
To: Andy Kallus and Peter Adolphson – Washington State Department of Ecology

From: Robert Trahan, Iain Wingard and John Herzog

CC: Port of Everett – Erik Gerking
Weyerhaeuser – Carol Wiseman
Floyd|Snider – Teri Floyd and Lynn Grochala

Date: January 20, 2017

File: 0676-020-05

Subject: Work Plan Addendum for Geochronology Sampling and Analysis at the Former Mill A Marine Area, Everett, Washington

This memorandum has been prepared as an addendum to the Remedial Investigation/Feasibility Study (RI/FS) Work Plan and Marine Area Sampling and Analysis Plan (SAP) for the Former Mill A Site (Site; GeoEngineers, 2014). This addendum is being provided on behalf of the Port of Everett (Port) and Weyerhaeuser Company (Weyerhaeuser) and describes additional sample collection for a geochronology study at the Site. This addendum has been prepared to provide data to meet the requirements specified in the Work Plan.

The sample collection and testing described in this addendum is being performed to evaluate net sedimentation rates in the Marine Area of the Site and to support identification and evaluation of remedial actions. The proposed sampling is based on the results of sediment sampling and chemical analysis performed as part of the RI for the Marine Area and discussions between the Port, Weyerhaeuser, Washington State Department of Ecology (Ecology), and Washington State Department of Natural Resources (DNR) in a meeting held on January 10, 2017.

The sampling described in this addendum includes the collection of sediment core samples from three separate locations for isotope analysis. Proposed sediment core locations are shown on Figure 1.

GEOCHRONOLOGY SAMPLING AND ANALYSIS

Sample Collection and Processing

Sediment cores will be advanced at three separate locations utilizing vibracore or rotary impact core technologies. The coring methodology has been selected based on previously observed material type, water depth and vessel traffic (i.e., active shipping channel). Coring activities will utilize a 3- to 4-inch-diameter core barrel containing dedicated (disposable) clear CAB (butryn) liners. The cores will be advanced to a depth of approximately 5 feet below mud line (bml) or refusal, whichever comes first. A minimum of three cores will be advanced at each proposed sample location to facilitate selection of a minimally disturbed core, of sufficient core length to complete the sediment deposition evaluation and a core that meets the Marine Area SAP acceptance criteria for sediment cores. Additional cores will be advanced at each location if the core acceptance requirements are not met. Additional core deployments will be within an approximate 10-foot radius of the initial deployment to the extent practicable. Vessel positioning, decontamination procedures,

field documentation and procedures for the collection and processing of subsurface sediment samples are described in the Marine Area SAP.

One core from each proposed geochronology study location will be processed to collect samples for isotope analyses. Core selection will be based on evaluation of the following:

- Appearance of sediment disturbance;
- Presence of anthropogenic material (e.g., wood); and
- Recovery of sediment in the core.

The objective of the evaluation will be to identify the core(s) from each location that exhibit the least amount of disturbance, the least amount of anthropogenic material, and highest sediment recovery for processing. The optimal core for processing would be one that exhibits no apparent disturbance, contains no anthropogenic material, and has the highest recovery.

The sediment core from each location exhibiting the lowest degree of disturbance, highest recovery and least amount of anthropogenic material will be photographed and visually classified in accordance with ASTM International (ASTM) D 2488 methods and the Unified Soil Classification System (ASTM D 2487). Material descriptions, including the visual absence or presence of biota, bioturbation and/or wood debris in the sediment core will be documented. If wood debris is present, the type or types of wood debris (i.e., saw dust, bark, chips, chunks, twigs, fibers, etc.), the estimated quantity (i.e., observed percent by volume) of each type of wood debris, and the depth interval where the wood is observed will be recorded.

Sample processing for isotope analysis will include sectioning the selected sediment core from each location into 2-centimeter (cm) intervals. Initially, every third sample interval from the mudline surface to approximately 1 foot below the contact between recent deposits and underlying more consolidated material will be submitted for isotope analysis. The remaining sample intervals will be archived for potential follow-up analysis to refine the geochronology evaluation.

Figure 1 identifies the proposed isotope sample locations. Coordinates for each sampling location are presented in Table 1.

TABLE 1. GEOCHRONOLOGY CORE SAMPLE LOCATIONS

Sample Location ¹	Sample Coordinates Washington State Planes (NAD83)		Estimated Mudline Elevation ² (ft MLLW)
	Easting	Northing	
MAF-GC-01	1299187.74	360195.62	-59.3
MAF-GC-02	1298966.20	359679.01	-64.3
MAF-GC-03	1298543.59	359095.65	-76.6

Notes:

¹ Sampling locations are shown on Figure 1.

² Estimated mudline elevation based on Pacific Geomatic Services, Inc. October 2014 bathymetric survey.

NAD83 = North American Datum of 1983

MLLW = mean lower low water

Laboratory Analysis

Selected samples will be submitted to the Teledyne-Brown Engineering (TBE) laboratory located in Knoxville, Tennessee for the following isotope analysis:

- Lead-210 (Pb-210)
- Cesium-137 (Cs-137)

Analysis will be performed on a standard turn-around time (TAT) basis. The TBE laboratory's standard TAT for Pb-210 and Cs-137 analysis is anticipated to be 25 days.

Data Quality Objectives

The laboratory analytical methods, maximum holding times, data quality indicators, and quality control frequencies for the isotope analyses are summarized in Tables 2 through 4.

TABLE 2. LABORATORY ANALYTICAL METHODS AND MAXIMUM HOLDING TIMES

Parameter	Method	Maximum Holding Time	Preservative
Pb-210	TBE 2015 ¹	10 years	None
Cs-137	EPA Method 901.1	1 year	None

Notes:

¹Teledyne-Brown Engineering Laboratory's in-house method 2015 (Appendix A)

EPA = Environmental Protection Agency

TABLE 3. DATA QUALITY INDICATORS FOR SEDIMENT ANALYSES

Parameter	Precision	Accuracy	Completeness	Sensitivity (Method Detection Limit)
Pb-210	± 30%	70-130%	95%	0.1 pCi/g dw
Cs-137	± 30%	70-130%	95%	0.1 pCi/g dw

Note:

pCi/g dw = picocurie per gram dry weight

TABLE 4. QUALITY CONTROL SAMPLES

Parameter	Field Duplicate	Method Blank	Matrix Replicates	Laboratory Control Standard	Matrix Spike	Matrix Spike Duplicate
Pb-210	N/A	1/20	N/A	1/20	1/10	1/20
Cs-137	N/A	1/20	N/A	1/20	1/10	1/20

Note:

N/A = Not Applicable

EVALUATION

Sedimentation Rate based on Cs-137 Activity

Cs-137 is not a naturally occurring radioisotope; rather it is produced during nuclear fission. The primary source of Cs-137 to sediment is the atmospheric fallout from historical testing of nuclear weapons. Typically, dates of two horizons (1954 and 1963) can be identified in Puget Sound sediment. The Cs-137 results will be evaluated to identify the first appearance of detectable Cs-137 activity, which generally marks the year 1954, and the maximum Cs-137 activity, which marks the year 1963 (Jeter 2000). The thickness of sediment between these two peaks, over a 9-year period, indicates the rate of sediment accumulation at the Site.

Sedimentation Rate based on Pb-210 Activity

Pb-210 is a natural isotope that is produced in the decay series of Uranium-238. Sediment contains a background level of Pb-210 that is “supported” by the decay of Radium-226 from sediment and rocks. Recently deposited sediment also includes an excess of “unsupported” Pb-210 that is produced by decay of Radon-222 gas and incorporated into sediment by atmospheric fallout (Maureen et al., 2006). It is the excess (“unsupported”) activity that is used to estimate the age sequence in undisturbed sediment.

The background or “supported” Pb-210 activity will be determined by averaging four deepest measurements at each location where Pb-210 activity does not change appreciably between samples over depth. The excess or “unsupported” Pb-210 activity will be determined by subtracting background Pb-210 activity from measured Pb-210 activity. Deeper levels in a core correspond to earlier times, so that radioactive decay is manifested as decreasing activity measurements with depth. For this evaluation, it is assumed that the sediment is not disturbed and that the sedimentation rate is constant over time.

Sedimentation Rate Based on the Radioactive Decay Coefficient for Pb-210

The correlation between the Pb-210 radioactive decay coefficient and the ratio of excess Pb-210 concentration versus depth and is the basis for determining sedimentation rates using the Jeter method (Jeter 2000). According to Jeter, the decay of Pb-210 translates into a logarithmic (\log_{10}) decrease in excess Pb-210 concentration with depth and assumes that the sedimentation rate and the rate of Pb-210 supply are steady, and the sediment is nearly uniform in physical properties. Because radioactive decay is an exponential process, excess Pb-210 concentration varies logarithmically with depth. Therefore, the sedimentation rate is equivalent to the radioactive decay coefficient divided by the slope of the trend line between the measured excess Pb-210 concentration versus depth.

Sedimentation Rate Based on the Radioactive Decay Constant for Pb-210

Based on a model of constant and uniform sediment accumulation (Battelle 1995), sedimentation rates can also be calculated from the Pb-210 radioactive decay constant. Using this technique, sedimentation rate is estimated by dividing the radioactive decay constant by the slope of natural logarithm of excess Pb-210 activities versus depth below the mixing layer. This method assumes: (1) sedimentation rate is constant; (2) loss of Pb-210 from sediment layers occurs only by radioactive decay; (3) mixing is confined to the surface mixed layer; and (4) intervals of sediment used for analysis have well defined depositional times that are short compared to the overall dating period.

SCHEDULE

The sampling and analysis will be performed following Ecology's approval of this Geochronology Sampling and Analysis Memorandum, Work Plan addendum. In water work activities (i.e., sediment core collection) will be performed within the allowable fish window for the site. Sampling is to be conducted prior to February 15, 2017.

REFERENCES

Battelle, 1995, "Historical Trends in the Accumulation of Chemicals in Puget Sound," Battelle/Marine Sciences Laboratory, Sequim, Washington, January 1995.

GeoEngineers, Inc., "Remedial Investigation/Feasibility Study Work Plan, Weyerhaeuser Mill A Former, Everett, Washington, Ecology Agreed Order No. DE 8979," prepared for the Washington State Department of Ecology on Behalf of Port of Everett, Weyerhaeuser Company and Washington State Department of Natural Resources, GEI File 0676-020-01, Dated October 16, 2014.

Jeter, 2000, "Determining the Ages of Recent Sediments Using Measurements of Trace Radioactivity," by Hewitt W. Jeter, Teledyne, New Jersey, March 2000.

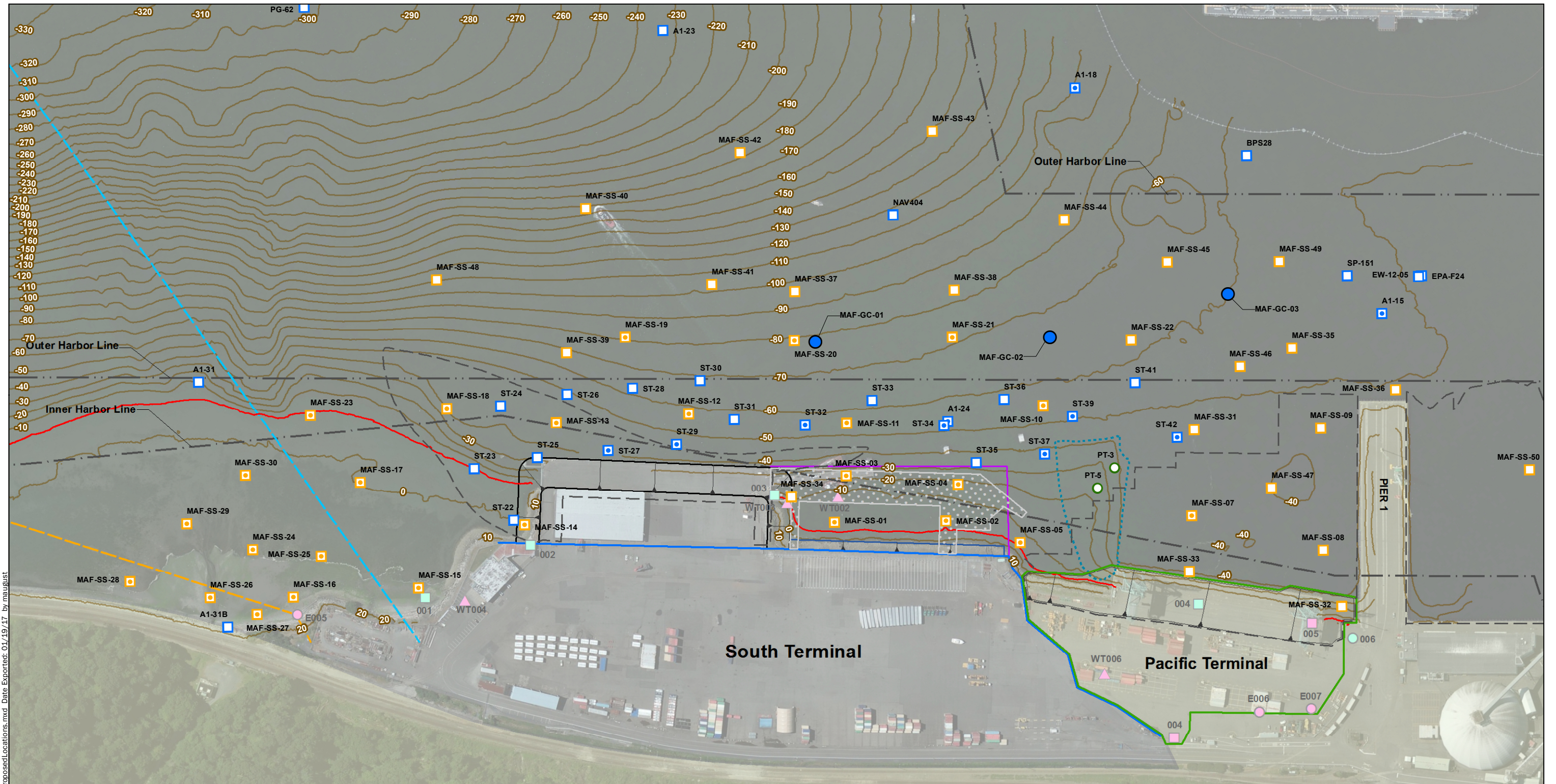
Maureen K. Corcoran and Julie R. Kelly, "Sediment-Tracing Technology: An Overview", September 2006.

RT:IW:JH:tt

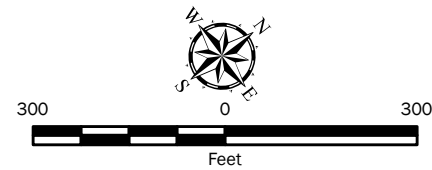
Disclaimer: Any electronic form, facsimile or hard copy of the original document (email, text, table, and/or figure), if provided, and any attachments are only a copy of the original document. The original document is stored by GeoEngineers, Inc. and will serve as the official document of record.

Attachments:

Figure 1. Proposed Sediment Core Investigation Locations for Isotope Analysis
Appendix A. Teledyne-Brown Engineering Laboratory's In-house Method 2015



P:\0_0676020\GIS\IsotopeAnalysis\067602005_F01_ProposedLocations.mxd Date Exported: 01/19/17 by maugust



Notes:
 1. The locations of all features shown are approximate. 2. This drawing is for information purposes. It is intended to assist in showing features discussed in an attached document. GeoEngineers, Inc. cannot guarantee the accuracy and content of electronic files. The master file is stored by GeoEngineers, Inc. and will serve as the official record of this communication.
 Data Source: Base aerial from Bing Maps, 2011.
 Projection: NAD 1983 StatePlane Washington North FIPS 4601 Feet

Legend

- Proposed Sediment Core Location for Isotope Analysis
- Remedial Investigation (RI) Surface Sediment Investigation**
- Surface Sediment Sample Location Only
- Surface Sediment Sample Location With Co-Located Sediment Core/Subsurface Sediment Sample (See Figure X)
- Previous Surface Sediment Investigation**
- Surface Sediment Sample Location Only
- Surface Sediment Sample Location With Co-Located Sediment Core/Subsurface Sediment Sample (See Figure X)
- Interim Action DMMP Sediment Characterization**
- Z-Layer Sample Location at Interim Action Surface
- Construction Berm Fill
- Historical Pier
- Interim Action Dredge Area
- Dredged Area
- Nearshore Confined Disposal (NCD) Facility
- 3 Feet Mean Low Low Water (MLLW) Elevation Contour Line
- Bulkhead
- Integrated Cleanup Containment and Terminal Use Area
- ▲ Historical Industrial Outfall
- Former Combined Sewer Outfall (CSO)
- Former Stormwater Outfall
- Current CSO
- Current Stormwater Outfall
- Current Kimberly-Clark/City of Everett/City of Marysville Outfall 100 (approximate)
- Historic Kimberly-Clark/Weyerhaeuser Outfall SW001 (approximate)

Proposed Sediment Core Investigation Locations for Isotope Analysis

Weyerhaeuser Mill A Former Everett, Washington

Figure 1

APPENDIX A
Teledyne-Brown Engineering Laboratory's
In-house Method 2015

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

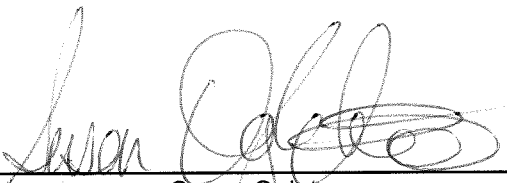
**TELEDYNE BROWN ENGINEERING
ENVIRONMENTAL SERVICES**

TBE-2015

Revision 4

Lead-210 Activity in Various Matrices

Prepared by:



Susan Ogletree
Technician

Date:

10/5/11

Reviewed by:




Lynne Perry
Quality Assurance Manager

Date:

10/5/11

Approved by:



Keith O. Jeter
Laboratory Operations Manager

Date:

10/5/11

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

Table of Contents

	Page
1.0 SCOPE AND APPLICABILITY	4
2.0 SUMMARY OF METHOD	4
3.0 DEFINITIONS	4
4.0 HEALTH AND SAFETY	4
5.0 CAUTIONS	5
6.0 INTERFERENCES	5
7.0 PERSONNEL QUALIFICATIONS	5
8.0 EQUIPMENT AND SUPPLIES	6
8.1 Apparatus and Materials	6
8.2 Standards and Reagents	7
9.0 PROCEDURE	7
9.1 Detection Capability	7
9.2 Sample Selection	8
9.3 Sample Preparation	8
9.4 Chemical Separation and Purification	9
9.5 Mounting of Precipitate	10
9.6 Sample Counting	12
9.7 Calculation of the Sample Activity or of the MDA	13
10.0 DATA AND RECORD MANAGEMENT	14
11.0 QUALITY CONTROL AND QUALITY ASSURANCE	14
12.0 REFERENCES	15

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

DOCUMENT ISSUE AND REVISION CONTROL FORM

DOCUMENT: TBE-2015, Lead-210 Activity in Various Matrices

SECTION: Environmental Analysis Department

COVERAGE: Environmental Analysis Program

ISSUE AND REVISIONS	PAGES	PREPARED BY	DATE	EFFECTIVE DATE	APPROVED BY
Revision 1	Revised 2.2, 4.3, 7.3, 9.2.1, 9.2.2, 10.1, 11.1, 11.3 thru 11.7, and deleted 11.8 thru 11.10	Bill Meyer	10/20/05	11/09/05	Bill Meyer
Revision 2	Sections 3.4, 6.2, 6.3, 8.2, 9.3.6	Lynne Perry	11/15/07	11/15/07	Keith Jeter
Revision 3	Sections 8.1, 8.2, MDA table 9.3.5, 9.3.6, 9.4.5, 9.4.8, 9.5.4, 9.5.5, 9.5.7, 9.5.9, 9.5.10, 9.5.11, 9.6.3, 9.6.4, 9.7.2, 10.1, 10.3, 11.7, 12.2	Lynne Perry	09/09/08	09/09/08	Keith Jeter
Revision 4	Sections 1.1, 5.1, 8.1&2, 9.3 note, 9.3.4, 9.3.6, 9.4.1&2, 9.4.8&9, 9.6, Deleted 9.5.10, 9.7.1	Lynne Perry	10/05/11	10/05/11	Keith Jeter

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

1.0 SCOPE AND APPLICABILITY

- 1.1 This procedure presents the radiochemical beta assay method for determining the leachable lead-210 (Pb-210) activity in sediments and soils.

2.0 SUMMARY OF METHOD

- 2.1 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 hydrochloric acid; activity in the interior of mineral grains may be excluded.
- 2.2 Stable lead and bismuth carriers are added to the dried sample and it is leached with 6 M hydrochloric acid. The sample is then filtered and the filtrate is evaporated, oxidized with nitric acid, and finally dissolved in 1.8 M hydrochloric acid. The solution is passed through an anion exchange column. Lead is eluted first with 9 M hydrochloric acid and with deionized water, then bismuth is eluted with 2 M sulfuric acid. The bismuth is precipitated as the oxychloride and is collected by vacuum filtration on a glass fiber disc. The bismuth yield is determined gravimetrically. The filter disc is mounted on a nylon planchet and covered with 3 mg/cm² aluminum absorber for beta assay in a low level, gas-flow proportional counter.

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
- 3.4 See procedure TBE-1004.

4.0 HEALTH AND SAFETY

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

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

- 4.2 When using or preparing reagents that consist of concentrated caustic or acidic 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 chemicals or hazardous reagents must be prepared and used only in a hood.
- 4.4 MSDS are available in locations convenient to the laboratories and from the Safety Manager. Refer to these for other specific safety instructions for chemicals and reagents.
- 4.5 Appropriate precautions, as specified in the TBE-ES Radiation Protection Program Manual, will be followed when handling radioactive material.
- 4.6 Before commencing any laboratory work activities at TBE-ES, all employees receive orientation and training on the TBE Knoxville Facility Safety Manual, and TBE Radiation Worker Training, as applicable.
- 5.0 CAUTIONS**
- 5.1 When adding concentrated acids, particularly sulfuric acid (H_2SO_4), to water, do so very slowly since significant heat is generated during dissolution. Prepare acid solution in a hood and cool the solution in an ice bath during dissolution, if required. Wear an apron, gloves and face shield, as appropriate.
- 6.0 INTERFERENCES**
- 6.1 NA.
- 7.0 PERSONNEL QUALIFICATIONS**
- 7.1 It is the responsibility of the analyst to heed any precautions noted by the procedure, to adhere to the instructions contained in the procedure, to report any deviation from this procedure, and to perform this procedure independently only when formally qualified.
- 7.2 It is the responsibility of the analyst performing this procedure to inspect worksheets and/or logbooks for accuracy and completeness, samples for correct volume and size, labels and tags for accuracy, equipment for correct operation, and to ensure that all calibrations for equipment used are current and not expired.

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

7.3 Analysts performing this procedure must be trained, qualified, and certified in accordance with the TBE-ES Quality Assurance Manual and procedure TBE-1007. Procedure specific training documentation for designated analysts is maintained in the QA office.

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

8.0 EQUIPMENT AND SUPPLIES

8.1 Apparatus and Materials

- Aluminum foil absorber (3 mg/cm²)
- Balance, Analytical
- Balance, top loading
- Beakers, 400 mL, 250 mL or as required
- Desiccator
- Filter discs, 2.8 cm fiberglass
- Filter discs, 15 cm fiberglass or paper
- Filter rack
- Funnel, 4" diameter
- Gas flow proportional counting system, low level
- Graduated cylinders, 100 mL
- Hot plate
- Ion exchange column with 200 mL reservoir
- Logbooks, worksheets, marking pens, labels, scissors and/or razor blades
- Magnetic stirring apparatus with stirring bars
- Nylon planchets, retaining rings
- Oven, hot air drying
- Petri dishes, 4-Way partitioned
- pH meter and pH 4 and pH 7 buffer solutions, or appropriate range pH paper
- Pipettes, Volumetric Spatula, stirring rods, watch glasses, glass wool
- Vacuum filtering apparatus

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

Standards and Reagents

- Amberlite IRA-400 C.P. Anion exchange resin, or equivalent
- Ammonium hydroxide (NH₄OH), concentrated: 15 M
- Bismuth carrier (nominal value 30 mg Bi/mL), standardized. See procedure TBE-2025, Preparation and Standardization of Carrier Solutions
- Ethanol, reagent grade
- Hydrochloric acid (HCl), concentrated: 12 M
- Hydrochloric acids (HCl), 6 M: dilute 500 mL of concentrated acid to 1 liter with deionized water
- Hydrochloric acid (HCl), 1.8 M: dilute 300 mL of 6 M hydrochloric acid to 1 liter with deionized water
- Hydrochloric acid (HCl), 9 M: dilute 750 mL of concentrated acid to 1 liter with deionized water
- Lead carrier (nominally 20 mg Pb/mL), standardization is not required. See TBE-2025, Preparation and Standardization of Carrier Solutions
- Nitric acid (HNO₃), concentrated: 16 M
- Sulfuric acid (H₂SO₄), concentrated: 18 M
- Sulfuric acid (H₂SO₄), 2 M: dilute 110 mL of concentrated acid to 1 liter with deionized water (Add acid to water with great care).

8.2 Use appropriate graduated cylinders and transfer pipettes during the preparation of the solutions cited above.

9.0 PROCEDURE

9.1 Detection Capability

Detection capability depends upon initial sample size, chemical yield, counting interval, and the background and efficiency of the counting instrument. Lower detection limits may be obtained by increasing the sample size or the counting interval.

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

Lead-210 Minimum Detectable Activity

Matrix	MDA	Sigma Level ^a	Sample Volume	Chem Yield	Counting Interval (minutes)	Counting Efficiency (cpm)	Bkgd (cpm)
Sediments & Soils ^b	0.2 pCi/gm	4.66	10 gm	0.5	100	0.23	0.3

a Sigma multiplier will be 4.66 unless otherwise specified by the customer

b A representative decay factor of 0.9 allows for one day delay in counting the planchet after lead separation.

c Efficiency of 0.23 for Bi-210 counting using a 3 mg/cm² aluminum absorber.

9.2 Sample Selection

9.2.1 Using the Request for Analysis with the LIMS number, locate the sample (or sample group) in the sample receiving and storage room. Log the samples out of the Receiving Room and return with them to the laboratory.

9.2.2 Begin entering into LIMS and/or the laboratory logbook the customer name, the sample ID numbers in order, the desired analyses, sample type, the sample preparation date and the initials of the analyst.

9.3 Sample Preparation

NOTE: Perform % moisture, if required, prior to aliquoting for lead analysis.

9.3.1 Write the sample ID number on the outside of a glass beaker using a laboratory marking pen.

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

9.3.3 Place the beaker in a hot air oven (105-120°C) overnight to dry.

9.3.4 After the sample is dry, remove and allow to cool.

9.3.5 Write the sample ID number on a clean 150 mL beaker. Weigh the beaker using a balance and enter this tare weight in LIMS/and or the laboratory logbook. Transfer approximately 2-10 grams of the dried sample to the beaker. Different sample weights may be used as necessary to meet customer requirements. Enter the aliquot weight in LIMS and/or the laboratory logbook.

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

- 9.3.6 Add 6 M HCl to the beaker, filling to the 100 mL mark. Using a calibrated pipette add 1.0 mL standardized Bi carrier to the beaker. Using a disposable pipette, add 1 mL Pb carrier to the beaker.
- 9.3.7 Cover the beaker with a watch glass and place on a moderate (approximately 200°F) hot plate. Allow the sample to leach for approximately 2 hours. Remove from the hot plate and allow to cool.
- 9.3.8 Fold a 15 cm fiberglass or paper filter disc in quarters to make a cone and place it in a 4-inch diameter funnel. Write the sample ID 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.
- 9.3.9 Add approximately 5 mL HNO₃ to the sample. Place the sample beaker on a moderate (approximately 200°F) hot plate and evaporate to dryness. Check that the sample does not spatter during evaporation and reduce hot plate temperature if necessary.
- 9.3.10 Add approximately 20 mL concentrated HCl to the sample. Place the sample beaker on a moderate (approximately 200°F) hot plate and evaporate to dryness. Check that the sample does not spatter during evaporation and reduce hot plate temperature if necessary.
- 9.3.11 Add 1.8 M HCl to the sample beaker, filling to the 150 mL mark. Warm gently on a low (approximately 100°F) hot plate to dissolve solids.
- 9.3.12 Remove the beaker from the hot plate and allow to cool. Stir the sample with a glass rod to homogenize the sample.
- 9.3.13 Further filtration may be required.

9.4 Chemical Separation and Purification

- 9.4.1 Obtain a ½-inch diameter 12-inch glass ion exchange column with a 200 mL reservoir and equipped with a stopcock at the bottom. Insert a small wad of glass wool above the stopcock.

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

9.4.2 Make a slurry of anion exchange resin (e.g. Amberlite IRA-400 C.P., or equivalent) and deionized water in a beaker. With the column stopcock open, pour the slurry into the column until the resin occupies approximately 10 inches of the column.

NOTE: New resin is used for each sample.

9.4.3 Condition the column by passing through approximately 60 mL of 1.8 M HCl. Close the stopcock when the liquid level reaches the top surface of the resin.

9.4.4 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.

9.4.5 Using extreme caution, pass approximately 150 mL of 9 M HCl through the column to elute the lead. Collect the effluent in a beaker and discard (unless advised to save the effluent by the Laboratory Operations Manager). Close the stopcock when the liquid level reaches the top surface of the resin. Enter the midpoint of the elution period in LIMS and/or the laboratory logbook.

9.4.6 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.

9.4.7 Measure 150 mL of 2 M H₂SO₄ and pass through the column to elute the bismuth. Collect the effluent in a clean, labeled 400 mL beaker.

9.4.8 Adjust the pH of the sample to 5 using a pH meter or pH paper. Add 35 mL concentrated NH₄OH to the sample beaker and add a magnetic stirring bar. Place the sample beaker on a magnetic stirring plate. Add NH₄OH using a disposable transfer pipette until a pH of 5 is obtained. If the pH exceeds 5, use 2 M H₂SO₄ to adjust the pH to 5.

9.4.9 Remove the sample beaker from the magnetic stirrer and add 2 mL of 6 M HCl using a disposable pipette. Bring to the 350 mL mark using deionized water.

9.4.10 Place the sample beaker on a moderate (approximately 200°F) hot plate and digest until the white BiOCl precipitate forms and falls to the bottom of the beaker (approximately 2 hours). Remove from the hot plate and allow to cool.

9.5 Mounting of Precipitate

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

- 9.5.1 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 ethanol.
- 9.5.2 Place the prepared discs in 4-way partitioned petri dishes that have been marked with sequence numbers (one number per partition, beginning with 1). Enter a corresponding sequence number beside each sample ID number entry in the laboratory logbook.
- 9.5.3 Place the petri dishes containing the prepared filters in a hot air oven (105-120°C) for 10 minutes or longer to dry. Remove petri dishes and allow to cool in a desiccator.
- 9.5.4 Weigh the filter discs on the analytical balance, using a clean spatula to handle them. Enter this tare weight beside the corresponding sequence number and sample ID number in LIMS and/or the laboratory logbook. Take care to replace each filter after weighing in the numbered petri dish partition from which it came.
- 9.5.5 Using a spatula, take the tared filters in sequence number order and transfer to the vacuum filtration apparatus. Wet with deionized water. Using LIMS and/or the laboratory logbook to establish the correspondence between sequence number and sample ID number, transfer each sample from its beaker onto the corresponding filter disc using the filtration apparatus.
- 9.5.6 Rinse the precipitate on the filter with deionized water and then with ethanol. Transfer each filter from the vacuum filtration apparatus back to the numbered petri dish partition from which it came. Place the petri dish in a hot air oven (105-120°C) for 10 minutes or longer until the filter is dry.
- 9.5.7 Remove the petri dish and allow to cool in a desiccator. Weigh the filters on the analytical balance and enter the weights beside the corresponding sequence numbers in LIMS and/or the laboratory logbook. Return each filter to its original partition in the petri dish. LIMS automatically calculates the mount weight and carrier yield.
- 9.5.8 Prepare a label for each sample, showing the analysis, the sample ID number and the customer. Attach each label to a nylon planchet.
- 9.5.9 Using LIMS and/or the laboratory logbook to establish the correspondence between sample ID number, and sequence number, transfer each filter to its planchet and fix in

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

place with a 2-inch piece of 3 mg/cm² aluminum absorber foil and a nylon ring. Trim excess aluminum with a razor blade or scissors.

9.5.10 Submit finished planchets and worksheets to the counting room for radioassay.

9.6 Sample Counting

Bismuth-210 mounts are typically counted 100 minutes for beta activity in a low level gas flow proportional counter.

- 9.6.1 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 Worksheet.
- 9.6.2 Arrange the worksheets in order according to counter number. Take the first sheet, locate the nylon planchet bearing the indicated sample ID number, and load the planchet into the detector tray indicated on the sheet.
- 9.6.3 Set the counter timer to 100 minutes and start the counters (different counting intervals may be used). Leave the worksheets on the tray in front of the counters
- 9.6.4 After all samples in the group have been counted, copy the raw data file and transfer to the appropriate calculation raw data folder. Remove the mounts from the counter trays and place them in the labeled container.

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

9.7 Calculation of the Sample Activity or of the MDA

9.7.1 Sample activity and the 2-sigma counting error are calculated as follows:

$$\frac{\text{Net pCi on Collection Date}}{\text{Unit Volume or Weight}} = \text{Net Activity} \pm \text{Counting Error} \quad \text{or,}$$

$$\frac{\text{Net pCi on Collection Date}}{\text{Unit Volume or Weight}} = \frac{\frac{N}{\Delta t} - \beta}{2.22(v)(y)(DF)(\epsilon)} \pm \frac{2 \times \sqrt{\frac{\frac{N}{\Delta t} + \beta}{\Delta t}}}{2.22(v)(y)(DF)(\epsilon)}$$

- Where:
- N = total counts from sample (counts)
 - Δt = counting time for sample (minutes)
 - β = background rate of instrument blank (cpm)
 - 2.22 = dpm/pCi or: 2.22×10^6 dpm/ μ Ci
 - v or w = volume or mass of sample analyzed
 - y = yield
 - DF = decay factor of Bi-210 from the mid-elution time to the mid-count time
 - ϵ = efficiency of the counter for Bi-210 beta counting, using a 3 mg/cm² aluminum absorber
 - 2 = sigma multiplier

9.7.2 If no activity is found, the MDA is reported and calculated as follows unless otherwise specified by the customer:

$$MDA = \frac{4.66 \sqrt{\frac{\beta}{\Delta t}}}{2.22(v)(y)(\epsilon)(DF)}$$

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

10.0 DATA AND RECORD MANAGEMENT

- 10.1 Data and records management is per procedure TBE-1003.
- 10.2 All laboratory data and ancillary information shall be documented in LIMS and/or laboratory logbooks or appropriate worksheets. Appropriate supervisory personnel shall review bound logbook entries as required by the TBE-ES Quality Assurance Manual and procedure TBE-4015.
- 10.3 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. Unless obvious, an explanation for the change is also required. Correction to recorded data in the electronic logbook requires a reason in order to save the change in LIMS.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

- 11.1 Sample collection, reagents and standards preparation, quality control and data acceptance, sample preparation and instrument procedures, calculations, precision and accuracy, reporting of results, attachments and references, as they apply to this procedure, are discussed in other procedures.
- 11.2 From a health and safety perspective, the TBE Knoxville Facility Safety Manual shall guide execution of this procedure.
- 11.3 Analysis blanks and spikes shall be run on each batch, as required by the TBE-ES laboratory QC program.
- 11.4 Sample duplicates shall be run to meet customer requirements or as required by the TBE-ES laboratory QC program.
- 11.5 A matrix spike consisting of a sample spiked with an appropriate standard (NIST traceable when possible) shall be run to meet customer requirements or as required by the TBE-ES laboratory QC program.

Procedure	Number: TBE-2015	Revision: 4
	Issue Date: 12/05/03 (reissue)	Revision Date: 10/05/2011
Responsible Individual:	Laboratory Production Manager	Review Date: 10/05/2014
Subject:	Lead-210 Activity in Various Matrices	

11.6 If any batch control sample fails laboratory established quality control criteria or fails to meet specific customer contract requirements, the samples comprising the controlled batch shall be reanalyzed.

11.7 Control samples may be statistically analyzed following the guidance of procedure TBE-4011.

12.0 REFERENCES

12.1 TBE Knoxville Facility Safety Manual, current version.

12.2 TBE-ES procedure TBE-3003, Calibration and Control of Alpha and Beta Counting Instruments.

12.3 TBE-ES Quality Assurance Manual, current version.

12.4 TBE-ES Radiation Protection Program Manual, current version.

12.5 U.S. Environmental Protection Agency, August 1980, *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA-600/4-80-032.

12.6 U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, revision 1, February 1979; *Quality Assurance for Radiological Monitoring Programs (Normal Operations) – Effluent Streams and the Environment*.