Attachment A

Treatability Studies Corrective Action Memoranda

BLASLAND, BOUCK & LEE, INC.
engineers, scientists, economists

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park
Suite 323
Albany, NY 12203
518) 862-2739
Dial Comm: 8 232-2739*
Telephone: (518) 862-2731

VIA FEDERAL EXPRESS AND ELECTRONIC MAIL

April 19, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Hudson River Remedial Design AOC - Notification of Treatability Study Field Work Initiation

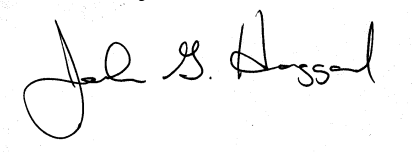
Dear Sir or Madam:

Paragraph 62 of the Remedial Design Administrative Order on Consent (AOC) (Index No: CERCLA-02-2003-2027) requires that GE notify EPA within 14 days of initiating field work. GE plans to begin mobilization to collect treatability study samples the week of April 26,

2004 and begin full scale sample collection on May 3, 2004. The field work start will be contingent on weather and seasonal constraints allowing safe performance of the field sampling.

Please let me know if you have any questions.

Sincerely,



John G. Haggard

JH/bg
Attachement

cc: Bob Gibson, GE
Mike Elder, GE
Paul Doody, BBL
Steve Garbaciak, BBL
Don Sauda, BBL
Mike Crystal, Sevenson

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT SEDIMENT SAMPLING AND ANALYSIS PROGRAM

Date: May 5, 2003
Organization Name: Blasland, Bouck & Lee, Inc. (BBL)
Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description: The Standard Operating Procedure (SOP) for Sample Collection for Treatability Tests was included as Appendix 1 of the Treatability Studies Work Plan (TS Work Plan, BBL, February 2004) that was approved by USEPA on February 13, 2004. The Sample Collection for Treatability Tests SOP referenced the SOP for Sediment Sampling Procedures in Appendix 1 of the Sediment Sampling and Analysis Program Quality Assurance Project Plan (SSAP QAPP, Quantitative Environmental Analysis, LLC [QEA] and Environmental Standards, Inc. [ESI], 2002). Because the samples being collected for the Treatability Studies involve collecting multiple samples in a ¼-acre sampling location, all the procedures in the SSAP QAPP are not relevant. Specifically, changes need to be made in the areas of data management, surveying the ¼-acre sampling locations, sediment probing, sample collection and decontamination of hand-held dredges, and aluminum coring tube length and sampling depth.

Michael Johnson from Malcolm Pirnie, a USEPA Oversight Contractor, has requested that the SOP for Sample Collection for Treatability Tests (TS Work Plan, Appendix 1) be revised to reflect the actual procedures to be followed for the work being performed.

Reported To: Bob Gibson, GE

Corrective Action: The attached SOP for Sample Collection for Treatability Tests has been modified to reflect the actual procedures to be followed for the work being performed. While the reference to the SSAP QAPP remains, modifications have been made in the areas of data management, surveying the ¼-acre sampling locations, sediment probing, sample collection and decontamination of hand-held dredges, and aluminum coring tube length and sampling depth. The Sample Collection for Treatability Tests SOP modification described herein was approved verbally by the USEPA Oversight Contractor, Michael Johnson from Malcolm Pirnie, on May 3, 2004.

Reviewed and Implemented By: Don Sauda (BBL)

cc: GE Project Manager: Bob Gibson
Other Distribution: Mark LaRue (QEA), Laurie Scheuing (QEA)

Standard Operating Procedure: Sample Collection for Treatability Tests

I. Scope and Application

This standard operating procedure (SOP) describes the procedures for Sample Collection for Treatability Tests. Sampling locations are discussed in the Treatability Studies Work Plan (TS Work Plan) (Blasland, Bouck & Lee, Inc. [BBL], 2004). Samples will include sediment samples in quantities ranging from 80 gallons (310 liters) to 140 gallons (530 liters), and 2,200 gallons (8,400 liters) of water.

Surface water samples will be collected from throughout the treatability studies program on an as-needed basis. The water sampling station will be located at River Mile 187.5. Composite sediment samples will be prepared from sediments at locations designated S1, S2, S3, and S4. These locations are shown on figures 4 through 10, included in the TS Work Plan. The coordinates, quantity and maximum collection depth for each sediment sample type are described on Table 1.

II. Equipment List

The following materials, as required, will be available during this procedure:

- Personal protective equipment (as required in the *Revised Health and Safety Plan* [Revised HASP]) (BBL, 2003);
- Sampling containers for aqueous samples;
- GPS survey equipment;
- Sampling pump and associated tubing for water collection;
- Sampling vessel with outboard motor;
- Hand-held dredge sampling device;
- Vibracoring device;
- 3-inch (outside diameter [o.d.]) aluminum coring tubes; and
- Field notebook.

III. Health and Safety Considerations

Refer to the Revised HASP (BBL, 2003).

IV. Sample Collection for Treatability Tests Procedure

Eight general sampling sites are discussed in the TS Work Plan. Discrete cores or hand-held dredge samples will be collected within each sampling site over an area of approximately 1/4-

acre (this area would be approximately equivalent to the area covered by a mechanical dredge filling one barge). Record general weather conditions relevant to sample integrity.

Sample collection procedures for water samples are described below:

Surface water samples will be collected from the Thompson Island sampling station located at River Mile 187.5, approximately one foot below the water surface. It is anticipated that surface water will be collected in conjunction with the baseline monitoring activities. Water samples will be collected throughout the treatability studies program on an as-needed basis to avoid difficulties associated with shipment and storage of large volumes of water.

Sample collection procedures for sediment samples are described below:

1. Obtain target composite sample size from the TS Work Plan for the 1/4-acre Treatability Studies sample location. Calculate target subsample sizes and number of coring tubes or hand-held dredges per subsample.

2. Mark the corner of each 1/4-acre Treatability Studies sample location. After the vessel is positioned for subsampling within a given sample location, proceed with sampling in a downstream to upstream manner.

3. Obtain subsamples by vibracoring or hand-held dredging following the SOP for Sediment Sampling Procedures in Appendix 1 of the SSAP QAPP (QEA and ESI, 2002) with the following modifications for collection of Treatability Studies samples:

    - All data will be recorded in field notebooks and summarized on Excel spreadsheets;
    - The GPS coordinates for the corners of each 1/4-acre sampling location are provided on Table 1. The GPS coordinates for each sample collected will be recorded;
    - A calibrated steel rod will be used to periodically probe the sediment surface in each sampling location to determine the sediment thickness and type;
    - The maximum collection depth at each sampling location is shown on Table 1;
    - The length of 3-inch (o.d.) aluminum coring tube will vary based on the maximum sample collection depth shown on Table 1;
    - The coring tube will be attached to the vibracoring apparatus and lowered through the water column to the river bottom;
    - The length of cores recovered in aluminum tubing will be directly measured using an aluminum measuring device;
    - A hand-held (ponar) dredge will be used to collect the S1 sample in River Section 3 and the dredge samples will emptied into 5 gallon plastic pails;
    - The hand-held dredge will be decontaminated before and after sampling activities in River Section 3, but not between collection of individual samples; and
    - At the end of each day, a copy of the Excel spreadsheet will be provided to the processing laboratory.

4. Record number of subsamples taken from each position. Chill to 4^{o}C.

5. Label each core or dredge sample and process for shipment to the Treatability Studies sample processing facility.

Repeat subsampling until all compositing locations are complete. Then move to the next 1/4-acre sampling location and complete all subsampling. Continue until all eight 1/4-acre composite samples are completed. Record any deviations from this SOP during sampling.

Sample Homogenization Procedures:

1. Place sediments to be homogenized in an appropriately sized, decontaminated, mixing device, such as a grout mixer.

2. Mix for at least 10 minutes, until sediments are combined to a uniform consistency with no unmixed agglomerations of sediment visible.

V. References

BBL. 2003. *Revised Health and Safety Plan* (Revised HASP). Hudson River PCBs Superfund Site. Prepared for General Electric Company, Albany, NY.

QEA and ESI. 2002. *Sediment Sampling and Analysis Program - Quality Assurance Project Plan* (SSAP-QAPP). Hudson River PCBs Superfund Site. Prepared for General Electric Company, Albany, NY.

Table 1 - Treatability Studies Sediment Sample Collection

Treatability Studies Sediment Sampling Locations	Coordinates (New York State Plane East, NAD 83)								Quantity of Sediment		Maximum Collection Depth	TSWP Figure
	NW Corner		SW Corner		SE Corner		NE Corner					
	Northing	Easting	Northing	Easting	Northing	Easting	Northing	Easting	(Gallons)	(Cubic Feet)	(Feet)	Reference
S2 - River Section 1	1,608,911	732,730	1,608,805	732,724	1,608,805	732,835	1,608,899	732,829	40	5.3	1.8	4
S3 - River Section 1	1,607,785	732,221	1,607,690	732,221	1,607,690	732,312	1,607,785	732,312	55	7.4	3.8	4
S1 - River Section 1	1,595,947	737,592	1,595,848	737,592	1,595,848	737,712	1,595,947	737,712	70	9.4	1.2	5
S4 - River Section 1	1,593,043	736,251	1,592,927	736,251	1,592,927	736,359	1,593,043	736,359	50	6.7	3.1	6
S4 - River Section 2	1,576,492	737,715	1,576,398	737,715	1,576,398	737,841	1,576,492	737,841	50	6.7	3.0	7
S2 - River Section 2	1,571,444	735,583	1,571,337	735,583	1,571,337	735,688	1,571,444	735,688	40	5.3	1.9	8
S3 - River Section 3	1,503,237	725,467	1,503,122	725,467	1,503,122	725,565	1,503,237	725,566	55	7.4	5.1	9
S1 - River Section 3	1,498,640	724,536	1,498,534	724,536	1,498,534	724,637	1,498,640	724,637	70	9.4	0.2	10
								Total:	***430***	***57.5***		

Notes:

TSWP - Treatability Study Work Plan (BBL, 2004)

S1 =	140	Gallons	0.7	Cubic Yards
S2 =	80	Gallons	0.4	Cubic Yards
S3 =	110	Gallons	0.5	Cubic Yards
S4 =	100	Gallons	0.5	Cubic Yards
Total =	430	Gallons	2.1	Cubic Yards

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park
Suite 323
Albany, NY 12203
518) 862-2739
Dial Comm: 8 232-2739*
Telephone: (518) 862-2731

VIA FEDERAL EXPRESS AND ELECTRONIC MAIL

June 2, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Hudson River Remedial Design AOC – Treatability Study Work Plan Standard Operating Procedures

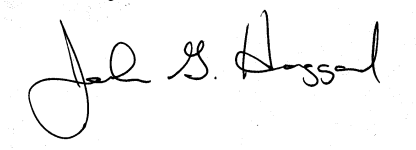
Dear Sir or Madam:

On February 13, 2004, the U.S. Environmental Agency (EPA) approved the Treatability Studies Work Plan (TS Work Plan) prepared by Blasland, Bouck & Lee, Inc. Since that time, the vendors to complete the Treatability Studies have been selected. As requested with your approval letter, please find enclosed for informational purposes the final Standard Operating

Procedures (SOPs) for the Treatability Studies. For your convenience, we have enclosed a complete set of Appendices for the TS Work Plan that include the final SOPs.

If any questions arise, please do not hesitate to contact either myself or Bob Gibson at (518) 862-2736.

Sincerely,



John G. Haggard

JGH/bg

cc: Bob Gibson, GE
Michael Elder, GE
Barbara Ippolito, GE
Don Sauda, BBL
Steve Garbaciak, BBL
Paul Doody, BBL

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 323
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

VIA FEDERAL EXPRESS AND FAX

June 29, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Hudson River – Treatability Study Work Plan - Corrective Action Memorandum No. 2

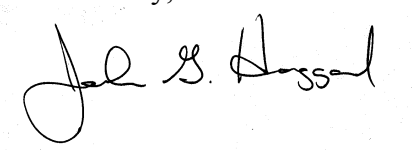
Dear Sir or Madam:

Attached please find Corrective Action Memorandum (CAM) No. 2 to the Treatability Study (TS) Work Plan. This CAM documents correction of a number of inconsistencies in the

TS Work Plan that were identified during our planning for the work and preparation of the final Standard Operating Procedures (SOPs) with our treatability study contractor.

We anticipate commencing testing work at Waste Stream next week. We therefore request your prompt review and approval of this CAM so that the treatability testing can proceed smoothly and without interruption. If you should have any questions, please feel free to give Bob Gibson a call at (518) 862-2736.

Sincerely,



John G. Haggard

JGH/bg
Enclosure

cc: Ben Conetta, USEPA
Robert Gibson, GE
Barbara Ippolito, GE
Don Sauda, BBL
Paul Doody, BBL

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT
TREATABILITY STUDIES PROGRAM

Date: June 29, 2004

Organization Name: Blasland, Bouck & Lee, Inc. (BBL)

Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description: The Treatability Studies Work Plan (TS Work Plan, BBL, February 2004) was approved by USEPA on February 13, 2004. Once a Treatability Studies Contractor was selected, final Standard Operating Procedures (SOPs) were prepared and submitted to USEPA on June 2, 2004. During preparation of the SOPs and planning for implementation of the work, a number of minor inconsistencies and corrections to the TS Work Plan were identified. This Corrective Action Memorandum (CAM) documents the resultant modifications to the work plan. The corrections, with references to the TS Work Plan Section(s) and the associated Data Quality Objective (DQO), are summarized below.

- Section 2.1.2 (DQO 1b) – The text states that total organic carbon (TOC) analysis will be conducted on suspended solids (SS) present in the water sample. Because TOC is being analyzed on filtered and unfiltered water samples, the TOC present in the SS will be equal to the difference in TOC between filtered and unfiltered water samples. Therefore, TOC analysis will not be done on the SS present in the water sample. This is consistent with Table 2 of the TS Work Plan. Additionally, large volumes of water would need to be filtered to generate enough sample volume to analyze the filtered solids for TOC. This is not practical or necessary for the treatability test program.

- Section 2.3 and Table 2 (DQO 3) – The text and Table 2 state that suspended particulate fraction samples will be analyzed for polychlorinated biphenyls (PCBs). Because PCBs are being analyzed on filtered and unfiltered water samples, PCBs present in the suspended particulate fraction will be equal to the difference in PCBs between filtered and unfiltered water samples. Therefore, PCBs analyses are not being done on the suspended particulate fraction. Additionally, similar to the previous item, large volumes would be required to generate the required sample volume for PCBs analysis on the suspended particulate fraction. One other clarification to this section is related to the analyses of TSS, pH, dissolved oxygen (DO), and turbidity described in subsections 2.3.1 and 2.3.2. The analyses are included in both subsections, however, the water generated from the same dredge elutriate test (DRET) will be used to determine PCB and non-PCB release to the water column from the dredge head. Therefore, TSS, pH, DO and turbidity will be analyzed once for each DRET run to satisfy the PCB release and non-PCB release DQOs defined in subsections 2.3.1 and 2.3.2, respectively.

- Section 2.4.2 and Table 2 (DQO 4b[1a] & 4c[1a]) – The text and Table 2 incorrectly state that grain-size distribution analyses will be conducted on each solid fraction (i.e., fraction retained on each sieve). In the initial size separation study, the standard set of screens (per ASTM D422) will be used and PCBs, pH, Target Analyte List (TAL) metals, specific gravity, and Atterberg limits will be measured in each fraction, provided the test generates enough volume for all the fractions. Fractions that do not result enough volume for discrete sampling will be composited with material from adjacent screens. Therefore, grain-size distribution analyses is integral to the test and will not be conducted on material separated using sieves. However, density separation may be conducted using techniques such as a hydrocyclone or high-density liquid methods. Should these density separation techniques be used, grain-size distribution analyses will be conducted on collected solid fractions.

- Section 2.5.3 and Table 2 (DQO 5a[3]) - The text and Table 2 state that General Electric Hudson River (GEHR) Modified Method 8082 will be conducted on effluent water samples from the rapid small-scale column tests (RSSCTs). However, to be consistent with other analyses specified in the TS Work Plan for low-level PCBs in water, the analyses will be changed to the Modified Green Bay Mass Balance Method.

The Sediment Sampling and Analysis Program - Quality Assurance Project Plan (SSAP-QAPP, Quantitative Environmental Analysis, LLC [QEA] and Environmental Standards, Inc. [ESI], 2002), Baseline Monitoring Program – Quality Assurance Project Plan (BMP-QAPP, QEA and ESI, 2004), and TS Work Plan all contain SOPs related to analytical testing. During preparation of the SOPs for the TS Work Plan, inconsistencies and corrections related to analytical methods were identified.

The methods for analyzing samples generated during the Treatability Studies for PCBs are specified in the text and on Table 2 of the TS Work Plan. These methods require slight modifications, as summarized below.

- GEHR Modified Method 8082 was originally developed for analysis of sediment samples collected in the SSAP. Since the objective of some tests is to evaluate partitioning of PCBs into the aqueous phase, GEHR Modified Method 8082 needs to be modified to analyze water samples. In order to run the method on aqueous samples, calibration standards for GEHR Modified Method 8082 need to be lower than those specified in the SSAP-QAPP. A description of these standards is given below.

    i. The initial calibration curve for Aroclor 1221, Aroclor 1242, and Aroclor 1254 will be adjusted lower to meet reporting requirements for water samples. The initial calibration curve will include standards at 5.00 parts per billion (ppb), 10.0 ppb, 20.0 ppb, 50.0 ppb, and 100 ppb. This will provide a reporting limit of 25.0 nanogram per liter (ng/L) for each Aroclor listed.

ii. A Method Detection Limit (MDL) study has been performed on the three Aroclors. A global MDL will be set at 8.85 ng/L for the three Aroclors listed. Sample data will be "J" flagged to this MDL value.

iii. The sample extracts will be set to a final volume of 5 milliliters (mL).

iv. The surrogates tetra-chloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB) will be added to all method blank samples, laboratory control spike samples, matrix spike samples, matrix spike duplicate samples, and field samples at a concentration of 25 ng/L and 250 ng/L, respectively.

v. Laboratory Control Spike (LCS) samples will be spiked with Aroclor 1242 at a concentration of 250 ng/L.

- PCBs will be measured in water samples by the Modified Green Bay Mass Balance Method or GEHR Modified Method 8082. These are specified in the text and Table 2, and will be completed in accordance with the SSAP-QAPP and BMP-QAPP. However, for the DRET (DQO 3a.[1]), water processing tests (DQO 5a[2]) and 5a[3]), and perhaps other analyses, liquid-liquid extraction (USEPA Method 3520C) is specified as an option to solid phase extraction (USEPA 3535) because the potential PCB concentrations in these samples may be much higher than samples collected during the SSAP and BMP programs. This will allow the analytical laboratory to dedicate the solid phase extraction equipment to other Hudson River sampling programs without encountering potential equipment contamination issues.

Reported To: Bob Gibson, GE

Corrective Action: The Treatability Studies will be completed based on the changes described above. Additionally, the attached Tables 8 and 9 from the TS Work Plan have been revised to document several changes to the analytical methods, sampling containers and Quality Assurance/Quality Control (QA/QC) in order to be consistent with the SOPs in the SSAP-QAPP, BMP-QAPP and TS Work Plan.

Reviewed and Implemented By: Don Sauda (BBL)

cc: GE Project Manager: Bob Gibson
Other Distribution: Amy Toth (Waste Stream)

General Electric Company
Hudson River PCBs Superfund Site
Treatability Studies Work Plan

Table 8 - Sample Containers, Preservation, and Holding Times

Parameter	Bottle Type	Preservation	Holding Time[2]
Solids			
PAH (SW-846 8270C/3545)	1-8oz glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	14 days to extraction
			40 days to analysis
PCBs (GEHR Modified SW-846 8082)	1-8oz glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	14 days to extraction
			40 days to analysis
PCDD/PCDF (USEPA 1613B)	1-8oz amber glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	30 days to extraction
			45 days to analysis
TAL Metals (SW-846 6010B)	1-4oz wide mouth glass jar	Cool to 4°C +/- 2°C	180 days to analysis
Mercury (SW-846 7471A)			28 days to analysis
TCLP-Volatiles (SW-846 1311/8260B)	1-4oz glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	14 days to TCLP extraction
			14 days to analysis
TCLP-Semivolatiles	1-8oz glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	14 days to TCLP extraction
(SW-846 1311/3510C/3520C/8270C)			7 days to extract prep
			40 days to analysis
TCLP-Pesticides	1-8oz glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	14 days to TCLP extraction
(SW-846 1311/3510C/3520C/8081A)			7 days to extract prep
			40 days to analysis
TCLP-Herbicides (SW-846 1311/8151A)	1-8oz glass jar with Teflon®-lined lid	Cool to 4°C +/- 2°C	14 days to TCLP extraction
			7 days to extract prep
			40 days to analysis
TCLP-Metals (Except Mercury)	1-4oz wide mouth glass jar	Cool to 4°C +/- 2°C	180 days to TCLP extraction
(SW-846 1311/3010A/6010B)			180 days to analysis
TCLP-Mercury (SW-846 1311/7470A)			28 days to TCLP extraction
			28 days to analysis
pH (USEPA 9045C)	1-4oz wide mouth glass jar	Cool to 4°C +/- 2°C	48 hours to analysis
Total P/PO_4 (USEPA 365.2)	1-4oz wide mouth glass jar	Cool to 4°C +/- 2°C	28 days to analysis
NH_3/TKN (USEPA 350.1/351.3)	1-4oz wide mouth glass jar	Cool to 4°C +/- 2°C	28 days to analysis
Grain size (ASTM D422/ASTM D1140)	large Ziploc® bag	NS	NS
Paint Filter (SW-846 Method 9095A)	1-4oz wide mouth glass jar	Cool to 4°C +/- 2°C	7 days to analysis
TOC (Lloyd Kahn)	1-125ml glass jar	Cool to 4°C +/- 2°C	28 days to analysis
Water Content (SM 2540G)	small Ziploc® bag	3 to 30°C	As soon as practical
Atterberg limits (ASTM D4318)	large Ziploc® bag	NS	NS
Unconfined Compressive Strength (ASTM D2166-00)	2 large Ziploc® bags	NS	NS
Specific Gravity (ASTM D854)	large Ziploc® bag	NS	NS
Bulk Density (ASTM D4531, modified)	large Ziploc® bag	NS	NS
Consolidation (ASTM D2435)	large Ziploc® bag	NS	NS
Water			
PAH (SW-846 Method 8270C/3520C)	2-1 liter amber glass bottles with Teflon®-lined lid	Cool to 4°C +/- 2°C	7 days to extraction
			40 days to analysis
PCDD/PCDF (USEPA 1613B)	2-1 liter amber glass bottles with Teflon®-lined lid	Cool to 4°C +/- 2°C	30 days to extraction
			45 days to analysis
PCBs (GEHR Modified SW-846 8082)	2-1 liter amber glass bottles with Teflon®-lined lid	Cool to 4°C +/- 2°C	7 days to extraction
			40 days to analysis
PCBs (Modified Green Bay Mass Balance)	2-1 liter amber glass bottles with Teflon®-lined lid	Cool to 4°C +/- 2°C	7 days to extraction
			40 days to analysis
TAL Metals (USEPA 200.8)	1liter plastic bottle	HNO_3 to pH<2 Cool to 4°C +/- 2°C	180 days to analysis
Mercury (USEPA 245.1)			28 days to analysis
Suspended Solids (TSS) (USEPA 160.2)	500ml plastic bottle	Cool to 4°C +/- 2°C	7 days to analysis
NH_3/TKN/NO_2/NO_3 (USEPA 350.2/351.3/353.3/354.1)	1liter plastic bottle	H_2SO_4 to pH <2, Cool to 4° +/- 2°C	28 days to analysis (48 hours for NO_2/NO_3)
Biochemical Oxygen Demand (USEPA 405.1)	1 liter plastic bottle	Cool to 4°C +/- 2°C	48 hours to analysis
Total Phosphorus/(USEPA 365.2)	1 liter plastic bottle	H2SO4 to pH<2, Cool to 4°C +/- 2°C	28 days to analysis
Chemical Oxygen Demand (USEPA 410.2)			28 days to analysis
Total Organic Carbon (Tekmar Dohrmann)			28 days to analysis
Turbidity (USEPA 180.1)	2-1 liter plastic bottles	Cool to 4°C +/- 2°C	48 hours to analysis

Notes:

1 USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste. SW-846 3rd ed. Washington, D.C. 1996.*
USEPA. Methods for Chemical Analysis of Water and Waste. EMSL-Cincinnati. 1983:
APHA. *Standard Methods for the Examination of Water and Wastewater.* Washington, DC. 1998.
ASTM International. 2003. *Annual Book of ASTM Standards 2003 Section 4 Construction* , Volume 04.08. West Conshohocken, PA. ASTM International
Department of the Army. 1986. *Engineering Manual Laboratory Soils Testing* . Washington, D.C. Department of the Army, Office of the Chief of Engineers

2 All holding times are measured from date of collection.

3 NS = Not Specified

4 NA = Not Applicable

5 Sample container requirements may be modified based on available sample volume.

General Electric Company
Hudson River PCBs Superfund Site
Treatability Studies Work Plan

Table 9 - Sample Quantities and Quality Control Frequencies

Parameter	Estimated Test Batches	Estimated Environmental Sample Quantity	Treatability Laboratory/Field QC Analyses[8]						Analytical Laboratory QC Sample						Total
			Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
			Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Solids															
PAH (SW-846 8270C/3545)	1	TBD	NA	--	1/batch	TBD	1/batch	TBD	1/batch	TBD	1/batch	TBD	NA	--	TBD
PCBs (GEHR Modified SW-846 8082)	31	TBD	NA	--	1/batch	TBD	1/batch	TBD	1/batch	TBD	1/batch	TBD	NA	--	TBD
PCDD/PCDF (USEPA 1613B)	12	TBD	NA	--	1/batch	TBD	1/batch	TBD	NA	--	NA	--	NA	--	TBD
TAL Metals (SW-846 6010B/7471A)	20	TBD	NA	--	1/batch	TBD	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
TCLP-Volatiles (SW-846 1311/8260B)	11	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	NA	--	TBD
TCLP-Semivolatiles (SW-846 1311/3510C/3520C/8270C)	11	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	NA	--	TBD
TCLP-Pesticides (SW-846 1311/3510C/3520C/8081A)	11	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	NA	--	TBD
TCLP-Herbicides (SW-846 1311/8151A)	11	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	NA	--	TBD
TCLP-Metals (SW-846 1311/3010A/6010B/7470A)	11	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	NA	--	TBD
pH (USEPA 9045C)	18	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Total P/PO_4 (USEPA 365.2)	1	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
NH_3/TKN (USEPA 350.1/351.3)	1	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
TOC (Lloyd Kahn)	16	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Paint Filter (SW-846 Method 9095A)	21	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Specific Gravity (ASTM D854)	19	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Grain size (ASTM D422/ASTM D1140)	9	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Atterberg limits (ASTM D4318)	19	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Unconfined Compressive Strength (ASTM D2166-00)	10	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Bulk Density (ASTM D4531, modified)	1	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Water Content (SM 2540G)	59	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Consolidation (ASTM D2435)	11	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Dredge Elutriate Test															
PCBs (Modified Green Bay Mass Balance)	NA	28	NA	--	NA	--	NA	--	1/20	2	1/20	2	NA	--	32
TAL Metals (USEPA 200.8/245.1)	NA	24	NA	--	NA	--	NA	--	1/20	2	NA	--	1/20	2	28
Suspended Solids (TSS) (EPA 160.2)	NA	24	NA	--	NA	--	NA	--	NA	--	NA	--	1/20	2	26
Total Organic Carbon (Tekmar Dohrmann)	NA	24	NA	--	NA	--	NA	--	1/20	2	NA	--	1/20	2	28
Water															
PAH (SW-846 Method 8270C/3520C)	42	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	1/batch	TBD	NA	--	TBD
PCBs (Modified Green Bay Mass Balance)	46	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	1/batch	TBD	NA		TBD
PCBs (GEHR Modified SW-846 8082)	60	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	1/batch	TBD	NA	--	TBD
PCDD/PCDF (USEPA 1613B)	53	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	NA	--	TBD
TAL Metals (USEPA 200.8/245.1)	96	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Chemical Oxygen Demand (USEPA 410.2)	14	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Biochemical Oxygen Demand (EPA 405.1)	42	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
NH_3/TKN/NO_2/NO_3 (USEPA 350.2/351.3/353.3/354.1)	42	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Total Phosphorus (EPA 365.2)	42	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Suspended Solids (TSS) (EPA 160.2)	78	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD
Total Organic Carbon (Tekmar Dohrmann)	89	TBD	NA	--	NA	--	1/batch	TBD	1/batch	TBD	NA	--	1/batch	TBD	TBD
Turbidity (USEPA 180.1)	43	TBD	NA	--	NA	--	1/batch	TBD	NA	--	NA	--	1/batch	TBD	TBD

Notes:
1. Test batches and sample counts are an approximation.
2. 1/batch = One QC sample per treatability study batch or one per 20 samples, whichever is more frequent.
3. Rinse blanks not required when dedicated sampling equipment is used.
4. Freq. = Frequency
5. NA = Not Applicable
6. No. = Number
7. QC = Quality Control
8. Treatability laboratory/analytical laboratory samples do not include control and/or replicate samples required by the treatability studies test standard operating procedures.
9. TBD = To be determined.

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park
Suite 319
Albany, NY 12203
(518) 862-2739
Dial Comm: 8 232-2739*
Telephone: (518) 862-2731

VIA FEDERAL EXPRESS AND FAX

June 30, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Hudson River Remedial Design AOC – Treatability Study Sample Collection Status

Dear Sir or Madam:

On April 19, 2004, we notified you of the initiation of sample collection activities for the Treatability Study (TS) work. The purpose of the present letter is to provide an update on the TS sampling activities currently underway. The initial sediment sample collection was completed between May 3 and 18, 2004. Following initial chemical and physical characterization of the

four material types, the S4 material (high PCBs, fine-grained) was found not to meet the specifications identified in the TS Work Plan. As a result, a second S4 sample location was immediately identified and collected on June 9, 2004. Following receipt of chemical and physical testing results, this material, like the first sample, was not found to be acceptable. A final S4 location was identified and sample collection was completed on June 23, 2004. Preliminary results from the chemical and physical testing of that sample were received late in the work day on June 28 and were subsequently reviewed by GE. On June 30 (today), GE determined, based on these results, that this material, designated S4B, meets the requirements identified in the TS Work Plan and is thus acceptable for use in the Treatbility Studies. PCB concentrations of S4B were 490 ppm (466 ppm in a duplicate sample) and the silt/clay fraction was greater than 80 percent by weight.

In accordance with the schedule in the TS Work Plan, samples must be delivered to the treatability test contractor within 7 days from receipt of acceptable characterization analyses for the S4 material. Since GE has just determined that this material is acceptable, and given the intervening Independence Day holiday, it is anticipated that delivery of the samples to the treatability test contractor will occur on July 7, 2004. The Treatability Studies will be completed 90 days thereafter or on October 5, 2004.

If you have any questions regarding this matter, do not hesitate to contact me at (518) 862-2739 or Bob Gibson at (518) 862-2736.

Sincerely,

John G. Haggard

JGH/bg
Enclosure

cc: Bob Gibson, GE
Michael Elder, GE
Barbara Ippolito, GE
Steve Garbaciak, BBL
Paul Doody, BBL
Don Sauda, BBL

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

July 23, 2004

Via Electronic Mail and
First Class Mail

John Haggard
General Electric Company
320 Great Oaks Office Park, Suite 323
Albany, New York 12202

Re: Corrective Action Memorandum 002 for Treatability Study Workplan
Hudson River PCBs Superfund Site

Dear Mr. Haggard:

This is to inform you that the United States Environmental Protection Agency (EPA) has reviewed GE's June 29, 2004 Corrective Action Memorandum 002 associated with the Treatability Study Workplan (TSWP) for the Hudson River PCBs Site. EPA is approving the Memorandum, but would like clarification concerning the laboratory methods for PCBs and some additional information.

Please confirm that the only change to laboratory methods for PCBs in water is for the rapid small-scale column tests (RSSCTs), which is to be changed to the Modified Green Bay Mass Balance Method and that all other analyses are expected to remain the same as cited in Table 2 of the final TSWP. In addition, please provide the modified SOP and the MDL study for GEHR Modified Method 8082.

Please call me at (212) 637-3952 if you have any questions.

Sincerely yours,

Doug Garbarini
Team Leader
Hudson River Team

cc: William Ports, NYSDEC

ORIGINAL

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 323
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

VIA FEDERAL EXPRESS AND FAX

August 6, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Hudson River – Treatability Study Work Plan - Corrective Action Memorandum No. 3

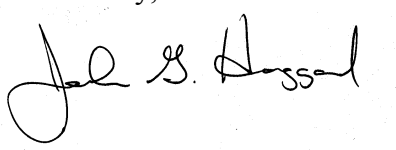
Dear Sir or Madam:

Attached please find Corrective Action Memorandum (CAM) No. 3 to the Treatability Study (TS) Work Plan. This CAM documents the final bulk sediment samples collected for

treatability testing and provides a new Standard Operating Procedure (SOP) for hydrocyclone performance testing.

We anticipate the hydrocyclone testing to be conducted at Waste Stream next week. We therefore request your prompt review and approval of this CAM so that the treatability testing can proceed smoothly and without interruption. If you should have any questions, please feel free to give Bob Gibson a call at (518) 862-2736.

Sincerely,



John G. Haggard

JGH/bg
Enclosure

cc: Ben Conetta, USEPA
Robert Gibson, GE
Barbara Ippolito, GE
Don Sauda, BBL
Paul Doody, BBL

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT
TREATABILITY STUDIES PROGRAM

Date: August 6, 2004
Organization Name: Blasland, Bouck & Lee, Inc. (BBL)
Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description:

1) The Standard Operating Procedure (SOP) for Sample Collection for Treatability Tests was included as Appendix 1 of the Treatability Studies Work Plan (TS Work Plan, BBL, February 2004) that was approved by USEPA on February 13, 2004. The Sample Collection for Treatability Tests SOP was modified in Corrective Action Memorandum No. 001, dated May 5, 2004. That SOP was approved verbally by the USEPA Oversight Contractor, Michael Johnson from Malcolm Pirnie, on May 3, 2004. Since that time, additional sediment sample volume was collected in order to meet the target PCB, bulk density and grain size requirements for each of the four sediment types to be used in the Treatability Studies.

2) The SOP for Size Separation Testing (Appendix 6 of the TS Work Plan) includes provisions for additional testing using density separations which simulate full-scale unit operations, such as a hydrocyclone. A new SOP for Hydrocyclone Performance Testing has been prepared.

Reported To: Bob Gibson, GE

Corrective Action:

1) The attached Table 1 from the SOP for Sample Collection for Treatability Tests has been modified to summarize the actual sample locations and volumes of sediment collected for Treatability Studies. The changes are summarized below.

- As shown on Table 1, S1 sediment was collected from River Section 1 (sample designated SX2) and River Section 3 (sample designated SX1). Additional S1 sediment was collected from River Section 1 (sample designated SX4) and River Section 3 (sample designated SX3) at the coordinates shown on Table 1. Collection of samples from these additional locations was verbally approved by the USEPA Oversight Contractor, Bryan Miner from the USACOE, on May 13, 2004. The S1 material being used in the Treatability Studies was formed by combining and homogenizing the SX2 and SX4 samples. The samples designated SX1 and SX3 are being temporarily stored in a refrigerated trailer at the GE facility in Fort Edward.

- As shown on Table 1, the S4 sample was formed by combining and homogenizing sediment from River Sections 1 and 2. Based on the PCB concentration, bulk density and grain size analysis, this sample did not meet the requirements for S4 material to be used in the Treatability Studies. Additional S4 sediment was collected from River Section 1 (sample designated S4A) at the coordinates shown on Table 1. Collection of samples from this additional location was verbally approved by the USEPA Oversight Contractor, Bryan Miner from the USACOE, on June 9, 2004. However, the S4A also did not meet the requirements for S4 material. Therefore, the samples designated S4 and S4A were not used in the Treatability Studies and are being temporarily stored in a refrigerated trailer at the GE facility in Fort Edward.

- Additional S4 sediment was collected from River Section 1 (sample designated S4B) at the coordinates shown on Table 1. In order to more precisely collect only S4 sediment, the samples were collected using a push coring technique with 3-inch (o.d.) Lexan® coring tubes. Collection of samples from this additional location using the push coring technique was verbally approved by the USEPA Oversight Contractor, Bryan Miner from the USACOE, on June 22, 2004. Based on the PCB concentration, bulk density and grain size analysis, this sample did meet the requirements for S4 material and was used in the Treatability Studies.

2) The attached Hydrocyclone Performance Testing SOP (Attachment 1) has been prepared to be included in the TS Work Plan as Appendix 33. An updated Table of Contents and Appendix 33 flysheet is also attached. It is anticipated that one of the sediment samples being temporarily stored in a refrigerated trailer at the GE facility in Fort Edward will be used during the Hydrocyclone Performance Testing. The testing is currently scheduled to begin the week of August 9, 2004.

Reviewed and Implemented By: Don Sauda (BBL)

cc: GE Project Manager: Bob Gibson
Other Distribution: Amy Toth (Waste Stream)

Table 1 - Treatability Studies Sediment Sample Collection

Treatability Studies Sediment Sampling Locations	Coordinates (New York State Plane East, NAD 83)								Maximum Collection Depth	Quantity of Sediment	
	NW Corner		SW Corner		SE Corner		NE Corner				
	Northing	Easting	Northing	Easting	Northing	Easting	Northing	Easting	(Feet)	(Gallons)	(Cubic Feet)
S1 - River Section 1 (originally designated SX2)	1,595,947	737,592	1,595,848	737,592	1,595,848	737,712	1,595,947	737,712	1.2	140	18.7
S1 - River Section 1 (originally designated SX4)	1,615,064	735,096	1,614,977	735,087	1,614,977	735,202	1,615,068	735,212	0.2		
S2 - River Section 1	1,608,911	732,730	1,608,805	732,724	1,608,805	732,835	1,608,899	732,829	5.3	82.5	11.0
S2 - River Section 2	1,571,444	735,583	1,571,337	735,583	1,571,337	735,688	1,571,444	735,688	1.9		
S3 - River Section 1	1,607,785	732,221	1,607,690	732,221	1,607,690	732,312	1,607,785	732,312	3.8	112.5	15.0
S3 - River Section 3	1,503,237	725,467	1,503,122	725,467	1,503,122	725,565	1,503,237	725,566	5.1		
S4B - River Section 1	1,610,060	733,457	1,610,060	733,457	1,610,060	733,457	1,610,060	733,457	3.0	105	14.0
S4B - River Section 1	1,609,990	733,416	1,609,990	733,416	1,609,990	733,416	1,609,990	733,416	3.0		
SX1 - River Section 3	1,498,640	724,536	1,498,534	724,536	1,498,534	724,637	1,498,640	724,637	0.2	60	8.0
SX3 - River Section 3	1,498,640	724,536	1,498,534	724,536	1,498,534	724,637	1,498,640	724,637	2.0	72.5	9.7
S4 - River Section 1	1,593,043	736,251	1,592,927	736,251	1,592,927	736,359	1,593,043	736,359	3.1	102.5	13.7
S4 - River Section 2	1,576,492	737,715	1,576,398	737,715	1,576,398	737,841	1,576,492	737,841	3.0		
S4A - River Section 1	1,595,855	737,819	1,595,855	737,819	1,595,855	737,819	1,595,855	737,819	0.2	95	12.7
								Total:		***770***	***102.8***

Notes:

1. Material sent to Treatability Studies Laboratories in July 2004

S1 =	140	Gallons	0.7	Cubic Yards
S2 =	82.5	Gallons	0.4	Cubic Yards
S3 =	112.5	Gallons	0.6	Cubic Yards
S4B =	105	Gallons	0.5	Cubic Yards
Total =	440	Gallons	2.2	Cubic Yards

2. Material being temporarily stored at GE Fort Edward Facility

SX1 =	60	Gallons	0.3	Cubic Yards
SX3 =	72.5	Gallons	0.4	Cubic Yards
S4 =	102.5	Gallons	0.5	Cubic Yards
S4A =	95	Gallons	0.5	Cubic Yards
Total =	330	Gallons	1.7	Cubic Yards

Appendices

Appendix 33

SOP – Hydrocyclone Performance Testing

BBL®
BLASLAND, BOUCK & LEE, INC.
engineers, scientists, economists

Standard Operating Procedure
Hydrocyclone Performance Testing

1. Scope and Application

This standard operating procedure (SOP) lists the steps to be performed to insure safe and effective operation of the hydrocyclone. Hydrocyclone tests are performed to separate fractions of a material based on density and particle size. The physical and chemical properties of the separated fractions can be measured. An SOP for hydrocyclone performance testing was not included in the Treatability Studies Work Plan (TS Work Plan) (Blasland, Bouck & Lee, Inc. [BBL], 2004 However, this procedure has been developed to meet the density separation objective (DQO 4b.(1a) and 4c.(1a), per Section 2.4.2 and the Size Separation Testing SOP (Appendix 6 of the TS Work Plan), pending the sieve test results.

2. Equipment List

The following materials, as required, will be available during this procedure:

- Hydrocyclone and fittings, including apexes and vortex finders;
- Calibrated pressure gauge with oil-filled dampener;
- Sample bottles;
- Graduated container to collect underflow and overflow streams;
- Analytical balance;
- Steel ruler; and
- Stopwatch or timer.

3. Health and Safety Considerations

All work will be in accordance with Sevenson Environmental Services, Inc.'s Corporate Health and Safety Plan. Should you have any question or concern about the sample or procedure, address this with your supervisor or health and/or safety officer prior to beginning work.

Hydrocyclones are to be assembled and raised into position in a safe manner using a hoist, if necessary. Inlet and overflow hoses should be securely affixed to their appropriate fitting so that they will not blow off under pressure. The pump should be started to generate the lowest possible pressure. The pressure should be slowly increased to the operating pressure while observing any signs of leaking or hoses blowing off.

4. Procedure

4.1. Review the test work to be performed. Take care to note the test objectives, equipment to be used, sequence of testing, the conditions (pressure, feed percent solids, underflow percent solids desires, etc.).

4.2. Hydrocyclone Test Procedure Pump/Sump/Hydrocyclone Setup

4.2.1. Remove the drain plug from the large sump, and open the drain valve on the small sump.

4.2.2. Thoroughly flush the sump until the water is clear. Reinstall the drain plug or close the valve.

4.2.3. Obtain the appropriate drawings and parts lists. Assemble the hydrocyclone taking care to ensure that the correct fittings are installed. Measure the apex and vortex finder diameters if old or unmarked parts are used.

4.2.4. If a hydrocyclone is already pre-assembled, disassemble to make sure that no internal parts are damaged, the correct inlet head liner is installed and is fitted properly, and that no residual solids are present in any of the internal crevices. It is especially important to remove the apex housing to insure that no residual material is to be found in that area.

4.2.5. When assembled, check to be sure that no reverse shelf exists. A piece of wire with a bend on the end can be used to feel for the shelf. If one is found, disassemble the hydrocyclone and readjust.

4.2.6. Install the hydrocyclone over the appropriate pump/sump. Install the overflow pipe or hose.

4.2.7. Install the calibrated pressure gauge with its accompanying oil filled pulsation dampener as close to the inlet as possible. (See “Gauge and Pulsation Dampener Guidelines” presented below).

4.2.8. Fill the sump with clean water.

4.2.9. Partially open the sump bypass valve.

4.2.10. Start the pump.

4.2.11. Slowly increase the pump speed to the target pressure, observing the pressure gauge and watching for leaks.

4.2.12. Fill the sump to the target level (typically a minimum 30 seconds of retention time), start the mixer, and add the appropriate amount sample. Mix thoroughly.

The bypass valve can be used to take a feed sample to measure feed percent solids. Adjust and measure percent solids as needed.

4.2.13. When the percent solids are correct, adjust the pump speed to give the target operating pressure. Observe the flow out of the apex. If roping (i.e., less than typical 120° spray) occurs due to solids overloading, use a larger diameter apex. If the underflow is too dilute based on percent solids concentration, use a smaller diameter apex.

4.2.14. Take grab samples of the underflow and overflow and observe the differences.

4.2.15. When the operating conditions and the underflow and overflow are deemed acceptable, take underflow and overflow samples. Both must be taken simultaneously. Sample full stream where possible using a plastic beaker or sample bottle to sample the underflow and a larger beaker or sample bottle to sample the overflow. Where underflow and overflow flow rates are large, sample cutters should be used simultaneously or two pails can be used if larger samples are required.

4.2.16. Recheck the feed pressure and perform the capacity determination as described below. Place the appropriate calibrated collection vessel under the apex and collect the underflow for a known amount of time. Measure the volume of the fluid collected. Return this to the sump. Direct the overflow into a larger calibrated vessel for a known amount of time. Measure the volume of fluid collected. Return the fluid to the sump. Determine the hydrocyclone flow rate at the pressure used based on performance data provided by Krebs Engineers. Immediately check the published capacity curve to be certain that this experimentally determined capacity is in agreement with the curve. If it is not, perform the capacity determination again. If the two numbers still do not agree, check the feed pressure gauge (see below).

4.3. Gauge and Pulsation Dampener Guidelines

The measuring devices used in hydrocyclone testing are the stopwatch, beakers, calibrated collection containers, and the pressure gauge with its associated pulsation dampener. The pressure gauge is the most important because the pressure drop across the hydrocyclone affects both performance and capacity. Therefore the pressure gauge must be accurate.

4.3.1. Obtain a pressure gauge known to be accurate to 5% or better. The gauge should be such that the desired working pressure is within the middle range of the gauge.

4.3.2. Mount a flush valve on the appropriate port in the pulsation dampener.

4.3.3. Fill the pulsation dampener with the appropriate oil making sure no air is present in the pulsation dampener.

4.3.4. Attach the gauge plus dampener to the hydrocyclone inlet. Make sure there are no leaks.

4.3.5. Before each set of tests, flush the dampener using a water line.

4.3.6. Observe normal gauge operation.

4.3.7. If any of the following occurs, the gauge must be removed, inspected, checked or recalibrated, and the pulsation dampener and mounting nipple must be flushed:

- Erratic needle movement;
- Gauge does not return to 0 psi with no pressure on it;
- Gauge pressure increase slowly or not at all as pump speed increases;
- Blown gauge; and
- Calculated hydrocyclone capacity does not correspond to published capacity.

5. References

BBL. 2004. *Treatability Studies Work Plan*. Hudson River PCBs Superfund Site. Prepared for General Electric Company, Albany, New York.

Krebs Engineers. 1999. *Hydrocyclone Performance Testing*.

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

VIA FEDERAL EXPRESS

August 24, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Corrective Action Memorandum No. 003 for Treatability Study Work Plan

Dear Sir or Madam:

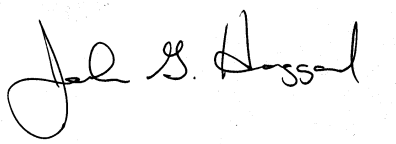
On August 6, 2004, the General Electric Company (GE) submitted Corrective Action Memorandum (CAM) No. 003 to the United States Environmental Protection Agency (USEPA). Among the items included with CAM No. 003 was a new Standard Operating Procedure (SOP) for hydrocyclone performance testing. On August 9, 2004, USEPA approved CAM No. 003 and

requested additional information on analytical tests, if any, planned for the overflow and underflow samples from the hydrocyclone performance testing.

The hydrocyclone performance testing includes provisions for analytical tests on the overflow and underflow samples. For the overflow, samples will be collected for analytical tests for PCBs and total suspended solids. In the underflow, it is anticipated that there will be distinct solid and liquid phases in collected samples. The liquid phase will be decanted off the solid phase so that analytical tests can be performed on each phase. For the liquid phase in the underflow, the volume will be measured and analytical tests will be performed for PCBs and total suspended solids. For the solid phase in the underflow, the weight will be measured and analytical tests will be performed for PCBs, percent solids and grain size distribution.

If you should have any questions, please feel free to give Bob Gibson a call at (518) 862-2736.

Sincerely



John G. Haggard

cc: Ben Conetta, EPA
Michael Elder, GE
Robert Gibson, GE
Don Sauda, BBL

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 323
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

VIA FEDERAL EXPRESS AND FAX

August 9, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Hudson River – Treatability Study Work Plan - Corrective Action Memorandum No. 4

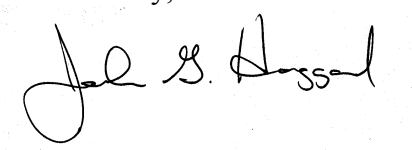
Dear Sir or Madam:

Attached please find Corrective Action Memorandum (CAM) No. 4 to the Treatability Study (TS) Work Plan. This CAM documents a series of modifications regarding the sediment

types that will be used in several of the treatability tests. The rationale for altering the sediment types to be evaluated in the various treatability tests is explained in the CAM and summarized in the attached table.

Many of the proposed substitutions will occur during this week's treatability testing. We therefore request your prompt review and approval of this. If you should have any questions, please feel free to give Bob Gibson a call at (518) 862-2736.

Sincerely,



John G. Haggard

JGH/bg
Enclosure

cc: Ben Conetta, USEPA
Robert Gibson, GE
Barbara Ippolito, GE
Don Sauda, BBL
Paul Doody, BBL

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT
TREATABILITY STUDIES PROGRAM

Date: August 9, 2004

Organization Name: Blasland, Bouck & Lee, Inc. (BBL)

Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description:

The Treatability Studies Work Plan (TS Work Plan) was originally prepared by BBL in December 2003. Based on comments from USEPA on January 16, 2004, the TS Work Plan was revised in February 2004 and approved by USEPA on February 13, 2004. Since that time, a number of activities have provided additional information related to the Treatability Studies. These activities include initiation of the Phase I Intermediate Design, selection of a Contractor to conduct the Treatability Studies, collection and analysis of river sediment and water samples for the Treatability Studies, and completion of approximately 30% of the Treatability Studies.

Using the most current information available, BBL is proposing a number of revisions for the material specified to be used in various Treatability Studies. The key revisions are discussed below.

- The S4 material being used in the Treatability Studies was designated S4B at the time of collection, so BBL proposes that this designation be maintained.
- In the TS Work Plan, Size Separation Tests were proposed for H1 and H2 slurries using S1 and S2 sediments. Since the test involves a wet washing step, the separation is not sensitive to initial percent solids of the material introduced to the sieves. Therefore, following completion of the Size Separation Tests for H1S1 and H1S2 slurries, BBL determined that running these tests using these same sediments prepared as H2 slurries would be a duplication. Instead, BBL proposes to use S3 and S4 sediment so data can be collected on all four sediment types. Additionally, the river water used to make H1 or H2 slurries is not necessary to meet the Data Quality Objectives (DQOs) for this test, so the S3 and S4 sediment can be directly used in Size Separation Tests. Therefore, raw sediment will be washed through the sieves using river water.
- DQO 5 in the TS Work Plan covers the tests needed to develop the water processing design including the Precipitation/Flocculation Filtrate Settling Tests, Filtrate Column Settling Tests, Multimedia Filtration (MMF) Tests, Carbon Column Tests, and Rapid Small-Scale Column Tests (RSSCT). One of the keys to these tests is to have a representative feed to the High-Volume

Plate and Frame Filter Press Tests used to produce filtrate for these tests. Based on initial testing with H2 slurries, the majority of the gravel and sand (approximately 85-50% with S1, S2 and S3 sediments) quickly settles out of the slurries and will be difficult to keep fluidized in the feed to the filter press. The resultant solids in the filter press feed will be much less than the 5% target and, therefore, is outside the specified range of conditions to be evaluated in the Treatability Studies. Therefore, BBL proposes that S4B, with an initial solids content of 5%, is the only sediment to be prepared as H2 slurry fed to the High-Volume Plate and Frame Filter Press Test and the subsequent water processing tests. The S4B sediment contains about 17% gravel and sand, so the H2S4B slurry can be kept fluidized. Additionally, it is proposed that the H1S2 slurry, with an initial solids content of 25%, which was not originally included in these tests, be added for MMF, carbon column and/or RSCCT testing.

- The TS Work Plan specified that H2S1 and H2S3 were two of the slurries to be used in the Dewatering Polymer Tests. BBL will substitute the H2S4B slurry for the H2S1 and use the settled solids from the Primary Sedimentation Test with H2S3 instead of the slurry directly.
- The TS Work Plan specified that the slurries to be used in the Primary Sedimentation Tests may be conditioned with polymer. BBL has subsequently decided that polymer is not going to be used for those tests. Therefore, the Primary Sedimentation Polymer Tests are not necessary.
- The TS Work Plan did not initially specify using the S1 sediment in the Primary Sedimentation Tests. BBL plans to add a test using H1S1 slurry.
- Per the TS Work Plan, H1S4B, H2S3, and H2S4B slurries were to be used for centrifuge tests. Also, the TS Work Plan specified two undefined slurries conditioned with polymer to be used in centrifuge tests. BBL has chosen H1S3 and H1S4B as the conditioned slurries. Additionally, BBL proposes to substitute the H1S3 slurry (without polymer) in place of H2S3 so that a comparison can be made between H1S3 and H1S4B slurries with and with polymer conditioning.
- The TS Work Plan specified H2S2 as one of the slurries to be used in the Mixing Energy Tests. BBL proposes to substitute the H1S4B slurry in place of H2S2, because of settling of the coarse fraction in this slurry.
- The TS Work Plan did not specify conducting Laboratory Belt Filter Press Tests. BBL proposes to add tests using H1S3 and H1S4B slurries conditioned with polymer. A Standard Operating Procedure (SOP-13) has been submitted to USEPA.
- The TS Work Plan specified density separation tests with high density liquids. Instead, BBL will conduct Hydrocyclone Performance Testing to evaluate density effects. BBL will use the S4 sediment originally collected that is not being used in other Treatability Studies. SOP-33 has been submitted to USEPA (CAM No. 3).

- BBL may consider dewatering the overflow from hydrocyclone testing of the S4 material (after "thickening" using settling, decanting and polymer conditioning) with the plate and frame filter press. This could yield another water sample for MMF, carbon column, and/or RSCCT testing.

The attached table summarizes the material originally proposed in the TS Work Plan along with the revisions currently being proposed. The above-described key revisions are shown along with other revisions to related Treatability Studies.

Reported To: Bob Gibson, GE

Corrective Action:

The Treatability Studies will be completed based on the changes described above. These changes will impact Treatability Studies scheduled to begin the week of August 9, 2004. Therefore, an expeditious approval of these changes by USEPA is requested.

Reviewed and Implemented By: Don Sauda (BBL)

cc: GE Project Manager: Bob Gibson
Other Distribution: Amy Toth (Waste Stream)

General Electric Company
Hudson River PCBs Superfund Site
Treatability Studies Revisions

Treatability Tests	TS Work Plan Section	Material to be used in Test	
		Original Plan	Revised Plan
Dredge Elutriate Tests	2.3.1 & 2.3.2	S1, S2, S3, S4	S1, S2, S3, S4B
Stabilization/Solidification Tests	2.4.1 & 2.4.9	M1S1, M1S2, M1S3, M1S4 H1S1(FC), H1S3, H1S4 (FC) H2S1, H2S3(FC), H2S4 (FC)	M1S1, M1S2, M1S3, M1S4B H1S1, H1S2, H1S3, H1S4B (all FC) H2S4B (FC)
Size Separation Tests	2.4.2	H1S1, H1S2, H2S1, H2S2	H1S1, H1S2, S3, S4B
Drainage Tests	2.4.3	H1S1, H1S2, H2S1, H2S2	H1S1, H1S2
Primary Sedimentation Polymer Tests	2.4.4	H1S2, H1S3, H2S1, H2S2, H2S3	
Primary Sedimentation Tests	2.4.5	H1S2, H1S3 H2S2, H2S3, H2S4	H1S1, H1S2, H1S3 H2S2, H2S3, H2S4B
Dewatering Polymer Tests	2.4.6	H1S1, H1S2, H1S3, H1S4 H2S1, H2S3 H1S3, H2S2, H2S4 (all SS)	H1S1, H1S2, H1S3, H1S4B H2S4B H1S3, H2S2, H2S3, H2S4B (all SS)
Mixing Sub-Studies Tests	2.4.6	H1S3, H2S1, H2S2(SS)	H1S3, H2S2(SS), H2S4B(SS)
Cake Release Screening Tests	2.4.6	H1S2, H1S3, H2S1, H2S3	H1S2, H1S3, H2S3(SS), H2S4B(SS)
Plate and Frame Filter Press Tests	2.4.7	H1S2, H1S3, H2S2(SS), H2S4(SS)	H1S2, H1S3, H2S2(SS), H2S4B(SS)
Cake Solids vs. Time Tests	2.4.7	H1S3, H2S1, H2S3	H1S3, H2S2(SS), H2S4B(SS)
High-Volume Plate and Frame Filter Press Tests	2.4.7	H1S1, H1S3, H1S4 H2S1, H2S2, H2S3, H2S4	H1S1, H1S2, H1S3, H1S4B H2S4B
Centrifuge Tests	2.4.8	H1S4, H2S3, H2S4 Two (w/P)	H1S3, H1S4B, H2S4B H1S3(w/P), H1S4B(w/P)
Mixing Energy Tests	2.4.10	H1S1, H1S2, H2S1, H2S2, H2S3	H1S1, H1S2, H1S4B, H2S1, H2S3
Precipitation/Flocculation Filtrate Settling Tests	2.5.1	H1S1, H1S3, H1S4 H2S1, H2S2, H2S3, H2S4	H1S1, H1S2, H1S3, H1S4B H2S4B
Filtrate Column Settling Tests	2.5.1	H1S1, H1S3, H1S4 H2S1, H2S2, H2S3, H2S4	H1S1, H1S2, H1S3, H1S4B H2S4B
Multimedia Filtration Tests	2.5.2	H1S1, H1S3, H1S4 H2S1, H2S2, H2S3, H2S4	H1S1, H1S2, H1S3, H1S4B H2S4B
Rapid Small-Scale Column Tests	2.5.3	H1S1, H1S3, H1S4 H2S1, H2S2, H2S3, H2S4	H1S1, H1S2, H1S3, H1S4B H2S4B
Carbon Column Tests	2.5.3	H1S1, H1S3, H1S4 H2S1, H2S2, H2S3, H2S4	H1S1, H1S2, H1S3, H1S4B H2S4B
Storage/Transportation Stability Shaker Tests	2.6.1	M1S1, M1S2, M1S3, M1S4 H1S1(FC), H1S3, H1S4 (FC) H2S1, H2S3, H2S4 (all FC)	M1S1, M1S2, M1S3, M1S4B H1S1, H1S2, H1S3, H1S4B (all FC) H2S4B (FC)
Laboratory Belt Filter Press Tests	SOP 13		H1S3(w/P), H1S4B(w/P)
Hydrocyclone Performance Testing	SOP 33		S4

Notes:

1) TS Work Plan = Treatability Studies Work Plan (Blasland, Bouck and Lee, Inc., February 2004)
2) FC = Filter Cake
3) SS = Settled Solids from Primary Sedimentation Tests
4) w/P = With Polymer
5) SOP = Standard Operating Procedure

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

August 9, 2004

Via Electronic Mail and
First Class Mail

John Haggard
General Electric Company
320 Great Oaks Office Park, Suite 323
Albany, New York 12202

Re: Corrective Action Memorandum 003 for Treatability Study Workplan
Hudson River PCBs Superfund Site

Dear Mr. Haggard:

This is to inform you that the United States Environmental Protection Agency (EPA) has reviewed GE's August 6, 2004 Corrective Action Memorandum 003 associated with the Treatability Study Workplan (TSWP) for the Hudson River PCBs Site. EPA is approving the Memorandum and is requesting additional information on tests, if any, that are planned for the overflow and underflow samples from the hydrocyclone performance tests. Results from the testing of the underflow and overflow samples (e.g., percent solids, particle size distribution, PCB concentrations in underflow vs. overflow; whether the underflow will drain rapidly by gravity so as to pass a paint filter test, etc.) could provide useful information for the design.

Please call me at (212) 637-3952 if you have any questions.

Sincerely yours,

Doug Garbarini
Team Leader
Hudson River Team

cc: William Ports, NYSDEC

ORIGINAL

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

Via Federal Express and Electronic Mail

September 20, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Treatability Study Completion Schedule

Dear Sir or Madam:

General Electric Company (GE) has received data that indicate that the waters prepared as feedstock for the rapid small-scale column tests (RSSCTs) have lower than expected concentrations of PCBs. The RSSCT data quality objective (DQO), which was established in the Treatability Study Work Plan (TSWP) Section 2.5.3, will not be satisfied if the tests are conducted using these waters. Therefore, GE would like to postpone these tests until more representative feedstock water can be prepared.

The RSSCTs contain granular activated carbon (GAC) and simulate breakthrough of carbon beds. GE has designed this study to simulate about 6 months of full-scale operation with 23-days of testing. The apparatus was set up at the treatability laboratory on September 9, 2004 and the tests were scheduled to be completed in advance of our original completion date of October 5, 2004.

The RSSCT feed water had been pre-treated with a multi-media filter (MMF), per the TSWP. The MMF achieved 96% to 99.5% removal of PCBs for all samples. The PCB concentrations in the RSSCT feedstock are all not high enough to run effective GAC breakthrough analyses. GE would like to test waters with PCBs greater than 300 ng/L. Therefore GE proposes the following corrective action:

- Conduct high-volume filter press tests using a slurry of sample S4A, which has a PCB concentration of 100 ppm, and was recently transferred from storage in Fort Edward to the treatability laboratory. Approximately 150 gallons of filtrate will be produced.
- Perform confirmatory PCB analysis (using Method GEHR8082 [total and dissolved]) of the filter press filtrate and a sample passing a 5 micrometer (µm) membrane;
- Reconfigure the columns for the new test plan; and
- If the filtered PCB concentration is greater than 300 ng/L, the entire feedstock will be filtered with a 5 µm membrane and run through the RSSCT columns.

It will take approximately 4 weeks to generate the water sample from the S4A sediment sample and receive confirmatory data. The water will be run through the rapid small-scale GAC columns for 23 days. A contingency of 9 additional days is planned if breakthrough is not indicated after the first 23 days of operation. The work will begin on September 20 and be completed on or before November 19, 2004, contingent upon generation of a representative feed water.

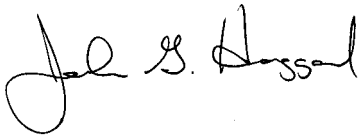
The TSWP specified that treatability studies would be completed within 90 days from receipt of samples by the treatability laboratory. The sediment samples arrived at the treatability laboratory on July 7, 2004, and thus the test program was scheduled to be completed on or before October 5, 2004. All treatability tests, except the RSSCTs, will be completed as planned. GE believes an extension for the RSSCTs is justified because:

- Conducting the test with waters which were prepared as feedstock do not meet the DQO for the RSSCTs, as established in the TSWP;
- The treatability tests are not on the project critical path; therefore, extending the date for completion of those tests will not delay delivery of the Phase 1 Intermediate Design Report; and
- No other treatability tests are linked to the completion of the RSSCT tests.

Therefore, in accordance with Paragraph 47.c of the Administrative Order on Consent for Remedial Design, GE requests that the date for completion of the treatability studies be revised

from October 5, 2004 to November 19, 2004. If you have any questions, please contact Scott Blaha (518-862-2738) or Bob Gibson (518-862-2736).

Sincerely,



John G. Haggard

JGH/bg

cc: Ben Conetta, EPA
Michael Elder, GE
Robert Gibson, GE
Don Sauda, BBL
Paul Doody, BBL
Barbara Ippolito, GE

September 30, 2004

Via Electronic Mail and
First Class Mail

John Haggard
General Electric Company
320 Great Oaks Office Park, Suite 323
Albany, New York 12202

Re: Treatability Study Completion Schedule
Hudson River PCBs Superfund Site

Dear Mr. Haggard:

This is to inform you that the United States Environmental Protection Agency (EPA) has reviewed GE's September 20, 2004 letter concerning the Treatability Study (TS) Completion Schedule for the Hudson River PCBs Site. Our comments are attached.

Your letter also refers to provisions in our agreement for revision of the completion date of the TS; you have requested an extension from October 5, 2004 to November 19, 2004 so that additional tests can be conducted. EPA has indicated that any additional information (or supplemental studies) GE believes is warranted for the design be identified and conducted without impact to the overall schedule. As the treatability study schedule is not on the critical path and the extension of the completion date will not delay the delivery of the Intermediate Design Report, the revised schedule is acceptable to EPA. This approach is consistent with EPA's previous correspondence concerning the TS, i.e., the need for any such studies should be identified early enough in the present TS process so that supplemental TS work would not be warranted subsequent to the Intermediate Remedial Design and no impacts to the schedule would be seen.

Please call me at (212) 637-3952 if you have any questions.

Sincerely yours,

Doug Garbarini
Team Leader
Hudson River Team

cc: William Ports, NYSDEC

Subject: Comments on Treatability Study Completion Schedule and RSSCT Test Influent

We have reviewed the GE correspondence dated September 20, 2004 regarding the Treatability Study Completion Schedule. Our comments on the document are provided below.

1. GE proposes to test the new batch of influent for the RSSCT testing via GEHR 8082. Please confirm that GEHR 8082 has adequate analytical sensitivity to detect PCBs at concentrations near 300 ng/L.

2. The effluent from the filter presses will be filtered using a 5 micron filter to avoid potential loss of PCBs across the multi media filter (MMF), as may have occurred during the prior testing attempt. GE should consider sending running a sample of the filter press effluent (following confirmatory analysis for dissolved and total PCBs) through the MMF again as a check on the previous findings.

3. It is possible that a cellulose 5 micron filter may adsorb dissolved phase PCBs from the planned RSSCT influent, and it appears that GE is addressing this issue by testing a sample of the filtrant prior to filtering the entire batch of RSSCT influent. If the utility of the 5 micron filter appears problematic, GE should consider using glass fiber filters.

4. Please provide further schedule information regarding the 4-week duration for the preparation of the RSSCT influent (e.g., time to run S4A slurry through filter presses, laboratory turnaround time on confirmatory samples, results review time, etc.).

5. It appears that the effluent from the RSSCT testing is to be analyzed for TOC and PCBs via GEHR 8082. Considering the currently available information regarding discharge requirements, it may be appropriate to analyze the effluent via Method 608 and the modified Green Bay Method, as well. GE should indicate whether all discharge compliance characterization is to be accomplished using the effluent from the Carbon Column Tests, and even if so, discuss the need for analysis via Method 608.

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

Via Federal Express

October 8, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

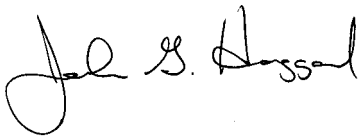
Re: Response to EPA Request for RSSCT Test Plan Information

Dear Mr. Garbarini:

General Electric Company (GE) has received a letter from the Environmental Protection Agency (EPA), dated September 30, 2004, providing comments regarding the Treatability Study (TS) completion schedule. In that letter, EPA approved the extension of the Rapid Small Scale Column Tests (RSSCTs), thus extending the TS completion date to November 19, 2004. It should be noted that the RSSCTs were prescribed in the TS Work Plan (February 2004) and are not additional or supplemental tests. EPA's letter also indicates any supplemental TS work should be identified early so that such work would not be necessary after the Intermediate Design. While GE may implement additional treatability studies in advance of submitting the Intermediate Design report (IDR), the Remedial Design (RD) Work Plan, which is part of the RD Administrative Order on Consent (RD AOC), provides explicitly (on page 2-20) that the IDR will include recommendations for supplemental treatability studies (if needed) and that the results of those supplemental studies will be documented in the Final Design Report. In accordance with that Work Plan, GE maintains the ability to identify supplemental treatability studies, in the Intermediate Remedial Design Report, and to perform those studies between Intermediate and Final Design.

EPA attached comments to the September 30, 2004 letter, which request additional information regarding the RSSCTs. A response is provided in the attachment to this letter. Please contact me or Scott Blaha (518-862-2738) for clarification.

Sincerely,



John G. Haggard

JGH/bg
Attachment
cc: Ben Conetta, EPA
Bill Ports, NYSDEC
Scott Blaha, GE
Michael Elder, GE
Robert Gibson, GE
Don Sauda, BBL
Paul Doody, BBL
Barbara Ippolito, GE

Response to EPA Comments (September 30, 2004) on Treatability Study Completion Schedule and RSSCT Test Influent

1. GE proposes to test the new batch of influent for the RSSCT testing via GEHR 8082. Please confirm that GEHR 8082 has adequate analytical sensitivity to detect PCBs at concentrations near 300 ng/L.

Response: GEHR 8082 has adequate sensitivity to detect PCBs at concentrations near 300 ng/L. A method detection study for analysis of aqueous samples by this method is attached to this response.

2. The effluent from the filter presses will be filtered using a 5 micron filter to avoid potential loss of PCBs across the multi media filter (MMF), as may have occurred during the prior testing attempt. GE should consider sending running (sic) a sample of the filter press effluent (following confirmatory analysis for dissolved and total PCBs) through the MMF again as a check on the previous findings.

Response: Additional testing of the MMF to support the Intermediate Design is not planned. More sediment would have to be collected and pressed to produce the large volume of water required to run the MMF test suggested by EPA. These activities would further extend of the schedule.

3. It is possible that a cellulose 5 micron filter may adsorb dissolved phase PCBs from the planned RSSCT influent, and it appears that GE is addressing this issue by testing a sample of the filtrant (sic) prior to filtering the entire batch of RSSCT influent. If the utility of the 5 micron filter appears problematic, GE should consider using glass fiber filters.

Response: A bag filter has been selected to pre-treat the MMF feed. A bag filter will be the most reliable method to filter the entire water volume in the laboratory. This pre-filtration step will reduce the potential for plugging the RSSCTs. A membrane with the same specifications of the bag filter was used for analytical purposes, to obtain representative results for a small sample volume. If the loss of PCBs across this membrane is significant, GE will consider other filter materials.

4. Please provide further schedule information regarding the 4-week duration for the preparation of the RSSCT influent (e.g., time to run S4A slurry through filter presses, laboratory turnaround time on confirmatory samples, results review time, etc.).

Response: The plan for the 4-week test preparation was, as follows:
September 20 – October 1: generate approximately 150 gallons of filtrate from the plate & frame press,
October 4- October 8: expedited turnaround for the PCB analysis and data review, and
October 11- October 15: pre-treat water with bag filter, and apparatus set-up.

5. It appears that the effluent from the RSSCT testing is to be analyzed for TOC and PCBs via GEHR 8082. Considering the currently available information regarding discharge

requirements, it may be appropriate to analyze the effluent via Method 608 and the modified Green Bay Method, as well. GE should indicate whether all discharge compliance characterization is to be accomplished using the effluent from the Carbon Column Tests, and even if so, discuss the need for analysis via Method 608.

Response: Per TS Corrective Action Memorandum No. 2 (dated June 29, 2004), the modified Green Bay Method will be applied during testing for the effluent samples and confirmatory analysis of the influent. Also, GE agrees to analyze the six rounds of effluent samples by Method 608.

GEHR 8082 was used as an initial indication of the PCB concentration in the RSSCT influent water. Data is pending for these analyses. The RSSCT influent will also be analyzed for mercury (Method 1631). GE will submit a Corrective Action Memorandum and laboratory SOPs to document these modifications.

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1016	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	46.6 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040212W1	02/12/04	040212W1	05/20/04	38.1	81.8%
2	040212W2	02/12/04	040212W2	05/20/04	41.8	89.8%
3	040212W3	02/12/04	040212W3	05/20/04	42.5	91.2%
4	040212W4	02/12/04	040212W4	05/20/04	39.8	85.4%
5	040212W5	02/12/04	040212W5	05/21/04	42.0	90.2%
6	040212W6	02/12/04	040212W6	05/21/04	45.2	97.1%
7	040212W7	02/12/04	040212W7	05/21/04	46.7	100.2%
8	040212W8	02/12/04	040212W8	05/21/04	48.4	104.0%

Number (n):	8	
AVG:	43.1	ng/L
STD (s):	3.48	ng/L
%RSD:	8.08%	
MDL:	10.43	ng/L
PQL:	52.2	ng/L
VALID:	valid	

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/20/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/20/04
QA/QC Officer:	Willam A. Kotas	Date:	5/20/04
Lab Director:	Robert E. Wagner	Date:	5/20/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1221	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	53.4 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040212W9	02/12/04	040212W9	05/21/04	36.3	68.0%
2	040212W10	02/12/04	040212W10	05/21/04	41.6	77.9%
3	040212W11	02/12/04	040212W11	05/21/04	39.4	73.8%
4	040212W12	02/12/04	040212W12	05/21/04	36.8	68.9%
5	040212W13	02/12/04	040212W13	05/21/04	41.8	78.2%
6	040212W14	02/12/04	040212W14	05/21/04	36.8	68.9%
7	040212W15	02/12/04	040212W15	05/21/04	37.1	69.5%
8	040212W16	02/12/04	040212W16	05/21/04	35.8	67.1%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	38.2	ng/L
STD (s):	2.39	ng/L
%RSD:	6.25%	
MDL:	7.16	ng/L
PQL:	35.8	ng/L
VALID:	0	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/21/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/21/04
QA/QC Officer:	Willam A. Kotas	Date:	5/21/04
Lab Director:	Robert E. Wagner	Date:	5/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1232	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	51.6 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040216W1	02/16/04	040216W1	05/22/04	48.8	94.5%
2	040216W2	02/16/04	040216W2	05/22/04	52.8	102.4%
3	040216W3	02/16/04	040216W3	05/22/04	51.8	100.4%
4	040216W4	02/16/04	040216W4	05/22/04	49.7	96.3%
5	040216W5	02/16/04	040216W5	05/22/04	52.4	101.6%
6	040216W6	02/16/04	040216W6	05/22/04	55.1	106.8%
7	040216W7	02/16/04	040216W7	05/22/04	49.3	95.6%
8	040216W8	02/16/04	040216W8	05/22/04	49.8	96.5%

Number (n):	8	
AVG:	51.2	ng/L
STD (s):	2.17	ng/L
%RSD:	4.25%	
MDL:	6.52	ng/L
PQL:	32.6	ng/L
VALID:	valid	

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/22/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/22/04
QA/QC Officer:	Willam A. Kotas	Date:	5/22/04
Lab Director:	Robert E. Wagner	Date:	5/22/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1242	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	49.5 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040213W1	02/13/04	040213W1	05/21/04	32.4	65.4%
2	040213W2	02/13/04	040213W2	05/21/04	36.4	73.6%
3	040213W3	02/13/04	040213W3	05/21/04	29.9	60.4%
4	040213W4	02/13/04	040213W4	05/21/04	38.3	77.4%
5	040213W5	02/13/04	040213W5	05/21/04	31.5	63.6%
6	040213W6	02/13/04	040213W6	05/21/04	35.0	70.8%
7	040213W7	02/13/04	040213W7	05/21/04	32.6	65.9%
8	040213W8	02/13/04	040213W8	05/21/04	33.7	68.1%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	33.7	ng/L
STD (s):	2.75	ng/L
%RSD:	8.14%	
MDL:	8.23	ng/L
PQL:	41.2	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/21/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/21/04
QA/QC Officer:	Willam A. Kotas	Date:	5/21/04
Lab Director:	Robert E. Wagner	Date:	5/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1248	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	50.0 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040213W9	02/13/04	040213W9	05/21/04	30.8	61.6%
2	040213W10	02/13/04	040213W10	05/21/04	33.4	66.8%
3	040213W11	02/13/04	040213W11	05/21/04	29.9	59.8%
4	040213W12	02/13/04	040213W12	05/21/04	32.3	64.6%
5	040213W13	02/13/04	040213W13	05/21/04	31.0	62.0%
6	040213W14	02/13/04	040213W14	05/21/04	38.6	77.1%
7	040213W15	02/13/04	040213W15	05/21/04	31.0	62.0%
8	040213W16	02/13/04	040213W16	05/21/04	36.7	73.5%

Number (n):	8	
AVG:	33.0	ng/L
STD (s):	3.11	ng/L
%RSD:	9.44%	
MDL:	9.33	ng/L
PQL:	46.7	ng/L
VALID:	valid	

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/21/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/21/04
QA/QC Officer:	Willam A. Kotas	Date:	5/21/04
Lab Director:	Robert E. Wagner	Date:	5/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1254	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	49.8 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040212W9	02/12/04	040212W9	05/21/04	58.9	118%
2	040212W10	02/12/04	040212W10	05/21/04	62.9	126%
3	040212W11	02/12/04	040212W11	05/21/04	62.7	126%
4	040212W12	02/12/04	040212W12	05/21/04	56.7	114%
5	040212W13	02/12/04	040212W13	05/21/04	60.5	121%
6	040212W14	02/12/04	040212W14	05/21/04	59.5	119%
7	040212W15	02/12/04	040212W15	05/21/04	60.7	122%
8	040212W16	02/12/04	040212W16	05/21/04	54.2	109%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	59.5	ng/L
STD (s):	2.95	ng/L
%RSD:	4.96%	
MDL:	8.85	ng/L
PQL:	44.2	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/21/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/21/04
QA/QC Officer:	Willam A. Kotas	Date:	5/21/04
Lab Director:	Robert E. Wagner	Date:	5/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_CLLE\[GC11_021604_1232_CLLE_.XLS]A

Date: 09-Sep-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1260	Analysis:	EPA METHOD 8082
Matrix:	WATER	Instrument:	GC-11
Extraction:	CLLE	Column:	DB-1
Spike conc:	49.6 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040212W1	02/12/04	040212W1	05/20/04	32.3	65.0%
2	040212W2	02/12/04	040212W2	05/20/04	35.4	71.3%
3	040212W3	02/12/04	040212W3	05/20/04	35.3	71.2%
4	040212W4	02/12/04	040212W4	05/20/04	32.7	65.9%
5	040212W5	02/12/04	040212W5	05/21/04	34.2	68.9%
6	040212W6	02/12/04	040212W6	05/21/04	35.9	72.4%
7	040212W7	02/12/04	040212W7	05/21/04	36.9	74.4%
8	040212W8	02/12/04	040212W8	05/21/04	37.4	75.4%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	35.0	ng/L
STD (s):	1.85	ng/L
%RSD:	5.27%	
MDL:	5.53	ng/L
PQL:	27.7	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Heather Carlson	Date:	5/20/04
Gas Chromatography Analyst:	Anthony Maiello	Date:	5/20/04
QA/QC Officer:	Willam A. Kotas	Date:	5/20/04
Lab Director:	Robert E. Wagner	Date:	5/20/04

Comments:

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

Via Electronic Mail & Federal Express

October 22, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

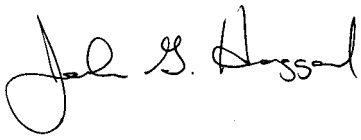
Re: Hudson River – Treatability Studies Work Plan - Corrective Action Memorandum No. 5

Dear Sir or Madam:

Attached please find Corrective Action Memorandum (CAM) No. 5 to the Treatability Studies (TS) Work Plan. This CAM proposes additional sediment and water samples collected for treatability testing.

We anticipate initiation of sample collection next week. We therefore request your prompt review and approval of this CAM so that the sample collection can be completed while access to the river is still available. If you should have any questions, please feel free to give Bob Gibson a call at (518) 862-2736.

Sincerely,



John G. Haggard

JGH/bg

Attachments

cc: Ben Conetta
Scott Blaha
Bob Gibson
Don Sauda
Barbara Ippolito
Paul Doody

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT
TREATABILITY STUDIES PROGRAM

Date: October 22, 2004

Organization Name: Blasland, Bouck & Lee, Inc. (BBL)
Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description:

Hydrocyclone tests were completed to study the separation of sediment based on density and particle size. A variety of cone configurations were tested to satisfied the data quality objectives (DQOs) described in the Treatability Study Work Plan (TS Work Plan, BBL, February 2004). GE would like to perform optimization studies to verify the most effective configuration and evaluate if the coarse fraction will have consistent PCB concentrations from test to test. More sediment must be collected to run these additional hydrocyclone tests.

The Standard Operating Procedure (SOP) for Sample Collection for Treatability Tests was included as Appendix 1 of the TS Work Plan that was approved by USEPA on February 13, 2004. The Sample Collection for Treatability Tests SOP was modified in Corrective Action Memorandum (CAM) No. 001, dated May 5, 2004. That SOP was approved verbally by the USEPA Oversight Contractor, Michael Johnson from Malcolm Pirnie, on May 3, 2004. Since that time, additional sediment sample volume was collected in order to meet the target PCB, bulk density and grain size requirements for each of the four sediment types to be used in the Treatability Studies. This was described in CAM No.003, dated August 6, 2004. CAM No. 003 was approved by USEPA on August 9, 2004.

CAM No. 003 also included an SOP for hydrocyclone performance testing. This SOP was also approved by USEPA on August 9, 2004, with a request for clarification of analytical tests during the hydrocyclone performance testing. This clarification was subsequently submitted to USEPA on August 24, 2004. In late August 2004, a series of hydrocyclone performance tests were conducted and additional tests are planned. However, in order to complete these tests, additional sediment and river water must be collected.

Reported To: Scott Blaha and Bob Gibson, GE

Corrective Action:

The attached Table 1 from the SOP for Sample Collection for Treatability Tests has been modified to summarize the sample locations and volumes of additional sediment to be collected for Treatability Studies. Samples were previously collected from these locations for the Treatability Studies. Additionally, approximately 900 gallons of river water will be collected from the Thompson Island sampling station located at river mile (RM) 187.5. The sediment and water will be collected in accordance with the TSWP as subsequently amended by CAMs. The S2 and S3 samples will be collected in aluminum core tubes. The S4B samples will be collected with a lexan core tube and spot checks for fraction of fines will be completed on the sampling vessel. These samples will be characterized by methods described in the TSWP (DQO 1).

The sampling is currently scheduled to begin the week of October 25, 2004 and is expected to be completed in about 2 weeks.

Reviewed and Implemented By: Don Sauda (BBL)

cc: GE Project Managers: Scott Blaha and Bob Gibson
Other Distribution: Amy Toth (Waste Stream)

Table 1 - Treatability Studies Sediment Sample Collection

Treatability Studies Sediment Sampling Locations	Coordinates (New York State Plane East, NAD 83)								Maximum Collection Depth	Quantity of Sediment	
	NW Corner		SW Corner		SE Corner		NE Corner				
	Northing	Easting	Northing	Easting	Northing	Easting	Northing	Easting	(Feet)	(Gallons)	(Cubic Feet)
S2 - River Section 1	1,608,911	732,730	1,608,805	732,724	1,608,805	732,835	1,608,899	732,829	5.3	100	13.4
S3 - River Section 1	1,607,785	732,221	1,607,690	732,221	1,607,690	732,312	1,607,785	732,312	3.8	100	13.4
S4B - River Section 1	1,610,060	733,457	1,610,060	733,457	1,610,060	733,457	1,610,060	733,457	3.0	50	6.7
									Total:	***250***	***33.5***

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

Via Federal Express and Electronic Mail

November 19, 2004

Douglas Garbarini
United States Environmental Protection Agency
Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866

RE: Rapid Small-Scale Column Test Status

Dear Mr. Garbarini:

In a September 20, 2004 letter, General Electric (GE) proposed to filter press additional slurry of sediment sample S4A to generate influent water for the rapid small-scale column tests (RSSCTs). The United States Environmental Protection Agency (EPA) approved this proposal in a letter dated September 30, 2004. The work began on September 20, 2004 and was to be completed on or before November 19, 2004, contingent upon generation of a representative feed water. As was the case in our initial attempt, the polychlorinated biphenyl (PCB) concentration was significantly reduced following settling and filtration operations while preparing the influent water for the RSSCTs. Representative water was not produced by this method.

It is critical that the RSSCT feedstock have the following qualities to meet the RSSCT data quality objectives (DQOs):

- Generated from Hudson River sediment and water, so that the PCB congener distribution and natural organic material will represent the full-scale feedstock for treatment by granular activated carbon (GAC);
- Sufficient PCB mass to produce breakthrough curves in 6-month minimum simulation (23 days); and
- Low suspended solids concentration to prevent column plugging during testing.

Potential PCB loss mechanisms are sorption, volatilization, and biodegradation. PCB losses will be limited by using Teflon materials, reducing storage times, and cooling the feedstock. Also, the dissolved phase PCB concentrations will likely be greater in the supernatant, produced by the method below, compared with the plate and frame press filtrate.

Therefore, GE proposes the following revised corrective action:

1. Mix S4B sediment, with approximately 350 milligrams per kilogram (mg/kg) of PCBs, with Hudson River water at a concentration of 1.76 grams sediment (dry weight basis) per liter of water for a period of one hour.
2. Allow the solids to settle for 30 minutes. Polymers may be used to enhance solids settling rate and performance.
3. Decant the supernatant at a slow rate to a Teflon-lined vessel.
4. Generate small batches required to meet RSSCT demand, to minimize storage time.
5. Conduct confirmatory PCB analysis (using Method GEHR 8082) and total suspended solids (TSS) for each batch of RSSCT feedstock generated.

These tests require significant preparation, including the characterization of recently collected sediment and equipment procurement. Also, the RSSCTs are run continuously and periodic monitoring is required. So, to avoid schedule conflicts with the holidays, GE proposes to begin the test on or about January 4, 2005. While water is targeted to be run through the RSSCT for 23 days, a contingency of 14 days is planned if breakthrough is not indicated after the first 23 days of operation. Therefore, the RSSCT will be completed on or before February 10, 2005, contingent upon generation of a representative feed water. This test schedule will not delay the submittal of the Intermediate Design Report.

In summary, GE believes an additional extension for the RSCCTs is justified because:

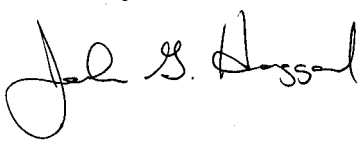
- Conducting the test with waters which were prepared as feedstock do not meet the DQO for the RSCCTs, as established in the Treatability Studies Work Plan (TSWP);
- The treatability studies are not on the project critical path; therefore, extending the date for completion of the RSSCTs will not delay delivery of the Phase I Intermediate Design Report; and
- No other treatability tests are linked to the completion of the RSSCT tests.

Therefore, in accordance with Paragraph 47.c of the Administrative Order on Consent for Remedial Design, GE requests that the date for completion of the treatability studies be revised from November 19, 2004 to February 10, 2005.

Additionally, as discussed in GE's October 8, 2004 letter to EPA providing additional information on the RSSCT, six rounds of RSCCT effluent samples will be analyzed for PCBs by the modified Green Bay Method and EPA Method 608. Also, the RSCCT influent water will be tested for mercury by EPA Method 1631. Laboratory Standard Operating Procedures (SOPs) and the Method Detection Limits (MDLs) for these analyses are included in the attachment to this letter.

If you have any questions, please contact Scott Blaha (518) 862-2738 or Bob Gibson (518) 862-2736.

Sincerely,



John G. Haggard

Attachments

cc: Ben Conetta, U.S. EPA
Bill Ports, N.Y. DEC
Don Sauda, BBL
Mike Elder, GE
Bob Gibson,GE
Scott Blaha, GE

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC FLUORESCENCE, METHOD 1631B, 1631C, 1631E AND MCAWW 245.7

(SUPERSEDES: REVISION 3, REVISION DATE 09/18/02)

Prepared by: _____ 12/18/02
Date

Approved by: _____ 12/19/02
Technical Specialist Date

Approved by: _____ 12/19/02
Quality Assurance Manager Date

Approved by: _____ 12/20/02
Environmental, Health and Safety Coordinator Date

Approved by: _____ 12/19/02
Laboratory Director Date

Proprietary Information Statement:

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

TABLE OF CONTENTS

LIST OF APPENDICES:

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS) using Method 1631B, Method 1631C, 1631E, and MCAWW Method 245.7.

1.2. The associated LIMs method codes are D4 (Method 1631B), E9 (Method 1631C), PR (Method 1631E), and D5 (Method 245.7). The sample preparation code for all methods is D4 (BrCl Oxidation).

1.3. CVAFS analysis provides for the determination of total mercury (organic and inorganic). The oxidant, bromine monochloride has been found to give quantitative recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Methods 1631B, 1631C, and 1631E (hereafter abbreviated to Method 1631 in this SOP) are applicable to the preparation and analysis of mercury in ground water, surface water, effluents and other aqueous samples. All matrices require sample preparation prior to analysis.

1.5. Method 245.7 is applicable to the determination of mercury in drinking, surface and saline waters and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.6. The STL North Canton reporting limit for mercury in aqueous matrices is 0.5 ng/L by Method 1631, and 5 ng/L by Method 245.7.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in aqueous solutions. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor and fluorescence at 253.7 nm. A representative portion of the sample is digested and oxidized in bromine monochloride. The excess free halogens are reduced with hydroxylamine hydrochloride. The mercury (+2) is reduced to its elemental state with stannous chloride and purged from solution with argon in a gas / liquid separator. For Method 1631, the mercury vapor is collected on a gold trap and then thermally desorbed to

the detector. For Method 245.7 the mercury vapor is transported directly from the gas / liquid separator to the detector. The mercury vapor passes through a cell positioned in the light path of an atomic fluorescence spectrophotometer. Fluorescence is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample fluorescence to the calibration curve (fluorescence vs. concentration).

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane and are oxidized by bromine monochloride. (Sample is preserved after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion and oxidation.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Gold, silver and iodide are known interferences. At mercury a concentration of 2.5 ng/L and at increasing iodide concentrations from 30 to 100 mg/L, test data have shown that mercury recovery will be reduced from 100 to 0 percent.

4.2. The use of a brominating digestion coupled with atomic fluorescence detection overcomes many of the chloride, sulfide and molecular absorbance interferences. No interferences have been noted for sulfide concentrations below 24 mg/L.

4.3. Water vapor may collect in the gold traps (Method 1631), and subsequently condense in the fluorescence cell upon desorption, giving a false peak due to scattering of the excitation radiation. Condensation can be avoided by predrying the gold trap and by discarding those traps that tend to absorb large quantities of water.

4.4. The fluorescent intensity is strongly dependent upon the presence of molecular species in the carrier gas that can cause *quenching* of the excited atoms.

4.5. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. The analytical instrument and sample / standards preparation area should be

protected from mercury vapor or particulates in the laboratory air. Samples, standards and blanks should only be opened in a clean area. Gloves must be powder free and should be checked for mercury contamination. Do not use powdered nitrile gloves as they have been shown to have either low level mercury contamination or interferences. Only clean gloves should touch the instrument and other equipment used to process blanks, standards and samples.

4.6. Samples known to contain mercury concentrations greater than 200 ng/L should be diluted prior to bringing them into the clean work area dedicated to processing low level mercury samples.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), 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.

5.3. 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. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

Hydrochloric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

Potassium bromate, bromine monochloride.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as a carbon filter.

5.4. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should 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.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders are located outside the laboratory and the gas led to the instrument through approved lines.

5.8. The CVAFS apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Atomic Fluorescence Spectrophotometer equipped with:

6.1.1. Fluorescence Cell with quartz ends. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.1.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.1.3. Peristaltic pump.

6.1.4. Flowmeter.

6.1.5. Recorder or Printer.

6.1.6. Gas /Liquid separator:

6.1.7. Drying devices: Nafion Dryer (used for all methods), soda lime trap (Method 1631).

6.1.8. Gold traps (2): quartz tube containing gold coated sand.

6.2. Sample bottles, 40 mL borosilicate glass VOC vials, QEC or equivalent, < 0.5 ng/L contamination when used for Method 1631 samples. In actual practice, should contribute less than 0.1 ng/L to facilitate meeting method blank criteria. Unless tested by the manufacturer for cleanliness and accuracy, 12 vials from each lot must be gravimetrically tested at the 40 mL point. Cleanliness is assessed by adding 0.2 mL BrCl (Section 7.15). Store the test vials at room temperature for at least 12 hours and analyze as samples. All vial results must be less than the reporting limit.

6.3. Argon gas supply, high purity, or equivalent. A gold trap may be used in-line to further purify the argon.

6.4. Calibrated automatic pipettes.

6.5. Disposable cups or tubes, low mercury content.

6.6. Starch / iodine paper.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a US Filter PureLab Plus deionized water system or equivalent. Reagent water must be free of mercury and interferences as demonstrated through the analysis of reagent and method blanks.

7.2. Stock (10 mg/L) mercury standards (in 5-10% HNO_3) are purchased. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 µg/L): Fill a 100 mL volumetric flask about half full with reagent water. Add 0.5 mL of BrCl solution (Section 7.15). Add 0.10 mL of the stock mercury standard (Section 7.2) and dilute to 100 mL with reagent water. The intermediate mercury standard should be replaced every 9 months.

7.4. Working mercury standard (1 µg/L): Fill a 40 mL vial about half full with reagent water. Add 0.2 mL of BrCl solution (Section 7.15). Add 4.0 mL of the intermediate mercury standard (Section 7.3) and dilute to 40 mL with reagent water. The working mercury standard should be replaced every 3 months.

7.5. The calibration standards listed in Table I must be prepared fresh daily from the working standard (Section 7.4) by transferring 0, 0.02, 0.04, 0.08, 0.2, 0.4, and 1.0 mL of a mercury standard into 40 mL vials and diluting to volume with reagent water; for Method 1631 use the working standard (Section 7.4), for 245.7 use the intermediate standard (Section 7.3). BrCl (Section 7.15) and $NH_2OH \bullet HCl$ (Section 7.13) reagent solutions are also added.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, some automated mercury systems may not require 40 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard (QCS) must be made from a different manufacturer or lot than that of the calibration standards.

7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed with all reagents that are used for sample preparation.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade and ultra trace mercury grade.

Note: Ultra trace mercury HCl (when commercially available) should be used to prepare the bromine monochloride solution. Trace metal grade HCl may be used to prepare the stannous chloride and 2% HCl rinse solutions provided that these solutions are purged with argon prior to use.

7.9. Autosampler rinse solution (2%): 400 mL trace metal grade HC1 diluted to 20 L reagent water. Purge overnight with argon.

7.10. Stannous chloride solution concentrate: Add 500 g of $SnCl_2 \bullet 2H_2O$ to 2.4 L trace metals concentrated hydrochloric acid. Allow the $SnCl_2 \bullet 2H_2O$ to completely dissolve. ACS Reagent grade suitable for mercury determination (< 1 ppb) recommended.

7.11. Stannous chloride working solution: Fill a 2.5 L glass bottle (HCl leached) with 2.25 L of reagent water. Add sufficient stannous chloride concentrate (Section 7.10) to bring the total

volume to 2.5 L. This produces a reductant solution that is 10% HCl and 2% $SnCl_2 \bullet 2H_2O$. Purge with argon (0.5 L/min) for at least 24 hours. Analyze a reagent blank with this solution prior to analysis of samples (Section 9.8).

7.12. Hydroxylamine hydrochloride solution: Dissolve 300 g of $NH_2OH \bullet HCl$ in reagent water. Dilute to 1 L. Add 1 mL of stannous chloride solution working solution and purge with argon (0.5 L/min) for at least 24 hours. Analyze a reagent blank made with this solution prior to analysis of samples (Section 9.8).

7.13. Potassium bromide: KBr, reagent grade, low mercury content is desirable. This dry reagent may be baked at 250°C for at least 8 hours to volatilize trace Hg(0) contamination.

7.14. Potassium bromate: $KBrO_3$, reagent grade, low mercury content is desirable. This dry reagent may be baked at 250°C for at least 8 hours to volatilize trace Hg(0) contamination.

7.15. Bromine monochloride preservative/oxidizing solution: In a ventilation hood, add 5.4 g KBr to 500 mL of ultra trace (low mercury) HCl. Allow the salt to dissolve. Slowly add 7.6 g $KBrO_3$. Halogen fumes will be emitted during this step. Adequate ventilation is essential to protect analyst safety. Analyze a reagent blank with this solution prior to analysis of samples (Section 9.8)

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for Hg is 48 hours from time of collection to the time of preservation with BrCl solution (Section 7.15). Holding time from time of collection to the time of preservation is extended to 28 days if the oxidation step is performed in the sample bottle. Samples to be analyzed for dissolved Hg must be filtered within 48 hours of collection, then preserved as above. Once preserved, holding time is 90 days from sample collection to analysis.

8.2. Aqueous samples are filled to the top of the container with no headspace, maintained at 0-4°C from time of collection until receipt and preserved with bromine monochloride (BrCl) (Section 8.1). The sample may be stored in either fluoropolymer or borosilicate glass. The sample should not be refrigerated after preservation. Preservation/oxidation is verified by the persistence of the yellow color of the bromine monochloride. Additional BrCl must be added if the preservative/oxidizer is consumed. Record any additional BrCl (Section 7.15) used.

9. QUALITY CONTROL

9.1. Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.2. Initial Demonstration of Capability

9.3. Prior to the analysis of any analyte using Method 1631 or Method 245.7, the following requirements must be met.

9.3.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below both the STL North Canton reporting limit. In addition the MDL for Method 1631 must be ≤ 0.2 ng/L.

9.3.2. Initial Demonstration Study (initial precision and recovery study)- This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.3.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.3.3. Carryover determination – Analyte system blanks immediately after calibration solutions containing successively larger concentrations of Hg – from this test determine the amount of Hg that will carry >0.5 ng/L of Hg into a succeeding system blank. When a sample one half or more of this determined amount is analyzed then a system blank must be analyzed to demonstrate cleanliness at the RL. Samples with detectable Hg analyzed after the high sample but before the system blank must be reanalyzed.

9.4. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate (2 MS/MSD pairs if the batch has more than 10 samples). In some cases, at client request, it may be

appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.5. Sample Count - Laboratory generated QC samples (Method Blanks, LCS, and MS/MSDs) are not included in the sample count for determining the size of a preparation batch.

9.6. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.7. If a sample requires additional BrCl beyond the normal amount (Section 11.1.5) an additional preparation blank should be prepared with the same amount of BrCl. The result of this prep blank will be added to the narrative of the associated sample. This prep blank does not have any specific acceptance criteria, but it should be proportional to the amount of BrCl used.

9.8. System / subtraction / reagent blank: The reagent blank consisting of all reagents used to prepare samples and standards will be used for background subtraction and system cleanliness monitoring. Three reagent blanks are prepared and analyzed with the daily initial calibration curve (ICal) . Apply the average calibration factor from the ICal to the average

raw response from these 3 reagent blanks. The calculated mercury concentration must be less than the reporting limit. The average raw response from these 3 calibration blanks will be subtracted from all raw response data from all other data prior to calculating concentration factor (for cal standards) or concentrations. Subsequent bubbler / reagent blanks are run as ICB and CCB in conjunction with the ICV (QCS) and CCV (OPR). These IC and CC blanks are used to monitor the cleanliness of the instrument and are calculated in the same manner as samples and are not used for background subtraction purposes. The absolute value of the calculated mercury concentration must be less than the reporting limit.

9.9. Laboratory Control Sample (LCS): One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, limits of 75- 125% recovery will be applied.

- In the instance where the LCS recovery is greater than the maximum and the sample results are < RL, the data may be reported with qualifiers**. Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.10. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each 10 samples in preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Method 1631 requires that each matrix be spiked at a 10% frequency. Some regulatory agencies interpret each discharge or sampling point as a separate matrix. It is the

client's responsibility to determine which sample(s) is to be matrix spiked each time samples are submitted for analysis. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 71 - 125 % recovery and 24% RPD for 1631 and 76 – 111% recovery and 18% RPD for 245.7 must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.11. Initial Calibration Verification (ICV/ICB) (QCS – quality control sample): Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 20% of the true value for that solution . An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.3.5) for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the ICV, ICB, CCV, and CCB with the calibration curve.

9.12. Continuing Calibration Verification (CCV/CCB) (on-going precision and recovery - OPR): Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV concentration must be at 5 ng/L for 1631 and 10 ng/L for 245.7. The CCV result must fall within 77-123% of the true value for that solution for 1631 and 76-111% for 245.7. A CCB is analyzed immediately following each CCV. (See Section 11.3.5 for required run sequence). The CCB

(system/reagent blank) must fall within +/- the reporting limit (RL) from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.13. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1 except that the oxidation time need does not need to be a minimum of 12 hours.

10.2. Due to the differences in calibration ranges separate calibration and calibration verification standards must be prepared for Methods 1631 and 245.7. See Section 7.5 and Table 1.

10.3. Calibration may be performed daily (every 24 hours), but is required only when indicated by instrument and preparation QC problems. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer (Table III). Allow the instrument to become thermally stable before beginning calibration (approximately 1-2 hours of warm-up is required if the lamp has been turned off). The most stable results are obtained if the lamp is left on full time. Refer to the CVAFS instrument manual for detailed setup and operation protocols.

10.5. Run 3 deionized water blanks to ensure that the instrument, reductant solution and rinse solutions are adequately clean. The raw blank response should be less than 15,000 for 1631 and 100 for 245.7.

10.6. Calibrate the instrument according to instrument manufacturer's instructions, using 6 standards and 3 calibration blanks. One standard must be at the STL North Canton reporting limit. Analyze standards in ascending order beginning with the blanks. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.7. The calibration factors must have less than 15% RSD or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable RSD. Also, the low standard must calculate back within ±25% of the true value.

10.8. Refer to Sections 9.11 and 9.12 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed with the digestion reagents as well as the field samples.

11.1.2. Open the outer sample bag, carefully dump the inner bag containing the sample bottles onto a clean bench top in the low level mercury area with a minimum of handling. Immediately discard the outer sample bag. Change gloves between each sample or work with another analyst using the clean hands-dirty hands technique.

11.1.3. Change gloves and open the remaining inner bag, remove the sample vials, label and place in the low level mercury prep area.

11.1.4. Mix the sample then transfer ~2.7 mL from each sample vial to a "1X dilution" labeled 10 mL glass culture tube for screening. This will leave 40 mL in the bottle. Confirm by checking meniscus and the 40 mL calibration point. Set the cap back on original sample vial. Repeat this process for all 40mL vial aliquots of the sample. Transfer 2 mL of sample from the 1X tube to the "5X dilution" tube that already contains 8 mL of reagent water. Transfer 0.05 mL of sample from the 1X tube to the "200X dilution" tube that already contains 10 mL of reagent water. Reseal the original sample vial caps if it will be more than 3 minutes before the next step is performed (Section 11.1.5).

Note: Typically 4 sample vials and 2 screening vials will be prepared per sample (6 sample vials for client requested MS/MSD samples).

11.1.5. Temporarily lift the cap and add 0.20 mL of BrCl (Section 7.15) to the 40 mL sample vial, reseal and mix. If the yellow tint from the BrCl disappears add an additional aliquot of BrCl. This iterative process may be repeated until a maximum of 2 mL has been added. Record the amount of BrCl used on the

bench sheet. If the 2 mL maximum was reached and the yellow BrCl color still does not persist consult supervisor to determine if sample dilution prior to preservation / oxidation is appropriate. At least one method preparation blank must be prepared for each different volume of BrCl added.

11.1.6. Add 0.05 mL BrCl to the dilution tube(s) from Section 11.1.4. Confirm the 5X and 200X dilution tubes have adequate BrCl. Add more as needed.

11.1.7. Store the sample vials at room temperature for at least 12 hours. If the yellow BrCl color disappears during the storage period, the oxidizer has been consumed. Add additional BrCl until the yellow color persists. Do not exceed a total of 2 mL. Consult laboratory Technical Director or supervisor if yellow color does not persist after 2 mL addition of BrCl. Record the total volume of BrCl added on the benchsheet. Starch / iodine paper may be used to detect excess halogens (i.e. BrCl) in colored samples where the yellow color of the BrCl can not be seen.

Note: The 12 hour oxidation time is not required for the sample aliquots in the screening tubes.

11.1.8. Prepare method blank and LCS vials using the same reagents as used for the samples.

11.2. Sample screening:

11.2.1. Add 0.05 mL of hydroxylamine solution (Section 7.12) and analyze the 200X screening aliquot of the sample using a single point calibration (10 ng/L) and Method 245.7.

11.2.2. If the sample response exceeds that of the 10 ng/L standard (i.e. sample concentration > 2000 ng/L), then low level analysis by either 245.7 or 1631 is not technically appropriate. Remove all vials associated with this sample from the low level prep and storage areas immediately. Consult supervisor.

11.2.3. If the estimated concentration is greater than 200 ng/L, consult supervisor about analysis by 245.7. If approved, calculate the appropriate dilution and proceed with 245.7 analysis. Alternately, prepare an appropriately large dilution of the sample before bringing it into the low level preparation area. Direct low level analysis by 1631 is not technically appropriate due to the likelihood of contamination.

11.2.4. If the 200X dilution screen was non-detect (i.e. <500 ng/L), add 0.05 mL of hydroxylamine solution (Section 7.12) and analyze the 5X dilution screen using a single point calibration (10 ng/L) and Method 245.7.

11.2.5. If the sample response (Note: this is a 5X dilution) exceeds that of the 5 ng/L standard then the sample concentration is beyond the normal calibration range of Method 1631. Either analyze the sample 245.7 (if allowed by the client) or prepare the appropriate dilution for 1631 analysis.

11.2.6. If the 5X dilution screen response is non-detect at 5 ng/L then the sample may be analyzed without dilution by either 245.7, or Method 1631 depending on the Reporting Limit needed by the client unless matrix interferences warrant dilution.

11.3. Sample Analysis:

11.3.1. When ready to begin analysis, add 0.10 mL of hydroxylamine hydrochloride solution (Section 7.12) to the samples to reduce the excess BrCl (the BrCl has been reduced when no yellow color remains). Cap and shake. Add the hydroxylamine solution in 0.10 mL increments until the BrCl is completely reduced. Record the total volume used on the benchsheet.

Note: Spiking is done before the addition of the hydroxylamine hydrochloride reagent.

11.3.2. With instrument control parameters set to appropriate values (See Table III), load samples into autosampler. Use 40 mL vials for Method 1631 and 14 mL or 40 mL tubes for 245.7.

11.3.3. Start autosampler sequence.

11.3.4. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.3.5. The following analytical sequence must be used:

Instrument Calibration
ICV (QCS)
ICB
CCV (OPR)
CCB
Maximum 10 samples

CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 3 and Table II (Appendix A) for the appropriate quality control criteria.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

Note: Instrument calibration need not be performed if the run QC parameters indicate that the system is in control.

11.4. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data are reviewed periodically throughout the run.

11.5. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.6. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.7. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1.Calibration Factors are calculated according to the equation:

$$CF(x) = \left(\frac{Area(x) - Area(b)}{Conc(x)} \right)$$

Where:

CF(x) = calibration factor of standard (x)
area(x) = area of standard (x)

conc(x) = concentration of standard (x)
area(b) = average area of 3 calibration blanks

12.2. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.3. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.4. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R\ = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.5. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.6. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Appropriate factors must be applied to sample values if dilutions are performed.

12.9. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.3.

13.2. Method performance is determined by the analysis of method blanks and laboratory control samples. The method blanks must meet the criteria in Section 9.6. The laboratory control sample should recover within 25% of the true value until in house limits are established.

13.3. Training Qualification:

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 experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Method 1631, Revision B: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, U.S.EPA, May 1999.

16.2. Method 1631, Revision C: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, U.S.EPA, March 2001.

16.3. Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, U.S. EPA, August 2002.

16.4. Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, U.S.EPA, January 2000.

16.5. QA-003, STL North Canton QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Performance Based Modifications from 1631B and 1631C.

17.1.1.1. An automated continuous flow CVAFS system (Section 6.1) is used rather than the manual purge and trap system described in Sections 6.3, 6.4, 6.5 and 6.6 of Method 1631B

17.1.1.2. The stannous chloride solution has been changed from 20% (Section 7.5 in method) to 2% (Section 7.11) $SnCl_2 \bullet 2H_2O$ to adjust for the volumetric differences between the manual and automated processes.

17.1.1.3. The calibration range covered by the method is from 0.5 to 100 ng/L (Section 10.1.1.2 in the method). Although the automated system is linear over this calibration range the current system has too much run-to-run carryover (> than the RL) when concentrations exceed 25 ng/L. Thus, the calibration range in this SOP is restricted to 0.5 to 25 ng/L.

17.1.1.4. This SOP uses 40 mL sample aliquots (Section 11.1.4 & Table III) for each analysis rather than the 100 mL described in the method (Section 11.1.1 in method). Reagent volumes are scaled accordingly.

17.1.1.5. The holding time from sampling to preservation has been extended to 28 days (Section 8.1) based on holding time studies performed and on file at this laboratory and EPA recommendations incorporated in Revision E of Method 1631.

17.1.2. Other Interpretations and Differences from Method 1631B and 1631C.

17.1.2.1. Conventional fixed concentration matrix spiking has been used in this SOP (Section 9.10) rather than the variable concentration spiking described in the method (Section 9.3 in method). Also, batch acceptability is determined by method blank and LCS criteria and not MS/MSD recovery and RPD.

17.1.2.2. The 3 bubbler blanks used for background subtraction are run with the initial calibration and are labeled calibration blanks. Subsequent bubbler blanks are run as ICB and CCBs to monitor system cleanliness but are not included in the average to determine the bubbler blank response to be subtracted from all other raw response data.

Dale Rushneck (technical consultant to the US EPA, Office of Water) did not object to this implementation of the subtraction and monitoring blanks. See Appendix H question #1.

17.1.2.3. The bubbler blank acceptance criteria is stated as 25 pg (Section 9.4.1 in the Method 1631B). The actual concentration will depend on sample size. The manual method describes the use of 100 mL sample aliquots. This would calculate to 0.25 ng/L. The automated system used by STL was able to meet the method sensitivity and accuracy criteria while using only 40 mL of sample. This would calculate to 0.625 ng/L. STL has chosen an intermediate blank criteria that corresponds to conventional

blank control criteria. The bubbler blank average used for subtraction must be less than 0.5 ng/L (without background subtraction). Subsequent bubbler blanks (ICV & CCV) calculated with background subtraction must have an absolute value less than 0.5 ng/L.

17.1.2.4. The method describes a bubbler blank that consists of reagent water, BrCl, $NH_2OH \bullet HCl$ and $SnCl_2 \bullet 2H_2O$ solutions which have been purged (Section 9.4.1.1 in the manual method). The EPA has recommended that a similar solution be separately prepared and used for the preparation of bubbler blanks, calibration standards and OPR standards. See Appendix H questions 2 & 4. STL disagrees with the EPA regarding the use of this solution for blanks and standards. STL's experience indicates standards in autosampler vials are not stable (when excess $SnCl_2 \bullet 2H_2O$ is present) for the several hours necessary to complete analysis in a typical autosampler sequence. Consequently all blanks and standards are prepared in the same manner as the samples and the $SnCl_2 \bullet 2H_2O$ is added automatically by the system immediately before analysis.

17.1.3. Additional interpretations and differences from Method 1631E.

17.1.3.1. Section 9.1.7 of the method requires (3) method blanks per analytical batch. The section also describes an analytical sequence that includes a CCV (OPR) only at the beginning and end of the sequence, and that includes no CCBs (system blanks) after calibration. This SOP requires only one method blank per preparation batch, but requires additional stability and cleanliness checks through the analysis of a CCV/CCB pair at the beginning, end and after every (10) analyses during an analytical run.

17.1.3.2. Section 9.2.1 of the method recommends that an MDL be determined whenever a new operator begins work. At this laboratory, a new operator receives proper, documented training and must prove competence through an initial demonstration of performance that includes the successful analysis of (4) LCSs (See Section 9.3.2).

17.1.3.3. Conventional MS/MSD techniques and criteria have been maintained in contrast to Section 9.3.4 of the method (See Section 17.1.2.1 of this SOP).

17.1.3.4. Section 9.4.3.1 of the method requires reagent blank concentrations to be <0.2 ng/L. In this laboratory, reagent blanks are analyzed as system calibration blanks and are held to the system blank criteria of <0.5 ng/L (See Section 9.8 of this SOP).

17.1.3.5. Section 9.4.5.1 of the method recommends that field blank analysis immediately before analyzing samples from the batch. Field blanks are analyzed as normal samples in this laboratory with no particular run order requirement.

17.1.3.6. Section 9.4.7 of this method recommends that 5% of the bottles in a lot be monitored. Bottle cleanliness in this laboratory is verified by the initial analysis of 5% of the bottles from three boxes of a lot of 40 mL sample vials, and then monitored through the routine analyses of system blanks (calibration blanks).

17.1.3.7. The volume descriptions for the equation in Section 12.3.2 of the method includes subtraction of the volume of reagent used in the standards and the samples. Since the volume of reagents used in samples and standards is typically the same (or differs insignificantly in rare cases), this subtraction is not included in the determination of Hg concentration in this laboratory.

17.1.4. Performance Based Modifications from Method 245.7.

17.1.4.1. The preservative / oxidizer solution (Section 7.15) from Method 1631B has been used in place of the bromate/bromide oxidizer solution (Section 7.7.4 in method).

17.1.4.2. The autosampler is rinsed with 2% HCI solution as recommended by the manufacturer rather than deionized water (Section 11.3.2 in method).

17.1.5. Other Interpretations and Differences from Method 245.7.

17.1.5.1. Reagent blank acceptance criteria is an absolute value less than the reporting limit (Section 9.8) rather than MDL (Section 9.2.1.3 in method)

17.1.5.2. Conventional fixed concentration matrix spiking has been used in this SOP (Section 9.10) rather than the variable concentration spiking described in the method (Section 9.5 in method). Also, batch acceptability is

determined by method blank and LCS criteria and not MS/MSD recovery and RPD.

17.1.5.3. All standards are prepared using the same reagents as the samples rather than only in reagent water (Section 10.1.1.2 in method). (See Section 10.1)

17.1.5.4. The digested sample is used for dilution since no undigested sample (Section 11.3.4 in method) is available as the BrCl solution both preserves and oxidizes the sample. Also, this form of the sample should be more homogeneous for total mercury analysis.

17.2. Modifications from previous SOP

17.2.1. See change form.

17.3. Facility Specific SOPs

17.3.1. Not applicable.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAFS raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Aqueous Sample Preparation - Mercury

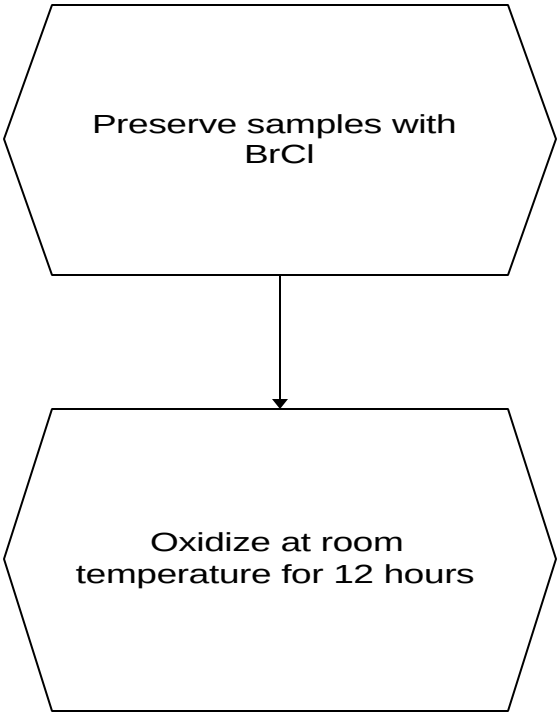
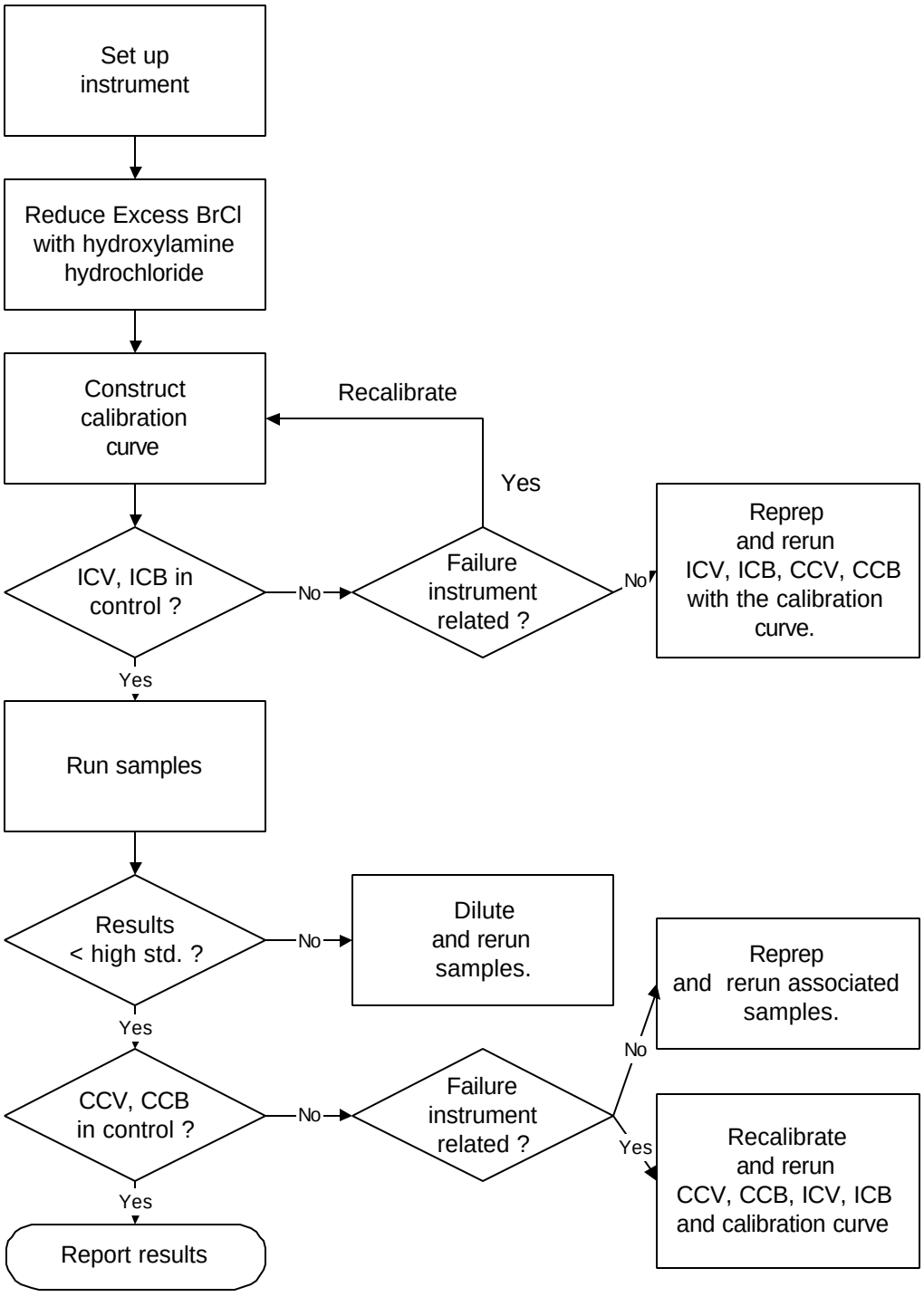


Figure 2. CVAF Mercury Analysis



APPENDIX A

TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD, QC STANDARD AND SPIKING LEVELS (ng/L)

	1631		245.7	
	Conc ng/L	µL Std (Sec.7.4)		µL Std (Sec.7.3)
Standard Water RL	0.5		5	
Standard Solid RL	NA		NA	
Std 1 (in triplicate)	0	0	0	0
Std 2	0.5	20	5	20
Std 3	1	40	10	40
Std 4	2	80	20	80
Std 5	5	200	50	200
Std 6	10	400	100	400
Std 7	25	1000	250	1000
ICV (QCS)	5	200 (Sec 7.6)	10	40 (Sec 7.6)
LCS/CCV (OPR)	5	200	10	40
MS	5	200	10	40

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA 1631	ACCEPTANCE CRITERIA 245.7	CORRECTIVE ACTION
ICV (QCS)	Beginning of every analytical sequence.	80-120 % recovery	80-120 % recovery	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.11).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL (0.5 ng/L)	The result must be within +/- RL (5 ng/L)	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.11).
CCV (OPR)	Every 10 samples and at the end of the run.	77-123 % recovery	76-111 % recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. (See Section 9.12).
CCB	Immediately following each CCV.	The result must be within +/- RL (0.5 ng/L)	The result must be within +/- RL (5 ng/L)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. (See Section 9.12).

Method Blank	One per sample preparation batch of up to 20 samples. Note: additional prep blank(s) required if additional BrCl needed in some sample(s)	The result must be within +/- RL (0.5 ng/L) Sample results greater than 20x the blank concentration are acceptable.	The result must be within +/- RL (5 ng/L)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.6 for additional requirements.

*See Sections 11.3.5 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA 1631	ACCEPTANCE CRITERIA 245.7	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	75-125 % recovery	75-125 % recovery	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.9).
Matrix Spike	Two per sample preparation batch of up to 20 samples.	71-125 % recovery. If the MS/MSD is out for an analyte, it must be in control in the LCS.	76-111 % recovery. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.10)
Matrix Spike Duplicate	See Matrix Spike	71 - 125 % recovery; RPD ≤ 24%. (See MS)	76-111 %; RPD ≤ 18%. (See MS)	See Corrective Action for Matrix Spike.

TABLE III. Summary of Instrument Parameters (Leeman Labs Hydra AF Gold +)

Instrument Parameter	1631	245.7
Argon flow (L/min)	0.5	0.4
Pump flow (mL/min)	10	10
Rinse (sec)	60	120
Uptake (sec)	240	35
Sample volume (mL)	40	11
Integration (sec)	0.70 (70 sec total)	35 sec total
Method	CVAFS with trap	CVAFS
Furnace 1 temp (°C)	450	
Furnace 2 temp (°C)	450	
Dry Time (sec)	5	
Desorption Time (sec)	70	
Stabilize Time (sec)	10	

APPENDIX B
EXAMPLE
STL NORTH CANTON Hg DATA REVIEW CHECKLIST

Example

STL North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ Analyst:_____ Instrument:_____
Prep Batches Run:_____
Circle Methods used: 1631B, 1631C, and 1631E : NC-MT-0001 Rev 4 **245.7 :** NC-MT-0001 Rev 4
245.7 screen :NC-MT-0001 Rev 4

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels (including 3 initial calibration blanks)?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. Samples preserved within holding time at lab?				
2. LCS done per prep batch and within QC limits?				
3. Method blank done per prep batch and < RL?				
4. MS run at required frequency (1 per 10 samples) and within limits?				
5. MSD or DU run at required frequency (1 per 10 samples) and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately?				
2. Current MDL data on file?				
3. Calculations and Transcriptions checked for error?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct?				

Analyst: _____ Date:_____
Comments:
_____
_____
_____

2nd Level Reviewer : _____ Date:_____
_____
_____
_____

Standard and Reagent Numbers

ICal/CCV _____ **ICV _____**

BrCl _____ NH2OH HCl _____ SnCl2 _____

APPENDIX C

MSA GUIDANCE

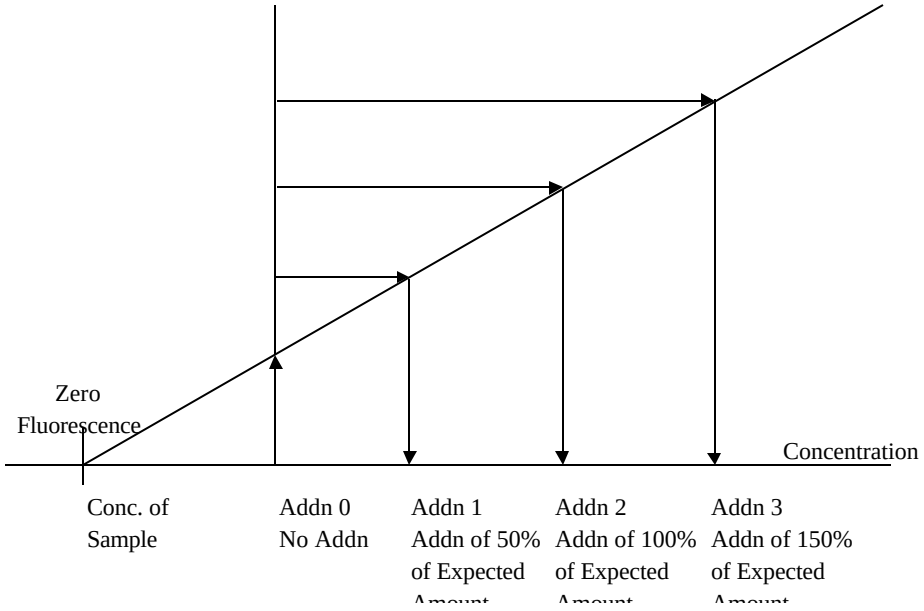
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the fluorescence (or response) of each solution is determined and a linear regression performed. On the vertical axis the fluorescence (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero fluorescence , the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Fluorescence or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on “Continuous”
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won’t Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal fluorescence or concentration	Incorrect standard used Incorrect dilution performed Dirty cell

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 hydrochloric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered Gloves must not be used in the mercury laboratory since the powder contains mercury, as well as other metallic analytes. Only powder free gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and discard.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman Labs Hydra AF gold plus)[(1)]

Daily	Semi-annually	Annually
	Check Hg lamp intensity.	Change Hg lamp.
		Check liquid/gas separator.
Check argon flow.		
Check pump tubing.		
Check drain.		
Check soda lime drying tube.		

APPENDIX G

INSTRUMENT SET UP

Hg Analysis (Leeman Labs Hydra AF gold plus)

TO SET UP INSTRUMENT FOR ANALYSIS

1. WinHG Rack File editor

2. New Rack file

    A. Enter sample workorder # into corresponding "Sample name" (limit 8 chars, no spaces)

    B. Enter client ID into "Extended ID"

    C. Save file with Date/letter name (e.g. 0324a) (limit 8 characters, no spaces)

3. WinHg Database

    A. Select most recent calibration of appropriate method (1631 or 245.7)

    B. Save Protocol As, method / current date (e.g. 16310324) (limit 8 characters, no spaces)

    C. Clear calibration data from new protocol

    D. Apply (i.e. Save changes)

    E. Upload protocol to Runner

3. WinHg Runner

    A. Sample tab

    B. Select appropriate rack file(s), click auto sample

APPENDIX H. Correspondence with US EPA Office of Water

From: Dale Rushneck [SMTP:dale.rushneck@home.com]
Sent: Thursday, March 29, 2001 3:07 PM
To: Bruce, Mark
Cc: Joan Cuddeback; Maria Gomez-Taylor
Subject: Fw: 1631B blank questions from STL

Hi Mark

Maria Gomez-Taylor has asked that I respond to your E-mail. If you'll remember from our previous exchange of E-mails in 1999, I'm a contractor to EPA assisting with technical issues.

My responses are embedded in your E-mail and begin with an **asterisk**. If you have further questions, please contact me as convenient.

Dale

DALE.RUSHNECK@HOME.COM
3104 Worthington Av
Fort Collins CO 80526-2752
Phone: 970-223-2013
Fax: 970-223-2008

> ----- Forwarded by Maria Gomez-Taylor/DC/USEPA/US on 03/28/01 09:37 AM
> -----
>
> mbruce@stl-inc.com
> To: Maria Gomez-Taylor/DC/USEPA/US@EPA
> 03/22/01 cc:
> 11:35 AM Subject: 1631B blank questions from STL
>
> To: Maria Gomez-Taylor
>
> STL is implementing Method 1631 Revision B. We have questions regarding a
> few areas in the method.
>
> 1) Bubbler blank subtraction : The method requires that at least 3 bubbler

> blank responses be averaged together and subtracted from all raw data
> produced during the initial calibration and analytical batch before final
> results are calculated (Sections 10.1.1.3 and 12.2). Is it acceptable to
> run the 3 bubbler blanks used for "average bubbler blank subtraction" at
> the beginning with the initial calibration and monitor subsequent bubbler
> blanks to ensure that system contamination is less than or equal to 0.25
> ng/L?
*The intent of the bubbler blanks is to monitor the background level in the instrument over the batch. So, although there is no prohibition against running the 3 bubbler blanks at the beginning of the batch, it is the laboratory's responsibility to make sure that the system doesn't become contaminated during the batch, thereby compromising the measurement of Hg in a sample. Therefore, we would suggest that the bubbler blanks be run throughout the batch.

> 2) Bubbler blank on automated system : The method describes a bubbler blank
> in a manual system. It would seem that in an automated system the closest
> analog is analyzing reagent water from an autosampler vial. Do you agree?
*We're having ongoing discussions of this subject. The current thinking is that the best way to simulate a bubbler blank is to add the reagents (BrCl, NH2OH, and SnCl2) sequentially to 100 mL of reagent water in a purge vessel, purge the water at the flow rate and for the time specified for the blank and samples, then use the purged water as the bubbler blank. Also, use this water for calibration.
> 3) Bubbler blank acceptance criteria : The method states that the average
> bubbler blank must be less than 25 pg. In a process using 100 mL sample
> volumes this translates to an effective concentration of 0.25 ng/L. In a
> process using only 10 mL sample volumes this translates to an effective
> concentration of 2.5 ng/L. We suspect that the EPA didn't really intend
> to> allow bubbler (and reagent) blanks to have Hg concentrations this high.
> Is> it correct to assume that the bubbler (and reagent) blank acceptance
> criteria should be 0.25 ng/L regardless of the sample volume used?
*Yes. However, all tests (MDL, IPR, OPR, blank, MS, MSD, etc.) must be run at the smaller volume and all QC acceptance criteria must be met.

> 4) Subtraction blank : Is it acceptable to use reagent blanks in place of
> reagentless bubbler blanks if the reagent blank is held to the same
> acceptance criteria as the reagentless bubbler blank? This reduces the
> complexity of the method by using only one type of analytical blank and

> increases the frequency of reagent blank runs thus exceeding the current
> method requirement for reagent blank frequency.
*No. Because the bubbler blank contains little or no Hg (it's been oxidized, reduced, and purged), calibration is established without an Hg background. If reagent blanks were used, an Hg background would be "calibrated out." If the background came from the reagent water, and a field blank or field sample contained less Hg, the result would be negative. It would be acceptable to use a method blank in place of the reagent blank because all reagents would go into the method blank.

> Thank you for your assistance in answering these questions.
>
>
> Mark L. Bruce Ph. D.
> Technical Director
> STL North Canton
> 4101 Shuffel Dr. N.W.
> North Canton, Ohio 44720
> Office : (330) 966-7267 Fax : (330) 497-0772 E-Mail :
> mbruce@stl-inc.com
> www.stl-inc.com
>

STL Reference Data Summary

Structured Analysis Code: I-**-PR-01-01

Target Analyte List: All Analytes

Matrix: WATER
Extraction: None specified.
Method: Mercury, 1631E
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 938							Spike List 939							
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1701	Mercury	0.5	ng/L	0.1	ng/L	20040112	C	Y	5	ng/L	77	125	18	C	Y	5	ng/L	71	125	24

STANDARD OPERATING PROCEDURE
NORTHEAST ANALYTICAL, INC.

NE016_04.SOP
REVISION NUMBER: 04

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF POLYCHLORINATED BIPHENYLS BY EPA METHOD 608

FEBRUARY 10, 2003

COPY #

STANDARD OPERATING PROCEDURE

Author: Anthony J. Maiello
Reviewed by:

William A. Kotas
QA Officer

Approved by:

Robert E. Wagner
Laboratory Director

Northeast Analytical, Inc.
Issuing Section: GC Organics Analysis
SOP Name:NE016_04.SOP
Date:10-February-2003
Revision:04

1.0 Title

EPA Method 608 -PCB Aroclor Analysis by Capillary Column GC

Standard operating procedure for the analysis of Polychlorinated Biphenyls by Gas Chromatography with Electron Capture Detection and Total Aroclor Quantification.

2.0 Purpose

The purpose of this SOP is to provide a detailed written document for measurement of Polychlorinated Biphenyls (PCBs) according to EPA Method 608 specifications.

3.0 Scope

3.1 This SOP is applicable in the determination and quantification of PCBs as outlined in EPA Method 608. It is applicable to the following matrices: water, wastewater, and municipal and industrial discharges.

3.2 The following compounds can be determined by this method:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor-1221	11104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

3.3 Samples are extracted with a pesticide grade solvent by Separatory Funnel or Continuous Liquid Liquid Extraction. The extracts are further processed by concentration and a series of clean-up techniques. The sample extract is then analyzed by injecting onto a gas chromatographic system and detected by an electron capture detector.

3.4 This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs by gas chromatography. Sample extraction and cleanup procedures are described separately in

additional laboratory Standard Operating Procedures.

4.0 Comments

4.1 One of the major sources of interference in the analysis of PCBs is from Organochlorine Pesticides that are co-extracted from the samples. Several of the commonly found pesticides and associated degradation products (DDT, DDE, DDD) overlap the PCB profile envelope and co-elute with several PCB GC peaks and therefore cannot be accurately measured. The analyst must be careful in chromatographic pattern review and flag peaks that are suspected of being contaminated so that they are not included in the total PCB values generated.

4.2 Laboratory contamination can occur by introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters respond on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification.

5.0 Safety

5.1 Safety glasses and disposable gloves must be worn when handling samples and extracts.

5.2 All manipulations of sample extracts should be conducted inside a chemical fume hood. The analyst should minimize manipulation of sample extracts outside of a fume hood.

5.3 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions of handling applicable solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

5.4 Samples remaining after analysis should be either returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance in this matter and also standard operating procedure NEO54, disposal of laboratory waste.

6.0 Requirements

6.1 Extensive knowledge of this standard operating procedure and EPA Method 608 is required.

6.2 The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

7.0 Equipment

7.1 Instrumentation

7.1.1 Gas chromatograph: Varian Model 3800, equipped with Model 1079 injector, temperature programmable oven, electron capture detector, Model 8200 autosampler (or equivalent).

7.1.2 Chromatograph Data System: A data system for measuring peak height and peak area. A Millennium_32 computer network based workstation (Waters Associates), will be employed to capture detector response and digitally store the chromatographic information. This system will allow for chromatographic review of data from the gas chromatograph, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

7.2 Glassware and Accessories

7.2.1 25 mL volumetric flasks, Class A, acid washed, (Baxter Cat. No. F4635-25) or equivalent.

7.2.2 5 mL volumetric flasks, Class A, acid washed (Baxter Cat. No. F4635-5) or equivalent.

7.2.3 10 mL volumetric flasks, Class A, acid washed (Baxter Cat. No. F 4635-10) or equivalent.

7.2.4 50 mL volumetric flasks, Class A, acid washed (Baxter Cat. No. F4635-50) or equivalent.

7.2.5 100 mL volumetric flasks, Class A, acid washed (Baxter Cat. No. F4635-100) or equivalent.

7.2.6 4 dram vials for sample extract storage
(Kimble Opticlear, part no. 60910, code no. 60910-4) or equivalent.

7.2.7 8 dram vials for sample extract storage (Kimble Opticlear, part no. 60910, code no. 60910-8) or equivalent.

7.2.8 Pasteur pipettes (Kimble, part no. 72050) or equivalent.

7.2.9 250 mL beakers, glass (Baxter Cat. No. B2650-250) or equivalent.

7.2.10 100 mL beakers, glass (Baxter Cat. No. B2650-100) or equivalent.

7.2.11 Disposable 10 mL pipettes (Baxter P4650-110) or equivalent.

7.2.12 Disposable 5 mL pipettes (Baxter P4650-15) or equivalent.

7.2.13 Disposable 1.0 mL pipette (Baxter P4650-11X) or equivalent.

7.3 Chemicals

7.3.1 Hexane, Burdick and Jackson, (Cat.No. 216-4) or equivalent.

7.3.2 Acetone, Burdick and Jackson, (Cat.No.010-4) or equivalent.

7.3.3 Toluene, Baker, (Cat.No. 9336-03) or equivalent.

7.3.4 Methylene Chloride, Burdick and Jackson, (Cat. No. 300-4) or equivalent.

7.4 Analytical Standard Solutions

7.4.1 Aroclor Stock Standard Solutions

7.4.1.1 Polychlorinated Biphenyls - Neat commercial material for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as electric power transformers and capacitors.

7.4.1.2 Stock standards are prepared from individual Aroclor formulations by weighing an exact amount of the neat material to the nearest 0.1 mg, and dissolving and diluting to volume in a 100 mL volumetric flask with hexane. See Attachment A, Table 1 for exact weights of each compound. For DCBP, dissolve neat

formulation in 10 mL of toluene and then transfer to a 100 mL volumetric flask bringing to volume with hexane.

7.4.1.3 The stock standards are transferred into Boston bottles and stored in a refrigerator at 0-6?C, protected from light.

7.4.1.4 Stock PCB standards must be replaced after one year, or sooner if comparison with certified check standards indicate a problem. See 8.5.3 for limits.

7.4.1.5 For quality control and general labeling requirements refer to standard operating procedure NE050, Preparation of Standards.

7.4.2 Calibration Standards

7.4.2.1 Calibration standards are prepared at five concentration levels using a prepared working standard. A high and low level calibration set is prepared for each Aroclor. See Attachment A, Table 2 for the preparation and exact concentrations of the working standards.

7.4.2.2 The following nominal* concentrations make up the five point initial calibration curve high and low level standard set:

	High Level	Low Level
Std 1.	20.0 ng/mL (0.020 PPM)	5.0 ng/mL (0.005 PPM)
Std 2.	100 ng/ml (0.100 PPM)	10 ng/mL (0.010 PPM)
Std 3.	250 ng/mL (0.250 PPM)	20 ng/mL (0.020 PPM)
Std 4.	500 ng/mL (0.500 PPM)	50 ng/mL (0.050 PPM)
Std 5.	1000 ng/mL (1.00 PPM)	100 ng/mL (0.100 PPM)

*Note: Calibration standards are prepared from neat Aroclors which are weighed to the nearest 0.1 mg. The actual concentration of each calibration standard is provided in the attached standard preparation tables (Attachment A).

7.4.2.3 The two surrogate compounds TCMX and DCBP are included in the A1254 calibration standards. The stock standard for TCMX is prepared by diluting 1 mL of TCMX solution (ULTRA, cat. #IST-440, at 2000 ug/mL or equivalent) into a 100 mL volumetric flask resulting in a solution of TCMX at 20 PPM. The stock standard for DCBP is prepared by diluting 10.0 mg of neat Decachlorobiphenyl (Chem Service I -2170 or equivalent) into a 100 mL volumetric flask resulting in a solution of DCBP at 100 PPM.

7.4.2.4 To prepare the working surrogate standard, pipet 5.0 mL of the 100ppm DCBP stock standard and 2.5 mL of the 20ppm TCMX stock standard into a 100 mL volumetric flask and bring to volume with hexane. The final concentration of this solution will be 5.0ppm of DCBP and 0.5ppm of TCMX. Refer to Attachment A, Tables 4 and 4A for instructions on preparation of the high and low level calibration standards containing A1254 and the surrogates. Refer to Attachment A, Tables 3 and 3A for instructions on preparing the remaining calibration standards.

7.4.2.5 Transfer all calibration standards to 8 dram vials and store in a refrigerator at 0-6?C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem. See 8.5.3 for limits.

7.4.3 Continuing Calibration Check Standards

7.4.3.1 Continuing calibration check standards for high and low level calibration curves are prepared independently from calibration standards, by using an alternate source purchased from reference standard vendors. Refer to Attachment B, Tables 1-3 for instructions on preparation of these standards.

8.0 Procedure

8.1 Gas Chromatographic Operating Conditions

8.1.1 Establish the gas chromatograph (GC) operating parameters as follows:

Autosampler parameters: Multi-vial mode, ECD Attenuation and range are 1.

Refer to Attachment C for all other GC operating procedures, alternate parameters may be established for other GC instrumentation to meet method requirements.

Note: GC helium gas flow is optimized after instrument maintenance by adjusting the helium flow to elute a PCB calibration standard to a known retention time.

8.2 Data Acquisition

8.2.1 Chromatographic information will be collected and processed utilizing a computer based data acquisition workstation (Waters Associates, Millennium_32 computer network based workstation) The GC workstation acquires the millivolt detector signal, performs an analog to digital conversion and stores the digital chromatogram on the computer network's disk. The chromatography software performs all data reduction including, long term data storage on magnetic media, chromatographic peak integration, all calculations, report generation, chromatogram plots, and calibration functions.

8.3 Initial GC Calibration

8.3.1 GC calibration will be performed by the external calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

8.3.2 Establish the gas chromatographic operating parameters outlined in Section 8.1 and prepare the calibration standards at the five concentrations outlined in Section 7.4.2.

8.3.3 Inject each calibration standard using the GC autosampler and the parameters outlined in section 8.1, which are those used for actual samples.

8.3.4 For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and group number for calibration purpose.

Aroclor	Group Number
A1221	1
A1232	2
A1016	3
A1242	4
A1248	5
A1254	6
A1260	7

8.3.5 Attachment D is an example of chromatograms of reference standards of each Aroclor for a DB1 column with peaks selected for calibration labeled.

8.3.6 For the initial calibration curve to be considered valid, the percent relative standard deviation must be less than 10% over the working range. The calibration curve is used for quantification in every case and is not replaced with the average calibration factor. See attachment E for an example of response factors and the calculation of the percent relative standard deviation.

8.4 Retention Time Windows

8.4.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Make three injections of each Aroclor at a midlevel concentration throughout a minimum 72-hour time period.

8.4.2 For the 5 peaks selected for calibration of each Aroclor, calculate the standard deviation resulting from the variation in the three retention times for that peak.

8.4.3 The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

8.4.4 If the standard deviation of the selected peak is zero, the standard deviation of the peak eluting after it is used. If it is the last eluting peak that has zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

8.4.5 Retention time windows established in section 8.4.3 to 8.4.4 may not be practical when samples containing matrix interferences are injected onto the GC column. The default R.T. Window of +/- 0.07 minutes is employed when the established windows are too narrow. Besides using retention time windows to assign peaks for quantification, the analyst should rely on their experience in pattern recognition of multi-response chromatographic response exhibited by PCB Aroclors.

8.4.6 Attachment F provides examples of calculated retention time windows generated by the above outlined procedures.

8.5 Gas Chromatographic Analysis

8.5.1 Prior to conducting any analyses on samples, calibrate the system as specified in Section 8.3

8.5.2 To start an analytical sequence, the 500 ppb (or 50 ppb) calibration standard is injected and analyzed for the seven Aroclors that the system is calibrated for, if more than 24-hours has elapsed since the last valid continuing calibration check standard. If less than 24-hours has elapsed since the last valid continuing calibration check

standard, select one 500 ppb (or 50 ppb) continuing calibration check standard. Selection of the continuing calibration check standard should be based on anticipated Aroclor contamination that the samples may exhibit. Selection of the continuing calibration check standard should also be alternated among the seven Aroclors.

8.5.3 The calculated value for the continuing calibration check standard must be ±20% for it to be valid and analysis to proceed. If this criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples which are not bracketed by valid check standards must be re-analyzed when the system is in-control.

8.5.4 The daily retention time windows must be established. Use the retention time for the selected 5 peaks of the continuing calibration check standard as the midpoint of the window for that day. If all seven Aroclors were analyzed as the initial continuing calibration check standard, then establish retention time windows using the retention time plus or minus the windows established in Section 8.4. If only one Aroclor was analyzed as the continuing calibration check standard (i.e., less than 24-hours had elapsed), use the retention time from this standard as the midpoint plus or minus the windows established in Section 8.4. to establish the daily retention time windows. For the remaining six Aroclors, go back to the previous time all seven Aroclors were analyzed as the initial calibration check standards and use those retention times plus or minus the windows established in Section 8.4 to develop daily retention time windows.

8.5.6 All succeeding continuing calibration check standards analyzed during an analysis sequence must also have a percent difference of ±15% or less when compared to the initial calibration generated from the 5 point calibration curve.

8.5.7 All succeeding standards in an analysis sequence should exhibit retention times that fall within the daily retention time window established by the first continuing calibration check standard(s) of that analytical sequence. If the retention times are outside the established windows instrument maintenance must be performed and recalibration may be required.

8.5.8 The following is the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run every tenth injection in the analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples analyzed.

ANALYTICAL SEQUENCE

INJECTION	MATERIAL INJECTED
1	Hexane Blank
2-36	Initial Calibration Standards
37-43	Continuing Calibration Check Standard
44-52	Samples analyses, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of 9 samples between continuing calibration check standards.

53	Continuing calibration check standard
54	and higher repeat inject. 44-53 sequence

8.6 Quality Control

8.6.1 This section outlines the necessary quality control samples that need to be instituted at the time of sample extraction. The data from these quality control samples is maintained to document the overall precision and accuracy of the data generated.

8.6.2 Each analyst must demonstrate competence in accuracy and precision on quality control samples before beginning unsupervised analysis of unknown samples (Initial Demonstration of Performance). This demonstration must be on-going and be repeated if there is any modification to the method.

8.6.3 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples an organic-free reagent water blank is processed

8.6.4 The method blank should exhibit PCB levels less than the practical quantification limit (PQL). If the method blank exhibits PCB contamination above the reportable PQL, the samples associated with the contaminated blank should be re-extracted and analysis repeated when appropriate. If there is no original sample available for re-extraction or if the associated sample concentrations greatly exceed the blank concentration, then all positive concentration results for the associated samples should be flagged with a "B" indicating blank contamination and a case narrative describing the situation prepared.

8.6.5 A matrix spike is to be analyzed at a rate of 1 matrix spike per every 10 samples. A duplicate sample may be prepared in lieu of a matrix spike when detectable PCB concentrations are known to be present.

8.6.6 Analyze one unspiked and one spiked sample. Calculate the percent recovery based on Aroclor concentration of both samples as follows:

A = concentration of spiked sample
B = concentration of unspiked sample (background)
T = known true value of the spike
Percent Recovery (p) = 100 (A-B) %/T

Compare the percent recovery calculated with project specified limits, the lab established limits, or the default lab limits of 70-130% where appropriate. If the concentrations of the matrix spikes are *greater* than five times the calculated sample amount then the quality control limits may be applied. If the concentrations of the matrix spikes are *less* than five times the sample than there are no established limits applicable. If the percent recovery falls outside the acceptance range for the given Aroclor used as the spiking analyte, then the matrix spike recovery failed the acceptance criteria. Inform quality control manager and document matrix spike recoveries.

A relative percent difference (RPD) is calculated for Duplicates. This is calculated as follows:

A = Concentration of original sample
B = Concentration of duplicate sample

RPD = [(A-B)/{(A+B)/2}] x 100
where (A-B) *is taken as an absolute value*

If the concentrations of the sample/duplicate set are *greater* Than five times the calculated PQL then an RPD of twenty percent or less is acceptable. If the concentrations of the matrix spike set are *less* than five times the PQL there are no established limits applicable to the RPD.

8.6.7 A QC reference check standard (laboratory control spike sample) is also prepared and analyzed. Spike one liter of laboratory organic free water, extract, and analyze. Calculate the percent recovery for the Aroclor spike and compare to the lab-established limits or the default limits of 70-130%. If the percent recovery for the QC reference check standard (laboratory control spike sample) is out of criteria, the analysis is out of the control for that analyte and the problem should be immediately corrected. The entire batch of samples will need to be re-extracted and re-run. If no samples are available for re-extraction the data is delivered to the client with a case narrative.

8.6.8 Method accuracy, based on matrix spike and laboratory control spike data , is maintained by the laboratory as part of the QC program. For each sample matrix, upper and lower warning and control limits for method performance are established. Upper and lower warning limits (p ± 2SD) and upper and lower control limits (p ± 3SD) are established. Laboratory established limits are compared to default limits and are updated with new data periodically.

8.6.9 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicates, duplicate, method blank, and QC reference check standard (laboratory control spike sample) at time of extraction. Surrogate compounds chosen for this method are Tetra-Chloro-meta-Xylene (TCMX) and Decachlorobiphenyl (DCB). The following are typical surrogate amounts added to normal encountered matrices. These amounts may be adjusted by the analyst, if PCB background levels are high and surrogates are being diluted out of analysis range. The surrogate spike amount added for water samples is normally: 1.0 mL of 0.05ppm TCMX/0.5ppm DCB

8.6.10 Surrogate percent recovery data for each matrix is tabulated as part of the on-going laboratory QC program. The standard deviation is calculated once enough surrogate data is available for each matrix, typically based on 25 to 30 samples. Upper and lower warning limits (p ± 2SD) and upper and lower control limits (p ± 3SD) are established. Laboratory limits are compared to default limits and are updated with new data periodically.

8.6.11 Only one surrogate analyte needs to meet established control limits for the analysis to be valid. The recovery data is compared to the project specified limits, lab-established limits or the default limits of 60-140% as appropriate. If percent surrogate recovery is not within limits for either surrogate, the following steps are required.

8.6.11.1 Review calculations that were used to generate surrogate percent recovery values to make certain there are no errors.

8.6.11.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

8.6.11.3 When appropriate, re-analyze the extracts that did not meet control limits, either at the previously analyzed dilution or at a more dilute level to assess if the sample matrix interfered with surrogate

measurement.

8.6.11.4 If the above steps do not give satisfactory results, re-extract and re-analyze the sample when appropriate. Report this data if surrogate recovery is within limits. If surrogate percent recovery is out of limits for the re-extracted samples, low or high surrogate recovery may be due to matrix affects and the data can be reported as estimated and the data user is notified in the form of a case narrative.

9.0 References

9.1 EPA Method 608 -U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", July 1988.

9.2 Standard Methods for the Examination of Water and Waste Water, 18th Edition 1992, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

9.3 New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1988, updated 1998.

9.4 "Guide to Environmental Analytical Methods", fourth edition, Genium Publishing Corporation, 1998.

10.0 Attachments

10.1 Attachment A: PCB Standards Preparation Tables

10.2 Attachment B: PCB Continuing Calibration Tables

10.3 Attachment C: GC Operating Conditions

10.4 Attachment D: Chromatograms of PCB standards.

10.5 Attachment E: Example Calculations.

10.6 Attachment F: Retention Time Windows

11.0 Glossary

Accuracy: Accuracy means the nearness of a result or the mean (±) of a set of results to the true value. Accuracy is assessed by analysis of reference samples and percent recoveries.

Analytical Batch: The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples which are analyzed together whereas the sample method sequence, the reagent lots, and manipulations are common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g. ground water, sludge, ash, etc.).

Aroclor: Polychlorinated biphenyls (PCBs) were commercially produced for a variety of uses including, transformers, capacitors, inks, paints, dedusting agents, and pesticides to list several. Monsanto Corporation was a major producer and sold PCBs under the trade name Aroclor.

Blank: A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples so sodium sulfate is used as a blank matrix. The blank is taken through the appropriate steps of the process. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field in sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

Calibration Check Standard: Standard used to determine the state of calibration of an instrument between periodic recalibration. A calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

CAS Number: An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210:[614] 447-3600.)

Laboratory Control Spike: A blank which has been spiked with the analyte(s) from an independent source in order to monitor the execution of the analytical method is called a check sample. The level of the spike shall be at the regulatory action level when applicable. Otherwise, the spike shall be at 5 times the estimate of the quantification limit. The matrix used shall be phase matched with the samples and well characterized: for example, reagent grade water is appropriate for an aqueous sample.

Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Environmental Sample: An environmental sample or field sample is a representative sample of any material (aqueous, nonaqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required. Environmental samples are normally classified as follows:
Drinking Water--delivered (treated or untreated) water designated as potable water;
Water/Wastewater--raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluent;
Sludge--municipal sludges and industrial sludges;
Waste--aqueous and nonaqueous liquid wastes, chemical solids, contaminated soils, and industrial liquid and solid wastes.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

Instrument Calibration: Analysis of analytical standards for a series of different specified concentrations; used

to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Matrix: The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

Matrix Spike: Aliquot of a sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate: A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

Method Blank: An analytical control consisting of all reagents, internal standards and surrogate standards, which is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

MSDS: Material safety data sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs. The thrust of the law is to have those who make, distribute, and use hazardous materials responsible for effective communication. See the Hazard Communication Rule, 29 CFR, Part 1910, 1200, as amended, Sec. g. See Schedule I, Sec. 12, of the Canadian Hazardous Products Act.

PCB: Polychlorinated biphenyls (PCBs) are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a frequent environmental pollutant.

Precision: Precision is the agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.

Quality Control: Set of measures within a sample analysis methodology to assure that the process is in control.

Standard Curve: A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

Stock Solution: Standard solution which can be diluted to derive other standards.

Surrogate: Surrogates are 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, calibration and check standards, samples (including duplicates and QC reference samples) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Surrogate Standard: A pure compound added to a sample in the laboratory prior to extraction so that the overall efficiency of a method can be assessed.

12.0 Pollution and Waste Management

12.1 Pollution Prevention: see NE168.SOP for details

12.2 Waste Management: see SOPS NE054.SOP, NE083.SOP and NE089.SOP for details.

13.0 Method Performance

13.1 Method performance statistics for matrix spikes and surrogate spikes and reference samples and are maintained by the QA/QC Department.

14.0 Method Detection Limits

14.1 Method Dectection Limits (MDL) studies are statistically determined by per 40CFR136 Appendix B. MDLs are initially established and are re-established if major equipment changes are undertaken. MDLs for this method generally range from 7 to 11 ug/L for each Aroclor.

15.0 Sample Collection, Preservation and Storage

15.1 Grab samples are collected in clean 1-Liter glass bottles with Teflon lined caps. Samples are shipped to the lab chilled (4 +/- 2 degrees Celsius) and are stored under refrigeration. The extraction hold time for aqueous samples is 7 days from the date collected.

ATTACHMENT A

Table 1
PCB Stock Standard Preparation Table

PCB Formulation	Supplier	Catalog #	Standard weight(mg)	Conc. (PPM)
A1016	Monsanto Neat Archive	NA	93.2	932.0
A1221	Monsanto Neat Archive	NA	106.8	1068.0
A1232	Monsanto Neat Archive	NA	103.3	1033.0
A1242	Monsanto Neat Archive	NA	99.0	990.0
A1248	Monsanto Neat Archive	NA	101.9	1019.0
A1254	Monsanto Neat Archive	NA	99.6	996.0
A1260	Monsanto Neat Archive	NA	99.2	992.0
DCBP (Surrogate)	Chem Service	F2170	10	100.0

Unless otherwise noted hexane is the solution used to make all dilutions.

Table 2
PCB Working Standard Preparation Table

PCB Stock Standards	Init. Volume (mL)	Final Volume (mL)	Conc. (PPM)
A1016	1.0	100	9.32
A1221	1.0	100	10.68
A1232	1.0	100	10.33
A1242	1.0	100	9.90
A1248	1.0	100	10.19
A1254	1.0	100	9.96
A1260	1.0	100	9.92

Table 3
PCB Calibration Standard Preparation Table (High Level Calibration Curve)

Initial Volume (mL)	Initial Conc. (ug/mL)*	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
5.0	(10.0)*	50	0.932	1.068	1.033	0.990	1.019	0.992
2.5	(10.0)*	50	0.466	0.534	0.5165	0.495	0.5095	0.496
1.25	(10.0)*	50	0.233	0.267	0.25825	0.2475	0.2548	0.248
1.00	(10.0)*	50	0.1864	0.2136	0.2066	0.198	0.2038	0.1984
0.500	(10.0)*	50	0.0932	0.1068	0.1033	0.0990	0.1019	0.0992
5.0	(0.250)*	50	0.01864	0.02136	0.02066	0.0198	0.02038	0.01984

*Nominal Concentration, see Table 2 for actual working standard concentrations for each Aroclor

Table 3A
PCB Calibration Standard Preparation Table (Low Level Calibration Curve)

Init. Volume (mL)	Initial Conc. (ug/ml)*	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
1.0	(1.0)*	100	0.09320	0.1068	0.1033	0.09900	0.1019	0.09920
2.5	(1.0)*	50.0	0.04660	0.05340	0.05165	0.0495	0.05095	0.04960
10	(0.20)*	100	0.01864	0.02136	0.02066	0.01980	0.02038	0.01984
1.0	(1.00)*	50.0	0.00932	0.01068	0.01033	0.00990	0.01019	0.00992
0.50	(0.500)*	50.0	0.00466	0.00534	0.00517	0.00495	0.00509	0.00496

* Nominal Concentration, see Table 2 for actual working standard concentrations for each Aroclor.

Table 4
PCB A1254 Calibration Standard Preparation Table (for High Level Curve)

Initial Volume (mL) A1254	Initial Conc. (ug/mL) A1254	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.0	9.96	0	50	0.996	0	0
10.0	9.96	4.0	100	0.996	0.020	0.200
25.0*	0.996	0	50	0.498	0.010	0.100
1.25	9.96	0.800	50	0.249	0.008	0.080
0.500	9.96	0.500	50	0.0996	0.005	0.050
0.100**	0.996	0.200	50	0.01992	0.002	0.020

*This initial volume is of the A1254 0.996 ppm calibration standard WITH surrogates.
**This initial volume is of the A1254 0.996 ppm secondary stock solution WITHOUT surrogates.

Table 4A
PCB A1254, TCMX and DCBP Calibration Standard Preparation Table (for Low Level Curve)

Initial Volume A1254 (mL)	Initial Conc. A1254 (ug/mL)	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.00	0.996	0.80	50	0.0996	0.00800	0.0800
2.50	0.996	0.50	50	0.04980	0.00500	0.0500
1.0	0.996	0.40	50	0.01992	0.00400	0.0400
0.50	0.996	0.250	50	0.00996	0.00250	0.0250
0.25	0.996	0.100	50	0.00498	0.00100	0.0100

ATTACHMENT B

Table 1
PCB Continuing Calibration Stock Standards

PCB	Supplier	Catalog #	Conc. (ug/mL)
A1016	Chem Service	F107AS	1000
A1221	Chem Service	F108AS	1000
A1232	Chem Service	F113AS	1000
A1242	Chem Service	F109AS	1000
A1248	Chem Service	F110AS	1000
A1254	Chem Service	F111AS	1000
A1260	Chem Service	F112BS	1000

Table 2
PCB Continuing Calibration Working Standards
prepared from 1000 PPM Stock Standards

PCB	Initial Volume (mL)	Final Volume (mL)	Concentration (PPM)
A1016	1.0	100	10.0
A1221	1.0	100	10.0
A1232	1.0	100	10.0
A1242	1.0	100	10.0
A1248	1.0	100	10.0
A1254	1.0	100	10.0
A1260	1.0	100	10.0

Table 3
PCB Continuing Calibration Standards (High Level)
prepared from 10 PPM CCV Working Standards

PCB	Initial Volume (mL)	Final Volume (mL)	Concentration (PPM)
A1016	2.5	50	0.500
A1221	2.5	50	0.500
A1232	2.5	50	0.500
A1242	2.5	50	0.500
A1248	2.5	50	0.500
A1254 and Surrogate*	2.5 and 1.0	50	0.500 and (0.010/0.100)*
A1260	2.5	50	0.500

*Surrogate stock solution 0.500 PPM TCMX and 5.0 PPM DCBP

Table 3A
PCB Continuing Calibration Standards (low Level)
prepared from 0.500 PPM CCV Standards

PCB	Initial Volume (mL)	Final Volume (mL)	Concentration (PPM)
A1016	1.0	10	0.050
A1221	1.0	10	0.050
A1232	1.0	10	0.050
A1242	1.0	10	0.050
A1248	1.0	10	0.050
A1254 and Surrogate*	1.0 and 0.100*	10	0.0500 and (0.005/0.050)*
A1260	1.0	10	0.500

*Surrogate stock solution 0.500 PPM TCMX and 5.0 PPM DCBP

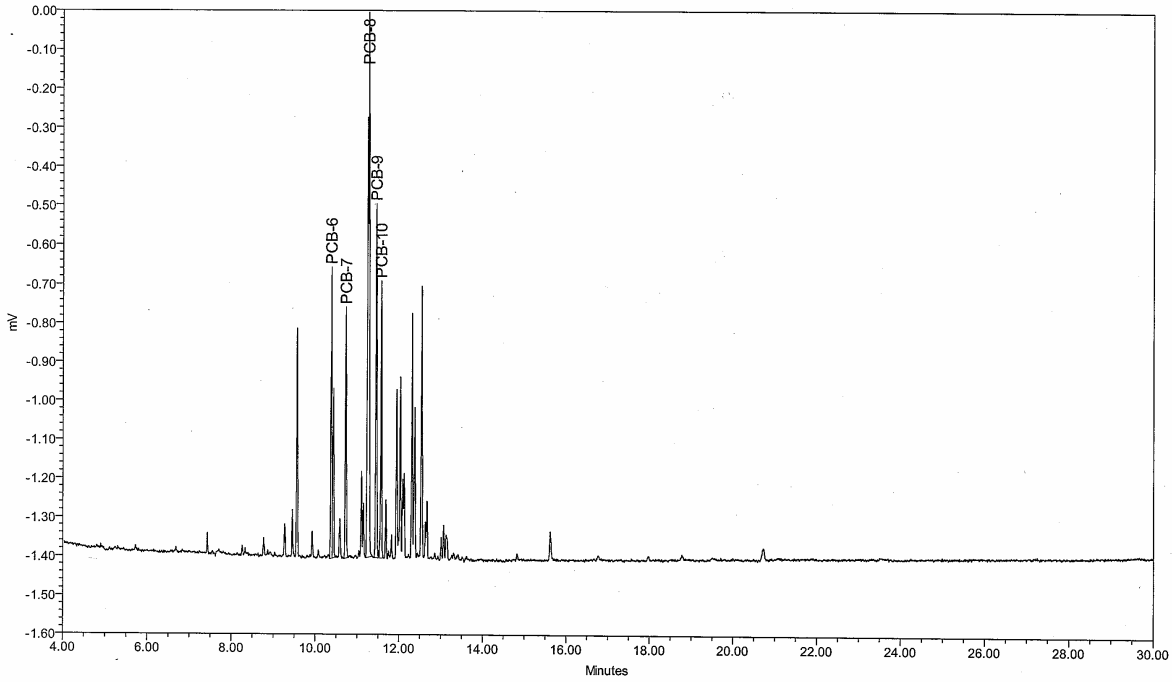
ATTACHMENT C

Gas Chromatograph Operating Procedures

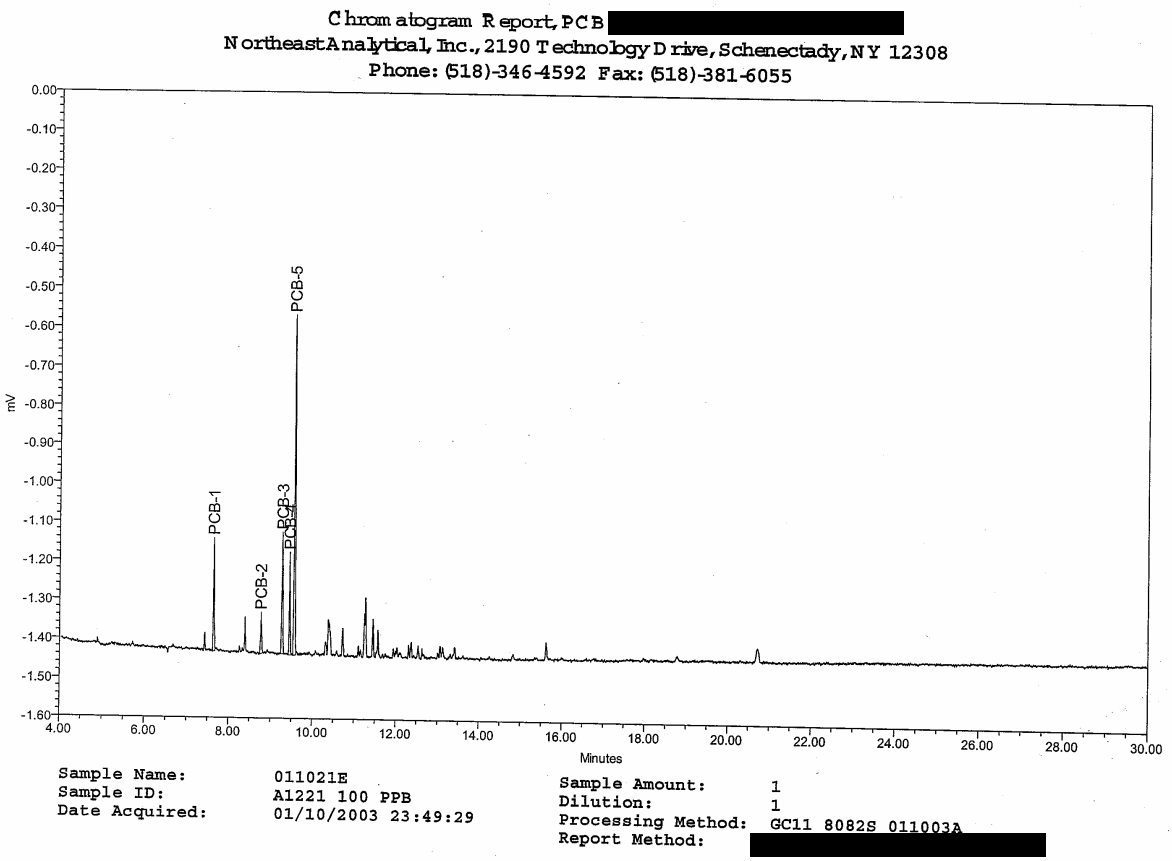
Column Type	Capillary	Capillary
Column ID	DB5-MS	DB-1
Vendor	J&W	J&W
Part Number	122-5532	122-1032
Column Length(m)	30	30
ID(mm)	0.25	0.25
Film Thick.(um)	0.25	0.25
1)Initial Col. Temp. (^{o}C)	140	140
1)Col. Hold Time (min.)	1.0	1.0
1)Col. Temp. Rate (^{o}C/min.)	10	10
1)Final Col. Temp. (^{o}C)	200	200
1)Col. Hold Time (min.)	NA	NA
2)Col. Temp. Rate (^{o}C/min.)	5	5
2)Final Col. Temp. (^{o}C)	245	245
2)Col. Hold Time (min.)	14.50	14.50
GC Col. gas flow rate (mL/min.)	17-24	17-24
ECD autozero	Yes	Yes
Detector Temp.(^{o}C)	300	300
Init. Injector Temp. (^{o}C)	300	300
A/S Vial Needle Depth	85	85
A/S Solvent Select	3	3
A/S Upper Air Gap	Yes	Yes
A/S Lower Air Gap	Yes	Yes
A/S Viscosity Factor	4	1
A/S Hot Needle Time (min.)	0.05	0.05
Autosampler(A/S) Model Number	8100	8100
A/S Injection Volume (uL)	1.3	1.3
A/S Injection Time (min.)	0.01	0.01
A/S Injection Rate (uL/sec.)	Fast 4.0	Fast 4.0
A/S Solvent Inj. plug size (uL)	0.2	0.2

ATTACHMENT D

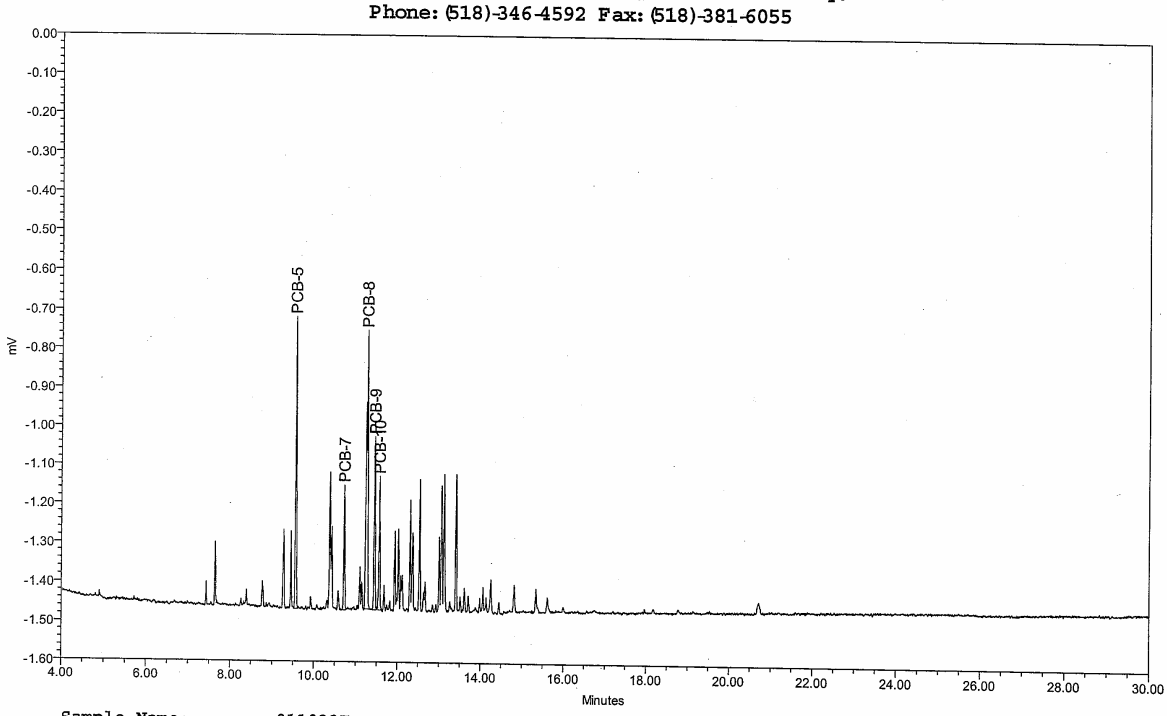
Chromatogram Report, PCB [redacted]
NortheastAnalytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055



Sample Name:	011016E	Sample Amount:	1
Sample ID:	A1016 100 PPB	Dilution:	1
Date Acquired:	01/10/2003 20:33:58	Processing Method:	GC11_8082S_011003A
		Report Method:	[redacted]


Chromatogram Report, PCB [redacted]
NortheastAnalytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055
mV
0.00
-0.10
-0.20
-0.30
-0.40
-0.50
-0.60
-0.70
-0.80
-0.90
-1.00
-1.10
-1.20
-1.30
-1.40
-1.50
-1.60
PCB-1
PCB-2
PCB-3
PCB-4
PCB-5
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
26.00
28.00
30.00
Minutes
Sample Name: 011021E
Sample ID: A1221 100 PPB
Date Acquired: 01/10/2003 23:49:29
Sample Amount: 1
Dilution: 1
Processing Method: GC11 8082S 011003A
Report Method: [redacted]

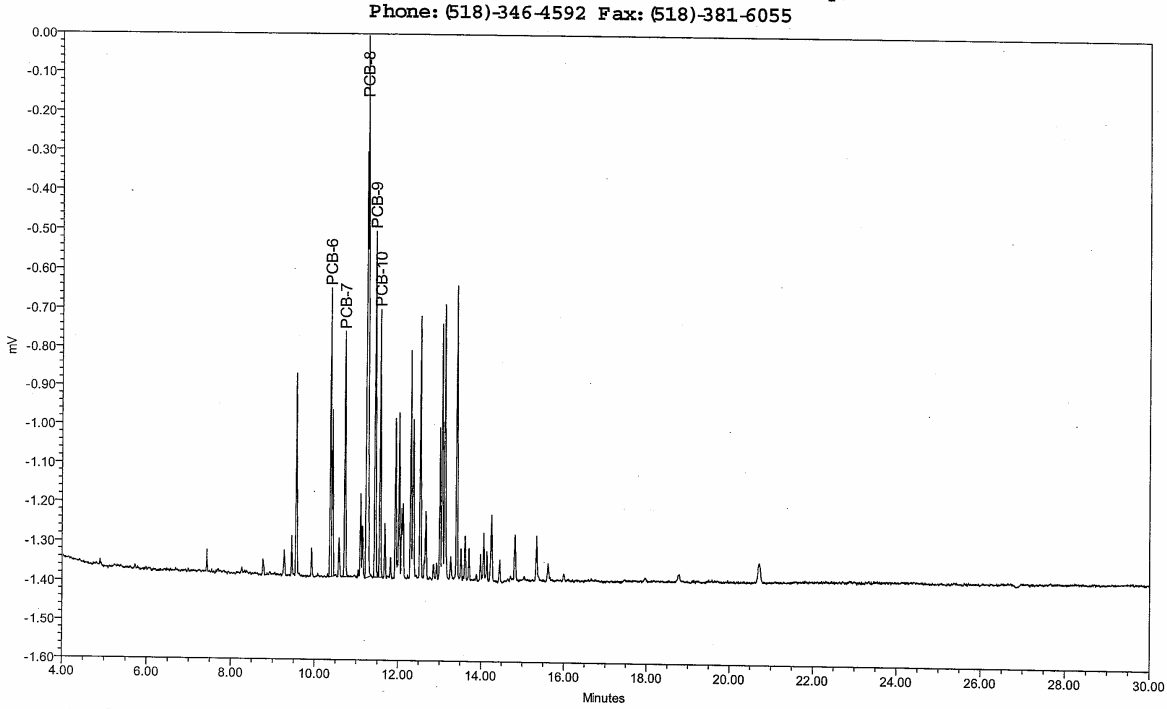
Chromatogram Report, PCB [redacted]
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055



Sample Name: 011032E
Sample ID: A1232 100 PPB
Date Acquired: 01/11/2003 03:04:48

Sample Amount: 1
Dilution: 1
Processing Method: GC11_8082S_011003A
Report Method: [redacted]

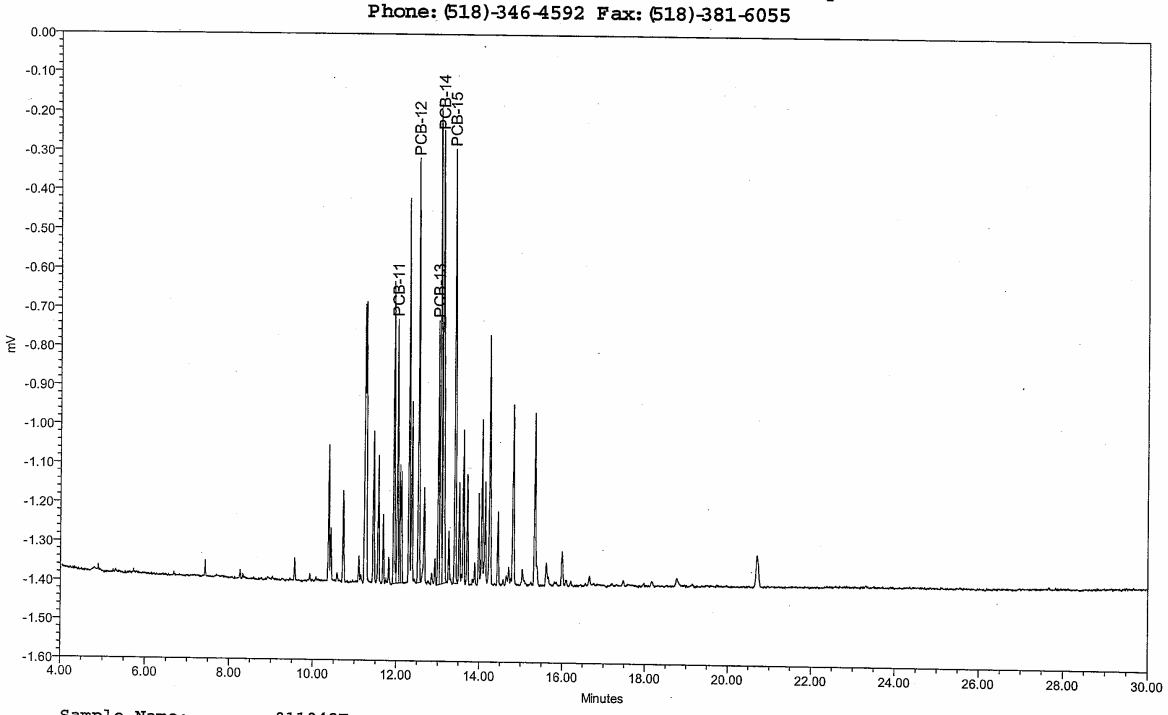
Chromatogram Report, PCB [redacted]
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055



Sample Name: 011042E
Sample ID: A1242 100 PPB
Date Acquired: 01/11/2003 06:20:14

Sample Amount: 1
Dilution: 1
Processing Method: GC11_8082S_011003A
Report Method: [redacted]

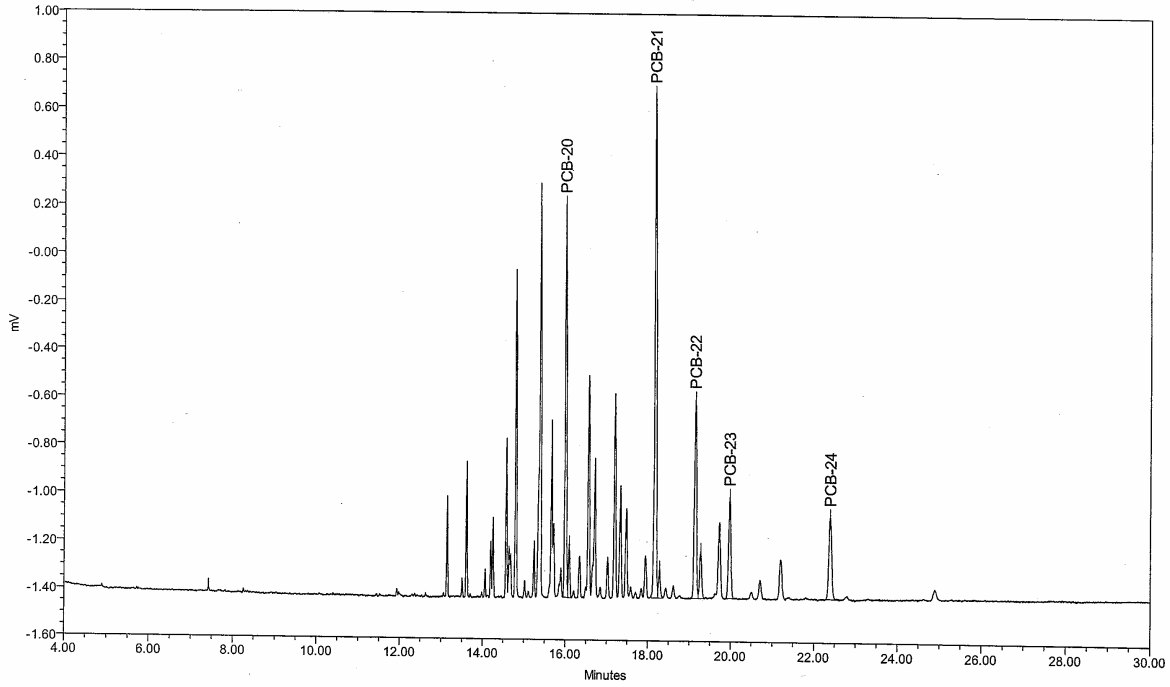
Chromatogram Report, PCB [redacted]
Northeast Analytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055



Sample Name: 011048E
Sample ID: A1248 100 PPB
Date Acquired: 01/11/2003 09:35:33

Sample Amount: 1
Dilution: 1
Processing Method: GC11 8082S 011003A
Report Method: [redacted]

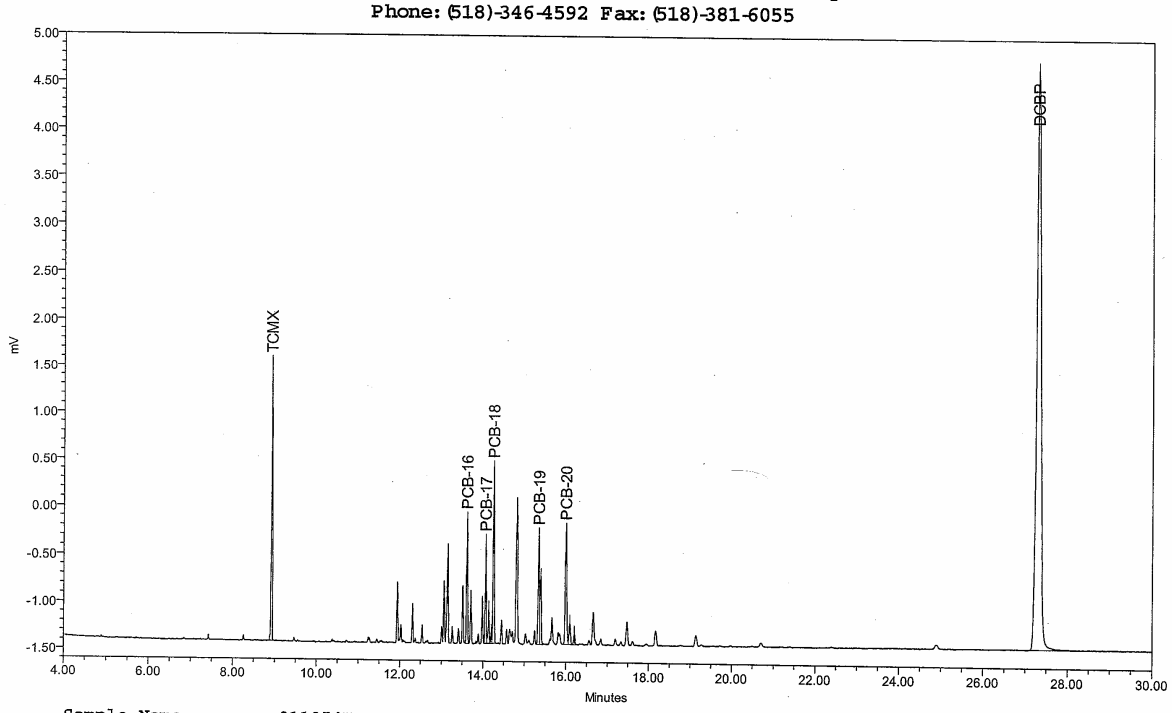
Chromatogram Report, PCB [redacted]
NortheastAnalytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055



Sample Name: 011060E
Sample ID: A1260 100 PPB
Date Acquired: 01/11/2003 16:06:17

Sample Amount: 1
Dilution: 1
Processing Method: GC11_8082S_011003A
Report Method: [redacted]

Chromatogram Report, PCB [redacted]
NortheastAnalytical, Inc., 2190 Technology Drive, Schenectady, NY 12308
Phone: (518)-346-4592 Fax: (518)-381-6055



Sample Name: 011054E
Sample ID: A1254 100 PPB
Date Acquired: 01/11/2003 12:50:53

Sample Amount: 1
Dilution: 1
Processing Method: GC11 8082S 011003A
Report Method: [redacted]

ATTACHMENT E

Example Calculations

Initial Calibration Curve by first Order Linear Regression with intercept:

$Y=A+Bx$

Y= Response µV-sec (area sum of 5 quant peaks)
A= Constant (Intercept) µV-sec
B= First Order Coefficient (slope)
x= Solution Concentration (ng/ml)

Unknown Sample Solution Concentration:

$$x = \frac{(Y_{(meas)}-A)}{B}$$

Unknown Sample Final Concentration:

$$\text{Conc. (ug/L)} = \frac{(x)(v_t)(D.F.)\ (1/1000)}{V(t)}$$

x= Solution Concentration (ng/ml)
v_t = Concentrated Extract Volume (ml)
D.F. = Analytical Dilution Factor
Vt = Sample Total Volume (L)

ATTACHMENT F

Northeast Analytical Inc

04/04/02

Retention Time Window Study
for Aroclors (PCB) by GC/ECD

[redacted]

Instrument: GC#
Column: DB1-30Meter

Analyte	PEAK	Standard 1 50 PPB R.T. Min	Standard 2 50 PPB R.T. Min	Standard 3 50 PPB R.T. Min	STD. DEV Min	%RSD	Window +/- Min.
Aroclor 1016		C__0401B	CS_0403	CS_0404			
	6	10.431	10.434	10.430	0.0021	0.020	0.006
	7	10.777	10.780	10.775	0.0025	0.023	0.008
	8	11.321	11.325	11.320	0.0026	0.023	0.008
	9	11.498	11.502	11.496	0.0031	0.027	0.009
	10	11.616	11.619	11.613	0.0030	0.026	0.009
Aroclor 1221		C__0401B	C__0403A	CS__0404			
	1	7.705	7.707	7.706	0.0010	0.013	0.003
	2	8.833	8.837	8.835	0.0020	0.023	0.006
	3	9.334	9.335	9.333	0.0010	0.011	0.003
	4	9.508	9.512	9.510	0.0020	0.021	0.006
	5	9.619	9.621	9.619	0.0012	0.012	0.003
Aroclor 1232		CS__0401	CS__0403	CS__0404			
	5	9.622	9.619	9.622	0.0017	0.018	0.005
	7	10.779	10.776	10.779	0.0017	0.016	0.005
	8	11.325	11.321	11.323	0.0020	0.018	0.006
	9	11.501	11.498	11.499	0.0015	0.013	0.005
	10	11.619	11.615	11.617	0.0020	0.017	0.006
Aroclor 1242		CS__0401	CS__0403	CS__0404			
	6	10.431	10.432	10.430	0.0010	0.010	0.003
	7	10.777	10.778	10.774	0.0021	0.019	0.006
	8	11.322	11.322	11.320	0.0012	0.010	0.003
	9	11.498	11.498	11.496	0.0012	0.010	0.003
	10	11.616	11.617	11.614	0.0015	0.013	0.005
Aroclor 1248		CS__0401	CS__0403	CS__0404			
	11	12.074	12.071	12.070	0.0021	0.017	0.006
	12	12.582	12.579	12.578	0.0021	0.017	0.006
	13	13.052	13.048	13.047	0.0026	0.020	0.008
	14	13.168	13.165	13.163	0.0025	0.019	0.008
	15	13.454	13.451	13.450	0.0021	0.015	0.006
Arolcor 1254		CS__0401	CS__0403	CS__0404			
	16	13.655	13.651	13.651	0.0023	0.017	0.007
	17	14.099	14.097	14.098	0.0010	0.007	0.003
	18	14.291	14.289	14.288	0.0015	0.011	0.005
	19	15.383	15.382	15.381	0.0010	0.007	0.003
	20	16.042	16.041	16.039	0.0015	0.010	0.005
Arolcor 1260		CS__0401	CS__0403	CS__0404			
	20	16.045	16.045	16.041	0.0023	0.014	0.007
	21	18.212	18.210	18.207	0.0025	0.014	0.008
	22	19.183	19.182	19.179	0.0021	0.011	0.006
	23	20.016	20.016	20.007	0.0052	0.026	0.016
	24	22.425	22.420	22.410	0.0076	0.034	0.023
TCMX (SURROGATE)	Surr.	8.988	8.986	8.987	0.0010	0.011	0.003
DCB (SURROGATE)	Surr.	27.277	27.273	27.270	0.0035	0.013	0.011

Q:\RTWIN\031302B.XLS

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE141_05.DOC
REVISION NUMBER: 05

STANDARD OPERATING PROCEDURE FOR EXTRACTION OF AQUEOUS SAMPLES FOR PCB ANALYSIS USING SEPARATORY FUNNEL EXTRACTION (SW-846 METHOD 3510C)

DECEMBER 10, 2003

COPY

STANDARD OPERATING PROCEDURE

Author: Heather L. Carlson

Reviewed by:

Heather L. Carlson
Aqueous Extraction Supervisor

Approved by:

William A. Kotas
Quality Assurance Officer

Northeast Analytical, Inc.
Issuing Section: Organics
SOP Name: NE141_05.doc
Date: 12/10/2003
Revision: 05

1.0 TITLE **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION AND CLEANUP OF AQUEOUS SAMPLES USING THE SEPARATORY EXTRACTION TECHNIQUE (EPA SW-846 METHOD 3510C) FOR SUBSEQUENT ANALYSIS BY US-EPA SW-846 METHOD 8082 OR EPA METHOD 608**

2.0 PURPOSE The purpose of this SOP is to provide the extraction chemist with the procedures required to perform extractions of PCBs from water/wastewater samples, using the Separatory Funnel extraction technique and to perform the subsequent extract volume reduction and cleanup.

3.0 SCOPE The following procedure is utilized by Northeast Analytical, Inc. for the extraction, extract concentration and cleanup of PCBs from water/wastewater samples using the Separatory Funnel extraction method for analysis by SW-846 Method 8082 and EPA Method 608.

4.0 COMMENTS The sample extraction chemist should be aware of sample hold times for this method, which is seven days from the date the samples were collected.

5.0 SAFETY The extraction chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and gloves must be worn when handling glassware and samples. Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The extraction chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before beginning the extractions. All equipment and solvents should be handled within a lab fume hood.

6.0 REQUIREMENTS The extraction chemist must have an understanding of the methods and requirements of US

EPA-SW-846 "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition Method 3510C. An approved instructor must also certify the extraction chemist to perform the procedure. The extraction chemist should have performed an acceptable precision and accuracy demonstration prior to performing this method without supervision.

7.0 EQUIPMENT:

7.1 **Separatory Funnel,** 2 Liter, with Teflon stopcock and Teflon stopper.

7.2 **Graduated cylinder,** 1000mL, 100mL Kimax, Kimble#20026-100

7.3 **Beakers,** Assorted Pyrex: 600mL and 1000mL, used for liquid containment and pipette storage.

7.4 **Boston bottles,** 125mL volume with Teflon lined cap.

7.5 **Disposable Glass Pasteur pipettes**: 9", Krackeler-Brand, Cat#67-450-900 (or equivalent)

7.6 **pH Indicator Strips:** pH Range: 0-14.0, EM Science # 9590 (or equivalent)

7.7 **1:1 Sulfuric acid:** (H_2SO_4)**,**
Preparation: To a beaker containing 500mL cold DI-water slowly and under constant stirring add 500mL concentrated H_2SO_4. Allow the mixture to cool after preparation.

7.8 **5N Sodium Hydroxide;** J.T.Baker # 5761-03

7.9 **Acetone,** High Purity Solvent (Burdick/Jackson) #UN1090. (or equivalent)

7.10 **Dichloromethane,** High Purity Solvent (Burdick/Jackson) #UN1593

7.11 **Hexane,** High Purity Solvent (Burdick/Jackson) #UN1208. (or equivalent)

7.12 **Toluene,** High Purity Solvent (Burdick/Jackson) #UN1294. (or equivalent)

7.13 **1.0 mL Gastight Syringe;** Hamilton #81317

7.14 **Drying column,** Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

7.15 **Glass Wool,** Silanized, Supelco #2-0410, solvent washed as per NE159.sop.

7.16 **Powder Funnel,** Pyrex

7.17 **Sodium Sulfate,** J.T.Baker #3375-05, Solvent washed as per NE039.sop.

7.18 **1:1 Magnesium Sulfate,** EM Science #MX0075-03/**Sodium Sulfate,** J.T.Baker #3375-05, Solvent washed as per NE091.sop

7.19 **Stainless Steel Spatula**

7.20 **TurboVap Evaporator concentrator tubes (TurboTubes),** Zymark 250mL, 0.5mL endpoint.

7.21 **TurboVap Evaporator,** Zymark #ZW640-3.

7.22 **Volumetrics**; 10 mL or 5 mL; Pyrex #5640

7.23 **Vials:** glass, 8-dram and 4-dram (with polyseal sealed cap), for sample extracts.

7.24 **Vial Rack**: Plastic rack used to hold vials, during all phases of the extract processing.

7.25 **Concentrated Sulfuric Acid,** Mallinckrodt #2468, #UN1830, Solvent washed as per NE174.doc

7.26 **Florisil,** J.T. Baker #M368-08, solvent washed as per NE094.doc.
SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.

7.27 **Mercury**; Triple distilled, Mercury Waste Solutions Inc., Solvent washed as per NE175.sop (or equivalent)

7.28 **Centrifuge:** International Equipment Co., Model CL. (or equivalent)

7.29 **Wrist Shaker:** Burrell wrist action shaker, Model 75 and 88. (or equivalent)

7.30 **Auto sampler vials**; Scientific resource Inc., # 99468-A or equivalent

8.0 PROCEDURES:

8.1. SAMPLE PREPARATION

8.1.1 Throughout the entire process it should be noted that if the extraction chemist encounters any problems or difficulties with any samples or steps involved, all work should **STOP!!!** Any problems should be brought to the attention of the supervisor and documented in the sample extraction logbook.

8.1.2 Before any steps are taken, the extraction chemist should first review the sample job folder and check the sample labels versus the original chain of custody. Any discrepancies should be noted in the sample extraction logbook.

8.1.3 Bring samples to room temperature by letting the samples warm up in the laboratory for a minimum of 30 minutes.

8.1.4 Mark the level of the sample on the outside of the sample container with a felt tip pen. Determine the pH of the sample by removing a small amount of sample with a Pasteur pipette (approximately 0.1 mL) and wet a pH indicator strip. The pH should be between 5 and 9. Adjust the pH with 1:1 Sulfuric Acid if the pH is greater than 9 or 5N Sodium Hydroxide if the pH is lass than 5. Record the original pH in the extraction logbook.

8.1.5 Using 1 Liter clear sample bottles, measure out two, 1 Liter aliquots of DI water to be used as an extraction blank and laboratory control spike.

8.2. SAMPLE EXTRACTION

8.2.1 In a chemical fume hood set up Separatory Funnels with Teflon stopcocks and Teflon stoppers for each sample on a funnel holder. Rinse with approximately 20mL of Acetone. Drain and discard the rinse solvent, then rinse again with approximately 20mL of Dichloromethane, drain and discard the rinse also. Allow the Separatory Funnels to dry completely.

8.2.2 Rinse one Boston bottle per sample with Dichloromethane and discard rinse solvent. Let the solvent evaporate by laying the bottles in the fume hood. Label each bottle with appropriate Sample ID, cover with tape to eliminate destroying of ID number.

8.2.3 Close the stopcock. Add 40mL of Dichloromethane to the Separatory Funnel using a 100mL graduated cylinder. Place one of the correctly labeled Boston bottles under each Separatory Funnel drain in case of any leaks.

8.2.4 Invert the sample bottle twice to three times to thoroughly mix the sample, then carefully transfer the sample into the Separatory Funnel, making sure not to splash or allow sample to run down the outside of the glassware.

8.2.5 Spike surrogate- and matrix-spike compound solutions directly into sample in the Separatory Funnel. Record the surrogate- and matrix-spike concentration, the amount spiked, and the spike solution reference code in the extraction logbook.

8.2.6 Stopper the Separatory Funnel, remove from rack and vent quickly by holding it upside down and opening the stopcock (**aiming it into the hood and away from any other samples**) to release vapor pressure. Keep a secure hold on the Teflon stopper at all times. Shake and vent until minimal if any vapor pressure is released when the stopcock is opened. Shake the Separatory Funnel vigorously for two minutes venting occasionally.

8.2.7 Return the Separatory Funnel back to the rack and remove the stopper. Allow the solvent and water phases to separate for a minimum of 10 minutes. After 2 or 3 minutes swirl the samples by moving the Separatory Funnel in a circular motion creating a vortex in the water which will sink any Dichloromethane trapped on the surface.

8.2.8 Slowly drain the bottom Dichloromethane layer into the pre-rinsed and correctly labeled 125mL Boston bottle.

8.2.9 Add 30 mL of Dichloromethane to sample bottle using a 100 mL graduated cylinder. Cap and shake the bottle to rinse. Transfer this rinse to the corresponding Separatory Funnel by carefully pouring.

8.2.10 Repeat steps 8.2.6 through 8.2.9 once more times for a total of three shakes.

8.2.11 Cap the Boston bottles and put into a refrigerator to chill the samples.

8.2.12 Empty the wastewater from the Separatory Funnels into a waste container for proper disposal according to Chemical Hygiene Plan.

8.2.13 Separatory Funnels must be rinsed with Acetone and dried in the hood before being washed with warm water, rinsed with RO water and muffled at 400?C in a Muffle furnace for a minimum of three hours before storage and reuse.

8.2.14 Fill up sample bottle with water to previous level. Pour water into a 1 Liter graduated cylinder and record sample volume in the sample extraction logbook. Discard sample container according to

guidelines in Chemical Hygiene Plan.

8.3 SAMPLE EXTRACT DRYING PROCEDURE

8.3.1 All water extracts must be dried to remove residual water prior to concentration and cleanup. Assemble the drying columns with a Teflon stopcock, Teflon stopcock seal, and Viton O-ring. There should be one column for each sample with was extracted.

8.3.2 Insert a small plug of glass wool in the bottom of the column, using a Hexane pre-rinsed 5 mL disposable pipette. The glass wool is to support the drying agent.

8.3.3 Using a Pyrex powder funnel, fill each column with approximately 2 inches of Sodium Sulfate in height from the bottom of the column.

8.3.4 Rinse the column and sodium sulfate with a full column of Hexane, draining the Hexane into a clean rinse bucket and leaving just enough to cover the Sodium Sulfate.

8.3.5 Rinse TurboTubes with Hexane, label one per sample and set in holders. Place one directly under each column.

8.3.6 Remove the samples from the refrigerator. Using a Hexane pre-rinsed stainless steel spatula, slowly add a 1:1 mixture of $MgSO_4$and Na_2SO_4. Cap the bottle and shake, adding more if necessary until the drying agent is free flowing. Depending on the amount of water in the sample it will take between 1 and 7 scoops of 1:1 $MgSO_4$ /Na_2SO_4 to absorb all the water.

8.3.7 Once the drying agent is free flowing, carefully pour the sample into the column. While the bottle is still inverted over the column rinse the threads with a disposable Pasteur pipette volume of Hexane to complete the transfer of extract.

8.3.8 Add three to four disposable Pasteur pipette volumes of Hexane to the Boston bottle, cap and shake. Pour the Hexane into the column and rinse the bottle rim. Repeat the bottle rinse twice more then rinse down the insides of the column with Hexane.

8.3.9 Open the stopcock and allow the sample to drain into the Turbo Tube. **DO NOT** let the drying column go dry until the end of 8.3.11.

8.3.10 Stop the column from draining just above the Sodium Sulfate. Rinse insides of the column with three to four disposable Pasteur pipette volumes of Hexane.

8.3.11 Using a graduated cylinder, pour 60 mL of Hexane into the column to completely rinse the column into the TurboTube. Let the column drain into the tube for several minutes after the solvent has eluted.

8.3.12 Add 200 uL of Toluene to the sample extract (6-8 drops from a disposable Pasteur pipette).

8.4 SOLVENT REDUCTION: TURBO VAP EVAPORATOR SYSTEM

8.4.1 The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow / vortex action. The unit maintains a

slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

8.4.2 Turn the unit on (switch is located on the backside of the unit) and allow to heat up to the specified temperature for individual solvent use. For this procedure the temperature is 38?C $\pm$ 2?C. This is indicated by the "Heating" display light, located above the temperature control knob on the right side of the unit. The system is at the proper temperature when the "At Temperature" light is lit. This is located above the "Heating" display light. To verify this temperature there is a thermometer located next to the TurboVap, which should be checked periodically to assure the desired temperature is being maintained.

8.4.3 As a precaution the TurboVap system regulators should be checked to assure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Make sure that the nitrogen gas pressure regulator is off. Bleed any residual gas until the regulator gauge reads "0" psi. Remove the empty TurboTube.

8.4.4 Wipe down inside of TurboVap with a Hexane wetted paper towel including top lid and pins. Place TurboTubes containing the sample extracts into the TurboVap and close lid. Slowly open the pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the volume reduction.

8.4.5 The process for solvent (Dichloromethane /Hexane) reduction takes approximately 30-45 minutes. **DO NOT** leave the unit unattended as extracts may be blown to dryness and loss of PCB as well as surrogate- and matrix-spike may occur. Immediately notify the extraction supervisor if an extract is blown to dryness and note the incident in the extraction logbook.

8.4.6 Concentrate the solvent to approximately 10 mL. Remove from the TurboVap and place in holder. Add about 90 mL of Hexane to the TurboTube.

8.4.7 Make sure the gas pressure regulator is off.

8.4.8 Repeat step 8.4.4. The process for solvent reduction takes approximately 30 minutes.

8.4.9 Concentrate to approximately 5 mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist almost entirely of Hexane, this process of replacing one solvent with another is called a solvent exchange.
NOTE*:* Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing before the 5.0 mL point is achieved. Samples, which stop reducing, should be removed as soon as possible.

8.4.10 Using a disposable Pasteur pipette quantitatively transfer the sample extract into a Hexane pre-rinsed 10 mL volumetric. Use Hexane to rinse the TurboTube and set to volume. Invert the volumetric several times to mix completely.

8.4.11 Transfer sample from the volumetric to a correctly labeled 4-dram vial.

8.4.12 All dirty glassware must be rinsed with Acetone and dried in the fume hood before being washed with warm water, rinsed with RO water and muffled.

8.5 SAMPLE EXTRACT CLEANUP PROCEDURE

Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances, which must be removed before accurate chromatographic analysis can be performed. Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The extraction chemist records the sequence and number of repeats of cleanup steps performed in the sample logbook. Sample extract cleanups are performed on set volume extracts. The set volume is 5mL or 10mL for water/wastewater samples.

8.5.1 Sulfuric Acid Wash

8.5.1.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

8.5.1.2 Add one disposable Pasteur pipette volume full of solvent washed concentrated Sulfuric Acid to each extract vial, and shake by hand in a chemical fume hood for 30 seconds. Then centrifuge for at least 1 minute on setting #4. Transfer the Hexane (upper) layer to a correctly labeled, pre-rinsed 4-dram vial.

8.5.1.3 Repeat 8.5.1.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required.
Note: It is entirely possible that all colored material will not be removed from the extract.

8.5.2 Florisil Adsorption (Slurry)

8.5.2.1 The Florisil slurry removes co-extracted polar compounds, residual water, and residual acid.

8.5.2.2 Add one spatula of Florisil to each extract vial.
SEE SUPERVISOR FOR THE APPROPRIATE FLORISIL DEACTIVATION CONCENTRATION TO BE USED.

8.5.2.3 In a fume hood vigorously shake the vial for approximately 30 seconds by hand. Swirl to get any Florisil off the walls of the vial, then allow to settle.

8.5.2.4 Transfer the Hexane (upper) layer to a correctly labeled, pre-rinsed 4-dram vial.

8.5.3 Removal of Sulfur Using Mercury

NOTE: Mercury is a highly toxic metal all operations involving Mercury should be performed within a chemical fume hood. Prior to using Mercury, the extraction chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the Material Safety Data Sheet (MSDS).

8.5.3.1 Add 1-3 drops of solvent washed Mercury (NE175.doc) to the sample extracts, and cap. Handshake for 15-30 seconds. If the Mercury changes color or breaks up into tiny balls and will not reform the original ball, change the Mercury. To change the Mercury transfer extract

into a new correctly labeled, and Hexane pre-rinsed 4-dram vial and add new Mercury to it. Repeat previous step. Place on wrist action shaker for 30 minutes. The sulfur is converted to Mercuric Sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental Sulfur.

8.5.3.2 Remove the sample extracts from the wrist shaker.

8.5.3.3 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean up or Florisil slurry (discussed in 8.5.1 and 8.5.2).

8.6 FINAL EXTRACT PREPARATION

8.6.1 Transfer the extract to a Hexane pre-rinsed and correctly labeled final 4-dram vial. Labeled with the client, the test, the dilution factor, the date, and the sample ID number.

8.6.2 Transfer approximately 1.0 mL of extract into a labeled 1.5 mL GC autosampler vial.

8.6.3 Submit the GC vials along with the Project folder containing the GC Queue Lab Sheet and a copy of the extraction logbook page to the GC analyst.

8.7 EXTRACT SCREENING AND DILUTION

8.7.1 PCB extracts are screened by GC to determine the approximate concentration of PCBs before final analysis. Prior site history and client supplied estimates of sample concentration may be used to determine what, if any, extract dilution is necessary. Extracts of unknown concentration are generally screened at a 5 or 10 to 100 fold dilution.

8.7.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of Hexane. For example, adding 1.0mL of the extract to 9.0mL Hexane performs a 1 to 10 dilution. The vial containing the diluted extract is labeled denoting the equivalent extract volume after the dilution; e.g. a 25mL extract diluted 1:10 is labeled "250X", an undiluted 25mL extract is labeled "25X". When high dilutions are prepared, secondary (serial) dilutions of the initial diluent are prepared; e.g. a 100-fold dilution is prepared by a 1:10 dilution of the initial extract, then a 1:10 dilution of the resulting diluent.

8.7.3 Perform the dilution by using an appropriate class A disposable volumetric pipette to transfer the extract and make-up volume of Hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

8.7.4 Transfer 1mL of the extract to a labeled GC auto sampler vial. Record the screening dilution on the GC Queue Lab Sheet (LIMS spreadsheet) along with the extract volume, and the sample volume. Submit the GC Queue Lab Sheet and the project folder with the sample extracts to the GC analyst.

9.0 POLLUTION PREVENTION

See NEA168.SOP

10.0 WASTE MANAGEMENT

See NEA054.SOP, NEA083.SOP & NEA089.SOP

11.0 REFERENCES

1. US EPA SW-846 “Test Methods for Evaluating Solid Waste” Volume 1B Laboratory Manual Physical/Chemical Methods Office of Solid Waste and Emergency Response, Third Edition Final Update III , December 1996.

2. Guide to Environmental Analytical Methods, 4th Edition Genium Publishing Corporation, Schenectady, NY 12304, 1998

12.0 ATTACHMENTS

Attachment 1. Method flow chart

Attachment 1.

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC11_012004_1016_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1016	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	46.6 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040120W1	01/20/04	040120W1R	06/17/04	32.1	68.9%
2	040120W2	01/20/04	040120W2R	06/17/04	39.9	85.7%
3	040120W3	01/20/04	040120W3R	06/17/04	33.8	72.6%
4	040120W4	01/20/04	040120W4R	06/17/04	32.6	70.0%
5	040120W5	01/20/04	040120W5R	06/18/04	30.5	65.4%
6	040120W6	01/20/04	040120W6R	06/18/04	34.8	74.7%
7	040120W7	01/20/04	040120W7R	06/18/04	31.7	68.1%
8	040120W8	01/20/04	040120W8R	06/18/04	33.0	70.9%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	33.6	ng/L
STD (s):	2.88	ng/L
%RSD:	8.58%	
MDL:	8.64	ng/L
PQL:	43.2	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:		Date: 6-21-04
Gas Chromatography Analyst:		Date: 6/21/04
QA/QC Officer:		Date: 6/21/04
Assistant Lab Director:		Date: 6/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC11_013004_1221_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1221	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	53.4 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040130W1	01/30/04	040130W1	06/07/04	32.9	61.7%
2	040130W1	01/30/04	040130W1	06/07/04	35.2	66.0%
3	040130W1	01/30/04	040130W1	06/07/04	33.0	61.8%
4	040130W1	01/30/04	040130W1	06/07/04	30.5	57.2%
5	040130W1	01/30/04	040130W1	06/07/04	35.5	66.4%
6	040130W1	01/30/04	040130W1	06/07/04	38.0	71.2%
7	040130W1	01/30/04	040130W1	06/07/04	33.3	62.3%
8	040130W1	01/30/04	040130W1	06/07/04	32.7	61.2%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	33.9	ng/L
STD (s):	2.28	ng/L
%RSD:	6.72%	
MDL:	6.83	ng/L
PQL:	34.1	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Nicholas Marcotte	Date: 6-21-04
Gas Chromatography Analyst:	Cynthia Marcotte	Date: 6/21/04
QA/QC Officer:	[illegible]	Date: 6/21/04
Assistant Lab Director:	R.E. Wagner	Date: 6/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC11_021704_1232_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1232	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	51.65 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040217W1	02/17/04	040217W1	06/08/04	36.7	71.1%
2	040217W2	02/17/04	040217W2	06/08/04	42.1	81.5%
3	040217W3	02/17/04	040217W3	06/08/04	38.2	74.0%
4	040217W4	02/17/04	040217W4	06/08/04	39.6	76.7%
5	040217W5	02/17/04	040217W5	06/08/04	42.2	81.7%
6	040217W6	02/17/04	040217W6	06/08/04	43.0	83.3%
7	040217W7	02/17/04	040217W7	06/08/04	37.4	72.4%
8	040217W8	02/17/04	040217W8	06/08/04	39.9	77.3%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	39.9	ng/L
STD (s):	2.36	ng/L
%RSD:	5.92%	
MDL:	7.08	ng/L
PQL:	35.4	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Renee Nelson	Date: 6/21/04
Gas Chromatography Analyst:	Cynthia Mairs	Date: 6/21/04
QA/QC Officer:		Date: 6/21/04
Assistant Lab Director:	R.E. Wagner	Date: 6/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC18F_061404_1242_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1242	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	50.0 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040614W1	06/14/04	040614W1	06/17/04	28.4	56.8%
2	040614W2	06/14/04	040614W2	06/17/04	27.1	54.1%
3	040614W3	06/14/04	040614W3	06/17/04	32.5	65.0%
4	040614W4	06/14/04	040614W4	06/17/04	27.2	54.4%
5	040614W5	06/14/04	040614W5	06/17/04	30.7	61.4%
6	040614W6	06/14/04	040614W6	06/17/04	30.1	60.2%
7	040614W7	06/14/04	040614W7	06/17/04	25.2	50.5%
8	040614W8	06/14/04	040614W8	06/17/04	25.3	50.5%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	28.3	ng/L
STD (s):	2.61	ng/L
%RSD:	9.24%	
MDL:	7.84	ng/L
PQL:	39.2	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist: Date: 6/21/04

Gas Chromatography Analyst: Date: 6/21/04

QA/QC Officer: Date: 6/21/04

Assistant Lab Director: Date: 6/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC11_032204_1248_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1248	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	50.0 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040322W1	03/22/04	040322W1	06/08/04	39.4	78.8%
2	040322W2	03/22/04	040322W2	06/08/04	38.9	77.8%
3	040322W3	03/22/04	040322W3	06/08/04	40.8	81.6%
4	040322W4	03/22/04	040322W4	06/08/04	40.0	79.9%
5	040322W5	03/22/04	040322W5	06/08/04	38.0	75.9%
6	040322W6	03/22/04	040322W6	06/08/04	43.7	87.4%
7	040322W7	03/22/04	040322W7	06/08/04	39.3	78.5%
8	040322W8	03/22/04	040322W8	06/08/04	45.5	91.0%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	40.7	ng/L
STD (s):	2.59	ng/L
%RSD:	6.35%	
MDL:	7.75	ng/L
PQL:	38.7	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:	Renee Nelson	Date:	6/21/04
Gas Chromatography Analyst:	Cynthia Masella	Date:	6/21/04
QA/QC Officer:	[signature]	Date:	6/21/04
Assistant Lab Director:	R.E. Wagner	Date:	6/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC11_013004_1254_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1254	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	49.8 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040130W1	01/30/04	040130W1	06/07/04	46.2	92.7%
2	040130W2	01/30/04	040130W2	06/07/04	48.0	96.4%
3	040130W3	01/30/04	040130W3	06/07/04	43.9	88.1%
4	040130W4	01/30/04	040130W4	06/07/04	45.8	92.0%
5	040130W5	01/30/04	040130W5	06/07/04	45.9	92.1%
6	040130W6	01/30/04	040130W6	06/07/04	49.4	99.2%
7	040130W7	01/30/04	040130W7	06/07/04	49.3	99.1%
8	040130W8	01/30/04	040130W8	06/07/04	52.4	105%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	47.6	ng/L
STD (s):	2.70	ng/L
%RSD:	5.68%	
MDL:	8.11	ng/L
PQL:	40.5	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:		Date: 6-21-04
Gas Chromatography Analyst:	Cynthia Marello	Date: 6/21/04
QA/QC Officer:		Date: 6/21/04
Assistant Lab Director:	R.E. Wagner	Date: 6/21/04

Comments:

Northeast Analytical, Inc.

Method Detection Limits

File Name: Q:\MDL\PCB\Gc11_2004_608\[GC11_012004_1260_SEPFUN_.XLS]A

Date: 18-Jun-04

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound:	A1260	Analysis:	EPA METHOD 608
Matrix:	WATER	Instrument:	GC-11
Extraction:	SEP FUNNEL	Column:	DB-1
Spike conc:	49.6 ng/L		

	NEA Sample ID	Extraction Date	File Name	Analysis Date	Measured Concentration ng/L	Percent Recovery (%)
1	040120W1	01/20/04	040120W1R	06/17/04	43.1	87.0%
2	040120W2	01/20/04	040120W2R	06/17/04	45.3	91.3%
3	040120W3	01/20/04	040120W3R	06/17/04	41.4	83.4%
4	040120W4	01/20/04	040120W4R	06/17/04	42.0	84.7%
5	040120W5	01/20/04	040120W5R	06/18/04	43.2	87.1%
6	040120W6	01/20/04	040120W6R	06/18/04	41.6	83.9%
7	040120W7	01/20/04	040120W7R	06/18/04	42.5	85.7%
8	040120W8	01/20/04	040120W8R	06/18/04	39.6	79.8%

One sided Student's t values (t) at the 99% confidence level.

Number (n)	(t) value
7	3.143
8	2.998

Number (n):	8	
AVG:	42.3	ng/L
STD (s):	1.67	ng/L
%RSD:	3.94%	
MDL:	4.99	ng/L
PQL:	25.0	ng/L
VALID:	valid	

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Sample Preparation Chemist:		Date: 6-21-04
Gas Chromatography Analyst:	Anthony Morillo	Date: 6/21/04
QA/QC Officer:		Date: 6/21/04
Assistant Lab Director:	R.E. Wayne	Date: 6/21/04

Comments:

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

Via Federal Express and Electronic Mail

November 22, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Additional TS Sample Collection

Dear Sir or Madam:

On October 22, 2004, General Electric (GE) submitted Corrective Action Memorandum (CAM) No. 5 to the Treatability Studies (TS) Work Plan. CAM No. 5 proposed additional sediment and water samples collected for treatability testing. The additional treatability work will include evaluation of separation operations, such as additional testing with the hydrocyclone. Therefore, the total PCB concentration in the

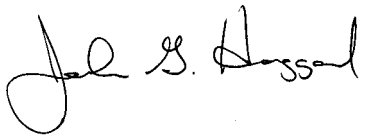
sediment must be greater than 50 mg/kg. The three treatability samples (S2-2, S3-2, S4B-2) have been collected and composited. Analytical samples were submitted for laboratory analysis on November 2, 2004.

The preliminary results for grain size indicate that the material collected represents the range of materials types that will be the focus of additional studies. Preliminary PCB analytical results indicated that PCB concentration for S2-2 and S4B-4 are relatively consistent with the S2 and S4 samples collected for the initial treatability studies. However, the preliminary PCB concentration in the S3-2 sample was an order of magnitude lower than the S3 sediment used for the initial studies and is therefore not useful for the additional size separation studies.

GE is proposing to collect additional S3 sediment at a different location in River Section 1 (sample designated S3-3) at the coordinates shown on Table 1 (attached). This location is a few hundred feet upriver from the most recent treatability sampling event (sample designated S3-2). The samples will be collected using a push coring technique with 3-inch Lexan® coring tubes. Additionally, the samples will cluster in the immediate vicinity of the target coordinates.

We anticipate initiation of sample collection during the beginning November 29. We therefore request your prompt review and approval of this additional sample collection, while access to the river is still available and weather conditions are acceptable. If you should have any questions, please contact Scott Blaha at (518) 862-2738 or Bob Gibson at (518) 862-2736.

Sincerely



John G. Haggard

Enclosure

Notes:
The S3-2 sample was collected based on CAM No. 5;
The S3-3 sample is proposed for collection during the week of 11/29/04.

Table 1 - Treatability Studies Sediment Sample Collection

Treatability Studies Sediment Sampling Locations (Sample ID)	Coordinates (New York State Plane East, NAD 83)								Maximum Collection Depth	Quantity of Sediment	
	NW Corner		SW Corner		SE Corner		NE Corner				
	Northing	Easting	Northing	Easting	Northing	Easting	Northing	Easting	(Feet)	(Gallons)	(Cubic Feet)
S3 - River Section 1 (S3-2)	1,607,785	732,221	1,607,690	732,221	1,607,690	732,312	1,607,785	732,312	3.8	100	13.4
S3 - River Section 1 (S3-3)	1,608,051	732,301	1,608,051	732,301	1,608,051	732,301	1,608,051	732,301	2.0	100	13.4

John G. Haggard, Manager
Hudson River Program

General Electric Company
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203
Fax: (518) 862-2731
Telephone: (518) 862-2739
Dial Comm: 8 232-2739*
E-Mail:John.Haggard@corporate.ge.com

Via Electronic Mail & Federal Express

December 10, 2004

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

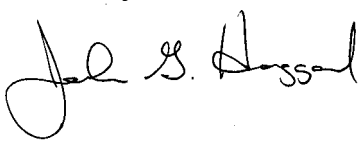
Re: Hudson River – Treatability Studies Work Plan - Corrective Action Memorandum No. 6 – Size Separation Studies

Dear Sir or Madam:

Attached please find Corrective Action Memorandum (CAM) No. 6 to the Treatability Studies (TS) Work Plan.

If you should have any questions, please feel free to give Scott Blaha a call at (518) 862-2738.

Sincerely,



John G. Haggard

JGH/bg

Attachments

cc: Ben Conetta
Scott Blaha
Bob Gibson
Don Sauda
Barbara Ippolito
Paul Doody

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT
TREATABILITY STUDIES PROGRAM

Date: December 9, 2004
Organization Name: Blasland, Bouck & Lee, Inc. (BBL)
Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description:

The plan for size separation testing was provided in the Treatability Studies Work Plan (TS Work Plan, BBL, February 2004) that was approved by USEPA on February 13, 2004. The objectives (DQO 4b.(1a) & 4c.(1a)) include performing size separation to evaluate different disposal options (e.g. non-TSCA). The size separation test plan was more fully described in Corrective Action Memos: CA003, CA004, and CA005. GE also responded to EPA comments regarding CA003 in a letter dated August 24, 2004.

Wet sieving and hydrocyclone testing were employed, as size separation tests, during the treatability study program. GE plans additional testing to acquire more data to evaluate the performance of size separation operations with respect to the DQOs and to provide a basis for the Intermediate Design. The separated fractions will be sampled and submitted to analytical laboratories for relevant assays. The products of these tests (i.e. separated sediment fractions) will be retained, and properly stored, at the treatability laboratory.

Reported To: Scott Blaha, GE

Corrective Action:

1) The SOP for wet sieving was provided in Appendix 6: Standard Operating Procedure- Size Separation Testing of the TS Work Plan. The SOP for this test has been revised, per the attachment.
2) The sediment samples used as feedstock for wet sieve testing, conducted to date, were stated in the TS Work Plan and CA004. For these additional tests proposed, some samples obtained previously and some recently obtained sediment samples, per CA005, will be sieved. The specific sediment designations are S2-2, S3-3, S3-4, and S4A and the preliminary characteristics of these sediments are given in Table 1.
3) The SOP for Hydrocyclone Performance Testing was provided with CA003 as Appendix 33: SOP-Hydrocyclone Performance Testing. The SOP for this test has been revised, per the attachment.
4) The sediment designated S4 was used in initial hydrocyclone tests conducted in August. The sediment samples to potentially be used as feedstock for additional hydrocyclone testing were stated in CA004 and CA005. Also, GE issued a letter (dated November 22, 2004) to report progress of sample collection described in CA005. In these additional tests proposed, samples S2-2 and S3-4 will

be fed, as a slurry, to the hydrocyclone test apparatus. Slurry concentrations of approximately 15% and 25% will be tested. The preliminary characteristics of these sediments are given in Table 1.

5) The tests will be started during the week of December 13, 2004 and analytical data will be received on or before January 30, 2005. Per the TS Work Plan, the program completion date was October 5, 2004 (90 days after the receipt of the initial samples at the treatability laboratory). GE has already requested an extension to this program date (letters dated September 20, 2004 and November 19, 2004), with the provision that these treatability studies will not influence the critical path schedule for delivery of the Intermediate Design Report (IDR), per Table 4 of the Remedial Design Work Plan (BBL, August 2003). The schedule for these proposed tests will meet also the provisional schedule described in these letters, and will not influence the critical path for the IDR.

Table 1: Preliminary Sediment Properties for Additional Size Separation Tests

Sample ID	**S2-2**	**S3-4[1]**	**S3-3**	**S4A**	**S3-2**	**S4B-2**
[PCB] mg/kg	73	148	156	100	13	351
Fines (<75 um)	20%	50%	26%	30%	34%	74%

Notes:

(1) S3-4 is a 60:40 composite of S3-2 and S4B-2, the data is the weighted average

(2) Data in this table is preliminary and has not been validated.

Reviewed and Implemented By: Don Sauda (BBL)

cc: Bob Gibson (GE)
Amy Toth (Waste Stream)

Attachments:
Appendix 6 (rev 2): SOP- Size Separation Testing
Appendix 33 (rev 1): SOP- Hydrocyclone Performance Testing

Standard Operating Procedure
Size Separation Testing

1. Scope and Application

This standard operating procedure (SOP) describes the procedures for size separation testing. These tests are part of treatability studies described in the Hudson River PCBs Superfund Site Treatability Studies Work Plan (TS Work Plan) (Blasland, Bouck & Lee, Inc. [BBL], 2004).

Hydraulically-transported sediments may be treated for preliminary removal of coarse (>75 microns [µm]) particulates. Removal of coarse/dense materials relieves loading of solids to dewatering facilities. It also offers the possibility of beneficial use of sand and coarse particles.

The size separation procedures described below are used to obtain a quantitative determination of the distribution of particle sizes in sediment. Size fractions are determined on a dry weight basis. Samples are passed through various sieve sizes using wet sieving procedures to ensure complete and accurate separation of attached particles.

2. Equipment List

- Sieve shaker, Gilson Model WV-2 or equivalent;
- Sieve brush;
- Drying pan and top;
- Drying oven;
- Analytical balance, mg accuracy;
- Sieves of various apertures;
- Scale;
- Pan, container, or bucket to retain fines;
- Squirt bottle;
- Distilled or deionized water; and
- Clock or stop watch.

3. Health and Safety Considerations

All work will be in accordance with Sevenson Environmental Services, Inc.'s Corporate Health and Safety Plan.

4. Size Separation Testing Procedure

4.1 Allow samples to warm to room temperature. Homogenize each sample mechanically.

4.2 Remove a representative aliquot (approximately 25 grams) and analyze for total solids content. This information can be used to estimate the dry weight of the aliquot used for particle size analysis. The efficiency of the entire analysis can then be evaluated by adding the dry weights of all sample fractions and comparing this sum with the estimated dry weight of the original aliquot.

4.3 Remove a second representative aliquot of about 300 grams for wet sieving. Greater sample sizes can be used if necessary; however, large samples will not go through the series of screens as efficiently as a smaller sample.

4.4 Add the sample to a Gilson Model WV-2 Wet-Vac Sieve Tester. This device has a rotary spray bar above the top sieve to wash water from a recirculation pump system down through a stack of screens as they are vibrating. The following US Series sieve sizes are anticipated to be used during the test: ¾-inch, 3/8-inch, #4, #10, #20, #40, #60, #80, #100, #200, and #400. Select sieves may not be used. Empty the individual sieves and repeat as necessary to generate sufficient volume for analytical testing. Collect a representative sample of the <#400 sieved material.

4.5 Submit samples from each sieve channels for the desired analyses. Some sieve channels may be combined for some or all of the analyses.

4.6 Following cleaning and drying of all sieves, determine a tare weight for each. Measure out a quantity of sediment to be used during the size separation test and submit a sample for % solids analysis. Use this data to determine the total dry weight of solids to be used in the size separation test.

4.7 Similar to step 4.4, add the sample to a Gilson Model WV-2 Wet-Vac Sieve Tester. The following US Series sieve sizes are anticipated to be used during the test: ¾-inch, 3/8-inch, #4, #10, #20, #40, #60, #80, #100, #200, and #400. Select sieves may not be used. When the sieving cycle is complete, place each sieve into a drying oven overnight. Alternatively, the contents of each sieve may be emptied into drying pan(s) with known tare weight(s). Use rinse water, as necessary, to make sure all solids are transferred to the drying pan(s). These drying pans will be placed into a drying oven overnight. The drying oven should be set to 100-105 ˚C. Cool the sample to room temperature. Weight the cooled sample to the nearest 0.1 mg.

4.8 Recover all the solids and wash water that contain the <#400 sieved material. Place the solids and wash water into a drying pan(s) with known tare weight(s). Use rinse water, as

necessary, to make sure all solids are transferred to the drying pan(s). Place the pan(s) into a drying oven overnight. The drying oven should be set to 100-105 ˚C. Cool the sample to room temperature. Weight the cooled sample to the nearest 0.1 mg.

4.9 Calculations

4.9.1 Calculate sample weight retained on each sieve following drying by subtracting the tare weight of the sieve (or drying pan) from the final weight of the sieve (or drying pan). Record this as weight retained on the specified sieve.

4.9.2 Calculate the percent weight retained on each sieve by dividing the weight retained by the initial dry sample weight, and multiplying by 100.

4.9.3 Calculate the percent weight passing each sieve by subtracting the sum of the weights of the material retained on each larger sieve from the initial dry sample weight. Divide that weight by the initial dry sample weight and multiply by 100.

4.9.4 Record raw data and calculated percentages in a data sheet such as that shown below.

Sample ID and Initial Dry Weight	Sieve #	Aperture (µm)	Tare (g)	Weight (g)	Weight Retained (g)	% Weight Retained	% Weight Passed
	10	2000					
	20	850					
	40	425					
	60	250					
	80	180					
	100	150					
	200	75					
	400	38					
	<400*	<38					

* Sample material passing the #400 sieve.

5. References

BBL. 2004. *Treatability Studies Work Plan*. Hudson River PCBs Superfund Site. Prepared for General Electric Company, Albany, NY.

Standard Operating Procedure
Hydrocyclone Performance Testing

1. Scope and Application

This standard operating procedure (SOP) lists the steps to be performed to insure safe and effective operation of the hydrocyclone. Hydrocyclone tests are performed to separate fractions of a material based on density and particle size. The physical and chemical properties of the separated fractions can be measured. An SOP for hydrocyclone performance testing was not included in the Treatability Studies Work Plan (TS Work Plan) (Blasland, Bouck & Lee, Inc. [BBL], 2004). However, this procedure has been developed to meet the density separation objective described in DQO 4b.(1a) and 4c.(1a) in Section 2.4.2 of the TS Work Plan and the Size Separation Testing SOP (Appendix 6 of the TS Work Plan).

2. Equipment List

The following materials, as required, will be available during this procedure:

- Hydrocyclone and fittings, including apexes and vortex finders;
- Calibrated pressure gauge with oil-filled dampener;
- Sample containers;
- Graduated containers to collect underflow and overflow streams;
- Analytical balance;
- Log sheet;
- Thermometer;
- Steel ruler; and
- Stopwatch or timer.

3. Health and Safety Considerations

All work will be in accordance with Sevenson Environmental Services, Inc.'s Corporate Health and Safety Plan. Should you have any question or concern about the sample or procedure, address this with your supervisor or health and/or safety officer prior to beginning work.

Hydrocyclones are to be assembled and raised into position in a safe manner using a hoist, if necessary. Inlet and overflow pipes and hoses should be securely affixed to their appropriate fitting so that they will not blow off under pressure. The pump should be started to generate the

lowest possible pressure. The pressure should be slowly increased to the operating pressure while observing any signs of leaking or hoses blowing off.

4. Procedure

4.1. Review the test work to be performed. Take care to note the test objectives, equipment to be used, sequence of testing, and the operating conditions (pressure, feed percent solids, etc.).

4.2. Hydrocyclone/Pump/Sump Setup and Operation

4.2.1. Open the drain valve on the feed sump.

4.2.2. Thoroughly flush the sump until the water is clear. Close the valve.

4.2.3. Obtain the appropriate drawings and parts lists. Assemble the hydrocyclone taking care to ensure that the correct fittings are installed. Measure the apex and vortex finder diameters if old or unmarked parts are used.

4.2.4. If a hydrocyclone is already pre-assembled, disassemble to make sure that no internal parts are damaged, the correct inlet head liner is installed and is fitted properly, and that no residual solids are present in any of the internal crevices. It is especially important to remove the apex housing to insure that no residual material is to be found in that area.

4.2.5. When assembled, check to be sure that no reverse shelf exists. A piece of wire with a bend on the end can be used to feel for the shelf. If one is found, disassemble the hydrocyclone and readjust.

4.2.6. Install the hydrocyclone over the sump. Install the overflow pipe or hose.

4.2.7. Install the calibrated pressure gauge with its accompanying oil filled pulsation dampener as close to the inlet as possible. (See “Gauge and Pulsation Dampener Guidelines” presented below).

4.2.8. Screen the sediment to be used in the test being conducted through a ¼-inch screen. Retain the >¼-inch material in a separate container. Mix the appropriate quantities of water and sediment from the Hudson River to meet the desired % solids slurry for the test being conducted.

4.2.9. Fill the sump with slurry to the designated volume (about 50 gallons). Additional slurry should be premixed and added to the sump following each test to replace the volume of slurry removed by sampling.

4.2.10. Fully open the hydrocyclone bypass valve and valve off flow to/from the hydrocyclone.

4.2.11. Start the pump and check for leaks.

4.2.12. Make sure sump is mixed thoroughly. A feed sample can be collected from the bypass line.

4.2.13. Fully open all valves to/from the hydrocyclone. Adjust the bypass valve to give the target operating pressure. Do not use valves to/from the hydrocyclone to adjust pressure. Observe the flow out of the apex. If roping occurs, adjust the apex to be larger, if adjustment is possible. If the underflow is too dilute, adjust the apex to be smaller, if adjustment is possible.

4.2.14. Take grab samples of the underflow and overflow and observe the differences.

4.2.15. When the operating conditions and the underflow and overflow are deemed acceptable, collect feed, underflow and overflow samples. All samples must be taken simultaneously. Sample full stream where possible using a sample bottle to sample the feed and underflow, and a larger sample container to sample the overflow. Where underflow and overflow flow rates are large, sample cutters should be used simultaneously or two pails can be used if larger samples are required.

4.2.16. Recheck the feed pressure and perform the capacity determination as described below. Place the appropriate calibrated collection vessel under the apex and time the underflow collection until the vessel reaches the target volume. Return this to the sump. Direct the overflow into a larger calibrated vessel and time the overflow collection until the vessel reaches the target volume. Return the fluid to the sump. Determine the hydrocyclone flow rate at the pressure used. Immediately check the published capacity curve to be certain that this experimentally determined capacity is in agreement with the curve. If it is not, perform the capacity determination again. If the two numbers still do not agree, check the feed pressure gauge (see below). Check the temperature of the slurry in the sump.

4.2.17. Record all data collected during the test on a log sheet.

4.2.18. When no additional tests are going to run using the slurry in the test unit, the overflow should be directed to a 55-gallon plastic drum while the underflow is collected in an appropriately sized container. The feed to the hydrocyclone should be continued until the pump is no longer maintaining the desired feed pressure. The pump should be shut down and all remaining slurry in the sump should be cleaned out as waste. The underflow and overflow should be retained.

4.3. Gauge and Pulsation Dampener Guidelines

The measuring devices used in hydrocyclone testing are the stopwatch, calibrated collection vessel, and the pressure gauge with its associated pulsation dampener. The

pressure gauge is the most important because the pressure drop across the hydrocyclone affects both performance and capacity. Therefore the pressure gauge must be accurate.

4.3.1. Obtain a pressure gauge known to be accurate to 5% or better. The gauge should be such that the desired working pressure is within the middle range of the gauge.

4.3.2. Mount a flush valve on the appropriate port in the pulsation dampener.

4.3.3. Fill the pulsation dampener with the appropriate oil making sure no air is present in the pulsation dampener.

4.3.4. Attach the gauge plus dampener to the hydrocyclone inlet. Make sure there are no leaks.

4.3.5. If necessary, flush the dampener using a water line.

4.3.6. Observe normal gauge operation.

4.3.7. If any of the following occurs, the gauge must be removed, inspected, checked or recalibrated, and the pulsation dampener and mounting nipple must be flushed:

- Erratic needle movement;
- Gauge does not return to 0 psi with no pressure on it;
- Gauge pressure increase slowly or not at all as pump speed increases;
- Blown gauge; and
- Calculated hydrocyclone capacity does not correspond to published capacity.

5. References

BBL. 2004. *Treatability Studies Work Plan*. Hudson River PCBs Superfund Site. Prepared for General Electric Company, Albany, New York.

Krebs Engineers. 1999. *Hydrocyclone Performance Testing*.

John G. Haggard
Manager, Hudson River Program

GE
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203

T 518 862 2739
F 518 862 2731
John.Haggard@ge.com

VIA FEDERAL EXPRESS AND ELECTRONIC MAIL

January 25, 2005

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

Re: Treatability Notification

Dear Sir or Madam:

In a November 19, 2004 letter, General Electric (GE) proposed to mix Hudson sediment and river water, then clarify and decant the water to generate feedstock for the rapid small-scale column tests (RSSCTs). The United States Environmental Protection Agency (EPA) verbally approved this plan on November 24, 2004. In the meantime, GE has procured materials and began preparing feedstock.

The treatability study laboratory has performed jar tests to validate the method proposed to generate a representative feedstock. The jar tests have confirmed that a representative feedstock can be prepared and the method is reproducible. This preparatory work has added approximately two

weeks to the test schedule; however, this additional time has reduced the risk of quality issues or delays during the RSSCT test.

Water will be run through the RSSCT columns for approximately 23 days, beginning January 25, 2005. A contingency of 14 days is planned if breakthrough is not detected after the first 23 days of operation. Therefore, the RSSCT will be completed on or before March 3, 2005.

GE believes an extension for the RSCCTs is justified because:

- The RSSCT studies are not on the project critical path; therefore, extending the date of completion will not delay delivery of the Phase I Intermediate Design Report; and
- No other treatability tests are linked to the completion of the RSSCT tests.

Therefore, in accordance with Paragraph 47.c of the Administrative Order on Consent for Remedial Design, GE requests that the date for completion of the treatability studies be revised to March 3, 2005.

If you have any questions, please contact Scott Blaha (518) 862-2738.

Sincerely,

John G. Haggard
Manager, Hudson River Program

John G. Haggard
Manager, Hudson River Program

GE
320 Great Oaks Office Park, Ste: 319
Albany, NY 12203

T 518 862 2739
F 518 862 2731
John.Haggard@ge.com

Via Federal Express

April 20, 2005

Team Leader, Hudson River Team
Emergency and Remedial Response Division
United States Environmental Protection Agency, Region 2
290 Broadway, 19th Floor
New York, New York 10007-1866
Attn: Douglas Garbarini, Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Chief, New York/Caribbean Superfund Branch
Office of Regional Counsel
United States Environmental Protection Agency, Region 2
290 Broadway, 17th Floor
New York, New York 10007-1866
Attn: Hudson River PCBs Superfund Site Attorney (1 copy)

Director, Division of Environmental Remediation
New York State Department of Environmental Conservation
625 Broadway, 12th Floor
Albany, New York 12233-7011
Attn: Hudson River PCBs Superfund Site (3 copies – 1 unbound)

Director, Bureau of Environmental Exposure Investigation
New York State Department of Health
547 River Street
Troy, New York 12180
Attn: Hudson River PCBs Superfund Site (2 copies)

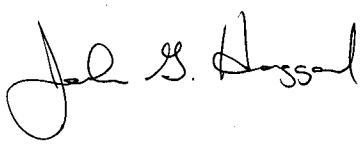
Re: Hudson River Treatability Study Work Plan- Corrective Action Memorandum No. 7

Dear Sir or Madam:

A Corrective Action Memorandum (CAM007) for the Treatability Study Work Plan is attached. This CAM proposes additional bench-scale filter press test simulations. We request your prompt approval of this plan so that we can begin the testing. These tests will not extend the schedule for Phase 1 Intermediate Design.

If you have any comments or questions, please contact Scott Blaha at (518) 862-2738.

Sincerely,



John G. Haggard
Manager, Hudson River Program

JHG/bg

Attachment

Cc: Ben Conetta
Scott Blaha
Bob Gibson
Don Sauda
Paul Doody
Barbara Ippolito

GENERAL ELECTRIC COMPANY
HUDSON RIVER DESIGN SUPPORT
TREATABILITY STUDIES PROGRAM

Date: April 19, 2005
Organization Name: Blasland, Bouck & Lee, Inc. (BBL)
Initiator's Name and Title: Donald Sauda, Treatability Studies Task Manager

Problem Description:

The plan for bench-scale filter press testing was provided in the Treatability Studies Work Plan (TS Work Plan, BBL, February 2004) that was approved by USEPA on February 13, 2004. The standard operating procedure (SOP) for this test is given in Appendix 12 of the TS Work Plan. In addition to the TS Work Plan, Corrective Action Memo (CAM) CA002 and CA004 further described bench-scale filter press testing.

Plans for hydrocyclone performance testing were provide in CAM CA006. During these tests, which were conducted in December 2004, samples of the hydrocyclone overflow were collected. Three of these samples will be used for bench-scale filter press testing.

Reported To: Scott Blaha, GE

Corrective Action:

The settled solids of hydrocyclone overflow will be conditioned with polymer and fed to the bench-scale filter press apparatus. The three samples are designated S2-2-HC-15-2-OF, S3-4-HC-25-2-OF, and S3-4-HC-15-1-OF. Several tests will be conducted with different polymer types and dosages.

Water content (Standard Methods [SM] 2540G) will be measured on the feed and filter cake.

The tests will be started on April 20, 2005. The tests, including receipt of analytical data, will be completed by May 6, 2005. This schedule will not influence the critical path schedule for delivery of the Intermediate Design Report (IDR), per Table 4 of the Remedial Design Work Plan (BBL, August 2003).

Reviewed and Implemented By: Don Sauda (BBL)

cc: Scott Blaha (GE)
Amy Toth (Waste Stream)