = atomic mass unit

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Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SW/GW					
Sampling SOP	S-1, S-2					
Analytical Parameter	Opex and Kempore					
Concentration Level	Low					
Analytical Method/ SOP Reference	Lab Specific HPLC method SW-846 8000B / L-11					
Laboratory Name	Lancaster Labs					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Reagent/Method Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike Sample	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	75-125%
LCS/LCSD	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%
Surrogates	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RSD≤ 15% for each analyte

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	GW					
Sampling SOP	S-1					
Analytical Parameter	Perchlorate					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 6850 / L-37					
Laboratory Name	Lancaster Labs					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Reagent/Method Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD < 20 if results ≥ 5x QL	Qualify data	Analyst and Data Validator	Precision	RPD < 20 if results ≥ 5x QL
Matrix Spike Sample	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%, RPD = 20%
LCS	One every analytical batch	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%
Other: Continuing Calibration Check	Every 10 samples	RSD≤ 15%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RSD≤ 15%

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16, S-14					
Analytical Parameter	VOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-8260B / L-3					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every 12 hours prior to sample analysis	No target compounds ≥ QL (except methylene chloride and cyclohexane must be <10x QL and acetone and 2-butanone must be < 2 x QL)	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL (except methylene chloride and cyclohexane must be <10x QL and acetone and 2-butanone must be < 2 x QL)
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	Percent recovery 70-130% RPD ≤ 30% with MSD	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 70-130% RPD ≤ 30% with MSD
Matrix Spike Duplicates	One per 20	Percent recovery 70-130% RPD ≤ 30% with MS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/precision/bias	Percent recovery 70-130% RPD ≤ 30% with MS
LCS	One per analysis day	Percent recoveries 70-130%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy	Percent recoveries 70-130%
Surrogates	3 per sample	Percent recoveries 70-130%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 70-130%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16, S-14					
Analytical Parameter	VOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-8260B / L-3					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Internal Standards (ISs)	6 per sample	Area counts: - ± 50% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	Area counts: - ± 50% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	Beginning of the analytical run or every 12hrs	Acceptance limits specified in method	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

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Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	SVOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8270C / L-4					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every extraction batch	No target compounds ≥ QL (except phthalates must be ≤ 5x QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL (except phthalates must be ≤ 5x QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL (except phthalates must be ≤ 5x QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL (except phthalates must be ≤ 5x QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One per SDG	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery
Matrix Spike Duplicates	One per SDG	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery and RPDs ≤30	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery and RPDs ≤30
LCS	One per batch	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds: 40-140% Recovery
Surrogates	6 per sample	Acid Compounds: 15-110% Recovery Base-Neutral Compounds:	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Acid Compounds: 30-130% Recovery Base-Neutral Compounds:

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10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	SVOCs					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8270C / L-4					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
		30-130% Recovery				30-130% Recovery
Internal Standards (ISs)	6 per sample	Area counts: -50% ±100% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	Area counts: -50% ±100% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard
Other: Continuing Calibration Check compounds (CCC)	Daily and every 10 samples	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	Beginning of the analytical run or every 12hrs	Acceptance limits specified in method	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	PCB					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8082 / L-28					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	Percent recovery 30-150% for Pesticides and a RPD ≤ 30% with MS. Percent recovery 40-140% for PCBs and a RPD ≤ 50% with MS.	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 30-150% for Pesticides and a RPD ≤ 30% with MS. Percent recovery 40-140% for PCBs and a RPD ≤ 50% with MS.
Matrix Spike Duplicates	One per 20	Percent recovery 30-150% for Pesticides and a RPD ≤ 30% with MSD. Percent recovery 40-140% for PCBs and a RPD ≤ 50% with MSD.	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/ Precision/ bias	Percent recovery 30-150% for Pesticides and a RPD ≤ 30% with MSD. Percent recovery 40-140% for PCBs and a RPD ≤ 50% with MSD.
LCS	One per batch	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy	Percent recovery 40-140%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	PCB					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8082 / L-28					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Surrogates	3 per sample	Percent recoveries Percent recovery 30-150%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries Percent recovery 30-150%
Continuing Calibration Verification (CCV)	Every 12 hours or 10 samples and at the beginning and end of analytical sequence	Calibration factor (CF) ≤ 15 %D from the mean initial calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	CF ≤ 25 %D from the initial calibration standard

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Extractable Petroleum Hydrocarbon (EPH)					
Concentration Level	Low					
Analytical Method/ SOP Reference	MADEP EPH-2004 / L-7					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	Percent recovery 40-140% RPD ≤ 50% with MSD	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 40-140% RPD ≤ 50% with MSD
Matrix Spike Duplicates	One per 20	Percent recovery 40-140% RPD ≤ 50% with MS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/Precision/bias	Percent recovery 40-140% RPD ≤ 50% with MS
LCS	One per batch	Percent recoveries 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy	Percent recoveries 40-140%
Surrogates	1 per sample	Percent recoveries 40-140%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 40-140%
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Daily and every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	%D between the initial calibration ≤ 25%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Extractable Petroleum Hydrocarbon (EPH)					
Concentration Level	Low					
Analytical Method/ SOP Reference	MADEP EPH-2004 / L-7					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
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10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Volatile Petroleum Hydrocarbon (VPH)					
Concentration Level	Low					
Analytical Method/ SOP Reference	MADEP VPH-2004 / L-8					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	Percent recovery 70-130% RPD ≤ 50% with MSD	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 70-130% RPD ≤ 50% with MSD
Matrix Spike Duplicates	One per 20	Percent recovery 70-130% RPD ≤ 50% with MS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 70-130% RPD ≤ 50% with MS
LCS	One per analysis day	Percent recoveries 70-130%	Reanalyze and qualify data	Analyst and Data Validator	Precision	Percent recoveries 70-130%
Surrogates	1 per sample	Percent recoveries 70-130%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recoveries 70-130%
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	%D between the initial calibration ≤ 25%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined NA = Not Applicable NS = Not Specified by method

Prepared by: TC
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10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	TAL Metals					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 6010B (ICP)/6020A (ICP/MS)/Hg by 7470A / L-2					
Laboratory Name	Test America Laboratories, Inc..					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One per preparation batch	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Reagent Blank**	ICB: immediately after ICV CCB: every 10 samples immediately after CCV	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD < 20 if results ≥ 5x QL	Qualify data	Analyst and Data Validator	Precision	RPD < 20 if results ≥ 5x QL
Laboratory Matrix Spike	One per 20	Percent recoveries 75-125%	Qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75-125%
LCS	One per batch	Percent recoveries 80-120%	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80-120%
ICV/CCV	Every ten samples, beginning and end of each run.	±10% of true value for all metals, except Hg is ±20%	Reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	90-110% except Hg is 80-120%
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs) (ICP/MS only)	Every sample	60-125% of IS in calibration blank	Dilute sample 5x, add IS and reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	60-125% of IS in calibration blank

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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	TAL Metals					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 6010B (ICP)/6020A (ICP/MS)/Hg by 7470A / L-2					
Laboratory Name	Test America Laboratories, Inc..					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Other: Serial Dilution	One per SDG	Within 10% of original determination	Qualify data	Analyst and Data Validator	Accuracy/bias	Within 10% of original determination
Other: Detection Limit Standard	Beginning and end of curve	Percent recoveries 70-130%	Recalibrate and reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 70-130%
Other: Interference Check Sample	Beginning and end of run or every 8 hours	Percent recoveries 80-120%	Recalibrate and reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80-120%

** Also referred to as initial and continuing calibration blanks.
TBD = To Be Determined
NA = Not Applicable

Prepared by: TC
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10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Hexavalent Chromium					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 3060A/7199, L-38					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One per preparation batch	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Reagent Blank**	ICB: immediately after ICV CCB: every 10 samples immediately after CCV	Absolute value of target metals must be < QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	Absolute value of target metals must be < QL
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	One per batch	RPD < 20 if results ≥ 5x QL	Qualify data	Analyst and Data Validator	Precision	RPD < 20 if results ≥ 5x QL
Predigestion Laboratory Matrix Spike	One per 20	Percent recoveries 75-125%	Reanalyze the entire batch; no re-analysis is sample concentration is >4X the spike concentration	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75-125%
Matrix Spike Duplicates	NA	NA	NA	NA	NA	NA
Post Verification Spike (PVS)	One per batch or one per 20	Percent recoveries 85-115%	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 85-115%
LCS (soluble)	One per batch or one per 20	Within certified accepted range	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75-125%
LCS (insoluble)	One per batch or one per 20	Percent recoveries 80-120%	Determine cause of problem, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80-120%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Hexavalent Chromium					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 3060A/7199, L-38					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
ICV/CCV	Every ten samples, beginning and end of each run.	±10% of true value	Reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias	90-110%

** Also referred to as initial and continuing calibration blanks.
NA = Not Applicable

Prepared by: TC
Checked by: CR
04/23/09

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Ammonia					
Concentration Level	Low					
Analytical Method/ SOP Reference	Quick Chem Method 10-107-06-1-A / L-20					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 0.1 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch prior to samples and after last calibration standard	< 0.1 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One per distillation batch	Percent recoveries 75 – 125%	Reanalyze and/or qualify data	Analyst and Data Validator	Accuracy	Percent recoveries 75 – 125%
Matrix Spike Duplicates	One per distillation batch	Percent recoveries 75 – 125%	Reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/precision	Percent recoveries 75 – 125%
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

TBD – To be determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Chloride, Sulfate, Nitrate, Nitrite					
Concentration Level	Low					
Analytical Method/ SOP Reference	USEPA 300 (Chloride, Sulfate, Nitrate, Nitrite) / L-13, L-17, L-18					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< QL for each anion	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch prior to samples and after last calibration standard	No target analytes ≥ ½ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One per batch	Percent recoveries 75 – 125%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 75 – 125%
Matrix Spike Duplicates	NA	RPD ≤ 20	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	RPD ≤ 20
LCS	One every analytical batch prior to samples and after last calibration	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Chloride, Sulfate, Nitrate, Nitrite					
Concentration Level	Low					
Analytical Method/ SOP Reference	USEPA 300 (Chloride, Sulfate, Nitrate, Nitrite) / L-13, L-17, L-18					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
	standard					
Surrogates	NA	NA	NA	NA	NA	NA

TBD – To be determined NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Total Organic Carbon (TOC)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Llody Kahn / L-39					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every analytical batch prior to samples and after last calibration standard	< 0.1 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch prior to samples and after last calibration standard	< 0.1 mg/L	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target analytes ≥ ½ QL
Laboratory Duplicate	One per batch	RPD ≤ 20	Reanalyze and/or qualify data	Analyst and Data Validator	Precision	RPD ≤ 20
Laboratory Matrix Spike	One per distillation batch	Percent recoveries 75 – 125%	Reanalyze and/or qualify data	Analyst and Data Validator	Accuracy	Percent recoveries 75 – 125%
Matrix Spike Duplicates	One per distillation batch	Percent recoveries 75 – 125%	Reanalyze and/or qualify data	Analyst and Data Validator	Accuracy/precision	Percent recoveries 75 – 125%
LCS	One every analytical batch prior to samples and after last calibration standard	Percent recoveries 85 – 115%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recoveries 80 – 120%

TBD – To be determined
NA – Not Applicable

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Formaldehyde, Acetaldehyde					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 8315 / L-32					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Reagent/Method Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike Sample	One every analytical batch	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	75-125%
LCS	One every analytical batch	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%
Surrogates	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RSD≤ 15% for each analyte

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Hydrazine, MMH, UDMH					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified Method 8315 / L-24					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Reagent/Method Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	One every analytical batch	No detection > the Reporting Limit	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No detection > the Reporting Limit
Laboratory Duplicate	One per batch	RPD < 20 if results ≥ 5x QL	Qualify data	Analyst and Data Validator	Precision	RPD < 20 if results ≥ 5x QL
Matrix Spike Sample	One every analytical batch	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	75-125%
LCS/LCSD	One every analytical batch	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	80-120%
Surrogates	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Check compounds (CCC)	Every 10 samples	RSD≤ 15% for each analyte	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RSD≤ 15% for each analyte

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

Prepared by: TC
Checked by: CR
04/23/09

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	Phthalic Anhydride (acid)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified 8000 - HPLC / L-36					
Laboratory Name	Test America Laboratories (Tallahassee)					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every prep batch	No target compounds ≥ QL	Re-clean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Lab Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One per 20 samples	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 60-140
Matrix Spike Duplicates	One per 20 samples	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery is 60-140 and RPDs ≤50
LCS	One per analytical batch	Within LIMS limits	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 60-140
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Verification (CCV)	Every 10 samples shots and at the end of the sequence	%D ≤ 20%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	CCV %D ≤ 20%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable NS = Not Specified by method

Prepared by: TC
Checked by: CR
10/14/08

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	N,N-dimethylformamide (DMF)					
Concentration Level	Low					
Analytical Method/ SOP Reference	Modified 8033 – GC/NPD / L-35					
Laboratory Name	Katahdin Analytical Services					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every prep batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Lab Duplicate	One per ten, if requested	%D ≤ 25%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	%D ≤ 30%
Laboratory Matrix Spike	One per 20	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 70-130
Matrix Spike Duplicates	One per 20	NS	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery is 70-130 and RPDs ≤50
LCS	One per analytical batch	Percent Recovery: 70-130	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 70-130
Surrogates	With every sample	Percent Recovery: 70-130	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent Recovery: 70-130
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: Continuing Calibration Verification (CCV)	Start of sample analysis and after every 10 samples	%D ≤ 25%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	CCV %D ≤ 25%
Instrument Tune with DFTPP	NA	NA	NA	NA	NA	NA

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

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EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	NDMA					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 Modified 8270C / L-6					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	One every batch	No target compounds ≥ QL	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	As needed to assess carryover from high concentration samples	No target compounds ≥ QL	Reclean, retest, reanalyze, and/or qualify data	Analyst and Data Validator	Accuracy/bias-Contamination	No target compounds ≥ QL
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Matrix Spike	One per 20	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 50-150% RPD ≤ 50% with MSD
Matrix Spike Duplicates	One per 20	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias	Percent recovery 50-150% RPD ≤ 50% with MS
LCS	One per batch	Percent recovery 40-140%	Reanalyze and qualify data	Analyst and Data Validator	Precision	Percent recovery 50-150%
Surrogates	per sample	Percent recovery 30-130%	Re-extract and reanalyze	Analyst and Data Validator	Accuracy/bias	Percent recovery 50-150%

EPA-NE QAPP Worksheet #24a

Fixed Laboratory Analytical QC Sample Table

Medium/Matrix	SO/SD					
Sampling SOP	S-2, S-3, S-15, S-16					
Analytical Parameter	NDMA					
Concentration Level	Low					
Analytical Method/ SOP Reference	SW-846 Modified 8270C / L-6					
Laboratory Name	Test America Laboratories, Inc.					
No. of Sample Locations	TBD					
Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Internal Standards (ISs)	per sample	Area counts: - ± 50% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	Area counts: - ± 50% of areas in associated continuing calibration standard Retention times: ± 30 seconds from retention times in associated continuing calibration standard
Other: Continuing Calibration Check compounds (CCC)	Daily and every 10 samples	RSD≤ 15% for each analyte, RRF ≥0.05	Reanalyze and qualify data	Analyst and Data Validator	Accuracy/bias and Precision	RRF ≥0.05, %D between the initial calibration RRF and CCC ≤ 25%
Instrument Tune with DFTPP	Beginning of the analytical run or every 12hrs	Acceptance limits specified in method	Re-tune instrument per manufacturers specifications	Analyst and Data Validator	NA	Limits specified in USEPA Region I Validation Guidelines (1996)

TBD = To Be Determined
NA = Not Applicable
NS = Not Specified by method

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14.0 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

This section of the QAPP is intended to summarize sources of previously collected data and information that will be used to make project decisions. These historical data were used to design the RI/FS sampling program. Worksheet #25 has been excluded from this QAPP. Information from historical investigations at the Olin Chemical Superfund Site is presented in detail in Section 2 of the Draft Focused RI Report (MACTEC, 2007).

15.0 DOCUMENTATION, RECORDS AND DATA MANAGEMENT

This section of the QAPP describes how project field activities, data collection, and information will be documented, tracked and managed from their generation in the field to final use and storage in a manner that ensures data integrity and defensibility.

15.1 PROJECT DOCUMENTATION AND RECORDS

A complete file of work assignment and contract related documents will be maintained by MACTEC and subcontractor project managers. The file will contain all contracts, work authorizations, change orders, invoices, and correspondence.

Technical records used to document field activities and project investigations will make up the majority of records generated during remedial investigations at the site. A summary of field records and data generation records is presented in Worksheet #26.

15.2 FIELD ANALYSIS DATA PACKAGE DELIVERABLES

Field data will be generated during measurements made during sample collection, and during analyses completed in the field. For the field analyses associated with sample collection which consist of measuring water quality parameters (temp, pH, conductivity, dissolved oxygen, REDOX, and turbidity), FID/PID screening, and water level measurements, data packages are not required. All field and QC sample results, calibrations, and calibration verifications will be recorded in the field logbook and/or on field data record forms, and on equipment calibration record forms found in Appendix B of the FSP.

For on-site screening analyses for ammonia (see Section 11), documentation of analyses and testing results will be recorded in instrument logbooks completed at the site. The logbooks will contain all records needed to document the sample analyses. Sample results and associated QC measurements will be summarized in a field screening data quality report. Any quality issues that are identified that impact data usability will be documented in the data quality report.

15.3 FIXED LABORATORY DATA PACKAGE DELIVERABLES

Data deliverables for samples analyzed at the off-site laboratory based on specifications described in the EPA CLP SOWs. For standard EPA SW-846 methods, CLP-like deliverables will be required. At a minimum, the data packages from the analytical chemistry laboratories will include the following:

1. Data package narrative
   - summary of analytical methods used
   - correlation of field sample identifications and laboratory sample identifications
   - data qualifier definitions
   - deviations from established QA/QC procedures with corrective action

2. Sample results
   - project name
   - field sample identification
   - lab ID
   - unit of measurement
   - batch number
   - collection/extraction/analysis dates
   - detection limits
   - dilution factors
   - percent moisture

3. Sample documentation
   - original chain-of-custody
   - shipping documents
   - cooler receipt forms

4. Quality Assurance/Quality Control
   - spike recoveries (surrogates, MS/MSDs, LCSs)
   - internal standard summary
   - initial calibration summaries
   - GC/MS tuning summaries
   - continuing calibration summaries

- QC blank summaries
- CLP From 10 – dual column summary (applicable GC methods)
- Serial dilution summary (metals analysis)
- Interference check (metals analysis)
- measures of precision (laboratory duplicates, MS/MSDs)
- control limits for accuracy and precision

5. Raw data including instrument printouts (run sequence/acquisition files, chromatograms and quantitation reports), instrument logbook pages, and sample preparation logs.

Results for all soil and sediment samples must be reported on a dry weight basis.

15.4 DATA REPORTING FORMATS

Project field data will be recorded in dedicated logbooks and on standardized forms. Documentation of field activities is described in Section 10. All information related to sample collection and analysis will be documented in logbooks, instrument printouts, or field record forms. All entries that are not generated by an automated data system will be made neatly and legibly in permanent, waterproof ink. Information will not be erased or obliterated. Corrections will be made by drawing a single line through the error and entering the correct information adjacent to the cross-out. All changes will be initialed, dated, and, if appropriate, accompanied by a brief explanation. Unused pages or portions of pages will be crossed out to prevent future data entry.

15.4.1 Electronic Deliverables

The contract laboratory shall provide a separate electronic data deliverable (EDD) to MACTEC. The format for this deliverable will be provided to the Laboratories prior to analysis of samples. This format allows the data to be loaded directly into the Olin Corporation database and MACTEC TED.

15.5 DATA HANDLING AND MANAGEMENT

Data from field activities and measurements may be entered into the TED data base and used during site assessments. Data from laboratory analyses will be entered directly into the project database using the electronic deliverable.

15.5.1 Data Entry and Verification

Data entry performed by MACTEC or its contractors will be proofed for accuracy. Verification will be carried out either by proofing printout or database records against the original data.

15.5.2 Data Transformation and Reduction

Data generated through field activities or by the subcontract laboratory, will be reduced and validated prior to reporting. Measurements and sample collection information will be transcribed directly into the field logbook or onto standardized forms. If errors are made, results will be legibly crossed out, initialed and dated by the person recording the data, and corrected in a space adjacent to the original (erroneous) entry. Periodic reviews of the field records by the MACTEC Field Operations Leader will ensure that:

- logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed;
- records are legible and in accordance with good record keeping procedures, i.e., entries are signed and dated, data are not obliterated, changes are initialed, dated, and explained;
- sample collection, handling, preservation, and storage procedures were conducted in accordance with the protocols described in the QAPP, and that any deviations were documented and approved by the appropriate personnel; and
- instruments were calibrated and operated in accordance with the procedures specified in the QAPP.

Laboratory data reduction procedures will be performed according to procedures in the laboratory's QA Manuals. These procedures are also summarized in Section 19.3.

15.5.3 Data Transfer and Transmittal

All laboratory data will be provided by the laboratory in both electronic and hard copy format. The electronic data will be imported into the Olin Corporation and MACTEC TED database. During data validation a quality assurance review of sample results will be completed to ensure that the data in the database match the hard copy provided by the laboratory.

Hard copy and EDDs from the laboratories will be transmitted to the MACTEC Data Manager and Project Chemist. Each hard-copy report and EDD will be logged into a MACTEC validation tracking log. As the package proceeds through data validation, review, and data management, the status of the package will be recorded in the log. Completion of validation and final disposition of the package will also be documented.

Laboratory data will be maintained in a computerized database to allow easy retrieval of information and electronic transfer of the data to other parties. As samples are obtained and shipped to laboratories for analysis, a new database record will be created for each sample number, containing the date of sampling, sample location, Field Sample ID, sample depth, sample type (field vs. QA/QC), analyses to be performed, and laboratory name. As laboratory analytical results are received, and validated, the results will be imported to the database.

15.5.4 Data Analysis and Reporting

Once data are entered into the TED and validation is completed, data reports will be generated as needed to support contamination assessments and report preparation. TED is a computerized database management system designed to manage a large amount of sampling and analytical data will be used. The data management system is intended to serve as the database for data generated in past phases of the project as well as the current phase. The data management system is designed to provide the technical project staff with a vehicle to sort, arrange, and thereby analyze data with speed and efficiency, while allowing user-specified reporting and graphics capabilities.

The data management system will allow the user to select criteria for construction of a table and can therefore generate unique data tables from which to analyze various problems and scenarios.

15.6 DATA TRACKING AND CONTROL

Management of field data is described in Section 10.2. Laboratory data will be maintained as described in the laboratory's QA Manuals. MACTEC is the custodian of the project files and will maintain the contents of the files, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews in a secured, limited access area.

EPA-NE QAPP Worksheet #26
Project Documentation and Records Table

Sample Collection Records	Field Analysis Records	Fixed Laboratory Records	Data Assessment Records	Other
Field Logbooks	Sample Receipt, Custody and Tracking Records	Sample Receipt, Custody and Tracking Records	Field Sampling Audit Checklists	
COC Records	Equipment Calibration Logs	Standard Traceability Logs	Data Validation Checklists	
Air Bills	Equipment Maintenance, Testing and Inspection Logs	Equipment Calibration Logs	Data Validation Reports	
Telephone Logs	Field Activity Forms	Sample Preparation Logs	PE Sample Score sheets	
Field Data Record Forms	Corrective Action Forms	Instrument Logs - Run Logs	Corrective Action Forms	
Corrective Action Forms	Screening Results Forms	Equipment Maintenance, Testing and Inspection Logs		
Field Forms	Instrument logbooks	Corrective Action Forms		
Telephone Logs	Calibration Standard Certificates	QC Sample Results Reports		
		Instrument Printout (raw data) for field samples, standards, QC checks and QC samples		
		Telephone Logs		
		MDL Study Records		
		Email		

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Checked by: CR
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16.0 ASSESSMENTS AND RESPONSE ACTIONS

16.1 PLANNED ASSESSMENTS

Worksheet #27b summarizes potential assessments during this investigation.

16.1.1 Internal Assessments

Technical system audits (TSAs) of both field and laboratory activities may be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the QAPP.

Field Sampling TSAs

A system audit of field activities including sampling and field measurements will be conducted and documented by the Project QA Officer (or their designee) at the start of the RI sampling. The purpose of this audit is to verify that all established procedures are being followed as planned and documented and to allow for timely corrective action, reducing the impact of the nonconformance. The audit will ensure that all personnel have read the QAPP and have signed Worksheet #4. The audit will cover field sampling records, field measurement results, field instrument operation and calibration records, sample collection, preservation, handling, and packaging procedures, adherence to QA procedures, personnel training, sampling procedures, decontamination procedures, review of sampling design versus the Field Sampling Plan, corrective action procedures, and chain-of-custody, etc. Follow-up surveillance will be conducted by the Field Operations Leader to verify that QA procedures are maintained throughout the investigation. An example of a field audit checklist is included as Figure 16-1.

Upon completion of the audit, the Project QA Officer will prepare a written audit report, which summarizes the audit findings, identifies deficiencies, and recommends corrective actions. In addition, a verbal debriefing will also be given to the Field Operations Leader and Project Manager at the time of the audit. The written report will be submitted to the MACTEC Project Manager, who will be responsible for ensuring that corrective measures are implemented.

Fixed Laboratory TSAs

Prior to the start of the sampling program, the MACTEC QA Officer will host a kick-off meeting with the Olin Laboratory Project Manager and Lab Manager from TestAmerica-Westfield to review the QAPP and the Sampling and Analysis Program.

No laboratory audits are planned for this RI. The MACTEC QA Officer will obtain at least one audit report for TAL from a client/govt certification agency that had been completed within the previous year. This audit report will be reviewed to assess laboratory audit issues and verify that any necessary corrective actions have been completed. Audits may be conducted by the MACTEC QA Officer or by a designated qualified individual under the direction of the MACTEC QA Officer if data quality concerns regarding lab performance arise. If a lab audit is deemed necessary, the fixed laboratory TSA will include a review of the following areas:

- QA organization and procedures (including the Laboratory QA Plan);
- personnel training and qualifications;
- facility security;
- sample log-in procedures;
- sample storage facilities;
- analyst technique
- adherence to referenced analytical methods and the QAPP;
- compliance with QA/QC objectives;
- equipment, instrumentation and supplies kept on reserve;
- instrument calibration and maintenance;
- data recording, reduction, review, and reporting; and
- cleanliness and housekeeping.

An example of a fixed laboratory TSA checklist is included as Figure 16-2. Preliminary results of the TSA will be discussed with the Laboratory Manager, Laboratory Project Manager, and Laboratory QA Manager during a verbal debriefing held at the facility. Assessment findings will be documented and reported as described in Section 16.2.

Fixed Laboratory Performance Evaluation Samples

A set of single-blind PE samples will be submitted to the laboratories during each sampling event for parameters where PE samples are available from the USEPA. PE samples will be obtained through the New England Regional Laboratory. The PE samples will be used as a quantitative assessment of the laboratory performance and the overall accuracy of the method. PE sample results will be submitted to EPA for scoring. Score sheets will be evaluated by the MACTEC project chemist and assessments of data accuracy will be included in the data validation reports for each sampling event.

Data Validation TSA

A review of the complete Data Validation Report will be conducted by the MACTEC QA Officer prior to submitting the report to EPA. This review will include a review of the reported data validation actions and observations, and a review of the Data Validation Report to ensure that all required components are present. This review will also ensure that the most recent version of the Region I Data Validation guidelines were followed and that all measurement performance criteria were met or evaluated.

Figure 16-1
Example of Field Sampling TSA Checklist

Project: **Site Location:** **Auditor:**	
1. Was project-specific training held?	
2. Are copies of project plan on site and available to personnel?	
3. Are samples being collected in accordance with the project plan?	
4. Do the numbers and locations of samples conform to the project plan?	
5. Are sample locations flagged, staked, or otherwise marked?	
6. Are samples labeled in accordance with the project plan?	
7. Is equipment decontamination in accordance with the project plan?	
8. Is field instrumentation being operated and calibrated in accordance with the project plan?	
9. Are samples being preserved and containerized in accordance with the project plan?	
10. Are QC samples in accordance with the types, collection procedures, and frequencies specified in the project plan?	
11. Are chain-of-custody procedures and documents in conformance with the project plan?	
12. Are field records complete, accurate, up-to-date, and in conformance to good record keeping procedures?	
13. Are modifications to the project plan being communicated, approved, and documented appropriately?	
Additional Comments:	
Auditor:	**Date:**

Figure 16-2
Example of Fixed Laboratory TSA Checklist

Project: Facility Location: Auditor:

Project: **Facility Location:** **Auditor:**	
Is there a written QA Program Plan/Manual?	
Is there a designated QA Officer?	
Are facilities and equipment adequate to perform the analyses of interest?	
Review procedures and engineering controls for minimizing cross contamination.	
Review most Percent interlaboratory PE sample results and Percent Agency audits.	
Review SOP system. Review techniques for conformance to approved SOPs.	
Are personnel qualified and trained? Is there a formal training program and are records of training and proficiency maintained?	
Is there a designated sample custodian? Is there a sample inspection checklist? Are sample log-in procedures defined in an SOP?	
Is the laboratory area secure?	
Review internal chain-of-custody procedures.	
Are instruments operated and calibrated in accordance with the methods referenced in the QAPP? Are records of calibration maintained?	
Is equipment maintained according to written protocols? Are routine and non-routine maintenance procedures documented?	
Are samples being analyzed in conformance to the methods referenced in the QAPP?	
Are QC samples and checks being performed at the frequencies stated in the methods referenced in the QAPP?	
Are records complete, accurate, up-to-date, and in conformance to good record keeping procedures?	
How are project-specific requirements communicated to the bench level?	
Review data reduction, review, and reporting processes.	
Review data archival process (paper and electronic).	
Review audit and corrective action program.	
Additional Comments:	
Auditor:	**Date:**

16.1.2 External Assessments

External assessments may also be competed by EPA Region 1.

16.2 ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

The results of the field sampling and fixed laboratory TSAs will be documented in written reports; in addition, verbal debriefings will also be held at the conclusion of all audits. The reports will be prepared by the auditor and will describe the scope of the TSA, summarize audit findings, and recommend corrective action. The report will be distributed to the appropriate personnel for response: the Field Operations Leader will be responsible for responding to the field sampling TSA report, and the Laboratory Manager will be responsible for addressing the fixed laboratory TSA report. Significant issues that are discovered during the TSA and which could potentially affect data quality or usability will be brought to the immediate attention of the MACTEC Project Manager.

The response to the TSA reports will include a description of the corrective action(s) to be implemented, the identities of the personnel responsible for implementing the corrective action, and the schedule for implementation/completion. All responses must be completed within two weeks of issuing the TSA report. The response will be reviewed by the MACTEC Project QA Officer and/or Project Chemist and, if all issues have been addressed appropriately and in a timely manner, no further action will be required. In the event that the corrective action(s) are inadequate or inappropriate, follow-up activities, including additional audits, or discussions with the MACTEC Project Manager, will be conducted by the Project QA Officer and/or Lead Chemist. The complete TSA report, including resolution of any deficiencies, will be included in the QA reports to management, as described in Section 17.

16.3 ADDITIONAL QAPP NON-CONFORMANCES

16.3.1 Field Non-Conformances

Corrective action in the field may be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP), or when sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. The field team may identify the need for corrective action. The Field Operations Leader will approve the corrective action and notify the Project Manager. The MACTEC Project Manager, in consultation with Olin and EPA if

necessary, will approve the corrective measure. The Field Operations Leader will ensure that the corrective measure is implemented by the field team. Corrective actions will be implemented and documented in the field record book. Documentation will include:

- a description of the circumstances that initiated the corrective action;
- the action taken in response;
- the final resolution; and
- any necessary approvals.

No staff member will initiate corrective action without prior communication of findings through the proper channels. If necessary, a problem resolution audit will be conducted.

16.3.2 Laboratory Non-Conformances

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, omissions or discrepancies with chain-of-custody documentation, low/high pH readings, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with laboratory analysts and Laboratory Section Leaders, it may be necessary for the Laboratory QA Manager to approve the implementation of corrective action.

The analyst may identify the need for corrective action. The Laboratory Section Leader, in consultation with the staff, will approve the required corrective action to be implemented by the laboratory staff. The Laboratory QA Manager will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, the MACTEC Lead Chemist will be notified. The Lead Chemist will notify the Project QA Officer, who in turn will contact all levels of project management for concurrence with the proposed corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action files, and the narrative data report. If the corrective action does not rectify the situation, the laboratory will contact the MACTEC Lead Chemist, who will determine the action to be taken and inform the appropriate personnel. If necessary, a problem resolution audit will be conducted.

16.4 DATA VALIDATION AND DATA ASSESSMENT NON-CONFORMANCES

The need for corrective action may be identified during either data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives. If the data validator or data assessor identifies a corrective action situation, the MACTEC Project Manager will be responsible for informing the appropriate personnel. All corrective actions of this type will be documented by the MACTEC Project Manager and maintained in the project files.

EPA-NE QAPP Worksheet #27b
Project Assessment Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) responsible for performing assessment, title and organizational affiliation	Person(s) responsible for responding to assessment findings, title and organizational affiliation	Person (s) responsible for identifying and implementing corrective actions (CA), title and organizational affiliation	Person (s) responsible for monitoring effectiveness of CA, title and organizational affiliation
Field Sampling Technical System Audit (if required)	1/ at startup sampling 1 / year	Internal	MACTEC	Chris Ricardi, Project QA Officer MACTEC	Field Operations Leader MACTEC	Peter Thompson, Project Manager MACTEC	Chris Ricardi, QA Officer MACTEC
Fixed Laboratory Technical Systems Audit (if required)	None scheduled	External	MACTEC	Chris Ricardi, Project QA Officer MACTEC	Field Operations Leader MACTEC, Laboratory QA Manager	Laboratory QA Manager	Laboratory QA Manager
Field Health and Safety Systems Audit (if required)	1/ at startup sampling 1 / year	Internal	MACTEC	Kendra Bavor, Health and Safety MACTEC	Field Operations Leader MACTEC	Peter Thompson, Project Manager MACTEC	TBD, Laboratory QA Manager Designated Health and Safety Officer
Performance Evaluation Sample Tracking and Analysis	1 per event	External	EPA	Leo Corbin/ EPA	Chris Ricardi, Project QA Officer MACTEC	Laboratory QA Manager	Laboratory QA Manager

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17.0 QA MANAGEMENT REPORTS

QA reports will be submitted to the MACTEC Project Manager to ensure that any problems identified during the sampling and analysis programs are investigated and the proper corrective measures taken in response. Worksheet #28 summarizes QA management reports which may be submitted during this investigation. The QA reports will include:

- all results of field and laboratory audits
- problems noted during data validation and assessment
- significant QA/QC problems, recommended corrective actions, and the outcome of corrective actions
- data usability assessments and
- the final project report.

QA reports will be prepared and submitted on an as-needed basis.

EPA-NE QAPP Worksheet #28
QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation, Title and Organizational Affiliation	Report Recipients, Title and Organizational Affiliation
Verbal Status Report	Weekly	At the end of every day of field activities	TBD , Field Operations Leader, MACTEC	Peter Thompson, Project Manager, MACTEC
Verbal or Written Status Report	As necessary	As necessary	Peter Thompson, Project Manager, MACTEC	Steve Morrow Project Manger, Olin Corporation; James DiLorenzo RPM, EPA
Corrective Action Report	As necessary	As necessary	Chris Ricardi, Project QA Officer, MACTEC	Peter Thompson, Project Manager, MACTEC
Field Sampling Technical Systems Audit Report	One/ at startup of sampling	Within 2-3 days of audit	Chris Ricardi, Project QA Officer, MACTEC	Peter Thompson, Project Manager, MACTEC Michael Murphy, Project Principal, MACTEC
Data Usability Assessment	One/ after all data generated and validated	TBD	Chris Ricardi, Project QA Officer, MACTEC	Peter Thompson, Project Manager, MACTEC Michael Murphy, Project Principal, MACTEC
Final Project Report (RI/FS)	One/ after RI/FS completed	TBD	Peter Thompson, Project Manager, MACTEC	Steve Morrow Project Manger, Olin Corporation; James DiLorenzo RPM, EPA

TBD – To Be Determined

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10/14/08

18.0 VERIFICATION AND VALIDATION REQUIREMENTS

All data generated through field activities, or by the laboratory operation, will be reduced and validated prior to reporting.

18.1 FIELD SAMPLING DATA

Field sampling data will be verified daily by each person performing the tasks. These data will be verified for completeness and correctness. Field sampling data will also be independently reviewed daily by the MACTEC Field Operations Leader to ensure that records are complete, accurate, and legible and verify that the sampling procedures are in accordance with the protocols specified in the QAPP. Personnel performing the verification tasks will sign the field notes after verification. Verification will include all field logbook notes, field sampling forms, and COCs. Details of the review of field records are included in Section 19.1.

18.2 FIELD ANALYSIS DATA

Each member of the sampling team performing field analysis tasks will verify their own data at the conclusion of each day for completeness and correctness. Field analysis data will also be independently verified daily by the MACTEC Field Operations Leader to ensure that records are complete, accurate, and legible and verify that the calibration procedures are in accordance with the protocols specified in the QAPP. Personnel performing the verification tasks will sign the field notes after verification. Verification will include all field logbook notes and equipment calibration forms. Details of the review of field analysis records are included in Section 19.2.

Field analytical records and data reports generated during the ammonia analyses will be reviewed by the MACTEC QA Officer prior to final reporting of results. This will include a review of instrument logbooks, samples COC and Tracking forms, and QC measurement results generated during the analysis. The QA Officer will review the final data quality report and ensure that all data quality issues are reported along with the sample results.

18.3 FIXED LABORATORY DATA

Internal Reviews at Laboratory

Prior to the release of any data from the laboratory, the data will be verified and approved by laboratory personnel. This review will consist of a tiered review by the person performing the work, a qualified peer, and by supervisory personnel. Details of the review are included in Section 19.3

Independent Review

An independent review of fixed laboratory data will be performed by MACTEC in order to determine the quality of the analytical data. With the exception of Opex, Kempore, NDMA, VPH, EPH and a sub-set of inorganic analyses of surface water, and soil, all chemical data will be validated using the Region I, EPA-New England Data Validation Functional Guidelines (USEPA, 1996), which will be modified as necessary to include method-specific criteria, as detailed in Section 7.2 throughout this QAPP. Opex, Kempore, NDMA, VPH, EPH and a sub-set of inorganic analytical data will undergo a Level I Olin Data Quality Evaluation. Details of the Level I Olin Data Quality Evaluation are included in Section 19.3.

Unless otherwise specified in a task-specific sampling plan, a Tier II validation will be completed for VOCs, SVOCs, and metals. Tier II validation will include a data package completeness check and an overall evaluation of data and potential usability issues will be included, and technical holding times and QC sample results (blanks, surrogate spikes, MS/MSDs, matrix duplicates, LCSs, and PEs) will be reviewed. Tier III validation will be completed on approximately 10 percent of the samples as specified in task work plans. Validation will be performed by MACTEC data validators, under the direction of the MACTEC Project QA Officer. Details of the chemical data validation are included in Section 19.3

For data sets generated with the remaining methods a chemist review will be performed on the data prior to final reporting in investigation reports. During the review, the project chemist will evaluate QC measurements associated with the data sets using goals discussed in Section 7.2 and the reference analytical methods. QC measurements including QC blank data, laboratory control sample results, matrix spike results if applicable, and duplicate results. Results will be qualified using general procedures described in the USEPA validation guideline and the judgment of the project chemist. Assessments, qualifications, and professional judgments will be summarized in data validation reports presented with each investigation report.

19.0 VERIFICATION AND VALIDATION PROCEDURES

Worksheet #29a summarizes the data verification processes for data generated during this investigation.

19.1 FIELD SAMPLING DATA

Sample collection information will be transcribed directly into the field logbook or onto standardized forms. If errors are made, results will be legibly crossed out, initialed and dated by the person recording the data, and corrected in a space adjacent to the original (erroneous) entry. Each member of the field sampling team will be responsible for an internal verification of the transcribed information. Periodic reviews of the field records by the MACTEC Field Operations Leader will ensure that:

- logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed;
- records are legible and in accordance with good record keeping procedures, i.e., entries are signed and dated, data are not obliterated, changes are initialed, dated, and explained; and
- sample collection, handling, preservation, and storage procedures were conducted in accordance with the protocols described in the QAPP, and that any deviations were documented and approved by the appropriate personnel.

19.2 FIELD ANALYSIS DATA

Field analysis information will be transcribed directly into the field logbook or onto standardized forms. If errors are made, results will be legibly crossed out, initialed and dated by the person recording the data, and corrected in a space adjacent to the original (erroneous) entry. Each member of the field sampling team will be responsible for an internal verification of the transcribed information. Periodic review of the field analysis records by the MACTEC Field Operations Leader will ensure that:

- logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed;
- records are legible and in accordance with good record keeping procedures, i.e., entries are signed and dated, data are not obliterated, changes are initialed, dated, and explained; and
- calibration procedures were conducted in accordance with the protocols described in the QAPP, and that any deviations were documented and approved by the appropriate personnel.

19.3 FIXED LABORATORY DATA

Internal Reviews at Laboratory

Prior to being released as final, laboratory data will proceed through a tiered review process. Data verification starts with the analyst or technician who performs a 100 percent review of the data to ensure the work was done correctly the first time. It is the responsibility of the analyst or technician to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- sample preparation and analysis information is correct and complete
- results are correct and complete
- the appropriate EPA method Specifications have been followed and are identified in the project records
- proper documentation procedures have been followed
- all nonconformances have been documented
- project-specific requirements have been met

Following the completion of the initial verification by the analyst or technician, a systematic check of the data will be performed by an experienced peer, Laboratory Section Leader, or designee. This check will be performed to ensure that initial review has been completed correctly and thoroughly. Included in this review will be an assessment of the acceptability of the data with respect to:

- adherence of the EPA methods and any project-specific methods and specific instructions;
- correct interpretation of data (e.g., mass spectra, chromatographic interferences, etc.);
- correctness of numerical input when computer programs are used (checked randomly) and numerical correctness of calculations and formulas (checked randomly);
- acceptability of QC data;
- documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.);
- documentation of dilution factors, standard concentrations, etc.;
- sample holding time assessment; and
- nonconforming events have been addressed by corrective action as defined on a nonconformance memo.

A third-level review will be performed by the Laboratory Project Manager before results are submitted to the client. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed will include:

- results are present for every sample in the analytical batch or reporting group;
- every parameter or target compound requested is reported;
- the correct units and correct number of significant figures are utilized;
- all nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory; and
- the final report is legible, contains all the supporting documentation required by the project, and is in either the standard format or in the client-required format.

A narrative to accompany the final report will be finalized by the Laboratory Project Manager. This narrative will include relevant comments, including data anomalies and non-conformances.

Data Validation (Independent Review)

MACTEC will be responsible for performing an independent validation of the analytical data. Project-specific procedures will be used to validate analytical laboratory data. Worksheet #29b summarizes validation criteria, data validator and data validation tier for each matrix, analytical parameter and concentration level. The basis for the validation will be the Region I, EPA-New England Data Validation Functional Guidelines (USEPA, 1996) and the Compendium of Quality Assurance and Quality Control Requirements and Performance Standards for Selected Analytical Methods Used in Support of Response Actions for the MCP (MADEP, 2004). USEPA Region I Data validation procedures for SW-846 Methods are modified to include method-specific criteria (i.e., measurement performance criteria detailed in this QAPP) from the MCP Methods. For other methods that are not addressed in USEPA Region I guidelines or the MCP Compendium, method performance criteria are developed for this project. Worksheets #11, 12b, 21 and 24a, the EPA methods, specify the QC criteria and holding time requirements for all analyses conducted under this program. These criteria will be used to evaluate and qualify the data during validation and will be substituted for the default validation criteria listed in the guidelines, when necessary. A summary of QC limits and precision limits are presented on Table 7.2 and 7.3. Data validation modifications are summarized on Worksheet #29c.

Unless otherwise specified in a task-specific sampling plan, a Tier II validation will be completed for all VOCs, SVOCs, Pesticides, PCBs, and metals. Tier II validation will include a data package completeness

check and an overall evaluation of data and potential usability issues will be included, and technical holding times and QC sample results (blanks, surrogate spikes, MS/MSDs, matrix duplicates, LCSs, and PEs) will be reviewed. Tier III validation will be completed on approximately 10 percent of the samples as specified in task work plans. If during Tier III validation, significant errors are discovered in the QC summary forms, calculations, or data reporting, a Tier III validation will be performed on a larger subset of data packages and raw data will be examined. Validation will be performed by MACTEC data validators, under the direction of the MACTEC Project QA Officer.

For data sets generated for EPH/VPH, and various general chemistry methods, an Olin Level I data review will be performed. The Olin Level I review checklists are presented in Appendix D. The Level I procedures are similar to a Tier II review. During the Level I validation the primary method requirements and QC checks of the analytical method are reviewed to evaluate the precision and accuracy of the results.

A project chemist review and validation will be performed on the remaining methods. This program includes a variety of analytical methods that do not have validation guidelines established by EPA. Methods include some SW-846 procedures and a subset of methods developed at subcontracted laboratories as performance based methods. During validation of data from these methods, the project chemist will evaluate QC measurements associated with the data sets using goals discussed in Section 7.2, Worksheets #11, 12b, 21 and 24a, or SOPs provided by the subcontract laboratories. The validation will be completed using the general approach for data validation described in USEPA Region 1 guidelines (USEPA,1996). All data sets will have a review of the primary method requirements and QC checks of the analytical method to evaluate the precision and accuracy of the results. Chemist Review validation will include the following reviews and evaluations of QC measurements:

- Data package narratives and sample summary information
- Sample collection, preservation, and holding times
- Chain of custody
- QC blanks
- Laboratory control sample results
- Matrix spike results
- Laboratory and/or field duplicate precision
- Surrogate recovery (if applicable)
- Internal standard recovery (if applicable)

A full validation will be completed on approximately 10 percent of the chemist review data sets. Full validation will include reviews of instrument calibration, raw data and logbook documentation, and calculation and transcription checks routinely completed during a Tier III validation as applicable to each method. If during full validation, significant errors are discovered in the QC summary forms, calculations, or data reporting, a full validation will be performed on a larger subset of data packages until the extent of the errors is identified.

Results Qualification

Results will be qualified using general procedures described in the USEPA validation guideline and the judgment of the project chemist. Assessments, qualifications, and professional judgments will be summarized in data validation reports presented with each investigation report. Data Validation checklists will be used by the data validator to document validation actions and qualifiers added to the data. Validation Checklists and Olin Level I data review checklists are presented in Appendix D of this QAPP.

Electronic Data Verification

Electronic data provided by the laboratory will be entered into the project database as described in Section 15. During data validation results entered into the project database will be compared to results reported on the hardcopy data packages to verify the accuracy of the laboratory electronic data. A minimum of ten percent of sample results for each method in each data delivery package will be compared to the electronic results in the database. If errors are discovered, review will be completed on a large subset of results until the extent of the errors is identified. Corrective actions will be initiated at MACTEC or at the laboratory to determine the source of the errors and implement corrective actions.

Data Validation Report

Upon completion of the validation, a report will be prepared. Validation reports will be organized by sample collection task and may include be designed to report on multiple sample delivery groups. The validation report(s) will include the following information:

- identity of the laboratory used for analysis
- a summary of analytical methods

- a summary of samples that are included in the sample set
- a listing of sample collection and analysis parameters that were evaluated
- PE sample scores and interpretations
- a discussion of data validation actions, qualifications, and observations
- a tabulation of validated samples results

Qualifiers applied to the data during validation will be entered into the electronic data deliverables in the database. Validated data will be used to generate tables and figures.

EPA-NE QAPP Worksheet #29a
Data Verification Process

Verification Task	Description	Responsible for Verification (Name, Organization)
COCs and Shipping Forms	Chain-of-Custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers for which they represent. When everything checks out, a copy of the COC will be retained in the site file, and the original and remaining copies will be taped inside the cooler for shipment.	MACTEC Field Operations Leader
COCs and Shipping Forms	COCs, forms, and shipping documentation will be reviewed to verify completeness in accordance with QAPP requirements. Sample coolers will not be sealed until this review has been performed.	MACTEC Field Sample Manager
Field Notes	Field notes will be reviewed on a daily basis to ensure notes are accurate, all necessary calibration information has been documented, and notes are complete.	MACTEC Field Operations Leader
Audit Reports	Upon report completion, a copy of all audit reports will be placed in the project file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the site file. At the beginning of each week, and at the completion of the site work, project file audit reports will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the Field Operations Leader will be notified to ensure action is taken.	Peter Thompson, MACTEC
Laboratory Data Packages*	All laboratory data packages will be verified internally by the laboratory performing the work for completeness prior to submittal. The laboratory shall complete DC-2 forms documenting the organization and contents of each data package.	Laboratories to be determined
Data Validation	All lab data reports will be technically reviewed for accuracy and completeness. Data validation is completed as specified in this QAPP.	MACTEC Project Chemist
Data Validation Reports	All data validation reports will be reviewed for completeness and technical content.	Chris Ricardi, MACTEC QA Officer.
Water Quality Field Data	All water quality measurements will be reviewed on a daily basis to ensure the results are representative and reasonable.	MACTEC Field Operations Leader

- Requires a signature after review has been completed.

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10/14/08

EPA-NE QAPP Worksheet #29b

Data Validation Summary Table

Medium/ Matrix	Analytical Parameter	Concentration Level	Validation Criteria	Validation Criteria Modified	Data Validation Tier Level	Modified Tier Level Used	Data Validator (Name, title and organizational affiliation)	Responsibility for Data Validations (Name, title and organizational affiliation)
SW,GW	VOCs	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	No	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	SVOCs	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	No	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	PEST, PCB	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	Yes	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	Herbicides	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	Yes	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	EPH	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Extractable Petroleum Hydrocarbons by Method MADEP-EPH-98-1	No	Olin Level I	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	VPH	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Volatile Petroleum Hydrocarbons by Method MADEP-VPH-98-1	No	Olin Level I	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW, GW	TAL Metals	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	No	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW, GW	Hexavalent Chromium	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Wet Chemistry Parameters by Various Methods	No	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW, GW	Nitrate, Nitrite, Fluoride, Bromide, Hardness	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Wet Chemistry Parameters by Various Methods	Yes	Olin Level I validation on all water data for wet chemistry methods	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW, GW	Alkalinity, Ammonia, Chemical Oxygen Demand (COD),	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Wet Chemistry Parameters by	No	Olin Level I validation on all water data for wet	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer

EPA-NE QAPP Worksheet #29b

Data Validation Summary Table

Medium/ Matrix	Analytical Parameter	Concentration Level	Validation Criteria	Validation Criteria Modified	Data Validation Tier Level	Modified Tier Level Used	Data Validator (Name, title and organizational affiliation)	Responsibility for Data Validations (Name, title and organizational affiliation)
	Chloride, pH		Various Methods		chemistry methods			
SW, GW	Specific Conductance, Sulfate, Total Dissolved Solids (TDS), Total Organic Carbon (TOC), Total Suspended Solids (TSS)	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Wet Chemistry Parameters by Various Methods	No	Olin Level I validation on all water data for wet chemistry methods	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	Formaldehyde, Acetaldehyde	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	Hydrazine, MMH and UDMH	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	Phthalic anhydride (acid)	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	N,N-dimethylformamide (DMF)	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	NDMA / NDPA	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	No	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	Nonylphenol (alkylphenols)	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SW,GW	Opex and Kempore	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
GW	Perchlorate	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	VOCs	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	Yes	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	SVOCs	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	Yes	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO	PCBs	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	Yes	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	EPH	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist,	No	Olin Level I	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer

EPA-NE QAPP Worksheet #29b

Data Validation Summary Table

Medium/ Matrix	Analytical Parameter	Concentration Level	Validation Criteria	Validation Criteria Modified	Data Validation Tier Level	Modified Tier Level Used	Data Validator (Name, title and organizational affiliation)	Responsibility for Data Validations (Name, title and organizational affiliation)
			Extractable Petroleum Hydrocarbons by Method MADEP-EPH-98-1					
SO,SD	VPH	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Volatile Petroleum Hydrocarbons by Method MADEP-VPH-98-1	No	Olin Level I	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	Metals	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	No	Tier II 10% Tier III	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	Hexavalent Chromium	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Wet Chemistry Parameters by Various Methods	No	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	Ammonia, Total Organic Carbon (TOC), Sulfate, Nitrate, Nitrite, Chloride	Low	Olin-Wilmington Level I Data Quality Evaluation. Standard Operating Procedure and Checklist, Wet Chemistry Parameters by Various Methods	No	Olin Level I validation on all water data for wet chemistry methods	Yes	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	Formaldehyde, Acetaldehyde	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	Hydrazine, MMH and UDMH	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	Phthalic anhydride (acid)	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	N,N-dimethylformamide (DMF)	Low	Performance based review / Professional judgment	Yes	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer
SO,SD	NDMA	Low	Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses	No	Chemist Review	No	MACTEC Chemist	Chris Ricardi, MACTEC QA Officer

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EPA-NE QAPP Worksheet #29c
Data Validation Modifications

Analyses to be performed for this project will be conducted using the methods referenced in Worksheet #20. Method performance criteria have been established. The QA/QC criteria are summarized in Worksheets #11, #12b, #21, #23a and #24a and will be used as control limits during data validation.

The basis for the validation will be the Region I, EPA-New England Data Validation Functional Guidelines (USEPA, 1996) and the Compendium of Quality Assurance and Quality Control Requirements and Performance Standards for Selected Analytical Methods Used in Support of Response Actions for the MCP (Bureau of Waste Site Cleanup; 1 Winter Street, Boston, Massachusetts 02108; WSC-CAM; May 2004 Massachusetts Department of Environmental Protection (MADEP), 2004.

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20.0 DATA USABILITY/RECONCILIATION WITH PROJECT QUALITY OBJECTIVES

20.1 DATA USABILITY

Prior to completing the RI/FS report an assessment will be complet4ed to determine if field and validated laboratory data collected during the investigation are consistent with the project quality objectives established for the project. The assessment of data usability will be completed at the end of each major sample collection event. The assessment will include a review of any field program issues, sample collection issues, field measurement issues, or laboratory data quality issues that were identified during the field sampling event and subsequent data review process. A data usability report (or subsection of the RI/FS report) will be completed that provides a discussion of field sampling problems that prevented collection of all samples, or other situations where data that were specified in the Work Plan and FSP were not obtained. Evaluation of on the PARCC parameters will be completed during data validation and chemistry reviews. Data may be qualified as estimated and potentially biased during data validation. Some result may be rejected based on the guidelines and QC results. RI Data gaps will be identified and interpretations on the significance of the data gaps and data quality uncertainties will be summarized in the RI report. Interpretations of the limitations on the use of the data, and the significance of data gaps will be included in the Data Usability Assessment.

20.2 PRECISION

The RPD between the matrix spike and matrix spike duplicate in the case of organic parameters, or sample and sample duplicate in the case of all parameters, is calculated to compare to precision objectives. MS/MSDs and laboratory duplicates will be used to assess analytical precision and the field duplicates will be used to assess project precision. The RPD will be calculated according to the following formula:

$$RPD = \frac{(Amount\ in\ Sample\,1 - Amount\ in\ Sample\,2)}{0.5\,(Amount\ in\ Sample\,1 + Amount\ in\ Sample\,2)}\,x\,100$$

The impact of analytical imprecision, project imprecision, and overall imprecision (when both analytical and project precision tests show problems) on data usability will be assessed. If the precision results yield data which are not usable, the Data Usability Assessment will identify how this problem will be resolved and the potential need for re-sampling will be discussed in the final project report.

20.3 ACCURACY

If field or laboratory contamination exists, the impact on the data will be evaluated during the Data Usability Assessment. The direction of bias for contamination will be identified.

In order to assure the accuracy of the analytical procedures, matrix spike samples will be utilized. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample, determines percent recovery (%R).

Accuracy is similarly assessed by determining %Rs for surrogate compounds added to each field and QC sample to be analyzed for organic parameters. Accuracy for all analyses will be further assessed through determination of %Rs for LCS, PEs, and calibration results, etc. If the Data Validation Reports indicate contamination and/or analytical biases, the impact on the data will be assessed.

%R for MS/MSD results will be determined according to the following equation:

$$\%R = \frac{(Amount\,in\,Spiked\,Sample - Amount\,in\,Sample)}{Known\,\text{Amount}\,\text{Added}}\,x\,100$$

%R for LCSs, LFBs, and surrogate compound results will be determined according to the following equation:

$$\%R = \frac{Experimental\,Concentration}{Known\,Amount\,\text{Added}}\,x\,100$$

Overall contamination and accuracy/bias will be reviewed for each matrix and analytical parameter. The Data Usability Assessment will include any limitations on the use of the data, if it is limited to a particular matrix, SDG, parameter, or laboratory. Any trends in PE results will be evaluated for each matrix and analytical parameter. If any PE results show false negatives or false positives, the impact of these results will be assessed. If the accuracy results yield data which are not usable, the Data Usability Assessment will identify how this problem will be resolved and the potential need for resampling will be discussed in the final project report.

20.4 REPRESENTATIVENESS

If field duplicates indicate spatial variability, the Data Usability Assessment will evaluate the impact on the data. Overall sample representativeness will be evaluated for each matrix and analytical parameter.

The Data Usability Assessment will include any limitations on the use of the data, if limited to a particular matrix, SDG, parameter, or laboratory. If the results of the evaluation of representativeness yield data which are not usable, the Data Usability Assessment will identify how this problem will be resolved and the potential need for resampling will be discussed in the final project report.

20.5 SENSITIVITY AND QUANTITATION LIMITS

Method and instrument sensitivity will be evaluated through the use of MDL studies for all analyses. MDL studies will be provided to MACTEC by the laboratories. MACTEC will evaluate the MDL studies to ensure the laboratories can meet required practical quantitation limits presented in Worksheet #9b.

Overall sensitivity will be reviewed for each matrix and analytical parameter. The impact on the lack of sensitivity or the reporting of higher quantitation limits by the laboratory will be assessed. The Data Usability Assessment will include any limitations on the use of the data, if limited to a particular matrix, SDG, parameter, or laboratory. If the results of the evaluation of sensitivity yield data which are not usable, the Data Usability Assessment will identify how this problem will be resolved and the potential need for resampling will be discussed in the final project report.

20.6 COMPLETENESS

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed or processed. Following completion of the testing, the percent completeness will be calculated by the following equation:

$$Completeness = \frac{(number\ of\ valid\ measurements)}{(number\ of\ measurements\ planned)} x100$$

Overall completeness will be reviewed for each matrix and analytical parameter. The Data Usability Assessment will include any limitations on the use of the data, if limited to a particular matrix, SDG, parameter, or laboratory. If the results of the evaluation of completeness yield data which are not usable, the Data Usability Assessment will identify how this problem will be resolved and the potential need for resampling will be discussed in the final project report.

20.7 DATA LIMITATIONS AND ACTIONS

The field and laboratory data collected during this investigation will be used to achieve the objectives identified in Section 7.0 of this QAPP. The QC results associated with each analytical parameter for each matrix will be compared to the objectives presented in this QAPP. Data generated in association with QC results meeting the stated acceptance criteria (i.e., data determined to be valid) will be considered usable for decision-making purposes. Limitations on the use of the data will be stated and explained, if necessary.

In addition, the data obtained will be both qualitatively and quantitatively assessed on a project-wide, matrix-specific, and parameter-specific basis. Results of the measurement error assessments will be applied against the site as a whole; any conclusions will be documented in the final report. Data generated in associated with QC results not meeting the stated acceptance criteria may still be considered usable for decision-making purposes, depending on certain factors. This assessment will be performed by the MACTEC Project Manager, in conjunction with the MACTEC Project QA Officer, and the results presented and discussed in detail in the final report. Factors to be considered in this assessment of field and laboratory data will include, but not necessarily be limited to, the following.

- conformance to the field methodologies and SOPs proposed in the QAPP;
- conformance to the EPA methods referenced in the QAPP;
- adherence to proposed sampling strategy;
- presence of elevated detection limits due to matrix interferences or contaminants present at high concentrations;
- presence of analytes not expected to be present;
- conformance to validation protocols included in the QAPP for both field and laboratory data;
- unusable data sets (qualified as “R”) based on the data validation results;
- data sets identified as usable for limited purposes (qualified as “J”) based on the data validation results;
- effect of qualifiers applied as a result of data validation on the ability to achieve the project objectives;
- status of all issues requiring corrective action, as presented in the QA reports to management;
- effect of nonconformance (procedures or requirements) on project objectives;
- adequacy of the data as a whole in meeting the project objectives; and
- identification of any remaining data gaps and need to reevaluate data needs.

Every attempt will be made to eliminate any sources of sampling and analytical error as early as possible in the program. An ongoing data assessment program throughout the program will also assist in the early detection and correction of problems, thereby ensuring that project objectives are met.

Reconciliation with the project objectives will have been considered to have been met if the measurement performance criteria from Section 7.0 are met. If the data usability indicates that the project quality objectives in Section 7.0 have not been met, then the project management team will meet to determine any additional work to be performed under this site management plan.

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22.0 LIST OF ACRONYMS AND ABBREVIATIONS

µg/L	micrograms per liter
Al	aluminum
AOC	Administrative Order of Consent
AS/SVE	air sparge/ soil vapor extraction system
BEHP	bis-2-ethylhexlphathalate
CA	Corrective Action
Ca	calcium
Cl	chloride
CLP	Contract Laboratory Program
COC	chain of custody
COD	Chemical Oxygen Demand
COPCs	contaminants of potential concern
Cr	chromium
CRA	Conestoga Rovers Associates
CSL	Calcium Sulfate Landfill
DAPL	dense aqueous phase liquid
DO	dissolved oxygen
DQIs	Data Quality Indicators
DQOs	data quality objectives
EB	Equipment Blank
EDD	electronic data deliverable
EPH	Extractable Petroleum Hydrocarbons
FDRs	Field Data Records
Fe	iron
FID	Flame Ionization Detector
FRI	Focused Remedial Investigation
FSP	Field Sampling Plan
GEI	Geotechnical Engineers, Inc.
HSO	Health and Safety Officer

LIST OF ACRONYMS AND ABBREVIATIONS - CONTINUED

IRA	Immediate Response Action
LCSs	laboratory control samples
LNAPL	light non aqueous phase liquid
LSPs	Licensed Site Professionals
MADEP	Massachusetts Department of Environmental Protection
MADEQE	Massachusetts Department of Environmental Quality Engineering
MCP	Massachusetts Contingency Plan
MDLs	method detection limits
mg/L	milligrams per Liter
MMBA	Maple Meadow Brook and Saw Mill Brook
Mn	manganese
MS	matrix spike
MSD	Matrix spike duplicates
MWRA	Massachusetts Water Resource Authority
MWSWs	municipal water supply wells
Na	sodium
NCP	National Contingency Plan
NDMA	N-nitrosodimethylamine
NDPA	n-nitrosodiphenylamine
ng/L	nanograms per Liter
NELAP	National Environmental Laboratory Accreditation Program
NEPCO	New England Pollution Control Company
NH3	ammonia
NPI	National Polychemicals, Inc.
NPL	National Priorities List
on-PWD	on-Property West Ditch
ORP	oxidation-reduction potential
PALs	Project Action Limits
PCBs	Polychlorinated Biphenyls
PE	performance evaluation
PESs	Performance evaluation samples
PID	Photoionization Detector
PP	Proposed Plan

LIST OF ACRONYMS AND ABBREVIATIONS - CONTINUED

PQOs	Project quality objectives
Property	Olin Property
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QLs	quantitation limits
RAMs	Release Abatement Measures
RGP	Remediation General Permit
ROD	Record of Decision
RPD	relative percent difference
SAP	Sampling and Analysis Plan
SO4	sulfate
SOPs	Standard Operating Procedures
SOW	Statement of Work
SVOCs	semi-volatile organic compounds
SW	Surface Water
SWMUs	Solid Waste Management Units
TAL	Test America Laboratories
TCL	Target Compound List
TDS	Total Dissolved Solids
TED	Technical Environmental Database
TMP	trimethylpentenes
TOC	Total Organic Carbon
TSAs	Technical system audits
TSS	Total Suspended Solids
umhos/cm	micromhos per centimeter
USEPA	United States Environmental Protection Agency
VOCs	volatile organic compounds
VPH	Volatile Petroleum Hydrocarbons

APPENDIX A
RESUMES

Career Summary

Mr. Thompson has over two decades of experience in hard rock geology, hydrogeology and environmental engineering. His responsibilities have included senior technical and project management roles on sites contaminated with volatile and semi-volatile organic contaminants, metals, pesticides, PCBs, Dense Non-Aqueous Phase Liquids, (DNAPL), Dense Aqueous Phase Liquids (DAPL), petroleum hydrocarbons and metal sulfide mining wastes. He has prepared numerous Remedial Investigations and Feasibility Studies under CERCLA and State lead programs, including two Technical Impracticability (TI) Waivers for PCE/TCE in fractured bedrock. Mr. Thompson is the technical leader of MACTEC's Portland, Maine Bedrock Investigation Group.

He co-authored the technical guidance document for the USEPA for Subtitle D Solid Waste Facility Criteria and participated as a lecturer for USEPA on the nationwide MSWLF Seminar Program. As a result of this work in fractured bedrock, Mr. Thompson was hired by the National Groundwater Association (NGWA) as an instructor for the NGWA bedrock characterization workshop, taught in Portland, Maine and Houston, Texas in 2005.

Education:

Master of Science Civil Engineering, 1989, University of New Hampshire, EIT
Bachelor of Arts Earth and Environmental Science, 1980, Wesleyan University

Certifications and Non Professional Licenses:
OSHA 40-hour HAZWOPER Safety Work Zone Safety, US, #NV9999
OSHA 8-hour Refresher Safety Work Zone Safety, US, #NV9999
OSHA 8-hour Supervisor Safety Officer Work Zone Safety, US, #NV9999

Project Experience

Project Manager: Maine Department of Environmental Protection Smith Junkyard Hazardous Waste Remedial Investigation / Feasibility Study (RI/FS), Meddybemps, Maine. Remedial Investigation and Feasibility Study in accordance with state and U.S. EPA requirements at 52-acre junkyard area which apparently received hazardous wastes that also affected surrounding properties. Responsible for preparation of proposal, work plans, managing Remedial Investigation field work, preparing RI report, Risk assessments, and Feasibility Study.

Senior Hydrogeologist: U.S. Department of Energy (DOE)/Air Force Base Conversion Agency (AFBCA), Loring Air Force Base Hazardous Waste Remedial Action Program (HAZWRAP), Installation Restoration Program (IRP), Limestone, Maine. Environmental services since 1986 under the Installation Restoration Program (IRP) on nearly 9,000 acres (14 square miles) of land in Aroostook County, Maine. Performed a Remedial Investigation/Feasibility Study (RI/FS) covering 14 sites on the base, remedial design (RD), Site Investigations (SIs) at four additional sites, Preliminary Assessments (PAs) at 22 potential sites, and basewide groundwater and surface water studies. As technical leader, coordinated preparation for the first ever comprehensive Regulatory Review Meeting on all Operable Units at Loring Air Force Base.

Senior Hydrogeologist: Quarry Pilot Project, Loring Air Force Base, AFBCA, Limestone, Maine. As the technical leader for this project, Mr. Thompson was responsible for directing the investigation and evaluation of remedial alternatives for DNAPL in a fractured limestone bedrock aquifer. This project required development of an innovative approach involving methanol extraction of borehole rock samples to assess the fracture specific distribution of PCE, TCE, BTEX and related compounds and relating this data back in a detailed conceptual model to establish presence / absence, distribution, and volume estimates for residual DNAPL. The work included detailed geologic mapping, completion of cored and air hammered boreholes, borehole geophysics including ATV, BIPS, HPFM, Caliper, natural Gamma, and electrical logs, and low stress aquifer testing. The evaluation of potential remedial technologies included evaluation of bedrock foc, matrix porosity, phase relationships, matrix diffusion and fracture conductivities, in relationship to the potential effectiveness of in-situ chemical oxidation by permanganate. Surface geophysical investigations used to identify subsurface structure and fracture conditions identified magnetic anomalies that were caused by approximately 300 buried drums, which were removed.

Senior Hydrogeologist: Operable Unit 12 RI/FS, Lockheed Martin Energy Systems Hazardous Waste Remedial Action Program (HAZWRAP), Loring Air Force Base, Limestone, Maine. Senior Hydrogeologist supporting the Basewide Groundwater Operable Unit (OU 12) which entailed investigation of all discrete and co-mingled bedrock groundwater plumes and overburden groundwater at LAFB. Significant contributions to the program included development of a basewide bedrock structural model which included mapping folds, fracture patterns and faults based primarily on rock core, geologic mapping and borehole geophysical data (caliper log, heat pulse flow meter, packer data, Acoustic Televiewer logs (ATV, etc.), high altitude photolineament analysis, rock core and geologic mapping). This structural model significantly improved interpretation of groundwater flow conditions, interaction between aquifer systems, contaminant migration and fate, and greatly improved regulatory confidence in the Air Forces' ability to evaluate and manage environmental impacts within the fractured bedrock aquifer underlying LAFB. Technical lead in finalizing the Basewide Groundwater RI and FS which included extensive fracture flow groundwater modeling concerning the effects of matrix diffusion and fracture hydraulics on the behavior of fuel and chlorinated solvent contaminants in fractured media. Also responsible for technical review of the Natural Attenuation Evaluation which included six fuel plumes, four chlorinated solvent plumes and five co-mingled fuel and chlorinated solvent plumes.

Senior Hydrogeologist: New York State Department of Environmental Conservation Superfund Hazardous Waste Disposal Sites Engineering Services Contract, Numerous State Sites, New York. Broad range of standby engineering services at inactive hazardous waste disposal sites on as-needed basis. Ten major sites under this $15 million contract included drycleaning operations, waste oil disposal sites and closed landfills. Services included site assessments, risk assessments, remedial investigations / feasibility studies (RI/FS), remedial design, construction oversight, monitoring and interaction with regulatory agencies and the public. Responsible for performing senior technical review of Site Assessment Reports, and RI / FS work plans.

Senior Hydrogeologist: Maine Department of Transportation Callahan Mine Superfund Site Remedial Investigation / Feasibility Study (RI/FS), Brooksville, Maine. Hydrogeologic technical lead for Remedial Investigation/Feasibility Study of 150-acre former copper mining site located on Penobscot Bay. RI includes study of metal-contaminated sediments, including ecological risk assessment of metals in marine and estuarine and terrestrial environments, contamination and hydrogeological assessment of source areas including waste rock and tailings piles. Responsible for development of work plans for groundwater investigations of waste rock piles, ore processing areas, tailings pile, and offsite areas; participated in characterization of metals contamination in marine sediment adjoining this former zinc and copper massive sulfide mine.

Senior Hydrogeologist: ABB - Combustion Engineering, Inc. Henry Knob Former Mine Site Remedial Investigation / Feasibility Study (RI/FS), Clover, South Carolina. Remedial Investigation and Feasibility Study (RI/FS) at site of former 185-acre kyanite mine, now with 7-acre acidic pond on site and with nearby mine tailings and former ore-processing facilities. Responsible for senior review of work plans, preparation of the project QAPP, review Interim RI deliverables.

Senior Hydrogeologist: KerrAmerican Mine Remediation Design and Construction Management Services, Blue Hill, Maine. Design and construction management services for site remediation at a former zinc-copper mine closed in the early 1980s and covering 80 acres along Carleton Stream; site encompasses a 19-acre plant site, 34-acre tailings impoundment area, 15-acre auxiliary pond, drainage ditch and other features. Responsible as both project geologist and technical director for development of the project, including file reviews, borrow pit and quarry studies, local property owner cooperation and public relations, site geologic studies, and development of project technical approaches for mine closure, and negotiation with regulators on behalf of client.

Senior Hydrogeologist: Eastland Woolen Mill Superfund Site CERCLA RI/FS, Corinna Maine, USACE, USEPA. As senior hydrogeologist prepared work plans, directed RI field investigation activities, evaluated all RI data and prepared the RI/FS report with other MACTEC engineers and scientists and participated in public meetings. The work included delineation and evaluation of remedies for chlorinated VOC, metals and PAH contamination in sediment, chlorinated VOC contamination in surficial and subsurface soils, and in underlying fractured bedrock. In the bedrock groundwater system, DNAPL, composed of mono-, di, and trichlorobenzene compounds impacted the bedrock aquifer to a depth of 300 feet below ground surface in a discretely fractured meta sedimentary bedrock. The RI was started in July of 1999 and was concluded in 2001. The RI Report and Feasibility Study were both finalized in July of 2002 and a ROD for the groundwater operable unit (OU 1) was signed in September 2002.

Senior Hydrogeologist: Eastland Woolen Mill Superfund Site Remedial Design, Corinna Maine, USACE, USEPA. Lead design of fractured bedrock treatment for the Site. Prepared a remedial design work plan that includes a comprehensive fractured bedrock chemical source characterization, hydraulic characterization, to understand the fractured flow transport characteristics and contaminant mass distribution.

Project Manager: Modeling Peer Review, Hows Corner Superfund Site, Maine DEP.

At the invitation of the Maine DEP, MACTEC agreed to peer review modeling conducted by Woodard and Curran for the Hows Corner Superfund Site PRP Group of the fate of PCE in a fractured bedrock recharge area. Serving as Project Manager, worked with the groundwater modeler to evaluate and suggest improvements to the model prepared for the RI Report for the site. The purpose was to develop suggestions for model improvements that would more realistically reflect the site conditions and provide a more reasonable tool for the PRP group to evaluate potential performance of proposed remedial alternatives.

Project Manager: Site Investigation, Biddeford Textile Mills, Biddeford Maine

Maine DEP. The Maine DEP, contracted MACTEC to conduct an investigation into fuel oil, metals and chlorinated solvent contamination within five subterranean canals, built in the late 1800's under a textile mill complex in Biddeford Maine. The data was developed to assist the Emergency Response Unit of the Maine DEP in understanding source of contamination relative to the location of operating businesses located overhead in the Mill complexes.

Project Manager: Maine DEP, Petroleum Investigation and Remediation Program. As project manager, he is responsible for planning and managing the investigation of six petroleum impacted sites including MTBE in fractured bedrock, the installation of a soil vapor extraction system to recover K-1 kerosene contamination in a dense, fractured till, execution of remedial activities for soils contaminated with No. 2 heating fuel oil, upgrading an site overburden groundwater product recovery pump system, evaluation of pilot testings at a site where migration of free product and dissolved fuel contaminants had impacted downgradient, private bedrock water supply wells, and completing a soil removal action at another site.

Project Manager: Windsor School Site, Windsor VT, VT DEC. As project manager, was responsible to VT DEC for development of work plan, managing groundwater monitoring program, and interpretation of results and preparation of reports at this PCP/ dioxin contaminated groundwater site.

Project Manager: S.D. Warren Co., Skowhegan, Maine. As project manager for this $2.3 MM landfill construction project, responsible for oversight of the construction services group engineering staff, management of third party geomembrane QA/QC subcontractor, communications with the Maine Department of Environmental Protection and client.

Project Manager: Confidential Client, Vermont. As project manager, developed the scope and oversaw the technical execution of a operational review of an industrial effluent treatment plant to determine operational and capital improvements needed to improve effluent quality. Also directed the design and permitting of an aboveground storage and dispensing facility for diesel and gasoline fuels.

Engineer: U.S. EPA - Office of Solid Waste, Washington, D.C. As an engineer, took a leading technical role in writing the technical guidance document to aid permit writers, designers and owner/operators in meeting the recently promulgated RCRA Subtitle D criteria governing

municipal solid waste landfill facilities. The document was peer reviewed nationally and published by EPA. The document covers such topics as facility siting, design, groundwater monitoring, corrective action and operations. MACTEC was not involved in the development of the regulations but provided technical guidance to cost effectively meet regulatory requirements.

Publications and Presentations

2007 Lesson's Learned From Two Decades of Detailed Characterization of Chlorinated Solvent Source Areas in Fractured Meta-Sedimentary Bedrock Settings in Maine. Presented at the Geologic Society of America, Characterization and Remediation of Contaminated Bedrock Aquifers April 2007, Durham, N.H., GSA Abstracts with Programs Vol. 39, No. 1

2006 Comparison of Multi-Level Monitoring Well Technologies, High Resolution Site Characterization and Remediation, GRAC Symposium, November 14-16, 2006. Long Beach, California. Michael Taraszki, PG, CHG - Oakland, California, Peter Thompson - Portland, Maine

2005. Course Instructor: USEPA/NGWA Workshop. Characterization and Remediation of Contaminated Groundwater in Fractured Bedrock. September 2005 – Portland Maine, November 2005 – Houston Texas

2005. Detailed Fractured Bedrock Characterization for Remedial Design at the Eastland Woolen Mill Superfund Site, Corinna, Maine Presented at 2005 NGWA Eastern Regional Groundwater Issues Focus Conference, Portland Maine. September 2005.

2004. The Regional Bedrock Structure at Loring Air Force Base, Limestone, Maine; The Unifying Model for the Study of Basewide Groundwater Contamination. Presented at 2004 NGWA Fractured Bedrock Conference, Portland Maine. September 2004.

1997. Applicability and Evaluation of Intrinsic Remediation as a Remedial Tool for Petroleum Contaminated Bedrock Aquifers; A case Study from Loring Air Force Base, Limestone, Maine. Co –Author Paper Presented at the fourth Lessons Learned in Remediation of Petroleum-Contaminated Sites in Maine Conference, April. 1997

1995. Risk Issues Affecting Strategies for Remediation of Fuel Oil Adjacent to Building Foundations Paper Presented at the third Lessons Learned in Remediation of Petroleum-Contaminated Sites in Maine Conference, April.

1994. Contributing Author: USEPA Seminar Publication; Design Operation and Closure of Municipal Solid Waste Landfills. EPA/625/R-94/008. September.

1993. Pulp and Paper Mill Landfill Gas and Odor Control. Paper presented at ME/NH Regional TAPPI meeting in March 1993 reviewed the sources, evolution, migration and control of landfill gases using active and passive gas control technologies.

1993. TerraProbe Probe Site Investigation and a Vapor Extraction Pilot Study at Lee, Maine Paper presented at the Lessons Learned In Petroleum Remediation Seminar sponsored by the Maine Department of Environmental Protection. June.

1992, - 1995. RCRA Subtitle D Lectures: Design, Operation and Closure of Municipal Solid Waste Landfills. Two day lecture series sponsored by U.S. EPA presented in seven cities including Omaha, Dallas, New York City, Denver, Chicago, Honolulu and Guam, Nogales, Mexico and Mexicalli, BC., Mexico.

1991. Principal Author: USEPA: Solid Waste Disposal Facility Criteria - Technical Manual: EPAA530-R-93-017. November. 1991

Career Summary

Mr. Murphy has 23 years of risk assessment, risk management, and site closure experience. Mr. Murphy is the Group Leader for Risk Assessment and Natural Resources. Mr. Murphy has conducted and managed numerous baseline risk assessments within federal and state Superfund and RCRA programs and has used the results of those assessments to assist in the development of practical, risk-based remedial requirements and strategic plans for cost-effective remediation and property redevelopment and re-use. Mr. Murphy has conducted, directed, or reviewed more than 100 Massachusetts Contingency Plan (MCP) human health risk assessments. Many of these risk assessments, in combination with Activity and Use Limitations, have been essential elements in the redevelopment of contaminated properties. Mr. Murphy has extensive experience in application of risk-based corrective action (RBCA) techniques to a wide range of environmental contamination problems. Mr. Murphy has extensive experience in communicating risk analysis information to regulators, advisory committees, public education meetings, and to communities potentially impacted by environmental contamination. He has also developed risk-based guidelines, standards, and policies for contaminants in air, water, vapor intrusion, soil, sediments, sludge, ash, and fish. He has directed database management activities to support risk assessment and site decision making. Mr. Murphy has more than 15 years of experience in assigning, scheduling, directing, and reviewing risk assessment projects. Mr. Murphy was a major contributor to the development of the risk assessment and risk management components of the Massachusetts Contingency Plan (MCP), including the redevelopment of soil and groundwater cleanup standards. Mr. Murphy has participated in the Massachusetts Superfund Advisory Committee meetings, has contributed to the Numerical Standards Workgroup and Indoor Air Workgroup in support of the most recent round of revisions to the soil and groundwater cleanup standards contained in the MCP. Mr. Murphy is a member of the Licensed Site Professional Association Technical Practices Committee.

Education:

M.S.P.H., Environmental Sciences and Engineering School of Public Health, University of North Carolina, 1980

B.A., Biology, Merrimack College, North Andover, Massachusetts, 1973

Project Experience

Group Leader for Risk Assessment and Natural Resources. Technical review responsibility for the public health risk assessments conducted in the northeast. Responsible for assigning and scheduling human health and ecological risk assessments and applied ecology projects in the Wakefield, Massachusetts, office. Key projects listed below highlight site redevelopment experience as well as risk communication and regulatory experience. Extensive USEPA and Massachusetts (MCP) experience is presented in the sections following.

USEPA Region I RI/FS and RFI/CMS Projects

Senior Risk Assessor: Battelle and US Army Corps of Engineers, New England Division Manufacturing Facility, Centredale Manor Superfund Site, Providence, Rhode Island – Directed development of sampling and analysis plans, data evaluation workplan, biota consumption risk assessment workplan, and baseline ecological risk assessment workplan under CERCLA requirements. Served as Baseline Human Health Risk Assessment (BHHRA) lead and resource manager and technical reviewer for the Baseline Ecological Risk Assessment (BERA). Developed Preliminary Remediation Goals for impacted media (biota, sediment, and surface soil). Met regularly with USEPA representatives and presenting work products. Dioxins and hexachloroxanthene are signature compounds for this site that encompasses multiple reaches of the Woonasquatucket River. Provided risk assessment support for the Feasibility Study and for preparation of Record of Decision (ROD) documents. Conducted residual risk analysis and evaluation of short-term risks associated with proposed remedial alternatives.

Senior Risk Assessor: Connecticut Yankee Atomic Power Company
Former Power Generation Facility (Haddam Neck Plant), Haddam, Connecticut – Conducted technical review for the Baseline Ecological Risk Assessment (BERA) prepared for the RCRA Facility Investigation (RFI) for the former atomic power facility. The BERA addressed 24 areas of concern (AOC), including terrestrial, wetlands, and aquatic habitats.

Senior Risk Assessor: Confidential Client
Former Research and Development Facility, Windsor, Connecticut – Conducted technical review for the Baseline Ecological Risk Assessment (BERA) prepared for the RCRA Facility Investigation (RFI) prepared for the former facility used for research, development, engineering, production, and servicing of nuclear and fossil fuel systems.

Senior Risk Assessor: US Army Corps of Engineers, New England Division
Manufacturing Facility, Eastern Woolen Mill Superfund Site, Corinna, Maine – Conducted senior technical review for the baseline human health risk assessment and served as the resource manager for both the human health and ecological risk assessment activities. The human health risk assessment evaluated numerous exposure points at and remote from the original source area in and along the adjacent river. Potential exposures to soil, surface water, sediment, and groundwater used as drinking water were evaluated. The signature compounds at the site were chlorobenzenes.

Senior Risk Assessor: US Army Corps of Engineers, New England Division
U.S. Army Environmental Center, Sudbury Annex, Massachusetts – Technical Advisor/Senior Technical Reviewer for investigation of base-wide arsenic soil contamination problem. Developed a site-specific bioavailability factor for arsenic in soils for use in risk assessment and remedial decision-making (CERCLA-Region I). In vitro bioaccessibility testing conducted in Wakefield, Massachusetts, laboratory.

Senior Risk Assessor: Confidential Manufacturing Client
Manufacturing Facility, Bridgeport, Connecticut – Developed risk assessment component of a proposal to achieve site stabilization as part of a voluntary (RCRA) Phase I investigation. Made recommendations for soil, soil gas, and groundwater sampling and analysis to support the risk assessment activities. Directed the risk assessment activities focusing on potential human exposures to chlorinated solvents migrating from groundwater into buildings and chlorinated solvents and petroleum potentially migrating to the adjacent, tidally influenced surface water body. Conducted interior building survey to identify preferential vapor migration pathways and likely

human exposure locations. Conducted risk assessment based on indoor air sampling results. Conducted technical review of ecological risk assessment. Presented risk-based Phase I results to USEPA Region I and Connecticut DEP representatives.

Senior Risk Assessor: Confidential Manufacturing Client
Manufacturing/Research Facility, Windsor, Connecticut – Developed human health risk assessment component of a proposal for the RCRA Facility Investigation (RFI). Made recommendations for soil, soil gas, surface water, sediment, and groundwater sampling and analysis to support the risk assessment activities. Directed the risk assessment activities focusing on potential human exposures to chlorinated solvents migrating from groundwater and soil into buildings and chlorinated solvents and petroleum potentially migrating to the adjacent, surface water bodies. Additional exposures evaluated include soil direct contact, surface water, and sediment direct contact. Presented proposed risk assessment approach to USEPA Region I and Connecticut DEP representatives. A total of 26 Areas of Concern were assessed. Risk assessment approach is a hybrid of the USEPA Superfund approach and the State of Connecticut Remedial Standards approach. Directed human health and ecological risk assessments and conducted technical review of Baseline Human Health Risk Assessment (BHHRA) and Baseline Ecological Risk Assessment (BERA).

Senior Risk Assessor: US Air Force and HAZWRAP
Loring Air Force Base, Caribou, Maine – Managed the preparation of federal Superfund baseline health and ecological risk assessments and development of risk-based remedial objectives for multiple operable units. Submitted written reports and presented results to and negotiated remedial requirements with USEPA Region I and Maine Department of Environmental Protection.

Senior Risk Assessor: Confidential Manufacturing Client
Paper Mill, Maine – Conducted Public Health and Environmental Risk Evaluation as part of a RCRA Facility Investigation Report. Prepared risk assessment for seven solid waste management units (SWMUs), represented client in meetings with USEPA Region I and Maine DEP representatives, responded to comments, and finalized report.

Senior Risk Assessor: HAZWRAP
Massachusetts Military Reservation, Cape Cod, Massachusetts – Managed the preparation of health and ecological risk assessments (CERCLA – Region I) for more than 15 sites. Directed the preparation and use of a system of linked spreadsheets containing standardized exposure and toxicity data, which when combined with site-specific chemical data, allowed for the rapid completion of human health and ecological risk evaluations. Prepared risk assessment reports and provided support for Decision Documents to support no further action decisions and Feasibility Study activities. Conducted senior technical review for comprehensive preliminary risk assessments for the leading edge of the Ashumet Valley Plume.

CERCLA RI/FS and RCRA RFI/CMS Projects Outside USEPA Region I

Senior Risk Assessor: Confidential Chemical Manufacturer
Former Chemical Manufacturing Facility, Saltville, Virginia – Conducted Monte Carlo risk analysis for consumption of freshwater fish that contain methylmercury in the North Fork of the Halston River at Saltville, Virginia. Risk analysis demonstrated that the risks were overestimated using default USEPA (CERCLA- Region IV) exposure assumptions. Monte Carlo analysis concluded that risks associated with potential fish consumption by fishermen were within allowable limits. Mercury was the major chemical of concern.

Senior Risk Assessor: US Navy, Southern Division
Southern Division Naval Facilities Engineering Command, Cecil Field, Jacksonville, Florida – Conducted Monte Carlo risk assessment for potential groundwater ingestion at Operable Unit 1. The default USEPA point estimate of non-cancer risk suggested that future use of the groundwater might be associated with unacceptable risks. The Monte Carlo analysis addressed intra-population variability in exposure parameters and also the uncertainty in estimated concentrations of inorganics in groundwater. The Monte Carlo risk assessment concluded that risks were more likely within an allowable range than was indicated by the point estimates of risk.

Senior Risk Assessor: US Navy, Southern Division
Southern Division Naval Facilities Engineering Command, Cecil Field, Jacksonville, Florida – Conducted (CERCLA) health risk assessment associated with consumption of self-caught fish at Lake Fretwell. Prepared letter report and communicated results to client and EPA Region IV and Florida DEP officials.

Senior Risk Assessor: US Navy, Southern Division
Southern Division Naval Facilities Engineering Command, Jacksonville, Florida – Managed and wrote (CERCLA) baseline human health risk assessment for Operable Unit 1 utilizing a Presumptive Remedy approach for the former landfill area.

Senior Risk Assessor: US Navy, Southern Division
Southern Division Naval Facilities Engineering Command, NTC Orlando, Florida – Successfully negotiated a no further action decision (CERCLA – Region IV) for surface soils at Operable Unit 1. Conducted a cost-effectiveness analysis and demonstrated in a presentation to USEPA and Florida Department of Environmental Protection that remediation of soils would have minimal risk reduction impact for the planned uses of the property.

Senior Risk Assessor: US Navy, Southern Division
Southern Division Naval Facilities Engineering Command, Florida, Georgia, Alabama, and Mississippi – Provided senior technical guidance and technical review for human health and ecological baseline risk assessments conducted for CERCLA and RCRA sites at several Naval facilities in the southeastern United States (USEPA Region IV). Bases for which risk assessments were performed include MCLB Albany in Georgia, NAS Jacksonville in Florida, NAS Meridian in Mississippi, Naval Submarine Base King's Bay in Georgia, Outlying Field Barin in Alabama, Naval Air Station Cecil Field in Florida, and U.S. Naval Station Mayport in Florida. Provided guidance concerning compliance with current risk assessment practices and federal and state regulatory requirements and guidance.

Senior Risk Assessor: Confidential Client
Former Coke Plant and Tar Plant Superfund Site, Ironton, Ohio – Conducted technical review for the 2006 Lagoon Area Vegetation and Benthic Macroinvertebrate Monitoring Report required under CERCLA.

Massachusetts Contingency Plan Projects

Senior Risk Assessor: Confidential Client
Former Manufacturing Site/Former Landfill, Everett, Massachusetts – For MCP Health and Ecological Risk Assessment to Support Site Redevelopment, conducted hot spot analysis, developed health-based target soil levels for a Release Abatement Measure, and conducted (Massachusetts Contingency Plan) Method 3 human health risk characterization. The risk characterization employed the newly developed Extractable Petroleum Hydrocarbon/Volatile Petroleum Hydrocarbon analytical techniques to address petroleum releases (fuel oils, mineral oils, Stoddard solvent) and the "adult lead model" to evaluate worker exposure and risk associated with lead in soils. Developed the risk-related components of the Activity and Use Limitation for the site. Provided technical and regulatory advice to client during real estate transfer process.

Senior Risk Assessor: U.S. Army Corps of Engineers, New England Division
Watertown Arsenal, Massachusetts – For MCP Human Health Risk Assessment to Support Site Redevelopment, directed and conducted Massachusetts Contingency Plan Method 3 human health risk characterization at a former power plant facility in Watertown, Massachusetts. Both light and heavy fuel oils were released at the site. The risk characterization utilizes Extractable Petroleum Hydrocarbon/Volatile Petroleum Hydrocarbon soil and groundwater data and an innovative method for utilizing historical TPH data to estimate Extractable Petroleum Hydrocarbon/Volatile Petroleum Hydrocarbon concentrations in soil.

Senior Risk Assessor: Confidential Chemical Industry Client
Chemical Facility, Massachusetts – Project Manager and Technical Lead for MCP Human Health Risk Assessment to Support Site Redevelopment, conducted (Massachusetts Contingency Plan) Method 3 risk assessment for soils, groundwater, surface water, and sediments. The evaluation included more than 100 chemicals of potential concern in soils (inorganics, organics, pesticides) and groundwater. Developed Reference Doses for uncommon chemicals detected at the site. Conducted imminent hazard evaluations for potential and actual impacts on public water supply. Contributed to development of a Monitoring Plan and Contingency Plan for the public water supply. Presented results to Massachusetts Department of Environmental Protection representatives. Conducted a focused risk assessment to support site redevelopment and building construction and property transfer. Completed risk assessment to support the re-use of excavated/stockpiled soils as upland soils. Completed exposure/risk assessment to support the discontinuance of a emissions control system for an operating soil vapor extraction system. Directed numerous human health and ecological risk assessments to support Immediate Response Action (IRA) and Release Abatement Measure (RAM) status reports and closure reports. Directed sampling of soil, sediment, and surface water to support risk assessment activities. Contributed to site closure strategies.

Senior Risk Assessor: U.S. Army Corps of Engineers, New England Division
Hingham Annex, Massachusetts – For MCP Human Health and Ecological Risk Assessments, provided senior technical review for Hingham Annex (Massachusetts Contingency Plan) Method 3 human health and ecological risk assessments. PAHs in sediment and petroleum in soils are the predominant oil and/or hazardous materials.

Senior Risk Assessor: U.S. Army Corps of Engineers, New England Division
General Services Administration Site, Watertown Arsenal, Massachusetts – Conducted a (Massachusetts Contingency Plan) Method 3 Human Health Risk Characterization. Assessment involved multimedia exposure and risk assessment for metals in soils chlorinated solvents.

Senior Risk Assessor: U.S. Army Corps of Engineers, New England Division
Watertown Arsenal, Massachusetts – Conducted an Imminent Hazard Evaluation related to surface soils contamination (arsenic) at Arsenal Park. Imminent hazard evaluation consisted of a (Massachusetts Contingency Plan) Method 3 risk assessment. Results of this assessment were used to identify extent of soil removal activities.

Senior Risk Assessor: Confidential Client
Manufacturing Facility, Massachusetts – For MCP Human Health Risk Assessment to Support Site Redevelopment, conducted (Massachusetts Contingency Plan) Method 3 human health risk characterization and developed the content of an Activity and Use Limitation in support of the sale and redevelopment of a manufacturing facility. The risk assessment includes the assessment of soil (petroleum, metals and chlorinated solvents), groundwater (chlorinated solvents and petroleum) and indoor air (chlorinated solvents) exposures and associated risks per the MCP. Developed scope of work for a comprehensive indoor air monitoring program to support the risk characterization. Presented results and conclusions of the risk assessment to purchaser of the property and associated consultants and attorneys. As project manager, directed indoor air monitoring and conducted Method 3 human health risk characterization to document a condition of no significant risk for the facility after conversion from industrial to residential use.

Manufacturing Facility, Haverhill, Massachusetts – Directed preparation of a Massachusetts Contingency Plan Method 3 Human Health Risk Assessment to support a Response Action Outcome. Release of metals and volatile organic compounds to soil was the focus of the assessment. The risk assessment followed a limited soil removal action. Current industrial and potential unrestricted future land uses were evaluated. The assessment concluded that a condition of No Significant Risk had been achieved and the site could be closed without any land use restrictions.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant Site, Lawrence, Massachusetts – Wrote risk assessment proposal for (Massachusetts Contingency Plan) Method 3 human health and ecological risk assessment. Directed project and conducted senior technical review of quantitative human health risk characterization and worked with client in developing MCP site closure strategy. Site closure included maintenance of current land use via institutional controls and riverbank stabilization.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant Site, Attleboro, Massachusetts – Directed project and conducted senior technical review of risk-based closure. Method 3 (Massachusetts Contingency Plan) risk assessment utilized hot spot analysis to determine location and volume of surgical soil removal necessary (Release Abatement Measure) to achieve a condition of no significant risk for various potential future land use options. Results of the risk assessment were used in determination of the most beneficial future use of the site and to determine content of an Activity and Use Limitation. Site was redeveloped as a paved parking facility.

Senior Risk Assessor: Boston Edison
Boston, Massachusetts – Wrote risk assessment proposal and conducted senior technical review for more than 10 Massachusetts Contingency Plan disposal sites. Method 2 risk assessments were conducted for these cable oil sites using EPH/VPH analytical data.

Senior Risk Assessor: Confidential Chemical Industry Client
Chemical Manufacturing/Processing Facility, Salem, Massachusetts – Developed proposal for (Massachusetts Contingency Plan) Method 3 risk characterization. Contributed to MCP site closure strategy development, including Release Abatement Measures and Activity and Use Limitations. Directed the risk characterizations that were a component of the Comprehensive Site Investigation Report. Chromium, pentachlorophenol, petroleum and chlorinated solvents in soils and groundwater and chromium in surface water and sediment are the focus of the risk assessment.

Senior Risk Assessor: Confidential Consulting Firm
Ten Mile River, Attleboro, Massachusetts – Conducted (Massachusetts Contingency Plan) Method 2 Risk Assessment including development of soil and groundwater standards for ethanol and mineral spirits at a site adjacent to the river. Negotiated Scope of Work and contract. Managed human health and ecological components of the project and wrote report.

Senior Risk Assessor: Confidential Client
Former Manufacturing Facility, Everett, Massachusetts – Conducted third-party review of human health and ecological risk assessments conducted for eight separate disposal areas. Provided comments on the consistency of the risk assessments with current risk assessment practice and with the requirements of the newly revised Massachusetts Contingency Plan. Identified those aspects that are likely to raise concerns at the Massachusetts Department of Environmental Protection and assessed the defensibility of the risk assessment in those areas. Prepared written comments and discussed those comments with client and other consultants preparing and revising the reports. Identified documentation needs for Activity and Use Limitations that are necessary to support the final cleanup plans that are based on the risk assessment reports.

Additional Projects

Confidential Corporate Client – Conducted a survey of the State and USEPA regulatory requirements for groundwater remediation with respect to vapor intrusion into buildings. Summarized the actual requirements and the technical basis of each set of requirements. Prepared a Discussion Paper comparing and contrasting the technical basis and level of protection offered industrial/commercial workers in potential vapor intrusion sites managed under Work Place Air Standards, USEPA risk-based approaches, and the various States' approaching for managing vapor intrusion. Also prepared a summary of the current status and technical basis of the toxicity values for trichloroethylene that may be utilized in evaluating the human health risks associated with vapor intrusion into buildings. Prepared a briefing for Corporate Remediation, Real Estate, Legal, and Facility Management personnel discussing those requirements and discussing and comparing levels of protection afforded industrial/commercial workers and residents by the various regulatory requirements. Prepared recommendations for collection of information necessary to evaluate potential vapor intrusion issues at a facility.

Prepared, in 2006, the Site Investigation Work Plan, and directed the field investigation and preparation of the Supplemental Site Investigation Report (SIR) per the Rhode Island Remediation Regulations for the "Park Parcel"in North Providence, Rhode Island. Investigation included sampling and analysis of surficial soils and surface water and sediment of the adjacent freshwater

pond. The Supplemental SIR included a Method 1 human health risk assessment for soils, a Method 3 (quantitative risk calculations) human health risk assessment for the freshwater pond, and a Screening level Ecological Risk Assessment (SLERA) for the surface water and sediments of the adjacent pond.

Former Silverware Manufacturer, Providence, Rhode Island – In 2004, developed, in conjunction with municipal officials, Rhode Island Department of Environmental Management representatives, private parties and our client, a health-protective configuration/design for a park with walking trails along the shore of the large pond adjacent to the site. Site remedies will be designed into the configuration/design of the park. Directed human health risk assessment activities to support development of Remedial Action Plan to support redevelopment of the former manufacturing facility property. Heavy metals, arsenic, petroleum, and volatile organic compounds were addressed in the risk assessment activities. Developed sampling and analysis plan top support human health and ecological risk assessment for surface water and sediments of the pond. Contributed to the development of an Environmental Land Use Restriction to support the passive recreational use of the property.

Also, for the adjacent parcel of land, directed human health risk assessment and development of remedial requirements for soil and groundwater to support the redevelopment of the former industrial property into a retail center, including a supermarket, several retail shops and a gasoline filling station. Contributed to the development of an Environmental Land Use Restriction to support the retail use of the property.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant (MGP) Site, Rochester, New Hampshire – Developed and implemented risk-based closure strategy. Overall site objectives are to ultimately meet applicable groundwater standards and continue to protect environmental receptors in the adjacent river via a combination of source removal and natural attenuation and to control potential risks associated with soil-via exposure pathway elimination and institutional controls. Directed human health and environmental risk assessment activities including development of risk-based soil target levels and negotiation of cleanup requirements with NH Department of Environmental Services.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant (MGP) Site, Exeter, New Hampshire – Prepared risk assessment component of the Site Investigation Report. Evaluated soil and groundwater data and recommended additional sampling of soil, groundwater, and soil gas to support risk assessment activities. Participated in meeting with NHDES to present the findings and conclusions of the assessment.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant (MGP) Site, Nashua, New Hampshire – Performed senior technical review for scoping and implementation of the Method 3 human health and environmental risk assessment scope of work for the portion of the Nashua River adjacent to the former MGP facility.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant (MGP) Site, Lewiston, Maine – Currently conducting streamlined risk assessment activities and developing risk-based closure strategy to support voluntary risk-based closure. Risk assessment includes assessment of soil, groundwater, potential vapor migration into buildings and potential impacts to sediment and surface water in the adjacent river. Impacts to the aquatic system are assessed via modeling of fate and transport of MGP constituents

in groundwater. Risk assessment will focus on current site uses that will be maintained via institutional controls.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant Waste Site, Scarborough, Maine – Developed and negotiated with state regulators soil cleanup goals. Cleanup goals were based on limited exposure potential to site soils to be maintained by institutional controls and physical barriers to direct exposure.

Senior Risk Assessor: Confidential Utility Client
Former Manufactured Gas Plant Site, Ridgewood, New Jersey – Conducted risk assessment associated with potential exposures to soils adjacent to a MGP site. Based on the risk evaluation, provided recommendations for limited removal actions to achieve risk reduction to acceptable levels. Carcinogenic PAHs and lead were the focus of the risk assessment.

Employer: Massachusetts Department of Environmental Protection, Boston, Massachusetts
Various Projects, Boston, Massachusetts – Responsible for prioritizing, scheduling, assigning, and supervising health risk analysis activities in the Office of Research and Standards. Also developed the risk analysis component of the Massachusetts Contingency Plan for waste disposal sites, directed and conducted risk analysis for waste disposal sites, municipal solid waste incinerators, solid waste disposal units, disposal and re-use of wastewater sludges and compost. Presented risk analysis information to advisory committees and public education meetings and communicated risk analysis results and interpretations to the public, municipal officials and other regulatory agencies. In addition, hired and trained staff.

Employer: Massachusetts Department of Environmental Quality Engineering, Boston, Massachusetts
Various Projects, Boston, Massachusetts – Assessed the fate and effects of environmental contaminants for decisions concerning the quality of the environment and public health protection. Developed health- and risk-based guidelines for contaminants in air, soil, water and ash. Also conducted and prepared reports for multimedia, multicontaminant health risk assessments using toxicological, epidemiological, chemical and fate-modeling principles. Developed and tested methods for setting guidelines, standards and policies for air, water, soil, and fish contaminants. Evaluated endangerment assessments and remedial action plans for hazardous waste sites, developed and recommended processes for health risk assessment and risk management to be incorporated into the Massachusetts Contingency Plan. In addition, presented recommendations to advisory committees and other public groups, planned and supervised activities of staff and student interns.

Employer: New England Interstate Water Pollution Control Commission, Boston, Massachusetts
Various Projects, New England – Assessed the fate and effects of environmental contaminants for decisions concerning the quality of the environment and public health protection. Also conducted health evaluations relative to air, water and soil contaminants and contaminated sites using toxicological, risk assessment, epidemiological, chemical and engineering principles. Developed and tested methods for setting guidelines for standards for contaminants in water, air, soil and fish and shellfish, as well as evaluate endangerment assessments and remedial action plans for hazardous waste sites. In addition, supervised staff and student interns.

Employer: Massachusetts Department of Environmental Quality Engineering, Boston, Massachusetts

University of Massachusetts, Boston, Massachusetts – Negotiated contracts, initiated, and managed the University of Massachusetts/DEQE research and training program, as well as monitored and reported technical progress and fiscal status of all research projects. Responsible for identifying agency research priorities and integrated them with research activities of other state and federal agencies.

Electronic Data Management

Wakefield, Massachusetts office – Collected and synthesized information concerning electronic database needs and requirements. Learned the basic principles of electronic database management and interfacing with graphics software packages. Proposed an organizational structure and process for all aspects of electronic management of site-related analytical data and graphics presentation of analytical data. This Microsoft Access™- -based system is now in use in the Wakefield office. This system is used to compile, review, and report site-specific analytical data throughout a project's duration in order to develop Site Conceptual Models, identify additional sampling needs, provide input for risk assessment activities, and prepare tabular and graphical presentations of data for reports and presentations.

Memberships, Professional Organizations, and Workgroups

Licensed Site Professional Association, Boston, MA
Licensed Site Professional Association, Technical Practices Committee
Massachusetts Department of Environmental Protection, Managing Asbestos in Soils Workgroup
Massachusetts Department of Environmental Protection, Ecological Risk Assessment Workgroup
Massachusetts Department of Environmental Protection, Indoor Air Workgroup

Publications and Presentations

2007. Reducing Uncertainty in Fish Consumption Risk Assessments – Potential Impacts on Development of Remedial Objectives, Sediment management Work Group, 2007 Spring Sponsor Forum, Cleveland, OH, May 3.
2005. Development of Risk-Based Sediment Preliminary Remediation Goals for the Centredale Manor Superfund Site, Third International Conference on Remediation of Contaminated Sediments, New Orleans, January..
2005. Overview of Ecological Risk Assessment. Presented at the Meeting of the Environmental Professionals of Connecticut, Connecticut Owner's Group, April 23.
2000. Redevelopment of a Former Landfill Site. The 16th Annual Conference on Contaminated Soils, Sediments and Water, University of Massachusetts at Amherst, October 17.
1996. Development of a site-specific health-based cleanup level for total petroleum hydrocarbon (TPH) contamination in soil. 1996 Society of Toxicology Annual Meeting, March 10, Anaheim, California (with Silverman, D.M, Pedersen, D.C.).
1996. The cost of attaining acceptable human health risk. American Defense Preparedness Association 22nd Environmental Symposium and Exhibition, Investing in Our Future: Reinventing Environmental Protection, March 21, Orlando, Florida (with Buckley, S.E.).
1994. Human health and ecological risk in the Superfund program - how has it been managed? Society of Risk Analysis Annual Conference, Baltimore, Maryland, December.
1994. Groundwater exposure assessment for human receptors - are Unfiltered monitoring well samples the best estimators of potential human exposures to inorganics? Society for Risk Analysis Annual Conference, Baltimore, Maryland, December (with Peters, J.H.).

1994. A comparison of three derivation methods for soil screening concentrations: ingestion of residential soil. Society for Risk Analysis Annual Conference, Baltimore, MD, December (with Silkowski, M.A., Buckley, S.E.).
1994. Impact of background screening on selection of contaminants of potential concern and risk estimates for soil. Ninth Annual Conference on Contaminated Soils, Amherst, MA, October (with J. Peters).
1993. Using risk assessment to develop site assessment and remediation strategies. Air and Waste Management Association Conference, Hartford, CT, October 19 (with Ellen Cool, Ph.D.).
1993. Integrated risk management: a tool for strategic decision-making. Presented at Air and Hazardous Waste Management Conference. Hartford, CT, October 20 (with Cool, Ellen G.).
1992. Derivation of a level of cadmium for Massachusetts type I sludge. Presented at 1992 Annual Meeting, Society of Toxicology, Seattle, WA, February (with Harnois, Marion, Paul Locke, and Carol West).
1992. What is the role of risk assessment in site management? Does it make a difference? Panel Discussion, Boston Risk Assessment Group and New England Chapter of the Society for Risk Analysis, January 22.
1991. Use of a streamlined risk assessment for 21 E waste site assessment and remediation. Presented at the Boston Risk Assessment Group and New England Chapter of the Society for Risk Analysis Seminar Series, October 16 (with Paul W. Locke).
1990. Expedited health risk assessment and decision-making processes for site remediation in Massachusetts. Proceedings of the International Specialty Conference, *How Clean Is Clean?* Air and Waste Management Association, Boston, MA, November.
1990. Evaluation of non-ingestion exposures from volatile organic compounds in drinking water. Presented at Drinking Water and Public Health Conference, Northeast Regional Environmental Public Health Center, Amherst, MA, May (with Martin, Karen A.).
1989. Using models to assess exposure from volatile organic compounds in drinking water: Groveland, Massachusetts Superfund Site. Presented at National Governor's Association Conference, *Assessing Exposure to Toxic Contaminants: Issues Facing State Government*, Salt Lake City, Utah, March (with Martin, Karen A.).
1988. Cyanides in residential soils: Is relocation necessary? Presented at the Regional Environmental Public Health Conference, Northeast Regional Environmental Public Health Center, Amherst, MA, May (with Karen Martin and Carol Rowan West).
1986. Development of a health-based ambient air guideline for polychlorinated dioxins and furans. Presented at Annual Meeting, Society for Risk Analysis, Boston, MA, November (with Carol Rowan West).

Career Summary

As a chemist and Quality Assurance Officer in the Government Programs Group, and as a QA/QC and chemistry lead for private commercial clients, Mr. Ricardi has 21 years experience in laboratory contracting, analytical chemistry and the validation, assessment, and management or data in support of remedial investigation, feasibility studies, and remedial actions. He is certified as an Environmental Analytical Chemist with the National Registry of Certified Chemists.

Mr. Ricardi's responsibilities include supervision of project chemists, data validation, analytical chemistry, and the preparation of Quality Assurance Project Plans (QAPPs) and chemical data quality reports. This includes numerous project located in USEPA Region I. He has worked on government programs under contract to the USACE, USAEC, HAZWRAP, USANG, and state agencies. He has extensive experience in hazardous waste site investigation and remediation, including preparation and review of field sampling and analysis plans and quality assurance plans, data validation and interpretation, designing and managing both onsite and offsite laboratory programs, laboratory audits and field audit, QA administration, and writing and reviewing investigation reports. He has extensive experience in USEPA analytical methodologies and interpretation of quality control data.

Education:

Bachelor of Science Science Wildlife Biology, 1982, University of Massachusetts

Certifications and Non Professional Licenses:

National Registry of Certified Chemists Environmental Analytical Chemist Laboratory, US, #2610
OSHA 40-hour HAZWOPER Safety Work Zone Safety, US, #NV9999
OSHA 8-hour Refresher Safety Work Zone Safety, US, #NV9999

Project Experience:

Project Chemist and Quality Assurance: Olin Corporation - 51 Eames Street, Wilmington, Massachusetts. Senior project chemist responsible for analytical laboratory program development and contracting, data validation, and data quality reporting, and the preparation of Work Plans and remedial reports under CERCLA.

Senior Chemist: Maine Department of Transportation Callahan Mine Superfund Site Remedial Investigation / Feasibility Study (RI/FS), Brooksville, Maine. Remedial Investigation/Feasibility Study of 150-acre former copper mining site located on Penobscot Bay. RI included study of metal-contaminated sediments, including ecological risk assessment of metals in marine and estuarine and terrestrial environments, contamination and hydrogeological assessment of source areas including waste rock and tailings piles. Responsible for Quality Assurance for a remedial investigation; prepared the Quality Assurance Project Plan based on the USEPA Region I guidance requirements, the design of the laboratory analytical program, prepared the analytical laboratory subcontract Statement of Work, contracted laboratory services, completed laboratory audits, and provided senior review of data validation and data quality reporting.

Quality Assurance / Quality Control Manager: U.S. Army Corps of Engineers - Baltimore District, Environmental Services Program Support at Region 12 Military Sites, Eastern U.S. Locations, Northeastern States. Environmental compliance services program support at present and former U.S. military installations throughout Northeast U.S. (Region 12) to support U.S. Army Installation Restoration Program and BRAC property transfer activities. Services provided at sites in Massachusetts, Connecticut, Maine, and in the Caribbean at Puerto Rico and St. Croix; services included Remedial Investigations / Feasibility Studies (RI/FS), risk assessments, wetlands mitigation, master planning / GIS services, groundwater sampling and development of Corrective Action Plans (CAP) and Records of Decision (ROD). Responsible for serving as QA Officer and Project Chemist for the Army Toxic and Hazardous Materials (USATHAMA) Program involving multiple sites, including: Natick Solider System Center, Massachusetts; Fort Devens, Ayer, Massachusetts; Fort Dix, Wrightstown, New Jersey, Badger Army Ammunition Plant, Wisconsin. Required: validation and management of laboratory data using the USAEC QA Program and IRDMIS and ERIS, production of quality control reports, assessing the nature, extent and behavior of organic and inorganic contaminants, and writing contamination and risk assessment reports.

Quality Assurance / Quality Control Manager: U.S. Army Corps of Engineers - New England District, Eastland Woolen Mill Site Remedial Investigation / Feasibility Study (RI/FS), Corinna, Maine. Multiple remedial investigations, assessments, feasibility studies and remedial design support activities at an abandoned textile mill site, on the National Priorities List, covering multiple city blocks, with 1,600 linear feet along the east branch of the Sebasticook River. Calculations indicate real-time data and maximum adaptability provided by the field analytical laboratory saved $900,000 in fixed base laboratory costs. Responsibilities included design of analytical chemistry programs, supervision of data validation, data management, and data quality reporting for over 3000 samples analyzed for VOCs, SVOCs, pesticides, PAH, PCBs including HRMS congeners and homologues, dioxins/furans, and metals. Designed and operated an on-site analytical laboratory testing soils collected during RI investigations and confirmation sampling during remedial actions, and air samples that were collected over a 2-year period to monitor fugitive emissions levels and potential exposure of local residents during remedial activities. Required data for development of human health and ecological risk assessments.

Quality Assurance / Quality Control Manager: U.S. Army Corps of Engineers - New England District, ID/IQ HTRW Investigation, Design, QC Services, Boston and Other Sites, Massachusetts. Hazardous, toxic, and radioactive waste (HTRW) investigations and engineering designs at 16 FUD and BRAC military installations in New England through indefinite delivery order contract. Work included RCRA / CERCLA site investigation studies; NEPA, UST, asbestos, and lead-based paint assessments; administrative records support, remedial design (landfill closure,

asbestos abatement, TPH removals); cost estimates; and construction plans / specifications and oversight. Responsible for providing QA/QC services for the following project sites under this contract: SSC Natick, Massachusetts; Fort Devens, Ayer, Massachusetts; Fort Dix, Wrightstown, New Jersey, Badger Army Ammunition Plant, Wisconsin; required validation and management of laboratory data using the USAEC QA Program and IRDMIS and ERIS, and production of quality control reports, assessing the nature, extent and behavior of organic and inorganic contaminants, and writing contamination and risk assessment reports.

Quality Assurance / Quality Control Manager: U.S. Department of Energy (DOE)/Air Force Base Conversion Agency (AFBCA), Loring Air Force Base Hazardous Waste Remedial Action Program (HAZWRAP), Installation Restoration Program (IRP), Limestone, Maine. Environmental services since 1986 under the Installation Restoration Program (IRP) on nearly 9,000 acres (14 square miles) of land in Aroostook County, Maine. Performed a Remedial Investigation/Feasibility Study (RI/FS) covering 14 sites on the base, remedial design (RD), Site Investigations (SIs) at four additional sites, Preliminary Assessments (PAs) at 22 potential sites, and basewide groundwater and surface water studies. Included an award-winning stream restoration project involving wetland remediation and restoration design for 34 acres of contaminated wetland / floodplain and 2.5 miles of stream bed. Responsible for development of work plans and quality assurance plans, analytical programs, data validation, data management, and RI reports completed under the HAZWRAP program; provided planning and review of internal processes related to collection and reporting of data; represented the Air Force in QA/QC related matters; responded to regulatory agency comments and concerns related to QA/QC; and supervised project chemists.

Project Chemist: U.S. Army Corps of Engineers - New England Division, NAE Total Environmental Restoration Contract (TERC) for New England Sites, Various New England Sites. As a subcontractor, completed work at 5 sites including Brownfield sites, Superfund sites, and a BRAC Site, including: HTRW services for Raymark Superfund Site, Stratford CT ; Silresim Superfund Site Lowell MA; New Bedford Harbor Superfund Project, New Bedford MA ; Watertown Arsenal, Watertown MA; and Stratford Army Engine Plant CT. Quickly mobilized resources and executed large, time-critical projects; provided USACE-approved on-site laboratory services; and delivered specialized expertise in remediation of solvent DNAPL, hexavalent chromium, and PCBs. Responsible for remedial investigations, monitoring programs, and remediation pilot studies at multiple AOCs for sites including Stratford Army Engine Plant; supervised validation of soil, groundwater, and air sample data for VOCs, SVOCs, pesticides, PAH, PCBs, and TAL metals and hexavalent chromium; included remedial investigation programs to identify the nature and extent of contamination, evaluation of data and State regulatory standards, and completion of a groundwater remediation pilot study on the effectiveness of in-situ chemical oxidation for VOCs and in-situ chemical reduction of hexavalent chromium.

Quality Assurance / Quality Control Manager: U.S. Army - Robert Morris Acquisition Center (RMAC), Soldier Systems Center A/E Services, Natick, Massachusetts. A/E services, including geotechnical investigation and preparation of Response Action Outcome (RAO) statement, for the Army's primary research complex for supplies and materials used by soldiers in the field. Responsible for providing consulting on analytical programs; managed data validation and assessment activities; and provided technical review on long-term-monitoring deliverables.

Quality Assurance / Quality Control Manager: U.S. Air Force Center for Environmental Excellence (AFCEE) Installation Restoration Program (IRP) Massachusetts Military Reservation 1983-2003, Massachusetts Mil. Reservation, Massachusetts. Long-term provision (since 1983) of engineering and environmental services for Installation Restoration Program (IRP) at a Superfund site, including complete CERCLA remedial investigation / feasibility studies, remedial design, construction management and project oversight for environmental investigation and remediation of 22,000-acre military complex operating since 1911 with airfields, cantonment area, landfills, housing, firing range areas, sewage treatment plant, fuel facilities and weapons storage areas. Contamination from petroleum fuel-related compounds, solvents, VOCs and metals threatening sole source aquifer underneath complex. Managed construction/installation of a 250,000-GPD facility for containment and treatment of a groundwater plume in addition to other key remedial activities. Responsible for quality assurance and senior technical review for a laboratory providing on-site analytical support for remedial investigations and remedial actions conducted by Jacobs Engineering Group, Inc. Played a key role in establishing analytical protocols, writing SOPs, and designing a computerized data management system to provide electronic deliverables in accordance with the Jacobs Environmental Management System and IRPIMS.

Senior Chemist: Confidential Client - Manufactured Gas Plant (MGP) Site Investigation, Feasibility Study, Remedial Design, Construction Management. Phase I and Phase II Site Investigations and feasibility study, analysis of remedial options and development of remedial approach to prevent migration of oil-like material (OLM, in DNAPL state) into river near a former coal gasification / manufactured gas plant (MGP). Provided construction management. Work conducted under state Voluntary Response Action Program. Provided asbestos abatement and removed asbestos at two buildings on site. Responsible for technical oversight of contract laboratories and validation of analytical chemistry data for Phase I and Phase II remedial investigations and subsequent remedial action; included the analysis of soil and water samples for VOCs, SVOCs, PAH, cyanides, PCBs, and petroleum hydrocarbons; validated air sampling data used to monitor fugitive emissions for aromatic VOCs, PAH, and dust using NIOSH methods.

Quality Assurance / Quality Control Manager: Honeywell International, Inc. Master Services Agreement for Remedial Investigation and Feasibility Studies (RI/FS) and Remedial Design 2002-2005, Various Locations, Nationwide U.S. Technical and design services for planning and implementing remedial actions and for remedial investigations and feasibility studies (RI/FS) at various locations under a Master Services Agreement for Honeywell International. Includes project management / control, historical research field investigation, analytical services, groundwater sampling, monitoring well installation, and environmental and construction oversight, data management, and report preparation as needed. Responsible for QA services for remedial investigations at a group of coal tar sites, including the Detroit Tar Site (Michigan), Fairfield Tar Site (Alabama), and the Ironton Tar Site (Ohio). Involved QA activities and analytical chemistry contracting, technical oversight of subcontractor laboratories, and quality reviews of all analytical data. Testing includes the development of analytical programs to test for VOCs, PAH, phenols, metals, cyanides in difficult sample matrices. Supervised laboratory programs that include air testing in support of evaluations of contaminant removal and fugitive emissions associated with vapor extraction systems, planned leaching tests associated with soil stabilization processes used during remedial actions.

Senior Chemist: Massachusetts Department of Environmental Protection Site Assessments and Remediation Support Services, Numerous locations statewide, Massachusetts. Environmental services for almost two decades (since 1986) involving more than 100 task order assignments executing complex and technically challenging environmental projects and sites, including initial site investigations (ISIs) and reviews; assessment and remedial actions at sites of oil and hazardous materials releases; potentially responsible party (PRP) identification and oversight; human health and ecological risk assessments; policy and program development and implementation; assessments and formerly used defense sites (FUDS); remedial construction implementation and oversight including innovative in-situ groundwater remediation technologies; and sediment characterization and modeling. Responsible for chemistry and data management support for the contract; evaluated laboratory results in accordance with MADEP protocols; assisted in laboratory management.

Project Experience While With Enseco, Inc., Arvada, Colorado, 1988

Consultant. Mr. Ricardi had a two-month contract agreement to design an introductory training program for the GC/MS division of Rocky Mountain Analytical Laboratory. Responsibilities included writing a training manual for analysts/chemists, and writing departmental Standard Operating Procedures for volatile and semivolatile organic analyses.

Project Experience While With Rocky Mountain Analytical Laboratory, Arvada, Colorado, 1985-1988

Organic Chemistry Analyst. Performed the analysis of volatile and semivolatile organic compounds using GC and GC/MS instrumentation. Methods included USEPA CLP protocol and custom methods for private clients.

Publications and Presentations

Ricardi, C. “The Truth Behind the Numbers: The Key to Effective Use of GRO and DRO Data.” Lessons Learned in the Remediation of Petroleum-Contaminated Sites in Maine. Conference Proceedings. Maine Department of Environmental Protection. April. 1997.

Ricardi, C. "Sport Fisheries vs. Acid Rain/The New Clean Air Act." New England Game and Fish. February. 1992.

Ricardi, C. "TCLP: Chemistry, Sampling and Uses." Air Quality and Waste Management Technical Conference. New England Section. Providence. RI. 1991.

Ricardi, C. "The Poisoning of Our Game and Fish." Sports Afield. January. 1991.

APPENDIX B
STANDARD OPERATING PROCEDURES OF FIXED LABORATORYANALYTICAL METHODS

LIST OF STANDARD OPERATING PROCEDURES OF FIXED LABORATORY ANALYTICAL METHODS INCLUDED IN APPENDIX B

- L-1 Trace Metals Analysis in Aqueous matrix by ICP-AES (6010B), ICP-MS (6020A), Cold Vapor Atomic Absorption analysis for Mercury (7470A)
- L-2: Trace Metals Analysis in Solid matrix by ICP-AES (6010B), ICP-MS (6020A), Cold Vapor Atomic Absorption analysis for Mercury (7471B)
- L-3: Analysis of Volatile Organic Compounds in Aqueous and Solid matrix by USEPA SW-846 8260B
- L-4: Analysis of Semi-Volatile Organic Compounds in Aqueous and Solid matrix by USEPA SW-846 8270C and USEPA SW-846 Low Level 8270C
- L-5: Determination of Nitrosamines by Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (GC/CI/MS/MS), EPA Modified Method 521.
- L-6: Analysis of Nitroso-dimethylamine (NDMA) in Water and Solid matrix by Modified by USEPA SW-846 8270C Low Level Method
- L-7: Analysis of Extractable Petroleum Hydrocarbons in Solid matrix by MADEP EPH-98-1.
- L-8: Analysis of Volatile Petroleum Hydrocarbons in Solid matrix by MADEP VPH-98-1
- L-11: Determination of Dinitrosopentamethylenetetramine (Opex) in Aqueous matrix by Method 8000B HPLC
- L-11: Determination of Azodicarbonamide (Kempore) in Aqueous matrix by Method 8000B HPLC
- L-12: Analysis of Sulfate by Ion Chromatography in Aqueous matrix by USEPA 300.0
- L-13: Analysis of Chloride by Ion Chromatography in Aqueous matrix by USEPA 300.0
- L-15: Analysis of Specific Conductivity, Alkalinity and/or pH using the ManSci MANTECH Autotitrator in Aqueous matrix by SM 2320B, SM 4500
- L-16: Analysis of Hardness in Aqueous matrix by SM 2340B
- L-17: Analysis of Nitrate by Ion Chromatography in Aqueous matrix by USEPA 300.0
- L-18: Analysis of Nitrite by Ion Chromatography in Aqueous matrix by USEPA 300
- L-19: Analysis of Total Dissolved Solids (TDS) by SM 2540C
- L-20: Analysis of Ammonia in Aqueous matrix by QuickChem Method 10-107-06-1-A

- L-21: Analysis of Chemical Oxygen Demand in Aqueous matrix by USEPA 410.4
- L-22: Analysis of Total Organic Carbon in Aqueous matrix by SM 5310B
- L-23: Analysis of Total Suspended Solids (TSS) by Standard Methods 2540D
- L-24: Determination of Hydrazine, MMH and UDMH by Modified Method 8315, LC/MS/MS.
- L-25: Analysis of Extractable Petroleum Hydrocarbons in Aqueous matrix by MADEP EPH-98-1
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- L-27: Analysis of Poly Chlorinated Biphenyls in Aqueous matrix by method USEPA SW-846 8082A
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- L-36: Analysis of Phthalic Acid and Maleic Acid in Water and Soil by HPLC
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- L-38 Analysis of Hexavalent Chromium in Aqueous Samples and Digestates from Solid Samples Via Ion Chromatography by SW-846 Method 7199
- L-39: Analytical Procedures for the Total Carbon, Non-purgeable Organic Carbon (Total Organic Carbon) and Total Iorganic Carbon by Lloyd Kahn.

STL-Westfield
Laboratory Quality Manual

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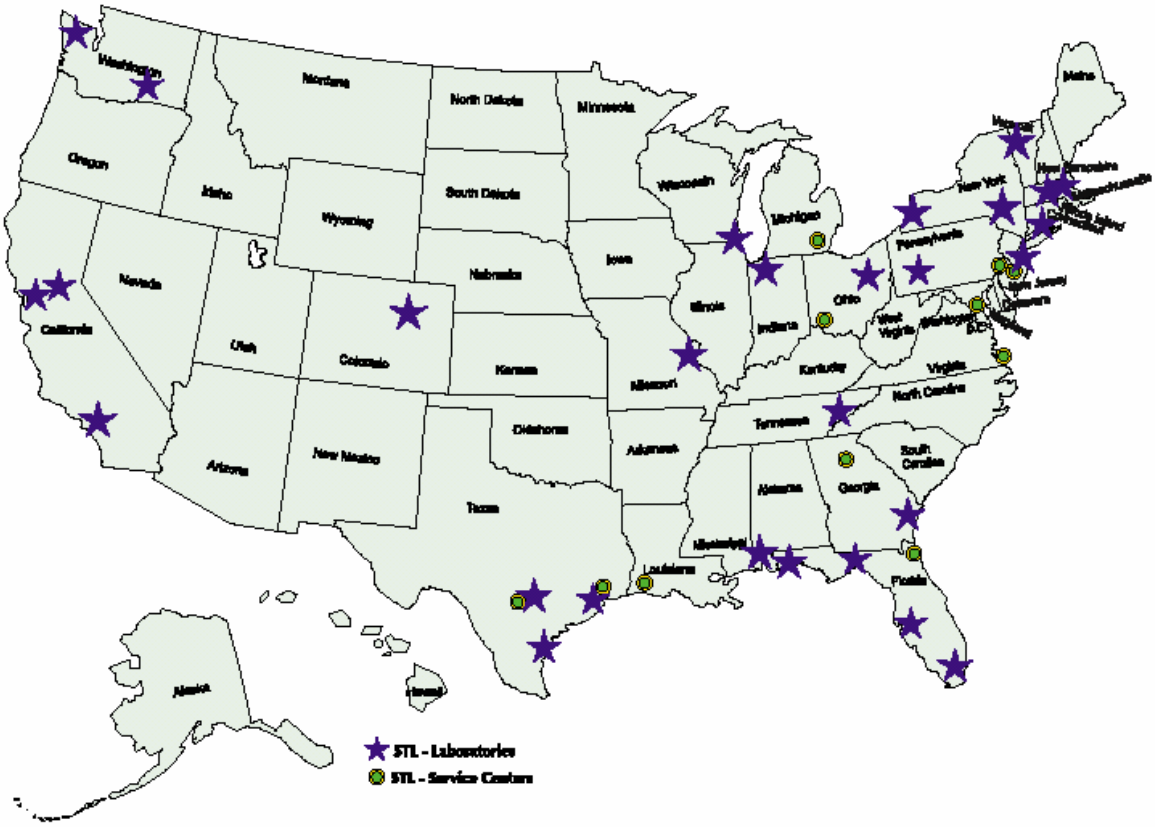
U.S. LABORATORY and SERVICE CENTER LOCATIONS



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STL's Facilities

STL's facilities are designed for efficient, automated high-quality operations. Super minicomputers and PC networks are used for sample tracking, work scheduling, status reporting and electronic data transfer to clients.

All STL facilities are equipped with structural safety features. Each employee is familiar with the location, use and capabilities of general and specialized safety features associated with their workspace. STL also provides and requires the use of certain items of protective equipment including safety glasses, protective clothing, gloves respirators, etc.

Because of the sensitivity of the work STL performs, access to all STL facilities is controlled through various security systems including locks, passwords, electronic access cards and manned reception areas.

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STL Westfield

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STL-Westfield, located in Midwestern Massachusetts, is 25 miles northwest of Hartford, CT, 94 miles west of Boston, MA and 76 miles east of Albany, NY. The laboratory is conveniently situated just off I-90, at Exit 3, in Westfield, MA.

Analytical Services

STL Westfield provides full-service environmental analytical services to client in Massachusetts, New England and beyond. The staff of 35 scientists and customer service specialists process over 3.5 million dollars of analysis annually. Analysis is performed under SW846, NPDES, SDWA and NIOSH methods for organics and inorganic target compounds in water, soil, sediment and air samples.

The laboratory specialized in “local” projects generally performed under state regulatory requirements. In addition, wastewater testing is performed for customers reporting to the EPA under the NPDES program. The laboratory prides itself on aggressive customer service that creates success for the customer by providing quality data on time. Our experienced project management team is not only familiar with state and federal regulations but also the reality of tight scheduling deadlines.

Several unique service features at the lab include:

- **Aquatic Toxicity Services-** the newly remodeled toxicity laboratory accommodates numerous fresh and salt-water species in both chronic and acute exposure evaluations.
- Two service locations within Massachusetts**-** in addition to the laboratory facility in Westfield, we operate a customer service facility in Billerica for customers in the eastern part of the state.
- Microscopy services**-** occasionally environmental customers require microscopy services such as unknown identification and coal ash/coal tar differentiation.
- Certification throughout New England **–** including Massachusetts, Connecticut, Maine, Rhode Island, Vermont, New York, New Jersey and Florida.
- **Massachusetts Contingency Plan (MCP) Analysis**

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Severn Trent Laboratories (STL) is the largest environmental laboratory company in the world with revenues in excess of $200 million and 28 laboratory locations in 20 states nationwide. These facilities have the combined experience of over 500 years in the environmental testing business.

Through continued investment in facilities, equipment, methods and people, STL has developed an unprecedented team of resources, experience and capabilities. With over 2000 chemists, microbiologists and environmental scientists, STL is well positioned to support a variety of clients including commercial, governmental and chemical industries.

STL's testing capabilities include chemical, physical and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air and saline/estuarine samples. Specialty capabilities include air toxics testing, mixed waste testing, tissue preparation and analysis, aquatic toxicology, dioxin/furan testing and microscopy.

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3.0 Description

3.1 Introduction

Severn Trent Laboratories (STL)-Westfield is a part of Severn Trent Services Inc. (STS), a major group of US based companies. Severn Trent, plc, owns both companies, and is an international provider of water and wastewater services headquartered in Birmingham, UK.

3.2 Terms and Definitions

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Audit: a systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results.

Confidential Business Information (CBI): information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

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Confirmation: verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Equipment Blank: a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Blank.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Federal Water Pollution Control Act (Clean Water Act, CWA): legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

Good Laboratory Practices (GLP): formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Detection Limit (IDL): the minimum amount of a substance that can be measured on a specific instrument, with a specified degree of confidence that the amount is greater than zero. The IDL is associated with the instrumental portion of a specific method only, and specific sample preparation steps are not considered in its derivation. A calculated IDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The IDL thus represents a range where qualititative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Initial Demonstration of Capability (IDC): procedure to establish the ability to generate acceptable accuracy and precision for analysts. This is also referred to as Initial Demonstration of Proficiency and Continuing Demonstration of Capability.

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Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): a document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Matrix: The substrate of a test sample. For purposes of batch and QC requirements determination, the matrix descriptions in Table 1 are used.

Table 1 Matrix Descriptions

Matrix	Description
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the

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point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Preservation: refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample.

Proficiency Testing: determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: a sample, the composition of which is unknown to the analyst, which is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits.

Proprietary: belonging to a private person or company.

Quantitation Limit (QL): the lowest point at which a substance can be quantitatively measured with a specified degree of confidence using a specific method. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL).

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Storage Blank: a blank matrix stored with field samples of a similar matrix.

Trip Blank: a blank matrix placed in a sealed container at the laboratory that is shipped and held unopened in the field and returned to the laboratory in the shipping container with the field samples.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

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Quality Assurance (Project) Plan (QAPP): a formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Control Sample: an uncontaminated sample matrix spiked with a known amount(s) of an analyte(s) from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Quality Management Plan (QMP): a formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Record Retention: the systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Resource Conservation and Recovery Act (RCRA): legislation under 42 USC 321 et seq. (1976).

Safe Drinking Water Act (SDWA): legislation under 42 USC 300f et seq. (1974), (Public Law 93-523).

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Selectivity: The capability of a method or instrument to respond to a target substance or constituent in the presence of non-target substances.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Systems Audit: a thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): legislation under 15 USC 2601 et seq., (1976).

Traceability: the property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

4.0 Organization and Personnel

4.1 QA Policy and Objectives

It is STL Westfield's policy to:

- provide high quality, consistent, and objective environmental testing services that meet all federal, state, NELAC and municipal regulatory requirements.
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- provide STL clients with the highest level of professionalism and the best service practices in the industry.
- build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- maintain a working environment that fosters open communication with both clients and staff.

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Management Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best overall service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a Quality System that is clear, effective, well communicated, and supported at all levels in the company. Simply put, STL-Westfield is committed to producing data of the utmost quality for our clients.

STL Mission Statement

Through the innovation and dedication of our people, together with the quality of our system, we will deliver levels of performance that delight our clients, retain confidence of our stakeholders and enables the profitable growth of our business.

Purpose

The purpose of the Quality Manual (QM) is to describe the STL Quality System and to outline how that system enables all employees of STL to meet the Quality Assurance (QA) policy. The QMP also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in the QMP.

4.2 QA Management

4.2.1 Organization and Responsibilities

A functional organizational chart of the individuals associated with Quality Assurance Management follows. The responsibilities of the individuals associated with Quality Assurance Management are described below. A list of STL Westfield personnel including education and experience is found in Table 6.

The General Manager is directly responsible for the daily operations of one or more operating facilities within STL. The GM's responsibilities include:

- Allocation of personnel and resources, long term planning, setting goals, and achieving the financial, business, and quality objectives of STL.

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- Ensures timely compliance with corporate management directives, policies, and management systems reviews.

The Laboratory Director oversees the daily operations of the laboratory. The duties and responsibilities of the Laboratory Director are:

- Supervision of staff, setting goals and objectives for both the business and the employees
- Achieving the financial, business, and quality objectives of the facility
- Ensures timely compliance with audits and corrective actions
- Responsible for maintaining a working environment, which encourages open, constructive problem solving and continuous improvement.

The Corporate QA Manager is responsible for establishing, implementing, and communicating STL's Quality System. The duties and responsibilities of the Corporate QA Manager are:

- Monitors compliance with the Corporate Quality Management Plan
- Conducts management system reviews
- Provides regulatory and technical updates to the STL facilities, assists in development of management plans and technical policies to be approved by the Chief Operating Officer (COO)
- Coordinates employee training within STL
- Available to any employee in STL to resolve data quality or ethical issues.

The QA Manager is responsible for reviewing and advising on all aspects of QA/QC. The duties and responsibilities of the QA Manager are to:

- Assist the Project Managers in specifying QA/QC procedures to be used during sample analysis.
- Implement quality control procedures and techniques to assure that the laboratory achieves established standards of quality.
- Evaluate data quality and maintain records.
- Monitor laboratory activities to determine conformance with the authorized quality assurance policy, and to implement appropriate steps to ensure adherence to quality assurance programs.
- Coordinate internal audits
- Review performance evaluation results.
- Administer intra-laboratory and inter-laboratory QA efforts.
- Responsible for implementing and communicating the Quality Manual
- Providing Quality Systems and Ethics training to all new personnel
- Prepare quality assurance report to management

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- Has final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of the analytical data.
-

The Technical Manager has overall responsibility to serve as a technical resource for the laboratory. The duties and responsibilities of the Technical Manager are to:

- serve as a technical resource to laboratory employees, as well as Project Managers, sales personnel, and clients.
- help manage the laboratory's LIMS system.
- reviews data produced in assigned departments and authorizes its release.
- works closely with the Report generation department and investigates issues raised by clients, QA, sales, and other departments to find root cause and implement corrective action and proper response.

The Project Manager has overall responsibility for management of the analytical requirements for sample analysis. The duties and responsibilities of the Project Manager are to:

- Administer and supervise all requirements of the analytical tasks to ensure meeting the client objectives on schedule.
- Act as liaison between the laboratory and the client to discuss and resolve any problems that may occur.
- Work with laboratory supervisors in planning and conducting progress meetings.
- Take part in corrective actions.

The individual responsible for sample management acts as sample custodian for the laboratory. Their duties and responsibilities are to:

- Sign for the incoming field samples and verify the data entered on the chain-of-custody forms.
- Advise the Project Manager of discrepancies, omissions or inappropriate samples.
- Prepare laboratory study documents and internal chain-of-custody documents.
- Oversee sample information entry into the laboratory sample database.

- Transfer sample and laboratory internal chain-of-custody documents to analytical personnel.

The System IT Manager is responsible for:

- The management and quality control of all computing systems.
- The installation, operation and maintenance of software and programs

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The Laboratory Supervisors are responsible for meeting all the technical and analytical terms and conditions for sample analysis. Their areas of responsibilities are to:

- Organize the personnel, equipment and materials in a manner required to fulfill the analytical requirements of sample analysis.
- Oversee all aspects of laboratory analyses and provide technical support when necessary.
- Review analytical data for validity and clarity.
- Maintain contact with the Project Manager in areas of technical concern, and advise the Project Manager of analytical progress, needs of potential problems that occur.
- Advise the laboratory director of progress, needs and potential problems that occur.
- Inform laboratory director if the daily review indicates a decline in data quality and implement actions.

The Sample Analysts are responsible for the analysis of samples. The analysts will:

- Schedule, prepare and analyze samples according to the method specific requirements indicated by the chain-of custody or daily backlogs.
- Advise the section supervisor of progress, needs and potential problems that occur.
- Verify that the laboratory QC and analytical procedures are being followed as specified.
- Review sample QC data, at least daily, including inspection of raw chromatograms and calibration curves.
- Inform section supervisors if the daily review indicates a decline in data quality and implement actions.

4.3 Quality System

4.3.1 Objectives of STL Quality System

The goal of the STL Quality System is to ensure that business operations are conducted with the highest standards of professionalism and integrity in the industry.

To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL provides the highest quality service available in the industry. A well-structured and well-communicated Quality System is essential in meeting

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this goal. STL's Quality System is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

The Corporate Quality Management Plan (QMP) is the basis and outline for STL's Quality System and contains general guidelines under which all STL facilities conduct their operations.

4.4 Project Document Control Procedures

Before releasing analytical results, the laboratory assembles and cross checks the information of custody records, lab bench sheets, analyst and instrument logs and other relevant data to ensure that data pertaining to each particular sample is consistent throughout the record.

4.4.1 Sample File Organization, Preparation and Review Procedures

Job Reports are created following completion of the job. One file sleeve will be assigned for each separate job. All documents, sample tags (if applicable), custody forms, and all other documentation pertaining to a particular job will be placed in the sleeve. Project files will be filed in numeric order and stored in a secure area with access limited to authorized personnel. Authorized personnel are limited to Supervisors, Lab Director, QA Officer and Project Managers or their designees.

4.5 Request, Tender, and Contract Review

4.5.1 Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL's intent to provide both standard and customized environmental laboratory services to our clients. To ensure project success, the technical staff performs a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

All contracts entered into by STL are reviewed and approved by the appropriate personnel at the facility or facilities performing the work. Any contract requirement or amendment to a contract communicated to STL verbally is documented and confirmed with the client in writing. Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL.

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All contracts, Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record.

4.5.2 Project Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site-specific testing programs. To achieve this goal, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively communicated to the laboratory personnel before and during the project.

Each STL facility has established procedures in order to ensure that communication is inclusive and effective. These include project memos, designation and meetings of project teams, and meetings between the laboratory staff and the client. STL has found it very effective to invite the client into this process. STL strongly encourages our clients to visit the laboratories and hold formal or informal sessions with employees in order to effectively communicate client needs on an ongoing basis, as well as project specific details for customized testing programs.

4.5.3 Subcontracting

Subcontracting is arranged with the documented consent of the client, in a timely response that shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility.
Proof of required certifications from the subcontract facility are maintained in STL project records. Samples are subcontracted under formal Chain of Custody (COC) or Work Share Agreement. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory.

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements.

STL will include subcontracted data as part of its final report in either a standard STL format or in its original form provided to STL by the subcontracting laboratory. In all cases, subcontracted data is clearly identified in the final report and case narrative.

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4.5.4 Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is done, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specify requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased.

4.5.5 Instrument Maintenance Activities and Schedules

A complete listing of instrumentation may be found in Table 8. Instrument preventative maintenance and careful calibration help to assure accurate measurements from laboratory instruments. Where applicable, all laboratory instrumentation is on a service contract with the instrument manufacturer or licensed service organization. The service contracts include regular preventative maintenance service calls on a scheduled basis.

Preventative maintenance procedures such as lubrication, source cleaning, detector cleaning and the frequency of such maintenance are performed according to the procedures delineated in the manufacturer's instrument manual or when deemed necessary by the analyst, see Table 9.0.

Instrument logbooks are in the laboratory at all times. They contain records of date instrument was put into service, condition of install instrument (new, used, reconditioned) usage, calibration, maintenance and repairs. Adequate supplies of spare parts such as GC columns, syringes, septa, injection port liners, and electronic parts are maintained in the laboratory so that they are available when needed.

After non routine maintenance has been performed a "Return to control" will be documented by listing the next valid run in the maintenance log.

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4.6 QA Document Control Procedure

4.6.1 Document Type

The following documents, at a minimum, are controlled at each STL Facility:

- Laboratory Quality Manual
- Standard Operating Procedures (SOP)
- Quality Management Plan
- Bench logs
- White papers
- Manuals
- Reference books

4.6.2 Document Control Procedure

Security and control of documents is necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by identification of the following items in the document: Document Name, Document Number, Revision Number, Effective Date, and Number of Pages. Controlled documents are authorized by the QA Department. Controlled documents are stored on an access-restricted directory.

There is a tracking form in Excel in the Q drive under the QAQC directory listing all controlled documents, control number, type of document, where they are located and date controlled.

Controlled documents are available for viewing at all locations where the operational activity described in the document is performed.

4.6.3 Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. A change form is generated and handed in to the QA department for review. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document are replaced with the current version of the document. The previous revision of the controlled document is archived by the QA Department.

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4.7 Records

4.7.1 Record Types

Record types are described in Table 2.

Table 2 STL Record Types

Raw Data Samples	Controlled Document	QC Records	Project Records	Administrative Records
Calibration	LQM	Audits/ Responses	COC Documentation	Accounting
Computer Tapes/Disk	QMP	Certifications	Contracts and Amendments	EH&S, Manual, Permits, Disposal Records
QC Samples	SOPs	Corrective Action	Correspondence	Employee Handbook
Sample data	Bench logs	Logbooks*	QAPP	OSHA 29 CFR Part 1910
Software (Version control)	White Papers, Work Instructions	Method & Software Validation, Verification	SAP	Personnel files, Employee Signature & Initials, Training Records
		Standards Certificates	Telephone logs, logbooks, E-mails	Technical and Administrative Policies

*Logbooks: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature

4.7.2 Record Retention

Table 3 outlines STL's standard record retention time. For raw data and project records, record retention is calculated from the date the project report is issued. For other records, such as Controlled Documents, QC, or Administrative Records, the retention time is calculated from the date the document is formally retired. Records related to the programs listed in Table 4 have lengthier retention requirements and are not subject to STL's standard record retention time.

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Table 3 STL Record Retention

Record Type		Archival Requirement
Raw Data	All*	10 Years from project completion
Controlled Documents	All*	10 Years from document retirement date
QC	All*	10 Years from archival
Project	All*	10 Years from project completion
Administrative	Personnel/Training	10 Years
	Accounting	10 Years from project completion

* Exceptions may be listed on a client by client basis

4.7.3 Programs with Longer Retention Requirements

Specific client projects and regulatory programs may have longer record retention requirements than the STL standard record retention length. In these cases, the longer retention requirement is noted in the archive. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to whom to contact for authorization prior to destroying the data.

4.7.4 Archives and Record Transfer

Archives are indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented.

STL ensures that all records are maintained as required by the regulatory guidelines and per the QMP upon facility location change or ownership transfer.

All observations and results recorded by STL Westfield are entered into the laboratory data entry system or into permanent laboratory logbooks. Data recorded are referenced with the sample laboratory number, date and analyst's initials in the indicated location on the bench sheet.

All logbooks and other document entries are made in ink. Any corrections made in a logbook will be made by crossing a single line through the error and entering the correct information. The person will subsequently date and initial the correction. Corrections made to other data records are made by crossing a single line through the error, entering the correct information and initialing and dating the correction.

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All chemicals and reagents received by STL Westfield must be dated upon receipt, and again dated and initialed upon opening.

The preparation of all standards and reagents must be in accordance with the written SOP. Preparation must be documented in a bound preparation log. Documentation must include the date, analyst initials, identification of stock source, the final concentration of the solution and the STL lot number.
Once in the possession of the Analyst, all logbooks are their responsibility to maintain. No logbooks are permitted outside the laboratory. Analysts are responsible for proper documentation in the logbooks. Once a Logbook has been completed it is returned to QA Manager for proper archiving.

4.8 Service to the Client

4.8.1 Sample Acceptance Policy

Prior to accepting samples a Chain-of-Custody must be completed with the following required information:

- Client address and contact
- Sampling location
- Date and time sampled
- Requested analyses
- Preservation
- Relinquished and received signatures.
- Temperature at receipt and if on ice

Samples are considered “compromised” if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification *with no ice present*.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non unique sample labelling.

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Note: All samples which require thermal preservation are considered acceptable if the arrival temperature is within 2°C of the required temperature or method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above freezing temperature of water to 6°C shall be acceptable.

Samples that are hand delivered immediately following collection may not meet these criteria, however they will be deemed acceptable, according to NELAC, if there is evidence that the chilling process has begun such as arrival on ice, etc.

When "compromised" samples are received, it is documented on the chain of custody and/or Job comment sheet and the client is contacted for instructions. If the client decides to proceed with analysis, the project report will clearly indicate any of the above conditions.

4.8.2 Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client.

4.8.3 Samples Tracking/Custody Procedures

- If the sampling event is conducted by an STL employee, the employee shall adhere to the requirements outlined in "General Aqueous Sampling and General Soil Sampling standard operation procedures. The SOPs detail proper sample collection techniques for inorganic, metals, organic and biological analysis.
- Samples are received at the laboratory by the sample custodian or designee who removes the samples from the shipping containers together with all accompanying documentation such as chain-of-custody (COC) forms, cooler receipt forms, analysis request forms, etc. An example chain-of custody form may be found in Appendix 1.
- The condition of the custody seal if required is examined and recorded on the COC.
- The temperature of the sample(s) upon receipt will be recorded on the COC.
- The pH of the sample(s) (when required) will be taken upon receipt. Any inappropriate pH reading will be recorded on the COC. Necessary pH

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adjustments will be made as required and documented on the Job comment sheet.

- The samples are inspected for general condition and the COC received with any samples is examined for discrepancies between package contents and the enclosed documents.
- Discrepancies, omissions, or inappropriate samples discovered will be noted and discussed with the Project Manager who will contact the client to resolve the problem.
- If the client cannot be reached, the samples will be assigned to appropriate cold storage until the problem is resolved.
- Samples delivered directly by the sample collector are received and inspected by the Sample Custodian or designee in the presence of the sample collector. Discrepancies, omissions, or inappropriate samples should be noted and discussed with the sample collector to resolve the problem.
- The samples will be entered by the sample custodian into the laboratory sample data-base upon successful completion of the sample log-in procedure. Sample entry will prepare a study document for all Jobs.
- Samples received by the Sample Custodian or designee will be assigned a unique laboratory Job number by the LIMS.
- Once the sample(s) are logged into the LIMS the Sample Custodian or designee will list the job number on the COC. The unique sample number will be printed out on a label and the label will be adhered to the clients sample container(s).
- All documents are then reviewed by a Project Manager to ensure that there are no transposition errors. The project manager or designee will validate the accuracy of the sample registration procedure by initialing the appropriate space(s) on the Study Document.
- All documents, sample tags, shipping labels, etc. will be included with the COC in the study document folder for that Job Number.
- Once in the possession of the Laboratory, all samples and extracts are stored and refrigerated in areas that are accessible only to Laboratory personnel. Access to all refrigerators during non-routine working hours is restricted to Supervisors or their designees.
- Access to the Laboratory facilities is limited to Laboratory personnel.
- All samples are stored at the Laboratory and are not disposed of until a minimum of 45 days after the project report has been sent unless prior arrangements have been made with the client.
- Samples are preserved per requirements or specific regulatory programs.
- All samples are stored according to the requirements outlined in the test method and in a manner such that they are not subject to cross contamination from their environment. Unless specified by the method or State regulations, a temperature range of 4 $\pm$ 2 ºC is used. Sample storage temperatures and microbiology incubators are monitored daily.

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4.8.4 Sample Disposal

STL Westfield retains samples for 45 days after sample receipt (approximately 30 days after the project report is sent). Some samples are required to be held longer periods based on client requirements. The laboratory must follow the longer retention time requirements require by regulation or client agreement. Sample may be return to the client upon written request. Unused portion of samples found to be hazardous according to State and Federal guidelines may be returned to the client upon completion of analytical work.

Samples shall be disposed of in accordance with federal, State and local regulations. The laboratory procedures for sample disposal are further described in the laboratory SOP *Hazardous Waste Management Procedures.*

4.9 Complaints

Client complaints are documented, communicated to management, and addressed promptly and thoroughly. Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a corrective action report. The Laboratory Director, Project Manager, and QA Manager are informed of all client complaints, and assist in resolving the complaint.

The nature of the complaint is identified, documented, and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA department is required to conduct a special audit to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is usually part of the overall action taken.

The number and nature of client complaints are reported to the Corporate QA Manager in the Corporate QA Monthly report submitted by each facility. The overall numbers of complaints received per facility are tracked and the appropriateness of the response to client complaints is assessed. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Management Systems Review. An example of our customer complaint form can be found in Appendix 1:

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4.10 Control of Non-conformances

Non-conformances include any out of control occurrence or a departure from documented procedures. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence in the form of a Non Conformance memo (NCM). The NCM can be generated from the LIMS at anytime during receipt of the sample(s) through the analytical process. The NCM will become part of the permanent job record.

When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. All records of reanalysis are kept with the project files.
Where non-conformances specifically affect a client's sample and/or data, the client is informed and action must be taken. Action can take the form of reporting and flagging the data, and including the non-conformance in the project narrative or cover letter.

4.11 Corrective Action

An important part of any quality assurance program is a well-defined, effective policy for correcting quality problems. This is depicted in the Figure 1. STL Westfield maintains a closed-loop corrective action system, which operates under the directions of the QA Manager. While the entire quality assurance program is designed to avoid problems, it also serves to identify and correct those that may exist. Usually these quality problems fall into two categories, immediate corrective action or long- term corrective action.

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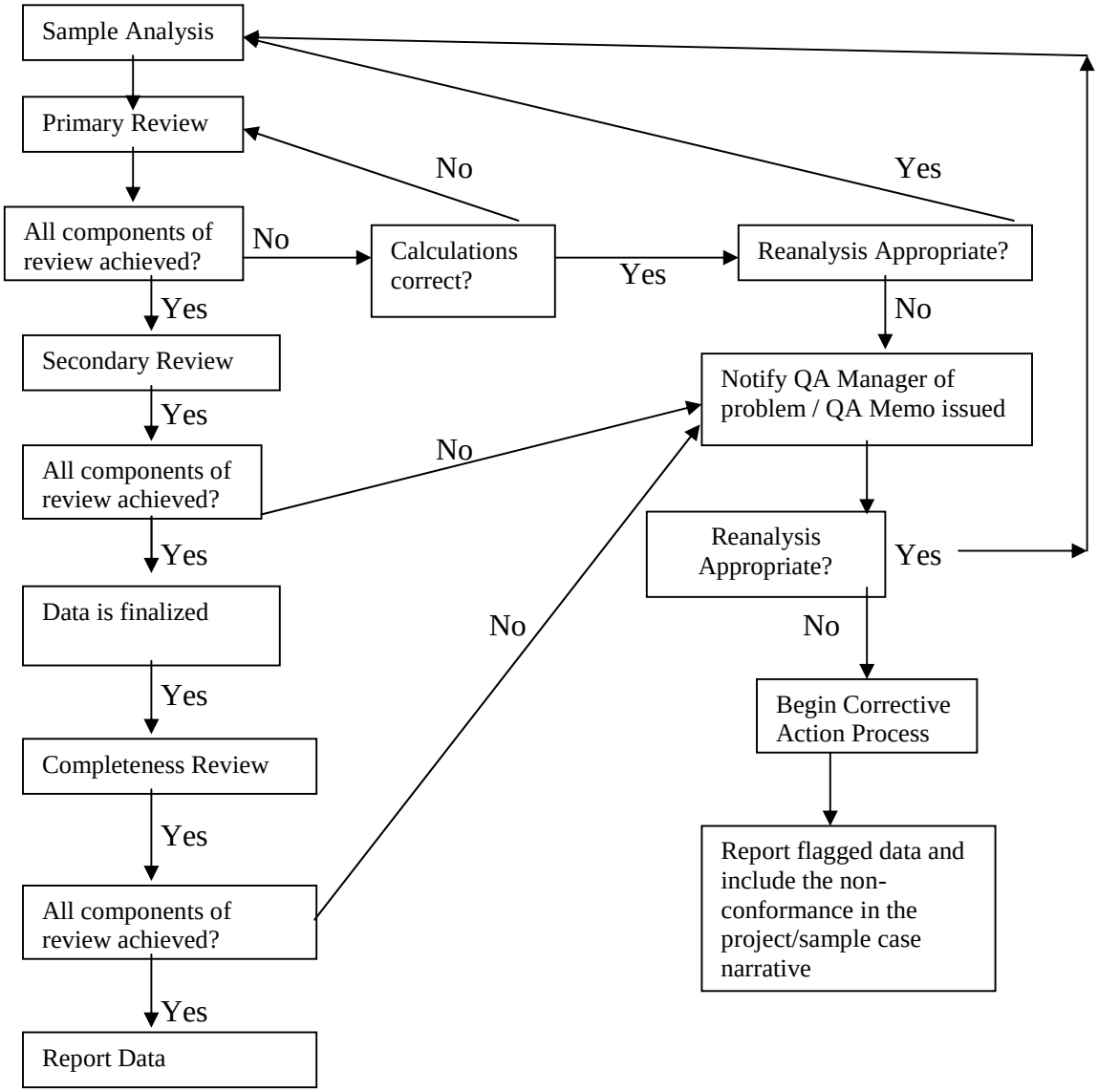
Figure 1: STL Westfield Decision Processes, Procedures and Responsibility for Initiation of Corrective Action



Specific quality control procedures are designed to help analysts detect the need for corrective action. Often an analyst's experience will be most valuable in identifying suspicious data or malfunctioning equipment; and an immediate corrective action may then be taken. The actions should be noted in laboratory notebooks but no other formal documentation is required unless further corrective action is necessary.

The need for long-term action may be identified by standard QC procedures, performance or system audits. Any quality problem, which cannot be solved by

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immediate corrective action, falls into this long-term category. STL Westfield uses a system to insure that the condition is reported to a person who is part of the closed-loop action and follow up plan (figure 1)

The essential steps in the closed –loop corrective action system are:

- The problem will be identified
- Responsibility for investigating the problem will be assigned.
- The cause of the problem will be investigated and determined.
- A corrective action to eliminate the problem will be determined
- Responsibility for implementing the corrective action will be assigned
- The effectiveness of the corrective action will be established and corrective action implemented
- The fact that the corrective action has eliminated the problem will be verified
- The complete process of establishing and implementing corrective action will be documented.

This process of corrective action will be used to make all corrections deemed necessary by the STL Westfield Project Manager or QA/QC Department.

4.12 Preventative Action

Preventative action is defined as noting and correcting a problem before it happens, because of a weakness in a system, method, or procedure. Preventative action includes analysis of the quality system to detect, analyze, and eliminate potential causes of non-conformances. When potential problems are identified, preventative action is initiated to effectively address the problem to eliminate or reduce the risk identified. The preventative action process takes the same format as the corrective action process.

5.0 Data Generation and Validation

5.1 Data Reduction

Analytical results will be reduced to the concentrations units specified in the analytical procedures. All calculations and data will be independently checked by senior laboratory staff upon secondary review of the data.

5.2 Data Validation

Data validation is the process by which analytical data are evaluated and accepted or rejected based on a set of criteria. STL Westfield personnel use the following criteria in the validation of laboratory data:

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- Use of published or approved analytical procedures
- Use of properly operating and calibrated instrumentation
- Precision and accuracy achieved comparable to that achieved in similar analytical programs
- Precision, accuracy and blank contamination meeting the analysis specified criteria as and/or the criteria found in the applicable method.
- Completeness of data set.

All data will be validated by laboratory supervisors prior to being released for reporting purposes to the STL Westfield Laboratory Director. The persons validating the data will have sufficient knowledge of the technical work to identify questionable values. All analyses requiring STL Westfield protocols will be validated in accordance with the requirements of those protocols.

5.3 Data Reporting and Authorization Procedures

Key personnel who will handle data gathering and evaluation are shown in the STL Westfield Organizational Chart. STL Westfield uses a computerized sample tracking for routine tracking and reporting of analysis data.

Special Note: According to 310 CMR, section 42.13(4), drinking water exceedances shall be reported to the client within 24 hours of obtaining validated data.

5.4 Data Review

Table 5.4.1 Primary Review

The primary review is often referred to as a “bench-level” review. In most cases, the analyst who generates the data (i.e. logs in, prepares and/or runs the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed. If directions to the analyst are not clear, the analyst must go to the Supervisor, QA Manager, or Project Manager, who must clarify the instructions.

Once an analysis is complete, the primary reviewer ensures that:

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- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- Internal COC procedures (when required) have been followed.

Any anomalous results and/or non-conformances noted during the Primary Review are communicated to the Supervisor and/or the QA Manager for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification.

5.4.2 Secondary Review

The secondary review is a technical review of a data set and is completed by the section supervisor. The secondary review is documented and the secondary reviewer is identified. The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs

If problems are found during the secondary review, the reviewer must work with the appropriate personnel to resolve them and notify the QA Manager.

5.4.3 Completeness Review

The completeness review is performed by Department Group Leaders and personnel from the QA/QC/Report Production Department. The completeness review includes the generation of a project narrative and/or cover letter that outlines anomalous data and non-compliances. The completeness review addresses the following items:

- Accuracy of Final Client Reporting Forms

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- Completeness
- Special Requirements/Instructions Fulfilled
- Is the project report complete?
- Correct assembly of the final report.
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?
- Have QC outages and/or non-conformances been noted and explained in the case narrative.

If problems are found during the completeness review, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the completeness reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures. This is accomplished by submitting a corrective action form to the appropriate personnel.

6.0 QA Program

6.1 Levels of QC Efforts

Every attempt will be made to have all data generated be valid data. The precision of laboratory analysis will be evaluated using sample duplicates and matrix spike duplicates. Analytical accuracy will be monitored using recovery of analytes from system monitoring compounds; matrix spikes, blank spikes, and reference check standards. These quality control measures and frequencies are summarized in Section 7.2. These QA efforts will assist in determining the reliability of the analytical data.

6.2 Accuracy and Precision

Accuracy is a measure of the degree of agreement between the analyzed value and the true or accepted reference value where it is known. Accuracy is usually expressed as a percent recovery. Precision is a measure of the mutual agreement among individual measurements of the sample parameter under similar conditions, usually expressed as a relative percent difference or as standard deviation. Accuracy and precision in the laboratory are assessed by the regular analysis of known standards and duplicate samples.

6.3 Completeness

Completeness is a measure of the amount of valid data obtained from the analytical measurement system, expressed as a percentage of the number of

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valid measurements that should have been or were planned to be collected. STL Westfield will make every attempt to generate valid data from all samples received. However, realistically, some samples may be lost in laboratory accidents or some results may be deemed questionable based on internal QC procedures. Due to the variable nature of the completeness value, the objective will be to have data completeness for all samples received for analysis as high as possible to meet completeness objectives as described by the client.

6.4 Representativeness

Representativeness is a measure of how closely the measured results reflect the actual concentration or distribution of the chemical compounds in the sample. Sampling may be performed by the client, a third party or STL field services personnel. Sample handling protocols (e.g., storage, preservation and transportation) have been developed to preserve the representativeness of the collected samples. Proper documentation will establish that protocols have been followed and that sample identification and integrity have been assured.

6.5 Comparability

Comparability is a QA objective wherein all sample data is comparable with other representative measurements made by STL Westfield or another organization. STL Westfield will achieve comparability by operating within the instrument linear range and by strict adherence to analytical protocols. The use of published analytical methods, standard reporting units and thorough documentation will ensure meeting this objective.

7.0 Quality Control

7.1 Internal Quality Control

Quality control is the routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. Quality Control checks are the application of STL Westfield's Quality Control program for laboratory analysis in order to ensure the generation of valid analytical results on project samples. These checks are performed by project participants throughout the program, under the guidance of the Quality Assurance Manager.

Special Microbiology Requirements:

The laboratory also monitors its reagent water by performing daily conductivity checks as well as a series of monthly and annual monitoring tests (HPC, metals, TRC, pH, et al) as outlined in Table 9020 I of Standard Methods.

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The laboratory verifies the operation of the autoclave weekly using suspensions. Autoclave cycles must be completed within 45 minutes a 15-minute sterilization time is performed.

As specified in Standard Methods 9030B, when weighting media, the analyst will measure the balance sensitivity by performing the deflection test (0.1g @150g).

7.2 Quality Control Samples

STL Westfield makes use of a number of different types of QC samples to document the validity of the generated data. The following types of QC samples are used routinely:

A. Blank Samples – Blanks are used to assess contamination introduced in transit, storage or in the laboratory.
   - Preparation Blanks – For inorganic analyses, these ASTM Type II reagent water blanks are prepared using the same reagents and analytical procedures as the samples, in order to assess possible laboratory contamination. For inorganic analyses, at least one sample in each preparation batch of 20 or fewer samples is prepared and analyzed for each analyte of interest, in order to assess possible laboratory contamination.
   - Laboratory Method Blanks – For organic analyses, theses blanks are “clean” samples, prepared in the laboratory to include surrogates, and analyzed according to a prescribed method in order to assess possible laboratory contamination.
   - Laboratory Holding Blank – For organic analyses, these blanks are placed in cold storage with the volatile organic samples during the holding time to assess contamination which may be introduced in storage.
   - Calibration Blanks – For all analyses, these blanks are used in instrument calibration and contain all the reagents used in preparing instrument calibration standards except the parameters of interest.

B. Initial and Continuing Calibration Verification – Verification samples are analyzed during each analysis run to assure calibration accuracy for each analyte. For inorganic analysis, theses are prepared from a source other than that used for calibration.

C. System Monitoring compound – For organic analyses, all samples are spiked with surrogate compounds prior to sample preparation in order to assess the behavior of actual components in individual samples during the entire preparative and analysis scheme. Surrogate standard compounds are chemically similar to compounds of interest (target compounds

D. Matrix Spikes/Analytical Spikes – For all analyses at frequencies particular to each method, spiking solutions are added to samples in order to evaluate any

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matrix effects of the sample on the analytical method. Matrix spikes and analytical spikes are performed using actual elements of interest or target compounds.

E. Duplicate Samples – For all analyses, a second aliquot of a sample carried through all sample preparation procedures to verify the precision of the analytical method. At least one sample in each analysis batch of 20 or fewer samples is analyzed in duplicate. Duplicates shall be rotated among client samples so that various matrix problems may be noted and addressed. A duplicate sample will be chosen from among the batch of samples as the sample that most closely represents the physical characteristics of the majority of samples within the batch.

F. Laboratory Control Samples – For inorganic analyses, at least one sample in each preparation batch of 20 or fewer samples is prepared and analyzed for each analyte of interest, in order to verify the preparation and analytical methods.

G. Blank Spikes – For organic analyses, reagent water is spiked with all the target analytes.

Reagents used in the laboratory are normally of analytical reagent grade or higher purity. Each lot of acid or solvent received is checked for acceptability prior to lab use. All reagents are labeled with the date received and date opened. The quality of the laboratory ASTM Class II reagent water is continuously monitored through the use of an in-line conductivity meter. The ASTM Class II reagent water used at STL Westfield is run through activated carbon, a cation resin, an anion resin, a mixed bed resin followed by a final mixed bed resin and is used for inorganic analyses. The organic free water is processed through a four-filtration step, including carbon filtering. The ASTM Class II reagent water and organic free water is verified daily by the analysis of the prep blank water for inorganics and by the analysis of the method blank for volatile organics.

7.3 Control Charts

In accordance with 310 CMR 42.08 (5) (b) 1b STL Westfield maintains quality control charts for each of the disciplines and categories in which the laboratory is certified by the Massachusetts Department of Environmental Protection, Office of Laboratory Certification. Control charts are plotted, following analysis, by means of the LIMS. All QC entered into the LIMS has the availability to be plotted. Each analytical department/ analyst has access the control chart program.

7.4 Internal Quality Assurance

To monitor quality, STL Westfield's QA/QC Department conducts internal Quality Assurance audits including:

A. QC Blind Samples- STL Westfield annually participates in the Performance Evaluation Programs conducted by a NIST approved provider and STL-Corporate QA. This program allows STL Westfield to monitor overall

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laboratory performance using a comprehensive set of double blind check samples that are received as real-time samples. The program evaluates program management services as well as analytical accuracy, and job report performance. STL-Westfield receives a detailed report indicating an overall laboratory quality performance grade, along with any corrective action measures that need to be addressed.

B. Internal Data Audit – On an ongoing basis a representative set of data is chosen randomly from work completed the previous month for one or more of STL Westfield commercial clients. This data review includes each laboratory section.

C. Internal Laboratory Audits – The QA Manager or corporate designee will perform laboratory audits on an annual basis. This involves evaluation of:
   - sample storage
   - chain of custody
   - instrument maintenance
   - documentation, data audits
   - precision
   - accuracy

In addition the QA/QC manager will meet frequently with the project managers and laboratory supervisors to review QA/QC data summaries and other pertinent information.

7.5 System and Performance Audits

7.5.1 System Audits

A system audit is an evaluation of the various components of a laboratory's measurements system to assess proper selection and use. This audit consists of an on-site review of a laboratory's quality assurance system and physical facilities for sampling, calibration and measurements. System audits are performed on a regular basis by the various regulatory agencies and annually by the STL Corporate QA. The audit frequently includes most or all of the components listed below:

- Personnel, facilities and equipment
- Chain - of - custody procedures
- Instrument calibration and maintenance
- Standards preparation and verification
- Analytical procedures
- Quality control procedures
- Data handling procedures
- Documentation control procedures

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- Sample management Procedures
- Project Management
- Report Generation and Review

7.5.2 Performance Audits

Performance audits provide a systematic check of laboratory operations and measurement systems by comparing independently obtained data with routinely obtained data. To fulfill the PT requirements for MA DEP certification and NELAC accreditation, STL Westfield routinely participates in laboratory performance evaluations received from the NYSDOH NELAP as part of the Potable and Non-Potable Water Chemistry Proficiency Programs. STL Westfield also use PT samples from other NIST approved PT providers for those analytes not included in the NY DOH PT samples, or as needed for corrective action to a failed proficiency. STL Westfield also analyzes proficiency samples from ERA to maintain certification for microbiology. A schedule for STL Westfield participation in these performance evaluations is detailed in Table 5.

Table 4 Laboratory Performance Evaluation Schedule (1 year)

Type	1st Quarter	2nd Quarter	3rd Quarter	4th Quarter
Potable Water		X		X
Non-Potable Solid & Hazardous Waste Chemistry	X		X	
LabProficiency Microbiology PT		X		X

7.6 Detection Limits

Definitions of Reporting Limits:

7.6.1 Instrument Detection Limit

The Instrument Detection Limit (IDL) is the level at which the instrument can reliably detect an analyte response. Method-specific sample preparation steps are not considered in the IDL calculation. IDL's are determined, as required, by specific client QAPP requirements.

Inorganic IDL Determination

Inorganic IDL's are determined by multiplying by the Student t-Test the appropriate value with n-1 degrees of freedom and the standard deviation

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obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x estimated IDL on three non-consecutive days with seven consecutive measurements per day.

Organic IDL Determination
Organic IDL's are determined by multiplying by the t-Test Student t-Test the appropriate value with n-1 degrees of freedom and the standard deviation obtained for three to seven replicate analyses of a standard solution (each analyte in reagent water).

The IDL is calculated by multiplying the standard deviation by the student t-Test (n-1) value.

No. of Samples	t-statistic
3	6.96
4	4.54
5	3.75
6	3.36
7	3.14

7.6.2 Method Detection Limit
The Method Detection Limit (MDL) is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. A MDL utilizes all preparatory steps in the final detection.

To obtain an MDL:

1. At least seven method blank samples are spiked at a concentration that is two to five times the IDL, or estimated MDL
2. The MDL is calculated by multiplying the t-Test value the appropriate value with n-1 degrees of freedom by the standard deviation obtained for three replicate analyses of a standard solution (each analyte in reagent water).

MDL's are determined annually, and when a new or changed instrument is utilized or when an analyst assumes a new method. The QA/QC Department maintains copies of MDL Forms for all tests performed at STL Westfield.

7.6.3 Reporting Limit (RL):
The level to which data is reported for a specific test method and/or sample. The RL is frequently 2 – 5X higher than the MDL but must be minimally at or above the MDL. The generic term reporting limit is equally referred to as the STL Westfield standard reporting limit.

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7.7 Training

STL is committed to furthering the professional and technical development of employees at all levels. Minimum training requirements for STL employees are outlined in Table 6.

Table 5 STL Employee Minimum Training Requirements

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Week 1	All
Basic Analytical Skills	Week 1	Technical
Ethics Training	Week 2*	All
Quality System	Quarter 1	All
Initial Demonstration of Capability (IDC)	Prior to unsupervised method performance	Technical

Note: Initial ethics training is given by HR upon hire and a more comprehensive training should follow within 2 weeks of hire by either the Lab Director, QAM or via the Corporate on-line ethics presentation.

- Technical training is accomplished within each laboratory by management (section group leaders) to ensure method comprehension. All new personnel are required to demonstrate competency in performing a particular method first by reading and signing off on the most recent SOP and by successfully completing an Initial Demonstration of Capability (IDOC) before conducting analysis independently on client samples.

  IDOCs and CDOCs are performed by analysis of four replicate QC check samples. Results of 4 successive LCS analyses can be used to fulfill the IDOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the data quality objectives of the specific test method or project data quality objectives. An IDOC Certification Statement is recorded and maintained in the employee's training or personnel file.

  Prior to an analyst assuming responsibility for an analysis, an Initial Demonstration of Capability (IDOC) must be performed and approved by the QA Supervisor.

  - The QA/QC Department will maintain a file of all IDOC's.
  - In addition to the IDOC, the analyst is also expected to read and be familiar with the analytical method and Laboratory SOP.
  - The Section Supervisor is responsible for the proper training of all analysts and technicians.

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An example of the IDOC form can be found in appendix 1.

7.8 Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL has established an Ethics Agreement. Each employee signs the Ethics Agreement, signifying agreed compliance with its stated purpose. STL-Westfield has an initial Ethics Training Seminar that is given to new employees upon the start of work (first day) by human resources in the form of a CD or movie. The initial training is followed up with a more comprehensive power point presentation within 2 weeks of hire by the Laboratory Director or Quality Assurance Manager or review of the Corporate Ethics Training Presentation via the STL intranet. The comprehensive training has a quiz associated with it and personnel are required to sign off the Employee Ethics Statement and the Confidentiality and Proprietary Information Agreement (Corp. Policy No: P-L-006 Rev: 4.2). Refresher training is given on a yearly basis to all employees by the Laboratory Director and/or the Quality Assurance Manager.

7.9 Management Reviews

7.9.1 QA Reports to Management

A monthly QA report is prepared by QA Manager and forwarded to the Laboratory Director, the General Manager, and the Corporate QA Manager. The reports include statistical results that are used to assess the effectiveness of the Quality System. The format of the monthly report can be found in Appendix 1.

A Corporate QA Monthly Report containing a compilation of the Facility QA reports statistics, information on progress of the Corporate QA program, and a narrative outlining significant occurrences and/or concerns is prepared by the Corporate QA Manager and forwarded to the Corporate Operating Officer.

7.9.2 Management Systems Review

A management systems review of each facility is performed annually by the Corporate QA Manager or his/her designee. The management systems review is focused on auditing compliance with the Quality System as defined in the QMP, compliance with regulatory criteria, and review of management and customer service practices. Specific issues identified in the monthly QA reports, such as outstanding corrective action issues, are included in the management systems review.

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A report on the management systems review is issued by the Corporate QA Manager within 30 calendar days of the audit. The audit is addressed to the facility Laboratory Director**,** QA Manager, GM, and COO.

Written responses are required within 30 calendar days of report issue. The response follows the format of the report, and corrective actions and time frames for their implementation are included for each deficiency. The response is directed to all individuals copied on the audit report. Where a corrective action requires longer than 30 days to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the Corporate QA Manager in the agreed upon time frame.

8.0 SOPs

Each STL facility maintains an SOP Index for all standard, non-standard, and laboratory developed methods. SOPs are also maintained for describing processes that are not related to a specific method. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a specific test method.

Method SOPs contain the following information:

Title Page with Document Name, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement. Certification of Test Method

Identification of Test Methods (Title) and Revision number

1. Applicable Matrix
2. Method Detection Limit
3. Scope and Application, including test analytes
4. Summary of the Test Method
5. Definitions
6. Interferences
7. Safety
8. Equipment, Supplies and Apparatus
9. Reagents and Standards
10. Sample Collection, Preservation, Shipment and Storage
11. Quality control
12. Calibration and Standardization
13. Procedure
14. Calculations
15. Method Performance
16. Pollution Prevention
17. Data Assessment and Acceptance Criteria for Quality Control Measures
18. Corrective Actions for Out-of-Control Data

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19. Contingencies for Handling Out-of-Control or Unacceptable Data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

8.1 Process SOPs: contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement.

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Safety
6. Procedure
7. References
8. Tables, Diagrams, and Flowcharts

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, and maintenance of an SOP index. SOPs, at a minimum, undergo review every 24 months. Where an SOP is based on a published method, the laboratory maintains a copy of the reference method.

8.1.1 Work Instructions- Can be used as an addendum to an SOP. It will have its own document control number and be updated based on that number not the SOP number. (Should be enclosed or laminated and hung on a wall or in a book.

8.1.1.1 Flow Chart, Step by step instructions…

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SOP Appendix
In some cases, a standard laboratory procedure is modified slightly for a specific client or project at the client or regulatory agency's request. In these cases, an Appendix to the SOP may be attached that indicates the modifications to the SOP that are specific to that project.

8.2 Laboratory Developed Methods

Laboratory developed methods are validated and documented according to the procedure described in section 8.4.

8.3 Non-standard Methods

Non-standard methods are validated and documented according to the procedure described in section 8.4.

8.4 Method Validation

Before analyzing samples by a particular method, the method is validated. Validation of the method is required for standard methods, non-standard methods, and laboratory developed methods. While method validation can take various courses, the following activities are generally required as part of method validation. Method validation records are designated QC records and are archived accordingly.

8.4.1 Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

8.4.2 Determination of Method Sensitivity
Method sensitivity is determined using detection limit studies. Method detection limit studies are performed using the criteria in 40 CFR Part 136 Appendix B. Instrument

detection limits are performed where required by specific data quality objectives or regulation.

8.4.3 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

8.4.4 Determination of Range

Where appropriate, a determination of the applicable range of the method is performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

8.4.5 Demonstration of Capability

All personnel are required to perform an initial demonstration of Capabilities (IDOC) proficiency on the instrument/method they will be using for analysis prior to testing samples. An IDOC is performed using at least four aliquiots prepared and analyzed according to the test method either concurrently or over a period of days. If a concentration is not specified the 1-4 times the quanitation should be used. In some cases a quality control sample would be obtained from an outside source.

On-going proficiency must be demonstrated annually. The continued demonstration of method performance is generally accomplished by batch specific QC samples such as Laboratory Control Samples and Method Blanks.

Toxicity analysis, the laboratory must obtain consistent results with standard reference toxicants (SRT). An initial DOC shall consist of five or more acceptable tests for each test method, species and endpoint with different batches of organisms. Appropriate negative controls are tested as specified in the appropriate method. (Initial DOC is established by maintaining control charts which record specific endpoints such as but not limited to, LC50, NOEC.

On going performance is demonstrated by routine SRT testing as specified in the method.

See Appendix 1 for DOC form.

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8.4.6 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, a SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

8.4.7 Departure from a documented SOP, Test Method or Policy

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure shall be documented and be authorized by the Laboratory Director, QA Manager and Department Supervisor. The client should be notified before any departure is permitted. Any such occurrence is documented in the cover letter and/or project case narrative.

8.5 Measurement Traceability

8.5.1 General

Traceability of measurements is assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that is peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard is subject to ongoing certifications of accuracy.

At a minimum, these include procedures for checking specifications for balances, thermometers, temperature, ASTM Type II reagent water systems, automatic pipettes and other volumetric measuring devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balances are calibrated on each day of use. All Alcohol and mercury thermometers are calibrated annually against a traceable reference thermometer. All digital thermometers are calibrated quarterly against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

Laboratory ASTM Type II reagent water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use.

Laboratory SOPs specify the required level of accuracy in volumetric glassware. In all cases, volumetric glassware meets the requirements specified in the published test method.

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8.5.2 Reference Standards

The receipt of all reference standards is documented. References standards are labeled with a unique Standard Identification Number, date received, and the expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All standards should be purchased with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The documentation of standard purity is archived, and references the Standard Identification Number.

All efforts are made to purchase standards that are $\geq$ 97.0% purity. If this is not possible, the weight of the standard is corrected for the purity when performing calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or Laboratory Control Sample (LCS) is used as the second source confirmation.

8.5.3 Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specific in method SOPs. Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the date the reagent was opened are documented.

9.0 Project Reports

9.1 General

The criteria described in Section 9.1.1 apply to all Project Reports that are generated under NELAC requirements.

9.1.1 Project Report Content

- Title
- Laboratory name, address, telephone number, contact person
- Unique Laboratory Project Number
- Total Number of Pages (report must be paginated)

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- Name and address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Method

9.1.2 The following are required where applicable to specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time $\leq$ 48 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging where results are reported below the quantitation limits.

9.1.3 Project Narrative

A Project Narrative is included at the client request or when required by the regulatory program and at a minimum includes an explanation of any and all of the following occurrences:

- Sample receipt description
- Non-conformances or Method Deviations
- “Compromised” sample receipt information
- QC criteria failures
- Subcontracted sample information

9.2 Project Release

The Laboratory Director or his/her designee authorizes the release of the project report with a final report cover signature. The designee includes the QA Manager, Reports Supervisor, Toxicity Technical Director and/or Reports Specialist and Project Managers.

Where amendments to project reports are required after issue, these shall be in the form of a separate document and/or electronic data deliverable. The revised report is clearly identified as such “Revised” with the date of revision and the signature of the Laboratory Director or his/her designee. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating what part of the original report for the project was revised. The original version of the project report must be kept intact and the revisions and cover letter included in the project files.

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The laboratory will contact a client within one business day of determining that a result has exceeded a specified regulatory limit or if there is doubt of the validity of a data point after an internal audit of said data point.

9.3 Subcontractor Test Results

Subcontracted data is clearly identified as such, and the name and location for the laboratory performing the test is included in the project report. Test results from more than one STL facility are clearly identified with the name and location of the STL facility that performed the testing and address for that facility.

9.4 Electronic Data Deliverables

Electronic Data Deliverables (EDDs) are routinely offered as part of STL's services. STL offers a variety of EDD formats including Excel spreadsheets, GIS-Key, Equis and custom formats.

EDD specifications are submitted to the corporate EDD department for review. Once the facility has committed to providing the diskettes in a specific format, the coding of the format is performed.

EDDs are subject to a secondary review to ensure their accuracy and completeness.

9.5 Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. More information on the range of project reports available can be obtained by contacting any STL facility. Regardless of the level of reporting, all projects undergo the same levels of review.

10.0 Analytical Methodology

Calibration Procedures and Frequency
Instrument or method calibration is performed in accordance with the specific analytical methods and as outlined below. Quality control measures and frequency are summarized in Section 8.1

10.1 Volatile Organics - GC/MS

A. GC/MS instruments are tuned to meet required mass spectral abundance criteria prior to both initial and continuing calibrations. This is accomplished via the analysis of 4-bromofluorobenzene (BFB).

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B. Initial calibration of GC/MS volatiles consists of at least 5 calibration standards, containing each target analyte, plus surrogate(s) and internal standard compounds. Calibration standards must include concentrations at the reporting level.
The validity of the initial calibration is verified by:
- % Relative Standard Deviation (RSD) of the relative response factor (RRF) for target compound or correlation coefficient from linear regression analysis.
- Minimum RRFs for each compound.
- Assessment of the relative retention time (RRT) shift for each compound between each standard run.
- Analysis of a standard obtained from a second source

C. Continuing Calibration Verification is performed when the Initial Calibration Verification is not performed on the day of analysis. The continuing calibration standard must be preceded by a successful instrument tune. The validity of the continuing calibration is verified by:
- Assessment of % difference (%D) of the RRFs versus the initial calibration average RRF for each compound.
- Minimum RRFs for each compound.
- RRT shift for each compound between successive calibration runs.
- The continuing calibration verification checks must include concentrations at the mid point concentration of the initial calibration.

Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the QAQC department.

D. Standards Preparation Procedures

Calibration Standards

- Purchase commercially available certified stock solutions.
- Prepare working standards by dilution of the stock standards.
- Verify the working standards by analysis of a calibration check standard prepared independently from standards.

<u>10.2 Volatile Organics- GC</u>

A. Initial calibration for GC Volatiles consist of at least 5 calibration standards containing each target analyte plus surrogate compounds. Calibration standards must include concentrations at or below the reporting level, if these limits/levels are known. The validity of the initial calibration is verified by:

- % Relative Standard Deviation (RSD) of the calibration factor for target compounds.

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- Assessment of the relative retention time (RRT) shift for each compound between each standard run.
- Analysis of a standard obtained from a second source

B. Continuing calibration is performed when the initial calibration is not performed on the day of analysis. A continuing calibration check must be performed at the beginning and end of each analytical batch. The validity of the continuing calibration is verified by:

- Assessment of % difference (%D) of the calibration factor versus the initial calibration average calibration factor for each compound
- RRT shift for each compound between successive calibration runs.
- The continuing calibration verification checks must include concentrations at the mid point concentration of the initial calibration.

C. Standard Preparation Procedures
Calibration Standards

- Purchase commercially available certified stock solutions.
- Prepare working standards by dilution of the stock standards.
- Verify the working standards by analysis of a calibration check standard prepared independently from standards.

Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the analytical department and/or QA...

<u>10.3 Semi-volatile Organics – GC/MS</u>

A. GC/MS instruments are tuned to meet required mass spectral abundance criteria prior to both initial and continuing calibrations. This is accomplished via the analysis of decafluorotriphenylphosphine (DFTPP).

B. Initial calibration of GC/MS semi-volatiles consists of at least 5 calibration standards containing each target analyte plus surrogate and internal standard compounds. Calibration standards must include concentrations at or below the reporting level, if these limits/levels are known. The validity of the initial calibration is verified by:

- % Relative standard deviation (RSD) of the relative response factor (RRF) for target compound or correlation coefficient from linear regression analysis.
- Minimum RRFs for each compound.
- Assessment of the relative retention time (RRT) shift for each compound between each standard run.

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- Analysis of a standard obtained from a second source.

C. Continuing Calibration Verification (CCV) is performed when the initial calibration is not performed on the day of analysis. The continuing calibration standard must be preceded by a successful instrument tune. The validity of the continuing calibration is verified by:

- Assessment of % difference (%D) of the RRFs versus the initial calibration average RRF for each compound.
- Minimum RRFs for each compound.
- RRT shift for each compound between successive calibration runs.

D. Standards Preparation Procedures

Calibration Standards

- Purchase commercially available certified stock solutions.
- Prepare working standards by dilution of the stock standards.

Verify the working standards by analysis of a calibration check standard prepared independently from standards.

Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the QAQC department.

<u>10.4 PCB Organics - GC/ECD</u>

A. Initial calibration for PCB compounds consists of at least 5 calibration standards, each containing arochlor 1016 and 1260 and 1 calibration standard of arochlors 1221, 1232, 1242, 1248, and 1254. Additional standards are required as part of the initial calibration sequence, including resolution check, instrument blank and initial calibration verification standard, (ICV). Validity of the initial calibration is evaluated by:

- RPD of true vs. calculated values of all single-component compounds and surrogates.
- % RSD of calibration factors for arochlors 1016 and 1260.
- The continuing calibration must be performed at the beginning and end of the analytical sequence.

The above criteria apply to both analytical columns.

B. Standards Preparation Procedures

Calibration Standards

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- Purchase commercially available certified stock solutions.
- Prepare working standards by dilution of stock standards.
- Verify the working standards by analysis of a calibration check standard prepared independently from standards.
-

Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the QAQC department.

10.5 Metals - ICP

A. Initial calibration for ICP metals consists of a method blank and 1 calibration standard.

B. The validity of the initial calibration is verified by the analysis of an independently prepared standard immediately after calibration.

B. Results for EPA method 200.7 must be within 95% to 105% (90% to 110% method SW846 6010B) of true value for each metal analyzed for analysis to begin.

C. Continuing calibration is performed by analysis of the Calibration Check Verification (CCV) standard at a frequency of 10% of sample volume, or every 2 hours, whichever is more frequent. The concentration of the CCV is at or near the mid-range of the calibration curve for each metal. The CCV is also analyzed after the last analytical sample. The validity of the calibration and analysis of preceding samples is verified by CCV results within the method requirements.

D. An MDL standard is analyzed with each analytical batch to verify the reporting limit.

E. Standards Preparation Procedures

Calibration Standards

- Prepare calibration standards by dilution of the stock standard.
- The calibration standards are prepared in reagent grade water, with the same acid concentrations as the digested sample.
-

Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the QAQC department.

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10.6 Metals - Mercury

A. Initial calibration for mercury consists of at least a blank and three standards (one standard at the reporting level). Calibration is performed on a daily basis (or once every 24 hours), and each time the instrument (cold-vapor AA) is set up. Standards are prepared in reagent-grade water, with the same acid concentration as the samples. The validity of the initial calibration curve ($r = \geq 0.995$) is verified by analysis of ICV standards immediately after calibration; results must be within 90% to 110% of true value.

B. Continuing calibration is performed by analysis of the CCV standard at a frequency of 10% of sample volume, or every 2 hours, whichever is more frequent. The concentration of the CCV is at or near the midrange of the calibration curve. The CCV is also analyzed after the last analytical sample. The validity of the calibration and analysis of the preceding samples is verified by CCV results between 90% and 110% (80% - 120% method SW846 7470A) of true value.

C. Standards Preparation Procedures
Calibration Standards

-Prepare the calibration standards by dilution of the stock standard at the time of analysis. Calibrate the instrument using a blank and a minimum of three standards.
-Prepare the standards in the same manner as samples using the same combination of reagents at the same concentration as that which the sample will have after preparation.

Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the QAQC department.

10.7 Conventional Wet Chemistry

A. For manual colorimetric analytical procedure an initial five standard calibration curve. Calibration curve ($r = \geq 0.995$) is performed on a daily basis (or once every 24 hours), and each time the instrument is set up. If the concentration of the continuing calibration standard, as calculated from the initial calibration curve, exceeds $\pm$ 10% of the theoretical concentration then that batch of samples must be rerun. If the rerun still exceeds $\pm$ 10%, then the system should be considered out of control and a new initial calibration curve must be generated.

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Note: Nelac requires the concentration of the continuing calibration standard be varied within the established calibration range. To fulfill this requirement, STL-Westfield will perform an annual variation for CCVs per method. The data from these runs will be documented and maintained by the QAQC department.

11.0 Common Preventative Maintenance

STL Westfield employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is preformed as needed and as recommended by the manufacturer. Instrument maintenance may be performed by the analyst, instrument specialist or outside technician. Maintenance logbooks shall be kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded. Notation of the date and maintenance activity is recorded each time service procedures are performed. The return to analytical control following instrument repair is documented in the logbook. Maintenance logbooks are reviewed and audited frequently (monthly) by the department supervisors and annually during the laboratory-wide system audit. Maintenance logbooks are retained as QC records for a period of 10 years after being archive.

Maintenance contracts may be held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory.

11.1 Equipment Verification and Calibration

A. All equipment shall be tested upon receipt to establish its ability to meet the QC guidelines contained in the test method for which the instrument is to be used. This testing shall be documented. Once the instrument is placed into service, ongoing calibration is demonstrated at the appropriate frequency as defined in the test method. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into service, this is documented in the instrument's maintenance logbook.
B. The instrument's logbook shall contain the following information: Identity, Date Installed, Manufacturer's Name, Model number, Serial number, and was as date and type of service maintenance performed.
C. All manufacturer's operations and maintenance manuals are to be kept up to date and accessible for the use of the instrument's operator. Documentation of the instrument's usage is maintained using analytical run and maintenance logbooks.
D. Routine maintenance procedures for laboratory instrumentation are given in Table 8.1. The frequencies of routine maintenance procedures for STL Westfield's field instrumentation are given in Table 8.2. The service intervals listed in Tables 8.1 and 8.2 are as follows: D=daily; W=weekly; M=monthly; Q=quarterly; SA=semi-annually; A=annually; AN=as needed.

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12.0 Analytical Procedures

The analytical procedures to be used in the analysis of samples may utilize one of the following references:

References

1. Methods for Chemical Analysis of Water and Wastewater", EPA-600/4-79-020, March 1983.
2. "Test Methods for Evaluating Soil Waste", USEPA-SW846, 3RD Edition, September 1986 with all current revisions.
3. Atomic Absorption – Furnace Technique, Perkin/Elmer Operating System
4. Federal Register, V. 50, No.3, January 4, 1985.
5. Federal Register, V. 49, No.209, October 26, 1984.
6. "Standard Methods for the Examination of Water and Wastewater", 16th Edition, 1986.
7. "Standard Methods for the Examination of Water and Wastewater", 18th Edition, 1992.
8. "Standard Methods for the Examination of Water and Wastewater", 19th Edition, 1995.
9. "Standard Methods for the Examination of Water and Wastewater", 20th Edition.
10. "Methods for the Determination of Organic Compounds in Drinking Water EPS-600/4-88-039, December 1988.
11. The Analysis of Trihalomethanes in Finished Water by the Purge and Trap Method, EMSL, Cincinnati, Ohio 45268, November 6, 1979.
12. Volatile Aromatic and Unsaturated Organic Compounds in Water by Purge and Trap Gas Chromatography, EMSL, Cincinnati, Ohio 45268, Revision 2.0, (1989).
13. Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series, EMSL, Cincinnati, Ohio 45268, revision 2.0 (1989).
14. Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector, EMSL, Cincinnati, Ohio 45268, Revision 4.0 (1989).
15. "ASTM, Petroleum Products, Lubricants, and Fossil Fuel, Vol. 5.01 D56-D1947, 1990.
16. "Analytical Handbook for the Laboratory of Organic Analytical Chemistry", Wadsworth Center for Laboratories and Research, New York State Department of Health, August 1991.
17. Lachat Quik Chem Methods, 10-107-06-1A, 10-204-00-1-A, 10-107-04-1A,10-210-00-1-A, 10-107-06-2-D, Lachat Instruments, Inc. 6645 West Mill Road Milwaukee, WI 53218-1239, revised 5/20/97, 6/6/96,6/24/97,8/6/96,10/7/97 respectively.
18. Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), Massachusetts Department of Environmental Protection, January 1998.

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19. Method for the Determination of Extractable Hydrocarbons (EH), Massachusetts Department of Environmental Protection, January 1998.
20. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma, Revision 4.4 EMMC Version, EPA 200.7, May 1994.
21. STL Quality Management, Plan M-Q-001, Revision 7, Revision Date: 14 July 2005, Effective Date: 4 November 2005.

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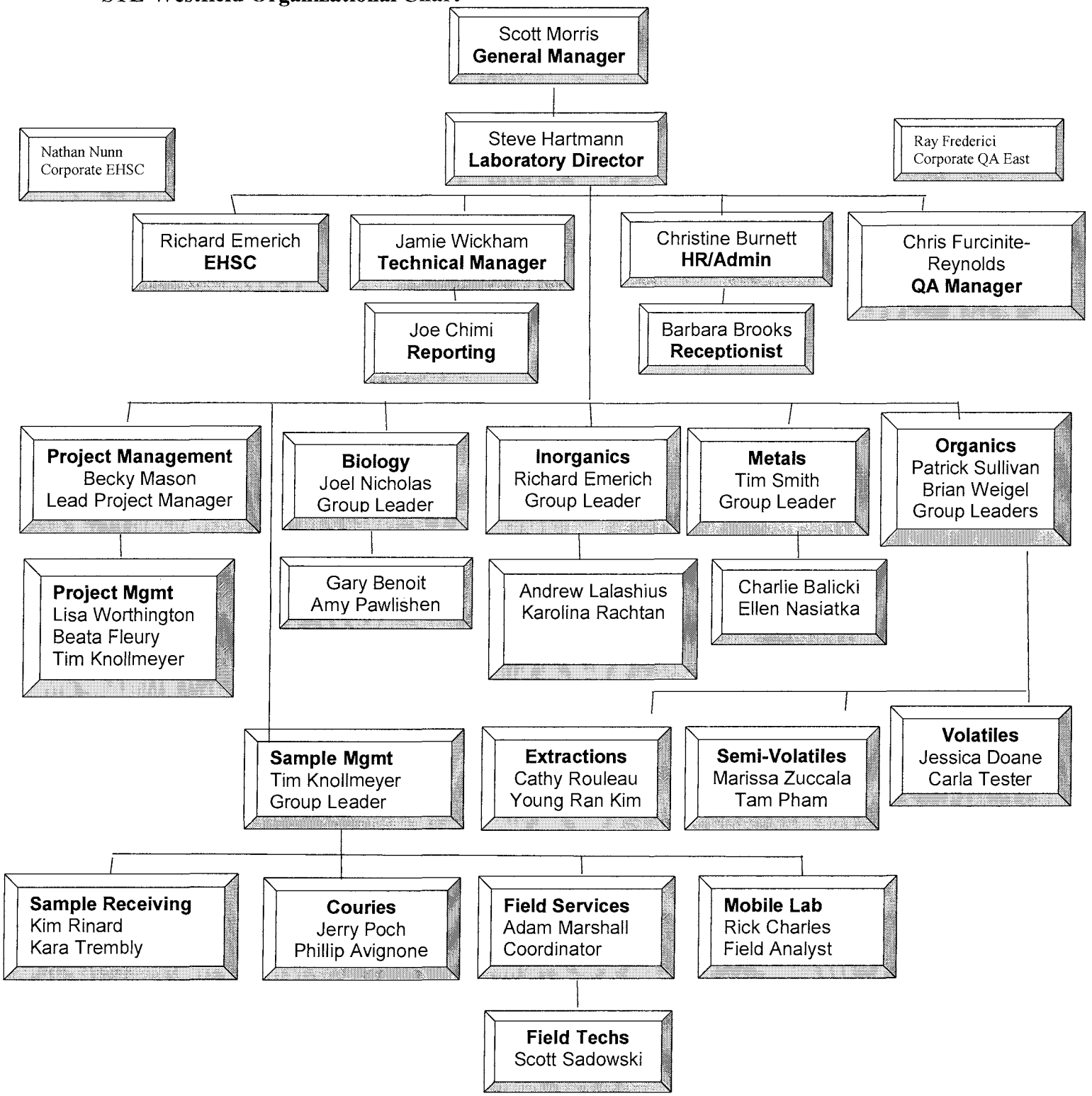
APPENDIX I

ORGANIZATIONAL CHART
EDUCATION and EXPERIENCE
INSTRUMENTATION AND SUPPORTING EQUIPMENT LIST
MAINTENANCE SCHEDULE
CORRECTIVE ACTION FORM
CUSTOMER COMPLIANT FORM
DEMONSTRATION OF CAPABILITIES
CHAIN OF CUSTODY
MONTHLY QA REPORT FORM

Note that all forms and tables are subject to change

STL-Westfield Organizational Chart



Education and Experience

Steven Hartman – B.S., Environmental Science, St. Joseph's College, ME
Responsibilities: Laboratory Director
Experience: 14 years

Christine Furcinite-Reynolds – B.S., Environmental Science, University of Massachusetts, Amherst, MA.
Responsibilities: Quality Assurance Manager
Experience: 20 years

James Wickham – B.S., Environmental Science & Geology, Bemidji State University, MN.
Responsibilities: Technical Manager
Experience: 20 years

Project Management:

Becky Mason – B.S., Natural Resource Economics, University of Massachusetts, Amherst, MA
Responsibilities: Lead Project Manager
Experience: 7 years

Lisa Worthington- B.S., Biology, Western New England College, MA
Responsibilities: Project Manager
Experience: XX years

Tim Knollmeyer – B.S., Political Science, University of Massachusetts, Amherst, MA
Responsibilities: Project Manager /Sample Management group leader
Experience: 15 years

Beata Fleury – B.S., Biology/Environmental Science, University of Massachusetts, Amherst, MA
Responsibilities: Project Manager
Experience: 5 years

Sample Receiving:

Kim Rinard – A.S., Environmental Science Field Tech., Holyoke Community College, Holyoke MA
Responsibilities: Sample-Receiving
Experience: 2 years

Kara Trembly - B.S., Biochemistry and Molecular Biology, University of Massachusetts - Amherst
Responsibilities: Sample-Receiving
Experience: <1 year

Organics:

Pat Sullivan– B.S., Biology, Westfield State College, MA
Responsibilities: Biology/WET Group Leader
Experience: 16 years

Brian Weigel – B.S., Biology/Premed, Grove City College
Responsibilities: Organics Group leader
Experience: 8 years

Carla Tester- B.S., Liberal Arts, Westfield State College, Westfield, MA.
Responsibilities: GC/MS Analyst
Experience: 7 years

Tam Pham – B.A., General Science, Westfield State College, Westfield, MA
Responsibilities: Organic prep technician
Experience: 4 years

Jessica Doane- B.S., Forensic Science, Bay Path College, Longmeadow MA
Responsibilities: GC/MS Volatiles
Experience: 2 years

Marissa Zuccala- B.S., Biology (minor chemistry), Westfield State College
Responsibilities: Organic Prep/Technician
Experience: 1 year

Young Ran Kim-
Responsibilities: Organic Prep
Experience: <1 year

Metals:

Tim Smith – B.S., Environmental Sciences, Johnson State, VT
Responsibilities: Metals Group leader
Experience: 18 years

Charlie Balicki - Westfield State College, MA
Responsibilities: Metals Analyst
Experience: 5 years

Ellen Nasiatka – B.A General Science, Westfield State College, Westfield, MA
Responsibilities: Metals Analyst/Prep technician
Experience: 2 year

Inorganic (Wet) Chemistry:

Richard W. Emerich– B.S., Chemistry Principia College, Elsha, IL
Responsibilities: Wet Chemistry Group Leader
Experience: 23 years

Andrew Lalashius- B.S., Biology, University of Massachusetts
Responsibilities: Wet Chemistry
Experience: 1year

Karolina Rachtan – B.S, Chemistry, USM and an M.S., Chemistry, University of Massachusetts
Responsibilities: Wet Chemistry
Experience: 1 year

Microbiology:

Joel Nicholas – B.S., Biology, University of Massachusetts, Amherst MA
Responsibilities: Microbiology/WET analyst
Experience: 4 year

Gary Benoit – Northampton High School
Responsibilities: Microbiology/WET analyst
Experience: 15 years

Amy Pawlishen- B.S, General Biology, American International College
Responsibilities: Microbiology/WET analyst
Experience: <1 year

Reports:

Joe Chimi – Malden High School
Responsibilities: Report Production, IT System Administrator
Experience: 8 year

Field Services:

Adam Marshall – B.S., Regional and Environmental Planning, Westfield State College, MA
Responsibilities: Field Services Coordinator
Experience: 9 years

Scott Sadowski- B.S., Environmental Science, Environmental Science, University of Massachusetts, Amherst, MA.
Responsibilities: Field Services
Experience: 6 years

Couriers:

Jerry Poch -
Responsibilities: Courier
Experience: 1year

Phillip Avignone-
Responsibilities: Courier
Experience: <1year

Human Resource:

Christine Burnett –
Responsibilities: Human Resource / Accounting
Experience: 15 years

Barbara Brooks-
Responsibilities: Front Office / Receptionist
Experience: 6 year

Years experience measured as of May 2007.

STL Westfield Instrumentation and Supporting Equipment List

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler	Method Performed
ICP	Varian	ICAP	2006	Yes	6010B, 200.7
	Thermo Jarrell Ash S/N 11090	61E Trace	2001	Yes	6010B, 200.7
ICPMS	Perkin Elmer S/N 1449912	6100PE	2004		6020, 200.8
GFAA	Perkin Elmer S/N 133309	5100 PC	1988	Yes	GFAA 200 Series GFAA 7000 Series 3113B
	Perkin Elmer S/N 132466	5100 PC	2003	Yes	GFAA 200 Series GFAA 7000 Series 3113B
Mercury Analyzer	Leeman Labs S/N 2045 112-0064-1	010-00073-1 Automated	2003	Yes	7471A, 7470, 245.1
Biology	(2) Market Forge Autoclave S/N 161883, 169797	STM-E		No	SM 9200s
	Orion pH Meter S/N 41468	420A		No	150.1
	Orion DO Probe	97-08-99		No	SM4500OC
	Fisher Digital Conductivity Meter			No	SM 2510B
	Olympus Microscope S/N 209124	BH2		No	
	Spencer Colony Counter	3330		No	SM 9200s
GC/MS Volatiles	Hewlett-Packard #1 S/N 2618A07889	5890 – GC 5972 – MSD		Yes	8260B, 624, 524-Water
	Agilent #2 S/N US00036050	6890 plus – GC 5973 – MSD		Yes	8260B
	Hewlett-Packard #3 S/N 3336A60155	5890II - GC 5972 – MS ,	2002	Yes	8260B
	Hewlett-Packard #4 S/N 3336A60501	5890II – GC 5972 – MSD		Yes	8260B
GC Volatiles	Hewlett-Packard #1 S/N 3126A36650	5890II - GC		Yes	502, 601/602, 8021
	Hewlett-Packard #2 S/N 3223A42969	5890II - GC	1999	Yes	502, 601/602, 8021

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler	Method Performed
Ion Chromatograph	LACHAT 5000 S/N A50000-403	LACHAT 5000	2001	Yes	300.0
Flow Injection System	LACHAT QUICKCHEM S/N A83000-1121	FIAA	1996	Yes	CN, NH3, NO2, NO3, PHENOLICS, TKN
TOC	LIQUITTOC ELEMENTAR S/N 35.00-5006	LIQUITTOC	2003	Yes	415.1, 9060 5310B
IGNITIBILITY	PENSKY-MARTIN S/N 1454	ASTM D-95		No	1010
TKN Digestion System	Lachat	BD-46	1996	No	LACHAT TKN
UV/VIS	JENWAY SPECTROMETER S/N 3043	6405 UV/VIS	2003	No	7196A, 410.4, UV254, 4500OP/PE, 4500NO, 3500FE, 4500Cr
Autotitrator (pH, Alkalinity, Conductance)	MAN-TECK ION PLUS S/N MSOH3-595	PC – Titrate 1104-00	2004	Yes	9040B, 9045C, 150.1, 2320B, 2510B, 9050A, 120.1
pH Meter	ACCUMET S/N C0007224	20		No	9040B, 9045C, 150.1
Dissolved Oxygen Meter	YSI 5000 S/N 96C4720I	5000		No	405.1
Turbidity	HF Scientific S/N 406184	Micro 100 Turbidimeter		No	180.1
Automated Distillation Apparatus	Westco S/N 1028	483-W001-01 Easy Dist		No	4500-CN-E, 9012A, 335.1, 335.3
COD	HACH S/N 911005946			No	410.4
	HACH S/N 910605021			No	410.4
	HACH S/N 900903265			No	410.4
GC Semi - Volatiles	Instrument B GC 6890 S/N US00027728 MS 5973 S/N US82322366 Tower 7683 US85004015			Yes	8270 Low Level
	Instrument J GC 6890 S/N US00008316 MS 5973 S/N US01140157 Tower 7673 S/N 3114A25556			Yes	625/8270

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler	Method Performed
	Instrument N GC 5890 II S/N 3336A0302 MS 5972 S/N HS07 Tower 61513A S/N US75706257			Yes	625/8270
	Instrument P GC 5890 II S/N 3336A52410 Tower 7673 S/N 3009A20958			Yes	ECD 608/8082/8081
	Instrument H GC 5890 II S/N 3203A40904 Tower 7673 S/N 3304A33716			Yes	ECD 608/8082/8081
	Instrument M GC 6890 S/N US00025858 Tower G7683 S/N (fr) US91907177 (bk) 3120A26315			Yes	ECD 8081/8082
	Instrument C GC 5890 II S/N 3310A49235 Tower 7673 S/N 3432A39688			Yes	FID DRO

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler	Method Performed
	Instrument D GC 5890 II S/N 2921A24544 Tower 7673 S/N (fr) 3531A43500 (bk) 312A26315			Yes	DUAL FID EPH
	Instrument E GC 5890 II S/N 2843AI9944 Tower 7673A S/N (fr) 2704A08I55 (bk) 3304A33720			Yes	FID DAI
	Instrument F GC 5890 II S/N 3235A46075 Tower 7673 S/N (fr) 3120A26315 (bk) 3104A24733			Yes	DUAL FID EPH
	Instrument G GC 5890 II S/N 3033A31429 A/S VARIAN-ARCHON 4552 S/N 12570			Yes	PID/FID VPH
	Instrument L GC 5890 II S/N A/S UP-PRES-200 S/N 95243009			Yes	PID/FID VPH

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP								
Pump Tubing	X							Inspect daily, replace as needed.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.
D-Shaped Mirrors						X		Inspect during annual Tech Support PM
MERCURY ANALYZER AND AUTOSAMPLER								
Pump Tubing	X							Inspect daily, replace as needed.
Standard Cup	X							Inspect daily, replace as needed.
Drying Tube							X	Replace as needed.
Mixing Coil		X						Inspect weekly, clean or replace as needed.
Sample Probe			X					Inspect monthly, clean or replace as needed.
Mercury Lamp							X	Clean or replace.
MARS MICROWAVE								
Pressure Control System	X							Flush.
Cavity and exhaust		X						Clean.
Door			X					Inspect seals and locks.
TURBIDIMETER				X				Quarterly Calibration
	X							Daily calibration check-QCS
CONDUCTIVITY METER							X	Replatinize cell when 1 umho/cm range exceeds 90-100%, and when erratic readings cannot be corrected.
	X							Calibrate with potassium chloride
	X							Instrument surfaces inspected and cleaned
pH METER		X					X	Clean or replace probe as needed
	X							Store electrode in buffer solution
	X							Daily Calibration
ION CHROMATOGRAPH								
Separator Column				X				Clean.
Guard Column				X				Clean.
Pump Pistons						X		Inspect.
Conductivity Cell						X		Inspect
AUTOANALYZER (/LACHAT)								
Pump Tubes	X							Inspect
Pump Tubes							X	Replace as needed or annually
Colorimeter filters		X						Inspect
Flush system								
ANALYTICAL BALANCES	X							Surfaces cleaned and covered
					X			Calibrated and cleaned by Manufacturer or certified company

	X							Checked with class "s" weights
BLOCK DIGESTOR						X		Check calibration.
UV/VIS SPECTROPHOTOMETER						X		Check for wavelength verification.

AUTOCLAVE								
Autoclave operation		X						Spore strips or ampules
Pressure Verification	X							Check and document; replace gauge or seals as needed.
Drain Autoclave	X							Visually inspect for leaks and signs of degradation.
Temperature Verification	X							Check with autoclave thermometer; document; replace thermometer as needed.
Cleaning				X				Wash with soapy water; visually inspect for leaks and degradation.
Times						X		Check with stop-watch; replace as needed.
Seals							X	Visually inspect and replace as needed.
TOC ANALYZER								
Catalyst							X	Inspect and Replace as needed.
Syringe			X					Perform zero point calibration.
GAS CHROMATOGRAPH – SEMIVOLATILES								
Septa							X	Replace as needed.
Column/Injector							X	Change sleeve and cut front of guard column. Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC SEMIVOLATILES								
Column/Injector							X	Change sleeve and cut front of column.
Septum							X	Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Splitless Disc							X	Replace.
Rough Pump							X	Oil changed by HP service.
Mass Spectrometer							X	Clean.

GAS CHROMATOGRAPH – VOLATILES								
Column							X	Replace.
Septum							X	Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH - MASS SPEC VOLATILES								
Column							X	Replace.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Rough Pump							X	Oil change by HP service.
Gas Manifold	X							Inspect daily, change when pressure reads <500 psi.

GAS CHROMATOGRAPH - SCREENING FOR VOLATILES								
Septa							X	Replace as needed.
Column							X	Replace.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
GAS CHROMATOGRAPH – DAI								
Column							X	Replace.
Septa							X	Replace as needed.
Gas Cylinder	X							Inspect daily, change when pressure reads <500 psi.
Hydrocarbon/Moisture Trap							X	Replace.
Filament	X							Checked for proper temperature when analysis required.
Injector Loop	X	X						Change daily for analysis of atmospheric gases. Change weekly for analysis of non-atmospheric gases.
Column							X	Replace.
Mass Spec								
Cryo-column	X							Inspect for breaks.
Septa							X	Replace as needed.
Column							X	Replace.
Rough Pump							X	Oil change by HP service representative.
Mass Spectrometer							X	Clean.
PURGE AND TRAP								
Sorbent Trap							X	Change.
Purge Flow		X						Inspect weekly. Adjust as needed and whenever concentrator maintenance is performed
AUTOSAMPLER - ORGANICS								
Syringe and Tubing							X	Cleaned
Needles and tubing							X	Replaced
Sample lines							X	Checked and rinsed
EXTRACT CONCENTRATOR								
Bath		X						Change water and scrub bath; dust outside.
Temperature Verification		X						Verify bath temperature and adjust.
Sensor Diagnostic Test		X						Check each position and adjust.

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	**Service Interval**							**SERVICE LEVEL**
	D	**W**	**M**	**Q**	**SA**	**A**	**AN**	
TCLP EQUIPMENT								
Semivolatiles/Metals Rotator		X						Check rotation.

{PRIVATE}

FIELD EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE

EQUIPMENT ITEM	Service Interval					SERVICE LEVEL
	D	W	M	Q	A	
CONDUCTANCE METER	X					Inspect daily, replatinize cell as needed.
pH METER	X					Inspect daily prior to sampling, add filling solution as needed.
DISSOLVED OXYGEN METER	X					Inspect probe daily prior to sampling, change as needed.

D = Daily
W = Weekly
M = Monthly
Q = Quarterly
A = Annually
SA = Semi Annual
AN = As Needed

STL

STL-WESTFIELD

CORRECTIVE ACTION FORM

AREA OF APPLICABILITY: Department

1. Person Responsible for Initiating Investigation	Date:
2. Identification of the Problem: .	
3. The cause of the problem has been determined to be:	
4. The corrective action to eliminate the problem has been determined to be:	
5. Person(s) responsible for implementing the corrective action	Date:
6. The effectiveness of the correction action has been established (attach copy if available) and the corrective action has been implemented.	Sign and Date

Section Supervisor:	
Laboratory Director:	Steve Hartman
Employee Name:	
QA Manager:	Christine Furcinite-Reynolds

Client complaint log

compliant log - Windows Picture and Fax Viewer

A12 360-0053

STL Westfield
Client Complaint and Handling
Tracking Form

A: Incomplete data package
B: Bottle shipment problem
C: Technical issue
D: Unexpected Results
E: QC failures
F: Turnaround time
G: Lab Responsiveness
H: Login incorrect
I: EDD issue
J: Other - specify

Complaint Number	CLIENT	Date Complaint Rec'd by LAB	Date LD/QA and PM Supervisor Notified	Date Responded back to client (1 business day)	Date Final Response to client	TYPE of Complaint: Correctable or Non Correctable	Category (see key)	Requires Revision Y/N	PM	Comments
360-0052										
360-0053										
360-0054										
360-0055										
360-0056										
360-0057										
360-0058										
360-0059										
360-0060										
360-0061										

Oct-dec 06 / Jan-Feb 07 / Mar-April 07 / May-June 07 / July-Aug 07 /

Demonstration of Capabilities

Microsoft Excel - 2006 DOC Template

A5 Laboratory Name: STL-Westfield

STL-Westfield Demonstration of Capability

NELAC Certification Statement

Laboratory Name: STL-Westfield
Laboratory Address: 53 Southampton Road, Westfield MA 01085

Analyst Name: Date:

Method: SOP#

Matrix: Water

We the undersigned certify that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification.
3. Copies of the test method and SOP are available for all personnel on site.
4. The data associated with the DOC are true, accurate, complete and self-explanatory.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is available for review by authorized inspectors.

Rich Emerich

Laboratory Supervisor Signature Date

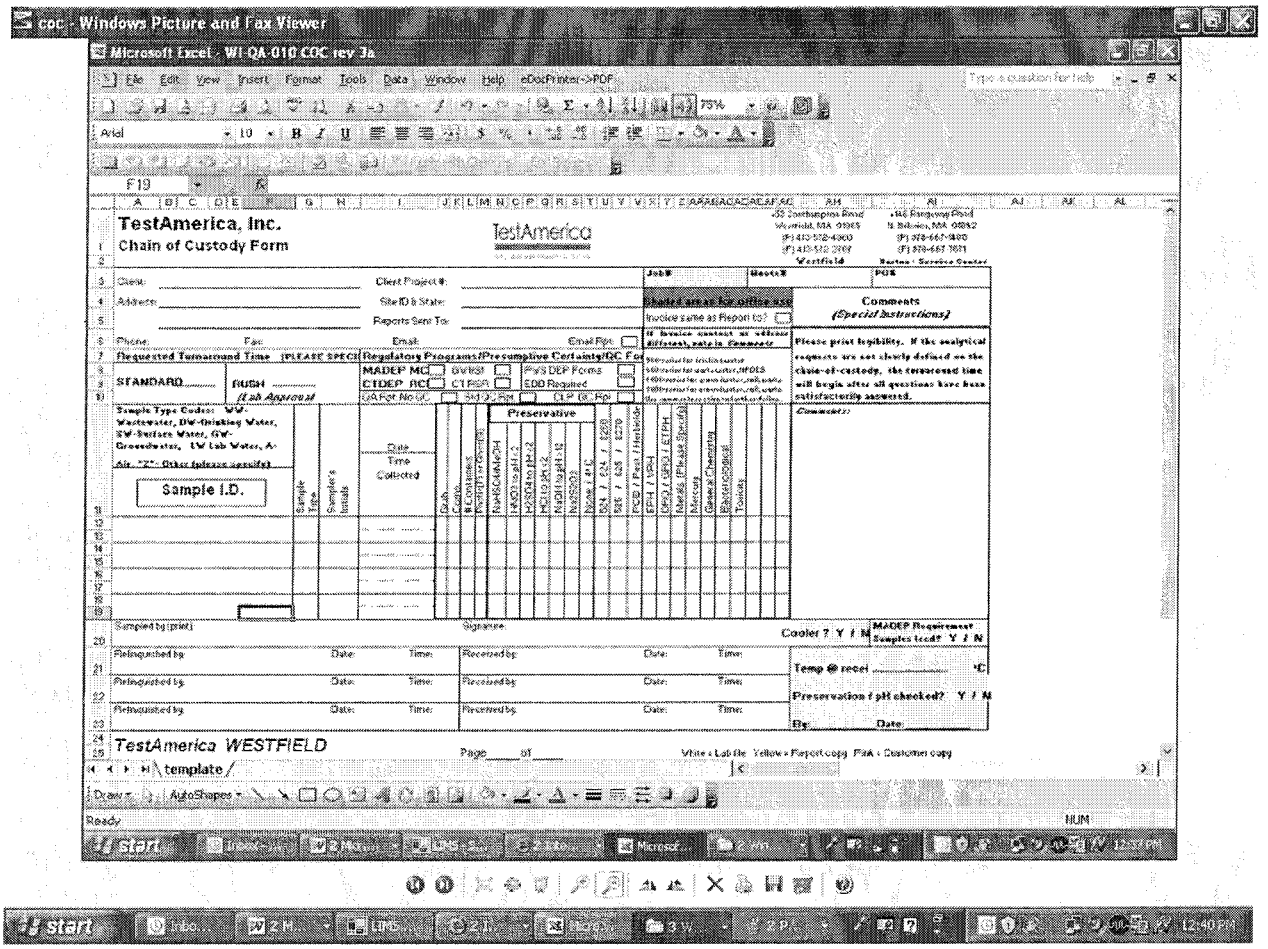
Quality Assurance Signature Date

ANALYTE	CONC.	IDC1	IDC2	IDC3	IDC4	SD	AVG	%REC	%RSD
						#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
						#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
						#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

Sheet1 (2) Sheet1 (3)

Ready NUM

Chain of Custody - Example



TestAmerica, Inc.

Chain of Custody Form

TestAmerica

Client:

Client Project #:

Address:

Site ID & State:

Reports Sent To:

Phone:

Fax:

Email:

Email Rpt:

Job#

Matrix#

PO#

Shaded areas for office use

Invoice same as Report to?

Comments

(Special Instructions)

Requested Turnaround Time (PLEASE SPECI

Regulatory Programs/Presumptive Certainty/QC For

STANDARD

RUSH

(Lab Approval)

MADEP MCP

GW/RSI

PWS DEP Forms

CTDEP RCP

CT PSR

EDD Required

QA Rpt No QC

Std QC Rpt

CLP QC Rpt

Please print legibility. If the analytical requests are not clearly defined on the chain-of-custody, the turnaround time will begin after all questions have been satisfactorily answered.

Comments:

Sample Type Codes: WW-Wastewater, DW-Drinking Water, SW-Surface Water, GW-Groundwater, LW Lab Water, A-Air, "O"-Other (please specify)

Sample I.D.

Sample Time

Sampler's Initials

Date

Time Collected

Preservative

Cooler ? Y / N

MADEP Requirement Samples Iced? Y / N

Sampled by (print)

Signature

Relinquished by

Date:

Time:

Received by

Temp @ recei

°C

Preservation / pH checked? Y / N

By:

Date:

TestAmerica WESTFIELD

Page ___ of ___

White = Lab file Yellow = Report copy Pink = Customer copy

template

Ready

NUM

Monthly metrics report

metrics report - Windows Picture and Fax Viewer

Microsoft Excel - STL_Metrics Rev4.0

D7 # Reports independent QA reviewed

	A	B	C	D	E	F	G	H	I
2	Monthly QA Report Metrics - STL --------				example				
3	Row	Objective	Period	Metrics	Jan'07	Feb'07	Mar'07	Apr'07	Ma
4	1		This month	# Reports for month	339				
5	2		This month	# Reports revised due to lab error	29				
6	3	<1%	This month	Revised Reports Ratio	5.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0
7	4		This month	# Reports independent QA reviewed	59				
8	5	if revised rep	This month	% Data Review: Reports	10.2	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0
9	6		Currently	# Technical staff (Analysts/technicians, including temps)	35				
10	7		Jan - to date	# of Analyst work product reviewed year-to-date	38				
11	8	83%	Jan - to date	% Analyst Data Authenticity Audits	100.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0
12	9		Current	# of analytical instruments	39				
13	10		Jan - to date	# of instrument data reviewed for data authenticity year-to-date	39				
14	11	96%	Jan - to date	% Electronic Data Authenticity Audits	100.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0
15	12	>5	This month	# Client Complaints	0	2	1	1	
16	13	>5	This month	# Client Compliments	2	2	1	1	
17	14		Jan - to date	# of PT analytes participated and received scores	2299				
18	15		Jan - to date	# of PT analytes not acceptable	56				
19	16	96%	Jan - to date	% PT Cumulative Score	97.6	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0
20	17	0	Jan - to date	# PT Repeat Analyte Failures Cummulative (analyte failed more than once in 4 consecutive studies)	0	0	0	1	
21	18		Currently	# SOPs	132				
22	19		Currently	# SOPs Reviewed/revised within 24 months	132				
23	20		Currently	# Methods without approved SOPs	0				
24	21	100%	Currently	SOP Status	100.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0
25	22	0	Jan - to date	Certification Losses due to performance/audit issues (# Methods affected)	1				
26	23	6	Currently	Internal Audit Closure (#months open)	5				
27	24	0	This month	Hold Time Violations (# samples affected)	5				

Jan Narrative / Metrics / PT Score Summary / External Audit Summary

APPENDIX II

CERTIFIED PARAMETERS

Note: This certified parameter list is subject to change, contact the laboratory to determine if you have the most recent list of certified parameters.

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
RICHARD F. DAINES, M.D.

Expires 12:01 AM April 01, 2008
Issued April 01, 2007
Revised July 03, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. STEVEN HARTMAN
TESTAMERICA WESTFIELD
53 SOUTHAMPTON ROAD
WESTFIELD, MA 01085

NY Lab Id No: 10843
EPA Lab Code: MA00014

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER All approved analytes are listed below:

Drinking Water Bacteriology	
Coliform, Total / E. coli (Qualitative)	Colilert
Drinking Water Metals I	
Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Drinking Water Metals II	
Aluminum, Total	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
Nickel, Total	EPA 200.8 Rev. 5.4
Drinking Water Metals II	
Thallium, Total	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Drinking Water Metals III	
Boron, Total	EPA 200.7 Rev. 4.4
Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4
Drinking Water Miscellaneous	
Methyl tert-butyl ether	EPA 524.2
Drinking Water Non-Metals	
Alkalinity	SM 18-20 2320B (97)
Calcium Hardness	EPA 200.7 Rev. 4.4
Color	SM 18-20 2120B (01)
Corrosivity	SM 18-19 2330
Fluoride, Total	EPA 300.0 Rev. 2.1
Hydrogen Ion (pH)	EPA 150.1
Nitrate (as N)	EPA 300.0 Rev. 2.1
Nitrite (as N)	EPA 300.0 Rev. 2.1
Orthophosphate (as P)	SM 18-20 4500-P E
Solids, Total Dissolved	SM 18-20 2540C (97)
Specific Conductance	SM 18-20 2510B (97)
Volatile Aromatics	
1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2

Serial No.: 34032

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Volatile Aromatics

1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2

Volatile Halocarbons

1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

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Acrylates

Acrolein (Propenal)	EPA 624
	EPA 8260B
Acrylonitrile	EPA 624
	EPA 8260B

Amines

2-Nitroaniline	EPA 8270C
3-Nitroaniline	EPA 8270C
4-Chloroaniline	EPA 8270C
4-Nitroaniline	EPA 8270C
Carbazole	EPA 8270C
Pyridine	EPA 8270C

Bacteriology

Coliform, fecal	SM 18-20 9222D (97)

Benzidines

3,3' -Dichlorobenzidine	EPA 625
Benzidine	EPA 625
	EPA 8270C

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 608
	EPA 8081A
4,4'-DDE	EPA 608
	EPA 8081A
4,4'-DDT	EPA 608
	EPA 8081A
Aldrin	EPA 608
	EPA 8081A

Chlorinated Hydrocarbon Pesticides

alpha-BHC	EPA 608
	EPA 8081A
alpha-Chlordane	EPA 8081A
beta-BHC	EPA 608
	EPA 8081A
Chlordane Total	EPA 608
	EPA 8081A
delta-BHC	EPA 608
	EPA 8081A
Dieldrin	EPA 608
	EPA 8081A
Endosulfan I	EPA 608
	EPA 8081A
Endosulfan II	EPA 608
	EPA 8081A
Endosulfan sulfate	EPA 608
	EPA 8081A
Endrin	EPA 608
	EPA 8081A
Endrin aldehyde	EPA 608
	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081A
Heptachlor	EPA 608
	EPA 8081A
Heptachlor epoxide	EPA 608
	EPA 8081A
Lindane	EPA 608
Toxaphene	EPA 608

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Chlorinated Hydrocarbon Pesticides

Toxaphene	EPA 8081A

Chlorinated Hydrocarbons

1,2,4-Trichlorobenzene	EPA 625
	EPA 8270C
2-Chloronaphthalene	EPA 625
Hexachlorobenzene	EPA 625
	EPA 8270C
Hexachlorobutadiene	EPA 625
	EPA 8270C
Hexachlorocyclopentadiene	EPA 625
Hexachloroethane	EPA 625
	EPA 8270C

Demand

Biochemical Oxygen Demand	SM 18-20 5210B (01)
Chemical Oxygen Demand	EPA 410.4 Rev. 2.0

Haloethers

4-Bromophenylphenyl ether	EPA 625
4-Chlorophenylphenyl ether	EPA 625
Bis (2-chloroisopropyl) ether	EPA 625
Bis(2-chloroethoxy)methane	EPA 625
Bis(2-chloroethyl)ether	EPA 625
	EPA 8270C

Mineral

Acidity	EPA 305.1
Alkalinity	SM 18-20 2320B (97)
Calcium Hardness	EPA 200.7 Rev. 4.4

Mineral

Calcium Hardness	SM 18-20 2340B (97)
Chloride	EPA 300.0 Rev. 2.1
Hardness, Total	EPA 200.7 Rev. 4.4
Sulfate (as SO4)	EPA 300.0 Rev. 2.1

Nitroaromatics and Isophorone

2,4-Dinitrotoluene	EPA 625
	EPA 8270C
2,6-Dinitrotoluene	EPA 625
	EPA 8270C
Isophorone	EPA 625
	EPA 8270C
Nitrobenzene	EPA 625
	EPA 8270C

Nitrosoamines

N-Nitrosodimethylamine	EPA 625
	EPA 8270C
N-Nitrosodi-n-propylamine	EPA 625
	EPA 8270C
N-Nitrosodiphenylamine	EPA 625
	EPA 8270C

Nutrient

Ammonia (as N)	LACHAT 10-107-06-1-B
Kjeldahl Nitrogen, Total	LACHAT 10-107-06-2
Nitrate (as N)	EPA 300.0 Rev. 2.1
	LACHAT 10-107-04-1-C
Nitrite (as N)	SM 18-20 4500-NO2 B (00)
Orthophosphate (as P)	SM 18-20 4500-P E

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All approved analytes are listed below:

Nutrient	
Phosphorus, Total	SM 18-20 4500-P E
Phthalate Esters	
Benzyl butyl phthalate	EPA 625
	EPA 8270C
Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270C
Diethyl phthalate	EPA 625
	EPA 8270C
Dimethyl phthalate	EPA 625
	EPA 8270C
Di-n-butyl phthalate	EPA 625
	EPA 8270C
Di-n-octyl phthalate	EPA 625
	EPA 8270C
Polychlorinated Biphenyls	
PCB-1016	EPA 608
	EPA 8082
PCB-1221	EPA 608
	EPA 8082
PCB-1232	EPA 608
	EPA 8082
PCB-1242	EPA 608
	EPA 8082
PCB-1248	EPA 608
	EPA 8082
PCB-1254	EPA 608
	EPA 8082
PCB-1260	EPA 608

Polychlorinated Biphenyls	
PCB-1260	EPA 8082
Polynuclear Aromatics	
Acenaphthene	EPA 625
	EPA 8270C
Acenaphthylene	EPA 625
	EPA 8270C
Anthracene	EPA 625
	EPA 8270C
Benzo(a)anthracene	EPA 625
	EPA 8270C
Benzo(a)pyrene	EPA 625
	EPA 8270C
Benzo(b)fluoranthene	EPA 625
	EPA 8270C
Benzo(ghi)perylene	EPA 625
	EPA 8270C
Benzo(k)fluoranthene	EPA 625
	EPA 8270C
Chrysene	EPA 625
	EPA 8270C
Dibenzo(a,h)anthracene	EPA 625
	EPA 8270C
Fluoranthene	EPA 625
	EPA 8270C
Fluorene	EPA 625
	EPA 8270C
Indeno(1,2,3-cd)pyrene	EPA 625
	EPA 8270C

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Issued April 01, 2007
Revised July 03, 2007

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. STEVEN HARTMAN
TESTAMERICA WESTFIELD
53 SOUTHAMPTON ROAD
WESTFIELD, MA 01085

NY Lab Id No: 10843
EPA Lab Code: MA00014

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER All approved analytes are listed below:

Polynuclear Aromatics

Naphthalene	EPA 625
	EPA 8270C
Phenanthrene	EPA 625
	EPA 8270C
Pyrene	EPA 625
	EPA 8270C

Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 8270C
2,4,6-Trichlorophenol	EPA 625
	EPA 8270C
2,4-Dichlorophenol	EPA 625
	EPA 8270C
2,4-Dimethylphenol	EPA 625
2,4-Dinitrophenol	EPA 625
	EPA 8270C
2-Chlorophenol	EPA 625
	EPA 8270C
2-Methyl-4,6-dinitrophenol	EPA 625
	EPA 8270C
2-Methylphenol	EPA 8270C
2-Nitrophenol	EPA 625
	EPA 8270C
3-Methylphenol	EPA 8270C
4-Chloro-3-methylphenol	EPA 625
	EPA 8270C
4-Methylphenol	EPA 8270C
4-Nitrophenol	EPA 625
	EPA 8270C

Priority Pollutant Phenols

Cresols, Total	EPA 8270C
Pentachlorophenol	EPA 625
	EPA 8270C
Phenol	EPA 625
	EPA 8270C

Purgeable Aromatics

1,2-Dichlorobenzene	EPA 624
	EPA 8260B
1,3-Dichlorobenzene	EPA 624
	EPA 8260B
1,4-Dichlorobenzene	EPA 624
	EPA 625
	EPA 8260B
Benzene	EPA 624
	EPA 8260B
Chlorobenzene	EPA 624
	EPA 8260B
Ethyl benzene	EPA 624
	EPA 8260B
Styrene	EPA 8260B
Toluene	EPA 624
	EPA 8260B
Total Xylenes	EPA 624
	EPA 8260B

Purgeable Halocarbons

1,1,1-Trichloroethane	EPA 624
	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 624

Serial No.: 34033

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Purgeable Halocarbons

1,1,2,2-Tetrachloroethane	EPA 8260B
1,1,2-Trichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethene	EPA 624
	EPA 8260B
1,2-Dichloroethane	EPA 624
	EPA 8260B
1,2-Dichloropropane	EPA 624
	EPA 8260B
Bromodichloromethane	EPA 624
	EPA 8260B
Bromoform	EPA 624
	EPA 8260B
Bromomethane	EPA 624
	EPA 8260B
Carbon tetrachloride	EPA 624
	EPA 8260B
Chloroethane	EPA 624
	EPA 8260B
Chloroform	EPA 624
	EPA 8260B
Chloromethane	EPA 624
	EPA 8260B
cis-1,3-Dichloropropene	EPA 601
	EPA 624
	EPA 8260B
Dibromochloromethane	EPA 624

Purgeable Halocarbons

Dibromochloromethane	EPA 8260B
Methylene chloride	EPA 624
	EPA 8260B
Tetrachloroethene	EPA 624
	EPA 8260B
trans-1,2-Dichloroethene	EPA 624
	EPA 8260B
trans-1,3-Dichloropropene	EPA 624
	EPA 8260B
Trichloroethene	EPA 624
	EPA 8260B
Trichlorofluoromethane	EPA 8260B
Vinyl chloride	EPA 624
	EPA 8260B

Purgeable Organics

2-Butanone (Methylethyl ketone)	EPA 8260B
2-Hexanone	EPA 8260B
4-Methyl-2-Pentanone	EPA 8260B
Acetone	EPA 8260B
Carbon Disulfide	EPA 8260B
Vinyl acetate	EPA 8260B

Residue

Solids, Total Dissolved	EPA 160.1
Solids, Total Suspended	EPA 160.2

Semi-Volatile Organics

2-Methylnaphthalene	EPA 8270C
Benzoic Acid	EPA 8270C

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All approved analytes are listed below:

Semi-Volatile Organics

Benzyl alcohol	EPA 8270C
Dibenzofuran	EPA 8270C

Wastewater Metals I

Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Iron, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Lead, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Magnesium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Nickel, Total	EPA 200.7 Rev. 4.4

Wastewater Metals I

Nickel, Total	EPA 200.8 Rev. 5.4
	EPA 6010B
Potassium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Sodium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Strontium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B

Wastewater Metals II

Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Antimony, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Chromium VI	EPA 7196A
	SM 18-19 3500-Cr D
Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 7470A

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53 SOUTHAMPTON ROAD
WESTFIELD, MA 01085

NY Lab Id No: 10843
EPA Lab Code: MA00014

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Wastewater Metals II

Selenium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B

Wastewater Metals III

Cobalt, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Thallium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Titanium, Total	EPA 200.7 Rev. 4.4

Wastewater Miscellaneous

Boron, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Color	SM 18-20 2120E
Cyanide, Total	EPA 9012A
	EPA 9014
	LACHAT 10-204-00-1-A
Hydrogen Ion (pH)	EPA 150.1
	EPA 9040B

Wastewater Miscellaneous

Phenols	LACHAT 10-210-00-1-A
Silica, Dissolved	EPA 6010B
Specific Conductance	SM 18-20 2510B (97)
Sulfide (as S)	SM 18 4500-S E

Sample Preparation Methods

EPA 3005A
EPA 3010A
EPA 3020A
EPA 3510C
EPA 5030B
EPA 9030B

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Amines	
2-Nitroaniline	EPA 8270C
3-Nitroaniline	EPA 8270C
4-Chloroaniline	EPA 8270C
4-Nitroaniline	EPA 8270C
Carbazole	EPA 8270C
Characteristic Testing	
Corrosivity	EPA 9045C
Ignitability	EPA 1010
Chlorinated Hydrocarbon Pesticides	
alpha-Chlordane	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081A
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA 8270C
2-Chloronaphthalene	EPA 8270C
Hexachlorobenzene	EPA 8270C
Hexachlorobutadiene	EPA 8270C
Hexachlorocyclopentadiene	EPA 8270C
Hexachloroethane	EPA 8270C
Haloethers	
4-Bromophenylphenyl ether	EPA 8270C
4-Chlorophenylphenyl ether	EPA 8270C
Bis (2-chloroisopropyl) ether	EPA 8270C
Bis(2-chloroethoxy)methane	EPA 8270C
Metals I	
Barium, Total	EPA 6010B
Metals I	
Cadmium, Total	EPA 6010B
Calcium, Total	EPA 6010B
Chromium, Total	EPA 6010B
Copper, Total	EPA 6010B
Iron, Total	EPA 6010B
Lead, Total	EPA 6010B
Magnesium, Total	EPA 6010B
Manganese, Total	EPA 6010B
Nickel, Total	EPA 6010B
Potassium, Total	EPA 6010B
Silver, Total	EPA 6010B
Sodium, Total	EPA 6010B
Metals II	
Aluminum, Total	EPA 6010B
Antimony, Total	EPA 6010B
Arsenic, Total	EPA 6010B
Beryllium, Total	EPA 6010B
Mercury, Total	EPA 7471A
Selenium, Total	EPA 6010B
Vanadium, Total	EPA 6010B
Zinc, Total	EPA 6010B
Metals III	
Cobalt, Total	EPA 6010B
Molybdenum, Total	EPA 6010B
Thallium, Total	EPA 6010B
Miscellaneous	
Cyanide, Total	EPA 9012A

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Miscellaneous

Hydrogen Ion (pH)	EPA 9045C

Nitroaromatics and Isophorone

2,4-Dinitrotoluene	EPA 8270C
2,6-Dinitrotoluene	EPA 8270C
Isophorone	EPA 8270C
Nitrobenzene	EPA 8270C

Nitrosoamines

N-Nitrosodi-n-propylamine	EPA 8270C

Phthalate Esters

Benzyl butyl phthalate	EPA 8270C
Bis(2-ethylhexyl) phthalate	EPA 8270C
Diethyl phthalate	EPA 8270C
Dimethyl phthalate	EPA 8270C
Di-n-butyl phthalate	EPA 8270C
Di-n-octyl phthalate	EPA 8270C

Polychlorinated Biphenyls

PCB-1016	EPA 8082
PCB-1221	EPA 8082
PCB-1232	EPA 8082
PCB-1242	EPA 8082
PCB-1248	EPA 8082
PCB-1254	EPA 8082
PCB-1260	EPA 8082

Polynuclear Aromatic Hydrocarbons

Acenaphthene	EPA 8270C
Acenaphthylene	EPA 8270C

Polynuclear Aromatic Hydrocarbons

Anthracene	EPA 8270C
Benzo(a)anthracene	EPA 8270C
Benzo(a)pyrene	EPA 8270C
Benzo(b)fluoranthene	EPA 8270C
Benzo(ghi)perylene	EPA 8270C
Benzo(k)fluoranthene	EPA 8270C
Chrysene	EPA 8270C
Dibenzo(a,h)anthracene	EPA 8270C
Fluoranthene	EPA 8270C
Fluorene	EPA 8270C
Indeno(1,2,3-cd)pyrene	EPA 8270C
Naphthalene	EPA 8270C
Phenanthrene	EPA 8270C
Pyrene	EPA 8270C

Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 8270C
2,4,6-Trichlorophenol	EPA 8270C
2,4-Dichlorophenol	EPA 8270C
2,4-Dimethylphenol	EPA 8270C
2,4-Dinitrophenol	EPA 8270C
2-Chlorophenol	EPA 8270C
2-Methyl-4,6-dinitrophenol	EPA 8270C
2-Methylphenol	EPA 8270C
2-Nitrophenol	EPA 8270C
3-Methylphenol	EPA 8270C
4-Chloro-3-methylphenol	EPA 8270C
4-Methylphenol	EPA 8270C
4-Nitrophenol	EPA 8270C

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Priority Pollutant Phenols

Pentachlorophenol	EPA 8270C
Phenol	EPA 8270C

Purgeable Aromatics

1,2-Dichlorobenzene	EPA 8260B
	EPA 8270C
1,3-Dichlorobenzene	EPA 8260B
	EPA 8270C
1,4-Dichlorobenzene	EPA 8260B
	EPA 8270C
Benzene	EPA 8260B
Chlorobenzene	EPA 8260B
Ethyl benzene	EPA 8260B
Styrene	EPA 8260B
Toluene	EPA 8260B
Total Xylenes	EPA 8260B

Purgeable Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260B
1,1,1-Trichloroethane	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8260B
1,1,2-Trichloroethane	EPA 8260B
1,1-Dichloroethane	EPA 8260B
1,1-Dichloroethene	EPA 8260B
1,2-Dichloroethane	EPA 8260B
1,2-Dichloropropane	EPA 8260B
Bromodichloromethane	EPA 8260B
Bromoform	EPA 8260B
Bromomethane	EPA 8260B
Carbon tetrachloride	EPA 8260B

Purgeable Halocarbons

Chloroethane	EPA 8260B
Chloroform	EPA 8260B
Chloromethane	EPA 8260B
cis-1,3-Dichloropropene	EPA 8260B
Dibromochloromethane	EPA 8260B
Dichlorodifluoromethane	EPA 8260B
Methylene chloride	EPA 8260B
Tetrachloroethene	EPA 8260B
trans-1,3-Dichloropropene	EPA 8260B
Trichloroethene	EPA 8260B
Trichlorofluoromethane	EPA 8260B
Vinyl chloride	EPA 8260B

Purgeable Organics

2-Butanone (Methylethyl ketone)	EPA 8260B
2-Hexanone	EPA 8260B
4-Methyl-2-Pentanone	EPA 8260B
Acetone	EPA 8260B
Carbon Disulfide	EPA 8260B
Vinyl acetate	EPA 8260B

Semi-Volatile Organics

2-Methylnaphthalene	EPA 8270C
Benzoic Acid	EPA 8270C
Benzyl alcohol	EPA 8270C
Dibenzofuran	EPA 8270C

Sample Preparation Methods

	EPA 3020A
	EPA 3050B

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Sample Preparation Methods

EPA 3550B
EPA 5035

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The Commonwealth of Massachusetts

Department of Environmental Protection

Division of Environmental Analysis
Senator William X. Wall Experiment Station

certifies

M-MA014 **TESTAMERICA WESTFIELD**
53 SOUTHAMPTON RD
WESTFIELD, MA 01085-5308

Laboratory Director: **STEVEN HARTMANN**

for the analysis of **NON POTABLE WATER (CHEMISTRY)**
POTABLE WATER (MICROBIOLOGY)
POTABLE WATER (CHEMISTRY)
pursuant to 310 CMR 42.00

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P. Contact the Division of Environmental Analysis to verify the current certification status of the laboratory.

Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.

Issued: **01 JUL 2007**
Expires: **30 JUN 2008**

Director, Division of Environmental Analysis

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2007

M-MA014 **TESTAMERICA WESTFIELD**
WESTFIELD MA

POTABLE WATER (MICROBIOLOGY) **Effective Date** **01 JUL 2007** **Expiration Date** **30 JUN 2008**

Analytes and Methods

HETEROTROPHIC PLATE COUNT	SM9215B
TOTAL COLIFORM	MF-SM9222B
TOTAL COLIFORM	ENZ. SUB. SM9223
FECAL COLIFORM	MF-SM9222D
E. COLI	EC-MUG-SM9221F
E. COLI	ENZ. SUB. SM9223

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2007

M-MA014 TESTAMERICA WESTFIELD
WESTFIELD MA

POTABLE WATER (CHEMISTRY) Effective Date 01 JUL 2007 Expiration Date 30 JUN 2008

Analytes	Methods
ANTIMONY	EPA 200.8
ARSENIC	EPA 200.8
BARIUM	EPA 200.7
BARIUM	EPA 200.8
BERYLLIUM	EPA 200.8
CADMIUM	EPA 200.7
CADMIUM	EPA 200.8
CHROMIUM	EPA 200.7
CHROMIUM	EPA 200.8
COPPER	EPA 200.7
COPPER	EPA 200.8
LEAD	EPA 200.8
MERCURY	EPA 245.1
NICKEL	EPA 200.8
SELENIUM	EPA 200.8
THALLIUM	EPA 200.8
NITRATE-N	EPA 300.0
NITRATE-N	LACHAT 10-107-04-1-A
NITRITE-N	EPA 300.0
NITRITE-N	SM 4500-NO2-B
FLUORIDE	EPA 300.0
SODIUM	EPA 200.7
SULFATE	EPA 300.0
CYANIDE, TOTAL	LACHAT 10-204-00-1-A
CALCIUM	EPA 200.7
ALKALINITY, TOTAL	SM 2320B
TOTAL DISSOLVED SOLIDS	SM 2540C
PH	EPA 150.1
TRIHALOMETHANES	EPA 524.2
VOLATILE ORGANIC COMPOUNDS	EPA 524.2
1,2-DIBROMOETHANE	EPA 504.1
1,2-DIBROMO-3-CHLOROPROPANE	EPA 504.1

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2007

M-MA014 **TESTAMERICA WESTFIELD**
WESTFIELD MA

NON POTABLE WATER (CHEMISTRY) **Effective Date** **01 JUL 2007** **Expiration Date** **30 JUN 2008**

Analytes and Methods

ALUMINUM	EPA 200.7	SODIUM	EPA 200.7
ALUMINUM	EPA 200.8	POTASSIUM	EPA 200.7
ANTIMONY	EPA 200.7	ALKALINITY, TOTAL	SM 2320B
ANTIMONY	EPA 200.8	CHLORIDE	EPA 300.0
ARSENIC	EPA 200.7	SULFATE	EPA 300.0
ARSENIC	EPA 200.8	AMMONIA-N	LACHAT 10-107-06-1-B
BERYLLIUM	EPA 200.7	NITRATE-N	EPA 300.0
BERYLLIUM	EPA 200.8	KJELDAHL-N	LACHAT 10-107-06-02-D
CADMIUM	EPA 200.7	ORTHOPHOSPHATE	SM 4500-P-E
CADMIUM	EPA 200.8	PHOSPHORUS, TOTAL	SM 4500-P-B,E
CHROMIUM	EPA 200.7	CHEMICAL OXYGEN DEMAND	EPA 410.4
CHROMIUM	EPA 200.8	BIOCHEMICAL OXYGEN DEMAND	SM 5210B
COBALT	EPA 200.7	TOTAL ORGANIC CARBON	SM 5310B
COBALT	EPA 200.8	CYANIDE, TOTAL	LACHAT 10-204-00-1-A
COPPER	EPA 200.7	NON-FILTERABLE RESIDUE	SM 2540D
COPPER	EPA 200.8	CHLORINE, TOTAL RESIDUAL	SM 4500-CL-F
IRON	EPA 200.7	PHENOLICS, TOTAL	LACHAT 10-210-00-1-A
LEAD	EPA 200.7	VOLATILE HALOCARBONS	EPA 624
LEAD	EPA 200.8	VOLATILE AROMATICS	EPA 624
MANGANESE	EPA 200.7	CHLORDANE	EPA 608
MANGANESE	EPA 200.8	ALDRIN	EPA 608
MERCURY	EPA 245.1	DIELDRIN	EPA 608
MOLYBDENUM	EPA 200.7	DDD	EPA 608
NICKEL	EPA 200.7	DDE	EPA 608
NICKEL	EPA 200.8	DDT	EPA 608
SELENIUM	EPA 200.7	HEPTACHLOR	EPA 608
SELENIUM	EPA 200.8	HEPTACHLOR EPOXIDE	EPA 608
SILVER	EPA 200.7	POLYCHLORINATED BIPHENYLS (OIL)	EPA 600/4-81-045
SILVER	EPA 200.8		
THALLIUM	EPA 200.7		
THALLIUM	EPA 200.8		
TITANIUM	EPA 200.7		
VANADIUM	EPA 200.7		
VANADIUM	EPA 200.8		
ZINC	EPA 200.7		
ZINC	EPA 200.8		
PH	SM 4500-H-B		
SPECIFIC CONDUCTIVITY	SM 2510B		
TOTAL DISSOLVED SOLIDS	SM 2540C		
HARDNESS (CACO3), TOTAL	EPA 200.7		
CALCIUM	EPA 200.7		
MAGNESIUM	EPA 200.7		

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STL WESTFIELD

STANDARD OPERATING PROCEDURE

Metals Analysis by Inductively Coupled Plasma Spectroscopy

EPA Method 6010B, SW-846

SOP ID: MES00200.MA

Revision (3)

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Approved Signatures and Dates

Christine Lorraine Reynolds

QUALITY ASSURANCE COORDINATOR

LABORATORY DIRECTOR

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1.0 TITLE

Metals Analysis by Inductively Coupled Plasma Spectroscopy EPA Method 6010B, SW-846

2.0 MATRIX

This method covers the determination of metals by Inductively Coupled Plasma Spectroscopy (ICP) by EPA Method SW646 6010B. Matrixes analyzed by this method include groundwater, surface water, aqueous samples, TCLP and EP extracts, industrial waste, soils, sludges, sediments and other solid wastes.

3.0 METHOD DETECTION LIMIT

Most metals can be reported to results between 10–50µg/L. Elements associated with the minerals (Ca, K, Mg and Na) typically are reported between 400-4000 µg/L. Detection limits and linear ranges for specific elements vary with wavelength selected.

4.0 SCOPE and APPLICATION

4.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) is used to determinate metals in solution. This method is suitable for the analysis of samples of liquid matrices for the determination of trace elements. Analysis is based on acid digestion with subsequent analysis.

Dissolved elements are determined in filtered and acidified samples.

5.0 SUMMARY

5.1 This method describes a technique for the simultaneous and sequential multi-element determinations of trace elements in solution. The basis of this method is the measurement of atomic emission by an optical spectroscopic technique. In inductively coupled plasma (ICP) analysis, samples are nebulized and the aerosol that is produced is transported into the plasma torch where excitation occurs. The ICP produces characteristic atomic line emission spectra. The spectra are then dispersed by a prism grating spectrometer and the intensities of the lines are monitored by the instruments detector. The array detector is a charge-coupled device (CCD) which is made up of a silicon chip with an array of photosensitive material on its surface. The detector converts photons, that are absorbed by the individual units of the array, into electrons that are collected and read as the sample signal. By calibrating the instrument against known standard concentrations and through the use of a data processor, the user is able to quantitate concentrations of unknown metal analytes in solution.

5.2 Total and total recoverable samples are first digested in a combination of nitric and hydrochloric acid. After digestion the samples are typically adjusted to 50 ml final volume (100ml final volume for soil digestates). Samples that have been field filtered can typically be analyzed as received if its turbidity is <1NTU.

6.0 DEFINITIONS

6.1 *Dissolved Metals*: Those elements, which pass through a 0.45µm membrane filter. The sample is acidified after filtration.

6.2 *Suspended Metals:* Those elements which are retained by a 0.45µm membrane filter

6.3 *Total Recoverable Metals*: The concentration of analyte determined by "direct analysis" of an unfiltered acid preserved drinking water sample with a turbidity of <1NTU. The concentration is determined on an unfiltered sample following treatment with hot, dilute acid digestion.

6.4 *Method Detection Limit (MDL)*: the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific digestion method. Quantitative results are not produced in this range, (40 CFR Part 136, Appendix B). The Method Detection Limit (MDL) is determined by performing at least 7 analyses of standards for all analytes measured at 2–5 times the required detection limit concentrations. The method detection limits are calculated as 3.143 times the standard deviation of the measured values.

6.5 *Instrument Detection Limit (IDL)*: the minimum amount of a substance that can be measured on a specific instrument, with a specified degree of confidence that the amount is greater than zero. The IDL is associated with the instrumental portion of a specific method only (non-digested), and specific sample preparation steps are not considered in its derivation. A calculated IDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The IDL thus represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

6.6 *Calibration Reporting Limit (CRI)*: The level to which data is reported for a specific test method and/or sample.

6.7 *Sensitivity*: The slope of the analytical curve: the relationship between the raw instrument signal and the concentration.

6.8 *Tuning Solution*: A multi-element solution containing elements that are representative of the emission range capable of being detecteded by the instrument. The solution is used to optimize the sensitivity of the instrument.

6.9 *Initial Calibration Verification (ICV)*: A multi-element standard of known concentrations prepared to verify instrument calibration. The solution must be an independent standard prepared from a different source than that used for calibration.

6.10 *Continuing Calibration Verification (CCV)*: A multi-element standard of a known concentration prepared to monitor and verify the instrument's daily continuing performance.

6.11 *Laboratory Control Sample (LCS)*: A multi-element standard of known concentration which is carried through the entire sample preparation and analysis procedure. The solution is used to verify the accuracy of the sample preparation

6.12 *Preparation Blank (Method Blank)*: High purity water containing the same acid

matrix as the calibration standards carried through the entire preparation process. This is used to monitor preparation contamination.

6.13 *Calibration Blank*: High purity water acidified with the same acid concentration present in the standards and samples. This may also be referred to as Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

6.14 *Matrix Spike (MS)*: Field sample to which a known amount of target analyte(s) is added.

6.15 *Water Sample*: For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, storm run-off, industrial or domestic wastewater.

6.16 *Calibration Standards*: a series of known standards solution used by the analyst for calibration the instrument.

6.17 *Linear Range:* The concentration range over which the analytical curve remains linear.

6.18 *Calibration curve:* A plot of the concentration of an analyte and the variable that is measured to indicate the presence of the analyte, in ICP-OES, usually intensity. The calibration curve is used to determine the analyte concentration in a sample.

6.19 *CID - Charge Injection Device*: a doped silicon based Charge Transfer Device used for the simultaneous detection of multiple wavelengths. The CID provides non-destructive reading of signals by using switching arrangements to measure the charge in a pixel directly but in general does not have the quantum efficiency of a CCD.

6.20 *Detector:* A device that absorbs photons and converts them into electrons. These photoelectrons can be measured to indicate the relative intensity of the incoming light.

6.21 *Inductive coupling*: Is the process of transferring energy to a system through the use of electromotive forces generated by magnetic fields. In ICP-OES a high-temperature discharge is generated by flowing a conductive gas (argon) through a magnetic field induced by a load coil that surrounds the tubes carrying the gas.

6.22 *Inter-element correction (I/E):* Correction applied to account for spectral interferences. A measurement must be made for the interfering element at its specific wavelength (which must be spectrally pure) to perform a correction at the analyte wavelength.

6.23 *Nebulization:* The process that converts a liquid to an aerosol.

6.24 *Plasma:* A plasma is a state of matter consisting of highly ionized gas which contains mainly electrons and ions but also contains atoms and molecules. The plasma has a high electron density but is electrically neutral overall and is regarded as the fourth state of matter.

7.0 INTERFERENCES

7.1 Spectral Interferences

7.1.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentrations elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

However, the first step in establishing the conditions required for good analytical performance is to eliminate potential problems rather than attempt to compensate for them. For example, the potential problems caused by spectral interferences can be eliminated by selection of the appropriate emission line.

The primary emission line of an element is typically selected to provide maximum sensitivity. However, if spectral overlap is found on this line, it is possible in most cases to select an alternative interferences free line for that element. Many spectral problems encountered in ICP can be avoided by the selection of emission lines that are free from spectral interferences.

7.2 Physical interferences

7.2.1 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in sample containing high dissolved solids.

7.2.2 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Chemical interferences are highly dependent on matrix type but are not normally a significant problem with the ICP technique (due to high plasma temperatures).

7.2.3 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. Suitable rinse time must be determined to reduce memory interferences.

7.2.4 Matrix interference can be assessed by the ICP Serial Dilution Analysis performed on a sample from each group of samples of similar concentration and matrix type. Agreement within 10% of the original determination after correction for dilution indicates an absence of matrix interference.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete

list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT and SUPPLIES

9.1 Varian 730 ES Spectrometer
9.2 Varian SPS Autosampler
9.3 Argon gas supply, welding grade or better.
9.4 Data processing software (Varian), hardware PC (Dell)
9.5 Peristaltic Pump Tubing
9.6 Plastic sample test tubes
9.7 Water recirculation unit for instrument cooling
9.8 Torch assembly and injector tube.

10.0 REAGENTS

10.1 All acids used shall be Trace Metal grade or equivalent

10.2 Hydrochloric Acid

10.3 Nitric Acid

10.4 Deionized distilled water equivalent to ASTM II reagent water, specification D1193, (Lab Water).

10.5 Rinse water: Nitric and HCL Acid Solution with Lab water.

10.6 Standard Solutions:

Calibration standards should be prepared as documented in this SOP. The traceability of the standards must be documented from source to use at the instrument.

10.6.1 Store calibration standards and other check solutions in the appropriate acid solution in plastic containers and at room temperature. Standards are made on an as needed basis and are verified daily.

10.7 Mixed Calibration Standard Solutions:

10.7.1 Calibration blank: Solution containing a concentration of nitric acid and hydrochloric acid.

10.7.2 Calibration Standard Solutions: A mixture of two multi-element solutions. STL Westfield calibration standards are a custom blend manufactured by Inorganic Ventures. The two combined solutions are diluted 1:100x. The LCS/LCD, MS/MSD, IPC (instrument performance check) and CCV are prepared from these two solutions.

10.8 Interference Check Samples (ICS)

10.8.1 ICS Solution A: a standard solution supplied by High Purity Standards or equivalent.

Concentration (mg/L)	ICS Solution Analyte**	Stock Standard Solution Concentration (mg/L)	Volume: Standard Solution (ml)	Solution Final (ml)*
100	Al	5000	1	50
100	Ca	5000		
100	Mg	5000		
40	Fe	2000		

10.8.2 ICS Solution AB: a standard solution supplied by Inorganic Ventures or equivalent.

Concentration (mg/L)	Analyte**	Stock Standard Solution Concentration (mg/L)	Volume: Standard Solution (ml)	Volume Stock Solution (Final)(ml)*
100	Al, Ca, Mg	5000	1.0	50
40	Fe	2000		
2.0	As, Pb, Tl,	1000	0.1	
1.0	Se	500		
0.6	Ag, Ba, Cd, Co, Cr, Cu, Ni, Zn	300		
0.4	Mn	200		
0.20	Be	100		

10.9 Varian Wavelength Calibration (Tune) Solution: A multi-element solution used to perform the instrument's calibration wavelength tune.

10.10 MCP 6010B CRI MDL solution. A multi-element solution diluted 1:100x. Final concentration reported at the MCP reporting limit.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Water samples are collected in plastic or glass 1000-mL sample containers. Soils are collected in 250-mL wide mouth jars. Water samples are preserved with Nitric acid to a pH of < 2.0. For the collection of dissolved metals, the sample must be filtered through a 0.45um pore diameter membrane filter at the time of collection or as soon thereafter as practically possible.

11.2 Samples are shipped to the laboratory on ice.

11.3 Samples are stored in either a secured water or soil refrigerator at 4^0C.

11.4 Holding time for digestion and analysis is 6 months from date of collection for all metals.

12.0 QUALITY CONTROL AND CORRECTIVE ACTION

QC Analysis	Frequency	Criteria	Corrective Action
Daily Performance Check	Daily	≤5%	Perform instrument maintenance and rerun standard.
Linear Dynamic Range	Each element semi-annually	±10%	Verify annually or whenever a change in analytical performance caused by a change in hardware or operating conditions.
Initial Calibration	Following tuning and prior to sample analysis	r = ≥ 0.995	Re-calibrate
ICB (Initial Calibration Blank)	Once at start of analytical run	< RL	Recalibrate. Re-check ICB; if still unacceptable, investigate possible contamination of reagents, Lab water.
ICV (Initial Calibration Verification)	Immediately after calibration, (Independent source)	90-110%	Recalibrate. Re-check ICV; if still unacceptable, investigate possible calibration std or ICV out of specification.
CRI	Daily, if not part of calibration curve	70-130% MCP Criteria	Recalibrate, re-analyze
LCS, LCSD	Once per batch of 20 field Samples or fraction thereof	80-120% RPD ≤20%	If unacceptable, re-analyze; if still out recalibrate or re-digest sample batch.
MS/MSD (Matrix Spike)	MS/MSD per 20 field samples	75-125% RPD ≤20%, 35% soils	If unacceptable, review LCS. If LCS acceptable, no corrective action require-matrix effect suspected.
CCV (Continuing Calibration Verification)	Every 10 samples or fraction thereof. Mid point range	90-110%	If unacceptable, re-analyze; if still out recalibrate. Sample values for blocks between acceptable CCV's may be reported; those that are not must be rerun.

CCB (Continuing Calibration Blank)	Every 10 samples or fraction thereof, after the CCV	< RL	If unacceptable, re-analyze; if still out recalibrate. Sample values for blocks between acceptable CCB may be reported; those that are not must also be rerun.
MB Method Blank/digestion Blank	One per batch of 20 samples	<RL	Re-analyzed, Re-digest (exceptions: If the analyte concentration is >10x the blank or data is qualified with a "B" flag. The analyte in the sample is <RL

* Once a year, analyze a CCV at a different concentration than what's normally prepared. This is a NELAC requirement.

12.2 ICP Interference Check Standard (ICS) Analysis: The Interference Check Samples consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents and solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP. Solution ICS A the interfering elements are control at ± 20%, while the non-interferning spectral lines are monitored for interelement correction accuracy. Results for the ICP analyses of Solution AB during the analytical runs must fall within the control limit of ± 20% of the true value for the analytes included in the Interference Check samples. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

TABLE 2
INTERFERANT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ag	0.6	Al	100
Ba	0.6	Ca	100
Be	0.2	Fe	40
Cd	0.6	Mg	100
Co	0.6		
Cr	0.6		
Cu	0.6		
Mn	0.4		
Ni	0.6		
Pb	2.0		
V	0.6		

Zn	0.6

12.3 Post Digest Spike (PDS): The PDS is used to determine the presence of matrix interferences. It also may determine the need for running the samples by the Method of Standard Additions (MSA). Run a PDS for every sample batch. Post Digestion Spike (PDS) addition is accomplished by adding analyte standard to a portion of a prepared sample, or it's dilution, at a sufficient concentration to yield a result at a minimum level of 20 times and a maximum of 100 times the MDL. Percent recovery should be 75-125% of the known value. If recovery of the analyte(s) is not within the specified limits, a matrix effect should be suspected.

12.4 Serial Dilution Test: If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrument detection limit in the original solution but <90% of the linear limit), an analysis of a 5x dilution should agree (after correction for the fivefold dilution) within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13.0 CALIBRATION and STANDARDIZATION

13.1 **Instrument Startup for Varian ICP**

13.1.1 Switch on the PC

Step 1

Close the plasma compartment door, ensuring that the locking lever is fully latched and turn on the power to the instrument (if you have not already done so).

Step 2

Place the sample tubing from the peristaltic pump into the wash solution and the drain tubing into the drain vessel.

Step 3

Turn on the plasma by clicking on the Main toolbar or pressing SHIFT+F4 on the keyboard. The pump will be initialized and the solution will begin aspirating.

Note
The plasma will take between 10–30 seconds to ignite. If it fails to ignite. The plasma needs to be turned on 30 minutes prior to analysis to allow it to stabilize

Step 4

Adjust the peristaltic pressure bars for even sample flow.

13.2 Perform Dark Current and Wavelength calibration prior to instrument calibration.

13.2.1 To perform a wavelength calibration:

Step 1

Check that the Polychromator boost purge is on. To do this, click (**Instrument Setup** button) on the Main toolbar to bring up the Instrument setup window. Click the **Status** tab and check that under the Polychromator listing Poly Boost is 'On'. If the Poly boost purge is 'Off' you should turn it on. To do this:

a. Choose **Poly boost on** from the Options menu in the main window.

b. Reopen the Instrument setup window. Under Polychromator, Poly boost should now say 'On'. Leave the poly boost purging for at least 20 minutes prior to performing the wavelength calibration.

Note
It is highly recommended that the Poly boost purge be on when performing a wavelength calibration.

Step 2

Open the Instrument setup window (if it is not already open), by clicking on the Main toolbar, and click the **W/L Calib** tab.

Step 3

With the wash solution aspirating, click Dark Current **scan**. It will take roughly 1 to 2 minutes to calibrate the detector. Once completed, a message will appear indicating the dark current measurement has been completed.

Note
Save the dark current measurement data by clicking **Store dark current**.

Step 4

Remove the sample tubing from the wash solution. Wipe the tubing and transfer it into the wavelength calibration solution.

Step 5

Once the wavelength calibration solution is aspirating, click **Calibrate** to perform the wavelength calibration. After a short while, a message will appear stating that the wavelength calibration is complete, as well as indicating the wavelength calibration range.

13.3 Instrument Shutdown

13.3.1 To shut down the instrument:

Step 1

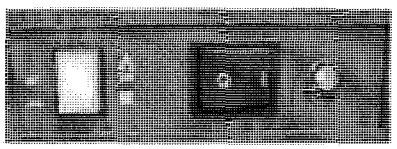
- Rinse the spraychamber by running water through the system for a few minutes.

Note
When running organic samples, it may be necessary to disconnect the spraychamber and clean it and dry it thoroughly between runs.

- Extinguish the plasma by clicking (or selecting Plasma Off from the **Analyze** menu when a worksheet is open). The peristaltic pump stops automatically when the plasma is extinguished.

Note
The yellow Plasma Emergency Off button, located next to the mains power switch on the front of the instrument, is designed to be used only in an emergency, for example if the torch is melting. It is not intended to be used every time you want to extinguish the Plasma.

Note
If used to extinguish the Plasma, you will need to reset the Plasma Emergency Off button to the ON position ( ) before the plasma can be re-ignited.



Plasma Emergency Off button

- Turn off the peltier by selecting **Peltier** Off from the **Analyze** menu (accessible only when in a worksheet).

Caution
The gas control unit supplies uninterrupted gas purge to the camera (Varian 720/730-ES) and polychromator assemblies to minimize the ingress of moisture. It is strongly recommended that gas supplies be left on at all times except during long periods of non-use. Always turn off the camera Peltier before turning off the instrument gas supply.

- Switch off the mains power switch (the green light will go off). This will turn off the complete instrument, including the Peltier (if not done so manually earlier) as well as the polychromator heater.

- Loosen the peristaltic pump tubes by releasing the pressure bars and lift the tubes out of the grooves. To do this:

a. Unclip and lower the pressure bar screws leaving the pressure bars resting loosely against the rollers.

b. Lift the tubing out of the grooves so that they are not being stretched over the rollers.

- Switch off the water cooler, laboratory exhaust system and any accessories (where applicable), and shut down the gas supplies at the cylinders.
- Exit the ICP Expert II software if it is no longer required, by selecting **Exit** from the **File** menu. Switch off the printer and monitor.

13.4 Calibration

13.4.1 Calibration Standard Solutions: A mixture of two multi-element solutions. STL Westfield's calibration standards are a custom blend manufactured by Inorganic Ventures. The two combined solutions are diluted 1:100x. The LCS/LCD, MS/MSD, IPC (instrument performance check) and CCV are prepared from these two solutions.

13.4.2 Autosampler set-up

The Autosampler (A/S) page for the Varian SPS A/S is accessed from the Sequence page by clicking the **Autosampler Setup** button.

From the dialog, you can configure the racks on the A/S and set up where each solution in the worksheet will be during analysis. This page is not available in manual sampling mode.

The page consists of the following:

Rack diagram

The rack diagram gives a visual representation of the racks in the A/s. Hold the cursor over a rack to display its type and use. The currently selected rack is outlined in red. To select a different rack, click on the required rack. Alternatively, you can select the rack type from the options displayed when you right-click on the **Rack diagram**.

Key to tube colors

Each type of tube in the rack diagram is displayed in a particular color. The **Key to tube colors** indicates which sort of tube is represented by each color.

Platen type

Click the platen type you are using. Choose from the default **Standard tray** or the **Wellplate tray**.

Rack properties

Set the properties for the currently selected rack. (The currently selected rack is outlined in red.)

Type

Specify the type of rack that is currently selected. The types available vary for the different A/S.

Use

Specify what the currently selected rack will be used for in the analysis. Choose from Sample, QC/Calibration, Dilution, Calibration and QC.

Starting tube

Enter the tube position where you have inserted the first sample. Typically the initial run consist of a calibration blank, followed by the calibration standards and QC check standards.

13.5 Torch Alignment

13.5.1 The following procedure can be used for both horizontal and vertical optimization of the torch assembly. This procedure must be performed whenever the torch is removed for cleaning. Aspirate the Varian Tune solution.

Performing a torch alignment scan:

Step 1

Place the sample pump tubing from the peristaltic pump into a 5 ppm Mn tune solution.

Step 2

From the Main Index window of the ICP Expert II software, do the following:

- Click **Instrument setup** on the Window menu. The **Instrument setup** window will be opened.
- Click the **Torch align** tab.

Step 3

Ensure that the **Scan method** paths points to the Torch Alignment file.

The Torch Alignment file is called TorchAlignment.vws, and is located in the following directory on the drive on which the ICP Expert II software is installed: \Program Files\Varian\ICP Expert\Supplied Worksheets

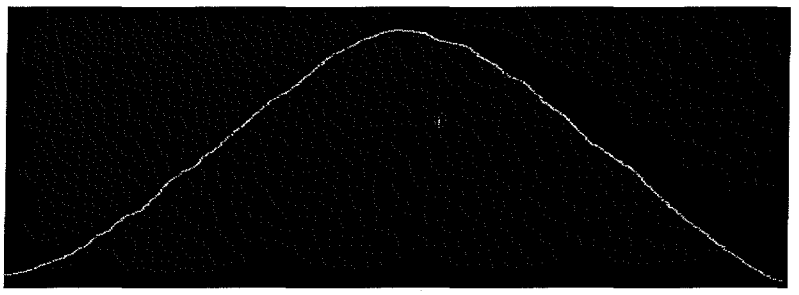
The TorchAlignment file consists of a single line, 257.610 nm Mn, that is recommended because it gives a representative viewing range scan for most elements.

Step 4

Perform a horizontal scan by clicking **Torch scan** on the **Torch align** page.

Perform a vertical torch scan **after** you have performed the horizontal scan. When the instrument scans the torch, it will move the pre-optics to the optimum positions for viewing the plasma. The optimum positions are displayed on the slide-bars.

When you perform a torch alignment scan, you should expect to see a scan result similar to the following:



14.0 PROCEDURES

14.1 Typical autosampler setup and analysis:

Wavelength calibration (both dark current and wavelength)
Calibration blk, and standards

1. IPC & ICV
2. ICB
3. CRI
4. ICSAB (required 6010)
5. ICSA (required 6010)
6. CCV
7. CCB
8. MB
9. LCS
10. LCD
11. Sample
12. Sample duplicate
13. Sample matrix spike
14. Post Digestion Spike
15. Serial Dilution
15. Sample
16. CCV
17. CCB
18-27. samples
28. CCV
29. CCB
30. ICSA
31. ICSAB

14.1.1 Dilute and reanalyze any element that exceeds the concentration range of the upper calibration curve.

14.1.2 Report the average of 2 integrations.

14.2 An Initial Demonstration of Capability (IDOC) must be made prior to using the above test method. CDOC (continuing DOC) are performed annually.

14.3 Please reference the appropriate sample preparation digestion procedure for Total and Total Recoverable metals.

15.0 CALCULATIONS

15.1 Aqueous samples.
Reported directly off the instrument printout in units of ug/L. The data is transferred and processed by the laboratory's LIMS (Library Information Management System)

15.2 Solid samples.
The concentrations determined in the digestate are to be reported in mg/kg on the dry weight basis of the sample.

$$mg/kg = \frac{C}{W} \times V \times \frac{100}{S}$$

Where:
C = Concentration (mg/L)
V = Final volume in mL after sample preparation
W = weight in g of wet sample
S = % Solids

15.3 If dilutions are performed, the appropriate factors must be applied to the sample values prior to results being reported.

16.0 METHOD PERFORMANCE

The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

17.0 POLLUTION PREVENTION

This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc)

and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented

18.2.1 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCE

22.1 "Method 6010B Inductively Coupled Plasma-Atomic Emission Spectroscopy" SW-846 Test Methods for Evaluating Solid Waste, December 1996, Revision 2.

22.2 QAM : Quality Assurance Manual for STL Westfield.

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STL WESTFIELD

STANDARD OPERATING PROCEDURES

Analytical Procedure for the Analysis of Inductively Coupled Plasma – Mass Spectrometry (ICP/MS)

SW846 6020
MADEP MCP 6020

SOP ID: MES02800.MA

Date Written: 02/01/2005
Revision Date: NA
Effective Date: 11/08/2006

Revision 0

Approved Signatures

QUALITY ASSURANCE OFFICER

LABORATORY DIRECTOR

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1.0 TITLE

Analytical Procedure for the Analysis of Inductively Coupled Plasma – Mass Spectrometry (ICP/MS)

2.0 MATRIX

This method provides procedures for the determination of a large number of elements in water samples and in waste extracts or digestates. When dissolved elements are required, the samples must be filtered, then acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion (prior to filtration and analysis) is required for groundwater, aqueous samples, industrial wastes, soils, sludge, sediments and other solid waste matrixes for which a Total ("acid-leachable") elements are required.

3.0 METHOD REPORTING LIMIT

The method-reporting limit for most elements measured by this technique is sub-µg/L (<1ppb). Typical instrument calibration curve covers a working range between 1 - 100µg/L. Sample results reported above 100µg/L are generated from a sample dilution.

4.0 SCOPE and APPLICATION

4.1 This procedure describes multi-element analysis by ICP-MS based on SW846 6020.

4.2 Method SW846 6020 lists fifteen elements approved for analysis by ICP/MS (Al, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl and Zn). MCP method 6020 lists thirteen approved elements (Sb, As, Ba, Be, Cd, Cr, Pb, Ni, Se, Ag, Tl, V and Zn).

4.3 Additional elements may be included in this procedure provided that the performance criteria presented in section 12 (Quality Control) and 16 (Method Performance) are met. However, project approval may be required from either a controlling agency or client for compliance testing beyond the elements stated in the promulgated methods.

4.4 The use of this method is restricted to spectroscopists who are knowledgeable in the recognition and the correction of spectral, chemical and physical interferences in ICP-MS.

5.0 SUMMARY

5.1 Prior to analysis, samples which require Total ("acid-leachable") results must first be digested using the appropriate sample preparation method. Well mixed aqueous samples, digestates or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into an R.F.plasma. The sample is decomposed and desolvated.

5.2 The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high vacuum chamber that houses a quadrapole mass spectrometer. The mass spectrometer is

capable of providing a resolution better than or equal to 0.9 amu peak width at 10% of the peak height for EPA 6020. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

5.3 Interferences must be addressed, assessed and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and the constituents of the sample matrix. Recommended elemental equations, which correct for many of these interferences, are listed in Table 1. Use of the internal standard technique is required by this method to compensate for suppressions and enhancements caused by sample matrices.

6.0 DEFINITIONS

6.1 Dissolved Metals- Those elements, which pass through a 0.45µm membrane filter. The sample is acidified after filtration.

6.2 Total Recoverable Metals (Method 3005A)- The concentration determined on an unfiltered sample following treatment with hot, dilute acid digestion.

6.3 Total Metals (Methods 3020A, 3050A)- The concentration determined on an unfiltered aqueous or solid sample following treatment with a hot acid digestion.

6.4 Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

6.5 Instrument Detection Limit (IDL) the minimum amount of a substance that can be measured on a specific instrument, with a specified degree of confidence that the amount is greater than zero. The IDL is associated with the instrumental portion of a specific method only, and specific sample preparation steps are not considered in its derivation. A calculated IDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The IDL thus represents a range where qualititative detection occurs on a specific instrument. Quantitative results are not produced in this range.

6.6 Reporting Limit (RL)- The level to which data is reported for a specific test method and/or sample. The reporting limit is influenced by sample volume used to prepare the sample for analysis as well as any dilution required to report the result. Reporting limits are typically set at 2 – 5x the MDL.

6.7 Sensitivity- The slope of the analytical curve: the relationship between the raw instrument signal and the concentration. However, when optimizing for sensitivity the manufacturer recommends the following specifications: Mg >20,000cps, Rh > 150, 000cps, Pb 208 >100,000 and background <30cps.

6.8 MS Tuning Solution- A multi-element solution containing analytes that are

representative of the mass range capable of being scanned by the instrument. The solution is used to optimize the sensitivity of the instrument and to verify that the mass resolution meets method criteria.

6.9 Initial Calibration Verification/Quality Control Sample (ICV)- a multi-element standard of known concentrations prepared to verify instrument calibration. The solution must be an independent standard prepared near the mid-point of the calibration curve at a concentration other than that used for calibration.

6.10 Continuing Calibration Verification (CCV)- A multi-element standard of known concentration prepared to monitor and verify the instrument's daily continuing performance.

6.11 Interference Check Standard (ICS)- A solution containing both interfering and analyte elements of known concentration that is used to verify background and interelement correction factors.

6.12 Laboratory Control Sample (LCS)- A multi-element standard of known concentration which is carried through the entire sample preparation and analysis procedure. The solution is used to verify the accuracy of the sample preparation

6.13 Preparation Blank- High purity water containing the same acid matrix as the calibration standards carried through the entire preparation process. Used to monitor preparation contamination.

6.14 Calibration Blank- High purity water acidified with the same acid concentration present in the standards and samples. Also referred to as Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

6.15 Matrix Spike (MS)- field sample to which a known amount of target analyte(s) is added.

6.16 Matrix Spike Duplicate (MSD)- a replicate matrix spike.

6.17 Matrix Duplicate (MD)- duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

6.18 Water Sample- For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, storm run-off, industrial or domestic wastewater.

6.19 Serial Dilution- A dilution of a sample by a known factor and when corrected by the dilution factor, the diluted sample should agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents within the sample matrix.

6.20 Internal Standard- An element within the same mass range as the element of interest but which is not normally found in the environment and does not interfere with the element of interest. The internal standard is added to every sample standard, blank, matrix spike, matrix spike duplicate, QC sample or sample extract at a known concentration, prior to analysis. Internal standards are used to monitor the amount of matrix interference on the sample/ element(s) of interest.

6.21 Initial Demonstration of Capability- A procedure to establish the ability to generate acceptable accuracy and precision for analysts. This is also referred to as Initial Demonstration of Proficiency and Continuing Demonstration of

Capability.

7.0 INTERFERENCES

7.1 Isobaric Interference- Isobaric interferences in the ICP/MS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (M/Z). Most interference of this type are corrected for by the instrument software.

7.2 Isobaric molecular and Doubly Charged Ion Interferences-

7.2.1 Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Table 2 lists isobaric interferences, which might possibly affect reportable elements. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, correction must be applied and the data flagged to indicate the presence of interference.

7.3 Physical Interferences – Physical interferences, caused by sample viscosity and surface tension, are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

7.3.1 Internal Standards should be added at a level to give approximately 50,000 – 800,000 counts of raw signal intensity. Target intensity is 300,000 counts. The mass of the IS should be within 50amu of the mass of the measured element.

7.3.2 Matrix effects will be monitored by comparing the IS intensity in the sample to the IS of the calibration blank. When performing field samples analysis by method 6020, the relative intensity (RI) must be between 30 – 120% of the initial calibration. QC samples must be between 80 –120%. If field samples fall outside of this range, perform a 5x (1:4) dilution to correct for matrix effect. Continue to perform a dilution until the IS criteria is met. Special note for drinking water samples: It is highly unusual for the IS to fail, primarily due to lack of matrix interferences in DW samples.

7.3.3 Memory effects are dependant on the relative concentration differences between samples and/or standards that are being analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference (high element carryover).

7.3.4 Analytical Note: Dissolved solids, deposited on the nebulizer tip and/or interface skimmers during sample processing will adversely affect instrument performance. A final total solids concentration should be below 0.2% (2000 mg/l) is recommended for an optimal system performance.

7.3.5 High salt concentrations (e.g., seawater samples) cause analyte signal suppression or enhancement, dependent upon the element. Samples with high salt content can cause both physical interference and isobaric molecular interferences

7.4 The use of Hydrochloric and Sulfuric acids should be minimized due to higher

incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, STL Westfield's Safety addendum and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The ICP RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 Argon Gas: high purity grade (99.99%)
9.2 ICP/MS capable of providing a resolution less than or equal to 0.9 AMU at 10%

peak height from 6-238 AMU and 1.0 AMU at 5% AMU at 5% peak height from 6-238 AMU with a data system that allows corrections for isobaric interferences and the application of the internal standard technique.

9.3 A multiple channel peristaltic pump.
9.4 A water chilled cooler.
9.5 Calibrated pipettes.
9.6 Auto-sampler with a/s disposable tubes, variable speed pump.
9.7 An electron multiplier detector.
9.8 Instrument computer with data capturing and instrument print capabilities.

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1 Storage and Shelf life

10.1.1 All standards must be stored in previously unused polyethylene or polypropylene containers. Standards stored at concentrations as received from an outside vendor must be replaced prior to the vendor's expiration dated provided. If no expiration date is provided, the standards may be stored up to one year from receipt. Standards must be replaced sooner if verification from an independent source indicates a problem.

10.2 The tuning solution is purchased as a custom multi-element mix. The concentrations typically used are 10µg/L of: Mg, Co, Rh, Cd, In, Ba, Co, Pb and U.

10.3 The initial calibration standards are purchased as a custom multi-element mix. The standards are diluted to working levels using dilute nitric acid. The custom mix may be either a 10 or 20mg/L solution. *Working standards should be prepared fresh daily, since concentrations can change on aging.* If not preparing fresh standards monitor the mix weekly for stability.

10.4 ICV- The ICV standards are obtained from an independent (different) source that the calibration standards. The custom mix may be either a 10 or 20mg/L solution. The ICV is prepared using dilute nitric acid.

10.5 CCV – The CCV standards are prepared from the same source as the calibration standards. The CCV standards are prepared in dilute nitric acid.

10.6 The Reporting Limit Verification standard(s) is prepared from the same stock as the calibration standards in dilute nitric acid. The RL standards must be equal to the concentration of the reporting limit.

10.7 The LCS/LCD/MS/MSD spiking solutions are made from the same stock as the calibration standards using dilute nitric acid.

10.8 Reagent Water: ASTM Type I for the elements of interest, generated from the lab's reagent water purification system capable of achieving 18.0 megohm-cm.

10.9 Nitric acid dilute (1.5%)- Carefully dilute 30ml concentration HNO3 into 2L with reagent water. This solution is used to dilute samples, calibration standards, QC samples and calibration blanks.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Aqueous (Total) samples are preserved with (1+1) nitric acid to a pH of <2 and may be stored in a pre-cleaned plastic container. Preservation must be verified

prior to analysis.

11.2 Aqueous (dissolved) samples are filtered through a 0.45um filter, prior to acid preservation.

11.3 Soil samples do not require preservation, but must be store at 4°C ± 2°C until time of preparation.

11.4 The holding time for metal samples (aqueous or soils) is 180 days from time of collection. See reference table in appendix.

12.0 QUALITY CONTROL

12.1 Ongoing data quality control checks are compared with method established performance criteria to determine if the instrument and results meet performance characteristics of the method. Control chart limits are maintained by the laboratory's LIMS and may be consulted to understand trends in QC samples, however control charts limits shall not exceed method-defined limits. Initial Demonstration of Capability (IDOC) is required prior to analyzing client's field samples. Annual MDLs (< RL) are mandatory and must be kept on file.

12.2 In instances where QC criteria are not met, a QC non-conformance may be generated to document deficiencies.

12.3 Method Blanks- One method blank must be processed per batch (20 field samples or less). The method blank results must be less than the reporting limit (RL). If a MB results does not meet this criteria, the source of the contamination should be determined and the problem corrected. The following corrective actions may be utilized when the MB does not meet criteria:

- The same analyte was not detected in the associated samples
- The MB concentration was 1/10 of the concentration found in the sample

Note: if the above criteria are not met and reanalysis is not possible, than the data must be qualified in the report's case narrative and the client notified.

12.4 LCS- One LCS must be processed with each preparation batch (note: MCP requires a LCS/LCSD be performed if a client specified MD or MSD is not utilized). The acceptance criteria for the LCS is 80-120%R for method 6020. RPD must not exceed 20% for aqueous samples and 30% for soils. The following corrective actions may be utilized when the LCS does not meet criteria:

- If the LCS exceeds these limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One exception to this rule is that if the LCS recovered above control criteria, and no target analyte was detected in the sample, then the data may be reported with proper documentation in the form of a QC non-conformance.

12.5 MS/MSD- One pair of MS/MSD or MS/MD must be processed with each QC batch. The control criteria are not to exceed 75 –125%R and 20% RPD for aqueous samples and 35%R for soils for analyte values greater than 100x the reporting limit. Corrective action when criteria are not met is to verify that the LCS was within control. If within control limits, no further action is required.

12.6 Daily Performance Standard – A 10ppb solution of elements covering the target

analytical mass range. The purpose of this standard is to check and maximize instrument sensitivity and performance. The daily check is performed using the tune solution, by the instrument's software, and must meet a control criteria on the RSDs of ≤5% (6020MCP RSDs of ≤3%) from a minimum of at least 4 replicates.

12.7 Tuning – Tuning must be performed daily prior to calibration. The tuning solution must contain elements representing all the mass regions of interest. The mass calibration criteria must be ≤ 0.1 amu difference from true, and resolution < 0.7 ±0.1 amu full width at 10% peak height with a RSD of ≤5% or re-optimization must occur.

12.8 ICV- The Initial Calibration Verification standard is a separate source check standard with is independent from the calibration standards. The mid-point of the calibration standard must recover within 90-110% of the expected value.

12.9 ICB/CCB – Initial Calibration and Continuing Calibration Blanks are matrix matched to the calibration standards and measure instrument drift and contamination. The ICB/CCB results must be < the RL. The CCB is analyzed every 10 samples.

12.10 CRI- The Calibration Reporting Limits standard measures instrument sensitivity and is analyzed daily following calibration. This standard need be analyzed only if a RL standard is not part of the calibration curve. The standard should recover within a recommended criteria of 70 – 130%.

12.11 CCV – Continuing Calibration Verification standard monitors calibration drift and accuracy. The standard, derived from the calibration standards, is analyzed every 10 samples and must recover with 90 – 110% of the expected value.

12.12 Linear Range Check Standard- For SW846 6020 MCP, this standard is performed daily at the beginning of the analytical run, only if a standard at the level of the linear range is not included in the calibration curve. The LRC standard must recover within 90-110% of expected value.

12.13 ICSA/AB – Interference Check Standard A & AB monitors the adequacy of isobaric interference corrections and must be analyze at the beginning of the analytical run or every 12 hours, whichever is more frequent. The % recovery should fall within 80 – 120% of the spiked values. There is no corrective action required for this standard because instrument corrections are based on natural isotope abundances that cannot be changed. If elements fall outside of these criteria, case narrate non-compliances.

12.14 Dilution Test – One dilution test is require per 20 field samples only if the analyte concentration is > 100x the RL. The dilution test measures the effect from sample matrix. Perform a 5x dilution and the difference between the undiluted sample and DF corrected sample should be ± 10% (6020MCP ± 15%). No corrective action is required for out of control situations.

12.15 IS – Internal Standards measure the effect of the sample matrix. The IS is added to each field sample and QC sample. For SW846 6020 and MCP, the IS must recover between 30-120% of the relative intensity (RI) of the calibration blank in field samples and 80-120% in QC samples. If outside of limits, reanalyze the calibration blank to confirm that the instrument has not drifted out of control. If criteria met, dilute samples 2x and re-analyze. Samples must be diluted until IS control criteria is met.

12.16 PDS- Post digested Spike addition for method SW846 6020 (not MCP) is performed for each QC batch of samples. An analytical spike is added to a portion of a prepared sample (or its dilution) and should recover between 75 – 125%R of known valve. If the PDS fails to meet this criterion, matrix interference should be suspected.

12.17 QC CRITERIA SUMMARY TABLE

QC Analysis	Frequency	QC Criteria	Corrective Action
Daily Performance Standard	Daily	≤5% 6020 6020MCP RSDs of ≤3%)	Perform instrument maintenance and rerun standard.
Tuning	Daily	0.7 ± 0.1amu	Re-optimize
Initial Calibration	Following tuning and prior to sample analysis [Minimum of blk, plus (3) non blk stds]	r = ≥ 0.995	Re-calibrate
ICB (Initial Calibration Blank)	Once at start of analytical run	< RL	Recalibrate. Re-check ICB; if still unacceptable, investigate possible contamination of reagents, reagent water.
ICV (Initial Calibration Verification)	Once at start of analytical run	90-110%	Recalibrate. Re-check ICV; if still unacceptable, investigate possible calibration std or ICV out of specification.
Reporting Limit Calibration Check Standard- CRI	Daily, if not part of calibration curve	70-130% - 6020 MCP	Recalibrate, re-analyze
LCS	Once per batch of 20 Samples or fraction thereof	80-120%	If unacceptable, re-analyze; if still out recalibrate or re-digest sample batch.
LCD	Once per batch of 20 Samples or fraction thereof	80-120%R, RPD ≤20 aqueous, ≤30% soils.	If unacceptable, re-analyze; if still out recalibrate or re-digest sample batch.
	Every 20 samples	RPD ≤20%	If RPD is out re-analyze. If still out

Sample Duplicate MD (Client specific for 6020 MCP)	or fraction thereof		of acceptable limits, report data with qualification.
Matrix Spike MS (Client specific for 6020 MCP)	Every 20 samples or fraction thereof for 6020	75-125%- Every batch	If unacceptable, review LCS. If LCS acceptable, no corrective action require- matrix effect suspected.
Matrix Spike Duplicate MSD (Client specific for 6020 MCP)	Every 20 samples or fraction thereof for 6020	75-125%R, RPD ≤20% aqueous, ≤35% soils.	If unacceptable, review LCS. If LCS acceptable, no corrective action require- matrix effect suspected.
CCV (Continuing Calibration Verification)	Every 10 samples or fraction thereof	90-110%	If unacceptable, re-analyze; if still out recalibrate. Sample values for blocks between acceptable CCVs may be reported; those that are not must be rerun.
Internal Standards IS	IS added to all QC and field samples	80-120% QC and 30-120% for field samples	Check CCB , if w/in limits perform dilution and re-analyze
Dilution Test	Every Batch of 20 field samples	Dilute 5x ±10% for analytes > 100 RL (6020MCP - ± 15%	None

CCB (Continuing Calibration Blank)	Every 10 samples or fraction thereof	< RL	If unacceptable, re-analyze; if still out recalibrate. Sample values for blocks between acceptable CCB may be reported; those that are not must also be rerun.
Linear Range Check	Only if not part of calibration curve	90-110% of true value	Re-analyze, re-optimize if still outside of control limits.
ICSA/AB	Daily at beginning of analytical run and every 12 hours whichever is more frequent	80-120% (6020)	No action required.
Method Blank MB	One per batch of 20 field samples	<RL	Re-analyzed, Re-digest

13.0 CALIBRATION and STANDARDIZATION

13.1 Instrument Startup ELAN 6100

13.1.1 Switch on the PC (the monitor, printer and autosampler are usually left on).

13.1.2 Once the Windows NT log-in dialog box appears type in your username and password to enter the Windows NT program manager.

13.1.3 Using the mouse, start the ELAN software via the following path: click on the Elan icon from the desktop.

13.1.4 Before igniting the plasma, check the condition of the sampler and skimmer cones, the plasma torch and the RF load coil, and ensure that the argon cylinder contains a sufficient amount of argon.

13.1.5 Ensure that the pump rollers of the peristaltic pump are clean and able to move freely.

13.1.6 Connect the pump tubing (both of the thin sample tubing and the thicker drain tubing) on the peristaltic pump, making sure that the flow of sample and waste will be in the correct direction. If the pump tubing is new, stretch it gently before attaching. If the pump tubing has been used previously, check it for flat spots, discoloration, brittleness and clogging. The pump tubing must be replaced regularly.

13.1.7 Replace the tubing clamps for each pump channel and swing the cam arm to close and apply tension to the clamps.

13.1.8. Place the sample capillary tube into a container of ~1.5 % HNO_3 rinse wash solution.

13.1.9 Click on the *Instrument icon* and then, if necessary, on the *Front Panel*

button to open the ELAN control panel. The System Status indicator should indicate "*Ready*" if all of the system hardware components are operating properly. If the System Status indicator says "*Not Ready*", click on the *Diagnostics button* to determine the cause of the problem.

13.1.10 Make sure the extractor fan is on. If this is not done, the instrument will overheat and shut itself down within a few minutes.

13.1.11 Switch on the peristaltic pump as follows: Click on the *Devices icon*. Click on the *Connect button*. Click on the *arrow button*. Check that the solution is flowing properly to the nebulizer and spray chamber, as well as to the waste container. Starting the pump before igniting the plasma fills the nebulizer and plasma torch with water vapor, thus removing any air pockets and ensuring a good first-time plasma ignition, which avoids puttering of the RF load coil onto the plasma torch.

13.1.12 To ignite the plasma, click on the *Plasma Start button* on the instrument control panel. The progress of the ignition sequence, which requires a little over a minute to complete, is depicted in the Ignition Sequence status bar. If the instrument has not been used for an extended period of time (i.e. more than two days), or if the nebulizer and/or spray chamber and/or plasma torch have been replaced, the ignition process should be stopped after about 75 % of the ignition sequence has been completed by clicking on the *Plasma Stop button*. After waiting for 15 seconds, click on the *Plasma Start button* again to ignite the plasma. This procedure allows for removal of excess air and improves plasma ignition.

13.1.13 Allow the instrument to warm up for approximately 30 minutes. The torch box temperature should reach about 44°C. Optimization can proceed while the instrument's warming up.

13.2 Instrument Shut Down

13.2.1 Before shutting down the instrument, aspirate ~1.5 % HNO_3 wash solution for 15 minutes to clean out the sample introduction system.

13.2.2 Remove the sample capillary tube or autosampler probe from the ~1.5 % HNO_3 wash solution.

13.2.3 Click on the *Devices icon* and then click on the *Stop button* to stop the peristaltic pump.

13.2.4 Click on the *Instrument icon* and then click on the *Plasma Stop button*.

13.2.5 Release the peristaltic pump tubing (both the sample, IS, drain and autosampler rinse tubing).

13.2.6. Close all ELAN software windows and the ELAN software itself. Then power off/shut down the PC.

13.3 Daily Optimization -The following optimizations procedures should be performed daily.

- Gas flow optimization – perform this software optimization. The curve should increase, attain a maximum point, then decrease. Select the maximum point of

signal. To reduce oxides, follow the operator's manual. This generally involves reducing the gas flow to lower the oxide ratios.

- Perform Lens Voltage Calibration- performed by the instrument's software, use the optimization solution, then automatically set the lens voltage to the optimum value when the procedure is complete. Voltage approaching "10", is indicative of the lens needing cleaning.
- Perform Auto Lens Calibration – performed by instrument software, use the optimization solution, then automatically set the lens voltage to the optimum value when the procedure is complete.
- X-Y Adjustment- the X-Y adjustment needs to be performed whenever the sampler or skimmer cones are cleaned or changed, or whenever the plasma torch has been removed or its configuration altered. Using the optimization solution, rotate the X and Y adjustment knobs to obtain the positions resulting in the maximum signal intensities. This seems to work best if the Y knob is done first. Alternately adjust the "X" and "Y" knobs until maximum intensity is achieved.

13.4 Perform Instrument Tune/ Mass Calibration Tune the instrument with the tuning solution containing elements representing all the mass regions of interests. The Mass Calibration. The mass calibration criteria must be ≤ 0.1amu difference from true, and resolution < 0.7amu full width at 10% peak height with a RSD of ≤5% or re-optimization must occur. Adjust mass calibration if it has shifted by more than 0.1amu from unit mass.

13.5 Perform Daily Performance Check

13.5.1 Frequency: Daily, after instrument mass calibration.

13.5.2 Daily performance standard should be a 10 µg/L standard of 3 or more elements representative of the analytical mass range. Its purpose is to check sample introduction; sensitivity; oxide and double charge interferences. This is a multiple check on instrument performance suggested by the manufacturers.

Manufacturer's recommended criterion*:

Background < 30cps	
Rh Sensitivity >150,000 cps	
Mg Sensitivity > 20,000 cps	CeO/Ce ≤ 3% (MCP Requirement)
Pb Sensitivity >100,000 cps	Ba++/Ba+ ≤ 3% (MCP Requirement)
Net intensities < 5% RPD	

*Poor sensitivity or gradual loss of sensitivity may be indicative that the sample and skimmer cones requiring cleaning or replacement. Cones can be cleansed with a 1% nitric acid wash or sonicated in a hot water bath for 5 minutes. Air dry before re-installing.

13.5.3 If Daily Performance Standard exceeds 5% criterion (<3% 6020MCP); perform instrument maintenance and re-run standard. If SW846 6020 MCP data are reported from an ICP-MS run in which the Daily

Performance Standard exceeded 3%, lab must narrate why the data are considered valid.

13.5 Initial Calibration. Calibrate the ICP/MS daily with a minimum of a blank and three non-blank standards, which should include a RL and LR standard. Perform triplicate integrations, with the average result to be reported.

13.6 Analyze the ICV solution immediately after initial calibration. Acceptance criteria is 90-110% of known value. Spike the solution near mid-range of the analytical curve (~50ppb).

13.7 Analyze ICB. Results must be < RL.

13.8 Analyze CRI (if not part of cal curve), LR (if not part of cal curve), ICSA/AB followed by CCV and CCB.

13.9 Analyze Method blank, LCS, LCSD, Samples, MS, MSD, MD.

13.10 Analyze Serial Dilution (1 per 20 field samples, 1:5X.

13.11 Analyze CCV, CCB every 10 samples and at close of analytical run.

13.12 Once a year, analyze a CCV at a different concentration than what's normally prepared. This is a NELAC requirement.

14.0 PROCEDURE

14.1 Preparation of Samples- Dissolved and Drinking water samples may be analyzed as received. Samples requiring Total aqueous analysis and Soils/sediments are digested prior to analysis.

14.2 Analysis of Samples- Report the average of three integrations. Flush the system with a rinse blank between samples and standards during the analytical run.

14.3 Dilute and reanalyze any element that exceeds the concentration range of the upper calibration curve.

15.0 CALCULATION

15.1 Data Calculations

ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \frac{\text{(Found ICV)}}{\text{(True ICV)}}$$

CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \frac{(\text{Found CCV})}{(\text{True CCV})}$$

The final concentration for an aqueous sample is calculated as follows:

$$\text{Result } (\mu g/L) = \frac{(C \times V1 \times D)}{V2}$$

Where:

C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

The final concentration for an solid sample is calculated as follows:

$$\text{Result } (\mu g/Kg) = \frac{(C \times V \times D)}{W}$$

Where:

C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in grams (g), of wet sample digested.

LIMS calculates final concentration on a "dry-weight" basis.

Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \frac{(SSR - SR)}{SA}$$

Where:

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

Note – When sample concentration is less than the detection limit, use SR = 0 for the purposes of calculating %R.

The Relative Percent Difference (RPD) of a sample duplicate is calculated according to the following equation:

$$RPD = 100 \times \frac{|(DU1 - DU2)|}{(DU1 + DU2) / 2}$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing client samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a thorough review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include the generation of a job case narrative which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the STL Westfield's facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCES

22.1 SW846 6020 ICP/MS, USEPA, Revision 0, September 1994
22.2 SW846 6020A ICP/MS, MADEP WSC-CAM, Revision No. 2, August 13, 2004.

Appendix

TABLE I: Recommended Elemental Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

M = Total ion count rate at the specified mass.

TABLE 2

Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS,SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

Note: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

TABLE 3

Tuning Solution

Method 6020

Element	Concentration (µg/L)
Solution A	
Mg	10.
Rh	10.
Pb	10.

Recommended Internal Standards

Method 6020	Method 200.8
Li	Sc
Sc	Y
Y	In
Rh	Tb
In	Bi
Tb	
Ho	
Bi	
Ge	

TABLE 4

Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. Suggested masses for method 200.8 are in "quotes".

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"**205**", 203	Thallium
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
72	Germanium
139	Lanthanum
118	Tin
35, 37	Chlorine
"98", 96, 92, **97**, 94	Molybdenum

Note: It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

TABLE 5

Sample Preservation, Container and Analytical Holding Time Specifications for Surface Water, Groundwater, Soil and Sediment Samples Analyzed for Metals by ICP-MS

Matrix	Sample Container(s) [1]	Preservative	Holding Time [2]
Total Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for Total Metals	HNO_3 to pH < 2,	180 days: all metals except mercury 28 days: mercury
Dissolved Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for field-filtered sample for Dissolved Metals	Filter (0.45 µm) on site; or at the laboratory ***(prior to acid preservation)*** within 24 hours of collection HNO_3 to pH <2,	180 days: all metals except mercury 28 days: mercury
Suspended Metals Groundwater and Surface Water	Submit Suspended Solids on Filter to Laboratory	Filter on site Filter 100 – 500 ml of unpreserved sample	180 days: all metals except mercury 28 days: mercury
Soils and Sediments	(1) 4-ounce glass jar	Cool, 4°C	180 days: all metals except mercury 28 days: mercury
Concentrated Waste Samples	125 mL wide mouth glass or plastic	Cool to 4°C	180 days: all metals except mercury 28 days: mercury

1 The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised.
2 From date of sample collection.

Data ICPMS Analysis Guidelines Attached

Data Review Checklist Attached

SEVERN TRENT STL

STL Westfield
ICP-MS Data Review Checklist
Level I and Level II

Instrument ID: **P/E Elan 6100**	Method 200.8 / 6020 SOP: MES 02700.MA 200.8 MES 02800.MA 6020
Date:	Analyst:

The calibration curve is generated using a blank and 3 non-blk calibration points, with 3 integrations and a linear curve fit with an "r" of ≥ 0.995.	YES	NO	NA
1. Gas flow optimization, Len Calibration, Tune, Daily Performance passed and attached?			
2. ICVs & CCVs within 10% of true value or recal and rerun?			
3. ICB & CCBs < reporting limit (RL) or recal and rerun?			
4. CRI analyzed at beginning of run and at RL? (6020: 70-130%)			
5. ICSA/AB analyzed at beginning of run?(6020: 80-120% known, <RL unkn)			
6. 10 samples or less analyzed between CCV/CCB?			
7. All parameters within linear range?			
8. LCS/LSD within limits? (200.8: 85-115%, 6020: 80-120%, RSD ≤20%)			
9. Method Prep Blank value < reporting limit or all samples >10x blank?			
10. Internal standard intensities for samples (unless followed by dilution) are within criteria of the Calibration Blank intensities and date/initialed? (200.8: 60-125%, 6020: 80-120% qc, 30-120% field samples)			
11. Appropriate dilution factors applied to data?			
12. Matrix spike (MS), spike dup (MSD) and or sample dupe (MD) within defined limits? (200.8 70 -130% every 10 samples, 6020: 75-125%, every 20 samples, RSD ≤20%)			
13. Serial dilution performed for 6020 MCP? (1:5X, diff= +/- 15%)			
14. QC memos and / or **MCP Checklists generated if needed?**			

COMMENTS: _____

_____

Calibration Std Manufacturer, Lot # and Expiration date: _____

ICV Manufacturer, Lot # and Expiration date: _____

MS/MSD Manufacturer, Lot # and Expiration date: _____

DATA ENTERED BY:	DATA REVIEWED BY:
DATE:	DATE:

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STL WESTFIELD

STANDARD OPERATING PROCEDURE

Mercury in Liquid / Solid Waste (Manual Cold-Vapor Technique) Method 7470A/7471A

SOP Number: MES00400.MA

Date Written: June 6, 1991
Revision Date: 04/10/06
Effective Date: 05/12/06

Revision (3)

Approved Signatures and Dates

Christine Furcinito-Reynolds 5-11-06

QUALITY ASSURANCE COORDINATOR

5.12.06

LABORATORY DIRECTOR

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1.0 TITLE

Mercury in Liquid / Solid Waste (Manual Cold-Vapor Technique) Method 7470A/7471A

2.0 MATRIX

Aqueous wastes, ground waters, surface waters, soil, sediments, sludge-like materials and mobility procedure extracts.

3.0 METHOD REPORTING LIMIT

0.2 µg/L for aqueous samples and 0.04mg/Kg for soil matrixes.

4.0 SCOPE AND APPLICATION

4.1 This Method (7470A/7471A) is applicable to the analysis of Mercury in aqueous wastes, ground waters, surface waters, soil, sediments, sludge-like materials and mobility procedure extracts. In addition to inorganic forms of mercury, most forms of organo-mercury may also be broken down into mercuric ions and analyzed by this method.

5.0 SUMMARY

5.1 Method 7470A/71A, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

6.0 DEFINITIONS

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Calibration: The process of comparing the response of an instrument to a standard over some measurement range.

Calibration Curve: A curve that plots the concentration of known analyte standards against the instrument response to the analyte.

Continuing Calibration Verification (CCV): A standard used to verify the continuing calibration of the instrument.

Continuing Calibration Blank (CCB): a blank matrix that is introduced onto the instrument for analysis and can monitor calibration baseline drift and carryover contamination effects.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Initial Calibration Verification: analytical standard, from a source other than that used to prepare the instrument calibration standards, analyzed immediately after the instrument calibration to verify the preparation and concentration of the instrument calibration standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD is a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Mercury : a heavy silver-white poisonous metallic element that is the only elemental liquid at ordinary temperatures and freezes into a soft solid at about -39° C. and is used especially in scientific instruments. Also called quicksilver, mercury is a metal of Group IIb of the periodic table. The compounds of mercury are either of +1 or +2 oxidation state. Mercury(II) or mercuric compounds predominate.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as, Limit of Detection (LOD). (40 CFR , Part 136, Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Summarized in either a QC memo or a Non Conformance memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is usually set at a value 2-5x the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

7.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

7.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and

			permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 Mercury Autoanalyzer: Model PS200, Leeman Lab
9.2 Computer: Compatible with Leeman Labs software.
9.3 Printer
9.4 Compressed Nitrogen or Argon: minimum of 60psi, Air products.
9.5 Water bath: Capable of achieving 95°C.
9.6 Test tubes: Fisher, 16X100 mm, 25 x 150mm
9.7 Class A volumetric flasks, assorted sizes: Fisher.
9.8 Class A volumetric pipets, assorted sizes: Fisher.

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1. Preparation Reagents and Solutions
10.1.1 Laboratory Pure Water: Distilled water system.
10.1.2 Nitric Acid (68-71%): Fisher, Trace metal grade
10.1.3 Sulfuric Acid (95-98%): Fisher, Trace metal grade
10.1.4 Potassium permanganate, crystal: Fisher
10.1.5 Potassium persulfate: Fisher
10.1.6 Sodium Chloride: Fisher, ACS grade
10.1.7 Hydroxylamine Sulfate: Fisher, ACS grade
10.1.8 Mercury Stock Standard Solution: Fisher, SPEX Plasma Grade 1000mg/L or equivalent.
10.1.9 Quality Control Check Sample (QCS/ICV): 1000 mg/L Mercury Stock Standard Solution, Alfa, Johnson & Mathey or equivalent.
10.1.10 Potassium permanganate 5% solution - Dissolve 50g of potassium permanganate crystal in 1000mL lab water.
10.1.11 Potassium persulfate 5% solution - Dissolve 50g of potassium persulfate in 1000mL lab water.
10.1.12 Sodium chloride - hydroxylamine sulfate solution - Dissolve 60g of sodium chloride and 60g hydroxylamine sulfate in lab water and dilute to 500mL.
10.1.13 Stannous sulfate: Add 25 g stannous sulfate to 250 mL of 0.5 N H_2SO_4. This mixture is a suspension and should be stirred continuously during use. (Stannous chloride may be used in place of stannous sulfate.)
10.1.14 Sodium chloride-hydroxylamine sulfate solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in reagent water and dilute to 100 mL. (Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.)

10.2 Stock Standards Solutions*

Calibration Standard Solution Concentration (mg/L)	Stock Standard Solution Concentration (mg/L)	Volume of Stock Standard Solution (ml)	Final Volume Lab water (ml)
25	1000	5.0	200
0.5	25	5.0	250
0.1	25	1.0	250

* Prepare QCS and Calibration Standards using this table. Add 0.15mL HNO_3 per 100mL lab water for each.

10.2.1 Mercury working standard: Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1ug per mL. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at pH <2 with nitric acid. This acid should be added to the flask, as needed, before addition of the aliquot.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Plastic and glass containers are both suitable for collection.

11.2 Aqueous samples must be acidified to a pH <2 with HNO3. If sample is to be dissolved it must be filtered prior to preserving with nitric acid. The maximum holding times for mercury is 28 days.

11.3 Non-aqueous samples shall be refrigerated to 4°C.

12.0 QUALITY CONTROL

12.1 A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are prepped and analyzed one per batch on a 5% frequency, whichever is more frequent.

12.2 A Method Blank (MB) is prepped and analyzed one per batch.

12.3 A Laboratory Control Standard (LCS) and LCD are prepped and analyzed one per batch.

12.4 The calibration curve correlation coefficient "r" must be ≥ 0.995. The calibration curve is made up of the following standards; 0.0, 0.2, 0.5, 1.0, 5.0 and 10.0 ppb.

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Initial Calibration	Daily	Corr. Coeff (r) ≥ 0.995	Recalibrate the system, check system, formulations. If still out of control, remake standards and recalibrate.
Initial Calibration Verification (ICV)	Immediately after calibration.	90-110 % recovery	Re-run ICV, If still out, recalibrate.
Continuing Calibration Verification (CCV)	Every 10 samples	80-120% recovery	Re-analyze, if out of control, remake standard and re-run. If still out re-calibrate and reanalyze samples not bracketed by passing CCV.
Continuing Calibration Blank (CCB)	Every 10 samples Immediately after CCV.	< RL	Check calibration, reagents and system, re-analyze. . If still out re-calibrate and reanalyze samples not bracketed by passing CCB.
Matrix Spike (MS)/ MS Duplicate (MSD)	One per batch of 20 samples	75-125%	Rerun MS/MSD, if needed. QC outliers may be reported on the final report or case narrate.
Method Duplicate (MD)	One per batch of 20 samples as MS/MSD or Duplicate.	Lab Limits RPD ≤20 %	Rerun MD, if needed. QC outliers may be reported on the final report or case narrate.
Method Blank (MB)	One per batch of 20 samples	< RL	Re-analyze, if out of control, QC outliers may be reported on the final report or case narrate. Consult with supervisor and/or QC department.
LCS/LCD	Per batch of 20 samples	80-120%	Re-analyze, if out of control, QC outliers may be reported on the final report or case narrate. Consult with supervisor and/or QC department.

13.0 CALIBRATION and STANDARDIZATION

13.1 Calibration Standard Solutions

Calibration Standard Solution Concentration (ug/L)	Working Standard Solution Concentration (mg/L)	Working Standard Solution Volume (ml)	Final Volume Lab water (ml)
0.2	0.1 mg/L	0.2	100
0.5	0.1 mg/L	0.5	100
1	0.1 mg/L	1	100
5	0.1 mg/L	5	100
10	0.1 mg/L	10	100
5 (ICV)	0.1 mg/L	5	100

13.2 Instrument Settings

13.2.1 Sample uptake time approximately 30 seconds
13.2.2 Sample analysis time is approximately 20 seconds
13.2.2 Sample rinse time approximately 70 seconds
13.2.3 Total analysis time is approximately 2 minutes per sample for 2 replicate readings

13.3 Instrument Startup for

13.3.1 Switch on the PC
13.3.2 Open carry gas tank valve, connect peristaltic tubing, insert sipper
13.3.3 Open software
13.3.4 Select and turn on system pump and gas flow in software
13.3.5 Open new data set and set date/time
13.3.6 Adjust Hg lamp optimization in software
13.3.7. Enter standard and sample information in software
13.3.8. Allow 15 minutes for warm-up
13.3.10 Select auto-run icon on software to begin analysis

13.4 Instrument Shut Down

13.4.1 Before shutting down the instrument, uncap stannous chloride from container. Allow to rinse for 15 minutes

13.4.2 Remove sample sipper tube and allow instrument to rinse

13.4.3 Discard standards and samples in auto-sampler tray

13.4.4 Unclamp peristaltic tubing from pump

13.4.5 Turn pump and gas off in software

12.4.5 Close carry gas valve

12.4.6 Close software

13.5 Daily Optimization -The following optimizations procedures should be performed daily:

13.4.1 Gas flow optimization – set at 0.60 LPM

13.4.2 Pump Flow – set at 5mL/min

13.4.3 Adjust Hg lamp for optimal intensity

13.4.4 Inspect and replace as necessary peristaltic pump tubing

13.6 Initial Calibration.

13.6.1 Calibrate the PS200 daily with a calibration blank and a 5 point calibration curve.

13.6.2 Perform two replicates, with a linear curve result to be $r \geq 0.995$.

13.7 Once a year, analyze a CCV at a different concentration level than what's normally prepared. This is a NELAC requirement.

14.0 PROCEDURE

14.1 Preparation of Aqueous Samples

14.1.1 Transfer 10mLs of the samples to 16x100 mm test tubes.

14.1.2 Add 10mLs of lab water to a 16x100 mm test tubes for the digest blank, and LCS and LCD.

14.1.3 LCS, LCD, MS, MSD Samples: add working standard solution to produce a final concentration of 5ppb.

14.1.4 Add 0.5mLs of sulfuric acid and 0.25mLs nitric acid, mixing after each addition, to each test tube.

14.1.5 Add 1.5mLs of 5% potassium permanganate solution to each test tube. Samples with high organic content typically require additional permanganate. Purple color should persist for at least 15 minutes. If purple color does not persist for at least 15 minutes a dilution of the sample may be necessary.

14.1.6 Add 0.8mLs of 5% potassium persulfate solution to each test tube, cover and heat in water bath at 95°C for 2 hours.

14.1.7 Cool the samples and add 0.6mLs sodium chloride-hydroxylamine sulfate solution to each test tube to reduce excess permanganate. **Do not** decolorize until the samples are ready to be analyzed. Sample is ready for analysis.

14.2 Preparation of Solid Samples

14.2.1 Weigh 0.2 grams for each sample. Transfer to a 25-150 mm test tube.

14.2.2 MB, LCS and LCD use Teflon chips for a "solid" matrix

14.2.3 MS/MSD Samples: Weigh 2 additional replicates of 0.2 grams. Transfer to separate test tubes. Spike appropriately for a 5ppb final concentration (before dilution factor applied)

14.2.4 Add 1ml of DI water to each test tube.

14.2.5 Add 3.75 ml of concentrated HCl to test tube.

14.2.6 Add 1.25 ml of concentrated $HN0_3$ to test tube.

14.2.7 Heat samples in a water bath at 95°C for 2 minutes. Allow to cool.

14.2.8 Slowly add 15ml of 5% potassium permanganate solution to each.

14.2.9 Cover each test tube with a cap having vent holes cut in to them.

14.2.10 Place samples in a water bath and heat at 95°C for 30 minutes.

14.2.11 Remove samples from the water bath and cool.

14.2.12 Just prior to analysis, add 6ml of Sodium Chloride-Hydroxylamine sulfate solution to reduce the excess permanganate. DO NOT decolorize until they are ready to be analyzed.

15.0 CALCULATION

15.1 All results are quantitated from the calibration curve.

15.2 For soils samples, the sample results need to be multiplied by x0.7326 before performing any calculations. This correction factor represents the difference in sample volume between the aqueous calibration standards and the volume used in preparing the soil samples.

15.3 Sample results are recorded in the laboratory LIMS system

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed

correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCE

22.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods; SW846 USEPA, September 1986 Method 7470A/7471A

23.0 TABLES, DIAGRAMS, FLOWCHARTS VALIDATION DATA

None Available

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STANDARD OPERATING PROCEDURE

Analysis of Volatile Organic Compounds

Method EPA SW-8260B

SOP Number: MSS00104.MA
Rev 5

Date Written: 09/07/95
Date Revised: 10/12/06
Effective: 10/18/06

Approved Signatures

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QUALITY ASSURANCE COORDINATOR

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1.0 TITLE

Analysis of Volatile Organic Compounds by EPA SW-846 Method 8260B

2.0 MATRIX

This method is used to determine volatile organic compounds in ground and surface water, aqueous sludge, wastes, soils, sediment and air samples.

3.0 METHOD REPORTING LIMIT

Generally 1 – 2 µg/L for aqueous, 2.5 µg/Kg low-level soils and 125 – 250 µg/Kg for high-level soil samples

4.0 SCOPE and APPLICATION

3.1 This method is utilized for the detection and measurement of purgeable organics and is applicable to various water matrixes, soil, sludge and wastes. This method is suitable for a working concentration range from 1.0 to 80 µg/L.

5.0 SUMMARY

5.1 The sample is analyzed for Volatile Organic Compounds by purging helium gas through the sample and concentrating the volatile compounds onto an appropriate trapping material. The resulting concentrated sample is then desorbed from the trap and analyzed using GC/MS instrumentation. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of calibration standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

6.0 DEFINITIONS

6.1 GC/MS – Gas Chromatograph/mass spectrometer: an analytical system complete with a temperature programmable GC suitable for splitless injection with appropriate interface for sample introduction and a MS capable of scanning from 35 – 300 amu every 2 seconds or less. The

system includes all required accessories, including an autosampler, concentrator, analytical column and carrier gas.

6.2 Calibration Standard - a fresh mixed set of stock standards used to generate the calibration curve.

6.3 Calibration Verification Standard – a standard prepared near the mid-point of the initial calibration curve (usually prepared at 20µg/L) and may be made from the same stock standard used to make the calibration curve.

6.4 Initial Calibration Verification – a stock set of standards prepared from a second source (different) of standards that was used to prepare the calibration curve.

6.5 Surrogate – an organic compound, which is similar to the target compound(s), in chemical composition and behavior, but not normally found in environmental samples. These compounds are used to evaluate analytical efficiency by measuring compound recovery. Poor recovery may indicate a problem with the sample composition (matrix interference).

6.6 Internal Standard – an organic compound, which is similar to the target compound(s), in chemical composition and behavior, but not normally found in environmental samples. .I.S is added to every sample, including method blanks, samples, QC samples and standards. I.S. response outside of established limits will adversely affect the quantitation and final concentration of target compounds.

6.7 Matrix Spike – an aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds (target analytes) and subjected to the entire analytical process. Poor spike recovery may indicate a problem with the sample composition (matrix interference).

6.8 Laboratory Control Sample – an aliquot of analyte-free reagent water fortified (spiked) with known quantities of specific compounds (target analytes) and subjected to the entire analytical process in order to indicate the appropriateness of the method efficiency.

6.9 Method Blank – an artificial sample of analyte-free reagent water designed to monitor the introduction of contaminates into the analytical process.

6.10 Holding Time - the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

6.11 Reporting Limit (RL) - The level to which data is reported for a specific test method and/or sample. The RL is generally related to the Quanitation Limit. The RL must be minimally at or above the MDL.

6.12 Method Detection Limit (MDL) - the minimum concentration of a substance that can be measured with a specified degree of confidence

that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. (40 CFR Part 136 Appendix B).

6.13 Trip Blank - a blank matrix placed in a sealed container at the laboratory that is shipped and held unopened in the field and returned to the laboratory in the shipping container with the field samples.

7.0 INTERFERENCES

7.1 Major contamination sources are volatile contaminants that are present in the laboratory environment such as Methylene chloride, acetone and other solvents. To address this issue, the Instrumentation Lab is equipped with it's own HVAC system and is maintained at a positive pressure compared with the surrounding rooms to minimize solvent contamination. Reagent blanks are analyzed with each run to determine contamination from laboratory sources. Blank subtraction cannot be used.

7.2 Impurities in the purging gas or the sorbent trap can be a source of contamination. Only chromatography grade gasses are used. All carrier gas lines are made of copper or stainless steel tubing to prevent the permeation of Methylene chloride of plastic tubing.

7.3 To prevent other sources of contamination, only PTFE sealants or devices that could come into contact with the standards or samples shall be used.

7.4 Interfering contamination can occur from carryover from preceding samples that contain a high concentration of a target analyte. Suspected high concentration samples should be analyzed at a dilution. If carry-over occurs, one or more reagent blanks should be analyzed between samples.

7.5 Samples may also be contaminated through the PTFE seals on the vials. Trip blanks (when received) are analyzed with each set of samples to determine if the samples have been contaminated through absorption.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

8.3 Proper precautions should be taken when formulating new standards, or handling suspected high concentration samples.

8.4 Procedures shall be carried out in a manner that protects the health and safety of all STL employees.

8.5 Eye protection that satisfies ANSI Z87.1, a laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be put on immediately. Appropriate gloves for the inorganic department and sample receiving shall be made of 100% powder-free, non latex and composed of Nitrile.

8.6 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

8.7 Exposure to chemicals must be maintained as low as reasonably achievable therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

8.8 The preparation of standards and reagents should be conducted in a fume hood with the sash closed as far as the operation will permit.

8.9 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL employee. The situation must be reported **immediately** to a laboratory supervisor.

9.0 APPARATUS

9.1 Hewlett Packard 5890 Series II Gas Chromatograph or Agilent 6890.

9.2 Hewlett Packard 5972 B Mass Spectrometer or Agilent 5973.

9.3 Hewlett Packard Enviroquant data acquisition Software.

9.4 Restek RTX VMS 30 m, 0.25 mm ID analytical column.

9.5 Tekmar 3000 or 2000 purge and trap concentrator or OI 4560 P+T or OI 4660.

9.6 OI 4551-A

9.7 Archon OI autosampler (4552)

9.8 40 mL vials pre-cleaned and tested.

9.9 Hamilton syringes of various volumes.

9.10 Miscellaneous Class A volumetric glassware for the preparation of reagents/standards, and diluting of samples.

9.11 Data collection system (computer), with printer.

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1 Reagent grade or better chemicals shall be used in all tests.

10.2 ASTM Type II reagent water; purged with helium to remove any volatiles.

10.3 Methanol, CH_3OH (MeOH) - Purge and trap grade or equivalent, from Fisher Scientific. Demonstrated to be free of analytes. Store apart from other solvents.

10.4 1:1 Hydrochloric acid (HCl) - Add a measured volume of concentrated HCl to an equal volume of organic free distilled water.

10.5 Primary Stock Standard Solutions - Are purchased as certified solutions using the following commercial sources. Multiple vendors are listed in Section 13.0 to facilitate replacement in out-of-stock conditions. The preferred standard formulation approach is to utilize a liquid, gases, & MTBE concentrate (200mg/L). A set of standards from two separate vendors or Multiple lot number are acceptable to comply with this SOP.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Water samples are collected in 40 mL glass VOA vials with Teflon septum caps in quadruplicate. Soil samples are collected either in 40 mL VOA vials or 4 oz. wide mouth glass containers with septum-lined caps in duplicate for old style analysis. Method 5035 requires 10g into 10 mL (1:1) of MeOH for high-level analysis and 5 g into 5mL of 20% solution of Na_2SO_4 for low-level analysis.

11.2 VOA vials for water samples are preserved with 1:1 HCl, pH <2.

11.3 Water samples should be poured into the vial without introducing air bubbles. The vial should be filled completely so that when capped no headspace is present. According to EPA SW846 8260B, due to the differing solubility and diffusion properties of gases in aqueous matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace appears in the form of micro bubbles and should not invalidate a sample for volatile analysis.

Studies conducted by the USEPA (EMSL-Ci) indicate that "pea-size" bubbles (i.e., bubbles not exceeding ¼ inch or 6mm in diameter) did not adversely affect volatiles data. These bubbles were generally encountered in wastewater samples, which are more susceptible to variations in gas solubility than are groundwater samples. These samples are usually

accompanied by a buildup of pressure within the vial (e.g. carbonate containing samples preserved with acid).

11.4 Soil sample containers should be tapped slightly as they are filled to try and eliminate as much free air space as possible or they are preserved in the field as stated above. Make sure that the vial opening is dirt free to make a tight seal. Samples can be frozen if within 48hours without preservation. Otherwise, the sample must be preserved within 48 hours with either sodium bisulfate (low-level) or methanol (high-level).

11.5 Samples are shipped to laboratory on ice or with blue ice.

11.6 Water samples are stored in the volatile lab refrigerator at 4°C. Soil samples are stored in a separate volatile lab refrigerator at 4°C.
Note: Soil samples preserved with water only must be stored @-10°C (frozen).

11.7 Samples are not opened until time of analysis. Water samples are not opened but analyzed directly on the auto samplers. Samples are screened or history is checked prior to running samples.

11.8 Holding time: 14 days from sample date for preserved water and all soils. Unpreserved aqueous samples are only allowed under NYDEC programs and must be analyzed within 7 days. VOA air samples must be analyzed within 72 hours of collection.

12.0 QUALITY CONTROL

12.1 GC/MS Tunes with BFB. Tune must be performed every 12 hours. Recommended corrective action if tune fails is to perform instrument maintenance, as necessary, retune instrument. All analysis shall be suspended until tuning non-compliance is rectified.

8260B QUALITY CONTROL SUMMARY

{PRIVATE }QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	At start-up and when cont. cal fails. Min. of 5 stds. Low std must be ≤ RL.	% RSD ≤30% on CCC's or "r" ≥0.99, Min RF on SPCC's. %RSD ≤15% or "r" ≥ 0.99 all other analytes. Must contain all target analytes. If LR used, the curve must not be force through the origin.	Recalibrate, until acceptance criteria is achieved. Non-conforming compounds are reported in a case narrative.
Initial Calibration Verification (ICV)	Second source std. run each time an initial calibration is run	Lab Limits of ±15%	Rerun ICV std. or correct problem and recalibrate
Method Blank (MB)	1X per 20 samples and after calibration stds. Matrix match and preserve specific	All analytes < Reporting Limit, except common contaminates which must be <5x RL	Reanalyzed, locate source of contamination & correct problem. Report non-conforming compounds report in case narrative.
Internal Standards	Min of 3 compounds	Area counts must be between 50 – 200% of the area counts in the associated Continuing Calibration std. Retention times of IS must be within ± 30 sec of RT in associated con cal std	Case narrate exceedances If one or more IS std is outside of limits, reanalyze sample unless obvious interference is present. See corr action for Surr.
Continuing Calibration (CCAL)	Near Mid-point std. 1X per 12 hr. period prior to analyzing samples. Frequently a 20 µg/L std is used. Must contain all target compounds.	CCC's %D or % drift must be ≤20%, ≤30 for other compounds SPCCs must meet the min. mean response factors Chloromethane, 1,1-Dichloroethane and Bromoform- **0.10**, Chlorbenzene and 1,1,2,2-Tetrachloroethane **-0.30**	Reanalyze. Recalibrate if (1) %D of any CCCs >20, or (2) if %D of >10% of other analytes >30. Report non-conforming compounds in case narrative.

MS\MSD	Per Batch of 20 samples or less. Matrix specific. Must contain all target analytes.	70-130%R RPD ≤ 30 for waters and soils	Check LCS %R ,if recoveries are acceptable note exceedances in Case Narrative.
Surrogates	All samples and QC analyses – 4 compounds (min 3 compounds)	70-130% Lab determined recoveries exceeding +/- 30% are acceptable for some difficult matrixes (wastes/sludge) but surrogate recoveries for these matrixes must be documented. If reanalysis within hold-time, report better of two analysis and case narrate. If reanalysis occurs outside of hold-time with acceptable recoveries, report both analyses and case narrate.	Note exceedances in case narrative. If one or more surrogate outside of limits, reanalyzed sample unless: (1) obvious interference present, (2) if one surr exhibits high rec and target compound not detected in sample (3) MEOH preserve sample % moisture >25% and surr rec >10%.**For sample not reanalyzed due to obvious interference, MCP requires chromatogram be reported in the data report.**
Laboratory Control Sample (LCS) / LCD (Duplicate)	Run one per batch of 20 samples or less. Frequently a 20 µg/L std is used. Must contain all target compounds. Matrix specific. Can also be used as CCAL	70-130% Recovery Reanalyze, recalculate, locate source of problem. RPD < 25%, except for difficult compounds which must be ≤ 50%.	Report non-conformances in case narrative.

13.0 CALIBRATION and STANDARDIZATION

Initial and Continuing Calibration Standards

{PRIVATE }Sources for Primary Standards 8260 Method			
{PRIVATE }Standard	Manufacturer	Cat #	Concentration (µg/L)
Internal Std Mix	Ultra	STM-520	2500
Surrogate Mix	Ultra	STM-530	2500
Primary Calibration Mix A	Ultra	CUS 5911	200
Primary Calibration Mix B	Ultra	CUS 4900	2000
Primary Calibration Mix C	Ultra	DVM 584	200

13.1 Primary stock standards are stored in the Volatile freezer at -10°C to -20°C and are used or replaced every 3 months or sooner if problems with the gas compounds are observed. Gases are replaced weekly.

13.1.1 All primary and secondary standards are separated into 2 separate aliquots to preserve integrity of the standards for a longer period.

13.2 Working Standards - Prepared daily for initial and continuing calibrations or calibration checks using the following formulations. Standards are prepared by adding the working stock standards to clean volumetric flasks containing 100 mL of organic free water.

{PRIVATE } Stock	0.5 µg/L	1 µg/L	5 µg/L	10 µg/L	20 µg/L	40 µg/L	80 µg/L
Primary Stock (200µL)	0.25	0.5	2.5	5	10	20	40

Note: The 0.5 is a 10x serial dilution bases off the 5µg/L stock and the 1 µg/L is a 10x serial dilution based off the 10 µg/L stock.

13.3 Working surrogate & internal stand solution is stored on the Archon under

pressure. Unopened stock standards are stored in the volatile freezer at -10°C to -20°C.

13.4 4-Bromoflurobenzene, used to tune the GC/MS, is part of the IS/SS standard. It is added to all runs and can be evaluated from any blank or standard preceding a CCV.

Initial Calibration Verification Standard

LAB Std ID	Manufacturer ID	Primary Concentration ng/µL	Catalogue #
Secondary A	Custom Restek	200	555124
Secondary B	Custom Accu Std	2000	M-8260-ADD-10X
Secondary C Gas mix	Accu Std	200	S-12300-02 M-502B

NOTE: Each mix (Lab Std ID) is added at 10µL into 100ml volumetric flask with VOA-water to make 20µg/L.

Secondary Source of Standards (Low Levels/Secondary Stock Stds.)								
Standard	Manufacturers							
	Absolute		AccuStandard		Supeleco		Ultra Scientific	
Secondary Cal Stds	Cat#	Conc. µg/mL	Cat#	Conc. µg/mL	Cat#	Conc. µg/mL	Cat#	Conc. µg/mL
A-54 liquids	30001	200	M-502A-R	200	4-7933	200	DWM-583-1	200
B-6 Gases	30002	200	M-502-B	200	4-7934	200	DWM-584-1	200
C&I Custom 10comp	-	-	S-4616	-	20002590	-	-	-
A&B 60comp	-	-	M-502	200	4-7932	200	DWM-580-1	200
Ketones 4comp	-	-	CLP-022K	200	-	-	-	-
C&I 8comp	-	-	M-8260-ADD	200	-	-	-	-

Spike Standard	91370	5	-	-	-	-	-	-
Individuals								
MTBE	-	-	S-078	200	-	-	NV-250	100
2CEVE	70074	1000	M-601C	200	4-8672	200	HC-1	100
Diethylether	70153	1000	-	-	-	-	NV-140	100
THF	70380	1000	S-457S	200	-	-	EPA-1236	1000
CS_2	70060	1000	APP-9-035	100	4-8361	200	-	-
Vinyl acetate	-	-	APP-9-211	100	-	-	NV-1	100
Ethyl Methacrylate	70381	1000	APP-9-105	100	-	-	EPA-1036	1000
Trans-1,4-Dichloro-2-butene	70486	1000	APP-9-068	100	1	1	HC-460-1	100

13.5 Surrogate and Internal Standards are purchased separately and used diluted, either separately or combined in secondary and daily standards.

A surrogate/internal standard (SS&IS) solution is prepared as follows 1200µL of each: Surrogate Std (Ultra – Part # STM-530-1, 2500µg/mL), Internal Std (Ultra – Part # STM-520-1, 2500µg/mL) is added to 3600µL of MeOH to make 6000ng/µL and added to Archon autosampler well.

Samples, Method Blanks and calibrations are spiked with 1µL from the auto sampler, resulting in a surrogate concentration of 20µg/L. The surrogate compounds are: 4-Bromofluorobenzene, Dibromofluoromethane, Toluene-d_8, and 1,2-Dichloroethane-d_4. The internal standards are: Fluorobenzene, 1,4-Dichlorobenzene-d4, Chlorobenzene-d5. Working surrogate & Internal standard solution is stored on the Archon under pressure. Unopened stock standards are stored in the Volatile freezer at –10°C to -20°C.

13.6 Preparation information for all standards, including purchased primary

standards, is documented in the Volatiles Standards Logbook. This information includes: names of standard, date of preparation, initials of analyst, primary stock standard name, source lot #, primary stock concentration, date opened, expiration date, amount of primary stock added, final volume into MeOH, and concentration of standard.

13.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE STANDARDS

STL spikes with a full list of MS/MSD compounds. Additional spiking compounds may be requested on a project-specific basis.

To prepare the MS/MSD for water, 10 µL of the 8260 spike Std. (200 µg/mL) is added to 100 mL of a water and poured into a 40mL vial to achieve a 20 µg/L or 100 µg/kg (wet weight) spike level. The standard is run on the Archon.

To prepare the MS/MSD for low level soil, add 2.5uL of cal mix A + B to soil vial and bring to a total volume of 25mL.

To prepare the MS/MSD for high level soil to be run on Archon, add 400µl of high level MeOH sample to 100 mL flask of reagent water and then 10µl spike is added as with the water sample.

13.8 LABORATORY CONTROL SAMPLE (LCS) / LCS DUPLICATE (LCSD)

To prepare the water or high level soil LCS/LCSD, 10 µL of the 8260 spike Std. (200 µg/mL) is added to 100 mL of reagent-free water and fill 2-40mL vials for the Archon auto sampler.

To prepare low level or high level soil LCS/LCSD, add 2.5uL to vial and bring to a total volume of 25mL.

13.9 METHOD BLANK WATER

Reagent water or other blank matrix, i.e. Poland Spring water are presently used for method blanks. 40 mL vials are filled for the Archon auto-sampler. For soil, 25mL of water is placed in a 40mL vial and placed on the Archon auto-sampler.

Note: STL Westfield has found that we have fewer contamination issues with the use of Poland Spring water. It is used for MB's, CCV, LCS/D and Trip blanks.

14.0 PROCEDURE

The volatile analyst receives daily worksheets which contain the previous day's samples logged into the laboratory LIMS and require 8260B analyses. The worksheet indicates the sample collection date as well as the due date. The analyst has the responsibility to insure that all VOA analyses are performed within the required holding times.

14.1 TUNING

A blank is analyzed and the BFB is evaluated from the SS/IS mixture at 20ng/uL. Using the criteria in Table 4. This is done prior to running any standards or samples. Samples, Standards, and QC Samples (ICV, spikes, duplicates) have 12 hours from time of BFB injection to be analyzed. A new BFB with passing criteria will need to be analyzed before running more samples.

Table 3
Tuning Acceptance Criteria

M/Z	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	>95% <100% of mass 174
177	5-9% of mass 176

14.2 INITIAL CALIBRATION

An Initial Calibration is run if there have been QC problems that need to be resolved, or if maintenance has been done on the instrument (source cleaning, etc.). QC problems would include, but not be limited to, failing Continuing Calibration Standards, surrogates out of control limits, ICV failing, and spike standards out of control. The initial calibration is run immediately after the BFB-tune has been analyzed & passes its criteria.

The initial GC/MS calibration is based on a seven-point curve ranging between 0.5 µg/L to 80 µg/L. The concentrations of the standards are 0.5 µg/L, 1 µg/L, 5 µg/L, 10 µg/L, 20 µg/L, 40 µg/L and 80 µg/L. A select few compounds are at higher concentrations. Each compound has a response factor (RF) calculated for each concentration point of the curve relative to one of the internal standards using the following equation.

$$RF = \frac{(Ax * Cis)}{(Ais * Cx)}$$

Ax = Area of the characteristic ion for the compound being measured.
Ais = Area of characteristic ion for the specific internal standard.
Cis = Concentration of the specific internal standard.
Cx = Concentration of the compound being measured.

A mean RF is calculated using the five RF values calculated from initial calibration curve. A check of the 5 system performance check compounds (SPCC's) is made before the calibration curve may be used. The SPCC's and minimum RF limits are as follows:

	RF
Chloromethane	0.1
1,1-Dichloroethane	0.1
Bromoform	0.1
1,1,2,2-Tetrachloroethane	0.3
Chlorobenzene	0.3

In addition the percent relative standard deviation (% RSD) is calculated for

each target analyte and the SPCC's. The criteria for target analyte %RSD is 15% except for the CCC compounds for which the %RSD must be equal to or less than 30%. The CCC compounds are:

1.1-Dichloroethere
Toluene
Chloroform
Ethylbenzene
1,2-Dichloropropane
Vinyl Chloride

If the %RSD of any target analyte is 15% (30% for CCC compounds) or less, then the response factor may be assumed to be constant over the calibration range and the average RF may be used for quantitation.

If the RSD criterion of a target analyte is greater than 15% the instrument operating conditions should be checked. If the problem appears to be associated with a single calibration standard, that one standard may be reanalyzed and the RSD calculated. If the problem appears to be associated with a single compound, than the appropriate standards, may be reanalyzed for that compound.

If the RSD criterion is still greater than 15% a regression may be employed. The instrument response must be treated as the dependent variable (y) and the concentration as the independent variable (x). In order to be used for quantitation purposes the r must be greater or equal to 0.99 or r^2 must be greater or equal to 0.9801. Additional information can be found in section 7 of Method 8000 rev. 2, 1996 vol. IB.

Evaluating retention times – The relative retention time of each target analyte in each sample should agree within 0.06 relative retention time units of the closest calibration standard.

For further information, please refer to the section on Quality Control.

14.3 SAMPLE ANALYSES

All samples are loaded onto the auto-samplers in the 40mL vials. If a

sample is being rerun for dilution or other requirements a new 40mL vial is used. Dilutions are prepared into various volumetric flasks. A 25mL portion of the diluted sample is purged. Appropriate dilutions should be made to maintain the highest positive detection in the upper half of the calibration curve's linear range.

Note: The samples are screened or history checked before running to determine concentration, then a new sample may be run at a dilution.

The autosamplers spike each sample, standard and blank with 1 µl of internal and surrogate standard solution.

Soil samples are weighed to the nearest 0.01 g on a top-loading balance. Soils are weighed directly into tard 40mL vials. Low-level Soil weights of 1 or 5 grams are preferable. Follow section 12.9.2 for water and standards addition. High-level soil weights: 10g into 10mL (1:1) of MeOH. A 40mL vial with 25mL of organic free water, will have 100µl of MeOH sample added to it.

Low level soils have their final volume brought up to 25mL.

14.4 QUALITATIVE ANALYSIS

The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum (after background correction) with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The EnviroQuant Sortware used by STL allows updating with the mid-level standard each time instrument is recalibrated. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the

presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs i.e. m&p Xylene.

Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

The analyst must also use professional judgement in the identification of compounds. Consultation with the organic supervisor should be done if there is difficulty in identification.

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Use the following guidelines for making tentative identifications:

1. Relative intensities of major ions in the reference spectrum (ions

greater than 10% of the most abundant ion) should be present in the sample spectrum.

2. The intensities of the major ions should agree within ±20%. (Example: for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co eluting compounds.

5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Where applicable the concentration of these non-target analytes should be reported at estimated.

15.0 CALCULATION

15.1 QUANTITATION

Quantitation is based on the internal standard method when the RSD of the compound is 15% or less. Three internal standards are spiked in the standards and samples at 20 µg/L. They are Fluorobenzene, Chlorobenzene-d5 and 1,4-Dichlorobenzene-d4. The sample concentration of each compound is calculated using the internal standard nearest to the retention time of that given compound using the following calculation.

$$\text{Concentration } \mu g/L = Ax \times Is / Ais \times RF$$

Ax = Area of Characteristic ion for compound being measured.
Is = Amount of internal standard injected (ng).

RF = Response factor for compound being measured.
Ais = Area of characteristic ion for the internal standard.

If the compound RSD criteria were not met then the regression equation description in section 12.5 of this SOP and section 7 Method 8000 shall be used.

The report factor is used to adjust the µg/L from the instrument report to its correct final µg/L result based on dilutions and weights. The report factor is entered into the laboratory LIMS system.

15.2 CALCULATION FACTOR DETERMINATION:

Water:

The report factor will always be equal to the dilution factor. The default report factor (no dilution) is equal to 1.

Soil:

The **low level** factor is determined as follows: assumed to be 5g, 1X, with a RF of 5.

$$CF = \frac{25\ mL}{} \times \frac{1000\ g}{(\%solids)} \times \frac{1L}{1\ kg} = \frac{5\ \mu g/kg}{1000mL}$$

$$\text{or} = \frac{25\ mL}{(\text{wt of sample})\ (\%\ \text{solids})} = 5\ \mu g/kg$$

Soil

The **high-level** report factor is determined as follows:

$$CF = \frac{\mu g}{L} \times \frac{25\ mL}{(\text{wt of soil})(\%\text{solids})} \times \frac{(5,10\ \text{or}15\ ml)}{0.100mL} \times \frac{1000g}{1kg} \times \frac{1L}{1000ml} = 250\ \mu g/kg$$

$$\text{or} = \frac{25\ mL}{(\text{wt of sample})(\%\ \text{solids})} \times \frac{(MeOH\ mL)}{0.1mL\ (\text{dilution})} = 250\ \mu g/kg$$

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution. Unused and expired standards must be disposed of in the appropriate manner consistent with STL Westfield sample disposal policy.

18.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA

Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCE

22.1 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846 Method 8260B, 3rd Edition, Revision 2 1996.

22.2 5035- Sample prep for soils "Closed-System Purge and Trap and Extraction for volatile Organics in Soil and Waste Samples." SW846 Method 5035, Revision 0 Dec. 1996.

22.3 Operation Manuals for the HP 5890 Series II GC, 5972 MSD, Enviroquant Data System, Hewlett Packard.

22.4 Operation Manuals for the OI Archon, Tekmar-Dorman Precept II auto samplers and purge and trap concentrator 2000 & 3000 Tekmar-Dorman Corporation and OI 4560 P+T Concentrator

22.5 Severn Trent Laboratories Westfield QA Manual.

22.6 SW-846 8206B, Volatile Organic Compounds by CG/MS for MCP. WSC CAM-11A of MADEP, Rev No. 4, May 28th, 2004.

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STL WESTFIELD

STANDARD OPERATING PROCEDURES

Analytical Procedure for the Analysis of Semi-volatiles by GC/MS

EPA SW846-8270C

SOP ID: MSS00300.MA

Revision (4)

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QUALITY ASSURANCE OFFICER

LABORATORY DIRECTOR

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1.0 TITLE : Analytical Procedure for the Analysis of Semi-volatiles by GC/MS

2.0 MATRIX: The method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, media and water samples.

3.0 METHOD REPORTING LIMIT: Aqueous samples (approximately) 10µg/L, Soils 333µg/Kg.

4.0 SCOPE and APPLICATION

4.1 Method 8270C is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of matrices including; solids, soils, air sampling media and water samples. Samples are prepared for analysis by separatory funnel, microwave extraction, or sonication, and analyzed by a Gas Chromatograph (GC) equipped with a Mass Selective Detector (MSD). Direct injection of a sample may be used in limited applications.

4.2 The target compounds determined by this method are listed in Table 1 below. Additional parameters may be analyzed upon client request. The additional compounds are limited to semivolatile organic compounds that can be sufficiently recovered by the extractions and detected by the GC/MSD system. QC criteria for additional compounds are compound and contract specific and not included in this procedure.

TABLE 1

TARGET COMPOUNDS

NUMBER	ANALYTE	CAS NUMBER
1	Acenaphthene	83-32-9
2	Acenaphthylene	208-96-8
3	Aniline	62-53-3
4	Anthracene	120-12-7
5	Benzidine	92-87-5
6	Benzo(a)anthracene	56-55-3
7	Benzo(b)fluoranthene	205-99-2
8	Benzo(k)fluoranthene	207-08-9
9	Benzo(a)pyrene	50-32-8
10	Benzo(g,h,i)perylene	191-24-2
11	Benzoic acid	65-85-0

NUMBER	ANALYTE	CAS NUMBER
12	Benzyl alcohol	100-51-6
13	Benzyl butyl phthalate	85-68-7
14	Bis(2-chloroethyl)ether	111-44-4
15	Bis(2-chlorethoxy)methane	111-91-1
16	Bis(2-ethylhexyl)phthalate	117-81-7
17	Bis(2-chloroisopropyl)ether	108-60-1
18	4-Bromophenyl phenyl ether	101-55-3
19	4-Chloroaniline	106-47-8
20	2-Chloronaphthalene	91-58-7
21	4-Chlorophenyl phenyl ether	7005-72-3
22	Chrysene	218-01-9
23	Dibenzo(a,h)anthracene	53-70-3
24	Dibenzofuran	132-64-9
25	Di-n-butylphthalate	84-74-2
26	1,3-Dichlorobenzene	541-73-1
27	1,2-Dichlorobenzene	95-50-1
28	1,4-Dichlorobenzene	106-46-7
29	3,3'-Dichlorobenzidine	91-94-1
30	Diethyl phthalate	84-66-2
31	Dimethyl phthalate	131-11-3
32	2,4-Dinitrotoluene	121-14-2
33	2,6-Dinitrotoluene	606-20-2
34	1,2 Diphenylhydrazine	122-66-7
35	Di-n-octylphthalate	117-84-0
36	Fluoranthene	206-44-0
37	Fluorene	86-73-7
38	Hexachlorobenzene	118-74-1

NUMBER	ANALYTE	CAS NUMBER
39	Hexachlorobutadiene	87-68-3
40	Hexachloroethane	67-72-1
41	Indeno(1,2,3-cd)pyrene	193-39-5
42	Isophorone	78-59-1
43	Naphthalene	91-20-3
44	2-Nitroaniline	88-74-4
45	3-Nitroaniline	99-09-2
46	4-Nitroaniline	100-01-6
47	Nitrobenzene	98-95-3
48	N-Nitrosodi-n-propylamine	621-64-7
49	Phenanthrene	85-01-8
50	Pyrene	129-00-0
51	1,2,4-Trichlorobenzene	120-82-1
52	4-Chloro-3-methylphenol	59-50-7
53	2-Chlorophenol	95-57-8
54	2,4-Dichlorophenol	120-83-2
55	2,4-Dimethylphenol	105-67-9
56	2,4-Dinitrophenol	105-67-9
57	2-Methylphenol	95-48-7
58	2-Methyl-4,6-dinitrophenol	534-52-1
59	2-Methylnaphthalene	91-57-6
60	4-Methylphenol	106-44-5
61	2-Nitrophenol	88-75-5
62	4-Nitrophenol	100-02-7
63	Pentachlorophenol	87-86-5
64	Phenol	108-95-2

NUMBER	ANALYTE	CAS NUMBER
65	2,4,5-Trichlorophenol	95-95-4
66	2,4,6-Trichlorophenol	88-06-2
67	Hexachlorocylopentadiene	77-47-4
68	N-Nitrosodiphenylamine	86-30-6
69	n-Nitroso-di-methylamine	62-75-9
70	Azobnezene	86-30-6

4.3 Method 8270C can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons (PAHs), chlorinated hydrocarbons, and pesticides, phthalates esters, organophosphates esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

4.4 This method is restricted to use by or under supervision of analysts experienced in the use of GC/MS and skilled in the interpretation of mass spectra.

4.5 The estimated quantitation limit of this method for determining an individual compound is approximately 330 - 1670 µg/kg (wet weight) for soil/sediment samples and 10 - 50 µg/L for ground water samples.

5.0 SUMMARY

5.1 A measured volume of sample, approximately 1000mL or 30g, is extracted with methylene chloride (refer to specific preparation method for solvent type) using a separatory funnel, sonication or microwave extraction. The methylene chloride is reduced to a final volume of 1.0mL. The extract is injected into a GC/MSD either directly or after application of appropriate clean-up procedures.

5.2 Analytes eluted from the capillary column are introduced into the MS by a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using five-point calibration curve.

5.3 The GC column used in this method is a capillary column, which affords greater sensitivity, selectivity, and permits easier elimination of sample artifacts from the GC system.

6.0 DEFINITIONS

Semi-Volatile Compound: SW-846 does not contain formal definitions for volatiles or semivolatiles and there is no "official" definition of volatile or semivolatile compounds that can be used across all EPA programs. Some compounds can even be considered either a volatile or a semivolatile depending on the analytical technique used (i.e., naphthalene, nitrobenzene, dichlorobenzene, hexachloroethane). Generally speaking, volatiles have been defined elsewhere as compounds with boiling points below 150°C and vapor pressures of greater than 0.1 mm Hg and semivolatile would have boiling points above 150°C. However, such definitions are not hard and fast, and should only be considered descriptive of a general trend.

GC/MS: The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph separates the molecules within the compound sample by allowing certain types of molecules to be collected by the mass spectrometer sooner than other types. The mass spectrometer receives each molecule from the GC unit, breaks each molecule into ionized fragments, and identifies each molecule from the charge and mass of the ionized fragments.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of

detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Method interferences can be caused by contaminants present in solvents, reagents, glassware, or other devices used in handling the sample during analysis. These sources are demonstrated to be free of interference by the analysis of method blanks performed with each analytical batch.

7.2 All solvents used in the course of the analysis should be from corporate approved lots. These lots have been tested for interferences.

7.3 Glassware must be carefully cleaned with hot soapy water followed by solvent rinse.

7.4 A common laboratory introduced contaminant is phthalate esters or other plasticizers, which can be extracted by the organic solvents used in the method. Plastic containers or

other hardware should be avoided in preference for glass. Only plastics made of Teflon should contact the sample during analysis.

7.5 Only water from the deionized water purification system should be used for sample dilutions and method blanks. This water is proven to be free of interfering contaminants.

7.6 Matrix interferences may be caused by contaminants that co-extract from the sample. The extent of matrix interference will vary from source to source. In some cases, extract dilution may be required prior to chromatographic analysis due to the high concentration of non-target compounds that cannot be removed from the sample, or to the presence of high concentration of target compounds.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, STL Westfield Safety Addendum and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 Gas Chromatograph Mass Selective Detector.
9.2 HP 5890 Series II GC, 5972 MSD and HP 6890 Series GC, 5973 MSD.
9.3 GC Columns
9.4 RTX w/5 30m X 0.25mm, 0.5µm Restex Corp.
9.5 Syringes
9.5.1 1.0µL gas tight (Hamilton or equivalent)

9.5.2 10µL gas tight (Hamilton or equivalent)
9.5.3 100µL gas tight (Hamilton or equivalent)
Syringes used in the preparation of initial stock solutions should be maintained separately from all other syringes.

9.6 Balance capable of weighing to 0.1 mg (Sartorius Top loader R200D; IEA Balance #4).
9.7 Data Collection Device
9.8 HP chemstation G1701BA Enviroquant data analysis software.
9.9 Algilent model 6890/5973 GCMS (Low Level)

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1 Reagent water is defined as water free of target compounds at concentrations equal too or less than the Practical Quantitation Limit.
10.2 Methylene Chloride from Corporate approved lots.
10.3 Standards
The production of standards is documented in the ABN standards log book. Lot numbers should be recorded for commercial blends and assigned for homemade stocks and dilutions. Laboratory assigned lot numbers are always based on the production date. The commercial blends and neat materials required to provide a full compliment of the 68 target compounds are listed in Table 2 at the end of this section. The table specifies materials for both quantitation standards and QC check standards.
10.4 Stock standards
The six surrogate standards (#12-17) are prepared at various concentrations in methanol (with acid-base indicator) by Ultra Scientific (ISM-333XC).
Internal standards are purchased as a certified solution at a concentration of 4000ppm. Sonicate internal standard one hour before use.
10.5 Intermediate Stocks
Standards for MSD analysis are generally in the ppm range for targets. Intermediate dilutions are required to accurately achieve these low concentrations. The dilutions are described in Table 3.
10.6 Working Standards
The following principles must be followed when preparing working standards from stock solutions.
10.6.1 Use properly cleaned and baked glassware.
10.6.2 Never add stock solutions to a dry piece of volumetric glassware. Inject the standard solution below the top of the solvent layer.
10.6.3 Liquid dilutions are performed with readily miscible solvents. Uniformity is achieved by inverting a volumetric flask four times or in the case of 2mL or less volumetric flasks, by thorough mixing with a disposable pipet. This mixing is accomplished with a minimum amount of aeration of the solution.
10.6.4 Transfer solutions to an appropriate size, contaminant free (i.e. new) screw cap vials. Check to ensure that the Teflon liner is in place. Store with a minimum amount of headspace and mark the vial at the solution level to safeguard against evaporation. Subsequent use of the solutions should be noted with another mark. Store the solutions in the standards refrigerator.
10.6.5 Record standard production in the standards notebook, and label all vials with the following information:

a) Description of contents
b) Concentration
c) Solvent
d) Lot number
e) Expiration date
f) Analysts initials

This information should be written on a tag and taped onto the standard's vial.
Table 2 summarizes the steps required for formulation of the six concentration levels required to calibrate the method target compounds.

10.7 Spiking Solutions

10.7.1 Samples analyzed under this SOP receive a 500uL spike of surrogate standard. Acid compounds are spiked at 100ug/mL and base/neutrals are spiked at 50ug/mL.

10.7.2 Laboratory control samples and matrix spikes also receive 500uL of the matrix spike solution. This results in a solution, which is 40 mg/L for all targets.

10.8 Expiration Dates for Standards

Unless superceded by the method, expiration dates are 6 months from date of preparation for semi-volatile standards and 1month for volatile standards. Standards purchased as certified solutions often have expiration dates of their own. If this date expires sooner than the 6 months than that is the expiration date. Standards with expired dates must be discarded. It is sometimes possible to get extended expiration dates by calling the manufacturer with the lot number. A certificate must be obtained before the standard can be used. Stock and working standards are replaced after one year or sooner if questions about their quality arise.

TABLE 2

Commercial Blends Required for Production of Method Standard Solutions

NUMBER	ITEM	VENDOR	CAT #	CONC	SOLVENT	USE
1	Base/Neutral Composite Mix	Accu-standard	CLP-HC-BN-R	2000 µg/mL	Benzene: Methylene Chloride	QC Check/ ICV
2	Benzidine & 3,3'-Dichlorobenzidine	Accu-standard	Z-014F	2000 µg/mL	Methanol	QC Check/ ICV
3	Acid composite Mix	Accu-standard	CLP-HC-A-R	2000 µg/mL	Methylene Chloride	QC Check/ ICV
4	Acid composite Mix #2	Accu-standard	Z-014E-R	2000 µg/mL	Methylene Chloride	QC Check/ ICV
5	Acetophenone	Accu-standard	APP-9-004-20x	2000 µg/mL	Methylene Chloride	QC Check/ ICV
6	Method 8270-Surrogate Std.	Accu-standard	M-8270-SS	4000 µg/mL	Methylene Chloride	Surrogate for Calibration
7	Custom Standard – 11analytes	Ultra Scientific	CUS-5253	2000 µg/mL	Dichlorom ethane	Calibration
8	Semi-Volatiles Mixture Additional Compounds	Ultra Scientific	SVM-8270	1000 µg/mL	Methylene Chloride / Benzene (3:1)	Calibration
9	GC/MS Tuning Std	Accu-standard	M-626-75-20x	1000 µg/mL	Methylene Chloride	GS/MS Tuning
10	8270 Surrogate std.	Ultra Scientific	ISM-333XC	Varied	Methonal	QC Check/ ICV
11	Internal Std	Accu-standard	Z-014J	4000 µg/mL	Methylene Chloride	Internal Std

TABLE 2 (Cont)

Semi-volatile Surrogate Standard- Ultra Scientific ISM-333XC -

NUMBER	ITEM	VENDOR	CAT #	USE
12	Phenol-d6	Ultra Scientific	17,606-0	Surrogate Standard For spiking
13	2-Fluoro-phenol	Ultra Scientific	F1,280-4	Surrogate Standard For spiking
14	2,4,6-Tribromo-phenol	Ultra Scientific	13,771-5	Surrogate Standard For spiking
15	2-Fluorobiphenyl	Ultra Scientific	10,274-1	Surrogate Standard For spiking
16	p-Terphenyl-d14	Ultra Scientific	36, 463-0	Surrogate Standard For spiking
17	Nitrobenzene-d5	Ultra Scientific	30,884-6	Surrogate Standard For spiking

TABLE 3

Formulation of Intermediate Concentration Stock Solutions.

NUMBER FROM TABLE 2	ITEM	Volume (mL)	QS (mL)	QS SOLVENT	FINAL CONCENTRATION (mg/L)
18 (1-5)	Extractables	1.0	25	Acetone	80
19 (1-5)	Matrix Spike Standard	1.0	25	Acetone	80
20 (6)	Surrogate Calibration	0.5mL	10	Methylene Chloride	200
21 (12-17)	Surrogate Spiking	100mL	NA	Methanol	Varied

TABLE 4

Working Standard Formulation for Method 8270

- Refers to Intermediate Dilutions From Table 3a.
IS - Refers to Internal Standard described in 8.3.3.

Volume (uL)		**Volume IS (uL)**	**QS (mL)**	**QS SOLVENT**	**FINAL CONCENTRATION mg/L Calibration**
6-8	25	10	1	Methylene Chloride	5
6-8	50	10	1	Methylene Chloride	10
6-8	100	10	1	Methylene Chloride	20
6-8	200	10	1	Methylene Chloride	40
6-8	250	10	1	Methylene Chloride	50
6-8	400	10	1	Methylene Chloride	80
6-8	500	10	1	Methylene Chloride	100
22 surrogate Spk	0.5mL (Bases) 1.0 mL (acids)		100mL	Acetone	50 (base/neutral) 100 (acids)

11.0 SAMPLE HANDLING & PRESERVATION

11.1 While it is generally not the responsibility of the analyst to collect the sample, awareness of method requirements in this regard can ensure that proper quality control is applied.

11.2 The method specified container is 1-L amber glass with a Teflon lined closure. Sample bottles are never reused.

11.3 Samples should be cooled to 4°C and protected from light. The sample pH is measured

prior to extraction and noted in the sample extraction log.

11.4 The sample holding time to extraction for samples is seven (7) days for waters and fourteen (14) days for soils and concentrated wastes after collection. The analysis must be completed within forty (40) days of sample collection.

11.5 Every effort will always be made to analyze samples at STL within the method specified holding times. Holding time problems must be reported to a supervisor and/or the project manager.

12.0 QUALITY CONTROL

12.1 Quality Control Evaluation

{PRIVATE }QC Analysis	Frequency	Acceptance Criteria	Corrective Action
DFTPP Tune Standard	Every 12 hours	See Table 7.0	Auto tune MS, check for injection port problems.
Initial Calibration	At start-up and when cont. cal fails	r-value ≥ 0.99 for all method analytes (or <15% RSD, CCC ≤ 30%)	Check for injection problems reanalyze std. or recalibrate
Initial Calibration Verification (ICV)	After each initial calibration	r-value ≥ 0.99 for all method analytes (or <15% RSD, CCC ≤ 30%)	Check for errors, rerun ICV or recalibrate
Laboratory Control Sample (LCS/LCSD)	Extracted with every batch or every 20 samples whichever is more frequent.	% R 40-140% for b/n, 30-130% for acids. RPDs <20% waters and <30% soils	Correct problem, rerun LCS or recalibrate
Method Blank (MB)	Extracted with every batch or every 20 samples whichever is more frequent.	All analytes < RL	Reanalyze MB, locate source of contamination or report samples with "B" flag. Re-extract if required by client
Continuing Calibration	Every 12 hours prior to analysis of samples	CCC ± 20% R. other compounds ≤ 30%	Recalibrate if %D of any CCC is >20 or if %D of >10% of other compounds >30%.
Matrix Spike/Spike Duplicate (MS/MSD) [Client Specific -	Extracted every 20 samples (BATCH QC)	% R 40-140% for b/n, 30-130% for acids RPDs <20% waters and <30% soils	Check LCS, if recoveries acceptable in LCS, matrix interference presumed

MCP]			
Surrogate	Minimum of 3 b/n and 3 acids	%R waters 30-130% b/n and 15-110% for acids. Soils %R 30-130%	If two or more from any one fraction are outside limits or if any one surr. recovers <10%; re-extract sample
Internal Standard	Min of 6 across RT.	50-200% area counts of associated CC std.	If one or more outside of CL, re-analyze. c/n exceedances.
MDL	Run yearly	Requires QA Officer; Lab Director Approval	Rerun if not approved
IDOC/DOC	New analyst and yearly after that	Requires QA Officer; Lab Director Approval	Rerun if not approved

TABLE 6

Estimated MDLs and PQLs for 8270 Compounds

NUMBER	COMPOUND	PQL (ug/L)	Aqueous MDL est (ug/L)	SOIL MDL est (ug/Kg)
1	Aniline	50	18	24.1
2	Phenol	10	2.2	56.0
3	Bis(2-chloroethyl)ether	10	0.52	51.5
4	2-Chlorophenol	10	4.4	51.3
5	1,3-Dichlorobenzene	10	0.64	48.3
6	1,4-Dichlorobenzene	10	0.60	47.4
7	1,2-Dichlorobenzene	10	0.53	45.2
8	Benzyl Alcohol	20	3.6	59.7
9	2-Methylphenol	10	4.3	57.2
10	bis-(2-Chloroisopropyl)ether	10	0.39	54.8
11	Hexachloroethane	10	0.85	46.8

NUMBER	COMPOUND	PQL (ug/L)	Aqueous MDL est (ug/L)	SOIL MDL est (ug/Kg)
12	N-Nitrosodinpropylamine	10	0.49	61.7
13	4-Methylphenol	10	4.6	60.8
14	Nitrobenzene	10	0.66	54.6
15	Isophorone	10	0.62	66.2
16	2-Nitrophenol	10	4.2	72.7
17	2,4-Dimethyphenol	10	4.1	68.8
18	Bis(2-chloroethoxy)methane	10	0.71	67.6
19	2,4-Dichlorophenol	10	4.4	62.9
20	Benzoic Acid	50	3.3	173.5
21	1,2,4-Trichlorobenzene	10	0.77	58.6
22	Naphthalene	10	0.68	58.4
23	4-Chloroaniline	20	3.2	253.8
24	Hexachlorobutadiene	10	0.87	58.8
25	4-Chloro-3-Methylphenol	20	5.6	63.6
26	2-Methylnaphthalene	10	4.2	62.6
27	Hexachlorocyclopentadiene-SPCC	10	1.7	59.3
28	2,4,6-Trichlorophenol	10	3.3	71.3
29	2,4,5-Trichlorophenol	10	4.2	72.4
30	2-Chloronaphthalene	10	0.76	61.4
31	2-Nitroaniline	50	3.0	64.6
32	Dimethylphthalate	10	0.43	61.8
33	Acenaphthylene	10	0.59	58.8
34	3-Nitroaniline	50	3.9	456.3
35	2,6-Dinitrotoluene	10	0.74	59.1
36	Acenaphthene	10	0.66	60.0

NUMBER	COMPOUND	PQL (ug/L)	Aqueous MDL est (ug/L)	SOIL MDL est (ug/Kg)
37	2,4-Dinitrophenol	50	12	34.2
38	Dibenzofuran	10	3.3	61.8
39	4-Nitrophenol	50	3.8	58.5
40	2,4-Dinitrotoluene	10	1.0	51.9
41	Diethylphthalate	10	0.81	60.0
42	Fluorene	10	0.84	61.2
43	4-Chlorophenyl-phenylether	10	0.52	63.6
44	4-Nitroaniline	50	11	89.7
45	4,6-Dinitro-2-methylphenol	50	5.0	50.1
46	N-Nitrosodiphenylamine	10	1.1	48.3
47	1,2-Diphenylhydrazine	10	0.82	69.3
48	4-Bromophenyl-phenylether	10	1.2	56.3
49	Hexachlorobenzene	10	0.85	62.3
50	Pentachlorophenol	50	5.7	107.3
51	Phenanthrene	10	0.62	53.6
52	Anthracene	10	0.71	51.3
53	Di-n-Butylphthalate	10	1.1	145.8
54	Flouranthene	10	0.91	49.2
55	Benzidine	10	ND	
56	Pyrene	10	0.60	49.0
57	Butylbenzylphthalate	10	1.3	46.5
58	Benzo(a)Anthracene	10	0.65	48.6
59	3,3'-Dichlorobenzidine	20	1.6	235.6
60	Chrysene	10	0.66	51.3
61	bis(2-ethylhexyl)Phthalate	10	4.9	48.0

NUMBER	COMPOUND	PQL (ug/L)	Aqueous MDL est (ug/L)	SOIL MDL est (ug/Kg)
62	Di-n-octylphthalate	10	1.9	64.7
63	Benzo(b)fluoranthene	10	1.3	45.7
64	Benzo(k)fluoranthene	10	1.8	63.1
65	Benzo(a)pyrene	10	0.54	44.1
66	Indeno(1,2,3-cd)pyrene	10	0.95	52.3
67	Dibenzo(a,h)anthracene	10	0.85	53.1
68	Benzo(g,h,i)perylene	10	1.0	59.2

Water MDLs, prep method 3510

12.2 At the start of each day that samples are to be run, it is necessary to establish that the GC/MS meets the instrument performance criteria. This is accomplished through the analysis of the 50 ng/uL of decafluorotriphenyl-phosphine(DFTPP). This solution should also contain DDT, benzidine, and pentachlorophenol. The GC/MS tuning solution should also be used to asses GC column performance and injection port inertness. DDT breakdown to DDD + DDE should not exceed 20%. Benzidine + pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. Peak tailing factors must be <3 for benzidine and <5 for pentachlorophenol.

This system check must be performed within 12 hours for 8270 samples.

TABLE 7.0

DFTPP: Masses and Abundance Criteria

Mass	Criteria
51	30 - 60 percent of Mass 198
68	Less than 2 percent of Mass 69
69	<100 of mass 198
70	Less than 2 percent of Mass 69
127	40 – 60 percent of Mass 198

Mass	Criteria
197	Less than 1 percent of Mass 198
198	Base peak, 100 percent relative abundance
199	5 - 9 percent of Mass 198
275	10 – 30 percent of Mass 198
365	0.8 – 100 percent Mass 198
441	0.01 - 100 Present but less than Mass 443
442	40-100 percent of Mass 198
443	17 - 23 percent of Mass 442

12.3 A method blank is extracted with each extraction batch of 20 samples or fewer. Results for each method compound must be lower than the method PQLs to permit reporting of that compound. Exceptions can be granted by the supervisory staff upon review of the relevant batch data for common laboratory contaminates (phthalates) which should be <5 times the reporting limit.

12.4 A Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Laboratory Control Sample (LCS, or QC Check) is analyzed for every batch or 20 samples whichever is more frequent. The sample or reagent water is spiked with 1.0 mL of the matrix spike blend as described and should result in an extract concentration of 50 mg/L base neutrals and 100 mg/L acids. The percent recoveries must be between 40 – 140 for the b/n compounds and 30-130 for the acid compounds. RPDs should be <20% for waters and < 30% for soils.

12.5 Surrogate Recovery
The surrogate standard blend as described is spiked into all samples, blanks, and target-compound spiked samples or blanks to assess extraction efficiency and method performance. Surrogate recoveries should meet the following criteria:

SURROGATE 8270C / 625	Water RECOVERY	Soil RECOVERY
2-Fluorophenol	15-110	30-130
Phenol-d6	15-110	For all surrogates
Phenol-d5	15-110	
2,4,6-Tribromophenol	15-110	
Nitrobenzene-d5	30-130	
2-Fluorobiphenyl	30-130	
Terphenyl-d14	30-130	

Samples analyzed according to Method 8270C which have surrogate recoveries outside

of control limits require corrective action. If two or more surrogates for any one fraction (b/n or acid) are outside limits or if any one surrogate recovers at ≤10%, re-extract the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

The sample chromatogram is inspected for the presence of matrix interference. If there is no evident matrix problem, the extract can be re-injected.

12.6 Internal Standards: The area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. The retention times of the internal standards must be within ± 30 seconds of the retention times in the associated continuing calibration standard. All Low Level samples are fortified with 20ug/mL of internal standard mix.

13.0 CALIBRATION and STANDARDIZATION

13.1 Initial Calibration: The GC/MSD must first pass the performance criteria described in section 12.2. Calibration standards are used to generate a six point calibration curve for the semi-volatile target compounds. The calibration is performed at 5, 10, 20, 40, 50, 80 and 100µg/mL. % RSD should be ≤15 or "r" should be ≥0.99 for all compounds except CCCs which must be ≤30. Low standard must be at reporting limit.

13.2 The method target compounds primary characteristic m/z and approximate retention times are listed in Table 5.

13.3 Response factors (RF) are calculated by the enviroquant software for each of the compounds according to the following formula:

$$RF = \frac{(As)(Cis)}{(Ais)(Cs)}$$

where,

As = Area of the characteristic m/z for the parameter to be measured.
Ais= Area of the m/z for the internal standard.
Cis= Concentration of the internal standard (mg/L).
Cs = Concentration of the parameter to be measured (mg/L).

13.4 Initial Calibration

The following are calibration check compounds (CCC):

Acenapthene	4-Chloro-3-methylphenol
1,4 Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo (a) pyrene	

System performance check compounds (SPCC) should have average response factors greater than 0.05. These compounds are:

N-Nitrosodinpropylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

If any outliers exist for either the calibration check compounds or the system performance check compounds, the calibration standards should be reanalyzed.

13.5 The calibration curve must be verified each day (or every 12 hours) by measurement of a continuing calibration verification sample (CCV) at 50 mg/L. The % difference drift must be ≤20% for the CCC and ≤30% for all others.

13.6 The working range of this method is limited to the range of the calibration curve. All extracts with analyte concentrations exceeding the upper limit must be diluted into the upper three fourths of the calibration curve. All diluted extracts with results in the lower one fourth of the calibration range should be reanalyzed at lower dilution so that the quantitation falls within the upper three fourths of the calibration curve.

TABLE 5

Approximate Retention Times of 8270 Compounds on RT X-5 GC/MS Column
IS denotes Internal Standard Compound
SS denotes Surrogate Standard Compound

NUMBER	COMPOUND	Approx. Retention Time (min)	Quantifying Ion (m/z)
1	1,4-Dichlorobenzene-d4 (IS)	4.93	152
2	2-Fluorophenol (SS)	3.92	112
3	Aniline	4.73	93
4	Phenol-d6 (SS)	4.71	99
5	Phenol	4.71	94
6	Bis(2-chloroethyl)ether	4.73	93
7	2-Chlorophenol	4.80	128

NUMBER	COMPOUND	Approx. Retention Time (min)	Quantifying Ion (m/z)
8	1,3-Dichlorobenzene	4.90	146
9	1,4-Dichlorobenzene	4.94	146
10	1,2-Dichlorobenzene	5.11	146
11	Benzyl Alcohol	5.08	108
12	2-Methylphenol	5.19	108
13	bis(2-Chloroisopropyl)ether	5.18	45
14	4-Methylphenol	5.13	108
15	Hexachloroethane	5.38	117
16	N-Nitrosodinpropylamine	5.32	70
17	Nitrobenzene-d5(SS)	5.47	77
18	Naphthalene-d8 (IS)	6.19	136
19	Nitrobenzene	5.47	77
20	Isophorone	5.69	82
21	2-Nitrophenol	5.79	139
22	2,4-Dimethyphenol	5.82	107
23	Bis(2-chloroethoxy)methane	5.90	93
24	2,4-Dichlorophenol	6.06	162
25	Benzoic acid	5.99	122
26	1,2,4-Trichlorobenzene	6.14	180
27	Naphthalene	6.22	128
28	4-Chloroaniline	6.31	127
29	Hexachlorobutadiene	6.42	225
30	4-Chloro-3-Methylphenol	6.93	107
31	2-Methyl Napthalene	7.1	142
32	Acenaphthene-d10 (IS)	8.77	164

NUMBER	COMPOUND	Approx. Retention Time (min)	Quantifying Ion (m/z)
33	Hexachlorocyclopentadiene	7.42	237
34	2,4,6-Trichlorophenol	7.56	196
35	2,4,5 Trichlorophenol	7.64	196
36	2-Fluorobiphenyl (SS)	7.66	172
37	2-Chloronaphthalene	7.83	162
38	2-Nitroaniline	8.05	65
39	Dimethylphthalate	8.38	163
40	Acenaphthylene	8.05	152
41	2,6-Dinitrotoluene	8.51	165
42	3-Nitroaniline	8.76	138
43	Acenaphthene	8.82	153
44	2,4-Dinitrophenol	8.93	184
45	Dibenzofuran	9.09	168
46	4-Nitrophenol	9.12	109
47	2,4-Dinitrotoluene	9.20	165
48	Diethylphthalate	9.63	149
49	Fluorene	9.71	166
50	4-Chlorophenyl-phenylether	9.71	204
51	4-Nitroaniline	9.87	138
52	2,4,6-Tribromophenol(SS)	10.20	330
53	Phenanthrene-d10 (IS)	11.45	188
54	4,6-Dinitro-2-methylphenol	9.94	198
55	N-Nitrosodiphenyla-mine	9.96	169
56	1,2 Diphenylhydrazine	9.99	77
57	4-Bromophenyl-phenylether	10.61	248

NUMBER	COMPOUND	Approx. Retention Time (min)	Quantifying Ion (m/z)
58	Hexachlorobenzene	10.88	284
59	Pentachlorophenol	11.25	266
60	Phenanthrene	11.50	178
61	Anthracene	11.58	178
62	Di-n-Butylphthalate	13.73	149
63	Fluoranthene	13.83	202
64	Chrysene-d12 (IS)	16.70	240
65	Pyrene	14.27	202
66	Benzidine	23.31	184
67	Terphenyl-d14 (SS)	14.65	244
68	Butylbenzylphthalate	15.70	149
69	Benzo(a)Anthracene	16.67	228
70	3,3'-Dichlorobenzidine	16.69	252
71	Chrysene	16.76	228
72	bis(2-ethylhexyl)phthalate	16.88	149
73	Perylene-d12 (IS)	19.41	264
74	Di-n-octylphthalate	18.05	149
75	Benzo(k)fluoranthene	18.77	252
76	Benzo(b)fluoranthene	18.71	252
77	Benzo(a)pyrene	19.31	252
78	Indeno(1,2,3-cd)pyrene	21.89	276
79	Dibenzo(a,h)anthracene	21.94	278
80	Benzo(g,h,i)perylene	22.65	276

13.1 Instrumental procedures.

13.1.1 Gas Chromatograph

Carrier Gas:	Helium
EPC:	
Column Head Pressure:	12.3 psi
Initial Hold:	0.0 min
Ramp Rate 1:	98.00 psi/min
Final Value:	80.0 psi
Final Hold:	0.30
Ramp Rate 2:	98.0 psi/min
Final Value:	12.3 psi (constant flow)
Final Hold:	0.0
Initial Temp:	45 C
Initial Hold:	0.0 min.
Ramp Rate 1:	30 c/min.
Ramp Temp:	100 C
Hold Time:	3.00 min.
Ramp Rate 2:	15 c/min.
Ramp Temp 2:	280 C
Hold Time:	2.00 min.
Ramp Rate 3:	30 c/min.
Final Temp:	325 C
Final Hold:	8 min.
Detector Temp:	320 C
Injector Temp:	250 C
Injection Volume:	1 uL

13.1.2 Mass Spectrometer

Mass Range:	35 – 550 amu
Scan Time:	1.65 scans/second

The mass spectrometer must be tuned to meet the instrument performance check criteria for 50ng DFTPP

14.0 PROCEDURE

14.1 Sample Analysis Procedures

Sample extracts are removed from sample storage - Refrigerator 2. Extracts are returned to this refrigerator after analysis.

Extracts are spiked with the internal standard solution from Table #2. 10uL of internal standard is added to each 1.00mL of sample extract to achieve a final concentration of 40 mg/L. A luL aliquot is injected onto the column using an HP7673A autosampler.

14.2 Data reduction of sample chromatograms is performed by HP-enviroquant Software. GC/MS data from the samples is compared with that of the standards. For any compound to be confirmed as present in the sample the following conditions must be met:

1) The relative retention time must fall within 0.06 units of the relative retention time of the standard compound in the continuing calibration or the last calibration standard, whichever is applicable.

2) The relative ion abundances of any confirming ions must fall within ±20% of those in the standard compound in the continuing calibration or the last calibration standard, whichever is applicable.

14.3 Each day that samples are run, a batch directory is created. The batch is assigned a name corresponding to the run date. Any methods used on this date will be saved into the batch file along with the run data for the samples. The sequence table from the sample run date would be saved with the date following by "S". "S" is assigned automatically.

14.4 A run log is maintained for the instrument. Hard copies of the sequence log are printed from the computer file and saved in a bound book. The log indicates injection order.

14.5 An instrument log is maintained which documents any maintenance performed on the GC/MS or computer. The log includes such information as column changes, source cleanings and the archiving.

15.0 CALCULATION

15.1 Analyte concentration in GC injections is determined by the HP software:

$$ConX = \frac{(Ax)(Conci\ s)}{(Ais)RF}$$

where: ConX = concentration of analyte x in extract (mg/L).
Ax = area of analyte x in chromatograph.
RF = Response Factor for the compound of interest (average).
Ais = Area of internal standard in chromatograph.
Concis = Concentration of internal standard in the extract (mg/L).

15.2 For water samples this extract concentration (ConX) is converted into the sample concentration by the equation:

$$SamX = \frac{(ConX)(Vex\ t)(DF)}{Vsample}$$

where: SamX = Concentration of analyte X in sample (ug/mL).
Vext = Volume of extract in L.

Vsample = Volume of sample in L.
DF = Extract dilution factor.

15.3 For soil samples this extract concentration (ConX) is converted into the sample concentration by the equation:

$$SamX = \frac{(ConX)(Vext)(DF)}{DWsample}$$

where: SamX = Concentration of analyte X in sample (ug/kg(dry)).
Vext = Volume of extract in mL.
DWsample = Dry weight of sample in kg.
DF = Extract dilution factor.

The report template assumes an extract volume of 1 mL and sample dry weight of 0.030 kg.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a “bench-level” review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary

review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention" and according to the facility hazardous waste procedures.

22.0 REFERENCES

22.1 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846 Method 8270C, 3rd Edition, Revision 3 1996.

22.2 Operation Manuals for the HP 5890 Series II GC, 5972B MSD, 5973 MSD, Agilent Chemstsation/enviroquant data system.

22.3 MADEP,CAM,SW8468270C. QA/QC Requirements and Performance Standards for SW-846 Method 8270C, Semi-Volatile Organics by GC/MS.2004.

Corrections and/or additions:
2/27/07 :Removed section 12.7, removed reference to method 625

ATTACHMENT 1
Low level 8270 & NDMA information sheet

SW846 8270C[M] LOW LEVEL ANALYSIS

By utilizing an Agilent model 6890GC/5973MS, which offers a more sensitive detector, lower reporting limits (RL) can be achieved by this method. Commonly referred to as SW846 8270C[M] (Low Level), the instrument is run in full scan mode.

SW846 8270C[M] LOW LEVEL – Standard Analysis for water and soil.

The 8270C[M] Low Level analysis is run as a standard analysis for those clients looking to achieve lower reporting limits than routine SW846 8270C analysis. The method criteria (QA/QC) of the standard SW846 8270C is applied to the Low Level analysis.
Calibration is performed at 0.1, 0.25, 0.5, 1.0, 5.0, 8.0, 10.0, 20.0ug/mL.
Internal standard concentration is 200ug/mL, with 10uL spike into each 1.0mL solvent extract. Internal standard concentration is 2.0ug/mL.

n-Nitroso-dimethylamine in Soil (NDMA)
An in-house method has also been developed by TestAmerica Westfield for analyzing NDMA (n-Nitroso-di-methylamine) in soil by 8270C Low Level. Through the use of increased sample volume (100grams) and/or reduced final solvent concentration volumes (0.5mL), in addition to increased solvent injection volumes (2-3uL), detection limits of <5.0ug/Kg have been achieved.
An eight-point calibration curve is analyzed, with a calibration range of 0.5 – 100.0ng on column.
Calibration is performed at 0.1, 0.25, 0.5, 1.0, 5.0, 8.0, 10.0, 20.0ug/mL.
Nitrobenzene-d5 is used for the surrogate and the internal standard is 1,4-Dichlorobenzene-d4. These compounds are spiked into all samples at 50ng and 40ng on column respectively.

Title: Determination of Nitrosamines by Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry [GC/CI/MS/MS]

Approvals (Signature/Date):

Douglas Weir — 02/20/09 — Date Technical Manager	Joe Schairer — 2/18/09 — Date Health & Safety Manager / Coordinator
Pamela Schemmer — 2/18/09 — Date Quality Assurance Manager	Karla Buechler — 2/19/09 — Date Laboratory Director

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1. SCOPE AND APPLICATION

1.1. The procedures outlined within this SOP are appropriate for the analysis of aqueous samples for N-Nitrosodimethylamine (NDMA), several related nitrosamines by HRGC/CI/MS/MS. There is also a procedure for the analysis of NDMA in solid samples.

Analyte	Chemical Abstract Services (CAS) Registry Number
N-Nitrosodimethylamine (NDMA)	62-75-9
N-Nitrosomethylethylamine (NMEA)	10595-95-6
N-Nitrosodiethylamine (NDEA)	55-18-5
N-Nitrosodi-n-propylamine (NDPA)	621-64-7
N-Nitrosodi-n-butylamine (NDBA)	924-16-3
N-Nitrosopyrollidine (NPYR)	930-55-2
N-Nitrosopiperidine (NPIP)	100-75-4

1.2. The reporting limit for NDMA is 2.0ng/L in aqueous samples and 5.0ng/g in soil samples. The reporting limit for all other nitrosamine compounds in the method is 10ng/L.

2. SUMMARY OF METHOD

2.1. Samples are spiked with an isotopically labeled internal standard. Aqueous samples are extracted with methylene chloride using separatory funnel extraction procedures. Soil samples are sonicated with methylene chloride. The extract is dried, concentrated to a volume of 0.5 mL, and analyzed by HRGC/CI/MS/MS. Preparation procedures are performed according to SOP WS-IDP-0020.

2.2. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic product ions.

2.3. Quantitative analysis is performed by GC/CI/MS/MS using selected ion current profile (SICP) areas and isotope dilution techniques.

3. DEFINITIONS

3.1. Collisionally Activated Dissociation (CAD) – This is a process of converting the precursor ion's translational energy into internal energy by collisions with neutral gas molecules to bring about dissociation into product ions.

3.2. Large Volume Injection (LVI) – An injection into a gas chromatograph that is larger than the typical 1 or 2uL injection used for hot vaporizing injectors. This method uses 20uL injections.

3.3. Precursor Ion -- For the purpose of this method, the precursor ion is the protonated molecule ($[MH]^+$) of the target analyte. In MS/MS, the precursor ion is mass selected and fragmented to produce distinctive product ions of smaller mass.

3.4. Product Ion -- For the purpose of this method, a product ion is one of the fragment ions produced in MS/MS by collisionally activated dissociation of the precursor ion.

3.5. Programmed Temperature Vaporizing Injector (PTV) – A PTV is a GC injector that is capable of rapid heating. Typical use of a PTV injector involves introducing the sample with the injector cool, then rapidly heating it at 100-200 °C per minute to volatilize the analytes onto the GC column. One advantage of this type of injection is that thermally labile analytes in a mixture can be transferred to the GC column at a lower temperature than in conventional hot injections. It is also useful for large volume injections.

3.6. Other definition of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.7. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw HRGC/HRMS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Note: Toluene, which is a common solvent used in the extraction lab, is suspected to cause matrix related interferences.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety

problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves should be used for methanol.

5.1.2. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.3. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.1.5. The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/CI/MS/MS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph/Mass Spectrometer/Data System (GC/MS/MS/DS)

6.1.1. The GC is a Varian model CP3800 fitted with a Varian CP8400 programmable temperature, large volume injection port (PT/LVI). The detector is a Varian Saturn 2200 ion trap MS/MS. 20uL injections are used.

6.1.2. Deactivated injection port liners are used, and it is highly recommended that they be replaced when necessary with a new deactivated liner. Cleaning and deactivation of injection port liners by the analyst is not recommended

6.1.3. The MS/MS is operated in the chemical ionization mode using methanol as the reagent gas. The conditions and settings detailed in this SOP refer to methanol as the CI gas. The scan time is set so that all compounds have a minimum of 5 scans across the chromatographic peak, when operated in MS/MS mode. Seven to ten scans across chromatographic peaks are strongly recommended.

6.1.4. An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software has the capability of processing stored GC/MS/MS data by recognizing a GC peak within any given retention time window. The software is Varian MS WorkStation version 6.9 SP1

6.2. GC Column - Column - 60 m × 0.25 mm i.d. fused silica capillary column coated with a 1.4 micron bonded film of polyphenylmethylsilicone, (Varian VF-5MS or equivalent). NOTE: Shorter or thinner film columns are not recommended for use with ion trap GC/MS/MS systems because the chromatographic peak widths will be insufficient to obtain a sufficient number of MS/MS scans during peak elution

7. REAGENTS AND STANDARDS

7.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride (DCM).

7.2. High purity Methanol (CH_3OH).

7.3. All calibration, daily internal standard, daily clean up recovery standards, and daily spiking solutions are valid for a maximum of one year from preparation. Secondary standards may not have expiration dates past that of parent solutions.

7.4. Standards are purchased as solutions or mixtures with certification of their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If the chemical purity is 98% or greater, correction for the purity is not needed. When not being used, standards are refrigerated at 4 ± 2°C in screw-capped vials with fluoropolymer-lined or Teflon® - lined caps. A mark is placed on the vial at the level of the solution so that solvent loss by evaporation can be detected. If solvent loss has occurred, the solution should be replaced.

7.5. Sealed ampoules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 1 year from the date the ampoule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.6. Stock Solutions

Prepare stock standards in methylene chloride or equivalent solvent as described below, or purchase as dilute solutions (Cambridge Isotope Laboratories, Cambridge, MA, or equivalent). Observe standard safety precautions. Refrigerate all standard solutions at 4 ± 2°C when not in use.

7.6.1. Dissolve an appropriate amount of assayed reference material in solvent. For example, weigh 1 to 2 mg of NDMA to three significant digits in a 10 mL vial with Teflon® - lined cap.

Stock Solution Composition/Concentration		
Stock Solution	**Component**	**Concentration (ug/uL, in methylene chloride)**
Recovery Standard (RS)	2-Chloropyridine	200
Internal Standard (IS)	D6-NDMA	100
	D14-NDPA	100
Precision and Recovery (PAR)	NDMA	100
	NMEA	100
	NDEA	100
	NDPA	100
	NDBA	100
	NPYR	100
	NPIP	100

7.6.2. Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Reference standards that can be used to determine the accuracy of calibration standards are available from Cambridge Isotope Laboratories and may be available from other vendors.

7.6.3. Secondary standard – Using stock solutions, prepare secondary standard solutions, if necessary they can be further diluted for spiking standard solutions (see below).

7.7. Precision and Recovery Standard (PAR): Also known as the matrix spike or native spike solution. Prepare using the appropriate standard made in Section 7.6 to yield a spiking solution with a concentration of 1 ng/uL in methylene chloride.

7.8. Internal Standard Solution (IS): Also known as the Daily Internal Standard Solution. From stock solutions or purchased mixtures, prepare this solution containing the labeled standards listed in Section 7.6.1 at a concentration of 10 ng/uL in methylene chloride.

7.9. Recovery Standard Solution (RS) - From stock solutions or purchased mixtures, prepare this solution in methylene chloride containing 2-chloropyridine at a concentration of 1.0 ng/uL in methylene chloride.

7.10. Calibration Standards (CS-1 through CS-8, Table 1)

Combine the spiking solutions in Sections 7.7 through 7.9 to produce the eight calibration solutions listed in Table 1. These solutions permit the relative response to be measured as a function of concentration. The CS5 (midpoint) standard is routinely used for calibration verification (Note: any other calibration point within the lower or

upper calibration solution may be used for calibration verification). Refrigerate at 4 ± 2°C when not in use.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document.

8.2. Samples are collected in amber glass containers (liter containers for aqueous samples and 4oz. jars for soil samples) following conventional sampling practices.

8.3. Samples are stored at 4 ± 2°C in the dark, from the time of collection until extraction.

8.4. Aqueous sample extraction is performed within 7 days of sampling. Solid sample extraction is performed within 14 days of sampling. Sample analysis is performed within forty (40) days of extraction.

8.5. Extracts are refrigerated at 4 ± 2°C when not in use.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.1. In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is permitted certain options to improve separations or lower costs of measurements. These options include alternate extraction, concentration, cleanup procedures, and changes in columns and detectors. Unique changes are to be documented using a non-conformance memo. Permanent modifications or additions should be documented using an addendum to this SOP.

9.2. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank and a laboratory control sample.

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC Program document (WS-PQA-003) for further details of the batch definition.

9.3. Control Limits

In-house historical control limits must be determined for laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery ±3 standard deviations.

9.3.1. All internal standard and LCS recoveries must be entered into QuantIMS or other database so that accurate historical control limits can be generated

9.3.2. Refer to WS-PQA-003 for further details of control limits.

9.4. Labeled Internal Standards

Every sample, blank, and QC sample is spiked with internal standards. Internal standard recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits and determine the effect of matrix on the method performance. The composition of the internal standard spiking solutions is detailed in Section 7.8. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to internal standard recoveries being outside QC limits as long as the signal-to-noise is greater than 10:1.

If the recovery is outside the limits in Table 2, the following corrective action should be taken:

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Evaluate the signal-to-noise ratio for the internal standard. Re-extraction of samples should be performed if signal-to-noise for any internal standard is less than 10:1 or if internal standard recoveries fall below 10%. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.4.1. Refer to WS-PQA-003 for further details of the corrective actions.

9.4.2. Recovery of internal standards from samples must be entered into the QuantIMS database for determining control limit adjustments.

9.5. Method Blanks

A laboratory method blank must be run along with each analytical batch of 20 or fewer samples. The method blank is normally analyzed immediately after the calibration standards. The method blank consists of reagent water for aqueous samples, and a clean solid matrix (sand, sodium sulfate, etc.) for solid samples. The method blank must not contain any analyte of interest at or above the minimum levels (ML) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples unless the sample results exceed 10X the blank value.
- If there is no target analyte greater than the minimum levels (ML) in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.5.1. The method blank must have acceptable internal standard recoveries. If recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If internal standard recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.5.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.5.3. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.5.4. Refer to WS-PQA-003 for further details of the corrective actions.

9.6. Laboratory Control Sample (LCS) (may also be noted as “Precision and Recovery (PAR)

9.6.1. An LCS is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The composition of the LCS (PAR) spiking mixture is detailed in Section 7.7.

9.6.2. If any analyte in the LCS is outside the target control limits, corrective action must occur. Corrective action may include:

- Check calculations,
- Check instrument performance,
- Reanalyze the LCS, and if still outside of control limits,
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.6.3. Data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples,
- All target requested analytes are within control, but other LCS compounds are out of control.

9.6.4. The analyst should evaluate the anomalous analyte recovery for possible trends.

9.6.5. If re-extraction and reanalysis of the batch does not occur due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.6. Refer to WS-PQA-003 for further details of the corrective action.

9.7. Duplicates – Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 1 L water sample. A duplicate injection of a sample extract may be performed at the client's request to display instrument precision. The results of the laboratory duplicates (concentrations of the compound requested) should agree within 20 percent relative difference. Report all results and flag any outliers, re-extraction is not required when RPD limits are not met.

9.8. The assessment of matrix effects on method performance, as required by NELAP, is met in all isotope dilution techniques with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair is extracted at the client's request only and is not required by this method. An MS/MSD pair consists of aliquots of a selected field sample spiked with the analyte of known identity and concentration. The MS/MSD are also spiked prior to extraction with the same labeled internal standard as the method blank, laboratory control sample, and field samples. The MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. The results obtained from the MS and MSD samples (percent recovery and concentrations of the requested analytes) should agree within 20 percent relative difference. Report all results, flag any outliers, and file a non-conformance memo for any outliers. Re-extraction is not required when an MS/MSD pair fails to meet one or more QC criteria. An LCS is extracted to show precision of the extraction and analysis process. (An LCS/LCSD pair may be requested by the client, in which case, these usually replace requests for an MS/MSD pair).

9.9. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.11. QC Program

Further details of QC and corrective action guidelines are presented in WS-PQA-003. Refer to this document if in doubt regarding corrective actions.

9.12. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

10.1. Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. Prepare standards containing each analyte of interest at a *minimum* of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. After initial calibration is successful, a second source standard is required for verification of the calibration curve.

10.2. Mass Calibration and CI Reagent Check

10.2.1. Daily, when the instrument is in use, mass calibrate the MS in CI mode using FC-43. On the Varian Saturn 2200, the manifold temperature is 50 ºC and the trap temperature is 150 ºC.

10.2.2. This calibration is conducted using the autotune function of the system. The system will monitor masses 28, 69, 131, 264, 414, 464, 502, and 614. The measured values must be within 0.5 amu of the target masses. Print a copy of the autotune report for inclusion in the instrument logbook.

10.3. Initial Calibration

10.3.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. A new calibration is not required after minor maintenance.

10.3.2. With the exception of the circumstances delineated in policy CA-T-P-002, "Selection of Calibration Points", it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points.

10.3.3. The same injection volume is used for both standards and samples.

10.3.4. All units used in the calculations must be consistently uniform, such as ng/mL and response in area or height.

10.3.5. A minimum of five analytical standards containing different concentrations of the native compounds (see Table 1) is used to generate the curve.

10.3.6. Create a method with the operating parameters that have been established for the GC and the MS (see Tables 3-5).

10.3.6.1. The method file for the initial calibration curve must include the date (mmddyy) in the name.

10.3.7. Open the 8400 Sample List module to set up a run sequence for the ICAL.

10.3.7.1. Activate the method (load the newly created method).

10.3.7.2. Identify as 'New Calibration Block'.

10.3.7.3. Insert the calibration standards (CS-1 … CS-5, at a minimum), along with the 2nd source standard into the run sequence. Set injection volume to 20uL.

10.3.7.4. Update the data file collection and recalculation file to the current date.

10.3.7.5. Start the sequence to begin the ICAL.

10.3.7.6. Check the S/N of CS-1 (criterion is 10:1)

10.3.7.7. Check the high point of the curve for peak saturation.

10.3.8. Calculate the relative response factor (RRF) of the unlabeled compound vs. its internal standard and of the internal standard vs. the recovery standard (Table 1), using the ions specified in Table 4.

Average RRF is determined by calculating the mean RF of each calibration point.

$$RRF = \frac{A_S \times C_{IS}}{A_{IS} \times C_S}$$

Where:

A_S = integrated abundance (peak area) of the quantitation ion of the analyte.

A_{IS} = integrated abundance (peak area) of the quantitation ion internal standard.

Q_S = quantity of analyte injected in ng or concentration units.

Q_{IS} = quantity of internal standard injected in ng or concentration units

10.3.9. Compute the mean RRF, standard deviation (SD) and percent RSD (%RSD) for each compound. Evaluate the %RSD for each compound to determine the acceptability of the ICAL.

10.3.9.1. The %RSD for native NDMA must be ≤ 20%.

10.3.9.2. The %RSD for other native nitrosamines must be ≤ 30%.

10.3.9.3. The %RSD for the labeled compounds must be ≤ 40%.

10.3.10. The acceptable Correlation Coefficient (r) for all compounds must be ≥ 0.995. The Varian 2200 software expresses the goodness of the fit as Coefficient of Determination (r^2). This must be ≥ 0.990.

10.3.11. The calibration curves for the compounds are generally expressed as a linear fit, force through zero, no regression weighting. Quadratic curves have the same Coefficient of Determination, 0.990 for acceptability.

10.4. Second Source Standard -- Each time that a new calibration curve is analyzed a second source standard from an external source is analyzed immediately following the curve.

10.4.1. The acceptance criterion is 25 %D from the expected concentration.

10.5. Documentation for the ICAL consists of printouts for the Calibration Block Report, Calibration Curve Report, Compound Report and Sample Report.

10.6. Continuing Calibration

At the beginning of each 12-hour analytical sequence, in which samples are analyzed, GC/CI/MS/MS system performance and calibration verification for all native and labeled compounds are performed.

10.6.1. Perform a Mass Calibration and CI Reagent Pressure check as per section 10.1.

10.6.2. Ensure that the appropriate method containing the most recent acceptable calibration curve is loaded in the 8400 Sample List.

10.6.3. Insert the Continuing Calibration Verification Standard (CCV) in the 8400 Sample List.

10.6.3.1. A mid-level standard, CS-5 (50pg/uL) is analyzed and quantitated using the regression curve calculated from the initial calibration. Other levels are frequently used to verify the calibration curve.

10.6.3.2. Be sure to update the data file path to the current date along with the Recalclist.

10.6.4. The acceptance criteria for the CCV:

10.6.4.1. NDMA is 80-20%.

10.6.4.2. Other Nitrosamines are 70-130%

10.6.4.3. Labeled compounds are 50-150%

10.6.4.4. Signal-to-noise for all compounds should meet 10:1

10.6.4.5. Relative Retention Time (RRT) within ± 0.06 min., relative to the analyte RRT in the ICAL.

10.6.5. Corrective action for the CCV includes:

10.6.5.1. Re-inject the CS-5 once. If after the second injection, the CCV fails, perform correction action.

10.6.5.2. Some possible corrective actions are: maintenance and/or recalibration of the MS, GC column and injection port maintenance, and preparation of new CAL standards. This list is not meant to be all inclusive.

10.6.5.3. If the CCV is analyzed after corrective action / maintenance, it *must* be analyzed twice and both *must* pass the acceptance criteria. Otherwise repeat the ICAL.

10.6.5.4. Changes in the GC injection or column may affect retention times. After maintenance, verify all retention times before recalibrating.

10.6.5.5. Major maintenance such as cleaning the MS, replacing filament assemblies, replacing the electron multiplier, changing the GC

column, etc. require returning to the initial calibration step (Section 10.2).

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, and chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a nonconformance memo and approved by a supervisor and QA/QC Manager. If contractually required, the client will be notified. The nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A nonconformance memo shall be used for this documentation.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Store extracts in the refrigerator at 4 ± 2°C until analysis.

11.4. Instrumental Analysis

11.4.1. Perform the appropriate calibration outlined in Section 10.

11.4.2. Analyze the sample extracts under the same instrument operating conditions used to perform the instrument calibration (See Section 10).

11.4.3. Record the analysis information in the instrument logbook. Peer review of each analytical sequence is required and must be documented with date and initials. Document and verify each of the following in the instrument logbook: Data File ID, Sample #, Sample ID, Final Volume, Method/Matrix, Sample Size, and Units.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative identification:

12.1.1. Identify a sample component by comparison of its product ion spectrum to a reference spectrum in the user-created data base.

12.1.1.1. All ions present above 30% relative abundance in the product ion mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within an absolute 20%.

12.1.1.2. The precursor ion will have a low relative abundance and is not used in the analyte identification criteria.

12.1.2. The signal-to-noise (S/N) ratio must equal to 2.5 for the unlabeled analyte and 10 for the labeled analyte in a sample extract, and greater than or equal to 10 for both labeled and unlabeled analytes in a calibration standard.

12.1.3. The relative retention time (RRT) of the compound must be within the RRT limits (See Table 2).

12.2. Quantitative determination: The Varian 2200 system utilizes regression curves to calculate the concentrations.

12.2.1. Using the quantitation ions identified in Table 5 for each analyte, calculate the concentration of the sample components using the regression curves established from the initial calibration.

12.2.2. Linear regression equation: (using internal standards).

The linear fit uses the following function:

$$[\, y = ax + b \,]^{*}\mathrm{IS}_{\mathrm{conc}} \quad \text{or} \quad [\, x = \frac{(y-b)}{a} \,]^{*}\mathrm{IS}_{\mathrm{conc}}$$

Where:

y = Instrument response ratio (analyte/internal standard)
x = Concentration
a = Slope
b = Intercept
$\mathrm{IS}_{\mathrm{conc}}$ = Internal standard concentration

12.2.3. Quadratic regression equation: (using internal standards).

The quadratic curve uses the following function:

$$[\, y = ax^{2} + bx + c \,]^{*}\mathrm{IS}_{\mathrm{conc}}$$

Where:

y = Instrument response ratio (analyte/internal standard)
x = Concentration
a = Curvature
b = Slope
c = Intercept
$\mathrm{IS}_{\mathrm{conc}}$ = Internal standard concentration

12.2.4. Adjust final analyte concentrations to reflect the actual sample volume used in

the preparation.

$$FC = Cx \ / \ Vol$$

Where:

FC = the final analyte concentration, ng/L
Cx = the instrument analyte concentration
Vol = the sample size used for the extraction (in liters)

12.3. Analysis of complex samples.

12.3.1. Some samples contain high levels (>500 ng/L) of the compounds of interest, interfering compounds, and/or polymeric materials. In the case of high levels of one or more analytes such that the concentration in the extract exceeds the upper calibration limit and/or causes saturation of the MS detector, the extract is diluted with methylene chloride to bring the concentration of the analyte in question below the limit of the detector and into the calibration range if possible. If high levels of co-extracted interfering compounds are a problem, it may be necessary to extract and analyze a smaller aliquot of the sample. Complex dilutions may be required per Section 12.5.5. The client will be contacted to determine whether or not they will require this procedure.

12.3.2. For internal standard recoveries outside the recommended recovery range the data should be evaluated for adverse impact. If no significant adverse impact was observed the data should be reported and an NCM filed. For samples that may be or are adversely impacted, an additional aliquot of the original extract or diluted extract should be analyzed. If the recoveries are still outside of the range for the re-injected extract and it is determined that significant adverse impact to data quality has occurred, the sample(s) may be re-extracted, or reprocessed from an archive portion if available. A smaller aliquot should be used for the re-extraction unless it is suspected that the recovery problem is not matrix related. If the re-extracted sample(s) still does not meet the limits for labeled internal standard recovery, the data is evaluated for adverse impact and an NCM filed. All recoveries outside of method criteria will be flagged. The best quality data is reported from one of the two extractions unless the client requests that both sets of data be reported.

12.4. Data review

12.4.1. Documentation for each analytical sequence is to include the following:

- Run sequence (displaying sample name, sample type, injection notes, injection volume).
- Sample report (displaying sample id, operator, acquisition date, instrument ID, data file ID and method name). The detail of the report

will have: peak name, RT, status, area, amount/RF, S/N and calibration equation.

- Compound report (multi-window report displaying compound peak and spectrum).

12.4.2. The analyst who performs the initial data calculations must initial and date the front quantitation summary of the raw data for each sample (the CCVs or ICALs have separate specific checklists) to document that they have performed the qualitative and quantitative analysis on the sample/QC data.

12.4.3. When an entire project lot or batch is completed, a checklist representing the entire data package is completed.

12.4.4. All manual integrations must be documented according the Manual Integration SOP, CA-Q-S-002, and the Laboratory Addendum.

12.4.4.1. Manual integrations on the Varian software can be identified by a 'U' (user defined end points) on the sample report under the column 'Status'.

12.4.5. A second analyst must verify all qualitative peak identifications. If discrepancies are found the data should be returned to the analyst who performed the initial peak identification for resolution. Any changes made directly by the reviewing analyst must be dated and initialed by the reviewing analyst.

12.4.6. A second analyst must check all hand calculations and data entry into calculation programs, databases, or spreadsheets at a frequency of 100 percent. If discrepancies are found, the data should be returned to the analyst who performed the initial calculation(s). Any changes made directly by the reviewing analyst must be dated and initialed by the reviewing analyst.

12.4.7. Both the analyst who performed the initial qualitative and quantitative analysis and the analyst who performed the second level review must check all items on the data review checklist.

12.5. Reporting Results

12.5.1. Aqueous analytical results are reported in ng/L (parts per trillion).

12.5.2. Report results at or above the minimum level (also known as the Lower Calibration Limit – See Table 2).

12.5.2.1. When target analytes meet method identification criteria and are free of interferences, they are reported as unqualified positives down to the Reporting Limit.

12.5.3. Report results below the minimum level as not detected.

12.5.3.1. Below the Reporting Limit, qualitatively confirmed analytes are reported as "estimated" down to the MDL. To denote the less certain quantitation the value is "J" flagged as estimated.

12.5.4. If the concentration of any target analyte exceeds the upper calibration range of the initial calibration curve, the laboratory will provide one dilution to bring the response below the saturation level and/or the upper calibration level as part of the initial analysis cost.

12.5.5. Additional dilutions or re-extraction at a smaller sample aliquot will be performed at client request only. (Note: Complex dilution is one where the pre-spiked internal standards are diluted out and must be re-added to the extract. The sample analyte concentrations are then calculated using an internal standard method and all results from this dilution are therefore qualified.)

12.5.6. Results are reported to two significant figures. For aqueous samples, the units are ng/L.

12.6. Internal standard: Compute the concentrations of the labeled analogs in the extract using the regression curve determined from the initial calibration and the equation in 12.2.1 or 12.2.2.

12.7. Calculate the internal standard percent recovery using the following equation:

$$\%\,\text{Recovery} = \frac{C_{IS}}{\text{Amount Spiked}} \times 100$$

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION PREVENTION

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001.

15.1. The autovials that contain the extract to be analyzed contain methylene chloride. After the septum is pierced by the instrument, any remaining methylene chloride (as well as analytes dissolved in the solvent) can evaporate into the air. Do not allow them to site on the tray for an extended period of time. Remove the autovials from the instrument after analysis, replaced the pierced septa with new septa, and return them to long-term storage until released for disposal.

16. REFERENCES/CROSS REFERENCES

16.1. EPA Document # EPA/600/R-05/054, Method 521 Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS), Revision 1, September 2004.

16.2. EPA Method 1625, Revision B, Semivolatile Organic Compounds by Isotope Dilution GC/MS, June 1984.

16.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, December 1996

17. METHOD MODIFICATIONS

17.1. This is an analytical method that was developed in-house, hence, there are no specific method modifications.

18. ATTACHMENTS

18.1. Table 1 - Concentrations of Analytes in Calibration and Calibration Verification Solutions

18.2. Table 2 – Acceptance Criteria for Performance Tests

18.3. Table 3 – Chemical Ionization Parameters for a Varian Saturn 2000 using methanol as the reagent gas

18.4. Table 4 – (4a,4b,4c) Operating parameters for the: a) Large Volume Injector (LVI) using a Programmable Temperature Vaporizing Injector (PTV); b) Injector split vent program; c) GC Oven temperature program.

18.5. Table 5 – Quantitation Ions

18.6. Table 6 – MS/MS Parameters for the Varian Saturn 2000 using Methanol as the CI Reagent.

19. REVISION HISTORY

19.1. WS-MS-0012, Revision 1, Effective 2/20/2009

Table 1
Concentration of analytes in calibration and calibration verification solutions

Compound	Concentration (ng/uL)							
	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8
Native								
NDMA	2	5	10	20	50	100	200	500
NMEA	2	5	10	20	50	100	200	500
NDEA	2	5	10	20	50	100	200	500
NDPA	2	5	10	20	50	100	200	500
NDBA	2	5	10	20	50	100	200	500
NPYR	2	5	10	20	50	100	200	500
NPIP	2	5	10	20	50	100	200	500
Labeled								
D6-NDMA	100	100	100	100	100	100	100	100
D14-NDPA	100	100	100	100	100	100	100	100
Recovery Standard								
2-Chloropyridine	200	200	200	200	200	200	200	200

TABLE 2
Acceptance Criteria for Performance Tests

Component	Conc. (ng/mL)	OPR (LCS) (%)	VER (CC3) (%)	RL/LCL /MDL (ng/L)	RRT Limits
N-Nitrosodimethylamine	50	70-130	80-120	2	0.06
N-Nitrosodimethylamine	50	60-140	60-140	5ng/g (soils)	0.06
N-Nitrosomethylethylamine	50	70-130	70-130	10	0.06
N-Nitrosodiethylamine	50	70-130	70-130	10	0.06
N-Nitrosodi-n-propylamine	50	70-130	70-130	10	0.06
N-Nitrosodi-n-butylamine	50	70-130	70-130	10	0.06
NNitrosopiperidine	50	70-130	70-130	10	0.06
N-Nitrosopyrrolidine	50	70-130	70-130	10	0.06
D6- N-Nitrosodimethylamine	100	25-150	50-150	--	0.06
D14- N-Nitrosodi-n-propylamine	100	25-150	50-150	--	0.06
2-Chloropyridine	200	--	--	--	

Table 3 **Chemical Ionization Parameters for a Varian Saturn 2000 GC/MS/MS Using Methanol or Acetonitrile as the CI Reagent**	
CI storage level (*m/z*)	15-19
Ejection amplitude (*m/z*)	10-15
Background mass (*m/z*)	40
Maximum ionization time (µsec)	2000
Maximum reaction time (µsec)	100-120
Target total ion current (TIC) (counts)	5000
Prescan Ionization time (µsec)	100-200
Emission current (µamps)	50
Election Multiplier offset (V)	+300

Table 4a
Chromatographic Conditions including LVI Using a PTV Injector

Temp (^oC)	Rate (^oC/min)	Time (min)
35	0	0.5
250	200	1.08
250	0	34.0

Table 4b
Injector Split Vent Program

Time (min)	Split Status
0	Open
0.5	Closed
3.5	Open
5.5	Open

Table 4c
GC Oven Temperature Program

Temp (^oC)	Rate (^oC/min)	Hold Time (min)
35	0	2.5
105	10	0
215	5.5	4.5
255	25	1.0

Table 5
Quantitation Ions

Analyte	Retention Time (min)	Precursor Ion (*m/z*)	Quantitation Ion/Product Ion (*m/z*)
NDMA	13.93	75	47+44 (58)
NMEA	16.25	89	61 (71,90)
NDEA	18.27	103	75 (83,101)
NPYR	23.99	101	55 (99,102)
NDPA	23.28	131	89 (112,129)
NPIP	25.06	115	69 (70,116)
NDBA	28.50	159	57+103 (102)
NDMA-d6 (Surrogate)	13.86	81	49 (63,64)
NDPA-d14 (Internal Standard)	23.06	145	97 (96)

Table 6
Typical MS/MS Parameters for the Varian Saturn 2000 Using Methanol as the CI Reagent

Analyte	Segment	Retention Time Window (min)	Scan Range (*m/z*)	Scan Rate (sec/scan)	Excitation Amplitude (volts)	Excitation Storage Level (*m/z*)	Excitation Time (msec)	Precursor Ion Isolation Time (msec)
NDMA	1	13.00-15.50	40-90	0.55	0.40	40.0	10	3.0
NDMA-d6 (Surrogate)	1	13.00-15.50	40-90	0.55	0.45	45.0	10	3.0
NMEA	2	15.50-17.30	52-95	0.55	0.35	55.0	10	3.0
NDEA	3	17.30-22.00	65-110	0.55	0.35	67.0	10	3.0
NDPA-d14 (Internal Standard)	4	22.00-23.70	80-150	0.56	0.45	92.0	10	3.0
NPYR	5	23.70-24.30	50-110	0.56	0.40	52.0	10	3.0
NDPA	5	22.00-23.70	80-150	0.56	0.40	84.0	10	3.0
NPIP	6	24.30-27.00	60-120	0.56	0.40	64.0	10	3.0
NDBA	7	27.00-32.00	50-165	0.58	0.40	52.0	10	3.0

Waveform type- Resonant; Default values for all other MS/MS parameters: Isolation window 3 amu, Low edge offset 6 steps, High edge offset 2 steps, High edge amplitude 30 V, Modulation range 2 steps, Modulation rate 3000 :sec/step, # of Frequencies 1, CAD frequency offset 0 Hertz, Ejection Amplitude 20 V; Ionization RF 48 *m/z*

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STANDARD OPERATING PROCEDURE

Analytical Procedures for the Analysis of Extractable Petroleum Hydrocarbons (EPH)

Method MADEP EPH

SOP ID: GCS00300.MA
Rev 3

Date Written: 03/05/97
Date Revised: 12/05/06
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Approved Signatures

Christine Lucinda Reynolds

QUALITY ASSURANCE COORDINATOR

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1.0 TITLE

Analytical Procedures for the Analysis of Extractable Petroleum Hydrocarbons (EPH)

2.0 MATRIX

Organic Sample Preparation Laboratory
Organic Instrument Laboratory

3.0 REPORTING LIMIT

3.1 Waters: Target RLs are 10ug/L and Carbon Ranges are 100ug/L.
3.2 Soils: Target RLs are 0.333mg/Kg, and Carbon Ranges are 3.33mg/Kg.

4.0 SCOPE AND APPLICATION

4.1 This SOP is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. Extractable aliphatic hydrocarbons are collectively quantitated within two ranges: C_9 through C_{18}, and C_{19} through C_{36}. Extractable aromatic hydrocarbons are collectively quantitated within the C_{11} through C_{22} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150° C and 500°C.

4.2 This method is also designed to measure the individual concentrations of targeted polynuclear aromatic hydrocarbons (PAH) in water and soil. A listing of these targeted analytes is provided in Table 1.

4.3 Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel, and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, and other petroleum products which contain a significant percentage of hydrocarbons lighter than C_9. This method, in and of itself, is also not suitable for the evaluation of petroleum products which contain a significant percentage of hydrocarbons heavier than C_{36}.

4.4 This SOP is based on a solvent extraction, silica gel solid-phase extraction/ fractionation process (SPE), and gas chromatography (GC) analysis using a flame ionization detector (FID). This method can also be analyzed using a GCMS as stated in Appendix I. This procedure should be used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

Table 1. Aromatic Hydrocarbon Standard/Targeted PAH Analytes

Number	Compound	CAS Number
1	Naphthalene	91-20-3
2	2-Methylnaphthalene	91-57-6
3	Acenaphthylene	208-96-8
4	Acenaphthene	83-32-9
5	Fluorene	86-73-7
6	Phenanthrene	85-01-8
7	Anthracene	120-12-7
8	Fluoranthene	206-44-0
9	Pyrene	129-00-0
10	Benzo(a)Anthracene	56-55-3
11	Chrysene	218-01-9
12	Benzo(b)Fluoranthene	205-99-2
13	Benzo(k)Fluoranthene	207-08-9
14	Benzo(a)Pyrene	50-32-8
15	Indeno(1,2,3-cd)Pyrene	193-39-5
16	Dibenzo(a,h)Anthracene	53-70-3
17	Benzo(g,h,i)Perylene	191-24-2

5.0 SUMMARY OF METHOD

5.1 A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated in a Turbovap, nitrogen blow down device. Sample cleanup and separation into aliphatic and aromatic fractions is conducted using commercially available silica gel cartridges. The two extracts produced are then re-concentrated to final volumes of 1 mL each (i.e., an aliphatic extract and an aromatic extract). The extracts are then separately analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram of aliphatic compounds is collectively

integrated within the C_9 through C_{18} and C_{19} through C_{36} ranges. The resultant chromatogram of aromatic compounds is collectively integrated within the C_{11} through C_{22} range, and is used to identify and quantitate individual concentrations of targeted PAH analytes.

5.2 Average calibration factors or response factors determined using an aliphatic hydrocarbon standard mixture are used to calculate the collective concentrations of C_9 through C_{18} and C_{19} through C_{36} aliphatic hydrocarbons. An average calibration factor or response factor determined using a PAH standard mixture is used to calculate a collective C_{10} through C_{22} aromatic hydrocarbon concentration. Calibration factors or response factors determined for individual components of the PAH standard mixture are used to calculate individual concentrations of targeted PAH analytes.

6.0 DEFINITIONS

6.1 *Aliphatic Hydrocarbon*: Any organic compound comprised solely of carbon and hydrogen characterized by a straight, branched or cyclic chain of carbon atoms. This class of organic compounds includes alkanes, alkenes, alkynes, cycloalkanes or cycloalkenes.

6.2 *Aromatic Hydrocarbon*: Any cyclic and conjugated organic compound comprised solely of carbon and hydrogen. Aromatic compounds of environmental significance are benzoids that contain benzene or fused benzene rings.

6.3 *Extractable Petroleum Hydrocarbon*: Any hydrocarbon that elutes within the C9 through C18 and C19 through C36 aliphatic, or the C11 through C22 aromatic ranges defined by the method. The definition of Extractable Petroleum Hydrocarbon specifically **excludes** all substituted aliphatic or aromatic hydrocarbon derivatives (non-hydrocarbons as defined by the EPH Method), the individual EPH Method Target and Diesel PAH Analytes, surrogates, and/or internal standards that co-elute within these method-specific ranges. The EPH Method is suitable for the separation and quantification of the aliphatic and non-target aromatic components of kerosene, fuel oil #s 2, 4 and 6, diesel fuel, jet fuel (JP-4, 5 and 8) and certain hydrocarbon-based, low to medium viscosity lubricating oils contained within the aforementioned method-defined ranges (C9 through C36). These aliphatic hydrocarbon ranges correspond to a boiling point range between approximately 150°C and 265°C. Consequently, the EPH Method, in and of itself, is not suitable for the evaluation of lower boiling petroleum products (gasoline, mineral spirits, or certain petroleum naphthas) or higher boiling petroleum products (asphalts, tars, etc) outside the dynamic range of this method.

6.4 *Total Petroleum Hydrocarbons (TPH)*: The collective concentration associated with the PAC for all peaks corresponding to any fractionated or unfractionated aliphatic and/or aromatic compounds eluting between 0.1 minutes before the retention time for n-C9 to 0.1 minutes after the Rt for n-C36, **excluding** the PAC for all substituted aliphatic or aromatic hydrocarbon derivatives, the individual EPH Method Target and Diesel PAH Analytes, surrogates, and/or internal standards that co-elute within this chromatographic range. The Department recommends that the analysis of the unfractionated EPH extract be used as a

conservative estimate of TPH, as this term is defined in 310 CMR 40.0006, when this parameter is used to support human health risk characterization or other MCP assessments and evaluation decisions.

6.5 *Accuracy*: the degree of agreement between an observed value and an accepted reference value.

6.6 *Batch*: environmental samples, which are prepared and/or analyzed together with the same process using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.7 *Holding Time*: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

6.8 *Instrument Blank*: a blank matrix that is introduced onto the instrument for analysis and can monitor calibration baseline drift and carryover contamination effects.

6.9 *Laboratory Control Sample (LCS)*: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

6.10 *Matrix Duplicate (MD)*: duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

6.11 *Matrix Spike (MS)*: field sample to which a known amount of target analyte(s) is added.

6.12 *Matrix Spike Duplicate (MSD)*: a replicate matrix spike.

6.13 *Method Blank*: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

6.14 *Method Detection Limit (MDL)*: the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR , Part 136, Appendix B).

6.15 *Non-conformance*: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

6.16 *Precision*: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data

quality indicator.

6.17 *Reporting Limit (RL):* The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above
the MDL.

6.18 *Quality Assurance (QA)*: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

6.19 *Quality Control (QC)*: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

6.20 *Raw Data*: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

6.21 *Sensitivity*: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

6.22 *Standard Operating Procedure (SOP)*: a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Like all GC procedures, this method is subject to a "false positive" bias in the reporting of targeted PAH analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a targeted PAH analyte. While cleanup procedures specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analysis by dissimilar columns, gas chromatography/mass spectrometry (GC/MS) analysis, or other suitable technique is available in cases where a targeted PAH analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where coelution of a non-targeted hydrocarbon compound is suspected.

7.2 Interferences in the GC analysis of petroleum hydrocarbons result from non-petroleum derived organics that are extracted along with the target compounds. No effort is made in this SOP to identify or account for this effect.

7.3 Samples that contain high molecular weight compounds may leave residues throughout the GC system, which degrade GC performance in subsequent injections.

7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is

analyzed, it must be followed by the analysis of a solvent blank to check for cross-contamination.

7.5 Interfering Peaks in Specified Aliphatic Hydrocarbon Ranges:
Hydrocarbons (and non-hydrocarbons), even with elution times within the defined chromatographic windows for the aliphatic hydrocarbon ranges specified by the EPH Method, need not be included in the PAC for these ranges unless they meet the definitions of aliphatic hydrocarbon and extractable petroleum hydrocarbon, as defined above. If the concentration of a hydrocarbon range is based on one (or just a few) peaks within the range and an indicative petroleum hydrocarbon peak pattern is not apparent, the laboratory should provide this information and alert the data user of the potential for a false positive result in the Environmental Laboratory case narrative. MCP sites with co-mingled non-petroleum hydrocarbons such as vegetable oils, synthetic oils and lubricants, and some naturally occurring humic materials are particularly susceptible to this type of interference.

7.6 Interfering Peaks in Specified Aromatic Hydrocarbon Range:
The EPH Method should be used with caution at sites with uncertain history and disposal practices, particularly at sites where other hazardous materials were used, stored and/or managed. Such contaminants, if encountered, may co-elute within the method-defined aliphatic and or aromatic ranges resulting in an overestimation of the concentration (i.e., positive interference).

7.7 Evaluation of Individual Hydrocarbons Not Associated with an Extractable Petroleum Hydrocarbon:
In general, it may be prudent for the client to confirm all FID data using SW-846 Method 8270C (GC/MS) if critical MCP decision-making (notification, compliance with cleanup standards, risk assessment, etc.) is based solely on the EPH Method (or any other non-specific GC analysis). If a positive interference is suspected from hydrocarbons and/or non-hydrocarbons not associated with EPH in either aliphatic or the aromatic fraction or with a Target or Diesel PAH Analyte, and such interference would adversely effect MCP decision-making, if confirmed, then SW-846 Method 8270C, Semi-Volatile Organics by GC/MS, should be employed to accurately identify and quantify the components that comprise a fraction or to resolve any uncertainty regarding the identification of a specific Target or Diesel PAH Analyte. It is recommended that the chromatographic conditions specified under SW-846 Method 8270C be modified for consistency with the conditions specified by the EPH Method to better allow for a direct comparison of the suspect FID peaks with the GC/MS system. This is particularly useful when comparing suspect. aliphatic hydrocarbons. The electron impact mass spectra for aliphatic hydrocarbon homologues are not particularly unique and chromatographic relative retention time data may also be required to confirm suspect EPH data.

8.0 SAFETY

8.1 Methylene chloride, PAHs, and the petroleum target blends are known or suspected carcinogens.

8.2 Proper precautions should be taken when formulating new standards, or handling suspected high concentration samples. Care should be taken when handling the electron capture detector. It should never be open as it contains radiochemical sources.

8.3 Procedures shall be carried out in a manner that protects the health and safety of all STL employees. Eye protection that satisfies ANSI Z87.1, a laboratory coat, and

appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Appropriate gloves for the inorganic department and sample receiving shall be made of 100% powder-free, non latex and composed of Nitrile.

8.4 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

8.5 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
The preparation of standards and reagents should be conducted in a fume hood with the sash closed as far as the operation will permit.

8.6 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL employee. The situation must be reported **immediately** to a laboratory supervisor.

9.0 APPARATUS AND MATERIALS

9.1 Hewlett Packard 5890 Series II (FID); Model 7673 ALS (Dual).
9.2 30-m x 0.32-mm I.D., 0.25-µm film RTx5 column (J&W) or equivalent. This column will allow for the adequate resolution of alkanes from n-C_9 to n-C_{36}. This column will also resolve the targeted PAH analytes listed in Table 1.
9.3 Hamilton 10 µL manual and ALS syringes.
9.4 HP Chemstation Data System
9.5 1 Liter amber glass bottles
9.6 4 oz (120mL) amber wide mouth glass jars
9.7 2-L Separatory funnels
9.8 Volumetric flasks, 10, 25, 50 and 100mL
9.9 Analytical balance
9.10 Blowdown apparatus: air or nitrogen

10.0 REAGENTS AND STANDARD PREPARATION

10.1 Solvents
10.1.1 Methylene chloride (MCL), Acetone and Hexane – JD Baker high purity.
10.2 Analytical Standards
10.2.1 Stock solutions of target petroleum blends (aromatic and aliphatic) are purchased commercially and diluted to working standards in two steps. The initial dilution consists of combining the respective standard with the appropriate surrogate and/or fractionation stock (see sections below) into a single 200 ppm mix:

Target Compound	Vendor	Conc. (mg/L)	Catalog Number	Spike Amt (µL)	Final Volume (mL)	Solvent	Stock Conc (mg/L)
Aliphatic Hydrocarbons	Accu-standard	1000	DRH-007S	1000	5	Hexane	200

COD*	Section 10.4	100,000	23,836-8	10	5	Hexane	200
PAHs	Accu-standard	1000	DRH-006S	1000	5	Hexane	200
OTP*	Section 10.4	100,000	T280-0	10	5	Hexane	200
2FBP**	Section 10.5	10,000	10,274-1	100	5	Hexane	200
2BN**	Section 10.5	10,000	18,364-4	100	5	Hexane	200

* Surrogate standard stocks. See section 10.4.
** Fractionation standard stocks. See section 10.5

These initial blends are diluted to working standards according to the following table:

Target Compound	Stock Volume (µL)	Solvent*	Final Volume (mL)	Concentration (µg/mL)
PAHs/AH	5	MeCL/Hex	2	0.5
PAHs/AH	10	MeCL/Hex	2	1
PAHs/AH	50	MeCL/Hex	2	5
PAHs/AH	100	MeCL/Hex	2	10
PAHs/AH	250	MeCL/Hex	2	25
PAHs/AH	500	MeCL/Hex	2	50
PAHs/AH	1000	MeCL/Hex	2	100
PAHs/AH	2000	MeCL/Hex	2	200

* PAH standards are prepared in methylene chloride, AH standards in hexane.

10.2.1 The standards must be replaced in 6 months or sooner if QC criteria in this SOP cannot be met.

10.3 Matrix Spike/Laboratory Control Sample Spiking Solution

10.3.1 The spiking solution for both matrix and laboratory control sample spiking is produced by diluting a commercially available blend:

Target Compound	Vendor	Conc. (mg/L)	Catalog Number	Spike Amt (µL)	Final Volume (mL)	Solvent	Spiking Soln Con (mg/L)
Matrix Spike	Accu-standard	1000	DRH-MA-MS-40X	2000	10	Acetone	200

10.3.2 The matrix spiking solution must be replaced in 6 months or sooner if QC criteria in this SOP cannot be met unless superceded by manufacture's date.

10.4 Stock solutions of the surrogate standard compounds are prepared according to the following table:

Target Compound	Vendor	Catalog Number	Amount of neat material	Solvent	Final Volume	Stock Concentration
COD	Aldrich	23,836-8	1.00 g	MCL	10 mL	100,000 µg/mL
OTP	Aldrich	T280-0	1.00 g	MCL	10 mL	100,000 µg/mL

COD =chloro-octadecane; OTP=orthoterphenyl.

10.4.1 The surrogate standard spiking solution must be replaced in 6 months or sooner if QC criteria in this SOP cannot be met.

10.4.2 All investigative and laboratory extracts are spiked with 1.0 mL of a 200 µg/mL blend of the stocks described above. The blend is made by adding 200 µL of each stock to 100 mL of acetone. The resulting concentration of the working solution is 200 µg/mL.

10.5 Fractionation Surrogate Standards are spiked into the sample extract in Sep-Pak prior to fractionation, in order to determine fractionation efficiency. The fractionation surrogate standards are prepared according to the following table.

Target Compound	Vendor	Conc. (mg/L)	Catalog Number	Amount	Final Volume (mL)	Solvent	Final Conc* (mg/L)
2-FBP	Aldrich	Neat	10,274-1	0.1000g	10 mL	MCL	10,000
2-BN	Aldrich	Neat	18,364-4	0.1000 g	10 mL	MCL	10,000
2-FBP	Stock (above)	10,000	--	1 mL	2 mL	Hexan e	5000
2-BN	Stock (above)	10,000	--	1 mL	2 mL	Hexan e	5000

* Combined to a single solution.
2FP=2-Fluorobiphenyl; 2BN=2-Bromonaphthalene

10.5.1 The fractionation standards must be replaced in 6 months or sooner if QC criteria in this SOP cannot be met.

10.5.2 All investigative and laboratory extracts are spiked with 10 µL of the 5000 µg/L blend after the extract is transferred to the Sep-Pak cartridge. The final extracts result in an expected GC concentration of 50 µg/mL.

10.6 Fractionation Check solution

10.6.1 The Fractionation Check Solution is used to monitor the fractionation efficiency of the Sep-Pak cartridges as well as check for sample preparation errors (e.g., insufficient/excessive hexane use).

10.6.2 The spiking solution for the Fractionation Check Solution is purchased as a ready to use ampul in hexane (cat# DRH-MS-ASL)

10.7 Standard preparation is documented in the department standards prep logbook. All

lot numbers and dilution procedures must be entered.

10.8 Calibration standards, and matrix and surrogate spike solutions must be verified before they are used in investigative samples. This is accomplished by calibrating the instrument for PAHs and HCs and injecting the matrix spiking solution against the calibration curve. The matrix spike solution must return ±15% of its nominal concentration. Failure to achieve this criterion requires corrective action.

11.0 SAMPLE CONTAINERS, COLLECTION, AND PRESERVATION

11.1 Aqueous samples are collected in 1 liter amber glass bottles with Teflon-lined screw caps.

11.2 Soil and sediment samples are collected in 4 oz. (120 mL) amber wide-mouth glass jars with Teflon-lined screw caps.

11.3 Aqueous samples must be preserved at the time of sampling by the addition of acid to reduce the pH of the sample to less than 2.0. This is accomplished by providing sample containers with 5 mL of 1:1 HCl. Following collection and addition of acid, the sample must be cooled to 4°C.

11.4 Soil and sediment samples must be cooled to 4°C immediately after collection.

11.5 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

11.6 Aqueous and solid samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

11.7 Alternatively, soils samples can be frozen for up to 1 year of collection. Samples must be prepared within 24 hours of thawing.

11.8 In the event that sample shipment is delayed in the field or in transport, Project Management will assess the laboratory's ability to process the samples within the prescribed holding time and advise the client if holding times cannot be met.

11.9 If the sample is received at the laboratory already out of protocol, Project Management notifies the client to determine if the sample should be analyzed.

12.0 QUALITY CONTROL

12.1 Method detection limits are determined in accordance with the Corporate SOP for Conducting MDL Studies. MDLs must be less than or equal to the reporting limit. MDLs, shown below, are typical of the method and are performed annually:

Number	Compound	Aqueous MDL µg/L	Soil MDL mg/kg
1	Naphthalene	3.1	0.15
2	2-Methylnaphthalene	3.5	0.09
3	Acenaphthylene	3.7	0.15
4	Acenaphthene	3.9	0.15
5	Fluorene	3.6	0.12
6	Phenanthrene	3.9	0.10
7	Anthracene	3.9	0.10
8	Fluoranthene	4.1	0.13
9	Pyrene	4.2	0.17
10	Benzo(a)Anthracene	4.4	0.12
11	Chrysene	4.6	0.13
12	Benzo(b)Fluoranthene	5.4	0.16
13	Benzo(k)Fluoranthene	5.5	0.17
14	Benzo(a)Pyrene	4.9	0.22
15	Indeno(1,2,3-cd)Pyrene	4.8	0.10
16	Benzo(a,h)Anthracene	5.2	0.12
17	Benzo(g,h,i)Perylene	6.1	0.09
18	Nonane; C9	2.9	0.07
19	Decane; C10	6.0	0.03
20	Dodecane; C12	4.4	0.06
21	Tetradecane; C14	5.2	0.07
22	Hexadecane; C16	6.2	0.07
23	Octadecane; C18	5.7	0.19
24	Nondecane; C19	5.5	0.06
25	Eicosane; C20	5.3	0.08
26	Docosane; C22	5.5	0.10
27	Tetracosane; C24	5.4	0.10
28	Hexacosane; C26	6.8	0.04
29	Octacosane; C28	5.5	0.04
30	Triacontane; C30	5.1	0.04
31	Hexatriacontane; C36	6.3	0.09

12.2 The method blank is analyzed to ensure there is no background level of contaminants above the PQL. If the method blank contains reportable TPH concentrations the batch may require re-extraction. Method blanks are extracted with every batch and every 20 samples.

12.3 A matrix spike, matrix spike duplicate and sample duplicate (optional – only if requested by client) should be extracted and analyzed every 20 samples, or per batch. The recovery and RPD limits are 40-140% and ±50 respectively. If the recoveries do not meet the criteria, the LCS recovery must be consulted. The LCS recovery limits are 40-140%. If the LCS recovery is acceptable, and the MS fails the same criteria, then matrix interference is assumed and the LCS defines acceptable batch QC. The first eluting CN compound (nonane) generally does not achieve the criteria above. The recovery values for this compound is 30-140%.

12.4 A Laboratory Control Sample (LCS) and duplicate (LCSD) is extracted and analyzed every 20 samples or per batch, which ever is sooner. The LCS recovery must meet the acceptance criteria of 40-140%, RPD ≤25%. If the LCS fails to meet the criteria, a reanalysis or re-extraction may be required. The first eluting CN compound (nonane-C9) generally does not achieve the criteria above. The recovery value for this compound is 30-140%.

12.5 Extraction surrogate recovery limits for the samples, blanks and spikes is 40-140%. If the surrogate fails, the sample may be re-analyzed or re-extracted. Some highly contaminated samples obscure the region of the chromatogram in which the surrogate compounds elute and thus make surrogate recovery measurements unreliable. For this reason, analysts must be alert to sample introduced matrix effects.

12.6 Fractionation surrogate recovery limits for the samples, blanks and spikes is 40-140%. If one of or both surrogates fail, the sample may be re-analyzed or re-fractionated. If the extract results in no detectable amounts of target compounds, the corrective action requirement is waived.

12.7 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

12.7.1 The n-nonane (C_9) peak must be adequately resolved from the solvent front of the chromatographic run.

12.7.2 The surrogates 2-FBP, 2-BN, COD and OTP and any internal standards used must be adequately resolved from any individual components in the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.

12.7.3 All peaks from target compounds in the Aliphatic Hydrocarbon standard must be resolved to baseline. In the Aromatic Hydrocarbon standard, baseline separation is expected for Phenanthrene, Anthracene, Benzo(a)Anthracene, Chrysene, Benzo(b)Fluoranthene and Benzo(k)flouranthene. Dibenzo(a,h)Anthracene, Indeno(1,2,3-cd) Pyrene are not expected to be chromatographically separated to baseline; however, sufficient separation should be obtained to 25% of the baseline.

12.8 The fractionation check is performed with each new lot of fractionation cartridges. The fractionation procedure is described below:

12.8.1 Pre-rinse a 5 g/20 mL commercially prepared silica gel Sep-Pak cartridge with 20mL of Acetone and vacuum dry. Next add 20mL of Methylene chloride and also vacuum dry. Finally rinse the cartridge with 60 mL of hexane. Let the solvent flow through the column until the head of the liquid in the column is just above the column frit of the Sep-Pak cartridge. Close the stopcock and discard the hexane rinsate.

12.8.2 Load 1.0 mL of the Fractionation Check Solution (or sample) onto the Sep-

Pak the solution. Just prior to exposure of the column frit to the air, elute the column with 19.0 mL more hexane. Collect the eluant in a 25-mL volumetric flask and label this fraction "aliphatics".

NOTE: The amount of hexane used during Sep-Pak extraction is critical. Excessive hexane will cause elution of aromatics into the aliphatic fraction. Insufficient hexane will cause low recoveries of the aliphatic fraction. The volume of hexane recommended (20.0 mL) may need to be adjusted to meet QC limits.

12.8.3 Following recovery of the aliphatic fraction, elute the column with 25 mL of methylene chloride and collect the eluant in a 25-mL volumetric flask. Label this fraction "aromatics".

12.8.4 Transfer the contents of the two 25-mL volumetric flasks into two separately labeled graduated concentrator tubes. Concentrate each solution to a final volume of 1.0 mL under a gentle stream of air or nitrogen from an air blow-down apparatus.

12.8.5 Transfer the final 1.0 mL extracts to two labeled glass auto sampler vials with teflon-lined rubber crimp caps. The sample is ready for analysis.

12.8.6 For each analyte within the Fractionation Check Solution, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%

12.9 ACCEPTANCE OF DATA

12.9.1 Silica cartridges must not be overloaded with excessive mass of hydrocarbons. Loading should be limited to 5mg total hydrocarbon per gram of silica gel. For a 1.0 mL extract fractionated on a 5 gram cartridge, this equates to a max loading of 25,000 µg/mL. If exceeded, extract needs to be re-fractionated with a smaller volume.

12.9.2 The daily GC performance criteria must be met prior to the analysis of investigative samples. Specifically, the response ratio of C_{28} to C_{20} must be at least 0.85.

Note:

The working calibration curve must be verified on each working day, and after every 20 samples, by the injection of a mid-level calibration standard. If the relative percent difference (RPD) for any analyte varies from the predicted response by more than ± 25%, a new calibration curve must be prepared for that analyte.

12.9.3 The method blank must verify that there is no contamination above the PQL. Failing this, the batch may require re-extraction. According to MADEP CAM EPH method [reference 11.2.6.2] using GC/MS, the laboratory has identified a single component contaminant that appears at the same retention time in all laboratory and field samples. This contaminant is derived from the method's SPE cartridge and is not included in the calculation of the C11-C22 Aromatics range.

12.9.4 LCS, surrogates and matrix spike recoveries must % recovery criteria. Failure to meet these recovery limits may necessitate some type of corrective action such as reanalysis or re-extraction.

12.9.4.1 LCS Fractionation Criteria

Target Compounds Naphthalene and 2-Methylnaphthalene must be monitored in the LCS for breakthrough into the Aliphatic fraction. **If either the concentration of Napthalene or 2-Methylnaphthalene**

in the Aliphatic fraction exceeds 5% of the total concentration (aromatic and aliphatic sum) for Naphthalene or 2-Methylnaphthalene in the LCS or LCSD, fractionation must be repeated on all samples associated with that QC set.

Example Naphthalene % Breakthrough Calculation

Naphthalene in Aromatic fraction (a): 48ug/L
Naphthalene in Aliphatic fraction (b): 1.5ug/L
Total Naphthalene Concentration (c) = (a +b): 49.5ug/L

% Breakthrough = b / c x 100

% Breakthrough = 1.5/49.5 x 100

% Naphthalene breakthrough = 3.0

This calculation is applied to 2-Methylnaphthalene also.

12.9.5 For MCP jobs, the analyst is expected to complete Attachment 1 and 2, which details specific method and method reporting requirements.

13.0 CALIBRATION

13.1 In order to demonstrate the absence of mass discrimination in the GC system, the response ratio of the hydrocarbon peaks C_{28} to C_{20} must be at least 0.85. This requirement is evaluated with any calibration standard and must be met prior to the analysis of investigative samples.

13.2 Inject each calibration standard using the GC parameters defined in section 13. The HP Chemstation software tabulates peak area against the mass injected using external standard approach. The response factor is equal to the area divided by the concentration. The % RSD or the standard deviation divided by the average must be $\leq$25% or alternatively, the response factors may be plotted using a linear regression model provided the correlation coefficient is $\geq$0.99.

13.3 A collective calibration curve or factor (CF) must also be established for each hydrocarbon range of interest. A CF is calculated for the C_9-C_{18} Aliphatic Hydrocarbons, C_{19}-C_{36} Aliphatic Hydrocarbons, and C_{10}-C_{22} Aromatic Hydrocarbons. This is accomplished by summing peak area of the target compounds within each range. The response factors for the various regions are determined by dividing total peak area by total mass of all target peaks within each range. Note that the response factors for the surrogates 2-BN, 2-FBP, COD and OTP must not be included when calculating a CF in the range in which they elute (e.g., COD is not included in the C_{19} - C_{36} Aliphatic Hydrocarbon range).

13.4 At a minimum, the working calibration curve or calibration factor must be verified on each working day, and after every 10 samples, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the relative percent difference (RPD) for any analyte varies from the predicted response by more than ± 25%, a new calibration curve must be prepared for that analyte.

13.5 The recommended sequence of analysis is as follows:

(1) Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration)
(2) Laboratory Method Blank
(3) Samples
(4) QC Samples

(5) Mid-range Calibration Check Standard (after every 10 samples)

13.6 To monitor fractionation bleed through of Naphthalene and 2-Methylnaphthalene, these 2 compounds must be calibrated in the Aliphatic calibration curve.

14.0 SAMPLE PREPARATION AND INSTRUMENT PROCEDURES

14.1 Sample Preparation

14.1.1 Water samples are prepared in accordance with the SOP method reference 3510.

14.1.2 Soil samples are prepared in accordance with the SOP method reference 3540 and 3546. Method 3550 **must** only be used for highly contaminated soils. **Although method 3546 (microwave extraction) is approved for MADEP work, at this time it is not approved for SW846 methods.**

14.2 The GC/FID conditions guidelines used for TPH determination are defined below. Actual parameters are stored in a method identified with the calibration date. These parameters can vary based of instrument performance.

Instrument:	GC/FID #F; Hewlett Packard 5890 GC (Dual ALS)	
Columns:	30 meter x 0.25 mm, 0.25 um; Rtx-5	
Injectors	Hewlett Packard 7673 ALS	
Purge:	Init=Off; On at 1.00 min.	
Column flow:	1.5 mL/min (He); Constant Flow	
Injector temperature:	290° C	
Detector temperature:	330° C	
Temperature program:	Initial temperature	50° C
	Hold time	1 min
	Ramp	30 C/min
	Final temperature	130°C
	Hold time	1 minutes
	Ramp	15 C/min
	Final temperature	300° C
	Ramp	15 C/min
	Final temperature	315° C
	Run time	32.0 minutes

14.3 GC maintenance may be required to meet method performance criteria. This step reduces wear on the GC system and reduces the number of recalibrations required. The following steps can be taken if necessary to restore the performance of the GC system:

- Septum replacement
- Liner replacement
- Removal of several inches of the front end of the column
- Replace ALS needle, rinse vials
- Clean FID

14.4 Solvent injections may be required in GC batches to ensure that each sample can be assessed without the influence of carryover. The GC column can become contaminated with slow eluting petroleum compounds that emerge in subsequent injections, causing baseline elevation or distortion.

14.5 The GC/FID Runlog is used to document all analyses including sample extracts, calibration and QC samples. GC data reduction activities are documented on the quantitation report for the aromatic fraction analysis.

14.6 Data Reduction

14.6.1 Retention Time Windows for single component PAH target compounds are determined by the HP Chemstation software based on the retention times observed in the 50ppm calibration standard on days that a calibration is performed or in the Continuing Calibration standard if applicable. A peak must fall within ±0.2 min of the time observed in order for the reference peak to be identified as a PAH.

14.6.2 Reference times for the CN ranges are determined from the retention times observed in the 50ppm calibration standard on days that a calibration is performed or in the Continuing Calibration standard if applicable.

14.6.3 The C_9 - C_{18} Aliphatic Hydrocarbon range begins 0.1 minute before nonane and ends immediately (0.01 min) before the elution of the C_{19} peak. The C_{19} - C_{36} Aliphatic Hydrocarbon range begins 0.01 before the elution of the C_{19} peak, and ends 0.1 minute after C_{36}

EPH marker compounds and windows are summarized in the following Table:

Range/ Hydrocarbon Standard	Beginning Marker Compound	Ending Marker Compound
C_9-C_{18} Aliphatic Hydrocarbons	Nonane	Just Before Nonadecane
C_{19}-C_{36} Aliphatic Hydrocarbons	Nonadecane	Hexatriacontane
C_{10}-C_{22} Aromatic Hydrocarbons	Naphthalene	Benzo (g,h,i) Perylene

14.7 Data Reporting

Routine Reporting Requirements for the EPH Method Required Analytical Deliverable

GC Performance	NO
Retention Time	NO
Initial Calibration	NO
Calibration Check Standard	NO
Laboratory Method Blank	YES
Laboratory Control Sample (LCS)	YES
LCS Duplicate	YES
Initial Calibration Verification (ICV)	NO
Matrix Spike (MS)	YES (if requested by data user)
Matrix Spike Duplicate (MSD)	YES (if requested by data user)
Matrix Duplicate	YES (if requested by data user)
Extraction Surrogates	YES
Fractionation Surrogates	YES
Fractionation Check Standard	YES
Aromatic Breakthrough Evaluation	YES
System Solvent Blank (for baseline correction only)	YES
GC/MS QC	YES (GC/MS only)

14.8 Analyte List for the EPH Method

Range/Target Analyte	CASN	MCP CLEANUP STANDARDS GW-1 (GW-2) µg/L (ppb)	S-1/GW-1 (S1/GW3) µg/g (ppm)
Extractable Petroleum Hydrocarbon Ranges:			
C9-C18 Aliphatic Hydrocarbons	NA	(1000)	1000
C19-C36 Aliphatic Hydrocarbons	NA	5000	2500
C11-C22 Aromatic Hydrocarbons	NA	200	200
Diesel PAH Analytes:			
Naphthalene	91-20-3	20	4
2-Methylnaphthalene	91-57-6	10	4
Phenanthrene	85-01-8	50 1	(100)
Acenaphthylene	208-96-8	300	100
Other Target PAH Analytes:			
Fluorene	86-73-7	300	400
Acenaphthene	83-3-29	20	20
Anthracene	120-12-7	2000	1000
Fluoranthene	206-44-0	200 1	1000
Pyrene	129-00-0	200	700
Benzo(a)Anthracene	56-55-3	12	0.7
Chrysene	218-01-9	22	7
Benzo(b)Fluoranthene	205-99-2	12	0.7
Benzo(k)Fluoranthene	207-08-9	12	7
Benzo(a)Pyrene	50-32-8	0.22	0.7
Indeno(123 cd)Pyrene	193-39-5	0.52	0.7
Dibenzo(ah)Anthracene	53-70-3	0.52	0.7
Benzo(ghi)Perylene	191-24-2	300	1000

1. Refers to MCP Method 1 . GW-3 Standard
2. Standard Reporting Limits for this compound may not be able to achieve regulatory compliance limit for water when using the EPH Method for quantification. A GC/MS . SIM, HPLC or some other more sensitive analytical procedure may be required to comply with regulatory requirements for these PAH compounds.

15.0 CALCULATIONS

15.1 Reportable PAH target compounds amounts are calculated after determining the concentration in the extracts:

$$C = \frac{AREA}{avg\ RF}$$

where,

C= GC concentration of extract (µg/mL)
Area= area of target compound in sample chromatogram
RF= Average Response Factor (from initial calibration)

Using this value of the extract concentration, the sample amounts are calculated according to:

$$PAH = \frac{C_{ext}\, x\, V_{ext}}{V_{sam}}\, xDF$$

Water (µg/L):

where,

PAH= Sample concentration of PAH target compound in water (mg/L)
C_{ext}=GC concentration of extract (µg/mL).
V_{ext}=Extract volume (mL; typically 2)
V_{sam}=Sample volume (L; typically 1)
DF= Extract dilution factor

Soil (mg/kg dry):

$$PAH = \frac{C_{ext}\, x\, V_{ext}}{DW_{sam}}\, xDF$$

where,

PAH= Sample concentration of PAH target compound in soil (mg/kg dry)
C_{ext}=GC concentration of extract (µg/mL).
V_{ext}=Extract volume (mL; typically 2)
DW_{sam}=Sample dry weight (kg; typically 0.030)
DF= Extract dilution factor

15.2 Reportable CN range concentrations are determined similarly to the PAH calculation above. The GC concentration in the calculation is determined by multiplying the CN area (corrected for surrogate and target compound concentrations) by the range response factor.
A single peak in both aromatic and aliphatic fractions chromatograms and has been identified as being introduced by the fractionation cartridge. This peak is also excluded from the area summation in this calculation.

15.3 Undiluted PQLs are determined by inserting the GC quantitation limits (15 µg/mL for PAHs and 50 µg/mL for CN) and the standard sample volume for water (1 L) and dry weight for soil (0.030 kg dry) into the equations above. For undiluted samples the target PQL's for PAHs are 5 µg/L and 0.333 mg/kg(dry) for water and soil samples, respectively. For undiluted samples the target PQL's for the CN ranges are 50 µg/L and 1.6 mg/kg(dry) for water and soil samples, respectively.

15.4 Dilution factors are determined by multiplying the extract dilution and sample preparation dilution factors.

15.5 REPORTING OF RESULTS

15.5.1 The RLs for hydrocarbon ranges shall be based upon the concentration of the lowest calibration standard for an individual analyte within the range of interest. The range RL will be set at 100x the concentration of the lowest calibration standard for the associated analyte. Calculated collective concentrations for EPH aliphatic and aromatic hydrocarbon ranges below the RL should be reported as < Range RL (i.e. <100 µg/L).

15.5.2 All samples are reported in µg/L for waters or mg/kg (dry) for soils. Samples that contain no targets compounds in concentration above the quantitation limits are reported as BQL.

15.5.3 The practical quantitation limits for undiluted samples are as follows:

Water samples: 5 µg/L for PAH target compounds; 50 µg/L for CN ranges.

Soil samples: 0.33 mg/kg (dry) for PAH target compounds; 3.3 mg/kg (dry) for CN ranges.

15.5.4 All results are reported to two significant figures. Calculations are performed with at least four significant digits until the final answer to avoid rounding errors.

15.5.5 The quantitation limit is elevated in those samples requiring dilution. The normal quantitation limit is multiplied by the dilution factor to determine the sample specific reporting limit. Soil quantitation limits are adjusted for %moisture.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.2.1 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall address the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative

20.2 Any unauthorized deviation from this procedure must be documented as a non-conformance with cause and corrective action described in a QC memo.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to STL Westfield's facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCES

22.1 "Method For The Determination Of Extractable Petroleum Hydrocarbons (EPH)", MA Department of Environmental Protection, WSC-CAM Revision 3, September 10,2004.

22.2 "Method For The Determination Of Extractable Petroleum Hydrocarbons (EPH)", MA Department of Environmental Protection, Revision 1.1, May ,2004

22.3 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, July 1992 Revision, USEPA, Method 3510.

22.4 Draft "Method for Determination of Diesel Range Organics", EPA UST Workgroup, November, 1990

22.4 Microwave Accelerated Reaction System (MARS) operating manual, CEM Corp.

22.5 "Method for Determining Diesel Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-141, 1992.

APPENDIX I

A1.0 Determination of Target PAH and Hydrocarbon Ranges by GCMS. Modified SW-846 method 8270C.

Target PAH compounds and Aromatic and Aliphatic hydrocarbon ranges may be quantitated **after fractionation** using GC/MS. Refer to Section 9.10 of MADEP EPH Method, Revision 1.1, May 2004.

A1.1 DFTPP must be the GCMS tuning standard. A DFTPP standard must be injected every 12 hours. Refer to method 8270 SOP for tuning criteria. Evaluation of DDT breakdown and peak tailing of Pentachlorophenol and Benzidine is not required.

A1.2 The recommended internal standards is 5-alpha-androstane (primary quantitation ion is 245), other internal standards may be used as appropriate (see method 8270 for additional Internal standards). Samples requiring GW1 detection limits require analysis of target PAH using GCMS 5973. Samples are initially analyzed by standard EPH protocol using GC-FID. A portion of the extract is analyzed by GCMS to determine low-level concentrations of target PAH.

A1.3 All Target PAH compounds must meet initial and continuing calibration requirements as stated in method 8270C (CAM IIB).

A1.4 Range calibrations must be based on the individual aliphatic and aromatic calibration standards that are included in the specified ranges as defined by the EPH marker compounds (section 13.6.3). Range calibration factors are determined by dividing the summation of the peak areas (total ion current) by the total concentration injected.

A1.5 The evaluation of a CCV is required at the beginning and end of each analytical sequence (12 hour tune window).

A1.6 The samples must be extracted as described in section 13.0.

A1.7 The fractionation procedure is described in section 12.8.

A1.8 The performance standards for the EPH Aliphatic and Aromatic Ranges and comparable performance standards for target PAH analytes are stated below:

QC ELEMENT	PERFORMANCE STANDARD	
	Target PAH	EPH Range
Data		
Initial Calibration %RSD	≤15	≤ 25
Opening CCV (%Drift)	≤20	≤25
Closing CCV (%Drift)	≤20	≤25
Method Blanks	<RL	<RL
Internal Standards	50-200% of Opening Standard	50-200% of Opening Standard
Surrogate Recovery	40-140%	40-140%
Fractionation Surrogate Recovery	Not required	40-140%
LCS Recovery *(C9 30-130%)*	40-140%	40-140%
LCS Duplicate (RPD)	<20% for water <30% for soil	<25%
MS/MSD *(client specific)*	40-140% RPD <50%	40-140% RPD <50%
LCS/LCSD Naphthalene or 2-Methylnaphthalene Breakthrough	≤5% of either in aliphatic fraction	≤5% of either in aliphatic fraction

Note: Naphthalenene and 2-Methylnaphthalene must be measured in EPH aliphatic fraction of each sample for GC/MS analysis. Sample must be re-fractioned if concentration of either compound >5% in aliphatic fraction.

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MS/MSD *(client specific)*	40-140% RPD <50%	40-140% RPD <50%
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Note: Naphthalenene and 2-Methylnaphthalene must be measured in EPH aliphatic fraction of each sample for GC/MS analysis. Sample must be re-fractioned if concentration of either compound >5% in aliphatic fraction.

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STANDARD OPERATING PROCEDURE

Analytical Procedures for the Analysis of Volatile Petroleum Hydrocarbons (VPH)

Method MADEP VPH

SOP ID: GCS00500.MA

Date Written: 06/05/98
Date Revised: 12/7/06
Date Effective: 12/8/06

Rev 3

Approved Signatures

QUALITY ASSURANCE COORDINATOR

LABORATORY DIRECTOR

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1.0 TITLE

Analytical Procedures for the Analysis of Volatile Petroleum Hydrocarbons (VPH)

2.0 MATRIX

Organic Instrument Laboratory

3.0 REPORTING LIMIT

3.1 Waters: Target RLs are 5ug/L and Carbon Ranges are 100ug/L.

3.2 Soils: Target RLs are 0.250mg/Kg, and Carbon Ranges are 5.0mg/Kg.

4.0 SCOPE AND APPLICATION

4.1 This SOP is designed to measure the collective concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in water and soil. Volatile aliphatic hydrocarbons are collectively quantitated within two ranges: C_5 through C_8, and C_9 through C_{12}. Volatile aromatic hydrocarbons are collectively quantitated within the C_0 through C_{10} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 26° C and 220°C.

4.2 This method is also designed to measure the individual concentrations of targeted volatile hydrocarbons in water and soil. A listing of these targeted analytes is provided in Table 1.

4.3 Petroleum products suitable for evaluation by this method gasoline. Other heavier petroleum products may contribute to the VPH fraction. However, VPH is not recommended as the primary analytical tool for petroleum products other than gasoline, which contain a significant percentage of hydrocarbons heavier than C_{12}.

4.4 This SOP is based on a purge and trap, gas chromatography (CG) procedure. This procedure should be used by, or under the supervision of, analysts experienced in volatile organics analysis that are skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

Table 1. Aromatic Hydrocarbon Standard/Targeted PAH Analytes

Number	Compound	CAS Number
1	Benzene	71-43-2
2	Toluene	108-88-3
3	Ethylbenzene	100-41-4
4	Methyl-t-butyl ether	1634-04-4
5	Naphthalene	91-20-3
6	Xylenes (total)	106-42-3

5.0 SUMMARY OF METHOD

5.1 A sample is analyzed using purge and trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection is achieved by a photoionization detector (PID) and flame ionization detector (FID) in series, Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of aromatic and aliphatic hydrocarbons. The PID chromatogram is used to determine the individual concentrations or targeted analytes (BTEX/MTBE/naphthalene) and collective concentrations of aromatic hydrocarbons within the C_9 through C_{10} range. The FID chromatogram is used to determine the collective concentration of aliphatic hydrocarbons within the C_5 through C_8 and C_9 through C_{12} ranges.

6.0 DEFINITIONS

6.1 *Aliphatic Hydrocarbon*: Any organic compound comprised solely of carbon and hydrogen characterized by a straight, branched or cyclic chain of carbon atoms. By definition, this class of organic compounds includes alkanes, alkenes, alkynes, cycloalkanes or cycloalkenes for the VPH methodology.

6.2 *Aromatic Hydrocarbon*: Any cyclic and conjugated organic compound comprised solely of carbon and hydrogen. Aromatic compounds of environmental significance are benzoids that contain benzene or fused benzene rings.

6.3 *Volatile Petroleum Hydrocarbon*: Any hydrocarbon that elutes within the C5 through C8, and C9 through C12 aliphatic ranges or the C9 through C10 aromatic ranges defined by the method. The definition of Volatile Petroleum Hydrocarbon specifically **excludes** all substituted aliphatic or aromatic hydrocarbon derivatives (non-hydrocarbons as defined by the VPH Method), the individual VPH Method Target Analytes and/or surrogates that co-elute within these method specific ranges. The VPH Method is suitable for the separation and quantification of the aliphatic and non-target aromatic components of gasoline, mineral spirits, certain petroleum naphthas and components of kerosene, jet fuel, heating oils, lubricating oils, and/or other petroleum products contained within the aforementioned method-defined ranges.

6.4 *Accuracy*: the degree of agreement between an observed value and an accepted reference value.

6.5 *Batch*: environmental samples, which are prepared and/or analyzed together with the same process using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.6 *Holding Time:* the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

6.7 *Instrument Blank*: a blank matrix that is introduced onto the instrument for analysis

and can monitor calibration baseline drift and carryover contamination effects.

6.8 *Laboratory Control Sample (LCS)*: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

6.9 *Matrix Duplicate (MD)*: duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

6.10 *Matrix Spike (MS)*: field sample to which a known amount of target analyte(s) is added.

6.11 *Matrix Spike Duplicate (MSD)*: a replicate matrix spike.

6.12 *Method Blank*: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

6.13 *Method Detection Limit (MDL):* the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR, Part 136, Appendix B).

6.14 *Non-conformance*: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

6.15 *Precision*: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

6.16 *Reporting Limit (RL):* The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

6.17 *Quality Assurance (QA)*: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

6.18 *Quality Control (QC)*: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

6.19 *Raw Data*: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche

copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

6.20 *Sensitivity*: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

6.21 *Standard Operating Procedure (SOP)*: a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Like all GC procedures, this method is subject to a "false positive" bias in the reporting of targeted volatile compounds, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated.

7.2 Interferences in the GC analysis of petroleum hydrocarbons result from non-petroleum derived organics that are extracted along with the target compounds. No effort is made in this SOP to identify or account for this effect.

7.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it must be followed by the analysis of a method blank to check for cross-contamination.

7.4 Interfering Peaks in Specified Aliphatic Hydrocarbon Ranges Hydrocarbons (and non-hydrocarbons), even with elution times within the defined chromatographic windows for the aliphatic hydrocarbon ranges specified by the VPH Method, need not be included in the PAC for these ranges unless they meet the definitions of aliphatic
hydrocarbon and volatile petroleum hydrocarbon, as defined above. If the concentration of a hydrocarbon range is based on one (or just a few) peaks within the range and an indicative petroleum hydrocarbon peak pattern is not apparent, the laboratory should provide this information and alert the data user of the potential for a false positive result in the Environmental Laboratory case narrative. MCP sites with chlorinated hydrocarbons, ketones, and/or commingled non-petroleum hydrocarbons are subject to this interference.

7.5 Interfering Peaks in Specified Aromatic Hydrocarbon Range
The VPH Method should be used with caution at sites with an uncertain history, particularly closed or abandoned Manufactured Gas Plants (MGPs). Styrene, a common contaminant of concern (COC) at many MGP sites, cannot be satisfactorily resolved from o-xylene under the chromatographic conditions specified for the VPH Method. If encountered, co-eluting styrene could cause an overestimation of o-xylene and a subsequent underestimation of the C9-C10 aromatic range when the overestimated o-xylene peak is subtracted from the PAC for the range. Other contaminant pairs routinely encountered at MCP sites that are difficult to resolve under the chromatographic conditions specified for the VPH Method include 1,2-dichloroethane/benzene and 1,1,1,2-tetrachloroethane/ethylbenzene.

7.6 Evaluation of Interfering Compounds Not Associated with a Petroleum Product.
In general, it may be prudent for the client to confirm all PID/FID data by SW-846

Method 8260B (GC/MS) if critical MCP decision making (notification, compliance with cleanup standards, risk assessment, etc.) is based solely on the VPH Method (or any other non-specific GC analysis). If a positive interference is suspected from hydrocarbons and/or non-hydrocarbons not associated with VPH in either an aliphatic or aromatic fraction or with a Target Analyte, and such interference would adversely affect MCP decision making, if confirmed, then SW-846 Method 8260B, Volatile Organics by GC/MS (WSC-CAM-II A) should be employed to accurately identify and quantify the components that comprise the fraction or to resolve the analyte pairs.

8.0 SAFETY

8.1 Target compounds described in this method are known or suspected carcinogens. All manipulations of these chemicals should be performed under a hood.

8.2 Proper precautions should be taken when formulating new standards, or handling suspected high concentration samples. Samples for analysis should also be considered hazardous and should be handled using the same precautionary conditions as known hazards.

8.3 Procedures shall be carried out in a manner that protects the health and safety of all STL employees. Eye protection that satisfies ANSI Z87.1, a laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Appropriate gloves for the inorganic department and sample receiving shall be made of 100% powder-free, non latex and composed of Nitrile.

8.4 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

8.5 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

8.6 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL employee. The situation must be reported **immediately** to a laboratory supervisor.

9.0 APPARATUS AND MATERIALS

9.1 Hewlett Packard 5890 Series II (FID/PID).

9.2 105-m x 0.53-mm I.D., 3.0-µm film DB502 column (J&W) or equivalent.

9.3 Tekmar LSC Model 3000 with a Supelco custom trap 7cm carbopack B/7cm Carbopack C.

9.4 Hamilton 10 µL, 25µL, 500µL, 1000µL syringes, a 5mL Luerlock hypodermic and a 5mL gas-tight syringe shutoff valve.

9.5 19 x 150mm culture tube.

9.6 Top loading balance.

9.7 Ultrasonic bath.

9.8 HP Chemstation Data System

9.9 Tekmar Precept II – 48 position autosampler

9.10 Varian Archon – 51 position autosampler.
9.11 40mL VOA vial with Teflon lined cap

10.0 REAGENTS AND STANDARD PREPARATION

10.1 Reagents
10.1.1 Reagent water: organic free water (ASTM Type 1 reagent grade water).
10.1.2 Solvent: methanol; purge and trap grade or equivalent. Store away from other solvents.
10.2 Analytical Standards
10.2.1 Stock solutions of target petroleum blends (aromatic and aliphatic) are purchased commercially and diluted to working standards. Ultra Scientific or equivalent vendor produces the commercial blend of target compounds. See list on next page for the Primary Calibration standard.

VPH Primary Calibration Stock Standard

Target Compound	Vendor	Conc. (ug/mL)
Pentane	Ultra Scientific	1000
2-Methylpentane	--	1000
Methyl-t-butyl ether	--	1000
2,2,4-Trimethylpentane	--	1000
Benzene	--	1000
Toluene	--	1000
Nonane	--	1000
n-Decane	--	1000
Ethylbenzene	--	1000
m & p-Xylene	--	2000
o-Xylene	--	1000
1,2,4-Trimethylbenzene	--	1000
n-Butylcyclohexane	--	1000
Naphthalene	--	1000

VPH Intermediate Primary Calibration Standard

Ultra Scientific Stock Standard (ug/mL)	Volume of Stock Standard (uL)	Final Volume In Methanol (mL)	Final Concentration (ug/mL)
1000	1000	10	100

Calibration levels are formulated using the intermediate solution describe above. Standard are prepared in a 50 mL volumetric flask using reagent grade water just prior to loading on the ALS. **

Level	Intermediate Standard Volume (uL)	Concentration of Level (ug/L)
1	0.5	1.0
2	1.25	2.5
3	2.5	5
4	12.5	25
5	50	100
6	100	200
7	200	400

** Levels can be made up in alternate volumes (i.e. 100mL).

10.2.2 The Matrix Spike standard is prepared from a second source vendor – Absolute Standards The formulation directions are identical to those used for the Primary calibration standard.

10.2.3 Surrogate Standard: The surrogate standard used in this method is 2.5-dibromotoluene. It is introduced into every sample and standard prior to purge and trap analysis to monitor the efficiency of the purge and trap and any sample matrix interference.

The stock solution of the surrogate standard is prepared according to the following instructions:

VPH Surrogate Stock Solution

Target Compound	Vendor	Catalog Number	Amount (g)	Final MeOH Volume (mL)	Stoch Conc. (ug/mL)
2,5-Dibromotoluene	Aldrich	25,098-8	1.0	25	40000

The VPH surrogate is made up into a working concentration to be used in automated samplers such as the Precept II and Varian Archon. The formulation is outline in the table below:

Surrogate Stock Concentration (ug/mL)	Volume of Stock (uL)	Final Volume in Methanol (mL)	Surrogate Working Concentration (ug/mL)
40000	312.5	25	500

1.0µL of this 500 ug/mL solution is added automatically with the automated ALS sampler (Percept and/or Archon). This spike is added to all laboratory and investigative samples. The concentration is 100ug/L.

10.2.4 Continuing Calibration Standard: Generally 100 ug/L. (See Level 4 under calibration preparation).

10.2.5 All standards must be replaced in 6 months or sooner if QC criteria in this SOP cannot be met.

10.2.6 Standard preparation is documented in the department standard prep logbook. All lot numbers and dilution procedures must be entered.

11.0 SAMPLE CONTAINERS, COLLECTION, AND PRESERVATION

11.1 Aqueous samples are collected in 40 mL VOA vials and preserved to pH<2 with 1:1 HCL.

11.2 Soil and sediment samples are collected in 40 mL pre-weighed VOA vials that contain 15 mL of methanol. These weights are recorded in a preparation logbook. The ratio of methanol to soil is 1 mL methanol to soil ± 25%. In all cases, the soil sample in the vial must be completely covered by methanol.

11.3 Additional samples without methanol must be obtained for the determination of soil moisture and dry weight correction factors.

11.4 Soil and sediment samples must be cooled to 4°C immediately after collection.

11.5 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

11.6 Aqueous samples are analyzed within 14 days, soil and sediment samples must be analyzed within 28 days of collection. Alternatively, soil and sediment samples collected in an Encore sampler (SW846-5035) can be frozen and stored for 1 year. Samples must be preserved in Methanol within 24 hours of thawing.

11.7 In the event that sample shipment is delayed in the field or in transport, Project

Management will assess the laboratory's ability to process the samples within the prescribed holding time and advise the client if holding times cannot be met.

11.8 If the sample is received at the laboratory already out of protocol, Project Management notifies the client to determine if the sample should be analyzed.

11.9 A reagent water trip blank should accompany each batch of water samples and a methanol trip blank for the soil samples.

12.0 QUALITY CONTROL

12.1 Method detection limits are determined in accordance with the Corporate SOP for Conducting MDL Studies. MDLs must be less than or equal to the reporting limit.

12.2 The method blank is analyzed to ensure there is no background level of contaminants above the PQL. If the method blank contains reportable VPH concentrations the batch may require re-analysis.

12.3 A matrix spike and matrix spike duplicate is optional (client specific) and may be extracted and analyzed every 20 samples, or per batch. The recovery and RPD limits are 70-130% and ±50%, respectively. If the recoveries do not meet the criteria, the LCS recovery must be consulted

12.4 A Laboratory Control Sample (LCS) and duplicate (LCSD) are analyzed every 20 samples, or per batch. The LCS/LCSD recovery must meet the acceptance criteria of 70-130%. If the LCS/LCSD fails to meet the criteria, a reanalysis is required. The RPD recovery limit is ≤20% for an aqueous LCS and ≤25% for a soils LCS.

12.5 Surrogate recoveries limits for the samples, blanks and spikes are 70-130%. If the surrogate fails, the sample may require reanalysis. Some chromatograms evidence matrix interference, which makes reliable SS recovery measurements impossible.

12.6 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

12.6.1 The pentane and MTBE peaks must be adequately resolved from the solvent front that may be present on the FID and PID chromatograms.

12.7 Collective peak area integration for the hydrocarbon ranges must be measured from the baseline and include the unresolved complex mixture areas. For the individual target analytes and surrogate compounds, a valley-to-valley approach should typically be used for integration purposes; this approach may be modified on a case-by-case basis by an experienced analyst.

12.8 Acceptance of Data

12.8.1 The working calibration curve must be verified on each working day, and after every 20 samples, by the injection of a mid-level calibration standard. If the relative percent difference (RPD) for any analyte varies from the predicted response by more than ± 25%, the continuing calibration sample must be reanalyzed or a new calibration curve must be prepared for that analyte. [Minimum of 5 standards must be used for the calibration curve. The low standard must be ≤ reporting limit (RL). %RSD should be ≤25 or "r" should be ≥0.99 for all compounds and ranges. Must contain all VPH range and target analytes. If regression analysis is used, the curve must not be forced through the origin. See Calibration section 13.0]

12.8.2 The method blank must verify that there is no contamination above the PQL. Failing this, the batch may be re-analyzed.

12.8.3 QC criteria defined in section 10.0 for calibration must be met. LCS/LCSD, surrogate and MS/MSD (optional) as described in section 12.0 must be met.

12.9 For MCP jobs, the analyst is expected to complete Attachment 1 and 2, which details specific method and method reporting requirements.

13.0 CALIBRATION

13.1 A minimum 5-point curve is analyzed using the standard prepared above. The calibration curve plots area vs. amount using average response factor. The average response factor must be <25%, alternatively, linear regression (not forced through origin) may be used, the correlation coefficient (r2) value must be >0.990. The equation of the line used for the calculations will be referred to as the LR (Linear Regression). RF's (or response factors) are also utilized for some quantitation operations.

13.2 Calibration standards are prepared by spiking 50.0 mL of volatile free water with the VPH calibration standard and surrogate as described above.

13.2.1 A collective calibration curve or factor (CF) must also be established for each hydrocarbon range of interest. A CF is calculated for the C_5-C_8 Aliphatic Hydrocarbons, C_9-C_{12} Aliphatic Hydrocarbons, and C_9-C_{10} Aromatic Hydrocarbons. The response factors for the various regions are determined by averaging the response factors in the appropriate ranges. Note that the response factors for the surrogates must not be included when calculating CF in the range in which they elute. The CF for the C_5-C_8 and C_9-C_{12} Aliphatic Hydrocarbon ranges is determined from the FID signal. The CF for the C_9-C_{10} Aromatic Hydrocarbons is determined from the PID signal. See the table below for VPH range marker compounds:

Hydrocarbon Range	Beginning Marker	Ending Marker
C5-C8 Aliphatic Hydrocarbons (FID)	0.1 min. before n-Pentane	0.1 min. before n-Nonane
C9-C12 Aliphatic Hydrocarbons (FID)	0.1 min. before n-Nonane	0.1 min. before Naphthalene
C9-C10 Aromatic Hydrocarbons (PID)	0.1 min. before o-Xylene	0.1 min. before Naphthalene

13.3 At a minimum, the working calibration curve or calibration factor must be verified on each working day, and after every 20 samples and or at the sequence closing, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the relative percent difference (RPD) or % Drift (%D) for any analyte varies from the predicted response by more than ± 25%, a new calibration curve must be prepared for that analyte.

13.4 The recommended sequence of analysis is as follows:

(1) Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration)
(2) Laboratory Method Blank
(3) QC Samples
(4) Samples
(5) Mid-range Calibration Check Standard (after every 20 samples)

14.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

14.1 **Water Samples (Precept II and Archon)**: Samples vials are placed directly into the autosampler tray and a sampler sequence of events is created. (See appropriate

Operating Manuals for instructions) These automated systems both add surrogate spike automatically during the sample transfer to the Purge and trap system.

14.2 Sample dilution is necessary when the concentration of the sample exceeds the calibration range.

Sample dilutions are prepared volumetrically. Partially fill a volumetric flask with reagent grade water. The portion of sample used for the dilution is measured in a gas-tight syringe and then gently add to the volumetric flask. The flask is then brought to volume with reagent grade water, cap, and invert three times. This sample dilution is then transferred to the appropriate vessel for analysis.

EXAMPLE: 5 times dilution (1:5 or 5x)
50mL volumetric flask is partially filled with reagent grade water
10.0 mL of sample is added to the flask.
The flask is then brought to final volume with reagent grade water.

14.3 **Soil Samples**: Soil and sediments are extracted with methanol. An aliquot of the extract is added to reagent water.

14.3.1 Weigh the sample vial to 0.01 g on a top loading balance and determine the weight of the soil/sediment sample; this determination requires knowledge of the empty/tared weight of the sample vial and volume /weight of the methanol preservative that was added to the sample vial.

14.3.2 The sample is shaken for approximately 2 minutes. Allow sediment to settle until a layer of methanol is apparent.
Purge 100 μL of the soil extract per 5.0 mL of volatile water
This procedure yields a 1:50 dilution for target compounds and 0.1 mg/L for the method surrogate.
Further dilution of sample may be accomplished by purging less of the extract.

14.3.3 Automated samplers such as the Precept II and Varian Archon require larger volumes of sample prepared.
Samples are prepared in a 40mL VOA vial. The ideal volume of methanol extract to be purged is 100uL per every 5mL of water.
The VOA vial(s) is partially filled with reagent grade water. 840uL of methanol extract is then added to the vial. The vial is then topped off with reagent grade water and capped.

14.4. The GC VPH run logbook is used to document all VPH analyses including calibration, investigative samples, and QC samples.

14.5 The GC FID/PID equipped with a purge and trap autosampler is used to analyze VPH compounds. The instrument and column and conditions are as follows:

Instrument:	GC FID/PID; Hewlett Packard 5890 GC	
Columns:	105 meter x 0.53 mm, 0.30 um; DB502	
Column flow:	10 mL/min	
Temperature program:	Initial temperature	40 °C
	Hold time	1 min
	Ramp rate	5 °C/min
	Second temperature	100 °C
	Hold time	0 min
	Ramp rate	12 °C/min

	Third temperature	160 °C
	Hold time	0 min
	Ramp rate	24 °C/min
	Final temperature	230 °C
	Hold time	7.08 min
	Total Run Time	28.0
Inlet	250 °C	
Detector A	230 °C	
Detector B	230 °C	

Tekmer Model 3000 and Autosampler settings:

Trap Standby Temp.	30 °C	
Purge gas		Helium
Purge time		9.0 min
Purge temp		Ambient
Desorb Temperature	250 C	
Desorb Time	4.0 min	
Bake Temperature		260 °C
Bake Time		5.0 min
Model 3000 Valve Temp		150 °C
Model 3000 Transfer Line Temp		150 °C

14.6 Data Reduction

14.6.1 The retention Time Windows for single component VPH target compounds are determined by the HP Chemstation software based on the retention times observed in the 100 ppb calibration standard on days that a calibration is performed or in the Continuing Calibration standard if applicable. A peak must fall within ±0.15 min of the time observed in order for the reference peak to be identified as a VPH.

Reference times for the hydrocarbon ranges are determined from the retention times observed in the 100 ppb calibration standard on days that a calibration is performed or in the Continuing Calibration standard if applicable.

The C_5-C_8 Aliphatic Hydrocarbon range begins 0.1 min before C_5 and ends immediately (0.01 min) before the elution of the C_9 peak. The C_9 - C_{12} Aliphatic Hydrocarbon range begins 0.01 before the elution of the C_9 peak, and 0.1 min before naphthalene therefore there is no overlap of the two ranges. The Aromatic Hydrocarbon range is defined as beginning 0.1 minutes after the RT of o-xylene compound and ending 0.1 minutes before the naphthalene.

14.7 Data Reporting

Routine Reporting Requirements for the VPH Method Required Analytical Deliverable
GC Performance - NO
Retention Time Windows - NO
Initial Calibration - NO
Calibration Check Standard - NO
Laboratory Method Blank - YES

Laboratory Control Sample (LCS) - YES
LCS Duplicate - YES
Field Matrix Spike (MS) - YES (if requested by data user)
Field Matrix Spike Duplicate (MSD) - YES (if requested by data user)
Field Matrix Duplicate (MD) - YES (if requested by data user)
Surrogates – YES

14.8 Analyte List for the VPH Method

Range/Target Analyte	CASN	MCP CLEANUP STANDARDS GW-1 (GW-2) µg/L (ppb)	S-1/GW-1 µg/g (ppm)
Volatile Petroleum Hydrocarbon Ranges:			
C5-C8 Aliphatic Hydrocarbons	NA(1)	400	100
C9-C12 Aliphatic Hydrocarbons	NA1	(1000)	1000
C9-C10 Aromatic Hydrocarbons	NA1	200	100
Target Analytes:			
Benzene	71432	5	10
Ethylbenzene	100414	700	80
Methyl-tert-butylether (MTBE)	1634044	70	0.3
Naphthalene	91203	20	4
Toluene	108883	1000	90
o-Xylene (3)	95476	(6,000)	500
m- Xylene (2,3)	108383	(6,000)	500
p- Xylene (2,3)	106423	(6,000)	500

1.NA = Not Applicable
2. May not be resolvable under chromatographic conditions required under this Method
3. May be reported and evaluated as mixed isomers

15.0 CALCULATIONS

15.1 The concentration of a specific analyte and hydrocarbon ranges in an aqueous sample may be calculated using equations 1 and 2, respectively.

Equation 1: Concentration (mg/L)= C_{ext}* DF

Equation 2: Concentration HC Range (mg/L)=Total Area * CF*DF

Where,
C_{ext}=GC concentration of sample (mg/L). Returned by HP chemstation using linear regression
DF= Dilution Factor
CF=Calibration Factor.

15.2 The concentration of a specific analyte and hydrocarbon ranges in a soil and sediment samples may be calculated using equations 3 and 4, respectively.

15.2.1 *Prior to calculations 3 and 4, the methanol extract sample must be corrected for sample moisture that may have biased the overall volume of methanol during the extraction. The increase in extraction solvent volume may be a direct result of the solubility of the entrained sample moisture (or water) in the methanol. The following formula is used to correct for water is the sample:*

mL Methanol + ((% moisture/100) x g of sample = mL solvent/water (Vt)

This result, (Vt), is used in equations 3 and 4.

Equation 3: Concentration (mg/L)= C_{ext}* F

Equation 4: Concentration HC Range (mg/L)=Total Area * CF*F

where,

C_{ext}=GC concentration of sample (mg/L). Returned by HP chemstation using linear regression
Rw=Volume of reagent water used in the purge and trap analysis
Vt=Volume of total extract
Vi=Volume of methanol extract added to reagent water for purge and trap analysis
As= weight of soil sample extracted (soil sample in vial)
Wd=% solids(dry wt/wet wt)
CF=Calibration Factor
F= (Rw*(Vt/Vi)/(As*Wd)

15.3 The C_5 - C_8 and C_9 - C_{12} ranges are calculated from the FID signal and the C_9-C_{10} ranges from the PID. Since the individual peaks MTBE, Benzene, Toluene, Ethylbenzene, P-Xylene and o-Xylene are reported from the PID, these amounts are subtracted from the C_5-C_8 and the C_9 - C_{12} amount so that they are not double counted. Hence C5-C8 excludes MTBE, Benzene and Toluene. C_9 - C_{12} excludes Ethylbenzene, p-Xylene and o-Xylene and the collective C_9-C_{10} aromatics.

15.4 Reporting of Results

15.4.1 The reporting limit (RL) for Target VPH analytes shall be based upon the concentration of the lowest calibration standard for the analyte of interest. The RL must be greater than or equal to the concentration of the lowest calibration standard. The RLs for hydrocarbon ranges shall be based upon the concentration of the lowest calibration standard for an individual analyte within the range of interest. The range RL will be set at 100x the concentration of the lowest calibration standard for the associated analyte. Calculated collective concentrations for VPH aliphatic and aromatic hydrocarbon ranges below the RL should be reported as < Range RL (i.e. <100 µg/L).

15.4.2 All samples are reported in ug/L for waters or mg/kg(dry) for soils. Samples that contain no targets compounds in concentration above the quantitation limits are reported as BQL.

15.4.3 The practical quantitation limits for undiluted samples are as follows:

Water samples: 5.0 ug/L for VPH target compounds excluding naphthalene (10 ug/L); 100 ug/L for hydrocarbon ranges.

Soil samples: 0.25 mg/kg(dry) for VPH target compounds excluding naphthalene (0.50 mg/kg(dry); 5.0 mg/kg(dry) for hydrocarbon ranges.

15.4.4 All results are reported to two significant figures. Calculations are performed with at least four significant digits until the final answer to avoid rounding errors.

15.4.5 The quantitation limit is elevated in those samples requiring dilution. The normal quantitation limit is multiplied by the dilution factor to determine the sample specific reporting limit. Soil quantitation limits are adjusted for %moisture.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimise pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1, 2, 3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.2.1 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall address the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative

20.2 Any unauthorized deviation from this procedure must be documented as a non-conformance with cause and corrective action described in a QC memo.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to STL Westfield's facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCES

22.1 "Method for The Determination of Volatile Petroleum Hydrocarbons (VPH)", MA Department of Environmental Protection, Revision 3, September 10th, 2004. MADEP WSC-CAM.

22.2 "Method for The Determination of Volatile Petroleum Hydrocarbons (VPH)", MA Department of Environmental Protection, Revision 1.1, May, 2004.

22.3 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, July 1992 Revision, USEPA, Method 8015.

22.4 Tekmar Precept II user manual.

22.5 Varian Archon 3552 user manual.

22.6 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, July 1992 Revision, USEPA, Method 5030.

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Determination of Azodicarbonamide in Water

Approvals:

Prepared by: _____ Date: 7/8/08
Senior Chemist

Approved by: _____ Date: 7/16
Pesticide Residue Analysis Management

Approved by: _____ Date: 7/17/08
Quality Assurance

Revision Log:

Revision: 04		Effective Date: This version
Section	**Justification**	**Changes**
Approvals	Formatting requirements per LOM-SOP-LAB-201	Moved signatures to beginning of document
Revision Log	Formatting requirements per LOM-SOP-LAB-201	Moved to beginning of document and reformatted to include Justification
Reference	Formatting requirements per LOM-SOP-LAB-201	Added reference – *Chemical Hygiene Plan*
Document	Formatting requirements per LOM-SOP-LAB-201	Placed sections in order
Safety Precautions and Waster Handling	Formatting requirements per LOM-SOP-LAB-201	Added generic sentence referencing *Chemical Hygiene Plan*

Ver. #	Effective Date	Change
03	06/08/04	Major changes are as follows: • Updated to Level 3 format • Added Interferences and Statistical Information/Method Performance sections
02	05/07/02	Major changes are as follows: • Cross Reference section added • Second column confirmation added • Deleted MDL reference • Updated the following sections: Personnel Training and Qualifications, Standards, and Sample Preparation
01	12/03/99	New

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Reference:

1. Standard Operating Procedure QA-XXXX, Pace, Inc. New England, 8/95.
2. SW-846 Method 8000B, Revision II, December 1996.
3. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-025	Monitoring of QC Data Acceptance Limits

Purpose:

The purpose of this procedure is to outline HPLC analysis procedures for azodicarbonamide (Kempore) in water and wastewater.

Scope:

The method allows for HPLC analysis using a direct aqueous injection of Kempore (CAS No. 123-77-3).

	LOQ µg/L
Kempore (azodicarbonamide)	1000

Basic Principles:

Water samples are analyzed using a solid phase cleanup procedure followed by filtration and HPLC analysis. Separation is accomplished using a C18 column and phosphate buffer mobile phase. A UV detector at 230 nm is used for quantitation.

Interferences:

Method interferences may be caused by impurities in solvents, reagents, glassware or other hardware used in sample processing. The UV detector is a general detector and many organics will respond. All glassware is rinsed with solvent before used and a method blank is performed with each batch of samples to demonstrate that the system is free of contaminants.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Avoid inhaling solvents and getting them on the skin. When handling standard materials, use gloves and protective clothing, including protective eyewear. Avoid breathing standards or getting them on the skin.

All solvent waste is collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal in to the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed.

Personnel Training and Qualifications:

Each analyst performing the instrumental analysis will work with an experienced analyst for a period of time until s/he can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret the chromatograms, and enter data into the LIMS. S/he will also follow the Department Training Manual for Analysts. Proficiency is measured through documented audits of the tasks listed and overchecking of data. An annual quad study for any of the Direct Aqueous Injection HPLC tests will suffice for proficiency.

Sample Handling:

Samples are collected in 100-mL amber glass containers with a Teflon™-lined cap. Store at 4° ± 2°C until the analysis. All samples must be analyzed within 7 days of collection.

1. Prepare a disposable filter assembly with a Dionex Onguard-H cartridge, followed by a 0.45-µm nylon filter. Add 5 mL of sample to the syringe. Discard the first 3 mL of sample, and collect the remaining 2 mL into a labeled HPLC vial.

2. Prepare a laboratory control sample by adding 1.0 mL of stock standard to a 10 mL volumetric containing HPLC grade water. Bring to volume, filter and analyze.

3. Prepare a method blank by filtering 5 mL of HPLC grade water through the filter train.

4. Prepare a Matrix spike/matrix spike duplicate by spiking exactly 10 mL of background sample with 1.0 mL of stock standard. Record the final volume as 11 mL for the MS/MSD. Filter through the sampling train as above, and analyze.

5. Note that samples and standards must be injected within 24 hours of prep. Degradation of Kempore in the standards and samples has been observed in past analyses.

Apparatus and Equipment:

1. HP-1100 Liquid Chromatograph, or equivalent, equipped with binary or quaternary pump system with UV detector

2. Analytical Balance, accurate to 0.1 mg

Reagents and Standards:

A. Reagents

1. HPLC grade water

2. Potassium phosphate monobasic, KH_2PO_4, ACS grade

3. Potassium phosphate dibasic, K_2HPO_4, ACS grade

4. azodicarbonamide – 97% purity, Aldrich

5. Nylon filters, 0.45-µm

6. Syringe filter assemblies, 5-mL disposable

7. Various sizes of Class A volumetric pipettes, flasks, and syringes

8. Syringes, 10-µL, 250-µL, 1-mL

9. HPLC vials, 2-mL with Teflon™-lined caps

10. Dionex Onguard-H sample pre-treatment cartridges, Part No. 039596

B. *Standards*

1. Stock standards are prepared by weighing on an analytical balance approximately 20 mg of neat Kempore into 200 mL of HPLC grade water. Sonicate for 1 hour to bring into solution. The mass is recorded to the nearest 0.0001 grams. The stock standard is good for one month. Ongoing stability studies have not been performed to determine the effects of long term storage of the standard.

2. Prepare calibration levels fresh with each new calibration. Using a syringe, add the aliquot noted below to a volumetric flask partially filled with HPLC water. Bring to volume after addition of the stock.

Standard Level	µL added	Stock	FV	Approx. µg/L	Solvent
Level 1	100	Kempore Stock	10	1000	HPLC Water
Level 2	250	Kempore Stock	10	2500	HPLC Water
Level 3	1000	Kempore Stock	10	10000	HPLC Water
Level 4	2500	Kempore Stock	10	25000	HPLC Water
Level 5	5000	Kempore Stock	10	50000	HPLC Water
MDL	20	Kempore Stock	10	200	HPLC Water

Standards should be stored at 4° ± 2°C.

Standards Database Entry:

Level 1: KEMP1_

Level 2: KEMP2_

Level 3: KEMP3_

Level 4: KEMP4_

Level 5: KEMP5_

Chromatographic Procedure:

Prepare phosphate buffer by weighing exactly 5.444 grams of KH_2PO_4 and 9.124 grams of K_2HPO_4 into 4 L of HPLC grade water. Filter through a 0.45-µm nylon filter prior to use. Mobile Phase expires in 6 months. Filter and degas at least monthly prior to use. Check pH prior to use. The allowable pH range is 6.5 to 7.5. When Kempore is defected on the primary column, second column confirmation is required. This can be accomplished as a second analysis, or simultaneously, using dual columns and two detectors. Double the injection volume is needed when dual column analysis is performed.

The following chromatographic conditions are optimum, but can be changed to improve linearity, sensitivity, and chromatography on each system.

Column: Supelco PAH, 25 cm × 4.6 mm × 5 µm

Mobile Phase: 100% Phosphate Buffer, 1.0 mL/min

Injection Size: 20 µL

Detection: UV at 230 nm

Column Temperature: 35°C

Second Column Configuration Options:

1. CapcellCN, 25 cm × 4.6 mm × 5 µm
   Mobile phase same as above

2. Thermo hypersil phenyl, 25 cm × 4.6 mm × 5 µm
   Mobile phase same as above

Calibration:

An external standard calibration is performed on the HPLC using 5 levels of standards ranging from 1000 to 50,000 µg/L. If the response factors over the calibration range have a relative standard deviation (RSD) ≤20%, the average RF may be used to calculate results. If the RSD of the response factors exceeds 20%, then a linear calibration curve must be used. If the coefficient of determination is less than 0.99 using linear, a quadratic calibration may be used. If a quadratic fit is used, a sixth point must be added to the curve. The MDL standard may be used for this purpose.

Continuing Calibration

A continuing calibration standard must be injected before and after every 10 samples. The mid-level standard is used and the calculated response must be within ±15% difference from the expected value or the system must be recalibrated. All samples injected since the last continuing calibration verification standard which met specifications must be reanalyzed after a new calibration.

Calculations:

$$\frac{Pk\ H \times FV \times DF}{RF \times IV} = Concentration\ \mu g/L$$

Where:

RF = Average response factor (Peak Height/µg/L)

FV = Final volume in mL

DF = Dilution factor

IV = Initial volume of sample extract (mL)

Statistical Information/Method Performance:

Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These are run on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Copies of the annual studies are maintained by the department supervisor. Updated to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Quality Assurance/Quality Control:

1. A reagent blank (using deionized water) is prepared with every batch of 20 samples or less to verify the absence of glassware or system contamination.

2. A laboratory control sample (spike reagent blank) is prepared with every batch of 20 samples or less to ensure acceptable extraction performance.

3. A matrix spike and matrix spike duplicate (using client's samples) are extracted every 20 samples or less.

4. See SOP-PP-002 on QC acceptance limits and corrective action. See SOP-PP-025 on monitoring QC limits.

Lancaster Laboratories
2425 New Holland Pike • Lancaster, PA 17601

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Determination of Dinitrosopentamethylenetetramine in Water

Approvals:

Prepared by: _____ Date: 7/8/08
Senior Chemist

Approved by: _____ Date: 7/10/08
Pesticide Residue Analysis Management

Approved by: _____ Date: 7/22/08
Quality Assurance

Revision Log:

Revision: 04		Effective Date:	This version
Section	**Justification**		**Changes**
Approvals	Formatting requirements per LOM-SOP-LAB-201		Moved signatures to beginning of document
Revision Log	Formatting requirements per LOM-SOP-LAB-201		Moved to beginning of document and reformatted to include Justification
Revision Log	*Incorrect numbering of versions*		Renumbered versions as version 01, 02, 03 (previously 01, 01, and 01)
Reference	Formatting requirements per LOM-SOP-LAB-201		Added reference – *Chemical Hygiene Plan*
Document	Formatting requirements per LOM-SOP-LAB-201		Placed sections in order
Safety Precautions and Waste Handling	Formatting requirements per LOM-SOP-LAB-201		Added generic sentence referencing *Chemical Hygiene Plan*

Ver. #	Effective Date	Change
03	06/15/04	Major changes are as follows: • Updated to Level 3 format • Added Interferences and Statistical Information/Method Performance sections
02	05/08/02	Major changes are as follows: • Cross Reference section added • Second column confirmation added • Deleted MDL • Updated Personnel Training and Qualifications • Updated Standards
01	12/03/99	New

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Reference:

1. Standard Operating Procedure QA-5550, Pace, Inc., New England, 10/95.
2. SW-846 Method 8000B, Revision II, December 1996.
3. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-025	Monitoring of QC Data Acceptance Limits

Purpose:

The purpose of this procedure is to outline HPLC analysis procedures for dinitrosopentamethylenetetramine (Opex) in wastewater.

Scope:

The method allows for HPLC analysis using a direct aqueous injection of Opex (CAS 101-25-7).

	LOQ µg/L
Opex (Dinitrosopentamethylenetetramine)	100

Basic Principles:

Water samples are pH adjusted to 9 with a hydroxide solution. Filtration is performed, followed by HPLC analysis. Separation is accomplished using a C18 column and ACN/phosphate buffer mobile phase. A UV detector at 230 nm is used for quantitation.

Interferences:

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. The UV detector is a general detector and many organics will respond. All glassware is rinsed with solvent before use and a method blank is performed with each batch of samples to demonstrate that the system is free of contaminants.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Avoid inhaling solvents and getting them on the skin. When handling standard materials, use gloves and protective clothing, including protective eyewear. Avoid breathing standards or getting them on the skin.

All solvent waste is collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal in to the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed.

Personnel Training and Qualifications:

Each analyst performing the instrumental analysis will work with an experienced analyst for a period of time until s/he can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret the chromatograms, and enter data into the LIMS. S/he will also follow the Department Training Manual for Analysts. Proficiency is measured through documented audits of the tasks listed and overchecking of data. An annual quad study for any of the Direct Aqueous Injection HPLC tests will suffice for measuring proficiency.

Sample Handling:

Samples are collected in 100-mL amber glass containers with a Teflon™ lined cap. Store at 4° ± 2°C until the analysis. All samples must be analyzed within 7 days of collection.

1. Transfer 10 mL of sample to a polypropylene cup using a 10-mL graduated cylinder. Determine the pH of the sample. Record the initial pH in the sample preparation log. Add 5 N NaOH, 5 µL at a time while recording the pH and volume added until a pH of 9 is obtained.

2. Prepare a disposable filter assembly with a 0.45 µm filter. Add 5 mL of sample to the syringe. Discard the first 3 mL of sample and collect the remaining 2 mL into a labeled HPLC vial.

3. Prepare a laboratory control sample by adding 0.1 mL of stock standard to a 10 mL volumetric containing HPLC grade water, which has been pH adjusted to 9. Bring to volume, filter and analyze.

4. Prepare a method blank by filtering 5 mL of HPLC grade water, which has been pH adjusted to 9.

5. Prepare a matrix spike/matrix spike duplicate by spiking two aliquots of a background sample with 0.1 mL of stock standard. Adjust pH to 9, while recording volume changes. Filter, and analyze.

Apparatus and Equipment:

1. HP-1100 Liquid Chromatograph, or equivalent, equipped with binary or quaternary pump system with UV detector

2. Analytical balance, accurate to 0.1 mg

3. pH Meter, accurate to 0.1

Reagents and Standards:

A. Reagents

1. HPLC grade water
2. HPLC grade acetonitrile
3. Potassium phosphate monobasic, KH_2PO_4, ACS grade
4. Potassium phosphate dibasic, K_2HPO_4, ACS grade
5. Dinitrosopentamethyelentetramine (supplied by client)
6. Sodium Hydroxide – ACS grade
7. Nylon filters, 0.45-µm
8. Syringe filter assemblies, 5-mL disposable
9. Various sizes of Class A volumetric pipettes, flasks, and syringes
10. Syringes, 10-µL, 250-µL, 1-mL
11. HPLC vials, 2-mL with Teflon™ lined caps
12. Polypropylene cups, 25-mL
13. 10-mL graduated cylinders, 1 per sample

B. Standards

1. Stock standards are prepared by weighing on an analytical balance approximately 15 mg of neat Opex into 200 mL of HPLC grade water which has been pH adjusted to 9 using 5N NaOH. The mass is recorded to the nearest 0.0001 grams. Stock standard are good for 6 months, along with working standards. Standards should be stored at 4° ± 2°C in a refrigerator.

2. Working standards – Prepare calibration standards by adding the aliquot listed below to a 10 mL volumetric flask that contains some of the dilution solvent. Bring to volume after addition of the stock.

Standard Level	µL added	Stock	FV	Approx. µg/L	Solvent
Level 1	15	Opex Stock	10	100	pH 9 Water
Level 2	30	Opex Stock	10	200	pH 9 Water
Level 3	60	Opex Stock	10	400	pH 9 Water
Level 4	100	Opex Stock	10	750	pH 9 Water
Level 5	150	Opex Stock	10	1125	pH 9 Water
MDL	1	Level 2	10	200	pH 9 Water

Standards Database Entry:

Level 1: OPEX1_

Level 2: OPEX2_

Level 3: OPEX3_

Level 4: OPEX4_

Level 5: OPEX5_

Chromatographic Procedure:

Prepare phosphate buffer by weighing on an analytical balance exactly 5.444 grams of KH_2PO_4 and 9.124 grams of K_2HPO_4 into 4 L of HPLC grade water. Filter through a 0.45-µm nylon filter prior to use. Mobile phase expires in 6 months. Filter and degas at least monthly prior to use. Check pH prior to use. The allowable pH range is 6.5 to 7.5. When Opex is detected using the primary column, second column confirmation must be performed. Simultaneously primary and confirmation analysis may be accomplished using a "T" splitter, and dual detectors.

The following chromatographic conditions are optimum, but can be changed to improve linearity, sensitivity, and chromatography on each system.

Primary Column:

Column: Supelco PAH, 25 cm × 4.6 mm × 5 µm

Mobile Phase: 25% ACN/Phosphate Buffer, 1.0 mL/min.

Injection Size: 20 µL

Detection: UV at 230 nm

Column Temperature: 35°C

Confirmation Column Options:

1. Capcell CN, 25 cm × 4.5 mm × 5 µm
   Mobile phase as listed above

2. Thermo hypersil phenyl, 25 cm × 4.6 mm × 5 µm
   Mobile phase as listed above

Calibration:

An external standard calibration is performed on the HPLC using 5 levels of standards ranging from 100 to 1000 µg/L. If the response factors over the calibration range have a relative standard deviation (RSD) less than or equal to 20%, the average RF may be used to calculate results. If the RSD of the response factors exceeds 20%, then a linear calibration curve must be used.

Continuing Calibration:

A continuing calibration standard must be injected before and after every 10 samples. The mid-level standard is used and the calculated response must be within ±15% difference from the expected value or the system must be recalibrated. All samples injected since the last continuing calibration verification standard which met specifications must be reanalyzed after a new calibration.

Calculations:

$$\frac{Pk\ Ht \times FV \times DF}{RF \times IV} = Concentration\ \mu g/L$$

Where:

RF = average response factor (Peak Height/µg/L)

FV = Final Volume in mL

DF = Dilution Factor

IV = Initial Volume of sample extract (mL)

Statistical Information/Method Performance:

Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These

are run on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Quality Assurance/Quality Control:

1. A reagent blank (using deionized water) is prepared with every batch of 20 samples or less to verify the absence of glassware or system contamination.

2. A laboratory control sample (spike reagent blank) is prepared with every batch of 20 samples or less to ensure acceptable extraction performance.

3. A matrix spike and matrix spike duplicate (using client's samples) are extracted every 20 samples or less.

4. See SOP-PP-002 on QC acceptance limits and corrective action. See SOP-PP-025 on monitoring QC limits.

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STANDARD OPERATING PROCEDURES

Analytical Procedure for the Analysis of Anions by Ion Chromatography

EPA Method 300.0 Part A

SOP ID: INS05600.MA

Revision (4)

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Approved Signatures

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1.0 TITLE: Anions by Ion Chromatography

2.0 MATRIX: drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent water, solids

3.0 METHOD DETECTION LIMIT: Analyte specific

4.0 SCOPE AND APPLICATION:

4.1 This method is applicable to the analysis of drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent water, solids (when prepared using the extraction procedure), and leachates (when no acid- or alkaline-based leaching solution is used).

5.0 SUMMARY:

5.1 Specific anions are separated and detected based on their affinities toward the stationary phase in an ion chromatographic column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species that produce a higher conductance, which is then measured by a detector—the conductance for each eluted ion is proportional to the magnitude of the conductivity measured. The method provides results that are equivalent to those of USEPA method 300.0 Part A and those of ISO methods 10304-1 and 10304-2.

6.0 DEFINITIONS:

Ion chromatography: is a form of liquid chromatography that uses ion-exchange resins to separate atomic or molecular ions based on their interaction with the resin.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is introduced onto the instrument for analysis and can monitor calibration baseline drift and carryover contamination effects.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES:

7.1 Particulates will block the capillary fluidics of the system; therefore all samples are filtered with a 0.45μm filter before analysis.

7.2 Interferences can be caused by substances with retention times that are similar to and/or overlapping those of the analyte of interest. Large amounts of an analytes can interfere with the peak resolution of a closely eluting analyte. Sample dilution and/or fortification can be used to solve many of these problems.

7.3 Method interferences may also be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts of elevated baseline in ion chromatographs.

7.4 Oils or petroleum products present in a sample may cause poor peak resolution. Such problems can often be corrected by diluting the sample by a x2 or x4 factor.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Sodium Fluoride is Highly Toxic.

Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
Sodium Fluoride	Poison	2.5 Mg/M3-TWA as F	Highly Toxic. Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 EQUIPMENT AND SUPPLIES:

9.1 Lachat IC5000 Ion Chromatograph with suppressor cartridge, auto-sampler, and supporting equipment

9.2 Disposable glass test tubes for the auto-sampler (13x100mm)

9.3 Glass Class "A" analytical pipets

9.4 Eppendorf adjustable pipet (100-1,000μL) and disposable tips

9.5 Eppendorf 100μL pipet and disposable tips

9.6 Class "A" volumetric flasks

9.7 Deionized water swish bottle

9.8 5μm pore size nylon syringe filters for auto-sampler (30mm diameter; Fisher Catalog #DDR50T3050)

9.9 0.8μm disk filters (Lachat/HACH Catalog #43011)

9.10 10cc plastic disposable syringe

9.11 5cc plastic disposable syringes

9.12 25mm Teflon 0.45μm pore size syringe filters (Millipore: Fisher Scientific Catalog # 09-740-38B).

10.0 REAGENTS AND STANDARDS

10.1 100 *M* Sodium Carbonate Solution: Create by dissolving 8.40g sodium carbonate (ACS grade or better; Fisher Catalog #S263-500) in about 600mL of deionized water in a 1500mL beaker. Once dissolved pour into a clean 1000mL graduate and bring the volume to 1000mL with deionized water. Filter through a 0.45 um filter. Store in a glass or plastic bottle, refrigerated. EXP: 8 weeks from date of creation.

10.2 100 *M* Sodium Bicarbonate Solution: Create by dissolving 10.60g sodium bicarbonate (ACS grade or better; Fisher Catalog #S233-500) in about 600mL of deionized water in a 1500 mL beaker. Once dissolved pour into a clean 1000mL graduate and bring thel volume to 1000mL with deionized water. Filter through a 0.45 um filter. Store in a glass or plastic bottle, refrigerated. EXP: 8 weeks from date of creation.

10.3 Eluent Solution: Create fresh daily. Into a 1 gallon plastic container, place 60mL of the stock 100 *M* Sodium Bicarbonate solution, 78mL of stock 100 *M* Sodium Carbonate solution, and 2862mL of deionized water. Degas with helium or argon for three minutes. Fill the glass eluent storage bottle (a clean, 2.5L glass bottle) and degas for an additional minute. IMPORTANT! Use only a glass bottle for the eluent reservoir (plastic bottles tend to form air bubbles that will cause the eluent pump to draw a vacuum).

10.4 Regenerant Solution: To about 600mL of deionized water in a 1L volumetric, add 14.0 mL of concentrated sulfuric acid (ACS grade or better; Fisher Catalog

#A300-500). Fill to mark with deionized water, stopper, and mix by inversion. EXP: 1 year.

10.5 Individual Stock Standards:

10.5.1 Purchase individual stock standard concentrates for the anions to be determined as follows:

1000 mg/L Br^-	Ultra Catalog #ICC-001 (100 mL)
1000 mg/L Cl^-	Fisher Catalog #LC13000-7 (125 mL)
1000 mg/L F^-	Fisher Catalog #LC14600-1 (500 mL)
1000 mg/L NO_3^-	Fisher Catalog #LC17900-7 (125 mL)
1000 mg/L NO_2^-	Fisher Catalog #LC17920-7 (125 mL)
1000 mg/L HPO_4^{-2}	Fisher Catalog #LC18590-1 (500 mL)
1000 mg/L SO_4^{-2}	Fisher Catalog #LC25510-1 (500 mL)

10.6 Mixed Working Standard "A": To about 500mL of deionized water in a deionized water rinsed **1000mL Class A volumetric flask**, transfer the indicated volume of each stock 1000mg/L standard from 10.5.1 above as follows. After all additions are made, fill to mark with Deionized water, stopper, and mix by inversion. Store refrigerated. EXP: 2 months when refrigerated.

Analyte	Volume Transferred	Final Concentration
Br^-	5.0 mL	5.00 mg/L
Cl^-	50.0	50.0
F^-	5.0	5.00
NO_3^-	5.0	5.00
NO_2^-	5.0	5.00
HPO_4^{-2}	5.0	5.00
SO_4^{-2}	100.0	100.0

10.7 Mixed Working Standards "B" through "G": Mixed Working Standards "B" through "G" are prepared from Mixed Working Standard "A" as follows, using Deionized water to dilute to final volume in appropriate Class A volumetric flasks. Store refrigerated. EXP: 1 month when refrigerated.

Stnd ID	Stnd A Used	Final Volume	Br^- mg/L	Cl^- mg/L	F^- mg/L	NO_3^- mg/L	NO_2^- mg/L	HPO_4^- 2 mg/L	SO_4^{-2} mg/L
A	--	--	5.0	50.0	5.0	5.0	5.0	5.0	100.0
B	50.0	100	2.5	25.0	2.5	2.5	2.5	2.5	50.0
C	20.0	100	1.0	10.0	1.0	1.0	1.0	1.0	20.0
D	20.0	250	0.40	4.00	0.40	0.40	0.40	0.40	8.0
E	10.0	500	0.10	1.00	0.10	0.10	0.10	0.10	2.0
F	1.00	100	0.050	0.50	0.050	0.050	0.050	0.050	1.0
G	1.00	500	--	--	--	--	0.010	--	--

10.8 Initial Calibration Verification (ICV) Stock Standard: Purchase a commercially prepared standard from Inorganic Ventures (Catalog #STLMA-ICAL-3). Place 1.00mL of ICAL-3 into a 10mL volumetric and dilute to mark with Deionized water. Stopper and mix by inversion several times. This dilution contains 2.5mg/L of fluoride, nitrite, nitrate, bromide, and ortho-phosphate, 25.0mg/L of chloride, and 50.0mg/L of sulfate, which provides approximately midpoint values for the working curve on the IC.

10.9 Spiking Standard: Purchase a commercially prepared standard from Inorganic Ventures (Catalog #STLMA-ICAL-5). Place about 4mL of sample into a 10mL volumetric, add 100μL of ICAL-5, and dilute to mark with sample. Stopper and mix by inversion several times. This spiked sample provides a spike of 1.0mg/L for fluoride, bromide, nitrite, nitrate and ortho-phosphate, 10.0mg/L for chloride and 20.0mg/L for sulfate.

10.10 Laboratory Control Sample (LCS) and Laboratory Control Duplicate (LCD) and Continuing Calibration Verification(CCV) Solution: At the same time as the working standards are created, create a LCS/LCD solution by pipeting the following standards into a deionized rinsed, **500mL Class A volumetric flask**, containing about 100mL of deionized water:

Analyte	Volume Transferred	Final Concentration
Br^-	2.0 mL	4.00 mg/L
Cl^-	20.0	40.0
F^-	2.0	4.00
NO_3^-	2.0	4.00
NO_2^-	2.0	4.00
HPO_4^{-2}	2.0	4.00
SO_4^{-2}	40.0	80.0

10.11 20% Methanol Solution: Prepare by mixing 20mL of methanol (certified grade or better) and 80mL of deionized water. This solution is used to lubricate the pump.

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

11.1 Samples are to be collected in a glass or plastic bottle and refrigerated to 4°C. Since IC measures many individual anions in one run, holding times are determined by the anion to be measured that has the shortest holding time (most commonly nitrite with a 48 hour hold time).

12.0 QUALITY CONTROL

12.1 Routine Quality Control Requirements

QC Analysis	Frequency	QC Criteria	Corrective Action
ICB (Initial Calibration Blank)	Once at start of analytical run	< RL for each anion	Recalibrate IC. Re-check ICB; if still unacceptable, investigate possible contamination of reagents, deionized water, or plumbing on the IC.

ICV (Initial Calibration Verification)	Once at start of analytical run	90-110%	Recalibrate IC. Re-check ICV; if still unacceptable, investigate possible contamination of reagents, deionized water, or plumbing on the IC.
LCS	Per batch of 20 field samples or fraction thereof	85-115%	If unacceptable, re-analyze; if still out recalibrate. Reanalyze samples.
LCD (optional)	Per batch of 20 field samples or fraction thereof	RPD ≤20%	If RPD is out re-analyze. If still out of acceptable limits, report data with qualification.
Matrix Duplicate (MD)[1]	Per batch of 20 field samples or fraction thereof	RPD ≤20%	If RPD is out re-analyze. If still out of acceptable limits, report data with qualification.
Matrix Spike (MS)	Per batch of 20 field samples or fraction thereof	75-125%	If unacceptable, spike new aliquot and re-analyze. If still out of acceptable limits, report data with qualification.
Matrix Spike Duplicate (MSD)	Per batch of 20 field samples or fraction thereof	RPD ≤20%	If RPD is out re-analyze. If still out of acceptable limits, report data with qualification.
CCV (Continuing Calibration Verification)	Every 10 samples or fraction thereof	90-110%	If unacceptable, re-analyze; if still out recalibrate. Reanalyze samples not bracketed by passing CCVs, values for blocks between acceptable CCVs may be reported.
CCB (Continuing Calibration Blank)	Every 10 samples or fraction thereof	< RL for each anion	If unacceptable, re-analyze; if still out recalibrate. Reanalyze samples not bracketed by passing CCBs, values for blocks between acceptable CCBs may be reported.

12.2 Annual Quality Control Requirements

ITEM	FREQUENCY	QC CRITERIA	REQUIREMENTS
Reporting Limit (RL)	n/a	Refer to the lowest reported working curve standard	RL must be ≥calculated MDL
Method Detection Limit Calculation (MDL)	Once per year	MDL must be ≤ RL	Spike 7 lab prepared samples at between 3 and 5 times the lowest working curve value to determine MDL.
Demonstration of Capability (DOC)	Once per year per analyst performing the analysis	75-125%	Analysis of 4 laboratory prepared samples ±25% of true value to demonstrate proficiency.
Varied CCV Concentration	Once per year	90-110%	A CCV at a concentration level different than that routinely used, record this evaluation in the CCV file.

13.0 CALIBRATION AND STANDARDIZATION

13.1 See PROCEDURE below.

[1] Performed only if specifically requested by the client.

14.0 PROCEDURE

14.1 IC Instrument Preparation:

14.1.1 Prepare a fresh eluent batch as described in Item 10.3. This eluent volume is sufficient to analyze about 65 injections. If more than 65 injections are anticipated, make a double batch of eluent, and switch from the 2.5L eluent container to the 6L Erlenmeyer eluent flask. Record eluent creation in the appropriate log book stored by the IC.

14.1.2 If the working standards are more than 60 days old, discard them and create fresh working standards according to Items 10.6 and 10.7 above, and fresh LCS/LCD (optional) and CCV solutions according to Items 10.8 and 10.10. Use the same volumetric flasks each time; simply rinse the flasks four times each with 10mL portions of Deionized water to clean them. Record the creation of fresh standards in the appropriate log book stored by the IC.

14.1.3 Change the auto-sampler filter by removing the filter from the lure-lock attachment atop the stainless steel sample probe, and replacing it with a fresh 30mm syringe filter (5.0μm pore size). Refer to Item 14.5.2 for how often to change this filter.

14.1.4 Prime the IC pump as follows:

14.1.4.1 Open the pump valve one-half of a turn counterclockwise.

14.1.4.2 Place three wadded Kimwipes below the pump outlet to absorb any eluent, and place the tip of a 10cc plastic syringe against the pump valve's nipple.

14.1.4.3 Press the "Prime" button to start the pump. **IMPORTANT!** The pump valve must be open **BEFORE** pressing the "Prime" button.

14.1.4.4 As the pump engages, withdrawn the syringe handle to create a vacuum; eluent will eventually be drawn into the syringe, which also will cause any air bubbles in the line to be removed as well. Once about 4mL of eluent is in the syringe, remove the syringe, discard the eluent, and repeat this process twice more to assure that all air is removed from the pump.

14.1.4.5 Press the "Prime" button to turn off the pump. Once the pump has stopped, close the pump valve finger-tight.

14.1.5 Check the level of sulfuric acid regenerant solution. If it is below 100mL make fresh solution and top off the reservoir bottle.

14.1.6 Check the level of 20% methanol solution, used to lubricate the pump. If it is below 60mL, replace the solution with a fresh batch.

14.1.7 Check the deionized gallon bottle; if it is not full, refill it with deionized water to the bottle's neck.

14.1.8 Start the IC pump by pressing the "Run/Stop" button. Press the "mode" button until the indicator light shows "PSI" for the LED readout. Allow the IC to pump eluent for at least 10 minutes. After this time, the psi value for the IC should be below 1500; if not, and the disk filter has yet to be replaced, stop the pump, replace the disk filter and check the psi value after doing so. If it is still not below 1500, the guard column and/or analytical column may need to be cleaned or replaced. In such a case, see the department supervisor for assistance.

14.1.9 If the IC has not been run for more than 30 hours, prime the suppressor by pressing the "Initialize" button on the left front of the IC. The light

should change to yellow as it begins its 6 minute acid washing of the suppressor cartridge. If the light should change to red, simply press the "Initialize" button again to re-start the cycle.

14.1.10 While the IC is equilibrating (eluent pumping through the system for 10 minutes) set up your batch in the IC software as follows:

14.1.10.1 Turn on the computer and printer on if they are not already operating.

14.1.10.2 Launch the Omnion software by clicking on the symbol in the quick menu bar at the bottom of the screen, or double clicking on the Omnion icon on the desktop.

14.1.10.3 Click "OK" when the Omnion logo appears. The auto-sampler will then do a test run, and the software will then launch afterward.

14.1.10.4 Click on "IC5000" from the menu. For user enter "demo" and click "OK."

14.1.10.5 Maximize the window for the control bar and for the chromatograph view.

14.1.10.6 Create a new batch for the analytical run by holding down the "Ctrl" key and typing a "B." The batch window will then appear, and the computer will ask if you want to save the current batch. Click on "Yes." The batch information window then appears. Change the current batch designator in the "Data Path" field to today's date in the format of "MMDDYY" (so that the entire data path looks like this: "C:\OMNIONIC\MMDDYY"); in the "Sample ID" filed, type the today's date in the format MMDDYY; click on the blue file folder icon to the right of the blank field beside "Method", selecting "5an300a1.met" and clicking "OK"; for the "Filename" field enter "(001)"; and in the bottom-most box, enter the number of samples to be run (generally overestimate this number; you can always delete extra lines in the batch later.). Click "OK" to generate the batch.

14.1.10.7 Under the "Sample ID" column, enter the following in sequence, starting in Row 1:

STND A
STND B
STND C
STND D
STND E
STND F
STND G
ICV (ICAL3 X10)
ICB
MB1
LCS1
LCD1 (optional)
ID for Sample #1
ID for Sample #2
...

ID for Sample #8
CCV1 (STND C)
CCB1
ID for Sample #9
...
ID for Sample #18
CCV2
CCB2
MB2
LCS2
LCD2 (optional)
ID for Sample #19
ID for Sample #20
...
CCV3
CCB3
...

NOTE: If requested by the client, an MD may be run in addition to the QC samples listed. These would be fit into the analytical batch according to the criteria specified for each.

14.1.10.8 Next scroll over to the "Run Type" column. Click on the box in Row 1 (the same row as the "STND A") and select "Calibration" from the menu, then click on "OK." The "Unknown" should change to a blue "Calibration" notation. Repeat this for all of the Standards.

14.1.10.9 Scroll over to the "Level" column. For STND A enter a "1"; for STND B enter "2"; C a "3"; D a "4"; E a "5"; F a "6"; and G a "7". All other samples should remain at the default "0" value. **IMPORTANT!** The level numbers must be entered for the computer to understand that these are working standards to be used for curve creation; if they are not entered, the computer will not generate a working curve.

14.1.10.10 Change the numbers in the "Vial" column as needed to properly coordinate the samples in the auto-sampler rack with the listing on the batch on the computer. The first rack (the left-most as you face the auto-sampler) is numbered 1 through 90, and correlates as positions 1001 through 1090 on the computer for the "Vial" column. Positions 1 through 16 correspond to the larger, plastic vials on the standards rack which is in a fixed position at the back of the auto-sampler tray. The second rack from the left is numbered as 2001 to 2090; the third rack is 3001 to 3090; and the last rack is 4001 to 4090. **IMPORTANT!** You cannot address the fourth rack (positions 4001 to 4009) until every position in the first three racks is full.

14.1.10.11 Generally, the LCS/CCV is placed in a glass test tube in position 15 of rack 1 (spot 1015). If there are more than 4

LCS/CCVs to be run in the batch, add a second test tube of CCV to position 1014.

14.1.10.12 Since the CCB and ICB use deionized water, the larger, plastic vial in Position 1 is utilized for these samples. Therefore, in the example in Item 14.1.11.7 above, the vial positions corresponding to the sample IDs may look something like:

Vial	Sample ID
1001	STND A
1002	STND B
1003	STND C
1004	STND D
1005	STND E
1006	STND F
1007	STND G
1008	ICV (ICAL3 X10)
1	ICB
1	MB1
2	LCS1
2	LCD1 (optional)
2001	ID for Sample #1
2002	ID for Sample #2
	...
2008	ID for Sample #8
1015	CCV1
1	CCB1
2009	ID for Sample #9
	...
2018	ID for Sample #18
1015	CCV2
1	CCB2
1	MB2
2	LCS2
2	LCD2 (optional)
2019	ID for Sample #19
2020	ID for MS/MSD
	...
1015	CCV3
1	CCB3
	...

Note that the vial ID is repeated for each LCS/CCV occurrence; just one vial is used as opposed to making several unique vials containing the same standard. The same holds true for the LCS/LCD (optional) and the CCB/MB and ICB.

Also note that the standards and QC samples are in Rack 1 or the back rack, while the samples have been segregated to Rack 2. This is done simply for better record keeping when preparing the samples, and allows the

standards to be set up and a run initiated while the analyst works on filtering and setting up the samples, thus saving front-end start-up time for the analysis.

IMPORTANT! If more than four LCS/CCVs are listed in a batch, also place a full test tube of CCV solution in Position 14 of Rack 1 (1014) and address this for CCV starting with CCV5.

14.1.10.13 Once the batch is completed, highlight any unused lines by placing the cursor of the mouse on the starting line and dragging it along, while holding the left mouse button down, to encompass all excess lines. Then press the "Delete" key to remove them.

14.1.10.14 From the "Method" menu item, select "Export Options." Change the current date in the data path to today's date in the format "MMDDYY" and click "OK." A window will appear to state the data path doesn't exist; click "yes" to create it.

14.1.10.15 Next from the "File" menu item, select "Save Batch As." The "File Name" field will be highlighted in blue showing currently "*.seq" in it. Click in the field to activate it, and scroll over with the arrow keys to the far left. Press the "Delete" key once to erase the "*" part of the field, and type in today's date in the format of "MMDDYY" so that the complete field reads "MMDDYY.seq".

14.1.10.16 Still in the "Save Batch As" window, next double click on the "omnionic" item in the "Directories" window, to reveal the list of directories. Scroll down with the arrow bars until you see the directory with today's date, and then double click on it to open. Click "OK" to save the batch in the export directory of the same date.

14.1.10.17 The batch is now complete and ready to be run.

14.1.10.18 Note that once an analytical batch has been started you can add additional samples to the batch even though the batch is being run by the instrument. This is useful if samples come in and the IC has already started the analysis. To add samples to a batch already in progress:

14.1.10.18.1 Do a "Cntrl-B" to open the batch window, and maximize it.

14.1.10.18.2 Scroll down to the first blank line. Enter the Sample ID under that column, and then enter the appropriate Vial number corresponding to the sample position in the sample rack. Then go to the fields in turn that are blank and copy the data from the lines above into them by doing Cntrl-C to copy and Cntrl-V to paste so that all fields are filled in. **REMINDER!** Always finish the batch with a close-out CCV/CCB pair, and be sure to maintain all other QC requirements (see item 12.0) when adding samples to a batch.

14.1.10.18.3 Go to the "File" menu and select "Save Batch." Click "Yes" when asked if you want to update the batch

that is running. **IMPORTANT!** No changes or additions to the batch will be recognized by the computer unless you "Save Batch" before closing the batch window!

14.1.11 Sample Preparation

14.1.11.1 Prepare the standards and QC samples to be run by filling a 13x100mm test tube for each, respectively, and placing them in the auto-sample tray in the position corresponding to the Vial ID for each sample as shown in the batch window.

14.1.11.2 **All samples MUST BE FILTERED** before analysis. Use the 0.45μm pore size, 25mm diameter syringe filters and disposable 5cc syringes to filter each sample aliquot.

14.1.11.3 Make appropriate QC samples according to the frequency specified in Item 12.0, and make spikes or MS/MSD according to item 10.9.

14.1.12 Setting Peak Retention Times

14.1.12.1 The IC should have been pumping eluent for at least 10 minutes as you move through setting up the batch. If not, allow it to continue pumping eluent until 10 minutes has elapsed.

14.1.12.2 In the run time window, which shows the real-time chromatograph of the last run completed, click on the blue colored arrow icon titled "Run Single." For the sample ID enter "TEST" and click "Yes" or "OK" for all subsequent windows that appear. The IC will then draw a sample from Position 1001 (STND A) and analyze just that single sample.

14.1.12.3 After the analytical run is complete, the pump shuts down automatically. After it has, press the "Run/Stop" button on the front panel of the IC to start the pump again. If a red light on the "Initialize" button comes on at any point, either after the TEST run or during an analytical run, refer to the "Troubleshooting" section.

14.1.12.4 Evaluate the chromatogram on the computer screen. The readout places a time window bracket at the top of each of the seven peaks produced from the run. Ideally, the peak's tip should be slightly to the LEFT of the midpoint of the window, so as to allow for proper identification of the peak by the computer as the run progresses and the eluent looses strength (which will tend to shift peaks to the right). To determine the peak retention time, place the cursor of the mouse over the tip; a line is automatically drawn vertically to assist in placement, and a time readout appears in the upper left of the screen. Record the time on a piece of paper for each peak.

14.1.12.5 To change peak retention times, hold the "Ctrl" key down and type a "P" to open the peak window. Using the mouse cursor, click in the appropriate time boxes for a compound and change the times as needed. Close the window out after making changes by clicking on the "X" in the upper right of the window, and note the changes automatically made to the current chromatogram on the screen. If the changes are too dramatic, or not enough, make further changes by this procedure until the peak times are correctly identified.

14.2 Processing an IC Analytical Run

14.2.1 To begin the analytical run, click on the green arrow icon labeled "Run Batch"; the computer will then open a window asking what the range of the run is to be. Generally, leave this as the default "First" and "Last"; if you wish to re-run a sample, or start part of the analytical run over, you could change the "First" to the Row number in the batch window for the re-start point, and/or change the "End" to the Row number in the batch window for the re-start endpoint for the run. **NOTE!** These numbers refer to the Row Number in the batch window, **NOT** the vial numbers under the Vial column.

14.2.2 The IC will then automatically analyze the samples in the order specified in the batch window, starting with Row 1 and ending at the last Row with data in the batch window.

14.2.3 The IC is currently set up to print out a chromatogram after each sample is run. Make sure that there is sufficient paper in the printer tray to accommodate the number of samples to be run.

14.2.4 Note that you can add new samples, or add dilutions of samples whose results were over-range to the batch even after it has begun processing.

14.2.5 Should the red light illuminate on the "Initialize" button on the IC, indicating a fault in the system, refer to the "Troubleshooting" section.

14.2.6 The IC should shut down the pump automatically once the analytical run has finished. If it does not, shut the pump down manually by pressing the "Run/Stop" button.

14.3 For solids and soils, perform the following extraction procedure to generate aqueous extract for analysis:

14.3.1 To a 250 mL beaker, 1add about 10 g of solid material (record the actual weight used) and 100mL of Deionized water. Mix the slurry with mechanical stirring for 60 min, after which time filter the extract through a Whatman 3 qualitative filter into a 100mL volumetric flask. Rinse the filter's contents twice with small (10mL) portions of Deionized water to make the transfer quantitative. Dilute the flask to the mark with Deionized water, stopper, and mix by inversion. Pour off into a 120mL plastic bottle, appropriately labeled with the sample ID number. Analyze the extract as you would a regular water sample.

14.4 Troubleshooting

14.4.1 Eluent Reservoir Runs Dry

14.4.1.1 If the eluent reservoir runs dry during an analytical run, stop the run by pressing the "Run-Stop" button on the IC and clicking on the "Stop" stop sign icon on the computer.

14.4.1.2 Make fresh eluent according and after degassing it, fill the eluent reservoir bottle.

14.4.1.3 Re-prime the pump and then allow the eluent to pump through the IC system for 10 minutes.

14.4.1.4 Perform a calibration and retention time check.

14.4.1.5 Re-start the analytical run from the sample immediately after the last passing, acceptable CCV/CCB result.

14.4.2 Red Initialization Light

14.4.2.1 Should the red light illuminate on the "Initialize" button on the IC, it means that a problem exists with the suppressor

cartridge—usually insufficient acid regeneration to produce adequate suppression of the conductivity baseline.

14.4.2.2 If an analytical run is underway, stop the run by clicking on the "Stop" stop-sign icon, and click "Yes" when the window prompts to abort the run. Press the "Initialize" button; the light should change to yellow and the IC should reinitialize the cartridge. After the 6 min regeneration cycle, the light should turn green, indicating that it is acceptable to continue.

14.4.2.3 To continue, open the batch window and determine the Row Number in the batch window for the last sample that produced an acceptable result (by reviewing the chromatogram printouts as needed). Click on the green arrow icon labeled "Run Batch"; when prompted for the range for the run, enter the Row Number for the last acceptable sample as previously determined, and then click on "OK." Click through each subsequent request(s) to overwrite data with a "Yes" response. The computer will start over at the sample specified, and finish out the analytical run.

14.4.2.4 If the light is red but no analytical run is underway, press the "Initialize" button to manually start the suppressor regeneration cycle. The light should change to yellow, and then after the 6 min cycle, turn green. If not, check the regenerant solution supply to make sure it is adequate. If it is adequate, but the suppressor is more than 6 months old, replace the suppressor unit. If this still does not result in a green light, or the light faults to red after just a few injections, call Lachat Customer Service for assistance.

14.4.3 Pressure Fault or Pressure over 1500 psi

14.4.3.1 Should the IC shutdown because of a pressure fault, chances are that the disk filter is clogged and needs replacement. Replace the filter and restart the system; if the pressure remains high (over 1500 psi) then chances are that one or more of the lines, or the guard column and/or the analytical column are clogged. In this case, refer to the "Maintenance" section.

14.4.3.2 If the system is running adequately, but the pressure is at or above 1500 psi, chances are that the analytical run will not go much further before stopping automatically. In such a case, wait for the sample in progress to finish eluting, manually stop the run by clicking on the "Stop" stop sign icon, and clicking "Yes" when asked to abort. Stop the pump if necessary by pressing the "Run/Stop" button. Replace the disk filter, and then turn the pump on by pressing the "Run/Stop" button. Allow the system to pump eluent for 10 minutes, and then click on the green arrow icon "Run Batch," and enter for the starting sample the Row Number from the batch window for the next sample to be analyzed, then click "OK."

14.4.3.3 If changing the disk filter fails to correct the problem, change out the entire disk filter unit for a fresh one (a spare new one is kept in the supplies drawer). If the pressure still remains high, contact Lachat Customer Service for assistance.

14.4.4 The ICB or CCB produce peak(s) for analyte(s) of concern.

14.4.4.1 Should the blank water (Deionized) water produce a peak or peaks for any of the seven ions of concern, it generally indicates that either:

14.4.4.1.1 The wash water reservoir is contaminated and needs to be changed out completely;

14.4.4.1.2 The sample cup containing the blank has become contaminated and needs to be rinsed and replenished with fresh Deionized water; or

14.4.4.1.3 The auto-sampler filter is contaminated and the auto-backflushing process it undergoes is not sufficient to remove the contamination, in which case the run should be stopped and the filter should be replaced.

14.4.4.2 If none of these suggested corrective actions corrects the problem, call Lachat Technical Support for further assistance.

14.5 Maintenance

14.5.1 Auto-sampler Filter

14.5.1.1 Auto-sampler filters should be replaced with every analytical run performed, unless the previous run was short (less than 20 injections), in which case the same filter may be used on consecutive runs. The auto-sampler filter is generally good for 60-80 injections.

14.5.2 Suppressor Cartridge

14.5.2.1 Replace the cartridge if a noisy baseline is continually noted, or if the red light illuminates on the "Initialize" button several times within just a dozen or so injections. In any case, replace the suppressor every three months.

14.5.3 Guard Column

14.5.3.1 If the pressure reading climbs past 1500 psi consistently, the guard column could be clogged. Perform the EDTA back flushing cleaning method on the column (as well as analytical column) to remove any precipitated polyvalent compounds. If the column still produces operating pressures above 1500 psi to start, replace it. In any case, replace the guard column once every three months or as needed.

14.5.4 Analytical Column

14.5.4.1 If the pressure reading climbs past 1500 psi consistently, the analytical column could be clogged. Perform the EDTA backflushing cleaning method on the column (as well as guard column) to remove any precipitated polyvalent compounds. If the column still produces operating pressures above 1500 psi to start, replace it. In any case, replace the analytical column once every six months.

14.5.5 Auto-sampler Injection Line

14.5.5.1 Replace the injection line on the auto-sampler annually, or more frequently if it becomes discolored or noticeably brittle.

14.5.6 Waste Tubing

14.5.6.1 Replace the waste line tubing annually or more frequently if it becomes discolored or noticeably brittle.

14.5.7 Analytical Path Tubing

14.5.7.1 The PTFE tubing that comprises the main bulk of the IC plumbing should be replaced annually with identical lengths and type of tubing. Replace ferrules and o-rings as needed at this time, based on a simple visual examination.

14.5.8 Eluent Uptake Line

14.5.8.1 The eluent uptake line should be replaced annually, as well as the fritted uptake filter at the end of the line. If the fritted end becomes yellow or otherwise discolored, replace it sooner than annually.

14.5.9 Cleaning the Guard and Analytical Columns

14.5.9.1 Over time, the columns become clogged with polyvalent compounds precipitating out. To correct this, periodically clean the columns by backflushing them with a 0.5 *M* EDTA solution, followed by a 0.5 *M* sodium carbonate solution flush, and then an eluent flush. Lachat has available a specific method for this procedure, located in the operating manual.

14.5.10 Other Maintenance

14.5.10.1 Perform other maintenance as specified by the IC operating manual or Lachat Technical Support, on an as needed basis.

15.0 CACULATIONS

15.1 The instrument provides data calculation automatically, and is imported into and Excel spreadsheet for easy viewing and reporting of data. Verify that all quality control parameters for the QC samples run meet criteria before permitting data to be reported. If any QC samples fail, re-analyze the samples for which the QC data failed. Report concentrations in mg/L directly from the Excel spreadsheet, making any adjustments for dilutions.

15.2 Import the run data directly into an Excel spreadsheet as follows:

15.2.1 Launch Excel.

15.2.2 Using the "Open File" command, locate the data file in the date-based folder for the analytical run in question. This will be under the "OmnionIC" drive under "My Computer," and the specific data for the run will be in the folder with the date for the data in question. **Select "View all files"** and then look for the file called "5an300a1" in the folder. Open it.

15.2.3 A window will appear indicating that the data is delineated and can be imported into Excel. Click the "Next" button, then the "Finish" button to import the data into Excel.

15.2.4 The data is now available in spreadsheet format. The analyst may make column width changes and other cosmetic changes to allow the data to fit on a landscape view page for printing, but may not remove or alter any of the base data provided by the IC instrument. Once ready for printing, print a copy for data entry into Labnet, and save the file as a Microsoft Excel Workbook using the date as the filename in the format of "MMDDYY."

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1, 2, 3 or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.2.1 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's Chain of Custody? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

18.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

18.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

19.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

19.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective

action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

20.0 WASTE MANAGEMENT

20.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCES

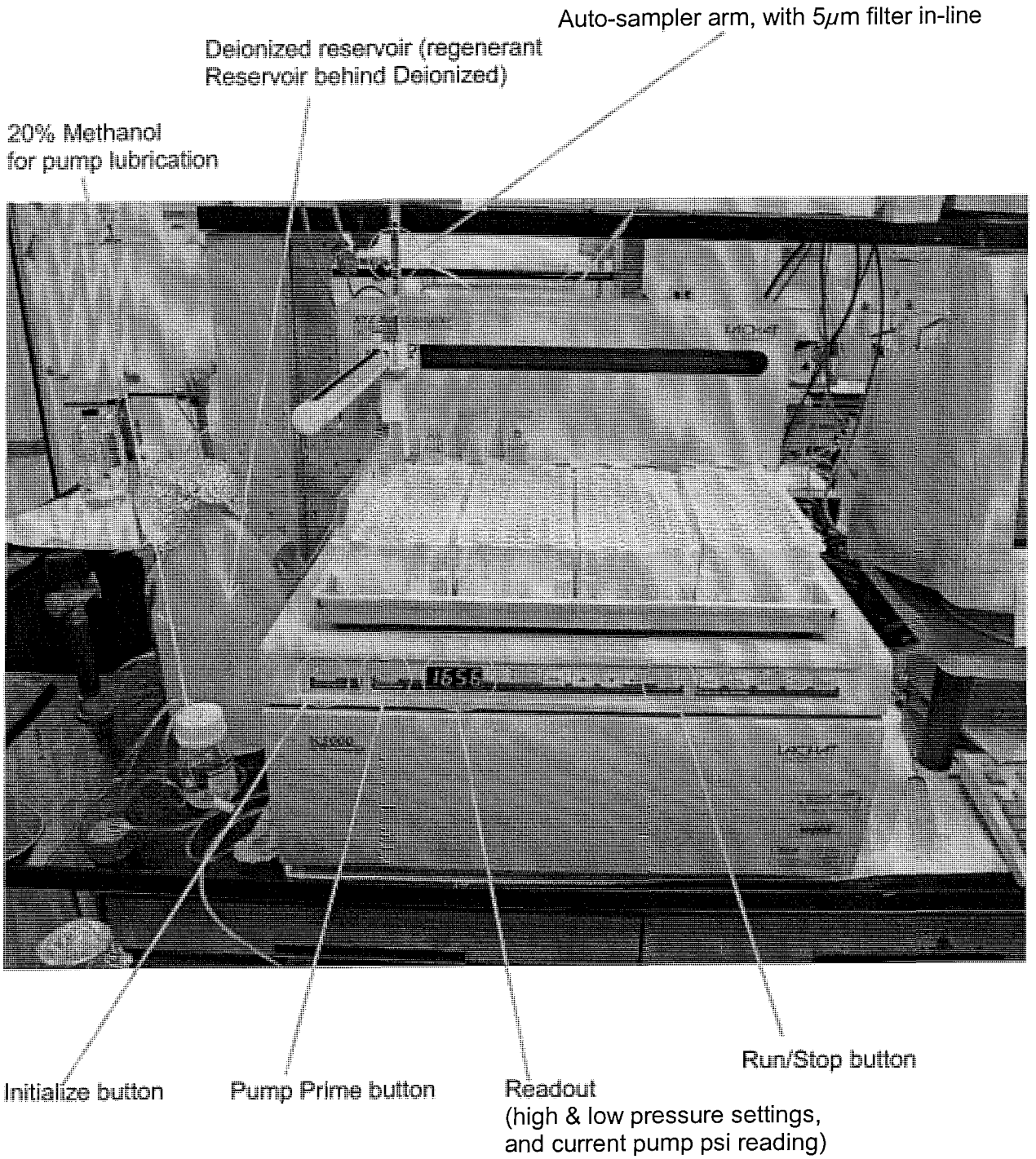
22.1 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, Method 365.3, (March 1983).

22.2 *Standard Methods for the Examination of Water and Wastewater*," 18th Edition, 1992, Method 4500 PE.

23.0 TABLES, DIAGRAMS, FLOWCHARTS VALIDATION DATA

Corrections and/or additions:
2/19/07replaced Milli-Q with deionized water, modified section 9.12 for Millipore, FS catalog#, adjusted volumes in section 10.6, removed section 10.10 CCV, added CCV to LCS information. Removed section 14.1.4, adjusted amounts for section 14.3.1, and removed section 14.5.1.1 – disk filter.

INSTRUMENT OVERVIEW DIAGRAM



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STANDARD OPERATING PROCEDURES

Analytical Procedure for the Automated Analysis of Specific Conductivity, Alkalinity and/or pH using the ManSci MANTECH Autotitrator

EPA Methods 120.1
EPA Methods 150.1
EPA SW-846 Method 9045C
Standard Methods 2320B
Standard Methods 2510B

SOP ID: INS07300.MA
Revision (1)

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Revision Date: 05/26/06
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Approved Signatures and Dates

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QUALITY ASSURANCE OFFICER

LABORATORY DIRECTOR

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1.0 TITLE: Specific Conductivity, Alkalinity and/or pH by Automation

2.0 MATRIX: Inorganic Chemistry

3.0 METHOD REPORTING LIMIT:
- 3.1 Alkalinity = 1.0 mg/L
- 3.2 Conductivity = 1.0µS/cm
- 3.3 pH = N/A

4.0 SCOPE:
- 4.1 The method is applicable to drinking, surface, ground, and saline waters; domestic and industrial wastes; deionized water leachates from solid materials; and acid rainwater.

5.0 SUMMARY:
- 5.1 The specific conductance of a sample is measured by use of a conductivity meter.
- 5.2 The pH of a sample is determined by potentiometric measurement using an approved pH probe and thermometer for temperature correction.
- 5.3 The alkalinity of a sample is measured by the addition of a specific concentration of acid to an endpoint of 4.5 (or for samples with <20mg/L alkalinity, to pH 4.3), the endpoint being determined by potentiometric measurement using an approved pH probe and thermometer for temperature correction.

6.0 DEFINITIONS

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Anomaly: a situation that was caused by the actions of the laboratory so that a process did not follow established standard operating procedure, protocol, or regulation (e.g. the analyst added the incorrect amount of matrix spike material to an investigative sample).

Batch: a set of investigative and/or quality control samples, which are prepared and/or analyzed together with the same process, and using the same lot(s) of reagents. A preparation batch is composed of between 1 and 20 investigative and/or quality control samples (or a different number of investigative and/or quality control samples as specified in an SOP) of the same general matrix, which meet the aforementioned criteria. An analytical batch is composed of prepared investigative and/or quality control samples, extracts, digestates and/or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Deficiency: a situation that was caused by the actions of the client or sampler so that a process did not follow established standard operating procedure, protocol, or regulation (e.g. some of the client's sample bottles arrive at the lab broken or the containers had the incorrect preservative for the tests to be run on the samples).

Holding Time: the maximum time that a sample may be held before it is prepared for and/or physically analyzed, while still considered as a valid sample pursuant to the specifications of the analytical method.

Instrument Blank: an aliquot of deionized water to which the same reagents are added as are added to the investigative and/or quality control samples, and is used to zero the instrument.

Laboratory Control Sample (LCS): a quality control parameter that is a deionized water sample that is spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as the investigative samples through all steps of the preparation and analytical procedures.

Laboratory Control Sample Duplicate (LCSD): a quality control parameter that is a duplicate of the LCS as described above, prepared separately from the LCS for purposes of judging method reproducibility.

Matrix Duplicate or Sample Duplicate (DU): a quality control parameter that consists of a duplicate aliquot of an investigative sample processed and analyzed independently; under the same laboratory conditions, as the original sample.

Matrix Spike (MS): a quality control parameter that consists of an investigative sample to which a known amount of target analyte(s) is added, in order to judge if there are any interferences intrinsic to the investigative sample that would prevent or inhibit the recovery of the target analyte(s).

Matrix Spike Duplicate (MSD): a quality control parameter that is a duplicate of the MS as described above, prepared separately from the MS for purposes of judging method reproducibility for the matrix in the investigative sample.

Method Blank: a quality control parameter that consists of deionized water that is processed simultaneously with, and under the same conditions as the investigative samples through all steps of the preparation and analytical procedures.

Method Detection Limit (MDL): the minimum concentration of a target analyte that can be measured with a specified degree of confidence, that is greater than zero, when using a specific method. By definition, the MDL has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD) as given in 40 CFR Part 136 Appendix B.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: a situation that is out of the ordinary that is worth noting, but may not necessarily have an impact on the analysis, handling and/or processing of the investigative samples (e.g. the client doesn't send a chain of custody form with the samples).

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets a set of defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from an analytical measurement activity or study, recorded in a laboratory notebook, on worksheets, in other records, in memoranda, in notes, or exact copies thereof, and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media (including dictated observations), and recorded data from automated instruments.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be at or above the MDL.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

7.0 INTERFERENCES

7.1 Molecules of organic compounds that do not dissociate in aqueous solution conduct a current very poorly.

7.2 Soaps, oily matter, suspended solids or precipitates may interfere with the response of the glass electrode. To correct for this, pause for a few seconds between each addition of titrant. Do not filter, dilute, concentrate or alter sample.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS**

for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
0.02 *N* HCl	Corrosive	N/A	This weak acid may cause irritation if comes into contact with the skin or eyes, or is accidentally consumed.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 Man-Tech Autotitrator system

9.2 50mL disposable autotitrator sample cups (Man-Tech Catalog #PC-1000-429/1000; $163.00 per case of 1000 as of 26Jan2006)

9.3 Deionized water swish bottle

9.4 Deionized water carboy for rinse water for the Man-Tech system

9.5 Waste carboy (of a size 50% larger than the rinse carboy capacity) inside a secondary containment bin.

10.0 REAGENTS

10.1 1,413µS/cm Meter Calibration Standard: Purchase a 1,413µS NIST traceable conductivity standard (Fisher Catalog #09-328-11). Alternatively, prepare by weighing out 0.7456g of KC1 (pre-dried at 105°C for 2 hours) and dissolve in about 900mL of Deionized water in a 1L volumetric that has been rinsed first with three, 20mL portions of denioinzed water. QS to 1 liter, stopper, and mix by inversion until all KCl is dissolved. Store at room temperature.

10.2 Laboratory Control Sample (LCS): Purchase a 1,409µS/cm NIST traceable standard (Lab Chem Catalog #LC18780-2). Store at room temperature.

10.3 pH 4, 7 and 10 Buffers: Obtain from consignment (via Fisher Scientific) color-coded buffer solutions for pH 4, pH 7 and pH 10.01.

10.4 pH LCS Buffer pH 6.00: Obtain from Lab Chem (Catalog #LC12330-4).

10.5 0.020 *N* HCl: Obtain NIST-traceable acid solution from Lab Chem (Catalog #LC15170-4).

10.6 Deionized water (fresh from the system; do not use water that has been standing).

11.0 PRESERVATION AND HOLD TIME

11.1 Collect samples in either glass or plastic bottles, and store samples at 4°C until analysis. Analyze as follows:

11.1.1 pH samples: immediately (within 24 hours).

11.1.2 Alkalinity samples: within 14 days of sample collection.

11.1.3 Conductivity samples: within 28 days of sample collection.

11.1.4 For samples with more than one parameter requested: go by the shortest holding time.

12.0 QUALITY CONTROL

12.1 Conductivity

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Method Blank	One per 20 samples or fraction thereof	<1.0µS/cm	Re-analyze; if still out, recalibrate.
Laboratory Control Sample (LCS)	One per 20 samples or fraction thereof	85-115%	Analyze a new aliquot of LCS; if still out check stock std solution
Sample Duplicate (DU)	One per 20 samples or fraction thereof	RPD ≤20%	Re-analyze; if precision criteria still not met determine if instrument calibration is correct.

12.2 pH

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per 20 samples or fraction thereof	85-115%	Analyze a new aliquot of LCS; if still out check stock std solution
Sample Duplicate (DU)	One per 20 samples or fraction thereof	RPD ≤20%	Re-analyze; if precision criteria still not met determine if instrument calibration is correct.

12.3 Alkalinity

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Method Blank	One per 20 samples or fraction thereof	<1.0µS/cm	Re-analyze; if still out, recalibrate.
Laboratory Control Sample (LCS)	One per 20 samples or fraction thereof	85-115%	Analyze a new aliquot of LCS; if still out check stock std solution
Sample Duplicate (DU)	One per 20 samples or fraction thereof	RPD ≤20%	Re-analyze; if precision criteria still not met determine if instrument calibration is correct.

13.0 CALIBRATION and STANDARDIZATION

13.1 See section 14.0 "Procedure"

14.0 PROCEDURE

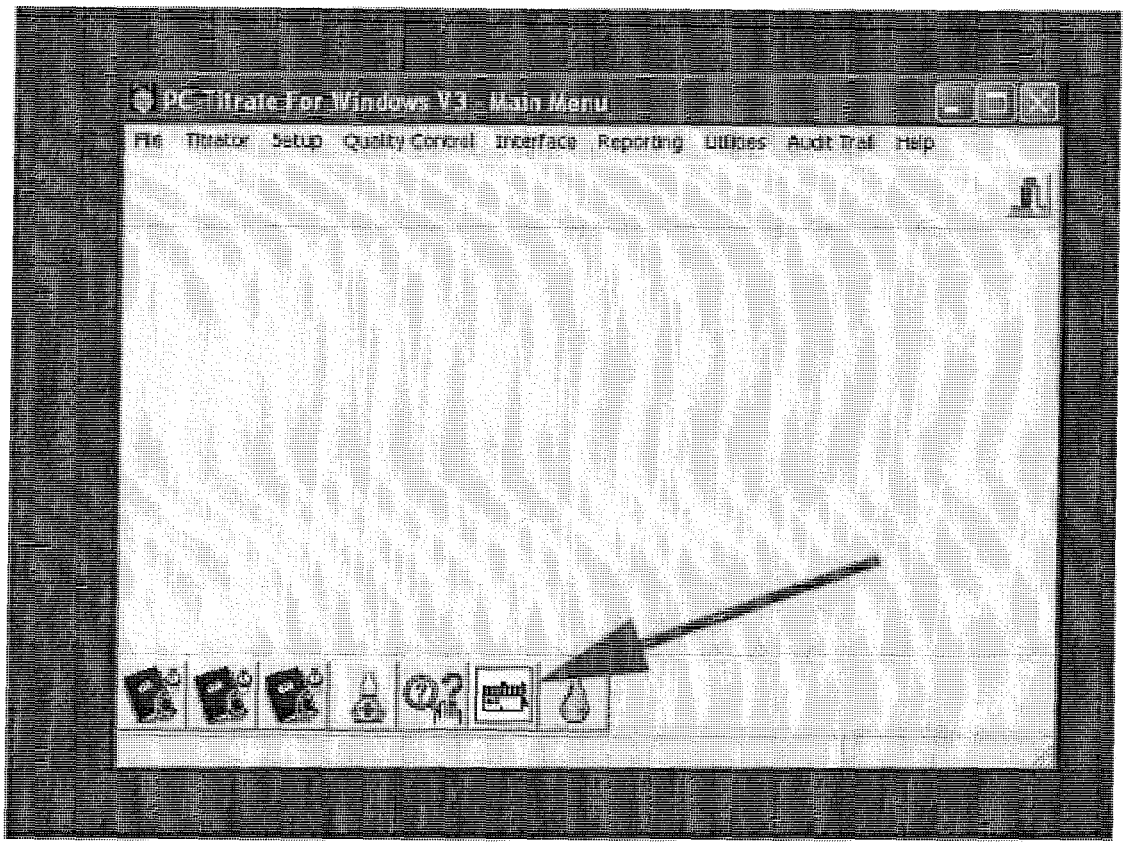
14.1 Analysis of samples consists of three main steps: building a batch using the PCTitrate software; servicing the instrument; and then loading samples and initiating the analytical run.

14.2 Building a Batch Using the Man-Tech PCTitrate Software

14.2.1 If the computer isn't already running, press the Start button. Once the Windows OS has finished initializing, use the mouse to double click on the PCTitrate icon on the Desktop.

14.2.2 The software will launch, showing a window similar to that in Figure 1.

FIGURE 1

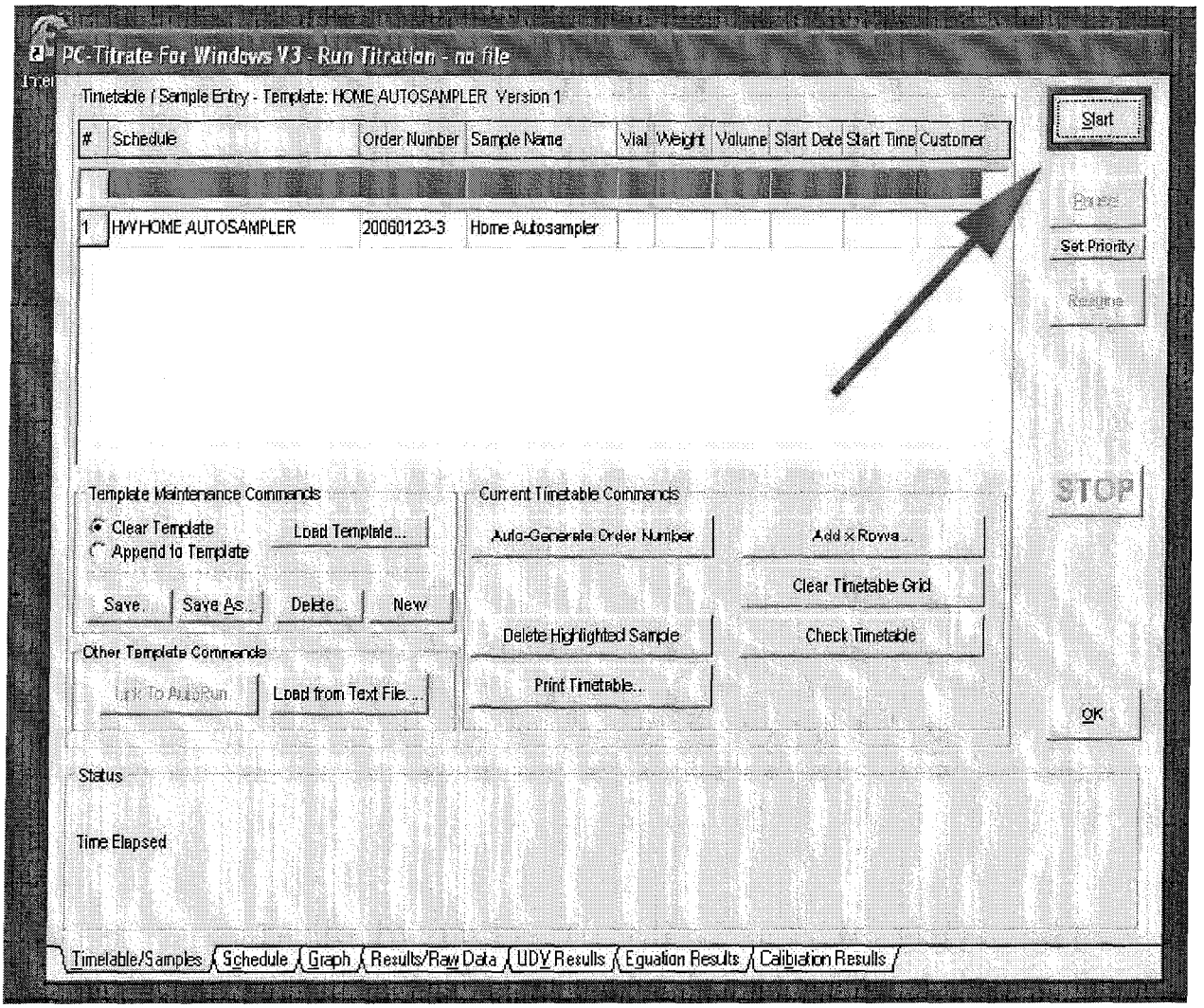


14.2.3 The software will automatically do a communications initialization between the computer and the instrument. You should hear some noises coming from the sampler arm and some electronic noises as it tests the pumps. Once that is done, click on the Autosampler Icon as indicated by the arrow in Figure 1.

14.2.4 This will bring up the Home Autosampler screen (Figure 2).

14.2.5 Click on the Start button as indicated by the arrow in Figure 2. This will initialize the autosampler and confirm the home position for it.

FIGURE 2

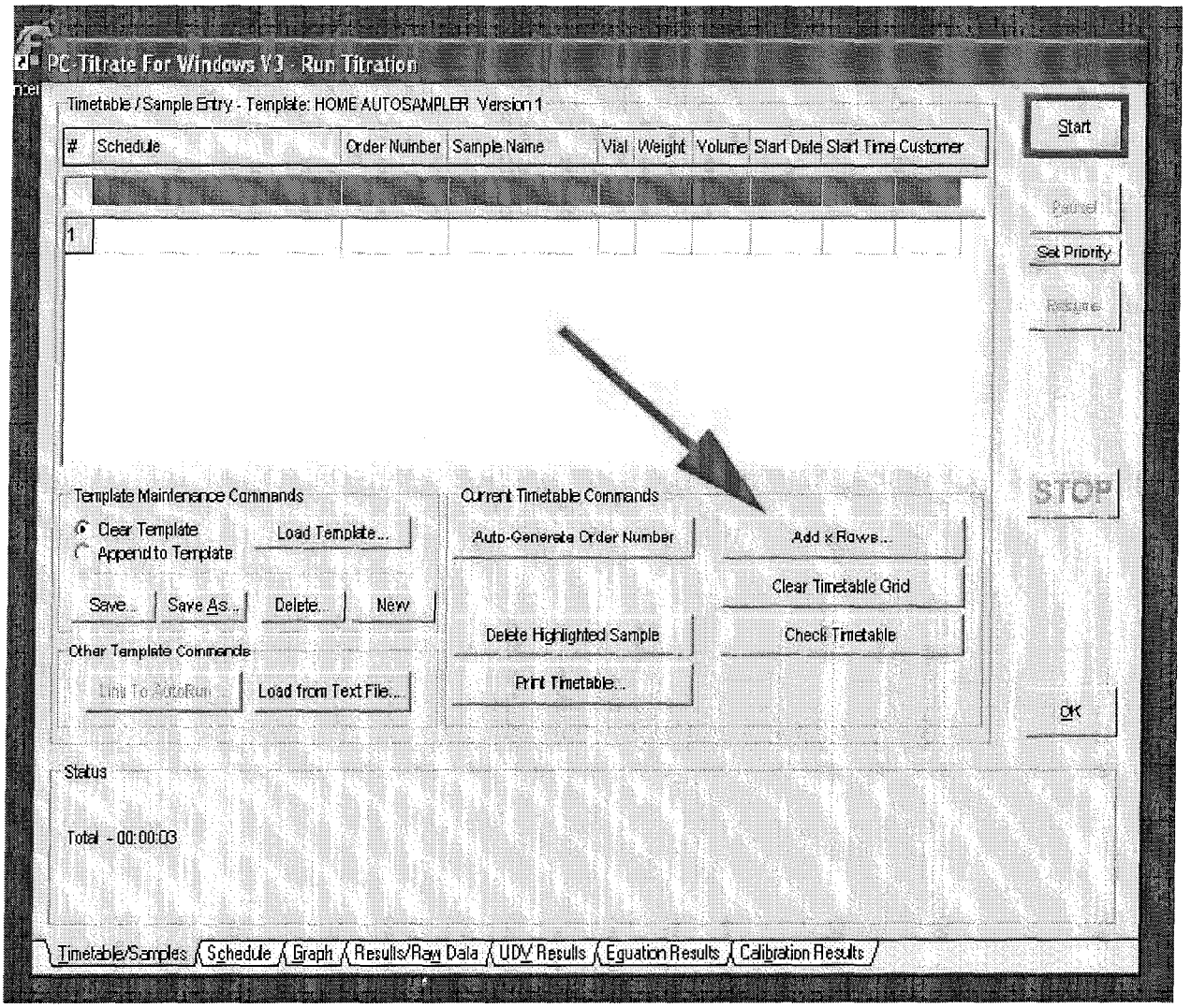


14.2.6 Once the initialization is complete for the Home Autosampler function, a new batch template (Figure 3) will be displayed.

14.2.7 Click on "Add x Rows" as shown by the arrow in Figure 3. Enter the number of quality control and investigative samples that you intend to analyze; if you don't know the exact number, estimate (you can always add or delete rows later as needed).

14.2.7.1 **NOTE!** The type and frequency of QC samples are listed in the table in Section 12.0. Be sure to include all required QC samples with your analytical batch. As of 1 April 2006, lab SOP is to no longer run an LCSD unless requested by the client; the standard QC parameters are MB, LCS, MS/MSD (or DU if the method isn't applicable for spikes).

FIGURE 3



14.2.8 Build an analytical batch in the software by first entering the names for the QC and investigative samples by the following hierarchy:

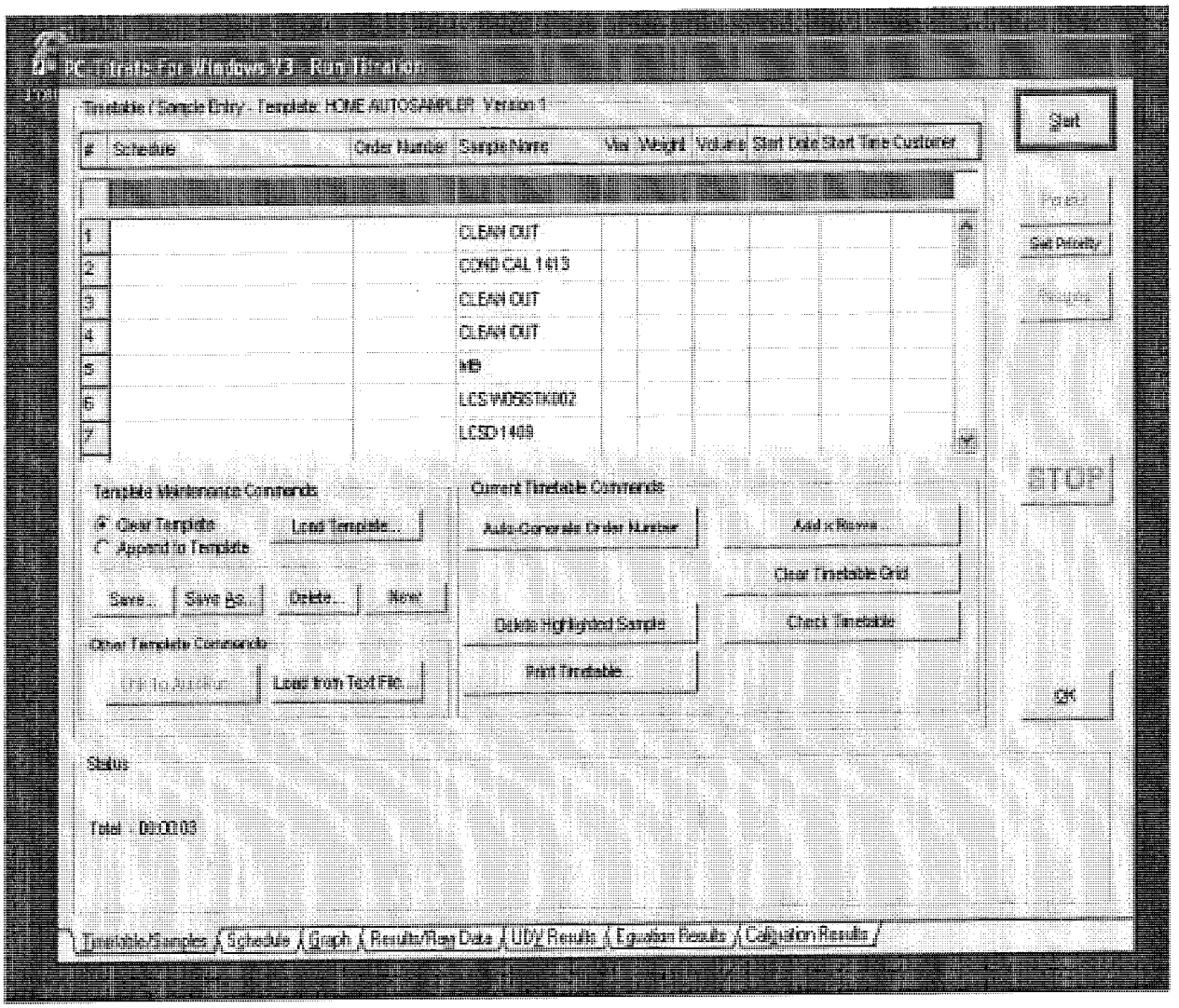
- 14.2.8.1 Conductivity samples
- 14.2.8.2 pH samples
- 14.2.8.3 Alkalinity samples

14.2.9 Thus if you plan to analyze samples that have a mix of all three parameters, you should first set up the calibration standard and QC for conductivity, then run the conductivity-only samples; then standardize the pH probe and run the LCS for pH; and finally, analyze the QC for alkalinity and run the remaining samples of all types. This order is important because the MB for conductivity and alkalinity may produce a slightly positive value if they are permitted to stand in the sample cup, exposed to the atmosphere, for too long a period of time.

14.2.10 Figure 4 shows the first lines of the analytical batch, where there is a “clean out” step to rinse off the probe, followed by the conductivity calibration standard, then two more “clean out” samples to rinse the

probe, followed by the Method Blank (MB) and the LCS samples. As of 5 April 2006, a duplicate of an investigative sample must be run in an analytical batch (replacing the LCSD). **IMPORTANT!** Use only fresh, right out of the tap deionized water for the MB. Note also that the LCS is from a different manufacturer or lot number as compared to the calibration standard.

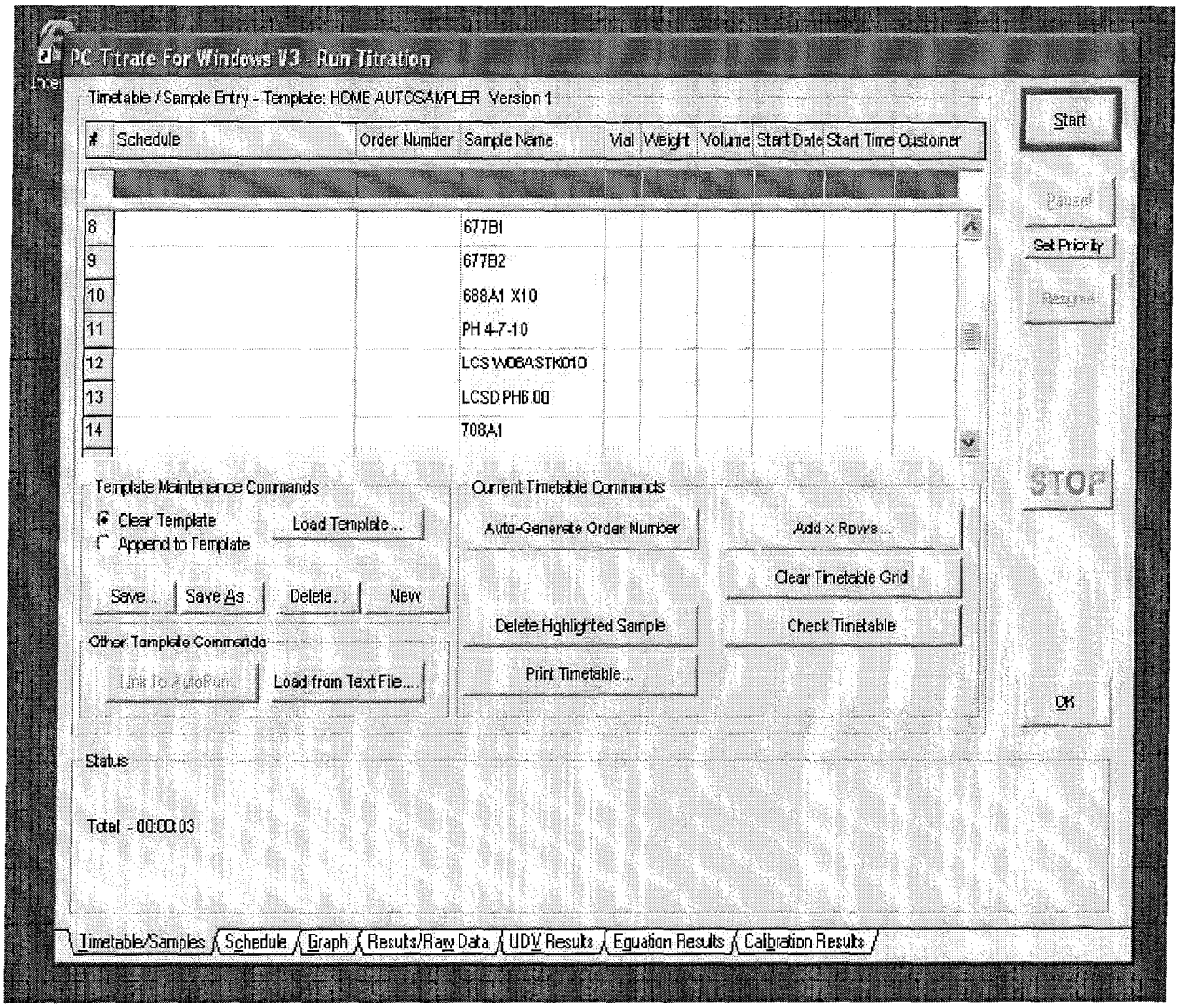
FIGURE 4



14.2.11 After the conductivity calibration and QC comes the investigative samples that need only conductivity analysis. (In this case, there are three of them, the last of which is a dilution at 10X. Refer to Figure 5.)

14.2.12 Next, set up the pH calibration, followed by the pH 6.00 LCS samples. Note that the pH calibration is listed only as one line, but actually sends the autosampler to three consecutive sample cup positions from which it draws, in order, the pH 4, 7 and 10 buffers. (Refer to Figure 5)

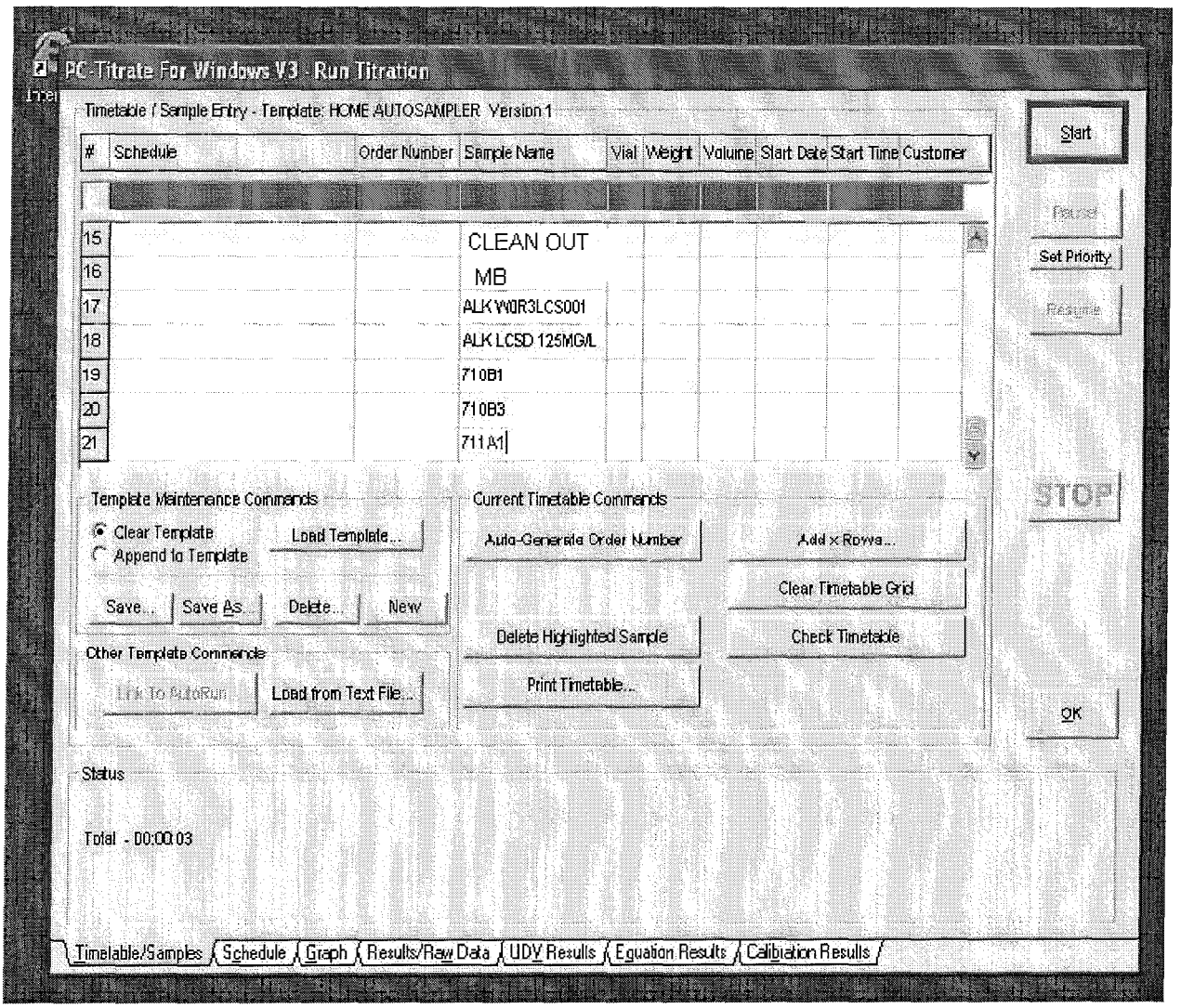
FIGURE 5



14.2.13 After the LCS, the investigative samples are listed. There should be one duplicate (DU) investigative sample per analytical batch. **NOTE!** You must do a pH calibration daily, but once it's done, the calibration is good for the day. **ALSO NOTE!** You must do a pH calibration before running alkalinity samples; therefore, if you plan to only run alkalinity on the instrument, be sure to first do a pH calibration.

14.2.14 Alkalinity samples or samples with a mix of pH, conductivity and alkalinity analysis come hereafter, starting with a "clean out" step to rinse the pH mixing chamber in advance of analyzing the alkalinity Method Blank (MB). **IMPORTANT!** Use only fresh, right out of the tap deionized water for the MB. (Refer to Figure 6.)

FIGURE 6

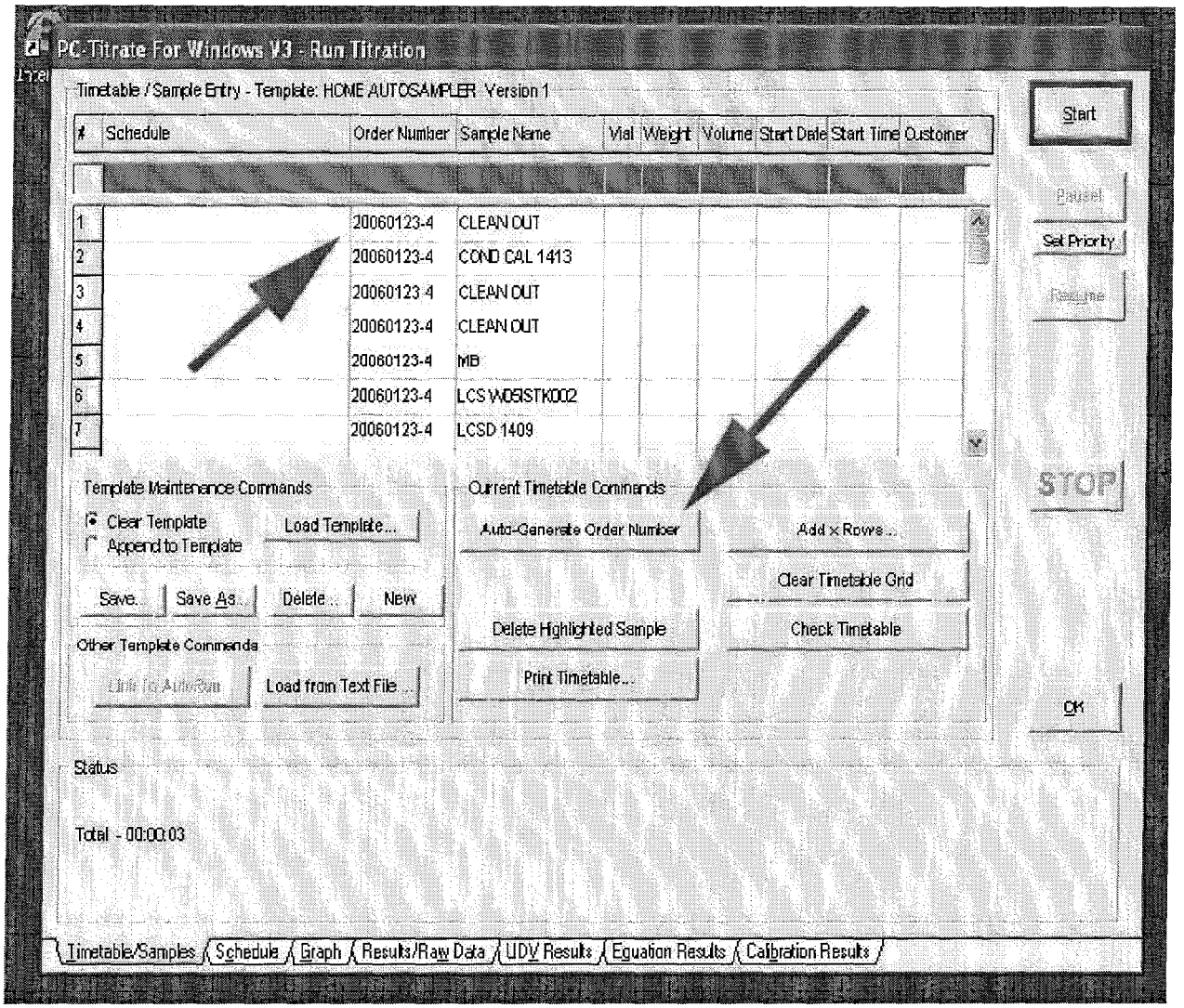


14.2.15 Once the batch has been completely built, scroll down within the software window to determine if any open lines still remain. If so, click in the "Sample Name" field for the blank line, and click the Delete Highlighted Sample button. Repeat as necessary to remove all unused lines at the end of the batch.

14.2.16 If you need to add more lines, click on the "Add x Rows" button, and enter the number of additional rows you need. Rows are <u>always</u> added at the end of the batch.

14.2.17 Next create the Order Number for the batch. The Order Number is a tracking number that the software uses, and is unique. Click in the Order Number field for the very first line of your batch. See Figure 7.

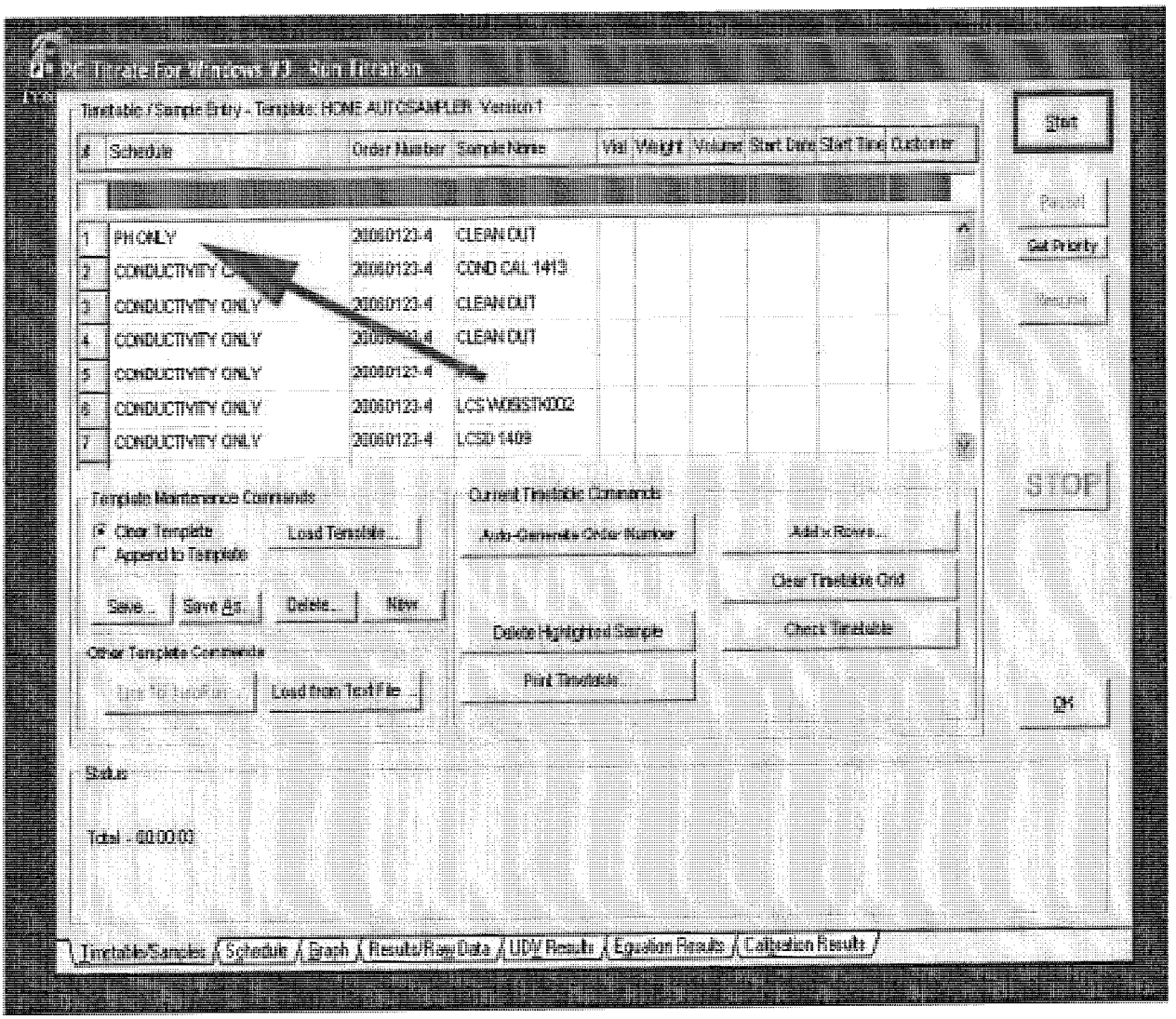
FIGURE 7



14.2.18 Once the Order Number is in the field of the first line, click with the mouse in the blank Order Number field directly under it. Next, hold the Shift key down; while holding the Shift key down, click on the Order Number just generated and hold the left mouse button down. Drag the cursor down the length of the column to the last open Order Number field on the last line, and release. The Order Number will automatically fill in to all open fields by this method.

14.2.19 Next, schedule the analysis commands for the instrument by scrolling back up to the first line, and double clicking with the mouse cursor in the first line's open Schedule field. A pop-up menu appears. Since our first clean out is designed to both clean the pH mixing chamber, the feeding line to it, and the conductivity probe, it's set as a PH ONLY command (though we won't be using the data generated for any purpose). Refer to Figure 8.

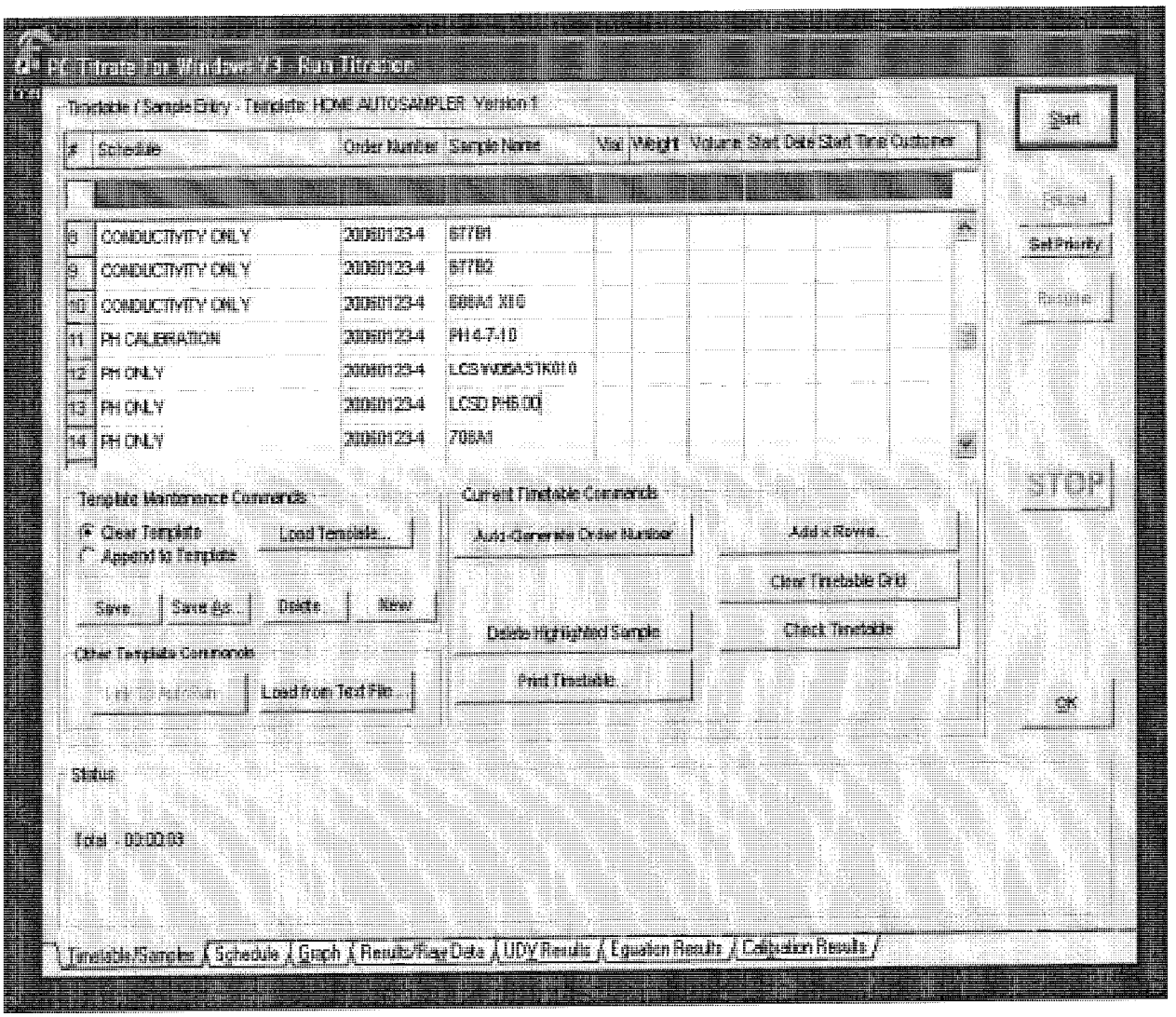
FIGURE 8



14.2.20 For the next line, double click and select CONDUCTIVITY CAL so that the instrument will know to calibrate to this sample cup. Thereafter, for each QC and investigative sample to be analyzed only for conductivity, select CONDUCTIVITY ONLY. (You can use the same click and drag method with the Shift key as mentioned in Step 14.3.18 for filling in multiple lines.)

14.2.21 For the calibration of the pH meter, double click in the Schedule field for that line and select PH CALIBRATION as shown in Figure 9.

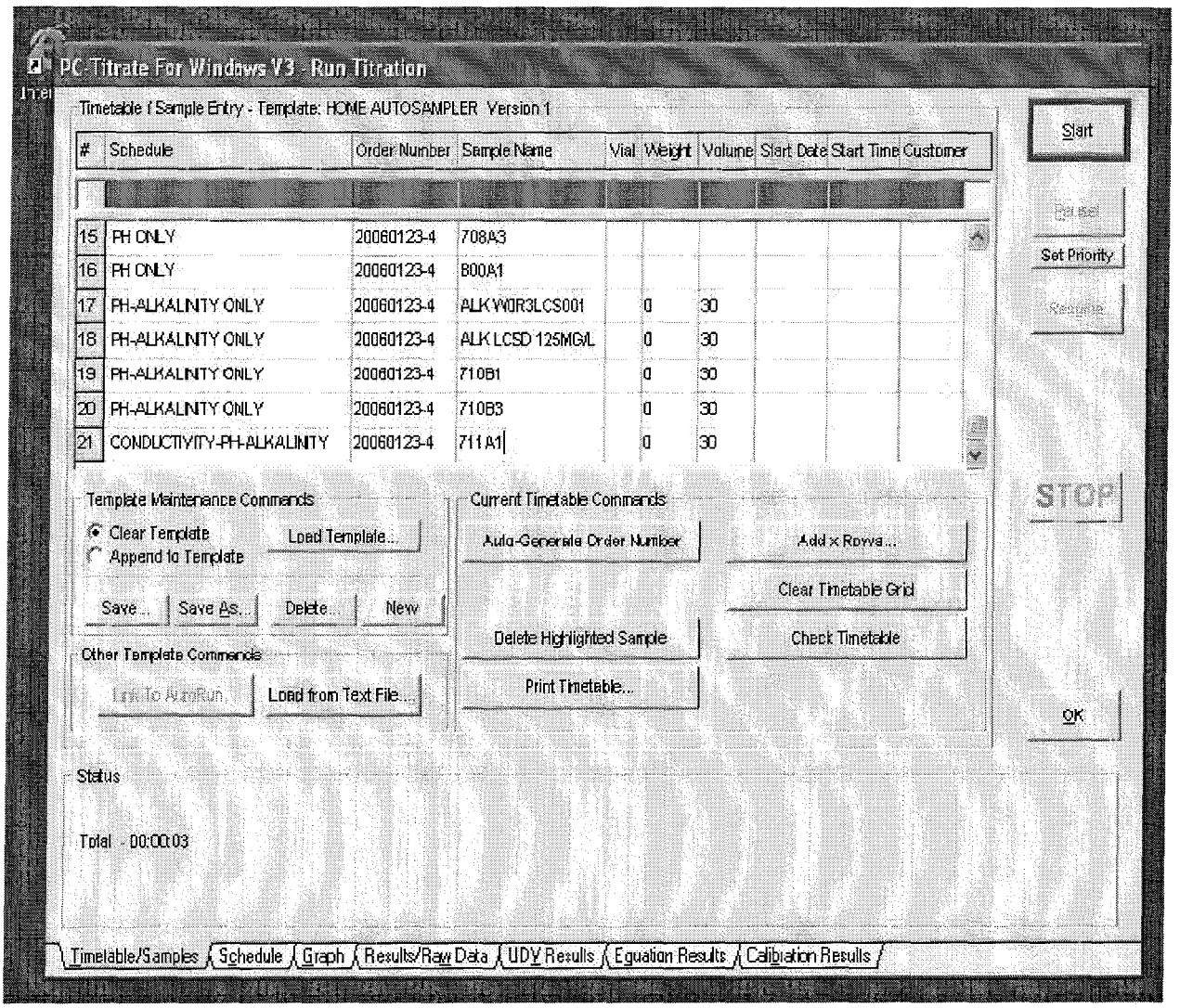
FIGURE 9



14.2.22 The QC and investigative samples needing only pH can then have PH ONLY selected for them.

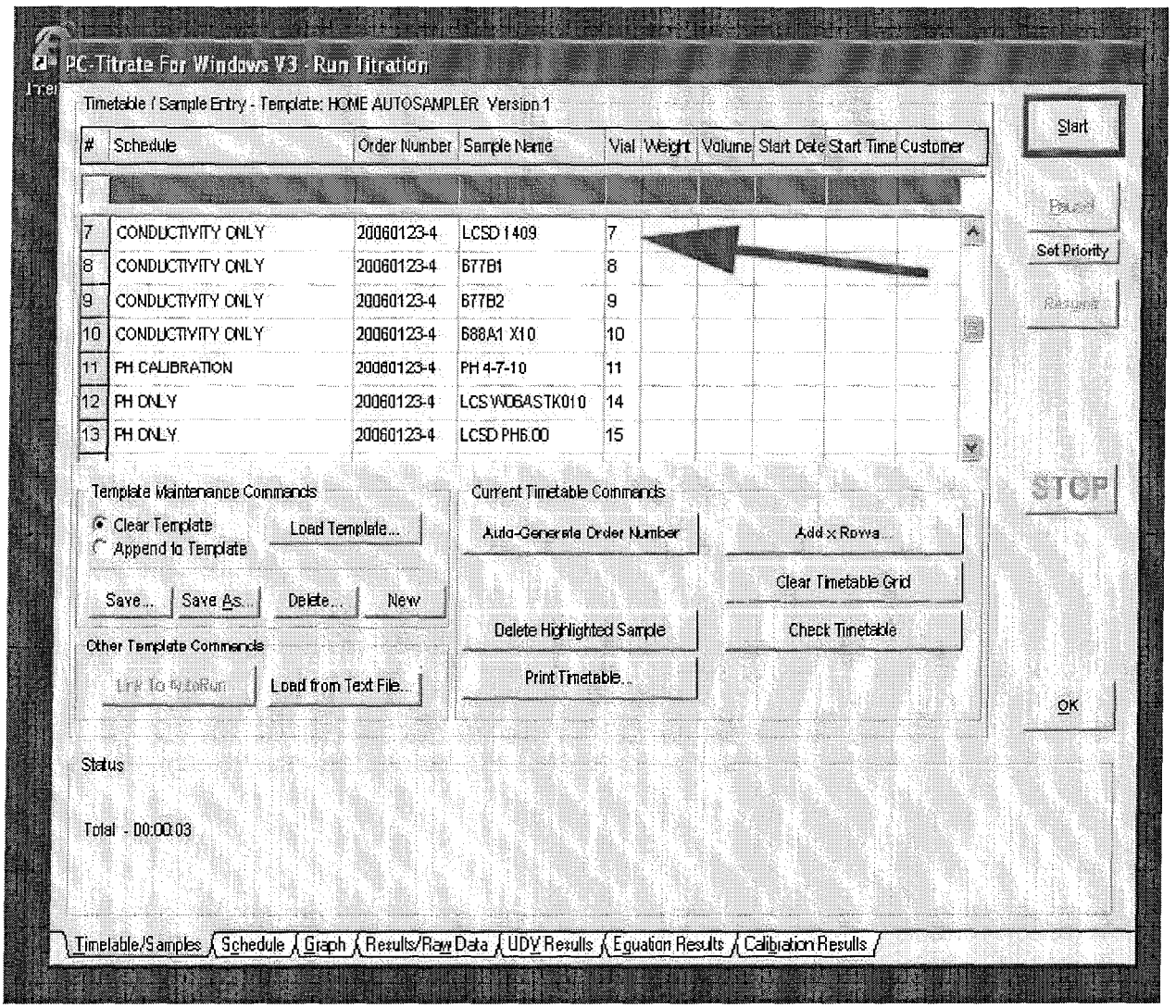
14.2.23 Finally, the "clean out," Method Blank (MB), LCS, and samples for alkalinity (and/or a mix of pH, alkalinity, and conductivity) are listed. Don't forget to run duplicates (DU) as required. (Refer to Figure 10.) **IMPORTANT NOTE!** An alkalinity-only analysis option isn't available: samples needing only alkalinity are run as PH-ALKALINITY ONLY.

FIGURE 10



14.2.24 Now that the sample names and types are listed and the samples have a test scheduled for each, you need to enter the sample cup position numbers for the batch. Scroll up to the first line of the batch, and click in the Vial field. Start with Vial position "1" and using the down-arrow key, move to the subsequent fields, entering numbers sequentially. (Refer to Figure 11.)

FIGURE 11



14.2.25 As aforementioned **NOTE THAT THE PH CALIBRATION USES ONE LINE BUT ADDRESSES THREE CUP POSITIONS.** Therefore, as shown in Figure 11, the PH 4-7-10 calibration starts in cup 11 and ends in cup 13; thus the next available cup for the LCS is cup 14.

14.3 Servicing the Instrument Before Initiating an Analytical Run

14.3.1 Before analyzing samples, perform the following maintenance operations:

14.3.1.1 Check the deionized water carboy on the floor under the instrument. If it is less than half-full, refill with fresh deionized water. (A full carboy has enough rinse water for a 55 cup run.)

14.3.1.2 Empty the wastewater carboy.

14.3.1.3 Check the pH mixing chamber atop the instrument. The pH probe should not have any precipitated crystals of material internally (from the filling solution), and the probe should be clean and free from oil or other residue.

14.3.1.3.1 If the probe has crystalline precipitate inside, remove the probe from the mixing chamber. Rinse and change out the electrolytic solution with the correct filling solution. Re-insert the probe so that the tip is one-quarter inch from the bottom of the mixing chamber.

14.3.1.3.2 If the probe has oily or other residue, remove the probe from the mixing chamber and clean it gently with a Kimwipe dampened with methanol. Repeat as necessary to clean the probe, and then re-insert the probe so that the tip is one-quarter inch from the bottom of the mixing chamber.

14.3.1.4 Check the tubing for the pumps. If any appears frayed, damaged or worn, replace it before the start of the run.

14.4 Loading Samples and Initiating the Analytical Run

14.4.1 Using the PCTitrate software template you created in Section 14.2, use disposable 50mL sample cups to place your standards, QC and investigative samples into the autosampler racks. The racks have been numbered in black marker by hand to indicate the positions. Rack numbers increment by unit value starting next to the glass rinse well and moving from back to front, left to right.

14.4.2 Fill each cup to the 45mL line. Place them in the appropriate locations in the racks. (For conductivity-only samples, filling to the 30mL line is sufficient.)

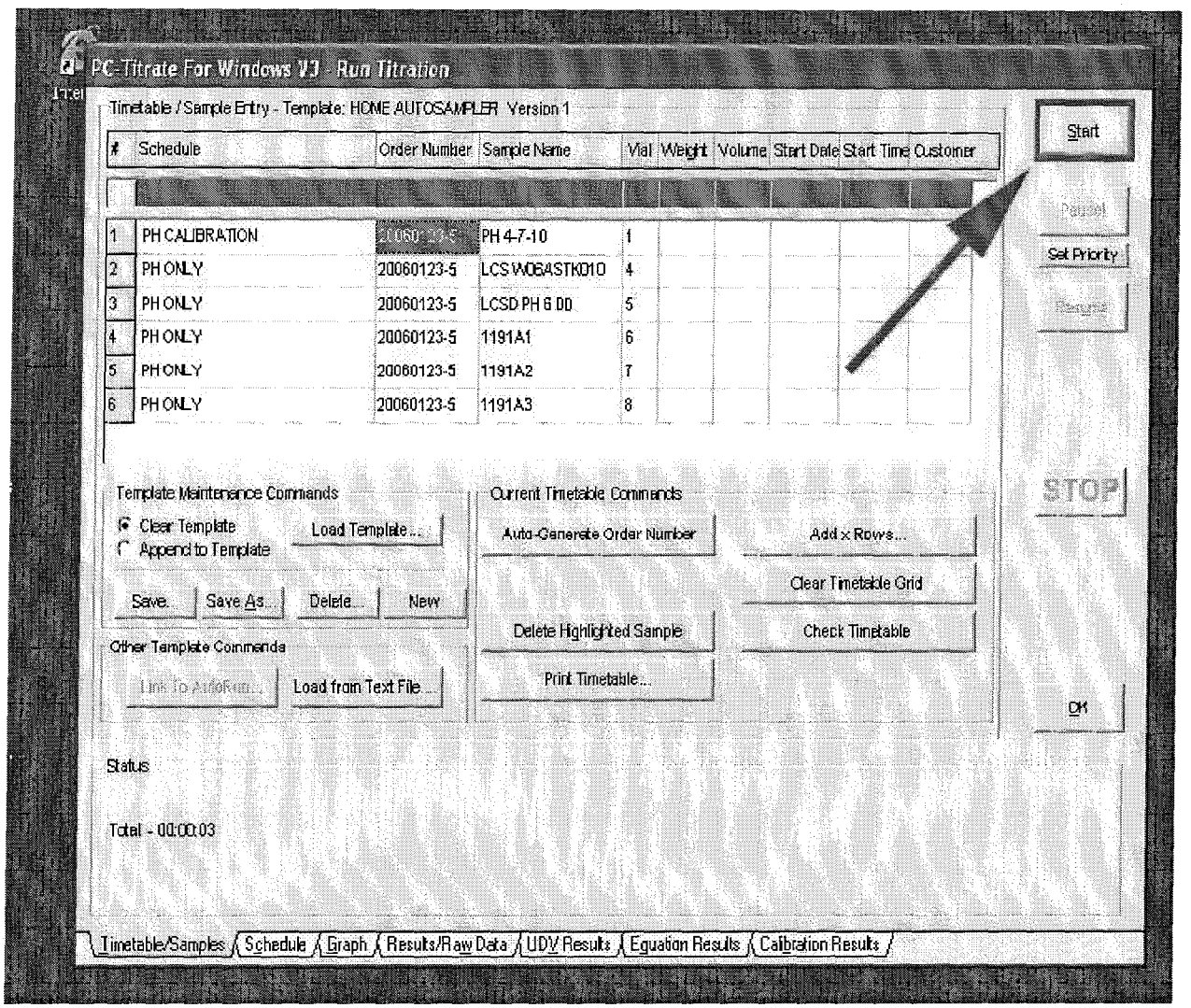
14.4.3 The instrument stores the pH probe in a pH 4 buffer and water mix once the analytical run is complete. Therefore, place a sample cup with 40mL of pH 4 buffer in the position in the racks <u>after the last scheduled sample.</u> DO NOT enter the pH 4 storage buffer cup as part of the analytical batch. The autosampler automatically goes to the next cup after completing the batch, expecting it to be the pH 4 buffer solution.

14.4.4 Once all samples are placed in the racks on the autosampler, return to the computer and click on the Start button (refer to Figure 12). The analytical run will begin.

14.4.5 **IMPORTANT NOTE!** The system uses a third-party conductivity meter for analysis of conductivity. Upon insertion into the conductivity calibration standard, and stabilization of the value, a window will appear on the screen to alert you to manually press the "CAL" button on the conductivity meter. It is suggested that you wait at the instrument for this step.

14.4.6 Once the message appears, press the "CAL" button on the conductivity meter, then use the mouse cursor to click on the "OK" button on the message. The instrument will now analyze all samples according to the schedule without further need for analyst monitoring.

FIGURE 12

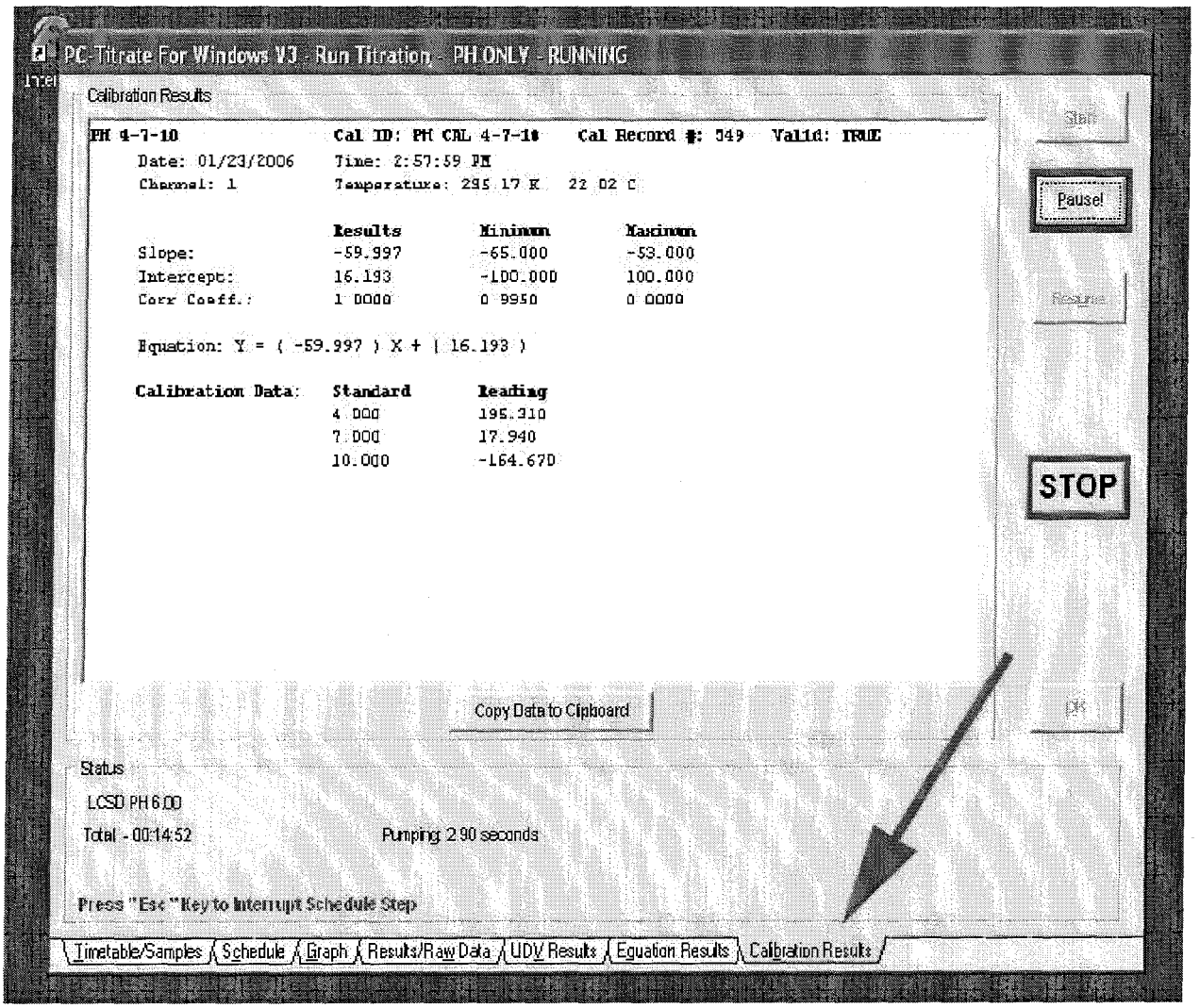


14.5 Monitoring Data in Real Time

14.5.1 At any time you can check the data being produced by the instrument by clicking on the Calibration Results or Equation Results tabs at the bottom of the screen.

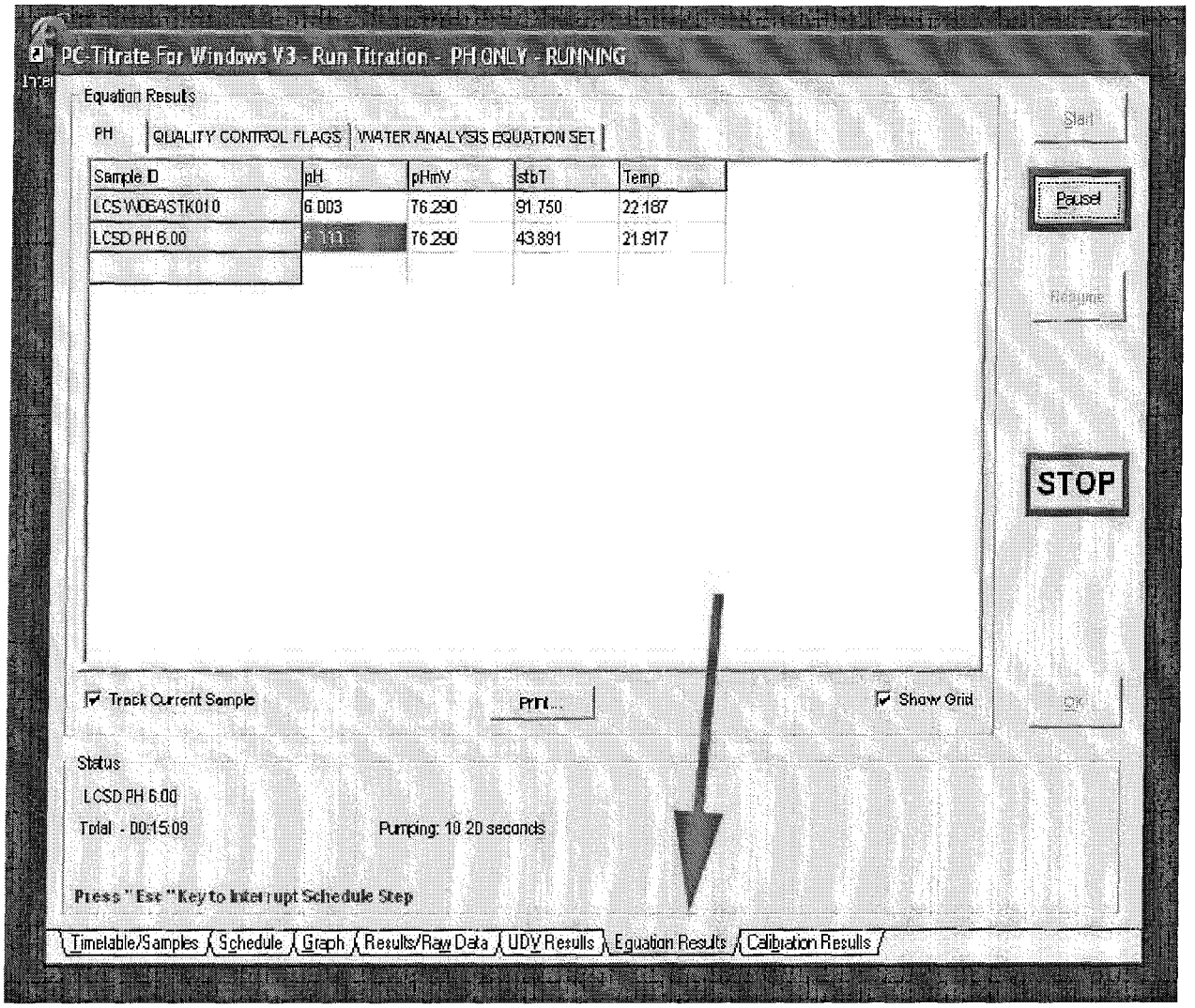
14.5.2 The Calibration Results tab will show the calibration for the pH curve and the associated criteria for the slop and intercept (refer to Figure 13). If the slope and/or intercept don't meet criteria, the run will stop and an error message will appear. Otherwise, the run will proceed automatically.

FIGURE 13



14.5.3 The Equation Results tab will show the calculated values for pH, alkalinity (and its associated sub-components of bicarbonate alkalinity, carbonate alkalinity, and phenolphthalein alkalinity), and conductivity as the data is produced for each sample. (Refer to Figure 14.)

FIGURE 14



14.5.4 The default is to show only pH data. Clicking on the WATER ANALYSIS EQUATION SET tab at the top (Figure 15) will show all the data. Note that some of the data may be negative or blank if you didn't schedule the particular sample for all of the analytes (Figure 16).

FIGURE 15

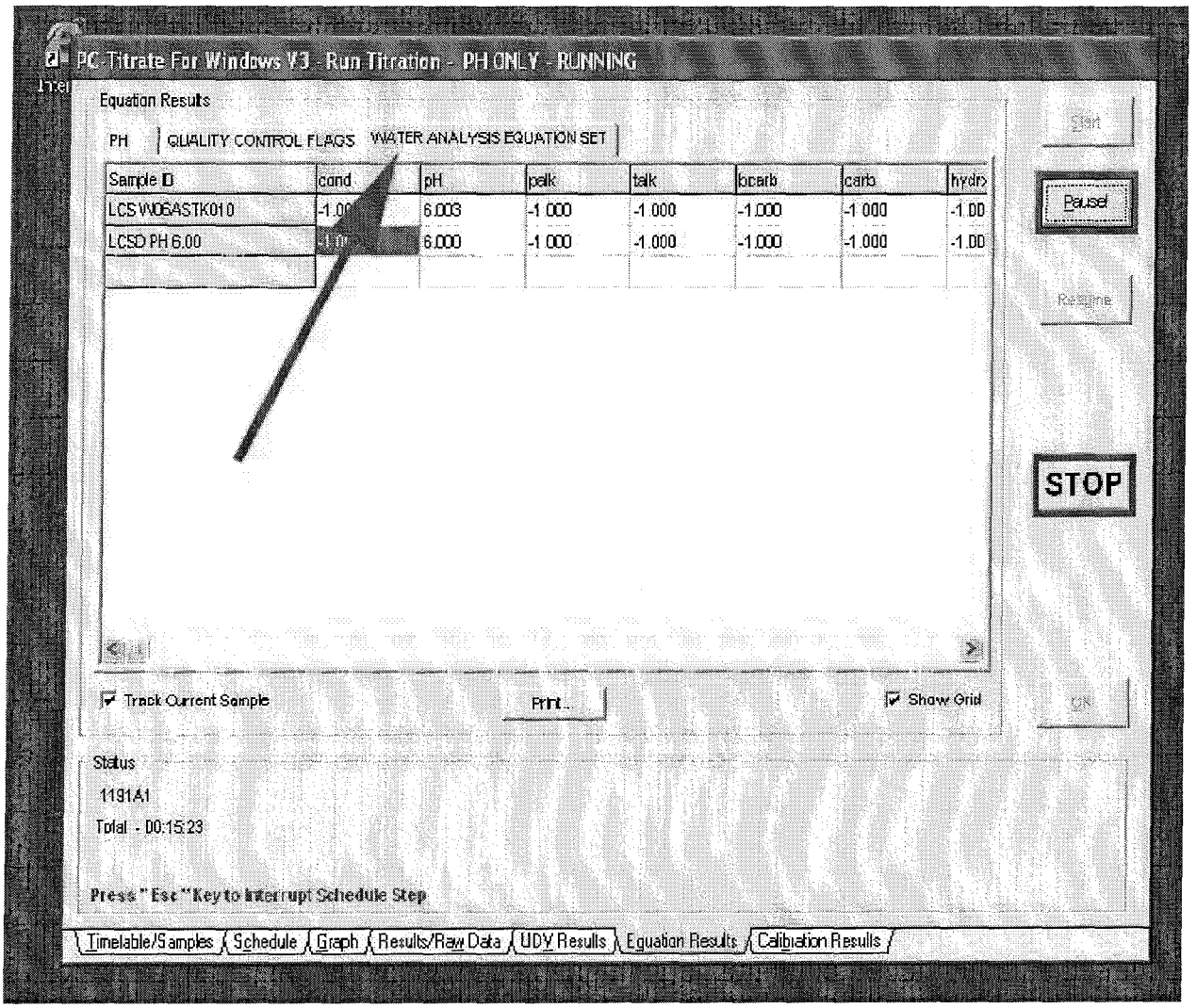
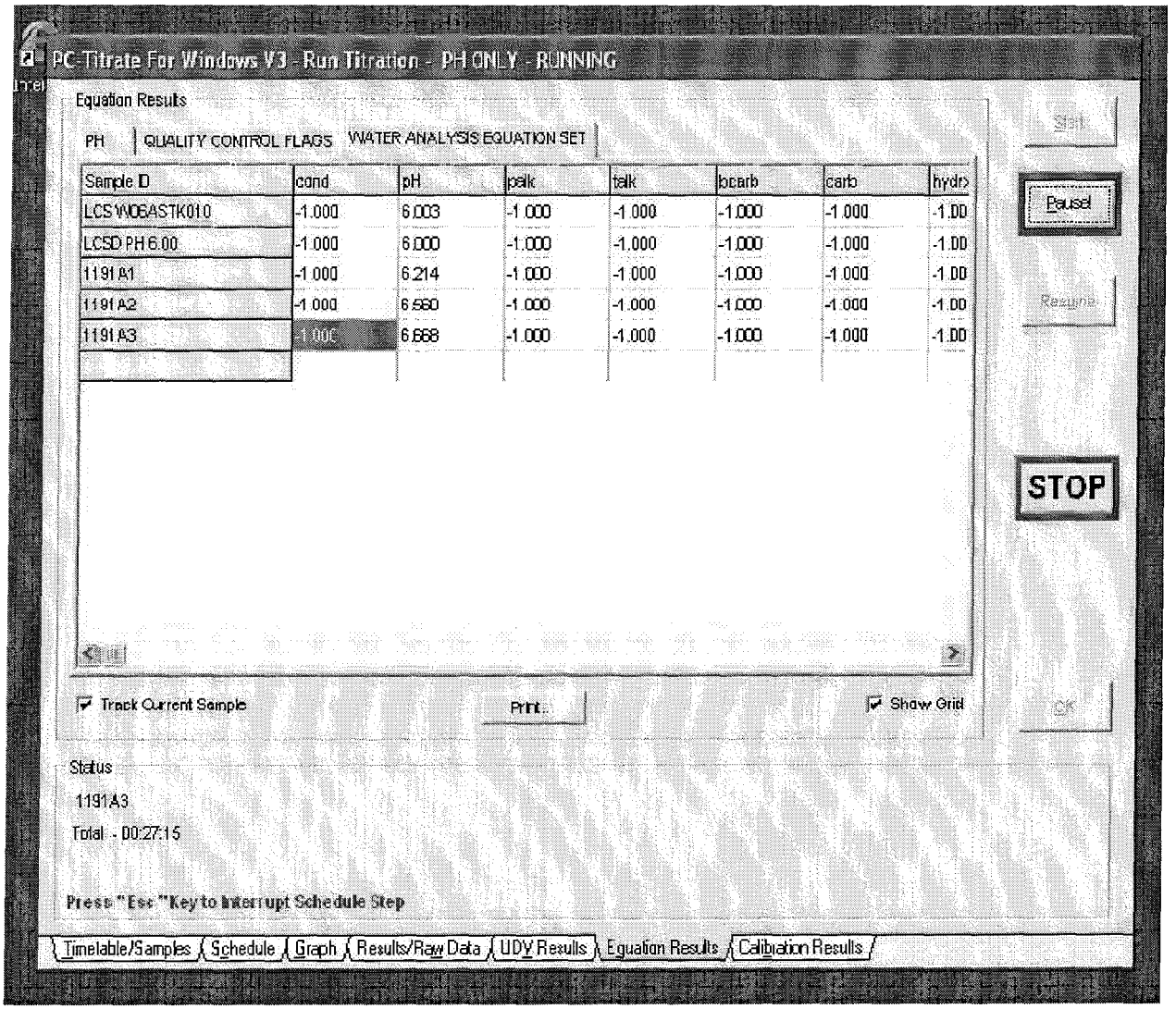

PC Titrate For Windows V3 - Run Titration - PH ONLY - RUNNING
Equation Results
PH
QUALITY CONTROL FLAGS
WATER ANALYSIS EQUATION SET
Sample ID
cond
pH
palk
talk
bcarb
carb
hydr
LCS W06ASTK010
6.003
-1.000
-1.000
-1.000
-1.000
-1.00
LCSD PH 6.00
6.000
-1.000
-1.000
-1.000
-1.000
-1.00
Start
Pause!
Resume
STOP
Track Current Sample
Print...
Show Grid
OK
Status
1191A1
Total - 00:15:23
Press "Esc" Key to Interrupt Schedule Step
Timetable/Samples
Schedule
Graph
Results/Raw Data
UDV Results
Equation Results
Calibration Results

FIGURE 16



14.5.5 Scrolling right will reveal additional data that may not be visible because of the limits to the size of the window.

14.6 How to Add Samples After Starting an Analytical Run

14.6.1 Click on the Pause! button (outlined in red) as shown in Figure 17. The instrument will complete the current sample being analyzed, then will stop and the Pause! button will become a Resume button outlined in green.

14.6.2 If you are not already in the batch table (Figure12), then click on the "Timetable/Samples" tab at the bottom of the software window to return to it.

14.6.3 Click on "Add x Rows" as shown in Figure 18.

FIGURE 17

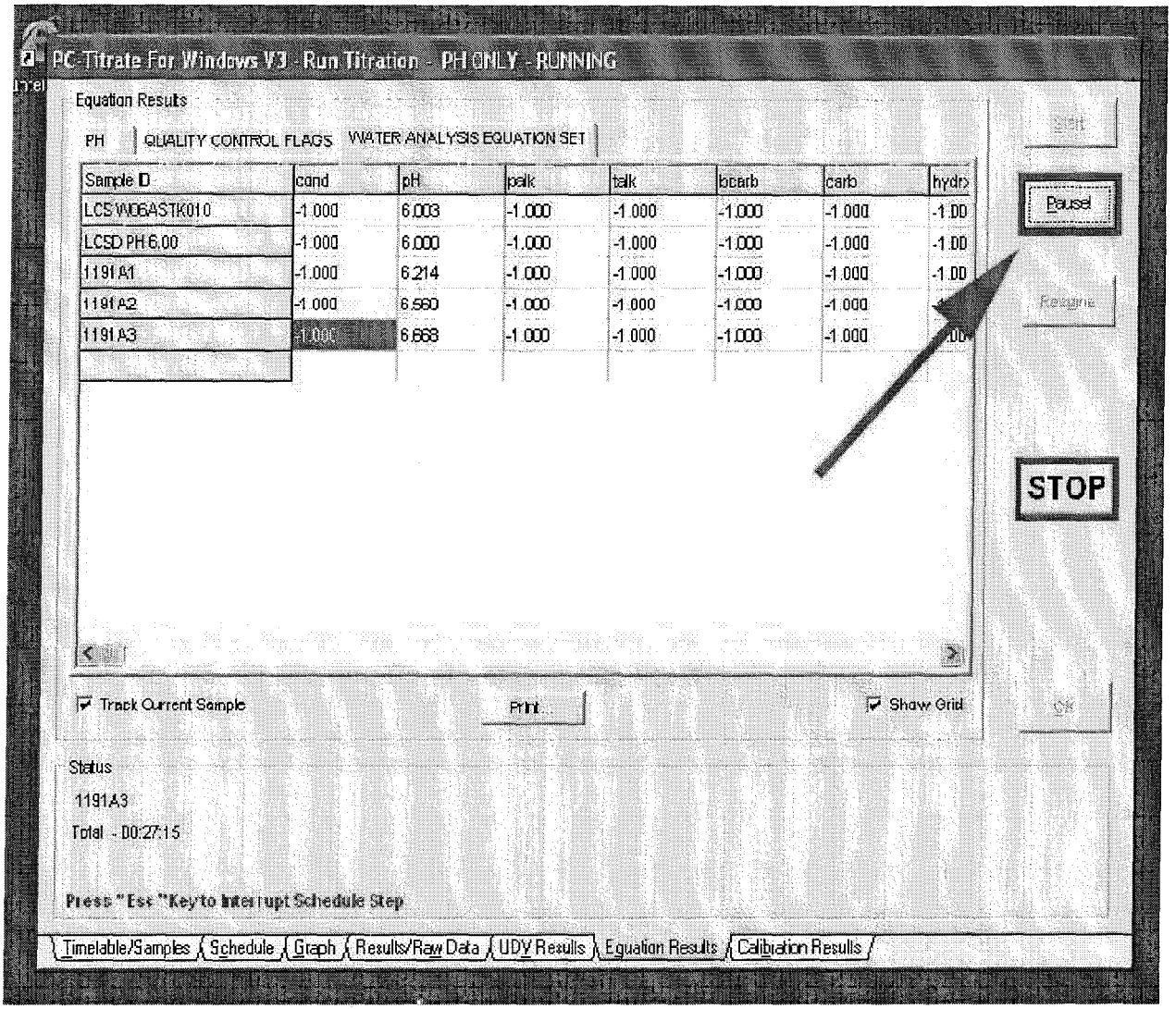
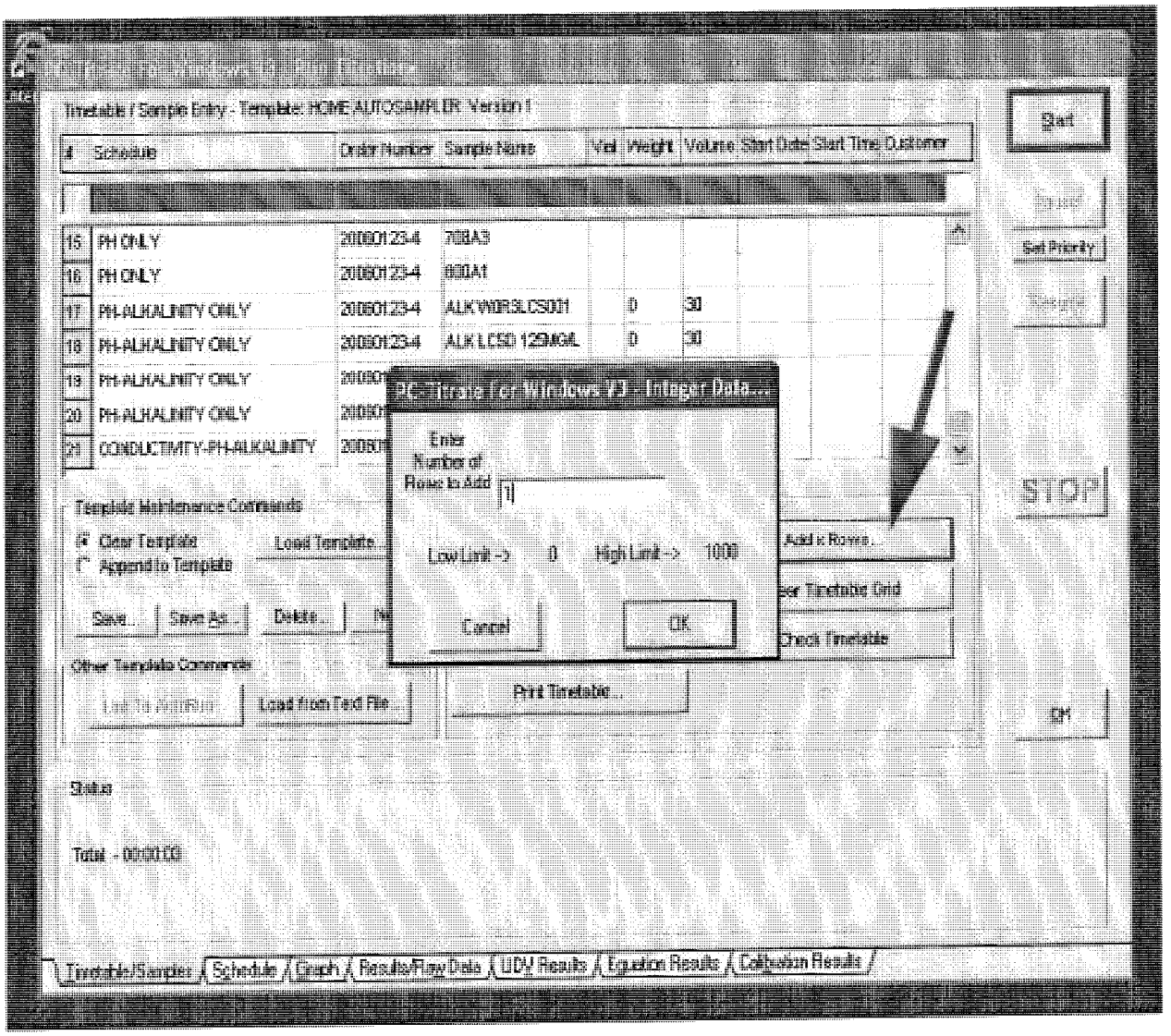
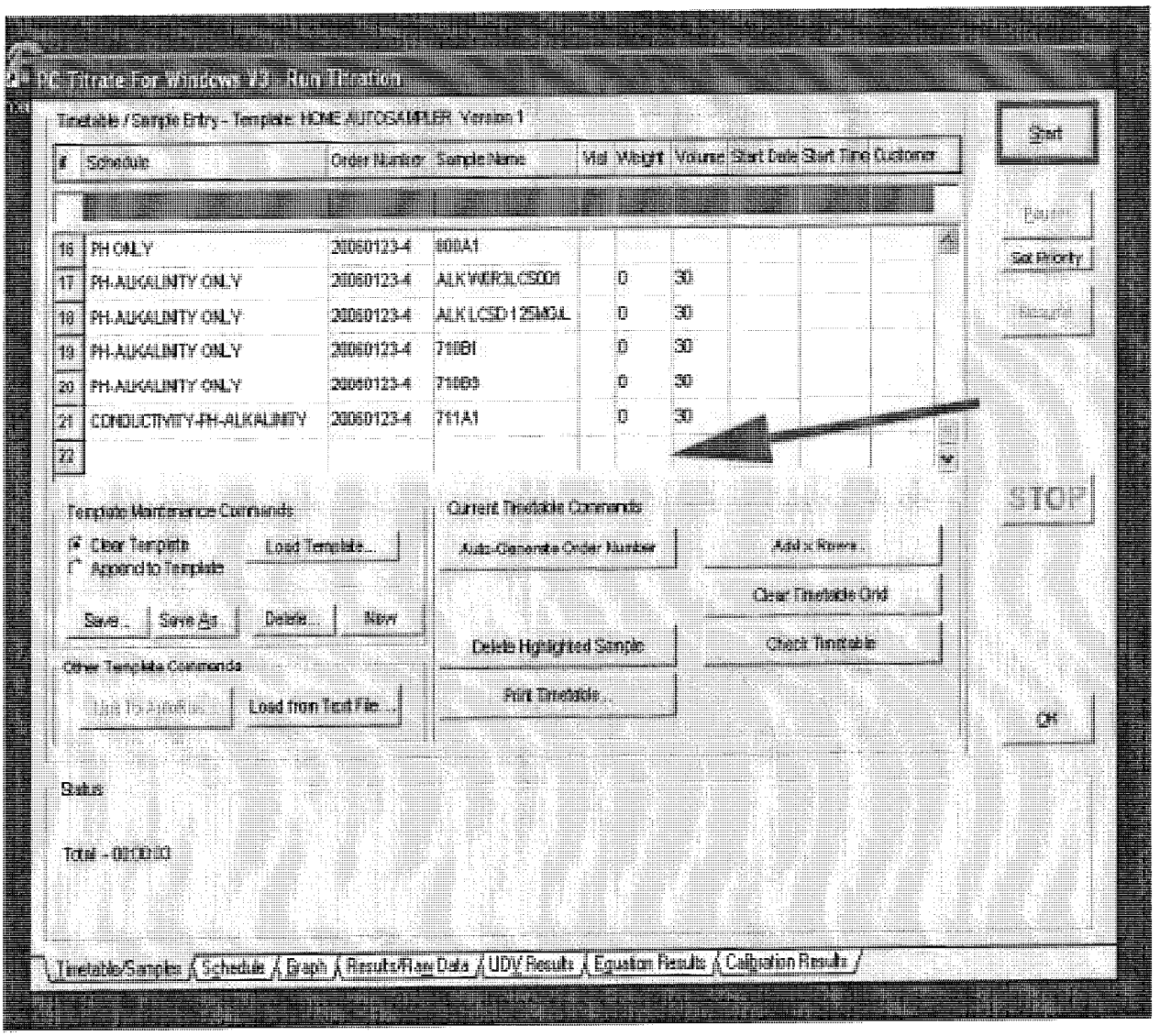

PC Titrate For Windows V3 - Run Titration - PH ONLY - RUNNING
Equation Results
PH
QUALITY CONTROL FLAGS
WATER ANALYSIS EQUATION SET
Sample ID
cond
pH
palk
talk
bcarb
carb
hydr
LCS W06ASTK010
-1.000
6.003
-1.000
-1.000
-1.000
-1.000
-1.00
LCSD PH 6.00
-1.000
6.000
-1.000
-1.000
-1.000
-1.000
-1.00
1191A1
-1.000
6.214
-1.000
-1.000
-1.000
-1.000
-1.00
1191A2
-1.000
6.560
-1.000
-1.000
-1.000
-1.000
1191A3
-1.000
6.668
-1.000
-1.000
-1.000
-1.000
Pause
STOP
Track Current Sample
Print...
Show Grid
Status
1191A3
Total - 00:27:15
Press "Esc" Key to Interrupt Schedule Step
Timetable/Samples
Schedule
Graph
Results/Raw Data
UDV Results
Equation Results
Calibration Results

FIGURE 18



14.6.4 A new window will appear asking for the number of new rows (samples) to add. Enter the number and click OK. Additional lines will appear as shown in Figure 19.

FIGURE 19



14.6.5 As you did for building the batch (Section 14.2), add the new samples, select the Schedule type for them, drag down the same Order Number to fill in the open fields, and enter the vial numbers for the new samples. Place sample cups in the racks containing the new, added samples, then click on the Resume! button. The Resume! button will change back to a red-outlined Pause! button, and the instrument will continue with analysis with the next vial in order.

14.7 Preparation of Soil Extracts for pH, Alkalinity and/or Conductivity Analysis

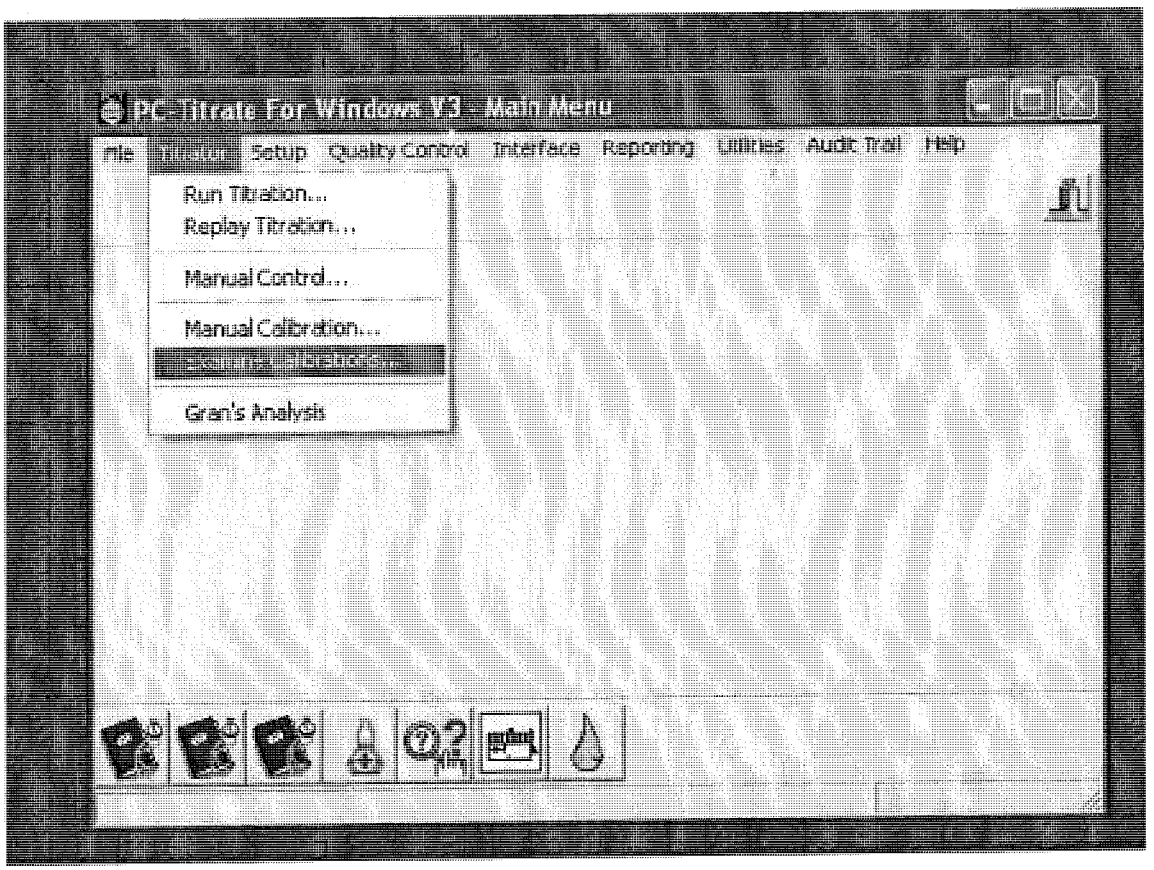
14.7.1 Prepare a Deionized Water Leachate (DI_LEACH) for a soil or solid sample needing analysis by placing 5g into a sample cup, adding 45mL of deionized water, affixing a screw cap finger-tight to the container, and then shaking the container for 1 minute. Allow the material to settle for 5 minutes, and then shake for 1 minute; repeat twice more.

14.7.2 Allow the material to settle to the bottom of the sample cup or container. Decant off the liquid, use a paint filter to filter the sample if large floatables or suspended particles are noted, and place into an autosampler cup. Place the sample into the rack on the autosampler and analyze as described in Section 14.2.

14.8 Reviewing Calibration for pH

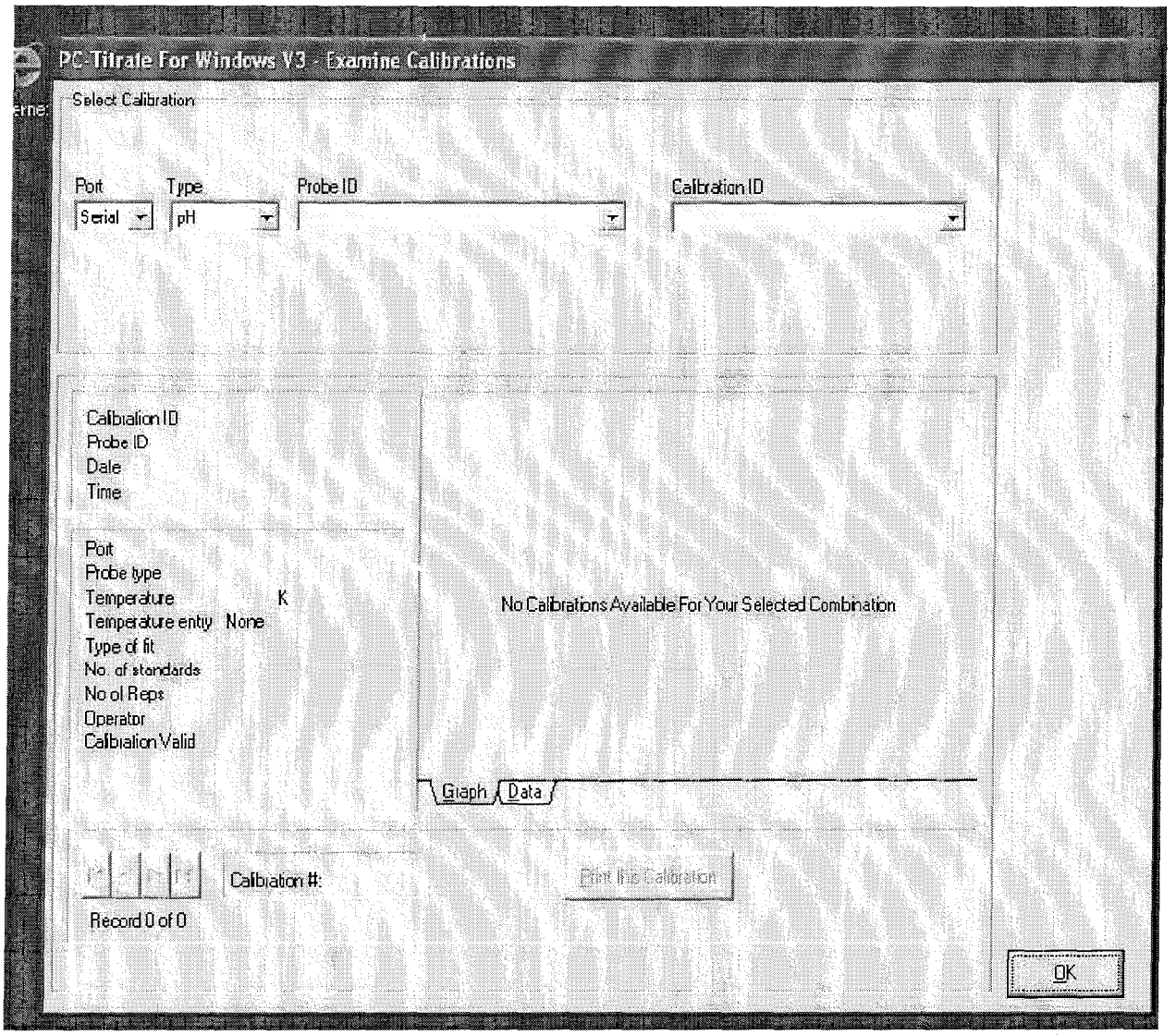
14.8.1 To review a calibration for any date, from the main PC Titrate menu, click on the "Titrator" menu item to display the drop down menu, then highlight and click on "Examine Calibrations" (See Figure 20).

FIGURE 20



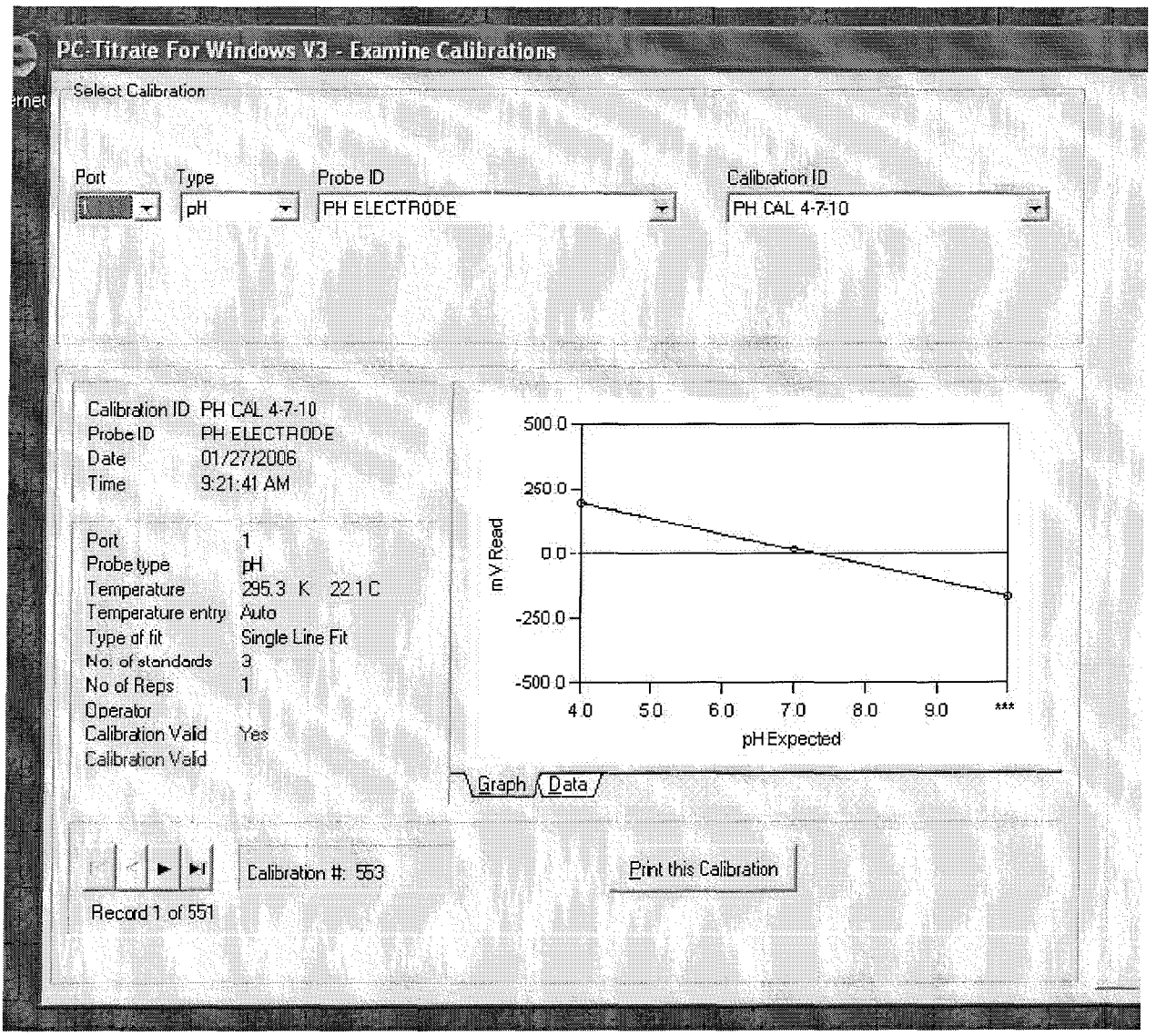
14.8.2 The Examine Calibrations screen will then be displayed (Figure 21).

FIGURE 21



14.8.3 In the drop-down menu for "Port" select "1" as shown in Figure 22 and the most recent calibration will be displayed.

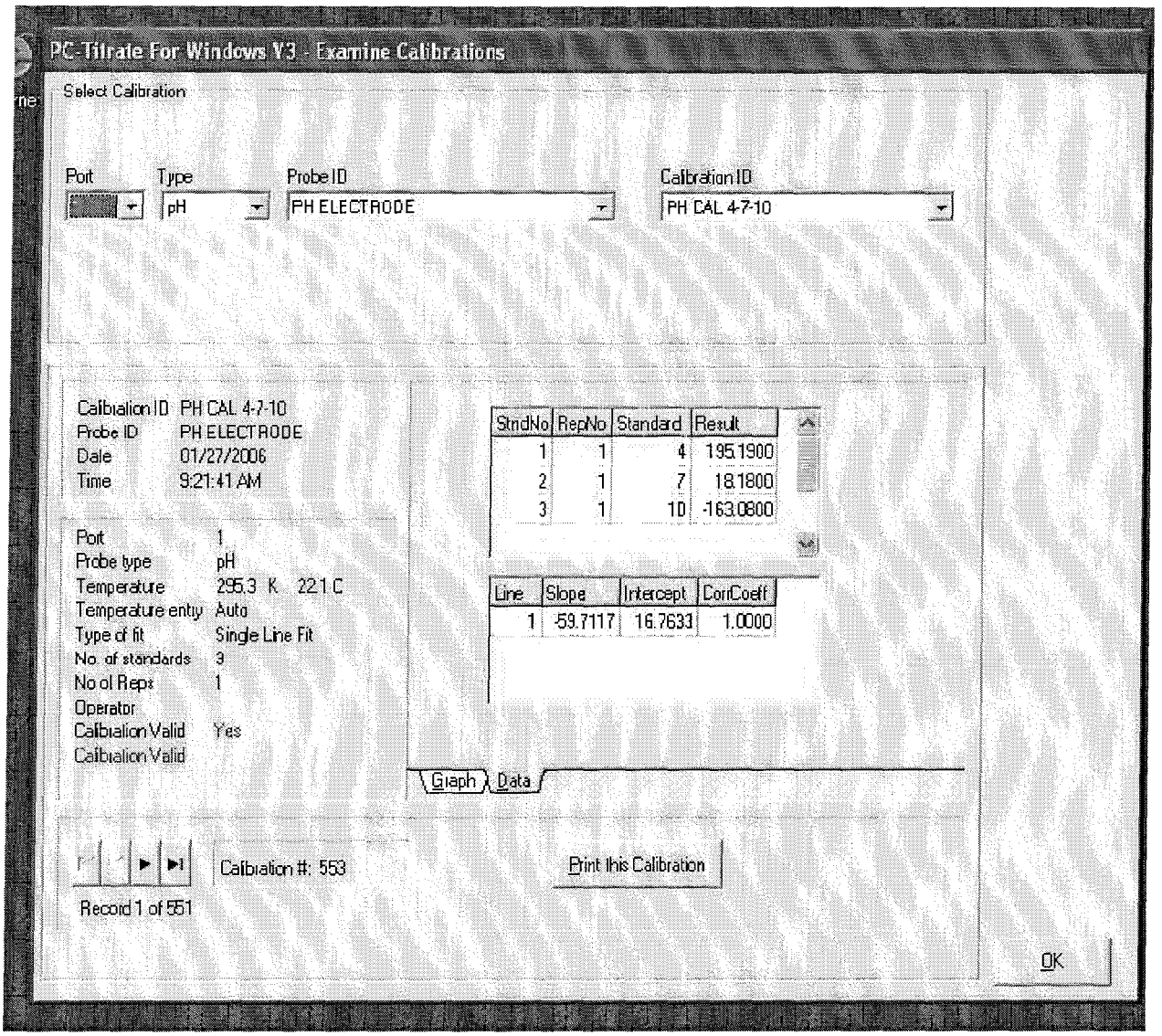
FIGURE 22



14.8.4 To review a calibration from an earlier date, click on the arrow button at the lower left of the screen. To print the calibration, click on "Print this Calibration" in the lower right.

14.8.5 Clicking on the "Data" tab below the calibration graph will display the criteria for the calibration curve and the x-intercept and slope for the calibration as shown in Figure 23.

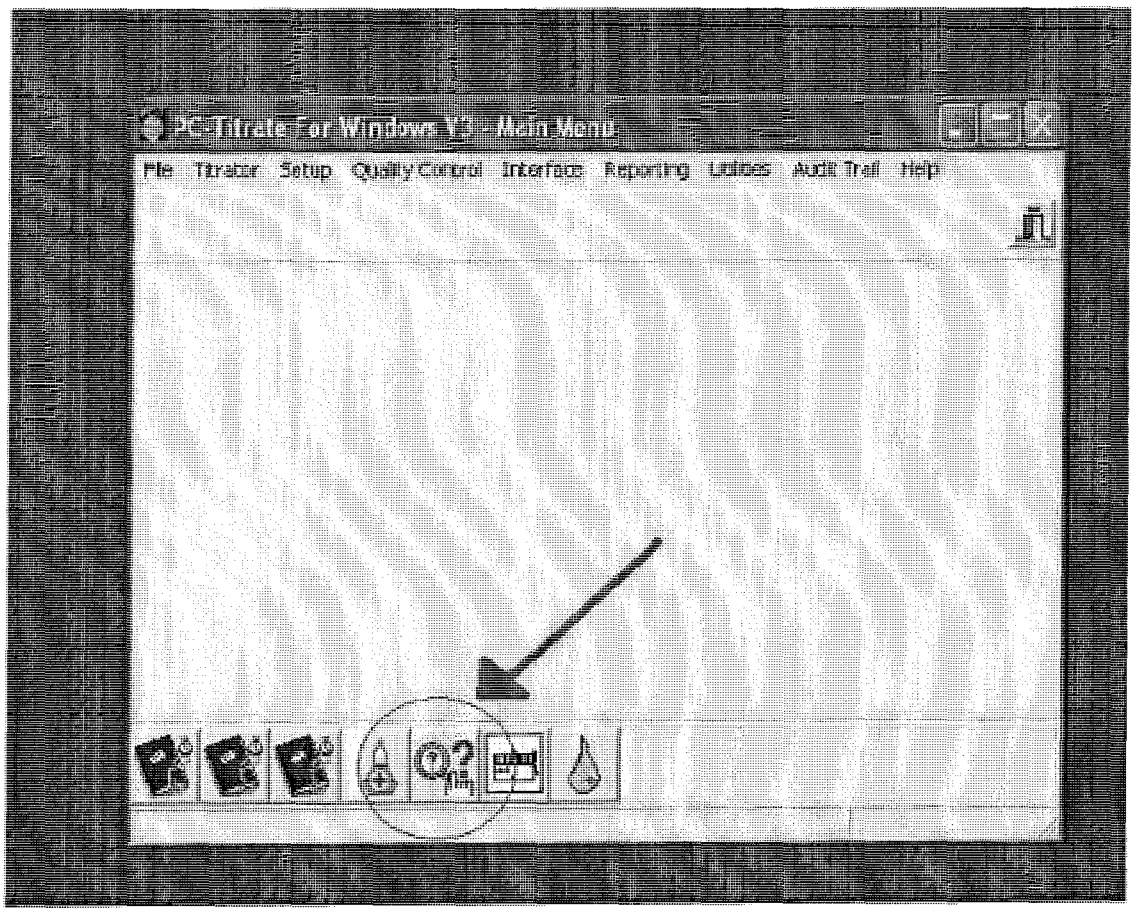
FIGURE 23



14.9 Retrieving Past (Historical) Data

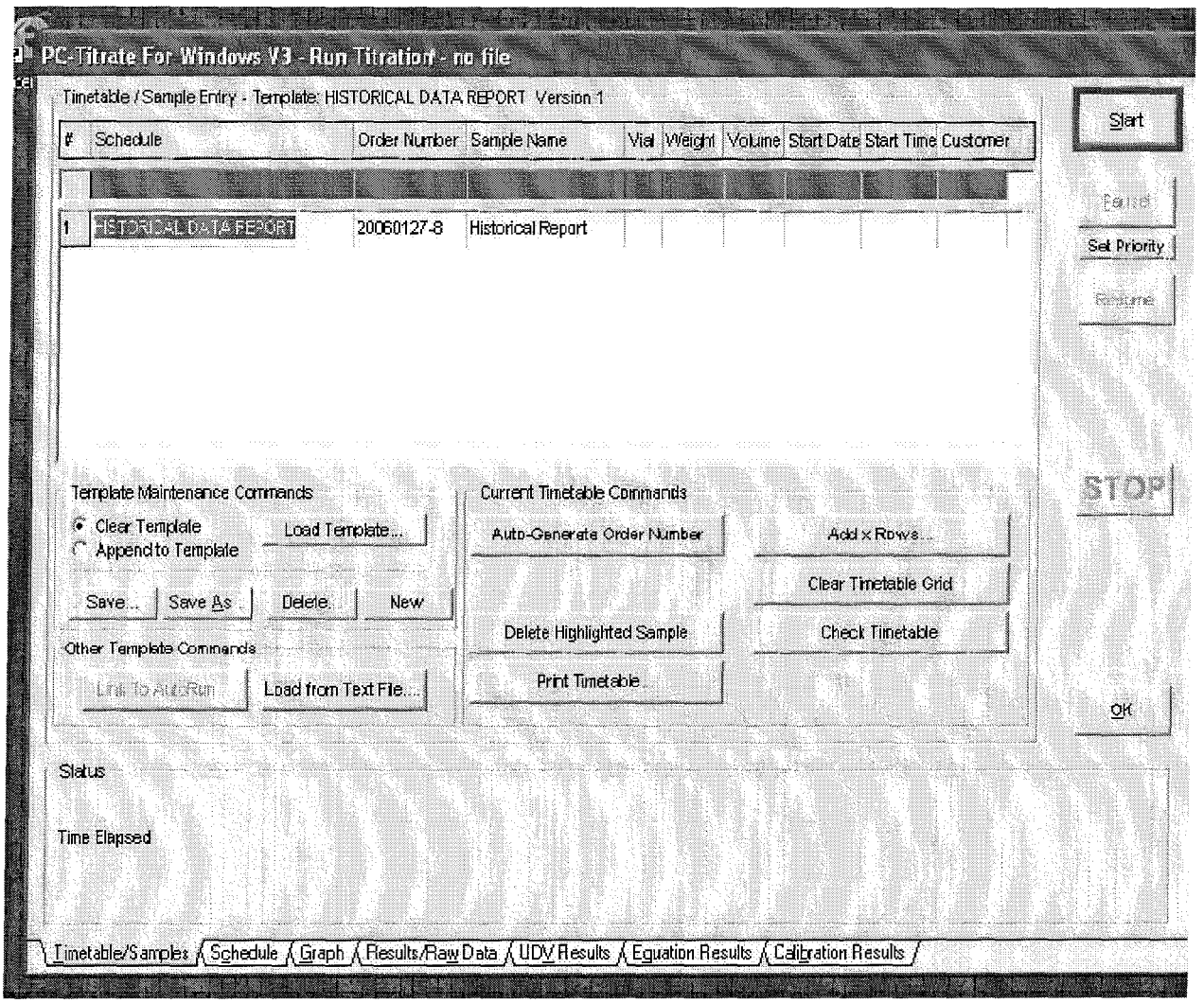
14.9.1 To obtain results from past analyses, return to the main menu. Click on the Inquiry Icon as shown in Figure 24.

FIGURE 24



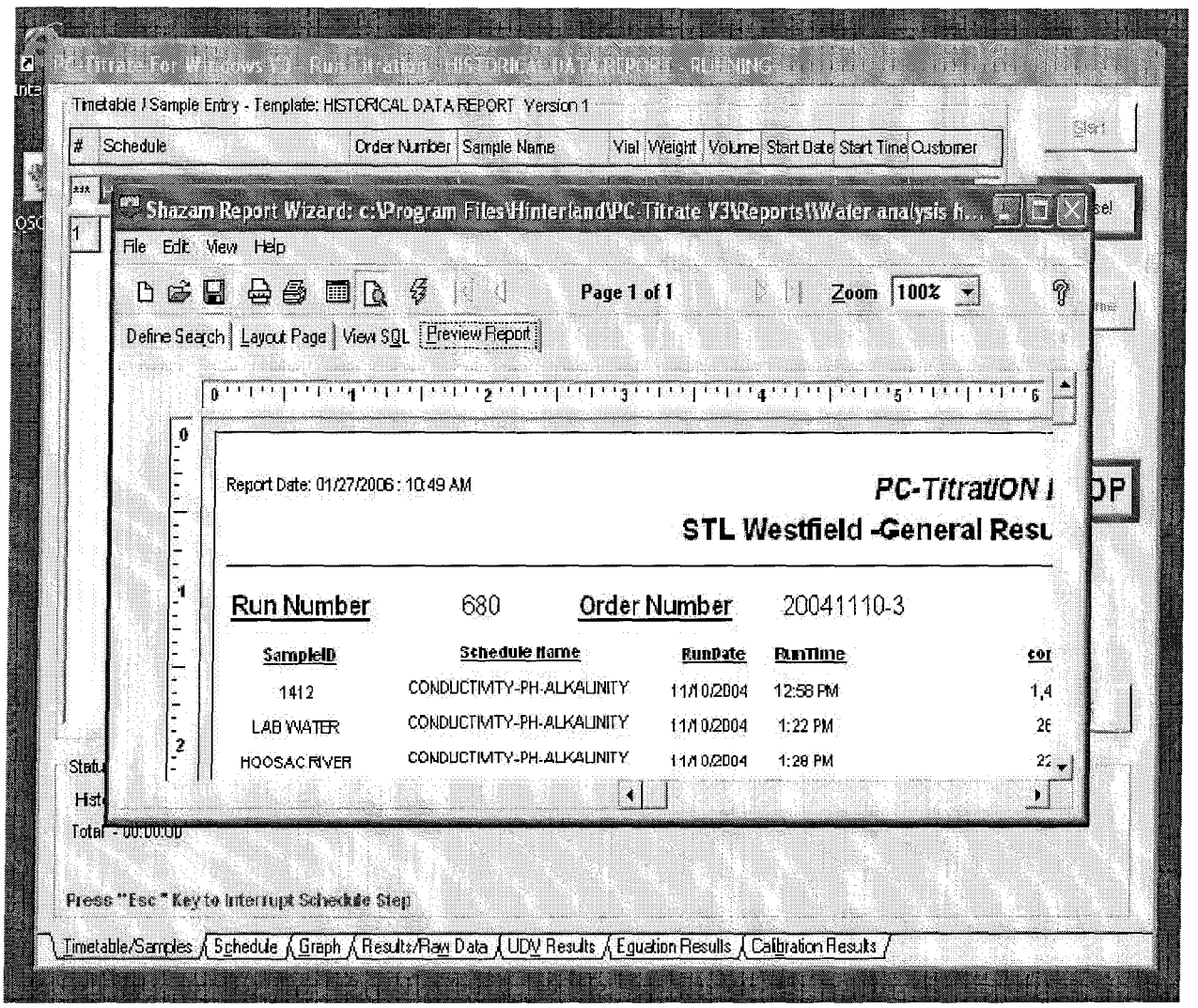
14.9.2 A new window will appear as shown in Figure 25. This will permit the generation of the historical data database.

FIGURE 25



14.9.3 Click on "Start" in order to generate the Shazam report for the <u>most recent</u> analytical batch. (See Figure 26.)

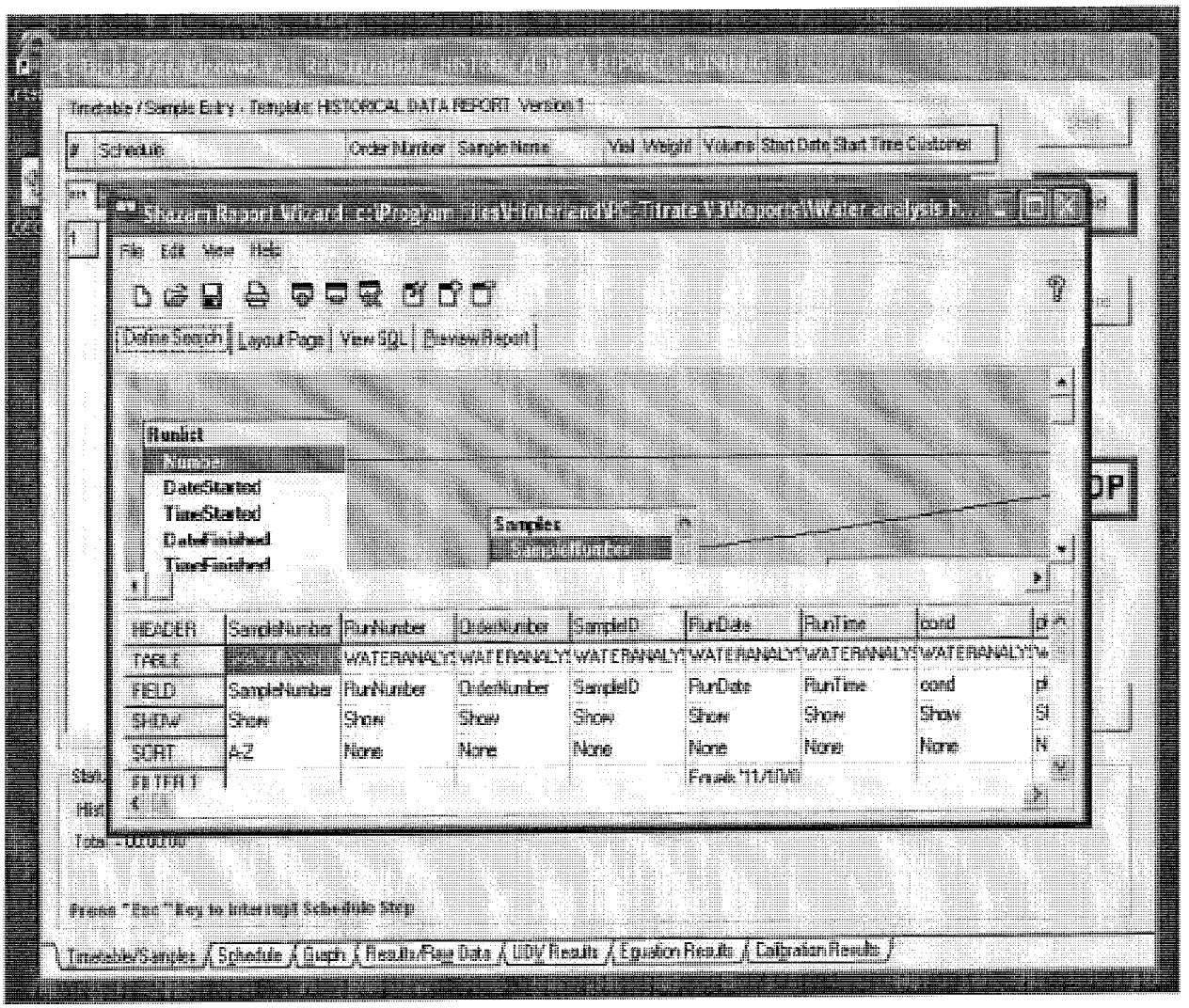
FIGURE 26



14.9.4 A copy may be printed by clicking on the printer icon in the menu bar.

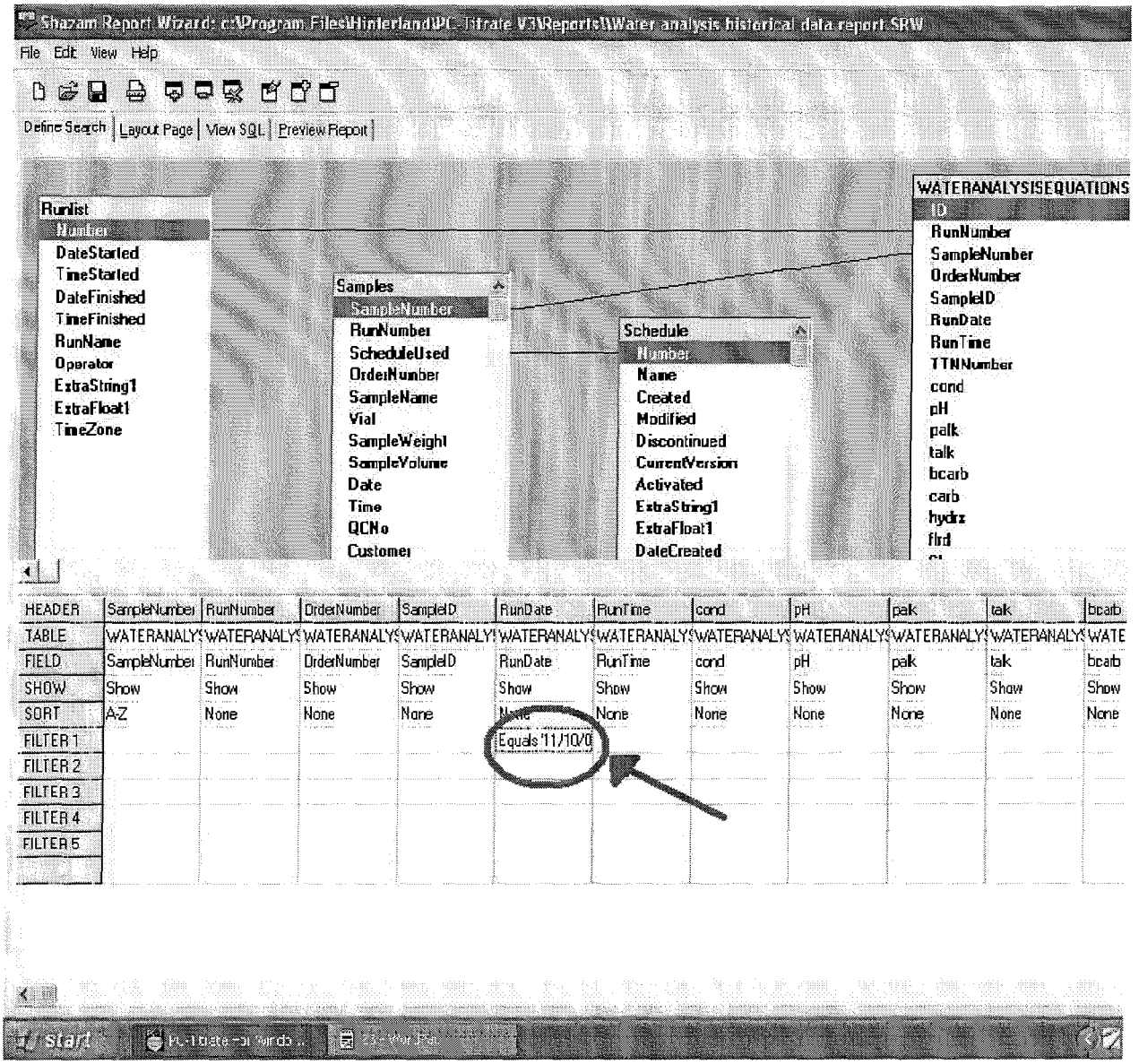
14.9.5 If this data is not what you wish to print, you may search for older data by clicking on the "Define Search" tab in the upper left. Doing so generates a new window as shown in Figure 27.

FIGURE 27



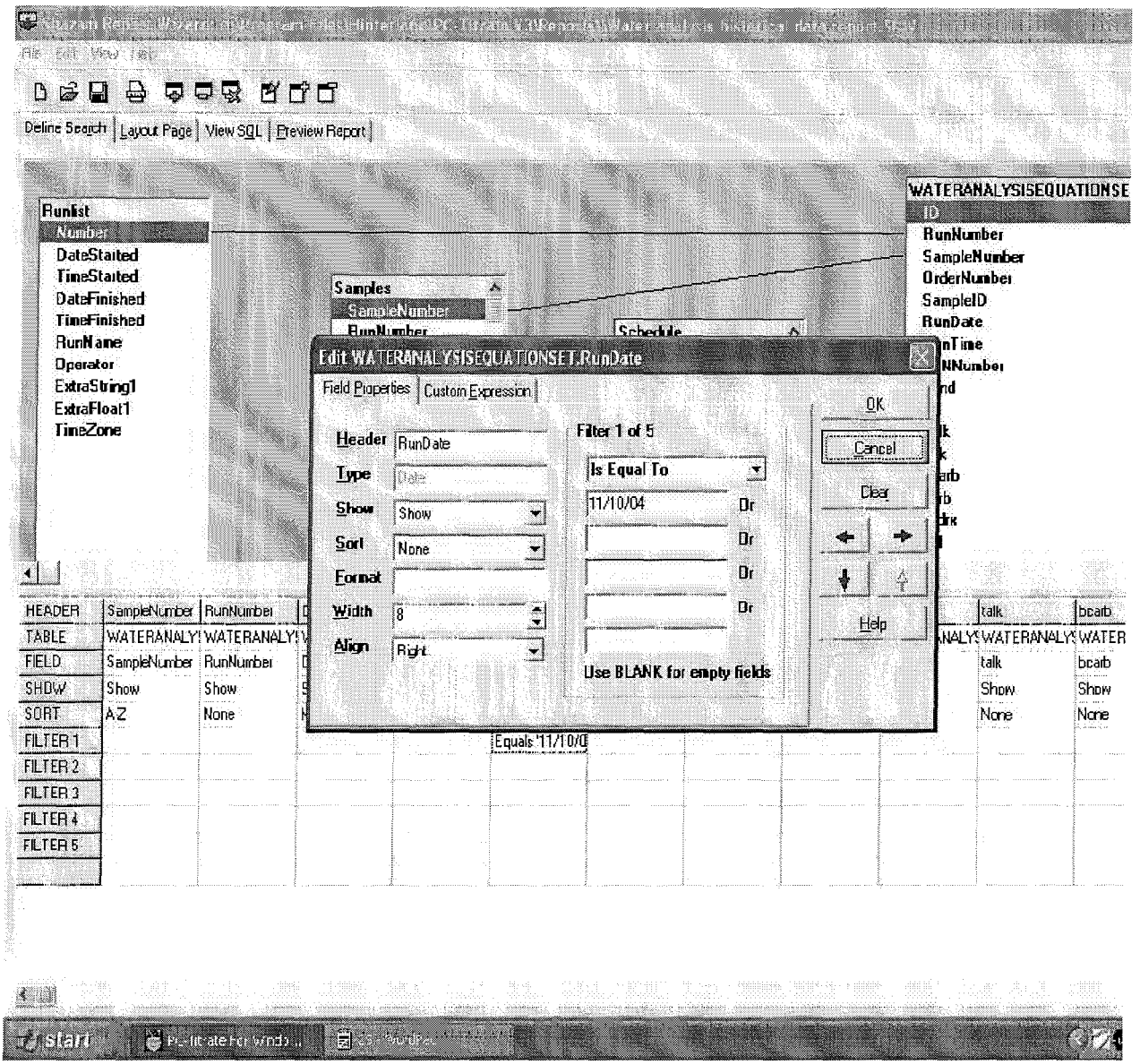
14.9.6 To find older data from this screen, expand the window to its full size, and click in the field at the intersection of the "RunDate" column and the "FILTER 1" row as shown in Figure 28 (circled item).

FIGURE 28



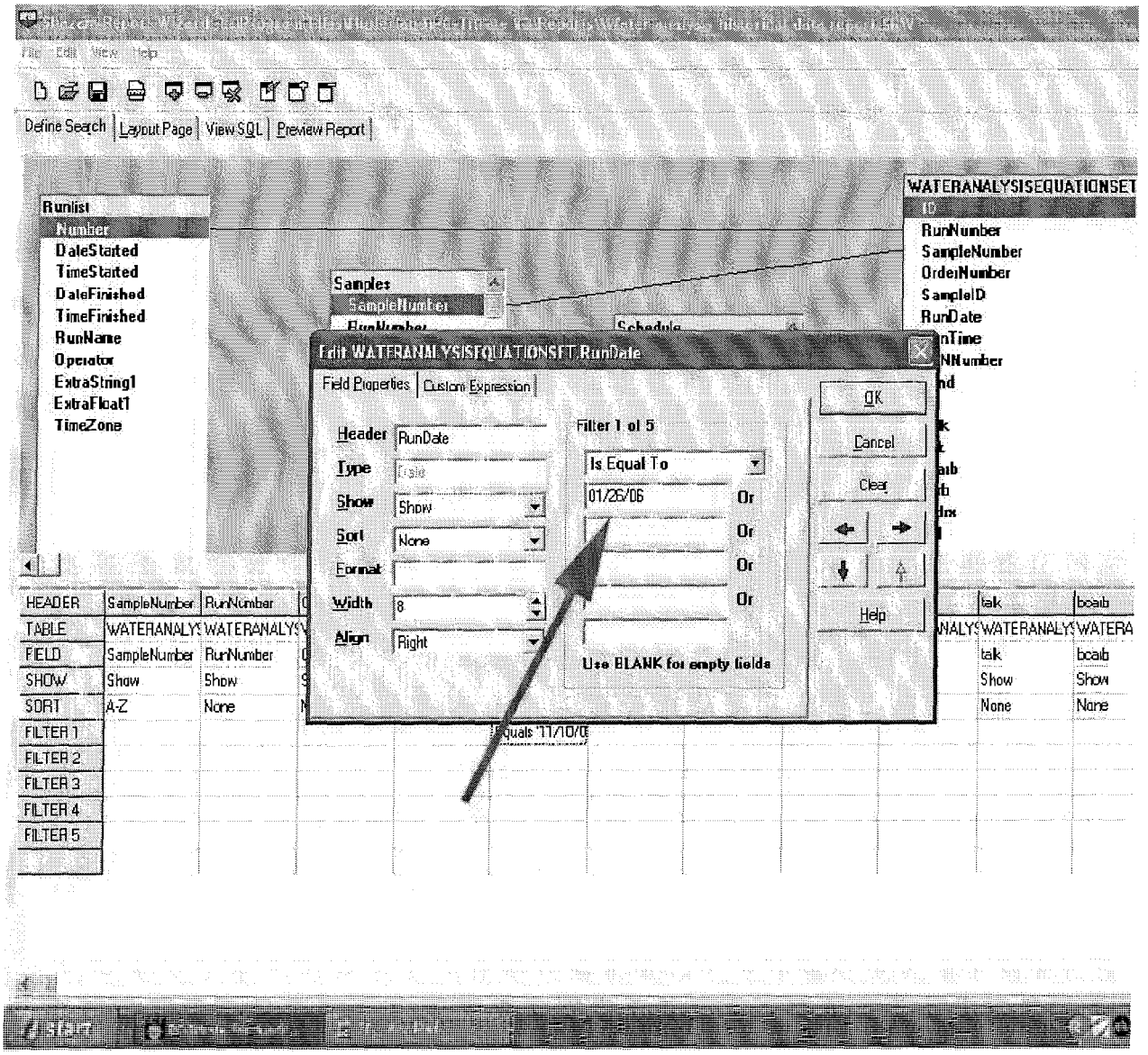
14.9.7 Clicking in this field (as shown in Figure 28) will open a new window as shown in Figure 29.

FIGURE 29



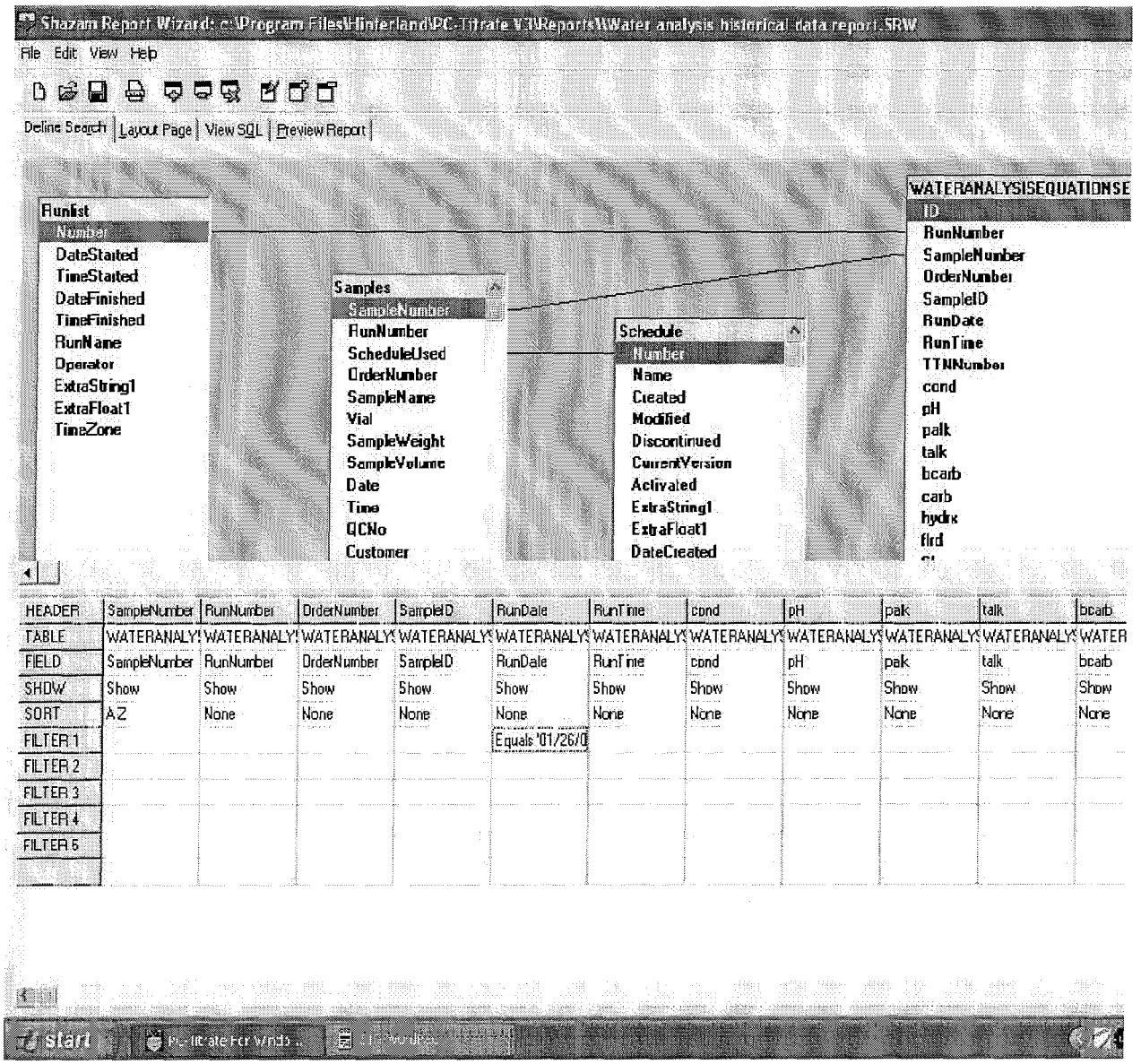
14.9.8 In the first field (that currently holds 11/10/04 in the figure above) delete out the current date held in that field, and enter the date for which you want to produce data. The format must be MM/DD/YY. For example, if you want data for 26 January 2006, enter “01/26/06” (as shown in Figure 30).

FIGURE 30



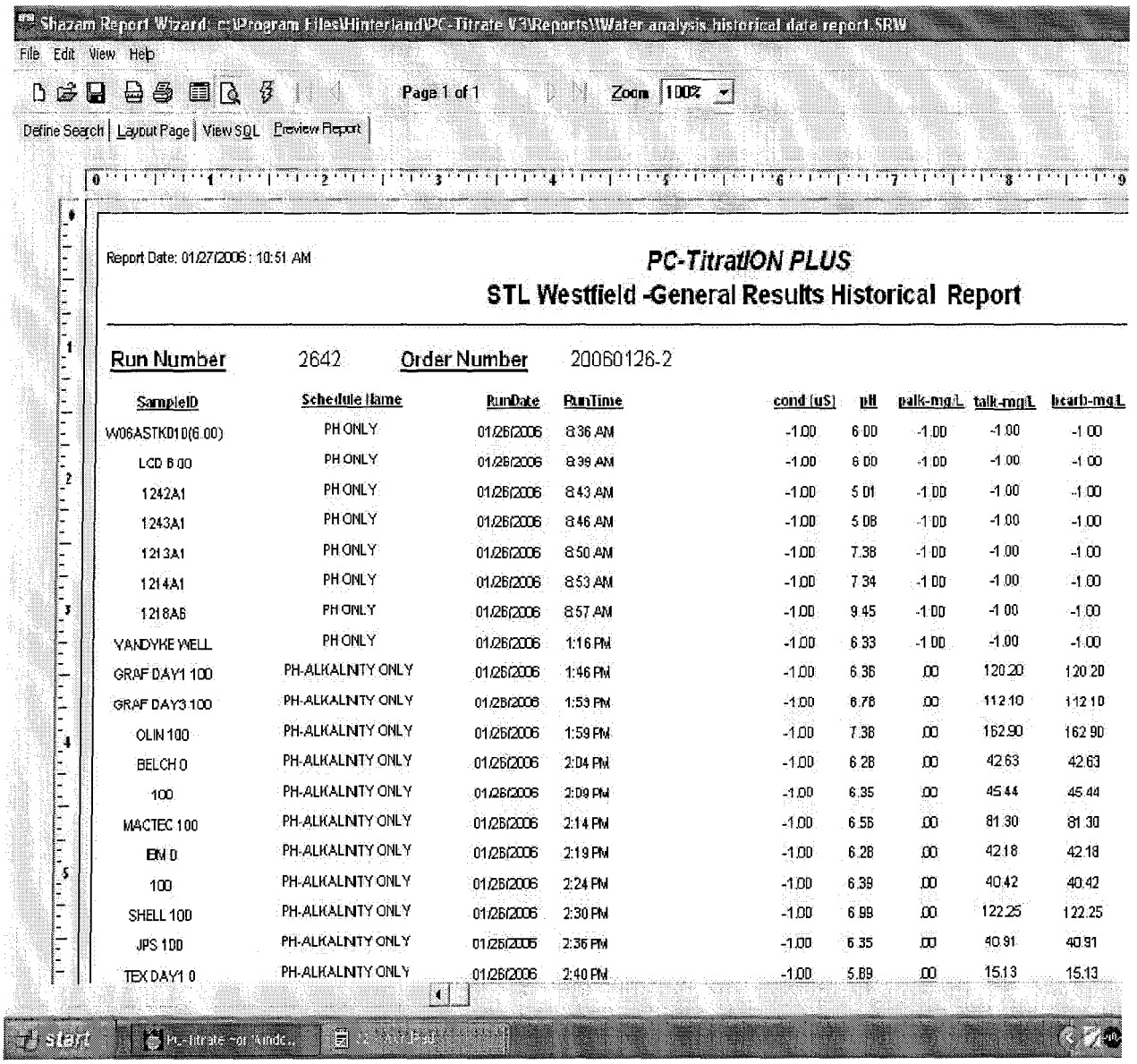
14.9.9 Click the "OK" button to lock in the data, producing a screen like that shown in Figure 31.

FIGURE 31



14.9.10 Click on the “Preview Report” tab at the upper left, and a report will open in a new window as shown in Figure 32.

FIGURE 32



SampleID	Schedule Name	RunDate	RunTime	cond (uS)	pH	palk-mg/L	talk-mg/L	bcarb-mg/L
W06ASTKD10(6.00)	PH ONLY	01/26/2006	8:36 AM	-1.00	6.00	-1.00	-1.00	-1.00
LCD 6.00	PH ONLY	01/26/2006	8:39 AM	-1.00	6.00	-1.00	-1.00	-1.00
1242A1	PH ONLY	01/26/2006	8:43 AM	-1.00	5.01	-1.00	-1.00	-1.00
1243A1	PH ONLY	01/26/2006	8:46 AM	-1.00	5.08	-1.00	-1.00	-1.00
1213A1	PH ONLY	01/26/2006	8:50 AM	-1.00	7.38	-1.00	-1.00	-1.00
1214A1	PH ONLY	01/26/2006	8:53 AM	-1.00	7.34	-1.00	-1.00	-1.00
1218A6	PH ONLY	01/26/2006	8:57 AM	-1.00	9.45	-1.00	-1.00	-1.00
VANDYKE WELL	PH ONLY	01/26/2006	1:16 PM	-1.00	6.33	-1.00	-1.00	-1.00
GRAF DAY1 100	PH-ALKALNITY ONLY	01/26/2006	1:46 PM	-1.00	6.36	.00	120.20	120.20
GRAF DAY3 100	PH-ALKALNITY ONLY	01/26/2006	1:53 PM	-1.00	6.78	.00	112.10	112.10
OLIN 100	PH-ALKALNITY ONLY	01/26/2006	1:59 PM	-1.00	7.38	.00	162.90	162.90
BELCH 0	PH-ALKALNITY ONLY	01/26/2006	2:04 PM	-1.00	6.28	.00	42.63	42.63
100	PH-ALKALNITY ONLY	01/26/2006	2:09 PM	-1.00	6.35	.00	45.44	45.44
MACTEC 100	PH-ALKALNITY ONLY	01/26/2006	2:14 PM	-1.00	6.56	.00	81.30	81.30
EM 0	PH-ALKALNITY ONLY	01/26/2006	2:19 PM	-1.00	6.28	.00	42.18	42.18
100	PH-ALKALNITY ONLY	01/26/2006	2:24 PM	-1.00	6.39	.00	40.42	40.42
SHELL 100	PH-ALKALNITY ONLY	01/26/2006	2:30 PM	-1.00	6.99	.00	122.25	122.25
JPS 100	PH-ALKALNITY ONLY	01/26/2006	2:36 PM	-1.00	6.35	.00	40.91	40.91
TEX DAY1 0	PH-ALKALNITY ONLY	01/26/2006	2:40 PM	-1.00	5.89	.00	15.13	15.13

14.9.11 Print the report by clicking on the printer icon. When finished, click on the Red X in the upper right corner of the window to exit. **IMPORTANT!** The software will ask you if you wish to save (see Figure 33). **YOU MUST CLICK THE "NO" BUTTON!** (See Figure 34.) If you click yes, all data up to that point will be deleted in favor of the single batch of data you just called up.

FIGURE 33

Page 1 of 1 Zoom 100%

Define Search | Layout Page | View SQL | Preview Report

Report Date: 01/27/2006 : 10:51 AM

PC-TitratION PLUS

STL Westfield -General Results Historical Report

Run Number 2642 **Order Number** 20060126-2

SampleID	Schedule Name	RunDate	RunTime	cond (uS)	pH	palk-mg/L	talk-mg/L	bcarb-mg/L
W06ASTKD1D(6.00)	PH ONLY	[illegible]	[illegible]	-1.00	6.00	-1.00	-1.00	-1.00
LCD 6.00	PH ONLY	[illegible]	[illegible]	[illegible]	6.00	-1.00	-1.00	-1.00
1242A1	PH ONLY	[illegible]	[illegible]	[illegible]	5.01	-1.00	-1.00	-1.00
1243A1	PH ONLY	[illegible]	[illegible]	[illegible]	5.08	-1.00	-1.00	-1.00
1213A1	PH ONLY	[illegible]	[illegible]	[illegible]	7.38	-1.00	-1.00	-1.00
1214A1	PH ONLY	[illegible]	[illegible]	[illegible]	7.34	-1.00	-1.00	-1.00
1218A6	PH ONLY	[illegible]	[illegible]	[illegible]	9.45	-1.00	-1.00	-1.00
VANDYKE WELL	PH ONLY	01/26/2006	1:16 PM	-1.00	6.33	-1.00	-1.00	-1.00
GRAF DAY1 100	PH-ALKALINITY ONLY	01/26/2006	1:46 PM	-1.00	6.36	.00	120.20	120.20
GRAF DAY3 100	PH-ALKALINITY ONLY	01/26/2006	1:53 PM	-1.00	6.76	.00	112.10	112.10
OLIN 100	PH-ALKALINITY ONLY	01/26/2006	1:59 PM	-1.00	7.38	.00	162.90	162.90
BELCH 0	PH-ALKALINITY ONLY	01/26/2006	2:04 PM	-1.00	6.28	.00	42.63	42.63
100	PH-ALKALINITY ONLY	01/26/2006	2:09 PM	-1.00	6.35	.00	45.44	45.44
MACTEC 100	PH-ALKALINITY ONLY	01/26/2006	2:14 PM	-1.00	6.56	.00	81.30	81.30
EM 0	PH-ALKALINITY ONLY	01/26/2006	2:19 PM	-1.00	6.28	.00	42.18	42.18
100	PH-ALKALINITY ONLY	01/26/2006	2:24 PM	-1.00	6.39	.00	40.42	40.42
SHELL 100	PH-ALKALINITY ONLY	01/26/2006	2:30 PM	-1.00	6.99	.00	122.25	122.25
JPS 100	PH-ALKALINITY ONLY	01/26/2006	2:36 PM	-1.00	6.35	.00	40.91	40.91
TEX DAY1 0	PH-ALKALINITY ONLY	01/26/2006	2:40 PM	-1.00	5.89	.00	15.13	15.13

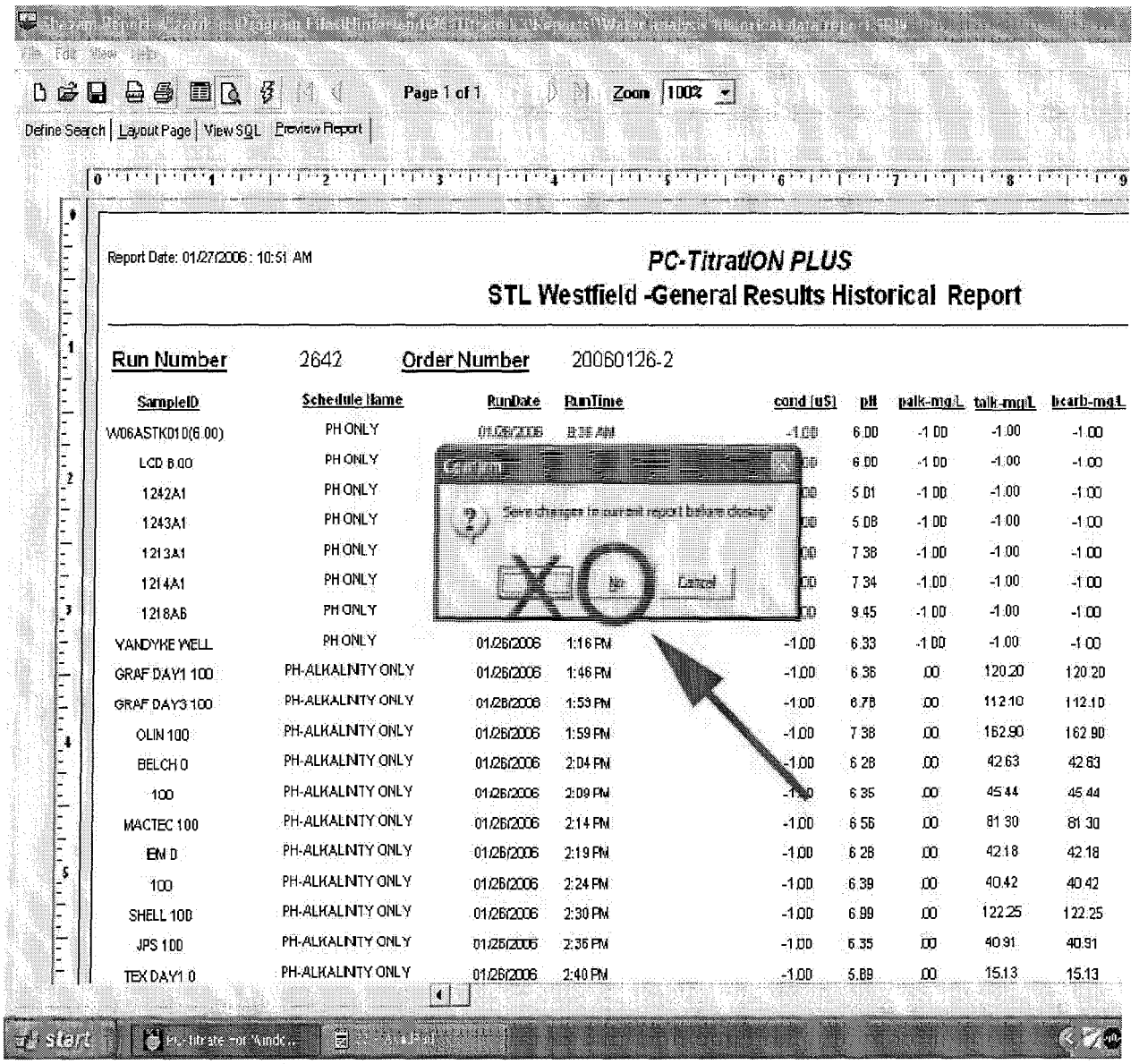
Confirm

Save changes to current report before closing?

Yes No Cancel

Start

FIGURE 34



Report Date: 01/27/2006 : 10:51 AM

PC-TitratION PLUS

STL Westfield -General Results Historical Report

Run Number 2642 **Order Number** 20060126-2

SampleID	Schedule Name	RunDate	RunTime	cond (uS)	pH	palk-mg/L	talk-mg/L	bcarb-mg/L
W06ASTKD1 0(6.00)	PH ONLY	[illegible]	[illegible]	-1.00	6.00	-1.00	-1.00	-1.00
LCD 6.00	PH ONLY	[illegible]	[illegible]	[illegible]	6.00	-1.00	-1.00	-1.00
1242A1	PH ONLY	[illegible]	[illegible]	[illegible]	5.01	-1.00	-1.00	-1.00
1243A1	PH ONLY	[illegible]	[illegible]	[illegible]	5.08	-1.00	-1.00	-1.00
1213A1	PH ONLY	[illegible]	[illegible]	[illegible]	7.38	-1.00	-1.00	-1.00
1214A1	PH ONLY	[illegible]	[illegible]	[illegible]	7.34	-1.00	-1.00	-1.00
1218A6	PH ONLY	[illegible]	[illegible]	[illegible]	9.45	-1.00	-1.00	-1.00
VANDYKE WELL	PH ONLY	01/26/2006	1:16 PM	-1.00	6.33	-1.00	-1.00	-1.00
GRAF DAY1 100	PH-ALKALINITY ONLY	01/26/2006	1:46 PM	-1.00	6.36	.00	120.20	120.20
GRAF DAY3 100	PH-ALKALINITY ONLY	01/26/2006	1:53 PM	-1.00	6.78	.00	112.10	112.10
OLIN 100	PH-ALKALINITY ONLY	01/26/2006	1:59 PM	-1.00	7.38	.00	162.90	162.90
BELCH 0	PH-ALKALINITY ONLY	01/26/2006	2:04 PM	-1.00	6.28	.00	42.63	42.63
100	PH-ALKALINITY ONLY	01/26/2006	2:09 PM	-1.00	6.35	.00	45.44	45.44
MACTEC 100	PH-ALKALINITY ONLY	01/26/2006	2:14 PM	-1.00	6.56	.00	81.30	81.30
EM 0	PH-ALKALINITY ONLY	01/26/2006	2:19 PM	-1.00	6.28	.00	42.18	42.18
100	PH-ALKALINITY ONLY	01/26/2006	2:24 PM	-1.00	6.39	.00	40.42	40.42
SHELL 100	PH-ALKALINITY ONLY	01/26/2006	2:30 PM	-1.00	6.99	.00	122.25	122.25
JPS 100	PH-ALKALINITY ONLY	01/26/2006	2:35 PM	-1.00	6.35	.00	40.91	40.91
TEX DAY1 0	PH-ALKALINITY ONLY	01/26/2006	2:40 PM	-1.00	5.89	.00	15.13	15.13

14.10 Preventive Maintenance

14.10.1 Autosampler

14.10.1.1 Over time the plastic autosampler racks will become caked with dried buffer solution and/or samples. To clean, remove them from the rack holder and rinse them under steaming-hot tap water, using a soapy wash brush to scrub off stubborn material. Rinse thoroughly with tap water and allow to air dry inverted on absorbent lab paper.

14.10.1.2 Clean the autosampler rack holder by wiping it down with a sponge soaked in hot, soapy water, then removing soapy residue with a sponge soaked in clean, hot tap water.

14.10.1.3 Clean the glass rinse well by first removing it from the zero-position in the first rack, then removing the plastic tubing from the top and bottom fittings, and then run hot tap water over and through it. Clean with a soapy-water brush to remove caked on material. Rinse with tap water and then deionized water. Allow to air dry inverted on absorbent lab paper, and once dry, re-assemble.

14.10.2 Conductivity Probe

14.10.2.1 Over time, the conductivity probe will have a layer of dirty material accumulate on its outer surface. To clean, remove it from the arm assembly by first loosening the retaining screw, then gently wiggling it out of the holder. Using a Kimwipe dampened with methanol, remove oil and material build-up from the outer surface. DO NOT touch the electrodes on the bottom interior of the probe. Re-install once clean.

14.10.3 Sampling Probe

14.10.3.1 The sampling probe allows sample to be taken up to the mixing chamber by a pump. Make sure that the probe tip isn't bent, and is attached to the conductivity probe with the black rubber o-rings, one placed high and one placed low. Adjust the height of the sampling probe by hand so that the bottom tip is about one-half inch above the bottom of the sample cup when the probe is in its fully lowered position.

14.10.3.2 If the sampling probe is bent, or doesn't appear to be drawing up liquid uniformly when the pump is in operation, replace it with a new one.

14.10.4 Mixing Chamber

14.10.4.1 Over time the mixing chamber will build up precipitated material on its inner surface, as well as on the thermometer, acid injection tip, pH probe, and stirring paddle. To clean, remove the various lines and probes from the chamber. **CAUTION!** Remove carefully. The thermometer can easily shatter if not lifted straight upward gently!

14.10.4.2 Clean the probes and paddle with a Kimwipe dampened with methanol. Allow them to air dry before re-assembling them.

14.10.4.3 The acid injection line should be set so that its tip is just below the liquid level when a run is in progress. The best way to judge this is to set up an alkalinity sample to run, and once the sample is drawn up by the instrument and the paddle begins to stir, adjust the acid injection line by hand.

14.11 Trouble-Shooting

14.11.1 The most common problems associated with the autotitrator are:

14.11.1.1 Clogged sample uptake line.

14.11.1.2 Sample uptake probe/conductivity probe misalignment or fault.

14.11.1.3 Electronic communication error.

14.11.2 Clogged Sample Uptake Line

14.11.2.1 The black sample uptake line and the circular pump head that pumps sample into the mixing chamber may become clogged if a sample with high suspended solids is encountered. It is

therefore important to filter any samples that have high solids content (e.g. solid/soil extracts, water samples with flocculent suspensions, etc.).

14.11.2.2 Should the pump clog, it most often occurs at the pump head. To clean:

14.11.2.2.1 Unscrew each of the nubs that connect the black plastic transfer lines to the pump head. Next unscrew the two Phillps-head screws on the head, and remove the black plastic cover piece. **DO NOT REMOVE THE SILVER METAL COLLAR AROUND THIS UNIT.**

14.11.2.2.2 Remove the stainless steel stem from the pump opening. Clean the area with a Q-tip.

14.11.2.2.3 Flush out the black lines using a syringe and warm tap water, backflushing to remove any blockage.

14.11.2.2.4 Re-insert the stainless steel stem into the unit so that the small leg of the "L" shape fits into the ball joint's hole inside the pump cavity. Re-attach the black plastic housing and re-screw the Phillips-head screws to finger-tight.

14.11.2.2.5 Re-attached the black plastic hoses and manually pump deionized water through the system to clean it.

14.11.3 Uptake Probe Misalignment

14.11.3.1 Should the conductivity probe (to which the uptake probe is attached with an o-ring) miss a cup, it is probably misaligned. Return it to the probe home position by either turning all equipment off, counting to ten, then turning it back on, or homing the autosampler with the software, whichever is available.

14.11.3.2 Once at the home position, make sure that the conductivity probe is not seated to deep or shallow in the white plastic arm. The uptake probe should be about 1.5 inches lower than the tip of the conductivity probe, and the o-ring should hold the two together, seated just about a half-inch up from the conductivity probe's bottom end.

14.11.3.3 Re-start the run and watch the probe. If it fails to clear a cup, move the probes up a bit in the arm so that they will clear in the future.

14.11.3.4 Sometimes the conductivity probe's cable catches on the cups in the sample racks. Make certain that the cable is in a starting position outside of the racks to avoid such.

14.11.4 Communications Error

14.11.4.1 Sometimes the instrument looses communication, or has an error or fault due to the instrument not communicating with the computer. To correct:

14.11.4.1.1 Turn all instrument components off.

14.11.4.1.2 Reboot the computer. Once the OS is finished launching, turn on all instrument components.

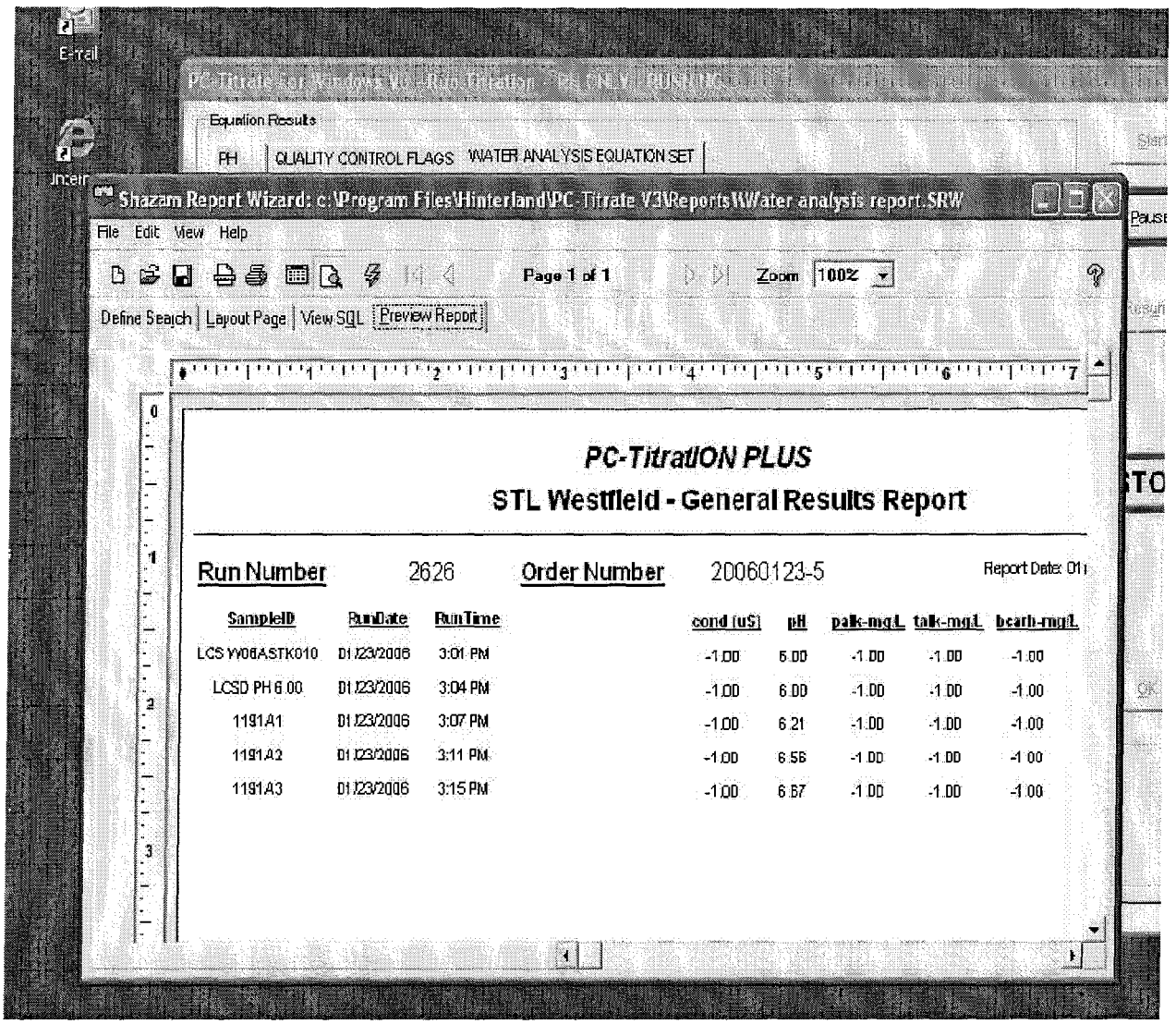
14.11.4.1.3 Try a Home Autosampler command. If it doesn't work, call technical support at ManSci for assistance. Otherwise, attempt a test run for alkalinity and observe

the instrument. If problems still exist, call technical support at ManSci for assistance.

15.0 CALCULATIONS & REPORTING DATA

15.1 The instrument automatically calculates pH, conductivity, and alkalinity values for the samples based on the calibrations it performs. A summary report automatically appears on the computer CRT once the batch has been analyzed. (Refer to Figure 35.)

FIGURE 35



15.2 You may print a copy of this report by clicking on the printer icon. Or you may simply close out the window, and a printed copy will automatically be generated. **IMPORTANT!** It is suggested that you manually instigate a printed copy so that you

may be sure that it prints properly before closing down the window. Make sure the printer is on, has paper, and is not in a fault mode before printing.

15.3 Report the reading directly from the conductivity meter for each sample. If a dilution of a sample was made, calculate the adjusted conductivity value for the sample.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.2.1 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCES

22.1 "Standard Methods for the Examination of Water and Wastewater," 18th Edition, 1992, Method 2320B.
22.2 "Standard Methods for the Examination of Water and Wastewater," 18th Edition, 1992, Method 2510B.
22.3 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79- 020, March 1983, method 120.1.
22.4 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79- 020, March 1983, method 150.1.
22.5 Method 9045C, "Soil and Waste pH", SW-846, Revision IIB, 1995.

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STANDARD OPERATING PROCEDURE

Analytical Procedure for the determination of Hardness by SM 2340 B

SOP ID: MES01600.MA

Date Written: 06/05/95
Revision Date: 11/08/06
Effective Date: 11/15/06
Revision (3)

Approved Signatures and Dates

QUALITY ASSURANCE COORDINATOR

LABORATORY DIRECTOR

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1.0 TITLE: Analytical Procedure for the determination of Hardness by SM 2340 B

2.0 MATRIX: Hardness can be computed from aqueous or solid sample results.

3.0 METHOD DETECTION LIMIT: The reporting limit for Hardness is 2.6mg/L. for aqueous samples and 66mg/Kg for soils.

4.0 SCOPE:

4.1 All waters and soil results can be reported for Hardness. Hardness is determined by computing it from the results of separate determinations of calcium and magnesium.

5.0 SUMMARY

5.1 This method describes the calculation equation for Hardness using SM 2340B. The determination of Calcium and Magnesium are performed by either EPA method 200.7 or SW846 6010B.

6.0 DEFINITIONS

Batch: environmental samples, which are prepared and/or analyzed together with the same process using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR , Part 136, Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 There are no interferences as such in the calculation of Hardness, however methods 200.7 and 6010 document chemical, spectral and physical interferences. Please refer to SOP number MES 00200MA 6010B and MES 00300MA 200.7 for more details.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

8.2 PRIMARY MATERIALS USED

There are no materials used in this method that have a serious or

significant hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

9.0 APPARATUS

9.1 Varian ICP, model 720
9.2 Computer for data collection and system printer.
9.3 Calculator

10.0 REAGENTS

10.1 There are no reagents as such in the calculation of Hardness; however methods 200.7 and 6010 document reagents used to perform those methods. Please refer to SOP number MES 00200MA 6010B and MES 00300MA 200.7 for more details.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Water samples are collected in plastic or glass 1000 ml sample containers, and preserved with Nitric acid to a pH of <2.0.
11.2 Holding time for samples collected for metal analysis is 6 months from date of collection.

12.0 QUALITY CONTROL

12.1 There are no quality control measurements as such in the calculation of Hardness; however methods 200.7 and 6010 document specific quality control limits used to perform those methods. Please refer to SOP number MES 00200MA 6010B and MES 00300MA 200.7 for more details.

13.0 CALIBRATION and STANDARDIZATION

13.1 There are no calibration and standardization as such in the calculation of Hardness; however methods 200.7 and 6010 document specific calibration procedures used to perform those methods. Please refer to SOP number MES 00200MA 6010B and MES 00300MA 200.7 for more details.

14.0 PROCEDURE

14.1 There are no procedures as such in the calculation of Hardness, however

methods 200.7 and 6010 document specific instrument procedures to ensure valid data generation. Pease refer to SOP number MES 00200MA 6010B and MES 00300MA 200.7 for more details.

15.0 CALCULATION

15.1 Hardness, mg equivalent.

$CaCO3/L = 2.497 \times (Ca, mg/L) + 4.118 \times (Mg, mg/L)$.

16.0 METHOD PERFORMANCE

16.1 The supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

17.0 POLLUTION PEVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.2.1 Primary review is referred to as a “bench-level” review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method

SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

22.0 REFERENCE

22.1 Standard Methods For The Determination Of Water And Wastes". 18th Edition, 1992.

22.2 QA Manual for STL Westfield.

23.0 TABLES, DIAGRAMS, FLOWCHARTS VALIDATION DATA

23.1 N/A

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STL WESTFIELD

STANDARD OPERATING PROCEDURES

Analytical Procedure for the Analysis of Residue, Filterable (Solids, Total Dissolved)
Method 2540C
(EPA 160.1)

SOP ID:INS00900.MA

Revision (5)

Date Written: 02/06/98
Revision Date: 2/19/07
Effective Date: 2/19/07

Approved Signatures:

QUALITY ASSURANCE OFFICER

LABORATORY DIRECTOR

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1.0 TITLE : Solids, Total Dissolved

2.0 MATRIX: Drinking, surface, and saline waters, domestic and industrial wastes.

3.0 METHOD REPORTING LIMIT: 10mg/L

4.0 SCOPE and APPLICATION

4.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes

4.2 The practical range of the determination is 10 mg/L to 20,000 mg/L

5.0 SUMMARY

5.1 A filtered aliquot of the sample is quantitatively transferred to a pre-weighed evaporating dish, evaporated and dried to constant weight at 180° C. The weight of the residue is then used to calculate the total dissolved solids content.

6.0 DEFINITIONS

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is introduced onto the instrument for analysis and can monitor calibration baseline drift and carryover contamination effects.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement

responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

7.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.

7.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

8.2 PRIMARY MATERIALS USED

There are no materials used in this method that have a significant or serious hazard rating NOTE: This list does not include all materials used in the method. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

9.0 APPARATUS

9.1 60 and 120mL capacity evaporating dishes
9.2 Drying oven, 105°C and 180°C
9.3 Desiccators
9.4 Analytical Balance
9.5 Filter holder, membrane filter funnel and Gooch crucible adapter

9.6 Suction flask, 500mL
9.7 100mL graduated cylinder
9.8 Gooch crucibles
9.9 2.1cm 0.45μm glass fiber filters
9.10 Forceps
9.11 Crucible tongs
9.12 Autoclave gloves
9.13 High temperature china marker

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1 Deionized water
10.2 Laboratory Control Sample (LCS): Weigh out 0.500g of oven-dried sodium chloride (ACS grade or better) and pour into a 1L volumetric flask containing about 900mL of Deionized water; QS to mark, stopper, and mix by inversion until the sodium chloride has been dissolved. LCS concentration is 500 mg/L.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Store samples at 4°C. Analyze within 7 days.

12.0 QUALITY CONTROL

12.1 Quality Control Evaluation

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Method Blank (MB)	Per batch of 20 samples or fraction thereof	<10mg/L	If the MB is >10 mg/L, reheat in the oven, desiccate, and take an additional weight reading to determine TDS value; if still >10, contact the QAQC department.
Laboratory Control Sample (LCS)	Per batch of 20 samples or fraction thereof	%R 85-115%	If out of recovery range, reheat in the oven, desiccate, and take an additional weight reading to determine TDS value; if still out of range, contact the QAQC department. Reanalyze batch

Laboratory Control Duplicate (LCD) (optional)	Per batch of 20 samples or fraction thereof	%R 85-115% RPD ≤ 20%	If RPD is >20%, reheat the original and MD in the oven, desiccate, and reweigh; if still >20%, report data qualified by a QA Memo.
Method Duplicate (DU)	Per batch of 20 samples or fraction thereof	%R 85-115% RPD ≤ 20%	If RPD is >20%, reheat the original and MD in the oven, desiccate, and reweigh; if still >20%, report data qualified by a QA Memo.

Demonstration of Capability (DOC)	Once per year per analyst performing the analysis	85-115%	Analysis of 4 LCSs, ±15% of true value to demonstrate proficiency.
Varied CCV Concentration-NELAC Requirement	Once per year	90-110%	A CCV at a concentration level different than that routinely used, record this evaluation in the CCV file.

13.0 CALIBRATION and STANDARDIZATION: NA

14.0 PROCEDURE

14.1 Select a number of dishes needed to perform the analysis, including a method blank (MB), laboratory control sample (LCS), a laboratory control duplicate (LCD) (optional), and a method duplicate (DU) for each 20 investigative samples or fraction thereof. Label each dish with a unique ID number using the high temperature (green) china marker (so the number will not come off during heating), and weigh each, recording the weight on the bench sheet.

14.2 Place the dishes in the 180°C oven for one hour; afterward cool in a desiccator for at least 2 hours. Weigh each dish and repeat this process until the weight of the dish is constant (where the variance in the weights is ≤0.5mg or until five weighings have been performed).

14.3 Prepare a 0.45μm glass fiber filter disc by washing the disc with 3 successive 10mL volumes of Deionized water. Discard rinse water. Rinse the receiving flask twice with 10mL Mill-Q water and invert on lab paper or a paper towel to dry.

14.4 Assemble the filtering apparatus, and begin suction.

14.5 Filter about 125 mL of a sample through the glass fiber filter, collecting the filtrate in the Deionized water rinsed vacuum flask.

14.6 Using a graduated cylinder, transfer 100mL of filtrate to a weighed 120mL

evaporation dish (or 50mL of sample to a 60mL dish) and evaporate to dryness in a 105°C oven. Rinse apparatus twice with 10mL portions of distilled water and discard the rinse water. Dry inverted on lab paper or a paper towel. Repeat 10.4-10.8 for all samples.

14.7 Once all liquid has evaporated from the dishes, transfer the dishes to a 180°C oven and allow them to bake for 1 hour.

14.8 After 1 hour at 180°C remove the dishes from the oven and cool them in a desiccator for at least 2 hours. Weigh each dish, recording the weight in the "Wt 180°C" column. Reheat at 180°C for 1 hour, cool in a desiccator, and reweigh. Repeat this process until a constant weight is obtained (a variance of no more than ≤0.5mg between weights, or until a total of five weighings have been taken).

14.9 After use, soak used dishes in ChemSolv to remove baked on residue. Scrub clean with soap and water, and rinse thoroughly with tap water. Heat cleaned evaporating dishes to 550±50°C for 1 hour to burn off any residue. Cool to room temperature on a porcelain square, then store in a desiccator until needed.

15.0 CALCULATION

15.1 Sample Analyte Quantitation

Calculate TDS as follows:

Total Dissolved solids, mg/L = [(A-B) x 1000] ÷ C
Where:
A= weight of sample + dish **in mg**
B= weight of dish **in mg**
C= weight of sample **in mL**

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

17.2 There are no materials used in this method that have a significant or serious hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

18.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines

anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to STL Westfield's facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required. Please refer to section 17- Pollution Control for more details.

22.0 REFERENCES

22.1 "Standard Methods for the Examination of Water and Wastewater," 18th Edition, 1992, Method 2540C.

22.2 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983, method 160.1.

Corrections and/or additions:
2/19/07-replaced Milli-Q with de-ionized water.

Analytical Procedure for the Analysis of Ammonia by Flow Injection

QuikChem Method 10-107-06-1-A (EPA approved)

Approvals (Signature/Date):

9 OCT 2008

Richard Emerich — Date
Department Manager & Health & Safety Manager / Coordinator

10/9/08

Christine Furcinite-Reynolds — Date
Quality Assurance Manager

10/10/08

Steve Hartmann — Date
Laboratory Director

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1.0 Scope and Application

This method is applicable to the determination of ammonia in surface waters and industrial wastewaters, as well as soils and sediments. The applicable aqueous working range is 0.1-20.0 mg/L.

1.1 Analytes, Matrix(s), and Reporting Limits

Matrix: Aqueous, Sediment and Soil Samples

Reporting limit: The standard aqueous reporting limit (RL) for ammonia is 0.1 mg/L, and 5.0 mg/Kg for soils.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

This method is based on the Berthelot reaction: ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630nm, and the color intensity is directly proportional to the ammonia concentration in a sample.

3.0 Definitions

"Ammonia" (also referred to as "total ammonia") is defined as the total amount of aqueous ammonium ion that can be converted to ammonia gas with the heat and alkaline conditions above pH 9 during a distillation process.

"Free ammonia" is defined as the total amount of aqueous ammonium ion that is able to be converted to ammonia gas when treated with an alkaline reagent so that the solution has a pH >9, and doing so without heat distillation.

The "receiving solution" is that into which the distillate for each ammonia sample is collected from the distillation process. It is mildly acidic in order to keep the ammonia in solution for analysis.

Ammonium ion (NH_4^+) and dissolved ammonia gas (NH_3) exist at equilibrium dependent on the pH of the solution. This relationship is complex; the easiest way to determine the approximate amounts of each species for a given pH is to use the free online calculator at http://cobweb.ecn.purdue.edu/~piwc/w3-research/free-ammonia/nh3.html.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is introduced onto the instrument for analysis and can monitor calibration baseline drift and carryover contamination effects.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCSD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts,

magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

4.0 Interferences

Color, turbidity and certain organic species may interfere. Turbidity in the receiving solution is removed by manual filtration. Intrinsic sample color is corrected for automatically by the autoanalyzer.

Calcium and magnesium ions may precipitate as insoluble salts if present in sufficient concentration, so a tartrate or EDTA solution is added to the sample in-line in order to prevent these problems.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

There are no specialized safety concerns associated with this method.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonia (stock std)	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes

Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- EASY-Dist Distillation Apparatus and associated glassware
- QuikChem 8000 flow injection analyzer and associated chemistry manifold, pump, and pump tubing
- Analytical balance capable of accurately weighing to the nearest 0.1mg

6.2 Supplies

- Class A volumetric flasks and volumetric pipettes
- Plastic 120mL containers
- 50mL Class A graduated cylinder
- Deionized water swish bottle
- Carbon-granule boiling chips
- Narrow range test paper (pH 8.0-10.0) and (pH 6.0-8.0)
- Glass stirring rod
- EASY-Dist ammonia distillation glassware
- 13x100mm test tubes

7.0 Reagents and Standards

7.1 Distillation Reagents

7.1.1 0.10 *N* Sodium Hydroxide Solution: Dissolve 4.0g of NaOH (Nitrogen–free) pellets in 200mL deionized water in a 500mL beaker. Once dissolved, transfer quantitatively to a 1000mL graduated cylinder and dilute to the 1000mL mark with deionized water. Cool and store in plastic container. Alternatively, purchase a commercially prepared solution (such as from Lab Chem). EXP: 3 years.

7.1.2 0.025 *M* Sodium Tetraborate Solution: Dissolve 2.5g $Na_2B_4O_7$ or 4.75g $Na_2B_4O_7.10H_2O$ in about 300mL of deionized water in a 500mL beaker. Once dissolved, pour into a 500mL graduated cylinder and dilute to the 500mL mark with deionized water. Store the solution in a plastic container. EXP: 3 years.

7.1.3 Borate Buffer Solution: In a 1L plastic bottle, mix 44mL of 0.1 *N* NaOH solution, 250mL of 0.025 *M* tetraborate solution and 206mL of deionized water. EXP: 1 year.

7.1.4 15 *N* Sodium Hydroxide Solution: Dissolve 150.0g of NaOH (Nitrogen-free) pellets in 150mL deionized water in a 250mL beaker. Once the cooled to room temperature dilute to 250mL using a graduated cylinder. Store the solution in plastic container. Alternatively, purchase a commercially available solution (such as from JT Baker or Lab Chem). EXP 3 years.

7.1.5 Receiving Solution, Sulfuric acid, 0.04*N*: Dilute 1.0mL conc. H_2SO_4 to 1 liter with deionized water. EXP: 3 years.

7.2 Analytical Reagents

NOTE! All solutions must be degassed with helium to remove dissolved oxygen and prevent bubble formation. Use helium at 140 Kpa (20 lb/in^2) through a degassing tube. Bubble one liter of solution for one minute.

7.2.1 Sodium Phenolate Solution

CAUTION: Wear gloves!! Phenol causes severe burns and is rapidly absorbed into the body through the skin.

In a 1.0L volumetric flask, dissolve 85mL of 91% liquified phenol (Fisher catalog # A931-500) or 83g crystalline phenol (C_6H_5OH) in about 600mL deionized water. While stirring, add 32g sodium hydroxide (NaOH, nitrogen-free). Cool; dilute to mark, stopper, and mix by inversion. **Do not degas this reagent.**

7.2.2 Sodium Hypochorite Solution: In a one liter glass bottle, mix 219mL of Regular Ultra Clorox bleach [6% sodium hypochlorite NaOCl, Clorox Company, Oakland, CA] and 281mL of deionized water. Cap the bottle and invert a few times to mix.

7.2.3 Ammonia Buffer: In a 1L volumetric flask containing about 600mL of deionized water, dissolve 50.0g disodium ethylenediamine tetraacetate (Na_2EDTA) and 5.5g sodium hydroxide (NaOH). Dilute to the mark, stopper, and invert to mix.

7.2.4 Sodium Nitroprusside: In a 1L volumetric flask containing about 600mL of deionized water, dissolve 3.50g sodium nitroprusside (Sodium Nitroferricyanide [$Na_2Fe(CN)_5NO{\cdot}2H_2O$]). Once dissolved, dilute to the mark with deionized water. Stopper the flask, and invert several times to mix.

7.2.5 Stock Standard 1000mg N/L as NH_3: Purchase a 1000ppm certified standard (Lab Chem; Fisher Scientific Catalog # LC17940-1).

7.2.6 100mg/L NH_3 Intermediate Stock Standard: To a 100mL volumetric flask, add 10.00mL of the 1000mg/L ammonia stock standard and dilute to the mark with deionized water. Stopper and mix by inversion.

7.2.7 10.0 mg/L NH_3 Intermediate Stock Standard: To a 100mL volumetric flask, add 1000μL of 1000mg/L ammonia stock standard and dilute to the mark with deionized water. Stopper and mix by inversion.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Plastic/ Glass	250mL	H_2SO_4, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136.3
Soils	Plastic/ Glass	50g	Cool 4 ± 2°C	28 Days	N/A

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Initial Calibration (NOT DISTILLED)	Daily	R≥ 0.995	If R value is not acceptable, re-analyze standards; if still problematic, check stock standards and reagents
Laboratory Control Sample (LCS) (DISTILLED)/ Laboratory Control Duplicate (LCD) (optional)	One per sample distillation batch of 20 field samples or less	85-115% RPD ≤20%	Identify problem and redistill all samples.
Initial Calibration Verification (ICV) (NOT DISTILLED)	One per analytical run	90-110%	Re-analyze; if still out recalibrate.
Initial Calibration Blank (ICB) (NOT DISTILLED)	One per analytical run	<0.1mg/L	Re-analyze; if still out, clean glassware and begin analysis over
Matrix Spike (MS) (DISTILLED)	One per distillation batch.	75-125%	If recovery is outside the QC criteria and the Blank Spike is within the acceptable QC limit, the recovery problem is judged to be either matrix or solution related and no further action is needed.
Matrix Spike Duplicate (MSD) (DISTILLED)	One per distillation batch.	75-125% RPD ≤ 20%	If RPD is out reanalyze the duplicate sample. If still out check original analysis. If matrix problem is encountered analyze an additional sample duplicate of another sample in the batch.
Continuing Calibration Verification (CCV) (NOT DISTILLED)	Every 10 samples and after the last sample of the analysis batch	90-110%	Re-Analyze another aliquot; if still out recalibrate and reanalyze all samples not bracketed by passing CCV.
Continuing Calibration Blank (CCB) (NOT DISTILLED)	Every 10 samples and after the last sample of the analysis batch	<0.1mg/L	Re-analyze; if still out, clean glassware and begin analysis over at last passing Cont. Cal. Blank

9.1.1 Annual Quality Control Requirements

ITEM	FREQUENCY	QC CRITERIA	REQUIREMENTS
Reporting Limit (RL)	n/a	0.10mg/L (aq) or 5mg/Kg (solid) [Equal to lowest working curve standard]	RL must be ≥calculated MDL
Method Detection Limit - Calculation (MDL)	Once per year	MDL must be ≤ RL	Spike 7 lab prepared samples at between 3 and 5 times the lowest working curve value to determine MDL.
Demonstration of Capability (DOC)	Once per year per analyst performing the analysis	75-125%	Analysis of 4 laboratory prepared samples ±25% of true value to demonstrate proficiency.
Varied CCV Concentration	Once per year	90-110%	A CCV at a concentration level different than that routinely used, and record this evaluation in the CCV file.

- Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.
- All data, regardless of regulatory program or level of reporting (Level1, 2, 3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.
  - Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.
  - Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.
  - Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.
  - The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-

conformances by using QA memos generated during primary and secondary review.

- Corrective Action for out of Control Data
  - If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

- Contingencies for Handling out of control or Unacceptable Data
  - If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

10.0 Procedure

NOTE! Perform steps 10.1.1.1 and 10.1.2.1 as quickly as possible to avoid loss of ammonia.

10.1 Preparation of Sample / Distillation Procedure

10.1.1 Water Samples

10.1.1.1 Measure 50mL of sample using a Class A graduated cylinder, and pour into a clean, 120mL plastic bottle. Adjust the pH of the sample aliquot to pH 9.5 by adding, drop wise, 15 *N* NaOH with gentle mixing, checking the pH periodically with narrow-range pH paper. Add 2.5mL borate buffer solution and swirl gently to mix. Check the pH and re-adjust to final pH of 9.5 as necessary.

10.1.2 Sediment/Soil Samples

10.1.2.1 Weigh out approximately 1.0g of sample and record the actual weight. Transfer the sample to a boiling tube and add 50mL of deionized water. Add four drops of 15 *N* NaOH, and then 2.5mL of borate buffer.

10.1.3 Transfer the pH-adjusted samples from the 120mL bottles into individual boiling tubes. Add 5-10 micro-porous carbon boiling chips to each. **IMPORTANT!** Use only the carbon chips, not Teflon or glass beads for distillation. Assemble the distillation apparatus according to the EASY-Dist manual procedure for ammonia.

10.1.4 Place 5.0mL of 0.04 *N* H_2SO_4 in 50mL receiving low-form graduated cylinder. Place a nipple and tube assembly into each graduated cylinder, then carefully work the graduated cylinder under the condenser stem outlet, pushing the nipple up to connect with the condenser stem. When properly assembled, the condenser tip of the distillation apparatus will be submerged in the receiving solution contained in the graduated cylinder.

> **NOTE:** Due to the minimal clearance between the condenser stem and the bottom of the graduated cylinder it is easier to place the stem in the cylinder, slide the cylinder under the condenser and then connect the stem to the condenser.

10.1.5 Set heater block temperature controls to the following settings:

RATE1:	15°C/min	RATE2:	0
TEMP1:	210°C	TEMP2:	210°C
TIME1:	2.0 Hour (max)	TIME2:	0

Timing Configuration: CONF1

The rate of distillate collection may vary with laboratory and condenser water temperatures. Use of aluminum foil instillation allows distillation of 20-30mL of distillate in one hour or less. The final volume of distillate may be collected in a shorter or longer period of time, adjust the TIME1 value accordingly, or manually stop the program.

10.1.6 Allow the distillation to proceed until a minimum of 25mL of distillate (total volume of 30mL in the graduated cylinder) as been collected. **NOTE!** Some tubes will distill faster than their neighbors. When a tube is finished distilling, remove the stopper at the top of the T-joint to let the steam vent. Prevent the tube from boiling to dryness by adding deionized water from a swish bottle periodically until all tubes have finished distilling.

10.1.7 Once all tubes have finished distilling, discontinue heating. Remove the stopper from the top of the sloped T-joint (if you haven't already done so for all tubes). The stopper removal is needed to prevent the collected distillate from being sucked back into the apparatus during cooling. Add some deionized water from a swish bottle so that the boiling tubes do not heat to dryness and fracture.

10.1.8 Disconnect the stem from the condenser. Remove the graduated cylinder from the apparatus and dilute the collected distillate to final volume of 50mL with deionized water in the collecting graduated cylinder for each respective sample.

10.1.9 Neutralize the distillate pH prior to analysis with 1.0 *N* NaOH to a pH of between 6 and 8, checking the pH with narrow range test paper.

10.2 Analysis of Samples:

Matrix	Sample Size
Waters	~50 mL of sample or a dilution
Soils/Wastes	1g/ 50mL DI water

10.2.1 Set up the QuikChem 8000 according to the methodology for ammonia as given in the QuikChem manual, using the proper chemistry manifold and connecting the appropriate tubing to the pump and manifold.

10.2.2 Create a new batch in the controlling software for the QuikChem 8000 for the analytical run. Each batch should be in the following format, where a method blank (MB), LCS, and a MS/MSD sample are run once per every 20 samples analyzed. A CCV/CCB combination is run every ten sample positions on the autosampler, and to close out the analytical run:

ICV
ICB/MB
LCS1
LCSD1 (optional)

Sample #1
Sample #1 MS
Sample #1 MSD
Sample #2
...
Sample #8
CCV1
CCB1
Sample #9
Sample #10
...
Sample #18
CCV2
CCB2
MB2
LCS2
LCSD2 (optional)
Sample #19
Sample #19 MS
Sample #19 MSD
...
CCV3
CCB3

10.2.3 Initiate the automatic analytical run using the "Run Batch" button in the controlling software. Working standards are loaded first on the autosampler from highest standard to lowest.

10.2.4 After the analytical run is complete, generate an analytical batch printout of the data to obtain the ammonia results in mg/L for each investigative and QC sample. Upload the data directly into TALS and submit the copy of the data with the review package for 2nd-level review.

10.2.5 After the run is complete, flush the pump tubing and chemistry manifold for 10 minutes with deionized water to purge any residual chemicals or sample from the system.

10.3 **Calibration**

10.3.1 All samples and certain QC samples must be distilled prior to analysis. See section 10.1 for the description distilled QC samples.

10.3.1.1 Prepare the reagents and standards as aforementioned.

10.3.2 Prepare the working standards as given in the table below.

Working Stock Standard Concentration	Volume of Working Solution	Final Volume in Distilled Water	Final Standard Concentration
XX	0mL	100mL	0 mg/L
10.0mg/L	1.0mL	100mL	0.10mg/L
10.0mg/L	5.0mL	100mL	0.50mg/L

10.0mg/L	10.0mL	100mL	1.0mg/L
100mg/L	2.0mL	100mL	2.0mg/L
100mg/L	8.0mL	100mL	8.0mg/L
100mg/L	20.0mL	100mL	20.0mg/L

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≤20% RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	Prior to/after every 10 injections

11.0 Calculations / Data Reduction

Upload the data into TALS directly. Print a copy of the analytical batch to accompany the review paperwork.
NOTE! Sample preparation and any applicable analytical dilutions must be entered into the TALS distillation batch for the system to properly calculate final concentrations and reporting limits.

11.1 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

11.2 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.3 Concentration = mg/kg or L = $\frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually, when a new instrument is put into production or a change in procedure is implemented.

12.2 Demonstration of Capabilities

Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. At least four aliquots shall be prepared (including any prep processes) and analyzed according to test method (either concurrently or over a period of days)

12.3 Training Requirements

- Analysts must read and understand this SOP before performing any distillation and/or analysis.
- The Department Manager and QA Manager must document this training in the analysts' training files.
- The department manager has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- Acceptable IDOC and annual DOC

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to WE-SAF-002.

- Alkaline waste generated by the Lachat auto-analyzer:
  - The waste, consisting of sample diluted with minute quantities of reagents, is collected in appropriately labeled 1gallon plastic bottles. The waste is then disposed into the "BASE/Cyanide Waste" drum in the hazardous waste room after the analysis is complete.

15.0 References / Cross-References

- QuikChem Method 10-107-06-1-A, Zellweger Analytics, revised May 20, 1997 (EPA approved).
- *Standard Methods for the Examination of Water and Wastewater*, 19th ed., 1995. Method 4500NH_3B (distillation) and 4500NH_3G (automated analysis). PP.4-78 to 4-82.

16.0 Method Modifications:

Item	Method xx	Modification
Sequentially number	*Include method references*	*Describe modification and reasons for modification. It may also be appropriate to reference where data is located to support modification.*

NA

17.0 Attachments

Attachment 1: None

18.0 Revision History

Chronological tracking of revisions.

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STANDARD OPERATING PROCEDURES

Analytical Procedure for the Analysis of Chemical Oxygen Demand (COD)

SOP ID: INS04300MA

Revision (5)

EPA Method 410.4
Standard Methods 5220D

Date Written: 02/23/99
Revision Date: 2/8/07
Effective Date: 2/8/07

Approved Signatures:

QUALITY ASSURANCE OFFICER

LABORATORY DIRECTOR

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1.0 TITLE : Analytical Procedure for the Analysis of Chemical Oxygen Demand (COD)

2.0 MATRIX: Surface waters, domestic and industrial wastes with low demand characteristics.

3.0 METHOD REPORTING LIMIT: Aqueous samples: 20mg/L

4.0 SCOPE and APPLICATION

4.1 This procedure is applicable to most water matrices as well as domestic and industrial wastes.

5.0 SUMMARY

5.1 Aqueous blanks, QC and investigative samples are measured and placed into sealed tubes that are heated in a block digester in the presence of sulfuric acid, mercury salts, and potassium dichromate at 150°C to permit oxidation of the dichromate. The amount of chemical oxidation changes the yellow-orange colored dichromate ion to the green chromic ion in proportion to the total chemical oxygen demand of the sample; the green color is measured spectrophotometrically at 600nm and a COD value determined for a sample as compared to a set of working standards.

6.0 DEFINITIONS

Chemical Oxygen Demand (COD): A measure of the oxygen-consuming capacity of inorganic and organic matter present in wastewater. COD is expressed as the amount of oxygen consumed in mg/l. Results do not necessarily correlate to the biochemical oxygen demand (BOD) because the chemical oxidant may react with substances that bacteria do not stabilize.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: an aliquot of reagent water to which the same reagents are added as are added to the investigative and/or quality control samples.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Issue summarized in a NC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. These can be inhibited by the addition of silver salts to the digestion matrix.

7.2 Biologically active samples should be tested as soon as possible or preserved for analysis for a later time. See preservation requirements. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.

7.3 Collect the samples in glass bottles, if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, STL Westfield Safety Addendum and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The disposable glass COD digestion tubes used in this analysis contain Mercuric Sulfate. A compound composed of Mercuric Oxide Red, which may affect the central nervous system, and Sulfuric acid

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercuric Oxide, Red	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 HACH Block Digester set at 150°C.
9.1 Spectrophotometer suitable for measurement at 600 nm using a 1cm path size
9.2 Analytical glassware
9.3 Eppendorf 100-1000μL pipeter
9.4 Eppendorf 10-100μL pipeter
9.5 Eppendorf fixed volume pipeters
9.6 Disposable pipeter tips
9.7 10mL or 15mL disposable sample cups
9.8 Test tube racks
9.8 Mechanical timer capable of timing a 120 min period

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1 Deionized Water
10.2 1,000mg/L Stock COD (Potassium Hydrogen Phthalate) Standard: Purchase the standard commercially (HACH Catalog #22539-29).
10.3 300mg/L Stock COD (Potassium Hydrogen Phthalate) Standard: Purchase the standard commercially (HACH Catalog #12186-29).
10.4 COD (0-150mg/L range) digestion vials with mercury (HACH Catalog # 21258-15)

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Samples must be preserved to pH $\leq$2 with H_2SO_4 and must be analyzed within 28 days.

12.0 QUALITY CONTROL

12.1 Quality Control Evaluation

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Initial Calibration	One per analytical batch	R $\geq$0.995	Re-analyze standards; if still problematic, check stock standards and reagents
Method Blank (MB)	One every 20 samples or fraction thereof	< 20mg/L (RL)	Re-analyze
Laboratory Control Sample (LCS)	One every 20 samples or fraction thereof	%R 85-115%	Analyze a new aliquot of LCS; if still out check stock std. Solution
Laboratory Control	One every 20 samples	%R 85-115%	Re-analyze; if precision criteria

Duplicate (LCSD) (optional)	or fraction thereof	RPD ≤20%	still not met determine if matrix or method is cause.
Initial Calibration Blank (ICB)	One per analytical batch	< 20mg/L (RL)	Re-analyze
Initial Calibration Verification (ICV)	One per analytical batch	90-110%	Re-analyze; if still out recalibrate
Continuing Calibration Verification (CCV)	One every 10 samples analyzed, and to close out the analytical run	90-110%	Re-analyze; if still out recalibrate and reanalyze all samples not bracketed by passing CCV
Continuing Calibration Blank (CCB)	One every 10 samples analyzed, and to close out the analytical run	<20mg/L (RL)	Re-analyze; if still out recalibrate and reanalyze all samples not bracketed by passing CCB
Matrix Spike (MS)	One every 20 samples or fraction thereof	%R 75-125%	If MS recovery indicates a suppressive interference the sample and MS should be diluted and re-analyzed.
Matrix Spike Duplicate (MSD)	One every 20 samples or fraction thereof	%R 75-125% RPD ≤20%	Re-analyze; if precision criteria still not met determine if matrix or method is cause.
Demonstration of Capability (DOC)	Once per year per analyst performing the analysis	85-115%	Analysis of 4 LCSs, ±15% of true value to demonstrate proficiency.
Varied CCV Concentration-NELAC Requirement	Once per year	90-110%	A CCV at a concentration level different than that routinely used, record this evaluation in the CCV file.

13.0 CALIBRATION and STANDARDIZATION: See Procedure.

14.0 PROCEDURE

14.1 Sample Preparation and Digestion

14.1.1 Select a number of COD vials for performing the analytical batch and place them in a test tube rack. Number each tube's top sequentially with a permanent marker pen. On the bench sheet, write in the vial number next to each sample to be prepared so that each standard and sample has a corresponding, uniquely identified vial assigned to it.

14.1.2 Prepare a set of working standards using the 1000mg/L COD Standard as stock according to the table below, producing a final volume of 2.0mL in each vial. When done adding liquid, screw the cap onto each vial finger-tight and mix by inversion.

Amount of 1000mg/L standard added to the COD vial (μL)	Amount of Deionized water added to the COD vial (μL)	Working standard concentration (mg/L)
0	2000	0
40	1960	20
80	1920	40
160	1840	80
240	1760	120
300	1700	150

14.1.3 Place 2.0mL of sample into an appropriately labeled COD vial. If the solution turns green, the COD value is out of range and the sample will need to be diluted. (Dilute samples in 4X and 10X dilutions to start, increasing the dilution as needed so that the solution does not turn green before digestion.) The final volume of sample must be 2.0mL—make up to the volume as necessary with Deionized water.

14.1.4 In addition to the standards and investigative samples, prepare the following QC samples:

14.1.4.1 Prepare one method blank (MB) per 20 samples, or fraction thereof, by placing 2.0mL of Deionized water into an appropriately numbered COD vial. This sample doubles as the ICB

14.1.4.2 Prepare one CCB per 10 samples, or fraction thereof, by placing 2.0mL of Deionized water into an appropriately numbered COD vial.

14.1.4.3 Prepare one ICV per analytical batch by placing 700μL of the 300mg/L COD standard into an appropriately numbered COD vial and adding 1300μL of Deionized water for an ICV of 105mg/L.

14.1.4.4 Prepare one LCS per 20 samples, or fraction thereof, by placing 200μL of the 1000mg/L COD standard into an appropriately numbered COD vial and adding 1800μL of Deionized water for a LCS of 100mg/L.

14.1.4.5 If requested by the client, prepare one LCSD per 20 samples, or fraction thereof, per 14.1.4.4 above.

14.1.4.6 Prepare a MS/MSD per 20 samples, or fraction thereof, by adding 100μL of the 1000mg/L COD standard to a COD vial containing 2.0mL of a sample, for a spike concentration of 50mg/L.

14.1.4.7 Prepare one CCV per 10 samples to be analyzed by placing 200μL of the 1000mg/L COD standard and 1800μL of Deionized water into an appropriately numbered COD vial for a CCV of 100mg/L.

14.1.5 Cap the vials finger-tight. Invert each vial to mix the contents and then place the vial in a pre-heated digestion block set at 150°C. Heat vials for 120 minutes, recording the start and stop times for heating on the bench sheet.

14.1.6 If any of the vials turn green with an intensity greater than the highest working standard when compared visually against a white background, dilute the sample and digest the new dilution as described above. Record any dilution information on the bench sheet.

14.1.7 Once all of the vials have cooled to room temperature, determine the abs of each vial's contents on a spectrophotometer set at 600nm using a 1cm cuvette, or if available use the HACH DR2800 to read the contents of each vial directly at 600nm. **NOTE!** Avoid shaking or undue agitation of the COD vials, which may cause precipitated material at the bottom of the tube to be re-suspended. If there is visible suspended material in any of the COD vials, centrifuge the vial for 10 minutes to remove the suspended interfering material.

14.1.8 Record each sample abs value on the bench sheet. Samples should be analyzed in the following order:

ICV
ICB
MB1
LCS1
LCSD1 (only performed on client request)
Sample #1
Sample #1 MS
Sample #1 MSD
Sample #2
...
Sample #7
CCV1
CCB1
Sample #8
Sample #9
...
Sample #17
CCV2
CCB2

MB2
LCS2
LCSD2 (only performed on client request)
Sample #18
Sample #18 MS
Sample #18 MSD
...

15.0 CALCULATION

15.1 Sample Analyte Quantitation

15.1.1 Use the generic Excel spreadsheet for determining the a, b, and c values for the polynomial fit for the working curve, then enter these values, together with the abs values and dilution factors for all samples, on the spreadsheet in order to have Excel calculate the COD values for each sample.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control

samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's Chain of Custody? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective

action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention" and according to the facility hazardous waste procedures.

22.0 REFERENCES

22.1 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, Method 410.4 (March 1983).
22.2 "Standard Methods for the Examination of Water and Wastewater," 18th Edition, Method 5220D.
22.3 "Water Analysis Handbook," HACH Company, 1989; pp.468-477.

23.0 TABLES, DIAGRAMS, FLOWCHARTS VALIDATION DATA

None

Corrections and/or additions:

Section 10.0 removed thee 800 mg/L standard

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STANDARD OPERATING PROCEDURES

Analytical Procedure for the Total Carbon, Non-purgeable Organic Carbon, Total Organic Carbon and Total Inorganic Carbon

Standard Methods 5310B
EPA Method 415.1

SOP ID: INS06900.MA
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QUALITY ASSURANCE OFFICER

LABORATORY DIRECTOR

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STANDARD OPERATING PROCEDURE

1.0 TITLE: Total Carbon, Non-purgeable Organic Carbon, Total Organic Carbon and Total Inorganic Carbon

2.0 MATRIX: Aqueous samples from drinking water, surface, domestic and industrial wastes.

3.0 METHOD DETECTION LIMIT: 1.0mg/L, MDL is estimated at 0.4mg/L.

4.0 SCOPE and APPLICATION:

4.1 This method is applicable to the measurement of Total Carbon, Non-purgeable Organic Carbon, Total Organic Carbon and Total Inorganic Carbon in aqueous matrices.

5.0 SUMMARY:

5.1 The amount of carbon in a sample is determined using an autoanalyzer. A sample is introduced to the autoanalyzer where it is heated to between 800 and 900°C in an oxygen atmosphere. This serves to transform all carbon to carbon dioxide, which is then carried through the autoanalyzer by the carrier gas ("zero-grade" air) to an infrared detector. An electric signal is produced from detector in response to the carbon dioxide detected, and this signal is integrated and compared to a working curve of standards for the range of carbon desired to determine the amount of carbon in the sample. Various types of carbon may be determined based on whether or not the sample is acidified, purged with zero-grade air before sampling, or otherwise treated: these include Total Carbon (TC), Non-purgeable Organic Carbon (NPOC), Total Organic Carbon (TOC), and Total Inorganic Carbon (TIC). For most aqueous samples, the Volatile Organic Carbon (VOC) component is minute, so that a result for NPOC is often assumed to be equal or similar enough to the TOC result for the latter to be reported as the former, and this is therefore standard procedure for STL—Westfield unless otherwise specified or requested by the client.

6.0 DEFINITIONS:

Total Organic Carbon (TOC): The carbon measured as a result of oxidation of the sample after the removal of inorganic carbon.

Total Inorganic Carbon (TIC): The difference between Total Carbon and Total Organic Carbon.

Total Carbon (TC): The carbon measured as a result of oxidation of the unpreserved and untreated sample.

NPOC: For purposes of this method, non-purgeable organic carbon (NPOC) is equivalent to the measurement of TOC.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An

analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: an aliquot of reagent water to which the same reagents are added as are added to the investigative and/or quality control samples.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

7.0 INTERFERENCES

7.1 The high temperature of combustion should cause 99+% of the carbon to convert to detectable carbon dioxide. In some instruments, high salt content in samples may suppress or adversely affect the results, and such samples suspected of having more than 1% total dissolved solids (TDS) are usually diluted to <1% TDS before being analyzed, and the sample reporting limit (RL) be appropriately increased by the dilution factor. However, with the liquiTOC does not actually require this dilution of high salt-content samples. [The only problem is that the reactor tube needs to be rinsed free of the salts after every 90 samples when analyzing brines or sea water.]

8.0 SAFETY

8.1 Procedures shall be carried out in a manner that protects the health and safety of all STL employees.

8.2 Eye protection that satisfies ANSI Z87.1, a laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Appropriate gloves for the inorganic department and sample receiving shall be made of 100% powder-free, non latex and composed of Nitrile.

8.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

8.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

8.5 The preparation of standards and reagents should be conducted in a fume hood with the sash closed as far as the operation will permit

8.6 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL employee. The situation must be reported **immediately** to a laboratory supervisor.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS/EQUIPMENT

9.1 liquiTOC Analyzer, computer workstation, autosampler, and associated peripherals and components
9.2 Teflon autosampler cups (reusable)
9.3 Deionized water-rinsed plastic 18mL plastic autosampler cups
9.4 Eppendorf 200μL autopipeter (or 100-1000 μL autopipeter)
9.5 Disposable pipeter tips
9.6 Tin foil squares
9.7 Volumetric glassware, triply rinsed with fresh Deionized water
9.7 Two-stage regulator for "zero air" compressed air cylinder

10.0 REAGENTS

10.1 Hydrochloric acid, concentrated (HCl), trace metals grade or betterDeionized water (fresh from the system)
10.2 "Zero-grade" air
10.3 <u>1000mg/L TOC Stock Standard:</u> Dry about 5g of anhydrous potassium hydrogen phthalate (KHP; also known as potassium biphthalate) in an oven at 103°C for 1 hour, and then allow it to cool to room temperature in a desiccator. Place 2.1254g of the oven-dried KHP into about 900mL of fresh deionized water in a 1L volumetric (that has first been triply rinsed with deionized water). Add 1mL of concentrated HCl to preserve. Stopper and mix by inversion or mechanically with stir bar and stir plate until the crystals are dissolved. Bring the solution to the mark with deionized water, stopper, and mix by inversion. (If a stir bar has been used, first remove the stir bar then bring the volume to the mark.) 1.00mL = 1.00mg of carbon. Store in brown glass. EXP: 12 months. Keep refrigerated.
10.4 <u>100.0mg/L Intermediate TOC Standard:</u> Using class A glassware, pipet 10.00mL of the 1000mg/L TOC Stock Standard into a 100mL volumetric flask (that has first been triply rinsed with deionized water) that contains about 50mL of fresh deionized water. Add 8 drops of concentrated HCl. Dilute to mark with deionized water, stopper, and mix by inversion. EXP: 6 months. Keep refrigerated.
10.5 <u>ICV TOC Stock Standard (second source standard):</u> Purchase a second source Phthalate concentrate, such as Inorganic Venture 1000ppm Phthalate (Catalog #ICKHP1-1: this contains 58.5% carbon, making it a 585mg/L Carbon standard). Alternatively, prepare a 1000mg/L TOC standard from a separate lot of oven-dried KHP as described in item 10.3.

10.6 23.4mg/L ICV Working Standard: Using class A glassware, transfer 4.00mL of the 1000ppm Phthalate purchased ICV stock standard to a 100mL volumetric flask (triply rinsed with Deionized water) containing about 80mL of fresh deionized water. Add 8 drops of concentrated HCl. Dilute to mark with deionized water, stopper, and mix by inversion. *EXP: 2 months. Keep refrigerated.*

10.7 32.0mg/L LCS/CCV Standard: Using class A glassware, transfer 8.00mL of the 1000mg/L TOC Stock Standard to a 250mL volumetric flask (that has first been triply rinsed with deionized water) containing about 200mL of deionized water. Add 16 drops of concentrated HCl. Dilute to mark with deionized water, stopper, and mix by inversion. *EXP: 2 months. Keep refrigerated.*

10.8 TOC Working Curve Standards: Using the 1000mg/L stock standard, create the following working curve standards. To each 100mL flask, add 8 drops of concentrated HCl to acidify and exclude dissolved carbon dioxide.

Amount of **100.0mg/L Intermediate** Standard	Amount of **1000mg/L Stock** Standard	Final Volume	Concentration of Working Standard
X	X	100mL	0 mg/L
1000µL	X	100mL	1.0
2000 µL	X	100mL	2.0
5.00mL	X	100mL	5.0
X	1000µL	100mL	10.0
X	2000 µL	100mL	20.0
X	4.00mL	100mL	40.0

11.0 PRESERVATION AND HOLD TIME

11.1 Aqueous or soil/solid samples should be collected in amber glass. For TOC analysis, preserve aqueous samples with 1+1 HCl so that the pH<2. For all other types of carbon analysis, *do not preserve* samples. Soil/solid samples are never preserved. All types of samples, aqueous or soil/solid, for whatever carbon type, should be held refrigerated at <6°C. Aqueous and soil/solid samples may be held for up to 28 days.

12.0 QUALITY CONTROL

QC ANALYSIS	**FREQUENCY**	**QC CRITERIA**	**CORRECTIVE ACTION**
Initial Calibration	Minimum of a Weekly Calibration	R≥ 0.995	Re-analyze standards; if still problematic, check stock standards and reagents
Initial Calibration Verification (ICV)	One per analytical batch	90-110%	Re-analyze; if still out recalibrate
Initial Calibration Blank (ICB) and Method Blank (MB)	One per analytical batch	<RL	Re-analyze; if still out begin analysis over

Laboratory Control Sample (LCS)	One per 20 samples or fraction thereof	85-115%	Analyze a new aliquot of LCS; if still out check stock std.
Matrix Spike (MS)	One per 20 samples or fraction thereof	75-125%	If MS recovery indicates a suppressive interference the sample and MS should be diluted and re-analyzed. If the addition of the spike extends the concentration beyond the calibration curve, the sample should be diluted and the calculated results adjusted accordingly.
Matrix Spike Duplicate (MSD)	One per 20 samples or fraction thereof	RPD ≤20%	Re-analyze; if precision criteria still not met determine if matrix or method is cause
Continuing Calibration Verification (CCV)	One every 10 samples analyzed, and to close out the analytical run	90-110%	Re-analyze another aliquot; if still out recalibrate and reanalyze all samples not bracketed by passing Cont. Cal. Std.
Continuing Calibration Blank (CCB)	One every 10 samples analyzed, and to close out the analytical run	<1.0mg/L	Re-analyze; if still out, clean glassware and begin analysis over at last passing Cont. Cal. Blank.

13.0 PROCEDURE/CALIBRATION/STANDARDIZATION

Total Organic Carbon (TOC)

13.1 Turn the computer and printer on. Once the computer has booted and the Windows desktop is displayed, turn the autosampler on, and then the analyzer. Open the valve on the zero-grade air tank and check the reading to be sure about 15psi of air is flowing into the line to the analyzer. **NOTE!** The autosampler carousel will rotate as it performs a check; do not remove or replace the carousel during this time. Furthermore, the analyzer will undergo a test—you can hear the syringe moving for about 2 minutes. Wait until that is complete before launching the operating software.

13.2 Launch the liquiTOC software by clicking on the desktop icon. Enter a "2" for the password in order to enter the software.

13.3 Check the Status Pane in the operating software—it should turn from red to green to indicate optimal conditions. If not, proceed to the troubleshooting section of the manufacturer's manual, or call tech support [Scott Hughes, elementar Americas, Inc at (856) 787-0055 x202].

13.4 The Status Pane has a number of bar graphs indicating the status of the current operating system. In standby mode and once the instrument has equilibrated, the gas flow at the Massflow and Flowmeter should be about 200mL/min±10mL/min, and the pressure should be about 1.0±0.3 bar; if not, adjust the gas flow at the zero-grade air tank as necessary. **NOTE**: If the Massflow and Flowmeter differ by more than 10 mL/min, it may be a sign of a leak in the system.

13.5 To create an analysis batch, first orient the autosampler controls so that the probes are in the washing well of the autosampler as follows:

13.5.1 Click on the little probe icon with the up-arrow indicated in the menu bar.

13.5.2 Alternatively, from either the Sample Pane Text or Sample Pane Graphics, select Options→ Diagnostic→ System Test which the produces the flow chart diagram of the physical layout of the analyzer. Next click on the autosampler picture to show a sub-window, which causes the probe arm to swing up and into the washing well. Close out the sub-window and the flow diagram window.

13.6 The software provides a Calibration Wizard to assist with the set up of the working curve, blank and "run-in" samples (which are used to clean the instrument and prepare it for blank determination). Launch the Wizard and enter the following set data for TOC:

13.6.1 # of measurements = 3

13.6.2 # of run-in = 6

13.6.3 # of blanks = 3

13.6.4 Conc range = 20ppm

13.6.5 # cal points = 6

13.6.6 Click on the selection for "Different solutions, unique volume" (this is the default setting)

13.6.7 NPOC conc of parent solution = 40

13.6.8 Click "Finish" to set the analytical run.

13.7 Return to the Sample Pane (Text). In order to make the working curve standards of correct concentration, you must overwrite the default, calculated values assigned by the calibration wizard. To do so:

13.7.1 Under the "NPOC [mg/L]" column, find the first of the three replicates for the first (lowest) working curve standard. Double click on it to bring up a data entry box. Delete the current entry and type a "1" for 1.0mg/L. Press the Enter key and repeat twice more so all three of the replicates now read "1.00."

13.7.2 Continue in a like manner, entering the next standard of "2.00," then "5.00," then "10.0," and finally "20.0": leave the last standard, 40.0 as is.

13.8 You can now enter QC and investigative samples. Start with the head-end QC samples as follows. The "ZZ " prefix (two Zs and a space) is required to tell the instrument that the samples are to be corrected for the intrinsic amount of carbon in the deionized water that was used to create the standards:

13.8.1 ZZ ICV 23,4

13.8.2 ZZ ICB

13.8.3 ZZ MB1

13.8.4 ZZ LCS1 32.0

13.9 To indicate that a correction for the deionized water carbon amount, these line items with "ZZ " will show up in green-colored text. All other items are in blue-colored text. **NOTE** that the MB and LCS (and CCV and CCB) need a unique, incremental number after each to differentiate them in the software; otherwise all LCSs, MBs, CCVs and CCBs will be grouped together, the software assuming they are all the same, single sample.

13.10 Click on the next space open for sample description. A window will appear asking for data input. Type in the identifier for each investigative sample to be run: since each sample is run in triplicate, click the "OK" two more times to set up that sample for triplicate analysis, and then enter the next sample description to be

analyzed. Samples should be analyzed in the following order, with appropriate QC samples as indicated (for frequency and type of QC samples, refer to the section 12.0). **Scan Sample ID numbers whenever possible to avoid mis-keying ID numbers.**

ZZ ICV 23.4
ZZ ICV 23.4
ZZ ICV 23.4
ZZ ICB
ZZ ICB
ZZ ICB
ZZ MB1
ZZ MB1
ZZ MB1
ZZ LCS1 32.0
ZZ LCS1 32.0
ZZ LCS1 32.0
Sample #1
Sample #1
Sample #1
Sample #1 MS
Sample #1 MS
Sample #1 MS
Sample #1 MSD
Sample #1 MSD
Sample #1 MSD
Sample #2
Sample #2
Sample #2
...
Sample #8
Sample #8
Sample #8
ZZ CCV1
ZZ CCV1
ZZ CCV1
ZZ CCB1
ZZ CCB1
ZZ CCB1
Sample #9
Sample #9
Sample #9
Sample #10
Sample #10
Sample #10
...

13.11 A "stop tag" is placed after the <u>first LCS</u>. Do this by selecting System→Stop Tag and entering the number of the line for the last triplicate of the highest standard, then click OK and close out the window. This allows the calibration to be

reviewed and quality control head end samples to be checked before the system continues with analyzing samples.

13.12 Place samples into the autosampler carousel in the order as dictated in the analytical batch, using the reusable PTFE (Teflon) cuvettes that have been rinsed three times with deionized water and air dried. Fill each cuvette with water so that there is about ½ inch of head space. For TOC (NPOC) add 100μL of concentrated HCl to the cuvette, then cover tightly with a tin foil square and place in the carousel in order.

13.12.1 "Run-in" samples are simply Deionized water, as are blanks, ICB/MBs, and CCBs.

13.12.2 Use concentrations for LCS, ICV and CCV that fall between 50 and 75% of the highest working standard. The ICV is to be prepared from a second, separate source to the working curve. The LCS and CCVs are prepared from the same standard as the working curve source.

13.12.3 MS and MSD are prepared by adding 400μL of 1000mg/L TOC standard to 40mL of sample in a 50mL mixing graduated cylinder. Stopper and mixing by inversion. The spiked concentration of MS or MSD is 10.0mg/L.

13.13 Save the batch by clicking on the Computer Disk icon and entering the date of the analysis for the filename in the format MMDDYY. Click "Yes" for saving graphical data.

13.14 Initiate the analytical run by clicking on the green "I" button at the top of the screen. Click "Yes" to start the continuous run, and the analyzer will begin analysis of the samples automatically. (The run can be stopped by clicking the green/red I/O button.)

13.15 Once the curve has been generated, if the Stop Tag has been correctly set after the first LCS, the system stops to permit review of the calibration. To accept and to calibrate the instrument:

13.15.1 Select Math→Calibrate and choose the 20ppm method. Click to close the window and the working curve calibration will appear in the Calibration Pane on the right for review.

13.15.2 Using the arrow button in the menu bar, click once to move to the first data point of the three for the blank. Click the "exclude" (red x) button to exclude this data point. (Policy is to exclude the first of the three replicates done for each analytical sample.)

13.15.3 Click the arrow button three times to move to the first of the next set of data points (for the first working standard). Click the exclude button. Continue this process until the first of each triplicate for each working curve standard has been excluded.

13.15.4 Determine if the acceptance criteria of R $\geq$0.995 by examining the chart data. If acceptable, continue; if not, re-calibrate.

13.15.5 Print the acceptable working standard curve by clicking on the print icon button in the menu bar. A window will appear asking you to title the data: enter the date in format of "MM/DD/YY CURVE" changing the day, month and year to the current one on the date of analysis.

13.15.6 Finally, to accept the curve and continue, click on the icon to forward the page (in the menu bar) and click on "OK" to lock the calibration into the system.

13.16 Since the system has stopped on the end of calibration and has gone to sleep, select Options→Wake Up to put the system back in operating mode.

13.17 Next change the stop tag to the last line of the analytical run. After that, and once the system is awake and is stable, the green "I" button will turn from gray to green; click on the green "I" button in the menu bar to re-start the analytical run in continuous mode with the first investigative sample.

13.18 After the analysis of the samples, the system will go to sleep automatically.

13.19 Evaluate and approve the data as follows:

13.19.1 In the graphics pane, select Math→Statistics→By Name to present the run data.

13.19.2 Click once on the first of the three readings for each QC and investigative sample, in turn, and then click on the small icon button and a red X to exclude that data point. Since a common analytical run includes both high, and low TOC content samples, we use the first of the triplicate analytical data as a "clean out" and thus this data is always discarded.

13.19.3 As a result of clicking on the exclude button for the first of each data point for all samples, the mean value for each sample will be recalculated and displayed automatically.

13.19.4 Print the data by clicking on the print icon. A window will appear asking you to title the data: enter "MM/DD/YY RESULTS."

13.19.5 Click once in the text pane.

13.19.6 Print this run summary as well by clicking on the print icon. A window will appear asking you to title the data: enter "MM/DD/YY RUN LOG."

13.19.7 Click on the Computer Disk icon to save all analytical data to the filename already chosen earlier.

13.20 Because the liquiTOC is so stable, one working curve can be generated for a week's worth of analyses. Simply start off each batch with an ICV, ICB/MB, and LCS; if the data meets QC criteria, the working curve stored in the system is viable. If not, rework the batch to make a new calibration curve as described herein.

Total Carbon (TC) and/or Total Inorganic Carbon (TIC)

13.21 To determine Total Carbon, follow steps 13.1 to 13.20 but **do not add hydrochloric acid** to the samples; the samples must be received unpreserved and analyzed unpreserved.

13.21.1 Samples that contain noticeable turbidity or suspended solids should not be filtered. For such samples, insert a small telfon-coating stir bar (ones reserved for TC analysis) into each PTFE sample cup so that the autosampler will stir and continually homogenize the sample during sample uptake by the instrument.

13.22 To determine Total Inorganic Carbon (TIC), run the sample for both TC and TOC. The difference between the two is TIC as follows:

$$TIC = TC - TOC$$

General Notes

13.23 The liquiTOC goes automatically into "sleep mode" after an analysis. The instrument and autosampler may be left turned on without harm between analyses; if leaving the instrument and autosampler on, it is suggested that the user quit out of the liquiTOC software after an analytical run is complete and the instrument is to be idle. The PTFE sample cups should be triply rinsed with Deionized water once the analytical run is completed, and air dried inverted on lab paper.

13.24 If a sample has a reading outside of the limits of the working curve, dilute the sample by an appropriate factor and analyze. Such samples can be added on to the end of the current analytical batch, with appropriate start-up and ending QC parameters (CCV, CCB, LCS, etc.). Report the final result for any such samples by multiplying the dilution factor times the diluted sample's reading, and adjust the reporting limit (RL) by the same factor.

13.25 Record the total injections counter value (found by clicking on the Maintenance bar graph on the operations screen; the tally is in the lower right corner of the Maintenance window), the type of analysis (TOC W for water, or TOC S for soil/solid), and other run data in the Run Log book for each analytical batch performed.

Troubleshooting & Maintenance

13.26 Troubleshooting

13.26.1 Should a communications error occur, save the current run by clicking on the computer disk icon, and then exit the software. Shut down the liquiTOC, then the autosampler; wait ten seconds, then turn the autosampler on, the liquiTOC on, and after the liquiTOC goes through it's start up (the instrument stops humming), launch the software. Open the file you were working on, at which point the software should automatically set itself to run the next sample in line. Click on the green-red button to re-initiate the automatic run.

13.26.2 Should the baseline be noisy, or a blank sample produce a concentration for TOC that is greater than the RL, the reactor chamber either needs to be cleaned or replaced. See the PM instructions below.

13.26.3 Should the flowmeter and massflow bar graphs not read 200±30 units, or if the two values are not within about 10 units of one another, there may be a flow problem or leak in the fludics of the system. Perform a leak check by selecting System→ Diagnostics→ Leak Test and

following the instructions given. Replace tubing or connectors as needed. If no leak is found, or if you need assistance, call Elementars Americas Inc at the number listed in Section 13.25.4 below.

13.26.4 For any other problems with the instrument, consult with Scott Hughes or technical support at Elementars America, Inc. at (856) 787-0022 x202.

13.27 Routine Preventive Maintenance

13.27.1 Record any maintenance performed in the instrument log book.

13.27.2 The liquiTOC has a maintenance monitoring feature that counts the number of injections and alerts the user as to when to perform routine maintenance on the instrument, or to check various operating conditions. This can be viewed by simply clicking on the "Maintenance" bar graph, located just under the bar graph read outs for the "Flowmeter" and "Massflow." The bar graph for Maintenance changes color to alert the operator that maintenance isn't yet required (green, will soon be required (yellow) and must be performed (red).

13.27.2.1 When open, the Maintenance window shows each key check or maintenance requirement. The frequency for maintenance is pre-set by the manufacturer. When a maintenance item is performed, double click in the corresponding counter space, type a zero ("0") to reset it, then click on the "OK" button to enter the value.

13.27.2.2 The auto-monitored maintenance items are as follows:

Auto-Monitored Maintenance Item	Actions to Be Taken
Refill Deionized Water Container (every 250 injections)	Check the deionized water supply to the instrument; add fresh deionized water if needed.
Check Reactor (every 500 injections)	Check the glass reaction chamber (that contains the platinum catalyst). If it is heavily encrusted with salts, or blocked or heavily discolored, rinse it with deionized water to clean it.
Renew Silver Wool (every 500 injections)	Renew the silver wool and copper metal granules in the halogen scrubber tubes on the inside left of the instrument. Copper granules alone may also be used if no silver wool is available.
Check Furnaces (every 1000 injections)	Check to be sure that the furnaces (the upper and lower zones on the reactor tube) are heating. Under normal operating conditions, the top segment should be 800C (and visibly glowing orange when examined), and the lower segment should ramp up from near room

	temperature to 800C when analyzing a sample.
Clean Reactor (every 1000 injections)	See "Check Reactor" above for cleaning procedure. Note that if the baseline is not stable, or the blanks begin reading a slightly positive value for TOC that is above the RL, the reactor tube and catalyst need to be replaced.
Screw Spindle Grease (every 500 injections)	Use a Kimwipe to wipe clean the screw spindle. Add a small dollop of white grease to the screw thread just above the motor (inside left panel of the instrument).
Waste Jug (every 500 injections)	Empty the waste jug every 500 injections or if it becomes close to being full.
Gas (every 5000 injections)	Replace the zero grade air cylinder every 5000 injections, or if the pressure drops below 500 psi.

13.27.3 In addition to the requirements for Maintenance listed above, perform the following at the frequency indicated, recording the maintenance performed in the instrument log book:

Maintenance Item	Actions to Be Taken
Check Desiccant (once per month)	If the desiccant has turned from blue to pink for more than two-thirds the fill length, replace the desiccant with fresh Magnesium perchlorate (Cat #05-001-059)
Check coil trap filter (once per month)	Check the polyfoam plug at the bottom of the glass coil water trap. If it is very dirty, or has salt deposits built up on it, change it with a new plug (Cat #03-002-399)
Check fluidics (once per month)	Check the lines and connections in both the left and right sides of the instrument to determine if any leaks have occurred. If so, replace worn parts as needed.
Check autosampler and reagent lines (once per month)	Check the lines and connections to make certain that the lines are not kinked or blocked. If any line is kinked or blocked, replace it with fresh

	tubing and/or connectors.
Replace the Reactor Chamber (as needed)	The reactor chamber is good for about 1500-2000 injections for water, or about 1000-1500 injections for soil. If the baseline becomes noisy, or a blank sample produces a TOC value over the RL, it is likely time to change the reactor chamber.

14.0 CALCULATIONS

14.1 The analyzer software automatically calculates the working curve; the linear fit for it and determines the concentration for the investigative and QC samples based on the curve. The data from an analytical run is printed out for documentation, and the data from it transferred to the LIMS system for reporting. All QC criteria must be met, else corrective action and/or acceptance of data with disclaimer is performed.

14.2 Note that it is standard operating procedure to discard the first of the triplicate data points for each sample analysis as cited above.

14.3 For each analytical batch, print a CURVE, RUN, and RESULTS page as described in the analysis procedure.

14.4 TIC is calculated manually from a run that includes both a TC and TOC analysis of the sample (see item 13.22).

15.0 METHOD PERFORMANCE

15.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

15.2 The reporting limit is 1.0mg/L for undiluted aqueous samples as provided by the liquiTOC 20ppm method. Lower detection limits are possible with method modifications to allow for more sample injection, and thus lower RLs.

15.3 Oily samples may cause erratic results. This is minimized by homogenization of the sample.

16.0 POLLUTION PREVENTION

16.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

17.0 DATA ASSESMENT and ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

17.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

17.2 All data, regardless of regulatory program or level of reporting (Level1, 2, 3, or 4), shall be subject to a through review that involves a primary, secondary and completeness review process. All levels of review must be documented.

17.2.1 A primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

17.2.2 A primary review must ensure that: sample preparation is accurate and complete; calculations were performed correctly; quantitation has been performed correctly; identifications are accurate; method SOPs have been followed; QC criteria has been met; dilution factors have been applied correctly; and QA memos have been generated for anomalous results, if necessary.

17.2.3 The secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

17.2.4 The completeness review shall address the following: is the job report complete; does the data meet client requirements; were data quality objectives met; were holding times met; were inter-method relationships reasonable; and was the project completed according to client's COC? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

18.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

18.1 If problems are found upon review with sample data the following shall apply:

18.1.1 All non-conformances that affect sample data shall become part of the affected project's permanent record.

18.1.2 When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

19.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

19.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, and based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

19.2 Any unauthorized deviation from this procedure must be documented as a non-conformance with cause and corrective action described in a QC memo

20.0 WASTE MANAGEMENT

20.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required.

21.0 REFERENCES

A. "Standard Methods for the Examination of Water and Wastewater," 18th Edition, 5310B. pp. 5-17 to 5-19.

B. SW846 Method 9060 "Total Organic Carbon," September 1986.

C. "High Temperature TOC/TN_b Analyzer Operating Instructions," elementar Americas, Inc., March 2003.

D. EPA Method 415.1, "Methods for the Analysis of Water and Waste," EPA-600/4-79-020, MARCH 1983.

Corrections and/or additions:

3/20/07: added control distribution box on cover, section 6-added definitions, revised the following sections: 13.8-13.10, 13.12, 13.12.3, 13.14 and 13.15.4. Removed sections 13.6.8 and 13.7, 13.7.3-13.7.6 sections renumbered.

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STANDARD OPERATING PROCEDURES

Analytical Procedure for the Analysis of Solids, Residue, Non-Filterable (Total Suspended) & Total Volatile Suspended

Standard Methods 2540D
(EPA Method 160.2)[1]

SOP ID: INS00200.MA

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QUALITY ASSURANCE OFFICER

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1.0 TITLE : Analytical Procedure for the Analysis of Solids, Total Suspended & Total Volatile Suspended

2.0 MATRIX: This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

3.0 METHOD REPORTING LIMIT: TSS reporting limit is 5mg/L, TVSS is 10.0mg/L

4.0 SCOPE and APPLICATION

4.1 The method is applicable to drinking, surface, and saline water, and domestic and industrial wastes. The applicable range of determination is 5mg/L to 20,000 mg/L.

5.0 SUMMARY

5.1 A well-mixed sample is filtered through a pre-weighed 0.45μm glass-fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C in order to determine the total suspended solids. This residue is then ignited at 500±0°C cooled, and weighed to a constant weight: the weight lost on ignition is the total volatile suspended solids.

6.0 DEFINITIONS

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is introduced onto the instrument for analysis and can monitor calibration baseline drift and carryover contamination effects.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD: a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Summarized in a Non conformance memo (NCM).

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Samples high in dissolved solids such as saline waters, brines, and some wastes, may produce a positive interference. Non-representative particulates should be removed from the sample.

7.2 Filtration apparatus filter material; pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

8.2 PRIMARY MATERIALS USED

There are no materials used in this method that have a significant or serious hazard rating. NOTE: This list does not include all materials used in the method. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

9.0 APPARATUS

9.1 Glass fiber filters, Whatman™ 2.1cm and 4.25cm circular, 0.45μm pore size, without organic binder
9.2 Aluminum weighing dish, 55mm circular, disposable.
9.3 Filtration apparatus
9.4 Membrane filter funnel
9.5 Suction Flask, 1000mL or greater capacity.
9.6 Analytical Balance capable of weighing to 0.1mg
9.7 Graduated cylinders, 500mL, 250mL, 100mL, and 25mL
9.8 Wide tip graduated pipets, 25mL
9.9 Forceps
9.10 Gooch crucibles
9.11 Vacuum pump
9.12 Drying oven, capable of maintaining 103°C
9.13 Muffle furnace, capable of maintaining 550°C
9.14 Autoclave gloves
9.15 Crucible tongs
9.16 Furnace tongs

10.0 PREPARATION OF WATER QUALITY REAGENTS

10.1 Deionized water
10.2 Whatman™ Ashless Powder.

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Store samples at 4°C and analyze within 7 days.

12.0 QUALITY CONTROL

12.1 Quality Control Evaluation

QC Analysis	Frequency	QC Criteria	Corrective Action
Method Blank (MB)	Per batch of 20 field samples or fraction thereof	<5 mg/L TSS <10mg/L TVSS	If the MB is >5 mg/L, reheat in the oven, desiccate, and take an additional weight reading to determine TSS value; if still >5, contact the QAQC department.
Laboratory Control Sample (LCS)	Per batch of 20 field samples or fraction thereof	%R 85-115%	If out of recovery range, reheat in the oven, desiccate, and take an additional weight reading to determine TSS value; if still out of range, contact the QAQC department. Reanalyze batch
Laboratory Control Duplicate (LCSD) (optional)	Per batch of 20 field samples or fraction thereof	%R 85-115% RPD ≤ 20%	If RPD is >20%, reheat the original and MD in the oven, desiccate, and reweigh; if still >20%, report data qualified by a QA Memo.
Method Duplicate (DU)	Per batch of 20 field samples or fraction thereof	%R 85-115% RPD ≤ 20%	If RPD is >20%, reheat the original and MD in the oven, desiccate, and reweigh; if still >20%, report data qualified by a QA Memo.

13.0 CALIBRATION and STANDARDIZATION: NA

14.0 PROCEDURE

14.1 Preparation of the glass-fiber filter

14.1.1 Choose a filter size appropriate to the analysis: 4.25cm for TSS or 2.1cm in conjunction with a Gooch crucible for TSS and/or VSS.

14.1.2 For TSS, using a pair of forceps, place a 4.25cm filter in the membrane filter funnel. For VSS, using a pair of forceps, place a 2.1cm filter into a uniquely numbered crucible and use the crucible

tongs to place the crucible in the vacuum assembly holder.

14.1.3 Apply a vacuum and wash filter with three successive 10mL volumes of de-ionized water.

14.1.4 For TSS, using the forceps place the washed filter on a numbered disposable aluminum weighing dish. For VSS, remove the crucible from the vacuum assembly using the crucible tongs. Heat either in oven at 103-105°C for 1 hour.

14.1.5 Remove after 1 hour and cool and store the weighing dishes and/or crucibles in desiccator until needed.

14.2 Preparation of LCS and LCSD (LCSD optional)

14.2.1 Prepare a Laboratory Control Sample (LCS) by placing 0.5000g of Whatman ashless powder into a 1L volumetric containing about 900mL of De-ionized water. QS to the mark. Stopper and mix by inversion. A volume of 100mL of this suspension carried through the analysis as indicated in 10.3 will provide a 500mg/L LCS.

14.2.2 Prepare a Laboratory Control Duplicate (LCSD) as indicated in above (optional).

14.3 TSS Analysis

14.3.1 Select a number of aluminum dishes and filters for your analytical run. Make sure none have the same ID number.

14.3.2 Weigh each aluminum dish and filter to the nearest 0.1mg. Record the weights in the tare weight column of the worksheet. Allocate one dish for a method blank (MB), one for a LCS, and one for a LCD. Run one MB, LCS LCSD (optional) and Method Duplicate (DU) per 20 samples analyzed, or fraction thereof.

14.3.3 Using a pair of forceps, remove the first sample's filter from the dish and place it in the filter apparatus. Apply suction and using a deionized water swish bottle, moisten the filter starting at the center and spiraling out to the edges, until the entire surface is wet so that suction is applied uniformly and completely to the filter.

14.3.4 Measure out an appropriate amount of sample using a graduated cylinder or wide-tip graduated pipet. (For effluents run between 100 and 500 mL; influents between 5 and 100 mL; clear surface waters 500-1000mL; noticeably turbid surface or groundwater, 25-100mL.)

14.3.5 Gently pour the sample into the center of the filter with a slow, steady stream. Ideally, the water should flow through the filter without accumulating in the filter funnel.

14.3.6 After the sample has been poured over the filter, rinse the graduate with 10mL of deionized water, and pour this rinse water over the filter.

14.3.7 Wash the filter with three, 10mL portions of deionized water. Allow the filter to air dry for 1 minute under vacuum.

14.3.8 Discontinue vacuum suction. Crack the funnel assembly-to-flask joint to reduce suction. Using the forceps, carefully remove the filter from the funnel assembly and place it back into its respective aluminum weigh dish.

14.3.9 Repeat steps 14.3.3-14.3.8 until all QC and investigative samples have been treated.

14.3.10 Place all weigh dishes containing the filters into an oven and heat at 103-105°C for at least 1 hour. Record the temperature and time at the start and end of the heating cycle. Remove from the oven after this time and cool to room temperature in a desiccator.

14.3.11 Determine the weight of each dish and filter, recording it on the worksheet as the "103°C weight."

14.3.12 Replace the dishes in the oven and heat for at least 1 hour. Record the temperature and time at the start and end of the heating cycle. Remove and cool in a desiccator.

14.3.13 Determine the weight for each dish and filter, recording the second weight in the "103°C weight" column below the first reading. If the weights are constant (≤0.5mg difference) then the sample is ready for reporting. If the weight difference is more than 0.5mg, re-heat for at least 1 hour, cool in a desiccator, and re-weigh. Continue this process until either a constant weight is achieved or a total of five readings are taken.

14.3.14 Using the appropriate Excel spreadsheet, enter the values for each parameter. Record the final TSS reading for each sample from the spreadsheet onto the worksheet, using "<5" for samples that have TSS values below 5.0 mg/L (the analysis RDL).

14.4 VSS Analysis

14.4.1 For VSS, Gooch crucibles with a 2.1cm Whatman 0.45μm filter are used.

14.4.2 Using the crucible tongs to handle them, weigh each crucible and filter to the nearest 0.1mg. Record the weights in the tare weight column of the worksheet. Allocate one crucible for a method blank (MB), one for a LCS, one for a LCSD (optional) and one for a DU. Run one MB, LCS LCSD (optional) and DU per 20 samples or fraction thereof.

14.4.3 Using the tongs, place the crucible for the first sample in the filter apparatus. Apply suction and using a deionized swish bottle, moisten the filter starting at the center and spiraling out to the edges, until the entire surface is wet so that suction is applied uniformly and completely to the filter.

14.4.4 Measure out an appropriate amount of sample using a graduated cylinder or wide-tip graduated pipet. (For effluents run between 100 and 500 mL; influents between 5 and 100 mL; clear surface waters 500-1000mL; noticeably turbid surface or groundwater, 25-100mL.)

14.4.5 Gently pour the sample into the center of the filter with a slow, steady stream. Ideally, the water should flow through the filter without accumulating in the crucible.

14.4.6 After the sample has been poured over the filter, rinse the graduate with 10mL of deionized water, and pour this rinse water over the filter.

14.4.7 Wash the filter with three, 10mL portions of deionized water. Allow the

filter to air dry for one minute under vacuum.

14.4.8 Discontinue vacuum suction. Crack the funnel assembly-to-flask joint to reduce suction. Using the crucible tongs, carefully remove the crucible from the funnel assembly.

14.4.9 Repeat steps 14.4.3-14.4.8 until all QC and investigative samples have been treated.

14.4.10 Place all crucibles into an oven and heat at 103-105°C for at least 1 hour. Remove from the oven after this time and cool to room temperature in a desiccator. Record the temperature and time at the start and end of the heating cycle.

14.4.11 Determine the weight of each crucible, recording it on the worksheet as the "103°C weight."

14.4.12 Replace the crucibles in the oven and heat for at least 1 hour. Remove and cool in a desiccator.

14.4.13 Determine the weight for each crucible, recording the second weight in the "103°C weight" column below the first reading. If the weights are constant (≤0.5mg difference) then the sample is ready for reporting. If the weight difference is more than 0.5mg, re-heat for at least 1 hour, cool in a desiccator, and re-weigh. Continue this process until either a constant weight is achieved or a total of five readings are taken.

14.4.14 Next place the crucibles in a muffle furnace set at 550±50°C for 20 minutes. **WARNING!** Use long crucible tongs and heat protective gloves when using the muffle furnace. Direct skin exposure to the furnace's heat may cause severe first or second-degree burns. Record the temperature and time at the start and end of the heating cycle.

14.4.15 After 20 minutes, remove the crucibles, place them on ceramic tiles, and allow them to air cool to room temperature. Once cool, place in a desiccator for at least 1 hour.

14.4.16 Determine the weight of each crucible, recording it on the worksheet as the "550°C weight."

14.4.17 Replace the crucibles in the furnace and heat for 20 minutes. Record the temperature and time at the start and end of the heating cycle. Remove, air cool, and then dry in a desiccator for at least 1 hour.

14.4.18 Determine the weight for each crucible, recording the second weight in the "550°C weight" column below the first reading. If the weights are constant (≤0.5mg difference) then the sample is ready for reporting. If the weight difference is more than 0.5mg, re-heat for 10 minutes, air cool, dry in a desiccator, and re-weigh. Continue this process until either a constant weight is achieved or a total of five readings are taken.

14.4.19 Using the appropriate Excel spreadsheet, enter the values for each parameter. Record the final VSS reading for each sample from the spreadsheet onto the worksheet, using "<5" for samples that have VSS values below 5.0 mg/L (the analysis RDL).

15.0 CALCULATION

15.1 Sample Analyte Quantitation

Use the appropriate Excel spreadsheet to determine the TSS and/or VSS values, or determine the values manually as follows:

Total Suspended Solids (mg/L) = [(A-B) * 1000] ÷ D

Volatile Suspended Solids (mg/L) = [(E-F) * 1000] ÷ D

Where:

A = dry weight of filter plus residue ("103°C weight") **in mg**
B = weight of filter ("tare weight") **in mg**
D = sample volume used, **mL**
E = weight of filter plus residue ("103°C weight") **in mg**
F = weight of filter plus residue ("550°C weight") **in mg**

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

17.2 There are no materials used in this method that have a significant or serious hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

18.0 DATA ASSESSMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control

samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1, 2, 3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.6 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTABLE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to STL Westfield's facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required. Please refer to section 17- Pollution Control for more details.

22.0 REFERENCES

22.1 "Standard Methods for the Examination of Water and Wastewater," 19th Edition, 1992, methods 2540D and 2540E, pgs 2-56 and 2-57.

22.2 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983, method 160.2.[1]

Corrections and/or additions:

1/10/07: Method reference added to cover sheet.

4/6/07: *Standard Methods* citation changed to 19th edition. Requirement for recording the time of start and end of heating cycle added. References to Item numbers in the Procedure section were corrected.

[1] This method reference is being withdrawn as of April 11, 2007.

(adjusted page settings and margins)

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Uncontrolled Copy

Determination of Hydrazine, Monomethylhydrazine and 1.1-Dimethylhydrazine in Aqueous Samples by LC/MS/MS

Approvals:

Prepared by: _____ Date: 10/29/08
Principal Chemist

Approved by: _____ Date: 10/29/08
Specialty Services Management

Approved by: _____ Date: 10/29/08
Quality Assurance

Revision Log:

Revision: 01		Effective Date:	This version
Section	Justification		Changes
			New

10342_01.DOC
102808

Reference:

1. *Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)*, USEPA 8315A.

2. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, USEPA SW-846.

3. *Hydrazine*, U.S. Department of Labor, Method 108.

4. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Reference (USEPA 8315A) Modifications:

1. LC/MS/MS instead of LC/UV is used for detection and quantification of hydrazine, monomethylhydrazine and 1.1-dimethylhydrazine derivative.

2. Rather than being target analyte, carbonyl compound (benzaldehyde) is used as derivatizing reagent to form hydrazones with hydrazine compounds.

Cross Reference:

Document	Document Title
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-0E-001	Glassware Cleaning for Organic Extractions
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-025	Monitoring of QC Data Acceptance Limits

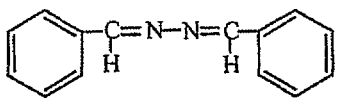
Scope:

The method is applicable for the determination of hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine in aqueous samples. The reporting limits of the three analytes are as followings:

Analyte	Reporting Limit (µg/L)
Hydrazine	0.2
Monomethylhydrazine	0.5
1,1-Dimethylhydrazine	0.5

Basic Principles:

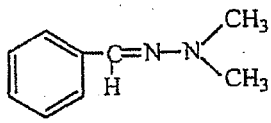
The sample is buffered with an acetate buffer to a pH of approximately 5. Hydrazine, monomethylhydrazine (MMH) and 1,1-dimethylhydrazine (UDMH) are derivatized with benzaldehyde to the corresponding hydrazones: benzalazine, benzaldehyde methylhydrazone and benzaldehyde dimethylhydrazone, respectively. The resulting solution is analyzed by LC/MS/MS operated in atmospheric pressure chemical ionization (APCI) mode for detection and quantification of the hydrazones.



Benzalazine

$C_{14}H_{12}N_2$

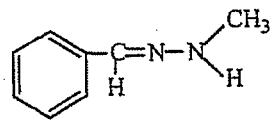
FW = 134.1809



Benzaldehyde Methylhydrazone

$C_9H_{12}N_2$

FW = 148.2078



Benzaldehyde Dimethylhydrazone

$C_8H_{10}N_2$

FW = 208.2628

NOTE: 1,1-Dimethylhydrazine is also known as unsymmetrical dimethylhydrazine.

Interferences:

Compounds which have similar structures to the compounds of interest, and similar molecular weights would potentially interfere. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants. Samples with a pH that is significantly acidic or basic and resists the buffering effect of the acetate buffer may show reduced performance.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Gloves, lab coats, and safety glasses should be worn when preparing standards, handling samples and operating the LC/MS/MS. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact, and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All solvent waste is collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal in to the lab-wide disposal facility. Vials containing expired hydrazine, methylhydrazine and 1,1-dimethylhydrazine standards, and HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a quad study that yielded acceptable recoveries for hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine, or completed another approved demonstration of capability (i.e. PT sample, blind sample, etc.). Personnel should spend several days working with an experienced technician or chemist who has demonstrated their proficiency of the extraction. Also, several batches of water samples should be performed under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Analysts performing the instrumental analysis will work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set-up sequences, perform calculations, interpret the chromatograms and mass spec data, and enter the data into the LIMS. Proficiency is measured through documented audits of the tasks listed and overchecking of data, as well as annual quad studies.

Sample Handling:

Collect water in clean, 40 ml amber glass vials/bottles containing 1.6 mL pH 5.0 acetate buffer (5M) as the preservative. There should be no headspace. Samples must be stored at 2° to 4°C and must be derivatized within 10 days of sampling. Extracts must be analyzed within 5 days of derivatization.

Apparatus and Equipment:

1. pH meter, accurate to 0.1 pH units

2. Sample shaker – Temperature controlled bath with platform shaker

3. Reaction vials – 4-mL glass vials with Teflon™-lined caps

4. Balance – Capable of weighing to 0.0001 g

5. Balance – Capable of weighing to 0.01 g

6. Stir plate

7. Beakers – assorted sizes

8. Volumetric flasks – Class A, assorted sizes

9. Glass pipettes – Class A, assorted sizes

10. Pipettes – Rainin, POS-D™ (Positive-Displacement) Pipette, 1-10 µL and 10-100 µL or equivalent

11. Pipettes – Rainin, Pipete-Lite LTS Pipette, 1-10 mL or equivalent

12. Pipettes – Eppendorf Research® Series Pipette, 10-100 µL and 100-1000 µL or equivalent

13. Graduated cylinders

14. Syringe filter – PALL, Acrodisc CR 25-mm syringe filter with 0.45-PTFE membrane, PN 4501 or equivalent.

15. Disposable polyethylene pipettes – 3 mL, SUN-SRI, Cat. No. 201 631 or equivalent

16. Syringe – 0-500 µL, Hamilton or equivalent

17. 1-liter bottles for mobile phase storage

18. HPLC autosampler vials: Thermo Scientific, 1.8 mL, Cat. No. 60180-205 or equivalent

19. HPLC with dual high-pressure pump and gradient capabilities – Thermo Electron Corporation, Accela or equivalent

20. HPLC autosampler – Thermo Electron Corporation, Accela or equivalent

21. LC/MS/MS equipped with atmospheric pressure chemical ionization source – Thermo Scientific, TSQ Quantum Access or equivalent.

22. HPLC Column – Thermo Scientific Hypersil Gold, 2.1 x 50mm, 3 µm, Part No. 25003-052130 or equivalent

23. Precolumn filter – Upchurch Scientific, Part No. A-315 or equivalent

24. Precolumn filter frit – Upchurch Scientific, 2 µm, Part No. A-101X or equivalent

25. In-line filter frit before injection valve – Analytical Scientific Instrument, 0.5 µm, Part No. 850-1347 or equivalent

26. Data System – Thermo Scientific LCquan for data acquisition, integration, and spectra or equivalent

Reagents and Standards:

A. Reagents

1. Methanol – HPLC grade or equivalent

2. Milli-Q Water

3. Ammonium acetate – ACS grade or equivalent

4. Glacial acetic acid – ACS grade or equivalent

5. Formic acid – ACS grade or equivalent

6. Benzaldehyde – Aldrich, ≥99.5%, Cat. No. 418099 - 100 mL, or equivalent

7. Hexylamine – Acros Organics, 99%, Cat. No. 204720050, or equivalent

8. 1M Hexylamine solution – Add 0.66 mL hexylamine into a 5-mL volumetric flask. Bring to volume with methanol and mix well. This solution is stored in the refrigerator and is stable for 30 days. This may be prepared in larger or smaller volumes.

9. 1% (v/v) Benzaldehyde derivatizing reagent – Add 0.1 mL of benzaldehyde into a glass vial containing 10 mL of methanol and mix well. This may be prepared in larger or smaller volumes depending on the size of the extraction group. This reagent must be prepared fresh daily.

10. pH 5.0 Acetate buffer (5M) - This buffer is stored at room temperature and is stable for 6 months. Prepare as follows:

    a. Add 28.6 mL of glacial acetic acid into a 100-mL volumetric flask containing approximately 50 mL of Milli-Q water. Bring to volume with Milli-Q water and mix well. This prepares 5M acetic acid solution. This solution is stored at room temperature and is stable for 6 months.

    b. Weigh 38.5 ± 0.04 g of ammonium acetate in a beaker. Add Milli-Q water to dissolve and quantitatively transfer it to a 100-mL volumetric flask. Bring to volume with Milli-Q water and mix well. This prepares 5M ammonium acetate solution. This solution is stored at room temperature and is stable for 6 months.

c. Place 60 mL of the 5M ammonium acetate solution into a 200 mL beaker. Add 40 mL of 5M acetic acid solution and mix well. Adjust the pH of the solution to 5.0 ± 0.2 with formic acid as needed.

d. Transfer the buffer to a glass bottle. Record the pH of the buffer on the bottle along with the date prepared and expiration date. The reagent is stable for 6 months.

9. pH 5.0 acetate buffer (0.2M) - To prepare pipette 10.0 mL of 5M acetate buffer into a 250-mL volumetric containing approximately 100 mL of Milli-Q water. Bring to volume with Milli-Q water and mix well. This solution is stored in the refrigerator or at room temperature and is stable for 6 months.

B. Standards

1. Hydrazine stock standard solution – Commercially prepared 100 µg/mL hydrazine standard in methanol (Cat. No. M-8330-ADD-8) can be purchased from AccuStandard, or equivalent vendor. Store in the refrigerator and protect from light. Refer to manufacturer's instruction for expiration date.

2. 1,1-dimethylhydrazine stock standard solution – Commercially prepared 1000 µg/mL 1,1-dimethylhydrazine standard in methanol (Cat. No. S-1627) can be purchased from Spex CertiPrep, or equivalent vendor. Store in the refrigerator and protect from light. Commercially prepared stock standard expires 90 days from date of preparation.

3. Monomethylhydrazine neat standard – Fluka, purum, ≥98.0%, (Cat. No. 418099-100mL) or equivalent vendor. The catalog, purity and lot must be recorded. Store the compound in the flammable cabinet.

4. Monomethylhydrazine stock standard solution, approximately 1000 µg/mL – On an analytical balance weigh ~20 mg neat compound (approximately 24 µL) into a 20-mL volumetric flask. Dilute to volume with methanol. Cap, mix, and label. Store in the refrigerator and protect from light. The stock standard solution expires 4 hours from the preparation time.

5. Standard solutions and spiking solution are prepared as noted below and can be prepared using different initial and final volumes as long as the resulting calibration standards and spiking solution are at the concentrations listed below.

6. Intermediate standard solution – Add 20.0 µL of hydrazine stock standard solution, X µL of monomethylhydrazine standard stock solution and 10.0 µL of 1,1,-dimethylhydrazine stock standard solution to a 20-mL volumetric flask with 2 mL of pH 5.0 acetate buffer (0.2M). Bring to volume with pH-5.0 acetate buffer (0.2M) and mix well. Store in the refrigerator and protect from light. Intermediate standard expires at the end of 1,1-dimethylhydrazine stock standard expiration date. The added volume of monomethylhydrazine stock solution is calculated as follows:

$$X\,(\mu L) = \frac{10{,}000}{\textit{Concentration of monomethylhydrazine stock solution}\ (\mu g/mL)}$$

Standard ID	Hydrazine Conc. (µg/mL)	MMH & UDMH Conc. (µg/mL)	Solvent
Intermediate standard	0.1	0.5	pH-5.0 acetate buffer (0.2M)

7. Working standards – Prepare the following working standards according to the dilution scheme presented below. To 20-mL volumetric flasks containing 2 mL of pH-5.0 acetate buffer (0.2M), add the volumes of intermediate standard solution as described below. Bring to volume with pH-5.0 acetate buffer (0.2M). Store in the refrigerator and protect from light. Working standards expire at the end of 1,1-dimethylhydrazine stock standard expiration date.

Standard ID	Prepared From	Initial Volume (µL)	Final Volume (mL)	Hydrazine Concentration (ng/mL)	MMH* & UDMH** Concentration (ng/mL)	Solvent
Level 1	Intermediate	20	20	0.1	0.5	0.2M Acetate Buffer
Level 2	Intermediate	50	20	0.25	1.25	0.2M Acetate Buffer
Level 3	Intermediate	100	20	0.5	2.5	0.2M Acetate Buffer
Level 4	Intermediate	200	20	1	5	0.2M Acetate Buffer
Level 5	Intermediate	500	20	2.5	12.5	0.2M Acetate Buffer
Level 6	Intermediate	1000	20	5	25	0.2M Acetate Buffer
Level 7	Intermediate	2000	20	10	50	0.2M Acetate Buffer
Level 8	Intermediate	5000	20	25	125	0.2M Acetate Buffer

8. Spiking solution – Add 125.0 µL of hydrazine stock standard solution, Y µL of monomethylhydrazine standard stock solution and 62.5 µL of 1,1,-dimethylhydrazine stock standard solution to a 10-mL volumetric flask with 2 mL pH-5.0 acetate buffer (0.2M). Dilute to volume with pH-5.0 acetate buffer (0.2M), and mix well. Store in the refrigerator and protect from light. Spiking solution expires at the end of 1,1-dimethylhydrazine stock standard expiration date.

$$Y\,(\mu L) = \frac{62{,}500}{\text{Concentration of monomethylhydrazine stock solution } (\mu g/mL)}$$

	Hydrazine Conc. (µg/mL)	MMH & UDMH Conc. (µg/mL)	Solvent
Spiking Solution	1.25	6.25	0.2M acetate buffer

Document all preparation data in the appropriate logbook.

Preparation of Glassware:

See SOP-OE-001.

Procedure:

A. Calibration standards preparation

A calibration curve must be prepared every 1 calendar day.

1. Measure 1.0 mL of the working standard Level 1 through Level 8 into reaction vial, respectively. Label the vials as Level 1 through 8.

2. Add 10.0 µL of 1M hexylamine solution into each reaction vial.

3. Derivatize the standards following the steps listed in Part C.

   NOTE: The response of derivatized calibration standards is not affected by the presence or absence of hexylamine. For soil samples, hexylamine is added to the calibration standards before derivatization. For convenience, the same derivatized calibration standards are used for both aqueous and soil samples even though hexylamine is not added to aqueous samples. If the calibration standards are prepared only for water sample analysis, addition of hexylamine could be omitted.

B. Sample Preparation

1. Using pipettes, measure 1.0 mL of each sample and add into the reaction vials. If the sample is known to contain high levels of hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine, less sample may be used provided it is diluted to 1.0 mL with pH-5.0 acetate buffer (0.2M).

2. Measure 1.0-mL aliquots of pH-5.0 acetate buffer (0.2M) to prepare a blank and laboratory control sample (LCS).

3. Using pipettes, add 10.0 µL spiking standard solution to LCS, matrix spike, and matrix spike duplicate.

C. Derivatization

1. Using a pipette, add 0.5 mL benzaldehyde derivatizing reagent to each reaction vial.

2. Cap each reaction vial and shake them for a few seconds to obtain thorough mixing.

3. Place the reaction vials in the platform shaker which is set at 40° ±4°C. Gently agitate the samples for 1 hour.

Allow the reaction vials to cool to room temperature. Use a 0.45-µm syringe filter to filter the standards and samples into autosampler vials.

NOTE: Polypropylene autosampler vial and polypropylene syringe filter should not be used. Whatman 25 mm GD/X syringe filter with PTFE filter media, 0.45 um pore size (Cat. No. 6875-2504) and PALL GHP Acrodisc 25 mm syringe filter with GF/0.45 um GHP membrane (Cat. No. 4558) should not be used due to sample loss during filtration.

D. LC/MS/MS Analysis

Tuning and calibration for the LC/MS/MS: Refer to the instrument manufacturers instructions for tuning and conditions. These values are stored in the tune file for future reference, and may not need to be changed unless loss of response is noted.

1. Chromatographic conditions

Below are the recommended chromatographic conditions for the reversed-phase separation. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

a. HPLC setup:

Column	Thermo Scientific Hypersil Gold, 2.1 x 50mm, 3 µm
Column Temperature	30°C
Autosampler Tray Temperature	4°C
Injection volume	10 µL
HPLC Run Time	18 minutes
Solvent Delay*	0-4.6 min, divert to waste

*Solvent delay needs to be adjusted if retention time changes.

b. HPLC gradient timetable

Time (min)	% of mobile phase A (Milli-Q water)	% of mobile phase B (methanol)	Flow (mL/min)
0.0	95	5	0.3
10	10	90	0.3
10.1	0	100	0.3
14	0	100	0.3
14.1	95	5	0.3
18	95	5	0.3

c. Wash bottle solvent: 96: 4% (v/v) methanol: water

2. Detector conditions, Thermo Scientific LC/MS/MS SRM mode

NOTE: Conditions listed are guidelines

Mode: APCI Positive Mode	Skimmer Offset: 0
Discharge Current: 4.0	Collision Gas Pressure (mTorr): 1.5
Vaporizer Temperature (°C): 350	Desolvation Gas: Nitrogen
Sheath Gas Pressure: 25	Collision Cell Gas: Argon
Ion Sweep Gas Pressure: 0	Scan Width (m/z): 0.002
Aux Gas Pressure: 5	Q1 (FWHM): 0.7
Capillary Temperature (°C): 250	

3. Acquisition method*

Time Segment** (min)	Analyte	Precursor	Product	Dwell (s)	Tube Lens	Collision Energy
4.6 – 6.1	Hydrazine	209	106	0.5	86	17
6.1 – 7.9	Monomethylhydrazine	135	77	0.5	86	20
7.9 - 18	1.1-Dimethylhydrazine	149	106	0.5	66	14

*The exact value of precursor ion, product ion, tube lens and collision energy is determined by infusing standards into mass spectrometer.

**Time segment needs to be adjusted if retention time changes.

4. Load the sample in the order listed below.

Example sequence:

1. Conditioner
2. Conditioner
3. Cal. 1
4. Cal. 2
5. Cal. 3
6. Cal. 4
7. Cal. 5
8. Cal. 6
9. Cal. 7
10. Cal. 8
11. Methanol solvent
12. Blank
13. LCS
14. 1234567
15. 1234567MS
16. 1234567MSD
17. thru 21. Continue running samples

22. Low level calibration standard as CCV
23. thru 32. Continue running samples
33. Mid level calibration standard as CCV

After every ten injections, a continuing calibration verification (CCV) standard is run, alternating between the low, mid and high level. Conditioner injections are run to equilibrate the system and are usually blanks or standards.

5. Calibration

Inject calibration levels 1 through level 8 after allowing the system to equilibrate at least 1 hour, and injection of 2 conditioners. Prior to running samples, check linearity. Fit the curve with either a linear or quadratic fit, with ignore the origin. If the correlation coefficient exceeds 0.99, the curve is acceptable. If neither fit type is acceptable, check the integration and correct as necessary. Recalibrate if none of these correct the issue. If a second calibration does not meet specifications, remake standards, or perform maintenance as needed. When quantitated using the calibration curve, each calibration point, except the lowest point for each analyte, should calculate to be within 70-130% of its true value. The lowest calibration point should calculate to be within 50-150 % of its true value. 1/X weighting is frequently used because the difference between calculated and true value is smaller than using other weighting options, although other weighting options are acceptable.

6. Continuing calibration

Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Since the autosampler is cooled at 4°C, calibration standards are allowed to be re-injected as CCVs. CCV concentrations alternating between the low, mid and high level of the calibration are recommended. The control limit for CCV is ±30% of the true value. When the CCV exceeds the control limits, the

analysis should be terminated and the problem corrected. Samples that are not bracketed by acceptable CCV runs must be reanalyzed. If the CCV recoveries are running high indicating increased sensitivity, and the associated samples are clean, the data may be reported with a comment.

7. Sample analysis

After the initial calibration, inject the blank, followed by the LCS, then samples. Bracket each set of ten samples with a continuing calibration verification (CCV) standard.

After injections are completed, check continuing calibration recoveries to make sure they are acceptable. Process each chromatogram and closely evaluate all integration, baseline anomalies, and retention time differences.

Quantitate results for the blank. No hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine above the reporting limit may be found in the blank for acceptable batch results.

Calculate the recoveries for the LCS by comparing concentrations observed to the true values. The advisory acceptance limits for LCS recovery are 70 to 130% for each analyte. The advisory method control limit for the relative percent difference (%RPD) between LCS duplicates is ≤30%. If the LCS is acceptable, proceed to sample quantitation.

The advisory method control limits for matrix spike recovery are 70 to 130% and %RPD for matrix spike duplicates is 30%. If matrix spike recoveries or %RPD for matrix spike duplicates are outside the control limits and all other QC data is within limits, the associated data should be flagged or noted in the comments section of the report.

Statistical control limits must be calculated for recoveries of LCS and matrix spike, and %RPD for LCS duplicates and matrix spike duplicates when sufficient data points have been collected.

If the peak area response exceeds the calibration range of the system, dilute an aliquot of the original aqueous sample with pH-5.0 acetate buffer (0.2M). Perform derivatization and LC/MS/MS analysis.

Compare the retention times of three analytes. The retention times should not vary by more than 0.2 minutes.

Calculations:

An external standard calibration curve is established using peak area. The concentrations are entered into the calibration table in ng/mL. The data system uses the following equation to calculate the concentration in the extract:

Single component using linear curve:

$$[(\textit{Sample Area} - \textit{Intercept}) / \textit{Slope}] \times \frac{FV}{IV} \times DF = \mu g/L \text{ as received}$$

Where:

FV (final volume) = 1 mL

IV (initial volume) = 1 mL

DF (dilution factor) = as needed

See SOP-PP-040 for details on all calculations/equations used to evaluate the initial and continuing calibration.

Statistical Information/Method Performance:

Reporting limits including method detection limits (MDL) and limits of quantitation (LOQ) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These are analyzed on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Results from all instruments are compared and pooled together to determine the reporting MDL. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Quality Assurance/Quality Control:

For each batch of samples extracted, a blank, a laboratory control sample (LCS) (Milli-Q water blank spiked with compounds to be determined carried through the entire procedure), a matrix spike, and a matrix spike duplicate must be extracted. If there is limited sample that prevents the preparation of the MS/MSD then an LCSD must be prepared instead. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. See SOP-PP-002 for QC acceptance criteria and SOP-PP-025 for monitoring QC limits. If any client, state, or agency has more stringent QC or batching requirements, these must be followed instead.

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Analysis #10346
Revision 01
Supersedes Date: None
Effective Date: FEB 10 2009

Determination of Hydrazine, Monomethylhydrazine and 1.1-Dimethylhydrazine in Soil Samples by LC/MS/MS

Approvals:

Prepared by: _____ Date: 1/21/2009
Principal Chemist

Approved by: _____ Date: 1/21/09
Specialty Services Management

Approved by: _____ Date: 1/27/09
Quality Assurance

Revision Log:

Revision	Effective Date	The version
Section	Justification	Changes
		New

10346_01.DOC
110408

Reference:

1. *Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)*, USEPA 8315A

2. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, USEPA SW-846.

3. *Hydrazine*, U. S. Department of Labor, Method 108.

4. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Reference (USEPA 8315A) Modifications:

1. LC/MS/MS instead of LC/UV is used for detection and quantification of hydrazine, monomethylhydrazine and 1.1-dimethylhydrazine derivative.

2. Rather than being target analyte, carbonyl compound (benzaldehyde) is used as derivatizing reagent to form hydrazones with hydrazine compounds.

3. 10 mM hexylamine solution is used as extraction fluid.

Cross Reference:

Document	Document Title
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-0E-001	Glassware Cleaning for Organic Extractions
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-025	Monitoring of QC Data Acceptance Limits
SOP-PP-040	Common Equations Used During Chromatographic Analyses

Scope:

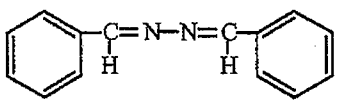
The method is applicable for the determination of hydrazine, monomethylhydrazine and 1.1-dimethylhydrazine in soil samples. The reporting limits of the three analytes are as follows:

Analyte	Reporting Limit (µg/kg)*
Hydrazine	2
Monomethylhydrazine	5
1.1-Dimethylhydrazine	5

*May be adjusted/raised if matrix issues are encountered during sample preparation.

Basic Principles:

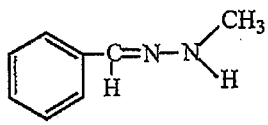
Soil samples are extracted with 10 mM hexylamine solution. The supernatant of soil extract is buffered with an acetate buffer to a pH of approximately 5. Hydrazine, monomethylhydrazine (MMH) and 1,1-dimethylhydrazine (UDMH) are derivatized with benzaldehyde to the corresponding hydrazones: benzalazine, benzaldehyde methylhydrazone and benzaldehyde dimethylhydrazone, respectively. The resulting solution is analyzed by LC/MS/MS operated in atmospheric pressure chemical ionization (APCI) mode for detection and quantification of the hydrazones.



Benzalazine

$C_{14}H_{12}N_2$

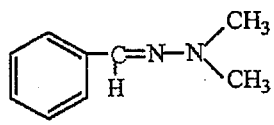
FW = 208.2628



Benzaldehyde Methylhydrazone

$C_8H_{10}N_2$

FW = 134.1809



Benzaldehyde Dimethylhydrazone

$C_9H_{12}N_2$

FW = 148.2078

NOTE: 1,1-Dimethylhydrazine is also known as unsymmetrical dimethylhydrazine.

Interferences:

Compounds which have similar structures to the compounds of interest, and similar molecular weights would potentially interfere. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants. Samples with a pH that is significantly acidic or basic and resists to pH adjustment may show reduced performance.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Hydrazine, monomethylhydrazine, and 1,1-dimethylhydrazine are toxic. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards, handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact, and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All solvent waste is collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. Vials containing expired hydrazine, methylhydrazine and 1,1-dimethylhydrazine standards, vials containing soil and extraction fluid, and HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications:

All personnel performing these techniques should have performed a quad study that yielded acceptable recoveries for hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine, or completed another approved demonstration of capability (i.e. PT sample, blind sample, etc.). Personnel should spend several days working with an experienced technician or chemist who has demonstrated their proficiency of the extraction. Also, several batches of water samples should be performed under the direct observation of another experienced preparation technician to assure the trainee is capable of independent preparation.

Analysts performing the instrumental analysis will work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set-up sequences, perform calculations, interpret the chromatograms and mass spec data, and enter the data into the LIMS. Proficiency is measured through documented audits of the tasks listed and overchecking of data, as well as annual quad studies.

Sample Handling:

Soil samples should be collected in 4-oz amber wide mouth jars with Teflon lined lids. Soil samples should be homogenized prior to analysis. Soil samples should be stored at 2° to 4°C and must be extracted and derivatized within 14 days of sampling. Extracts must be analyzed within 72 hours of derivatization.

Apparatus and Equipment:

1. pH meter, accurate to 0.1 pH units

2. Sample shaker – Temperature controlled bath with platform shaker

3. Sample shaker – Platform shaker capable of shaking at 300 rpm

4. Centrifuge

5. Reaction vials – 4-mL glass vials with Teflon™-lined caps

6. Extraction vials –20-mL glass vials with Teflon™-lined caps

7. Balance – Capable of weighing to 0.0001 g

8. Balance – Capable of weighing to 0.01 g

9. Stir plate

10. Beakers – assorted sizes

11. Volumetric flasks – Class A, assorted sizes

12. Glass pipettes – Class A, assorted sizes

13. Pipettes – Rainin, POS-D™ (Positive-Displacement), 1-10 µL (Cat. No. MR-10) and 10-100 µL (Cat. No. MR-100) or equivalent

14. Pipette tips – Rainin, 1-10 µL (Cat. No. C-10) and 10-100 µL (Cat. No. C-100) or equivalent

15. Pipette – Rainin, Pipet-Lite LTS, 1-10 mL, Cat. No. RL-10mL or equivalent

16. Pipette tip – Rainin, 1-10 mL, Cat. No. RC-L10ML or equivalent

17. Pipettes – Eppendorf Research®, 10-100 µL and 100-1000 µL or equivalent

18. Pipette tips – Fisher Scientific, 10-100 µL (Cat. No. 02707505) and 100-1000 µL (Cat. No. 02707508) or equivalent

19. Graduated cylinders

20. Syringe filter – PALL, Acrodisc CR 25 mm syringe filter with 0.45 PTFE membrane, PN 4501 or equivalent.

21. Disposable polypropylene syringe – 3 mL, SUN-SRI, Cat. No. 400 004 or equivalent

22. Syringe – 0-500 µL, Hamilton or equivalent

23. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent

24. 1 liter bottles for mobile phase storage

25. HPLC autosampler vial – Thermo Scientific, 1.8 mL, Cat. No. 60180-205 or equivalent

26. HPLC with dual high-pressure pump and gradient capabilities – Thermo Electron Corporation, Accela or equivalent

27. HPLC autosampler – Thermo Electron Corporation, Accela or equivalent

28. LC/MS/MS equipped with atmospheric pressure chemical ionization source – Thermo Scientific, TSQ Quantum Access or equivalent.

29. HPLC column – Thermo Scientific Hypersil Gold, 2.1 x 50mm, 3µm, Part No. 25003-052130 or equivalent

30. Precolumn filter – Upchurch Scientific, Part No. A-315 or equivalent

31. Precolumn filter frit – Upchurch Scientific, 2 µm, Part No. A-101X or equivalent

32. In-line filter frit before injection valve – Analytical Scientific Instrument, 0.5 µm, Part No. 850-1347 or equivalent

33. Data System – Thermo Scientific LCquan for data acquisition, integration, and spectra or equivalent

Reagents and Standards:

A. Reagents

1. Methanol – HPLC grade or equivalent

2. Milli Q Water

3. Ammonium acetate – ACS grade or equivalent

4. Glacial acetic acid – ACS grade or equivalent

5. Formic acid – ACS grade or equivalent

6. Benzaldehyde – Aldrich, ≥99.5%, Cat. No. 418099-100mL, or equivalent.

7. Hexylamine – Acros Organics, 99%, Cat. No. 204720050, or equivalent.

8. 1M Hexylamine solution – Add 0.66 mL hexylamine into a 5-mL volumetric flask. Bring to volume with methanol and mix well. This solution is stored in the refrigerator and is stable for 30 days. This may be prepared in larger or smaller volumes.

9. 1% (v/v) Benzaldehyde derivatizing reagent – Add 0.1 mL of benzaldehyde into a glass vial containing 10 mL of methanol and mix well. This may be prepared in larger or smaller volumes depending on the size of the extraction group. This reagent must be prepared fresh daily.

10. pH-5.0 Acetate buffer (5M) - This buffer is stored at room temperature and is stable for 6 months. Prepare as follows:

    a. Add 28.6 mL of glacial acetic acid into a 100-mL volumetric flask containing approximately 50 mL of Milli Q water. Bring to volume with Milli Q water and mix well. This prepares 5M acetic acid solution. This solution is stored at room temperature and is stable for 6 months.

    b. Weigh 38.5 ±0.04 g of ammonium acetate in a beaker. Add Milli Q water to dissolve and quantitatively transfer it to a 100-mL volumetric flask. Bring to volume with Milli Q water and mix well. This prepares 5M ammonium acetate solution. This solution is stored at room temperature and is stable for 6 months.

    c. Place 60 mL of the 5M ammonium acetate solution into a 200-mL beaker. Add 40 mL of 5M acetic acid solution and mix well. Adjust the pH of the solution to 5.0 ±0.2 with formic acid as needed.

    d. Transfer the buffer to a glass bottle. Record the pH of the buffer on the bottle along with the date prepared and expiration date. The buffer is stable for 6 months.

11. pH-5.0 acetate buffer (0.2M) - Pipette 10.0 mL of 5M acetate buffer into a 250-mL volumetric containing approximately 100 mL of Milli Q water. Bring to volume with Milli Q water and mix well. This solution is stored in the refrigerator or at room temperature and is stable for 6 months.

12. Extraction fluid (10 mM hexylamine solution) – Add 2.5 mL of 1M hexylamine solution into a 250-mL volumetric flask containing approximately 50 mL of Milli Q water. Bring to volume with Milli Q water and mix well. This solution is stored at room temperature and is stable for 2 weeks. This may be prepared in larger or smaller volumes.

B. Standards

1. Hydrazine stock standard solution – Commercially prepared 100 µg/mL hydrazine standard in methanol (Cat. No. M-8330-ADD-8) can be purchased from AccuStandard, or equivalent vendor. Store in the refrigerator and protect from light. Refer to manufacturer's instruction for expiration date.

2. 1,1-dimethylhydrazine stock standard solution – Commercially prepared 1000 µg/mL 1,1-dimethylhydrazine standard in methanol (Cat. No. S-1627) can be purchased from Spex CertiPrep, or equivalent vendor. Store in the refrigerator and protect from light. Commercially prepared stock standard expires 90 days from date of preparation.

3. Monomethylhydrazine neat standard – Fluka, purum, ≥98.0%, (Cat. No. 418099-100mL) or equivalent vendor. The catalog, purity and lot must be recorded. Store the compound in the flammable cabinet.

4. Monomethylhydrazine stock standard solution, approximately 1000 µg/mL – On an analytical balance weigh ~20 mg neat compound (approximately 24 uL) into a 20-mL volumetric flask. Dilute to volume with methanol. Cap, mix, and label. Store in the refrigerator and protect from light. The stock standard solution expires 4 hours from the preparation time.

5. Standard solutions and spiking solution are prepared as noted below and can be prepared using different initial and final volumes as long as the resulting calibration standards and spiking solution are at the concentrations listed below.

6. Intermediate standard solution – Add 20.0 µL of hydrazine stock standard solution, X µL of monomethylhydrazine standard stock solution and 10.0 µL of 1,1,-dimethylhydrazine stock standard solution to a 20-mL volumetric flask with 2 mL of pH-5.0 acetate buffer (0.2M). Bring to volume with pH-5.0 acetate buffer (0.2M) and mix well. Store in the refrigerator and protect from

light. Intermediate standard expires at the end of 1,1-dimethylhydrazine stock standard expiration date. The added volume of monomethylhydrazine stock solution is calculated as followings:

$$X\,(\mu L) = \frac{10{,}000}{\text{Concentration of monomethylhydrazine stock solution } (\mu g/mL)}$$

Standard ID	Hydrazine Conc. (µg/mL)	MMH & UDMH Conc. (µg/mL)	Solvent
Intermediate standard	0.1	0.5	pH-5.0 acetate buffer (0.2M)

7. Working standards – Prepare the following working standards according to the dilution scheme presented below. To 20-mL volumetric flasks containing 2 mL of pH-5.0 acetate buffer (0.2M), add the volumes of intermediate standard solution as described below. Bring to volume with pH-5.0 acetate buffer (0.2M). Store in the refrigerator and protect from light. Working standards expire at the end of 1,1-dimethylhydrazine stock standard expiration date.

Standard ID	Prepared From	Initial Volume (µL)	Final Volume (mL)	Hydrazine Concentration (ng/mL)	MMH & UDMH Concentration (ng/mL)	Solvent
Level 1	Intermediate	20	20	0.1	0.5	0.2M Acetate Buffer
Level 2	Intermediate	50	20	0.25	1.25	0.2M Acetate Buffer
Level 3	Intermediate	100	20	0.5	2.5	0.2M Acetate Buffer
Level 4	Intermediate	200	20	1	5	0.2M Acetate Buffer
Level 5	Intermediate	500	20	2.5	12.5	0.2M Acetate Buffer
Level 6	Intermediate	1000	20	5	25	0.2M Acetate Buffer
Level 7	Intermediate	2000	20	10	50	0.2M Acetate Buffer
Level 8	Intermediate	5000	20	25	125	0.2M Acetate Buffer

8. Spiking solution – Add 125.0 µL of hydrazine stock standard solution, Y µL of monomethylhydrazine standard stock solution and 62.5 µL of 1,1,-dimethylhydrazine stock standard solutions to a 5-mL volumetric flask with 2 mL methanol. Dilute to volume with methanol, and mix well. Store in the refrigerator and protect from light. Spiking solution expires at the end of 1,1-dimethylhydrazine stock standard expiration date.

$$Y\,(\mu L) = \frac{62{,}500}{\textit{Concentration of monomethylhydrazine stock solution}\ (\mu g/mL)}$$

	Hydrazine Conc. (µg/mL)	MMH & UDMH Conc. (µg/mL)	Solvent
Spiking Solution	2.5	12.5	methanol

Preparation of Glassware:

See SOP-OE-001.

Procedure:

A. Calibration standards preparation

A calibration curve must be prepared every 1 calendar day.

1. Measure 1.0 mL of the working standard Level 1 through Level 8 into reaction vial, respectively. Label the vials as Level 1 through 8.

2. Add 10.0 µL of 1M hexylamine solution into each reaction vial.

   NOTE: The response of derivatized calibration standards is not affected by the presence or absence of hexylamine. For soil samples, hexylamine is added to the calibration standards before derivatization. For convenience, the same derivatized calibration standards are used for both aqueous and soil samples even though hexylamine is not added to aqueous samples.

3. Derivatize the standards following the steps listed in Part D.

B. Determination of spiking solution concentration

1. Measure 1.0 mL of pH-5.0 acetate buffer (0.2M) into a reaction vial. Label the vial as spike checking.

2. Add 10.0 µL of 1M hexylamine solution into reaction vial.

3. Add 10.0 µL of spiking solution into reaction vial.

4. Derivatize the spike checking solution following the steps listed in Part D.

   NOTE: Spiking solution is prepared in methanol. During method development, the initial rapid decrease of monomethylhydrazine response was observed. It was also noticed that 1,1-Dimethylhydrazine response decreased occasionally. So the concentration of spiking solution is checked every time when spiking solution is used.

C. Extraction of soil samples

1. Weigh 1.0 ±0.01 g of sample into a labeled extraction vial. Record the initial weight of the sample and any comments about the sample on the extraction log.

2. Weigh 1.0 ±0.01 g of Ottawa sand into labeled extraction vials to prepare a blank and Laboratory Control Sample (LCS). Record the initial weight of the sand.

3. Using pipettes, add 50.0 µL spiking solutions to LCS, matrix spike (MS), and matrix spike duplicate (MSD).

4. Add 10.0 mL of extraction fluid to each sample, blank, and LCS.

   NOTE: The sequence of adding spiking solution and extraction fluid may not be changed.

5. Place the extraction vials in the platform shaker and agitate the samples at 300 rpm for 10 minutes at room temperature.

6. Place the extraction vials in a centrifuge at 2800 – 3000 rpm for 10 minutes at room temperature.

   NOTE: Extraction vial may break when centrifuge at speed over 3000 rpm.

7. Using pipette, measure 1.0 mL supernatant of soil extracts and add into the reaction vials.

   NOTE:

   a. Pre-wetting of pipette tip should be omitted in order to not disturb the supernatant.

   b. If the sample is known to contain high levels of hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine, less sample may be used provided it is diluted to 1.0 mL with extraction fluid.

8. Add 40.0 µL of pH-5.0 acetate buffer (5M) into each reaction vial.

D. Derivatization

1. Using a pipette, add 0.5 mL benzaldehyde derivatizing reagent to each reaction vial.

2. Cap each reaction vial and shake them for a few seconds to obtain thorough mixing.

3. Place the reaction vials in the platform shaker which is set at 40° ±4°C. Gently agitate the samples for 1 hour.

   Allow the reaction vials to cool to room temperature. Using 0.45 um syringe filter to filter the standards and samples into autosampler vials.

   NOTE: Polypropylene autosampler vial and polypropylene syringe filter should not be used. Whatman 25 mm GD/X syringe filter with PTFE filter media, 0.45 um pore size (Cat. No. 6875-2504) and PALL GHP Acrodisc 25 mm syringe filter with GXF/0.45 um GHP membrane (Cat. No. 4558) should not be used due to sample loss during filtration.

E. LC/MS/MS Analysis

Tuning and calibration for the LC/MS/MS: Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference, and may not need to be changed unless loss of response is noted.

1. Chromatographic conditions

   Below are the recommended chromatographic conditions for the reversed-phase separation. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

a. HPLC setup:

Column	Thermo Scientific Hypersil Gold, 2.1 x 50mm, 3µm
Column Temperature	30°C
Autosampler Tray Temperature	4°C
Injection Volume	10 µL
HPLC Run Time	18 minutes
Solvent Delay*	0-4.6 min, divert to waste

*Solvent delay needs to be adjusted if retention time changes.

b. HPLC gradient timetable

Time (min)	% of mobile phase A (MilliQ water)	% of mobile phase B (methanol)	Flow (mL/min)
0.0	95	5	0.3
10	10	90	0.3
10.1	0	100	0.3
14	0	100	0.3
14.1	95	5	0.3
18	95	5	0.3

c. Wash bottle solvent: 96: 4% (v/v) methanol: water

2. Detector conditions, Thermo Scientific LC/MS/MS SRM mode

NOTE: Conditions listed are guidelines

Mode: APCI Positive Mode	Skimmer Offset: 0
Discharge Current: 4.0	Collision Gas Pressure (mTorr): 1.5
Vaporizer Temperature (°C): 350	Desolvation Gas: Nitrogen
Sheath Gas Pressure: 25	Collision Cell Gas: Argon
Ion Sweep Gas Pressure: 0	Scan Width (m/z): 0.002
Aux Gas Pressure: 5	Q1 (FWHM): 0.7
Capillary Temperature (°C): 250	

3. Acquisition method*

Time Segment** (min)	Analyte	Precursor	Product	Dwell (s)	Tube Lens	Collision Energy
4.6 – 6.1	Hydrazine	209	106	0.5	86	17
6.1 – 7.9	Monomethylhydrazine	135	77	0.5	86	20
7.9 - 18	1.1-Dimethylhydrazine	149	106	0.5	66	14

*The exact value of precursor ion, product ion, tube lens and collision energy is determined by infusing standards into mass spectrometer.

**Time segment needs to be adjusted if retention time changes.

4. Load the sample in the order listed below.

Example sequence:

1. Conditioner
2. Conditioner
3. Cal. 1
4. Cal. 2
5. Cal. 3
6. Cal. 4
7. Cal. 5
8. Cal. 6
9. Cal. 7
10. Cal. 8
11. Methanol solvent
12. Blank
13. LCS
14. 1234567
15. 1234567MS
16. 1234567MSD

17. thru 21. Continue running samples
22. Low level calibration standard as CCV
23. thru 32. Continue running samples
33. Mid level calibration standard as CCV

After every ten injections, a continuing calibration verification (CCV) standard is run, alternating between the low, mid and high level. Conditioner injections are run to equilibrate the system and are usually blanks or standards.

5. Calibration

Inject calibration levels 1 through level 8 after allowing the system to equilibrate at least 1 hour, and injection of 2 conditioners. Prior to running samples, check linearity. Fit the curve with either a linear or quadratic fit, with ignore the origin. If the correlation coefficient exceeds 0.99, the curve is acceptable. If neither fit type is acceptable, check the integration and correct as necessary. Recalibrate if none of these correct the issue. If a second calibration does not meet specifications, remake standards, or perform maintenance as needed. When quantitated using the calibration curve, each calibration point, except the lowest point for each analyte, should calculate to be within 70-130% of its true value. The lowest calibration point should calculate to be within 50-150 % of its true value. 1/X weighting is frequently used because the difference between calculated and true value is smaller than using other weighting options, although other weighting options are acceptable.

6. Continuing calibration

Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Since the autosampler is cooled at 4°C, calibration standards are allowed to be re-injected as CCVs. CCV concentrations alternating between the low, mid and

high level of the calibration are recommended. The control limit for CCV is ±30% of the true value. When the CCV exceeds the control limits, the analysis should be terminated and the problem corrected. Samples that are not bracketed by acceptable CCV runs must be reanalyzed. If the CCV recoveries are running high indicating increased sensitivity, and the associated samples are clean, the data may be reported with a comment.

7. Sample analysis

After the initial calibration, inject the blank, followed by the LCS, then samples. Bracket each set of ten samples with a continuing calibration verification (CCV) standard.

After injections are completed, check continuing calibration recoveries to make sure they are acceptable. Process each chromatogram and closely evaluate all integration, baseline anomalies, and retention time differences.

Quantitate results for the blank. No hydrazine, monomethylhydrazine and 1,1-dimethylhydrazine at or above the reporting limit may be found in the blank for acceptable batch results.

Calculate the recoveries for LCS, matrix spike and matrix spike duplicate by comparing concentrations observed to the true values that are calculated using the concentration of spiking solution determined daily. The advisory acceptance limits for LCS recovery are 70 to 130% for each analyte. The advisory method control limit for the relative percent difference (%RPD) between LCS duplicates is ≤30%. If the LCS is acceptable, proceed to sample quantitation.

The advisory control limits for matrix spike recovery are 11 to 102% for hydrazine, 10 to 92% for monomethylhydrazine and 10-116% for 1,1-dimethylhydrazine. The advisory method control limit for %RPD between the matrix spike duplicates is ≤30%. If matrix spike recoveries or %RPD for

matrix spike duplicates are outside the control limits and all other QC data is within limits, the associated data should be flagged or noted in the comments section of the report.

Statistical control limits must be calculated for recoveries of LCS and matrix spike, and %RPD for LCS duplicates and matrix spike duplicates when sufficient data points have been collected.

If the peak area response exceeds the calibration range of the system, re-extract the soil sample. Dilute an aliquot of the fresh extract with extraction fluid. Perform derivatization and LC/MS/MS analysis.

Compare the retention times of three analytes. The retention times should not vary by more than 0.2 minutes.

Calculations:

An external standard calibration curve is established using peak area. The concentrations are entered into the calibration table in ng/mL. The data system uses the following equation to calculate the concentration in the extract:

Single component using linear curve:

$$[(\textit{Sample Area} - \textit{Intercept}) / \textit{Slope}] \times \frac{FV}{IW} \times DF = \mu g / L \text{ as received}$$

Where:

FV (final volume) = 10 mL
IW (initial weight) = 1 g
DF (dilution factor) = as needed

See SOP-PP-040 for details on all calculations/equations used to evaluate the initial and continuing calibration.

Statistical Information and Method Performance:

Reporting limits including method detection limits (MDL) and limits of quantitation (LOQ) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These are analyzed on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Results from all instruments are compared and pooled together to determine the reporting MDL. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Quality Assurance/Quality Control:

For each batch of samples extracted, a blank, a laboratory control sample (LCS) (Ottawa sand spiked with compounds to be determined carried through the entire procedure), a matrix spike, and a matrix spike duplicate must be extracted. If there is limited sample that prevents the preparation of the MS/MSD then a laboratory control sample duplicate (LCSD) must be prepared instead. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. See SOP-PP-002 for QC acceptance criteria and SOP-PP-025 for monitoring QC limits. If any client, state, or agency has more stringent QC or batching requirements, these must be followed instead.

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STANDARD OPERATING PROCEDURE

Analytical Procedures for the Analysis of PCB Aroclors

Method SW846 8082A

SOP ID: GCS00202.MA

Revision ~~(1)~~ (3) CFR 7/13/06

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Approved Signatures

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QUALITY ASSURANCE COORDINATOR

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1.0 TITLE

Analytical Procedures for the Analysis of PCB Aroclors by Method SW846 8082A

2.0 MATRIX

Method 8082 is used to determine the concentrations of polychlorinated biphenyls (PCBs)as Aroclors or as individual PCB congeners in extracts from solid and aqueous matrices.

3.0 METHOD REPORTING LIMIT: Aqueous samples: 0.30µg/L, Soils: 100µg/Kg

4.0 SCOPE AND APPLICATION

4.1 This procedure is used to determine the concentrations of pcb's as arochlors in extracts from solid and liquid matrices. The seven target arochlors are listed in table 1. This method is applicable to water, soil, sediment, and sludge. Sample extracts maybe subject to various clean-up procedures to remove interferences from non-target matrices. The procedure used will depend on the nature of the interference. Compound identification is based on pattern recognition of arochlors and relies on analyst expertise. Arochlor concentration is confirmed automatically with dual column injections. The practical quantitation limit for this method is 0.3-25.0 ug/L for waters and 100-5000 ug/kg for soils.

5.0 SUMMARY OF METHOD

5.1 Either 1liter of water or 10 grams of soil is extracted by using the appropriate matrix-specific extraction technique. Liquids samples are extracted at neutral pH with methylene chloride using a separatory funnel procedure (SW846 3510). Soils are extracted with hexane-acetone (1:1) using soxhlet (method SW846 3540), microwave digestion (SW846 3546) ultrasonic procedure (SW846 3550). The extraction solvent is concentrated, and exchanged to hexane. The final volume of the extract is brought to 5 mL for waters and 10mL for soils. Depending on the appearance of the extract, e.g. if it is highly colored, the extract may be subject to clean-up with sulfuric acid or florisil to remove interferences. A 2uL aliquot of the extract is injected into a Hewlett Packard gas chromatograph fitted with two 0.32id capillary columns connected with a deactivated glass "Y-splitter" preceded by a 5-meter fused silica guard column. Each column is connected to a separate electron capture detector. Sample extracts are analyzed simultaneously on each column. The connection to the Y-splitter can be problematic and a leak free seal difficult to obtain. Alternatively, both columns can be fitted with a 2-hole ferrule conversion fitting.

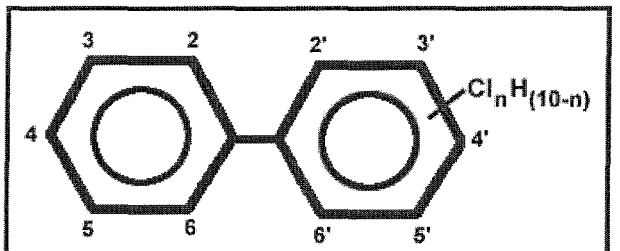
TABLE 1
EPA 8082A TARGET COMPOUNDS

NUMBER	ANALYTE	CAS NUMBER
1	Aroclor 1016	12674-11-2
2	Aroclor 1221	11104-28-2
3	Aroclor 1232	11141-16-5
4	Aroclor 1242	53469-21-9
5	Aroclor 1248	12672-29-6
6	Aroclor 1254	11097-69-1
7	Aroclor 1260	11096-82-5
8	Aroclor 1262	37324-23-5
9	Aroclor 1268	11100-14-4

6.0 DEFINITIONS

Polychlorinated Biphenyl (PCB) : PCB (or PCBs) is a category, or family, of chemical compounds formed by the addition of Chlorine (Cl2) to Biphenyl (C12H10), which is a dual-ring structure comprising two 6-carbon Benzene rings linked by a single carbon-carbon bond. The nature of an "aromatic" (Benzene) ring allows a single attachment to each carbon. This means that there are 10 possible positions for chlorine substitution (replacing the hydrogens in the original Biphenyl). Species with a single chlorine substituent are called "Monochlorobiphenyl" (or just "Chlorobiphenyl"). Species with two chlorines are called "Dichlorobiphenyl", and those with three through ten chlorines, in order, are called: "Tri...", "Tetra...", "Penta...", "Hexa...", "Hepta...", "Octa...", "Nona...", and "Decachlorobiphenyl".

See the diagram below



Polychlorinated Biphenyl (PCB)

PCB Congener : Any single, unique, well-defined chemical compound in the PCB category is called a "Congener". The name of a congener specifies the total number of chlorine substituents and the position of each chlorine. For example: 4,4'-Dichlorobiphenyl is a congener comprising the Biphenyl structure with two chlorine substituents, one on each of the two carbons at the "4" (also called "para") positions of the two rings. There are 209 PCB congeners.

PCB Mixture : With few exceptions, PCB was manufactured as a complex mixture of congeners, through progressive chlorination of batches of Biphenyl until a certain target percentage of Chlorine by weight was achieved.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD is a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement

responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

7.0 INTERFERENCES

7.1 Interference's in this method can result from contaminated solvents, reagents, glassware, or other sample processing hardware, as well as contaminated GC gases, parts, columns, and detectors. Interference's can also result from compounds co-extracted from the sample matrix to which the detector will respond. In this case a variety of extract clean-up procedures are available to eliminate the problem. Phthalate esters are particularly problematic to this method. These compounds are present in many plastics and can be leached out especially when coming into contact with extraction solvents. Avoiding contact with any plastic materials during sample extraction can minimize this problem. Exhaustive clean up of solvents reagents and glassware may be required to eliminate background levels of phthalates. Method blanks are treated identically to samples and are processed with every batch to ensure no contamination is present. Glassware must be scrupulously cleaned with detergent and hot water, rinsed with tap water and organic free reagent water, followed by drying in an oven at 400°C or by rinsing with methanol or acetone to dry and methylene chloride to remove impurities.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, the STL facility addendum and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or

significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 Hewlett Packard 5890 Series II Gas Chromatograph equipped with dual electron

capture detectors and model 7673 autosampler. Complete with septa, inlet liner, carrier and make-up gas helium, argon/methane.

9.2 Hewlett Packard Chemstation/Enviroquant PC based data system.
9.3 Column #1: Restek Rtx-CLPesticides 30meter x 0.32mm ID; 0.5um df
9.4 Column #2: Restek Rtx-CLPesticides2 30meter x 0.32mm ID; 0.25um df
9.5 Guard column: Restek Siltek 5meter x 0.32mm ID
9.6 Siltek press tight deactivated "Y-splitter"
9.7 2-liter Teflon separatory funnel
9.8 Soxhelet glassware
9.9 Hotplates
9.10 Tekmar model TM500 ultrasonic disrupter
9.11 CEM MARS5 (Microwave Accelerated Reaction System)
9.12 Low pressure liquid nitrogen tank
9.13 Condensers with water supply
9.14 Zymark Turbo-vap II, nitrogen blowdown device and associated glassware.
9.15 Syringes, volumetric flasks, graduated cylinder, autosampler vials, instrument supplies, pipets and other accessories
9.16 Analytical balance capable of weighing 0.1mg.

10.0 REAGENTS

10.1 Reagent grade (ACS) chemicals shall be used in all tests.
The date received must be recorded on all reagent labels. Consult the Safety manual for proper handling and storage of reagents. Whenever the lab receives new lots of reagents they must be screened to show they are free of contaminants. Analyze an equivalent portion of the reagent as would be used for a sample to demonstrate it is free of contaminants. The following is a list of reagents that may be used in analysis:
10.2 Organic free reagent water.
10.3 Pesticide grade acetone, methanol, methylene chloride, hexane, toluene, diethyl ether.
10.4 Florisil cartridges (supelco cat# 5-7053)
10.5 Sulfuric acid
10.6 Sodium sulfate
10.7 Dimethyldichlorosiloxane
10.8 Primary standards are purchased as certified solutions or as pure material.
Table 2 lists the commercial sources available that provide the necessary quantitation and calibration materials for this method.
10.9 Intermediate stock standards. Standards used for calibration are generally in the ppb range. Intermediate solutions are required to accurately achieve these low concentration levels. Table 3 lists the dilutions of the stock standard materials that may be used to accomplish this.
10.10 Working standards are prepared from the intermediate stocks and are recorded in

the GC working standards logbook with description of contents, date of preparation, initial and final volumes, concentration, dilution solvents, lot numbers, and expiration dates. Each working standard is assigned a unique lot number based on page number and volume of the logbook. This number must also be recorded onto the instrumental software data file to enable traceability from the final quantitation report. Unless super-ceded by the method, expiration dates are 6 months from date of preparation for semi-volatile standards and 1month for volatile standards. Standards purchased as certified solutions often have expiration dates of their own. If this date expires sooner than the 6 months than that is the expiration date. Standards with expired dates must discarded. It is sometimes possible to get extended expiration dates by calling the manufacturer with the lot number. A certificate must be obtained before the standard can be used.

10.11 Standards integrity is verified by purchasing second source standards of a different lot number.

10.12 Calibration standards are prepared at a minimum of five levels for the aroclors 1016 and 1260. Table 4 is a summary of the steps required to formulate these five levels. All other aroclor calibrations are based on single points.

10.13 Spiking solutions used to monitor the extraction performance are prepared in acetone. A 1.0mg/L solution of tetrachloro-m-xylene and decachlorobiphenyl is used for the surrogate spike. From this solution, an aliquot of 1.0mL for soils and 0.5mL for water is spiked into all samples, blanks, and matrix spikes, giving a final extract concentration of 100ug/L. A 10.0mg/L solution of aroclor 1016 and 1260 is used for matrix spiking. From this solution, an aliquot of 1.0mL for soils and 0.5mL for water is spiked into matrix spikes and Lab control spikes to give an extract concentration of 1000ug/L.

TABLE 2
Commercial Blends Required for Production of Method Standard Solutions

#	ITEM	VENDOR	CAT #	CONC.	SOLVENT	USE
1	Equity aroclor mix 1016/1260 tcmx/dcb	Supelco	86-1181	500 mg/L 1016/1260 50 mg/L tcmx/dcb	Acetone/methanol (40:60)	QC check
2	Aroclor1016	Accu-standard	C-216S-H-10X	1000mg/L	hexane	Quantitation
3	Aroclor1260	Accu-standard	C-260S-H-10X	1000mg/L	hexane	Quantitation
4	Tcmx/dcb	Accu-	CLP-032-	200 mg/L	Acetone	Quantitation

		standard	R			
5	Aroclor1221	Accu-standard	C-221S-H-10X	1000 mg/L	hexane	Quantitation
6	Aroclor1232	Accu-standard	C-232S-H-10X	1000 mg/L	hexane	Quantitation
7	Aroclor1242	Accu-standard	C-242S-H-10X	1000 mg/L	hexane	Quantitation
8	Aroclor1248	Accu-standard	C-248S-H-10x	1000 mg/L	hexane	Quantitation
9	Aroclor1254	Accu-standard	C-254S-H-10X	1000 mg/L	hexane	Quantitation
10	Aroclor1262	Accu-std	C-262S-H-10X	1000 mg/L	hexane	Quantitation
11	Aroclor1268	Accu-std	C-268S-H-10X	1000 mg/L	hexane	Quantitation

TABLE 3

Formulation of Intermediate Concentration Stock Solutions.

NUMBER FROM TABLE 2	ITEM	Volume (µL)	Final Vol. (mL)	SOLVENT	FINAL CONCENTRATION (mg/L)
1	Aroclor1016/1260 TCMX/DCB	200	10	Acetone	10.0 1.0
2	Aroclor1016	100	10	hexane	10.0
3	Aroclor1260	100	10	hexane	10.0
4	Tcmx/dcb	50	10	hexane	1.0
5	Aroclor1221	100	10	hexane	10.0
6	Aroclor1232	100	10	hexane	10.0

NUMBER FROM TABLE 2	ITEM	Volume (µL)	Final Vol. (mL)	SOLVENT	FINAL CONCENTRATION (mg/L)
1	Aroclor1016/1260 TCMX/DCB	200	10	Acetone	10.0 1.0
7	Aroclor1242	100	10	hexane	10.0
8	Aroclor1248	100	10	hexane	10.0
9	Aroclor1254	100	10	hexane	10.0
10	Aroclor1262	100	10	hexane	10.0
11	Aroclor1268	100	10	hexane	10.0

TABLE 4

Standard Formulation for EPA Method 8082.
- Refers to Intermediate Dilutions From Table 3.

Volume #1 (µL)	Final Vol. (mL)	SOLVENT	FINAL CONCENTRATION (µg/L)
10	1.0	Hexane	100
20	1.0	Hexane	200
50	1.0	Hexane	500
100	1.0	Hexane	1000
200	1.0	Hexane	2000
Volume #5-9 (µL)	**(mL)**	**SOLVENT**	**FINAL CONCENTRATION (µg/L)**
100	1.0	Hexane	1000

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Water samples are collected in 1 liter amber bottles. Soil samples are collected in 250 mL wide mouth jars. Both are preserved at 4°C.
11.2 Samples are shipped to the laboratory on ice.
11.3 Samples are stored in a walk-in cooler at 4°C.
11.4 **Holding time**: Aqueous extraction is required to be started within seven days of sample collection and soil extraction is required to be started within fourteen days of sample collection.
11.5 All sample extracts must be analyzed within forty days of extraction.

12.0 QC ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	At start-up and when cont. cal fails. (6-points)	r-value ≥ 0.99 for all method analytes (or <15% RSD on RF's)	Check for injection problems reanalyze std. or recalibrate
Initial Calibration Verification (ICV)	After each initial calibration Second source Std.	± 15%	Check for errors, rerun ICV or recalibrate
Laboratory Control Sample (LCS/LCSD)	Extracted with every batch or every 20 samples whichever is more frequent.	% R 40-140% RPDs <20% waters and <30% soils	Recalibrate, rerun or If problem is not corrected re-extract
Method Blank (MB)	Extracted with every batch or every 20 samples whichever is more frequent.	All analytes < RL	Reanalyze MB, locate source of contamination or report samples with a "B" flag. Re-extract if required by client
Continuing Calibration	After every 10 samples	%R ≤15%	Recalibrate and reanalyze any samples following the CCV
Matrix Spike/Spike Duplicate (MS/MSD)	Extracted every 20 samples (MCP – client specific)	% R 40-140% RPDs <50% for Aroclors and <30% for Congeners	Check LCS, if recoveries acceptable in LCS, matrix interference

			presumed
Surrogate	All samples and QC analyses – 2 compounds	%R 30-150%	Reanalyze sample, if still out re-extract unless obvious matrix interference is present.
Internal Standard	QC and samples	50-200% area counts of associated CC std.	If one or more outside of CL, re-analyze. c/n exceedances.
MDL	Run yearly	Requires QA Officer; Lab Director Approval	Rerun if not approved

12.1 Preventative Maintenance

The analytical system requires periodic maintenance. The frequency of maintenance will depend on the type of samples being analyzed. The more complex the matrix, the more contaminants are deposited in the injection port, analytical column and detector. Normal maintenance would require replacing the injection liner and removing 6 inches from the beginning of the analytical column. Active sites from sample matrix may accumulate in these areas causing low recovery. More extensive maintenance would be to bake ECD at 400°C at an extended period of time. Any abnormalities should be brought to the attention of the organic supervisor. If contamination is suspected in the instrument, blanks should be analyzed for confirmation. If contamination is present, notify organic supervisor for appropriate corrective actions.

13.0 CALIBRATION AND STANDARDIZATION INSTRUMENT CALIBRATION

13.1 Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration. The initial calibration is developed using a equal mixture of aroclors 1016 and 1260 along with surrogates tcmx and dcb at 1/10 the concentration of the aroclors. A minimum of five concentration levels that bracket the linear range of the detector are required. Unless otherwise required by a specific project, the analysis of all other pcb aroclors employ a single point calibration. A single calibration standard of each of the aroclors 1221, 1232, 1242, 1248, and 1254 near the mid-point of the expected calibration range is included with the initial calibration as well as the continuing calibration sequence to assist in pattern recognition.

13.2 A 2ul injection of each calibration level is made and the resulting peak areas divided by the mass injected gives a calibration factor. A minimum of three peaks from each aroclor is used for quantitation. Peaks selected must be at least 25% of the height of the largest peak in the given aroclor pattern. The mean and the standard deviation of the calibration factors for each selected peak in the five levels is calculated. If the percent relative standard deviation or the standard deviation divided by the average of the calibration factors is ≤ 20% than the response of the instrument is considered linear and the mean response factor can be used to quantitate samples. Alternatively a linear regression curve fit can be used provided the correlation coefficient is ≤ 0.99.

14.0 PROCEDURES

14.1 Work lists: Work &hold time lists are used to keep track of sample analysis backlog and hold time expirations. Each list shows sample id's, client names, due date, date sampled, and hold days remaining. These lists are generated from the laboratories LIMS system and are generated on a daily basis.

14.2 Walk-in Cooler: Samples are found in the walk-in cooler located near sample receiving.

14.3 Extraction Logbooks:
In the appropriate log book, fill in all relevant information for the extraction batch including, initials, date, sample ph prior to adjustment, sample ID's, volume used, reagent and standard lot numbers, date concentrated, final volume, and any comments or observations noted. In addition, the left-hand margin is used to keep track of batch QC. At the beginning of a batch, samples are numbered 1-20. When 20 is reached another full set of QC is extracted. Method blanks are extracted every day and one every additional 20 samples extracted in a day. When a logbook is created, a unique volume number is assigned and each page is paginated. Logbook pages should never be removed. If a mistake is made, void the page by drawing a single line through it and initial and date the bottom.

14.4 Glassware:
All glassware used must be scrupulously cleaned before use in trace analysis. Wash glassware in hot soapy water followed by rinsing with de-ionized water. Bake all glassware prior to re-use.

14.5 Sample preparation
For a more detailed explanation of the specific extraction procedures (SW846 3510, 3540, 3546 or 3550), consult the related extraction sop.

14.6 Water:
One liter of water sample is measured into a separatory funnel. Before extraction samples are brought to room temperature and the pH is recorded in the extraction logbook. If the pH is outside the neutral range it is noted on the report. No adjustment is made to the pH. 0.5 mLs of surrogate solution is added to each blank, sample, and spike. 0.5 mLs of matrix spiking solution is added to each lab

control spike and matrix spike. After extraction samples are concentrated to approx. 1ml then solvent exchanged with 20mls of hexane. The final volume of hexane extract is brought to 5.0mls. Samples are now ready for analysis and/or clean-up. EPA Method SW 3510C.

14.7 Soil:

Ten grams of soil sample is measured to .01grams and recorded into the extraction logbook. Enough sodium sulfate is added until a free flowing mixture is obtained without clumping. 1.0 mls of surrogate solution is added to each blank, sample, and spike. 1.0 mls of matrix spiking solution is added to each lab control spike and matrix spike. The samples are then extracted with hexane-acetone (1:1) using either soxhlet extractors, microwave extraction or ultrasonication extraction. The extraction solvent is then concentrated to 10mls in hexane. Solvent exchange is not necessary because all the acetone is driven off during concentration. Samples are now ready for analysis and/or clean-up. EPA Method SW-846 3540C, 3546 or 3550B.

For project specific requirements, the following optional step is used prior to extraction of PCB samples

Dry a portion of the soil sample at 60 degrees C for at least 12 hours. Place 20 grams of the sample in the extraction device. Extract sample with Methylene chloride.

14.8 Extract clean-up

Extracts may be subject to various clean-up procedures to eliminate interfering compounds. The analyst may choose to clean-up an extract initially based on appearance or after analysis shows an interfering matrix in the chromatogram. If a sample requires a clean-up procedure then all associated QC needs the same clean-up performed.

The following procedures are used for pcb extract clean-up

14.8.1 Sulfuric acid may be used to remove many common interferences. This procedure is usually the most effective removal of interferences in pcb extracts. Add 1ml or less of sulfuric acid to a 2ml vial. Using a pipet slowly add approximately 1ml of extract, if excessive heat or bubbling occurs add the extract more slowly. Let layers settle out and pipet cleaned extract off the top. If an emulsion forms and layers don't separate, use less acid and more extract. Perform clean-up on all QC samples also.

14.8.2 Copper may be used to remove elemental sulfur which obscures much of the chromatographic regions of interest.

Concentrate the sample to exactly 1.0 mL or other known volume. If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable

pipet leaving the excess sulfur in the concentration vessel. Transfer 1.0 mL of the extract to a calibrated centrifuge tube.
Add approximately 2 g of cleaned copper powder to the centrifuge tube. (The copper will fill the tube to approximately the 0.5 mL mark).
Vigorously mix the extract and the copper powder for at least 1 min on the mechanical shaker. Allow the phases to separate.
7.1.4 Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1.0 mL of extract.

14.9 GC conditions
This procedure uses a dual column dual injection technique that enables a primary and confirmatory analysis to be run simultaneously. The following table is a summary of instrument operating conditions. Helium is used as a carrier gas and argon-methane (P5) is used as the ECD make-up gas. Samples must be run under conditions identical to the calibration.

	HP 5890 Series II
Column Head Pressure (psi)	10 (Initial Conditions)
Column Flow (mL/min)	1 (Constant Flow - EPC)
Make-up Flow (mL/min)	60
Anode Purge Gas Flow	Fully open
Septum Purge Flow (mL/min)	2
Split Vent Flow (mL/min)	50
Initial T (degrees C)	100 for 1 min
Rate (1) (degree C/min)	8
Final T (degrees C)	280 for 10 min
Injector T (degrees C)	250
Detector T (degrees C)	320

15.0 CALCULATIONS

Sample analysis:
15.1 Absolute retention times are used for tentative compound identification. Retention

time windows are ±0.1 minutes by default. Alternatively the retention time window can be established by using ± 3sd of three standards run within a 72 hour period. Generally with electronic pressure control of the inlet, retention times will be very consistent and vary less than 0.02 minutes. Tentative identification based on retention time must be confirmed on a second GC column of dissimilar stationary phase. A tentative peak is considered confirmed when it falls within the retention time window of each column and the % difference of the results are <40%.

15.2 The calibration curve must be verified initially with a standard from a separate source (ICV) and every 12-hour shift or once every 20 samples by the measurement of a calibration standard or CCV. The % difference between the initial calibration mean response factors and the continuing calibration response factors must be <15% or a new calibration curve must be prepared for any compounds that fail. Additionally a closing calibration verification must be analyzed at the end of the analysis sequence and all samples up to the last passing CCV must be reanalyzed.

15.3 Samples extracts are injected at 2ul aliquots and the resulting peak areas are used to calculate analyte concentration. External calibration is used to calculate concentration. The area of a confirmed peak is divided by the average response factor or alternatively applied to a linear regression formula. This number is µg/L in the concentrated extract, which is the number from the instrumental quant report. This number is multiplied by the volume of the extract in liters (e.g. 10ml=0.01L). This is the microgram equivalent in the extract which is then divided by the initial volume of sample extracted. For waters this number is expressed in liters (usually 1). For soils this is expressed as kilograms of dry sample (usually 0.01). Dry sample weight is obtained by multiplying the kilogram weight by the determined % solids expressed as 1 being equal to 100%.

15.4 The working range of this method is limited to the range of the calibration curve. All extracts with analyte concentrations exceeding the upper limit must be diluted so the result falls within the range of the calibration curve.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.2.1 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.2.2 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.2.3 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

18.2.4 The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Officer should be contacted if additional information is required. See section 17 for more information on pollution control.

22.0 REFERENCE

22.1 EPA SW-846 Manual and Method 8082, Rev. 0, 1996.
22.2 Operation Manuals for the HP 5890 Series II GC, Enviroquant user quide.
22.3 Operation Manuals for the HP autosampler 7673A, Hewlett Packard.

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STANDARD OPERATING PROCEDURE

Analytical Procedures for the Analysis of Organochlorine Pesticides

Method SW846 8081A

SOP ID: GCS00100.MA

Revision (2)

Date Written: 04/01/99
Date Revised: 03/24/05
Date Effective: 1/10/06

Approved Signatures

QUALITY ASSURANCE COORDINATOR

LABORATORY DIRECTOR

STL Westfield
Standard Operating Procedure (SOP)
Change Form**{PRIVATE }**

SOP Number/Revision No.: 3 Last Mod ID (circle): NA / _____
SOP Title: organochlorine Pest.
Affected SOP Section Number(s): 14.6
Effective Date: January 06

CONTROLLED DISTRIBUTION

COPY # :

ISSUED TO :

Revision Number with Mod ID: 4

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. ***Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.

☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.

☑ Procedural Changes (Define Below) – Re-Training Required.

- Remove reference to Cont. L/L extractor
- Correct wording on pH (method 3510 states adj.) Sec. 7.3 "extract with pH range of 5-9.

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Approval Signature/Date: Laboratory Supervisor

Approval Signature/Date: Quality Manager or Designee

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1.0 TITLE

Analytical Procedure For Organochlorine Pesticides by Gas Chromatography, EPA SW-846, Method 8081A

2.0 MATRIX

Solid and liquid matrices

3.0 METHOD REPORTING LIMIT

3.1 Waters – 0.01µg/L with the exception of Toxaphene at 5µg/L and Chlordane at 0.05µg/L.

3.2 Soils – 10 µg/kg, with the exception of Methyoxychlor at 20, µg/kg Toxaphene at 500µg/kg and Chlordane at 50 µg/kg.

4.0 SCOPE AND APPLICATION

4.1 This procedure is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices using capillary column chromatography with electron capture detectors (ECD). The target compounds are listed in table 1. This method is applicable to water, soil, sediment, and sludge.

4.2 The two multi-component mixtures listed as target analytes in table 1 are chlordane and toxaphene. These mixtures are identified by pattern recognition and require a higher level of expertise in identification and quantitation.

4.3 Sample extracts maybe subject to various clean-up procedures to remove interferences from non-target matrices. Compound identification is confirmed automatically with dual column injections. The practical quantitation limit for this method is 0.1-2.0 ug/L for waters and 10-200 ug/kg for soils.

4.4 This procedure is no longer suitable for PCB analysis, which was combined, in previous methods. These are analyzed by method 8082, which include specific clean-up and quantitation procedures designed for PCB analysis.

5.0 SUMMARY OF METHOD

5.1 Either 1liter of water or 10 grams of soil is extracted by using the appropriate matrix-specific extraction technique. Liquids samples are extracted at neutral pH with methylene chloride using a separatory funnel procedure (3510). Soils are extracted with hexane-acetone (1:1) using soxhlet (method 3540) or ultrasonic procedure (3550). The extraction solvent is concentrated, and exchanged to hexane. The final volume of the extract is brought to 10mL. Depending on the

appearance of the extract, e.g. if it is highly colored, the extract may be subject to cleanup with florisil or mercury to remove interferences. 2uL of the extract is injected into a Hewlett Packard gas chromatograph fitted with two 0.32id capillary columns connected with a deactivated glass "Y-spliter" preceded by a 5-meter fused silica guard column. Each column is connected to a separate electron capture detector. Sample extracts are analyzed simultaneously on each column. The connection to the Y-splitter can be problematic and a leak free seal difficult to obtain. Alternatively, both columns can be fitted with a 2-hole ferrule conversion fitting. Compound identification occurs when a peak falls within a defined retention time window on both columns.

TABLE 1
EPA 8081A TARGET COMPOUNDS

NUMBER	ANALYTE	CAS NUMBER
1	Aldrin	309-00-2
2	alpha-BHC	319-84-6
3	beta-BHC	319-85-7
4	gamma-BHC (Lindane)	319-86-8
5	delta-BHC	58-89-9
6	Chlordane	57-74-9
7	4,4'-DDD	72-54-8
8	4,4'-DDE	72-55-9
9	4,4'-DDT	50-29-3
10	Dieldrin	60-57-1
11	Endosulfan I	959-98-8
12	Endosulfan II	33212-65-9
13	Endosulfan Sulfate	1031-07-8
14	Endrin	72-20-8
15	Endrin Aldehyde	7421-93-4
16	Heptachlor	76-44-8
17	Heptachlor Epoxide	1024-57-3

18	Methoxychlor	72-43-5
19	Toxaphene	8001-35-2

6.0 DEFINITIONS

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualititative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the

relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks

7.0 INTERFERENCES

7.1 Interference's in this method can result from contaminated solvents, reagents, glassware, or other sample processing hardware, as well as contaminated GC gases, parts, columns, and detectors. Interference's can also result from compounds co-extracted from the sample matrix to which the detector will respond. In this case a variety of extract clean-up procedures are available to eliminate the problem. Phthalate esters are particularly problematic to this method. These

compounds are present in many plastics and can be leached out especially when coming into contact with extraction solvents. Avoiding contact with any plastic materials during sample extraction can minimize this problem. Exhaustive clean up of solvents reagents and glassware may be required to eliminate background levels of phthalates. Method blanks are treated identical to samples and are processed with every batch to ensure no contamination is present. Glassware must be scrupulously cleaned with detergent and hot water, rinsed with tap water and organic free reagent water, or by rinsing with methanol or acetone to dry and methylene chloride to remove impurities.

8.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9.0 APPARATUS

9.1 Hewlett Packard 5890 Series II Gas Chromatograph equipped with dual electron capture detectors and model 7673 autosampler. Complete with septa, inlet liner, carrier and make-up gas helium, argon/methane.
9.2 Hewlett Packard Chemstation/Enviroquant PC based data system.
9.3 Column #1: Restek Rtx-CLPesticides 30meter x 0.32mm ID; 0.5um df
9.4 Column #2: Restek Rtx-CLPesticides2 30meter x 0.32mm ID; 0.25um df
9.5 Guard column: Restek Siltek 5meter x 0.32mm ID
9.6 Siltek press tight deactivated "Y-splitter"
9.7 2-liter separatory funnels
9.8 Soxhelet glassware
9.9 Hotplates
9.10 Tekmar model TM500 ultrasonic disrupter
9.11 Low pressure liquid nitrogen tank
9.12 Condensers with water supply
9.13 Zymark Turbo-vap II, nitrogen blowdown device and associated glassware.
9.14 Syringes, volumetric flasks, graduated cylinder, autosampler vials, instrument supplies, pipets and other accessories
9.15 Analytical balance capable of weighing 0.1mg.

10.0 REAGENTS and STANDARDS

10.1 Reagent grade (ACS) chemicals shall be used in all tests.
The date received must be recorded on all reagent labels. Consult the Safety manual for proper handling and storage of reagents. Whenever the lab receives new lots of reagents they must be screened to show they are free of contaminants. Analyze an equivalent portion of the reagent as would be used for a sample to demonstrate it is free of contaminants. The following is a list of reagents that may be used in analysis:
10.2 Organic free reagent water.
10.3 Pesticide grade acetone, methanol, methylene chloride, hexane, toluene, diethyl ether.
10.4 Florisil cartridges (supelco cat# 5-7053)
10.5 Sodium sulfate
10.6 Dimethyldichlorosiloxane
10.7 Primary standards are purchased as certified solutions or as pure material.
Table 2 lists the commercial sources available that provide the necessary quantitation and calibration materials for this method.
10.8 Intermediate stock standards. Standards used for calibration are generally in the ppb range. Intermediate solutions are required to accurately achieve these low concentration levels. Table 3 lists the dilutions of the stock standard materials that

may be used to accomplish this.

10.9 Working standards are prepared from the intermediate stocks and are recorded in the GC working standards logbook with description of contents, date of preparation, initial and final volumes, concentration, dilution solvents, lot numbers, and expiration dates. Each working standard is assigned a unique lot number based on page number and volume of the logbook. This number must also be recorded onto the instrumental software data file to enable traceability from the final quantitation report. Unless super-ceded by the method, expiration dates are 6 months from date of preparation for semi-volatile standards and 1month for volatile standards. Standards purchased as certified solutions often have expiration dates of their own. If this date expires sooner than the 6 months than that is the expiration date. Standards with expired dates must discarded. It is sometimes possible to get extended expiration dates by calling the manufacturer with the lot number. A certificate must be obtained before the standard can be used.

10.10 Standards integrity is verified by purchasing second source standards of a different lot number.

10.11 Calibration standards are prepared at a minimum of five levels for each pesticide. Table 4 is a summary of the steps required to formulate these five levels. Two multi-component pesticides, chlordane and toxaphene are quantitated by single point calibration only.

10.12 Spiking solutions used to monitor the extraction performance are prepared in acetone. A 1.0mg/L solution of tetrachloro-m-xylene and decachlorobiphenyl is used for the surrogate spike. From this solution, an aliquot of 1.0mL is spiked into all samples, blanks, and matrix spikes, giving a final extract concentration of 100ug/L. A 1.0mg/L solution of each single component pesticide is used for matrix spiking. From this solution, an aliquot of 1.0mL is spiked into matrix spikes and lab control spikes to give an extract concentration of 100ug/L.

TABLE 2

Commercial Blends Required for Production of Method Standard Solutions

#	ITEM	VENDOR	CAT #	CONC.	SOLVENT	USE
1	Single Component Blend	Supelco	4-8913	2000 mg/L	Tol/Hex (50:50)	Quantitation
2	Single Component Blend 2nd Source	Supelco	4S-8913	2000 mg/L	Tol/Hex (50:50)	QC Check

3	Toxaphene	Ultra Scientific	PP-271	100 mg/L	Hexane	Quantitation
4	Chlordane	Ultra-Scientific	PP-151	100 mg/L	Hexane	Quantitation
5	DDT/Endrin mix	Supelco	4-8282	500 mg/L	Methanol	QC Check
6	TCMX/DCBmix	Accu-standard	CLP-032-R	200 mg/L	Acetone	Quantitation

TABLE 3

Formulation of Intermediate Concentration Stock Solutions.

NUMBER FROM TABLE 2	ITEM	Volume (µL)	Final Vol. (mL)	SOLVENT	FINAL CONCENTRATION (mg/L)
1	Pesticide mix	50	100	hexane	1.0
6	TCMX/DCB	500			1.0
2	Pesticide mix 2nd Source	50	100	Hexane (acetone for spiking)	1.0
6	TCMX/DCB	500			
3	Toxaphene	100	10	hexane	1.0
4	Chlordane	100	10	hexane	1.0
5	DDT/Endrin mix	20	100	hexane	0.1
6	TCMX/DCB mix	50	10	acetone	1.0

TABLE 4

Standard Formulation for EPA Method 8081A.

- Refers to Intermediate Dilutions From Table 3.

Volume #1,6 or #2,6 (µL)	Final Vol. (mL)	SOLVENT	FINAL CONCENTRATION (µg/L)
10	1.0	Hexane	10
20	1.0	Hexane	20
50	1.0	Hexane	50
100	1.0	Hexane	100
200	1.0	Hexane	200

11.0 SAMPLE HANDLING & PRESERVATION

11.1 Water samples are collected in 1 liter amber bottles. Soil samples are collected in 250 mL wide mouth jars. Both are preserved at 4°C.

11.2 Samples are shipped to the laboratory on ice.

11.3 Samples are stored in a secured refrigerator at 4°C.

11.4 Holding time: Waters are required to be started within seven days of sample collection and soils and concentrated wastes are required to be started within fourteen days of sample collection.

11.5 All sample extracts must be analyzed within forty days of extraction.

12.0 QC ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	At start-up and when cont. cal fails	r-value ≥ 0.99 for all method analytes (or <20% RSD on RF's)	check for injection problems reanalyze std. or recalibrate
Initial Calibration Verification (ICV)	After each initial calibration	See table #2 All analytes ± 15% R.	Check for errors, rerun ICV or recalibrate
Laboratory Control Sample (LCS/LCSD)	Extracted with every batch or every 20 samples whichever is more frequent.	% R must be between 40-140% RPDs be <20 waters and <30% soils	Correct problem, rerun LCS or recalibrate
Method Blank (MB)	Extracted with every batch or every 20 samples whichever is more frequent.	All analytes < RL	Clean system, reanalyze MB or report samples with a "B" flag
Continuing Calibration	Prior to samples, every 12 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. 50 ug/L if linearity permits.	All analytes ± 15% R.	Correct Problem rerun Cont. Cal. If still out recalibrate & rerun sample
Matrix Spike/Spike Duplicate (MS/MSD) [Client Specific]	Extracted every 20 samples (BATCH QC)	Percent recoveries must be between 30-150% RPDs should be <30 for single component and <50 for multicomponent analytes.	Check LCS, if recoveries acceptable in LCS, matrix interference presumed
Surrogate	Minimum of 2, one that elutes at beginning of GC run and one that elutes at end of GC run.	Percent recoveries must be between 30-150% for both surrogates on both columns	If same surrogate outside on both columns, re-extract sample
MDL	Run yearly	Requires QA Officer; Lab Director Approval	Rerun if not approved

13.0 CALIBRATION AND STANDARDIZATION

13.1 Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration. After the instrument sits idle for an extended period, injection of a high concentration pesticide standard approx 20 times more concentrated than the mid level standard may help to reduce active sites. To ensure the injection port is not active a solution containing DDT and endrin only is injected in to the GC. The presence of DDD, DDE, endrin aldehyde, or endrin ketone indicates breakdown. Using the formula below the sum of the breakdown products must be less than 15% before analysis can begin.

DDT %breakdown = DDD + DDE / DDD + DDE + DDT

Endrin % breakdown = End ald + End ket / End ald + End ket + End

The initial calibration consists of five concentration levels that bracket the linear range of the detector. Unless otherwise required by a specific project, the analysis of multi-component analytes employs a single point calibration. A single calibration standard of chlordane and toxaphene near the mid-point of the expected calibration range is included with the initial calibration of the single component analytes for pattern recognition

13.2 A 2ul injection of each calibration level is made and the resulting peak areas divided by the mass injected gives a calibration factor. The mean and the standard deviation of the calibration factors for each analyte in the five levels is calculated. If the percent relative standard deviation or the standard deviation divided by the average of the calibration factors is ≤ 20% than the response of the instrument is considered linear and the mean response factor can be used to quantitate samples. Alternatively a linear regression curve fit can be used provided the correlation coefficient is ≤ 0.99

13.3 PREVENTIVE MAINTENANCE

13.3.1 The analytical system requires periodic maintenance. The frequency of maintenance will depend on the type of samples being analyzed. The more complex the matrix, the more contaminants are deposited in the injection port, analytical column and detector. Normal maintenance would require replacing the injection liner and removing 6 inches from the beginning of the analytical column. Active sites from sample matrix may accumulate in these areas causing low recovery. More extensive maintenance would be to bake ECD at 400°C at an extended period of time. Any abnormalities should be brought to the attention of the organic supervisor. If contamination is suspected in the instrument, blanks should be analyzed for confirmation. If contamination is present, notify organic

supervisor for appropriate corrective actions.

14.0 PROCEDURES

14.1 Work lists:
Work/hold time lists are generated by the laboratory's LIMS and are used to keep track of sample backlog and hold time expirations. Each list shows sample id's, client names, due date, date sampled, and hold days remaining. Any computer in the lab connected to LIMS is capable of generating these backlogs.

14.2 Walk-in Cooler: Samples are found in the walk-in cooler located near sample receiving.

14.3 Extraction Logbooks:
In the appropriate log book, fill in all relevant information for the extraction batch including, initials, date, sample ph prior to adjustment, sample ID's, volume used, reagent and standard lot numbers, date concentrated, final volume, and any comments or observations noted. In addition, the left-hand margin is used to keep track of batch QC. At the beginning of a batch, samples are numbered 1-20. When 20 is reached another full set of QC is extracted. Method blanks are extracted every day and one every additional 20 samples extracted in a day. When a logbook is created, a unique volume number is assigned and each page is paginated. Logbook pages should never be removed. If a mistake is made, void the page by drawing a single line through it and initial and date the bottom.

14.4 Glassware:
All glassware used must be scrupulously cleaned before use in trace analysis. Wash glassware in hot soapy water followed by rinsing with de-ionized water. A solvent rinse this then employed prior to use. Glassware is ready to use.

14.5 Sample preparation
For a more detailed explanation of the specific extraction procedures mentioned, consult the related sop.

14.6 Water:
One liter of water sample is measured into either a separatory funnel or continuous liquid-liquid extractor. Before extraction samples are brought to room temperature and the pH is recorded in the extraction logbook. If the pH is outside the neutral range it is noted on the report. No adjustment is made to the pH. 1.0 mLs of surrogate solution is added to each blank, sample, and spike. 1.0 mLs of matrix spiking solution is added to each lab control spike and matrix spike. After extraction samples are concentrated to approx. 1-5ml then solvent exchanged with 20mls of hexane. The final volume of hexane extract is brought to 10.0mls. Samples are now ready for analysis and/or clean-up. EPA Extraction Method SW 3510C.

14.7 Soil:
Ten grams of soil sample is measured to 0.01grams and recorded into the extraction logbook. Enough sodium sulfate is added until a free flowing mixture is

obtained without clumping. 1.0 mls of surrogate solution is added to each blank, sample, and spike. 1.0 mls of matrix spiking solution is added to each lab control spike and matrix spike. The samples are then extracted with hexane-acetone (1:1) using either soxhlet extractors, microwave digestionor ultrasonication. The extraction solvent is then concentrated to 10mls in hexane. Solvent exchange is not necessary because all the acetone is driven off during concentration. Samples are now ready for analysis and/or clean-up. EPA Method SW-846 3540C, 3546 or 3550B.

14.8 Extract clean-up

Extracts may be subject to various clean-up procedures to eliminate interfering compounds. The analyst may choose to clean-up an extract initially based on appearance or after analysis shows an interfering matrix in the chromatogram.
The following procedures are used for pesticide extract clean-up:

14.8.1 Elemental sulfur obscures much of the chromatographic regions of interest. It is removed by shaking 1ml of the extract with 1-2 drops of mercury in a glass vial with teflon lined screw cap. Shake mixture for 1-2 mins., let settle, transfer 1 ml to autosampler vial. The presence of sulfur is indicated by the appearance of black mercuric sulfide precipitate. Extended mixing times or multiple additions of mercury may be required.

14.8.2 Polar interferences are removed by passing the extract through florisil cartridges. With the valve in the closed position, place a 1-gram florisil cartridge onto a vacuum manifold and set vacuum to 10in of mercury. Condition the cartridge by adding 4ml of hexane to each cartridge. Slowly open the valve to allow hexane to pass through the sorbent bed to the lower frits. Allow a few drops to pass through to remove air bubbles. Close valve and allow hexane to soak the sorbent bed for five minutes. Do not turn off vacuum. Slowly open valve to allow the hexane to pass through cartridge. Close the valve when there is still 1mm of solvent above the sorbent bed. Do not allow the cartridge to go dry. Transfer 1ml of extract to the cartridge. Open the valve to allow the extract to pass through at 2ml/min. Close the valve after the entire extract passes through but before the cartridge becomes dry. Add 9ml of acetone/hexane (10/90 v/v) to the cartridge, open valve, and slowly collect the eluate into a clean vial. Reconcentrate to 1ml in the turbovap.

14.9 GC Conditions

This procedure uses a dual column dual injection technique that enables a primary and confirmatory analysis to be run simultaneously. The following table is a summary of instrument operating conditions. Helium is used as a carrier gas and argon-methane (P5) is used as the ECD make-up gas. Samples must be run under conditions identical to the calibration.

	HP 5890 Series II
Column Head Pressure (psi)	10 (Initial Conditions)
Column Flow (mL/min)	1 (Constant Flow - EPC)
Make-up Flow (mL/min)	60
Anode Purge Gas Flow	Fully open
Septum Purge Flow (mL/min)	2
Split Vent Flow (mL/min)	50
Initial T (degrees C)	100 for 1 min
Rate (1) (degree C/min)	8
Final T (degrees C)	280 for 10min
Injector T (degrees C)	250
Detector T (degrees C)	320

15.0 CALCULATIONS

Sample analysis:

15.1 Absolute retention times are used for tentative compound identification. Retention time windows are ±0.1 minutes by default. Alternatively the retention time window can be established by using ± 3sd of three standards run within a 72 hour period. Generally with electronic pressure control of the inlet, retention times will be very consistent and vary less than 0.02 minutes. Tentative identification based on retention time must be confirmed on a second GC column of dissimilar stationary phase. A tentative peak is considered confirmed when it falls within the retention time window of each column and the % difference of the results are <40%.

15.2 The calibration curve must be verified initially with a standard from a separate source (ICV) and every 12 hour shift or once every 20 samples by the measurement of a calibration standard or CCV. The % difference between the initial calibration mean response factors and the continuing calibration response factors must be <15% or a new calibration curve must be prepared for any compounds that fail. Additionally a closing calibration verification must be analyzed at the end of the analysis sequence and all samples up to the last passing ccv must be reanalyzed.

15.3 Samples extracts are injected at 2ul aliquots and the resulting peak areas are used to calculate analyte concentration. External calibration is used to calculate

concentration. The area of a confirmed peak is divided by the average response factor or alternatively applied to a linear regression formula. This number is µg/L in the concentrated extract, which is the number from the instrumental quant report. This number is multiplied by the volume of the extract in liters (e.g. 10ml=0.01L). This is the microgram equivalent in the extract, which is then divided by the initial volume of sample extracted. For waters this number is expressed in liters (usually 1). For soils this is expressed as kilograms of dry sample (usually 0.01*%solids). Dry sample weight is obtained by multiplying the kilogram weight by the determined % solids expressed as 1 being equal to 100%.

15.4 The working range of this method is limited to the range of the calibration curve. All extracts with analyte concentrations exceeding the upper limit must be diluted so the result falls within the range of the calibration curve.

15.5 Identification of Toxaphene and Chlordane is based on pattern recognition. 3-5 major characteristic peaks (>25% of the largest peak) are selected for quantitation. The sum of the peak area is used to calculate concentration based on a response factor generated from the same peaks present in the calibration standards of chlordane or toxaphene.

16.0 METHOD PERFORMANCE

16.1 The group supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

16.2 Method Detection Limit Study (MDL). An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually.

16.3 Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

17.0 POLLUTION PREVENTION

17.1 This method does not contain any specific modifications that serve to prevent or minimize pollution.

18.0 DATA ASSESMENT & ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

18.1 Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their

frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.

18.2 All data, regardless of regulatory program or level of reporting (Level1,2,3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.

18.3 Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.

18.4 Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.

18.5 Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.

The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

19.1 If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL or UNACCEPTALBE DATA

20.1 If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective action, based on the client's direction, may be taken. Action can also take the

form of reporting data, and including the non-conformance comment in the project narrative.

21.0 WASTE MANAGEMENT

21.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention" and according to the facility hazardous waste procedures.

22.0 REFERENCE

22.1 EPA SW-846 Manual and Method 8081A, Rev. I, 12/1996.
22.2 Quality Assurance and Quality Control Requirements for *SW-846 Method 8081A, Chlorinated Pesticides by Gas Chromatography* (GC) for the Massachusetts Contingency Plan (MCP),WSC-CAM, Rev 4, Aug 20, 2004.
22.2 Operation Manuals for the HP 5890 Series II GC, Enviroquant user quide.
22.3 Operation Manuals for the HP autosampler 7673A, Hewlett Packard.

23.0 TABLES, DIAGRAMS, FLOWCHARTS VALIDATION DATA: NONE

Title: DETERMINATION OF CARBONYL COMPOUNDS IN LIQUIDS AND SOILS BY HPLC (EPA SW-846 METHOD 8315)

Approvals (Signature/Date):

Chris Amason Technical Manager	4.29.08 Date	Jim Robbins Health & Safety Manager / Coordinator	4/29/08 Date
Lou Schneider Quality Assurance Manager	4/29/08 Date	Todd Baumgartner Laboratory Director	4/29/08 Date

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1.0 SCOPE AND APPLICATION

1.1 This method can be used to determine the concentration of formaldehyde (CAS #50-00-0), acetaldehyde (CAS #75-07-0), and propionaldehyde (CAS #123-38-6) by high performance liquid chromatography (HPLC).

1.2 This procedure is applicable to water, leachates and soil samples.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Sample preparation is described in SOP TL-LC-046: *Derivatization and Extraction of Liquids and Soils for Carbonyl Compounds.*

The extracts are analyzed by reversed-phase liquid chromatography using ultraviolet (UV) detection.

2.2 This procedure is based on the guidance provided in EPA SW-846 Method 8315. The reporting limits (RL) are listed in the current version of TL-QA-001: *TestAmerica Tallahassee Method/Analyte List.* The method detection limits (MDL) and control limits are listed in the current version of TL-QA-002: *TestAmerica Tallahassee Quality Control Limit Summary*, and in the TALS LIMS.

2.3 Definitions

2.3.1 Elute: to extract one material from another, usually by means of a solvent (in this case, ACN)

2.3.2 Isocratic: a chromatographic separation that employs a single solvent of constant composition

2.3.3 Laboratory Fortified Blank (LFB): An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required MDL. Also referred to as a laboratory control standard (LCS).

2.3.4 Laboratory Fortified Sample Matrix (LFM): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. Also referred to as a matrix spike (MS).

2.3.5 Laboratory Reagent Blank (LRB): An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus. Also referred to as a method blank (MB).

2.3.6 Procedural Standard Calibration: A calibration method where aqueous calibration standards are prepared and processed (e.g. extracted and derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiency in the processing procedure.

2.3.7 Standard Operating Procedure (SOP): An instructional document relating to the operation of facilities or equipment, or providing "how-to" directions for performing a specific activity or function.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the facility addendum to the CSM, and this document.

3.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

There are no specialized safety concerns associated with this method.

3.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Formaldehyde	Flammable, Poison	0.37 mg/m3 per 10 hrs	May cause sore throat, coughing, and shortness of breath. If ingested can cause severe abdominal pain, violent vomiting, headache, and diarrhea. Larger doses may produce decreased body temperature, pain in the digestive tract, shallow respiration, weak irregular pulse, unconsciousness and death. Methanol component affects the optic nerve and may cause blindness. May cause irritation to skin with redness, pain, and possibly burns. Skin absorption may occur with symptoms paralleling those from ingestion. Vapors cause irritation to the eyes with redness, pain, and blurred vision.
DNPH	Flammable solid	0.6 mg/m3 per 10 hrs	Explosive if dry. Absorption or ingestion into the body leads to the formation of mathemoglobin, which in sufficient concentration causes cyanosis. Onset may be delayed 2 to 4 hours or longer. Causes eye and skin irritation.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Analytical interferences may arise from improperly cleaned glassware. All glassware must be scrupulously cleaned before use according to SOP TL-AN-060: *Glassware Cleaning Procedures*. After drying, seal and store glassware in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

Acetone forms a stable DNPH derivative that may interfere with the chromatographic analysis of the aldehydes or, if present in high concentrations, may interfere with the complete derivatization of the aldehydes. Glassware should not be cleaned with acetone.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or elevated baselines in liquid chromatograms. All of the materials and reagents must be routinely demonstrated to be free from contaminants under the conditions of the analysis by the preparation and analysis of extracted MBs.

4.3 Matrix-related interferences may be caused by contaminants that are extracted from the sample matrix or from co-eluting organic analytes in the sample. Dilution may be required prior to analysis to reduce or eliminate the interferences.

4.4 All extracts for samples and associated quality control (QC) items must be filtered prior to analysis to eliminate particulate matter. Particulates in the mobile phase or sample extracts may cause serious instrument problems. All solutions and extracts should be filtered before use.

4.5 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in an all glass system may be required.

4.6 Interfering contamination may occur when a sample containing low concentrations of target analytes is analyzed immediately following a sample containing relatively high concentrations of target analytes. Programming a gradient "rinse" step of up to 100% organic phase can be used to eliminate interference in an isocratic run.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

5.1 Aqueous and solid samples must be collected in glass bottles or jars with Teflon-lined caps and maintained at 4° C (less than 6° C with no frozen samples) out of direct light. Samples should be iced for shipment. A minimum volume of 200mL should be collected for aqueous samples and a minimum of 40g for solids. Additional volume should be provided for MS samples and dry weights. No preservative is added (orange dot). Aqueous samples must be derivatized and extracted within three days of sample collection. Solid samples must be derivatized within a minimal hold time. Analysis of the derivatized extracts should be completed within three days of extraction for both aqueous and solid samples. This information is summarized in a table in Appendix A.

5.2 Extracts are stored at 4 °C in amber vials.

6.0 MATERIALS AND APPARATUS

6.1 Waters HPLC isocratic 501 pump, gradient 600E pump or equivalent

6.2 Waters 484 or 486 UV detector or equivalent detector with variable wavelength detection capable of achieving the required detection limit

6.3 Waters 717 autosampler, Waters 712 Wisp or equivalent

6.4 Waters HPLC column heater and controller or equivalent

6.5 Recommended analytical column – 15cm x 4.6mm C18 (Supelco LC-PAH or equivalent)

6.6 Microsyringes – appropriate volumes for preparation of standards and dilutions (Hamilton gastight or equivalent)

6.7 Volumetric glassware (Class A) – appropriate volumes

6.8 Autosampler vials – 4mL and 10mL amber with screw caps and septa

6.9 Guard columns – C18

6.10 Helium – For degassing solutions and solvents

6.11 Syringe – 60mL Gastight with 1/16" stainless steel tubing, for pump priming

7.0 REAGENTS

7.1 Reagents must be tracked in accordance with SOP TL-AN-041: *Standard Materials and Reagent Traceability.*

7.2 Reagent water – HPLC grade water (Barnstead Easypure RF or equivalent 18.3MΩ purified water)

7.3 Acetonitrile – HPLC grade

7.4 Methanol – HPLC grade

8.0 STANDARDS

8.1 Standards must be tracked in accordance with SOP TL-AN-041: *Standard Materials and Reagent Traceability.* General guidelines on the preparation of standards are given in SOP TL-AN-043: *Standard Preparation.*

8.2 The standards are procedural standards, meaning they are derivatized and extracted the same as the samples. The standard preparation steps are given in SOP TTL-LC-046: *Derivatization and Extraction of Liquids and Soils for Carbonyl Compounds.*

9.0 SAMPLE PREPARATION

9.1 See SOP TL-LC-046: *Derivatization and Extraction of Liquids and Soils for Carbonyl Compounds.*

10.0 ANALYTICAL PROCEDURES

10.1 Calibration: See SOP TL-AN-067: *Evaluation of Calibration Curves* for general guidance on how to evaluate the calibration data.

10.2 The following table summarizes the recommended concentrations of the five-point calibration curve. Specific information on how to prepare the calibration standards can be found in SOP TL-LC-046. Calibration standards are good for up to three days after being extracted.

Cal. Standard	Intermediate Conc. (µg/mL)	Volume of Intermediate (µL)	Final Volume (mL)	Conc of Cal. Standard (µg/mL)
1	100	20	4.0	0.50
2	100	50	4.0	1.25
3	100	100	4.0	2.5
4	100	500	4.0	12.5
5	100	1000	4.0	25

10.3 Initial Calibration (ICAL)

10.3.1 Initial calibration must be performed at instrument start-up and when continuing calibration checks fail acceptance criteria. Each instrument must be initially calibrated by the analysis of a minimum of five calibration standards. The standards are prepared using the guidance in Section 10.2 and in SOP TL-LC-046 and analyzed using the guidance described below. External standard calibration procedures are used for this method.

- Analyze the calibration standards using the instrument conditions optimized for the target compounds. The injection volume must be kept constant for all analyses – calibration standards, QC sample extracts and client sample extracts.

- Identify each target compound and tabulate the RT, concentration (µg/mL) and the corresponding area for each target compound. The calibration curve may be evaluated as a regression curve or as the response ratio [calibration factor (CF)].

10.3.2 Regression Curve

The concentration (µg/mL) of each target compound is routinely plotted on the x-axis and the corresponding response is plotted on the y-axis.

Linear fits may be used to define the concentration/response relationship. If the correlation coefficient of the regression curve or the coefficient of determination of a higher order fit is greater than 0.99, the curve can be used to quantify samples. The analyst must ensure that the type of regression curve selected accurately defines the concentration/response relationship over the entire calibration range. If the low standard is below the RL, it is not necessary to force a linear fit through the origin.

Note that some client-specific QAPs may require a correlation of 0.995 or greater.

10.3.3 NOTE ON 8000-SERIES METHODS: *Grand Mean Exception*
If the %RSD for any target compound in the initial calibration exceeds 20%, a calibration curve must be constructed and evaluated for that compound. In addition to meeting the correlation coefficient requirements in 13.3.2, the average %RSD for all of the compounds in the initial calibration **must** be less than or equal to 20% for the initial calibration to be used. No single target compound may be out of these limits by more than three times the accepted %RPD. In this case, a single compound must not be out by more than 60%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required since the lab has demonstrated that the lowest standard in the calibration curve (the equivalent of the RL) can be detected.

10.3.4 To use the response ratio (CF) calculate the CF for each target compound at each calibration level. Calculate the average calibration factor (CF_{avg}) for the ICAL levels. Calculate the standard deviation of the calibration factors. Finally, calculate the relative standard deviation (%RSD) of the levels for each target. Examples of these calculations are in Section 11.1.

For the 8000-series methods, if the %RSD of each target compound is less than or equal to 20%, the calibration is assumed to be linear through the origin and the CF_{avg} is used for quantitation.

10.3.5 When more calibration standards are analyzed than required, individual compounds may be eliminated from the lowest or highest concentration level(s) only. If points or levels are eliminated, analyte concentration in samples must fall within the range defined by the resulting curve. In no case should individual points in the middle of a calibration be eliminated without eliminating the entire level.

10.4 The retention time window (RTW) for each target compound must be determined using the guidance provided in SOP TL-AN-066: *Determination of Retention Time Windows for Chromatographic Analyses.* The width of the RTW used to make identifications should be based upon measurements of actual RT variations of standards over the course of a day. Three times the standard deviation of a RT can be used to calculate a suggested window size for a compound. The RTW must be documented and used to calculate the daily RTW that is used to identify the target compounds in samples. A column heater should be used to stabilize the column temperature. This will improve RT reproducibility. Per Sop TL-AN-066, historically stable default windows may be used.

10.5 Initial Calibration Verification Standard (ICVS)

Since the standards are procedural, that is, they undergo all the same preparation steps as the samples, a traditional ICVS is not used. The lab control standards are prepared from a second source and serve as the ICVS. The LCS recovery criteria are applied.

10.5.1 Continuing Calibration Verification (CCV)

10.5.2 A CCV check must be performed every 12 hours or every 20 samples, whichever is more frequent, and at the beginning and end of the run by injecting a mid-level standard. The mid-level standard chosen for the CCV must be periodically switched to prove that the curve can be verified at all levels. Evaluate the percent drift or percent difference of the ICAL. If the drift or difference is less than or equal to 15%, the curve is verified. If the first CCV fails criteria, a second standard may be analyzed. If it also fails, the ICAL must be repeated. All samples analyzed after a failed standard must be reanalyzed. Examples of how to perform these calculations are in Section 11.2.

10.5.3 NOTE ON 8000-SERIES METHODS: *Grand Mean Exception*
If the %difference or %drift for any target compound in the CCV exceeds 15%, the calibration curve can still be used for quantitation if the average %difference or average %drift for all of the compounds in the CCV is less than or equal to 15%. No single target compound may be out of these limits by more than three times the accepted %difference or %drift. In this case, a single compound must not be out by more than 45%.

NOTE: If a target compound that passes by the "grand mean exception" is detected (>RL), the PM is notified via an anomaly report or case narrative. If the targets are <RL, no notification is required.

NOTE: The use of the "grand mean exception" is discouraged and will be eliminated upon the adoption of Method 8000C.

10.6 For projects that require Florida DEP QAS criteria, the lowest concentration calibration standard (at or near the RL) must be analyzed the same day that sample extracts are analyzed [practical quantitation limit (PQL) verification standard] to demonstrate that the RL can be achieved. This criterion is met if the target compounds are detected.

10.7 Sample analysis

10.7.1 Instrument Conditions

10.7.2 The analytical conditions below are provided for guidance. These conditions may be modified to

optimize sensitivity and separation. The analyst must document the analytical conditions if different from those described below.

10.7.3 Analysis Conditions:

Analytical Column = 15cm x 4.6mm C18 (Supelco LC-PAH or equivalent)
Column Temperature = 25°C
Guard Column = C18
Isocratic Mobile Phase = 65:35 Methanol:HPLC water OR 45:55 ACN:HPLC water – degassed with helium.
Detector = 484 or 486 Waters UV
Wavelength = 360nm
Flow Rate = 1.5mL/min
Injection Volume = 10µL
Run Time = 10min
Acquisition Method = A8315

10.7.4 Instrument Start-up

10.7.5 Pump: Place solvent lines in fresh solvent that has been degassed with helium for at least 30 minutes. Remove air from the solvent lines by inserting the 60mL syringe fitting into the tube on the drain valve. Open the drain valve just until you can withdraw solvent. Withdraw about 20mL of mobile phase, close the valve, remove the syringe and discard the solvent. Repeat until the line is free of air bubbles. Continuing presence of bubbles indicates that the solvent is not degassed well enough. When the lines are free of bubbles, turn the flow rate up to the proper rate by 0.5mL/min increments, allowing the pressure to stabilize between increases.

10.7.6 Detector: Turn on the detector power when the rest of the system is turned on. The wavelength is displayed on the front panel. Check for the correct settings.

10.7.7 Allow at least 30 minutes for system equilibration before injecting samples. Temperature stability is critical. During equilibration, a thorough leak-check should be conducted. Check to be sure that all of the parameters are set to the proper values. Make sure the sequence is entered and downloaded. Auto-zero the detector before injecting.

10.7.8 Qualitative Sample Analysis

10.7.9 Remove the samples from the refrigerator, shake them and allow the solids to settle while equilibrating to room temperature. Never inject cold samples. Sample extracts are injected into the HPLC system following calibration. The same injection volume is used for all standards and samples.

A sequence should be set as described in the SOP Summary in AppendixX A. The sequence continues until all samples have been analyzed or until the CCV fails the acceptance criteria. The sequence must end with an acceptable CCV.

Samples should be run in the following order: method blanks, spikes, clean samples and finally, highly contaminated samples.

10.7.10 The evaluation of chromatograms for the target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), whether the peak response falls within the working range of the calibration and the integration of the peaks. Manual integrations must be documented in accordance with SOP TL-AN-065: *Manual Integrations.* The analyst must also take into account the results from the QC items before reporting quantitative data.

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?

10.7.11 Identification of the target compounds is based on RT. The analyst should scan the sample chromatogram for the target compounds on the analytical column. The retention time windows calculated around the CCV retention times are used for the identification of the target compounds. Identify a sample component by comparison of its RT to the RT of a reference chromatogram. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms. If the RT of an unknown compound corresponds, within limits, to the RT of a standard compound, then identification is considered positive. If, in the professional judgment of the analyst and supervisor, a peak within the RTW can be reasonably excluded as a target, the result may be reported as a "non detect".

NOTE: It is important to note that the RTW applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant RT shifts; therefore, all peaks that have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are diluted and re-analyzed.

10.7.12 The data system automatically labels and calculates the concentration of each peak that corresponds to a target compound. Peak height or area is proportional to analyte concentration. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences and the overall shapes of the peaks.

Identification requires expert judgment when sample components are not resolved chromatographically. When peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or a valley between two or more maxima) or any time doubt exists over the identification of a peak on a chromatogram, appropriate alternate techniques need to be employed to help confirm peak identification. For example, more positive identification may be made by the use of an alternative detector that operates on a chemical or physical principle different from that originally used; e.g. mass spectrometry.

If the concentration is below the detection limit, the RL for that compound is reported (Section 11.3). Peaks over-range are handled using dilutions as detailed below.

10.7.13 If the concentration of any target compound exceeds the calibration range (that is, the highest level standard included in the ICAL), a dilution must be performed on the extract and the diluted extract is reanalyzed. Dilutions should be made in ACN. A dilution should be made to bring the area of the peak of interest into the upper half of the calibration curve; that is, between the mid-level calibration standard and the highest level standard. An example of this calculation is in Section 11.4. If the DNPH peak is present on the chromatogram, the extract can be diluted for re-analysis. If no DNPH peak is detected, the extraction is repeated with a smaller sample aliquot.

10.7.14 The analyst must clearly show how the reported sample results were determined. All analyst interactions with the data system must be clearly traceable either by paper documentation or an electronic audit trail. False hits identified as targets by the data system should be marked as "undetected" via the data system. Manual calculations and integrations must be documented (paper and/or electronic) in accordance with SOP TL-AN-065: *Manual Integrations* and available for inspection. The analyst and reviewer must sign and date the appropriate review document(s) to signify that the reported data are consistent with the raw data.

10.8 Identification "Tools"

10.8.1 Chromatogram "Overlay"

A useful "tool" that employs a similar idea to the relative retention time (RRT) is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual clues to the identification of the target compound. The RT should remain fairly constant under the same instrument conditions. The analyst must be alert for the presence of matrix interferences and evaluate the data on the column before making an identification.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Initial Calibration Calculations

Calculate the CF for each target compound at each calibration level:

$$CF = \frac{response}{concentration} = \frac{area}{ug/mL}$$

Response = peak area or peak height

Calculate the average CF for the ICAL levels:

$$CFavg = \frac{CF1 + CF2 + CF3 ... + CFn}{n}$$

Note that n must be greater than or equal to 5 for the 8315 method.

Calculate the standard deviation of the calibration factors:

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF_{avg}})^2}{n-1}}$$

Where
CF_i = CF of the individual calibration level
CF_{avg}= average CF

Calculate the %RSD of the levels for each target:

$$\%\,RSD = \frac{standard\ deviation}{\overline{CF_{avg}}} \otimes 100$$

11.2 Continuing Calibration Calculations

11.2.1 If a regression curve has been chosen to quantify the samples; the percent difference is calculated for the CCV:

$$\%difference = \frac{Ct - Cccv}{Ct} \otimes 100$$

Where
Ct = expected concentration of the CCV
Cccv = concentration of CCV determined from regression curve

11.2.2 If the CF_{avg} has been used to evaluate the calibration curve, the percent difference is calculated as follows:

$$\%difference = \frac{CFavg - CFccv}{CFavg} \otimes 100$$

Where
CFccv = CF of the target compound from CCV standard
CFavg = average CF of the target compound (ICAL)

11.3 Calculations – External Standard

11.3.1 Aqueous Samples

Aqueous Samples – Regression curve option

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

Where
C_{curve} = concentration of analyte from curve (µg/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Aqueous Samples – Calibration factor option

$$Concentration(ug/L) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{V}$$

Where
Response = area (or height) of the target
CF_{avg} = average calibration factor (ICAL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

11.3.2 Solid Samples

Solid Samples – Regression Curve Option

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

Where
C_{curve} = concentration of analyte from curve (µg/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
Solids = (percent solids)/100

Solid Samples – Calibration factor option

$$Concentration(ug/kg,dw) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{W \otimes solids}$$

Where
Response = area (or height) of the target
CF_{avg} = average calibration factor
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
Solids = (percent solids)/100

11.4 Dilutions are made as follows:

$$DF = \frac{FV}{Vs}$$

Where
FV = final volume of the dilution (mL)
Vs = volume of sample used to prepare the dilution (mL)

11.5 Spike Calculations

11.5.1 Lab control standard recovery is calculated as follows:

$$\frac{\text{µg/L LCS actual value}}{\text{µg/L LCS expected value}} \times 100\% = \%\ \text{Recovery}$$

11.5.2 Matrix spike recovery is calculated as follows:

$$\frac{\text{µg/L MS actual value - µL/L sample}}{\text{µg/L MS expected value}} \times 100\% = \%\ \text{Recovery}$$

11.5.3 %RPD for spikes is calculated as follows:

$$\frac{\text{\% REC Spike 2 - \% REC Spike 1}}{\text{Average \% Recovery}} \times 100\ \% = \%\ \text{RPD}$$

11.5.4 Theoretical Aqueous Spike Concentration

$$Aqueous(ug/L) = \frac{0.15mL \otimes 100ug/mL}{0.100L} = 150ug/L$$

11.5.5 Theoretical Solid Spike Concentration

$$Soil(ug/kg) = \frac{0.15mL \otimes 100ug/mL}{0.020kg} = 750ug/kg$$

12.0 QUALITY CONTROL AND QUALITY ASSURANCE

12.1 This procedure is restricted to use by or under the supervision of analysts experienced in the use of liquid chromatography. Each analyst must demonstrate the ability to generate acceptable results (within the prescribed accuracy and precision limits) using this method by the analysis of reagent soil and water samples that have been spiked with the target compounds. Each analyst must participate (individually or as part of a work cell) in the analysis and evaluation of **initial demonstration of capability (IDOC**) samples to demonstrate minimum proficiency in this procedure. The IDOC samples are processed in the same manner as routine samples and evaluated according to SOP TL-CA-092: *Evaluation of DOCs*. Each analyst or work cell must subsequently perform an annual **continuing demonstration of capability (CDOC)**, which may consist of 4 LCS replicates or an "Acceptable" score on a PT sample.

The IDOC is used primarily to preclude a laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this method the quality of data will improve beyond those required here.

The analyst is permitted to modify HPLC columns and conditions to improve separations or lower analytical costs. Each time such method modifications are made, the analyst must repeat the IDOC procedures.

12.2 The **analytical batch** is discussed in SOP TL-AN-002: *Analytical Batching and Evaluation of QC Data*, and these criteria are summarized in the SOP Summary included in this SOP. Calculation of QC is also given in SOP TL-AN-002. The analytical batch consists of 20 or fewer samples of a similar matrix that are processed together along with the QC items. The QC items consist of a MB, LCS, LCSD, MS and MSD. If insufficient sample is provided for matrix spikes, the project manager must be notified via an NCM. Batch requirement information is summarized in a table in Appendix A.

12.3 The **method detection limit (MDL)** must be determined initially, when conditions change that might affect the response, or every three years, in accordance with SOP TL-CA-090: *Determination of the Method Detection Limit (MDL)*. The MDL must be verified annually by analysis of an MDL verification standard (MDLV) at a concentration of 1-5 times the calculated MDL. The MDLV must undergo all the same preparation steps as the samples, standards, and QC samples. The acceptance criterion is that the peaks must be detected without manual integration.

12.4 Corrective actions for out-of-control data are summarized in the Quality Control Checks table in Appendix B. This table describes checks, acceptance criteria and the recommended corrective action for various QC activities such as initial and continuing calibration, MB, spikes, reporting limits, method detection limits and IDOCs.

12.5. When data is out-of-control or does not meet the acceptance criteria, a nonconformance memo (NCM) must be written and the PM must be notified. Corrective Action is summarized in Appendix B.

12.6 Data that does not meet acceptance criteria may be conditionally reported with the use of data flags on the final report or through the use of a case narrative attached to the final report.

12.7 Re-extraction and/or reanalysis of the samples in the batch will not be performed under the following conditions:

1) The recovery of the spike exceeds the upper control limit and the compound is not detected in any sample in the analytical batch.

12.8 The laboratory may adopt additional QC practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. For example, field or laboratory duplicates may be analyzed to assess the precision of the environmental measurements or field reagent blanks may be used to assess contamination of samples under site conditions, transportation and storage.

13.0 PREVENTATIVE MAINTENANCE AND TROUBLESHOOTING

13.1 HPLC systems are subject to pressure buildup due to particulate matter in the mobile phase or sample extracts. If this is indicated by recurring pressure increases, all solvents, water and extracts should be filtered using all glass systems and nylon 0.45µm membrane filters.

13.2 The guard column should be evaluated and changed as pressure increases. The frequency of changing the guard column will depend on the nature of the samples analyzed.

13.3 Routine pump maintenance includes inspection and/or replacement of seals regularly. The entire system should be visually leak-checked daily following start- up and equilibration. Spare fittings and valves should be kept on hand for quick repairs.

13.4 The column should be stored in an appropriate mobile phase with the ends capped when not in use.

13.5 Routine detector maintenance includes leak-checking the fittings. Any other maintenance (i.e., lamp replacement) would be performed only if indicated by a problem such as loss of response or sudden increase in noise. Detector lamp replacement is performed when indicated by a significant decrease in signal to noise ratio.

13.6 Retention time shifts are frequently caused by plugged mobile phase inlet frits or air in the pump head check valves.

13.7 Baseline noise can be the result of air in the pump head check valves or poor detector lamp energy.

13.8 Peak broadening can be the result of a dirty guard column or an analytical column nearing the end of its useful life.

13.9 When analytical problems arise, several resources can be utilized including HPLC troubleshooting guides, instrument manuals and professional HPLC technical advisors.

14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention." All excess samples, sample leachates, and reagents must be disposed in accordance with SOP CA70: Waste Management.

14.1 WASTE STREAMS PRODUCED BY THE METHOD

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment

The following waste streams are produced when this method is carried out.

WASTE STREAMS PRODUCED BY THE 8315 METHOD	
Waste Stream Description	**Category (Preferred Treatment)**
Flammable solvents mixed with water from the HPLC	RCRA Hazardous Waste (This mixture is collected in 4L waste containers and added to the aeration system in the hazardous waste area. The water is purged for 12 hours and then discharged to the sewer system.)
Sample Extracts in Vials	RCRA Hazardous Waste (Either the vials are dumped into the drum marked "Vials" in the hazardous waste area, or the vials are emptied into the 4L waste containers and treated as above.)

15.0 REFERENCES

15.1 *Test Methods for Evaluating Solid Waste - Physical/Chemical Methods*, SW-846, Third Edition, Update III

15.2 TL-QAM: *TestAmerica Tallahassee Quality Assurance Manual* (QAM), current revision

16.0 TABLES, DIAGRAMS, AND FLOWCHARTS

16.1 Appendix A: 8315 METHOD SUMMARY

16.1.1 HOLD/STORAGE

Parameter	Aqueous	Soil/Solid
Routine Container	500mL amber glass fitted with Teflon-lined cap	250mL or 500mL glass with Teflon-lined cap
Preservative	None	None
Hold time	Three days from date of collection and three days from date of extraction	Seven days from date of collection and three days from date of extraction
Storage	4 °C (less than 6 °C with no frozen samples) from collection to extraction and analysis	4 °C (less than 6 °C with no frozen samples) from collection to extraction and analysis

16.1.2 EXTRACTION (See SOP LC46)

Aqueous Samples: 100mL of sample is derivatized with DNPH; the derivative is collected on an SPE cartridge and eluted with ACN. The extract is analyzed by reverse phase HPLC using UV detection.

Solid Samples: Soil samples are weighed out to 20g and leached with a buffered solution for 2 hours. The leachate is filtered, derivatized and analyzed in the same manner as aqueous samples.

16.1.3 QC REQUIREMENTS

BATCH QC
MB
LCS/LCSD
MS/MSD (frequency of 5% of samples)

16.1.4 SURROGATES

There are currently no surrogates available for the 8315 method.

16.1.5 ANALYSIS

Analysis is done by HPLC with UV detection; external standard calibration is used.

16.1.6 ANALYTICAL SEQUENCE

STANDARD/SAMPLES
Initial Calibration
Up to twenty sample analyses (not counting QC samples)
Continuing calibration verification (CCV) – every 12 hours or after every 20 or fewer samples
RL standard (daily if required)
Up to ten sample analyses (not counting QC samples)
CCV – sequence must end with a CCV

Sequence continues until all samples have been analyzed or the CCV fails the acceptance criteria. The 8000-series method requires that the sequence must be capped with an acceptable CCV.

16.2 Appendix B

QUALITY CONTROL CHECKS

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration (ICAL) – The lowest point should be at or near the RL – other points define the working range.	Prior to sample analysis or when CCV fails	**8000-series (min 5 points)** 1) Plot regression curve (CC>=0.99) OR 2) %RSD of each target <= 20% (see SOP for exception)	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument or column maintenance and reanalyze standards
Initial Calibration Verification Standard (ICVS) – Use LCS	Within sequence (after ICAL)	LCS criteria	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument or column maintenance and reanalyze standards
Continuing Calibration Verification (CCV)	**8000-series**: Every 12 hours or every 20 sample shots and at the beginning and end of the sequence	Percent difference or drift <= 15% (See SOP for exception for 8000-series methods)	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument or column maintenance and reanalyze standards
Method Blank	Per batch	All targets reported less than RL in Table 5 of the STL LQM	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze - Follow guidance in STL SOP AN02 and Table 13.1 in LQM - Perform instrument or column maintenance, recalibrate and reanalyze

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Lab control sample (LCS) and lab control sample duplicate (LCSD)	Per batch	Recoveries within LIMS/TL-QA-002 limits	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze - Follow guidance in SOP TL-AN-002 - Perform instrument or column maintenance, recalibrate and reanalyze
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	5% of samples	Recoveries within LIMS/TL-QA-002 limits	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze - Follow guidance in SOP TL-AN-002 - Perform instrument or column maintenance, recalibrate and reanalyze
Reporting Limit (RL) Standard – lowest level calibration standard	Daily – required for Florida DEP	Detected with reasonable sensitivity	- Reanalyze RL standard - Remake and reanalyze RL standard - Perform instrument or column maintenance, recalibrate and reanalyze associated samples
Initial Demonstration of Capability (The analyst has to perform the IDOC for either of the analogous 600 or 8000 series methods – not both)	Per analyst or work group	Within the 8000-series method limits	- Reanalyze QC sample for the targets that failed to meet the criteria
Method Detection Limit (MDL)	See SOP TL-CA-090	Evaluate according to SOP TL-CA-090	Evaluate according to SOP TL-CA-090
Method Detection Limit Verification (MDLV)	Annually	Evaluate according to SOP TL-CA-090	Evaluate according to SOP TL-CA-090

17.0 REVISION HISTORY

Revision 6: 05/09/2008

- Changed all STL references to TestAmerica
- Incorporated new cover page, logo, and naming conventions
- Name: Changed from EX to LC
- Revised all SOP references to new naming convention
- Corrected name of CSM (added "Environmental Health and...")
- Deleted all references to LQM, added references to TL-QAM, TL-QA-001, TL-QA-002, And TALS LIMS
- Added Section 17.0 Revision History
- Changed preparation SOP reference from EX46 to TL-LC-046
- 10.3.2, 10.3.3, 10.3.4: Changed order to indicate regression curve as first choice; CF may also be used
- 10.5: Revised text to indicate second-source LCS is used in place of traditional ICV
- 10.5.3: Added note regarding use of grand mean exception to be discontinued upon adoption of 8000C
- 10.7.3: Changed "computer method: to "acquisition method"
- 10.7.7: Changed "30 minutes to one hour" to "at least 30 minutes"
- 10.7.12, 10.7.14: Deleted "pre-Target" text
- 10.8.1: Deleted section (co-injection not used), renumbered 10.8.2 as 10.8.1
- 12.2: Changed text regarding notification of insufficient sample for MS/MSD
- 12.3: Added MDL verification requirements
- 12.4: Deleted mention of surrogates
- 13.3: Changed frequency from "every six months" to "regularly"
- 16.1.1: Changed soil hold time from "Minimal time" to "Seven days"
- 16.1.6: Deleted ICVS
- 16.2: Re-defined ICVS as LCS, added MDLV requirements

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Laboratory Method for the Determination of Herbicides in All Matrices

Approved By:

QA Officer: Melina Mulli Date: 12-3-2007

Laboratory Director: Date: 12/3/2007

Revision History

Revision	Description of Change	Approval	Date

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1. Title

Laboratory Method for the Determination of Herbicides in All Matrices.

EPA References:

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8000B, Revision 2, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8151A, Revision 1, December 1996

EPA Method 615-The Determination of Chlorinated Herbicides in Municipal and Industrial Wastewater.

Quality Systems Manual For Administrative, Organic and Limited Inorganic Departments of ANALYTICS Environmental Laboratory, LLC. Current Revision

Analytics Environmental Laboratory (AEL) SOP QA-L001-Sample Receipt, Preservation and Storage

AEL SOP QA- H001-Laboratory Method for the Preparation of Herbicide Standards

AEL SOP QA-H002: Separatory Funnel Liquid/Liquid extraction for the Determination of Chlorinated Acid Herbicides in Aqueous samples

AEL SOP QA-H003: Sonication Extraction for the Determination of Chlorinated Acid Herbicides in Solid samples

AEL SOP QA-H004: Extraction for the Determination of Chlorinated Acid Herbicides in Non Aqueous Liquid samples

AEL SOP QA-M009-Laboratory Method for Balance Calibration

AEL SOP QA-D001- Standard Operating Procedure for Data Review and Validation

AEL SOP-W001- Method for Containing, Cataloging, and Disposing of Solid/Liquid Waste

2. Scope and Application

This method is used for the determination of chlorinated phenoxy acid herbicides that are partitioned into an organic solvent (Diethyl Ether or Methylene Chloride:Acetone) and derivatized to methyl esters. The resulting methyl esters are amenable to gas chromatography. The parameters listed (Table 1) can be detected by this method. The quantitation range for aqueous sample is 1.0 μg/L-10 μg/L and 16.7 μg/kg-666.7μg/kg for solids. This method is restricted to use by or under the supervision of analysts experienced

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in the use of a gas chromatograph and in the interpretation of GC data. Organic acids, especially chlorinated acids, may result in interferences with the determination.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevations in baselines. Method blanks must routinely be analyzed to show an absence of these interferences.

3.2 Matrix interferences may include contaminants that are co-extracted from the sample. These may include but are not limited to, phthalate esters, petroleum hydrocarbons, and other organic acids. Alkaline hydrolysis may be used to remove some of these interferences. However, hydrolysis may result in the loss of some of the target analytes.

3.3 Sample extracts should be dry prior to methylation or else poor recoveries may be obtained.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure will be minimized by wearing solvent resistant gloves, safety goggles, a lab coat and by working in a fume hood when samples or standards are handled.

4.3 Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory.

5. Method Summary

A 2 µL volume of the sample extract is injected into the gas chromatograph (GC) using a Hewlett Packard auto-ampler. Analytes are detected with an electron capture detector (ECD). Quantification of target compounds is performed by external standard technique, using Hewlett Packard Enviroquant software. Target Compounds are confirmed by additional analysis on a dissimilar capillary column. Refer to Figure 1 for the method summary.

6. Sample Collection, Handling and Preservation

6.1 Solid samples should be collected in glass jar with a screw top Teflon lid.

6.2 Aqueous samples should be collected in 1 Liter Amber glass containers with teflon-lined caps.

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6.3 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to AEL SOP QA-L001 for proper cooler handling instructions.

6.4 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to AEL SOP QA-L001 for laboratory receipt and log-in procedures.

6.5 Aqueous samples must be extracted within 7-days of collection.

6.6 Solid samples must be extracted within 14 days of collection.

6.7 Sample extracts must be analyzed within 40 days of extraction.

6.8 Extracts must be stored in a freezer at –10°C to –20°C protected from light.

7 Definitions

7.1 µL= micro-liter

7.2 ml = milliliter

7.3 L= liter

7.4 µg= microgram

7.5 g= gram

7.6 kg= kilogram

7.7 GC= Gas Chromatograph

7.8 ECD= Electron Capture Detector

7.9 DCAA= 2,4-dichlorophenylacetic acid

7.10 MW= Molecular Weight

7.11 °C= Degrees Celsius

7.12 wt.= weight

7.13 vol.= volume

7.14 Conc.= concentration

7.15 psi= pounds per square inch

7.16 CF= Calibration Factor

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7.17 inj.= injections

7.18 CAS= Chemical Abstract Service

7.19 ALS= Automatic Liquid Sampler

7.20 RSD= Relative Standard Deviation

7.21 min= minute

7.22 ID= Internal Diameter

7.23 CV= Calibration Verification

8. Apparatus and Materials

8.1 GC System

8.1.1 Gas Chromatograph: Agilent Technologies 6890 N or equivalent

8.1.2 Auto-sampler Sequence:

Hewlett Packard 7683 auto-sampler or equivalent
ALS Conditions from HP Chemstation:

Method:	HERB.M
Sample washes	2
Sample Pumps	3
Injector volume	2.0 µL
Syringe size	10.0 µL
On column	off
Nano-liter adapter	off
Post-Inj. solvent A washes	2
Post-Inj. solvent B washes	2
Viscosity delay	1 second
Plunger speed	fast

8.2 GC Columns

8.2.1 Primary column:
Stx- CLPesticides 2, 30 m, 0.25 mm ID, 0.2 µm film thickness or equivalent

8.2.2 Confirmatory column:
Rtx – CLPesticides, 30 m, 0.25 mm ID, 0.25µm film thickness or equivalent

8.2.3 Guard column fused silica 10 meter, 0.25 mmID, Phenomenex Part #7CG-G000-00-GZ0 or equivalent

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8.2.4 Oven Temperature Program:

Inlet A	250°C
Inlet B	off
Detector A	250°C
Detector B	250°C
Oven equib time	0 minutes
Initial temperature	80°C
Initial time	1.50 minutes

Level	Rate (°C/min)	Final Temp (°C)	Final Time (min)
1	100	120	0.00
2(A)	40.0	310	0.85

Next run time 7.50 minutes

8.3 Gases

Helium, Grade 5, for carrier gas
Argon Methane (95:5), Grade 5, for make-up gas for ECD

Carrier Gas Flow rate:	6.2 mL/min
Purge Vent Flow rate:	8 mL/min
Split Vent Flow rate:	75 mL/min
Make-up Gas Flow rate:	45-50 mL/min

Inlet Purge	Init Value	On Time	Off Time	Split-less Injection
A	off	0.50	0.00	yes
B	off	0.50	0.00	yes

8.4 Electronic Pressure Program

Constant Flow:	ON
Constant Flow Pressure	47.1 psi
Constant Flow Temp	80 °C

Total program time 7.50 minutes

8.5 Data System: Hewlett Packard Environmental Chemstation

8.6 Instrument run logbook

8.7 Sample preparation logbook

8.8 Instrument maintenance logbook

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9. Standards and Reagents

9.1 Stock Standard Solutions: use the following standards or equivalent

9.1.1 Herbicide Curve Stock: 100 ug/mL, Accustandard M-8151

9.1.2 Herbicide Surrogate Stock: 100 ug/mL, Accustandard M-515-SS

9.1.3 Herbicide Second Source Stock: 100 ug/mL, Ultra HBM-8151m-1

9.1.4 Herbicide Surrogate Second Source Stock: 100 ug/mL, Absolute Standard #30022

9.2 Preparation of Working Solutions and Standards

9.2.2 Standards Preparations Logbook. All information pertinent to standards preparation must be recorded in black ink in a permanently bound logbook with numbered pages entitled "Herbicide Standard Prep". When a standard is prepared, it is assigned a unique, sequential number as follows: "HXXX, where H indicates that the standard is for herbicide analysis and XXX is the next sequential number (e.g. H001). Record this number on the left side of the page. The preparation date and analyst's initials are recorded in the lower right had corner of the preparation area. To the right of the standard number, describe the standard, including the concentration. Assign an expiration date and record it. Record the volume of the standard used, the standard description, manufacture, manufacturer's expiration date, catalog number and the lot number and the lot number. Record the amount, manufacture and lot number of the dilution solvent (Figure 3).

9.2.3 Store standards with a minimum of headspace in the freezer designated for herbicide standard storage. Label standard containers with the standard number, a brief description of the standard, the preparation date, analyst's initials, and the expiration date.

9.2.4 Expiration Dates

Stock standards may be stored in sealed ampules in the freezer until the manufacturer's expiration date. Stock standards should be replaced sooner if comparison with previous calibration standards indicates poor performance. Once opened, stock standards are good for six months. Expiration dates are for prepared intermediates standards are based on the oldest stock standard used, but will be no longer than 6 months from the preparation date. Replace standards if poor performance is indicated.

Note: Equivalent Dilutions may be used.

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9.2.5 Herbicide Calibration Curve

Std.Concentration ug/mL	Stock Standard	Stock standard Conc.	Vol. Of Stock Std. Sol.	Final Volume of Ether
Herb Std 0.025	Herb. Stock Std. (9.1.1)	100 ug/mL	2.5 uL each	10 mL
Herb Std 0.05			5.0 uL each	10 mL
Herb Std 0.1	Herb. Sur. Stock (9.1.2)	100 ug/mL	10 uL each	10 mL
Herb Std 0.2			20 uL each	10 mL
Herb Std 0.3			30 uL each	10 mL
Herb Std 0.5			50 uL each	10 mL
Herb Std 0.75			75 uL each	10 mL
Herb Std 1.0			100 uL each	10 mL

9.2.6 Herbicide Second Source Check Standard

Std.Concentration ug/mL	Stock Standard	Stock standard Conc.	Vol. Of Stock Std. Sol.	Final Volume of Ether
0.5 ug/mL	Herb. Second Source Stock (9.1.3) Herb Sur. Second Source Stock (9.1.4)	100 ug/mL 100 ug/mL	50 uL of each	10 mL

9.3 Hexane - Pesticide quality or equivalent for auto-sampler rinseate #2 and column washing.

9.4 Acetone - Pesticide quality or equivalent for column washing.

9.5 Methylene Chloride - Pesticide quality or equivalent for column washing.

9.6 Methanol – Purge &Trap grade or equivalent auto-sampler rinseate if needed.

9.7 Ether- Pesticide Quality or equivalent for dilutions and rinseate #1.

10. Procedure

10.1 Refer to the following Standard Operation Procedures for sample preparation.

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AEL SOP QA-H002: Separatory Funnel Liquid/Liquid extraction for the Determination of Chlorinated Acid Herbicides in Aqueous samples

AEL SOP QA-H003: Sonication Extraction for the Determination of Chlorinated Acid Herbicides in Solid samples

AEL SOP QA-H004: Extraction for the Determination of Chlorinated Acid Herbicides in Non Aqueous Liquid samples

10.2 Instrument Run Logbook

A bound paginated laboratory notebook is assigned to each instrument for use as a sample run log. The date is noted above each day's analyses along with the analyst's initials. Each day starts a new page. Each run is recorded with the following information: the run number, the auto-sampler vial number, the AEL sample # or standard #, the dilution, the method file name, relevant comments (i. e. rerun for low surrogate, standard passes, dilution necessary, etc.). Each instrument is identified with a unique letter. Data files (run numbers) are assigned using this letter followed by the next consecutive number. The letter designation for this instrument is "H". Also on the run log is a "loading check" and "unloading check". Prior to loading the vials, check to ensure that the tape label agrees with the label on the vial. When unloading the samples, verify that the order in the auto-sampler tray agrees with that in the run log. Record initials in the loading check boxes. See Figure 2 for an example of the run log page.

10.3 Initial Calibrations

Before sample analysis can begin, the instrument must be calibrated for each parameter. Calibration standards are prepared as described in section 9.2 above. The lowest level standard must be at or below the quantitation limit for each target compound. The remaining levels are chosen based on the linear range of each parameter with the highest level not exceeding the concentration limits of the column or detector. For herbicides, analysis of a minimum of five calibration levels is required, **unless a quadratic curve fit is used, which requires a minimum of six calibration levels.** Refer to Table 2 for the specific calibration levels.

10.3.1 The method is named by incorporating the date the calibration was run as follows: HMMDDY where;

H is the designation for analysis of Herbicides
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the last digit of the year

Saving the previous 5-point using the new name creates the new name. Calibration standards will be quantitated automatically upon completion of the analytical run. Load the data file of the first standard in the 5-point.

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10.3.2 Evaluate the low standard for analyte sensitivity. Peak area should be approximately three times the baseline noise level. Analytes may be selectively dropped from the low standard as long as the curve still contains five levels.

10.3.3 Verify that the computer is properly integrating all analytes and selecting the correct peaks. All manual integrations must be conducted in accordance with AEL SOP QA-M022.

10.3.4 Update each of the calibration levels by loading each datafile and using the "InitCal" menu pull-down "Update Levels". It is important to save the method after updating.

10.3.5 Determination of Linearity

Quantitation of herbicides is based on the area or height of the analyte peak at the known retention time. Linearity is determined from the calibration factor (CF) of the standards or the generation of a calibration curve using either linear regression or quadratic fit.

10.3.5.1 Linear regression or quadratic fit is routinely used for herbicide analysis. If a linear calibration curve is used the correlation coefficient (r) must be 0.995 (coefficient of determination r^2 0.990) or greater to meet acceptance criteria. For a quadratic fit, the coefficient of determination must be (r^2) 0.995 or greater to meet acceptance criteria. **Quadratic fits requires a minimum of 6 calibration standards. It is never acceptable to force a calibration curve through the origin. Nor is it acceptable to include the origin (0,0) as a sixth calibration point.**

10.3.5.2 Alternatively calculate a calibration factor for each compound. The calibration factor is the peak area (or height) of the compound divided by the mass of the compound injected. When the relative standard deviation (RSD) of the calibration factors is <20% the standard curve is considered to be linear and the average calibration factor can be used for quantitation.

10.3.5.3 If the linearity criteria is acceptable, file the initial calibration printout, the analyte plots for compounds set to linear or quadratic or the custom report evaluating curve fits, and the standard quantitation reports in the Herb QC book.

10.3.6 Determination of Retention Time (RT) Windows: Herbicides are identified using absolute retention times. Retention time windows are generated as follows:

1. Make three injections of a standard over the course of 72 hours. Serial injections or injections over a period of less than 72 hours may result in retention time windows which are to tight.

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2. Calculate the mean and standard deviation (SD) of the absolute retention time (RT) for each analyte in all three standards.

3. The width of the RT window is defined as the mean RT +/- (3*SD). Note: When the SD=0 a default SD of 0.01 shall be used.

4. The center of the RT window is determined for each analyte by using the absolute RT for each analyte once per initial calibration and at the beginning of each analytical window. For initial calibration the mid-point standard is used to determine the RT window and for analytical windows the calibration verification standard to open the window is used for RT window establishment.

RT windows shall be determined annually or when a new column is installed.

10.3.7 Each time the five point calibration is analyzed an herbicide standard prepared from an independent source is analyzed (Table 2) to verify the calibration. The measured value must agree within 20% of the true value for the calibration to be considered valid.

10.3.8 Calibration verification (CV)

Calibration verifications are conducted on a daily basis, prior to sample analysis, every 20th analysis (10 recommended to minimize reanalysis), and at the end of each sequence to validate the calibration of the system for each parameter. NOTE: DOD projects require calibration verification be analyzed after every 10 field samples and at the end of the analysis sequence.

10.3.8.1 A continuing calibration standard of varied concentration shall be used for this purpose. If the response (or calculated concentration) is within ±15% of the initial calibration, then the initial calibration is considered valid.

10.3.8.2 Alternatively, if the average of the responses of <u>all</u> analytes is within 15% and no individual analyte has %D>30% the initial calibration is considered valid.

10.3.8.3 If the average of <u>all</u> response (or calculated concentration) is >15%, the calibration verification fails and corrective action must be taken. It may be necessary to analyze a new calibration.

10.3.9 Corrective Action

If the calibration verification or initial calibration is unacceptable, the analyst must consider that the analytical system is out of control and corrective action is needed. Once the problem is determined, correct it and re-analyze

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the initial calibration if necessary. Instrument corrective action must be documented in the instrument's maintenance logbook. Each log entry should contain the date of the action, the initials of the analyst, a description of the problem and the corrective action taken and the result of the corrective action. Hard copies of the unacceptable initial calibration and/or calibration verification standard should be stored in the QC notebook with an explanation of the problem and how it was corrected.

10.4 Analyzing Samples

10.4.1 Inject 2µL of sample or blank extract onto the GC column using the Hewlett Packard 7683 auto-sampler. Analyze the sample using the GC conditions in the method HERB. Record the sample analyses in the instrument's run logbook (Section 10.2).

10.5 Quantitation of Sample Analytes

10.5.1 Target compounds are identified qualitatively based on the retention time comparison with the calibration standard. All analytes must fall within the RT windows defined above for positive qualification. The final interpretation of peak identification is based on the discretion of an experienced GC analyst. Target compounds are quantitated using a method file stored on the HP Enviroquant. The method file stores calibration factors and retention times. The calibration files are named as follows: HMMDDY where;

H= is the designation for Herbicide analysis
MM = is a two number designation for the month
DD = is a two number designation for the day
Y = is a one number designation for the year

Example: H06071, for the calibration analyzed on June 7, 2001.

10.5.2 Enviroquant quantitates the sample extract result by calculating the corresponding analyte concentration for the area found based on the calibration curve (or average calibration factor).

10.5.3 Dilutions

If the concentration of any target compounds exceeds the upper calibration, the extract must be diluted in Ether to achieve an on column concentration that will be in the upper half of the calibration range. Analyze the diluted extract and recalculate the sample result. Dilutions are documented in the Herbicide Sequence Logbook, noting the volume of extract used, the volume of Ether used and the Ether lot number.

10.5.4 The sample result is calculated, correcting to sample weight and percent solids or volume using the following equation:

$$\text{Sample result} = \frac{\text{Sample extract conc. } (\mu g/ml) * \text{actual vol of extract (ml)}}{} \times DF$$

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(μg/L or μg/kg) sample wt or vol (kg or L) * % solids/100

where:

1) Sample extract conc (μg/ml) is found on the raw data report from HP Enviroquant.
2) vol of extract (ml) and sample wt or vol is found on the extraction transfer sheet (Figure 3).
3) % solids is found on the extraction transfer sheet (Figure 3).

10.5.5 If target compounds are detected on the primary analysis, a confirmatory analysis is performed simultaneously to verify the presence of the target compound. This confirmation analysis is performed on a different column type, i. e. Stx -CLPesticides. The result from the confirmatory analysis should be within 40% of the result from the primary analysis. Report the higher of the two results unless there are overlapping peaks or other anomalies present. In these cases clearly document on the data file the reason for reporting the lower result and initiate a QC memo so results can be reported with a comment to this affect. When the % difference is >40% the data user should be advised of the disparity. This is done by flagging the result with a "D" and entering a comment on the results page.

11 Quality Control

11.1 Each time the five point calibration is analyzed an herbicide standard prepared from an independent source is analyzed (Table 2) to verify the calibration. The measured value must agree within 20% of the true value for the calibration to be considered valid.

11.2 The initial calibration is verified at the beginning of each day, every 20th sample analysis (10 recommended to minimize reanalysis required for DOD projects), and at the end of each sequence with a calibration verification (CV). If the calibration verification is unacceptable, the problem must be corrected and documented. It may be necessary to analyze a new initial calibration. Each sample analysis must be bracketed with an acceptable initial or calibration verification standard. However, if the samples are "U" and the ending calibration verification fails high, the proceeding runs are considered acceptable. Initiate a QC Documentation Memo for approval by the Quality Control Officer and the Laboratory Director. See Figure 4.

11.3 Per sample

11.3.1 A surrogate (2,4-Dichlorophenylacetic acid) is added to every sample, sample matrix spike, sample matrix spike duplicate, laboratory control sample and blank before extraction to test the efficiency of the extraction procedure. Percent recoveries are calculated by dividing the amount found by the amount added. The recovery acceptance ranges are listed in the instrument method per matrix. Whenever a surrogate is outside of the acceptable range, an investigation of the extraction and analytical procedures is conducted. If the surrogate recovery is outside the laboratory acceptance criteria the following steps should be taken:

1.) Re-inject the extract to verify the surrogate result.

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2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.3.2 Surrogate recoveries are charted by matrix in batches of 20. The analyst uses this information to observe trends, which might indicate a problem with the instrument or extraction procedure. The following conditions shall be considered out-of-control events for all control charts:
- One point outside control limits
- Three consecutive points outside warning limits
- Six consecutive points such that each point is larger (or smaller) than its immediate predecessor
- Eight consecutive points on the same side of the centerline

11.4 Per extraction batch- These are AEL's general acceptance criteria. Projects that have required QAPPs take precedence to AEL's general limits. These requirements are indicated using controlled project sheets to be filled out by the laboratory project manager.

11.4.1 A method blank must be performed with every sample batch (20 samples or daily). The blank is analyzed prior to the associated samples to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit (1/2 RL for DOD projects) of any compound the following steps should be taken:
1.) Re-inject the extract to verify the results.
2.) Re-extract the samples in the batch if sample volume and holding time permits. It may be possible to accept samples that are "U" with the approval of the QA Officer.
3.) If insufficient sample exists to re-extract, the contaminant will be qualified with a "B" flag.
4.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.4.2 A laboratory control sample (LCS) must be extracted with every sample batch 20 samples or daily, whichever is more frequent. The LCS is spiked with 1.0 mL of the matrix spiking solution. The laboratory control samples are subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy. The GC/ECD analyst will calculate the percent recoveries using the following formula:

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$$\text{Recovery}=\%\text{Rec}=\frac{Cs-Cu}{Cn}X100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the unspiked sample aliquot (use 0 for non-detects)
Cn = Nominal (true) concentration

If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. An LCS with recoveries above the laboratory acceptance criteria is considered acceptable for samples with no analytes detected. The following steps should be taken for recoveries below the acceptance limits or samples that have analytes detected:

1.) Re-inject the extract to verify the result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.5 Per QC Batch-These are AEL's general acceptance criteria. Projects that have required QAPPs take precedence to AEL's general limits. These requirements are indicated using controlled project sheets to be filled out by the laboratory project manager.

11.5.1 A matrix spike/matrix spike duplicate sample (MS/MSD) must be extracted with every QC batch (20 samples or monthly). The MS/MSD's are spiked with 1.0 mL of the matrix spiking solution. MS/MSD samples are subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy and precision. Spike recoveries are calculated and charted in accordance with AEL SOP QA-M023 in batches of 20 by matrix. Acceptable water or soil spikes shall be within the limits listed in table 3. Matrix spikes (MS) and Matrix spike duplicate (MSD) recoveries are calculated by the following formula:

$$\text{Recovery}=\%R=\frac{Cs-Cu}{Cn}X100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the un-spiked sample aliquot (use 0 for non-detects)
Cn = Nominal (true) concentration

If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. A MS/MSD with recoveries above the laboratory acceptance criteria is considered

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acceptable for samples with no analytes detected. The following steps should be taken for recoveries below the acceptance limits or samples that have analytes detected:

1.) Re-inject the extract to verify the result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

For each set of spikes extracted the Relative Percent Difference (RPD) must be calculated. Precision is estimated from the RPD of the concentrations (not recoveries) measured in the MS/MSD pairs. To be acceptable the RPD should be
$\leq$ 25%for aqueous samples and $\leq$ 30%for solid samples. Calculate the RPD according to the formula below.

$$RPD = \frac{|C1-C2|}{\frac{C1+C2}{2}} X100$$

11.6 Minimum Detection Limits

Minimum detection limit studies or method detection limit check samples are performed according to AEL SOP QA-M021.

11.7 Run log sequences are confirmed when loading and unloading sample extracts.

11.8 Data Validation- All data is reviewed completely before reporting to the client. Data review and Validation is conducted following AEL SOP QA-D001.

12. Routine Instrument Maintenance

12.1 Inlet Maintenance

A diagram of the GC inlet can be found in the Agilent Technologies 6890N manual Volume 2-Inlets. All inlet maintenance is recorded in the maintenance logbook for the instrument.

12.1.1 The inlet septum is replaced after 20-40 injections. An 11 mm septum is used. (Supelco thermogreen™ LB-2 Septa catalog #2-0654 or equivalent).

12.1.2 A clean deactivated/silanized split-less single taper liner should be installed each day, or as needed. Install the liner in the GC with a new O-ring (Hewlett Packard catalog #5180-4182 or equivalent), being careful not to over-tighten the nut. Refer to AEL SOP QA-M011 for cleaning and silanization of inlet liners.

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12.1.2 The injection port inlet seal and washer must be replaced periodically. This should be checked bi-monthly. If the gold seal is black or pitted a new seal and washer is required. Refer to Agilent Technologies 6890N manual Volume 2-Inlets for proper seal/washer placement.

12.2 Column Maintenance

All column maintenance is recorded in the maintenance logbook for the instrument.

12.2.1 The front end of the columns and the guard column are inspected when instrument performance is poor. Compound and particle deposits will degrade the chromatography by the broadening of peaks and the reduction of sensitivity. One loop (about 12 inches) of the front end can be clipped from each of the columns and/or the guard column to remove these contaminants.

12.2.2 Periodic baking of the column at its upper temperature limit will clean the column of contaminants that have remained on the column during the day's normal operating temperatures. This should be performed for a maximum of 24 hours.

12.2.2 When the previous two steps do not lead to improvement of the chromatography it may be necessary to replace the guard column. If chromatography still doesn't improve a new column may also be required. Generally, a column will last 6 - 10 months under normal operating conditions. Document the installation of a new column in the instrument logbook. Each entry includes the date the column was installed a description of the column which includes manufacturer, column length, internal diameter, film thickness, catalog and lot numbers and maximum temperature. Conditions under which the column was conditioned and any changes in installation procedures shall be documented as well.

12.3 The molecular sieve in-line trap for the helium gas is changed annually.

12.4 Charcoal trap for ECD vent is changed annually.

12.5 ECD Leak tests are performed every six months. Testing materials are purchased from National Leak Test or a similar company. Procedures vary so it is necessary to read the instructions received with each kit.

12.6 Gas tanks are replaced when tank pressure falls below 500 psi.

12.6 Periodic tightening of the detector's make-up gas fitting is necessary. The oven temperature programming loosens the fitting. Check weekly.

13. Pollution Prevention and Waste Management

13.1 See AEL SOP QA-W001 for handling of waste and unused portions of sample.

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TABLE 1

Target Compounds

Compound	CAS#
Dalapon	75-99-0
Dicamba	1918-009
2,4-D	94-75-7
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dichloroprop	120-36-5
2,4-DB	94-82-6
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
Acifluorfen	50594-66-6
Bentazon	25057-89-0
Chloramben	133-90-4
DCPA,Diacid	2136-79-0
3,5-Dichlororbenzoic acid	51-36-5
4-Nitrophenol	100-02-7
Picloram	1918-02-1
Pentachlorophenol	87-86-5
DCAA (surrogate)	19719-28-9

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TABLE 2

Calibration Levels

Herbicide (HERB):

Analytes Level:	1	2	3	4	5
Dalapon	0.025µg/mL	0.05µg/mL	0.1µg/mL	0.2µg/mL	0.3µg/mL
Dicamba	0.025	0.05	0.1	0.2	0.3
2,4-D	0.025	0.05	0.1	0.2	0.3
2,4,5-TP (Silvex)	0.025	0.05	0.1	0.2	0.3
2,4,5-T	0.025	0.05	0.1	0.2	0.3
Dichloroprop	0.025	0.05	0.1	0.2	0.3
2,4-DB	0.025	0.05	0.1	0.2	0.3
Dinoseb	0.025	0.05	0.1	0.2	0.3
MCPP	2.5	5.0	10	20	30
MCPA	2.5	5.0	10	20	30
Acifluorfen	0.025	0.05	0.1	0.2	0.3
Bentazon	0.025	0.05	0.1	0.2	0.3
Chloramben	0.025	0.05	0.1	0.2	0.3
DCPA, diacid	0.025	0.05	0.1	0.2	0.3
3,5-Dichlorobenzoic acid	0.025	0.05	0.1	0.2	0.3
4-Nitrophenol	0.025	0.05	0.1	0.2	0.3
Picloram	0.025	0.05	0.1	0.2	0.3
Pentachlorophenol	0.025	0.05	0.1	0.2	0.3
DCAA (Surrogate)	0.025	0.05	0.1	0.2	0.3

Analytes Level:	6	7	8
Dalapon	0.5µg/mL	0.75µg/mL	1.0µg/mL
Dicamba	0.5	0.75	1.0
2,4-D	0.5	0.75	1.0
2,4,5-TP (Silvex)	0.5	0.75	1.0
2,4,5-T	0.5	0.75	1.0
Dichloroprop	0.5	0.75	1.0
2,4-DB	0.5	0.75	1.0
Dinoseb	0.5	0.75	1.0
MCPP	50	75	100
MCPA	50	75	100
Acifluorfen	0.5	0.75	1.0
Bentazon	0.5	0.75	1.0
Chloramben	0.5	0.75	1.0
DCPA, diacid	0.5	0.75	1.0
3,5-Dichlorobenzoic acid	0.5	0.75	1.0
4-Nitrophenol	0.5	0.75	1.0
Picloram	0.5	0.75	1.0
Pentachlorophenol	0.5	0.75	1.0
DCAA (Surrogate)	0.5	0.75	1.0

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TABLE 2 (Cont.)

Second Source:

Analytes	Concentration (µg/mL)
Dalapon	0.5
Dicamba	0.5
2,4-D	0.5
2,4,5-TP (Silvex)	0.5
2,4,5-T	0.5
Dichloroprop	0.5
2,4-DB	0.5
Dinoseb	0.5
DCAA	0.5
MCPP	50
MCPA	50
Acifluorfen	0.5
Bentazon	0.5
Chloramben	0.5
DCPA, diacid	0.5
3,5-Dichlorobenzoic acid	0.5
4-Nitrophenol	0.5
Picloram	0.5
Pentachlorophenol	0.5
DCAA (Surrogate)	0.5

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FIGURE 1

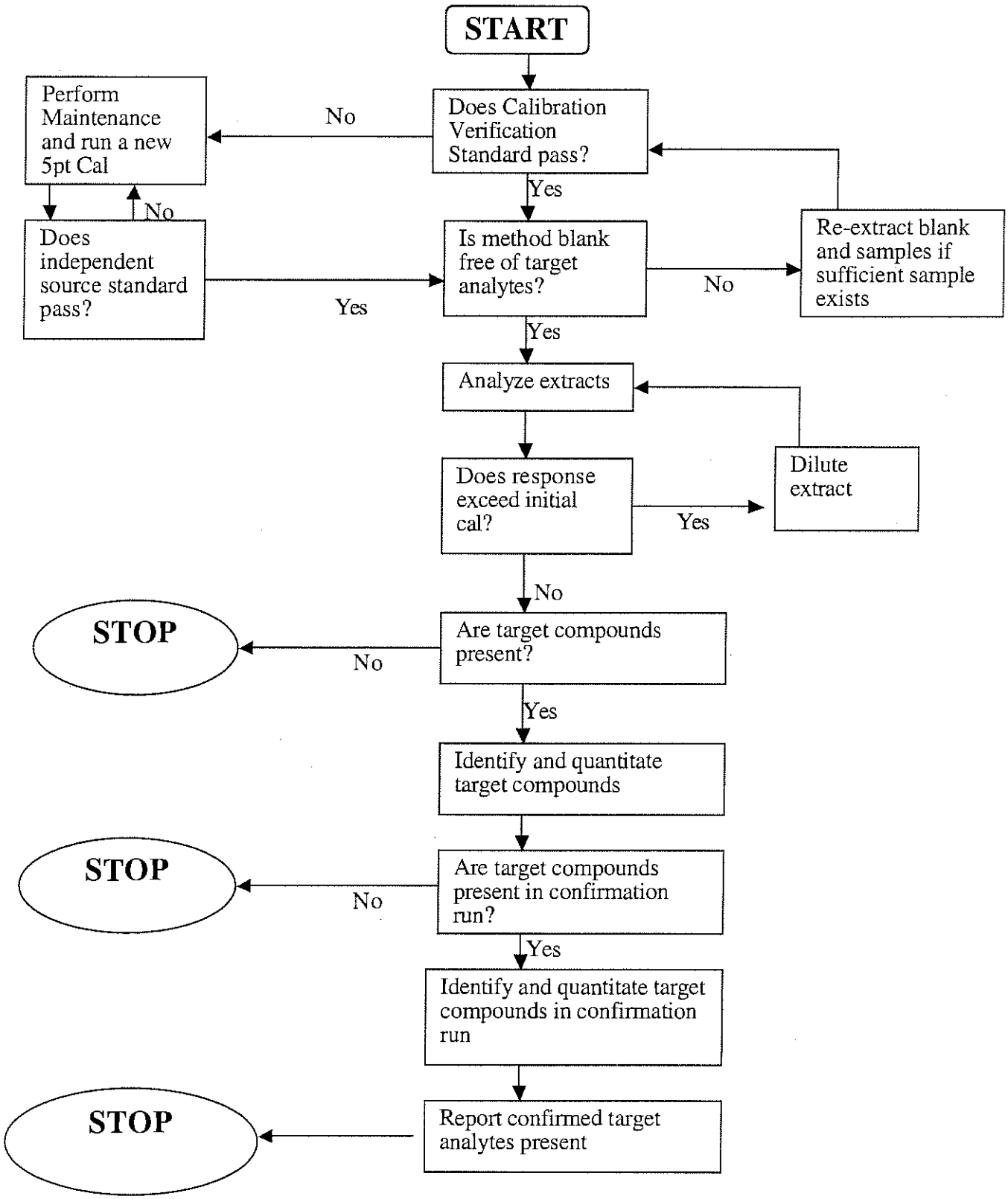

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FIGURE 2

PEST/PCB/HERB
Instrument F
Sequence Log Book

Load: _____
Unload: _____

Data File	Vial #	Sample	Method	Comments

Standard Dilutions: 1:2- 500 uL sample +500 uL hexane
1:5- 200 uL sample +800 uL hexane
Hexane Lot : _____ 1:10- 100 uL sample + 900 uL hexane
1:20- 50 uL sample + 950 uL hexane
1:100- 10 uLsample +990 uL hexane
1:1000- 1 uLsample +1000 uL hexane

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FIGURE 3

Transfer Date: 11/21/97

Initials: MR

EXTRACT TRANSFER SHEET

Organochlorine HRB

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
39059-1	615	11/20/97	Aqueous	1060		10.0		
38209-1	T8151W	11/20/97	Sludge	200		10.0		

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FIGURE 4

Quality Assurance Deviation

Analysis Code:
QC Batch ID:
(attach copy or list samples affected by this memo)

Out of Control Event with Brief Explanation

☐ Method Blank

☐ Continuing Calibration or Initial Calibration Issue

☐ Laboratory Control Spike

☐ Matrix Spike/MSD

☐ Surrogate Recovery

☐ Other

SOP Corrective Action Followed:

☐ Re-extract in Hold time

☐ Re-inject to confirm

☐ Other

☐ Document SOP corrective action or "See QA Memo" on raw data file

QA Officer Review: _____ _____

UNCONTROLLED

Alkylphenols by GCMS-SIM

This is a procedure for the analysis of solvent extractable alkylphenol compounds by gas chromatography/ mass spectrometry (GC/MS) in the Selective Ion Monitoring Mode (SIM) in aqueous matrices. Quantitation is based on the internal standard calibration technique.

Sample Preparation

Samples are collected in the field in 1-L amber glass bottles preserved with H2SO4 to a pH -2. In the laboratory, the samples are extracted by separatory funnel with methylene chloride after addition of sodium chloride and spiking with extraction surrogates. The extract is dried and concentrated prior to analysis on the GC/MS-SIM.

Calibration

The GC/MS system is calibrated using initial and continuing calibration standards. These must meet the minimum criteria before any associated data is considered acceptable. An initial calibration (ICAL) is performed as needed, by analyzing a minimum of five standards containing the analytes at varying levels. Calibration is verified at the beginning of every 12 hour shift with a continuing calibration verification (CCV).

Calibration and quantitation are based upon the internal standard method. The concentration of the native analyte is calculated based on the response of the internal

standard in the method. Surrogate standards are spiked into the samples prior to extraction and labeled internal standards are added to the extract prior to analysis.

Sample Identification and Quantitation

Identification of an analyte is determined using the analyte's mass/charge ratio and the relative retention of the analyte compared to the CCV. For an identification to be made, a peak must appear within the mass of the analyte and within 0.06 RRT units of the RRT in the daily standard.

TABLE 1 TESTAMERICA WEST SACRAMENTO CONTROL LIMITS FOR LABORATORY CONTROL SAMPLES

Method	Parameter	Analyte	Aqueous samples (ug/L)	CAS Number
GC/MS-SIM	Alkylphenols	NP (technical mix)	10.0	84852-15-3
		NP1EO (technical mix)	20.0	
		NP2EO (technical mix)	40.0	
		p-tert-Octyl Phenol	2.0	140-66-9
		Bisphenol-A	2.0	80-05-7

TABLE 2 TESTAMERICA WEST SACRAMENTO CONTROL LIMITS FOR LABORATORY CONTROL SAMPLES (1)

Analytical Method	Spiking Compounds	Spike Concentration (ng)	Laboratory –Established Control Limits	
			Percent Recovery (%)	Relative Percent Difference (%)
GC/MS-SIM	NP	4000	40-140	<35
	NP1EO	4000	40-140	<35
	NP2EO	4000	40-140	<35
	p-tert-OP	4000	40-140	<35
	Bisphenol-A	4000	40-140	<35
	n-NP (surrogate)	4000	40-140	
	n-NP1EO (surrogate)	4000	40-140	
	Acenaphthene-d10 (Internal Standard)		50-200	
	Phhenanthrene-d10 (Internal Standard)		50-200	

(1) The control limits are subject to change at the discretion of the laboratory.

TABLE 3 **TESTAMERICA WEST SACRAMENTO SUMMARY OF CALIBRATION PROCEDURES**

Method	Parameter	Calibration	Frequency	Acceptance Criteria	Corrective Action
GC/MS-SIM	Alkylphenols by GC/MS-SIM	Tune using Decafluorotriphenylphosphine (DFTPP)	Prior to sample analysis	Masses within 0.45 amu of target masses.	1) Retune Instrument. 2) Reanalyze DFTPP.
		Multipoint Calibration (minimum 5 points) (ICAL)	Initially and as required	RSD ≤ 35% for all compounds (labelled and surrogates);	1) Evaluate system 2) Recalibrate
		Initial Calibration Verification (Second Source Standard)	With each ICAL	%D ≤ 30% for all target compounds	1) Rerun the ICV 2) Remake or acquire a new ICV 3) Evaluate the instrument conditions 4) Evaluate the Initial Calibration Standards
		Continuing Calibration Verification (CCV)	Beginning of every 12 hour shift when samples are analyzed.	%D ≤ 25% for target compounds (except NP, NP1EO, NP2EO)	1) Assess impact on data 2) Repeat, if adverse impact. 3) Reanalyze samples subsequent to failed CCV. 4) Perform new ICAL as needed.

TABLE 4 **TESTAMERICA WEST SACRAMENTO SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES**

Method	Method Type	QC Type	Frequency	Requirement	Corrective Action
GC/MS-SIM	Alkylphenols by GC/MS-SIM	Extraction Surrogates	Every sample, method blank, and LCS	Recovery within 40-140%	1) Check chromatogram for interference. If found, flag data. 2) Check instrument and reanalyze extract if a problem is found and corrected. 3) Re-extract and reanalyze adversely affected samples.
		Method Blank	1 per analytical batch, not to exceed 20 field samples, per matrix.	No target analyte > RL.	1) Reanalyze method blank if instrument carryover is suspected. 2) If still exceeds and analyte concentration in sample < RL or > 10x blank concentration, narrate and report results. 3) If non-compliant and analyte concentration is between RL and 10x blank concentration, re-extract and reanalyze affected samples.
		LCS	1 per analytical batch, not to exceed 20 field samples, per matrix.	Recovery within laboratory limits (Table 3)	1) Review calculations. 2) Evaluate data for usability. 3) If samples results are ND and RL are met, no action required. 4) If samples have positives >RL, re-extract and reanalyze samples for analytes outside the acceptance criteria.
		LCSD	Performed if insuffient sample volume for MS/MSD. One per analytical batch, not to exceed 20 field samples, per matrix	Recovery/RPD with laboratory limits (Table 2)	See LCS.
		MS/MSD	1 pair per analytical batch, not to exceed 20 field samples, per matrix	Recovery/RPD with laboratory limits (Table 2)	See LCS.

TABLE 4 TESTAMERICA WEST SACRAMENTO SUMMARY OF SAMPLE COLLECTION, PRESERVATION AND STORAGE

Method	Matrix	Hold Time	Container/Preservation
GC/MS-SIM	Aqueous	28 days from sampling to extraction. 40 days from extraction to analysis.	pH 2 with H2SO4, protect from light/4°C

TITLE: DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED

Prepared By: Peter Ly Date: 9/16/02

Approved By:

Group Supervisor: Peter Ly Date: 9/16/02

Lab Operations Mgr: John C. Burton Date: 9/16/02

QA Officer: Deborah J. Nadeau Date: 9.20.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Updated for Target. changed inj. vol. to 1ul. Changed LCS/D prep, Aq. Hold time, vol. of Surr. spike add to samples and QC. Updated GC conditions and formulas. Minor changes throughout.	LAD	030405	030405
02	updated instrument conditions. Fixed typos	LAD	04/06	04/06
03	Added vial waste streams to sect. 1 updated laboratory water information in Sect. 5.1. Added M1 information to sect. 7.8.3. Added 8000 changes to sect. 7 and Table 1.	LAD	08/07	08/07

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-309-03**, titled **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-309-03**, titled **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

Recipient: _____ Date: _____

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. technical personnel for the determination of Dimethylformamide (DMF) in surface water, ground water, and extracted sediments.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix. For Solid samples, clean sand is used as the blank matrix.

CALIBRATION CHECK (IND): Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized. An LCS is always analyzed after an initial calibration prior to any samples being analyzed.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. LIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of dimethylformamide using GC/NPD. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin technical personnel involved in analysis of dimethylformamide using GC/NPD, to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the Organic Vial Waste (P).

2.0 SUMMARY OF METHOD

This method for Dimethylformamide provides gas chromatographic conditions for the detection of PPM concentrations. Prior to the use of this method, appropriate sample preparation techniques must be used. A 1 ul aliquot of sample is injected into a gas

chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by a nitrogen phosphorous detector.

3.0 INTERFERENCES

Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe must be rinsed between samples with acetone followed by laboratory reagent grade water. Analyze a reagent blank after highly contaminated samples. Use clean vials for standard and sample preparation.

No chemical interferences have been documented for this method. However, when standards or samples containing DMF concentrations greater than 0.025 mg/L are analyzed, analyte carryover does occur. Two or three laboratory reagent grade water blanks should be analyzed immediately after any standard or sample with a DMF concentration greater than 0.025 mg/L. More reagent blanks may need to be analyzed after higher concentrated samples or standards. Before analyzing a sample that preceded a sample or standard containing DMF, the reagent blank preceding the following sample must not detect any DMF above the PQL.

Client history, when available, is essential when determining analytical run sequences. Grouping samples together by approximate concentrations will mean fewer blank analyses will be needed.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: GC Hewlett Packard 5890 series I or II connected to the Turbochrom data system, or equivalent.

4.2 Column: Supelcowax 30m x 0.53mm x 1um: Catalog #2.5301, or equivalent.

4.3 Silanized 4mm ID injection liner.

4.4 Detector: Nitrogen Phosphorous Detector (NPD).

4.5 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.6 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.7 Vials: various sizes and types including 2.0 mL crimp tops, 10.0 mL screw caps, and 40.0 mL VOA vials.

4.8 Balances: Analytical, 0.0001 g

4.9 Refrigerator for storage of samples and standards.

4.10 Epindorf Adjustable pipettors are used to measure the 1.0-mL aliquot of water or sample. The pipettor's should be calibrated at least once per month following specific instruction. Please reference SOP CA-103 Calibration of Adjustable Pipettors.

4.11 Nitrogen phosphorous bead for the NPD.

5.0 REAGENTS

5.1 Laboratory reagent grade water

5.2 Standards

5.2.1 Stock standard solutions: Neat solutions of Dimethylformamide and Diethylformamide, purchased from suppliers like Chemserv or other acceptable retailers. Expiration dates are manufacturers dates as indicated on the standard vial. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds.

5.2.2 Intermediate standard solutions: Prepared through the dilution of the stock standards with purged laboratory reagent grade water. Expiration is 6 months from the date of preparation or the manufacturers expiration date, whichever is sooner. Information is documented in the standards prep logbook. The individual concentrations of the intermediate standards of Dimethylformamide and Diethylformamide are 10,000 mg/L. The Dimethylformamide standard is further diluted to concentrations of 100 mg/L and 10 mg/L. The Diethylformamide standard is further diluted to a concentration of 100 mg/L. The Independent Calibration Verification standard/Laboratory Control Spike intermediate solution is prepared at a concentration of 100 mg/L.

5.2.3 Calibration standard solutions: Prepare at a minimum five different calibration standards using the intermediate DMF solution of 10 mg/L and the intermediate DEF solution of 100 mg/L. The calibration levels should be 0.003/0.10 mg/L, 0.02/0.25 mg/L, 0.05/0.50 mg/L, 0.10/1.0 mg/L, 0.50/4.0, and 1.0/NA mg/L of DMF/DEF.

5.2.4 LCS/LCSD and MS/MSD Spike Solutions: Prepare a solution of Dimethylformamide from a source that is different than that of the calibration standards at a concentration of 10 mg/L.

5.2.5 Surrogate standard solutions: The solution of Diethylformamide is prepared at a concentration 100 mg/L.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples are collected in 40 mL VOA vials with no headspace. Soils are collected in a glass soil jar. Both aqueous and solids are stored at 4 (±2) °C until time of analysis or extraction.

6.2 The extraction hold time for soil samples is 14 days from date of sampling.

6.3 The analytical hold time for aqueous samples is 14 days from date of sampling (unless otherwise specified from the client).

6.4 The analytical hold time for soil extractions is 40 days from date of extraction.

7.0 PROCEDURES

7.1 Water Sample Preparation

7.1.1 The method blanks are prepared by using 1.0 mL of laboratory reagent grade water. Samples are prepared by using a 1.0 mL aliquot. Add 5 ul of the 100 mg/L surrogate spiking solution to all of the 1.0 mL aliquots. The final surrogate concentration is 0.50 mg/L.

7.1.2 The LCS/LCSD is prepared by using 1.0 mL laboratory reagent grade water. Add 10 ul of the LCS/LCSD spiking solution at 10 mg/L (which contains DEF at 10 mg/L). The final DMF and surrogate concentrations are 0.10 mg/L.

7.2 Soil Sample Preparation

Note: For the analysis of 2-Methoxyethanol, the surrogate and spike standards may be added at the same time. Refer to the current revision of Katahdin SOP CA-310.

7.2.1 Prepare soil samples by weighing 1 to 2 grams of sample into a 40.0 mL VOA vial. For laboratory method blanks and laboratory control samples add 1 gram of clean sand. Add 40 mL of laboratory reagent grade water to each VOA vial.

7.2.2 For method blanks and samples, add 200 ul of the 100 mg/L surrogate spiking solution. For 2-Methoxyethanol analysis, add 10 ul of the 10,000 mg/L 2-Butoxyethanol solution. The final concentrations of the DEF surrogate are 0.50 mg/L or 20 mg/Kg.

7.2.3 Prepare the LCS/LCSD and MS/MSD by adding 100 ul of the LCS/LCSD spiking solution at 100 mg/L and 100 ul of the surrogate solution at 100 mg/L. For 2-Methoxyethanol analysis, add 10 ul of the 10,000 mg/L 2-Methoxyethanol solution. The final DMF and DEF concentrations are 2.5 mg/L and 10 mg/Kg.

7.2.4 The samples are then placed on a mechanical shaker and shaken for 12 to 18 hours. Let the samples settle for approximately 10 minutes, then transfer a portion of the top liquid layer to a 2.0 mL crimp cap vial for analysis.

7.3 INSTRUMENT SET UP

7.3.1 Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Helium Carrier: 5.0 mL/min.
Helium Auxiliary: 30 mL/min.
Air: 100-120 mL/min.
Hydrogen: 3-4 mL/min.
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 60; 10 degrees/min to 160
Run time: 10 min
Injection size: 1 ul
Column head pressure: 15 pKa

7.4 Initial Calibration

7.4.1 The GC system is calibrated using the external standard calibration procedure. A minimum of five calibration levels are prepared according to the concentrations listed in section 5.2.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height or area for each compound. A calibration curve can be prepared in Target using the peak height or area against the concentration of the standard. A linear calibration applying a first order polynomial equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient (r) must be greater than or equal to 0.995. For linear models, Target calculates the coefficient of determination (r^2). This value is also equal to the correlation coefficient squared. The value for r^2 must be greater than or equal to 0.990.

The linear equation is:

$$y = mx + b$$

where: y = Instrument response
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

7.4.2 The working calibration curve must be verified daily and after every 10 samples by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 25%, a new calibration curve must be prepared for that analyte.

7.4.3 An LCS must be analyzed after an initial calibration prior to any samples being analyzed.

7.5 Retention time windows

7.5.1 Three injections are made of Dimethylformamide throughout the course of a 72-hour period.

7.5.2 A standard deviation is calculated using the three retention times of the peak.

7.5.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms.

7.5.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.5.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply a nominal window. This is done in order to avoid any false negative hits because of the window being to narrow. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms.

7.6 Gas chromatographic analysis

7.6.1 All instrument injections are performed using the direct injection technique with an autosampler set for a 1 ul injection volume.

7.6.2 Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with instrument calibration as listed in section 7.4 followed by sample aliquots interspersed with mid-concentration calibration standards.

Before any samples are analyzed the instrument must be calibrated by analyzing a five-point calibration or a mid-concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 25%. Each sample analysis must be bracketed with an acceptable initial calibration and a closing CV, or an opening CV and a closing CV. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. All samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard analyzed <u>after</u> a group of samples exhibits a response for an analyte that is <u>above</u> the acceptance limit, i.e. >25%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the analyses for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits <u>was</u> detected in a sample analysis, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 25% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.6.3 Absolute retention time windows are established using the mid-point of the window of that day if after analyzing the mid-point it is determined that one or more of the analytes fall outside of the previously established absolute retention time window. The daily retention time window equals the mid-point ±three times the standard deviations.

7.6.4 The identification of Dimethylformamide is based on agreement between the retention time of a peak in the sample chromatogram with the retention time window established through the analysis of standards of the target analyte. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window.

7.6.5 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.6.6 If the amount recovered is not detectable or below the PQL, then the compound is not considered to be present in the sample and is reported as <PQL.

7.6.7 When a GC system is determined to be out of control because either a CV can not pass or a five point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the column, or replacing the column. If there is a significant loss of response or the baseline is very high or unstable, the nitrogen phosphorous bead may need to be replaced. This information is recorded in the instrument run log (Figure 3). When an instrument requires more severe maintenance like replacing the NPD or an electronic board, this information is written in the instrument maintenance logbook.

7.7 Calculations

7.7.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.

7.7.2 Final concentrations are calculated based on the sample matrix using one of the two calculations listed below.

7.7.2.1 Water: Concentration (mg/L) = Amt * DF * (Vt/Vo)

7.7.2.2 Soil/Sediment: Concentration (mg/kg) = Amt * DF * (Vt/Vo) * (100/(100-M))

where: Amt = concentration calculated by Target in mg/L
Vt = Volume of total extract (L)
Vo = Sample Weight or Volume (Kg or L)
M = % Moisture
DF = Dilution at instrument

7.8 Data Review

7.8.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly

impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.9.

7.8.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recovery of the surrogate. The sample chromatogram is reviewed for any interferences before determining whether to accept a sample based on the surrogate recoveries. If the surrogate recovery is affected by matrix interference, the sample result may be accepted with narration. If the recovery for the surrogate is not acceptable because the recovery is high and the sample does not contain any analytes above the PQL, the data is narrated. If the recovery for the surrogate is low and there is no apparent matrix effect, the sample is reextracted or reprepped.

For method blanks, if the recovery of the surrogate is low or high, and the blank does not contain any Dimethylformamide above the PQL, and the recovery of the surrogate in the sample(s) is acceptable, the data is narrated. If the recovery in the blank is low and it does not contain any Dimethylformamide above the PQL, and the recovery in the samples are acceptable but the sample contains Dimethylformamide above the PQL, the sample may be reextracted or reprepped.

For laboratory control samples (LCS), if the only discrepancy in the analytical batch is with the LCS, and the Dimethylformamide recovery is acceptable, the data is narrated. If the recoveries of both the surrogate and the Dimethylformamide are low, the associated samples may need to be reprepped.

7.8.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

In Target Review, each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), manual integration is performed in Target Review. A "m" qualifier will automatically be printed on the quantitation report summary indicating that a manual integration was performed. The analyst will date and initial the "m" on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.8.4 Target Compound Detection

The chromatogram is evaluated to determine if Dimethylformamide is detected. The concentration of the analyte is then evaluated to determine if it is above the PQL and within the calibration range.

7.9 Reporting

7.9.1 After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked with Dimethylformamide and Diethylformamide. The spike concentrations are:

	WATER mg/L	SOILS mg/Kg
Dimethylformamide	0.10	10

The surrogate spike concentrations in the final extract are:

	WATER mg/L	SOILS mg/Kg
Diethylformamide	0.50	10

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Please note that established acceptance limits that are wider than 70-130% may not be allowable for certain states, federal

programs, or clients. For South Carolina, the acceptance limits for the spiked analytes will be 70-130% or narrower.

If the spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reprepped. However, if the recovery is high and the associated samples do not contain the specific compound, the data can possibly be accepted with narration.

If the spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reprepped.

8.5 Corrective Action Report (CAR): Whenever data is not acceptable because of a failing LCS or surrogate recovery, a CAR must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Department Manager and with the QAO. The Practical Quantitation Limit (PQL) concentrations for all the target analytes are listed in Figure 3.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL. Refer to the current revisions of USEPA Method 8033 for other method performance parameters and requirements.

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Methods 8033 US EPA, Update III, June 1997.
Katahdin Analytical Services, Inc., SOP CA-208, Traceability of Standards - Organics

Katahdin Analytical Services SOP CA-106, "Standards Prep and Documentation", current revision.
Katahdin Analytical Services SOP QA-805, "Laboratory Technical Personnel Training", current revision.

Katahdin Analytical Services SOP QA-103, "Calibration of Adjustable pipettors", current revision.

Katahdin Analytical Services SOP QA-806, "MDL and IDL Studies", current revision

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TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of 20 or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC:i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/LCSD	One set per prep batch	Statistically derived limits	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CV	If calibration curve previously analyzed, analyze daily before samples and after every 10 samples.	± 25% D	(1) Evaluate the samples: If the %D>+25% and sample results are <PQL, narrate. If %D>±25% and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate	One sample duplicate per ten samples if requested	RPD ≤20	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
5pt calibration	Initial cal prior to sample analysis	5 pt calibration - correlation coefficient (R) ≥ 0.995 or coefficient of determination (R2) ≥ 0.990	Repeat initial calibration
MDL study	Once per year	Ideally, PQL = at least 3 X the MDL.	Repeat MDL study

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-309-03	METHOD 8033 (GC/NPD)
Apparatus/Materials	1.0 Dimethylformamide	1.1 Acetonitrile
Reagents	5.2.1 … Expiration dates are manufacturers dates as indicated on the standard vial…Standards are stored at 4°C ± 2°C. 5.2.2 …Expiration is 6 months…	5.6.2 The stock standard solution is stable at 20-25 °C for about 3 months.
Sample preservation/ handling		
Procedures	7.4.2 If the response for any analyte varies from the expected response by more than ±25%, anew calibration curve must be prepared for that analyte.	8000B 7.7 if the response of an analyte is within ± 15%…
Procedures	5.2.5 Surrogate Standard solutions: The solution of diethylformamide is prepared at a concentration of 100 mg/L.	There is no mention of a surrogate in the specific method.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

FIGURE 1

EXAMPLE OF GC RUNLOG PAGE FOR DMF

Katahdin Analytical Services, Inc
GC Laboratory Instrument Runlog
Instrument GC11

Reviewed by/Date:_____

Method (circle):
SW846 8015M - Glycols
SW846 8015M - Alcohols
SW846 8015M - 2-MOE
SW846 8033M - DMF
Signal 30 **Power**_____

Date	Init	Result File	Sample Number	Accept Y/N	Method Channel A: B:	Column	Comments
6.19.07	SJC	BAF2201	SA2807-12	Y	DMFB029A	227	
		202	-13				
		203	Water	N			
6.20.07		204	CV 4	Y			
		205	Water	N			
		206	SA2807-14	Y			
		207	-15				
		208	-16				
		209	-17				
		210	-18				
		211	Water	N			
		212	CV 4	Y			
		213	Water	N			
		214	d				
		215	CV 4	Y			
		216	Water	N			
		217	WG40226-1	Y			
		218	-2				
		219	Water	N			
		220	WG40226-3	Y			
		221	Water	N			
		222	SA2644-1	Y			
		223	WG40226-4				
		224	Water	N			
		225	SA2644-2	Y			
		226	-3				
		227	SA2806-1				

	STANDARD ID	uL added	STD. CODE	STD. CODE
1	Surrogate	5	GCV2021	
2	LCS	10	GCV2024	
3	CV		GCV2028	
4				

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

FIGURE 2

DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date _____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Sample Data Summary Included (Level III & IV). _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration **(Narrate as needed).** _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

_____

First correction ☐ → Review and replace appropriate SDS Forms ☐ .

Second correction ☐ → Review and replace appropriate SDS Forms ☐ .

Last saved by kasgc04/13/07

TITLE: **DIRECT INJECTION METHOD FOR THE DETERMINATION OF DIMETHYL-FORMAMIDE USING GC/NPD – EPA SW-846 METHOD 8033 MODIFIED**

FIGURE 3

PQLS

COMPOUND	PQL (mg/L)	PQL (mg/Kg)
Dimethylformamide	0.003	0.20

Title: **PHTHALIC ACID AND MALEIC ACID IN WATER AND SOIL BY HPLC (TestAmerica Tallahassee)**

Approvals (Signature/Date):

6.5.08	06-05-08
Chris Amason — Date Technical Manager	Jim Robbins — Date Environmental Health & Safety Coordinator
Lou Schneider 6/5/08	06-05-08
Lou Schneider — Date Quality Assurance Manager	Todd Baumgartner — Date Laboratory Director

This SOP was previously identified as SOP No. LC65.

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1.0 SCOPE AND APPLICATION

1.1 This method is used for the determination of phthalic acid and maleic acid by high performance liquid chromatography (HPLC). This procedure is applicable to water and soil samples.

1.2 The routine reporting limits (RL) are listed in TL-QA-001: *TestAmerica Tallahassee Method/Analyte List.* The method detection limits (MDL) and accuracy and precision control limits for the target compounds are found in the TestAmerica LIMS System (TALS LIMS).

1.3 An example chromatogram is provided in Appendix B to show the approximate retention times of the target compounds.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 A known volume of liquid sample, nominally 50mL, is adjusted to a pH range of three to ten. The sample passes through C8 and amino solid phase extraction cartridges that have been pre-washed with the phthalic acid and maleic acid eluting solution and HPLC water. The analytes are eluted from the cartridges with the eluting solution, filtered and analyzed by HPLC with UV detection at 200nm.

Soil samples are leached with water for thirty minutes. The resulting leachate is then subjected to the same preparation steps and analytical procedures as aqueous samples.

2.2 No EPA method exists for the analysis of the target analytes. This procedure was developed by TA.

2.3 Definitions

2.3.1 Elute: to extract one material from another, usually by means of a solvent (in this case, phthalic acid and maleic acid eluting solution)

2.3.2 Isocratic: a chromatographic separation that employs a single solvent of constant composition

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the facility addendum to the CSM, and this document.

3.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

There are no specialized safety concerns associated with this method.

3.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetonitrile	Flammable Poison	40 ppm – TWA	Early symptoms may include nose and throat irritation, flushing of the face and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Hexane	Flammable Irritant	500 ppm – TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm -- TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur: symptoms may parallel inhalation exposure. Irritant to the eyes.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain and severe skin burns. May cause redness, pain, blurred vision, eye burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns or scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat and labored breathing. Symptoms of redness, pain and severe burning can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 INTERFERENCES

4.1 Analytical interferences may arise from improperly cleaned glassware. All glassware must be scrupulously cleaned before use according to SOP TL-AN-060: *Glassware Cleaning Procedures.*

4.2 All of the materials and reagents must be demonstrated to be free from contaminants by the analysis of reagent blanks (method blanks).

4.3 All extracts for samples and associated QC must be filtered prior to analysis to eliminate particulate matter.

4.4 Matrix-related interferences may be caused by contaminants that are extracted from the sample matrix or from co-eluting analytes in the sample. Dilution may be required prior to analysis to reduce or eliminate the interferences.

4.5 The drying oven used to determine the percent solids should be contained in a hood or in an area of the lab that is isolated from the preparation and analysis areas. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample.

5.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

5.1 Samples should be collected in glass or plastic jars and maintained at 4 °C out of direct sunlight (less than 6 °C with no frozen samples). Samples should be iced for shipment. A minimum volume of 100mL should be collected for aqueous samples and a minimum of 10g for soils. Additional volume should be provided for matrix spike samples and dry weights. No preservative is added (orange dot). Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction. Soils must be extracted within 14 days of collection and analyzed within 40 days of extraction. This information is summarized in a table in Appendix A.

6.0 MATERIALS AND APPARATUS

6.1 Waters HPLC isocratic 501 pump, gradient 600E pump or equivalent

6.2 Waters 484 or 486 UV detector or equivalent detector with variable wavelength detection capable of achieving the required detection limits

6.3 Waters 717 autosampler, Waters 712 WISP or equivalent

6.4 HPLC column heater and controller, Waters or equivalent

6.5 Recommended analytical columns:
Primary analytical column: RTF C8 4.6mm x 150mm or equivalent.
Confirmatory analytical column: HP Anion EX 4.6mm x 100mm or equivalent

6.6 Microsyringes – appropriate volumes for preparation of standards and dilutions

6.7 Volumetric glassware (Class A) -- appropriate volumes

6.8 Analytical balance accurate to ± 0.0001g

6.9 Autosampler vials -- 4mL and 10mL amber with screw caps and septa.

6.10 pH paper

6.11 Class A glass pipettes -- appropriate sizes

6.12 Solid phase cartridges – amino (NH2) (Waters or equivalent) and C8 (Waters, VWR or equivalent)

6.13 Syringe filters -- 0.45µm nylon (Whatman or equivalent).

6.14 40mL glass VOC vials

6.15 Tumbler

6.16 Centrifuge

6.17 Disposable syringes

6.18 Pasteur pipettes

6.19 SPE vacuum filtration apparatus

6.20 Spatulas

6.21 Graduated cylinders – appropriate sizes

6.22 75mL sample reservoirs

6.23 Guard columns – C8 and Anion Exchange

6.24 Helium -- For degassing solutions and solvents

6.25 Syringes -- 60mL Gastight with 1/16" stainless steel tubing, for pump priming

7.0 REAGENTS

7.1 The recipes used for reagent preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. All reagents should be transferred, recorded, labeled and stored according to SOP TL-AN-045: *Laboratory Notebooks*. Reagents must be tracked in accordance with SOP TL-AN-041: *Standard Materials and Reagent Traceability*.

7.2 Reagent water -- HPLC-grade water

7.3 Acetonitrile -- HPLC grade

7.4 Methanol -- HPLC grade

7.5 Hexane

7.6 Ammonium Phosphate -- monobasic

7.7 Phosphoric Acid (85%) -- reagent grade

7.8 Potassium Phosphate – monobasic

7.9 Sulfuric Acid

7.10 Sodium Hydroxide

7.11 Reagents Preparation

7.11.1 Sulfuric Acid (1:1) -- Slowly add 500mL reagent grade sulfuric acid to 500mL reagent water.

7.11.2 Sodium Hydroxide (10N) -- Dissolve 400g of reagent grade sodium hydroxide in reagent water to a final volume of 1000mL.

7.11.3 SPE cartridge eluting solution -- Add 57.5g of HPLC grade ammonium phosphate, monobasic, and 20mL of HPLC grade phosphoric acid to 1L of HPLC grade water. Stir to dissolve the salt. Store in the refrigerator.

7.11.4 Confirmation mobile phase -- Combine 9.2g of ammonium phosphate, monobasic, and 4mL of phosphoric acid (85%) and dilute to a final volume of 4L with reagent grade water. Degas with helium.

7.11.5 Primary mobile phase -- Mix 600mL of buffer (confirmation mobile phase) with 1400mL of acetonitrile. Degas with helium.

8.0 STANDARDS

8.1 The recipes used for standard preparation must be clearly documented as a controlled posting or as a narrative in the traceability log. All diluted standards should be transferred, recorded, labeled and stored according to SOP TL-AN-045: *Laboratory Notebooks*. Standards must be tracked in accordance with SOP TL-AN-041: *Standard Materials and Reagent Traceability*. General guidelines on the preparation of standards are given in SOPTL-AN-043: *Standard Preparation*.

8.2 Neat standards of phthalic acid and maleic acid -- highest purity available, primary and second source

8.3 Standards Preparation

8.3.1 Stock Standards: Prepare stock standards of each target compound at about 1000µg/mL. Maleic acid is prepared in eluting solution (7.11.3) and phthalic acid is prepared in methanol. Accurately weigh about 0.0100 grams into a glass weighing funnel, then transfer to a 10mL volumetric flask by rinsing with the appropriate solvent. Fill to volume with appropriate solvent. No purity correction is needed if the analyte is at least 96% pure. Transfer the stock standard to a 10mL amber vial and store at 4 °C. Prepare this standard every six months, or sooner if degradation is noted.

8.3.2 Intermediate standard: Prepare an intermediate standard from which to make the curve at 100µg/mL in eluting solution. Prepare this standard every month, or sooner if degradation is noted.

8.3.3 Calibration Standards: Prepare three calibration standards in the prepared SPE cartridge eluting solution at the following concentrations: 0.10µg/mL, 0.50µg/mL and 10µg/mL. Calibration standards are good for up to seven days from the date of preparation. Refer to the table in Section 10.1.2 for further details.

Curve levels may be adjusted according to project requirements as long as the low-level standard supports the reporting limit. The low level standard should be at or below the PQL and the high level standard should define the linear range of the detector. A 20-fold increase from low to high is common for HPLC.

8.3.4 Spike solution (QC Standard): From independently prepared stock standards, prepare a 100µg/mL spike solution in methanol. Prepare this standard every month, or sooner if degradation is noted.

8.3.5 The analyst is cautioned that these are maximum hold times and not to be considered an absolute guarantee of standard quality. The analyst must use sound judgment when deciding whether to use a standard. If the analyst is in doubt about the quality of a standard material, a new material must be obtained or the standard material verified. Do not compromise data quality to extend a standard's life – when in doubt, throw it out.

9.0 SAMPLE PREPARATION

9.1 Aqueous Sample Preparation

9.1.1 Prepare all glassware according to SOP AN60. Rinse all glassware with methanol or acetonitrile prior to use and allow to dry. Remove samples from cooler and allow to equilibrate to room temperature before beginning prep.

9.1.2 Check the pH of the samples with wide range pH paper and record.

9.1.3 Homogenize the sample and transfer 50mL to a plastic beaker. Repeat for all samples. If sufficient sample is available, transfer 50mL of the sample chosen for the matrix spike (MS) and matrix spike duplicate (MSD) into each of two plastic beakers.

9.1.4 Add 50mL of HPLC grade water to each of three plastic beakers. One will be for the method blank (MB) and the others for the lab control spike (LCS) and lab control spike duplicate (LCSD).

9.1.5 Spike the lab spikes and matrix spikes with 20µL of the 100µg/mL spiking solution.

9.1.6 Adjust the pH between three and ten with 1:1 sulfuric acid or 10N sodium hydroxide. Add the reagents drop-wise and mix thoroughly after each addition, checking the pH with wide range pH paper. Record the final pH.

9.1.7 Optional cleanup for "dirty" samples: Add the 50mL sample and 10mL of hexane to a 100mL separatory funnel. Shake vigorously for 30 seconds and vent. Drain the water layer into a plastic beaker and discard the hexane layer.

9.1.8 Assemble the solid phase extraction apparatus. Connect the sample reservoir to a C8 cartridge and connect an amino cartridge to the C8 cartridge so that the samples will pass through the C8 cartridge first and then through the amino cartridge. Label the amino cartridges. Connect the amino end to the SPE manifold. Condition the cartridges as follows:

-- Add about 5mL of the eluting solution (Section 7.11.3) to the reservoir and turn on the vacuum until the solution is drawn through the cartridges leaving a small amount of liquid in the reservoir. Do NOT let the cartridges go completely dry.

-- Add about 10mL of HPLC water to the reservoir and pass about 5mL through the cartridges. Do NOT let the cartridges go completely dry. Turn off the vacuum.

9.1.9 Add the sample to the reservoir. If the hexane clean up was employed, drain the lower layer into the reservoir and discard the hexane layer. Turn on the vacuum and watch for the samples to be seen passing through the cartridges. Allow the samples to pass through both cartridges dropwise.

9.1.10 After the sample has passed through the cartridges, allow the vacuum to remain on for an additional minute or more. Discard the C8 cartridges but retain the amino cartridges for elution of the target compounds.

9.1.11 Attach a 10mL disposable plastic syringe to the amino cartridge and remove the plunger from the syringe. Attach a 0.45µm polypropylene or nylon filter to the other end of the amino cartridge. Add 4.0mL of the eluting solution to the syringe. Insert the plunger and elute the cartridge slowly (drop-wise), collecting the extracts in properly labeled 4mL amber HPLC vials.

9.2 Solid Sample Preparation

9.2.1 Prepare all glassware according to SOP TL-AN-060. Rinse all glassware with methanol or acetonitrile prior to use and allow to dry. Remove samples from cooler and allow to equilibrate to room temperature before beginning prep.

9.2.2 Homogenize the sample and weigh 5.0g into a labeled 40mL VOC vial. Repeat for all samples. Weigh two additional 5.0g aliquots for the MS and MSD.

NOTE: The proper and thorough homogenization of soil samples is absolutely essential for producing representative analysis results. See SOP TL-AN-070: *Homogenization, Compositing, and Segregation of Samples* for further guidance on sample homogenization.

9.2.3 In most cases you will need to perform dry weights by weighing out 10.0g of sample into a labeled metal weigh boat and allowing it to dry overnight.

9.2.4 Add 25mL of HPLC grade water into three labeled 40mL vials to serve as the method blank and lab spikes.

9.2.5 Spike the lab spikes and matrix spikes with 20µL of the 100µg/mL spiking solution.

9.2.6 Add 25mL of HPLC grade water to each soil sample and place the samples and QC in a vial rack.

9.2.7 Tumble the vial rack for thirty minutes.

9.2.8 Centrifuge the soil samples until clear (about 2 minutes at high speed) or allow them to settle for several hours in the refrigerator.

9.2.9 Draw off as much of the leachate as possible with a 10mL disposable pipette into a plastic beaker. Check and record the pH. Proceed with the extraction in the same manner as aqueous samples, starting at Section 9.1.6.

10.0 PROCEDURE

10.1 Calibration: SOP TL-AN-067: *Evaluation of Calibration Curves* provides general guidance on how to evaluate the calibration data.

10.1.2 The following table summarizes the recommended concentrations of the three-point calibration curve. Specific information on how to prepare the calibration standards can be found in Section 8.3. Calibration standards are good for up to seven days after being made.

Cal Standard	Intermediate Conc (µg/mL)	Volume of Stock (µL)	Final Volume (mL)	Conc (µg/mL)
1	100	10	10	0.10
2	100	50	10	0.50
3	100	1000 (1.0mL)	10	10

10.1.3 Initial Calibration

10.1.3.1 Each instrument must be initially calibrated by the analysis of a minimum of three calibration standards. The standards are prepared using the guidance in Sections 10.1.2 and 8.3 and analyzed using the guidance described in Section 10.5. External standard methodology is used for this method.

- Analyze the calibration standards using the instrument conditions optimized for the target compounds. The injection volume must be kept constant for all analyses -- calibration standards, QC sample extracts and client sample extracts.

- Identify each target compound and tabulate the retention time (RT), concentration (µg/mL) and the corresponding area for each target compound.

10.1.3.2 The initial 3-point calibration may be evaluated using either the average response factor (or calibration factor, CF) or linear regression fit.

10.1.3.3 Linear Regression Curve

The concentration (µg/mL) of the target compound is plotted on the x-axis and the corresponding response is plotted on the y-axis.

Linear regression fit may be used to define the concentration/response relationship. If the correlation coefficient (r^2) of the regression curve is greater than 0.990, the curve can be used to quantify samples. The analyst must ensure that the regression curve selected accurately defines the concentration/response relationship over the entire calibration range.

10.1.3.4 Alternatively, calculate the calibration factor (CF) for each target compound at each calibration level. Calculate the average calibration factor for the initial calibration levels. Calculate the standard deviation of the calibration factors. Finally, calculate the relative standard deviation (%RSD) of the levels for each target. Examples of these calculations are in Section 11.1.

For this method, if the %RSD of each target compound is less than or equal to 10%, the calibration is assumed to be linear through the origin and the average calibration factor (RFavg) is used for quantitation.

10.1.3.5 The retention time window (RTW) for each target compound must be determined using the guidance provided in SOP TL-AN-066: *Determination and Evaluation of Retention Time Windows for Chromatographic Analyses.* The RTW must be documented and used to calculate the daily retention time window that is used to identify the target compounds in samples. Alternatively, default RT windows may be used for capillary column analyses.

10.2 Continuing Calibration Verification

10.2.1 A continuing calibration verification check must be performed after every 10 field samples and at the beginning and end of the run by injecting a mid-level standard. Evaluate the percent drift or percent difference from the initial calibration curve. If the drift or difference is less than or equal to 20%, the curve is verified. If the first continuing calibration check fails criteria, a second standard may be analyzed. If it also fails, the initial calibration must be repeated.

10.2.2 For projects that require Florida DEP QAS criteria, the lowest concentration calibration standard (at or near the RL) must be analyzed the same day that sample extracts are analyzed (PQL verification standard) to demonstrate that the reporting limit (RL) can be achieved. This criterion is met if the target compound is detected.

10.3 Instrument Conditions

10.3.1 The analytical conditions below are provided for guidance. These conditions may be modified to optimize sensitivity and separation. The analyst must document the analytical conditions if different from those described below.

10.3.2 Primary Analysis Conditions:
Analytical column = RTF C8 4.6mm x 150mm or equivalent
Column Temperature = 30°C
Guard Column = C8
Isocratic Mobile Phase = 30:70 20mM ammonium phosphate (monobasic), pH = 2:Acetonitrile – degassed with helium
Detector = 484 or 486 Waters UV detector or equivalent
Wavelength = 200nm
Flow Rate = 1.0mL/min
Injection Volume = 50µL
Run Time = 15min
Nelson Method = PHTH-MAL

10.3.3 Confirmation Analysis Conditions:
Analytical Column = HP Anion EX 4.6mm x 100mm or equivalent
Column Temperature = 30°C
Guard Column = Anion Exchange
Isocratic Mobile Phase = 20mM ammonium phosphate (monobasic) – degassed with helium
Detector = 484 or 486 Waters UV or equivalent
Wavelength = 200nm
Flow Rate = 1.5mL/min
Injection Volume = 20µl
Run Time = 12min
Nelson Method = MAL-CON

10.4 Instrument Startup

10.4.1 Pump: Place solvent lines in fresh solvent that has been degassed with helium. Remove air from the solvent lines by inserting the 60mL syringe fitting into the tube on the drain valve. Open the drain valve just until you can withdraw solvent. Withdraw about 20mL of mobile phase, close the valve, remove the syringe and discard the solvent. Repeat until line is free of air bubbles. Continuing presence of bubbles indicates that the solvent is not degassed well enough. When lines are free of bubbles, turn the flow rate up to the proper rate by 0.5mL/min increments, allowing pressure to stabilize between increases.

10.4.2 Detector: Turn on the detector power when the rest of the system is turned on. The wavelength and range are displayed on the front panel. Check for correct settings.

10.4.3 Allow at least 30 minutes for system equilibration before injecting samples. Temperature stability is critical. During equilibration, a thorough leak-check should be conducted. Check to be sure that all of the parameters are set to the proper values. Make sure the sequence is entered and downloaded. Auto-zero the detector before injecting.

10.5 Qualitative Sample Analysis

10.5.1 Sample extracts are injected into the HPLC system following calibration. The same injection volume is used for all standards and samples.

A sequence should be set as described in the SOP Summary in Appendix A. The sequence continues until all samples have been analyzed or until the CCV fails the acceptance criteria. The sequence must end with an acceptable CCV.

Samples should be run in the following order: method blanks, spikes, clean samples and finally, highly contaminated samples.

10.5.2 The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times), whether the peak response falls within the working range of the calibration and the integration of the peaks. Manual integration must be documented in accordance with SOP TL-AN-065: *Manual Integrations*. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data.

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. The analyst should ask:

- Is there previous data or current information about the sample that would aid in evaluating the data?
- Do the peaks look normal?
- Are peaks properly integrated?
- Are co-eluting peaks or matrix interferences present?

10.5.3 Identification of the target compounds is based on retention time. The analyst should scan the sample chromatogram for the target compounds on the primary analytical column. The retention time (RT) windows calculated around the CCV retention times are used for the identification of the target compounds. If, in the professional judgment of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as a target, the result may be reported as a "non detect".

NOTE: It is important to note that the retention time window applies only to peaks that are within the calibration range of the curve. Peak areas that exceed the established linear range of the calibration curve may result in significant retention time shifts; therefore, all peaks that have significant areas and elute closely to a target compound should be tentatively identified as a target compound and evaluated as such. Peaks over-range are diluted and re-analyzed.

10.5.4 Verify that the data system has correctly labeled and calculated a concentration for each peak that corresponds to a target compound. Observe the general appearance of the chromatogram for possible dilutions, matrix interferences and the overall shapes of the peaks.

If the concentration is below the detection limit, the reporting limit (RL) for that compound is reported (Section 11.3). The RL is calculated for all target compounds that are not detected on the primary analytical column. Any data system generated hits "called off" by the analyst should be flagged by the data system as to the reason. Peaks over-range are handled using dilutions as detailed below (10.5.6).

10.5.5 A compound is tentatively identified as a target compound if a peak falls within the target's retention time window on the primary analytical column. The extract is then analyzed on the confirmation column. If the target is detected on both columns, it is quantified and reported. The relative percent difference should be calculated. An example of this calculation is in Section 11.4.

Report the higher of the two results if the relative percent difference (RPD) is less than 40%. Report the lower of the two results and flag the results if the RPD is greater than 40% or notify the project manager via a case narrative.
NOTE: The relative percent difference between any two numbers will be a maximum of 200%. A large relative percent difference may be acceptable at concentrations near the reporting limit. If in doubt about whether to report a peak as a quantitative result, consult the section supervisor.

If the %RPD is greater than 40%, evaluate the chromatograms to determine if matrix interferences are present on one or both columns. If interference is detected, flag the result to note the disparity between the results. Alternatively, dilute the extract to a level that removes the interference and report the RL from this dilution.

10.5.6 If the concentration of any target compound exceeds the calibration range (that is, the highest level standard included in the initial calibration), a dilution must be performed on the extract and the diluted extract is reanalyzed. Dilutions should be made in phthalic acid and maleic acid diluting solution. A dilution should be made to bring the area of the peak of interest into the upper half of the calibration curve; that is, between the mid-level calibration standard and the highest level standard. An example of this calculation is in Section 11.5.

NOTE: If a peak is over range on the primary column, evaluate the confirmation column. If no peak is detected or if the concentration is within the calibration range, the analysis at a dilution is not necessary.

10.5.7 The general guidance for the evaluating of chromatographic data is summarized in a table in appendix A. The table assumes that the calibration criteria have been met and that the sample has acceptable associated lab spike recoveries.

10.5.8 The analyst must clearly show how the reported sample results were determined. False hits identified as targets by the data system should be "called off" and a reason code assigned by the analyst. Manual calculations and integrations must be documented.

10.6 Identification Tools

10.6.1 Co-Injection

Another useful "tool" is to add a known amount of the target analyte to a portion of the extract. The analysis of this "fortified extract" may provide chromatographic information that supports or refutes the initial identification. The analyst is cautioned to use this approach with discretion and with consultation with the department supervisor. As a general rule, spike a portion of the extract with an amount of target analyte that will result in about a two-fold increase in response.

NOTE: Do not perform this procedure until you have exhausted all other avenues and have consulted with the supervisor or other manager with LC experience.

10.6.2 Chromatogram "Overlay"

One useful tool that employs a similar idea to the relative retention time (RRT) is to "overlay" the sample chromatogram with the calibration standard. If the chromatograms are scaled the same, the overlay provides good visual cues to the identification of the target compound.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Initial Calibration Calculations

Calculate the calibration factor (CF) for each target compound at each calibration level:

$$CF = \frac{response}{concentration} = \frac{area}{ug/mL}$$

Response = peak area or peak height

Calculate the average calibration factor for the initial calibration levels:

$$CFavg = \frac{CF1 + CF2 + CF3 ... + CFn}{n}$$

Note that n must be greater than or equal to 3 for phthalic acid and maleic acid.

Calculate the standard deviation of the calibration factors:

$$Standard\ \ Deviation = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF_{avg}})^2}{n-1}}$$

Where
CF_i = calibration factor of the individual calibration level
CF_{avg}= average calibration factor

Calculate the relative standard deviation (%RSD) of the levels for each target:

$$\%\ RSD = \frac{standard\ deviation}{\overline{CF_{avg}}} \otimes 100$$

11.2 Continuing Calibration Calculations

11.2.1 If a regression curve has been chosen to quantify the samples; the percent difference is calculated for the CCV:

$$\%difference = \frac{Ct - Cccv}{Ct} \otimes 100$$

Where
Ct = expected concentration of the CCV
Cccv = concentration of CCV determined from regression curve

11.2.2 If the average calibration factor has been used to evaluate the calibration curve, the percent difference is calculated as follows:

$$\%difference = \frac{CFavg - CFccv}{CFavg} \otimes 100$$

Where
CFccv = calibration factor of the target compound from continuing calibration check standard
CFavg = average calibration factor of the target compound (ICAL)

11.3 Calculations -- External Standard

11.3.1 Aqueous/Liquid Samples

Aqueous/Liquid Samples -- Regression curve option

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{V}$$

Where
Ccurve = concentration of analyte from curve (µg/mL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

Aqueous/Liquid Samples -- Calibration factor option

$$Concentration(ug/L) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{V}$$

Where
Response = area (or height) of the target
CF_{avg} = average calibration factor (ICAL)
F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)

11.3.2 Soils/Solids Samples

Soils/Solids Samples -- Regression Curve Option

$$Concentration(ug/L) = Ccurve \otimes \frac{F \otimes DF}{W \otimes solids}$$

Where
Ccurve = concentration of analyte from curve (µg/mL)
F = final volume of the extract (mL)
DF = dilution factor
W = weight of sample extracted (kg)
Solids = (percent solids)/100

Soils/Solid Samples -- Calibration factor option

$$Concentration(ug/kg, dw) = \frac{response}{CF_{avg}} \otimes \frac{F \otimes DF}{W \otimes solids}$$

Where
Response = area (or height) of the target
CF_{avg} = average calibration factor

F = final volume of the extract (mL)
DF = dilution factor
V = volume of sample extracted (L)
W = weight of sample extracted (kg)
Solids = (percent solids)/100

11.4 The relative percent difference should be calculated as follows:

$$\%RPD = \left| \frac{(Cprim - Cconf)}{\frac{(Cprim + Cconf)}{2}} \right| \otimes 100$$

Where
Cprim = concentration of the target compound on the primary column
Cconf = concentration of the target compound on the confirmation column

11.5 Dilutions are made as follows:

$$DF = \frac{FV}{Vs}$$

Where
FV = final volume of the dilution (mL)
Vs = volume of sample used to prepare the dilution (mL)

12.0 QUALITY CONTROL

12.1 This procedure is restricted to use by or under the supervision of analysts experienced in the use of liquid chromatography. Each analyst must demonstrate the ability to generate acceptable results (within the prescribed accuracy and precision limits) with this method by the analysis of reagent soil and water samples that have been spiked with the target compounds. Each analyst must participate (individually or as part of a work group) in the analysis and evaluation of **initial demonstration of capability (IDOC)** samples to demonstrate minimum proficiency in this procedure, prior to unsupervised performance of the procedure. Annually thereafter, each analyst must perform a **continuing demonstration of capability (CDOC)**, which, in this case, is a repeat of the IDOC. The DOC samples are processed in the same manner as routine samples and evaluated according to SOP TL-CA-092: *Evaluation of DOCs.*

12.2 The procedures for determining the MDL are described in SOP TL-CA-090: *Determination of the Method Detection Limit (MDL).* The (MDL) must be determined initially, whenever conditions change that might affect the instrument sensitivity, or every three years, in each matrix of concern.

12.3 The MDL must be verified annually by analysis of an MDL verification standard (MDLV) at a concentration of 1-2 times the calculated MDL. The MDLV must undergo all the same preparatory steps as the samples. The acceptance criterion for the MDLV is that it must be detected without manual integration.

12.4 The analytical batch consists of twenty or fewer samples of a similar matrix that are processed together along with the quality control (QC) items. The QC items consist of a method blank, lab control spike and duplicate (LCS and LCSD) and matrix spike and duplicate (MS and MSD). The LIMS prep batch will be noted when insufficient sample is provided for matrix spikes, and an NCM generated in accordance with SOP TL-CA-085: *Nonconformance and Corrective Action Procedures.* Batch requirement information is summarized in a table in Appendix A. The QC is evaluated in accordance with SOP TL-AN-002: *Analytical Batching and Evaluation of QC Data.*These criteria are summarized in the SOP summary included in Appendix C. Calculation of QC data is also given in SOP TL-AN-002.

12.5 Re-extraction and/or reanalysis of the samples in the batch will not be performed under the following conditions:

1) When less than five compounds are spiked, as in phthalic acids and maleic acids, all target compounds must be within the control limits.

2) The recovery of the spike exceeds the upper control limit and the compound is not detected in any sample in the analytical batch.

12.6 Corrective actions for out-of-control data are summarized in the Quality Control Checks table in Appendix C. This table describes checks, acceptance criteria and the recommended corrective action for various QC activities such as initial and continuing calibration, method blank, spikes, reporting limits, MDLs and IDOCs.

12.7 When data is out-of-control or does not meet the acceptance criteria, a nonconformance memo (NCM) must be generated and the Project Manager must be notified. In some cases an anomaly may be the appropriate course of action.

13.0 PREVENTATIVE MAINTENANCE AND TROUBLESHOOTING

13.1 Instrument Preventative Maintenance

13.1.1 HPLC systems are subject to pressure buildup due to particulate matter in the mobile phase or sample extracts. If this is indicated by recurring pressure increases, all solvents, water and extracts should be filtered using all-glass systems and nylon 0.45µm membrane filters.

13.1.2 The guard column should be evaluated and changed as pressure increases. The frequency of changing the guard column will depend on the nature of the samples analyzed.

13.1.3 Routine pump maintenance includes inspection and/or replacement of seals every six months or more frequently if problems occur. The entire system should be visually leak-checked daily following start up and equilibration. Spare fittings and valves should be kept on hand for quick repairs.

13.1.4 The column should be stored in an appropriate mobile phase with the ends capped when not in use.

13.1.5 Routine detector maintenance includes leak-checking the fittings. Any other maintenance (i.e., lamp replacement) would be performed only if indicated by a problem such as loss of response or sudden increase in noise. Detector lamp replacement is performed when indicated by a significant decrease in signal to noise ratio.

13.2 Troubleshooting

13.2.1 Retention time shifts are frequently caused by plugged mobile phase inlet frits, air in pump head check valves or incomplete or inconsistent equilibration of the mobile phase in between injections in a gradient analysis.

13.2.2 Baseline noise can be the result of air in pump head check valves or poor detector lamp energy.

13.2.3 Peak broadening can be the result of a dirty guard column or an analytical column nearing the end of its useful life.

14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention." All excess samples, sample leachates, and reagents must be disposed in accordance with SOP TL-CA-070: Waste Management.

14.1 WASTE STREAMS PRODUCED BY THE METHOD

The following table lists waste streams generated by this method:

WASTE STREAMS PRODUCED BY THE METHOD	
Waste Stream Description	**Category (Preferred Treatment)**
Waste flammable solvent (acetonitrile, methanol, hexane)	RCRA Hazardous Waste (Waste flammable solvent is accumulated in a labelled satellite accumulation container under a hood, then added to the drum marked "Flammable Solvent" in the waste management area.)
Acidic waste	RCRA Hazardous Waste (Excess acidic waste or reagents are neutralized, then discharged to the sewer system (down the drain with water running).)
Buffered mobile phase and water from HPLC	Non-RCRA Hazardous Waste (Mixture is collected in 4L waste containers and then poured down the sink and flushed with a large quantity of water.)
Autosampler Vials containing eluting solution	RCRA Hazardous Waste (Employees collect vials and dispose of them in the main hazardous waste drum for vials in the back of the lab.)

15.0 REFERENCES

15.1 TL-QAM: *TestAmerica Tallahassee Quality Assurance Manual* (QAM), current revision.

16.0 TABLES, DIAGRAMS, AND FLOWCHARTS

16.1 Appendix A

PHTHALIC ACID AND MALEIC ACID METHOD SUMMARY

HOLD/STORAGE

Parameter	Aqueous	Soil/Solid
Routine Container	125mL amber glass with Teflon-lined cap	125mL amber glass with Teflon-lined cap
Preservative	None	None
Hold time	7 days from date of collection and 40 days from date of extraction	14 days from date of collection and 40 days from date of extraction
Storage	4°C (less than 6°C with no frozen samples) from arrival to extraction and analysis	4°C (less than 6°C with no frozen samples) from arrival to extraction and analysis

EXTRACTION

Aqueous: A known volume of liquid sample, nominally 50mL, is adjusted to a pH range of three to ten. The sample passes through C8 and amino solid phase extraction cartridges that have been pre-washed with the phthalic acid and maleic acid eluting solution and HPLC water. The analytes are eluted from the cartridges with eluting solution and analyzed.

Soils: Soil samples are leached with water for 30 minutes. The resulting leachate is then subjected to the same preparation steps and analytical procedures as aqueous samples.

QC REQUIREMENTS

BATCH QC
Method blank
LCS/LCSD
MS/MSD (frequency of 5% of samples)

SURROGATES
There are currently no surrogates available for the phthalic acid and maleic acid method.

ANALYSIS
Analysis is done by HPLC with UV detection; external standard calibration is used.

SEQUENCE

STANDARD/SAMPLES
Initial Calibration
Initial Calibration Verification (ICV)
Ten sample analyses (field samples, excluding QC*)
Continuing Calibration Verification (CCV)
RL standard (daily if required)
Ten sample analyses
Continuing Calibration Verification (CCV) – sequence must end with a CCV

Sequence continues until all samples have been analyzed or the CCV fails the acceptance criteria. The sequence must end with an acceptable CCV.

* Matrix spikes are considered QC samples, not field samples.

EVALUATION

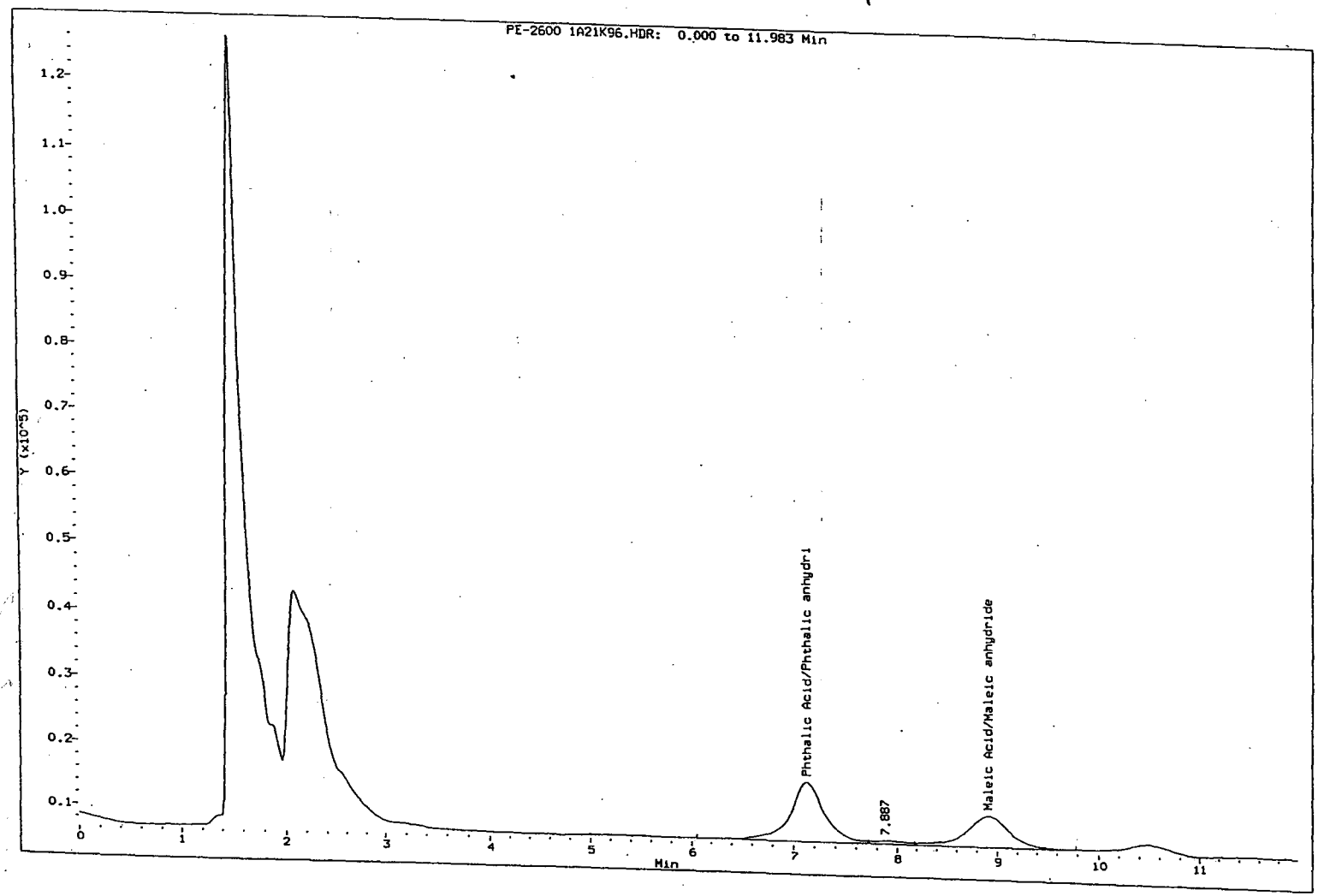
PEAK INFORMATION	ACTION	REPORT
No peaks found on primary or secondary column		Report < RL
Peak found within RTW on primary column	Peak is tentatively identified as the target	If concentration < RL, report < RL If concentration > RL, evaluate confirmation column.
Peak found within RTW on confirmation column	Peak is confirmed as the target. NOTE: If, in the professional judgment of the analyst and supervisor, a peak within the retention time window can be reasonably excluded as the target compound, the result may be reported as a "non detect".	If concentration < RL, report <RL If concentration >RL, calculate %RPD -if %RPD <40%, report higher concentration of primary and confirmation analyses. -if %RPD >40%, flag result to note the disparity. The default procedure is to report the lowest when %RPD>40%.. Case narrative or note to PM may be required for complex matrices.

16.2 Appendix B

16.2.1 **PRIMARY COLUMN EXAMPLE CHROMATOGRAM**

Data File: \\TALLCO1\chem\LC\TLCKUV1.i\1KA217.b\1A21K96.d/1A21K96.HDR
Injection Date: 22-JAN-2007 12:20
Instrument: TLCKUV1.i
Client Sample ID: M2.4-12-2-2

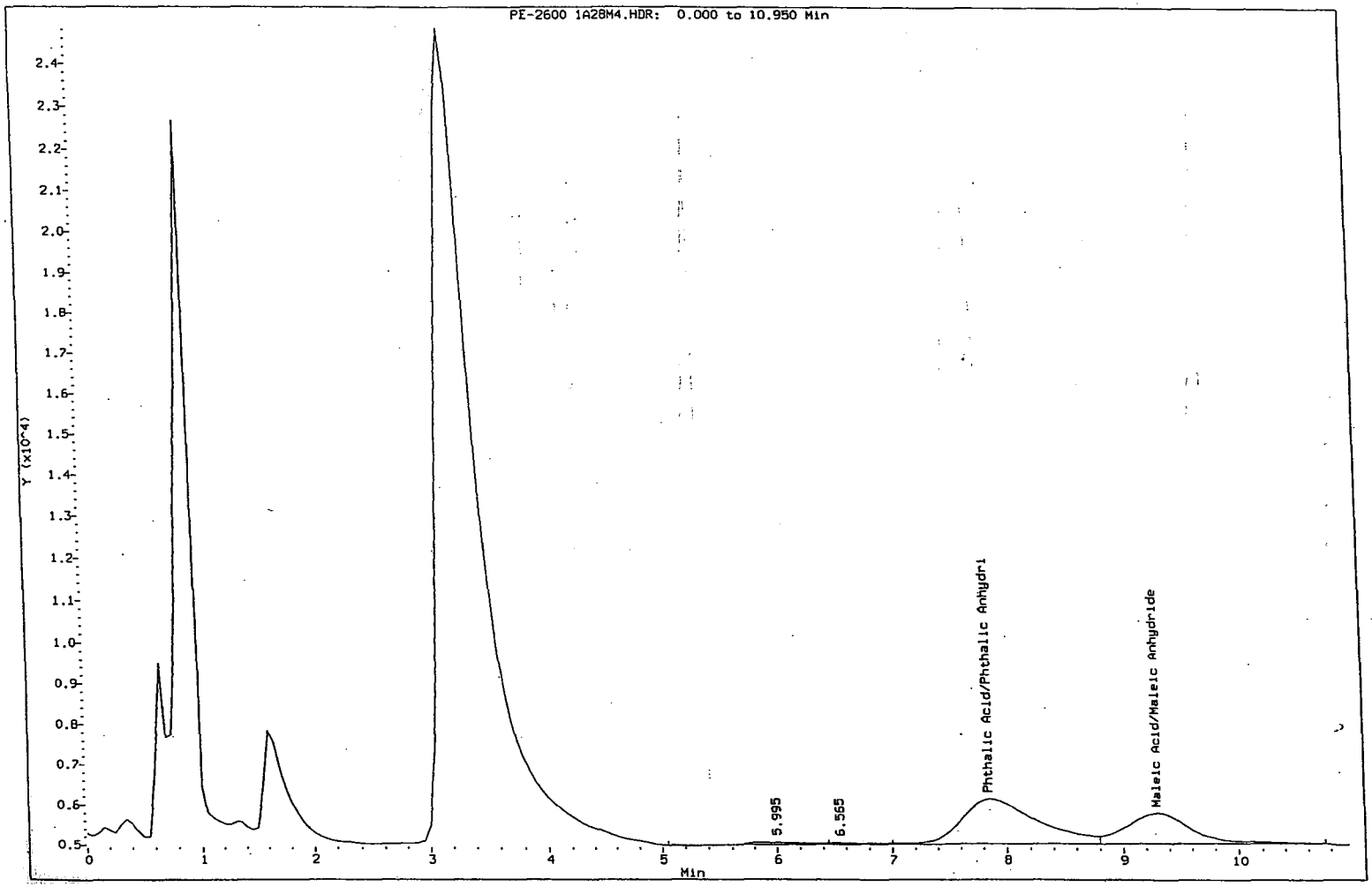
Primary



16.2.2 CONFIRMATION COLUMN EXAMPLE CHROMATOGRAM

Data File: \\TALLC02\chem\LC\TLCMUV1.i\1MA2B7.b\1A28M4.d/1A28M4.HDR
Injection Date: 28-JAN-2007 10:44
Instrument: TLCMUV1.i
Client Sample ID: M2.4-12-3-2

Confirmation



16.3 Appendix C
QUALITY CONTROL CHECKS

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration -- lowest point at or near RL -- other points define working range	Prior to sample analysis or when CCV fails	Minimum 3 points 1) Plot regression curve (CC>=0.990) OR 2) %RSD of each target <= 10%	- Evaluate chromatogram and integrations. - Check calculations. - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument or column maintenance and reanalyze standards
Initial calibration verification (ICV)	Immediately following initial calibration	Percent difference or drift <= 20%	- Evaluate chromatogram and integrations. - Check calculations. - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument or column maintenance and reanalyze standards
Continuing calibration verification (CCV)	Every ten sample shots and at the end of the sequence	Percent difference or drift <= 20%	- Evaluate chromatogram and integrations. - Check calculations. - Reanalyze standard(s) - Remake and reanalyze standard(s) - Perform instrument or column maintenance and reanalyze standards
Method blank	Per batch	All targets reported less than RL in LIMS	- Evaluate chromatogram and integrations. - Check calculations. - Reanalyze - Follow guidance in SOP TL-AN-002 - Perform instrument or column maintenance, recalibrate and reanalyze

QC CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Lab control sample (LCS) / Lab control sample duplicate (LCSD)	Per batch	Recoveries within LIMS limits	- Evaluate chromatogram and integrations. -Check calculations. - Reanalyze - Follow guidance in SOP TL-AN-002 - Perform instrument or column maintenance, recalibrate and reanalyze
Matrix spike (MS) and matrix spike duplicate (MSD)	5% of samples	Recoveries within LIMS limits	- Evaluate chromatogram and integrations. Check calculations. - Reanalyze - Follow guidance in SOP TL-AN-002 - Perform instrument or column maintenance, recalibrate and reanalyze
Reporting limit (RL) standard -- lowest level calibration standard	Daily – required for Florida DEP	Detected with reasonable sensitivity	- Reanalyze RL standard - Remake and reanalyze RL standard - Perform instrument or column maintenance, recalibrate and reanalyze associated samples
Initial demonstration of Capability	Per analyst or work group	Within the phthalic acid and maleic acid method limits	- Reanalyze QC sample for the targets that failed to meet the criteria
Method detection limit (MDL)	See TL-CA-090	Evaluate according to SOP TL-CA-090	Evaluate according to SOP TL-CA-090
Method detection limit verification (MDLV)	Immediately following MDL and annually	Evaluate according to SOP TL-CA-090	Evaluate according to SOP TL-CA-090

17.0 REVISION HISTORY

Revision 4, 6/22/2007:

- Section 8.3.1 was changed to reflect stock standard preparation in SPE cartridge eluting solution instead of methanol.
- All references to Severn Trent Laboratories or STL in the header, logo, copyright notice, and body of the SOP have been replaced with TestAmerica or TA.

Revision 5: 6/13/2008

- Incorporated new cover page, logo, and naming conventions
- Revised all SOP references to new naming convention
- Corrected name of CSM (added "Environmental Health and...")
- Deleted all references to LQM, added references to TL-QAM, TL-QA-001, and TALS LIMS
- 8.3.1: Revised procedure to reflect maleic acid stock prepared in eluting solution, phthalic acid stock prepared in methanol
- 9.1.7: Revised to separatory funnel back-extraction
- 10.1.3.5: Added last sentence – "Alternatively, default RT windows..."
- 10.4.3: Changed "30 minutes to one hour" to "at least 30 minutes"
- 12.3: Added MDLV requirements
- 12.6: Deleted reference to LQM
- 16.1 SEQUENCE: Added ICV
- 16.3 QC CHECKS: Added ICV and MDLV

Determination of Perchlorate in Aqueous Samples and Soil Samples by LC/MS/MS

Approvals:

Prepared by: _____ Date: 3/26/08
Principal Chemist

Approved by: _____ Date: 3/26/08
Pesticide Residue Analysis Management

Approved by: _____ Date: 3/31/08
Quality Assurance

Revision Log:

Revision: 03		Effective Date:	This version
Section	**Justification**		**Changes**
Approvals	Formatting requirements per LOM-SOP-LAB-201		Moved signatures to beginning of document
Revision Log	Formatting requirements per LOM-SOP-LAB-201		Moved to beginning of document and reformatted to include Justification
Document	Formatting requirements per LOM-SOP-LAB-201		Placed sections in order
Reference	New method supplied by EPA		Add method 6850,
Basic Principles	Not performed		Delete post extraction matrix spike requirements,
Sample Collection, Preservation, and Storage	Formatting Requirements & new procedure		Renamed section "Sample Handling"; sample bottle changed per new method as well as hold times
Apparatus and Equipment	New equipment		Updated all sections to reference Agilent not Waters instrument,
Reagents and Standards	New method		Rewrote most of preparation steps,
Standards Database Entry	Not needed		Deleted
Procedure for Sample Preparation	New procedure		Rewrote most of section; reorganized into standard format and re-identified as subsection A. under Procedure section
Procedure for LC/MS/MS Analysis	New instrument		Rewrote most of section including instrument conditions, tuning criteria, initial and continuing calibration, sample analysis; reorganized into standard format and re-identified as subsection B. under Procedure section

Ver. #	Effective Date	Change
02	03/14/06	Major changes as follows: • Updated stds prep section • Updated CCAL criteria • Updated ICAL set up • Incorporated PAs
01	09/22/04	New

63866557_03.DOC
032408

Reference:

1. *Test Methods for Evaluating Solid Waste*, EPA SW846, Method 6850, January 2007.

2. *Chemical Hygiene Plan*, Lancaster Laboratories, current version.

Cross Reference:

Document	Document Title
Analysis #6568	Extraction Procedure for Perchlorate in Soil Samples by LC/MS/MS
LOM-SOP-ES-203	Determining Method Detection Limits and Limits of Quantitation
SOP-PP-002	QC Data Acceptability and Corrective Action
SOP-PP-025	Monitoring of QC Data Acceptance Limits
SOP-PP-040	Common Equations Used During Chromatographic Analyses

Scope:

The method is applicable for the determination of trace levels of the following perchlorate in water and soil:

Analyte	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/Kg)
Perchlorate	1	5*

*Based on MDL study in sand. May be adjusted/raised if matrix issues are encountered during sample preparation

Basic Principles:

Water samples are filtered through a 0.45-µm syringe filter, followed by direct injection into a LC/MS/MS system followed by multiple reaction monitoring. Soil samples are extracted with water, followed by filtering through a 0.45-µm syringe filter and LC/MS/MS analysis. Quantitation is performed using an isotopic internal standard.

Interferences:

Compounds which have similar structures to the compounds of interest, and similar molecular weights would potentially interfere. Due to the use of multiple reaction monitoring (MRM) this is not likely.

Matrix effects, such as interferences, are possible due to the presence of hardness ions in the samples. In an effort to minimize this effect, a clean up procedure using two barium and one hydrogen OnGuard cartridge may be used. Sample dilutions may also be performed to minimize such matrix effects.

Method interferences may be caused by impurities in solvents, reagents, glassware or other hardware used in sample processing. All glassware is rinsed with solvent before used and a method blank is performed with each batch of samples to demonstrate that the system is free of contaminants. The MS/MS detector is a very selective detector which minimizes the chance for false positives.

Safety Precautions and Waste Handling:

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Gloves, lab coats, and safety glasses should be worn when preparing standards, handling samples and operating the LC/MS/MS. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact, and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All solvent waste is collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal in to the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed.

Personnel Training and Qualifications:

Analysts performing the instrumental analysis will work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set-up sequences, perform calculations, interpret the chromatograms and mass spec data, and enter the data into the LIMS. Proficiency is measured through documented audits of the tasks listed and overchecking of data, as well as annual quad studies.

Sample Handling:

Samples may be collected in glass or plastic unpreserved containers. Collect waters in clean 125 ml polyethylene bottles. There should be headspace. Samples should be stored at 2° to 4°C and must be injected within 28 days of sampling. Soil samples should be collected in 4-oz amber wide mouth jars with Teflon lined lids. Soil samples should be homogenized prior to analysis. Soil samples should be stored at 2° to 4°C and must be extracted within 28 days of sampling. Extracts must be analyzed within 28 days of extraction.

Apparatus and Equipment:

1. HPLC with dual high-pressure pump and gradient capabilities – Agilent or equivalent
2. HPLC autosampler – Agilent or equivalent
3. HPLC oven – Agilent or equivalent
4. LC/MS/MS equipped with electrospray ionization source
5. Column: Dionex IonPac AG16 Guard 2 x 50mm
6. Data System: Agilent Masshunter for data acquisition, integration, and spectra or equivalent

7. Filters: 0.45-µm Teflon filters

8. Glass pipettes, various sizes for standard preparation and dilutions

9. Volumetric flasks, Class A, various sizes for standard preparation and sample dilutions

10. 1-liter bottles for mobile phase storage

11. Balance – Analytical, capable of accurately weighing 0.0001 g

12. HPLC autosampler vials

13. Barium and Hydrogen Cartridges –

    Dionex OnGuard – Ba Part #046072, or equivalent

    Dionex OnGuard – H Part #039596, or equivalent.

Reagents and Standards:

1. Ammonium Hydroxide 30% – Baker Analyzed

2. Deionized, Milli-Q or bottled HPLC grade water

3. Acetonitrile, Fisher Optima or equivalent

4. 100 mm Ammonium Hydroxide Solution: 11 mL of ammonium hydroxide to 500 mL of deionized water and dilute to 1 liter in volumetric flask. Store at room temperature. Buffer is good for 3 months (measured as 90 days).

5. 2 mm Ammonium Hydroxide mobile phase. Prepare by adding 20 mL of 100 mm Ammonium Hydroxide to 500 mL Milli-Q and 500 mL Acetonitrile. Mobile phase is good for one month (measured as 30 days).

6. Stock Standards: 1000 ppm commercially prepared standards can be purchased from Ultra Scientific or GFS Chemicals. The internal standard can be purchased from Isotec, Inc. as a salt in 100-mg vials. The internal standard is custom synthesized. Commercially prepared stock standards expire after 2 years. The isotopic salt should be re-evaluated annually for signs of degradation. Intermediate solutions and working standards expire in 1 year.

7. Working Standards: Working standards are prepared fresh annually.

Standard ID	Stock	Initial Vol. mL	Final Vol. mL	Solvent	Concentration (µg/L)
Intermediate	ClO4	0.1	100	HPLC water	1000
Level 7	Intermed	2.5	100	HPLC water	25
Level 6	Intermed	1	100	HPLC water	10
Level 5	Intermed	0.4	100	HPLC water	4
Level 4	Intermed	0.2	100	HPLC water	2
Level 3	Intermed	0.1	100	HPLC water	1
Level 2	Intermed	0.04	100	HPLC water	0.4
Level 1	Intermed	0.02	100	HPLC water	0.2
ICV/Spike Interm	ClO4	0.1	100	HPLC water	1000
ICV/CCV	ICVInterm	0.4	100	HPLC water	4
ICV/CCV low	ICVInterm	0.04	100	HPLC water	0.4
IS stock	NaClO4	0.1g	100	HPLC water	1000000
IS spike	IS stock	0.1	100	HPLC water	1000

Procedure:

A. Sample Preparation

NOTE: For both soil and water preparations, routine QC per SW846 methods is performed (method blank, laboratory control spike/spike duplicate and matrix spike/matrix spike duplicate). Alternatively, a matrix spike per sample can be performed to evaluate matrix affects on an individual sample basis.

1. Water preparation

   a. Dispense 10.0 mL of sample or standard into a 15 mL disposable centrifuge tube.

   b. Add 100 uL of IS spiking solution to the sample tube, cap and shake until mixed well.

      NOTE: The final concentration of IS in the sample must be exactly the same as that in the calibration standards. Proportionally smaller volumes of sample and IS may be used to reduce laboratory waste or when limited sample volume is supplied to the laboratory.

   c. Filter the sample solution using a plastic syringe fitted with a 0.45-um or 0.20-um PTFE membrane filter. Dispense the sample into an autosampler vial for analysis.

      NOTE: Filtration may be omitted for calibration standards and for samples that have been previously filtered in the field.

   d. Prepare a blank by adding 100 µL of internal standard solution to 10 mL Milli-Q.

e. Prepare a Laboratory control sample by adding 0.05 mL of 1000 ppb standard solution and 0.1 mL of Internal Standard solution to a final volume of 10 mL.

f. Prepare internal standard solution spiked calibration standards by spiking 10 mL of each standard with 0.1 mL of internal standard solution.

2. Soil preparation – see Analysis #6568.

B. LC/MS/MS Analysis

Tuning and calibration for the LC/MS/MS: Refer to the instrument manufacturers instructions for tuning and conditions. These values are stored in the tune file for future reference, and may not need to be changed unless loss of response is noted.

1. Chromatographic conditions

Below are the recommended chromatographic conditions for the reversed-phase separation. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process:

Column	Dionex IonPac AG16 Guard 2 x 50mm
Gradient	50% ACN/50% 2mm ammonium hydroxide, 35°C
Flow	0.3 mL/minute
Injection size	50 µL

2. Detector conditions, Agilent LC/MS/MS MRM mode

NOTE: Conditions listed are guidelines

Mode: ES-	Source Temperature (C): 100
Multiplier (V): 650	Desolvation Temperature (C): 350
Capillary (V): 4000	Nebulizer Flow (PSI): 30
	Desolvation Gas Flow (L/min): 10

3. Aquisition method

Ion Mode: ES-

Inter Channel Delay (sec): 0.1

Ch	Precursor	Product	Dwell(s)	Fragmentor	Coll Energy	Delays
1($^{35}ClO_4$)	99.0	83.0	0.1	150	25	.03
2($^{37}ClO_4$)	101.0	85.0	0.1	150	25	.03
3($Cl^{18}O_4$)	107.0	89.0	0.1	150	25	.03

4. Calibration

Inject calibration levels 1 – 7 after allowing the system to equilibrate at least 1 hour, and injection of 3 conditioning standards. (At least 5 points must be used to construct the calibration curve per SW846 methods.) Prior to running samples, check linearity. As a first choice, select a linear fit, with ignore the origin. If the correlation coefficient is above 0.995, the curve is acceptable. If a non-linear curve is observed, select a quadratic fit, with origin ignored. At least 6 points must be added to the curve to use quadratic. If the correlation coefficient exceeds 0.99, the curve is acceptable. If neither fit type is acceptable, check the integration and correct as necessary. Recalibrate if none of these correct the issue. If a second calibration does not meet specifications, remake standards, or perform maintenance as needed.

After the calibration standards have been analyzed, the accuracy of the calibration must be verified by the analysis of an initial calibration verification (ICV) standard. The ICV is prepared at a concentration level within the calibration range and using a second source standard. The control limit for the ICV is ±15% of the true value. When the ICV exceeds the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

5. Continuing calibration

Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. The CCVs are prepared from a second source at levels within the calibration range of the method. CCV concentrations alternating between the low- and mid-range of the calibration are recommended. The control limit for the low-range CCV is ±50% and for the mid-range CCV is ±15% of the true value. When the CCV exceeds the control limits, the analysis should be terminated and the problem corrected. Samples that are not bracketed by acceptable CCV runs must be reanalyzed. If the CCV recoveries are running high indicating increased sensitivity, and the associated samples are clean, the data may be reported with a comment.

6. Sample analysis

After the initial calibration, inject the blank, followed by the LCS, then samples. Bracket each set of ten samples with a mid-level calibration check. If dilutions are necessary, it is best to inject those prior to the undiluted samples. Note that a peak smoothing value of 5 scans/6 smooths should be used for integration.

After injections are completed, check continuing calibration recoveries to make sure they are acceptable. Process each chromatogram and closely evaluate all integration, baseline anomalies, and retention time differences.

Quantitate results for the blank. No perchlorate above the reporting limit may be found in the blank for acceptable batch results. Calculate the recoveries for the LCS by comparing concentrations observed to the true values. The advisory acceptance limits for LCS are 80 to 120% for each component. Statistical limits must be calculated when sufficient data points have been collected. These should be updated twice per year. If the LCS is acceptable, proceed to sample quantitation.

Matrix spike method control limits are 80 to 120% for water matrices and 70 to 130% for solid matrices. If matrix spike recoveries are outside the control limits and all other QC data is within limits a matrix effect is suspected. The associated data should be flagged or noted in the comments section of the report.

The method control limit for duplicates or matrix spike duplicates is 15% for samples concentrations near the mid-range of the calibration and 50% for sample concentrations near the low-range of the calibration curve.

If the peak area response exceeds the calibration range of the system, dilute a fresh aliquot of the sample and reanalyze.

The internal standard area counts for each sample and QC standard must be monitored and compared against the average of the internal standard area counts of the calibration standards. If the internal standard area counts exceeds ±50%, the data should be considered suspect. The system should be checked for corrective action needed. Reanalysis can take place. If there is significant internal standard suppression a matrix effect is indicated and sample dilution should be performed.

Compare the retention times of the m/z83 peak and the m/z89 peak. The retention times should not vary by more than 0.2 minutes.

Evaluate the relative abundances of the m/z83 and m/z85 ions in the chromatogram. To confirm the presence of perchlorate, the 83/85 peak area counts should be within ±30% of the average peak area count of the mid-range standard.

Calculations:

An internal standard calibration curve is established using peak area. The concentrations are entered into the calibration table in µg/L. The data system uses the following equation to calculate the concentration in the extract:

Single component compounds:

1. Using the average calibration factor (AVGCF) from the initial calibration:

$$\frac{(Sample\ Area/Internal\ std.\ Area)}{AVGCF} \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

2. Using linear curve from the initial calibration:

$$\left[\left(\frac{Sample\ Area}{Internal\ std.\ Area} - Y - intercept\right) / Slope\right] \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

Where:

FV = final volume

IV = initial volume

DF = dilution factor

See SOP-PP-040 for details on all calculations/equations used to evaluate the initial and continuing calibration.

Statistical Information and Method Performance:

Reporting limits including method detection limits (MDL) and limits of quantitation (LOQ) are generated according to LOM-SOP-ES-203. MDLs are determined by taking seven spiked replicates through the entire extraction and analysis procedure. These are run on each instrument used for the analysis. The results are tabulated using an Excel spreadsheet. Results from all instruments are compared and pooled together to determine the reporting MDL. Copies of the annual studies are maintained by the department supervisor. Updates to the LIMS are made as needed by the QA department and only as directed by the supervisor. The department database is updated via a download from the LIMS. Each analyst has hard copies of the current reporting limits for each analysis.

Quality Assurance/Quality Control:

A reagent blank must be extracted with every batch of samples, not to exceed 20. A laboratory control sample (a spiked reagent blank), a matrix spike, and a matrix spike duplicate must be extracted for every batch, not to exceed 20 samples. If sample volume is not available to perform a MS/MSD, a laboratory control spike duplicate will be prepared to demonstrate precision and accuracy for the batch. See SOP-PP-002 for QC acceptance criteria and SOP-PP-025 for monitoring QC limits.

Title: THE ANALYSIS OF HEXAVALENT CHROMIUM IN AQUEOUS SAMPLES AND DIGESTATES FROM SOLID SAMPLES VIA ION CHROMATOGRAPHY [SW846 7199]

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1.0 Scope and Application

1.1. SW846 Method 7199 may be used to determine the hexavalent chromium content in aqueous samples or digestates of solid samples. Solid samples must be first digested according to TestAmerica Edison SOP No. ED-WET-010 (The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium - USEPA SW846 Method 3060A).

1.2. This method should be used by analysts experienced in the use of ion chromatography and in the interpretation of ion chromatograms

1.3. The reporting limit is 2.0 mg/kg for solid samples and 1.0 ug/L for aqueous samples.

1.4. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

An aqueous sample or a soil digestate is filtered through a 0.45um filter; pH is adjusted to 9.0-9.5 using the buffer solution. A measured volume of the sample is introduced to the ion chromatograph. A guard column removes organics from the sample before the Cr(VI) as CrO_4^{2-} is separated on an anion exchange separator column. Post-column derivatization of the Cr(VI) with Diphenylcarbazide is followed by detection of the colored complex at 530nm.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Contamination – A trace amount of Cr is sometimes found in reagent grade salts. Since a concentrated buffer solution is used for pH adjustments of samples, reagent blanks must be analyzed to assess for potential Cr contamination. Improperly cleaned glassware or contact of caustic/acidic reagents or samples with stainless steel or pigmented material may also cause contamination.

4.2. Reduction of Cr(VI) to Cr(III) can occur in the presence of reducing agents in an acidic medium of pH <6.5. However, at a pH of 6.5 or greater, CrO_4^{2-} which is less reactive than $HCrO_4^-$ is the predominant species.

4.3. Overloading of the analytical column capacity with high concentrations of chloride and sulfates, will cause a loss of Cr (VI). The column specified in this method can handle samples containing up to 5% sodium sulfate or 2% sodium chloride. Poor recoveries from fortified samples and tailing peaks are typical display of column overload.

4.4. For IC analysis, only compounds that co-elute with Cr (VI) and affect the Diphenylcarbazide reaction will cause interference. Periodic analysis of DI water blanks is used to demonstrate that the analytical system is essentially free from contamination. Ensure that all samples are covered at all times to minimize exposure to air.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document. Eye protection, lab coat and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. All questions pertaining to any safety procedure should be brought to the department manager or TestAmerica EH & S Coordinator.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit	Signs and symptoms of exposure

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if it comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Ammonium Hydroxide	Corrosive Poison	50 ppm – TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm – TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methanol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Ion Chromatograph system – Metrohm MIC – 2/830 : equipped with high performance liquid chromatograph pump, sample injection valve, post-column reagent delivery and a visible lamp detector containing no metal parts in contact with the flow path, capable of operating at 520 – 530 nm.

- Recorder, integrator /computer for receiving analog or digital signals for recording detector response (peak height or area) as a function of time.

6.2. Supplies

- IC column : Guard columns placed before the separator column containing a sorbent capable of removing strongly absorbing organics and particles that would otherwise damage the separator column (METROSEP RP / Standard C18).

- A separator or analytical column packed with a high capacity anion exchange resin capable of resolving CrO_4^{2-} from other sample constituents (METROSEP A Supp 5 150/4.0)

- Helium gas supply (High purity, 99.9955)

- Calibrated pipettes: Eppendorf or equivalent.

- Class A volumetric flasks

- Graduated cylinders and storage bottles

- 10 ml disposable syringes and 0.45 um syringe filters.

- Calibrated pH meter

- Sonicator – Fisher scientific FS60

7.0. Reagents and Standards

7.1. Reagent Water - DI water with a specific conductivity less than 1.0 uohms/cm at 25 °C.

7.2. The following Reagent grade chemicals are used for all solutions required in this test. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available. All reagents are stored at room temperature:

7.2.1. Ammonium Hydroxide, NH_4OH (sp.gr.0.902)
7.2.2. Ammonium Sulfate, $(NH_4)_2SO4$
7.2.3. 1,5-Diphenylcarbazide
7.2.4. Methanol, HPLC grade
7.2.5. Sulfuric Acid, concentrated (sp.gr. 1.84).

7.3. Primary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.4. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of 1000 mg/L Cr+6 primary stock standard to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.5. Hexavalent Chromium Primary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/LCr+6 primary stock standard to a 100ml volumetric flask and

dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.6. Hexavalent Chromium Spiking Solution (1.0 mg/L Cr+6) - Prepare by adding 10.0ml of 10 mg/L Cr+6 primary spiking solution (see section 7.5) to a 100ml volumetric flask and dilute to volume with dilution water. Prepare fresh with the calibration standards. Store at room temperature.

7.7. Secondary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). *The source of this standard must be from a different lot or manufacturer than the Primary Potassium Dichromate Stock Standard detailed in Section 7.3*. Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.8. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of 1000 mg/L Cr+6 Secondary Standard (see section 7.7) to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.9. Hexavalent Chromium Secondary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Secondary Standard (see section 7.7) to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.10. Laboratory control sample, LCS soluble (soil and water) - obtained from an independent source. Dilute an aliquot according to the instructions and analyze with samples. For storage and stability information, refer to manufacturer's instructions.

7.11. Laboratory Control sample, LCS insoluble (soil) - A 10-20 mg of $PbCrO_4$ is added to a separate sample aliquot and is analyze in the same manner as the samples. For storage and stability information, refer to manufacturer's instructions.

7.12. Eluent - Dissolve 33 g of ammonium sulfate in 500 ml of DI water and add 6.5 ml of ammonium hydroxide. Dilute to 1L with DI water. Using a sonicator degas the solution prior to use. Reagent is stable for 1 week. Store at room temperature.

7.13. Post-column reagent - Dissolve 0.5 g of 1,5 Diphenylcarbazide in a beaker containing 100 ml of HPLC grade methanol. In a 1 L volumetric flask, add 28 ml of 98% sulfuric acid into 500 ml DI water, mix, and add the 1,5 Diphenylcarbazide solution. Dilute to 1000 ml with DI water. Using a sonicator degas the solution for approximately 30 minutes. Reagent is stable for 4-5 days. Store at room temperature.

7.14. Buffer solution - Dissolve 33 g of ammonium sulfate in 75 ml of DI water and add 6.5 ml of ammonium hydroxide, dilute to 100ml with DI water. Reagent is stable for 6 months. Store at room temperature.

7.15. Dilution water - A batch of DI water must be prepared by adjusting the pH within the range of 9-9.5 using the buffer solution. Use this solution for diluting working

standards and high level samples. Reagent is stable for 1 week. Store at room temperature.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters[3]	HDPE[2]	250 mL	Cool 4 ± 2°C	24 Hours	SW 7199
Soils	Glass/plastic	25 grams	Cool 4 ± 2°C	30 Days[4]	SW 3060A

[1] Inclusive of digestion and analysis

[2] High-density polypropylene container

[3] For determination of dissolved Cr(VI), the sample should be filtered through a 0.45-um filter. Adjust the pH of the sample to 9-9.5 by dropwise addition of buffer solution, checking the pH with the pH meter.

[4] Digestates from SW-846 3060A must be analyzed within 168 hours from time of digestion. Store samples at 4°C before analysis or digestion.

9.0. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1. Method/Preparation Blank: For every QC batch, prepare a Method Blank that consists of a reagent water, pH adjusted to 9-9.5 with the same volume of buffer as used for the sample and is carried through the entire analytical procedure. This will identify any system / process contamination. **For soil samples, a digestion solution is used for Method Blank.**

9.1.2. Laboratory Control Sample (LCS) – *Water Samples*: A soluble LCS (see Section 7.10) is prepared and analyzed with each QC batch or with every 20 samples, whichever is more frequent. *Soil Samples*: An LCS soluble (see Section 7.10) and an LCS insoluble (see Section 7.11) is prepared and analyzed with each QC batch or with every 20 samples, whichever is more frequent. The LCS must be prepared from a source independent of the standards used in the calibration curve (i.e., different manufacturer or separate lot). The LCS is used to monitor the accuracy of the entire analytical process. The recovery for the LCS soluble must be within the certified accepted range. The LCS insoluble must be recovered within 80% to 120% of the true value. If the result deviates from the true value, a second analysis should be performed. If it is still outside the limits,

terminate the analysis until the source of problem is identified and corrected.

9.1.3. Duplicates: For every QC batch of a similar matrix, a duplicate will be prepared and analyzed in exactly the same manner as the original sample. The relative percent difference (RPD) between the sample and duplicate should be =20%.

9.1.4. Predigestion Matrix Spike: For each *water* QC batch: a predigestion matrix spike (Concentration = 40 ug/L) is prepared by adding 2 ml of 1mg/L Cr6+ primary standard (see Section 7.6) into 50 ml of sample. Spike recoveries should be between 85-115% of the true value. For every *solid* QC batch, both a predigestion matrix spike soluble and a predigestion matrix spike insoluble are prepared as described in TestAmerica Edison SOP No. ED-WET-010 (The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium - USEPA SW846 Method 3060A). The percent recovery for each should be between 75-125%. If the soil predigestion matrix spike recoveries are not within the acceptance limits, the entire batch must be rehomogenized, redigested and reanalyzed. (NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance).

If upon reanalysis, the matrix spikes are not within the recovery limits of 75%-125%, but the LCS is within the criteria specified in Section 9.1.2, the data are reported and the spike recoveries are detailed in the Non-Conformance Summary. Additional laboratory characterization of each sample in the batch for Oxidation Reduction Potential (see TestAmerica Edison SOP No. ED-WET-066, Redox by SM2580) and pH (see TestAmerica SOP No. ED-WET-061, pH Soil by SW846 9045C) is required to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), or Biochemical Oxygen Demand (BOD) may be required on a case by case basis at the direction of the Wet Chemistry Manager or Supervisor.

9.1.5. Post Verification Spike (PVS): For every QC batch of a similar matrix, a post verification spike (PVS) will be analyzed. For *soils*, this consists of first, analyzing a sample and then spiking a separate aliquot of the same sample with two times the concentration of Cr (VI) found in the sample or 40 mg/kg whichever is greater. The PVS recovery must be within 85-115%. If the recovery is outside the acceptance limit, reanalyze using the digestate of the sample duplicate. For *water* samples, analyze a sample and then spike a separate aliquot with two times the concentration of Cr (VI) found in the sample or 5 ug/L whichever is greater. If necessary, dilute the PVS to fall within the range of the calibration curve. If the PVS recovery is not 85-115%, dilute the sample and re-analyze.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV) – The ICV (a 25.0 ug/L calibration standard) is analyzed immediately after an acceptable calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within the 10% limit of expected value. It is prepared by adding 0.25ml of a 10 mg/L Cr6+ secondary spiking solution into 100 ml volumetric flask and diluted up to 100ml dilution water. If the measured concentration exceeds the 10% limit, a second analysis should be performed. If the result still exceeded the +/- 10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.1. Continuing Calibration Verification (CCV) - To verify that the instrument is properly calibrated on a continuing basis, run a CCV (25.0 ug/L) after every ten samples. CCV is prepared the same way as the ICV (see Section 9.2.1). If the measured value exceeds by more than 10% from the true value, reanalyze the calibration check standard. If this check is still out of the 10% limit, the instrument must be recalibrated and the previous ten samples reanalyzed. The instrument response from the calibration check may be used for recalibration purposes.

9.2.2. Continuing Calibration Blank (CCB) – A Continuing Calibration Blank (CCB) must be analyzed after every 10 samples. The value of the CCB should be less than the MDL.

10.0. Procedure

10.1. Sample Preparation

10.1.1. Allow pH adjusted samples to equilibrate to room temperature prior to analysis. Samples for dissolved Cr (VI) should be filtered and pH adjusted before analysis.

10.1.2. Digestates from SW846 3060A are pH adjusted within the range of 9-9.5, using the buffer solution, then brought to a final volume of 100 ml with the dilution water. Record the final pH of the digestate solutions in the appropriate log book. If the pH drops below 9.0, discard the extract and redigest the sample.

10.1.3. In order to prevent overloading the analytical column capacity with high concentrations of sodium hydroxide and sodium bicarbonate, all soil digestates and QC samples are diluted 10x prior to initial IC analysis using the dilution water. The reporting limit is adjusted accordingly for all reported soil samples. Note: the standard soil reporting limit listed in Section 1.3 is already adjusted for this dilution.

10.2. Calibration

Prior to calibration and analysis of samples the instrument operating conditions must be set as follows:

Analytical Column	METROSEP A Supp 5 150/4.0 150 mm L X 4.0 mm ID
Sample Loop Size	100 uL
Eluent Flow	0.75 mL/min
Post Column Reagent Flow	0.75 mL/min
Detector	530 nm Visible

Everyday or before each analysis, prepare fresh calibration standards. Calibrate the instrument using a minimum of a calibration blank and four calibration standards. The coefficient of correlation should be 0.999 or greater for all curves.

Table 1: Calibration Standard Preparation: WATER

Standard Cr(VI) Concentration (ug/L)	Volume (ml) of 1.0 mg/L Cr (VI) spiking solution**	Volume (ml) of dilution water (7.15)
1.0	0.10	100
5.0	0.50	100
25	2.5	100
50	5.0	100

Table 2: Calibration Standard Preparation: SOIL

Standard Cr(VI) Concentration (ug/Kg)	Volume (ml) of 1.0 mg/L Cr (VI) spiking solution**	Volume (ml) of dilution water (7.15)
5.0	0.50	100
10	1.0	100
25	2.5	100
50	5.0	100

** Cr(VI) 1.0 mg/L - Refer to section 7.6 for the preparation procedure

Note: Dilution water is used for the calibration blank

10.3. Sample Analysis

10.3.1. Each sample should be injected in duplicate and the RPD of the duplicates should be less than 20%. If the 20% criteria are not met, document this fact in the Non-Conformance Summary.

10.3.2. For aqueous samples, make the initial injection of all the samples. Once each sample has been injected, follow with the second injection and report the higher results. Analysis that is out of the hold time must be documented in the Non-Conformance Summary.

10.3.3. Dilute and reanalyze all samples that have a response for the peak area higher than the highest calibration standard. Check pH of diluted samples and adjust if necessary.

11.0. Calculations / Data Reduction

11.1. The concentration of the aqueous sample can be determined from the calibration curve. Sample concentrations are calculated based on the linear regression calculation and are automatically performed by the instrument. Reporting units are in ug/L.

11.2. For solid samples, use the following calculations:

$$\text{Cr(VI), mg/kg, dry wt.} = \frac{(A/1000) \times C \times 10}{B} \times (100/\ \%\ \text{solid})$$

Where A – concentration of sample, ug/L

B – Weight of sample in grams

C – Final volume of sample in ml

11.3. Print worksheet, calibration curve and quality control and attach computer printout copies plus prep work log.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

14.2.1. Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

14.2.2. Water Retain Samples -These materials are originally received aqueous samples. These solutions are then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9 with sodium bicarbonate (Siedler Chemical SC-0219-25). This solution is discharged into the municipal sewer system.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1996; SW-846, *Method 7199, Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography*.

15.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1996; SW-846, *Method 3060A, Alkaline Digestion for Hexavalent Chromium.*

15.3. TestAmerica Edison SOP No. ED-WET-010, *The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium - USEPA SW846 Method 3060A*, most current revision.

15.4. TestAmerica Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.5. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0 Revision History

- Revision 2, March 23, 2007
  - Section 8.5.1. Added the following text (in italics) to clarify the action taken when pH of the digestate drops below the desired range.

    If the pH drops below 9.0, discard the extract and redigest the sample.

  - Section 8.4.3 and 9.4. Revised to identify the two LCS (soluble and insoluble) used for this method and specified which LCS is applicable for the type of matrix.
  - Section 9.6. Added the procedure for evaluating matrix spikes which are outside of method acceptance limits.
- Revision 3, January 22, 2008
  - Updated to new format as per TestAmerica's SOP format.
  - Added the shelf-life and storage requirements to reagents in section 7, Reagents and standards.

- Added J.T.Baker (Catalog Nos. 3090, 3093 or equivalent) as source of primary and secondary standards. Deleted reference to Inorganic Ventures as a source of the primary standard.
- Replaced the source standard used for the preparation of 10 mg/L Cr+6 (primary and secondary spiking solution) from 100 mg/L Cr+6 to 1000 mg/L Cr+6.
- Deleted matrix spike low and matrix spike high as a QC sample requirement.
- Added Predigestion matrix spike as QC sample for aqueous samples.
- Added the preparation procedure and QC concentrations for sample QC (LCS, MS& PS) and Instrument QC (ICV & CCV).
- Standardized all analytical units to ug/L, mg/L, ug/kg and mg/kg.
- Added the following to the References section: SW846 Method 3060, TestAmerica Edison SOP ED-WET-010 (SW846 Method 3060), TestAmerica Edison SOP ED-GEN-022 (Training SOP) and TestAmerica Laboratory Quality Manual (ED-QA-LQM).

- Revision 4, April 9, 2008
  - Section 7.12. Correct spelling of “Eluant” to “Eluent”
  - Section 10.1.2. Revised procedure to record final pH of digestates
  - Added Section 10.1.3. Included explanation for 10x dilution of all soil samples and how the reporting limit is affected.
  - General instrument parameters for the IC have been added to Section 10.2.
- Revision 5, April 22, 2008
  - Section 9.1.4. Revised the spiking amount added and the sample volume used for the Predigestion Matrix spike to reflect current laboratory practices.

Analytical Procedure for the Total Carbon, Non-purgeable Organic Carbon (Total Organic Carbon) and Total Inorganic Carbon

Standard Methods 5310B
SW846 9060
Lloyd Kahn

Approvals (Signature/Date):

3 NOV 2008

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11/3/08 11/4/08

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1.0 Scope and Application

This method is applicable to the measurement of Total Carbon, Non-purgeable Organic Carbon (also known as Total Organic Carbon) and Total Inorganic Carbon in aqueous matrices.

1.1 Analytes, Matrix(s), and Reporting Limits

Matrix: Aqueous samples;drinking water, surface, domestic and industrial wastes, soils and solids.

Reporting Limit: Aqueous 1.0mg/L, soil/solids 0.1%

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

The amount of carbon in a sample is determined using an autoanalyzer. A sample is introduced to the autoanalyzer where it is heated to between 800 and 900°C in an oxygen atmosphere. This serves to transform all carbon to carbon dioxide, which is then carried through the autoanalyzer by the carrier gas ("zero-grade" air) to an infrared detector. An electric signal is produced from detector in response to the carbon dioxide detected, and this signal is integrated and compared to a working curve of standards for the range of carbon desired to determine the amount of carbon in the sample. Various types of carbon may be determined based on whether or not the sample is acidified, purged with zero-grade air before sampling, or otherwise treated: these include Total Carbon (TC), Non-purgeable Organic Carbon (NPOC) [also known as Total Organic Carbon (TOC)], and Total Inorganic Carbon (TIC). For most aqueous samples, the Volatile Organic Carbon (VOC) component is minute, so that a result for NPOC is often assumed to be equal or similar enough to the TOC result for the latter to be reported as the former, and this is therefore standard procedure for TESTAMERICA—Westfield unless otherwise specified or requested by the client.

3.0 Definitions

Total Organic Carbon (TOC): The carbon measured as a result of oxidation of the sample after the removal of inorganic carbon.

Total Inorganic Carbon (TIC): The difference between Total Carbon and Total Organic Carbon.

Total Carbon (TC): The carbon measured as a result of oxidation of the unpreserved and untreated sample.

NPOC: For purposes of this method, non-purgeable organic carbon (NPOC) is equivalent to the measurement of TOC.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include

prepared samples originating from various environmental matrices and can exceed 20 samples.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis and still be considered valid as promulgated in the method.

Instrument Blank: an aliquot of reagent water to which the same reagents are added as are added to the investigative and/or quality control samples.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. LCD, a replicate LCS.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum concentration of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific method. An MDL, by definition, has an uncertainty of +100%, and is the point at which the possibility of detection of false negatives and false positives is equal. The MDL thus represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range. Also referred to as Limit of Detection (LOD). (40 CFR Part 136 Appendix B).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. Frequently summarized in a QC memo.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator.

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Raw Data: any original information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

4.0 Interferences

4.1 The high temperature of combustion should cause 99+% of the carbon to convert to detectable carbon dioxide. In some instruments, high salt content in samples may suppress or adversely affect the results, and such samples suspected of having more than 1% total dissolved solids (TDS) are usually diluted to <1% TDS before being analyzed, and the sample reporting limit (RL) be appropriately increased by the dilution factor. However, with the liquiTOC does not actually require this dilution of high salt-content samples. [The only problem is that the reactor tube needs to be rinsed free of the salts after every 90 samples when analyzing brines or sea water.]

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- liquiTOC Analyzer,
  - computer workstation,
  - autosampler, and associated peripherals and components
    - solids analysis assembly
- Analytical balance capable of reading to 0.1mg
- Two-stage regulator for "zero air" compressed air cylinder

6.2 Supplies

- Teflon 18mL plastic autosampler cups (reusable)
- Eppendorf 100-1000µL adjustable autopipetter
- Disposable pipetter tips
- Aluminum/Tin foil squares
- Volumetric glassware, triply rinsed with fresh deionized water
- Quartz and brass sample boats
- 25mL glass beakers (HCL washed, deionized rinsed three times, oven dried)
- Small scoopula
- Sample boat tongs

7.0 Reagents and Standards

7.1 Hydrochloric acid, concentrated (HCl): Trace metals grade or better.

7.2 Deionized water (fresh from the system)

7.3 "Zero-grade" compressed air

7.4 1000mg/L TOC Stock Standard: Dry about 5g of anhydrous potassium hydrogen phthalate (KHP; also known as potassium biphthalate) in an oven at 103°C for 1 hour, and then allow it to cool to room temperature in a desiccator. Place 2.1254g of the oven-dried KHP into about 900mL of fresh deionized water in a 1L volumetric (that has first been triply rinsed with deionized water). Add 2mL of concentrated HCl to preserve. Stopper and mix by inversion or mechanically with stir bar and stir plate until the crystals are dissolved. Bring the solution to the mark with deionized water, stopper, and mix by inversion. (If a stir bar has been used, first remove the stir bar then bring the volume to the mark.) 1.00mL = 1.00mg of carbon. Store in brown glass. EXP: 12 months. Store refrigerated.

7.5 100.0mg/L Intermediate TOC Standard: Using class A glassware, pipet 10.00mL of the 1000mg/L TOC Stock Standard into a 100mL volumetric flask (that has first been triply rinsed with deionized water) that contains about 50mL of fresh deionized water. Add 8 drops of concentrated HCl. Dilute to mark with deionized water, stopper, and mix by inversion. EXP: 12 months. Store refrigerated.

7.6 ICV TOC Stock Standard (second source standard): Purchase a second source Phthalate concentrate, such as Inorganic Venture 1000ppm Phthalate (Catalog #ICKHP1-1: this contains 58.5% carbon, making it a 585mg/L Carbon standard). Alternatively, prepare a 1000mg/L TOC standard from a separate lot of oven-dried KHP as described in item 7.4. EXP: Manufacturer's expiration or 12 months. Store refrigerated.

7.7 23.4mg/L ICV Working Standard: Using class A glassware, transfer 4.00mL of the 1000ppm Phthalate purchased ICV stock standard to a 100mL volumetric flask (triply rinsed with Deionized water) containing about 80mL of fresh deionized water. Add 8 drops of concentrated HCl. Dilute to mark with deionized water, stopper, and mix by inversion. EXP: 12 months. Store refrigerated.

7.8 32.0mg/L LCS/CCV Standard: Using class A glassware, transfer 8.00mL of the 1000mg/L TOC Stock Standard to a 250mL volumetric flask (that has first been triply rinsed with deionized water) containing about 200mL of deionized water. Add 16 drops of concentrated HCl. Dilute to mark with deionized water, stopper, and mix by inversion. EXP: 6 months. Store refrigerated.

7.9 TOC Working Curve Standards: Using the 1000mg/L stock standard, create the following working curve standards. To each 100mL flask, add 8 drops of concentrated HCl to acidify and exclude dissolved carbon dioxide. EXP: 6 months. Store refrigerated.

Amount of **100.0mg/L Intermediate** Standard	Amount of **1000mg/L Stock** Standard	Final Volume	Concentration of Working Standard
X	X	100mL	0 mg/L
1000µL	X	100mL	1.0
2000 µL	X	100mL	2.0
X	500µL	100mL	5.0
X	1000µL	100mL	10.0
X	2000 µL	100mL	20.0
X	4.00mL	100mL	40.0

7.10 TOC Soil Standard: Purchase a sediment or soil standard of certified concentration for total carbon (High Purity Standards Catalog #CRM-LO-A) having about 1% total carbon content.

7.11 LCS Soil Standard: Purchase a sediment or soil standard of a different lot or different manufacturer (NIST Buffalo River Sediment Catalog #8407) having between 0.5 and 3.5% total carbon.

7.12 Ottawa sand, HCl and deionized water rinsed, and oven baked. (Store desiccated.)

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	Amber glass	120mL (3 40ml vials)	HCL, pH < 2; Cool 6°C	28 Days	40 CFR Part 136.3
Soils	Amber glass	50g	Cool 6°C	28 Days	Lloyd Kahn method and SW846

For all other types of carbon analysis besides TOC, do not preserve samples.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Initial Calibration Blank (ICB) and Method Blank (MB)	One per analytical batch	<RL	Re-analyze; if still out begin analysis over
Laboratory Control Sample (LCS) and LCSD (optional; at client's request)	One per 20 samples or fraction thereof	85-115%	Analyze a new aliquot of LCS; if still out check stock std.
Matrix Spike (MS)	One per 20 samples or fraction thereof	75-125%	If MS recovery indicates a suppressive interference the sample and MS should be diluted and re-analyzed. If the addition of the spike extends the concentration beyond the calibration curve, the sample should be diluted and the calculated results adjusted accordingly.
Matrix Spike Duplicate (MSD)	One per 20 samples or fraction thereof	75-125% RPD ≤20%	Re-analyze; if precision criteria still not met determine if matrix or method is cause

9.2 Instrument QC

QC ANALYSIS	FREQUENCY	QC CRITERIA	CORRECTIVE ACTION
Initial Calibration	Minimum of a Weekly Calibration	R≥ 0.995	Re-analyze standards; if still problematic, check stock standards and reagents
Initial Calibration Verification (ICV)	One per analytical batch	90-110%	Re-analyze; if still out recalibrate
Continuing Calibration Verification (CCV)	One every 10 samples analyzed, and to close out the analytical run	90-110%	Re-analyze another aliquot; if still out recalibrate and reanalyze all samples not

			bracketed by passing Cont. Cal. Std.
Continuing Calibration Blank (CCB)	One every 10 samples analyzed, and to close out the analytical run	<1.0mg/L	Re-analyze; if still out, clean glassware and begin analysis over at last passing Cont. Cal. Blank.

- Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and or analyzed with a specific batch and their frequency. Control samples are uniquely identified within the batch (MB, LCS, MS, MSD, etc) and an individual batch number uniquely identifies each batch.
- All data, regardless of regulatory program or level of reporting (Level1, 2, 3, or 4), shall be subject to a through review which involves a primary, secondary and completeness review process. All levels of review must be documented.
  - Primary review is referred to as a "bench-level" review. In most cases, the analyst who generates the data is the primary reviewer. One of the most important aspects of primary review is to make sure that all method specific requirements are met and job specific requirements are understood.
  - Primary review ensure that; sample preparation is accurate and complete, calculations were performed correctly, quantitation has been performed correctly, identifications are accurate method SOPs have been followed, QC criteria has been met, dilution factors have been applied correctly, and QA memos have been generated for anomalous results, if necessary.
  - Secondary review shall be a complete technical review of the data. If problems are found during secondary review, the reviewer will work with the appropriate personnel (analyst, PM, LD, QA) to resolve them.
  - The completeness review shall addresses the following; is the job report complete, does the data meet client requirements, were data quality objectives met, were holding times met, were inter-method relationships reasonable and was the project completed according to client's C-O-C? The completeness review may include a generation of a job case narrative, which outlines anomalous data and non-conformances by using QA memos generated during primary and secondary review.

- Corrective Action for out of Control Data
  - If problems are found upon review with sample data the following shall apply: All non-conformances that affect sample data shall become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, may be required.

- Contingencies for Handling out of control or Unacceptable Data
  - If the sample data is deemed to be out of control, consultation with the Supervisor, Manager, Project Manager, Laboratory Director, or QA Manager for direction may be required. Where non-conformances specifically affect a client's sample and/or data, the client may need to be directly informed and corrective actions, based on

the client's direction, may be taken. Action can also take the form of reporting data, and including the non-conformance comment in the project narrative.

10.0 Procedure—Total Organic Carbon (TOC)

10.1 Instrument set up

10.1.1 Turn the computer and printer on. Once the computer has booted and the Windows desktop is displayed, turn the autosampler on, and then the analyzer. Open the valve on the zero-grade air tank and check the reading to be sure about 15psi of air is flowing into the line to the analyzer. **NOTE!** The autosampler carousel will rotate as it performs a check; do not remove or replace the carousel during this time. Furthermore, the analyzer will undergo a test—you can hear the syringe moving for about 2 minutes. Wait until that is complete before launching the operating software.

10.1.2 Launch the liquiTOC software by clicking on the desktop icon. The User Log-in window will then show; enter a "2" for the password in order to enter the software. See Figure 1 and 2.

FIGURE 1

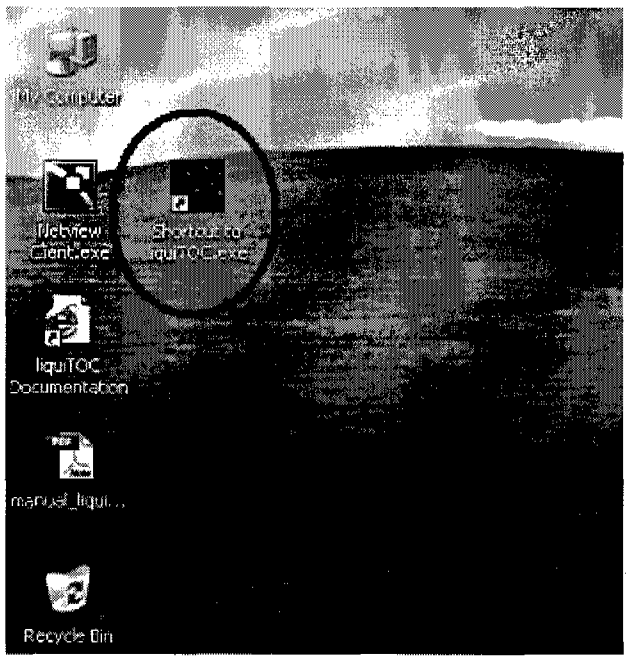
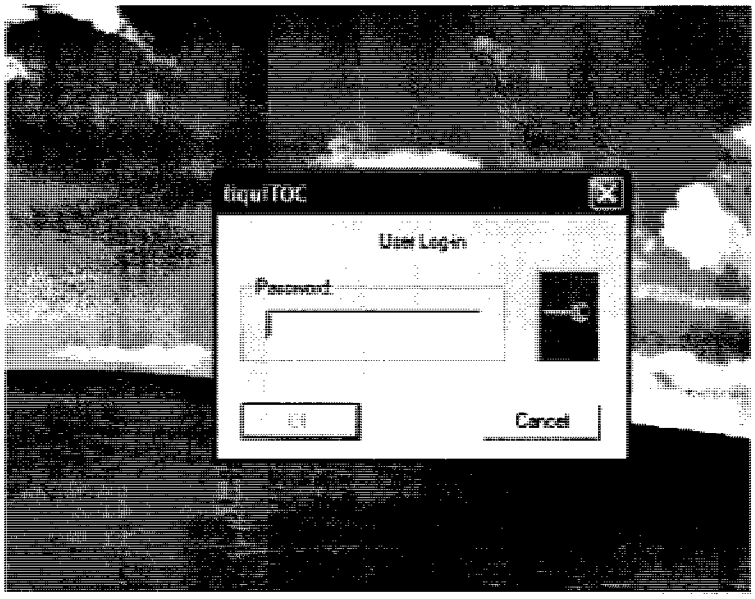
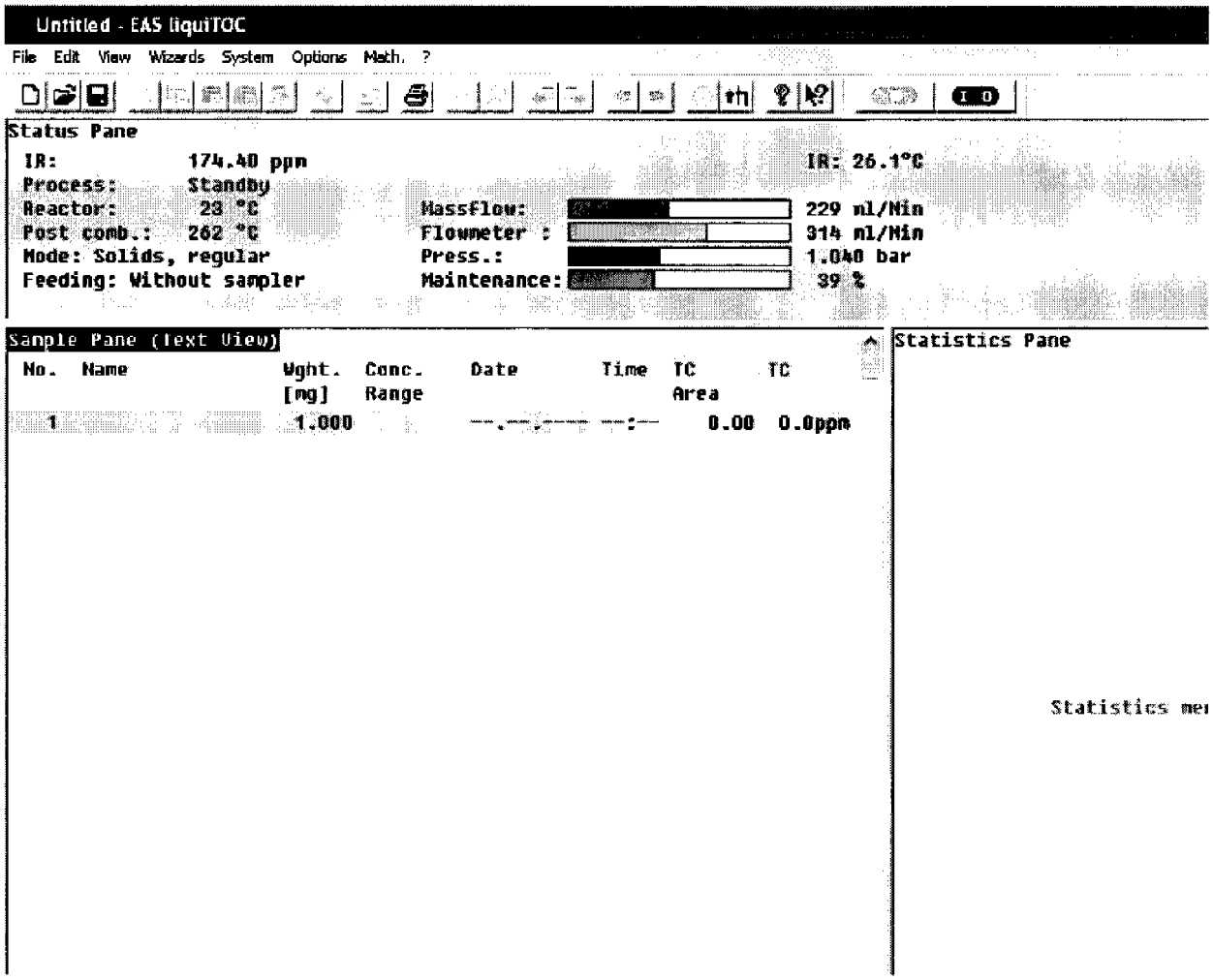


FIGURE 2



10.1.3 The operating software will then launch (See Figure 3). Check the Status Pane in the operating software—it should turn from red to green to indicate optimal conditions. If not, proceed to the troubleshooting section of the manufacturer's manual, or call tech support [Scott Hughes, elementar Americas, Inc at (856) 787-0055 x202].

FIGURE 3



10.1.4 The Status Pane has a number of bar graphs indicating the status of the current operating system. In standby mode and once the instrument has equilibrated, the gas flow at the Massflow and Flowmeter should be about 200mL/min ± 10mL/min, and the pressure should be about 1.0 ± 0.5bar; if not, adjust the gas flow at the zero-grade air tank as necessary. **NOTE**: If the Massflow and Flowmeter differ by more than 10 mL/min, it may be a sign of a leak in the system.

10.1.5 To create an analysis batch, first orient the autosampler controls so that the probes are in the washing well of the autosampler as follows:

10.1.5.1 Click on the little probe icon with the up-arrow indicated in the menu bar.

10.1.5.2 Alternatively, from either the Sample Pane Text or Sample Pane Graphics, select Options→ Diagnostic→ System Test which the produces the flow chart diagram of the physical layout of the analyzer. Next click on the autosampler picture to show a sub-window, which causes the probe arm to swing up and into the washing well. Close out the sub-window and the flow diagram window.

10.1.6 The software provides a Calibration Wizard to assist with the set up of the working curve, blank and "run-in" samples (which are used to clean the instrument and prepare it for blank determination). Launch the Wizard and enter the following set data for TOC. Refer to Figures 4 through 8.

10.1.6.1 # of measurements = 3

10.1.6.2 # of run-in = 6
10.1.6.3 # of blanks = 3
10.1.6.4 Conc range = 20ppm
10.1.6.5 # cal points = 6
10.1.6.6 Click on the selection for "Different solutions, unique volume" (this is the default setting)
10.1.6.7 NPOC conc of parent solution = 40
10.1.6.8 Click "Finish" to set the analytical run.

FIGURE 4

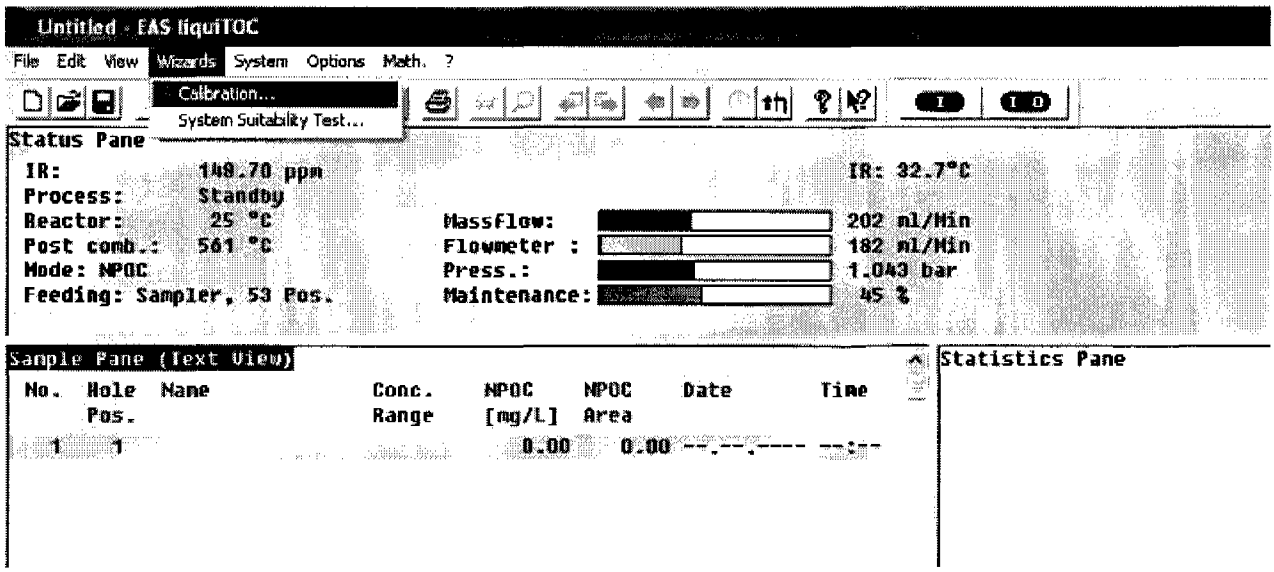


FIGURE 5

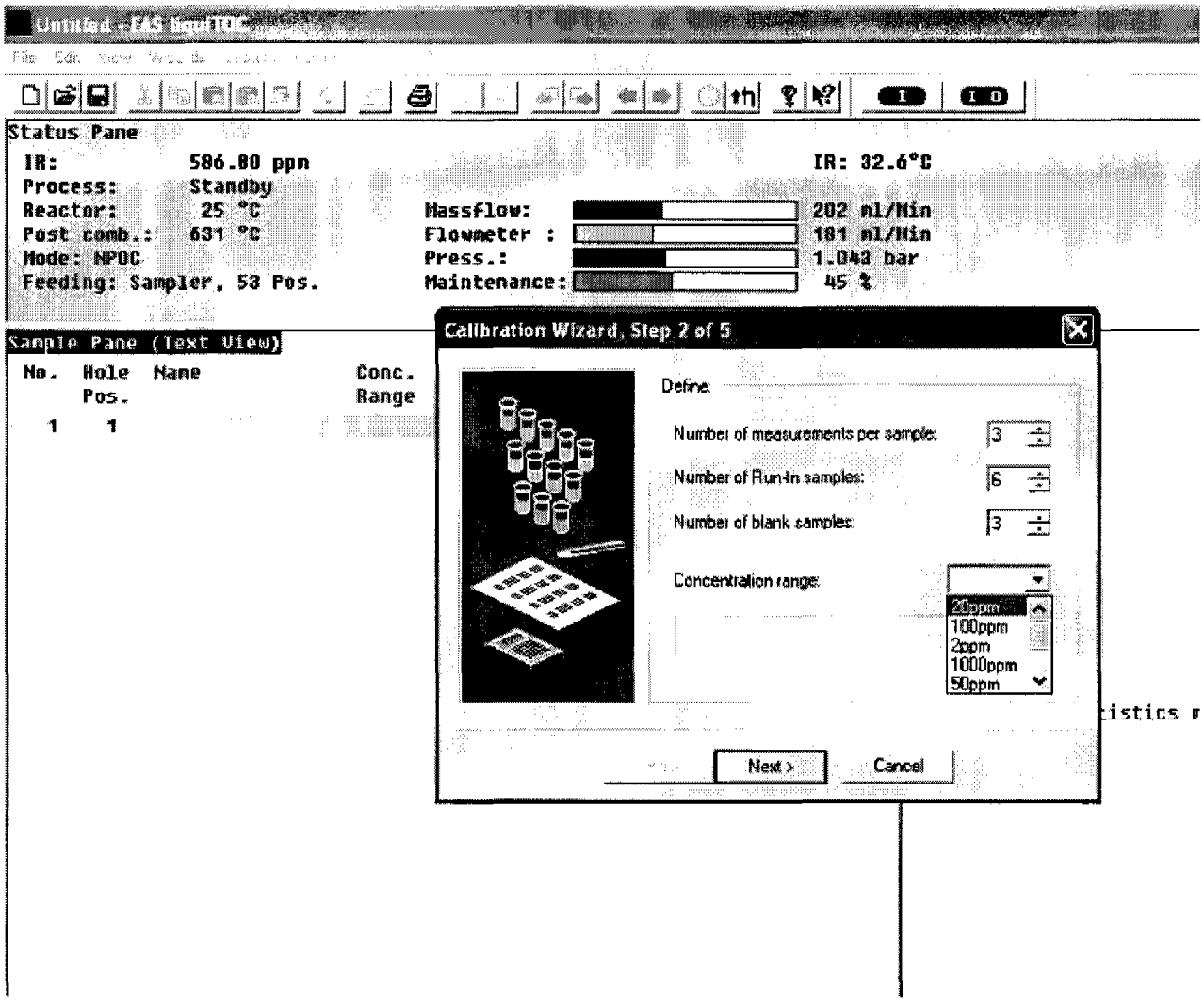

Untitled - EAS liquTOC
Status Pane
IR: 586.80 ppm
Process: Standby
Reactor: 25 °C
Post comb.: 631 °C
Mode: NPOC
Feeding: Sampler, 53 Pos.
IR: 32.6°C
Massflow: 202 ml/Min
Flowmeter : 181 ml/Min
Press.: 1.043 bar
Maintenance: 45 %
Sample Pane (Text View)
No. Hole Pos. Name Conc. Range
1 1
Calibration Wizard, Step 2 of 5
Define:
Number of measurements per sample: 3
Number of Run-in samples: 6
Number of blank samples: 3
Concentration range:
20ppm
100ppm
2ppm
1000ppm
50ppm
Next >
Cancel

FIGURE 6

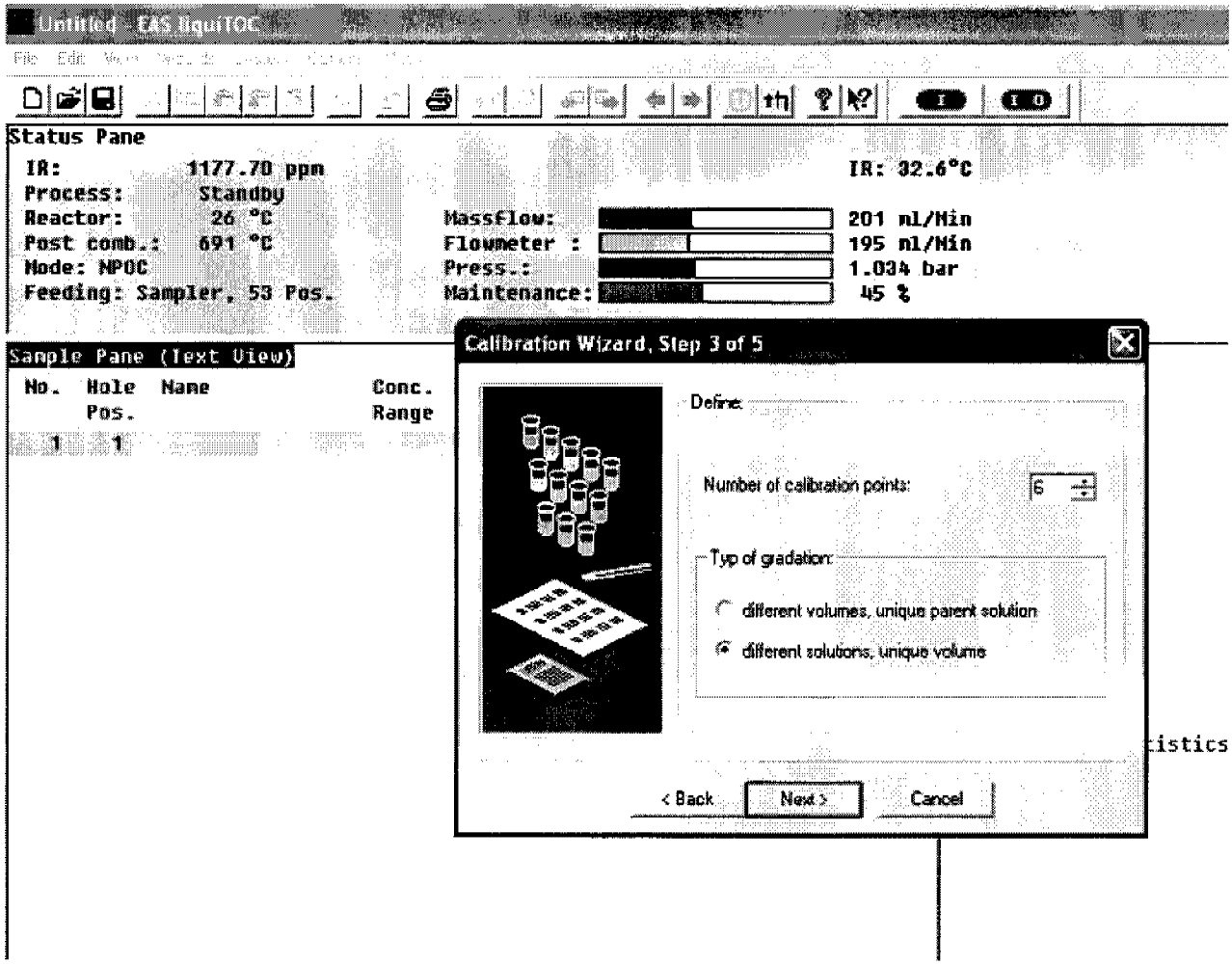

Untitled - EAS liquiTOC
Status Pane
IR: 1177.70 ppm
Process: Standby
Reactor: 26 °C
Post comb.: 691 °C
Mode: NPOC
Feeding: Sampler, 53 Pos.
IR: 32.6°C
Massflow: 201 ml/Min
Flowmeter : 195 ml/Min
Press.: 1.034 bar
Maintenance: 45 %
Sample Pane (Text View)
No. Hole Pos. Name Conc. Range
1 1
Calibration Wizard, Step 3 of 5
Define:
Number of calibration points: 6
Typ of gradation:
different volumes, unique parent solution
different solutions, unique volume
< Back
Next >
Cancel

FIGURE 7

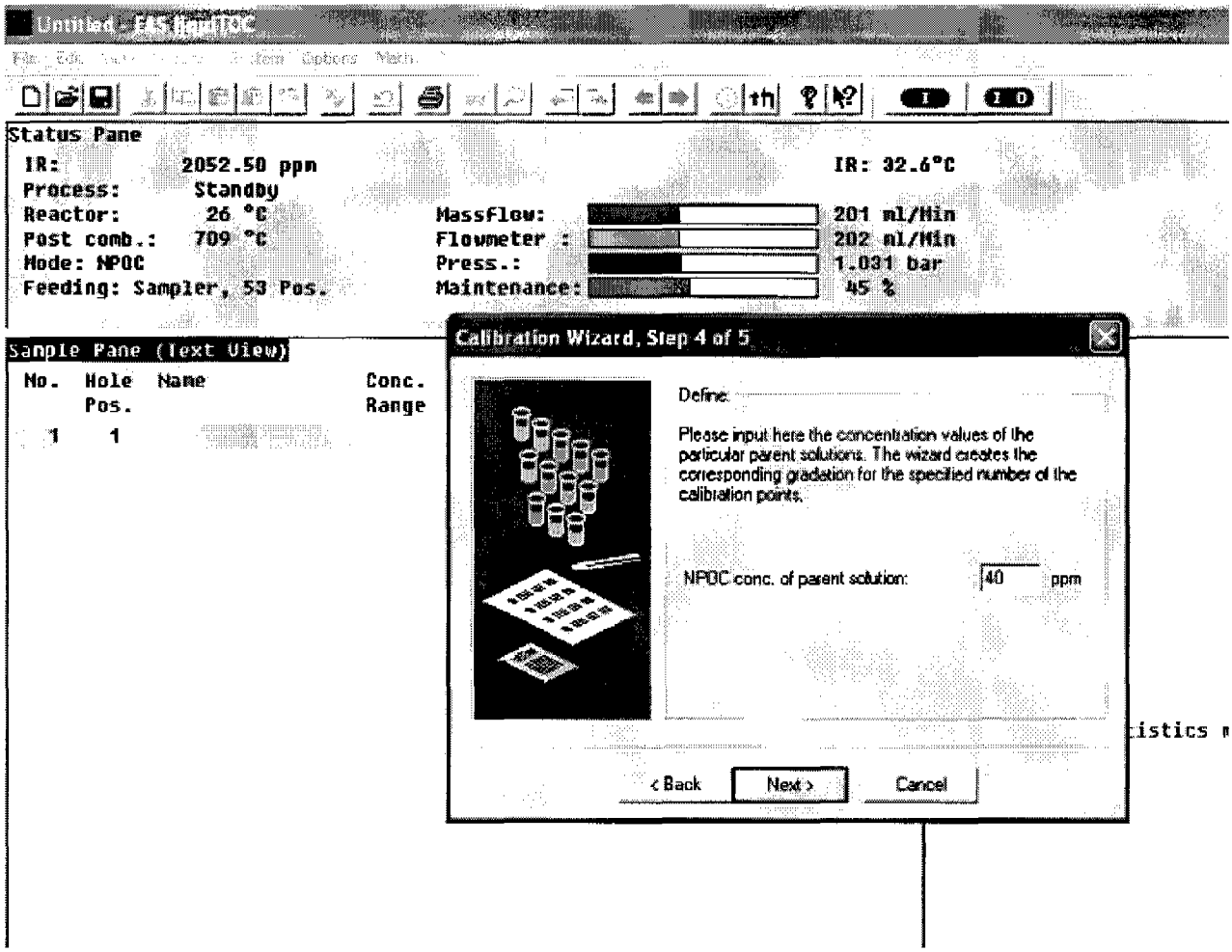
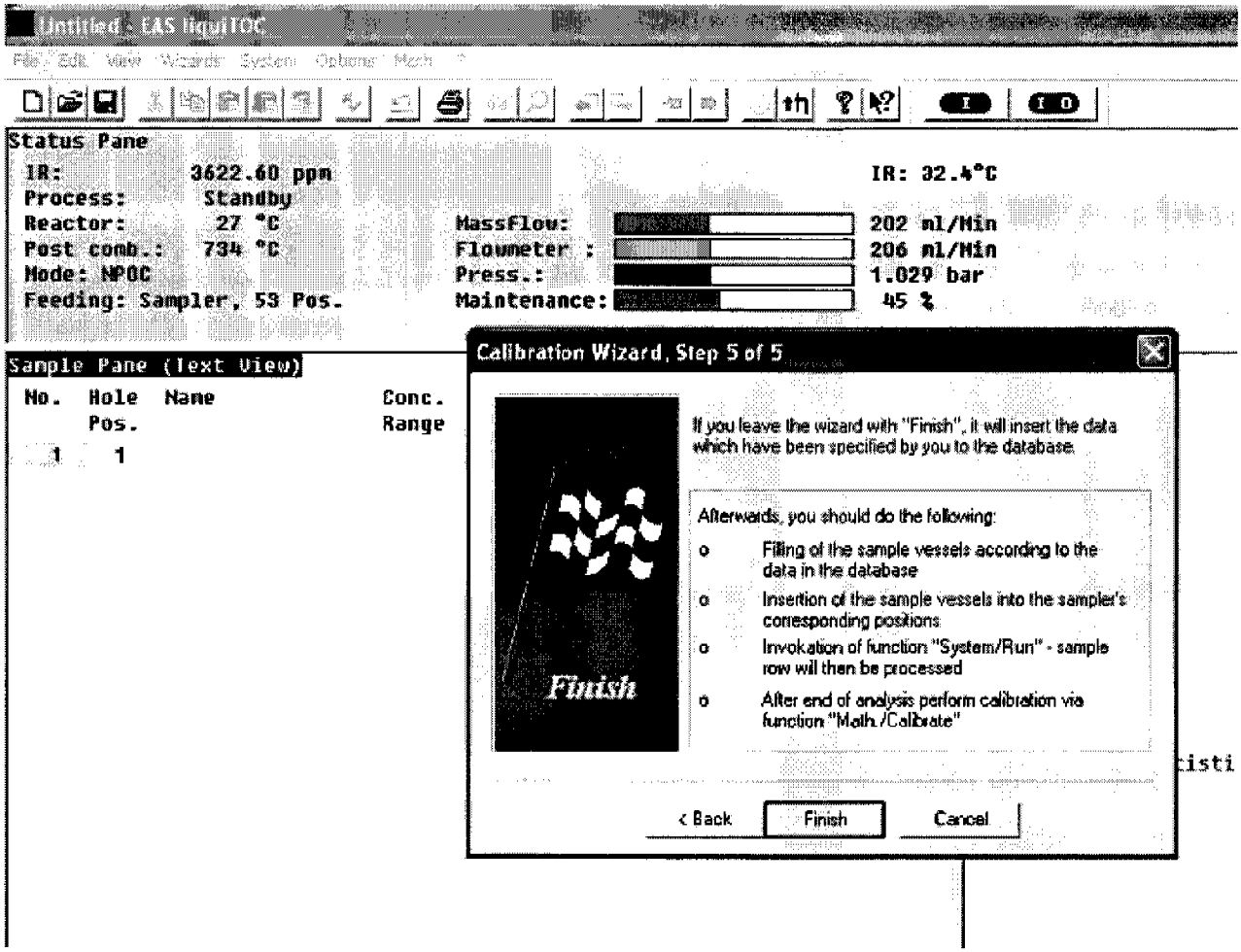

Untitled - EAS liquiTOC
Status Pane
IR: 2052.50 ppm
IR: 32.6°C
Process: Standby
Reactor: 26 °C
Post comb.: 709 °C
Mode: NPOC
Feeding: Sampler, 53 Pos.
Massflow: 201 ml/Min
Flowmeter : 202 ml/Min
Press.: 1.031 bar
Maintenance: 45 %
Sample Pane (Text View)
No. Hole Pos. Name Conc. Range
1 1
Calibration Wizard, Step 4 of 5
Define:
Please input here the concentration values of the particular parent solutions. The wizard creates the corresponding gradation for the specified number of the calibration points.
NPOC conc. of parent solution: 40 ppm
< Back
Next >
Cancel

FIGURE 8

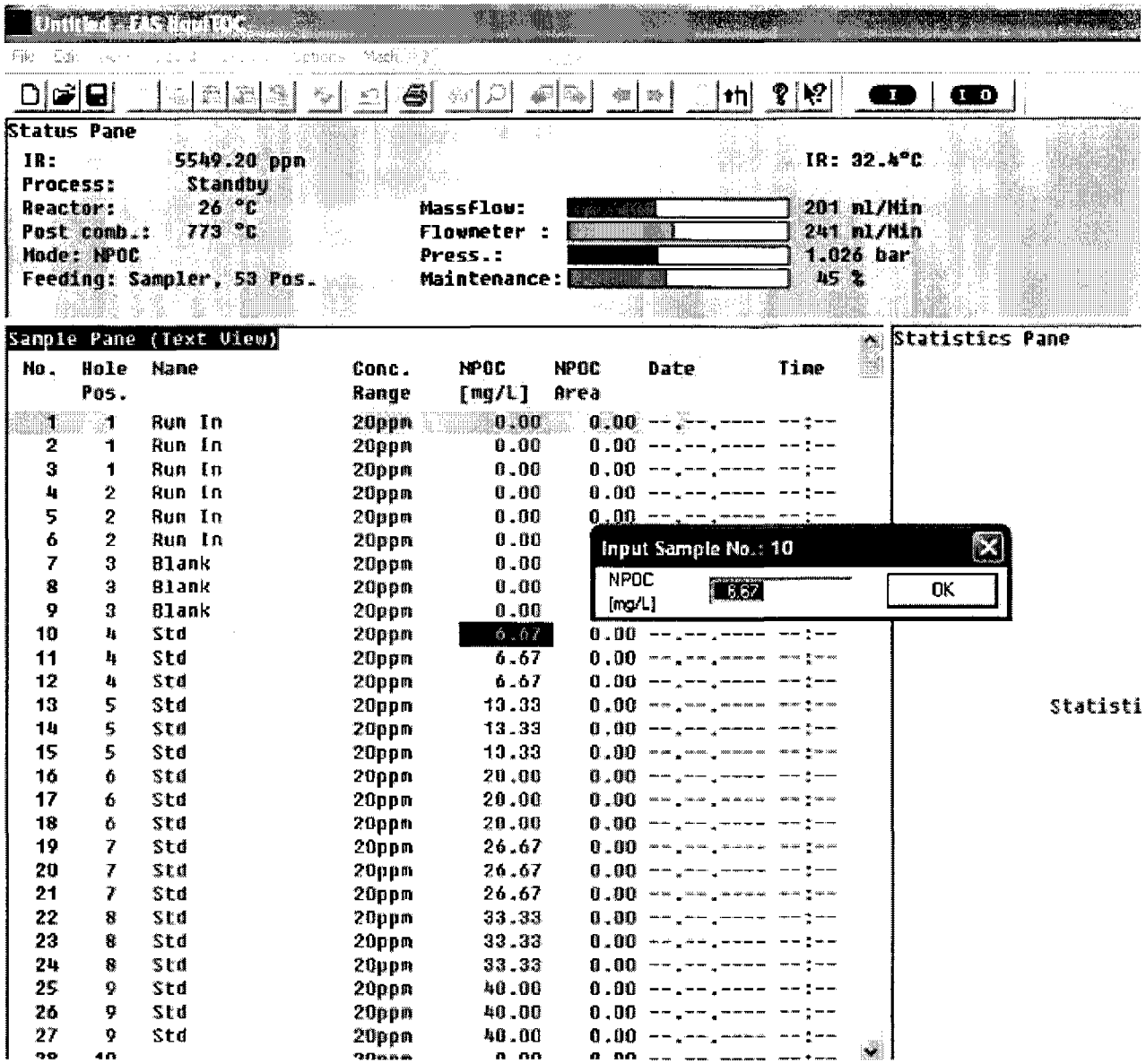

Untitled - EAS liquiTOC
Status Pane
IR: 3622.60 ppm
Process: Standby
Reactor: 27 °C
Post comb.: 734 °C
Mode: NPOC
Feeding: Sampler, 53 Pos.
IR: 32.4°C
MassFlow: 202 ml/Min
Flowmeter : 206 ml/Min
Press.: 1.029 bar
Maintenance: 45 %
Sample Pane (Text View)
No. Hole Pos. Name Conc. Range
1 1
Calibration Wizard, Step 5 of 5
If you leave the wizard with "Finish", it will insert the data which have been specified by you to the database.
Afterwards, you should do the following:
Filling of the sample vessels according to the data in the database
Insertion of the sample vessels into the sampler's corresponding positions
Invokation of function "System/Run" - sample row will then be processed
After end of analysis perform calibration via function "Math./Calibrate"
Finish
< Back
Finish
Cancel

10.1.7 Return to the Sample Pane (Text). In order to make the working curve standards of correct concentration, you must overwrite the default, calculated values assigned by the calibration wizard. To do so (refer to Figure 9):

10.1.7.1 Under the "NPOC [mg/L]" column, find the first of the three replicates for the first (lowest) working curve standard. Double click on it to bring up a data entry box. Delete the current entry and type a "1" for 1.0mg/L. Press the Enter key and repeat twice more so all three of the replicates now read "1.00."

10.1.7.2 Continue in a like manner, entering the next standard of "2.00," then "5.00," then "10.0," and finally "20.0": leave the last standard, 40.0 as is.

FIGURE 9



10.1.8 You can now enter QC and investigative samples (refer to Figure 10 and 11). Start with the head-end QC samples as follows. The "ZZ " prefix (two Zs and a space) is required to tell the instrument that the samples are to be corrected for the intrinsic amount of carbon in the deionized water that was used to create the standards:

10.1.8.1 ZZ ICV 23.4
10.1.8.2 ZZ ICB
10.1.8.3 ZZ MB1
10.1.8.4 ZZ LCS1 32.0

FIGURE 10

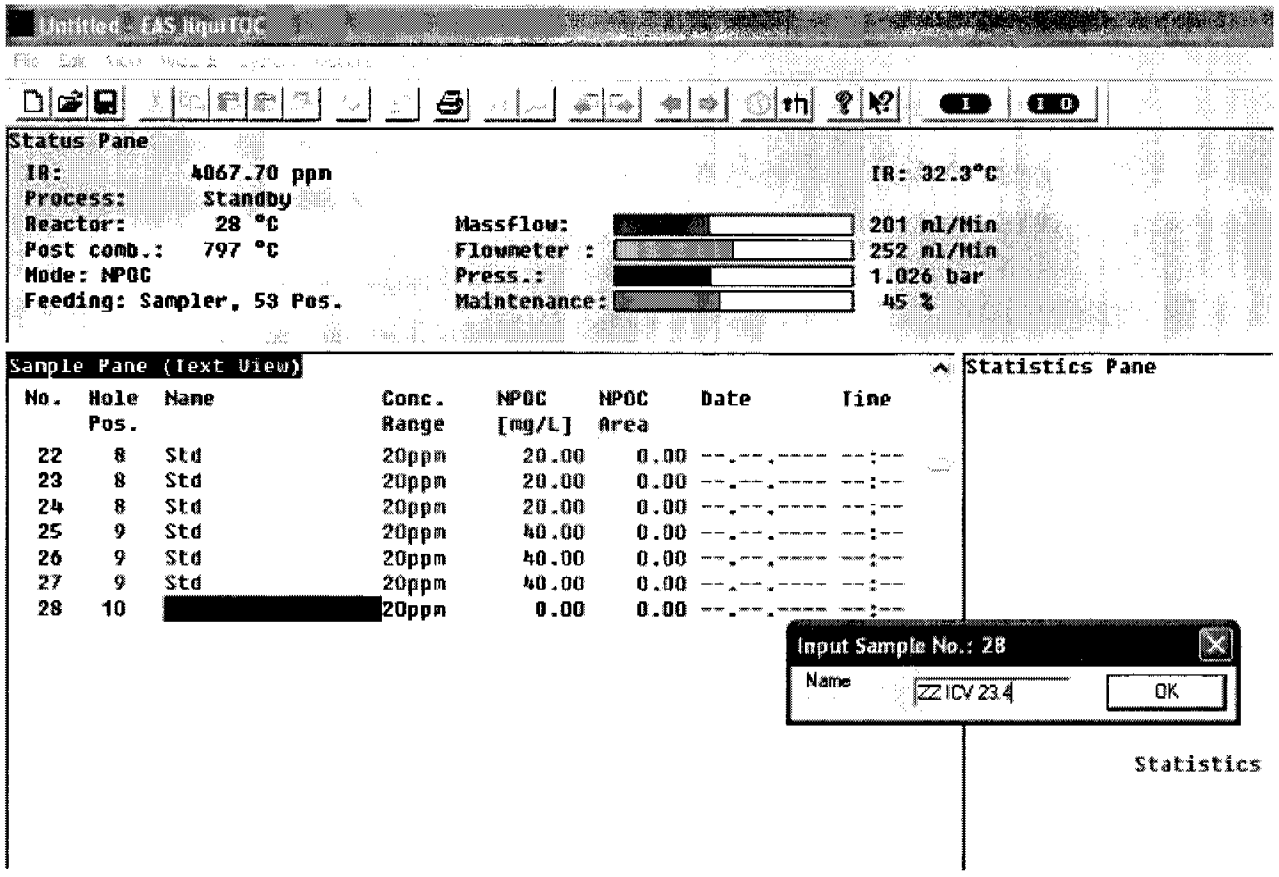
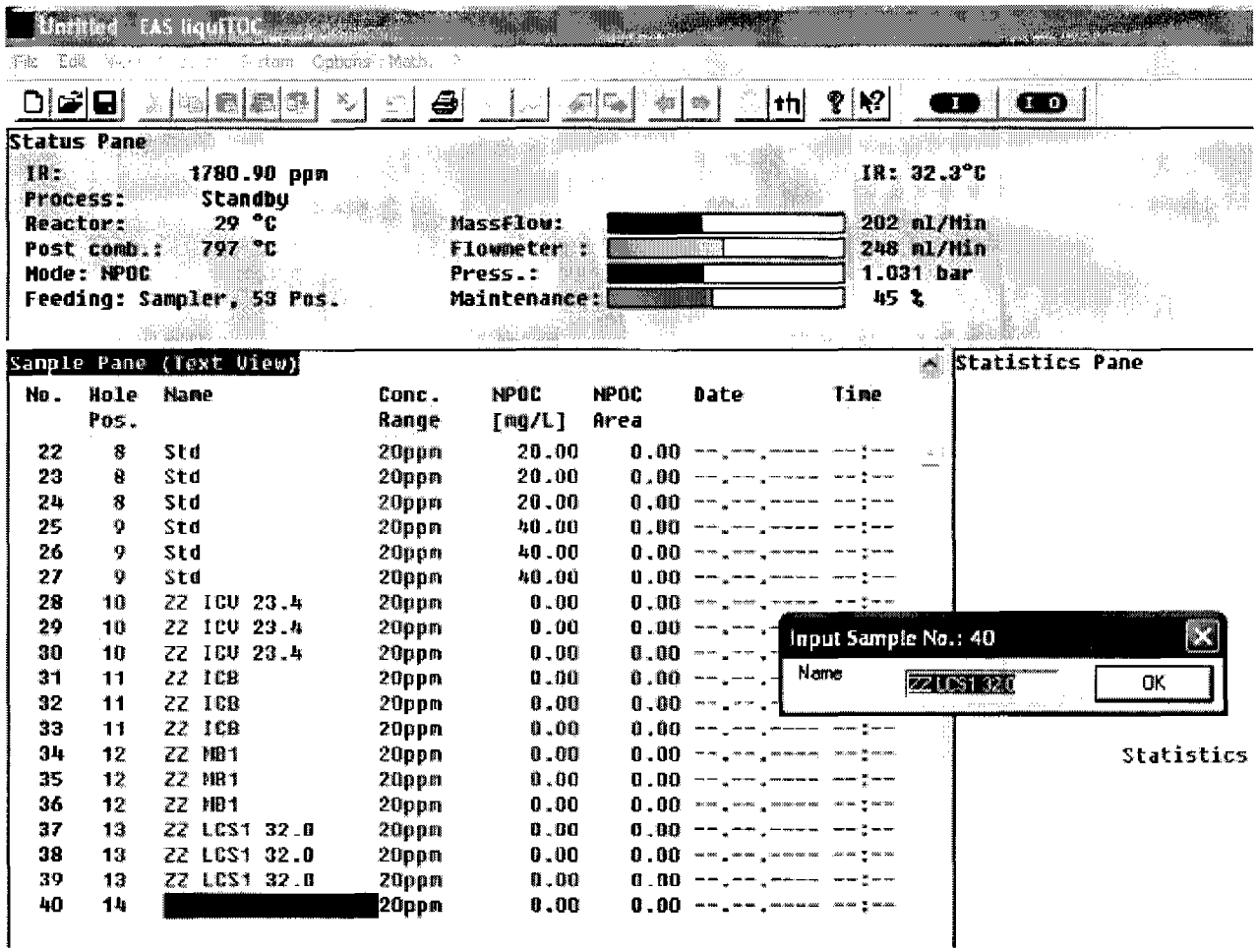


FIGURE 11



10.1.9 To indicate that a correction for the deionized water carbon amount, these line items with "ZZ " will show up in green-colored text. All other items are in blue-colored text. **NOTE** that the MB and LCS (and CCV and CCB) need a unique, incremental number after each to differentiate them in the software; otherwise all LCSs, MBs, CCVs and CCBs will be grouped together, the software assuming they are all the same, single sample.

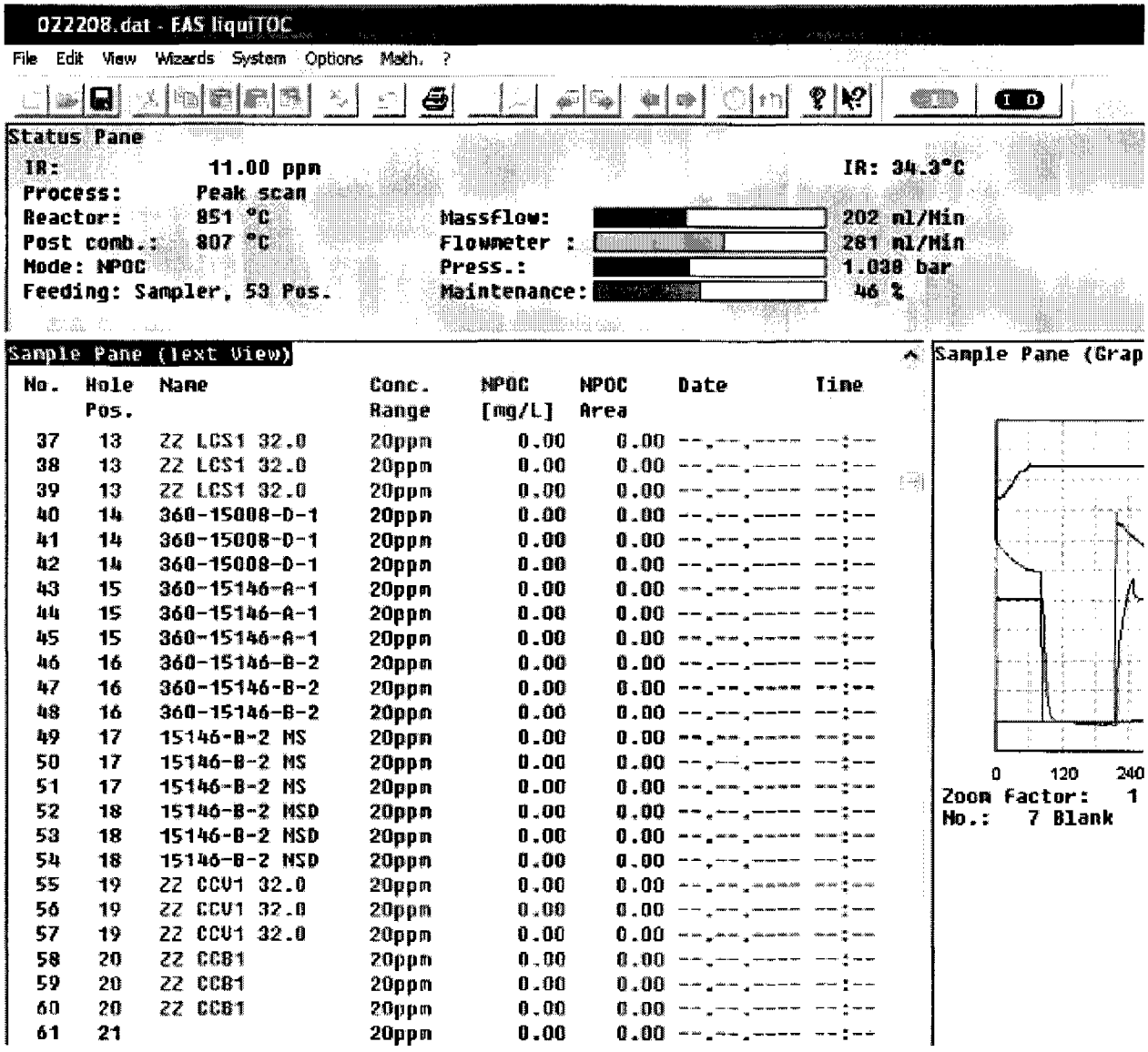
10.1.10 Click on the next space open for sample description. A window will appear asking for data input. Type in the identifier for each investigative sample to be run: since each sample is run in triplicate, click the "OK" two more times to set up that sample for triplicate analysis, and then enter the next sample description to be analyzed. Samples should be analyzed in the following order, with appropriate QC samples as indicated (for frequency and type of QC samples, refer to the section 9.0). **Scan Sample ID numbers whenever possible to avoid mis-keying ID numbers.**

ZZ ICV 23.4
ZZ ICV 23.4
ZZ ICV 23.4
ZZ ICB
ZZ ICB

ZZ ICB
ZZ MB1
ZZ MB1
ZZ MB1
ZZ LCS1 32.0
ZZ LCS1 32.0
ZZ LCS1 32.0
Sample #1
Sample #1
Sample #1
Sample #1 MS
Sample #1 MS
Sample #1 MS
Sample #1 MSD
Sample #1 MSD
Sample #1 MSD
Sample #2
Sample #2
Sample #2
...
Sample #8
Sample #8
Sample #8
ZZ CCV1
ZZ CCV1
ZZ CCV1
ZZ CCB1
ZZ CCB1
ZZ CCB1
Sample #9
Sample #9
Sample #9
Sample #10
Sample #10
Sample #10
...

10.1.11 A batch with investigative samples and QC may look like something shown in Figure 12 Note that regular samples are shown in blue text in the Sample Pane (Text View).

FIGURE 12



No.	Hole Pos.	Name	Conc. Range	NPOC [mg/L]	NPOC Area	Date	Time
37	13	ZZ LCS1 32.0	20ppm	0.00	0.00	--.--.----	--:--
38	13	ZZ LCS1 32.0	20ppm	0.00	0.00	--.--.----	--:--
39	13	ZZ LCS1 32.0	20ppm	0.00	0.00	--.--.----	--:--
40	14	360-15008-D-1	20ppm	0.00	0.00	--.--.----	--:--
41	14	360-15008-D-1	20ppm	0.00	0.00	--.--.----	--:--
42	14	360-15008-D-1	20ppm	0.00	0.00	--.--.----	--:--
43	15	360-15146-A-1	20ppm	0.00	0.00	--.--.----	--:--
44	15	360-15146-A-1	20ppm	0.00	0.00	--.--.----	--:--
45	15	360-15146-A-1	20ppm	0.00	0.00	--.--.----	--:--
46	16	360-15146-B-2	20ppm	0.00	0.00	--.--.----	--:--
47	16	360-15146-B-2	20ppm	0.00	0.00	--.--.----	--:--
48	16	360-15146-B-2	20ppm	0.00	0.00	--.--.----	--:--
49	17	15146-B-2 MS	20ppm	0.00	0.00	--.--.----	--:--
50	17	15146-B-2 MS	20ppm	0.00	0.00	--.--.----	--:--
51	17	15146-B-2 MS	20ppm	0.00	0.00	--.--.----	--:--
52	18	15146-B-2 MSD	20ppm	0.00	0.00	--.--.----	--:--
53	18	15146-B-2 MSD	20ppm	0.00	0.00	--.--.----	--:--
54	18	15146-B-2 MSD	20ppm	0.00	0.00	--.--.----	--:--
55	19	ZZ CCV1 32.0	20ppm	0.00	0.00	--.--.----	--:--
56	19	ZZ CCV1 32.0	20ppm	0.00	0.00	--.--.----	--:--
57	19	ZZ CCV1 32.0	20ppm	0.00	0.00	--.--.----	--:--
58	20	ZZ CCB1	20ppm	0.00	0.00	--.--.----	--:--
59	20	ZZ CCB1	20ppm	0.00	0.00	--.--.----	--:--
60	20	ZZ CCB1	20ppm	0.00	0.00	--.--.----	--:--
61	21		20ppm	0.00	0.00	--.--.----	--:--

10.1.12 It is recommended that a "stop tag" is placed after the first LCS. Do this by selecting System→Stop Tag and entering the number of the line for the last triplicate of the highest standard, then click OK and close out the window. This allows the calibration to be reviewed and quality control head end samples to be checked before the system continues with analyzing samples.

10.1.13 Place samples into the autosampler carousel in the order as dictated in the analytical batch, using the reusable PTFE (Teflon) cuvettes that have been rinsed three times with deionized water and air dried. Fill each cuvette with water so that there is about ½ inch of head space. For TOC (NPOC) add 100µL of concentrated HCl to the cuvette, then cover tightly with a tin foil square and place in the carousel in order.

10.1.13.1 "Run-in" samples are simply deionized water, as are blanks, ICB/MBs, and CCBs.

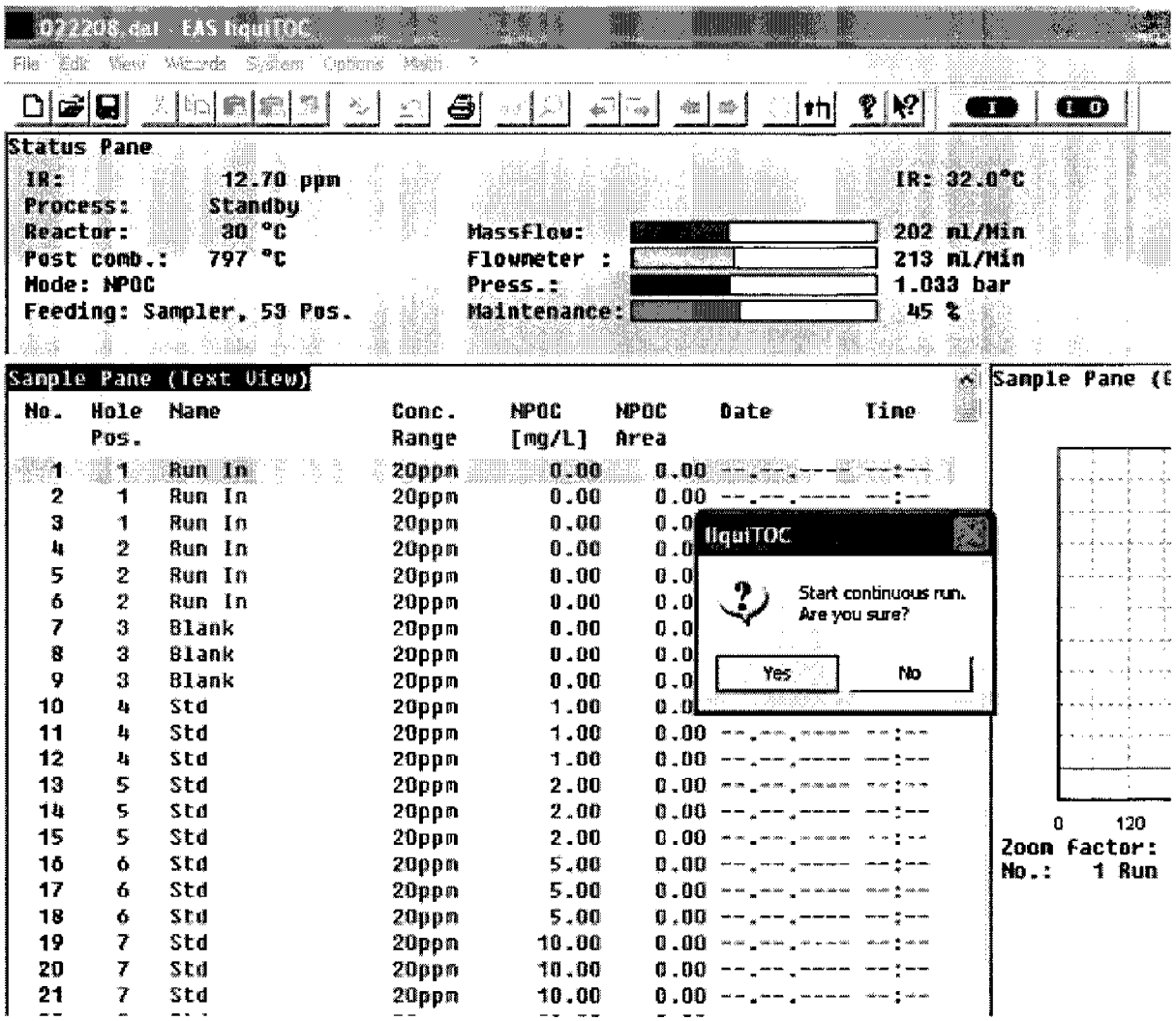
10.1.13.2 Use concentrations for LCS, ICV and CCV that fall between 50 and 75% of the highest working standard. The ICV is to be prepared from a second, separate source to the working curve. The LCS and CCVs are prepared from the same standard as the working curve source. (See preparation method listed above.)

10.1.13.3 MS and MSD are prepared by adding 400µL of 1000mg/L TOC standard to 40mL of sample in a 50mL mixing graduated cylinder. Stopper and mixing by inversion. The spiked concentration of MS or MSD is 10.0mg/L.

10.1.14 Save the batch by clicking on the Computer Disk icon and entering the date of the analysis for the filename in the format MMDDYY. Click "Yes" for saving graphical data.

10.1.15 Initiate the analytical run by clicking on the green "I" button at the top of the screen. Click "Yes" to start the continuous run, and the analyzer will begin analysis of the samples automatically. (The run can be stopped by clicking the green/red I/O button.) Refer to Figure 13.

FIGURE 13



10.1.16 Once the curve has been generated, if the Stop Tag has been correctly set after the first LCS, the system stops to permit review of the calibration. To accept and to calibrate the instrument:

10.1.16.1 Select Math→Calibrate and choose the 20ppm method. Click on the "Close" button to close the window and show the working curve calibration in the Calibration Pane on the right for review. (See Figures A, B and C below.)

FIGURE A

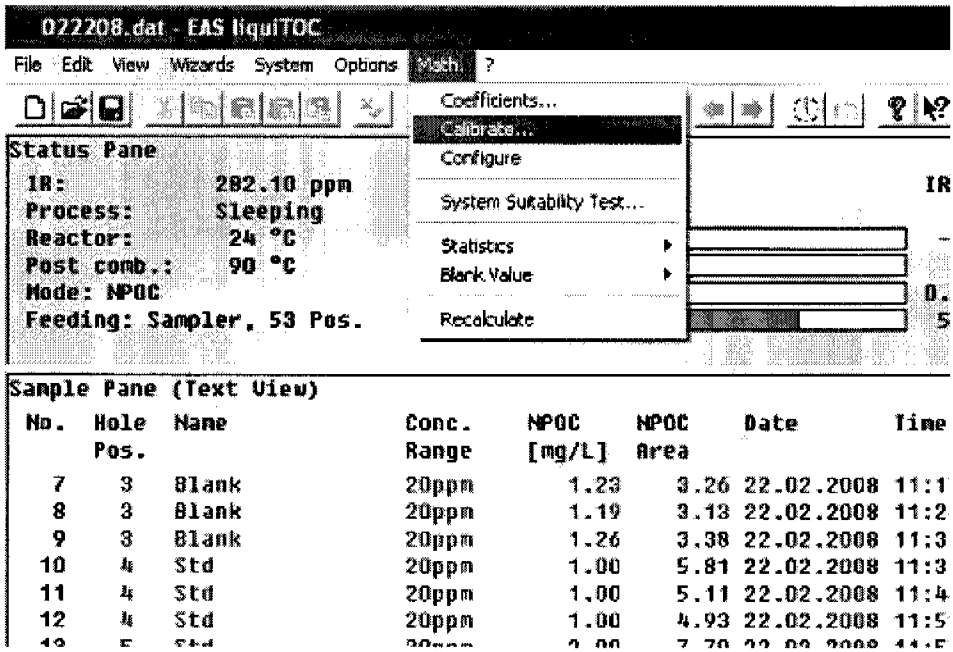


FIGURE B

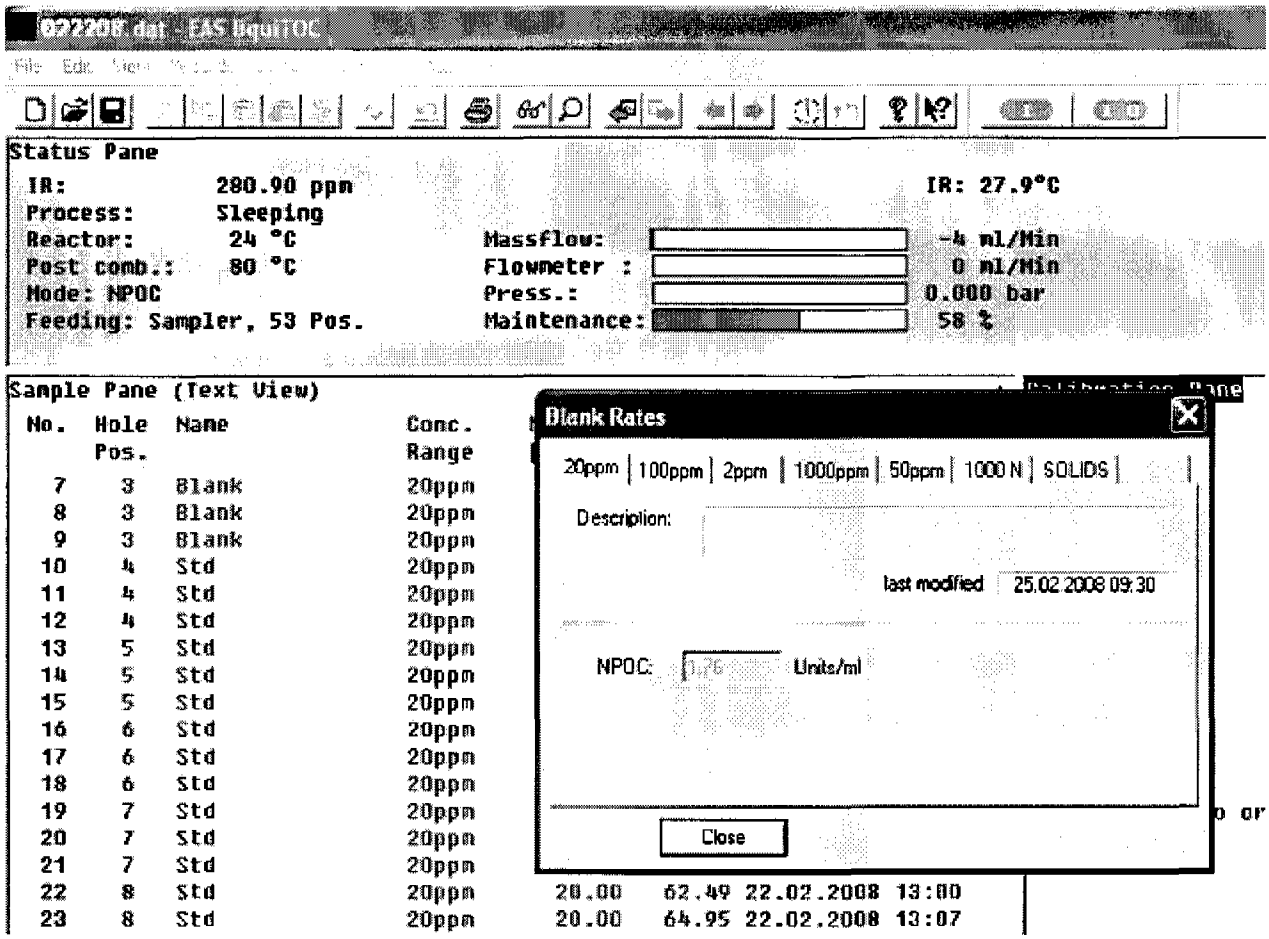

Status Pane
IR: 280.90 ppm
Process: Sleeping
Reactor: 24 °C
Post comb.: 80 °C
Mode: NPOC
Feeding: Sampler, 53 Pos.
Massflow: -4 ml/Min
Flowmeter : 0 ml/Min
Press.: 0.000 bar
Maintenance: 58 %
IR: 27.9°C
Sample Pane (Text View)
No. Hole Pos. Name Conc. Range
7 3 Blank 20ppm
8 3 Blank 20ppm
9 3 Blank 20ppm
10 4 Std 20ppm
11 4 Std 20ppm
12 4 Std 20ppm
13 5 Std 20ppm
14 5 Std 20ppm
15 5 Std 20ppm
16 6 Std 20ppm
17 6 Std 20ppm
18 6 Std 20ppm
19 7 Std 20ppm
20 7 Std 20ppm
21 7 Std 20ppm
22 8 Std 20ppm 20.00 62.49 22.02.2008 13:00
23 8 Std 20ppm 20.00 64.95 22.02.2008 13:07
Blank Rates
20ppm | 100ppm | 2ppm | 1000ppm | 50ppm | 1000 N | SOLIDS
Description:
last modified 25.02.2008 09:30
NPOC: Units/ml
Close

FIGURE C

022208.dat - EAS liquiTOC

File Edit View Wizards System Options Math. ?

Status Pane

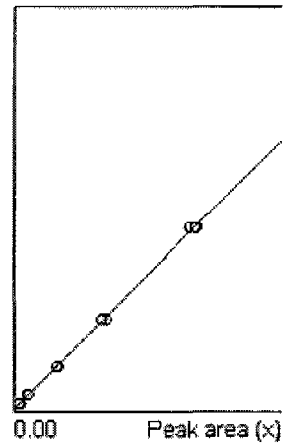
IR: 281.80 ppm — IR: 28.0°C
Process: Sleeping
Reactor: 24 °C — Massflow: -4 ml/Min
Post comb.: 74 °C — Flowmeter : 0 ml/Min
Mode: NPOC — Press.: 0.000 bar
Feeding: Sampler, 53 Pos. — Maintenance: 58 %

Sample Pane (Text View)

No.	Hole Pos.	Name	Conc. Range	NPOC [mg/L]	NPOC Area	Date	Time
7	3	Blank	20ppm	1.23	3.26	22.02.2008	11:17
8	3	Blank	20ppm	1.19	3.13	22.02.2008	11:24
9	3	Blank	20ppm	1.26	3.38	22.02.2008	11:31
10	4	Std	20ppm	1.00	5.81	22.02.2008	11:37
11	4	Std	20ppm	1.00	5.11	22.02.2008	11:44
12	4	Std	20ppm	1.00	4.93	22.02.2008	11:51
13	5	Std	20ppm	2.00	7.70	22.02.2008	11:57
14	5	Std	20ppm	2.00	8.32	22.02.2008	12:04
15	5	Std	20ppm	2.00	7.97	22.02.2008	12:11
16	6	Std	20ppm	5.00	18.31	22.02.2008	12:18
17	6	Std	20ppm	5.00	17.30	22.02.2008	12:25
18	6	Std	20ppm	5.00	17.45	22.02.2008	12:32
19	7	Std	20ppm	10.00	32.96	22.02.2008	12:39
20	7	Std	20ppm	10.00	34.22	22.02.2008	12:46
21	7	Std	20ppm	10.00	32.80	22.02.2008	12:53
22	8	Std	20ppm	20.00	62.49	22.02.2008	13:00
23	8	Std	20ppm	20.00	64.95	22.02.2008	13:07
24	8	Std	20ppm	20.00	64.38	22.02.2008	13:13
25	9	Std	20ppm	40.00	123.51	22.02.2008	13:20
26	9	Std	20ppm	40.00	126.37	22.02.2008	13:27
27	9	Std	20ppm	40.00	127.06	22.02.2008	13:34
28	10	ZZ ICV 23.4	20ppm	24.87	81.85	22.02.2008	13:41
29	10	ZZ ICV 23.4	20ppm	24.33	80.08	22.02.2008	13:48
30	10	ZZ ICV 23.4	20ppm	24.26	79.87	22.02.2008	13:55
31	11	ZZ ICB	20ppm	1.65	6.17	22.02.2008	14:01
32	11	ZZ ICB	20ppm	0.66	2.95	22.02.2008	14:08
33	11	ZZ ICB	20ppm	0.85	3.56	22.02.2008	14:15

Calibration Pane

NPOC Calibration, '20ppm'

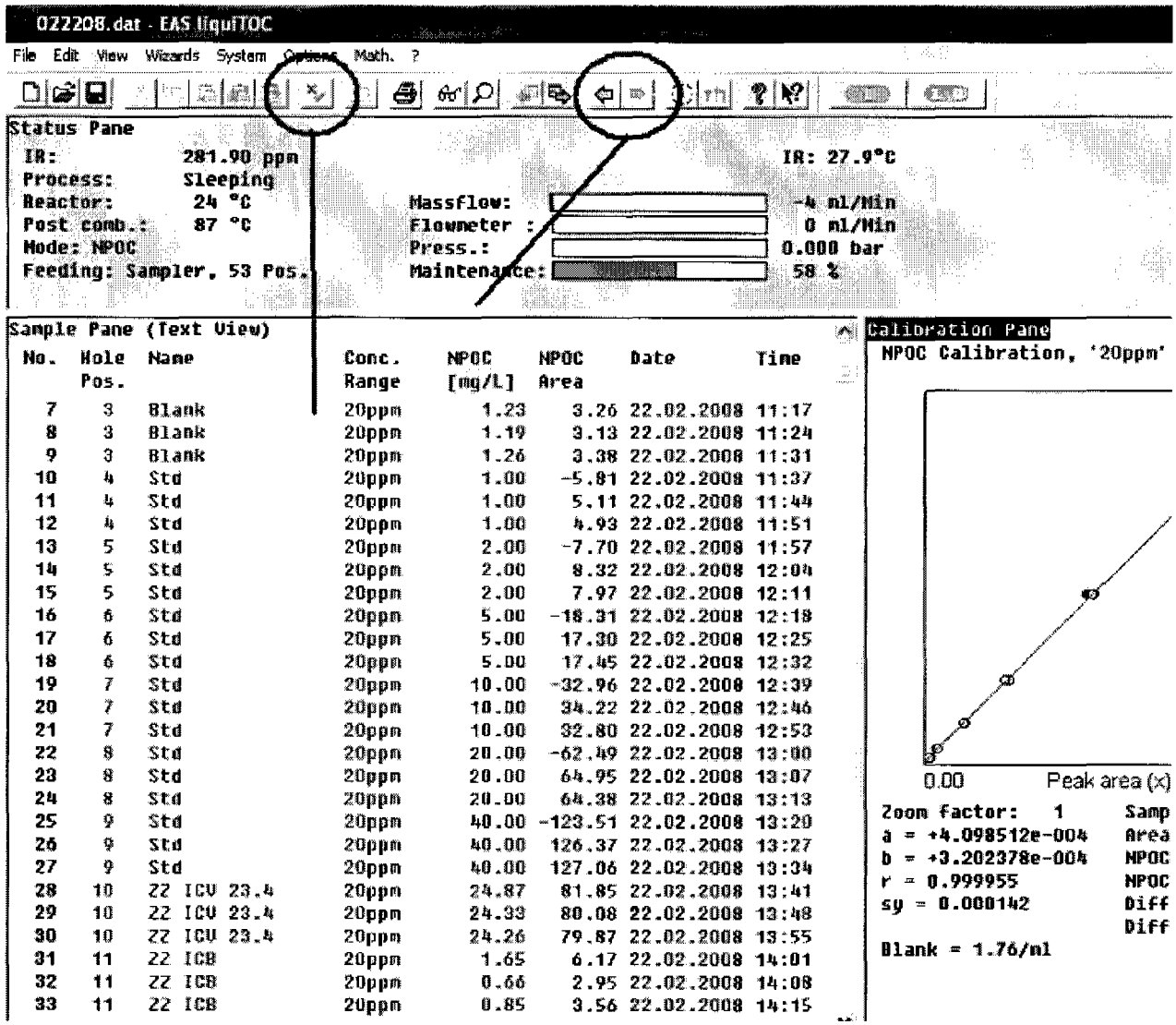


Zoom factor: 1
a = +3.610893e-004
b = +3.236045e-004
r = 0.999790
sy = 0.000297

Blank = 1.76/ml

10.1.16.2 Using the arrow button in the menu bar, click once to move to the first data point of the three for the blank. Click the "exclude" (red x) button to exclude this data point. (Policy is to exclude the first of the three replicates done for each analytical sample.) Refer to Figure D.

FIGURE D



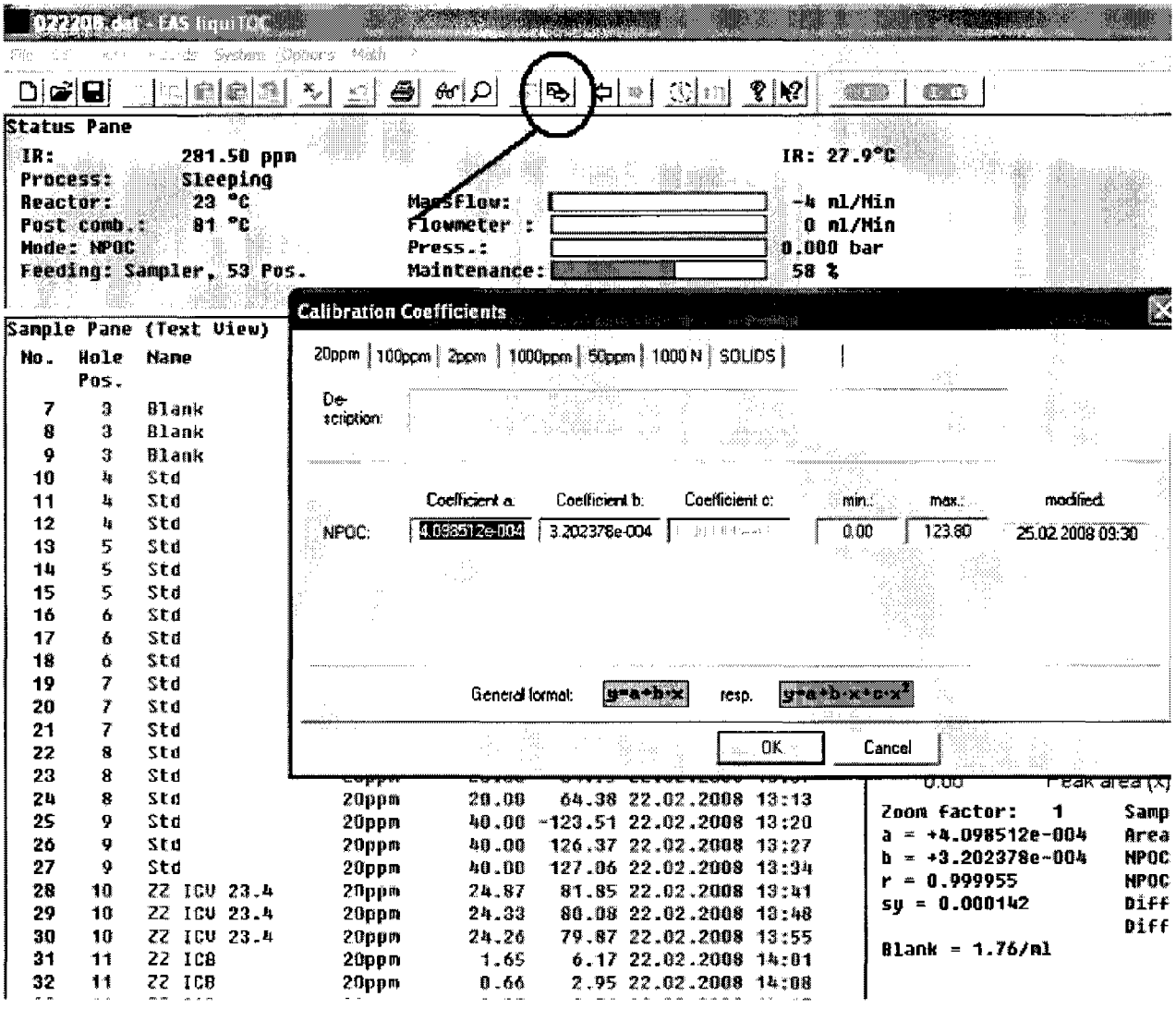
10.1.17 Click the arrow button three times to move to the first of the next set of data points (for the first working standard). Click the exclude button. Continue this process until the first of each triplicate for each working curve standard has been excluded. Excluded samples will have their data point appear in solid red on the curve graph.

10.1.18 Determine if the acceptance criteria of R ≥0.995 by examining the chart data. If acceptable, continue; if not, re-calibrate.

10.1.19 Print the acceptable working standard curve by clicking on the print icon button in the menu bar. A window will appear asking you to title the data: enter the date in format of "MM/DD/YY CURVE" changing the day, month and year to the current one on the date of analysis.

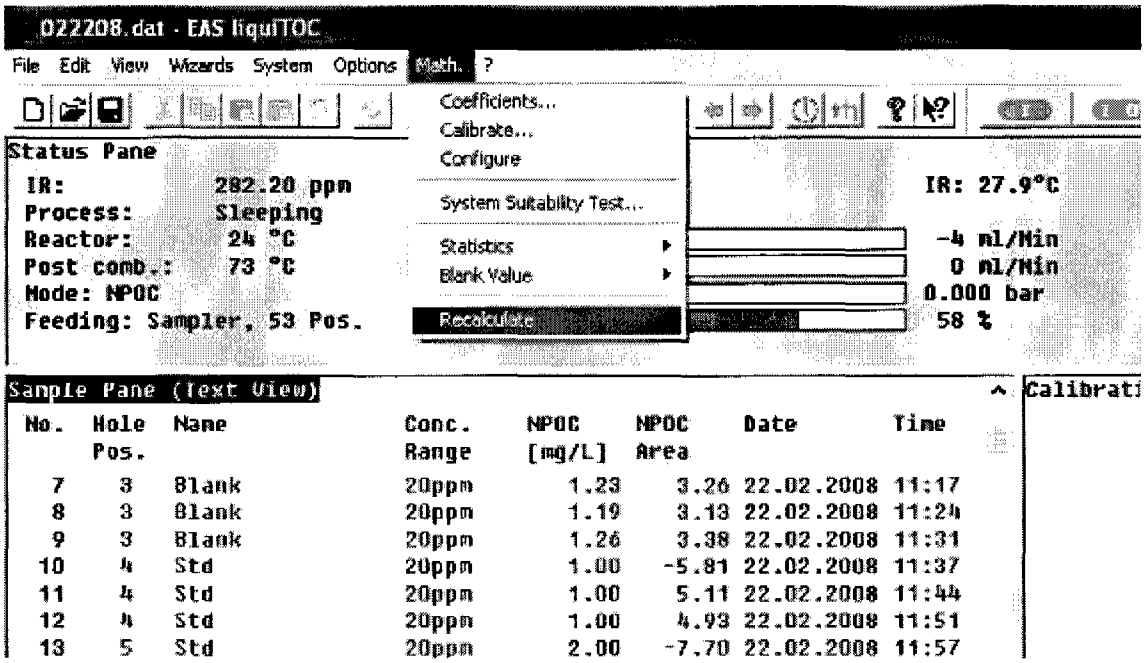
10.1.20 Finally, to accept the curve and continue, click on the icon to forward the page (in the menu bar; circled in red in Figure E below) and click on "OK" to lock the calibration into the system.

FIGURE E



10.1.21 Click into the left Sample Pane (Text View) window (the words should be highlighted with a blue bar). Referring to Figure F, select Math→Recalculate in order to update the working curve for the data analyzed so far (the head-end QC of ICV, ICB, MB and LCS). Make sure that the QC is all within acceptable range for each respective QC sample. (If not, refer to the troubleshooting section.)

FIGURE F



10.1.22 Since the system has stopped on the end of calibration and has gone to sleep, select Options→Wake Up to put the system back in operating mode.

10.1.23 Next change the stop tag to the last line of the analytical run. After that, and once the system is awake and is stable, the green "I" button will turn from gray to green; click on the green "I" button in the menu bar to re-start the analytical run in continuous mode with the first investigative sample.

10.1.24 After the analysis of the samples, the system will go to sleep automatically.

10.1.25 Evaluate and approve the data as follows:

10.1.25.1 In the graphics pane, select Math→Statistics→By Name to present the run data, and click on "Yes" when warned about overwriting data. Refer to Figure G and H.

FIGURE G

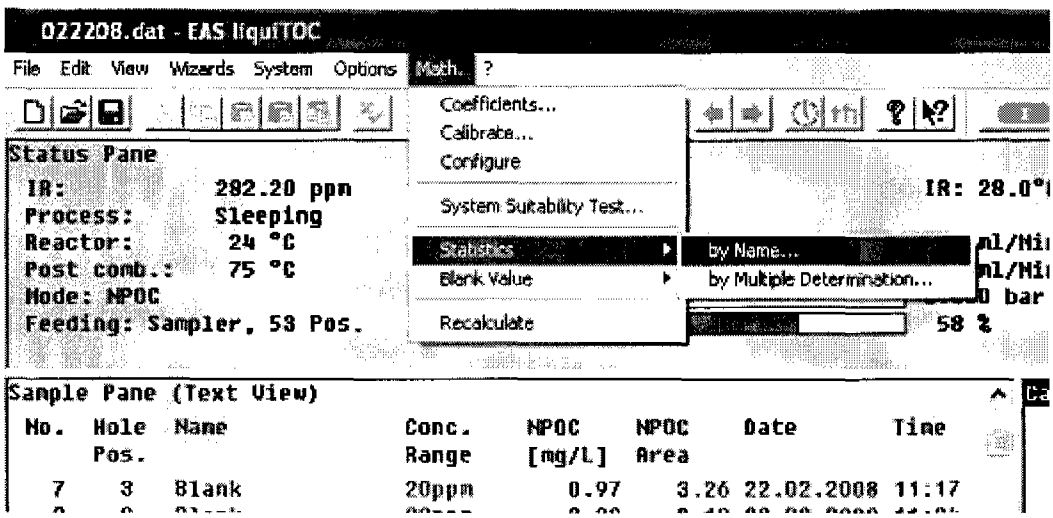
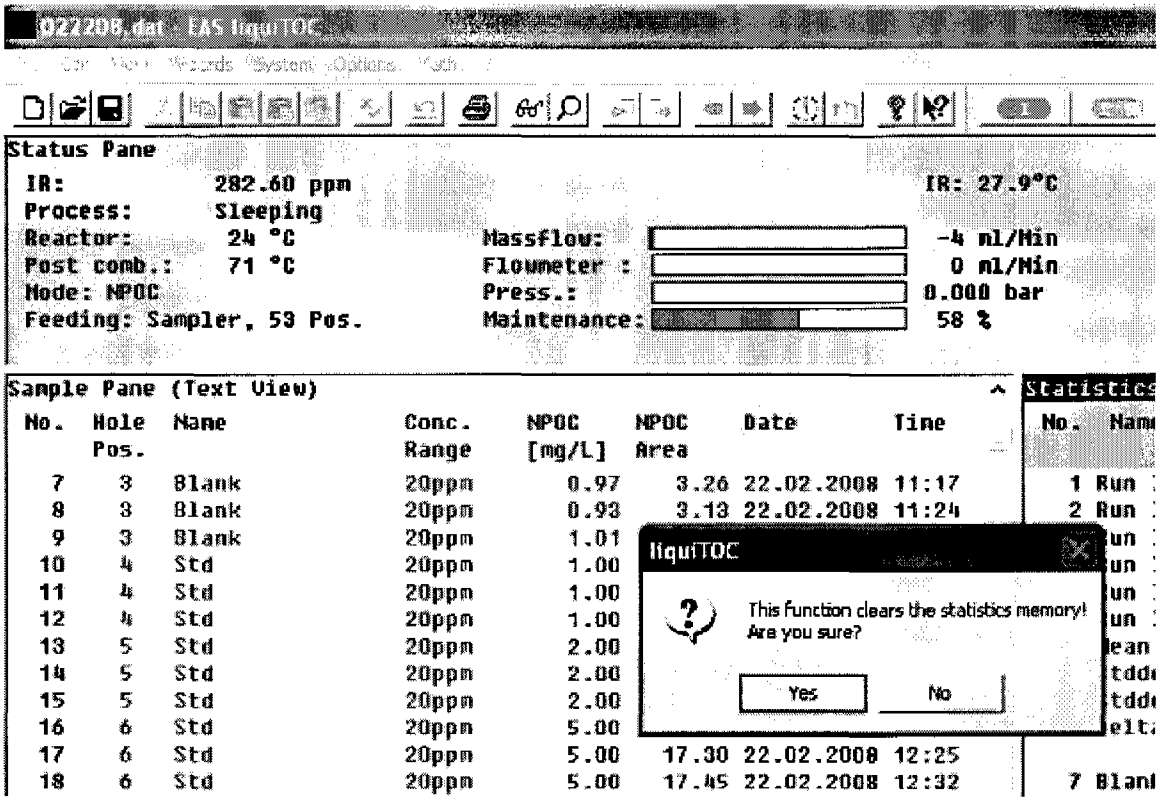
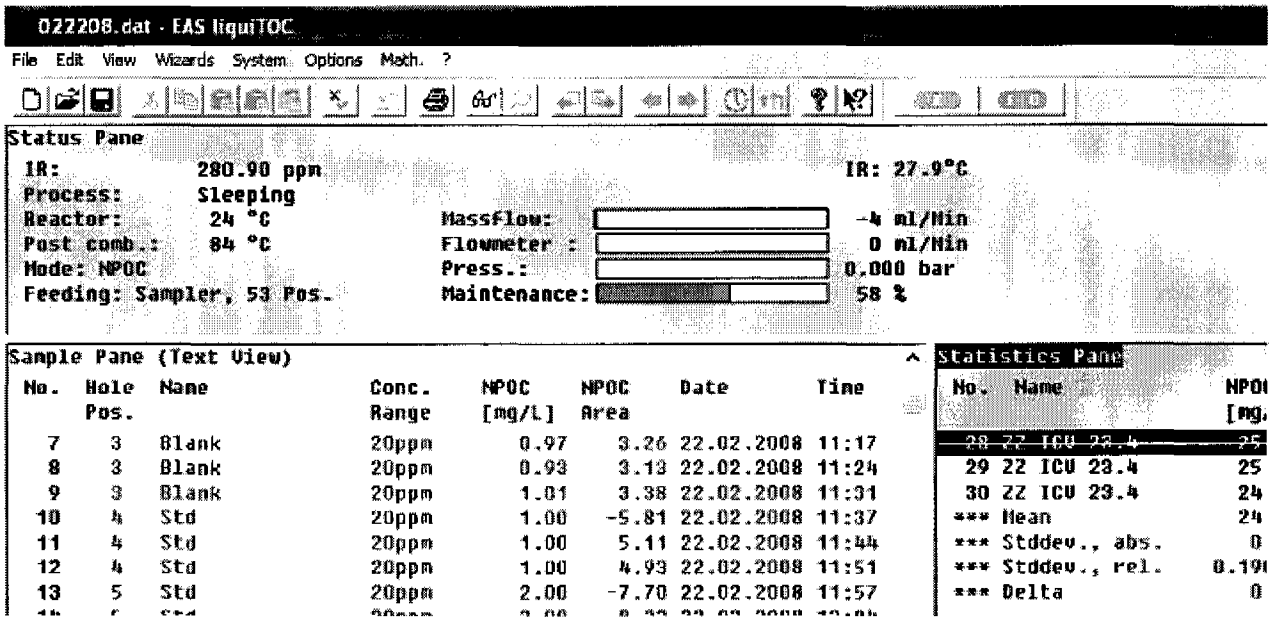


FIGURE H



10.1.25.2 Click once on the first of the three readings for each QC and investigative sample, in turn, and then click on the small icon button and a red X to exclude that data point. Since a common analytical run includes both high, and low TOC content samples, we use the first of the triplicate analytical data as a "clean out" and thus this data is always discarded. Refer to Figure I.

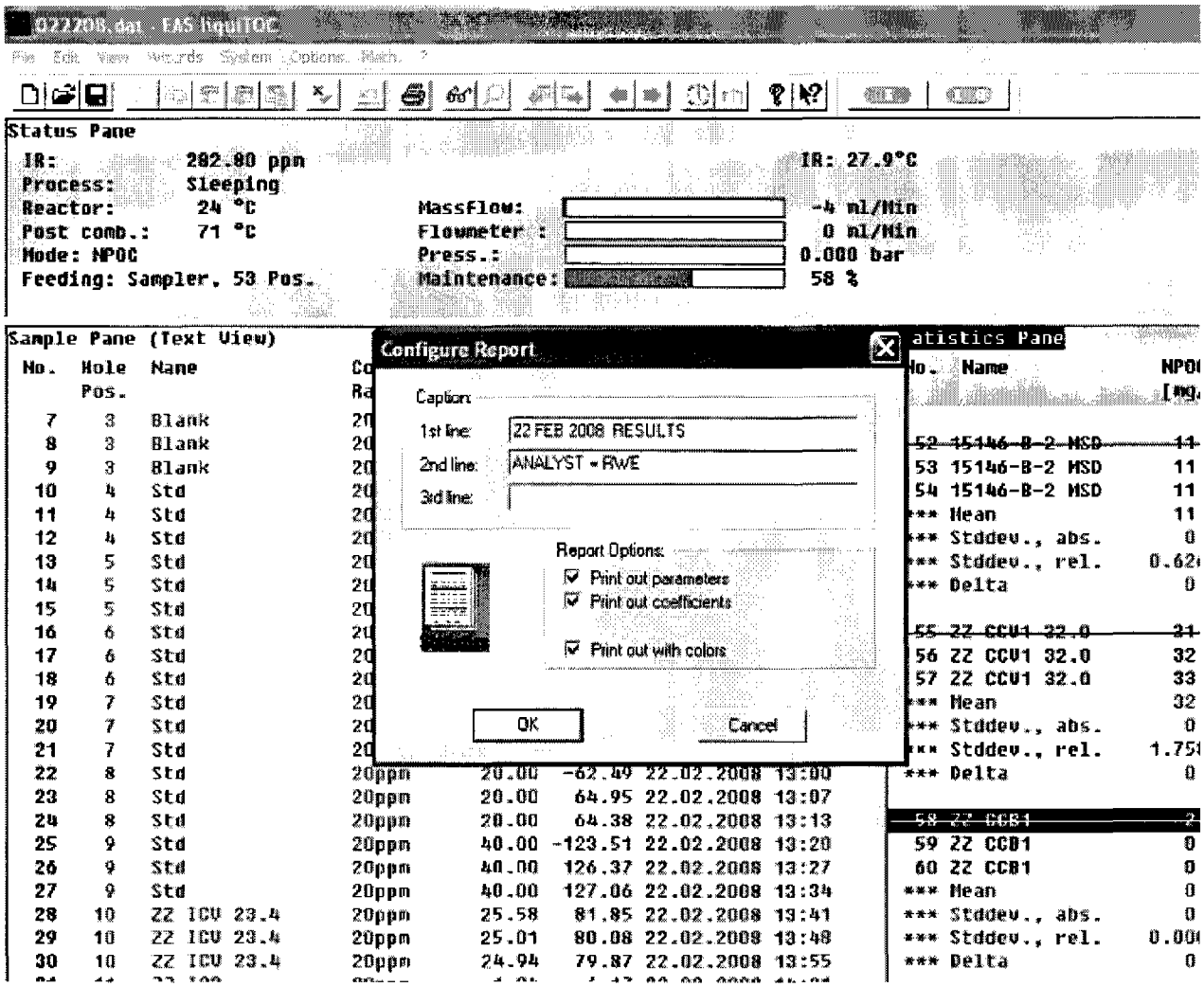
FIGURE I



10.1.25.3 As a result of clicking on the exclude button for the first of each data point for all samples, the mean value for each sample will be recalculated and displayed automatically.

10.1.25.4 Print the data by clicking on the print icon. A window will appear asking you to title the data: enter "MM/DD/YY RESULTS." Refer to Figure J,

FIGURE J



10.1.25.5 Click once in the text pane.

10.1.25.6 Print this run summary as well by clicking on the print icon. A window will appear asking you to title the data: enter "MM/DD/YY RUN LOG."

10.1.25.7 Click on the Computer Disk icon to save all analytical data to the filename already chosen earlier.

10.1.26 Because the liquiTOC is so stable, one working curve can be generated for a week's worth of analyses. Simply start off each batch with an ICV, ICB/MB, and LCS; if the data meets QC criteria, the working curve stored in the system is viable. If not, rework the batch to make a new calibration curve as described herein.

10.2 Total Carbon (TC) and/or Total Inorganic Carbon (TIC)

10.2.1 To determine Total Carbon, follow steps 10.1 to 10.1.26 but **do not add hydrochloric acid** to the samples; the samples must be received unpreserved and analyzed unpreserved.

10.2.1.1 Samples that contain noticeable turbidity or suspended solids should not be filtered. For such samples, insert a small telfon-coating stir bar (ones reserved for TC analysis) into each PTFE sample cup so that the autosampler will stir and continually homogenize the sample during sample uptake by the instrument.

10.2.2 To determine Total Inorganic Carbon (TIC), run the sample for both TC and TOC. The difference between the two is TIC as follows:

$$TIC = TC - TOC$$

10.3 Total Carbon (TC) and Total Organic Carbon (TOC) in Soil or Solid Material

10.3.1 Analysis of solid samples is done on the same instrument, but with a different set up that requires manual feeding of samples, one at a time. After the system has been allowed to cool to room temperature (put into sleep mode, allowed to run with the cooling fans for 30min, and then turned off), the aqueous reaction chamber may be swapped out for the solids reaction chamber and associated apparatus. Refer to the manufacturer's instrument manual for how to install the solids analysis equipment.

10.3.2 Pre-Treatment of Samples

10.3.2.1 Solid samples for TOC must be pre-treated. Place about 5g of material into a 25mL beaker. Grind as needed to break up large chunks. Add just enough deionized water to cover the solid material, and then add 12 drops of concentrated HCl. Using a stainless steel spatula, mix the solid, water and acid.

10.3.2.2 Heat the mixture in an oven set at 103°C overnight so that it fully dries. Cool the dried material to room temperature in a desiccator.

10.3.3 Prior to sample analysis, grind the solid into powder (or as close to that consistency as possible) using the flat of a stainless steel scoopula or spatula.

10.4 Differences in Methods

10.4.1 Both the Lloyd Kahn and EPA 9060 methods are identical in terms or sample preparation and physical analysis. They do differ in terms of replicates and reporting of data, as follows:

10.4.1.1 For the 9060 method, all samples (including QC) are run in quadruplicate. The individual results for each burn of the sample and the average is reported.

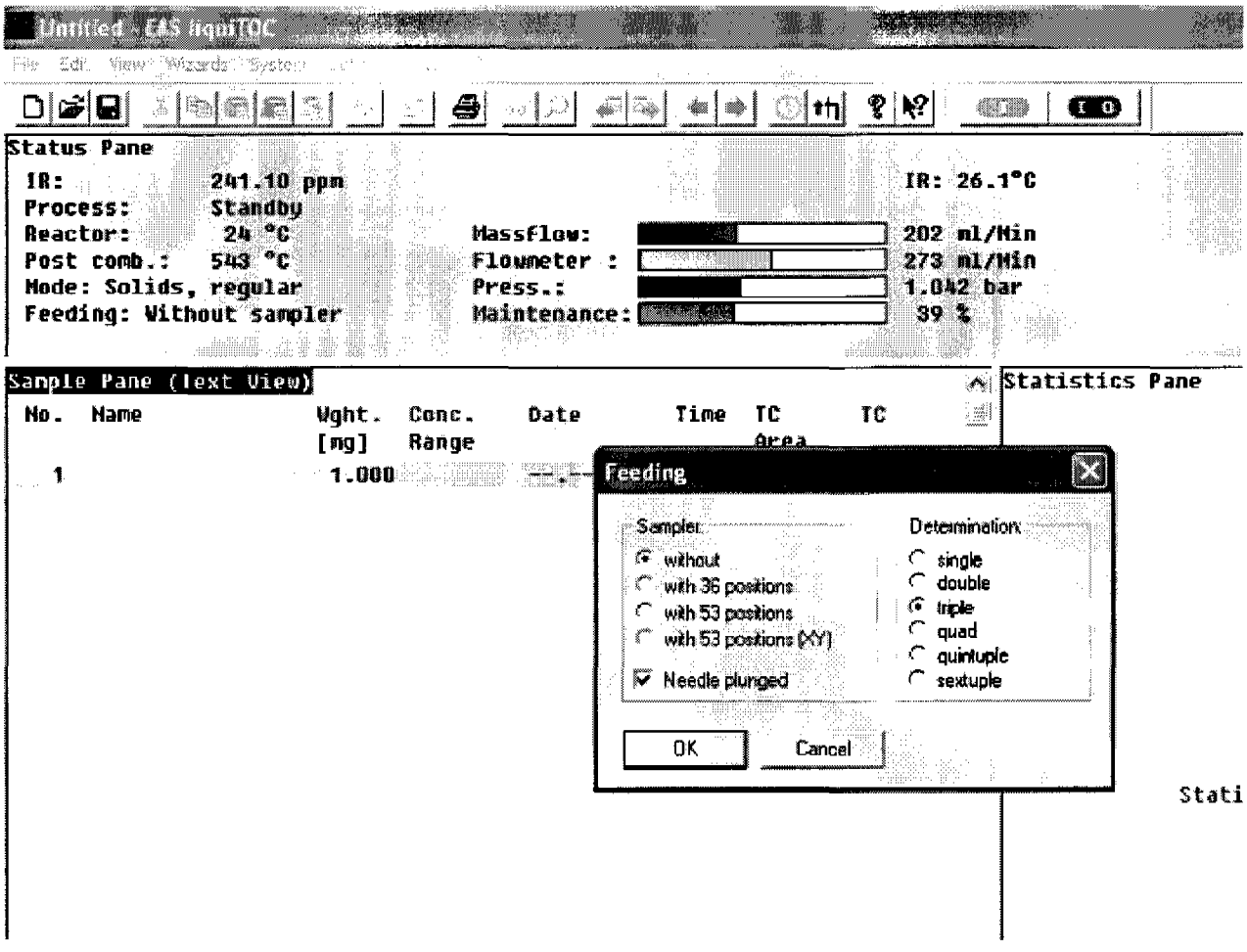
10.4.1.2 For the Lloyd Kahn method, all samples (including QC) are run in duplicate. Only the average value for the two analytical burns is reported.

10.5 Set-up and Analysis of Solid Samples

10.5.1 Change out the aqueous set-up for the solids set-up. **CAUTION! Parts may be hot!**

10.5.2 Launch the software as described for water analysis above. Once the Sample Pane window appears, select System → Feeding to bring up the autosampler Feeding window as shown in Figure 14.

FIGURE 14



10.5.3 Select "without" to turn the autosampler off. Click the "OK" button to exit. The autosampler will then go through an initialization for about 45 seconds after which time you may proceed to the next step.

10.5.4 Select System → Mode and click "Yes" in the next window to proceed to the Analysis Mode window as shown in Figure 15 and 16.

FIGURE 15

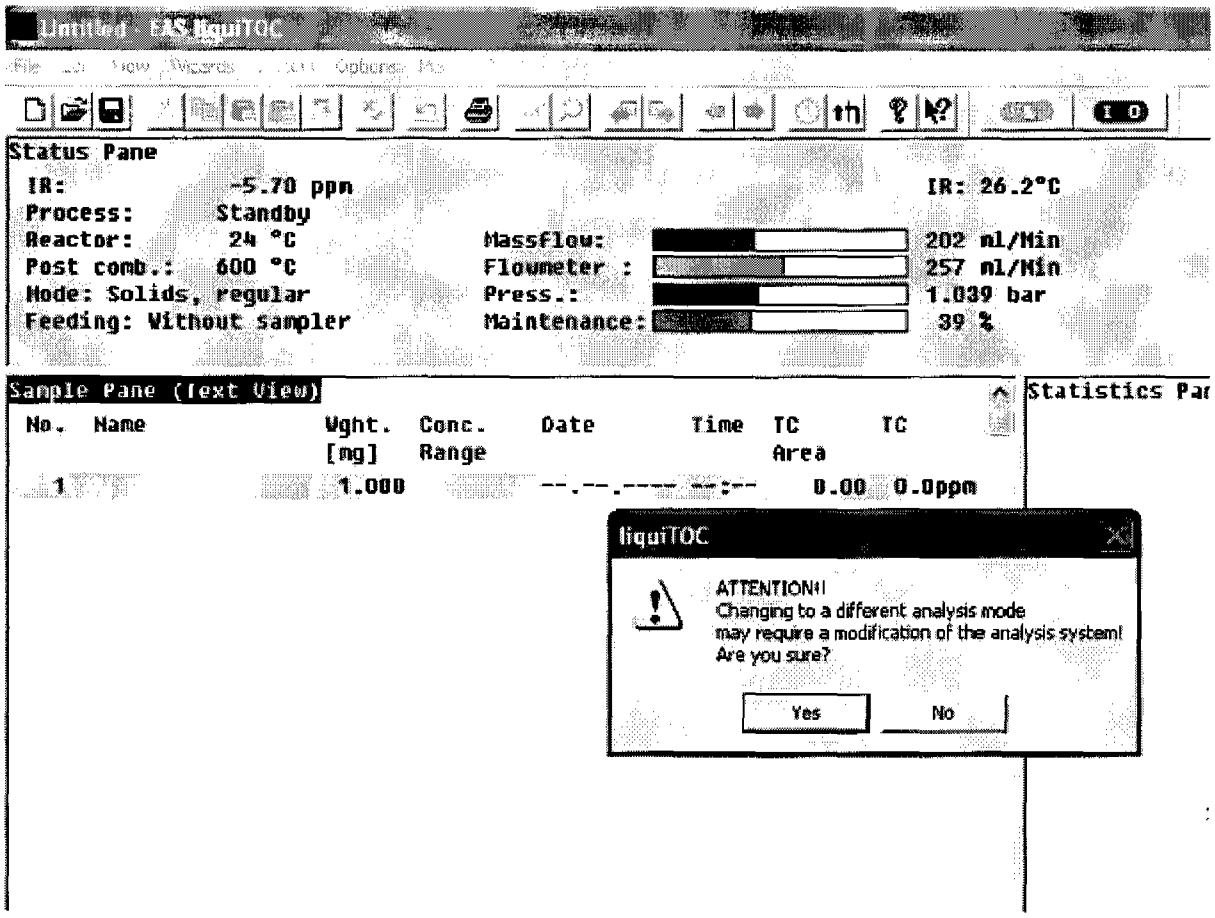
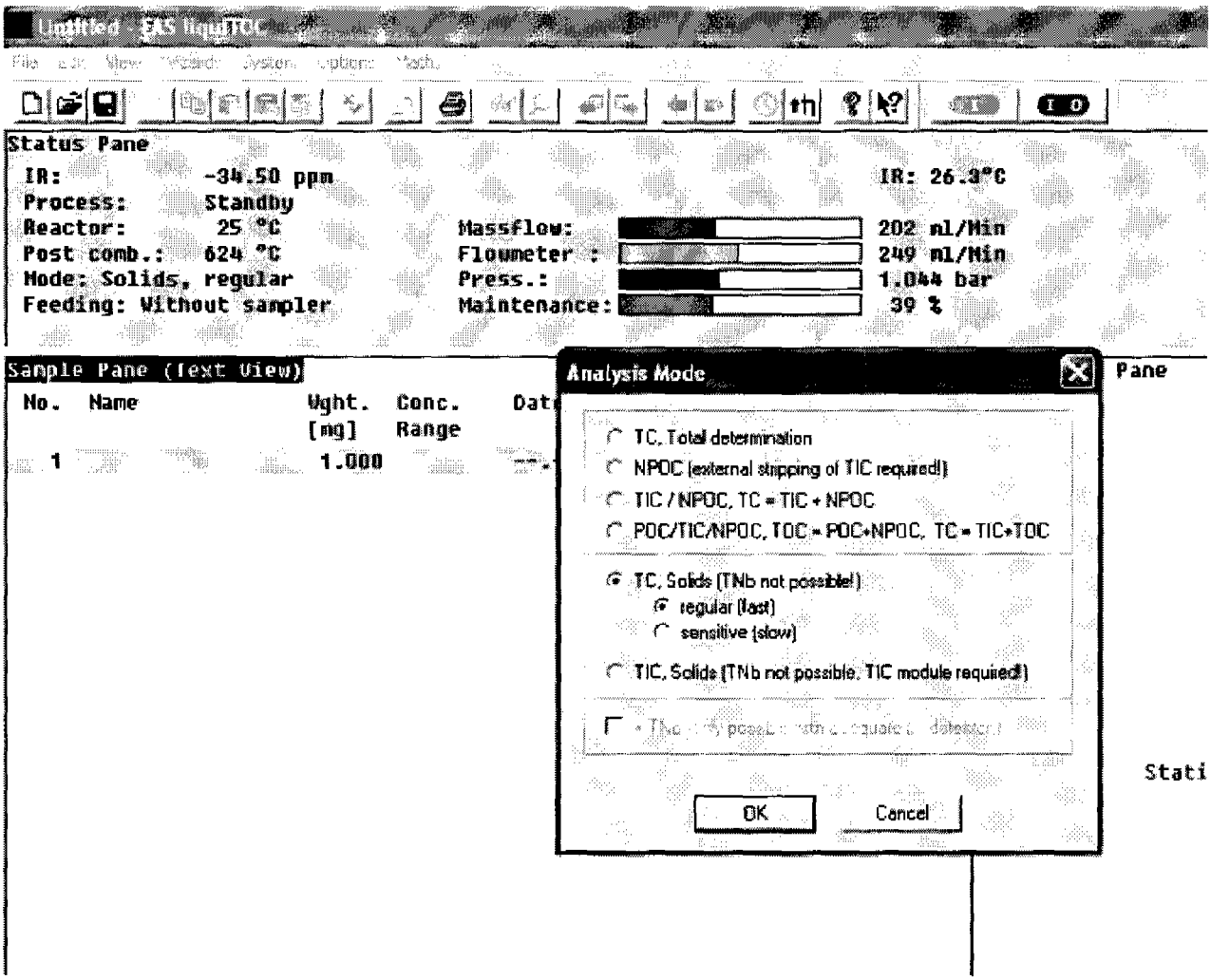

Untitled - EAS liquiTOC
Status Pane
IR: -5.70 ppm
IR: 26.2°C
Process: Standby
Reactor: 24 °C
Post comb.: 600 °C
Mode: Solids, regular
Feeding: Without sampler
Massflow: 202 ml/Min
Flowmeter : 257 ml/Min
Press.: 1.039 bar
Maintenance: 39 %
Sample Pane (Text View)
Statistics Par
No. Name Wght. [mg] Conc. Range Date Time TC Area TC
1 1.000 --.--.---- --:-- 0.00 0.0ppm
liquiTOC
ATTENTION!!
Changing to a different analysis mode
may require a modification of the analysis system!
Are you sure?
Yes
No

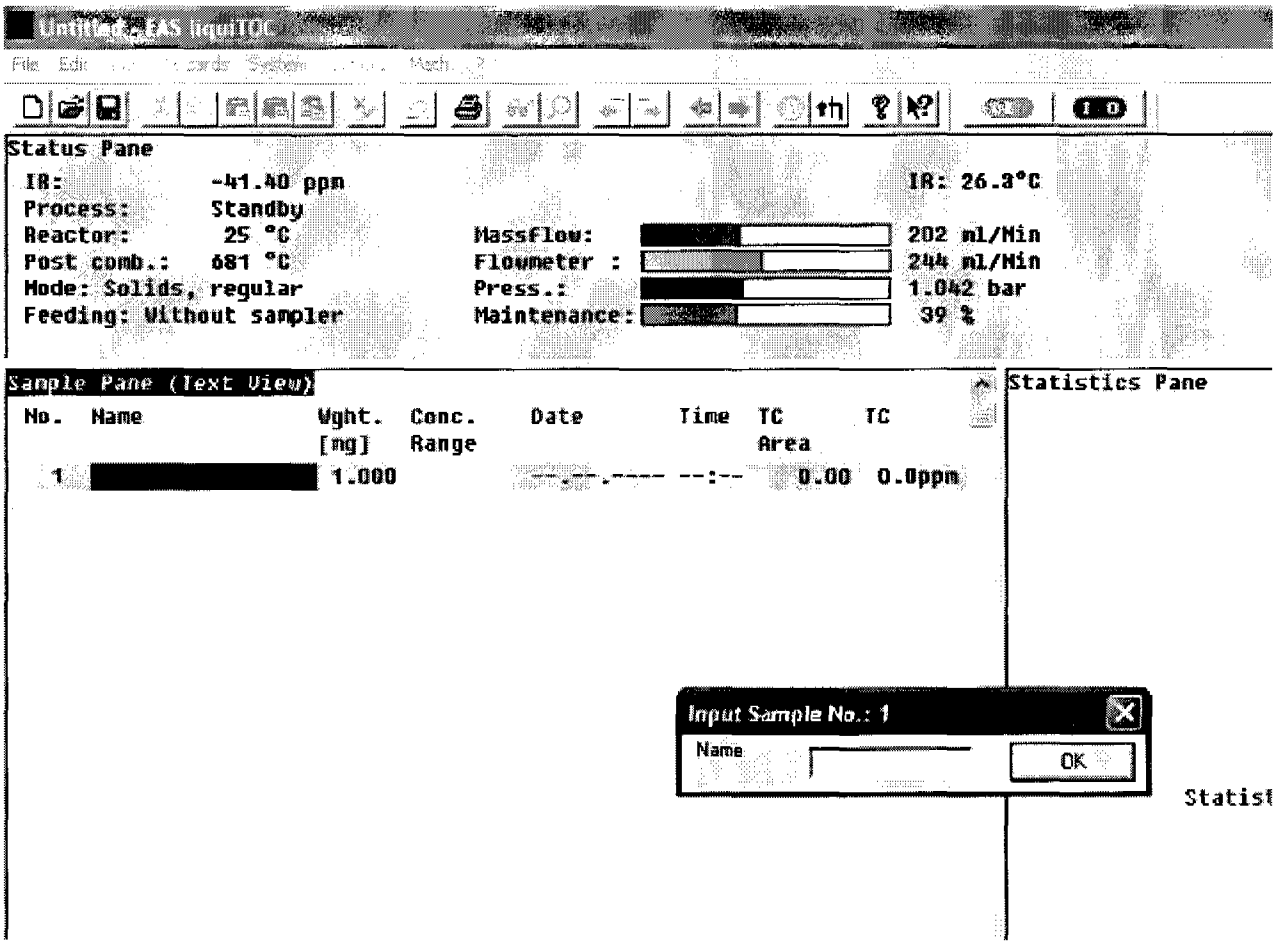
FIGURE 16



10.5.5 Select "TC, Solids" from the menu and "regular (fast)" by clicking on the radio buttons. Click "OK" to save the changes. The instrument will now cycle through an initialization and once complete, will return to the Sample Pane.

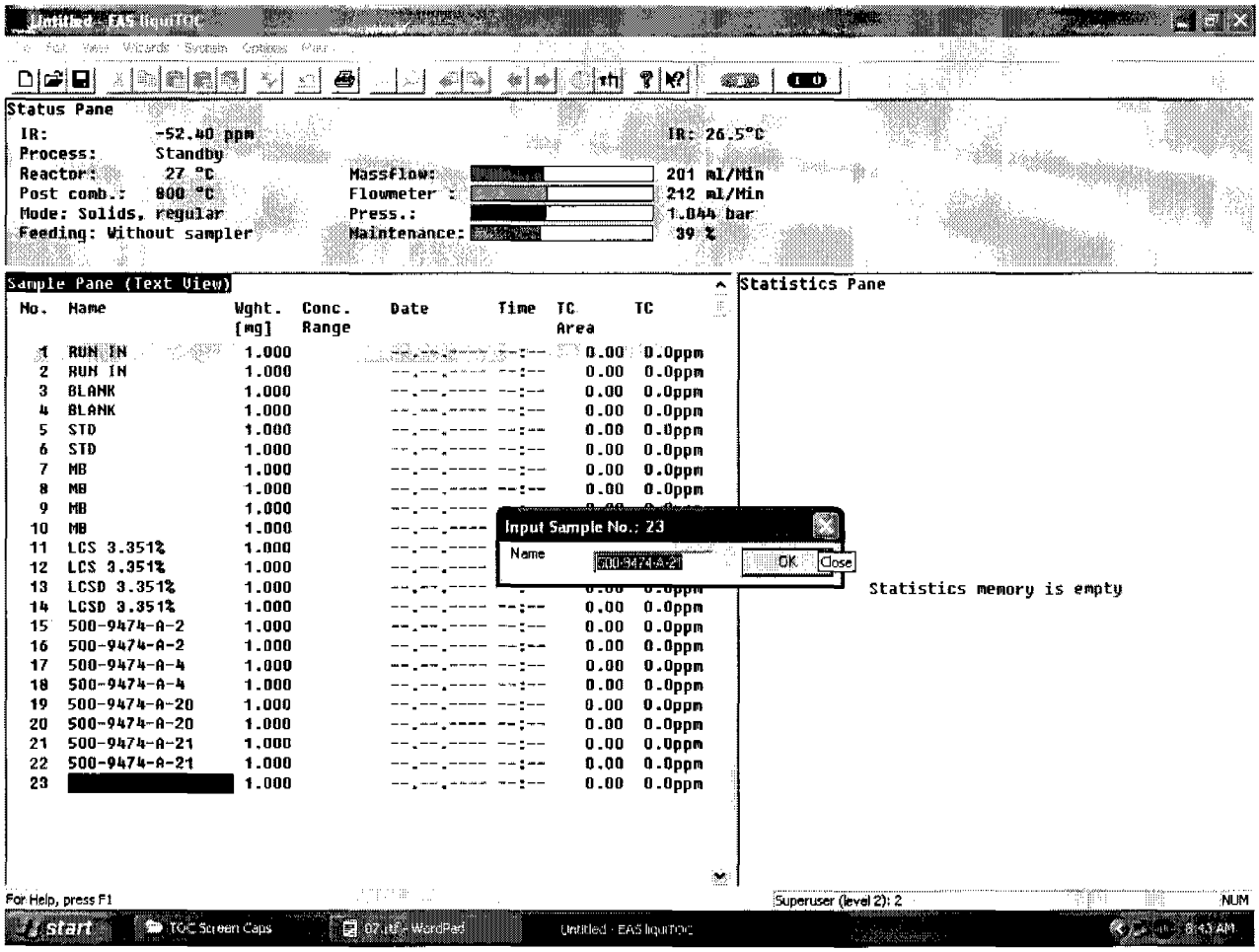
10.5.6 Double click in the "Name" field of the first line to enter the names for the QC and investigative samples. Doing so brings up the window as shown in Figure 17.

FIGURE 17



10.5.7 Start with two "RUN IN" samples followed by two "Blank" samples. (Run in samples are used to simply clean out the cuvettes using the heating program.) Follow this with two "Std" samples, and then MB, LCS, LCSD and investigative samples. For Lloyd Kahn, enter each sample twice; for 9060 enter each sample four times. Refer to Figure 18 (showing a Lloyd Kahn run).

FIGURE 18



10.5.8 Once the samples are entered, double click in the "Conc. Range" field for the first line. From the drop down menu in the new window, select "SOLID" and click "OK." This will fill in the SOLID method for all samples in the batch and instruct the instrument to look for a manual input of each sample in turn. Refer to Figure 19 and 20.

FIGURE 19

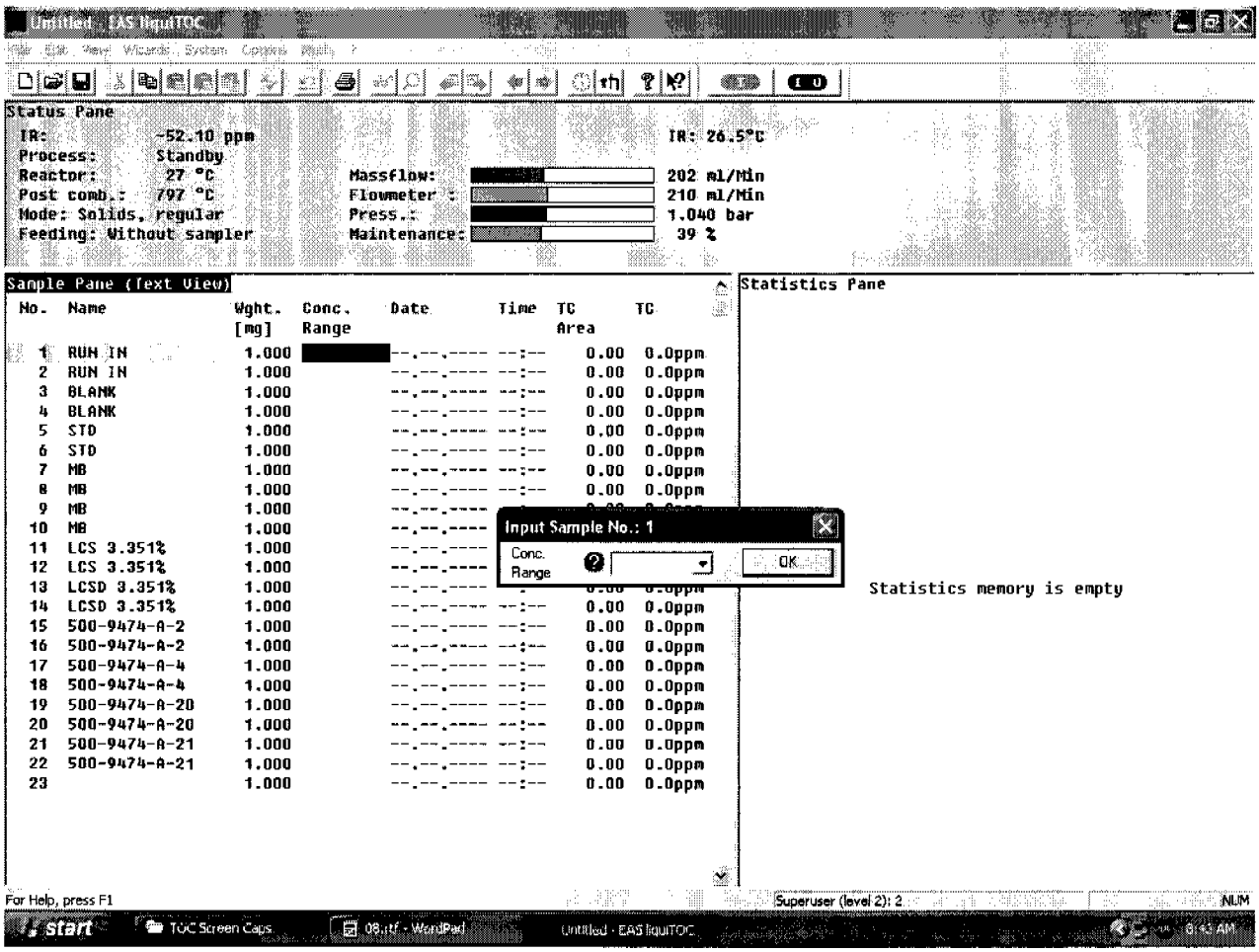
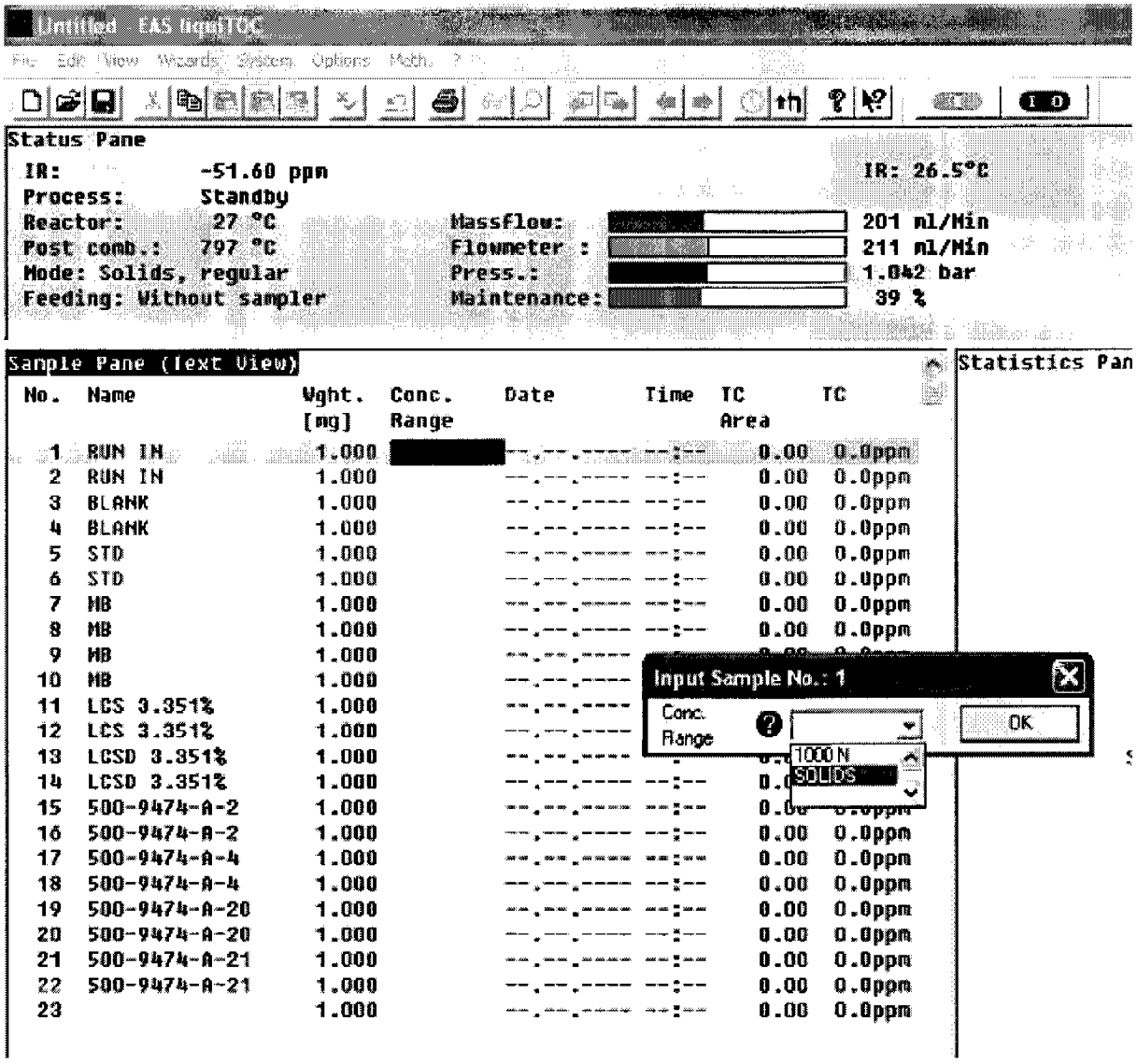
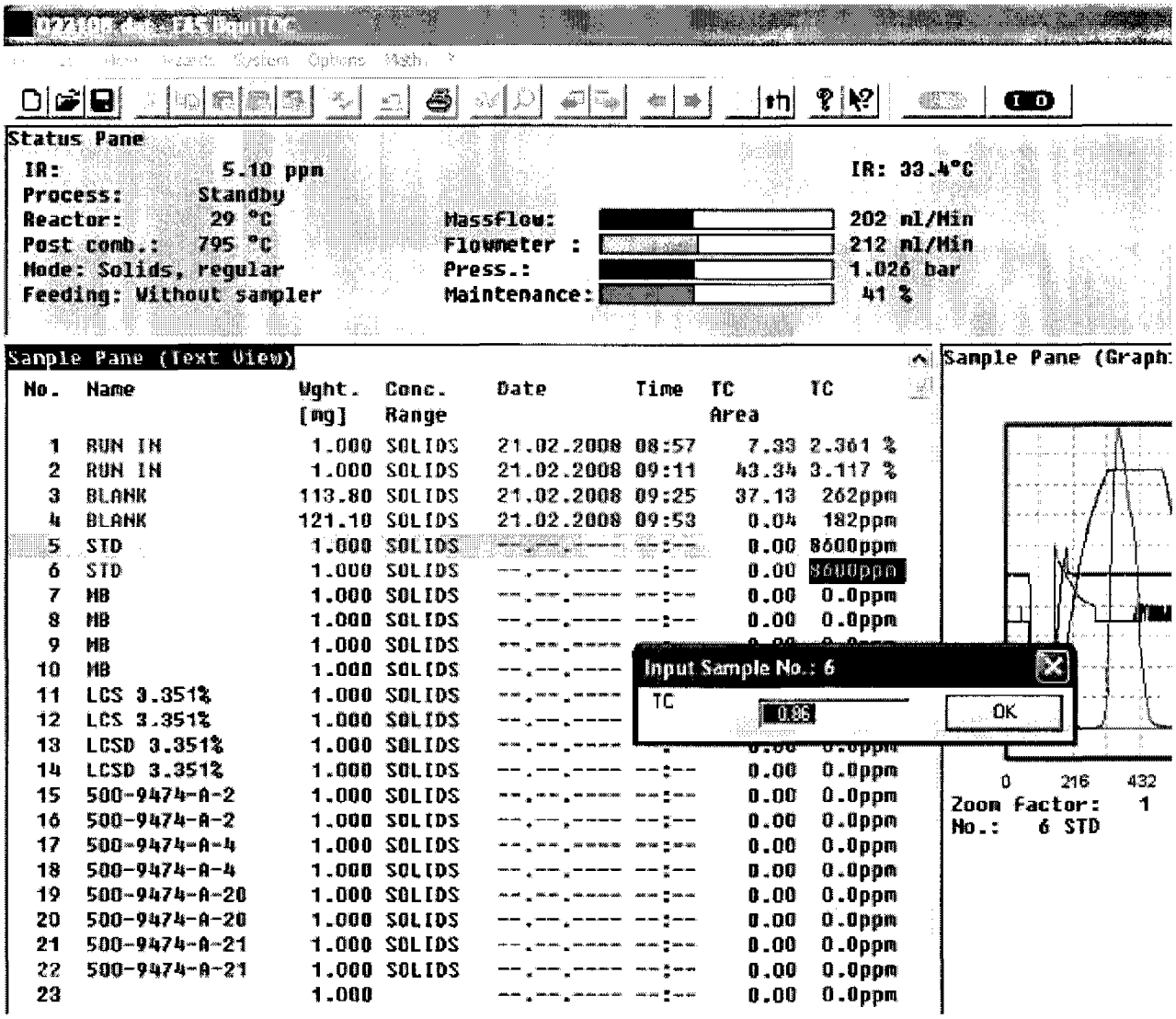

Untitled - EAS liquiTOC
Status Pane
IR: -52.10 ppm
Process: Standby
Reactor: 27 °C
Post comb.: 797 °C
Mode: Solids, regular
Feeding: Without sampler
Massflow: 202 ml/Min
Flowmeter: 210 ml/Min
Press.: 1.040 bar
Maintenance: 39 %
IR: 26.5°C
Sample Pane (Text View)
No. Name Wght. [mg] Conc. Range Date Time TC Area TC
1 RUN IN 1.000 --.--.---- --:-- 0.00 0.0ppm
2 RUN IN 1.000 --.--.---- --:-- 0.00 0.0ppm
3 BLANK 1.000 --.--.---- --:-- 0.00 0.0ppm
4 BLANK 1.000 --.--.---- --:-- 0.00 0.0ppm
5 STD 1.000 --.--.---- --:-- 0.00 0.0ppm
6 STD 1.000 --.--.---- --:-- 0.00 0.0ppm
7 MB 1.000 --.--.---- --:-- 0.00 0.0ppm
8 MB 1.000 --.--.---- --:-- 0.00 0.0ppm
9 MB 1.000 --.--.----
10 MB 1.000 --.--.----
11 LCS 3.351% 1.000 --.--.----
12 LCS 3.351% 1.000 --.--.----
13 LCSD 3.351% 1.000 --.--.----
14 LCSD 3.351% 1.000 --.--.---- --:-- 0.00 0.0ppm
15 500-9474-A-2 1.000 --.--.---- --:-- 0.00 0.0ppm
16 500-9474-A-2 1.000 --.--.---- --:-- 0.00 0.0ppm
17 500-9474-A-4 1.000 --.--.---- --:-- 0.00 0.0ppm
18 500-9474-A-4 1.000 --.--.---- --:-- 0.00 0.0ppm
19 500-9474-A-20 1.000 --.--.---- --:-- 0.00 0.0ppm
20 500-9474-A-20 1.000 --.--.---- --:-- 0.00 0.0ppm
21 500-9474-A-21 1.000 --.--.---- --:-- 0.00 0.0ppm
22 500-9474-A-21 1.000 --.--.---- --:-- 0.00 0.0ppm
23 1.000 --.--.---- --:-- 0.00 0.0ppm
Input Sample No.: 1
Conc. Range
OK
Statistics Pane
Statistics memory is empty
For Help, press F1
Superuser (level 2): 2
NUM
start
TOC Screen Caps
08.rtf - WordPad
Untitled - EAS liquiTOC
8:43 AM

FIGURE 20



10.5.9 Finally, enter the base concentration for the soil calibration standard by double clicking on the "0.0ppm" value under the "TC" column for the first of the "STD" (as shown in Figure 20 above this would be Line No. 5). Enter the true concentration for the standard by typing "0.86" and clicking "OK" twice to lock it in for both samples. **NOTE:** The system converts anything under 1% to ppm (10,000ppm = 1%) so it will convert the entry to 8600ppm automatically. Refer to Figure 21.

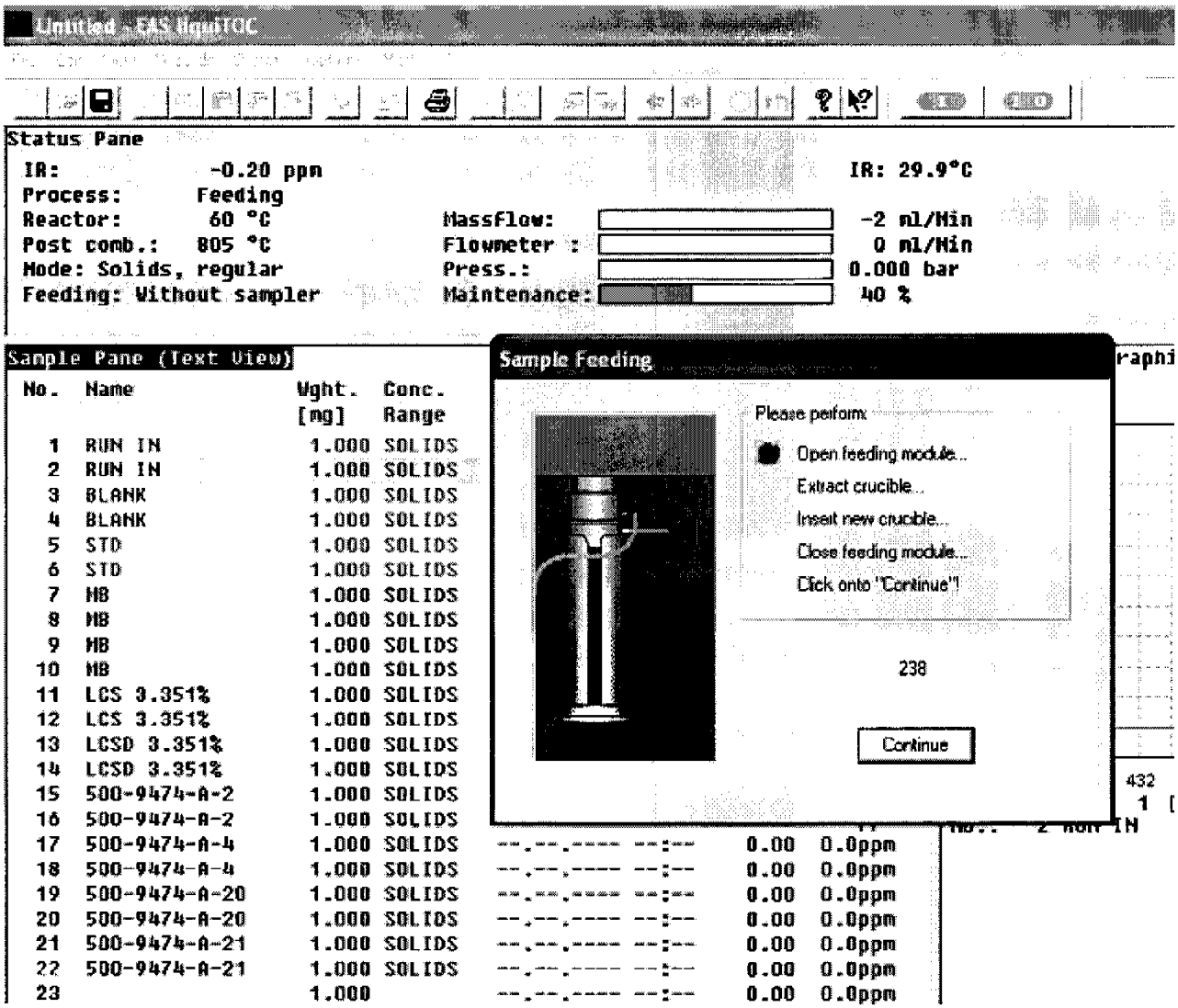
FIGURE 21



10.5.10 RUN IN samples use no sample in the cuvette; it is simply a burn-out run to clean the cuvette. Start the analysis by clicking on the green and red "I/O" button at the top of the page. The system will ask you if you're sure you wish to start a single sample run—click "Yes" to proceed.

10.5.11 The system will begin its pre-heat and baseline phase of the analysis. Once it's time for you to insert the cuvette, an alarm will sound and a graphic will appear on the screen as shown in Figure 22.

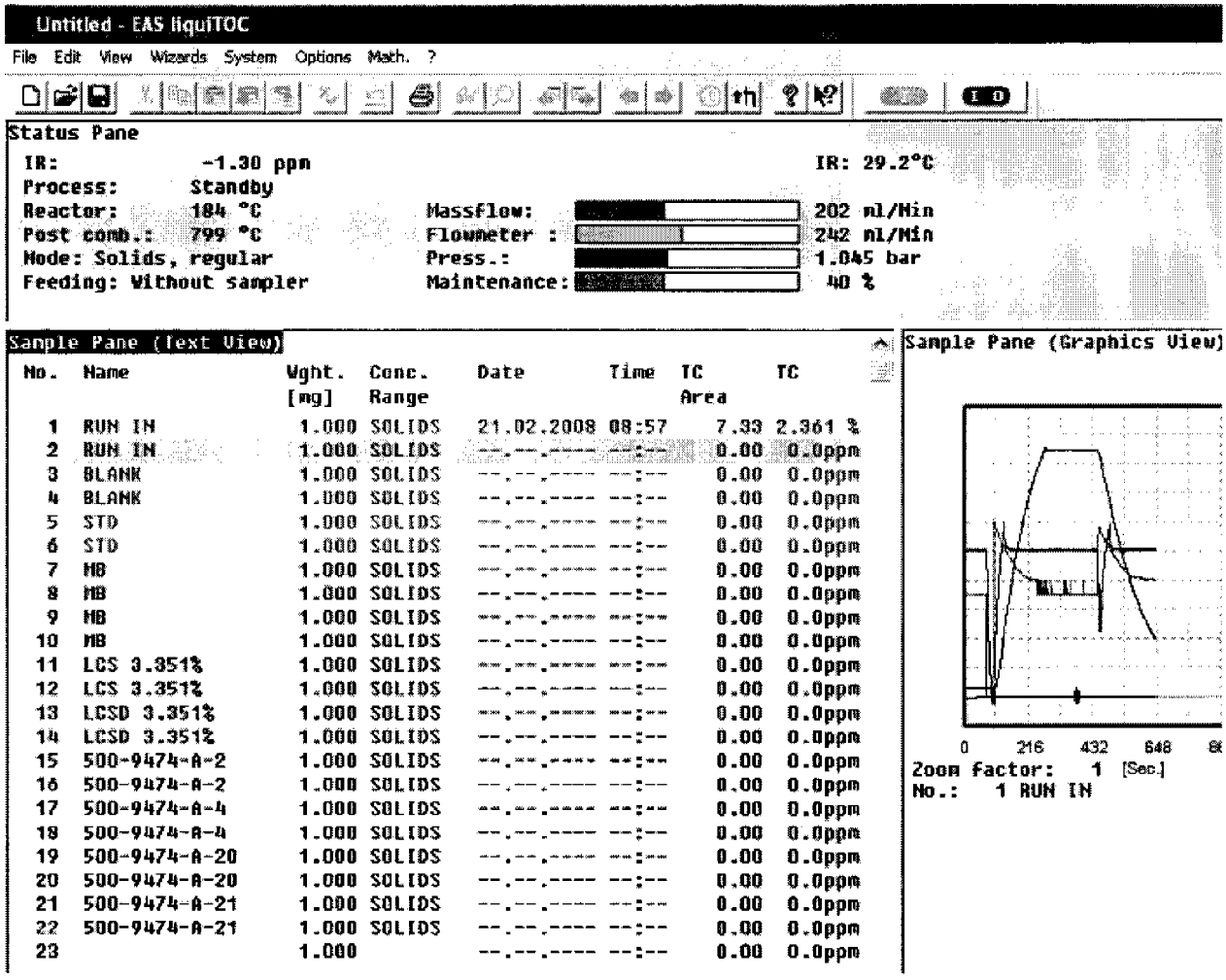
FIGURE 22



10.5.12 Open the front door of the liquiTOC, lower the pedestal, swap out the cuvettes, raise the pedestal back into position, and then close the door and press the "ENTER" key on the keyboard (or click on the "Continue" button).

10.5.13 Once the sample has been analyzed, the data will appear in the left-hand Sample Pane (Text View) and a graphical interpretation of the run will appear on the right. Refer to Figure 23.

FIGURE 23



No.	Name	Wght. [mg]	Conc. Range	Date	Time	TC Area	TC
1	RUN IN	1.000	SOLIDS	21.02.2008	08:57	7.33	2.361 %
2	RUN IN	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
3	BLANK	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
4	BLANK	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
5	STD	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
6	STD	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
7	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
8	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
9	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
10	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
11	LCS 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
12	LCS 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
13	LCSD 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
14	LCSD 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
15	500-9474-A-2	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
16	500-9474-A-2	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
17	500-9474-A-4	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
18	500-9474-A-4	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
19	500-9474-A-20	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
20	500-9474-A-20	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
21	500-9474-A-21	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
22	500-9474-A-21	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
23		1.000		--.--.----	--:--	0.00	0.0ppm

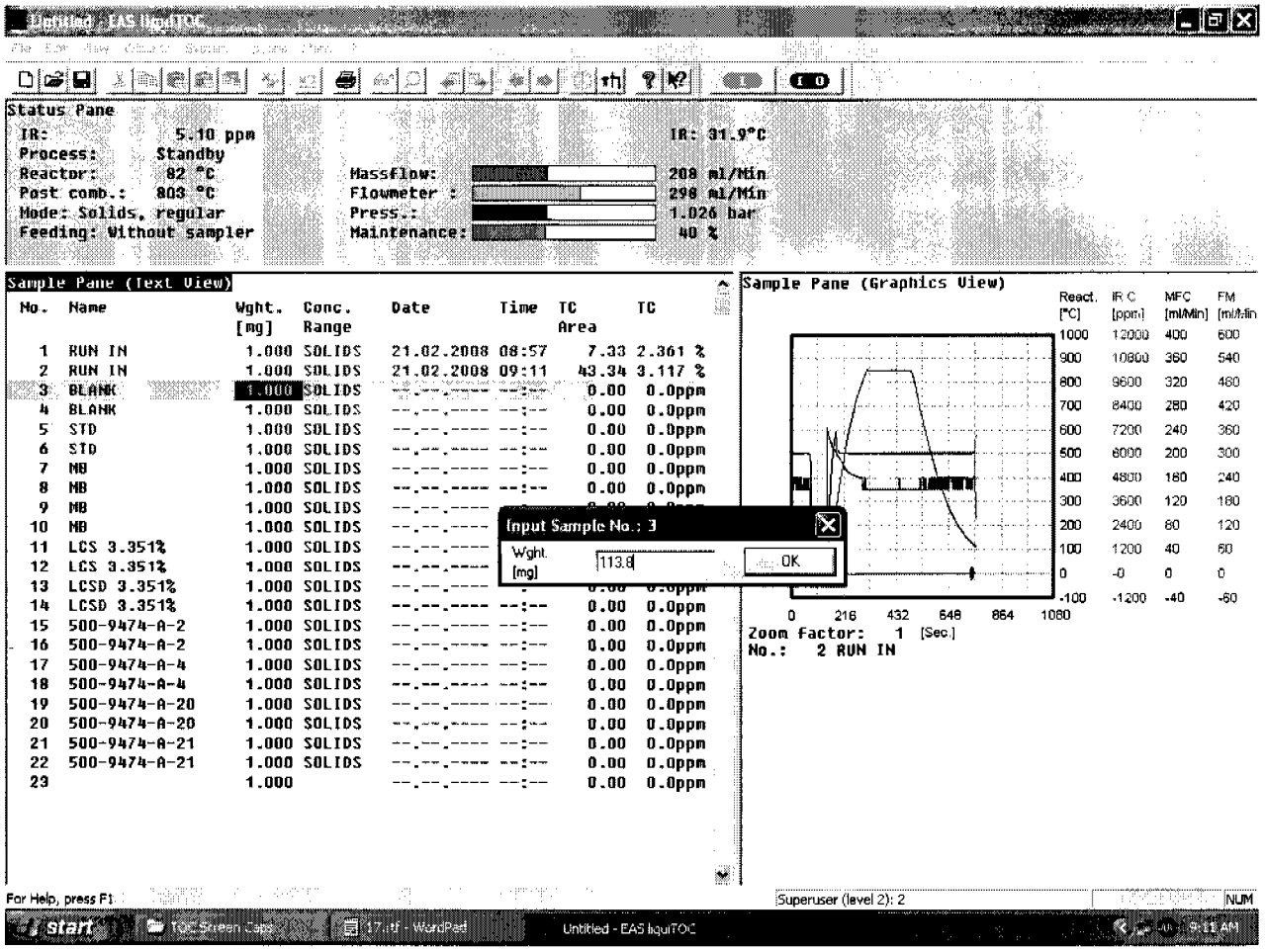
10.5.14 Do a second RUN IN to clean the other cuvette as described above.

10.5.15 For the BLANK samples, we use a blank soil sample that is made up of compositing all our old samples into one container. This material has already gone through the liquiTOC and afterward has been heated at 550°C for an hour to remove all carbon.

10.5.16 Place the cuvette on the analytical balance and tare it. Add about 100mg of blank soil sample to the cuvette, recording the exact weight of the sample. Return with the cuvette to the instrument and temporarily hold the cuvette with sample in the cuvette tray provided.

10.5.17 Double click in the "Wght. [mg]" field for the first Blank sample. The Wght. [mg] window will open; type in the weight recorded and click the "OK" button. Close out the window by clicking on the red "X" in the window. Refer to Figure 24.

FIGURE 24



10.5.18 Click on the green and red "I/O" button to start the next run. Repeat this for the second Blank sample.

10.5.19 For the STD samples, a two-point curve is all that is required pursuant to manufacturer's instructions. The "TC" concentration for the STD has been set to 0.86% (8600ppm) already above. For making a viable curve, use about 25mg for the low STD (Line No. 5) and about 300mg for the high STD. Enter the actual weights for each STD as described above. Refer to Figures 25 and 26.

FIGURE 25

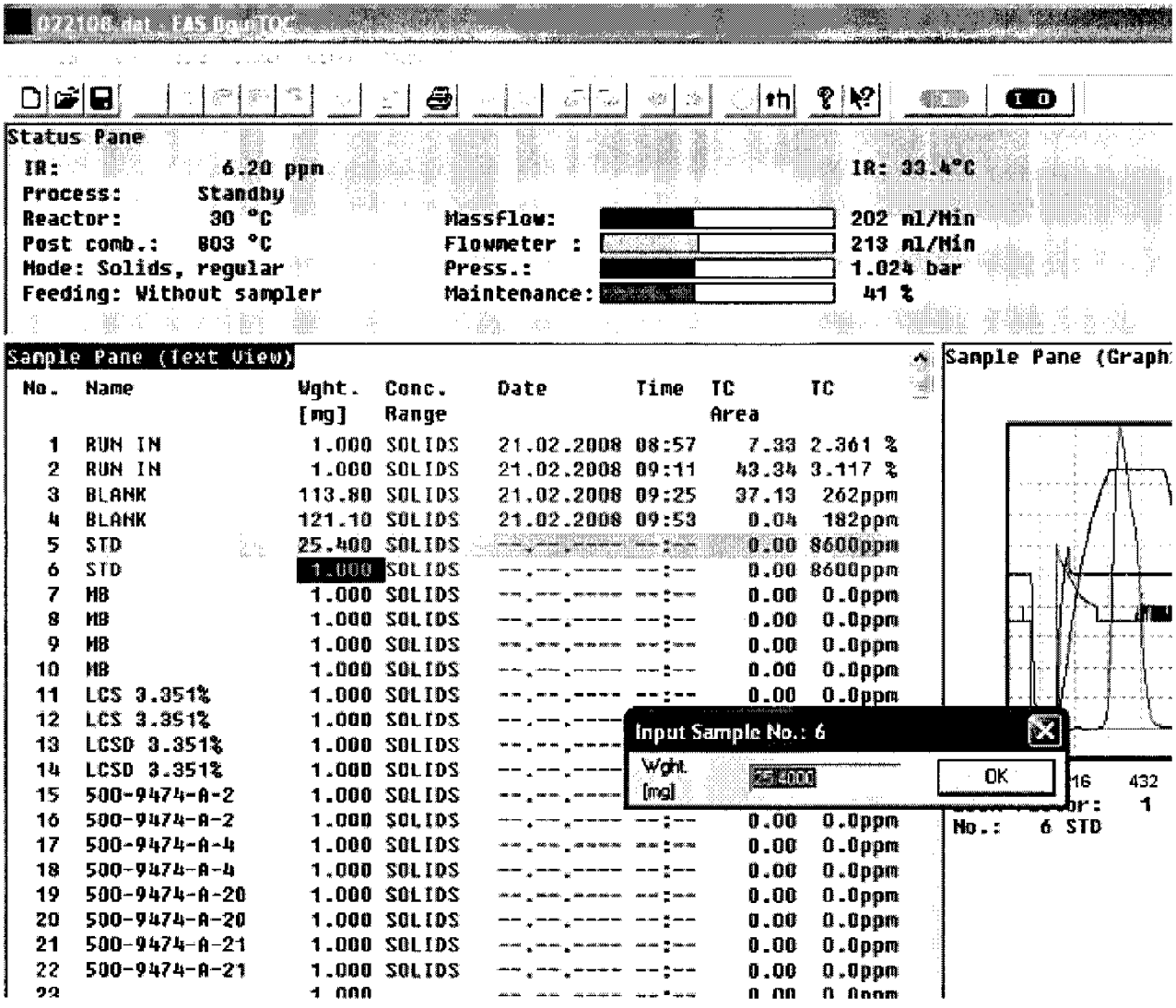
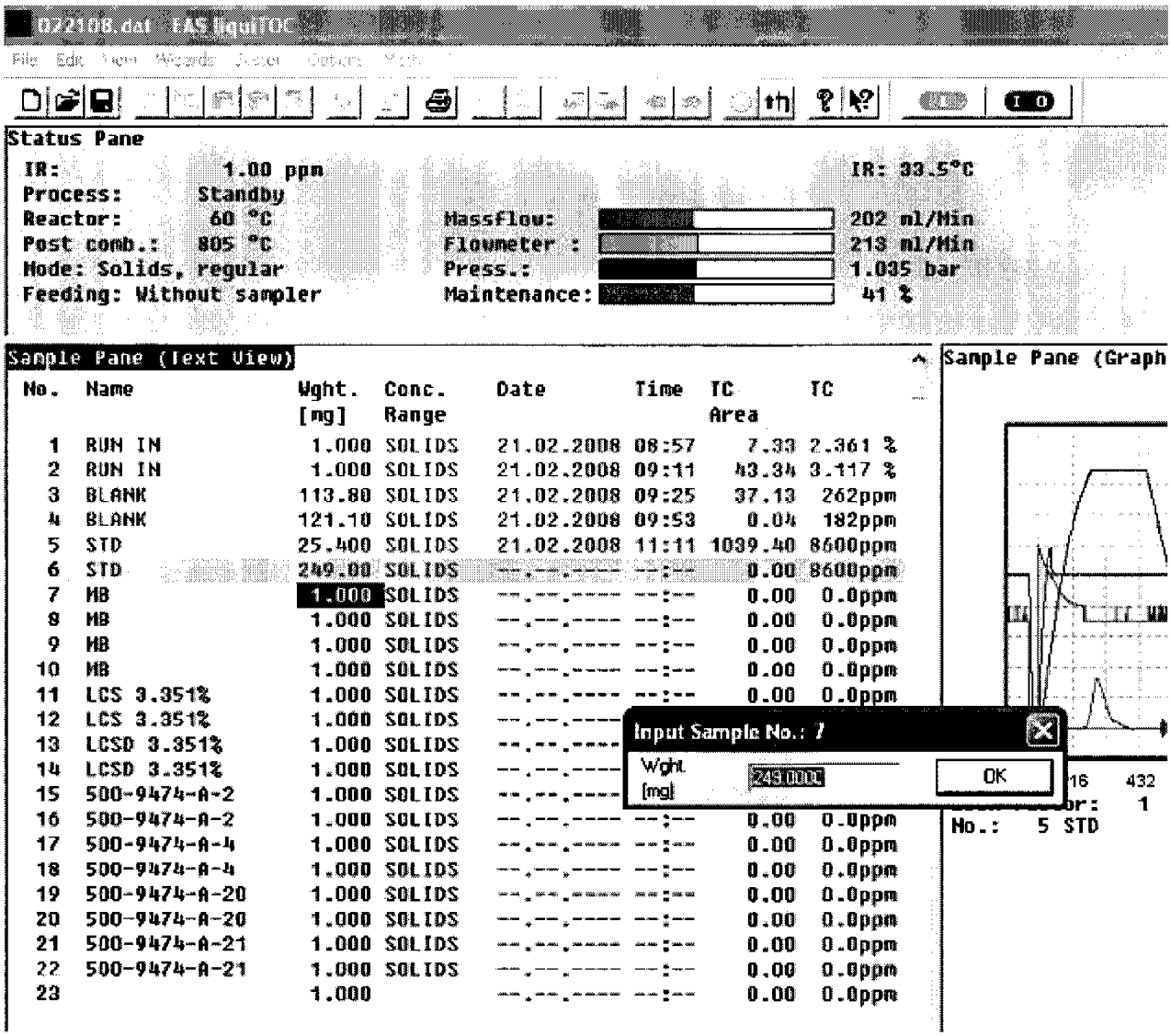
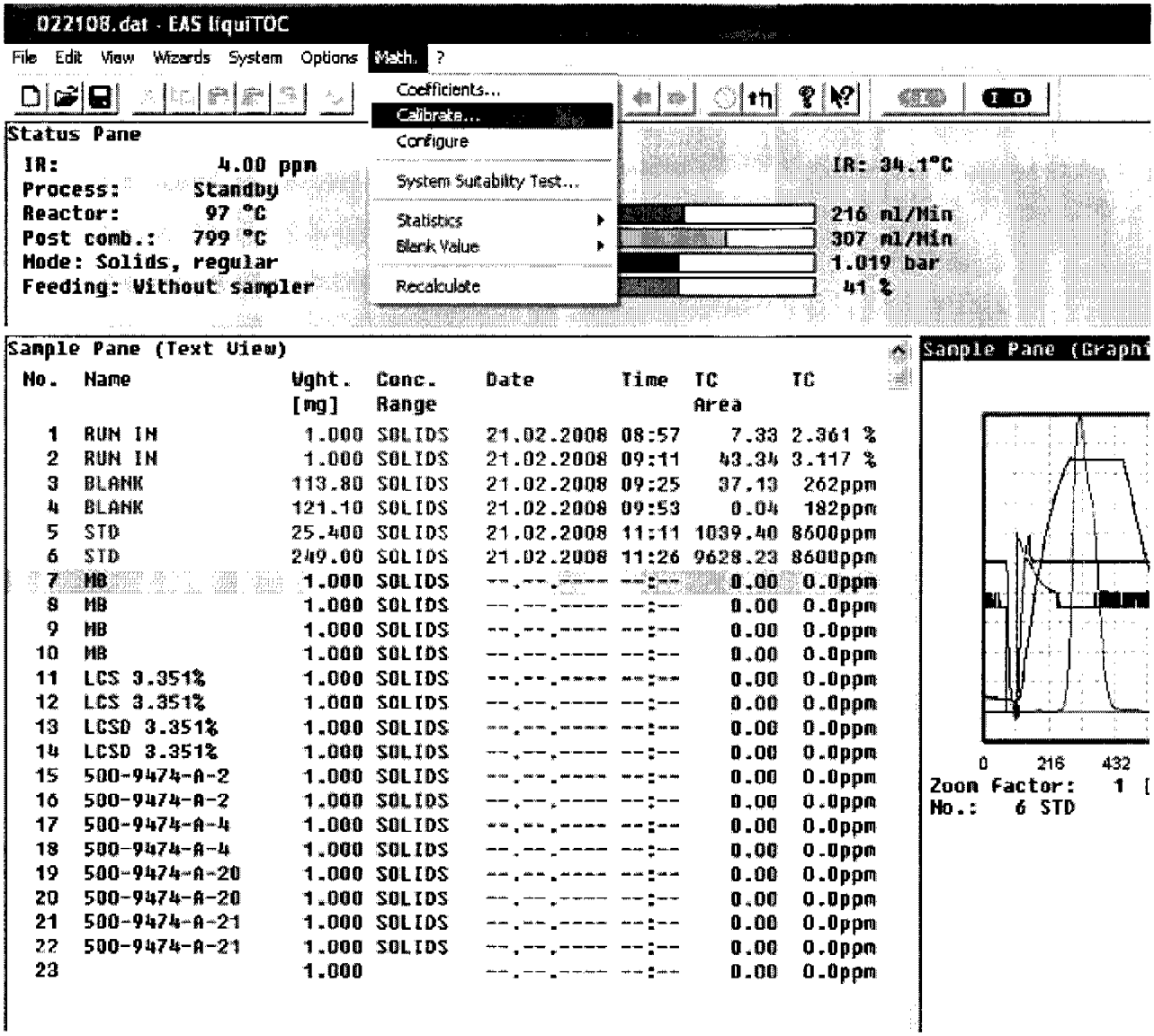

Status Pane
IR: 6.20 ppm
IR: 33.4°C
Process: Standby
Reactor: 30 °C
Post comb.: 803 °C
Mode: Solids, regular
Feeding: Without sampler
Massflow: 202 ml/Min
Flowmeter : 213 ml/Min
Press.: 1.024 bar
Maintenance: 41 %
Sample Pane (Text View)
Sample Pane (Graph
No. Name Wght. [mg] Conc. Range Date Time TC Area TC
1 RUN IN 1.000 SOLIDS 21.02.2008 08:57 7.33 2.361 %
2 RUN IN 1.000 SOLIDS 21.02.2008 09:11 43.34 3.117 %
3 BLANK 113.80 SOLIDS 21.02.2008 09:25 37.13 262ppm
4 BLANK 121.10 SOLIDS 21.02.2008 09:53 0.04 182ppm
5 STD 25.400 SOLIDS 0.00 8600ppm
6 STD 1.000 SOLIDS 0.00 8600ppm
7 MB 1.000 SOLIDS 0.00 0.0ppm
8 MB 1.000 SOLIDS 0.00 0.0ppm
9 MB 1.000 SOLIDS 0.00 0.0ppm
10 MB 1.000 SOLIDS 0.00 0.0ppm
11 LCS 3.351% 1.000 SOLIDS 0.00 0.0ppm
12 LCS 3.351% 1.000 SOLIDS
13 LCSD 3.351% 1.000 SOLIDS
14 LCSD 3.351% 1.000 SOLIDS
15 500-9474-A-2 1.000 SOLIDS
16 500-9474-A-2 1.000 SOLIDS 0.00 0.0ppm
17 500-9474-A-4 1.000 SOLIDS 0.00 0.0ppm
18 500-9474-A-4 1.000 SOLIDS 0.00 0.0ppm
19 500-9474-A-20 1.000 SOLIDS 0.00 0.0ppm
20 500-9474-A-20 1.000 SOLIDS 0.00 0.0ppm
21 500-9474-A-21 1.000 SOLIDS 0.00 0.0ppm
22 500-9474-A-21 1.000 SOLIDS 0.00 0.0ppm
Input Sample No.: 6
Wght. [mg]
OK
No.: 6 STD

FIGURE 26



10.5.20 Once the two STD samples have been run, calibrate the instrument. To do so, select Math. → Calibrate as shown in Figure 27.

FIGURE 27



10.5.21 This will bring up the calibration window. Click on the "SOLIDS" tab and then click the "OK" button to lock in the calibration and present the graphic on the right-hand pane as shown in Figure 28 and 29.

FIGURE 28

022108.dat - EAS liquiTOC

File Edit View Wizards System Options Math ?

Status Pane

IR: 2.70 ppm
Process: Standby
Reactor: 86 °C
Post comb.: 804 °C
Mode: Solids, regular
Feeding: Without sampler

IR: 34.0°C
Massflow: 204 ml/Min
Flowmeter : 292 ml/Min
Press.: 1.024 bar
Maintenance: 41 %

Sample Pane (Text View)

No.	Name	Wght. [mg]	Conc. Range			
1	RUN IN	1.000	SOLIDS			
2	RUN IN	1.000	SOLIDS			
3	BLANK	113.80	SOLIDS			
4	BLANK	121.10	SOLIDS			
5	STD	25.400	SOLIDS			
6	STD	249.00	SOLIDS			
7	MB	1.000	SOLIDS			
8	MB	1.000	SOLIDS			
9	MB	1.000	SOLIDS			
10	MB	1.000	SOLIDS			
11	LCS 3.351%	1.000	SOLIDS			
12	LCS 3.351%	1.000	SOLIDS			
13	LCSD 3.351%	1.000	SOLIDS			
14	LCSD 3.351%	1.000	SOLIDS			
15	500-9474-A-2	1.000	SOLIDS			
16	500-9474-A-2	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm
17	500-9474-A-4	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm
18	500-9474-A-4	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm
19	500-9474-A-20	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm
20	500-9474-A-20	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm
21	500-9474-A-21	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm
22	500-9474-A-21	1.000	SOLIDS	--.--.----	--:--	0.00 0.0ppm

Blank Rates

20ppm | 100ppm | 2ppm | 1000ppm | 50ppm | 1000 N | SOLIDS

Description:

last modified 11.02.2008 13:47

TC: Units/ml

Close

FIGURE 29

022108.dat - EAS liquiTOC

File Edit View Wizards System Options Math. ?

Status Pane

IR: 1.90 ppm
Process: Standby
Reactor: 78 °C
Post comb.: 804 °C
Mode: Solids, regular
Feeding: Without sampler

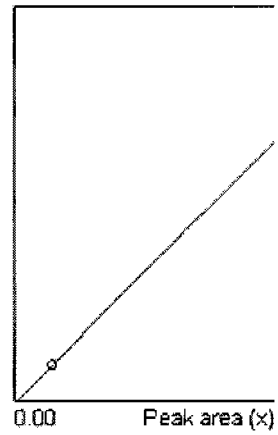
IR: 34.0°C
Massflow: 202 ml/Min
Flowmeter : 278 ml/Min
Press.: 1.029 bar
Maintenance: 41 %

Sample Pane (Text View)

No.	Name	Wght. [mg]	Conc. Range	Date	Time	TC Area	TC
1	RUN IN	1.000	SOLIDS	21.02.2008	08:57	7.33	2.361 %
2	RUN IN	1.000	SOLIDS	21.02.2008	09:11	43.34	3.117 %
3	BLANK	113.80	SOLIDS	21.02.2008	09:25	37.13	262ppm
4	BLANK	121.10	SOLIDS	21.02.2008	09:53	0.04	182ppm
5	STD	25.400	SOLIDS	21.02.2008	11:11	1039.40	8600ppm
6	STD	249.00	SOLIDS	21.02.2008	11:26	9628.23	8600ppm
7	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
8	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
9	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
10	MB	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
11	LCS 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
12	LCS 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
13	LCSD 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
14	LCSD 3.351%	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
15	500-9474-A-2	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
16	500-9474-A-2	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
17	500-9474-A-4	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
18	500-9474-A-4	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
19	500-9474-A-20	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
20	500-9474-A-20	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
21	500-9474-A-21	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
22	500-9474-A-21	1.000	SOLIDS	--.--.----	--:--	0.00	0.0ppm
23		1.000		--.--.----	--:--	0.00	0.0ppm

Calibration Pane

TC Calibration, 'SOLIDS'

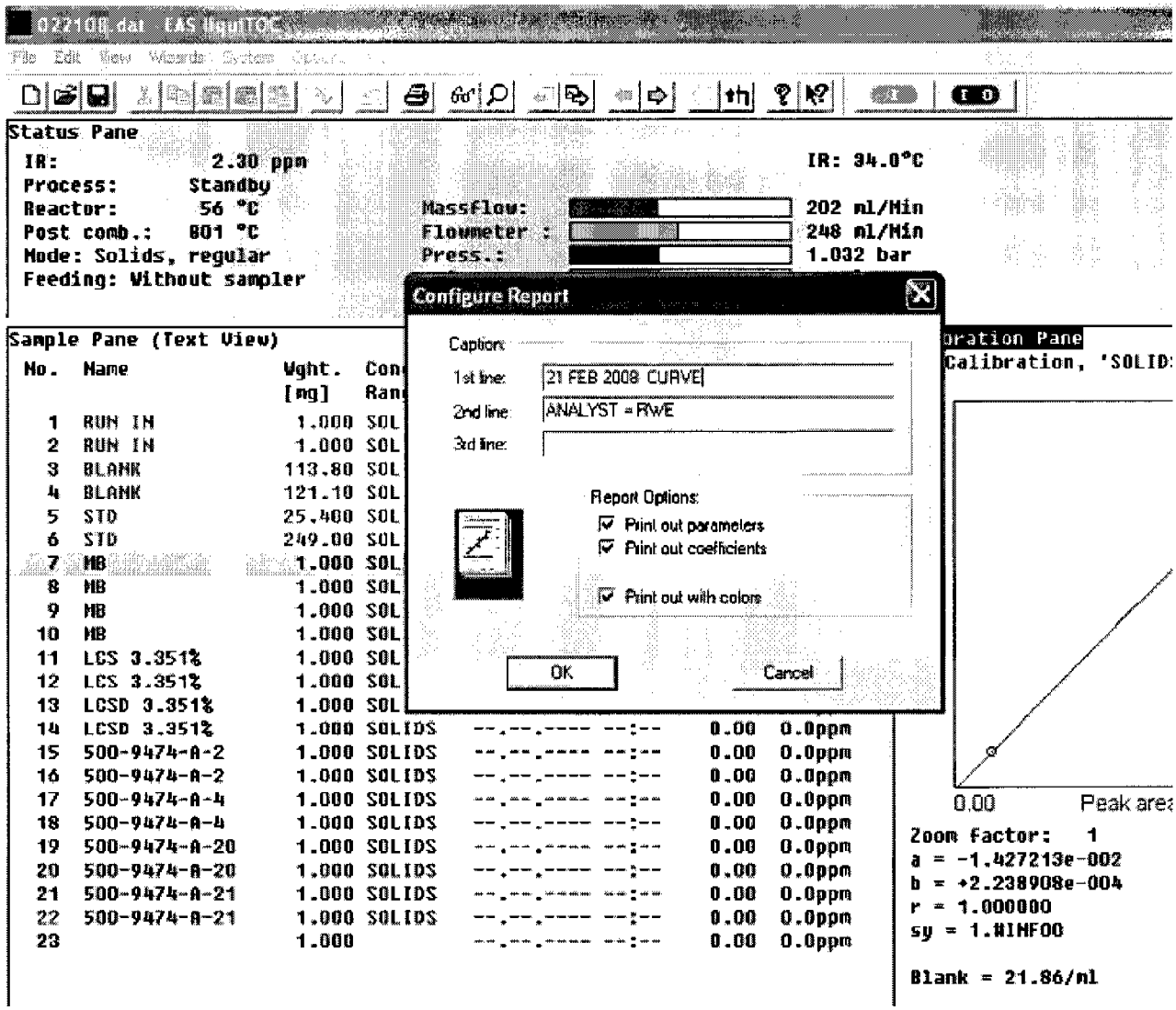


Zoom factor: 1
a = -1.427213e-002
b = +2.238908e-004
r = 1.000000
sy = 1.#INF00

Blank = 21.86/nl

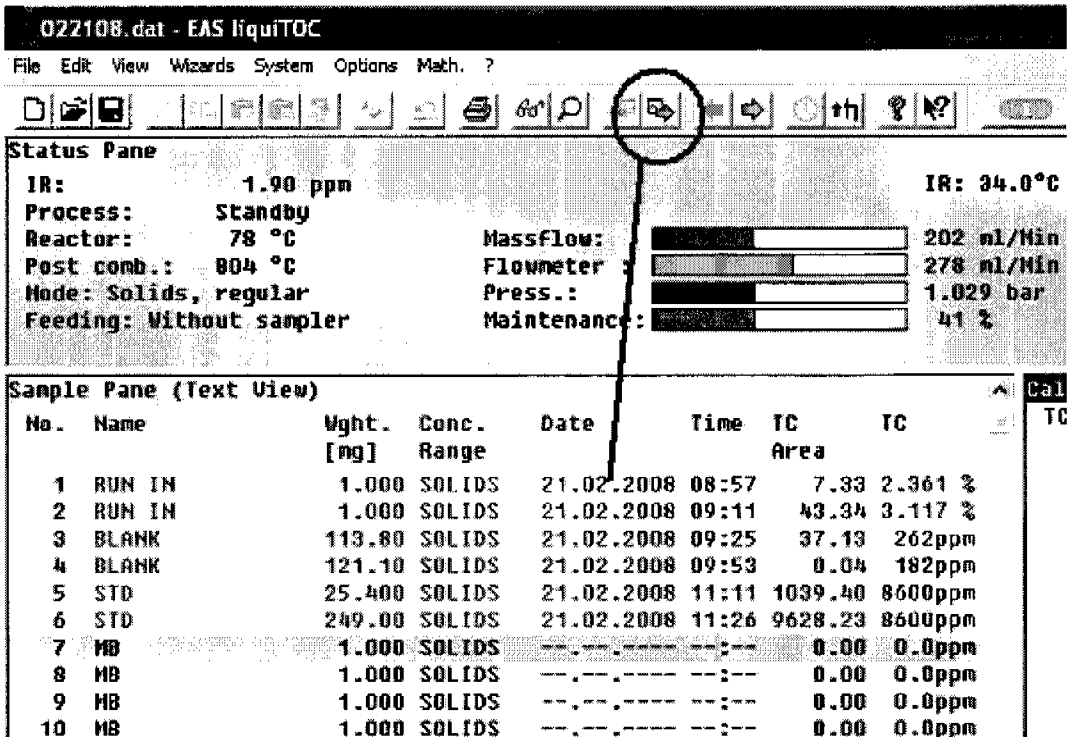
10.5.22 Print the calibration curve by clicking in the Calibration Pane window (to highlight the "Calibration Pane" text) and then click on the printer icon. Enter the date and the word "CURVE" to denote that you are printing the standard curve for the analytical batch. Refer to Figure 30.

FIGURE 30



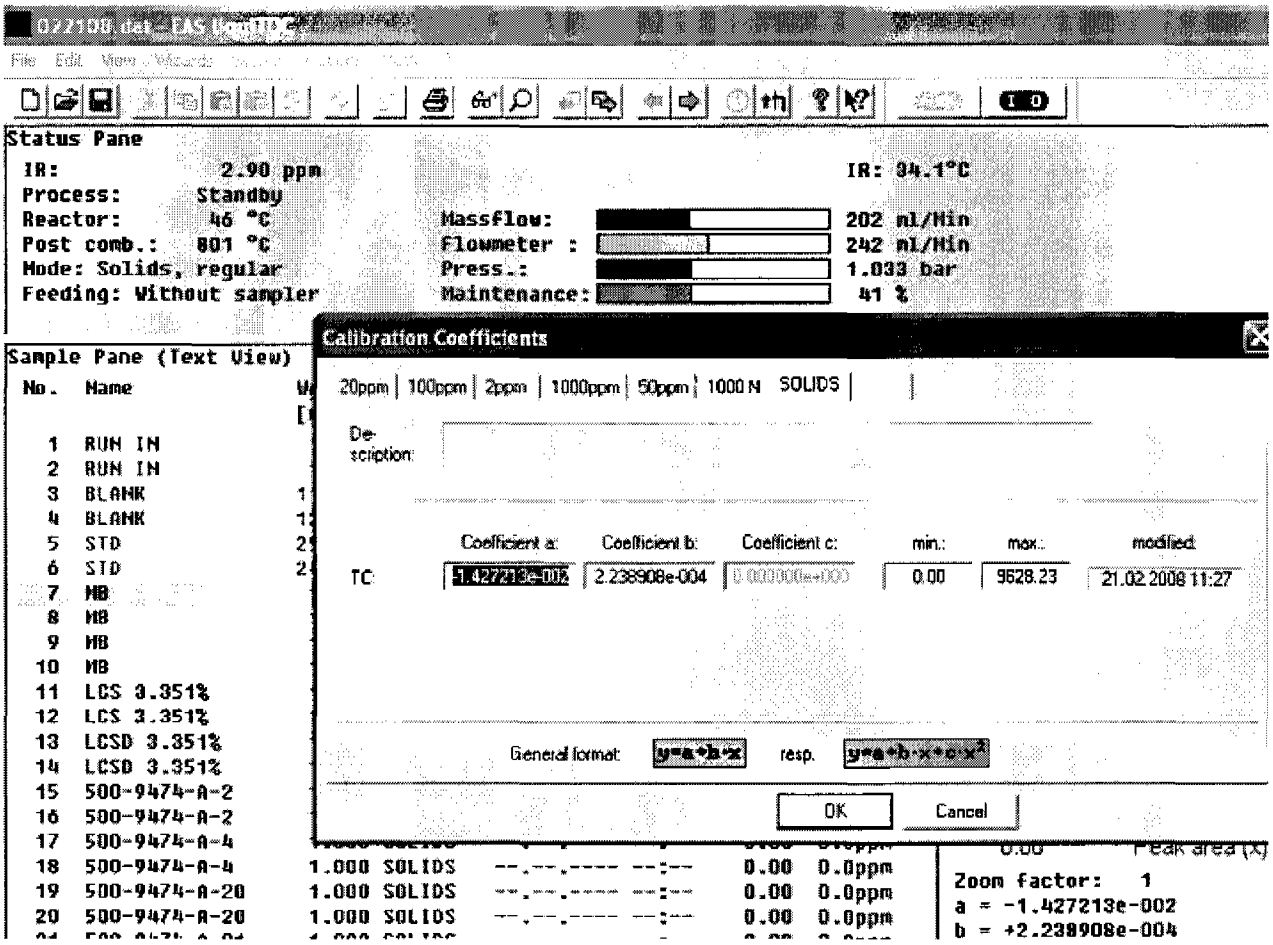
10.5.23 You must lock the calibration into place by clicking on the chart/arrow icon at the top of the page. This tells the computer to use that calibration point from here on. Refer to Figure 31.

FIGURE 31



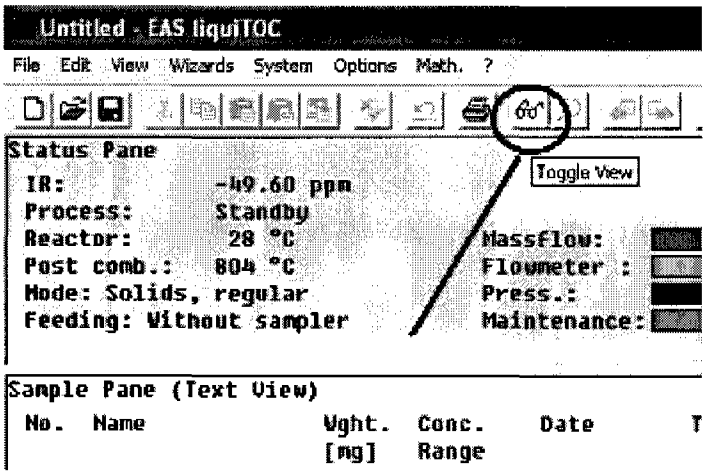
10.5.24 A new window appears; click the "SOLIDS" tab and then click the "OK" button to save the calibration. Refer to Figure 32.

FIGURE 32



10.5.25 With the Calibration Pane still highlighted, click on the eyeglasses icon to scroll through the options to return to the graphical option as shown in Figure 33.

FIGURE 33



10.5.26 It is recommended that you save the batch at this time (refer to the method for analysis of aqueous samples) and click on the icon of the Floppy Disk each time to save each new data point. If the system fails or faults out for any reason, data not manually saved will be lost.

10.5.27 Continue analysis of QC and investigative samples in this manner until all samples have been analyzed. Work up the summary report for the data and run log as given in the method for aqueous sample analysis.

General Notes

10.6 The liquiTOC goes automatically into "sleep mode" after an analysis. The instrument and autosampler may be left turned on without harm between analyses; if leaving the instrument and autosampler on, it is suggested that the user quit out of the liquiTOC software after an analytical run is complete and the instrument is to be idle. The PTFE sample cups should be triply rinsed with deionized water once the analytical run is completed, and air dried inverted on lab paper.

10.7 If a sample has a reading outside of the limits of the working curve, dilute the sample by an appropriate factor and analyze. Such samples can be added on to the end of the current analytical batch, with appropriate start-up and ending QC parameters (CCV, CCB, LCS, etc.). Report the final result for any such samples by multiplying the dilution factor times the diluted sample's reading, and adjust the reporting limit (RL) by the same factor.

10.8 Record the total injections counter value (found by clicking on the Maintenance bar graph on the operations screen; the tally is in the lower right corner of the Maintenance window), the type of analysis (TOC W for water, or TOC S for soil/solid), and other run data in the Run Log book for each analytical batch performed.

Troubleshooting & Maintenance

10.9 Troubleshooting

10.9.1 Should a communications error occur, save the current run by clicking on the computer disk icon, and then exit the software. Shut down the liquiTOC, then the autosampler; wait ten seconds, then turn the autosampler on, the liquiTOC on, and after the liquiTOC goes through it's start up (the instrument stops humming), launch the software. Open the file you were working on, at which point the software should automatically set itself to run the next sample in line. Click on the green-red button to re-initiate the automatic run.

10.9.2 Should the baseline be noisy, or a blank sample produce a concentration for TOC that is greater than the RL, the reactor chamber either needs to be cleaned or replaced. See the PM instructions below.

10.9.3 Should the flowmeter and massflow bar graphs not read 200±30 units, or if the two values are not within about 10 units of one another, there may be a flow problem or leak in the fluidics of the system. Perform a leak check by selecting System→ Diagnostics→ Leak Test and following the instructions given. Replace tubing or connectors as needed. If no leak is found, or if you need assistance, call Elementars Americas Inc. for technical assistance.

10.9.4 For any other problems with the instrument, consult with Scott Hughes or technical support at Elementars America, Inc. at (856) 787-0022 x202.

10.10 Routine Preventive Maintenance

10.10.1 Record any maintenance performed in the instrument log book.

10.10.2 The liquiTOC has a maintenance monitoring feature that counts the number of injections and alerts the user as to when to perform routine maintenance on the instrument, or to check various operating conditions. This can be viewed by

simply clicking on the "Maintenance" bar graph, located just under the bar graph read outs for the "Flowmeter" and "Massflow." The bar graph for Maintenance changes color to alert the operator that maintenance isn't yet required (green, will soon be required (yellow) and must be performed (red).

10.10.2.1 When open, the Maintenance window shows each key check or maintenance requirement. The frequency for maintenance is pre-set by the manufacturer. When a maintenance item is performed, double click in the corresponding counter space, type a zero ("0") to reset it, then click on the "OK" button to enter the value.

10.10.2.2 The auto-monitored maintenance items are as follows:

Auto-Monitored Maintenance Item	Actions to Be Taken
Refill Deionized Water Container (every 250 injections)	Check the deionized water supply to the instrument; add fresh deionized water if needed.
Check Reactor (every 500 injections)	Check the glass reaction chamber (that contains the platinum catalyst). If it is heavily encrusted with salts, or blocked or heavily discolored, rinse it with deionized water to clean it.
Renew Silver Wool (every 500 injections)	Renew the silver wool and copper metal granules in the halogen scrubber tubes on the inside left of the instrument. Copper granules alone may also be used if no silver wool is available.
Check Furnaces (every 1000 injections)	Check to be sure that the furnaces (the upper and lower zones on the reactor tube) are heating. Under normal operating conditions, the top segment should be 800C (and visibly glowing orange when examined), and the lower segment should ramp up from near room temperature to 800C when analyzing a sample.
Clean Reactor (every 1000 injections)	See "Check Reactor" above for cleaning procedure. Note that if the baseline is not stable, or the blanks begin reading a slightly positive value for TOC that is above the RL, the reactor tube and catalyst need to be replaced.
Screw Spindle Grease (every 500 injections)	Use a Kimwipe to wipe clean the screw spindle. Add a small dollop of white grease to the screw thread just above the motor (inside left panel of the instrument).

Waste Jug (every 500 injections)	Empty the waste jug every 500 injections or if it becomes close to being full.
Gas (every 5000 injections)	Replace the zero grade air cylinder every 5000 injections, or if the pressure drops below 500 psi.

10.10.3 In addition to the requirements for Maintenance listed above, perform the following at the frequency indicated, recording the maintenance performed in the instrument log book:

Maintenance Item	Actions to Be Taken
Check Desiccant (once per month)	If the desiccant has turned from blue to pink for more than two-thirds the fill length, replace the desiccant with fresh Magnesium perchlorate (Cat #05-001-059)
Check coil trap filter (once per month)	Check the polyfoam plug at the bottom of the glass coil water trap. If it is very dirty, or has salt deposits built up on it, change it with a new plug (Cat #03-002-399)
Check fluidics (once per month)	Check the lines and connections in both the left and right sides of the instrument to determine if any leaks have occurred. If so, replace worn parts as needed.
Check autosampler and reagent lines (once per month)	Check the lines and connections to make certain that the lines are not kinked or blocked. If any line is kinked or blocked, replace it with fresh tubing and/or connectors.
Replace the Reactor Chamber (as needed)	The reactor chamber is good for about 1500-2000 injections for water, or about 1000-1500 injections for soil. If the baseline becomes noisy, or a blank sample produces a TOC value over the RL, it is likely time to change the reactor chamber.

11.0 Calculations / Data Reduction

- The analyzer software automatically calculates the working curve; the linear fit for it and determines the concentration for the investigative and QC samples based on the curve.

The data from an analytical run is printed out for documentation, and the data from it transferred to the LIMS system for reporting. All QC criteria must be met, else corrective action and/or acceptance of data with disclaimer is performed.

- Note that it is standard operating procedure to discard the first of the triplicate data points for each sample analysis as cited above.
- For each analytical batch, print a CURVE, RUN LOG and RESULTS page as described in the analysis procedure.
- TIC is calculated manually from a run that includes both a TC and TOC analysis of the sample (see item 10.2.2).

11.1 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

11.2 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial detection limit study must be performed on the instrument before samples can be analyzed. MDL studies are conducted annually, when a new instrument is put into production or a change in procedure is implemented.

12.2 Demonstration of Capabilities

Initial Demonstration of Capability (IDOC). All personnel are required to perform an IDOC proficiency on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. At least four aliquots shall be prepared (including any prep processes) and analyzed according to test method (either concurrently or over a period of days)

12.3 Training Requirements

- Analysts must read and understand this SOP before performing any distillation and/or analysis.
- The Department Manager and QA Manager must document this training in the analysts' training files.
- The department manager has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- Acceptable IDOC and annual DOC

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to WE-SAF-002.

This instrument produces slightly acidic wastewater from aqueous samples and deionized water rinse. This waste requires no special treatment, and thus may be disposed of into the sewer system via any lab sink.

Solid matrix samples from the processing of solid/soil samples is collected in a beaker and disposed of into non-hazardous soil waste collected in the hazardous waste room.

15.0 References / Cross-References

- "Standard Methods for the Examination of Water and Wastewater," 18th Edition, 5310B. pp. 5-17 to 5-19.
- SW846 Method 9060 "Total Organic Carbon," September 1986.
- "High Temperature TOC/TN_b Analyzer Operating Instructions," elementar Americas, Inc., March 2003.
- Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method) July 27, 1988.

16.0 Method Modifications:

Item	Method xx	Modification
001	*9060*	*Used to determine TOC in soil and solids*

17.0 Attachments

Attachment 1:

18.0 Revision History

Chronological tracking of revisions.