Superfund Records Center
SITE: Centredale
BREAK: 3.04
OTHER: 48783

QUALITY ASSURANCE PROJECT PLAN - ADDENDUM 2

Allendale Pond And Lymansville Pond Sediment Collection Study
Centredale Manor Restoration Project Site
North Providence, Rhode Island

EPA Addendum to:
Battelle Tasks 19-22 QAPP – ADDENDUM (9/27/02)
In Support of Centredale RI/FS

Submitted By:

ESAT – Region I
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04/14/03

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ADDENDUM 2 - NOTICE

This QAPP Addendum 2 is based on Battelle Centredale Manor Tasks 19-22 QAPP – Addendum (9/27/02). All QAPP elements for this project are detailed in this document.

1.0 TITLE AND APPROVAL PAGE (EPA WORKSHEET #1)

Site Name/Project Name: *Centredale Manor Restoration Project Superfund Site/Allendale Pond and Lymansville Pond Sediment Collection Study*
Site Location: *Allendale Pond and Lymansville Pond, North Providence, Rhode Island*

Document Title: *Centredale Manor Tasks 19--22 Quality Assurance Project Plan -- Addendum 2*

Lead Organization (Agency, State, Tribe, Federal Facility, PRP, or Grantee): *EPA Region I*

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Preparation Date (Day/Month/Year): 4/01/03

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Signature/Date
Andy Beliveau/EPA Region I
Printed Name/Organization

Lead Organization's Project Manager: 5/6/03
Signature/Date
Alan Humphrey/EPA ERTC/REAC
Printed Name/Organization

Approval Signature: 5/13/03
Signature/Date
Maureen Corcoran/Geomorphologist
Printed Name/Title
USACE WES
Approval Authority

Other Approval Signatures: 5/20/03
Signature/Date
for Laureen Borochaner/USACE NAE Project Officer
Printed Name/Title

Document Control Number: *Not applicable.*

2.0 CONTENTS AND DOCUMENTATION FORMAT

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NA - Not applicable; NI - Not Included, because of the volume of discussion.

ATTACHMENTS

(Attachment A through G applicable to the May 23, 2001 QAPP)
(Attachment H through K applicable to the September 27, 2002 QAPP)

L Pertinent Fixed Laboratory SOPs ..... not paginated
M Pertinent Field Sampling SOPs ..... not paginated
N Teledyne Brown Engineering Resume Summaries ..... not paginated

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This is a Project-Specific Quality Assurance Project Plan.

EPA-NE QAPP Worksheet #3 - Rev. 04/03

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Document Control Number
Laureen Borochaner	Project Manager	U.S. Army Corps of Engineers, New England District	978-318-8802	NA
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June Mirecki	Geochemist	U.S. Army Corps of Engineers WES	601-634-4003	NA
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Mark Guilmain	Project QA Officer	Battelle Duxbury	781-952-5316	NA
Deirdre Dahlen	Sample Analysis Task Manager	Battelle Duxbury	781-952-5253	NA
Karen Tracy	TCDD Task Leader	Battelle Columbus	614-424-4028	NA
Mary Schrock	TCDD Lab Manager	Battelle Columbus	614-424-4976	NA
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Linda Bingler	Metals and MeHg Task Leader	Battelle Marine Sciences Laboratory (MSL)	360-681-3627	NA
Deborah Coffey	QA Officer	Battelle MSL	360-681-3645	NA
Ken Davis	Grain Size and TOC Task Leader	Applied Marine Sciences, Inc.	281-554-7272	NA
Jennifer Davis	QA Manager	Applied Marine Sciences, Inc.	281-554-7272	NA
Alan Humphrey	Sampling Project Officer	EPA ERTC/REAC	732-321-6748	NA
Mark Sprenger	Project Officer	EPA ERTC/REAC	732-906-6826	NA
Rebecca Charles	Pb/Cs Project Manager	Teledyne Brown Engineering Environmental Services	865-934-0379	NA

EPA-NE QAPP Worksheet #4 - Rev. 04/03

Project Personnel Sign-Off Sheet

Organization: Teledyne Brown Engineering -- Environmental Services

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read	QAPP Acceptable as Written
Rebecca Charles	Project Manager, Radionuclide Analysis	865-934-0379			
Marty Keller	QA Manager	865-934-0371			
Pat Marshall	Sample Custodian	865-934- 0382			
Marty Webb	Production Manager	865-934-0375			
Lauren Larson	Gamma Spectroscopist	865-934-0390			

EPA-NE QAPP Worksheet #5a - Rev. 04/03

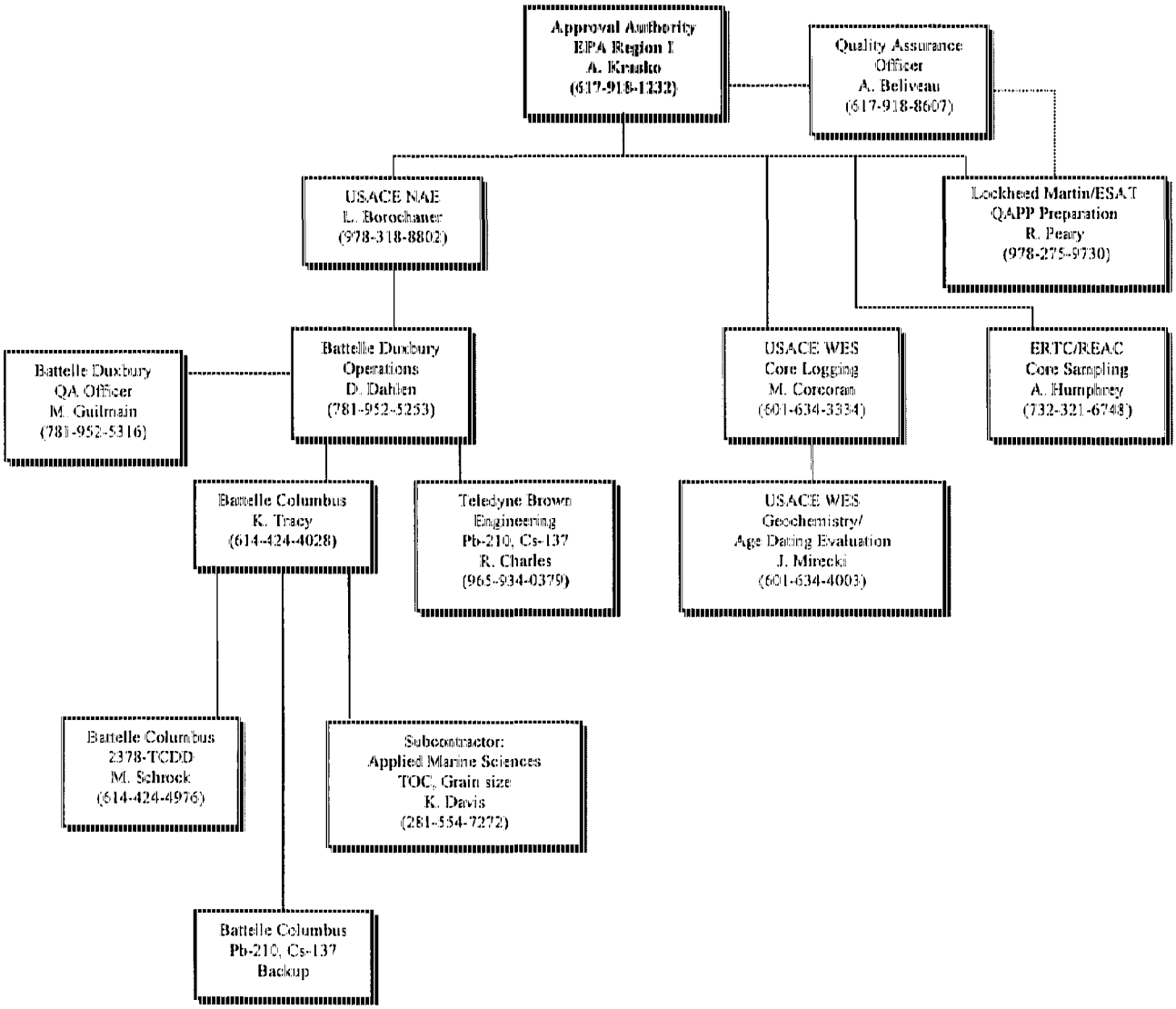

Figure 1. Project Organizational Chart.

EPA-NE QAPP Worksheet #5b - Rev. 04/03

Communication Pathways

Communication pathways will follow the project organization chart (Figure 1). Ms. Anna Krasko is the US EPA Project Manager. Mr. Andy Beliveau is responsible for the overall quality and conduct of the project. Ms. Anna Krasko will be the primary contact with the USACE NAE, USACE WES, and ERTC/REAC Project Managers.

Mr. Mark Guilmain will serve as Battelle's Quality Assurance (QA) Officer, and is responsible for identifying areas for corrective action, coordinating the QA activities such as systems and data audits, and preparing reports to management for this project. He will be assisted by the QA Officers/Managers at each of the participating laboratories.

As indicated in Figure 1, Task Leaders have been assigned for each of the major project tasks. Mr. Alan Humphrey of ERTC/REAC is responsible for core sampling. Ms. Maureen Corcoran of USACE WES is responsible for the core sub-sampling. Ms. Deirdre Dahlen of Battelle Duxbury is responsible for sample analyses. The Task Leaders will serve as the point of contact and will direct task activities and monitor task performance to ensure adherence to technical standards, budget, and schedule. They also will be responsible for apprising Ms. Anna Krasko of progress and notifying her of any significant problems or delays.

The need for corrective action may be identified during analysis, during QA reviews, or during management reviews. EPA Worksheets #21 and #24a define the corrective action(s) options if quality control data and calibration exceedences are identified. Battelle SOP 4-035 describes Battelle Duxbury's formal Corrective Action program. The Battelle QA Officer follows up all internal corrective action.

All communications will be conducted using electronic mail, phone, telefaxes, and/or reports.

EPA-NE QAPP Worksheet #6 - Rev. 04/03

Personnel Responsibilities and Qualifications Table

Name (a)	Organizational Affiliation	Responsibilities	Location of Personnel Resumes, if not included	Education and Experience Qualifications
Maureen Corcoran	U.S. Army Corps of Engineers Waterways Experiment Station (WES)	Core logging and core sub-sampling	NA	NA
June Mirecki	U.S. Army Corps of Engineers WES	Core geochemical and dating analyses	NA	NA
William Nelson	EPA Atlantic Ecology Division (AED)	Coordinate AED activities	NA	NA
Barbara Bergen	EPA AED	Coordinate AED core transport	NA	NA
Alan Humphrey	EPA ERTC/REAC	Project Officer for vibracoring	NA	NA
Mark Sprenger	EPA ERTC/REAC	Project Officer	NA	NA
Rebecca Charles	Teledyne Brown Engineering Environmental Services (TBE-ES)	Project Manager Radionuclide Analysis	NA	NA
Marty Keller	TBE-ES	QA Manager	NA	NA
Pat Marshall	TBE-ES	Sample Custodian	NA	NA
Marty Webb	TBE-ES	Production Manager	NA	NA
Lauren Larson	TBE-ES	Gamma Spectroscopist	NA	NA

(a) If the individual identified here is not available at the time of project commencement, then alternate staff -- with comparable training -- will perform project work.

EPA-NE QAPP Worksheet #8b - Rev. 04/03

Problem Definition/Site History and Background

QAPP Addendum 2

This QAPP Addendum 2 is based on Battelle Centredale Manor Tasks 19-22 QAPP -- Addendum (09/27/02). All QAPP elements for this project are detailed in this document.

General Objectives

The purpose of this study is to collect sediment samples from selected locations in Allendale Pond and Lymansville Pond (Figure 3). Samples will be analyzed for a small suite of parameters, identified in EPA-NE QAPP Worksheet #9a, in support of the Remedial Investigation (RI)/Feasibility Study (FS). The objective is to determine the nature and extent of contamination of the pond sediments by locating the layer of sediment corresponding to the time when the largest amounts of dioxin entered the river. An additional objective is to determine which layers of sediment do not contain significant amounts of dioxin. The information will also be used to determine which layers need to be removed, if necessary, and what are the physical characteristics of the layers to be removed.

The project will consist of sampling in both the Allendale Pond and the Lymansville Pond. The samples will be analyzed for Pb-210, Cs-137, 2378-TCDD, TOC, and grain size (see figure 2). A second round of sampling (Phase II) may be undertaken if it is determined that additional data are required based on the results from the initial phase of testing.

Data Quality Objectives

The Overall DQO for this project is to generate data of sufficient quality to define where the dioxin contamination is located in sediments laterally and vertically so that decisions can be made about the remedial alternatives that would be undertaken in the clean up of the site.

The DQO for sediment coring, slicing, and age dating is to generate data of sufficient quality to locate where in the sediment strata the dioxin was deposited.

The DQO for sediment dating is to generate data of sufficient quality to define the sedimentation rates in the Allendale and Lymansville Ponds.

The DQO for TOC/grain size is to generate data of sufficient quality to support the decision of how sediments will be physically remediated.

The DQO for 2,3,7,8-TCDD analysis of sediment core slices is to determine the total amounts of sediment that may need to be removed/remediated to meet human health risk based limits for the site.

EPA-NE QAPP Worksheet #8b – Rev. 04/03 (continued)

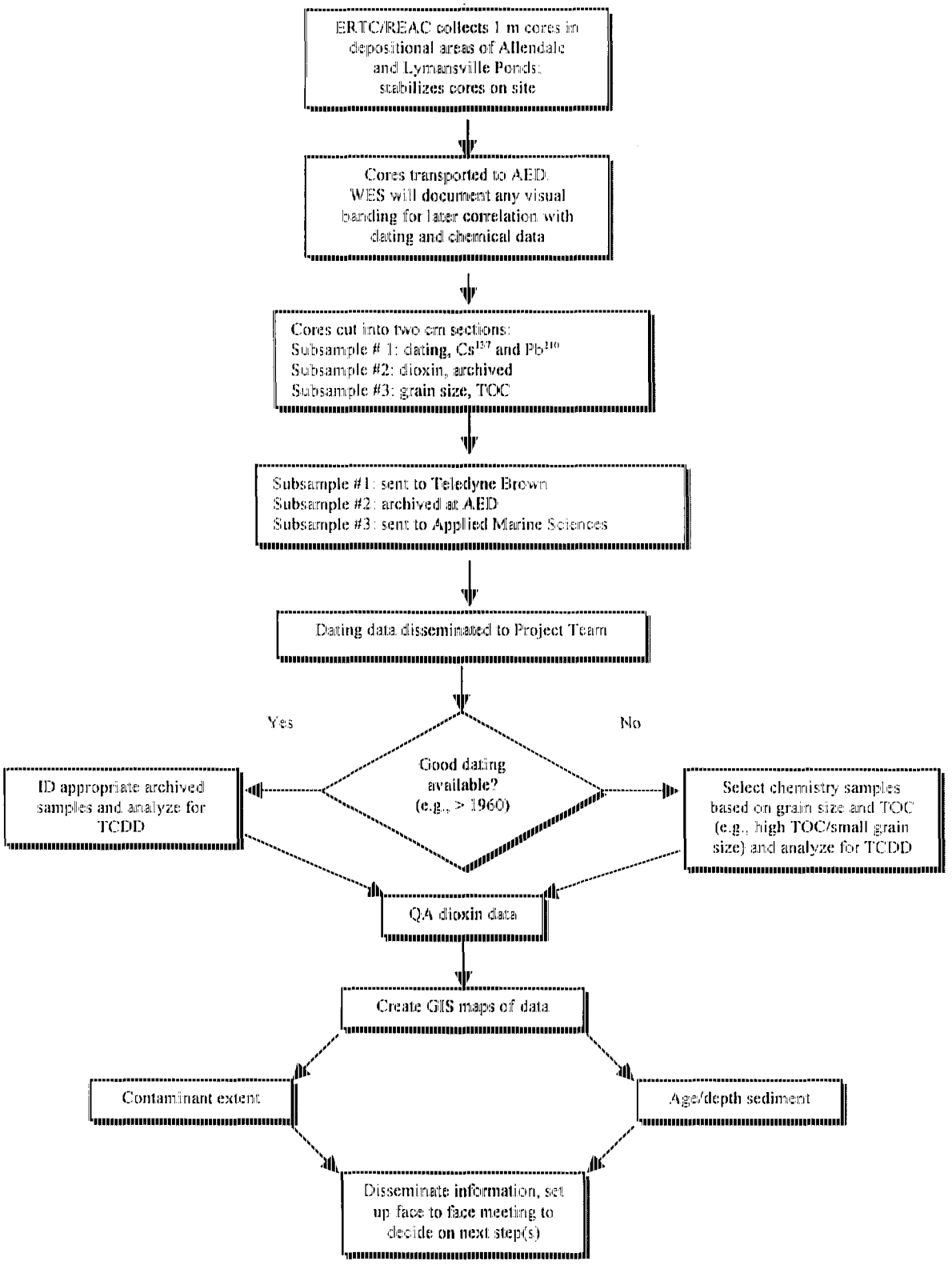

Figure 2. Logic Diagram for Sampling, Sub-sampling, Aging, and Analysis.

EPA-NE QAPP Worksheet #8b – Rev. 04/03 (continued)

Problem Definition/Site History and Background

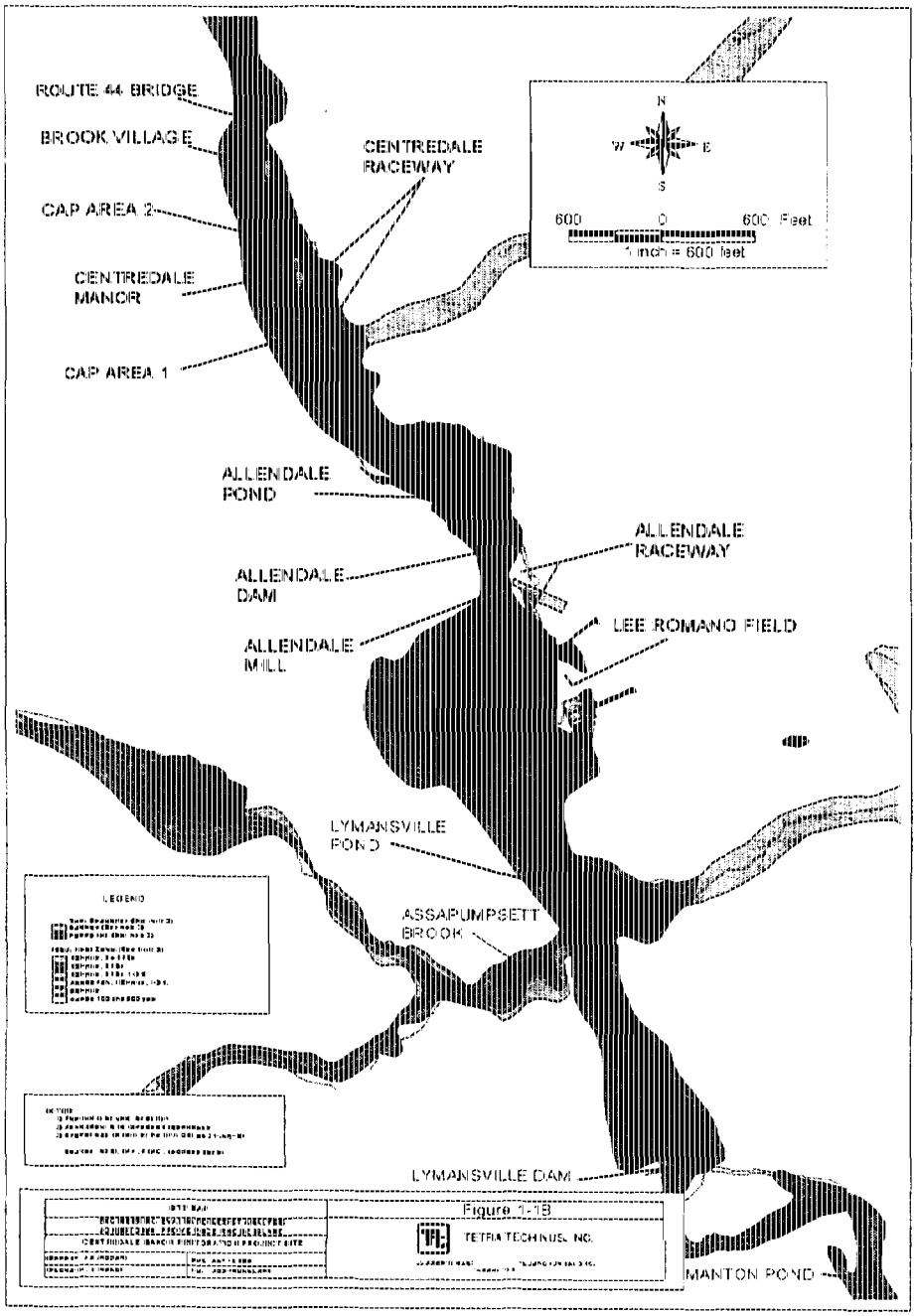

Figure 3. Allendale and Lymansville Ponds.

EPA-NE QAPP Worksheet #9a - Rev. 04/03

Project Description and Schedule

Sampling Tasks:
Samples will be collected from a Dioxin superfund site. Unused samples are regulated for F-listed waste and should be shipped back to Battelle Duxbury.

Field sampling activities, including sampling locations and collection techniques, are discussed in EPA-NE QAPP Worksheet #12. Briefly, sediment samples from multiple sampling locations located in Allendale Pond and Lymansville Pond (Figure 3) will be collected for physical and chemical testing (Table 1). Additional details regarding sample collection are provided in EPA-NE QAPP Worksheets #9c and #12b.

Table 1. Sampling Locations, Numbers of Samples, Required Analytical Parameters, and Performing Laboratories

Sample Matrix	Sampling Locations	Total Number of Samples (a)	Analysis Parameters	Performing Laboratory
Sediment - Cores - Allendale Pond	Approx. 20	60 (3 each of 20 cores)	Pb-210 and Cs-137 Dating	Teledyne Brown Engineering Environmental Services
		40 (2 each of 20 cores)	2,3,7,8-TCDD	Battelle Columbus
		20 (1 each of 20 cores)	Grain size, TOC	Applied Marine Sciences, Inc.
		60 (3 each of 20 cores)	Archival	U.S. EPA AED
Sediment - Cores - Lymansville Pond	Approx. 10	40 (4 each of 10 cores)	Pb-210 and Cs-137 Dating	Teledyne Brown Engineering Environmental Services
		20 (2 each of 10 cores)	2,3,7,8-TCDD	Battelle Columbus
		10 (1 each of 10 cores)	Grain size, TOC	Applied Marine Sciences, Inc.
		40 (4 each of 10 cores)	Archival	U.S. EPA AED

(a) Not including field QC.

EPA-NE QAPP Worksheet #9a - Rev. 04/03 (continued)

Project Description and Schedule

Sample Storage and Holding Times for Chemical Analyses:
All samples will be shipped by overnight carrier, on ice, from the field to participating laboratories. Upon arrival at the laboratory, samples will be logged into laboratory's sample tracking system and the laboratory will maintain possession of the original sample custody logs that accompany the samples. Samples will be prepared for physical and chemical testing within specified holding times (Table 2).

Table 2. Sample Type, Storage and Holding Time Requirements for Chemical Parameters

Sample Type	Sample Shipping Conditions (from Field to Labs)	Laboratory Storage Conditions	Holding Times
PE Samples (a)	Ambient	Follow directions received with PE samples	Follow directions received with PE samples
Sediments	Cold (<6 °C)	Frozen (below –10 °C)	2378-TCDD: 1 year (c)
		Refrigerated (at approximately 4±2 °C)	Grains size, TOC: 28 d (b)
		Refrigerated (at approximately 4±2 °C)	Pb-210, Cs-137: not determined

(a) PE samples will be shipped to participating laboratories for those parameters where there is no available SRM, but there is an available PE sample.
(b) EPA, 1992. EMAP Estuaries 1992 Virginian Province Quality Assurance Project Plan.
(c) Per EPA Method 1613, Rev.B for dioxin/furan, aqueous and solid samples may be stored for up to one year if stored as described in the method.

Sample Disposition – As noted previously, samples will be collected from a Dioxin superfund site and unused sample are regulated for F-listed waste. As a result, rather than archiving samples for 6-months past delivery of final data (standard procedure at Battelle), unexpended samples will be returned to Battelle Duxbury upon completion of the sample analysis task (*i.e.*, final data through internal QA).

EPA-NE QAPP Worksheet #9a - Rev. 04/03 (continued)
Project Description and Schedule

Sediment Analysis Tasks:
Sediments will be tested for 2,3,7,8-TCDD, Pb-210, Cs-137, TOC, and grain size. Both Allendale Pond and Lymansville Pond will be sampled (Table 1). Definitive data will be produced for each analytical task.

A second round (phase II) of sampling from both ponds for the same parameters may be necessary and will be determined by the quality and extent of data obtained from phase I.

All analytical tasks will be performed in a fixed laboratory following standard operating procedures (SOPs). SOPs were provided previously (Battelle, 2001)[1]. SOPs for new analytical parameters and/or laboratories that did not participate in fall 2002 sampling/analysis (e.g., Teledyne Brown) are provided with this QAPP Addendum. General descriptions of analytical methods are described below.

2,3,7,8,-TCDD Only

Sediment samples (Table 1) will be extracted and analyzed for the PCDD congener 2,3,7,8-TCDD only, following the general procedures in EPA Method 1613, Revision B, as described in Battelle Columbus SOPs ASAT.II-001-02 and ASAT.II-002-02 with modifications noted below.

Approximately 1- 10 g (wet weight) of each soil will be spiked with an isotopically labeled analog of 2,3,7,8-TCDD. Samples will be extracted with methylene chloride by an Accelerated Solvent Extraction (ASE) procedure (SOP ASAT.II-009-00). If a sample, under visual observation and high percent moisture, shows that ASE is not applicable, then a 16 hour minimum soxhlet extraction will be used. The entire extract will be put through cleanup (acid and base partitioning) for this analyte.

All extracts for 2,3,7,8-TCDD analysis will be spiked with $^{37}Cl_4$-2,3,7,8-TCDD cleanup standard, partitioned against acid solutions, and processed through acid/base silica, alumina, and carbon Celite columns. Extracts will be spiked with $^{13}C_{12}$-1,2,3,4-TCDD recovery standard and concentrated to a final volume of 20 µL.

Sample extracts will be analyzed by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) in the selected ion monitoring mode (SIM) at a resolution of approximately 10,000. Analysis for 2,3,7,8-TCDD will be on a DB-5 or equivalent column. The analyte will be quantified by isotope dilution or by the method of internal standards.

[1] Battelle 2001. Tasks 19-22 QAPP Field Sampling, Chemical and Toxicity Testing. May 23, 2001. 509 pgs + app.

EPA-NE QAPP Worksheet #9a - Rev. 04/03 (continued)
Project Description and Schedule

Pb-210 Age Dating

The Pb-210 activity of soils and sediments is determined radiochemically by separating the daughter product Bi-210 and assaying its beta activity. The method measures the Pb-210 fraction from which Bi-210 may be dissolved by leaching with hot hydrochloric acid (activity in the interior of mineral grains may be excluded).

Stable lead and bismuth carriers are added to the dried sample and it is leached with hot 6N hydrochloric acid. The sample is then filtered and the filtrate is evaporated, oxidized with nitric acid, and finally dissolved in 1.8N hydrochloric acid. The solution is passed through an ion exchange column. Lead is eluted first with 9N hydrochloric acid and with deionized water, then, bismuth is eluted with 2N sulfuric acid. The bismuth is precipitated as the oxychloride and is collected by vacuum filtration on a 1-inch glass fiber disc. The bismuth yield is determined gravimetrically. The filter disc is mounted on a nylon planchet and covered with 3 mg/cm^2 aluminum absorber for beta assay in a low level, gas-flow proportional counter. The Pb-210 concentration is then calculated and reported in pCi/g.

Cs-137 Age Dating

The gamma-ray spectrometers are calibrated with mixed gamma-ray standards (NIST traceable where available). The efficiency versus energy curve is determined on each germanium detector for each of the standard sample geometries.

An aliquot of sample is placed into a standard geometry (150 ml or 300 ml plastic bottle, or 1-L Marinelli container for soil/sediment samples). The standard geometry containing the sample is counted using a shielded Ge(Li) gamma-ray detector. The radionuclide concentration (Cs-137 only, for this project) is then calculated and reported in pCi/g.

Documentation and Records:
The core samples will be shipped under Chain-of-Custody from the sampling site to EPA AED, Narragansett. The core samples will be logged immediately after sampling and delivery to EPA AED, and the results will be documented in the core log by USACE WES. Sub-sampling of the cores will be done by USACE WES and will also be recorded in the core log. Sub-samples will be shipped to the laboratories for the appropriate analyses under additional Chain-of-Custody. For purposes of documentation, a copy of the original Chain-of-Custody from the core samples should accompany any additional sub-sample Chain-of-Custody. All archived core sub-samples will remain in the custody of EPA AED until sent for later analysis (TCDD or other, as needed) or until disposal.

EPA-NE QAPP Worksheet #9b - Rev. 04/03

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

Medium/Matrix: Sediment
Region I Matrix Code (from EPA-NE DQO Summary Form): SE
Analytical Parameter: 2,3,7,8-TCDD
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP[1]: L-23

Analyte	CAS Number	Project Action HH Goal* (pg/g dry weight)	Project Action ECO Goal* (pg/g dry weight)	Analytical Method[1]		Achievable Laboratory Limits (DRY WT – pg/g)	
				MDLs	Method QLs[2]	MDLs[3]	RLs[3]
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	0.0684	0.008	NA	2	1.0	1.0
Total TCDD	41903-57-5	As above	As above	As above	NA	NA	NA
Actual data will be evaluated against the lowest calibration standard; see EPA-NE Worksheet #30. Values detected at a level below the lowest calibration standard will be J flagged; Non-detects will be reported as the EDL[4] and U flagged.							

NA – Not available/applicable.

[1] Method 1613B.
[2] Analytical method MDLs and RLs documented in validated methods. These limits are based on a sample size of 10 g wet weight and an average sample % dry weight of 50% to give a final sample size of 5 g dry weight.
[3] Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method. These limits are based on an average sample size of 5 g dry weight. The MDL values are from a seven replicate MDL study performed in 2002 (using Automated Solvent Extraction technique); and the RL values are based on the lowest calibration standard, sample size (10g wet weight and an average sample % dry weight of 50% to give a final sample size of 5 g dry weight, and extract volume 20µL).
[4] EDL is defined as Estimated Detection Limit, see Method 8290 for the method of calculating EDLs.

* Recommended detection limits used to support the human health (HH) biota consumption risk assessment and the baseline ecological (ECO) risk assessment. These values were provided by Harding ESE for perspective rather than as a requirement for the analytical methods. If detection limits cannot be achieved, this will be addressed in the uncertainty discussions in the risk assessment. Goals presented to three significant figures.

EPA-NE QAPP Worksheet #9b - Rev. 04/03 (continued)

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

Medium/Matrix: Sediment
Region I Matrix Code (from EPA-NE DQO Summary Form): SE
Analytical Parameter: Pb-210, Cs-137 (Age Dating)
Concentration Level: Low
Field Analytical or Fixed Laboratory Method/SOP: L-112/L-113

Analyte	CAS Number	Project Action HH Goal* (dry weight)	Project Action ECO Goal* (dry weight)	Analytical Method		Achievable Laboratory Limits (DRY WT-pCi/g)
				MDLs	Method QLs	MDC[1]
Pb-210	NA	NA	NA	NA	NA	0.1
Cs-137	As above	As above	As above	NA	NA	0.5 (0.2)[2]

NA – Not available/applicable.

[1] Minimum Detectable Concentration (MDCs) based on typical sample volumes, efficiencies and count times. Actual data will be evaluated against posteriori MDC. Achieved MDCs will be reported with the study data.

[2] Lower detection levels can be achieved with extended count times.

* Recommended detection limits used to support the human health (HH) biota consumption risk assessment and the baseline ecological (ECO) risk assessment. These values were provided by Harding ESE for perspective rather than as a requirement for the analytical methods. If detection limits cannot be achieved, this will be addressed in the uncertainty discussions in the risk assessment. Goals presented to three significant figures.

EPA-NE QAPP Worksheet #9c - Rev. 04/03

Field and Quality Control Sample Summary Table – Allendale Pond

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference[1]	No. of Sampling Locations	No. of Field Dup Pairs	Organic		Inorganic		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks	No. of SRM or PE Samples[2]	Total No. of Samples to Lab[3]
						No. of MS	No. of MSD	No. of MS	No. of MSD					
Sediment	2,3,7,8-TCDD	Low	1613B/L-23	20 Cores (2 each of 20 cores = 40 samples)	1	1 MS/MSD set per analytical batch [a]		0	0	0	0	0	1 SRM/PE per analytical batch [a]	44
Sediment	Pb-210	Low	TBE-032-30/L-112	20 Cores (3 each of 20 cores = 60 samples)	0	0	0	1 per batch	0	0	0	0	0	60
Sediment	Cs-137	Low	TBE-2008/ L-113	20 Cores (3 each of 20 cores = 60 samples)	0	0	0	0	0	0	0	0	0	60
Sediment	Grain Size	Low	ASTM D422/L-46	20 Cores (1 each of 20 cores = 20 samples)	1	0	0	0	0	0	0	0	0	21
Sediment	TOC	Low	9060/L-49	20 Cores (1 each of 20 cores = 20 samples)	1	0	0	0	0	0	0	0	1 SRM per analytical batch [a]	22

(See next page for footnotes.)

EPA-NE QAPP Worksheet #9c - Rev. 04/03 (continued)

Field and Quality Control Sample Summary Table -- Lymansville Pond

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference[1]	No. of Sampling Locations	No. of Field Dup Pairs	Organic		Inorganic		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks	No. of SRM or PE Samples[2]	Total No. of Samples to Lab[3]
						No. of MS	No. of MSD	No. of MS	No. of MSD					
Sediment	2,3,7,8-TCDD	Low	1613B/ L-23	10 Cores (2 each of 10 cores = 20 samples)	1	1 MS/MSD set per analytical batch [a]		0	0	0	0	0	1 SRM/PE per analytical batch [a]	22
Sediment	Pb-210	Low	TBE-032-30/L-112	10 Cores (4 each of 10 cores = 40 samples)	0	0	0	1 per batch	0	0	0	0	0	40
Sediment	Cs-137	Low	TBE-2008/ L-113	10 Cores (4 each of 10 cores = 40 samples)	0	0	0	0	0	0	0	0	0	40
Sediment	Grain Size	Low	ASTM D422/L-46	10 Cores (1 each of 10 cores = 10 samples)	1	0	0	0	0	0	0	0	0	11
Sediment	TOC	Low	9060/L-49	10 Cores (1 each of 10 cores = 10 samples)	1	0	0	0	0	0	0	0	1 SRM per analytical batch [a]	12

[1] Complete SOP references in EPA-NE QAPP Worksheet #20.

[2] SRM samples will not be collected in the field -- instead these are fixed laboratory analytical QC samples supplied by a certified agency and prepared in the laboratory to demonstrate comparability. In cases where an SRM does not or is not available for a specific analytical parameter/media -- then a PE sample will be provided by NAE and shipped to the participating laboratory for analysis with the study samples.

[3] Total number of samples to the lab only represents total numbers of samples **collected in the field and shipped to the participating lab**. This includes authentic (study) samples and field QC samples (*e.g.*, field duplicates, and extra water for preparation of MS/MSD). Total number of samples to the lab does **not** include fixed laboratory QC samples (*e.g.*, MS/MSD) for soil. In this case, aliquots of sample will be used from an authentic sample to prepare the fixed laboratory QC sample -- separate and distinct samples will not be collected to satisfy the fixed laboratory QC sample requirements. Fixed laboratory QC sample (*e.g.*, MS/MSD) types and frequency are defined in EPA-NE QAPP Worksheet #11b or 24.

[a] Analytical batch contains 20 or fewer field samples.

EPA-NE QAPP Worksheet #9d - Rev. 04/03

Analytical Services Table

Medium/ Matrix	Analytical Parameter	Concentration Level	Analytical Method/SOP[1]	Data Package Turnaround Time [2]	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/ Organization (Name and Address: Contact Person and Telephone Number)
Sediment	2,3,7,8,-TCDD (a)	Low	1613B/L-23	30-45 days	Karen Tracy Battelle Columbus 505 King Avenue Columbus, OH 43201 (614) 424-4028	Backup GC/HRMS system available at Battelle Columbus
Sediment	Pb-210	Low	TBE-032-30/ L-112	30 days	Rebecca Charles Teledyne Brown Engineering – Environmental Services 2508 Quality Lane Knoxville, TN 37931 (865) 934-0379	Battelle Columbus 505 King Avenue Columbus, OH 43201 (614) 424-4028
Sediment	Cs-137	Low	TBE-2008/ L-113	30 days	As above	As above
Sediment	Grain Size	Low	ASTM D422/ L-46	30 days	Ken Davis Applied Marine Sciences 502 North Highway 3 League City, TX 77573 (281) 554-7272	GeoPlan Associates Peter Rosen 30 Mann St. Hingham, MA 02043 (616) 740-1340
Sediment	TOC	Low	9060/L-49	30 days	As above	As above

[1] Specify appropriate reference number/letter from the Field Analytical Method/SOP Reference Table (EPA-NE QAPP Worksheet #17) and from the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).
[2] Data Package Turnaround Time may increase should large numbers of samples (e.g., >40) be received for analysis.
(a) Battelle Columbus will also determine moisture content in order to present soil data on a dry weight basis.

EPA-NE QAPP Worksheet #10 - Rev. 04/03

Project Schedule Timeline Table

Activities	Dates (MM/DD/YY) [a]		Deliverable	Deliverable Due Date
	Anticipated Date(s) of Initiation	Anticipated Date of Completion		
QAPP Addendum Preparation				
Draft	February 7, 2003	April 7, 2003	QAPP	April 7, 2003
USACE NAE and EPA Review	April 7, 2003	April 14, 2003		
Final	April 14, 2003	April 21, 2003		April 21, 2003
Field Sampling[1]				
Sediment Cores	May 5, 2003	May 9, 2003	N/A	N/A
Sample Analysis[1]				
Physical and Chemical Testing	May 10, 2003	July 25, 2003	Data Report	September 16, 2003
Third Party Validation (Tier II/III)	June 15, 2003	August 31, 2003		
Letter Data Report	August 1, 2003	September 15, 2003		

[1] Participating laboratories will be responsible for sample custody and data quality control through validation process.
[a] If due date falls on a weekend, then the deliverable will be submitted on the following Monday.
N/A Not applicable.

Project Schedule Timeline Table – Phase II

Activities	Dates (MM/DD/YY) [a]		Deliverable	Deliverable Due Date
	Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Field Sampling[1]				
Sediment Cores	To Be Determined (Fall 2003)	To Be Determined	N/A	N/A
Sample Analysis[1]				
Physical and Chemical Testing	To Be Determined	To Be Determined	Data Report	To Be Determined
Third Party Validation (Tier II/III)				
Letter Data Report				

[1] Participating laboratories will be responsible for sample custody and data quality control through validation process.
[a] If due date falls on a weekend, then the deliverable will be submitted on the following Monday.
N/A Not applicable.

EPA-NE QAPP Worksheet #11a - Rev. 04/03

Project Quality Objectives/Decision Statements

Project Quality Objectives:
The project quality objectives for physical and chemical testing is to generate data of a quality sufficient to be used for the RI/FS. The data needed for the RI/FS must be definitive data of very high quality (tight measurement performance criteria). Sensitivity requirements for selected parameters (e.g., grain size) have not been defined.

Project quality objectives and measurement performance criteria for physical and chemical testing are defined below.

Measurement Performance Criteria:
Method performance criteria (MPC) chosen to ensure that the definitive data will be of high quality are defined by analysis parameter in EPA-NE QAPP Worksheet #11b.

Data quality may be defined in terms of accuracy, precision, representativeness, completeness, comparability, and sensitivity.

- *Accuracy* is the agreement between an observed value and an accepted value. Analytical accuracy is monitored for analytical chemistry measurements as specified in EPA Worksheet #11b.

  Applicable samples: LCS, MS, MSD, SRM, SIS (each sample).
  Applicable analyses:
  ⇒ 2,3,7,8-TCDD
  ⇒ TOC (SRM only)
  ⇒ Pb-210 (LCS, MS only)
  ⇒ Cs-137 (LCS only)

- *Precision* is defined as the degree of reproducibility among individual measurements of the same property, obtained under similar conditions. Measures of analytical precision will be determined in all phases of the program.

  Applicable samples: MS/MSD, DUP.
  Applicable analyses:
  ⇒ 2,3,7,8-TCDD (no DUP)
  ⇒ Grain Size (DUP. only)
  ⇒ TOC (DUP. only)
  ⇒ Pb-210/Cs-137 (DUP. only)

- *Representativeness* is the degree to which data accurately and precisely represent a characteristic of a population. Representativeness is addressed primarily in the sample design, through the selection of sampling sites and procedures that reflect the project goals and environment being sampled. It is ensured by the proper handling, homogenizing, compositing, and storage of samples and analysis within the specified holding times so that the material analyzed reflects the material collected as accurately as possible.

  Applicable analyses: 2,3,7,8-TCDD, Grain size, TOC, Pb-210 and Cs-137.

EPA-NE QAPP Worksheet #11a - Rev. 04/03 (continued)

Project Quality Objectives/Decision Statements

- *Completeness* is defined as the amount of data collected as compared to the amount that is needed to make valid decisions. 100% completeness is targeted for all analyses. Study objectives will not be compromised if 95% of the analytes are reported.

  Applicable analyses: 2,3,7,8-TCDD, Grain size, TOC, Pb-210 and Cs-137.

- *Comparability* is the measure of the confidence with which one data set can be compared to another. Comparability can be measured using split samples, comparing data to historical, or as will be done for this project -- by using established laboratory methods that are comparable to other low-level methods and by analyzing Standard Reference Material (SRM) samples, as available. Using SRMs from batch to batch of samples throughout this project shows how data compares at the concentration of the SRM.

  Applicable samples: SRM
  Applicable analyses:
  ⇒ 2,3,7,8-TCDD
  ⇒ TOC

- *Sensitivity* is the capability of methodology or instrumentation to discriminate among measurement responses for quantitative differences or a parameter of interest. Sensitivity expressed as the detection limits is presented in (EPA Worksheet #9b).

  Applicable analyses: 2,3,7,8-TCDD, TOC, Pb-210 and Cs-137.

- *Quantitation Limits*. Recommended project action goals are documented in EPA-NE QAPP Worksheet #9b. These are recommended detection limits, not <u>required</u> limits.

All deviations from protocols described in this QAPP will be documented and approved by the Project Manager and discussed in the final report. All data that do not meet the listed MPCs will be submitted to the Project Manager, or his designee, for review and assessment of the potential impact of the results.

Affected samples may be reanalyzed at the Project Manager's discretion. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a, Table 5 of 9/27/02 QAPP Addendum), and the rationale for accepting the analysis will be thoroughly documented.

EPA-NE QAPP Worksheet #11b - Rev. 04/03

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	2,3,7,8-TCDD
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	1613B/ L-23	Accuracy	<5× MDL, or associated samples >5× blank values (>10× blank values for totals)	Blank	A
			PD≤30% from certified values (for certified values >5× MDL)	Standard Reference Material	A
			Scored by EPA	EPA PE	A
			Within Method 1613 Table 6 OPR requirements for LCS 50-120% R for MS/MSD (Analyte concentration in MS/MSD must be >5× background concentration to be used for data quality assessment)	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			25-150% R	Internal Standards	A
		Precision	NA (Not required when MS/MSD is present)	Laboratory (Analytical) Sample Duplicates	A
			RPD≤30% (Using Recovery data; Concentration of spiked analytes in MS/MSD must be >5× background concentrations to be used for data quality assessment)	MS/MSD	A
		Comparability	See SRM above	Intercomparison exercises (*e.g.*, SRM analyses), follow defined SOPs	A

NA = Not Applicable

MDL = Method Detection Limit; PD = Percent Difference; R = Recovery; RPD = Relative Percent Difference

[1] Reference SOP Number from EPA-NE QAPP Worksheet #13.
[2] Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20.
[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability)

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	Grain Size
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	ASTM D422/ L-46	Accuracy	Not applicable	Blank	A
			Not applicable -- *No SRM for grain size in sediment/soil available*	Standard Reference Material	A
			Not applicable	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			Not applicable	Surrogates	A
		Precision	RPD ≤ 50% (for analytes detected at level > 10× MDL)	Laboratory (Analytical) Sample Duplicates	A
			Not applicable	MS/MSD	A
		Comparability	Not applicable, see SRM above	Intercomparison exercises (*e.g.*, SRM analyses)	A

MDL = Method Detection Limit; RPD = Relative Percent Difference.
[1]Reference SOP Number from EPA-NE QAPP Worksheet #13.
[2] Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20.
[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	TOC
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	9060/ L-49	Accuracy	<10× MDL (with signal:noise >10:1)	Blank	A
			PD ≤ 5%	Standard Reference Material	A
			Not applicable	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			Not applicable	Surrogates	A
		Precision	RPD ≤ 50% (for analytes detected at level >10× MDL)	Laboratory (Analytical) Sample Duplicates	A
			Not applicable	MS/MSD	A
		Comparability	See SRM above	Intercomparison exercises (e.g., SRM analyses), follow defined SOPs	A

MDL = Method Detection Limit; RPD = Relative Percent Difference; PD = Percent Difference.
[1] Reference SOP Number from EPA-NE QAPP Worksheet #13.
[2] Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20.
[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	Pb-210
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	TBE-032-30/ L-112	Accuracy	< Sample-specific MDC, or associated samples >5× blank values	Blank	A
			Not applicable – *No SRM or PE available*	Standard Reference Material	A
			Analytes meet the following: 70-130% R	Laboratory Control Sample	A
			60-140% R (Concentration of spiked analytes in MS must be >5× background concentrations to be used for data quality assessment)	Matrix Spike	A
		Precision	RPD≤50% (Analytes detected at level >5× MDC)	Laboratory (Analytical) Sample Duplicates	A
			Not Applicable (MS only)	MS/MSD	A
		Comparability	Not applicable, see SRM above	Intercomparison exercises (*e.g.*, SRM analyses)	A

RL = Reporting Limit; R = Recovery; MDC = Minimum Detectable Concentration; RPD = Relative Percent Difference

1 Reference SOP Number from EPA-NE QAPP Worksheet #13.

2 Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20.

3 Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	Cs-137
Concentration Level	Low

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	TBE-2008/ L-113	Accuracy	< Sample-specific MDC, or associated samples >5× blank values	Blank	A
			Not applicable – *No SRM or PE available*	Standard Reference Material	A
			Analytes meet the following: 70-130% R	Laboratory Control Sample	A
			Not performed for Cs-137	Matrix Spike	A
		Precision	RPD≤50% (Analytes detected at level >5× MDC)	Laboratory (Analytical) Sample Duplicates	A
			Not performed	MS/MSD	A
		Comparability	Not applicable, see SRM above	Intercomparison exercises (*e.g.*, SRM analyses)	A

RL = Reporting Limit; R = Recovery; MDC = Minimum Detectable Concentration; RPD = Relative Percent Difference.
[1] Reference SOP Number from EPA-NE QAPP Worksheet #13.
[2] Reference analytical method/SOP Number from EPA-NE QAPP Worksheet #20.
[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAP P Worksheet #12b - Rev. 04/03

Sampling Locations, Sampling and Analysis Method/SOP Requirements Table

Sampling Location [1]	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc Level	No. of Samples (Identify field duplicates and replicates)		Sampling SOP[2]	Analytical Method/ SOP[2]	Minimum Sample Volume [3]	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Allendale Pond	See site map (figure 3)	Sediment	2 from each core	2,3,7,8-TCDD	Low	40	QC: 2 DU	S-17, S-18	1613B/L-23	½ full	125 mL pre-cleaned jar	Cold (<6°C)	1 year
Allendale Pond	See site map (figure 3)	Sediment	10 from each core	Pb-210	Low	60	NA	S-17, S-18	TBE-032-30/L-112	½ full	125 mL pre-cleaned jar	Cold (<6°C)	30 d
Allendale Pond	See site map (figure 3)	Sediment	10 from each core	Cs-137	Low	60	NA	S-17, S-18	TBE-2008/L-113	½ full	125 mL pre-cleaned jar	Cold (<6°C)	30 d
Allendale Pond	See site map (figure 3)	Sediment	1 from each core	Grain size, TOC	Low	20	QC: 1 DU	S-17, S-18	Grain Size L-46; TOC L-49	½ full	500 mL pre-cleaned glass jar	Cold (<6°C)	28 d
Allendale Pond	See site map (figure 3)	Sediment	10 from each core	Archive for TCDD sample selection	NA	60	NA	S-17, S-18	NA	½ full	125 mL pre-cleaned jar	Cold (<6°C)	NA

EPA-NE QAP P Worksheet #12b - Rev. 04/03 (continued)

Sampling Locations, Sampling and Analysis Method/SOP Requirements Table

Sampling Location [1]	Location ID Number	Medium/ Matrix	Depth (Units)	Analytical Parameter	Conc Level	No. of Samples (Identify field duplicates and replicates)		Sampling SOP[2]	Analytical Method/ SOP[2]	Minimum Sample Volume [3]	Containers (Number, size and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Lymansville Pond	See site map (figure 3)	Sediment	2 from each core	2,3,7,8-TCDD	Low	20	QC: 2 DU	S-17, S-18	1613B/L-23	½ full	125 mL pre-cleaned jar	Cold (<6°C)	1 year
Lymansville Pond	See site map (figure 3)	Sediment	10 from each core	Pb-210	Low	40	NA	S-17, S-18	TBE-032-30/L-112	½ full	125 mL pre-cleaned jar	Cold (<6°C)	30 d
Lymansville Pond	See site map (figure 3)	Sediment	10 from each core	Cs-137	Low	40	NA	S-17, S-18	TBE-2008/L-113	½ full	125 mL pre-cleaned jar	Cold (<6°C)	30 d
Lymansville Pond	See site map (figure 3)	Sediment	1 from each core	Grain size, TOC	Low	10	QC: 1 DU	S-17, S-18	Grain Size L-46; TOC L-49	½ full	500 mL pre-cleaned glass jar	Cold (<6°C)	28 d
Lymansville Pond	See site map (figure 3)	Sediment	10 from each core	Archive for TCDD sample selection	NA	40	NA	S-17, S-18	NA	½ full	125 mL pre-cleaned jar	Cold (<6°C)	NA

NA = Not Applicable; DU = Field Duplicate;

[1] All field sampling locations are critical; see Field Sampling Plan for specific locations.

[2] See the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13), Field Analytical Method/SOP Reference Table (EPA-NE QAPP Worksheet #17), and Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

[3] Sample volume listed for soil (all parameters) is sufficient to allow extra material for preparation of Fixed Laboratory quality control (QC) samples. Supplemental QC samples identified above.

(a) PE samples, supplied by NAE/EPA, will be shipped directly to participating laboratories from EPA. These samples will not be shipped with study samples. See EPA-NE QAPP Worksheet #22a.

EPA-NE QAPP Worksheet #13 - Rev. 04/03

Project Sampling SOP Reference Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Identification	Modified for Project Work Y or N	Comments
S-17	Soil Sampling Appendix E -- Visual Identification of Soil Samples EM 1110-1-1906 (9/30/96)	USACE WES	Core log, core slicing equipment	N	Attachment
S-18	Draft Coring Methodology U.S. EPA ERTC/REAC March 2003	ERTC/REAC	Vibracore and penetrometer assembly, decontaminated Lexan core liners, core caps, GPS	N	Attachment

EPA-NE QAPP Worksheet #14 - Rev. 04/03

Field Sampling Equipment Calibration Table

Equipment	Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference*
Vibracore	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable

* Specify appropriate reference letter/number from the Project Sampling SOP Reference Table (EPA-NE QAPP Worksheet #13).

EPA-NE QAPP Worksheet #15 - Rev. 04/03

Field Equipment Maintenance, Testing and Inspection Table

Sampling Equipment/ Instrument	Maintenance Activity	Testing Activity	Inspection Activity	Responsible Person	Frequency	Acceptance Criteria	Corrective Action	SOP Reference
Vibracore	Cleaning	NA	Visual inspection	Alan Humphrey (ERTC/REAC)	Prior to use and after each use	Decon. completed	Re-Clean	NA
Core Slicing Equipment	Cleaning	NA	Visual inspection	Maureen Corcoran (USACE WES)	Prior to use and after each use	Decon. completed	Re-Clean	NA

NA = Not applicable.

EPA-NE QAPP Worksheet #16 - Rev. 04/03

Sample Handling, Tracking and Custody Requirements

SAMPLE COLLECTION, PACKAGING AND SHIPMENT (Sediment Cores)
Sample Collection: Alan Humphrey, ERTC/REAC -- Sediment cores
Sample Packing: Alan Humphrey, ERTC/REAC -- Sediment cores
Coordination of Shipment: Alan Humphrey, ERTC/REAC -- Sediment Cores Barbara Bergen, EPA AED -- Sediment cores
Type of Shipment: Same day transfer to EPA-AED, Narragansett using AED vehicle.
SAMPLE COLLECTION, PACKAGING AND SHIPMENT (Sediment Core Sub-samples)
Sample Collection: Maureen Corcoran, USACE WES -- Sediment core logging and sub-samples.
Sample Packing: Maureen Corcoran, USACE WES -- Sediment core sub-samples.
Coordination of Shipment: Maureen Corcoran, USACE WES -- Sediment core sub-samples. Barbara Bergen, EPA AED -- Sediment core sub-samples
Type of Shipment (Courier): Overnight Courier.
SAMPLE RECEIPT AND ANALYSIS
Responsible Organization: Battelle Columbus (receive samples for 2,3,7,8-TCDD analysis) Teledyne Brown (receive samples for Pb-210 and Cs-137dating analyses) Applied Marine Sciences (receive samples for TOC and Grain Size analyses)
Sample Receipt: Henry Pham, Sample Custodian Battelle Columbus Pat Marshall, Sample Custodian Teledyne Brown Ken Davis, Sample Custodian Applied Marine Sciences, Inc
Sample Custody and Storage: Henry Pham, Sample Custodian Battelle Columbus Pat Marshall, Sample Custodian Teledyne Brown Ken Davis, Sample Custodian Applied Marine Sciences, Inc
Sample Preparation: Mark Misita, Henry Pham and Susan Winnard (assisted by Wesley Baxter), 2,3,7,8-TCDD at Battelle Columbus Lauren Larson, Pb-210 and Cs-137 at Teledyne Brown Ken Davis, TOC and Grain Size at Applied Marine Sciences **Laboratory QC samples defined on EPA-NE QAPP Worksheet #24a must be prepared with the Study Samples. MS, MSD and laboratory duplicate samples must be prepared from Centredale project samples.**

EPA-NE QAPP Worksheet #16 - Rev. 04/03 (continued)

Sample Handling, Tracking and Custody Requirements

Sample Determinative Analysis: Joe Tabor (assisted by Mark Misita), 2,3,7,8-TCDD at Battelle Columbus Lauren Larson, Pb-210 and Cs-137 at Teledyne Brown Ken Davis, TOC and Grain Size at Applied Marine Sciences
SAMPLE ARCHIVAL
Field Sample Storage (No. of days from sample collection): Varies by parameter and matrix for chemical analyses -- see Worksheet #9a (Table 2)
Sample Extract/Digestate Storage (No. of days from extraction/digestion): varies by parameter and matrix for chemical analysis-- see Worksheet #9a (Table 2)
SAMPLE DISPOSAL
Responsible Organization: Each participating laboratory is responsible for disposal of samples received for required analyses
Responsible Personnel: Project Manager and Sample Custodian

EPA-NE QAPP Worksheet #20 - Rev. 04/03

Fixed Laboratory Analytical Method/SOP Reference Table

Ref Number	Fixed Laboratory Performing Analysis	Title, Revision Date and/or Number	Definitive or Screening Data	Region I NESTS Method Code	Analytical Parameter	Instrmt	Mod for Project Work Y or N
L-23	Battelle Columbus	The Analysis of Polychlorinated Dibenzo-p-dioxin/Polychlorinated Dibenzofuran (PCDD/PCDF) Using High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) Using Modified Method 8290 (ASAT.II-001-02)	Definitive	NA	2,3,7,8-TCDD	GC/ HRMS	N
L-112	Teledyne Brown	Determination of Leachable Lead-210 In Soils And Sediments (Procedure 032-30, Rev. [illegible])	Definitive	NA	Pb-210	Low level, gas-flow proportional counter	N
L-113	Teledyne Brown	Gamma Emitting Radioisotope Analysis (Procedure TBE-2008, Rev. [illegible])	Definitive	NA	Cs-137	Gamma-Ray Spectrometer	N
L-114	Teledyne Brown	Calibration and Control of Alpha and Beta Counting Instruments (Procedure TBE-3003, Rev. [illegible])	Definitive	NA	Pb-210	Low level, gas-flow proportional counter	N
L-115	Teledyne Brown	Calibration and Control of Gamma-Ray Spectrometers (Procedure TBE-3001, Rev. [illegible])	Definitive	NA	Cs-137	Gamma-Ray Spectrometer	N
L-116	Teledyne Brown	Balance Calibration Check (Procedure PRO-Q-008, Rev. [illegible])	Definitive	NA	Pb-210/Cs-137	Balance	N
All SOPs listed in shaded rows (blue) are included with this QAPP Update; all other SOPs were provided with the May 23, 2001 QAPP							

EPA-NE QAPP Worksheet #21 - Rev. 04/03

Fixed Laboratory Instrument Maintenance and Calibration Table

Instrument	Activity	List Maintenance, Testing and Inspection Activities	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[1]	Method/ SOP Ref*
Analytical Balance	Moisture Content	Daily performance check (or with each use).	Daily using two Class "S" weights bracketing expected weight range	Within 0.01 g of value	Calibration and service performed as needed or at regular intervals by professional metrology technician.	Louie Balogh (lead analyst)	PRO-Q-008/ L-116
Low Background Proportional Counter	Pb-210	Daily source checks and backgrounds	Yearly	3 Standard deviations of established mean	Remedial maintenance, new initial calibration, Document and justify	Marty Webb (Production Supervisor)	TBE-3003/ L-114
High Purity Germanium Detector	Cs-137	Daily calibration checks	Yearly	3 Standard deviations of established mean	Remedial maintenance, new initial calibration, Document and justify	Marty Webb (Production Supervisor)	TBE-3001/ L-115

IC = Initial Calibration; CC = Continuing Calibration; RSD = Relative Standard Deviation; PD = Percent Difference; LB = Lab Blank; DL = Detection Limit; RL = Reporting Limit; ICV = Initial Calibration Verification; CCV = Continuing Calibration Verification.

[1] If specified project personnel not available; alternate and equally trained staff will perform task/corrective action

* Specify appropriate reference letter/number from Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).

EPA-NE QAPP Worksheet #22a - Rev. 04/03

Field Sampling QC Table

Sampling SOP	S-17, S-18
Medium/Matrix	Sediment
Analytical Parameter	2,3,7,8-TCDD
Concentration Level	Low
Analytical Method/ SOP Reference	1613B/L-23
Sampler's Name	Alan Humphrey
Field Sampling Organization	ERTC/REAC
No. of Sample Locations	Up to 20 cores per phase

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (see EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Note – Unacceptable Field QC sample results are not a true measure of Fixed Laboratory accuracy and precision performance.

Field QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blanks/ Rinsate Blanks	NA	NA	NA	NA	NA	NA
Bottle Blanks	NA	NA	NA	NA	NA	NA
Trip Blanks	NA	NA	NA	NA	NA	NA
Cooler Temperature Blanks	1 per cooler (bottle with clean sand – not a sample)	NA	Inform Project Manager; Document in QA/QC Narrative	Laboratory Sample Custodian	NA	< 6 °C
Field Duplicate Pairs (Duplicate Subsamples)	1 per 20	NA	Inform Project Manager; Document in QA/QC Narrative	GC/HRMS Analyst	Precision	Guidance Limits Only: RPD ≤ 50% (for TCDD >10× MDL)
Collocated Samples	NA	NA	NA	NA	NA	NA
Field Splits	NA	NA	NA	NA	NA	NA
PES sent to Laboratory	NA	NA	NA	NA	NA	NA
Other:	NA	NA	NA	NA	NA	NA

RPD = Relative Percent Difference; MDL = Method Detection Limit

EPA-NE QAPP Worksheet #22a - Rev. 04/03 (continued)

Field Sampling QC Table

Sampling SOP	S-17, S-18
Medium/Matrix	Sediment
Analytical Parameter	Grain Size and TOC
Concentration Level	Low
Analytical Method/ SOP Reference	ASTM D422/L-46 9060/L-49
Sampler's Name	Alan Humphrey
Field Sampling Organization	ERTC/REAC
No. of Sample Locations	Up to 20 cores per phase

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (see EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Note -- Unacceptable Field QC sample results are not a true measure of Fixed Laboratory accuracy and precision performance.

Field QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blanks/ Rinsate Blanks	NA	NA	NA	NA	NA	NA
Bottle Blanks	NA	NA	NA	NA	NA	NA
Trip Blanks	NA	NA	NA	NA	NA	NA
Cooler Temperature Blanks	1 per cooler (bottle with clean sand -- not a sample)	NA	Inform Project Manager; Document in QA/QC Narrative	Laboratory Sample Custodian	NA	< 6 °C
Field Duplicate Pairs (Duplicate Subsamples)	1 per 20	NA	Inform Project Manager; Document in QA/QC Narrative	Ken Davis	Precision	Guidance Limits Only: RPD ≤ 50% (for values >10× MDL)
Collocated Samples	NA	NA	NA	NA	NA	NA
Field Splits	NA	NA	NA	NA	NA	NA
PES sent to Laboratory	NA	NA	NA	NA	NA	NA
Other:	NA	NA	NA	NA	NA	NA

RPD = Relative Percent Difference; MDL = Method Detection Limit

EPA-NE QAPP Worksheet #22a - Rev. 04/03 (continued)

Field Sampling QC Table

Sampling SOP	S-17, S-18
Medium/Matrix	Sediment
Analytical Parameter	Pb-210
Concentration Level	Low
Analytical Method/ SOP Reference	TBE-032-30/L-112
Sampler's Name	Alan Humphrey
Field Sampling Organization	ERTC/REAC
No. of Sample Locations	Up to 20 cores per phase

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (see EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Note – Unacceptable Field QC sample results are not a true measure of Fixed Laboratory accuracy and precision performance.

Field QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	NA	NA	NA	NA	NA	NA
Bottle Blanks	NA	NA	NA	NA	NA	NA
Trip Blanks	NA	NA	NA	NA	NA	NA
Cooler Temperature Blanks	1 per cooler (bottle with clean sand – not a sample)	NA	Inform Project Manager; Document in QA/QC Narrative	Laboratory Sample Custodian	NA	< 6 °C
Field Duplicate Pairs (Duplicate Subsamples)	1 per 20	NA	Inform Project Manager; Document in QA/QC Narrative	Analyst	Precision	Guidance Limits Only: RPD ≤ 50% (for analytes detected at level > 10× MDC)
Collocated Samples	NA	NA	NA	NA	NA	NA
Field Splits	NA	NA	NA	NA	NA	NA
PES sent to Laboratory*	NA	NA	NA	NA	NA	NA
Other:	NA	NA	NA	NA	NA	NA

RPD = Relative Percent Difference; RL = Reporting Limit; MDC = Minimum Detectable Concentration

EPA-NE QAPP Worksheet #22a - Rev. 04/03 (continued)

Field Sampling QC Table

Sampling SOP	S-17, S-18
Medium/Matrix	Sediment
Analytical Parameter	Cs-137
Concentration Level	Low
Analytical Method/ SOP Reference	TBE-2008/ L-113
Sampler's Name	Alan Humphrey
Field Sampling Organization	ERTC/REAC
No. of Sample Locations	Up to 20 cores per phase

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (see EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Note – Unacceptable Field QC sample results are not a true measure of Fixed Laboratory accuracy and precision performance.

Field QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Equipment Blanks/ Rinsate Blanks	NA	NA	NA	NA	NA	NA
Bottle Blanks	NA	NA	NA	NA	NA	NA
Trip Blanks	NA	NA	NA	NA	NA	NA
Cooler Temperature Blanks	1 per cooler (bottle with lab water – not a sample)	NA	Inform Project Manager; Document in QA/QC Narrative	Laboratory Sample Custodian	NA	< 6 °C
Field Duplicate Pairs (Duplicate Subsamples)	1 per 20	NA	Inform Project Manager; Document in QA/QC Narrative	Gamma-Ray Spectrometer Analyst	Precision	Guidance Limits Only: RPD ≤50% (for Cs-137 >10× MDC)
Collocated Samples	NA	NA	NA	NA	NA	NA
Field Splits	NA	NA	NA	NA	NA	NA
PES sent to Laboratory	NA	NA	NA	NA	NA	NA
Other:	NA	NA	NA	NA	NA	NA

RPD = Relative Percent Difference; QL = Quantitation Limit; MDC = Minimum Detectable Concentration

EPA-NE QAPP Worksheet #24a - Rev. 04/03
Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Sediment
Sampling SOP	S-17, S-18
Analytical Parameter	2,3,7,8-TCDD
Concentration Level	Low
Analytical Method/ SOP Reference	1613B/L-23
Laboratory Name	Battelle Columbus
No. of Sample Locations	2 each from approx. 30 cores

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/sample set	NA	Reextract, reanalyze, or justify	GC/HRMS Analyst	Accuracy	<5× MDL, or associated samples >5× blank values (>10× blank values for totals)
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike/Matrix Spike Duplicate	1/sample set	NA	Review with Laboratory Manager: re-analyze or justify in project records	Task Leader	Accuracy/ Precision	50-120% R RPD ≤30% (Analyte concentration in MS/MSD must be >5× background concentration to be used for data quality assessment)
LCS	1/sample set	Method 1613B, Table 6, "OPR" requirements	Review with Laboratory Manager: re-analyze or justify in project records NA	Task Leader	Accuracy	Method 1613B, Table 6, "OPR" requirements
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	1 per sample	NA	Review with Laboratory Manager: re-analyze or justify in project records	GC/HRMS Analyst/ Preparation Analyst	Accuracy	25 – 150% R
Other: SRM	1/sample set	NA	Reextract, reanalyze or justification documented	Task Leader	Accuracy, Comparability	PD ≤30% from certified values (for cert. Analytes >5× MDL)

MDL = Method Detection Limit; R = Recovery; RPD = Relative Percent Difference; OPR = Ongoing Precision and Recovery; PD = Percent Difference.

EPA-NE QAPP Worksheet #24a - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Sediment
Sampling SOP	S-17, S-18
Analytical Parameter	Pb-210
Concentration Level	Low
Analytical Method/ SOP Reference	TBE-032-30/L-112
Laboratory Name	Teledyne Brown
No. of Sample Locations	Up to 30 cores

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	1/sample set	NA	Reanalyze, or justify	Analyst	Accuracy	< Sample-specific MDC, or associated samples >5× blank values
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA (As required)	NA	NA	NA	NA	NA
Laboratory Duplicate	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Precision	RPD ≤ 50% (Analytes detected at level >5× MDC)
Laboratory Matrix Spike/Matrix Spike Duplicate	1 MS/ sample set	NA	Reanalyze or justification documented	Laboratory Analyst	Accuracy/ Precision	60-140% R (Concentration of spiked analytes in MS must be >5× background concentrations to be used for data quality assessment)
LCS	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Accuracy	70-130% R
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: SRM	NA	NA	NA	NA	NA	NA

Sample Set = 20 or fewer authentic study samples.

MDC = Minimum Detectable Concentration; R = Recovery; RPD = Relative Percent Difference

EPA-NE QAPP Worksheet #24a - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Sediment
Sampling SOP	S-17, S-18
Analytical Parameter	Cs-137
Concentration Level	Low
Analytical Method/ SOP Reference	TBE-2008/L-113
Laboratory Name	Teledyne Brown
No. of Sample Locations	Up to 30 cores

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	1/sample set	NA	Reanalyze, or justify	Analyst	Accuracy	< Sample-specific MDC, or associated samples >5× blank values
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA (as required)	NA	NA	NA	NA	NA
Laboratory Duplicate	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Precision	RPD ≤ 50% (Analytes detected at level >5× MDC)
Laboratory Matrix Spike/Matrix Spike Duplicate	Not performed for Cs-137	NA	NA	NA	NA	NA
LCS	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Accuracy	70-130% R
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: SRM	NA	NA	NA	NA	NA	NA

Sample Set = 20 or fewer authentic study samples.
MDC = Minimum Detectable Concentration; R = Recovery; RPD = Relative Percent Difference

EPA-NE QAPP Worksheet #24b - Rev. 04/03

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method/SOP: L-23 (Sediment); 2,3,7,8-TCDD

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits (Dry Weight -pg/g)	Analytical Precision		Analytical Accuracy/Bias	
		Laboratory Replicate	MS/MSD	SRM	LCS/MS/MSD
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1	Not applicable	RPD ≤ 30% (a)	PD ≤ 30% (b)	LCS: Within Method 1613B Table 6 OPR MS/MSD: 50 -- 120% (a)
Total TCDD	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable
TCDD Internal Standard	Not applicable	Not applicable	Not applicable	25 -- 150% R	

RPD = Relative Percent Difference; PD = Percent Difference; R = % Recovery; MS = Matrix Spike;
MSD = Matrix Spike Duplicate; SRM = Standard Reference Material; MDL = Method Detection Limit;
LCS = Laboratory Control Sample; OPR = Ongoing Precision and Recovery
(a) Concentration of spiked analytes in MS/MSD must be >5× background concentration to be used for data quality assessment
(b) SRM; For certified values >5× MDL

EPA-NE QAPP Worksheet #24b - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method/SOP: L-46 (Sediment); Grain Size

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits (Dry Weight – %)	Analytical Precision	Analytical Accuracy/Bias	
		(Laboratory Replicates Only)	SRM	LCS/MS/MSD
% gravel	0.01	RPD ≤ 50% (for values >10× MDL)	Not applicable	Not applicable
% coarse sand	As above	As above	As above	As above
% medium sand	As above	As above	As above	As above
% fine sand	As above	As above	As above	As above
% silt	As above	As above	As above	As above
% clay	As above	As above	As above	As above
Grain size distribution curve	As above	Not applicable	As above	As above

RPD = Relative Percent Difference; MDL = Method Detection Limit; SRM = Standard Reference Material; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate

Sampling SOP: S-17, S-18
Analytical Method/SOP: L-49 (Sediment); TOC

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits (Dry Weight – %)	Analytical Precision	Analytical Accuracy/Bias	
		(Laboratory Replicates Only)	SRM	LCS/MS/MSD
% TOC	0.01-0.001	RPD≤ 50% (for values >10× MDL)	PD ≤ 5%	Not applicable

RPD = Relative Percent Difference; MDL = Method Detection Limit; SRM = Standard Reference Material; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; PD = Percent Difference

EPA-NE QAPP Worksheet #24b - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method/SOP: L-112 (Sediment); Pb-210

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits (Dry Weight -pCi/g)	Analytical Precision		Analytical Accuracy/Bias		
		Laboratory Replicate	MS/MSD	SRM	LCS	MS
Pb-210	0.1	RPD ≤ 50% (a)	Not Applicable	Not Applicable	70-130% R	60-140% R (b)

RPD = Relative Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; R = Recovery.

(a) Replicates: for values >5× MDC (Minimum Detectable Concentration)
(b) Concentration of spiked analyte in MS must be >5× background concentration to be used for data quality assessment

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method/SOP: L-113 (Sediment); Cs-137

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits (Dry Weight -pCi/g)	Analytical Precision		Analytical Accuracy/Bias		
		Laboratory Replicate	MS/MSD	SRM	LCS	MS
Cs-137	0.5	RPD ≤ 50% (a)	Not Applicable	Not Applicable	70-130% R	Not Applicable

RPD = Relative Percent Difference; LCS = Laboratory Control Sample; MS = Matrix Spike; MSD = Matrix Spike Duplicate; R = Recovery.

(a) Replicates: for values >5× MDC (Minimum Detectable Concentration)
(b) Concentration of spiked analytes in MS must be >5× background concentration to be used for data quality assessment

EPA-NE QAPP Worksheet #26 - Rev. 04/03

Project Documentation and Records Table

Sample Collection Records	Field Analysis Records	Fixed Laboratory Records	Data Assessment Records	Other
Field Logs	Well purging log (pH, T, conductivity, etc.)	Sample Receipt, Custody and Tracking Forms	Performance Audit Reports (sample preparation, chemical analysis)	
Custody Forms	Soil boring/sample description logs	Standard Preparation Logs (certificates of analysis, QC checks)	Lab-wide Systems Audit Reports	
Sample Labels	Sediment core logs	Equipment Calibration Logs	PE Sample Results	
Custody Seals		Sample Preparation Records	Corrective Action Forms	
Telephone Logs		GC/ECD, GC/MS and GC/HRMS Logbooks (acquisition, maintenance, tuning)	Control Charts	
Photograph log		Calibration Reports	Telephone Logs and/or electronic mail	
GPS log		Sample Quantification Reports		
		Sample Chromatograms		
		Laboratory Logbooks (acquisition, maintenance)		
		Final Report Tables (authentic samples and QC results)		
		Corrective Action Logs (miscellaneous documentation)		
		Study Records (e.g., Data Package)		
		Sample Disposal Records		
		Telephone Logs and/or electronic mail		

EPA-NE QAPP Worksheet #27b - Rev. 04/03

Project Assessment Table

Assessment Type	Frequency	Internal or External (1)	Organization Performing Assessment	Person(s) responsible for performing assessment, title and organizational affiliation	Person(s) responsible for responding to assessment findings, title and organizational affiliation	Person (s) responsible for identifying and implementing corrective actions (CA), title and organizational affiliation	Person (s) responsible for monitoring effectiveness of CA, title and organizational affiliation
Fixed Laboratory Data Package Audit	Each Pb/Cs data package	Internal and External	Teledyne Brown Engineering Environmental Services	Marty Keller QA Manager Teledyne Brown	Lauren Larson Analyst Teledyne Brown	Marty Webb Production Manager Teledyne Brown	Marty Keller QA Manager Teledyne Brown

[1] *Note* -- All data packages will receive internal validation and audits. All non-HRMS data packages (excluding percent moisture) will receive an external (E) Tier II validation.

EPA-NE QAPP Worksheet #27c - Rev. 04/03

Project Assessment Plan

QAPP Title:	Centredale Manor QAPP
Assessed Organization:	Battelle Columbus
Location of Assessment:	Battelle Columbus, Columbus, OH
Dates of Assessment:	Completion of analytical task
Assessment Team Members:	Zachary J. Willenberg
Type of Assessment:	Data audit
Assessment Scope:	Audit data package for completeness and accuracy
Documents to be Reviewed:	2,3,7,8-TCDD data packages (custody, sample processing data, GC/HRMS data and calibrations, and final report tables)
Notification Date(s):	At completion of audit
Proposed Schedule:	Estimated: August 1-15, 2003
Assessment No.:	
Contract No.:	

EPA-NE QAPP Worksheet #27c - Rev. 04/03 (continued)

Project Assessment Plan

QAPP Title:	Centredale Manor QAPP
Assessed Organization:	Applied Marine Sciences, Inc.
Location of Assessment:	Applied Marine Sciences, Inc. League City, TX
Dates of Assessment:	Completion of analytical task
Assessment Team Members:	Jennifer Davis
Type of Assessment:	Data audit
Assessment Scope:	Audit data package for completeness and accuracy
Documents to be Reviewed:	Grain Size and TOC data package (custody, sample processing data, instrument data and calibration records (if appropriate), final report tables
Notification Date(s):	At completion of audit
Proposed Schedule:	Estimated: August 1-15, 2003
Assessment No.:	
Contract No.:	

EPA-NE QAPP Worksheet #27c - Rev. 04/03 (continued)

Project Assessment Plan

QAPP Title:	Centredale Manor QAPP
Assessed Organization:	Teledyne Brown Engineering -- Environmental Services
Location of Assessment:	Teledyne Brown Engineering -- Environmental Services
Dates of Assessment:	Completion of analytical task
Assessment Team Members:	Marty Keller
Type of Assessment:	Data audit
Assessment Scope:	Audit data package for completeness and accuracy
Documents to be Reviewed:	Pb-210 and Cs-137 data package (custody, sample processing data, instrument data and calibration records (if appropriate), final report tables
Notification Date(s):	At completion of audit
Proposed Schedule:	Estimated: August 1-15, 2003
Assessment No.:	
Contract No.:	

EPA-NE QAPP Worksheet #28 - Rev. 04/03

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation, Title and Organizational Affiliation	Report Recipients, Title and Organizational Affiliation
Data Audit	1/data package (2,3,7,8-TCDD)	At end of analytical task	Zachary J. Willenberg QA Officer Battelle Columbus	Sample Preparation, HRGC/HRMS Analyst, Laboratory Manager, Task Leader Battelle Columbus
Data Audit	1/data package (Grain Size, TOC)	At end of analytical task	Jennifer Davis QC Manager Applied Marine Sciences, Inc	Ken Davis Grain Size and TOC Task Leader Applied Marine Sciences
Data Audit	1/data package (Pb-210, Cs-137)	At end of analytical task	Marty Keller QA Manager Teledyne Brown	Analyst, Production Manager Teledyne Brown

EPA-NE QAPP Worksheet #29a - Rev. 04/03

Data Verification Process

Verification Task	Description	I/E [1]	Responsible for Verification (Name, Organization)
QA Audit (2,3,7,8-TCDD, Moisture)	Described in EPA-NE QAPP Worksheet #9a (Assessment/Audit Tasks) and EPA-NE QAPP Worksheet #27a, b	I	Mark Misita (Sample Preparation) Joe Tabor (HRMS Analyst) Karen Tracy (Task Leader) Mary Schrock (Laboratory Manager) Zachary J. Willenberg (QA Officer) Battelle Columbus
QA Audit (Pb-210/Cs-137)	As above	I	Rebecca Charles (Project Manager) Marty Keller (QA Manager) Teledyne Brown Engineering Environmental Services

[1] *Note* -- All data packages will receive internal validation and audits. All non-HRMS data packages (excluding percent moisture) will receive an external (E) Tier II validation.

EPA-NE QAPP Worksheet #29b - Rev. 04/03

Data Validation Summary Table

Medium/ Matrix	Analytical Parameter	Conc Level	Validation Criteria[1] (a)	Validation Criteria Modified[2] (a)	Data Validation Tier Level (a)	Modified Tier Level Used[3]	Data Validator (Name, title and organizational affiliation)	Responsibility for Data Validations (Name, title and organizational affiliation)
Sediment	2,3,7,8-TCDD	Low	Data will receive third party validation following EPA Region I Validation guidelines	NA	Tier III	NA	Steve Stodola Dioxin Data Validation Coordinator (QA Chemist, OEME) USEPA Region I	Karen Tracy (Dioxin/Furan Task Leader) Battelle Columbus
Sediment	Pb-210 and Cs-137	Low	As above	NA	Tier II[4]	NA	As above	Rebecca Charles (Project Manager) Teledyne Brown Engineering Environmental Services
Sediment	Grain Size and TOC	Low	As above	NA	Tier II	NA	As above	Ken Davis (Grain Size/TOC Task Leader) Applied Marine Sciences, Inc.

[1] If the most recent revision of the Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses will not be used to validate project data, then document this fact and, on EPA-NE QAPP Worksheet #29a, provide a detailed description of the alternate validation criteria and/or procedures that will be used.
[2] If the Region I validation criteria will be modified to meet project objectives, then document this fact and, on EPA-NE QAPP Worksheet #29a, provide a detailed description of the modified validation criteria that will be used.
[3] If a modified validation Tier will be used to validate project data, then document this fact and, on EPA-NE QAPP Worksheet #29a, provide a detailed description of the Tier modifications that will be used.
4 A modified TIER II validation according to QC criteria found in Worksheets #11b, #24a, and #24b will be used.

(a) Note – All data packages will also receive internal validation and audits, as described in EPA-NE QAPP Worksheets #9a (Assessment/Audit Tasks), #27a, #27b, #27c, #28 and #29a.

EPA-NE QAPP Worksheet #29c - Rev. 04/03

Data Validation Modifications

Participating laboratories (*i.e.*, Battelle Columbus, Applied Marine Sciences, Inc., and Teledyne Brown Engineering Environmental Services) will be responsible for performing internal data validation and verification of the data package. Data validation and verification procedures will follow internal laboratory SOPs and are further described in EPA-NE QAPP Worksheet #9a. The appropriate Task Leader will document and justify modifications to data validation/verification procedures, followed up by notification to the Project Manager/QA Officer for approval.

EPA-NE QAPP Worksheet #30 - Rev. 04/03

Data Usability Assessment

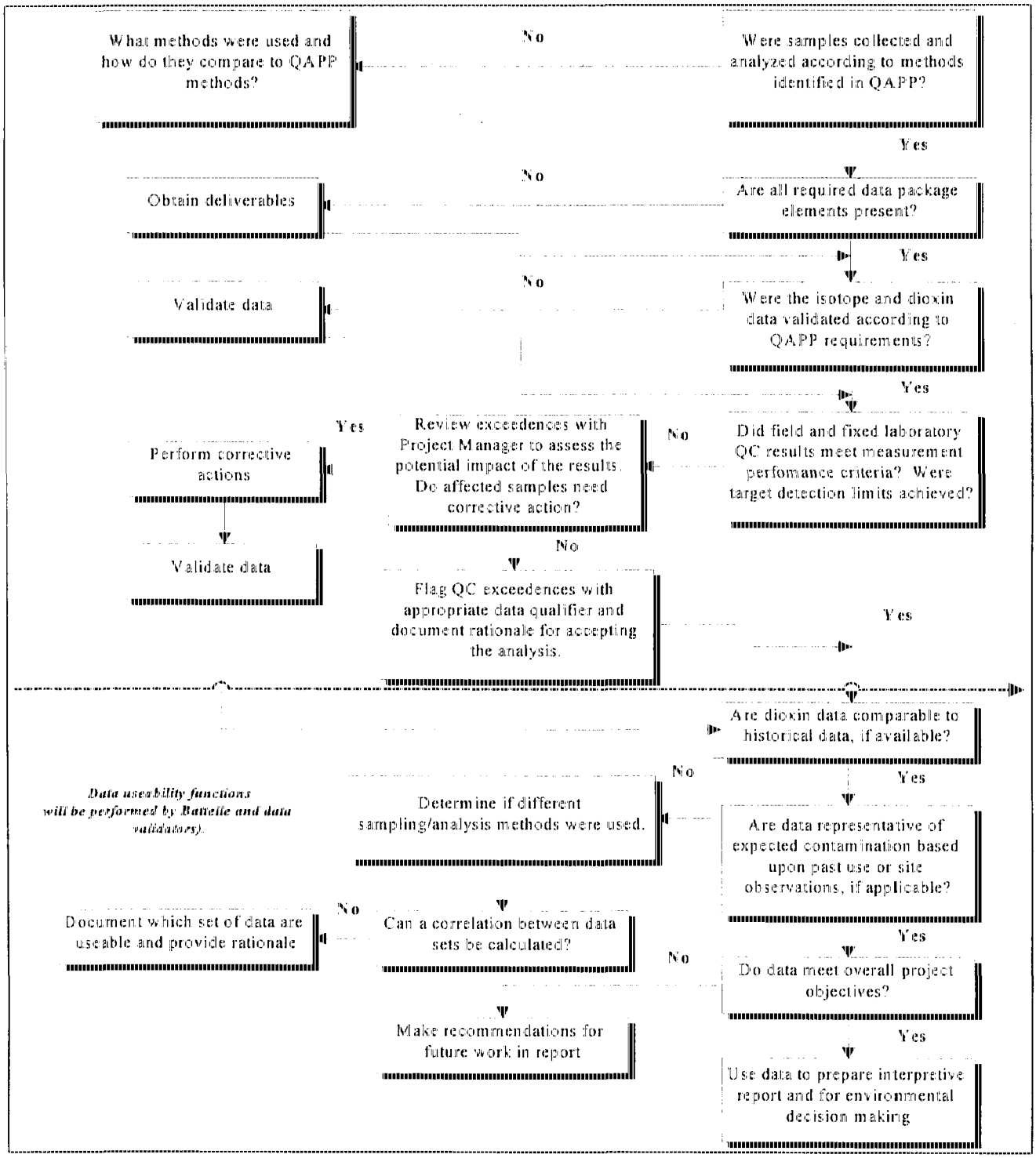

Figure 4. Preliminary Data Review Decision Tree.

Attachment L

Teledyne Brown Engineering
Environmental Services

Procedure 032-30

Determination Of Leachable Lead-210 In Soils And Sediments

Rev. 1

Date of Issue: 01/22/2001

Approved By:

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DETERMINATION OF LEACHABLE LEAD-210 IN SOILS AND SEDIMENTS

1.0 INTRODUCTION

The Pb-210 activity of soils and sediments is determined radiochemically by separating the daughter product Bi-210 and assaying its beta activity. The method presented here measures the Pb-210 fraction from which Bi-210 may be dissolved by leaching with hot hydrochloric acid; activity in the interior of mineral grains may be excluded.

Stable lead and bismuth carriers are added to the dried sample and it is leached with hot 6N hydrochloric acid. The sample is then filtered and the filtrate is evaporated, oxidized with nitric acid, and finally dissolved in 1.8N hydrochloric acid. The solution is passed through an anion exchange column. Lead is eluted first with 9N hydrochloric acid and with deionized water, then bismuth is eluted with 2M sulphuric acid. The bismuth is precipitated as the oxychloride and is collected by vacuum filtration on a 1-inch glass fiber disc. The bismuth yield is determined gravimetrically. The filter disc is mounted on a nylon planchet and covered with 3 mg/cm^2 aluminum absorber for beta assay in a low level, gas-flow proportional counter.

2.0 DETECTION CAPABILITY

Detection capability depends upon sample size, chemical yields during processing, counting time, and the efficiency and background of the counting instrument. The minimum detectable activity (MDA) for leachable Pb-210 in soils and sediments is nominally 0.2 picocuries per gram at the 4.66 sigma level. This figure is based on a sample weight of 10 grams (dry), a chemical yield of 0.5, and a counting interval of 100 minutes.

A counter background of 0.3 cpm and an efficiency of 0.23 for Bi-210 counting (using a 3 mg/cm^2 aluminum absorber) are employed in the calculation. A representative decay factor of 0.9 allows for one day delay in counting the planchet after lead separation.

3.0 SAMPLE SELECTION PROCEDURE

(a) Using the Sample Receipt Form with the Teledyne Brown Engineering sample number, locate the sample (or sample group) in the Sample Receiving and Storage room. Sign for the samples, and take them to the Environmental Radiochemistry Laboratory.

(b) Make an entry in the Lead-210 Data Book showing customer name, sample type, and sample numbers.

4.0 SAMPLE PREPARATION PROCEDURE

This section describes how soil and sediment samples are dried, leached in acid, filtered, evaporated and dissolved to prepare them for chemical separations. Wear a laboratory coat and safety glasses while carrying out the steps below. Disposable gloves and special precautions should be used for hazardous samples.

(a) Write the TBE-ES sample number on the outside of a glass beaker using a laboratory marking pen.

(b) Using a clean spatula add approximately 25 of sample into the beaker. (If analyses other than Pb-210 are required, additional sample may be decanted to accommodate them).

(c) Place the beaker under the heating lamp hood or in a hot air oven overnight to dry.

(d) Write the sample number on a clean 250 ml beaker. Weigh on a balance. Without removing the beaker, set the scale for 10.0 grams more than the tare weight. Carefully scoop the dried sample from its numbered container to the beaker until balance is again achieved, resulting in approximately 10 grams of sample in the beaker. Different sample weights may be used as necessary. Record aliquot weight used in the Lead-210 Data Book.

(e) Add 6N HC1 to the beaker, filling to the 100 ml mark. Using separate carrier pipets, add 1.00 ml standardized Bi carrier @ approximately (0.370 gm BiOCl/ml) and 1 ml Pb carrier to the sample beaker.

(f) Cover the beaker with a watch glass and place on a moderate (300°F) hotplate. Allow the sample to leach for approximately 2 hours. Remove from the hotplate and allow to cool.

(g) Fold a 15 cm fiberglass or paper filter disc in quarters to make a cone and place it in a 4-inch diameter funnel. Mark the sample number on a clean 400 ml beaker and place it under the funnel in a filter rack. Gravity filter the sample. Rinse the solids with deionized water and collect the washings with the filtrate. Discard the filter and solids.

(h) Add approximately 5 ml HNO_3 to the sample. Place the sample beaker on a moderate (300°F) hotplate and allow to gently evaporate to dryness. Check that the sample does not spatter during evaporation and reduce hotplate temperature if necessary.

(i) Remove beaker from the hotplate and allow to cool. Add approximately 20 ml conc. HC1 and again evaporate to dryness.

(j) Add 1.8N HC1 to the sample beaker, filling to the 150 ml mark. Warm gently on a hotplate to dissolve solids. Further filtration may be required. The sample is now ready for the chemical separation described next.

5.0 CHEMICAL SEPARATION AND PURIFICATION PROCEDURES

(a) Obtain a 1/2-inch diameter 10-inch length ion exchange column surmounted by a 200 ml reservoir and equipped with a stopcock at the bottom. Insert a small wad of glass wool above the stopcock.

(b) Make a slurry of anion exchange resin (eg Amberlite IRA-400 C.P.) and water in a beaker. With the column stopcock open, pour the slurry into the column until the resin occupies most of the column. Note: new resin is used for each sample.

(c) Condition the column by passing through approximately 60 ml of 1.8N HC1. Close the stopcock when the liquid level reaches the top surface of the resin.

(d) Pass the sample solution through the column with the stopcock fully open. Collect the effluent in a beaker and discard. Close the stopcock when the liquid level reaches the top surface of the resin.

(e) Exercise caution and work in a hood in this step because of the strong acid used. Pass approximately 150 ml of 9N HC1 through the column to elute the lead. Collect the effluent in a beaker and discard with caution (unless advised to save the effluent by the laboratory supervisor). Close the stopcock when the liquid level reaches the top surface of the resin. Record the midpoint of the elution period in the laboratory notebook.

(f) Pass approximately 150 ml of deionized water through the column. Close the stopcock when the liquid level reaches the top surface of the resin. Discard the effluent.

(g) Measure 150 ml of 2M H_2SO_4 in a graduated cylinder and pass through the column to elute the bismuth. Collect the effluent in a clean, labeled 400 ml beaker.

(h) Adjust the pH to 5 as follows: Add 35 ml conc NH_4OH to the sample beaker from a graduated cylinder. Add a magnetic stirring bar and place on the pH apparatus (previously standardized with pH 7.0 buffer). Add NH_4OH using an eyedropper until a pH of 5 is obtained. Add 2M H_2SO_4 with an eyedropper if the pH exceeds 5.

(i) Remove the sample beaker from the pH apparatus and add 2 ml of 6N HC1 using a disposable pipet. Dilute with deionized water, filling to the 350 ml mark.

(j) Place the sample beaker on a hotplate (approximately 500°F surface temperature) and digest until the white particles BiOC1 precipitate form and fall to the bottom of the beaker (approximately 2 hours). Remove from the hotplate and allow to cool.

6.0 MOUNTING OF PRECIPITATE

(a) Prepare a 2.8 cm glass fiber filter disc for each sample by mounting it on a vacuum filtration apparatus and rinsing with deionized water and methanol.

(b) Place the prepared discs in 4-way partitioned petri dishes which have been marked with sequence numbers (one number per partition, beginning with 1). Write a corresponding sequence number beside each sample number entry in the Lead-210 Data Book.

(c) Place petri dishes containing prepared filters in an approximately 110°C hot air oven for 10 minutes or longer to dry. Remove petri dishes and allow to cool in a desiccator.

(d) Weigh the filter discs on the analytical balance within 5 minutes of removal from the desiccator, using a clean spatula to handle them. Record this tare weight beside the corresponding sequence number and sample number in the laboratory data book. Take care to replace each filter after weighing in the numbered petri dish partition from which it came.

(e) Using a laboratory spatula, take the tared filters in sequence number order and transfer to the vacuum mounting apparatus. Wet with deionized water. Using the laboratory data book to establish the correspondence between sequence number and sample number, filter each sample from its beaker onto the corresponding filter disc.

(f) Rinse precipitate on filter with deionized water and then with ethanol. Transfer each filter from vacuum mounting apparatus back to the numbered petri dish partition from which it came. Place petri dish in a hot air oven for 10 minutes or longer.

(g) Remove petri dish and allow to cool in a desiccator. Weigh filters on the analytical balance within 5 minutes of removing from the desiccator. Write the weights beside the corresponding sequence numbers in the laboratory data book. Return each filter to its original partition in the petri dish.

(h) Write a gummed label for each sample, showing the analysis (Pb-210), the sample number and the customer. Fix each label to the back of a nylon planchet.

(i) Using the laboratory data book to establish the correspondence between sample number, and sequence number, transfer each filter to its planchet and fix in place

with a 2-inch piece of 3 mg/cm^2 aluminum absorber foil and a nylon ring. Trim excess aluminum with a razor blade or scissors.

(j) Subtract filter tare weight from final weight for each sample. Write the difference (mount weight) in the laboratory data book. Divide the mount weight by the corresponding carrier yield figure (written on the bismuth carrier flask) to obtain chemical yield. Enter the yield fraction in the laboratory data book.

(k) Using the laboratory data book as a guide, begin filling out a Radiochemical Work Sheet for each sample. Enter sample number, company name, analysis, aliquot used, mid-elution time, and analyst's initials.

(i) Submit finished planchets and work sheets to the Counting Room for radioassay.

7.0 SAMPLE COUNTING PROCEDURES

Bismuth-210 mounts are counted usually 100 minutes for beta activity in low level, gas flow proportional counters.

(a) Assign a low level, gas-flow proportional counter to each prepared bismuth mount by writing the counter number in the space provided on the Radiochemical Work Sheet.

(b) Arrange the work sheets in order according to counter number. Take the first sheet, locate the nylon planchet bearing the indicated sample number, and load the planchet into the detector tray indicated on the sheet.

(c) Set the counter timer to 100 minutes and start the counters (different counting intervals may be used). Write the counting start date and time on the Radiochemical Work Sheets in the spaces provided. Leave the work sheets on the tray in front of the counters

(d) After completion of the count, write the number of counts indicated for each detector, and the counting interval in minutes, beside the corresponding counter numbers on the Radiochemical Work Sheets. Remove the mounts from the counter trays and place them in the labeled container.

8.0 CALCULATION OF THE SAMPLE ACTIVITY OR OF THE MDA

8.1 The equation used for calculating activity and the 2 sigma counting error is:

$$\frac{Net\ pCi\ on\ counting\ date}{gram} = \underbrace{\frac{(N/\Delta t - \beta)}{2.22(v)(y)(DF)(\varepsilon)}}_{\textbf{net activity}} \pm \underbrace{\frac{2\sqrt{(N/\Delta t + \beta)/\Delta t}}{2.22(v)(y)(DF)(\varepsilon)}}_{\textbf{counting error}}$$

where:

N	=	total counts from sample (counts)
Δt	=	counting time for sample (min)
β	=	background rate of counter (cpm)
2.22	=	$\frac{dpm}{pCi}$
v	=	weight of sample analyzed (grams)
y	=	chemical yield of the mount or sample counted
DF	=	decay factor of Bi-210 from the mid-elution time to the the mid-count time
ε	=	efficiency of the counter for Bi-210 beta counting, using a No. 1 ($3mg/cm^2$) absorber

8.2 Establishing and reporting activities which are less than or equal to the detection limit:

If the net activity (previously defined) is equal to or is less than a specified multiple of the background counting error, the activity on the collection date is below the limits of detection and is called "less than" (L.T.) or "minimum detectable activity" (MDA).

The L.T. value can be specified by stating only the counting error at a predetermined multiple (σm) of the one sigma statistics. A sigma multiple (σm) of 4.66 is used for

calculation of the L.T. values unless another multiple such as 2.83 is specified.

$$\text{thus L.T.} = \frac{\sigma m \sqrt{\beta / \Delta t}}{2.22(v)(y)(DF)(\varepsilon)}$$

TELEDYNE BROWN ENGINEERING ENVIRONMENTAL SERVICES

Procedure TBE-2008

Gamma Emitting Radioisotope Analysis

Rev. 0

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Approved by:

Keith O. Jeter Date 3/29/02
Keith O. Jeter, Operations Manager

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DETERMINATION OF GAMMA EMITTING RADIOISOTOPES

1.0 SCOPE & APPLICABILITY

1.1 This procedure presents the methods for determining gamma emitting radioisotopes by high purity germanium detectors with high resolution spectrometry in specific media: air particulate filters, charcoal filters, milk, water, vegetation, soil/sediments, biological media, etc.

1.2 No chemical separation and purification procedures are required for gamma ray analysis. This is a nondestructive analysis, and after completion of the assay, the aliquot can be used for other analyses. However, to identify a specific target gamma emitter, chemical separation can be employed to isolate the desired gamma emitter(s) when other gamma emitters are present in high concentrations (see Section 1.4).

1.3 For water samples, a procedure is included to pre-concentrate the samples by evaporation for greater sensitivity. Advantages gained include:

- Permits the analysis of more than one liter of water in comparison to the restriction of one liter maximum for the standard Marinelli geometry
- Filter geometry is significantly a more efficient geometry since the sample is concentrated directly in front of and close proximity to the detector

1.4 For in-plant samples, a radiochemical method that includes chemical separation and purification is presented for determining the Ce-141 and Ce-144 activities of in-plant samples that have high concentrations of other gamma emitters.

2.0 SUMMARY OF METHOD

2.1 GEOMETRIES. Each sample to be assayed is put into a standard geometry for gamma counting such as 1-liter wrap-around Marinelli containers, 300 mL or 150

mL bottles, charcoal cartridge or 2-inch filter paper source geometries. Calibration and counting efficiencies of the gamma counting system for these geometries must have been determined with standard (known) radionuclide activity traceable to the National Institute of Standards and Technology.

To improve the sensitivity for measurement of gamma emitting radionuclides in water matrices, the sample can be evaporated from any known volume of one liter or greater and the residue collected in a standard filter geometry.

2.2 COUNTING. Samples are counted on large (>55 cc volume) germanium detectors connected to dedicated data acquisitions and data computation systems. All resultant spectra are stored electrically.

2.3 CALCULATION. The analysis of each sample consists of calculating the specific activities of all detected radionuclides or the detection limits from a standard list of nuclides. If water samples were pre-concentrated by evaporation, the specific activities or detection limits are divided by the volume of water represented in the filter geometry.

2.4 IN-PLANT SAMPLES FOR CE-141/CE-144. The radiochemical isolation of cerium reduces the gamma counting background and improves the sensitivity of analysis by a factor of 100 or more compared to direct gamma spectral analysis. This method is an adaptation of the Los Alamos Collected Radiochemical Procedures La-1721, 3rd edition, September 1967.

2.4.1 Solid samples are leached in acid, then filtered. Aqueous samples need no special preparation. Stable cerium carrier is added to the sample, then cerium is purified by precipitating first as fluoride, then as hydroxide, and finally as oxalate. The cerium oxalate is collected by vacuum filtration, dried and weighed to determine chemical yield. The precipitate is mounted on a nylon planchet and is covered with Mylar film.

2.4.2 The sample planchet is analyzed on a high resolution germanium detector. Ce-141 is inferred by its emission at 145 keV (48.4% gamma abundance). Ce-144 is inferred by its emission at 134 keV (10.8% gamma abundance). Results are calculated using a counting efficiency curve derived by analyzing multiple nuclide standards prepared in the same counting geometry.

3.0 DEFINITIONS

3.1 MSDS -- Material Safety Data Sheet

3.2 NIST -- National Institute of Standards and Technology

3.3 TBE-ES -- Teledyne Brown Engineering -- Environmental Services

4.0 HEALTH & SAFETY WARNINGS

4.1 At a minimum, personnel performing this procedure are required to wear the following protective equipment: laboratory coats, safety glasses, and disposable gloves.

4.2 Appropriate precautions, as specified in the Laboratory Radiation Protection Program (RPP) Manual, will be adhered to when handling radioactive material.

5.0 CAUTIONS -- N/A

6.0 INTERFERENCES -- N/A

7.0 PERSONNEL QUALIFICATIONS

Analysts performing this procedure must be trained, qualified, and certified in accordance with the TBE-ES Quality Control Manual IWL-032-365, Sections 2.2 through 2.9 inclusive.

Analysts in training may perform this procedure only under the direct supervision and observation of a senior technician certified to perform this procedure.

8.0 EQUIPMENT & SUPPLIES

8.1 Gamma-Ray Spectrometer consisting of high resolution germanium detectors connected to Nuclear Data acquisition and data computation systems. For each detector, 2048 channels (1 KeV per channel) or 4096 channels (0.5 KeV per channel) are assigned for pulse height analysis).

8.2 Standard sample container geometries, as appropriate:

- 1-Liter wrap around Marinelli containers
- 300 mL or 150 mL bottles
- 2-inch filter paper for air particulates
- Charcoal cartridges
- Nylon planchets (see Section 8.6)

8.3 Evaporation supplies for filter geometry

- Beakers: 1, 2, or 4-liter graduated
- Hot plate
- 2-inch stainless steel planchet
- Kimwipes, or equivalent
- Marking pen, to write on beaker and planchets
- Fiber sample trays
- Heat lamps, Heat hood
- Parafilm
- Paper envelope to store prepared planchets

- HNO_3, concentrated, in a dropping bottle
- Distilled water in a wash bottle
- Laboratory aerosol, dispensable by drop

8.4 In-Plant Samples Ce-141/Ce-144: Preparation

8.4.1 Aqueous

- Centrifuge tubes, 50-mL disposable
- Pipets, 10-mL disposable and 1-mL Eppendorf or glass volumetric
- Rubber pipet bulb
- pH paper
- Hydrochloric acid (HCl) to adjust pH if needed
- Deionized water, in a wash bottle
- Cerium carrier solution, standardized

8.4.2 Resins and other solids

- Beakers, 150-mL
- Gravity filtration apparatus and glass fiber filters
- Balance, analytical
- Hood
- Spatula
- Heat lamps or drying oven
- Poly bottles
- Pipets, disposable (10-mL)
- Centrifuge tube, 50-mL plastic and centrifuge tube rack
- Hydrochloric acid (HCl), 6 M: for resins
- Nitric acid (HNO_3), 8 M: for non-resin solids

- Cerium carrier solution, standardized

8.5 In-Plant Samples Ce-141/Ce-144: Chemical Separation & Purification

- Glass rod
- Hot water bath: 250 or 400-mL beaker half full of water on a moderate hot plate
- Deionized water, in a wash bottle
- Hydrofluoric acid (HF), conc. **!! Extreme Hazard !! No Skin Contact !!**
- Boris Acid (H_3BO_3), saturated solution
- Nitric acid (HNO_3), concentrated
- Sulfuric acid (H_2SO_4), 2 M
- Ammonium oxalate, saturated

8.6 In-Plant Samples Ce-141/Ce-144: Mounting the Precipitate

- Filter paper, 2.8 cm No. 42 ashless disc
- Vacuum filtration apparatus
- Petri dishes, 4-way partitioned
- Hot air oven
- Dessicator
- Balance, analytical
- Spatula
- Gummed labels
- Nylon planchets, 2-inch with Mylar film and nylon ring
- Scissors or razor blade
- Deionized water
- Ethanol

9.0 PROCEDURE

9.1 Detection Capability

Gamma ray spectroscopy, using a germanium detector, provides a high resolution method of distinguishing many gamma emitting nuclides in a single sample.

Each of the most commonly observed nuclides listed in the ensuing Table 9.1 has at least one gamma ray with a unique energy. Consequently, each nuclide in the Table may be identified in the presence of any or all of the others. The Table 9.1 also lists the nominal detectable limits for three of the standard sample container geometries.

Table 9.1A. Gamma Spectroscopy Detection Sensitivities[1] by High Resolution Germanium for Environmental Samples

Nuclide	Milk and Water[2] (pCi/L)	Animal, Fish, Soil Vegetation, etc. (pCi/g)	Filters (pCi/total filter)
Be-7	50	0.2	20
K-40	80	0.4	50
Mn-54	5	0.02	2
Co-58	5	0.02	2
Fe-59	10	0.04	3
Co-60	5	0.02	2
Zn-65	10	0.04	5
Zr-95-Nb-95	5	0.04	3
Ru-103	5	0.02	2
Ru-106	50	0.2	20
I-131	15	0.1	4
Cs-134	5	0.02	2
Cs-137	5	0.02	2
Ba-140/La-140	10	0.2	3
Ce-141	10	0.1	3
Ce-144	40	0.2	20
Ra-226	80	0.1	10
Th-228	10	0.02	10

[1]The detection limits are referenced to the count time and are based on two standard deviations of the background statistics.

[2]For water samples that have been pre-concentrated by evaporation onto a planchet, divide the values by the volume of water represented in the filter geometry.

Table 9.1B Ce-141/Ce-144 Minimum Detectable Activity (MDA)

Matrix	MDA[1]	Sigma Level[2]	Sample Volume	Chem Yield	Counting Interval (hour)	Counting Efficiency (cpm)	Backgd (cpm)
Ce-141[3]	$7x10^{-6}$ µCi/mL	4.66	10 mL	0.80	6		
	$4x10^{-3}$ µCi/g	4.66	2 g	0.80	6		
Ce-144[4]	$3x10^{-5}$ µCi/mL	4.66	10 mL	0.80	6		
	$2x10^{-4}$ µCi/g	4.66	2 g	0.80	6		

[1]Assumes there is no delay between collection and counting

[2]Sigma multiplier will be 4.66 unless otherwise specified by the client

[3] Half-life for Ce-141 is 32.5 days; therefore, delay in counting would significantly increase the MDA

[4] Half-life for Ce-144 is 284 days; therefore, delay in counting would increase the MDA

9.2 Sample Selection

9.2.1 Using the sample receipt form with the TBE-ES sample number, locate the sample (or sample group) in the sample receiving and storage room. Sign for the samples on the Receiving Room Log and return with them to the environmental laboratory.

9.2.2 Begin filling out the Radiochemical Preparation Logbook, entering the customer name, the sample numbers in order, the desired analyses, sample type, collection dates, the sample preparation date and the initials of the analyst.

After processing all samples within the sample group, begin filling out the Radiochemical Work Sheet – Gamma Spectroscopy. Using the laboratory logbook as a guide, fill in the customer name, collection date, sample type, analyst's initials, preparation date and aliquot used. Write the sample number of each sample in numerical order, and indicate the desired analysis (gamma spec).

9.2.3 Make an entry in the Gamma Spec laboratory logbook showing customer name, sample number, sample type, collection dates and desired analyses.

9.3 Sample Preparation

A laboratory sample for this SOP is defined as the material collected for analysis. A test source is prepared from laboratory sample material for purpose of determining its radioactive constituents. This is accomplished by putting the laboratory sample in a geometry suitable for the counting instrument, in this case a standard geometry that is user-friendly to the gamma spectrometer. The geometries used for the test source should be identical to the geometry of the calibration source, to the extent possible.

Important considerations in preparing test sources for gamma-ray spectrometry are geometry (shape), size, and homogeneity (uniformity) of source.

9.3.1 MILK AND WATER: Load environmental water and milk samples into 1-liter Marinelli containers.

9.3.2 WATER, larger volumes: For water volume exceeding the capacity of the 1-liter Marinelli, evaporate and mount the residue on a 2-inch stainless steel planchet, as follows:

9.3.2.1 Mark the sample number with a laboratory marking pen onto a clean, graduated 1, 2 or 4 liter beaker.

9.3.2.2 Shake the sample container to distribute any particulate matter evenly. Decant 1 liter or more of sample into the beaker and record the sample volume (and customer name and sample identification number) on the beaker with a marking pen.

9.3.2.3 Add approximately 1 mL concentrated HNO_3 to the sample from a dropping bottle. Place the beaker on a hot plate under the hood in the Gamma Preparation Room and set the hot plate for approximately 200°F temperature.

9.3.2.4 Evaporate the sample until the volume is reduced to 1-5 ml. Take care to reduce hot plate temperature as the sample volume

decreases in order to avoid loss by spattering from the beaker. Remove from hot plate.

9.3.2.5 Prepare a 2-inch stainless steel planchet for each water sample by first wiping it clean with a Kimwipe. Write sample number, customer name, and volume on the back of planchet with a marking pen.

9.3.2.6 Transfer the solution from each sample beaker to its correspondingly numbered planchet. Wash the beaker sparingly with deionized water using a wash bottle and collect the washings in the planchet.

9.3.2.7 Place the filled planchets in the fiber sample tray under heat lamps in the Light Hood. Add 1 drop of laboratory aerosol to each planchet. Evaporate to dryness. Remove and allow to cool.

9.3.2.8 Stretch parafilm over the planchet.

9.3.2.9 Insert the planchet into a new clean paper envelope on which the sample number, customer name, and volume have been inscribed.

9.3.3 AIR PARTICULATE FILTERS: Position two-inch diameter filter papers in front of the detector without change in geometry.

9.3.4 VEGETATION AND BIOLOGICAL MEDIA, e.g., food crops, fish, soils, etc.: Load into tared 300 or 150 mL plastic bottles or 1-liter Marinelli containers. Determine and record the net weight of the sample.

9.3.5 CHARCOAL CARTRIDGES: Position charcoal cartridges on the face only of the detector or on the face of the detector and up to four (depending on the number of charcoal cartridges in the weekly set) around

the cylindrical surface of the detector. If I-131 is observed, individually recount each cartridge, positioned on the face of the detector.

9.3.6 IN-PLANT SAMPLES FOR CE-141/CE-144: Refer to Appendix A for sample preparation and chemical separation and purification.

9.4 Calibration of Equipment For Gamma Ray Spectroscopy

The standard sample container geometries are the 1-liter Marinelli container, 300 mL and 150 mL polyethylene bottles, 2-inch diameter filter paper, a charcoal cartridge, and a 2" stainless steel planchet. Mixed gamma ray standards traceable to the National Institute of Science and Technology or best available are used to calibrate the various standard geometries.

Each standard is initially counted on each germanium detector and an efficiency versus energy curve is determined for each geometry for the energy range of approximately 50 KeV to 2 MeV.

On an on-going basis, once a week the check source standard is counted on each detector for energy, efficiency, calibration and resolution. A more detailed calibration procedure is described In PRO-042-44, "Calibration of Gamma Ray Spectrometers."

9.5 Sample Counting

9.5.1 Verify that the samples contain the same sample numbers as on the accompanying Radiochemical Work Sheet----Gamma Spec.

9.5.2 Write counting sequence numbers on the work sheet following the order that the sample numbers appear on the sheet. Begin with the number 1 if starting a new sample counting group; otherwise, use the number which follows the last sequence number assigned.

9.5.3 Write the counting start date and the number of the gamma-ray spectrometer used on the Radiochemical Work Sheet.

9.5.4 Measure an aliquot of sample in a standard geometry (one that has been calibrated). Record the amount on the work sheet.

9.5.5 Place the standard geometry with the sample aliquot on a shielded Ge(Li) detector and gamma count for a period of time that will meet the required sensitivity of measurement.

9.5.6 After the counting period the Nuclear Data system performs a peak search and identification. Print the gamma spectrum and/or store the spectrum on the appropriate computer-compatible device.

9.5.7 Calculate the radioactivity of the gamma emitters present in the sample.

9.5.8 For greater counting efficiency, reduce the size of the test source, as in Step 9.3.2. This will allow a greater amount of laboratory sample to be counted in a more favorable geometry.

9.6 Calculation of the Sample Activity or of the MDA

9.6.1 The Nuclear Data system performs a peak search and identification of all photopeak energies. The photopeak regions of the spectrum are integrated and the area under the baseline continuum is subtracted to determine the true photopeak area. Isotopes are identified by their appropriate photopeaks, and ratios to each other when more than one gamma photon is emitted by an isotope in the sample.

9.6.2 Radionuclide concentrations, A, (or detection limit based on the background if no peak is observed) are calculated by for a library of isotopes in pCi:

$$A = \frac{C}{2.22} \times BEV$$, where:

C = net count rate, cpm, in the peak area above the baseline continuum

B = the gamma ray abundance of the radionuclide being measured (gammas/disintegration)

E = detector efficiency (counts/gamma) for the particular photopeak energy being considered

V = volume (or mass) of sample aliquot used (liters or gram)

2.22 = conversion factor from dpm/pCi

9.6.3 The calculation includes the efficiency of the detector for each gamma ray energy and for the sample geometry, the percent abundance for the gamma ray, the sample size, length of count, and references the results to the collection date.

9.6.4 The results are typed by a line printer and then transcribed onto a computer compatible format and manual data entry or directly by tape into the Interim Report system.

10.0 DATA AND RECORDS MANAGEMENT

10.1 All laboratory data and ancillary information shall be documented in bound laboratory logbooks or appropriate worksheets in permanent ink. Appropriate supervisory personnel shall review logbook entries and worksheets as required by the TBE-ES Quality Assurance Program.

10.2 Corrections to recorded data in logbooks or on worksheets shall be noted by drawing through the incorrect data with a single line and recording the date of the correction and the initials of the person making the correction. The correct data will be recorded in an unambiguous location in the immediate proximity of the incorrect data.

11.0 QUALITY CONTROL & QUALITY ASSURANCE

11.1 Sample duplicates shall be run to meet client requirements or, at a minimum, as required by the TBE-ES laboratory QC program.

11.2 Analysis blank and spikes shall be run to meet client requirements or, at a minimum, as required by the TBE-ES laboratory QC program.

11.3 A matrix spike consisting of a sample spiked with an appropriate standard (NIST traceable when possible) shall be run to meet client requirements.

11.4 If any batch control sample fails laboratory established quality control criteria (TWL-032-365, Section 9.1.2) or fails to meet specific client contract requirements, the samples comprising the controlled batch shall be reanalyzed.

11.5 Alpha and beta counters used in this analysis shall be controlled as set out in PRO-032-27.

11.6 The Laboratory Operations Manager or designee will interpret control charts. Five percent of the plotted values are expected to fall outside the $+2\sigma$ precision band on statistics alone. If a check source falls outside the $\pm 3\sigma$ precision band, another reading of the check source shall be made. If the second reading also falls outside the $\pm 3\sigma$ band, the counter is judged to be out of control & shall be taken out of service.

11.7 A counter control chart may show trends without being out of control by the above criteria. The Laboratory Operations Manager or a qualified designee shall interpret these trends and take corrective action. It is good practice to investigate trends which approach a 2 sigma control line. The instrument maintenance log shall be used to document the occurrence and interpretation of trends, and any corrective action taken.

11.8 When a counter is out of control, the Laboratory Operations Manager or the person he/she designates will examine the check source for defects. Re-

calibration of the counter may be necessary. New calibrations should be compared to previous calibrations to identify major changes in counter operations. Corrective action shall be documented in the maintenance log for that instrument.

11.9 When a counter is out of control for a given analysis, it may not be used for that analysis. A label must be placed on the instrument indicating its status.

12.0 REFERENCES

12.1 EPA-600/4-80-032, Prescribed *Procedures for Measurement of Radioactivity in Drinking Water*, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, August 1980. Procedures, except SM-19-7110B, are based on Method 901.1 "Gamma Emitting Radionuclides," augmented for non-aqueous matrices by TBE-ES technical personnel.

A.1 In-Plant Sample Ce-141/Ce-144 Preparation and Chemical Separation and Purification

A1.1 Sample Preparation

This section describes how samples are aliquoted and prepared for chemical separations. Wear a laboratory coat, disposable gloves and safety glasses while carrying out the steps below.

A1.1.1 Aqueous Samples

A1.1.1.1 Write the Teledyne sample number or login number on a 50 ml disposable plastic centrifuge tube using a laboratory marking pen. Also write the analysis on the tube.

A1.1.1.2 Shake the sample container to mix, then withdraw 10 ml of sample using a disposable pipet and rubber bulb. Transfer the liquid to the labeled sample tube. Different aliquots may be used according to sample availability or desired detection limit. Write the measured aliquot in the laboratory data book.

A1.1.1.3 Test the sample for acidity by dipping a stirring rod into it, then touching to pH paper. If the sample is not acidic, add several drops HCl from a dropping bottle, then stir and test again. Add deionized water from a wash bottle to bring the volume near 20 ml.

A1.1.1.4 Add 1.00 ml of standardized cerium carrier solution (nominally 10 mg Ce/ml) to the sample using an Eppendorf pipet or a glass volumetric pipet. Proceed to Section 9.4 for chemical separation procedures.

A1.1.2 **Resin Samples**

A1.1.2.1 Resin samples are leached in 6M HC1 without heating, then are filtered. The filtrate is diluted to 100 ml, then measured aliquots are taken for various analyses.

A1.1.2.2 Write the Teledyne sample number on a new 150 ml beaker. Obtain the tare weight of the beaker using the analytical balance. Record this figure in the laboratory notebook along with the client name, sample number and sample type.

A1.1.2.3 Working in a hood, use a laboratory spatula to scoop a representative aliquot of the sample from its container into the labeled beaker. The size of the aliquot will vary according to sample availability, overall sample activity and desired detection limit.

A1.1.2.4 Reweigh the sample beaker and record this gross weight in the laboratory notebook. Subtract the beaker tare weight and record the sample aliquot weight. Also note whether the sample is dry or wet.

NOTE: If the sample is wet and client wants results on a dry basis, place the sample beaker under heat lamps or in a drying oven and dry it before taking the final weight. In rare cases the client wants results on both a wet and dry basis. In these cases take a final weight on both a wet and a dry basis.

A1.1.2.5 Working in a hood, pour 6 M HC1 from a beaker into the sample beaker, covering the sample with about 75 ml of liquid. Allow the sample to leach for 2-5 hours without heating.

A1.1.2.6 Fold a glass fiber filter in quarters and place in a plastic funnel. Place the funnel in the mouth of a new poly bottle which has been marked at an appropriate volume level and which has been labeled

with the client name and sample number. Filter the sample into the bottle, washing the resin with 6 M HCl and with water. Discard the filter and resin into a radioactive solid waste container.

A1.1.2.7 Add deionized water to the bottle, filling to the volume mark. Screw on the cap and shake to mix.

A1.1.2.8 Use a disposable pipet to draw a measured aliquot (usually 10 ml) from the sample bottle, transferring it to a 50 ml plastic "C" tube labeled with the sample number and the analysis. Record the measured aliquot and the dilution information in the laboratory notebook.

A1.1.2.9 Using a calibrated fixed or adjustable volume pipet, add 1.00 ml standardized cerium carrier (nominally 10 mg Ce/ml) to the sample. Proceed to Section 9.4 for chemical separation.

A1.1.3 Other Solid Samples

Solid samples other than resin are leached in 8M HNO_3 with heating, then are filtered. The filtrate is diluted to an appropriate volume, then measured aliquots are taken for various analyses.

A1.1.3.1 Write the Teledyne sample number on a new 150 ml beaker. Obtain the tare weight of the beaker using the analytical balance. Record this figure in the laboratory notebook along with the client name, sample number and sample type.

A1.1.3.2 Working in a hood, use a laboratory spatula to scoop a representative aliquot of the sample from its container into the labeled beaker. The size of the aliquot will vary according to sample availability, overall sample activity and desired detection limit.

A1.1.3.3 Reweigh the sample beaker and record this gross weight in the laboratory notebook. Subtract the beaker tare weight and record the sample aliquot weight. Also note whether the sample is dry or wet.

NOTE: If the sample is wet and client wants results on a dry basis, place the sample beaker under heat lamps or in a drying oven and dry it before taking the final weight. In rare cases the client wants results on both a wet and dry basis. In these cases take a final weight on both a wet and a dry basis.

A1.1.3.4 Working in a hood, pour 8 M HNO_3 from a beaker into the sample beaker, covering the sample with about 50 ml of liquid. Place the beaker on a moderate hot plate (setting near 2) and allow the sample to leach for 2 hours or longer, adding deionized water as necessary. Remove the sample beaker and allow to cool.

A1.1.3.5 Fold a glass fiber filter in quarters and place in a plastic funnel. Place the funnel in the mouth of a new 150 ml poly bottle which has been marked at an appropriate volume level and which has been labeled with the client name and sample number. Filter the sample into the bottle, washing the solids 8 M HNO_3 and with water. Discard the filter and resin into a radioactive solid waste container.

A1.1.3.6 Add deionized water to the bottle, filling to the volume mark. Screw on the cap and shake to mix.

A1.1.3.7 Use a disposable pipet to draw a measured aliquot (usually 10 ml) from the sample bottle, transferring it to a 50 ml plastic centrifuge tube labeled with the sample number and the analysis. Record the measured aliquot and the dilution information in the laboratory notebook.

A1.1.3.8 Using a calibrated pipet, add 1.00 ml standardized cerium carrier (nominally 10 mg Ce/ml) to the sample. Proceed to Section 9.4 for chemical separations.

A1.2 Chemical Separation and Purification

This section describes the isolation of cerium by precipitating the fluoride, then the hydroxide, then the oxalate. Wear a laboratory coat, safety glasses and disposable gloves while carrying out the following steps. **Exercise extreme caution when working with HF.**

A1.2.1 Precipitation of CeF3

A1.2.1.1 Add deionized water from a wash bottle to the sample tube to bring the liquid level to near 20 ml.

A1.2.1.2 Wearing a laboratory coat, disposable gloves and a face shield or safety glasses with side shields, and **exercising extreme caution**, add approximately 3 ml concentrated HF from its plastic squeeze container to the sample tube. Stir with a glass rod, but do not leave the rod in the sample tube. Using a deionized water wash bottle, rinse the rod while withdrawing it from the tube.

A1.2.1.3 Make a hot water bath by partly filling a 250 ml or 400 ml beaker with tap water and heating on a moderate hot plate (setting near 3). Place the sample tubes in the bath to precipitate CeF_3. Remove sample tubes from the beaker and place in a rack to cool.

A1.2.1.4 Equalize the liquid levels in the sample tubes and centrifuge for 10 minutes. Decant and discard the supernate into a radwaste beaker.

A1.2.1.5 Rinse the precipitate by adding approximately 5 ml deionized water to the sample tube from a wash bottle. Stir with a glass rod, then rinse the rod while withdrawing it from the tube. Equalize liquid

levels and centrifuge again for 10 minutes. Decant and discard the supernate into a radwaste beaker.

A1.2.1.6 Dissolve the CeF_3 precipitate by adding 1 ml saturated boric acid (H_3BO_3) solution, then 1 ml concentrated HNO_3. Stir (and heat in a water bath if necessary) until the precipitate dissolves.

A1.2.2 Precipitation of $Ce(OH)_3$

A1.2.2.1 Add deionized water from a wash bottle to the sample tube and bring the liquid level to near 20 ml. Stir and add concentrated NH_4OH from a dropping bottle until the solution is basic and particles of $Ce(OH)_3$ form. Do not heat.

A1.2.2.2 Centrifuge the sample for 10 minutes immediately after the preceding step. Discard the supernate into a radwaste beaker.

A1.2.2.3 Add 5 ml 2M H_2SO_4 to the sample tube and stir with a glass rod to break up the precipitate. Add 5 ml deionized water and heat in a water bath for a half hour or longer to dissolve the precipitate. Some particles may remain undissolved.

A1.2.3 Precipitation of $Ce_2(C_2O_4)_3$

A1.2.3.1 With the sample tube still in the water bath from the previous step, add approximately 20 ml saturated ammonium oxalate from a beaker. Stir, then continue heating in the water bath for 20 minutes or longer to precipitate cerium oxalate.

A1.2.3.2 Remove the sample tube and place it in a rack to cool. Proceed to section 6.0 to mount this final precipitate for gravimetric yielding and gamma counting.

A1.3 Mounting the Precipitate

A1.3.1 Prepare a 2.8 cm No. 42 ashless filter paper disc for each sample by mounting it on a vacuum filtration apparatus and rinsing with deionized water and ethanol.

A1.3.2 Place the prepared discs in 4-way partitioned petri dishes which have been marked with sequence numbers (one number per partition, beginning with 1). Write corresponding sequence numbers beside each sample number entry in the laboratory data book. The sequence number indicates the correspondence between a filter and the sample which will be mounted on it.

A1.3.3 Place petri dishes containing prepared filters in an approximately 100°C hot air oven for 10 minutes or longer to dry. Remove petri dishes and allow to cool in a desiccator.

A1.3.4 Weigh the filter discs on the analytical balance using a clean spatula to handle them. Record this tare weight beside the corresponding sequence number and sample number in the laboratory data book. Take care to replace each filter after weighing in the numbered petri dish partition from which it came.

A1.3.5 Using a laboratory spatula, take the tared filters in sequence number order and transfer to the vacuum mounting apparatus. Wet with deionized water. Using the laboratory data book to establish the correspondence between sequence number and sample number, filter each sample from its centrifuge tube onto its corresponding filter disc.

A1.3.6 Rinse precipitate on filter with deionized water and then with ethanol. Transfer each filter from vacuum mounting apparatus back to the numbered petri dish partition from which it came. Place petri dish in a 105-120°C hot air oven for 10 minutes or longer.

A1.3.7 Remove petri dish and allow to cool in desiccator. Weigh filters on the analytical balance within 5 minutes of removing from the desiccator. Write the weights beside the corresponding sequence numbers in the laboratory data book. Return each filter to its original partition in the petri dish.

A1.3.8 Write a gummed label for each sample designated Ce-141, Ce-144, or both. All labels must contain the sample number and customer. Fix each label to the back of a nylon planchet.

A1.3.9 Using the laboratory data book to establish the correspondence between sample number and sequence number, transfer each filter to its planchet and fix in place with a 2-inch piece of Mylar film and a nylon ring. Trim excess Mylar with scissors or a razor blade.

A1.3.10 Subtract filter tare weight from final weight for each sample. Write the difference (mount weight) in the laboratory data book. Divide the mount weight by the corresponding carrier yield figure (written on the cerium carrier flask) to obtain chemical yield. Enter the cerium yield percentage in the laboratory data book.

A1.3.11 Using the laboratory data book as a guide, begin filling out a Radiochemical Work Sheet for each sample. Enter sample number, company name, analysis (Ce-141, Ce-144, or both), collection date, aliquot used, sample type, chemical yield, and analyst's initials.

A1.3.12 Submit finished planchets and work sheets to the Counting Room for radioassay.

A1.3.13 Proceed to: Section 9.4.

TELEDYNE BROWN ENGINEERING ENVIRONMENTAL SERVICES

Procedure TBE-3003

Calibration and Control of Alpha and Beta Counting Instruments

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Calibration and Control of Alpha and Beta Counters

1.0 SCOPE & APPLICABILITY

1.1 Calibration of counters used to determine α and/or β activity is performed to establish the counting efficiency of the instrument. The counting efficiency establishes the number of disintegrations registered in the detector and electronics of a counting instrument compared to the number emitted by the source. Efficiencies are plotted as a function of sample mass residue.

Counters used for gross alpha and gross beta determination are calibrated by counting aliquots of radioactive standard solutions which have been evaporated together with varying amounts of chemical salts to derive self-absorption curves. Beta counters used for analyzing chemically separated species are calibrated by precipitating a standard solution of the desired isotope along with an inert carrier. The standard is mounted in the same geometry as a sample and is counted to derive counter efficiency.

1.2 Routine control of alpha and beta counters is established by counting check sources and blanks. Check source results are plotted on control charts inscribed with lines showing the mean, ±2s, and ±3s precision levels. Background readings are recorded on control charts inscribed with lines showing the maximum and minimum acceptable levels.

2.0 SUMMARY OF METHOD

2.1 Automatic proportional counters are used for measurement of gross alpha and gross beta analyses for all sample media. The preparation of different sample types is described in separate procedures. The final "mounting" is in a 2-inch steel planchet, which is positioned in the counting instrument. Alpha and beta standards are also prepared and measured in 2-inch planchets.

2.2 Alpha standards are prepared by diluting Am-241 or Th-230 standard solutions (traceable to NIST) and by evaporating measured aliquots in planchets. The efficiency of the

instrument is then determined by dividing the cpm measured by the known dpm value. Routine measurements of check sources are plotted on control charts.

2.3 Absorption (called self-absorption) of alphas by the sample mass in the planchet (thus reducing the count rate) is determined as follows.

A set of alpha standards of known activity of Am-241 or Th-230 is prepared by adding known amounts of standard to 2-inch planchets. Chlorides are converted to nitrates in all aliquots by evaporating with concentrated HNO_3 in glass beakers. Each of the prepared standard planchets is evaporated with varying amounts of Na_2CO_3 salt. This salt has been shown to have the same self-absorption properties as finely divided silt. Residue weights between 0 and 1.5 grams are distributed in 2-inch planchets. From the radiometric determinations a self-absorption curve is constructed with the apparent instrument efficiency monotonically decreasing with increasing "mount weight." An algebraic expression of this curve is stored in a computer and is referenced during data reduction to yield the counting efficiency corresponding to the measured mount weight.

2.4 Determinations of beta counting efficiencies and self-absorption curves are similar to the methods used for alpha. Beta standards are prepared by diluting Cs-137 standard solutions (traceable to NIST). Measured aliquots of these solutions are evaporated in planchets to prepare standards in the same geometry used for counting samples. Self-absorption curves are prepared by evaporating standard solutions with varying weights of Na_2CO_3 salts. Beta check sources are counted routinely and the results are plotted on control charts.

3.0 DEFINITIONS

3.1 MSDS -- Material Safety Data Sheet

3.2 NIST -- National Institute of Standards and Technology

3.3 TBE-ES -- Teledyne Brown Engineering -- Environmental Services

4.0 HEALTH & SAFETY WARNINGS

4.1 At a minimum, personnel performing this procedure are required to wear the following protective equipment: laboratory coats, safety glasses, and disposable gloves.

4.2 When using or preparing reagents that consist of concentrated caustic or acid materials or solutions producing excessive heat, the analyst is required to wear an apron over his/her laboratory coat and an appropriate face shield over his/her safety glasses.

4.3 All potentially hazardous or hazardous reagents must be prepared and/or used only in a hood.

4.4 MSDSs (Material Safety Data Sheets) are available in locations convenient to the laboratories and in the Safety Manager's Office. Refer to these for other specific safety instructions for chemicals and reagents.

4.5 Appropriate precautions, as specified in the Laboratory Radiation Protection Program (RPP) Manual, will be followed when handling radioactive material.

5.0 CAUTIONS

5.1 Samples which appear to be hygroscopic are dried again under heat lamps just prior to counting.

6.0 INTERFERENCES

6.1 Chlorides present in a sample are incompatible with the stainless steel planchet.

6.2 Heavy concentrations of organic material may interfere with analysis via self absorption

7.0 PERSONNEL QUALIFICATIONS

7.1 Analysts performing this procedure must be trained, qualified and certified in accordance with the TBE-ES Quality Control Manual IWL-032-365, Sections 2.2 through 2.9

inclusive. Procedure specific training documentation for designated analysts is maintained in the QA office.

7.2 Analysts in training may perform this procedure only under the direct supervision and observation of a senior technician certified to perform this procedure.

8.0 EQUIPMENT & SUPPLIES

8.1 Alpha and Beta Counter Calibration

- Each α and β counting instrument, e.g., low level gas flow proportional counters
- Work sheets
- Aerosol solution
- Deionized water
- Volumetric glassware, as needed to obtain standard activities near 1,000 dpm
- Stainless steel counting planccets, 2" and 1"
- Heat lamps
- Dessicator
- Analytical balance
- NIST-traceable standard solutions, with certificates: Am-241 (for alpha), Cs-137 (for beta) ---- others can be used to accommodate client requirements
- Chemical salt solution, e.g., 0.1 g/mL sodium carbonate (Na_2CO_3)

8.2 Beta Counter Calibration

- Each beta counter instrument, e.g., low level gas flow proportional counters
- Deionized water
- Glass stirring rod
- Volumetric glassware, as needed to obtain standard activities near 1,000 dpm
- Centrifuge tubes
- Filter discs
- Nylon planchet
- Mylar to cover prepared planchets
- NIST-traceable standard solution of desired nuclide, with certificate

- Carrier, as defined in the radiochemical procedure
- Other reagents, as needed for preparation of the final precipitate, as stated in the respective radiochemical procedure (e.g., for I-131, precipitation of palladium iodide section of the radioiodine procedure)

9.0 PROCEDURE

9.1 Calibration of Gross Alpha and Gross Beta Counters

The laboratory manager or a person he designates will perform calibrations. This is done once per year and when control charts indicate efficiency change.

9.1.1 Obtain an NIST traceable standard solution of Am-241 (for alpha) or Cs-137 (for beta). Other nuclides may be used as standards in order to accommodate clients' requirements.

9.1.2 Dilute the standard quantitatively in volumetric glassware to obtain an activity near 1000 dpm in a few ml volume. Keep a record of all dilutions in the Standards Log along with copies of the certificate. Entries should be dated and initialed.

9.1.3 Label 10 or more stainless steel counting planchets and obtain their tare weights using an analytical balance. Pipet equal aliquots of diluted standard (approximately 1000 dpm) into each.

9.1.4 Add varying amounts of chemical salt solution (e.g., Na_2CO_3, 0.1 g/mL) to the planchets such that a residue weight range of 0 – 0.15 g (or greater) is represented. Add a drop of aerosol solution to each planchet, then add deionized water to bring the liquid level to that usually obtained when processing samples.

9.1.5 Evaporate the filled planchets to dryness under heat lamps. Allow them to cool and store them in a desiccator. Re-weigh to determine residue mass for each planchet and record these figures. To determine whether moisture has been assimilated by the residue, the weighing operation may be repeated at times during the calibration period

9.1.6 Count the evaporated standards for approximately 50 minutes (or 10,000 counts) each on the instrument to be calibrated. Determine dpm of the standards at count time by applying the decay factor from reference date to count date. Calculate counter efficiency by subtracting background count rate (cpm) from the count rate of the standard (cpm) and then dividing by the activity of the standard (dpm).

9.1.7 Record planchet tare weights, final weights, and residue masses on a work sheet. Also record the identity of the radioactive standard used and the aliquots taken. Include a copy of this sheet in the maintenance log of the instruments calibrated. Also include the instrument printout from the standards, and the calculated efficiency for each residue mass used.

9.1.8 Plot counting efficiency against residue mass. Fit a mathematical function to the data and draw this interpretive curve on the plot. Record the plot and the equation of the function in the instrument maintenance log. Implement the equation in the computer programs used in data reduction such that a sample activity will be calculated using a counting efficiency appropriate to its residue mass.

9.2 Calibration of Beta Counters Used for Nuclide Analysis

The laboratory manager or a person he designates will perform calibrations. Step 9.2.11 (determining Cs-137 efficiency and scaling to obtain values for other nuclides) must be performed at least once a year and when control charts indicate efficiency changes. Other steps (preparing a new standard of a nuclide) must be performed once every 3 years.

9.2.1 Obtain an NIST traceable standard solution of the nuclide and a copy of its certificate.

9.2.2 Dilute the standard quantitatively in volumetric glassware to obtain an activity near 1000 dpm in a few ml volume. Keep a record of all dilutions in the standards log along with a copy of the certificate. Entries should be dated and initialed.

9.2.3 Normally 4 or more standard mounts are prepared at a time. Pipet aliquots of diluted standard into centrifuge tubes that are numbered sequentially. The aliquots should represent near 1,000 dpm at the anticipated count time. Record the aliquot used on a calibration data sheet.

9.2.4 Refer to the radiochemical procedure for the nuclide being tested. Using a pipet, add standardized carrier to each tube. The quantity of carrier may be diminished from that indicated in the procedure so that the resulting standard will contain a precipitate mass which is typical for the analysis. For example, if I-131 yields are typically 75%, three-fourths of the usual iodide carrier volume may be used in preparing the I-131 standard. Record the precise volume used and the carrier concentration.

9.2.5 Add approximately 5 ml deionized water to each tube, and stir with a clean glass rod. Refer to the radiochemical procedure to find instructions for final precipitation. For example, for preparing an I-131 standard refer to the "precipitation of palladium iodide" section of the radioiodine procedure.

9.2.6 Carry out the final precipitation described in the procedure. Collect the precipitate on a filter disc and determine gravimetric yield as indicated. Mount on a nylon planchet and cover with Mylar according to the procedure. Record filter tare weights, final weights, and chemical yields on the calibration data sheet.

9.2.7 Count the prepared standards for 10 minutes in a single counter to determine uniformity. Divide each standard's count by its yield and compare the quotients. Reject a standard if its count differs from the rest of the group by 3 standard deviations ($3\sqrt{N}$) or more.

9.2.8 Count a standard in each detector to be calibrated for a minimum of 10,000 counts. Record counter number, standard number, counting time, counts, and counter background. Determine dpm of the standard by applying decay and yield

corrections to the activity derived by dilution. Divide net cpm by standard dpm to derive efficiency. Record all data in the maintenance log of the instrument.

9.2.9 Determine Cs-137 efficiency for the counter by carrying out step 9.2.8 using Cs-137 standards.

9.2.10 Calculate and record an efficiency ratio of the nuclide under test to that of Cs-137. Post a revised list of counter efficiencies for use in data reduction.

9.2.11 If a counter must be recalibrated when standards for various nuclides are not available, calibrate for Cs-137 (Step 9.2.9) and apply the most recent efficiency ratios to obtain values for the other nuclides. Record all details of calibration and efficiency determination in the instrument maintenance log. Post a revised list of counter efficiencies for use in data reduction.

10.0 DATA AND RECORDS MANAGEMENT

10.1 All laboratory data and ancillary information shall be documented in bound laboratory logbooks or appropriate worksheets in permanent ink. Appropriate supervisory personnel shall review logbook entries and worksheets as required by the TBE-ES Quality Assurance Program.

10.2 Corrections to recorded data in logbooks or on worksheets shall be noted by drawing through the incorrect data with a single line and recording the date of the correction and the initials of the person making the correction. The correct data will be recorded in an unambiguous location in the immediate proximity of the incorrect data.

10.3 Each standard lists the manufacturer of the nuclides, the certificate numbers, nuclides, and activities of the nuclides at a reference time. The counting of the standards are recorded in a log containing the sequential listing of every sample (including backgrounds and standards) by detector serial number. The start date and time and duration of count are recorded. The spectrum is stored and transferred to various data storage media for archival purposes.

11.0 QUALITY CONTROL & QUALITY ASSURANCE

11.1 Control of Gross Alpha and Gross Beta Counters

Control of gross alpha and gross beta counters is established by analyzing check sources and blanks, comparing the readings to their acceptance regions and plotting them on control charts. The check source for monitoring gross beta performance is one of the Cs-137 mounts described in Section 11.2. The check source for monitoring gross alpha performance is the same as the evaporated Am-241 standard described in Section 9.1. Blanks are plain 2-inch diameter stainless steel planchets of the kind used for samples.

11.1.1 Include a blank planchet in each group of samples counted. Also include the check source (alpha and/or beta, as appropriate) in each group. Check sources must be read after changing counting gas cylinders. Plateau must be run (Department of Energy requirement).

11.1.2 Record the count rates of blanks and check sources in the monthly log. Gross alpha backgrounds are acceptable if they are in the range 0.02-0.20 cpm. Gross beta backgrounds are acceptable if they are in the range 0.5-3.0 cpm. Compare check source readings to the control limits written in red at the top of the log page to determine whether they are in the acceptance region.

NOTE: The check source acceptance region is the ±3 sigma band calculated from check source readings taken near the calibration date. The acceptance region is corrected for radioactive decay each month to derive control limits that are written in red in the monthly log.

11.1.3 If check source or blank readings fall outside the acceptance region, repeat the measurement. If the second reading also falls outside the acceptance region the counter is judged to be out of control for that analysis.

11.1.4 When a counter is out of control for a given analysis (alpha or beta) it may not be used for that analysis. Samples counted after the previous acceptable reading

must be recounted on a different counter for verification. A label must be placed on the instrument indicating its status if it is not in use.

11.1.5 When a counter is out of control, the laboratory manager or the person he designates may examine the check source for defects. He may:

- Investigate the electronic system for malfunctions
- Adjust the detector potential or re-analyze the plateau
- Dismantle the detector and replace or clean elements
- Determine a new efficiency for the counter and revise the control levels.

11.1.6 Corrective action must be documented in the maintenance log for that instrument.

11.1.7 Plot check source and blank results on their respective control charts after each measurement. The check source plots show the mean, ±2s and ±3s control lines derived from check source readings taken near the calibration date (see note above). The background plots show the control limits stated in Section 11.1.2.

11.1.8 The laboratory manager or a person he designates will interpret control charts. A counter control chart may show trends without being out of control by the above criteria. The laboratory manager or his designee will interpret these trends and take corrective action according to his judgment. It is good practice to investigate trends which approach a 2 sigma control line. The instrument maintenance log should be used to document the occurrence and interpretation of trends, and any corrective action taken.

11.2 Control of Beta Counters Used for Nuclide Analysis

Control of beta counters is established by analyzing check sources and blanks, comparing the readings to their acceptance regions, and plotting them on control charts.

Check sources are 1-inch diameter planchets containing Cs-137 deposits. A separate check source is assigned to each counter. They are not used to derive efficiencies but serve to monitor consistency of counter performance.

There are 2 kinds of blanks. The "regular" blank consists of a plain paper filter disc mounted with Mylar film on a 1-inch diameter planchet. The "absorber" blank consists of a fiberglass filter disc mounted in the same way. This latter blank is counted with an 80 mg/cm2 aluminum absorber for deriving backgrounds for the Sr-89 analysis.

11.2.1 Check source readings are taken each working weekday and after each change of counting gas. Insert the appropriately labeled source into each counter and set the timing interval for 10 minutes. After counting is complete, record the cpm beside the corresponding date in the monthly log. Compare check source readings to the control limits written in red at the top of the log page to determine whether they are in the acceptance region.

NOTE: The check source acceptance region is the ±3 sigma band calculated from check source readings taken near the calibration date. The acceptance region is corrected for radioactive decay each month to derive control limits that are written in red in the monthly log.

11.2.2 If a check source reading falls outside the acceptance range, repeat the measurement. If the second reading also falls outside the acceptance range, the counter is judged to be out of control.

11.2.3 Count regular and absorber blanks overnight to determine counter backgrounds. Read and record these as often as the counting load permits, generally once or twice per week. In addition, count a regular 200 minute blank every week (usually on Monday). If its 2-sigma counting error band does not include the previous overnight regular blank background, a new overnight blank must be counted on that night. Regular blank readings are acceptable if they are in the range of 0.1 to 1.5 cpm. The laboratory manager may choose not to use a counter if its background is in the high part of the acceptance range.

11.2.4 When a counter is out of control, it may not be used for analyzing samples. Samples counted after the previous acceptable reading must be recounted on a

different counter for verification. A label must be placed on the instrument to indicate its status if it is not in use.

11.2.5 When a counter is out of control, the laboratory manager or the person he designates may examine the check source for defects. He may:

- Investigate the electronic system for malfunctions
- Adjust the detector potential
- Dismantle the detector and replace or clean elements
- Determine a new efficiency for the counter and revise the control levels

11.2.6 Work on the counting system is documented in the maintenance log.

11.2.7 Plot check source and blank results on their respective control charts after each measurement. The check source plots show the mean, ±2s and ±3s control lines derived from check source readings taken near the calibration date (see note above). The background plots show the control limits stated in Section 11.2.3.

11.2.8 The laboratory manager or a person he designates will interpret control charts. A counter control chart may show trends without being out--of--control by the above criteria. The laboratory manager or his designee will interpret these trends and take corrective action according to his judgment. It is good practice to investigate a trend which approaches a 2--sigma control line. The instrument maintenance log should be used to document the occurrence and interpretation of trends, and any corrective action taken.

12.0 REFERENCES

12.1 There are no external references that pertain to this procedure.

TELEDYNE BROWN ENGINEERING ENVIRONMENTAL SERVICES

Procedure TBE-3001

Calibration and Control of Gamma-Ray Spectrometers

Rev. 0

Date of Original Issue: 03/29/02

Approved by:

Keith O. Jeter Date 3/29/02
Keith O. Jeter, Operations Manager

Martin R. Keller Date 3/29/02
Martin R. Keller, Quality Assurance Manager

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CALIBRATION AND CONTROL OF GAMMA-RAY SPECTROPHOMETERS

1.0 SCOPE & APPLICABILITY

1.1 Gamma spectroscopy is used to identify and quantify gamma-emitting radionuclides. Advantages of this technique include:

- Ability to simultaneously measure more than one radionuclide
- Potential for quick turnaround times
- Minimization of sample manipulation
- Option for nondestructive measurements when sensitivity and interferences are not at issue
- Option to employ chemical separation, purification, or size reduction (e.g., evaporation, co-precipitation, compression, or ashing) procedures to enable greater counting efficiencies
- Ability to use relatively large sample sizes to reduce measurement uncertainty associated with subsampling at the laboratory.

1.2 Calibration of the gamma-ray spectrometer establishes the counting efficiency of the instrument. These counting efficiencies are specific to the radionuclide (or energy), the geometrical relationship between the source and detector (e.g., source-to-detector distances), and characteristics of the source material, particularly those that affect absorption and scattering of the radiation, such as homogeneity, density. Several different calibrations on a given detector may be needed to accommodate requirements for detection sensitivity, specificity, and media.

1.3 For this procedure, five sample geometries are used for the analysis of various media: a one liter plastic Marinelli, a two inch diameter filter geometry, 300 ml and 150 ml plastic bottles, a charcoal cartridge and a 2" stainless steel planchet geometry.

2.0 SUMMARY OF METHOD

2.1 Calibration standards are prepared for each of five sample geometries, then all gamma detectors are calibrated with each standard once per year or after a detector is repaired or newly purchased. Once a week the check source standard is counted on each detector for efficiency, energy and resolution checks.

2.2 Calibration verification shall include checks on counting efficiency and the relationship between channel number and gamma ray energy performed on a day of use basis along with performance checks on peak resolution using the full width of peak at half maximum (FWHM). Background measurements shall be performed each day of use, on at least a monthly basis.

2.3 Radioactive standards for the yearly recalibrations of efficiencies are prepared in the same geometries. Radionuclide reference standards traceable to the National Institute of Standards and Technology are used.

3.0 DEFINITIONS

3.1 MSDS -- Material Safety Data Sheet

3.2 NIST -- National Institute of Standards and Technology

3.3 TBE-ES -- Teledyne Brown Engineering -- Environmental Services

4.0 HEALTH & SAFETY WARNINGS

4.1 At a minimum, personnel performing this procedure are required to wear the following protective equipment: laboratory coats, safety glasses, and disposable gloves.

4.2 When using or preparing reagents that consist of concentrated caustic or acid materials or solutions producing excessive heat, the analyst is required to wear an apron over his/her laboratory coat and an appropriate face shield over his/her safety glasses.

4.3 All potentially hazardous or hazardous reagents must be prepared and/or used only in a hood.

4.4 MSDS's (Material Safety Data Sheets) are available in locations convenient to the laboratories and in the Safety Manager's Office. Refer to these for other specific safety instructions for chemicals and reagents.

4.5 Appropriate precautions, as specified in the Laboratory Radiation Protection Program (RPP) Manual, will be followed when handling radioactive material.

5.0 CAUTIONS

5.1 The primary precaution in preparing standards is to maintain the activity level below that which would create more than a five percent "dead time" effect in the electronics.

5.2 The temperature of the room is maintained at constant temperature with thermostatically controlled air conditioning and heating units.

6.0 INTERFERENCES-N/A

7.0 PERSONNEL QUALIFICATIONS

7.1 Analysts performing this procedure must be trained, qualified, and certified in accordance with the TBE-ES Quality Control Manual IWL-032-365, Sections 2.2 through 2.9 inclusive. Procedure specific training documentation for designated analysts is maintained in the QA office.

7.2 Analysts in training may perform this procedure only under the direct supervision and observation of a senior technician certified to perform this procedure.

8.0 EQUIPMENT & SUPPLIES

8.1 Gamma-Ray Spectrometer consisting of high resolution germanium detectors connected to Nuclear Data acquisition and data computation systems. For each

detector, 2048 channels (1 KeV per channel) or 4096 channels (0.5 KeV per channel) are assigned for pulse height analysis).

8.2 Standard sample container geometries, as appropriate:

- 1-Liter wrap around Marinelli containers
- 300 mL or 150 mL bottles
- 2-inch filter paper for air particulates
- Charcoal cartridges
- 2-inch stainless steel planchets

8.3 Use appropriate graduated cylinders and transfer pipets during the preparation of the solutions cited in 8.2 above.

9.0 PROCEDURE

9.1 Calibration Source Preparation

9.1.1 Calibrations are conducted using standard sources with identical counting geometries and the same or similar matrices as the test source for analysis. Calibration sources can be any reproducible shape or size, but the radionuclides must be uniformly distributed throughout. Important considerations are geometry (shape), size, and homogeneity (uniformity) of the source.

Calibration sources are purchased from a commercial vendor. Accompanying certificates document the known activity and stated uncertainty for each radionuclide.

Each standard in solution, upon arrival at TBE-ES after purchase, is quantitatively diluted using a reagent matrix specified by the instructions

which accompany the standard. Each individual counting standard is prepared by volumetric aliquoting of an approximate activity size. See section 5.0 above.

9.1.2 Using the prepared standards for each geometry, calibrate all gamma detectors as described in the ensuing sections with each standard once per year or after a detector is repaired or newly purchased. Once a week the check source standard is counted on each detector for efficiency, energy, and resolution checks.

9.2 Detailed Calibration Procedure

Each detector is calibrated for efficiency, energy and resolution at full width half maximum (FWHM) once per year at various energies for each geometry. This procedure is repeated after a detector is repaired or newly purchased. Efficiencies for other energies are calculated by a computer using a curve-fitting program using the above energy calibrations as reference points.

9.2.1 The energy calibration is performed by adjusting electronic discriminators and amplifier gains so that each channel of a 2048 channel pulse height analyzer represents one KeV of energy and/or each channel of a 4096 channel pulse analyzer represents 0.5 KeV and/or 1 KeV of energy of energy respectively depending on desired photon detection.

9.2.2 Counting efficiencies for the various gamma energies (photopeaks) are determined from the activity counts of the NIST-traceable standards.

9.2.3 A counting efficiency versus gamma energy curve is determined for each container geometry and for each detector that is to be used for sample analysis. Known amounts of various radionuclides that emit gamma photons with energies well spaced and distributed over the normal range of analysis are used for this calibration.

9.2.4 The detector efficiency, E, at a given photopeak energy for a given geometry is determined by using a known quantity or concentration (for a volume geometry) of a gamma emitting radionuclide. This is calculated by the data system, as follows: as follows

$$E = \frac{C}{A \times B}$$, where:

C = net count rate, cpm (integfrated counts in the photopeak above the baseline continuum divided by the counting time in minutes)

A = activity of radionuclide added to the given geometry container (dpm)

B = the gamma ray abundance of the radionuclide being measured (gammas/disintegration)

9.3 Detailed Stability Check Procedure

Each week the check source standard is counted on each detector. The efficiencies, resolutions, and energy settings of the appropriate peaks are calculated and periodically plotted on a control chart. The length of count is preset to a time sufficient to obtain counting statistics of 10% or better. If an electrical power interruption occurs, the check source standard is counted on one detector of each of the data acquisition systems after the power has been restored, for efficiency, energy, and resolution checks. After determining that each system is functioning properly, a visual inspection of each spectrum in the acquisition mode is made to assure that each detector is counting. After the repair or purchase of a gamma detector, the efficiencies of each geometry are performed and revised if necessary. The $\pm 2\sigma$ and $\pm 3\sigma$ precision levels are determined by averaging 15-20 measurements of the stability check source.

9.4 Detector Backgrounds

9.4.1 When a gamma-emitting isotope is present in a sample the gamma-ray peak is identified by the software program. The software calculates the

number of counts in the peak attributable to the isotope by subtracting background counts from the total count in the peak region. This is done by setting a region on either side of the peak, determining the number of counts in each region and performing a linear interpolation and subtraction from the peak region. The background counts are part of the continuous background spectrum of the detector. In some cases the background can also have a contribution from the Compton effect of a high energy gamma-ray peak in the sample. Each gamma detector also has a few gamma-ray peaks in the continuous background spectrum. The gamma-ray peaks are from naturally occurring isotopes present in the detector and surrounding materials. The most common isotopes are K-40, Ra-226, and Th-228.

9.4.2 Once every two weeks, each detector background is determined by operating the system without a sample for a 1000 minute period. The gamma-ray peaks for the detected isotopes are identified and the counts per second of each peak are determined and stored on a disk. Later when the computations are being performed for sample activities the background information is retrieved and subtracted from the appropriate peaks before the remainder of the calculation is performed.

10.0 DATA AND RECORDS MANAGEMENT

10.1 All laboratory data and ancillary information shall be documented in bound laboratory logbooks or appropriate worksheets in permanent ink. Logbook entries and worksheets shall be reviewed by appropriate supervisory personnel as required by the TBE-ES Quality Assurance Program.

10.2 Corrections to recorded data in logbooks or on worksheets shall be noted by drawing through the incorrect data with a single line and recording the date of the correction and the initials of the person making the correction. The correct data

will be recorded in an unambiguous location in the immediate proximity of the incorrect data.

10.3 Each standard lists the manufacture of the nuclides, the certificate numbers, nuclides, and activities of the nuclides at a reference time. The counting of the standards are recorded in a log containing the sequential listing of every sample (including backgrounds and standards) by detector serial number. The start date and time and duration of count are recorded. The spectrum is stored and transferred to various data storage media for archival purposes.

11.0 QUALITY CONTROL & QUALITY ASSURANCE

11.1 The efficiencies of the selected energies are plotted on a control chart inscribed with lines showing the mean, $\pm 2\sigma$, and $\pm 3\sigma$ precision levels. The laboratory manager or a person he designates will interpret the control charts. Five percent of the plotted values are expected to fall outside the $\pm 2\sigma$ precision band based on statistics alone. If a value falls outside the $\pm 3\sigma$ precision band, another reading of the standard should be made. If the second reading also falls outside the $\pm 3\sigma$ band, the detector is judged to be out of control. An efficiency control chart can exhibit a trend without being out of control by the above criteria. Interpretation of trends is performed by the laboratory manager or by the person he designates. It is good practice to investigate trends which approach one of the $\pm 2\sigma$ precision lines. The mean, $\pm 2\sigma$, and $\pm 3\sigma$ precision lines are determined by averaging the last 15-20 efficiencies (for each selected energy) from the previous year of the weekly stability check measurements. The resolution (full width at half maximum) of each selected peak must not exceed 5.0 KeV. If it does, another measurement with the standard should be made. If the resolution still exceeds 5.0 KeV, the detector is judged to be out of control.

11.2 The energy of each selected peak must be within $\pm$ 3 KeV of the correct gamma-ray peak energy. If one peak energy is outside $\pm$ 3 KeV of the correct value,

another measurement should be made. If the energy is still outside ± 3 Kev, the detector is judged to be out of control.

11.3 When a detector is determined to be out of control, the laboratory manager or the person he designates will evaluate the detector and the electronic system to determine the cause of the problem.

11.4 The detector is to be labeled not to be used to analyze samples while designated out of control. Corrective action must be documented in the maintenance log kept in the laboratory. If a sample (or samples) had been counted during a time period for which the detector was judged out of control, the sample (or samples) are counted a second time on a detector which is within control limits.

12.0 REFERENCES

12.1 There are no external references which pertain to this procedure.

TELEDYNE BROWN ENGINEERING ENVIRONMENTAL SERVICES

Procedure PRO-Q-008

Balance Calibration Check

Rev. 0

Date of Original Issue: 11/28/01

Approved by:

Keith O. Jeter, Operations Manager — Date 11/28/01

Martin R. Keller, Quality Assurance Manager — Date 11/28/01

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Appendix I 7

1.0 SCOPE & APPLICABILITY

1.1 Control standards for balances are necessary to assure high quality results for gravimetric measurements. The purpose of this procedure is to define the steps to be followed when checking balance calibration and to establish criteria for acceptable performance of the balances. Only balances meeting established control limits are to be used. Balances not meeting these standards are tagged "Out Of Service" and are removed from use until repaired or replaced.

2.0 SUMMARY OF METHOD

2.1 Balance calibration checks utilizing a minimum of three (3) selected certified weights will be made each day the balance is to be used. The Laboratory Manager or Supervisor of each area is responsible for assuring that balance calibration checks are made for the balances in each area and that logbooks are maintained.

3.0 DEFINATIONS

3.1 MSDS -- Material Safety Data Sheet

3.2 NIST -- National Institute of Standards and Technology

3.3 TBE-ES -- Teledyne Brown Engineering -- Environmental Services

4.0 HEALTH & SAFETY WARNINGS

4.1 No specific Health and Safety warnings apply to balance calibration.

5.0 CAUTIONS

5.1 Samples which appear to be hygroscopic are dried again under heat lamps just prior to counting.

6.0 INTERFERENCES-N/A

7.0 PERSONNEL QUALIFICATIONS

7.1 Analysts performing this procedure must be trained, qualified, and certified in accordance with the TBE-ES Quality Control Manual IWL-032-365, Sections 2.2 through 2.9 inclusive. Procedure specific training documentation for designated analysts is maintained in the QA office.

7.2 Analysts in training may perform this procedure only under the direct supervision and observation of a senior technician certified to perform this procedure.

8.0 EQUIPMENT & SUPPLIES

8.1 • Calibration check weights certified to NIST standards

8.2 Use appropriate graduated cylinders and transfer pipets during the preparation of the solutions cited in 8.1 above.

9.0 PROCEDURE

9.1 Balance checks

9.1.1 Balances are calibrated by an external agency on an annual basis. Calibration check weights are certified by an external agency on a triennial basis. A calibration check will be made before each use day using this procedure and certified weights selected by the Laboratory Manager.

9.1.2 A logbook is provided with the balance.

9.1.3 Check the level of the balance on the level built into the balance. Adjust balance legs until the bubble is centered in the window.

9.1.4 Inspect the balance pan and chamber for cleanliness. If the balance is dirty, note this fact in the log and report the condition of the balance to the Lab Supervisor. Clean the balance as necessary before making checks.

9.1.5 Close the balance chamber and zero the balance. Using a clean cotton or nylon glove or the forceps provided with the weights, place the first designated weight on the center of the balance pan. Never allow the weights to contact

any surface other than the weight box, the forceps, the balance pan, or the glove.

9.1.6 Perform the weighing, allowing sufficient time for the pan to stabilize, and record the measured weight in the log under the column listing measured value.

9.1.7 Complete the calibration check using the remaining designated weights.

9.1.8 Analytical Balances

9.1.8.1 Analytical balances shall be labeled with the manufacturer's serial number. Balances are services and cleaned annually by the manufacturer or a qualified representative to meet original specifications. Calibration weights are re-certified triennially to NIST (class S) weights by the manufacturer or by a qualified representative. If "as received" readings from the calibration vendor show out of tolerance conditions, the Quality Assurance Manager shall initiate an NCR and determine needed rechecks of prior work that used the balance.

9.1.8.2 A certificate of service and calibration shall be issued and each unit shall be labeled and dated with the last service and calibration period.

9.1.8.3 The balance shall be zeroed and tested with certified class S weights traceable to NIST each day that the balance is in use and the results shall be recorded in a log. The analyst will zero the balance before weighing a set of samples. If the balance is not functioning correctly, it shall be removed from use. The balance must be serviced and calibrated before it is put back in use.

10.0 DATA AND RECORDS MANAGEMENT

10.1 All laboratory data and ancillary information shall be documented in bound laboratory logbooks or appropriate worksheets in permanent ink. Logbook entries and worksheets shall be reviewed by appropriate supervisory personnel as required by the TBE-ES Quality Assurance Program.

10.2 Corrections to recorded data in logbooks or on worksheets shall be noted by drawing through the incorrect data with a single line and recording the date of the correction and the initials of the person making the correction. The correct data will be recorded in an unambiguous location in the immediate proximity of the incorrect data.

11.0 REFERENCES

11.1 There are no external references which pertain to this procedure.

APPENDIX I

	Model	Serial	QC ID#
Sartorius	LC1200S	10101858	QC104
Sartorius	AC210S	10100741	QC105
Sartorius	LC1200S	40010004	QC106
Denver	A160	0053202	QC117

Attachment M

EM 1110-1-1906
30 September 1996

US Army Corps
of Engineers

ENGINEERING AND DESIGN

Soil Sampling

ENGINEER MANUAL

Appendix E
Visual Identification of Soil Samples

E-1. Field Identification Techniques

Visual identification techniques reported herein generally yield results which are consistent with the Unified Soil Classification System (ASTM D 2487, 1993; ASTM D 2488, 1993; U.S. Army Engineer Waterways Experiment Station, 1960).[1] Because these techniques are primarily visual, subtle discrepancies may exist between the identifications obtained in the field and the classifications determined in the laboratory. However, the results are meaningful provided the inspector makes careful and consistent identifications. See Chapter 13 for details on handling and storage of samples and maintaining sampling records. The inspector's equipment required to conduct these tests is limited to a pocket knife, scale, magnifying glass, and a small container of diluted hydrochloric acid. Table E-1 can be used as a checklist for conducting a systematic visual identification of a soil sample; it is also useful for locating the appropriate table and/or figure which describe(s) a test procedure for visually identifying the soil.

E-2. Grain Size

The inspector must first determine whether the material is coarse grained or fine grained. To make this determination, spread a representative sample on a flat surface. Determine whether or not the predominant size fraction is discernible with the naked eye. Coarse-grained soils vary from particles in excess of 75 mm (3 in.) in diameter to particles just discernible with the unaided eye, such as table salt or sugar, whereas fine-grained soils are microscopic and submicroscopic. The predominant material of peat or muck is decaying vegetation matter. Table E-2 and Figure E-1 may aid in determining the grain size of the soil in question. If the predominant material is coarse grained, follow the procedures outlined in paragraph E-2*a*; if the soil is fine grained, follow the procedures outlined in paragraph E-2*b*.

a. Coarse-grained soils.

(1) *Coarse fraction.* Once the soil has been determined to be coarse grained, further examination is required to determine the grain size distribution, the grain shape, and the density of the in situ deposit (if applicable). The gradation of coarse-grained soils can be described as well graded, poorly graded, or gap graded.

Table E-3 and Figure E-2 can be used in selecting the appropriate descriptive terms. Soil particles can also be described according to a characteristic shape. Particle shape may vary from angular to rounded to flat or elongated. Appropriate descriptive terms are listed in Table E-4; particle shapes are illustrated in Figure E-3. The density of an in situ deposit of a coarse-grained soil is also valuable information. Results obtained by pushing a reinforcing rod into a surface deposit or from the Standard Penetration Test may indicate the density of an in situ deposit. Appropriate descriptive terms may be selected from Table E-5.

(2) *Fine fraction.* The plasticity characteristics of the fine fraction of a coarse-grained material also need to be determined. The tests for fine-grained soils (paragraph E-2*b*) which are described in Table E-6 and illustrated in Figures E-4 through E-10 can be used to characterize the fine fraction of the soil sample in question.

b. Fine-grained soils.

(1) *Coarse fraction.* The coarse-grained fraction, where applicable, should be described in terms of the size of the predominate grain size, i.e., sand or gravel. Paragraph E-2*a*, Table E-2, and Figure E-1 may aid in selecting appropriate descriptive terms.

(2) *Fine fraction.* Several tests may be useful in determining the plasticity characteristics of fine-grained soils or fractions thereof; these tests include the dilatancy or reaction to shaking test, the dry strength test, and the toughness and plasticity tests. For each test, the fraction which passes the No. 40 U.S. Standard Sieve (0.42 mm) is used; this fraction corresponds to the fraction which is required for determination of Atterberg limits. For the purpose of the visual tests, however, screening is not important; the removal of coarse particles is adequate. Tests to determine the plasticity characteristics of the fine fraction are described in Table E-6, the dilatancy test is illustrated in Figures E-4 through E-7, the dry strength test is presented in Figure E-8, and toughness and plasticity tests are given in Figures E-9 and E-10, respectively.

c. Other tests. The dispersion (settlement in water) test and the bite test can be used to determine the

[1] References cited in this appendix are included in Appendix A.

presence of and relative amounts of sand, silt, and clay fractions (see Table E-6). Several other tests, such as the odor and the peat tests for determining the presence of organic matter, the acid test for determining the presence of a calcium carbonate cementing agent, and the slaking test for determining whether the "rocklike" material is shale, are listed in Table E-6. Strength descriptors of a clay sample are listed in Table E-7.

E-3. Soil Moisture and Color

Soil moisture and color are important indicators of soil conditions. For example, visible or free water from a soil sample can infer the proximity of a water table. The color of a moist soil sample tells much about the minerals and chemicals present in the soil, the drainage conditions, and the presence of organic matter. Soil color charts prepared for the U.S. Department of Agriculture (USDA) by the Munsell Color Company, New Windsor, NY 12553, are helpful for describing the color of soil samples. Soil moisture conditions or water contents can be described following the criteria presented in Table E-8. The importance of color for identifying and classifying moist fine- grained soils is shown in Table E-9.

E-4. Mass Structure and Mass Defects of Soil Formations

Mass structure and mass defects yield data about the geotechnical engineering behavior of a soil formation in question. For example, a varved clay would most likely have different engineering properties from an homogeneous deposit of one of the constituent soils. Likewise, slickensides indicate a clay deposit has been overconsolidated because of desiccation, surcharge loading, or both; an overconsolidated clay would have different engineering properties from a normally consolidated deposit of the same material. Descriptive terms for mass structure and mass defects of a soil formation are presented in Tables E-10 and E-11, respectively.

E-5. Description of Soils

As presented in the footnote in Table E-1, the description of a soil sample should contain appropriate terms to characterize the soil type and grain size, its moisture content and color, and mass structure and defects. Commonly used names and descriptions for selected soils are presented in Table E-12.

Table E-1
Order of Description for Soils

Criteria	Table No.	Figure No.
Soil types and particle sizes	E-2	E-1
Coarse-grained soils		
Description of gradation of coarse-grained soils	E-3	E-2
Description of grain shape of coarse-grained soils	E-4	E-3
Density of coarse-grained soils	E-5	
Fine-grained soils		
Field identification procedures for fine-grained soils	E-6	
Strength or consistency of clays	E-7	E-4/E-10
Moisture content	E-8	
Role of color for identification of moist fine-grained soils	E-9	
Terms for describing mass structure of soils	E-10	
Terms for describing mass defects in soil structure	E-11	
Commonly used descriptive soil names	E-12	

Example: Sand, fine, silty, tan, poorly-graded, dense, wet, subrounded, very friable with occasional clay lenses.

Table E-2
Soil Types and Particle Sizes

Principal Soil Type	Descriptive Term	Size	U.S. Standard Sieve	Familiar Example
Coarse-grained Soils	Cobble	76 mm or larger	Greater than 3 in.	Grapefruit or orange
	Coarse gravel	76 mm to 19 mm	3 in. to 3/4 in.	Walnut or grape
	Fine gravel	19 mm to 5 mm	3/4 in. to #4	Pea
	Coarse sand	5 mm to 2 mm	#4 to #10	Rock salt
	Medium sand	2 mm to 0.4 mm	#10 to #40	Openings of a window screen
	Fine sand	0.4 mm to 0.074 mm	#40 to #200	Table salt or sugar
Fine-grained Soils	Silt or clay	Microscopic and submicroscopic		
Organic	Peat or muck			Decaying vegetable matter

Note: Particles which are retained on the No. 200 U.S. Standard Sieve (0.074 mm) can just be discerned with the naked eye at a distance of about 25 cm (10 in.).

Table E-3
Description of Gradation of Coarse-Grained Soils

Descriptive Term	Meaning
Well graded	A good representation of all grain sizes is present
Uniformly or poorly graded	All grains are approximately the same size
Gap graded	Intermediate grain sizes are absent

Table E-4
Description of Grain Shape of Coarse-Grained Soils

Descriptive Term	Example
Angular	Irregular with sharp edges such as freshly broken rock
Subangular	Irregular with smooth edges
Subrounded	Irregular but smooth as a lump of molding clay
Rounded	Marble or egg shaped, very smooth
Flaky	Sheet of paper or flake of mica
Flat	Ratio of width to thickness greater than 3
Elongated	Ratio of length to width greater than 3

Table E-5
Density of Coarse-Grained Soils

Descriptive Term	Blows per Foot[1,2]	Field Test
Very loose	Less than 4	----------
Loose	4-10	Easily penetrated with a 13-mm- (1/2-in.-) diam reinforcing rod pushed by hand
Medium dense	10-30	Easily penetrated with a 13-mm- (1/2-in.-) diam reinforcing rod driven with a 2.3-kg (5-lb) hammer
Dense	30-50	Penetrated 0.3 m (1 ft) with a 13-mm- (1/2-in.-) diam reinforcing rod driven with a 2.3-kg (5-lb) hammer
Very dense	Greater than 50	Penetrated only a few centimeters with a 13-mm- (1/2-in.-) diam reinforcing rod driven with a 2.3-kg (5-lb) hammer

[1] From SPT (Appendix B, Table B-1).
[2] 1 ft = 0.3048 m.

Table E-6
Field Identification Procedures for Fine-Grained Soils[1]

Test	Test Procedures and Interpretation of Results
Dilatancy	Prepare a pat of moist soil with a volume equivalent to a 25-mm (1-in.) cube (Figure E-4). Add water, if necessary, to make the soil soft but not sticky, i.e., (reaction to the "sticky limit"). Place the pat of soil in the open palm of one hand and shake horizontally; strike vigorously against the other hand several times. If the reaction is positive, water appears on the surface of the pat; the consistency of the pat then becomes livery; and the surface of the pat becomes glossy (Figure E-5). Next, squeeze the sample between the fingers (Figure E-6). The water and gloss should disappear from the surface of the pat; the soil will stiffen and crack or crumble (Figure E-7). The rapidity of the appearance of water on the surface of the soil during shaking and its disappearance during squeezing help to identify the character of the fines in the soil. Very fine clean sands give the quickest and most distinct reaction, inorganic silts give a moderately quick reaction, and plastic clays have no reaction.
Dry strength	Mold a pat of soil to the consistency of putty. If the soil is too dry, add water; if it is too sticky, the specimen should be allowed to dry by evaporation. After the consistency of the pat is correct, allow the pat to dry (by oven, sun, or air). Test its strength by breaking and crumbling between the fingers (Figure E-8). The dry strength increases with increasing plasticity. High dry strength is characteristic of high plasticity clays. Silty sand and silts have only slight dry strengths, but can be distinguished by feel when powdered; fine sands feel gritty whereas silts feel smooth like flour. It should also be noted that shrinkage cracks may occur in high plasticity clays. Therefore, precautions should be taken to distinguish between a break which may occur along a shrinkage crack or a fresh break which is the true dry strength of the soil.
Toughness and plasticity	A specimen of soil which is about the size of a 25-mm (1-in.) cube should be molded to the consistency of putty; add water or allow to dry as necessary. At the proper moisture content, roll the soil by hand on a smooth surface or between the palms into a thread about 6-mm (1/8-in.) diam (Figure E-9). Fold the thread of soil and repeat the procedure a number of times. During this procedure, the water content of the soil is gradually reduced. As drying occurs, the soil begins to stiffen and finally loses its plasticity and crumbles at the plastic limit. After the thread has crumbled, the pieces should be kneaded together and a kneading action should be applied until the lump crumbles. For higher clay contents, threads are stiffer and lumps are tougher at the plastic limit than for lower plasticity clays. A complementary test is the ribbon test. A roll of soil about 13- to 19-mm (1/2- to 3/4-in.) diam by 75 to 125 mm (3 to 5 in.) long should be prepared at a moisture content just below the "sticky limit". Flatten the roll of soil to thickness of 3 to 6 mm (1/8 to 1/4 in.) between the thumb and forefinger (Figure E-10). For high plasticity clays, a ribbon 20 to 25 cm (8 to 10 in.) long can be formed; shorter lengths correspond to lower plasticity clays whereas a ribbon cannot be formed when using non-plastic soils.
Dispersion test (settlement in water)	Place a few hundred grams of soil in a jar containing water. Shake the jar containing the mixture of soil and water and then allow the soil to settle. The rate of settling can be used to judge the predominate soil type(s) whereas the thicknesses of the various soils can be used to judge the gradation of the soil. Sands settle in 30 to 60 seconds, silts settle in 30 to 60 minutes, and clays may remain in suspension overnight. The interface between fine sands and silts occurs where individual grains can not be discerned with the unaided eye. The cloudiness of the water indicates the relative clay content.
Bite test	Place a pinch of soil between the teeth and grind lightly. Fine sands grate harshly between the teeth; silts have a gritty feeling but do not stick to the teeth; clays tend to stick to the teeth, but do not have a gritty feeling.
Odor	Organic soils have a musty odor which diminishes upon exposure to air. The odor can be revived by heating a moist sample or by exposing a fresh sample.
Peat	Peat has a fibrous texture and is characterized by partially decayed sticks, leaves, grass, and other vegetation. A distinct organic odor is characteristic of peat. Its color generally ranges from dull brown to black.
Shine	A moist, highly plastic clay will shine when rubbed with a fingernail or pocketknife blade; a lean clay will have a dull surface.
Acid test	The presence of calcium carbonate in a soil can be determined by adding a few drops of dilute (3:1 ratio of water to acid) hydrochloric acid to the soil. The relative amount of calcium chloride in the soil can be determined by the effervescence (fizzing reaction) which occurs. Degrees of reaction range from none to strong. For some very dry non-calcareous soils, the illusion of effervescence as the acid is absorbed by the soil can be eliminated by moistening the soil before the acid is applied.
Slaking test	Certain shales and other soft "rocklike" materials disintegrate upon drying or soaking. The test is performed by placing the soil in the sun or oven to dry completely. After the sample has been dried, it should then be soaked in water. The degree of slaking should be reported.

[1] These tests must be performed on the minus No. 40 sieve size (0.42 mm) particles, which is the division between medium and fine sand. For field classification purposes, screening is not intended; simply remove the coarse particles that interfere with the tests.

Table E-7
Strength or Consistency of Clays

Descriptive Term	Blows[1] per Foot[2]	Unconfined Compressive Strength kPa	(tsf)	Field Test
Very soft	< 2	< 25	(< 0.25)	Core (height twice diameter) sags under its own weight while standing on end; squeezes between fingers when fist is closed
Soft	2-4	25-50	(0.25-0.5)	Easily molded by fingers
Medium	4-8	50-100	(0.5-1.0)	Molded by strong pressure of fingers
Firm	8-15	100-190	(1.0-2.0)	Imprinted very slightly by finger pressure
Very firm	15-30	190-380	(2.0-4.0)	Cannot be imprinted with finger pressure; can be penetrated with a pencil
Hard	> 30	> 380	(> 4.0)	Imprinted only slightly by pencil point

[1] From SPT (Appendix B, Table B-1).
[2] 1 ft = 0.3048 m.

Table E-8
Moisture Content

Condition	Estimated Water Content percent	Example
Dry	0 - 10	Absence of moisture; well below optimum water content for fine-grained soils
Moist	10 - 30	Fine-grained - damp, near optimum water content Coarse-grained - no visible water
Wet	30 - 70	Fine-grained - well above optimum water content Coarse-grained - visible water
Water bearing	-------	Water drains freely, below water table

Table E-9
Role of Color for Identification of Moist Fine-Grained Soils

General:

Detect different soil strata
Detect soil type based upon experience in local area
Colors become lighter as water content decreases

Soil Type:

Inorganic soils have clean, bright colors: light gray, olive green, brown, red, yellow, or white
Organic soils have dark or drab shades: dark gray, dark brown, or almost black

Presence of Chemicals:

Iron oxides: red, yellow, or yellowish brown
Silica, calcium carbonate, or aluminum compounds: white or pinkish

Drainage Conditions:

Poor: grayish blue and gray or yellow mottled colors

Table E-10
Terms for Describing Mass Structure of Soils

Descriptive Term	Definition
Homogeneous	Uniform properties
Heterogeneous	Mixtures of soil types not in layers or lenses
Stratified	Alternate layers of different soils or colors
Laminated	Repeating alternate layers of different soils or colors 3 to 6 mm (1/8 to 1/4 in.) thick
Banded	Alternate layers in residual soils
Lensed	Inclusions of small pockets of different soils

Table E-11
Terms for Describing Mass Defects in Soil Structure

Descriptive Term	Definition
Slickensides	Fracture of failure planes (polished surfaces) seen in stiff clays
Root holes	Holes remaining after roots have decayed
Fissures	Cracks from shrinkage, frost, etc.; specimen breaks along a definite plane of fracture
Weathered, oxidized	Irregular discolorations
Concretions	Accumulations of carbonates or iron compounds
Blocky	Cohesive soil broken into small angular lumps which resist further breakdown

Table E-12
Commonly Used Descriptive Soil Names (Continued)

Common Name	Description
Adobe	Soils such as calcareous silts and sandy-silty clays which are found in semiarid regions of southwestern United States and North Africa.
Alluvium	Deposits of mud, silt, and other material commonly found on the flat lands along the lower courses of streams.
Argillaceous	Soils which abound in clays or clay-like materials.
Bentonite	A clay of high plasticity formed by the decomposition of volcanic ash.
Boulder clay (L)[1]	A name, used in Canada and England, for glacial till.
Buckshot (L)	Clays of southern and southwestern United States which crack into small, hard lumps of more or less uniform size upon drying.
Buff's liver (L)	The term name used in some sections of the United States to describe an inorganic silt of slight plasticity, which, when saturated, quakes like jelly from vibration or shock.
Calcareous	Soils which contain an appreciable amount of calcium carbonate, usually from limestone.
Caliche	The term which describes deposits of silt, clay, and sand cemented by calcium carbonate deposited by evaporation of groundwater; this material is found in France, North Africa, and southwestern United States.
Coquina (L)	Marine shells which are held together by a small amount of calcium carbonate to form a fairly hard rock.
Coral	Calcareous, rock-like material formed by secretions of corals and coralline algae.
Diatomaceous earth	A white or light gray, extremely porous, friable, siliceous material derived chiefly from diatom remains.
Dirty sand (L)	A slightly silty or clayey sand.
Disintegrated granite	Granular soil derived from decomposition and weathering of granite rock.
Fat clay (L)	Fine colloidal clay of high plasticity.
Fullers earth	Highly plastic white to brown clays of sedimentary origin which are used commercially to absorb fats and dyes.
Gumbo (L)	Highly plastic silty and clayey soils which become impervious, sticky, and soapy or waxy when saturated.
Hardpan	A general term used to describe a hard, cemented soil layer which does not soften when wet.
Lateritic soils	Residual soils, usually red in color, which are found in tropical regions. In their natural state, these soils have a granular structure with low plasticity and exhibit good drainage characteristics; when remolded in the presence of water, they often become plastic and clayey.
Loam clay	Silty clays and clayey silts of low to medium plasticity.

[1] (L) refers to a name which is used in local areas, only.

Table E-12 (Concluded)

Common Name	Description
Limerock (L)	A soft, friable, creamy white limestone found in the southeastern United States; it consists of marine remains which have disintegrated by weathering.
Loam	An agricultural term used to describe sandy silty topsoils which contain a trace of clay, are easily worked, and are productive of plant life.
Loess	A silty soil of eolian origin characterized by a loose, porous structure, and a natural vertical slope; it covers extensive areas in North America, Europe, and Asia.
Marl	A soft, calcareous deposit mixed with clays, silts, and sands, and often contains shells or organic remains; it is common in the Gulf Coast area of the United States.
Micaceous soils	Soil which contains a sufficient amount of mica to give it distinctive appearance and characteristics.
Muck (mud)	Very soft, slimy silt which is found on lake or river bottoms.
Muskeg	Peat deposits found in northwestern Canada and Alaska.
Peat	Fibrous, partially decayed organic matter or a soil which contains a large proportion of such materials; it is extremely compressible and is found in many areas of the world.
Red dog (L)	The residue from burned coal dumps.
Rock flour	A low plasticity, sedimentary soil composed of silt-sized particles which may become quick at high moisture contents.
Shale	A thinly laminated rock-like material which has resulted from consolidation of clay under extreme pressure; some shales revert to clay on exposure to air and moisture.
Talus	A fan-shaped accumulation of fragments of rock that have fallen near the base of a cliff or steep mountainside as a result of weathering.
Topsoil	The top few inches of soil which contains considerable organic matter and is productive of plant life.
Tufa	A loose, porous deposit of calcium carbonate which usually contains organic remains.
Tuff	Stratified, compacted deposits of fine materials, such as cemented dust and cinders, ejected from volcanoes. Tuffs are prevalent in the Mediterranean area.
Varved clay	A sedimentary deposit which consists of alternate thin (less than 13 mm) layers of silt and clay.
Volcanic ash	Uncemented volcanic debris which consists of particles less than 3 mm diameter; upon weathering, a clay of high compressibility is formed.

BORING LOG
FIELD DATA

Project _____ Site _____ Date _____
Location _____ Job No. _____
Drill Rig _____ Inspector _____ Operator _____ Surface El _____ Boring No. _____

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet _____ of _____ Sheets

DRAFT CORING METHODOLOGY
U.S. EPA ERTC/REAC
MARCH 2003

INTRODUCTION

Vibracoring (or vibrocoring) is a technology and a technique for collecting core samples of underwater sediments and wetland soils. The vibrating mechanism of a vibracorer, sometimes call the "vibrahead", operates on hydraulic, pneumatic, mechanical or electrical power from an external source. The attached core tube is driven into sediment by the force of gravity, enhanced by vibration energy. When the insertion is completed, the vibracorer is turned off, and the tube is withdrawn with the aid of hoist equipment.

A variety of vibrahead types have been developed. In general the frequency of vibrations is in the range of 3,000 to 11,000 vibrations per minute (VPM), and the amplitude of movement is on the order of a few millimeters (mm). The vibrations cause a thin layer of material to mobilize along the inner and outer tube wall, reducing friction and easing penetration into the substrate. The liquid spaces in the matrix allow sediment grains to be displaced by the vibrating tube. Vibracoring works best on unconsolidated, waterlogged, heterogeneous sediments and soils. Silty sediments of mixed grain size are easiest to core. Vibracoring is less effective for relatively dry clays, packed sand or any consolidated (cemented) materials.

In non-vibratory coring methods a tube is simply pushed, dropped, propelled or hammered into the substrate. One might think that hammer-coring is essentially a very low-frequency, high-amplitude version of vibracoring, but the effect of slow hammering is not the same. In vibracoring, the high frequency vibration transfers more energy to the sediment, greatly reducing wall friction both inside and outside of the tube. The result is longer and more representative cores. In addition, vibracoring is quicker, less labor intensive, and the submersible units can be used over a wide range of water depths. Among the different types of vibracoring systems, the submersible electrical and hydraulic units are less constrained by depth than mechanical or pneumatic systems.

POSITIONING VESSEL

1. Sample locations will be selected prior to commencement of the survey. Each sample station will be located using real-time differential global positioning system (dGPS) and a labeled marker buoy will be deployed on station.
2. The coordinates for the coring location for that day will be entered into the dGPS unit on board the survey vessel as way points. The survey vessel will travel to and anchor as close to each marker buoy as possible.
3. After the vessel is anchored on the marker buoy, actual sample locations will be identified by using data provided by real-time dGPS unit on the survey vessel immediately prior to collection of the sample.
4. If the position is unacceptable, the vessel position is adjusted and the position rechecked.
5. Once the survey vessel is anchored in an acceptable position, core samples will be collected. All non-dedicated equipment that may potentially come in contact with the sediments will be decontaminated between sample locations as specified below.
6. At the end of the sampling day, the data loaded in the dGPS units are checked to verify the existence of all locations in which data were collected. Sampling locations will be plotted onto a master chart as the samples are collected and checked with the dGPS data as a further verification that the correct locations and sampling schedule are being followed, and as a visual reference of the progress of the survey.

SAMPLING PROCEDURES

Decontamination of Equipment

Decontamination of non-dedicated sampling equipment (i.e., the polybutyrate core tube liners, aluminum core tubes, and the core nose and catcher assemblies) will be performed prior to sampling and between each sample location. Visible sediment from outside the core barrel, on the vibracore unit, on the outside of the submersible pump and pump hose will be removed with pressurized water and a brush. After all visible contamination has been removed, surfaces that will come in contact with the sediments will be rinsed with distilled water and then acetone. After the acetone has evaporated, all equipment will be wrapped with aluminum foil. All waste materials will be contained during decontamination and transferred to appropriate drums for disposal.

Collection of the Chemical Cores

1. Obtain water depth (depth to top of sediment) from the onboard dual frequency fathometer (if dual frequency fathometer indicates two surfaces, use the lower of the two surface depths). Calculate required penetration depth.
2. The plastic covering and aluminum foil at the top of the core barrel and liner will be removed. The core barrel will be inserted into the head of the vibracorer as it lies horizontally on the deck and secured in the vibracore.
3. The operator will slowly winch the vibracorer into its deployment orientation.
4. The vibracorer will then be then slowly lowered into the water by the deployment equipment.
5. As the vibracorer approaches the sediments, the motor and the vibracorer penetrometer equipment will be turned on. The vibracorer is then allowed to slowly penetrate the sediments.
6. On completion of the required penetration, or upon vibracore refusal, the motor will be turned off and the vibracorer slowly raised. The actual vibracore penetration depth is recorded.
7. Clean the vibracorer and coring assembly by hosing down the equipment with water prior to being brought on board. Care should be taken not to direct water into the open end of the core barrel.
8. The vibracorer will then be returned to its deck storage location and the core nose will be immediately sealed by placing a plastic cap over the open end. The core will be carefully unbolted and taken to the core extraction area.
9. The core liner will be extracted from the core barrel and vibracoring recovery (e.g., total sediment core footage) will be measured using a tape measure and recorded to the nearest 0.1 foot.
10. Evaluate whether core penetration and recovery are acceptable.
11. If the recovery is acceptable, record this as the "primary" core and move the sample vessel a few feet prior to collecting any duplicate cores, if needed.
12. For each core collected record all pertinent information (as summarized below) in a field log book.
13. The core tubes will be capped and stored upright until transfer to the support launch for return to the sample processing area. The top of the core tube will be cut at the core sediment surface prior to capping. After capping, the top and bottom ends will be marked. Tubes will be transported to the processing facility by the support launch and will be maintained in an upright position wrapped in a cooling vest (sheets of iced gel).

Procedures for Unacceptable Chemical Core Recovery

1. If the penetration depth or recovery ratio (total sediment core footage divided by penetration depth) are unacceptable for the specified chemical analyses, the location of the sampling vessel will be shifted a few feet to collect a second sample.
2. Establish the new location and record GPS coordinates of actual sample location.
3. Collect a vibracore at the adjusted location.
4. A maximum of two vibracores for chemical analysis will be collected. If none of the cores meet the penetration or percent recovery ratio objectives, the longer of the two recovered cores will be selected as the primary core for chemical analysis.

Core Processing

1. Cores will be stored until processing in a vertical position at 4 degrees celcius (°C). Cores will be maintained under custody of U.S. EPA/ERTC or REAC until transferred to WES.
2. Cores will be transported to AED for processing. Process areas should be designated for handling hazardous materials. All personnel in the processing area, must be dressed in appropriate health and safety gear as specified in a site specific health and safety plan (HASP).
3. Remove top core cap for each core liner, and assess whether core lengths are to be processed as cohesive or non-cohesive sediments.
4. Cores will be placed horizontally on the processing table.
5. Using the circular saw a longitudinal cuts will be made through the core liner.
6. The cores will be visually described in a field log book and photo-documented.
7. The smear zone will be removed by scraping approximately 1/4 inch of exposed sediment. Dispose of this material as specified below.
8. Cores will be cut into 2 centimeter (cm) subsections. If there is inadequate sample volume, a new interval will be established. Each subsection will be placed in a decontaminated stainless steel bowl and homogenized. The sample material will be split into two subsamples (subsample #1 for dating and subsample #2 for dioxin (4-ounce), total organic carbon (TOC) (4-ounce) and grain size analyses (32-ounce). Each subsample will be transferred to the appropriate labeled glassware.
9. Subsample #1 will be submitted under chain of custody to subcontract lab for dating (Cs137,Pb210, and Be7).
10. Subsample #2 will be archived at AED. Analysis of this sample will be contingent upon the results of dating. The holding time until extraction for dioxin analyses is 30 days from the date of sampling. Extracts may be held for an additional 45 days prior to analysis.
11. All remaining sediment, and materials (that are not going to be decontaminated and reused) that came in contact with sediment, will be placed in appropriate drums for storage and disposal.

DOCUMENTATION

Field notes will also be kept during sampling activities. The following information will be recorded in a bound field logbook:

- Names of personnel on the vessel(s)
- Vessel Name
- Weather and tidal conditions
- Date and time of sampling
- Location and sample station number
- Vibracore Location Number
- Vibracore Number
- Measured Water Depth
- Vibracore Penetration & Recovery Table
- Calibration information

EQUIPMENT LISTS

Survey Vessel

- Dual frequency fathometer
- Plastic sheeting
- Permanent marker or grease pencil
- Vibracore and penetrometer assembly and deployment equipment (e.g., Aframes, winches, generator)
- Decontaminated polybutyrate (e.g., Lexan) core liners, fully assembled with decontaminated stainless steel core noses and core catchers
- Cooling vests, or equivalent
- Assorted nautical equipment (e.g., anchors, lines, personal flotation devices)
- Field logbooks
- Appropriate decontamination equipment
- Tape measure
- Submersible pump and hose
- DGPS with external antennae
- Core extrusion table
- Hacksaw and spare blades
- Core caps
- Wrap-around polyethylene "sausage-case" sleeving
- Appropriate personal health and safety equipment
- Appropriate decontamination equipment

Core Processing Laboratory

- Core processing table
- Processing laboratory notebook and associated coring documentation
- Tape measure
- Hacksaw and spare blades
- Power driver circular saw and spare blades
- Core caps
- Sampling equipment: stainless steel bowls and spoons
- Refrigerator, at 4°C for sample storage/archive
- Sample Glassware
- Appropriate waste disposal equipment
- Appropriate personal health and safety equipment
- Appropriate decontamination equipment

Attachment N

TELEDYNE BROWN ENGINEERING
A Teledyne Technologies Company

Resume Summaries for Technical Personnel

Laboratory Manager	Keith O. Jeter
Responsibility:	Responsible for overall management of all environmental laboratory services for the Knoxville, Tennessee radioanalytical laboratory
Academic Training:	Bachelor of Science in Chemistry, Louisiana Tech University, 1986
Experience:	Ten years experience as laboratory and instrument manager for Duratek Inc.'s radiological and trace metals laboratory. Other experience includes task leader for radiological laboratory analysis of environmental monitoring samples at Oak Ridge National Laboratory (ORNL) and research chemist for Lion Oil Company in El Dorado, Arkansas.
QA Manager	Marty Keller
Responsibility:	Responsible for all aspects of the Laboratory Quality Assurance program including assuring that all client Statements of Work (SOW) requirements are met. Reports directly to upper management.
Academic Training:	Bachelor of Arts in Microbiology/Biochemistry, University of Tennessee, 1985. Certified by the American Society for Quality Control as Lead Auditor.
Experience:	Previous experience as a Quality Assurance Manager with ThermoRetec radiological laboratory. Senior Quality Assurance Engineer for Bechtel National, Inc.
Project Manager	Rebecca Charles
Responsibility:	Responsible for all aspects of client SOW (from sample receipt through data report delivery). Validates laboratory data reports against client requirements and serves as liaison between client and the laboratory.
Academic Training:	Bachelor of Arts in Zoology, University of Tennessee, 1978 & Associate of Science Degree in Health Physics.
Experience:	Seven years experience with International Technology (Quanterra Environmental Services) as a radiological chemist, laboratory supervisor, and laboratory project manager. Three years experience with the Tennessee Department of Environment and Conservation performing environmental sampling, regulatory oversight and technical review.
Project Manager	Lynne Perry
	Responsible for preparation of Radioactive Environmental Monitoring Reports and preparation of Quality Assurance reports. Serves as liaison between clients and the laboratory.

Academic Training:	Central Virginia Community College, Associate of Science, 1981. Professional Nuclear Quality System Lead Auditor Certification, 2000.
Experience:	Quality Assurance Manager for ThermoRetec Albuquerque Laboratory. Radiochemist for IT Corporation at both the Oak Ridge Radiochemistry laboratory and at the Waste Pit Remedial Action Project (WPRAP) in Fernald, Ohio. Provided radiochemical data validation for Science Applications International Corporation. Over 30 years of laboratory experience.
Production Manager	Martin Webb Oversees sample throughput and day to day operations of laboratory. Supervises chemist and technicians, assigns tasks, manages all laboratory resources and equipment.
Education:	Associate of Science, Roane State Community College.
Experience:	Ten years laboratory/health physics experience. Health physics dosimetry, ALARA trainer, lead analyst for Duratek. Laboratory/green is clean supervisor at US Ecology. Experience includes operation of radiochemical and analytical laboratory equipment, dose monitoring and supervision of operations.
Lead Chemist	Chris Foell Responsible for scheduling, supervision of analysts and performance of radiochemical separations for higher level samples including nuclear power plant effluents as required by 10CFR50 and samples for disposal under 10CFR61.
Education:	B.S. Geology, Boston College, M.S. Geology, Indiana University. Analytical and biochemistry coursework, University of New Orleans. Phi Beta Kappa
Experience:	Nine years experience in radiochemistry at Oak Ridge Research Institute/Lockheed Martin Energy Systems. Experience with DOE hazardous waste, defense, and industrial hygiene/health physics programs. Addition laboratory and research experience with the U.S. Department of Agriculture-Agriculture Research Service. Experience in geological field work and data interpretation.
Lead Chemist	Louie Balogh Responsible for scheduling, supervision of analysts and performance of radiochemical separations for environmental and bioassay samples.
Education:	Bachelor of Science in Business Management, Tusculum College, 2000
Experience:	Senior Analyst, Project Manager and Laboratory Supervisor for Thermo NuTech's radiological laboratory. Over 12 years of chemical laboratory experience including positions at Etrac, Inc., ATEC Associates and Foamex, Inc.

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<u>Gamma Spectroscopist</u>	I. Lauren Larsen
Responsibilities:	Responsible for technical issues involving gamma spectrometry.
Academic:	Master of Science in Oceanography, Oregon State University, Corvallis, OR. Bachelor of Science in General Science, Portland State College, Portland, OR.
Experience:	Over 35 years in radioanalytical determinations including gamma-ray spectrometry, alpha spectrometry and liquid scintillation counting with ORNL and Oregon State University.

UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

SAMPLING AND ANALYZING SEDIMENT CORES FOR ^{210}Pb GEOCHRONOLOGY

by

E. Ann Martin and Cynthia A. Rice

OPEN-FILE REPORT 81-983

This report is preliminary and has not been reviewed for conformity with U.S. Geological Survey editorial standards or stratigraphic nomenclature. Use of brand names in this report does not imply endorsement by the USGS.

1981

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Introduction

Various radiometric techniques have been applied to determine various geologic time scales for dating sediments. Few of these methods are applicable to recent sediments. Among those that apply, the most commonly used are based on the analysis of ^{210}Pb, $^{228}Th/^{232}Th$, $^{228}Ra/^{226}Ra$, and a variety of manmade isotopes resulting from the detonation of nuclear devices (for example, ^{90}Sr and ^{137}Cs). Our purpose is to describe the essential considerations in planning field sampling, analysis of sediments, and data reduction for determining sedimentation rates on the basis of ^{210}Pb analyses. Although this paper discusses only the ^{210}Pb method, many of the principles and suggestions regarding sampling are applicable to other radiometric methods.

The vertical distribution of ^{210}Pb can be used as a means to establish a sediment chronology; ^{210}Pb geochronology has recently been accepted as an important radioisotope technique for studying rates of recent sediment accumulation. The maximum dating range is controlled by the amount of excess ^{210}Pb present over the background concentration of ^{210}Pb in the sediment column and by the precision of the analytical methods used to analyze for the isotope. In most environments, this maximum dating range is 100-150 years before the sample was collected.

Background

The first reported use of ^{210}Pb dating was in the early 1960's to determine snow accumulations in the permanent snowfields of Greenland (Goldberg, 1963). This radiometric method has since been used to examine sediment accumulation rates in various environments including Alpine glaciers and Antarctic ice sheets (Windom, 1969; Crozaz and others, 1964); rivers and estuaries (Benninger, 1976; Benninger and others, 1975, 1976; Martin, 1979); marshes (Armentano and Woodwell, 1975; McCaffrey, 1977); basins and lakes (Koide and others, 1973; Edgington and Robbins, 1976; Matsumoto, 1975; Thomson and others, 1975; Farmer, 1978; Martin and Rice, 1980); continental shelves (Nittrouer and others, 1979; Shokes, 1976; Shokes and Presley, 1976; Bothner and Locker, 1977; Holmes and Martin, 1977, 1978); and continental slopes (Koide and others, 1972; Bruland and others, 1974). Recent uses of ^{210}Pb-dated cores have involved studies of pollution histories (Goldberg and others, 1978, 1979; Chow and others, 1973; Schubel and Hirschberg, 1977; Christensen and others, 1978; Skei and Paus, 1979; Matsumoto and Wong, 1977; and Nriagu and others, 1979).

Theory and Assumptions

The ^{210}Pb methods used for determining sedimentation rates are based on the occurrence of excess radioactive lead, ^{210}Pb, a member of the uranium-238 decay series (fig. 1). Radon-222, which has a half life of 3.8 days (Lederer and others, 1968), diffuses from the soil into the atmosphere and remains chemically inert until it decays through four short-lived nuclides (^{218}Po, ^{214}Pb, ^{214}Bi, and ^{214}Po) to ^{210}Pb (fig. 2). This isotope is rapidly removed from the atmosphere into the hydrosphere by rain, snow, and dry fallout and provides a measurable flux to the land and waters of approximately 0.5 to 1.0 dpm (disintegrations per minute) $^{210}Pb/cm^2/yr$ depending on the latitude and distance from continental land masses

(Benninger, 1976, Carpenter and others, in press). ^{210}Pb is also supplied to the hydrosphere by stream runoff and decay of ^{226}Ra in the water column, but these sources are insignificant when compared with the atmospheric source (Benninger, 1976). There seems to be no significant formation of ^{210}Pb as a result of nuclear detonations (Beasley, 1969). From the water column, it is rapidly removed to the bottom sediments. Several mechanisms have been proposed for the removal of ^{210}Pb from the hydrosphere, including adsorption onto particulate clays or organic matter and coprecipitation with Fe and Mn oxides. The chemical behavior of lead makes it immobile in the sediments. ^{210}Pb, which has a half life of 22.26 years (Höhndorf, 1969), undergoes a beta decay to ^{210}Bi in the sediment column. This daughter undergoes another decay to ^{210}Po, which has a half life of 138.4 days (Lederer and others, 1968), and alpha decays to stable ^{206}Pb.

As with other radiometric dating methods, three general assumptions must be met:

1) The ^{210}Pb flux to the sediments has been constant during the time interval of the dating range. This is called the inheritance assumption.
2) There has been negligible migration of ^{210}Pb in the sedimentary column after deposition. This is called the closed-system assumption.
3) The sedimentation rate has been constant during the period of time to be determined.

Two methods are commonly used to measure ^{210}Pb activity. Both methods involve the measurement of another radioactive isotope and the assumption of secular equilibrium between ^{210}Pb and the measured isotope.

Method 1: Beta counting. The beta counting method involves the recovery of radioactive ^{210}Pb from solutions by anion exchange chromatography; ordinary lead is used as a carrier. After several concentration steps, the ^{210}Pb is precipitated as a sulfate or a chromate. The ^{210}Bi daughter is measured as it undergoes a beta decay to ^{210}Po. This method is used by many investigators (Krishnaswami and others, 1971; Koide and others, 1972; Shokes, 1976).

Method 2. Alpha counting. Another widely used technique, and the one preferred by the authors, involves measurement of ^{210}Po activity (Robbins and Edgington, 1975; Flynn, 1968; Millard, 1963; Nozaki and Tsunogai, 1973). If ^{210}Pb and ^{210}Po are in secular equilibrium, which has been shown to exist in some sediments (Benninger, 1976; Nittrouer and others, 1979), the alpha decay of ^{210}Po can be used to measure the ^{210}Pb activity of both the ^{210}Pb produced by the decay of ^{226}Ra in the sediment column (supported ^{210}Pb) and the ^{210}Pb added from external sources (excess ^{210}Pb). In recently deposited sediments that contain large amounts of organic matter, the $^{210}Po/^{210}Pb$ activity may be >1.0. This disequilibrium can be verified by analyzing the ingrowth of ^{210}Pb 6 months after the original analysis and comparing the second activity with the initial activity. A calibrated ^{208}Po spike is used to calculate the activity of the ^{210}Po, and standard methods of data reduction are used to obtain ^{210}Pb activities. The analytical method and data reduction are explained in detail in the "Laboratory Methods" section of this report.

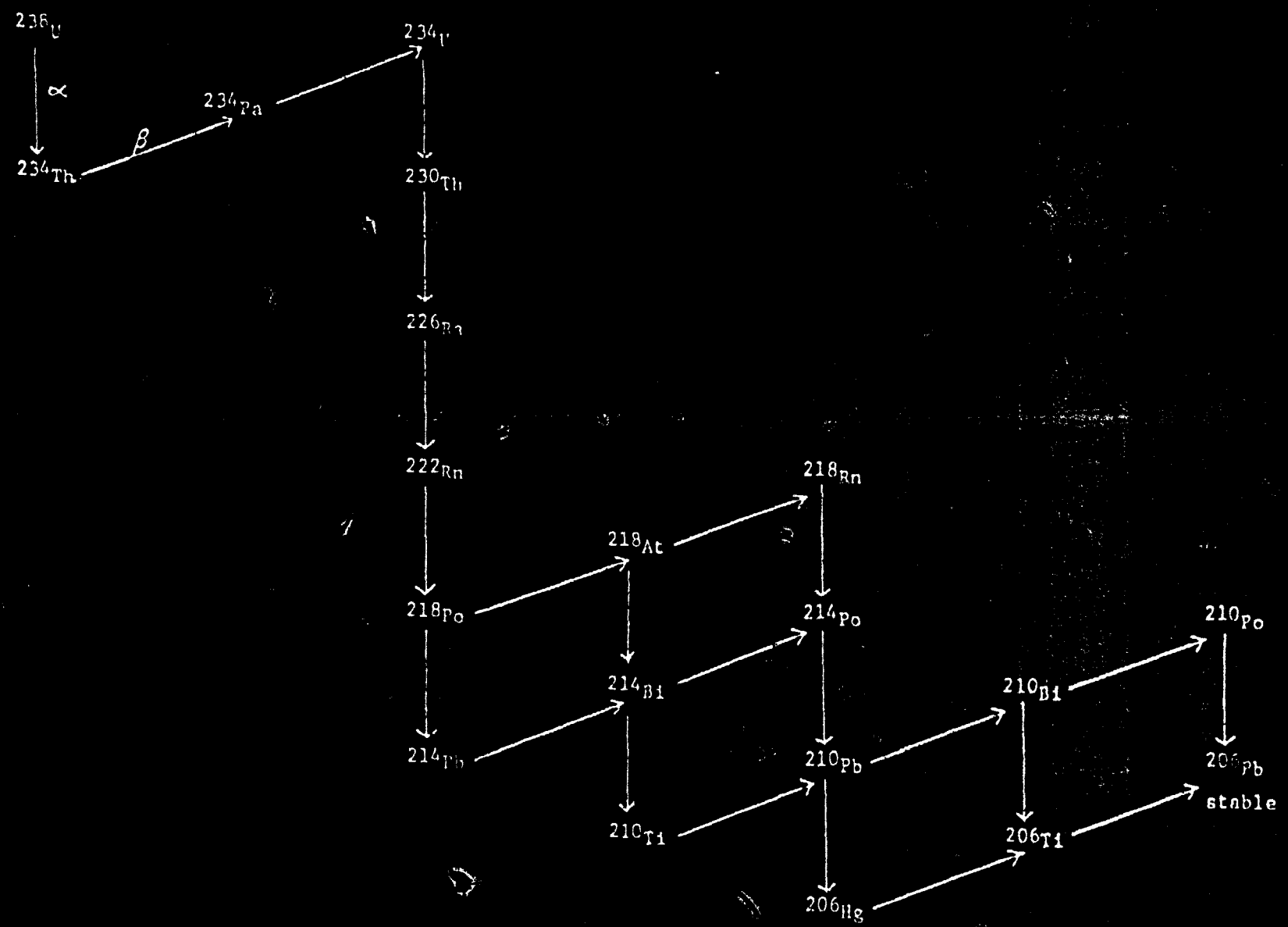


Fig. 1. Radioactive decay of ^{238}U to stable ^{206}Pb.

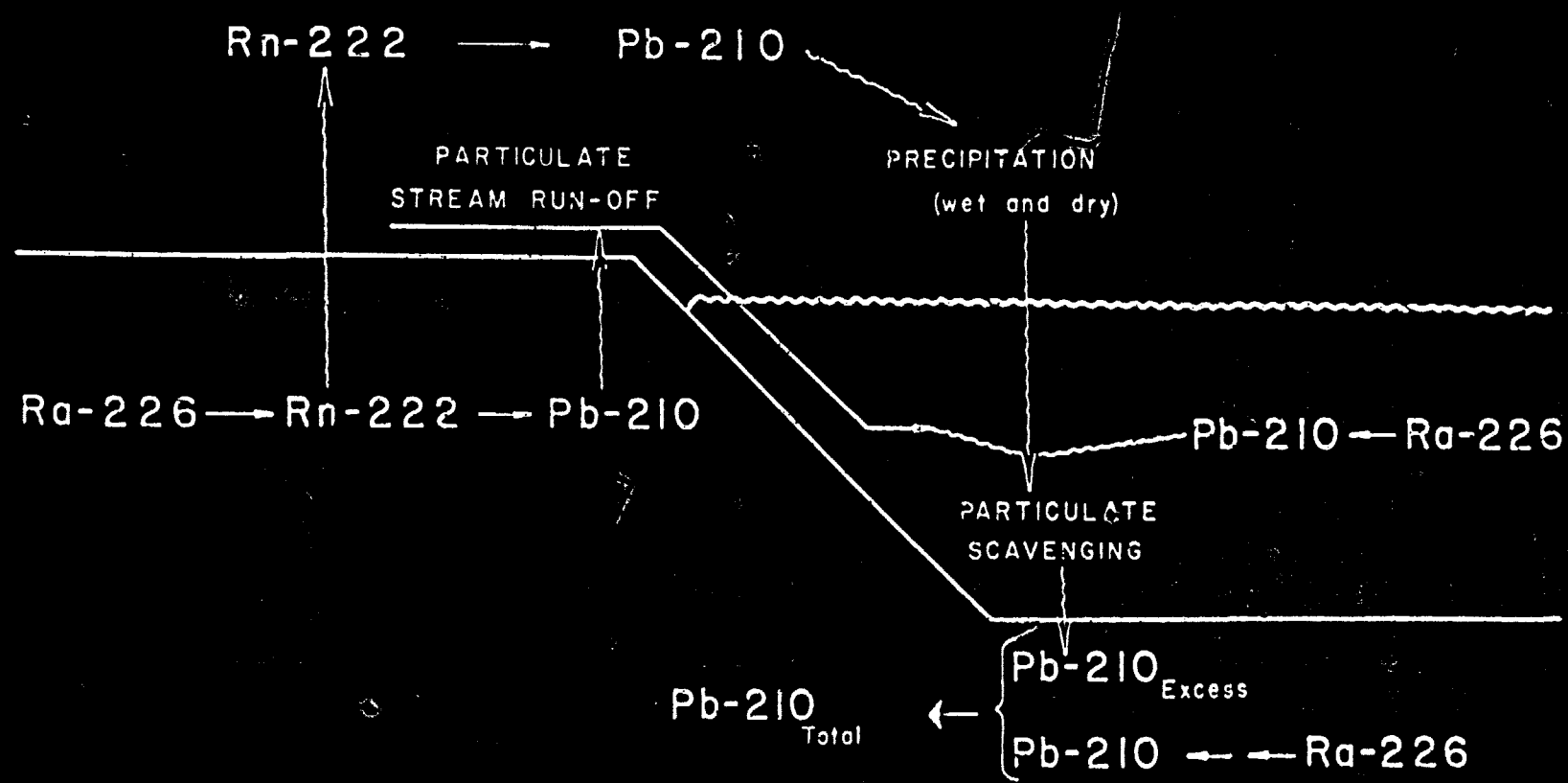


Fig. 2. Geochemical cycle for ^{210}Pb.

Sampling

1. Site Selection and Core Criteria

A. Sampling sites should be representative of larger areas of the system to be studied. If the study area is a small lake, site selection is not too difficult; if the study area is a complex one, such as an estuarine system, the selection becomes more complicated.

B. Sites should be located in areas where sediment deposition is suspected to have been constant (no major surges of sediment supply or periods of sediment erosion). The textural composition of a vertical section of the sediment should be as homogeneous and fine grained as possible and should consist predominantly of silt and clay.

C. Post-depositional disturbances, such as bioturbation, wave and wind actions, and gas generation, should be absent. Areas having anoxic bottom waters at least part of the year generally have less biological activity. Also, the area should be in a relatively natural state; that is, free of all dredging activities including dredge dumping.

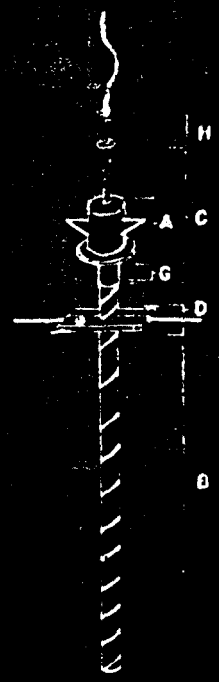
D. If the level of ^{226}Ra-supported ^{210}Pb activities is to be estimated from ^{210}Pb profiles, the cores should include sediment that is at least 150 years old. If the suspected sedimentation rate is greater than 1 cm per year, a 1 m core will not be sufficient to determine background by this method. A sediment rate can sometimes be estimated from the suspended load in the system or from subbottom profiles, if they are available. If no background sedimentation information is available, a trial core may save a later wasted effort. If a preliminary core is not possible, use the "best educated guess."

E. The diameter of the core must be large enough to obtain approximately 10 g of dried sample material within a depth interval that gives the desired resolution in the ^{210}Pb profile. Moisture content data, if available for the sediment in the study area, would be useful in making this determination.

Optimum cores are from minimally disturbed and preferably anoxic areas, have the uppermost sediment layer intact, contain uniformly textured sediments, are of sufficient length for background determinations, and have large enough core diameter that sufficient sample material can be obtained for analyses after drying.

2. Sampling Devices

A. Diver-obtained cores are excellent in quiet, shallow environments where water depths are less than 100 ft and there are no strong currents. Figure 3 shows the parts of the diver-coring system used by the authors. The weight of the hammer (A) can be adjusted to the coring environment. We have several hammers to fit this setup; their weights vary from 2 to 20 pounds. The authors use a split hydroplastic core barrel which has been taped together and attached to a diver corer. The presplit barrel (B) is inserted into the sleeve (C). After the barrel has been inserted into the

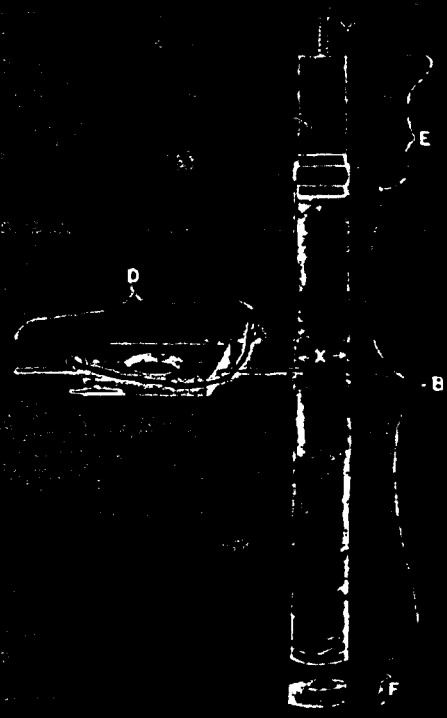


LEGEND

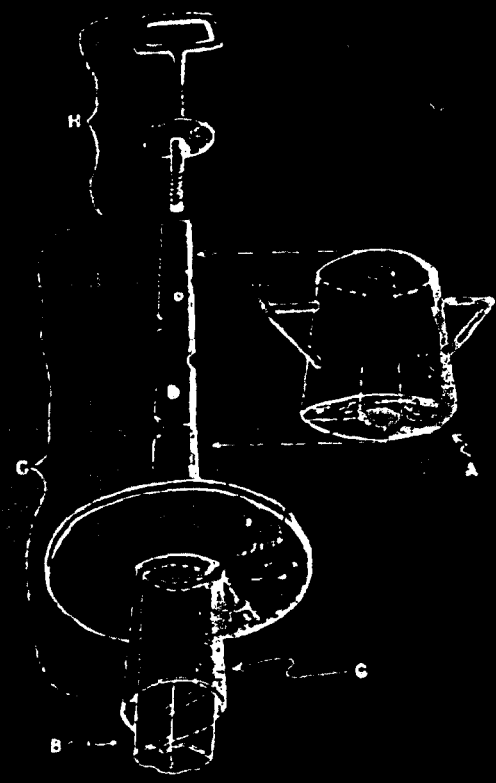
A. Hammer 25/50 lbs. (14" high)
B. Split core barrel

C. Hammer support (23 1/2" long), barrel support (6 1/4" long)
D. Core retriever
E. Core sealer (1" gasket)
F. Core cap
G. Sleeve (barrel support, 6 1/4" long)
H. Hammer retainer/cable attachment (13" long)

ASSEMBLED DIVER CORING DEVICE



CORE BARREL WITH RETRIEVER, SEALER, AND CAP



DIVER-CORER ASSEMBLY

Fig. 3. Coring Device operated by divers to obtain shallow cores.

sediment column, the hammer and sleeve are removed, an aluminum handle (D) is attached to the core barrel, a plug (E) is inserted into the top of the core barrel, and the core is retrieved from the sediment. A PVC (polyvinyl chloride) cap (F) is placed on the bottom of the barrel before the core is transported to the surface. The presplit barrel allows the core to be split easily, in many cases with a stainless steel wire, resulting in minimum disturbance of the sediment. The advantage of this system over other coring devices is that the core barrel can be pushed slowly into the sediments, thus minimizing compaction and disturbance of the sediments. This system works well in soft sediments, such as those found in many lakes, bays, and estuaries. It has been used successfully in the Potomac River and Estuary and Upper Klamath Lake, Oregon.

B. Box cores are good for recovering undisturbed cores in deeper water where diver-obtained cores are not possible. These have been used successfully on the Washington shelf (Nittrouer, 1978) and the south Texas shelf (Holmes and Martin, 1978). Slow entry and clear venting allow recovery of an undisturbed sediment column.

C. Conventional piston coring devices generally disturb the strata and usually result in incomplete recovery; as much as 2-3 ft of the surface strata may be lost. If loss of the upper layers of the sediment column is not taken into account, a systematic error in the age of various sediment layers may result. In many cores, lost sediment layers may represent the total range of ^{210}Pb chronology.

D. Gravity coring devices often recover cores in which the integrity of the sediment column has not been maintained; significant disturbances occur, predominantly near the sediment-water interface. Small-diameter (less than 3 in.) gravity corers cause significant shortening or compaction during penetration, especially if the sediment is fine grained. Slowed entry improves the quality of a gravity core, but, even under the best of conditions, the resulting core does not accurately represent the sediment column.

Generally, the following criteria should be observed in selecting a coring device:

1) Precautions should always be taken to assure that the top layer of the sediment is not lost.
2) Every effort should be made to ensure that each core has been recovered with only a minimum of disturbance of the sediment column.
3) Rapid-entry small-diameter gravity- and piston-coring techniques should be avoided in favor of diver-obtained cores or slow entry, clear vented large-diameter coring devices such as box cores.

3. Field Sampling Methods

When possible, cores should be subsampled in the field before transportation to a laboratory. During transportation, disturbances such as exposure to large fluctuations in temperature and general movement of the cores may cause loss of core integrity. The following are suggestions for sampling procedures.

A. Do not freeze the core until it has been subsampled for ^{210}Pb analyses. Freezing distorts the structure by expanding intervals, especially those containing large amounts of interstitial water. Freezing may result in errors in assigning activities to specific intervals and, consequently, errors in rates calculated from these activities. If the cores need to be maintained for other analyses at a temperature colder than can be achieved in a refrigerator, subsample the core and then freeze the subsamples.

B. If the study area is gas charged, subsample rapidly to avoid as many of the disturbances caused by expanding gas as possible.

C. Plan to obtain X-radiographs of the core aboard the sampling vessel if facilities are available, or plan to transport an unsampled core half to an X-ray facility. The top and bottom of the core barrel should be clearly marked to avoid orientation complications later. X-radiographs permit detection of bioturbation, structural discontinuities, and evidence of disturbance incurred during the coring operation. Detection of features caused by disturbance is especially useful when sampling recent estuarine sediments; zones of fine sediment deposition usually lack laminations and textural differences that are visible to the eye. Some of the effects of mixing can be detected only on X-radiographs until textural analyses have been completed.

D. Take as many subsamples as it is feasible to analyze. Accurate, closely spaced sampling intervals with a minimum of contamination are required for optimum resolution. Close sampling allows for assessment of sedimentary events such as flood deposits and for elimination of erroneous rates which are skewed too heavily by isolated events.

E. Avoid sampling the outer edges of the core, that is, the sediment touching the outside wall of the coring device. Sample only the inside semicircle material from split cores. When possible, scrape and discard sediment from exposed surfaces to avoid overlapping contamination from the side of the coring device and to prevent complications caused by downcore "slop."

F. Obtain a sufficient amount of sediment so that replicate analyses can be made. Knowledge of the water content of the sediments is helpful in determining the amount of sediment required.

G. Use a noncontaminating sampling knife such as a teflon spatula to obtain the subsample. If the sediment is too stiff, a stainless steel knife may be used. The spatula or knife should be cleaned after each subsample to avoid contamination. Subsample containers should be clean (acid-washed if possible) and capable of being sealed to avoid water loss. Snap-cap vials or whirl-pak bags are adequate for most samples.

4. Laboratory Methods

Lead-210 Procedure (modified from Flynn, 1968) (alpha count)

A. Wet subsamples are placed into precleaned and preweighed porcelain evaporating dishes, allowed to dry under heat lamps at 109°C, cooled, and

reweighed to determine the water loss (these data are used later to normalize depths in the core).

B. Dried samples are ground to a fine powder in a ceramic mortar to obtain a homogeneous sample for analyses.

C. Subsamples (5 g) are weighed into precleaned, preweighed small crucibles and heated in a muffle furnace at 110°C for 6 hours until a stable weight is obtained. The samples are then allowed to cool in a desiccator and reweighed.

D. The dry sample is transferred to a 100-ml Teflon beaker using about 5 ml of reagent-grade 16N nitric acid (HNO_3). An appropriate amount of calibrated ^{208}Po spike is then added and the sample is swirled to mix the spike.

E. The solution is evaporated to dryness under heat lamps at 109°C. The dried sample is washed from the sides of the beaker with 8N hydrochloric acid (HCl) and swirled again to insure proper mixing. The solution is evaporated to dryness again and allowed to cool.

F. One-milliliter aliquots of 30 percent hydrogen peroxide (H_2O_2) are added until the sample is thoroughly wet, and this solution is again evaporated to dryness under heat lamps. The sample should not be placed under the heat lamps until the peroxide reaction has subsided. The cooling and peroxide steps are repeated two additional times.

G. The sample is then subjected to two washings with 8N HCl and evaporated to dryness between each washing. This is to remove all traces of the nitric acid which would interfere with autoplating onto the silver planchet.

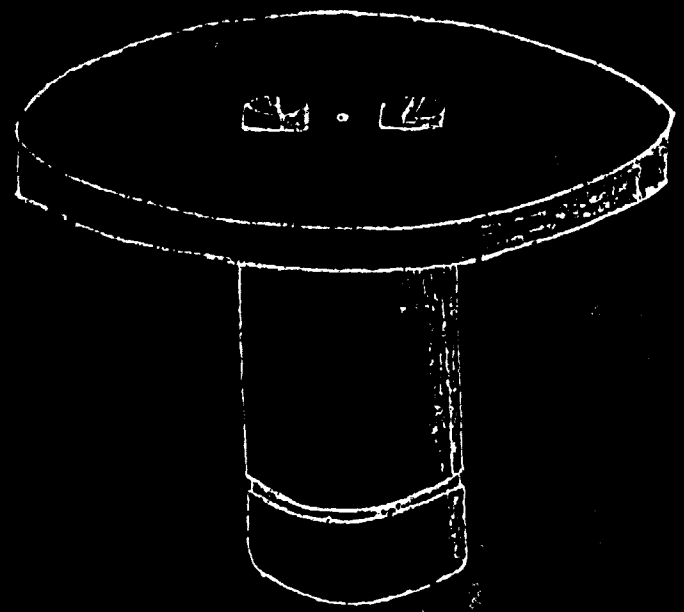
H. Five milliliters of 8N HCl are added to the final dried sample, and it is transferred to a glass beaker using additional amounts of HCl and deionized H_2O.

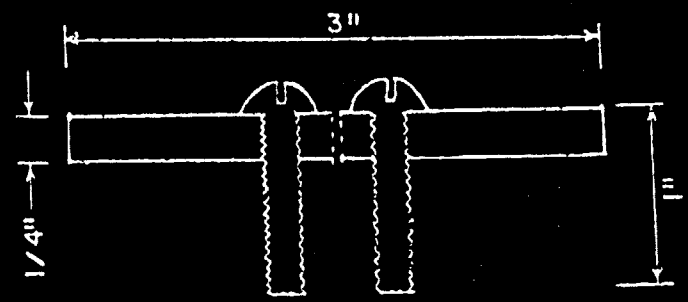
I. To minimize the interference of Fe^{+3}, Cr^{+6}, and other oxidants, 5 ml of 20% hydroxylamine hydrochloride and 2 ml of 25% sodium citrate are added. Also, 1 ml of a holdback carrier, $BiNO_3$, is added to prevent the deposition of ^{212}Bi.

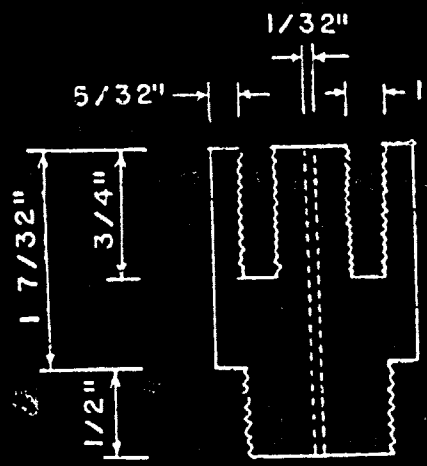
J. The pH of the resulting solution is adjusted to between 1.8 and 1.9 using HCl and ammonium hydroxide (NH_4OH) to tolerate any tellurium and selenium whose presence decreases the plating efficiency.

K. The beaker is placed on a stirring hot plate and heated to between 85°C and 90°C for 5 minutes to reduce any Fe^{+3}, Cr^{+6}, or other oxidants that might be present.

L. The Teflon plating device is added as shown on figure 4. It holds a silver foil disc 0.0127 cm thick and allows plating on only one side. The plating device covers the beaker, and only minimum evaporation occurs during the plating process.







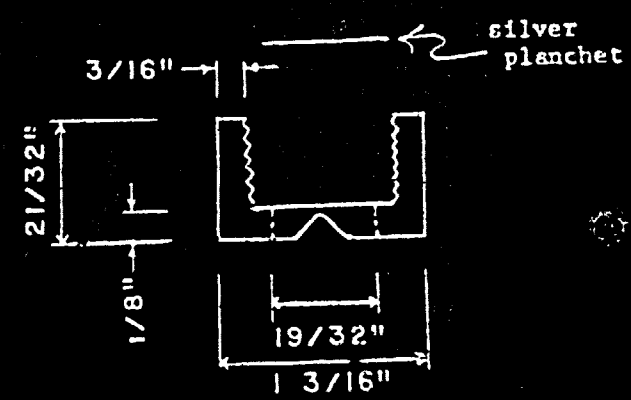


Fig. 4. Apparatus used for autoplating (modified from Flynn, 1968).

M. The heating and stirring continues for a minimum of 90 minutes.

N. The apparatus is disassembled, and the silver disc is washed with deionized H_2O, dried, and then counted on an alpha counting system.

An alternative to this method involves a dry ashing step in place of Step C. We find that ashing some samples at 400°C results in no significant loss of ^{210}Po. The deviation with replicate samples for the dry ashing procedure was less than the deviation observed with the wet ashing procedure using subsamples from the same core. This alternative is used only when the organic content is large enough to cause interferences with the extractions and plating steps of the procedure.

Textural Considerations

One of the primary assumptions of ^{210}Pb dating is that the initial activity at the top of the core has remained constant throughout the time interval to be dated. Variation caused by textural changes violates this assumption. Generally, higher activities are associated with finer sediments (Nittrouer and others, 1979). Cores interbedded with layers of sand and mud may show activity changes which are associated with complex stratigraphy and are not actual changes in sedimentation patterns. Cores containing layers high in nondecomposed organic matter (such as wood chips or peat moss) also may have very complicated profiles. Therefore, only cores that are fairly homogeneous in texture should be analyzed by radiometric techniques in general and by ^{210}Pb activity dating in particular.

Textural measurements should be made on a number of intervals of each core to ensure that activity changes in the profiles are not the result of a complex stratigraphy. These measurements may make possible the interpretation of complicated activity profiles when it is not possible to obtain cores which are homogeneous throughout their length.

Bioturbation and Other Mixing

Knowledge of physical and chemical processes that disturb the sediments after they have been deposited is very important in the interpretation of ^{210}Pb activity profiles. These postdepositional processes include bioturbation, erosion and mixing by wave and current action, and gas generation. In shallow areas, even plant roots and bottom trawling by fishermen can disturb the sediment column. Deposit feeders can cause higher water contents within the sediment that will allow it to be eroded more easily. Few environments are completely undisturbed by some type of postdepositional mixing, and these processes must be considered when studying rates of sediment accumulation.

Recently, several studies have been conducted to assess the influence of bioturbation on sedimentological records (Davis, 1974; Guinasso and Schink, 1975; Peng and Broecker, 1979; DeMaster and Cochran, 1977). These studies show that bioturbation can influence accumulation rates determined by radiometric dating techniques in areas where sediment accumulation is slow and the degree of reworking of the sediment column is intense.

However, in environments of rapid accumulation or those having a minimum of biological sediment mixing, the influence is small.

In some areas, the problems caused by mixing can be overcome. If the sedimentation rate in the study area is rapid (in the range of centimeters per year) and the mixing is not too deep, a rate may be calculated for deposits below the disturbed layer of the sediment column. However, if the rate is slow (in the range of millimeters per year), extreme caution should be exercised in evaluating the activity profile. If all the excess ^{210}Pb has decayed in the mixed layer (that is, the mixed layer encompasses the sediment column that represents 100-150 years), no rate can be calculated. In areas where all the excess ^{210}Pb has decayed, accumulation rates are sometimes undeterminable owing to the thickness of the sediment mixing layer and the slow deposition rates. Mixing models also may be used to separate the influence of mixing from the decay of the excess ^{210}Pb. Several of these models have been proposed and used with varying degrees of success (Guinasso and Schink, 1975; DeMaster and Cochran, 1977).

Steps in Data Reduction

1. The ^{210}Pb activity is determined for each sampling interval of each core.
2. To ensure that the activity of each sample does not reflect variations in salt content, a correction is made for the salt that is added to the sample weight during drying.
3. The water content is normalized to the lowest value observed in the bottom portion of the cores. Normalizing factors are applied to each interval of each core. The normalized intervals are restacked to form a water-normalized core.
4. Background activity (^{226}Ra-supported ^{210}Pb) is subtracted from each individual activity. Background levels are estimated for the cores that were not of sufficient length to allow a background to be assessed directly, or a ^{226}Ra measurement is subtracted from each interval.
5. Excess activities (activities minus background) are plotted against normalized depths for each core, and sedimentation rates are calculated over intervals of straight-line decay.

Step 1: Determining Activity

When evaluating the raw activity data, the following factors need to be considered:

a) The ^{208}Po spike must be calibrated and the activity must be recalculated for each set of activities to account for the continuous decay of the spike.
b) The time lapse between plating and counting must be taken into account.
c) Tail corrections for resolving overlapping ^{208}Po and ^{210}Po peaks are applied when necessary.
d) Statistical factors must be considered (a low count means low precision).

A typical spectrum obtained by alpha counting for ^{208}Po and ^{210}Po is shown in figure 5. When the polonium spectrum shows the tail of the ^{210}Po peak underlying the ^{208}Po peak, the peaks should be resolved. If both peaks are assumed to have the same shape, they can be separated algebraically. The method used by the authors is outlined by Benninger (1976).

At least 1000 counts should be obtained for ^{210}Pb spectra; obtaining this many counts will give counting errors of slightly less than 3% (500 counts give a counting error of less than 5%). In addition to obtaining enough counts, the researcher should evaluate secular equilibrium by measuring the ^{210}Po activity of the solution again after 6 months, during which time the ^{210}Pb has decayed to form new ^{210}Po. This evaluation depends on the assumption that all of the original ^{210}Po was removed from the solution during plating.

Step 2: Correcting for salt (Required only for saline environments)

A correction needs to be made for the weight of salts added to the sample from interstitial water during drying. The following procedure, taken from Nittrouer (1978), is used to make the salt corrections in the Corpus Christi laboratory:

If A_{210} is the ^{210}Pb activity of the sample analyzed, w_1 is the mass of the sediment sample, and w_2 is the dry mass of the sediment analyzed less the salt factor w_s, then the salt-corrected activity would be calculated by:

$$A_{210sc} = \frac{A_{210} w_1}{w_2}$$

The dry mass of the sediment sample minus the salt is calculated by:

$$w_2 = w_1 - (C_w S) \frac{w_1}{C_s}$$

where C_w = water content of sample $\frac{\text{mass of water}}{\text{mass of wet sediment}}$

S = salinity of interstitial water

$C_w S$ = mass of salt per unit mass of wet sediment

C_s = sediment content of sample or $1 - C_w$

w_1 = wet mass of sediment analyze

The salinity of the interstitial water for each sampling interval would be required for optimum results. Measuring salinity would involve placing a subsample of each sampling interval in a mud squeezer, extracting the interstitial water, and measuring the salinity either by refractometry or by a titration method. In reality, measuring of salinity by this method is impractical because of the amount of time and sampling material needed.

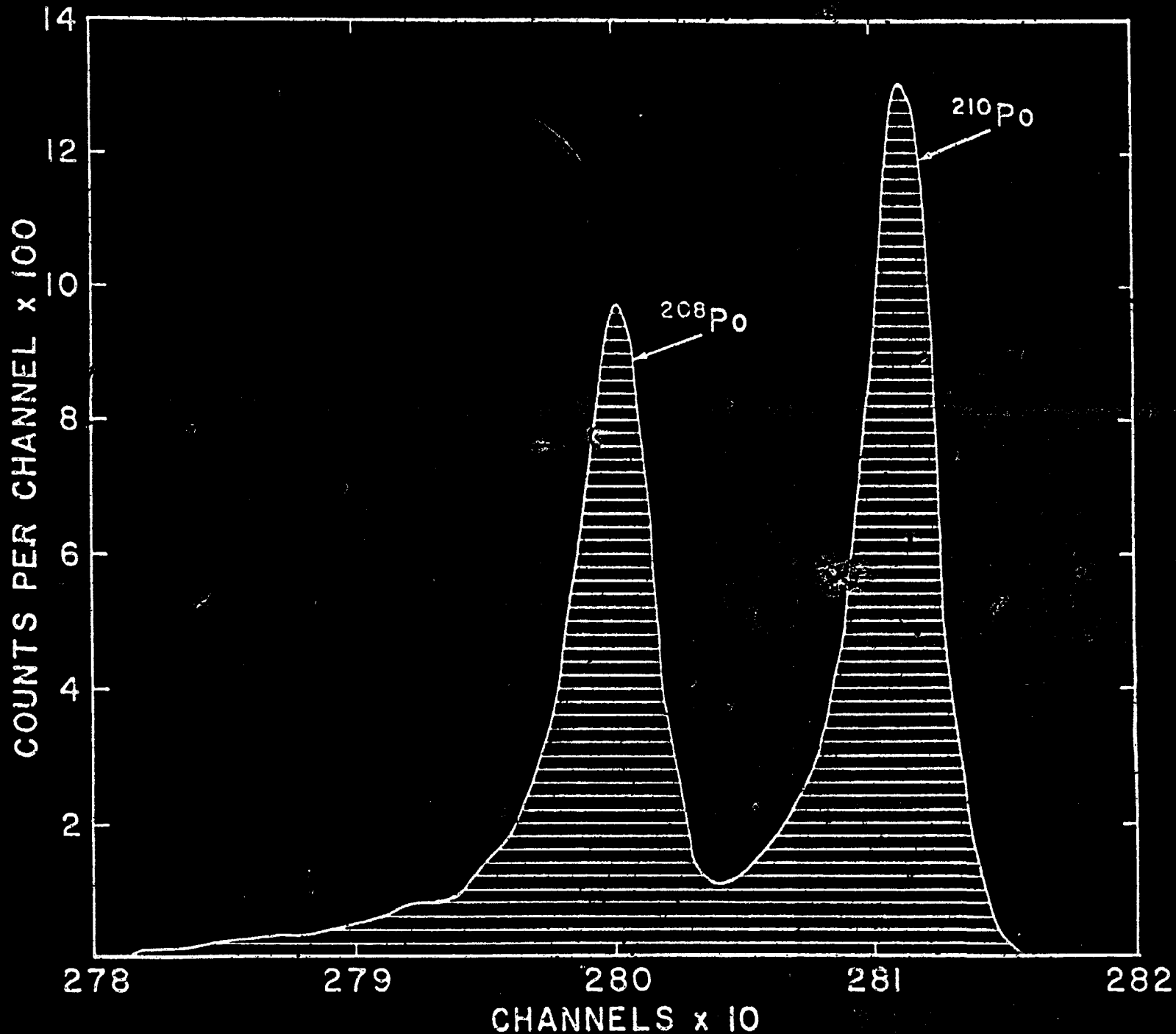


Fig. 5. Spectrum for ^{208}Po and ^{210}Po.

One of the following methods is usually used to assess the salinity:

1. The salinity of the water directly overlying the sediment at each sampling site is assumed to be equal to the salinity of the interstitial water in the core. This is a reasonable assumption in most areas if the core is short (less than 2 m) and if salinity has not fluctuated.

2. The salinities of several intervals of each core are determined by extracting the interstitial water and measuring it directly. The salinity values for the samples located between the measured subsamples are then estimated from the values directly above and below each sampling interval. If there is sufficient time and sample material, this would probably be the most accurate approach.

3. Salinity data from another study in the area can be used to estimate the salinity of the interstitial water. This is the least desirable approach; the validity of the salinity correction would depend on many factors, such as the reliability of the data used and the proximity of the data collection to the core locations of the study.

Step 3. Normalizing water content

If the sediments have a high water content in the surface strata and compaction in the lower strata, the sedimentation rate can change within the core length even though the amount of sediment deposited each year is constant. Therefore, a correction for the water content should be made. Some investigators assume that the bottom of the core represents the "normal" condition and adjust all of the water-content data to the lowest water-content observed in the bottom of the core. In some cores, lower water contents may be observed higher up in the core, but these generally result from textural differences (for example, sand layers). These layers are not considered typical of the water content of the core. In figure 6, the width of each block represents the water content of the core. In core A, the water content is higher in the top of the core and decreases down the core until an almost constant water content is reached. If the core is adjusted to the lowest water content, the result is a compacted core (B) in which the top intervals of the core are adjusted to the bottom intervals. If the top portion of the core is treated as "normal," the water content is adjusted to the highest water content observed in the core, and the result is an expanded core (C). If an average water content is used to correct the core, the result is a core in which the top portion of the sediment column is compacted and the bottom portion is expanded (D). Another method of correcting, which is not shown in figure 6, attempts to normalize the entire core to reflect no water content; the dry sediment intervals are restacked to form a dry core. This is sometimes referred to as an accumulation of dry sediment deposited per unit surface area per unit time. All the normalizing methods for water content have merit, and the one chosen by the investigator depends on the information he is seeking and the types of data he is attempting to merge. The importance of calculating rates on a uniform porosity has recently been discussed (Farmer, 1978; Behrens, 1980). The important thing to remember is to evaluate how the data were reduced and how the rates (expressed as cm/yr or g/cm^2/yr or whatever is most useful) were calculated.

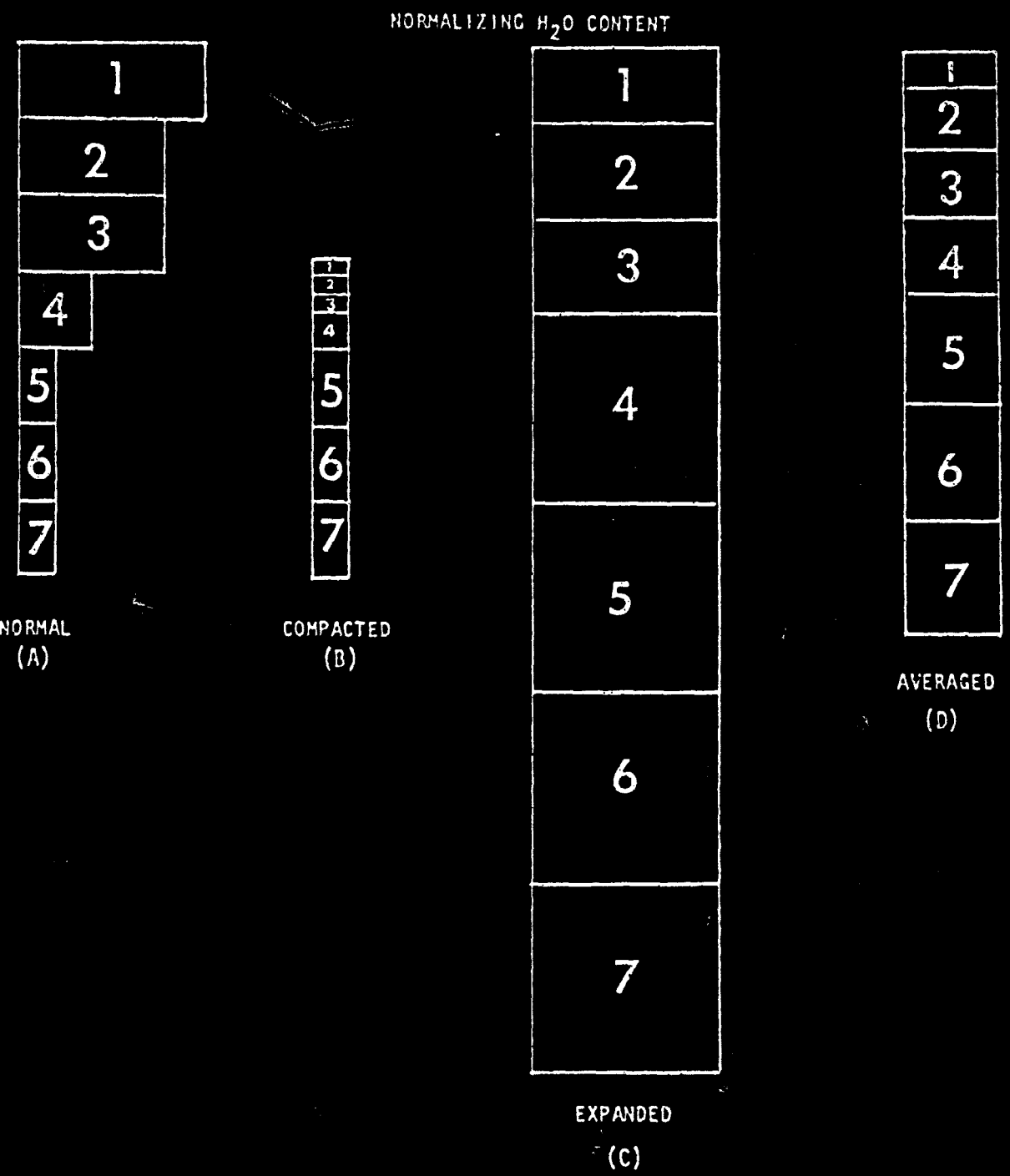


Fig. 6. Methods of normalizing a core for water content. See text for discussion.

The authors adjust the water content to the lowest water content observed in the bottom of the core; therefore, our data are reported for (compaction) normalized cores. If the measurements of water content in cores show a uniform interstitial water/sediment ratio, adjustments for sedimentation rates based on water content do not significantly change the rates.

Step 4: Adjusting for background ^{226}Ra activity

The presence of ^{210}Pb supported by the decay of ^{226}Ra in deeper layers of the sediment column must be taken into account. This ^{226}Ra is generally determined by one of three methods. Method (a) is used by the Corpus Christi laboratory in determining background activities; however, the values assigned to each set of cores should be checked by one of the other two methods:

a. Indirect

The supported ^{210}Pb activity found to be in equilibrium with its parent ^{226}Ra is indirectly determined by assuming that radioactive equilibrium is attained in the lower sections of the core (generally older than 150 yrs) which show constant ^{210}Pb activity with depth. This supported activity, which is assumed to represent ^{226}Ra activity, is subtracted from the total activity in the upper portions of the core to obtain excess ^{210}Pb activities. This excess ^{210}Pb activity decreases at a rate controlled by its half life.

b. Indirect

The sample solutions analyzed for ^{210}Pb content are purged of existing ^{222}Rn; ingrowth of radon and daughter products are counted in a scintillation counter or chamber. ^{226}Ra is assumed to be in equilibrium with ^{222}Rn, and the value obtained is subtracted from the total activities determined for ^{210}Pb.

c. Direct

The amount of ^{226}Ra in the sediment column is determined directly by separating, electroplating, and alpha counting the ^{226}Ra on the same sample intervals that were analyzed for ^{210}Pb activity (Koide and Bruland, 1975).

Values for ^{226}Ra ranging from 0.2 to 9.0 dpm/g have been reported by investigators. The ^{226}Ra activity in nearshore recent sediments can vary by a factor of 10. The ^{226}Ra correction becomes more important after significant decay of the excess ^{210}Pb has occurred. When the initial activity ratio of excess ^{210}Pb to ^{226}Ra is low, less than 5.0, calculated ages greater than 100 years can differ by as much as 50 percent. ^{226}Ra values may vary from one location to another and from one environment to another.

Step 5: Calculating sedimentation rates

Sediment rates are calculated by plotting the log (ln) of the excess ^{210}Pb activities versus normalized depths and calculating time horizons

over intervals of constant slope using the equation:

$$A_d = A_o e^{-\alpha t}$$

where A_d = activity of ^{210}Pb at a depth d
A_o = activity of ^{210}Pb at a higher reference point
α = decay constant of ^{210}Pb (0.0311 yr^{-1})
t = age of the sediment sample

A calculator program for simple linear regression is used to determine the slope. The resulting sedimentation rates are presented as unit length per unit of time (cm/yr or mm/yr). To determine the accumulation of sediment in mass per unit area per unit time ($g/cm^2/yr$), which is useful in calculating sediment budgets and sediment fluxes, the following relationship used by Nittrouer (1978) is employed:

$$R = (1 - Cw_2)\, P_s r$$

where R = rate in mass/area/time (for example, $g/cm^2/yr$)
r = rate expressed as length/time (for example, cm/yr)
Cw_2 = normalized water content of the core
P_s = density of the sediment

Caution: This equation can be used <u>only</u> when the core has been normalized.

The density (P_s) of the sediment can be measured directly by evaluating each sampling interval or by assuming an average density for the sediment in the study area.

Examples of ^{210}Pb Profiles

There are two ways to construct ^{210}Pb profiles. The total activity may be plotted against the corrected depth to show the region of radioactive decay and the background value of ^{226}Ra-supported ^{210}Pb activity. A second profile may be constructed by plotting the excess ^{210}Pb activity versus corrected depth (the region of radioactive decay over which the sedimentation rate is calculated). Displayed in this manner, the steeper the slope of the line, the slower the sedimentation rate. Figures 7 and 8 are typical total and excess ^{210}Pb activity profiles.

The profile shown in figure 9 is from a core obtained from Howard Bay, a shallow body of water connected to Upper Klamath Lake, Oregon. In this example, a region at the top of the core is mixed. The profile in this section of the core may be attributed to bioturbation (mixing that reflects burrowing and feeding activities). The X-radiograph for this core reveals a significant number of worm burrows in the top 20 cm. If there had been no X-radiograph made of the core, the top 20 cm could have been mistakenly interpreted as a sedimentation rate that was much more rapid than the rate below this section of the core. These worm burrows were numerous but very small and were not visible by simple visual observation.

The profile in figure 10 is from a core taken in the Potomac River. With the exception of the top few centimeters, the entire core displays the same ^{210}Pb activity. This constant activity most likely reflects the background activity supported by ^{226}Ra in the sediment; it would appear

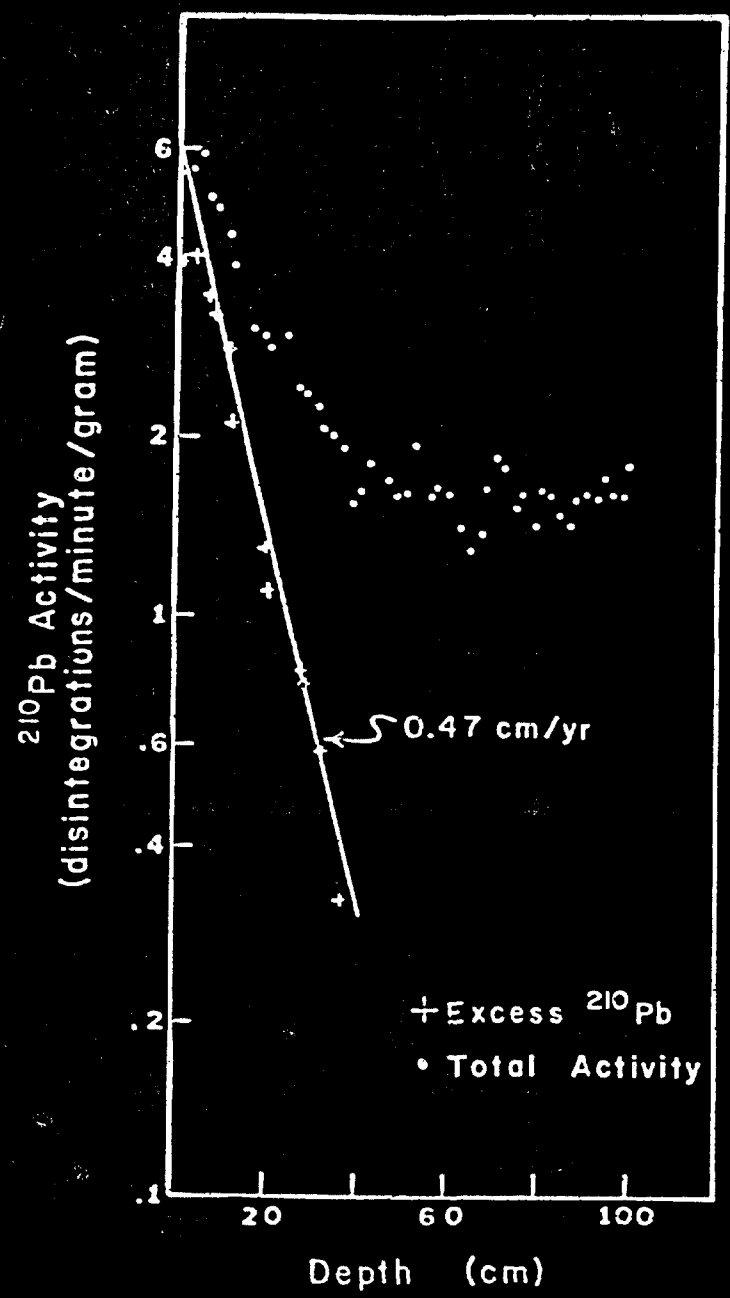


Fig. 7. Total and excess ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in a core collected from the Potomac Estuary.

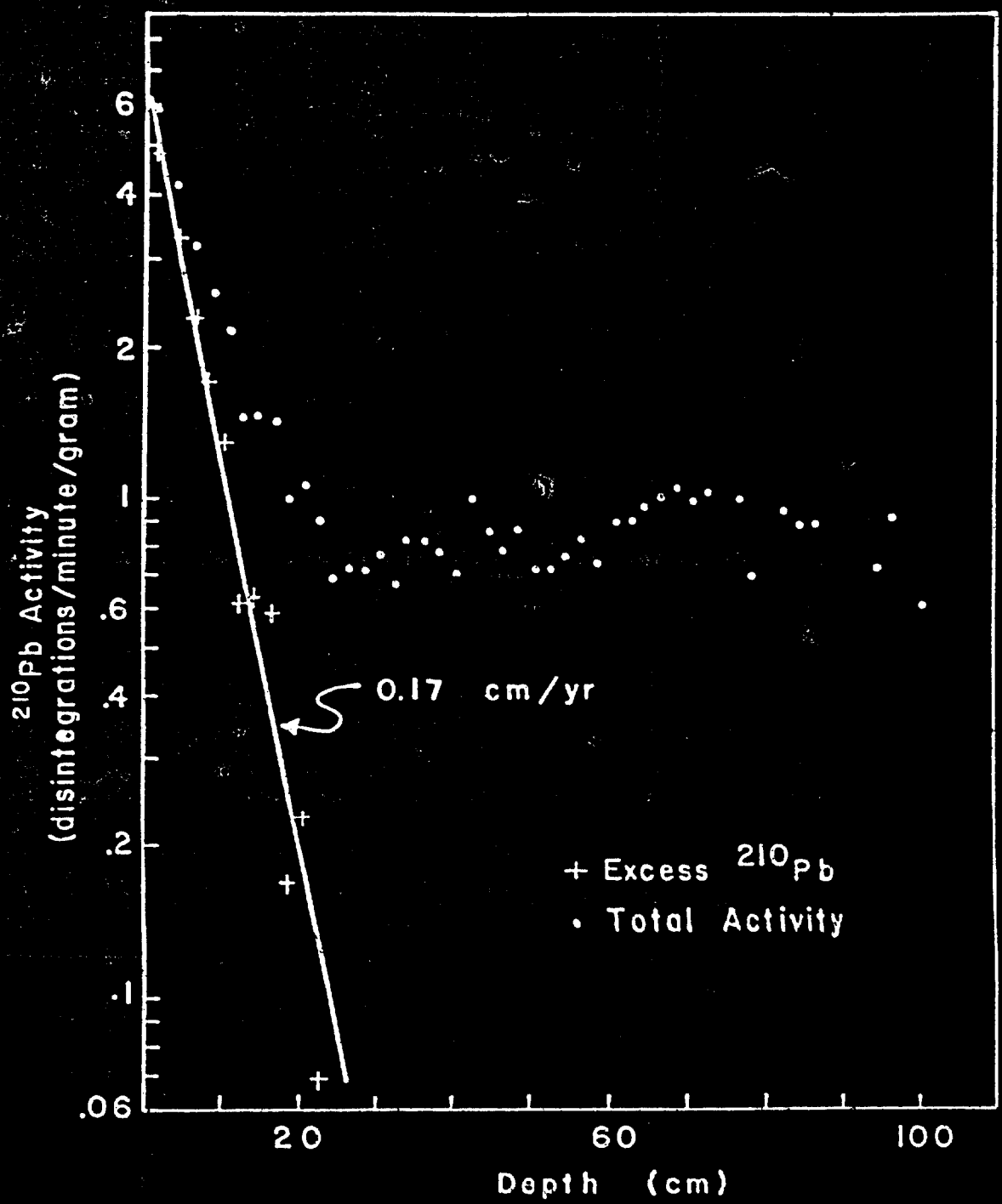


Fig. 8. Total and excess ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in a core collected in the Potomac Estuary.

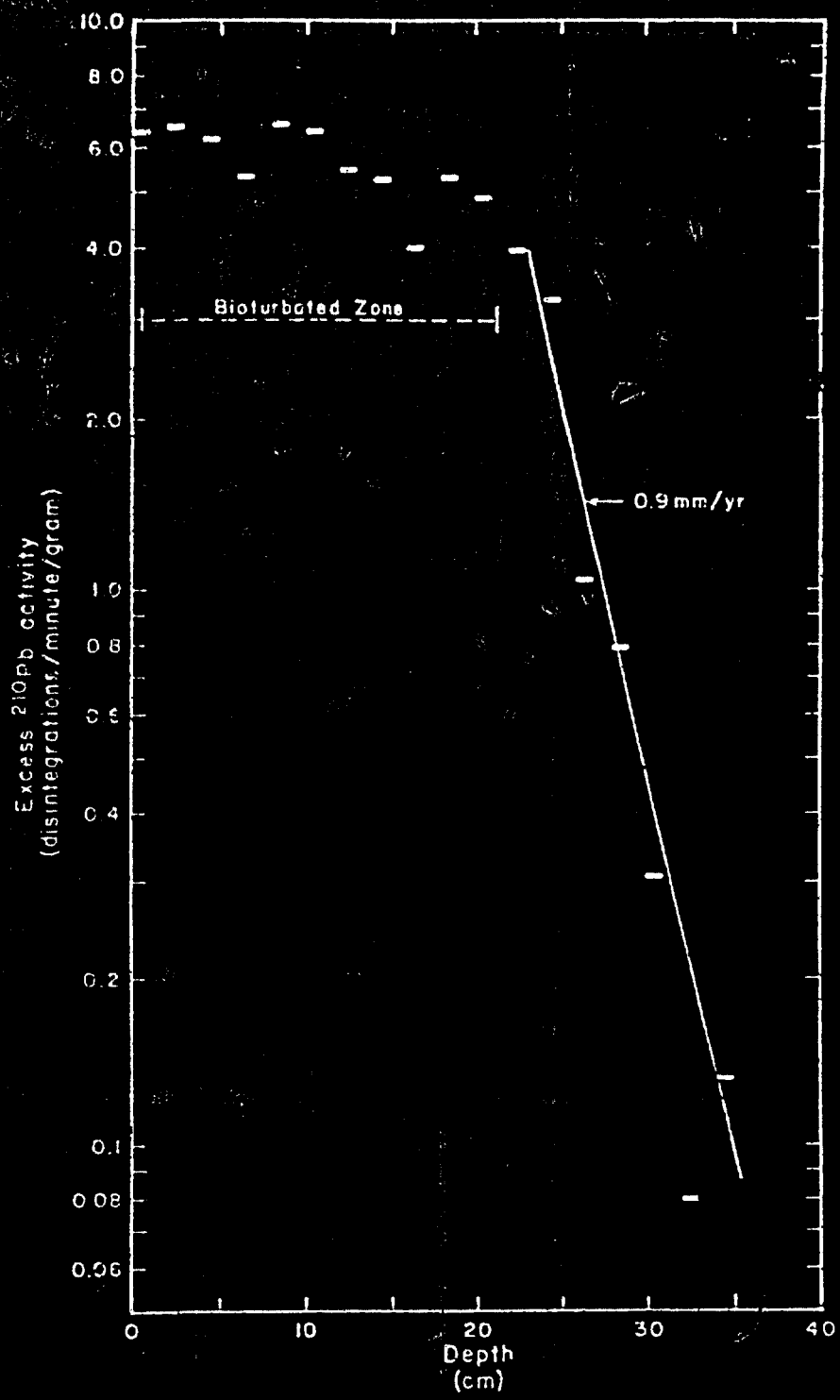


Fig. 9. Excess ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in a core obtained in Howard Bay, Upper Klamath Lake, Oregon.

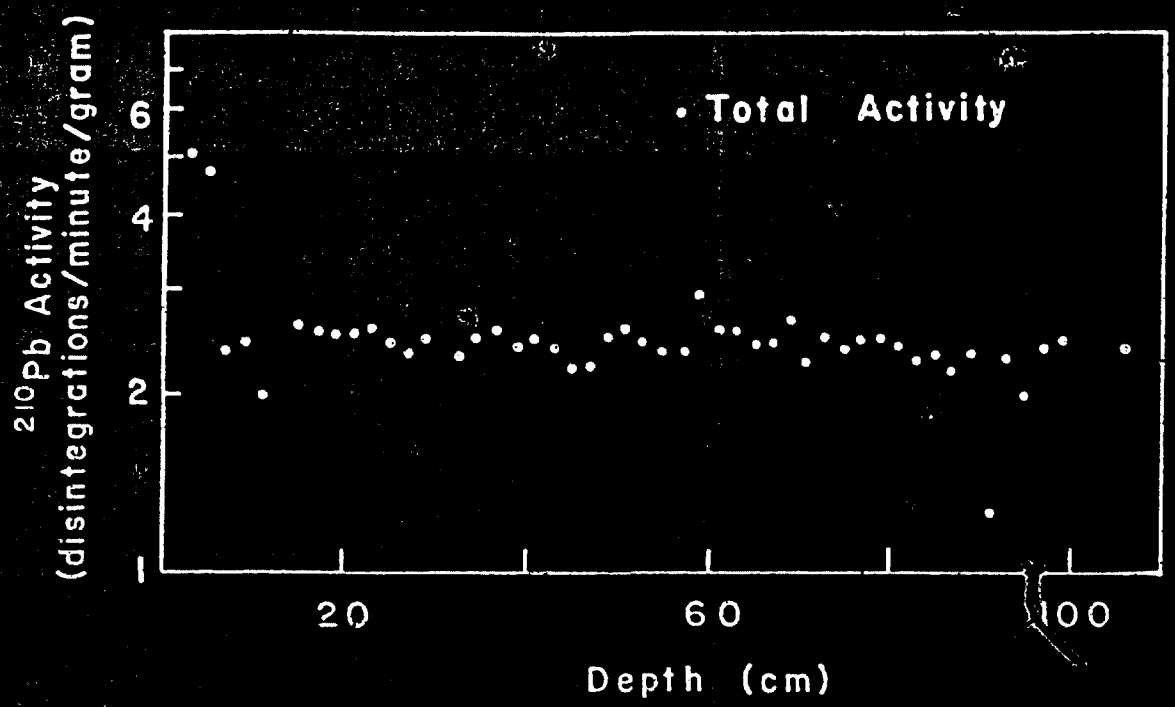


Fig. 10. ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in a core obtained from the Potomac River.

that no sedimentation has taken place in the 100-150-year span of time that is datable by this method.

Figure 11 shows a core in which there are two sedimentation rates: a recent more rapid rate and an older, slower rate of deposition. Changes in land use or natural environmental factors that cause changes in the amount of sediment supplied to a deposition area can be reflected in the profile of a core.

Events such as storms may sometimes be detected in the activity profile. Figure 12 shows an example of what may be a storm deposit. The area of constant activity in the center of the profile could have been deposited during a single episode. It is not always possible in an activity profile to obtain the resolution necessary to distinguish a short-term event. In areas of low accumulation rates (<1 mm/yr), the region in which the ^{210}Pb activity decays is very thin, and it is impossible to subsample at close enough intervals and to obtain sufficient material to perform analyses.

Figure 13 shows total ^{210}Pb activity profiles for two box cores collected in the Gyre Basin, Gulf of Mexico. One core (A) shows an excess of ^{210}Pb activity in the top 10 cm of the core. Core B displays a relatively constant ^{210}Pb activity for the entire length of the core. If nothing else were known about the cores, one might assume that deposition is taking place at core site A and no deposition is taking place at core site B. However, it is reported that the top few inches were lost from box core B. Therefore, it cannot be concluded that the absence of excess ^{210}Pb activity is a result of no sediment accumulation. This example illustrates the care that needs to be exercised when interpreting an activity profile. Many events can be indicated by activity profiles, and to give examples of each one would be impossible. Each profile should be treated as an individual problem and approached with as many additional data as are available.

Summary

^{210}Pb dating, using the excess ^{210}Pb over that expected from radioactive equilibrium with ^{226}Ra, provides a useful geochronological tool for recently (100-150 yr) deposited sediments. However, potential complications and limitations should be considered. It is important to obtain as much information as possible about the texture, salinity, moisture content, and background of the sediment column before attempting to obtain cores. The sampling endeavor is critical to obtaining good cores for dating. It is important that the core be as undisturbed as possible.

^{210}Pb dating should be supported by other sedimentological tools to provide independent estimates of the accumulation rate. Other methods include pollen horizon calculations, X-radiography, the use of other radioisotopes, and counting of varves in laminated sediment. Background activities assigned to cores should also be validated by an independent check of the ^{226}Ra activity.

Consider these general guidelines when making a decision to determine sedimentation and accumulation rates using ^{210}Pb geochronology.

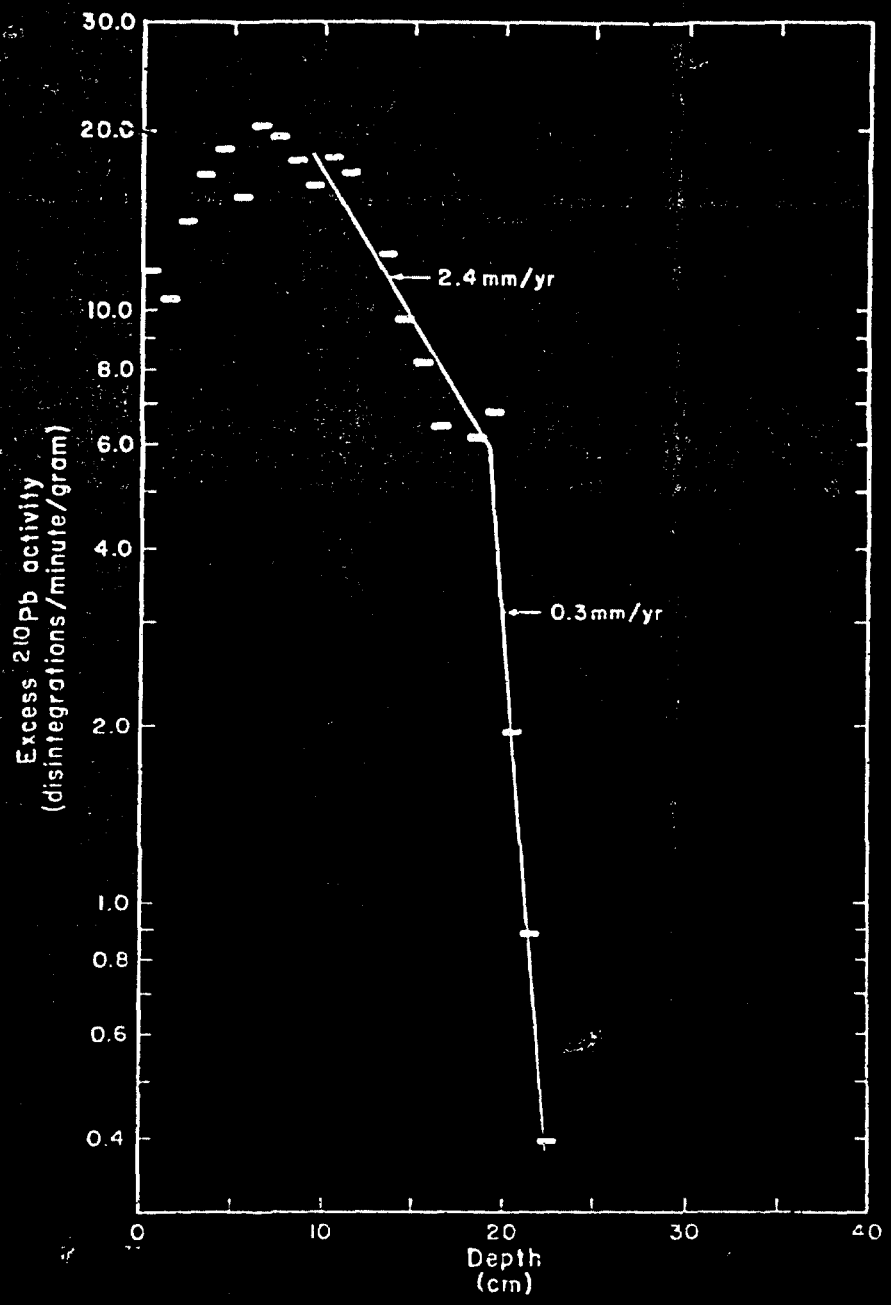


Fig. 11. Excess ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in a core collected from Upper Klamath Lake, Oregon.

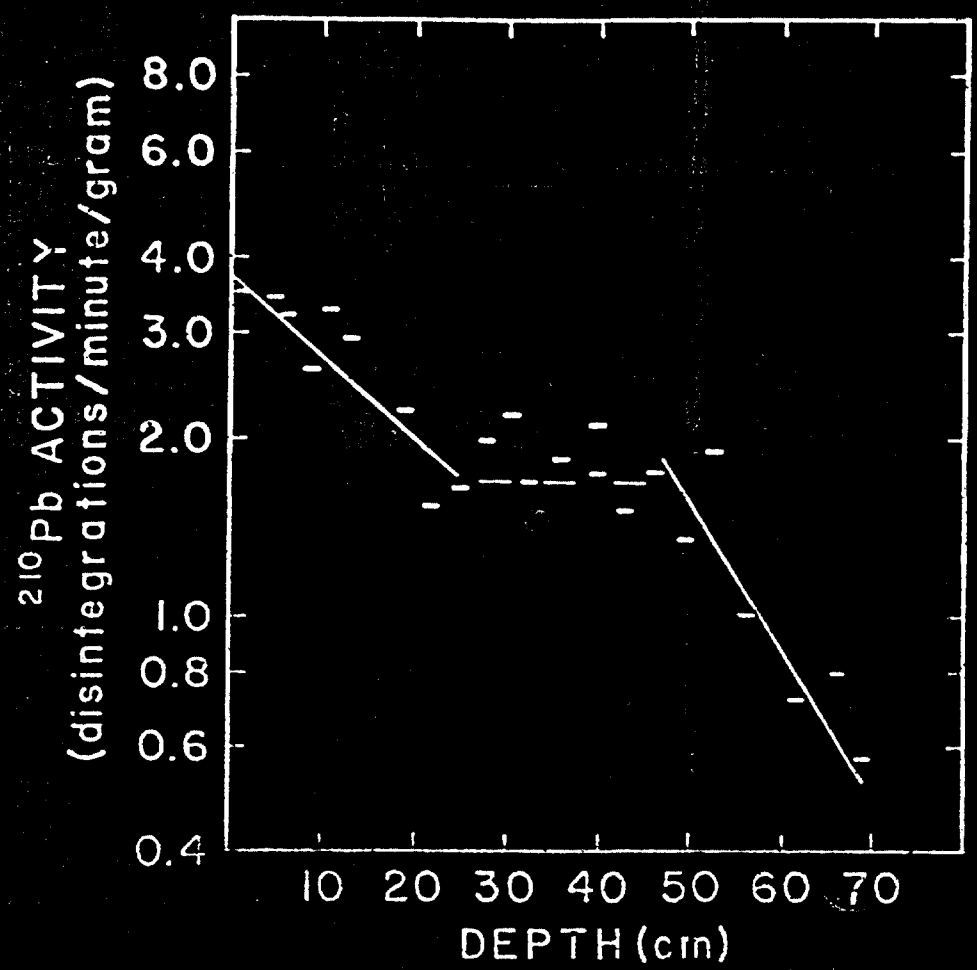


Fig. 12. Total ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in a core collected from the Potomac River.

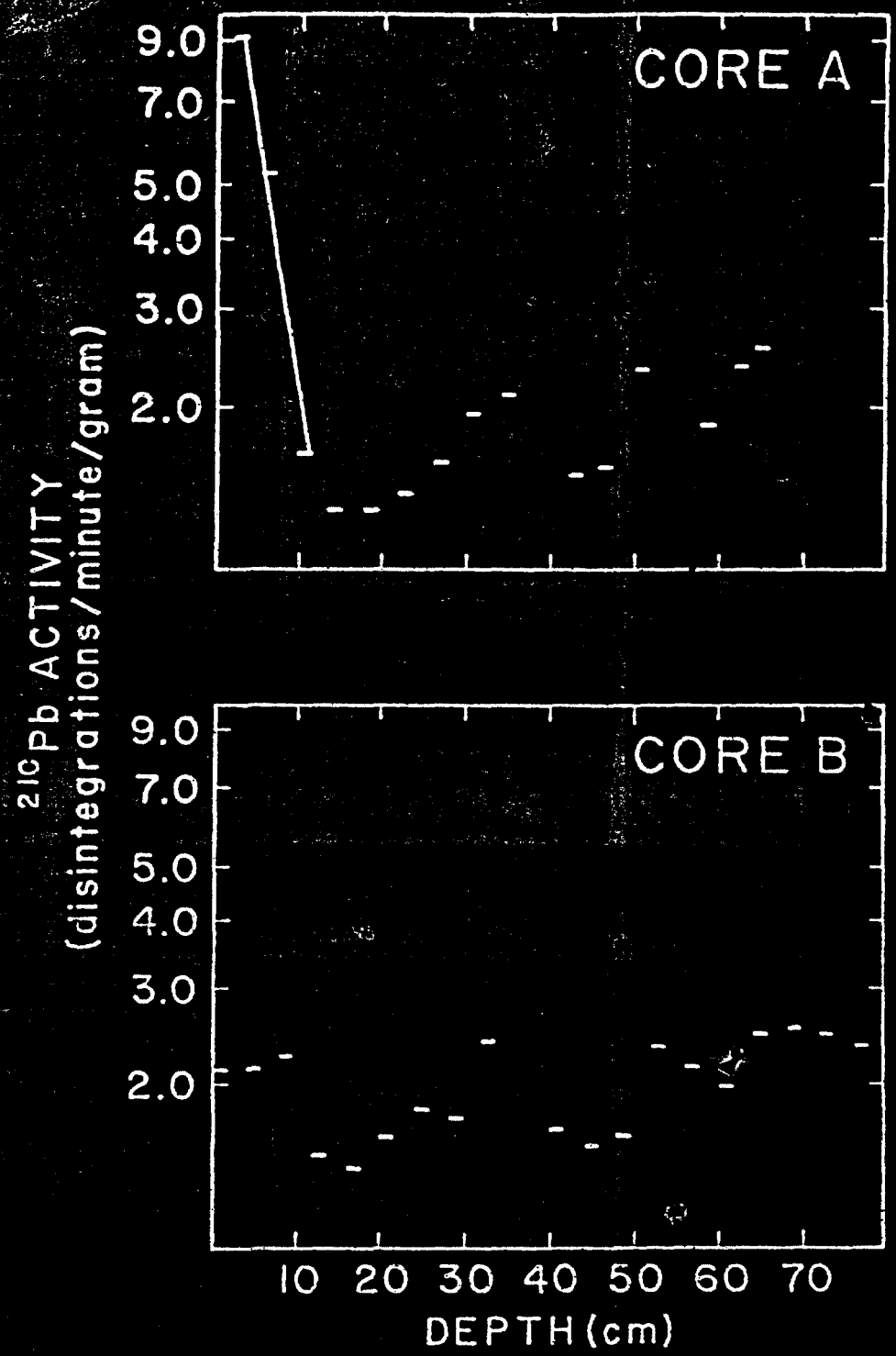


Fig. 13. Total ^{210}Pb activity (disintegrations per minute per gram) versus depth (cm) in two box cores from the Gyre Basin, Gulf of Mexico.

1) ^{210}Pb geochronology should <u>not</u> be used when:

a.) The grain size of the sediment column varies signficantly throughout the length of the core.

b.) The ratio of initial activity of the excess ^{210}Pb to the ^{226}Ra-supported ^{210}Pb in the sediment column is .5:1 or less.

c.) The area being investigated has a very low sedimentation rate (<0.1 cm/yr) and cannot be sampled in intervals small enough to provide a decay profile.

d.) The sediment column is deeply mixed by bioturbation or by other physical or chemical processes. Bioturbated cores include those that appear in the field to be undisturbed but for which X-radiographs show an intense network of burrows.

e). The study environment has a history of dredging and/or spoil dumping.

2) Conversely, optimum conditions exist for ^{210}Pb dating when sediment cores are obtained that are:

a.) Uniform in texture throughout the core length.

b.) Deposited at a rate greater than 1 mm/yr.

c.) Undisturbed by physical or chemical processes after they were deposited.

d.) Undisturbed by the coring device.

e.) Sampled in the field and X-radiographed.

f.) Characterized by an initial (surface) ^{210}Pb activity that is at least five times greater than the background activity of the sediment column.

If the above guidelines are followed and care is taken in the field sampling, it should be possible to obtain good excess ^{210}Pb profiles. Then, by means of the applicable correction factors discussed in the "Data Reduction" section, sedimentation and accumulation rates can be determined with a high degree of confidence. This method gives excellent supporting documentation for other sedimentation studies; it does not provide absolute answers, but can be a useful geochronological tool.

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EPA-NE QAPP Worksheet #9b - Rev. 04/03 (continued)

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

Medium/Matrix: Sediment
Region I Matrix Code (from EPA-NE DQO Summary Form): SE
Analytical Parameter: Geotechnical Tests
Concentration Level: NA
Field Analytical or Fixed Laboratory Method: ASTM D2216-98/D2974-00, ASTM D854-02, ASTM D4318-00

Analyte	CAS Number	Project Action HH Goal*	Project Action ECO Goal*	Analytical Method		Achievable Laboratory Limits
				MDLs	Method QLs	RLs
Water Content (ASTM D2216-98/D2974-00)	NA	NA	NA	NA	NA	NA
Specific Gravity (ASTM D854-02)	NA	NA	NA	NA	NA	NA
Atterberg Limits (ASTM D4318-00): - Liquid Limit - Plastic Limit - Plasticity Index	NA	NA	NA	NA	NA	NA

NA -- Not available/applicable.

EPA-NE QAPP Worksheet #9c - Rev. 04/03

Field and Quality Control Sample Summary Table – Allendale Pond

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference[1]	No. of Sampling Locations	No. of Field Dup Pairs	Organic		Inorganic		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks	No. of SRM or PE Samples[2]	Total No. of Samples to Lab[3]
						No. of MS	No. of MSD	No. of MS	No. of MSD					
Sediment	2,3,7,8-TCDD	Low	1613B/L-23	20 Cores (2 each of 20 cores = 40 samples)	1	1 MS/MSD set per analytical batch [a]		0	0	0	0	0	1 SRM/PE per analytical batch [a]	44
Sediment	Pb-210	Low	TBE-032-30/L-112	20 Cores (3 each of 20 cores = 60 samples)	0	0	0	1 per batch	0	0	0	0	0	60
Sediment	Cs-137	Low	TBE-2008/ L-113	20 Cores (3 each of 20 cores = 60 samples)	0	0	0	0	0	0	0	0	0	60
Sediment	Grain Size	Low	ASTM D422/L-46	20 Cores (1 each of 20 cores = 20 samples)	1	0	0	0	0	0	0	0	0	21
Sediment	TOC	Low	9060/L-49	20 Cores (1 each of 20 cores = 20 samples)	1	0	0	0	0	0	0	0	1 SRM per analytical batch [a]	22
Sediment	Water Content	NA	ASTM D2216-98/ D2974-00	20 Cores (1 each of 20 cores = 20 samples)	0	0	0	0	0	0	0	0	0	20
Sediment	Specific Gravity	NA	ASTM D854-02	8 (Selected from the 20 cores)	0	0	0	0	0	0	0	0	0	8
Sediment	Atterberg Limits	NA	ASTM D4318-00	8 (Selected from the 20 cores)	0	0	0	0	0	0	0	0	0	8

(See next page for footnotes.)

EPA-NE QAPP Worksheet #9c - Rev. 04/03 (continued)

Field and Quality Control Sample Summary Table -- Lymansville Pond

Medium/ Matrix	Analytical Parameter	Conc. Level	Analytical Method/ SOP Reference[1]	No. of Sampling Locations	No. of Field Dup Pairs	Organic		Inorganic		No. of Trip Blanks	No. of Bottle Blanks	No. of Equip. Blanks	No. of SRM or PE Samples[2]	Total No. of Samples to Lab[3]
						No. of MS	No. of MSD	No. of MS	No. of MSD					
Sediment	2,3,7,8-TCDD	Low	1613B/ L-23	10 Cores (2 each of 10 cores = 20 samples)	1	1 MS/MSD set per analytical batch [4]		0	0	0	0	0	1 SRM/PE per analytical batch [4]	22
Sediment	Pb-210	Low	TBE-032-30/L-112	10 Cores (4 each of 10 cores = 40 samples)	0	0	0	1 per batch	0	0	0	0	0	40
Sediment	Cs-137	Low	TBE-2008/ L-113	10 Cores (4 each of 10 cores = 40 samples)	0	0	0	0	0	0	0	0	0	40
Sediment	Grain Size	Low	ASTM D422/L-46	10 Cores (1 each of 10 cores = 10 samples)	1	0	0	0	0	0	0	0	0	11
Sediment	TOC	Low	9060/L-49	10 Cores (1 each of 10 cores = 10 samples)	1	0	0	0	0	0	0	0	1 SRM per analytical batch [4]	12
Sediment	Water Content	NA	ASTM D2216-98/ D2974-00	10 Cores (1 each of 10 cores = 10 samples)	0	0	0	0	0	0	0	0	0	10
Sediment	Specific Gravity	NA	ASTM D854-02	4 (Selected from the 10 cores)	0	0	0	0	0	0	0	0	0	4
Sediment	Atterberg Limits	NA	ASTM D4318-00	4 (Selected from the 10 cores)	0	0	0	0	0	0	0	0	0	4

[1] Complete SOP references in EPA-NE QAPP Worksheet #20.

[2] SRM samples will not be collected in the field -- instead these are fixed laboratory analytical QC samples supplied by a certified agency and prepared in the laboratory to demonstrate comparability. In cases where an SRM does not or is not available for a specific analytical parameter/media -- then a PE sample will be provided by NAE and shipped to the participating laboratory for analysis with the study samples.

[3] Total number of samples to the lab only represents total numbers of samples **collected in the field and shipped to the participating lab**. This includes authentic (study) samples and field QC samples (*e.g.*, field duplicates, and extra water for preparation of MS/MSD). Total number of samples to the lab does not include fixed laboratory QC samples (*e.g.*, MS/MSD) for soil. In this case, aliquots of sample will be used from an authentic sample to prepare the fixed laboratory QC sample -- separate and distinct samples will not be collected to satisfy the fixed laboratory QC sample requirements. Fixed laboratory QC sample (*e.g.*, MS/MSD) types and frequency are defined in EPA-NE QAPP Worksheet #11b or 24.

[4] Analytical batch contains 20 or fewer field samples.

EPA-NE QAPP Worksheet #9d - Rev. 04/03

Analytical Services Table

Medium/ Matrix	Analytical Parameter	Concentration Level	Analytical Method/SOP[1]	Data Package Turnaround Time [2]	Laboratory/Organization (Name and Address: Contact Person and Telephone Number)	Backup Laboratory/ Organization (Name and Address: Contact Person and Telephone Number)
Sediment	2,3,7,8,-TCDD (a)	Low	1613B/L-23	30-45 days	Karen Tracy Battelle Columbus 505 King Avenue Columbus, OH 43201 (614) 424-4028	Backup GC/HRMS system available at Battelle Columbus
Sediment	Pb-210 (a)	Low	TBE-032-30/ L-112	30 days	Rebecca Charles Teledyne Brown Engineering -- Environmental Services 2508 Quality Lane Knoxville, TN 37931 (865) 934-0379	Linda Bingler Battelle MSL 1529 Sequim Bay Rd. Sequim, WA 98382 (360) 681-3604
Sediment	Cs-137 (a)	Low	TBE-2008/ L-113	30 days	As above	As above
Sediment	Grain Size	Low	ASTM D422/ L-46	30 days	Ken Davis Applied Marine Sciences 502 North Highway 3 League City, TX 77573 (281) 554-7272	Peter Rosen GeoPlan Associates 30 Mann St. Hingham, MA 02043 (616) 740-1340
Sediment	TOC	Low	9060/L-49	30 days	As above	As above
Sediment	Water Content	NA	ASTM D2216-98/ D2974-00	30 days	As above	As above
Sediment	Specific Gravity	NA	ASTM D854-02	30 days	As above	As above
Sediment	Atterberg Limits	NA	ASTM D4318-00	30 days	As above	As above

[1] Specify appropriate reference number/letter from the Field Analytical Method/SOP Reference Table (EPA-NE QAPP Worksheet #17) and from the Fixed Laboratory Method/SOP Reference Table (EPA-NE QAPP Worksheet #20).
[2] Data Package Turnaround Time may increase should large numbers of samples (e.g., >40) be received for analysis.
(a) Battelle Columbus will also determine moisture content in order to present soil data on a dry weight basis.

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	Water Content
Concentration Level	NA

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	ASTM D2216-98/ D2974-00	Accuracy	Not applicable	Blank	A
			Not applicable – *No SRM for water content in sediment/soil available*	Standard Reference Material	A
			Not applicable	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			Not applicable	Surrogates	A
		Precision	ASTM D2216-98 - RPD ≤ 8%	Laboratory (Analytical) Sample Duplicates	A
			ASTM D2974-00 - RPD ≤ 20%		
			Not applicable	MS/MSD	A
		Comparability	Not applicable, see SRM above	Intercomparison exercises (*e.g.*, SRM analyses)	A

RPD = Relative Percent Difference.
[1]Reference SOP Number from EPA-NE QAPP Worksheet #13.
[3]Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	Specific Gravity
Concentration Level	NA

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	ASTM D854-02	Accuracy	Not applicable	Blank	A
			Not applicable -- *No SRM for specific gravity in sediment/soil available*	Standard Reference Material	A
			Not applicable	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			Not applicable	Surrogates	A
		Precision	D ≤ 0.08	Laboratory (Analytical) Sample Duplicates	A
			Not applicable	MS/MSD	A
		Comparability	Not applicable, see SRM above	Intercomparison exercises (*e.g.*, SRM analyses)	A

D = Difference between duplicate values.
[1]Reference SOP Number from EPA-NE QAPP Worksheet #13.
[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAPP Worksheet #11b - Rev. 04/03 (continued)

Measurement Performance Criteria Table

Medium/ Matrix	Sediment
Analytical Parameter	Atterberg Limits
Concentration Level	NA

QC results are evaluated against the measurement performance criteria (MPC) and all data that do not meet the listed MPCs will be submitted to the Project Manager for review and assessment of the potential impact of the results. Affected samples may be reanalyzed. Data that are accepted outside these criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rational for accepting the analysis thoroughly documented in the QA/QC narrative.

Sampling Procedure[1]	Analytical Method	Data Quality Indicators (DQIs)[3]	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-17, S-18	ASTM D4318-00	Accuracy	Not applicable	Blank	A
			Not applicable - *No SRM for Atterberg limits in sediment/soil available*	Standard Reference Material	A
			Not applicable	Laboratory Control Sample; Matrix Spike/Spike Duplicate	A
			Not applicable	Surrogates	A
		Precision	LL - D ≤ 9%	Laboratory (Analytical) Sample Duplicates	A
			PL - D ≤ 7%		
			PI - D ≤ 6%		
			Not applicable	MS/MSD	A
		Comparability	Not applicable, see SRM above	Intercomparison exercises (e.g., SRM analyses)	A

D = Difference between duplicate values.
LL = Liquid Limit; PL = Plastic Limit; PI = Plasticity Index.
[1]Reference SOP Number from EPA-NE QAPP Worksheet #13.
[3] Data Quality Indicators (a.k.a. PARCC parameters, *i.e.*, precision, accuracy/bias, sensitivity, data completeness, comparability).

EPA-NE QAPP Worksheet #24a - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Sediment
Sampling SOP	S-17, S-18
Analytical Parameter	Water Content
Concentration Level	NA
Analytical Method	ASTM D2216-98/D2974-00
Laboratory Name	Applied Marine Sciences
No. of Sample Locations	30

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	NA	NA	NA	NA	NA	NA
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Precision	ASTM D2216-98 - RPD ≤ 8% ASTM D2974-00 - RPD ≤ 20%
Laboratory Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	NA	NA	NA
LCS	NA	NA	NA	NA	NA	NA
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: SRM	NA	NA	NA	NA	NA	NA

Sample Set = 20 or fewer authentic study samples.
RPD = Relative Percent Difference.

EPA-NE QAPP Worksheet #24a - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Sediment
Sampling SOP	S-17, S-18
Analytical Parameter	Specific Gravity
Concentration Level	NA
Analytical Method	ASTM D854-02
Laboratory Name	Applied Marine Sciences
No. of Sample Locations	12

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Method Blank	NA	NA	NA	NA	NA	NA
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Precision	D ≤ 0.08
Laboratory Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	NA	NA	NA
LCS	NA	NA	NA	NA	NA	NA
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: SRM	NA	NA	NA	NA	NA	NA

Sample Set = 20 or fewer authentic study samples.
D = Difference between duplicate values.

EPA-NE QAPP Worksheet #24a - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Medium/ Matrix	Sediment
Sampling SOP	S-17, S-18
Analytical Parameter	Atterberg Limits
Concentration Level	NA
Analytical Method	ASTM D4318-00
Laboratory Name	Applied Marine Sciences
No. of Sample Locations	12

Data that are accepted outside the measurement performance criteria will be flagged with the appropriate data qualifier (EPA-NE QAPP Worksheet #9a) and the rationale for accepting the analysis will be thoroughly documented in the QA/QC narratives.

Laboratory QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	NA	NA	NA	NA	NA	NA
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	1/sample set	NA	Review with Project Manager; re-analyze or justify in project records	Analyst	Precision	LL - D ≤ 9% PL - D ≤ 7% PI - D ≤ 6%
Laboratory Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	NA	NA	NA
LCS	NA	NA	NA	NA	NA	NA
LFB	NA	NA	NA	NA	NA	NA
Surrogates	NA	NA	NA	NA	NA	NA
Internal Standards (ISs)	NA	NA	NA	NA	NA	NA
Other: SRM	NA	NA	NA	NA	NA	NA

LL = Liquid Limit; PL = Plastic Limit; PI = Plasticity Index.
Sample Set = 20 or fewer authentic study samples.
D = Difference between duplicate values.

EPA-NE QAPP Worksheet #24b - Rev. 04/03 (continued)

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method: ASTM D2216-98/D2974-00 (Sediment); Water Content

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits	Analytical Precision		Analytical Accuracy/Bias		
		Laboratory Replicate	MS/MSD	SRM	LCS	MS
Water Content	Not Applicable	ASTM D2216-98 - RPD ≤ 8% ASTM D2974-00 - RPD ≤ 20%	Not Applicable	Not Applicable	Not Applicable	Not Applicable

RPD = Relative Percent Difference.

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method: ASTM D854-02 (Sediment); Specific Gravity

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits	Analytical Precision		Analytical Accuracy/Bias		
		Laboratory Replicate	MS/MSD	SRM	LCS	MS
Specific Gravity	Not Applicable	D ≤ 0.08	Not Applicable	Not Applicable	Not Applicable	Not Applicable

D = Difference between duplicate values.

Fixed Laboratory Analytical QC Sample Table

Sampling SOP: S-17, S-18
Analytical Method: ASTM D4318-00 (Sediment); Atterberg Limits

Analyte	Achievable Laboratory Sensitivity/ Quantitation Limits	Analytical Precision		Analytical Accuracy/Bias		
		Laboratory Replicate	MS/MSD	SRM	LCS	MS
Liquid Limit	Not Applicable	D ≤ 9%	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Plastic Limit	As Above	D ≤ 7%	As Above	As Above	As Above	As Above
Plasticity Index	As Above	D ≤ 6%	As Above	As Above	As Above	As Above

D = Difference between duplicate values.