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Memorandum

To: Anne Summers and Marcel Hermans – Port of Portland

From: Mike Palermo – Palermo Consulting, Inc.; Ben Hung and Todd Thornburg – Anchor Environmental, L.L.C.

CC: Krista Koehl – Port of Portland; Tom Schadt, John Verduin, PE, and Elizabeth Appy – Anchor Environmental, L.L.C.

Date: June 15, 2007

Re: Proposed Sampling and Analysis Plan for a Sequential Batch Leachate Test
Port of Portland – Terminal 4 Early Action

This memorandum presents the proposed sampling and analysis plan (SAP) to conduct a sequential batch leaching test (SBLT) on sediments collected in Slip 3 and Berth 414. Based on discussions with EPA, Mike Palermo Consulting, Inc. and Anchor Environmental L.L.C. (Anchor) have reviewed the previously completed pancake column leaching test (PCLT) results and methodology as reported in the 2004 *Terminal 4 Early Action Characterization Report* (Blasland, Bouck & Lee, Inc.; BBL 2005; sample T4-CM2-Met). As discussed with EPA, the recommendation was that a SBLT be performed with material collected from representative areas within the Slip 3 and Berth 414 dredge prism. This recommendation was based in part on the fact that the source material used in the previously completed leachate analysis was a composite that included sediments outside of the proposed dredge prism, such as portions of Wheeler Bay and Berth 414. The composite was prepared in advance of the selection of the remedial alternative and before the areas specified for dredging had been finalized.

The SBLT will provide a measure for the concentrations of constituents in the groundwater that is in contact with the contaminated sediment layer of the CDF, for use in the long-term groundwater model. The SBLT and PCLT are both valid leaching tests for this purpose. The advantage of the SBLT, however, is that it can be conducted in significantly less time and with smaller sediment sample volumes compared to the PCLT. As a result, it will be possible to perform the SBLT using archived sediment samples from Terminal 4 in frozen storage at the analytical lab (in compliance with sample holding times).

The SBLT will be completed in accordance with the procedure developed by the U.S. Army Corps of Engineers (USACE) Waterways Experiment Station, as described in Appendix B of the Upland Testing Manual (USACE 2003). The general sample handling and analysis procedures will follow previously approved procedures specified in the 2004 *Terminal 4 Early Action Engineering Evaluation/Cost Analysis Work Plan* (BBL 2004), Appendix A, Field Sampling Plan and Quality Assurance Project Plan. Analytical Resource Inc. (ARI) will perform the leachate testing and laboratory analysis; analytical parameters will include target metals, semivolatile organics, pesticides, and PCBs.

Sampling and Processing Procedures

Bulk Sediment Composite. The bulk sediment composite sample was prepared according to the sampling and processing procedures described in the 2007 *Proposed Sampling and Analysis Plan for Additional Modified Elutriate Testing* (Anchor 2007). This bulk sediment composite sample was prepared from archived sediment core intervals representative of the dredge prism as discussed in the meeting with EPA on May 10, 2007. The archived intervals were taken during pre-construction sampling in July 2006 and sheetpile wall sampling during January 2007.

Challenge Water. The SBLT involves “challenging” the sediment sample with water to produce a leachate sample for testing. Per USACE 2003, the appropriate supply water is deoxygenated, distilled-deionized (DDI) water. Therefore, laboratory supplied DDI water will be the supply water in the SBLT.

Laboratory Analysis

ARI in Tukwila Washington will perform the SBLT, as well as the analysis of the elutriate water.

Sequential Batch Leaching Test. The SBLT will be completed in general accordance with the procedure developed by the USACE Waterways Experiment Station, as described in Appendix D of the Upland Testing Manual (USACE 2003). A water-to-sediment ratio of 4-to-1 will be used to prepare the SBLT slurry, consistent with both the anticipated characteristics of the hydraulic dredge inflow to the CDF, as well as the slurry concentration recommended in the SBLT test procedure. The sediment-water mixture will then be tumbled for a 24-hour period to

ensure intimate contact and encourage chemical equilibrium between sediment and water phases.

The leachate is drawn off and processed to recover dissolved and colloidal constituents. The sample will be centrifuged before filtering as described in ARI SOP 1114, which is a modification of the Standard Operating Procedure (SOP) for porewater extraction (see Appendix A). In accordance with the USACE protocol, metals are filtered through a 0.45-micron filter. To avoid adsorption onto the filter surface, organics are pre-filtered at 4 microns then filtered through a 1-micron glass fiber filter. After the leachate is extracted, the same sediment will be subjected to another cycle of leaching with new DDI supply water. A total of four consecutive leaching cycles will be performed.

SBLT Leachate Water. ARI will perform the following analysis on SBLT leachate water:

- Total suspended solids
- Metals (arsenic, cadmium, chromium, copper, lead, mercury, nickel, silver, and, zinc)
- Semivolatile organics (including SIM PAHs)
- Pesticides (low level)
- PCBs (low level)

The specific analytes, methods, reporting limits and detection limits for the leachate water are provided in Table 1.

Use of the Data

The SBLT is intended to evaluate the potential quality of groundwater that migrates through the dredged material in the CDF and subsequently through the CDF berm. The results will be used for input in the long-term groundwater modeling of the CDF as well as to evaluate potential impacts to surface water from any discharge through the berm from the CDF.

References

Anchor. 2007. *Proposed Sampling and Analysis Plan for Additional Modified Elutriate Testing*.
Prepared for Port of Portland, Portland, Oregon.

BBL. 2005. *Terminal 4 Early Action Engineering Evaluation/Cost Analysis, Public Review Draft*. Report prepared for Port of Portland, Portland, Oregon.

BBL. 2004. *Work Plan, Terminal 4 Early Action Engineering Evaluation/Cost Analysis*. Report prepared for Port of Portland, Portland, Oregon. February 23.

USACE. 2003. *Evaluation of Dredged Material Proposed for Disposal at Island, Nearshore, or Upland Confined Disposal Facilities – Testing Manual*. US Army Corps of Engineers. January 2003.

Tables

Table 1 – Parameters, Methods, Reporting Limits and Detection Limits for Leachate Samples

Appendix

Appendix A – Centrifugation SOP

Table 1
Leachate Analytical Parameters, Methods, Method Reporting Limits, and Method Detection Limits

Parameter	Analytical Method	Method Detection Limit	Method Reporting Limit
Conventionals (mg/L)			
Total Suspended Solids	EPA 160.2	-	1
Metals (µg/L)			
Arsenic	EPA 200.8	0.05	0.2
Cadmium	EPA 200.8	0.02	0.2
Chromium	EPA 200.8	0.03	0.5
Copper	EPA 200.8	0.12	0.5
Lead	EPA 200.8	0.15	1
Mercury	EPA 7470-LL	0.001	0.02
Nickel	EPA 200.8	0.15	0.5
Silver	EPA 200.8	0	0.2
Zinc	EPA 200.8	1.05	4.0
Semivolatile Organics (µg/L)			
Naphthalene	8270-SIM	0.029	0.1
2-Methylnaphthalene	8270-SIM	0.024	0.1
Acenaphthylene	8270-SIM	0.022	0.1
Acenaphthene	8270-SIM	0.024	0.1
Fluorene	8270-SIM	0.026	0.1
Phenanthrene	8270-SIM	0.025	0.1
Anthracene	8270-SIM	0.023	0.1
Fluoranthene	8270-SIM	0.02	0.1
Pyrene	8270-SIM	0.036	0.1
Benz(a)anthracene	8270-SIM	0.024	0.1
Chrysene	8270-SIM	0.039	0.1
Benzo(b)fluoranthene	8270-SIM	0.044	0.1
Benzo(k)fluoranthene	8270-SIM	0.056	0.1
Benzo(a)pyrene	8270-SIM	0.041	0.1
Indeno(1,2,3-cd)pyrene	8270-SIM	0.48	0.1
Dibenz(a,h)anthracene	8270-SIM	0.046	0.1
Benzo(g,h,i)perylene	8270-SIM	0.52	0.1
Dimethyl phthalate	8270-SIM	0.023	0.1
Diethyl phthalate	8270-SIM	0.083	0.1
Di-n-butyl phthalate	8270-SIM	0.112	0.2
Butylbenzyl phthalate	8270-SIM	0.022	0.1
Bis(2-ethylhexyl) phthalate	8270-SIM	0.194	0.2
Di-n-octyl phthalate	8270-SIM	0.046	0.1
Pesticides (µg/L)			
4,4'-DDE	8081A-LL	0.000337	0.0005
4,4'-DDD	8081A-LL	0.000305	0.0005
4,4'-DDT	8081A-LL	0.000357	0.0005
2,4'-DDE	8081A-LL	-	0.0005
2,4'-DDD	8081A-LL	-	0.0005
2,4'-DDT	8081A-LL	-	0.0005
PCBs (µg/L)			
Aroclor 1016	8082-LL	0.002	0.01
Aroclor 1221	8082-LL	-	0.01
Aroclor 1232	8082-LL	-	0.01
Aroclor 1242	8082-LL	-	0.01
Aroclor 1248	8082-LL	-	0.01
Aroclor 1254	8082-LL	-	0.01
Aroclor 1260	8082-LL	0.0014	0.01
Aroclor 1262	8082-LL	-	0.01
Aroclor 1268	8082-LL	-	0.01

Pore water extraction for TBT

1.0 PURPOSE AND SCOPE

This procedure describes the method, materials, equipment, and special conditions required to extract interstitial water from sediments, for the analysis of TriButyl Tin species. This procedure is specifically for extraction for the analysis of Butyl Tin species, but may be modified for other analytes, provided such modifications are noted on the extraction data sheet.

2.0 EQUIPMENT

- 2.1 Refrigerated Centrifuge, and Polycarbonate bottles
- 2.2 Balance for balancing the centrifuge bottles
- 2.3 Nitrogen chamber, with Oxygen meter
- 2.4 Miscellaneous spoons, pipettes, etc.

3.0 DEFINITIONS

- 3.1 Interstitial Water
 - 3.1.1 Interstitial water is the water that fills the pore spaces of sediment. The water is assumed to be in chemical equilibrium with the sediment. Only a portion of the water will be extracted using centrifugation. Other methods of extraction produce larger yields, (i.e., pressing) but are prone to shift the chemical equilibrium and are not recommended.

4.0 DOCUMENTATION

- 4.1 Pore Water Extraction Data Form, 1114F

5.0 PROCEDURE

- 5.1 Decontamination

At a minimum, decontamination of equipment shall include the following steps:

- wash the equipment with a detergent and rinse with tap water to remove any residue;
- rinse with a 5% nitric acid solution
- rinse with DI water
- air dry
- rinse with solvent, hexane for polycarbonate bottles and methylene chloride for all other equipment.

- 5.2 Centrifuge Settings

The process of centrifugation separates the solids and liquid phases based on their specific gravity. During the spinning process, the denser materials move to the bottom of the tube while the lighter materials float on top. In order to be extracted, there must be a significant difference between the specific gravity of the matrix and the material to be separated. Some organic materials may not be significantly denser than the pore water and may not separate. An example of this is seed pods which generally will float on top of the water layer.

The rate of separation in a centrifuge is based on the difference in specific gravity of the materials, particle size, temperature, and speed and radius of the rotor. Also, the design of the centrifuge rotor will effect the calculation, as the angle of the rotor, and thus the effective radius, will change the centrifugal force.

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Based on project specific information, the time of spin may be calculated from the temperature, the size of the particles to be separated, and the constants associated with each centrifuge and rotor, the time of each spin cycle may be calculated from the following equation:

$$t = \frac{(6.3E09) \eta \log_{10} R/S}{N^2 D^2 \Delta s}$$

Where:

- t is the time of spin in minutes
- η is the viscosity of the fluid phase, in poises
- R is the radius of rotation of the top of the sediment in the bottle, cm
- S is the radius of rotation of the surface of the suspension in the bottle, cm
- N is the speed of the rotor, rpm
- D is the particle diameter, microns
- Δs is the difference in specific gravity between the solids and the suspending liquid

In general for pore water extraction of sediments, a time of 2-3 minutes is all that is required. However, to account for highly organic sediments, a time of 30 minutes has been assumed by the U.S. Army Corps of Engineers. The speed necessary for the first extraction step has been calculated at 3,000 rpm for the IEC centrifuge and the speed for the second clean up spin in the Beckman centrifuge is 7,000 rpm.

3.3 Extraction

The amount of pore water required will be determined based on project specific detection limits. Appropriate centrifuge bottles shall be selected based on the analytes of interest, the amount of pore water required and the weight and speed limits of the rotor / centrifuge.

Sediment samples will be maintained at 4°C prior to centrifuging. Care must be taken at each step to ensure that the temperature of the sample is maintained at or below 10°C during processing. Proper set up and planning are required to keep each step as short as possible and keep the samples and pore waters in the proper temperature environment.

The centrifuges should be turned on at the same time as the nitrogen in the anaerobic chamber is turned on. The temperature of each centrifuge should be set to 4°C, and they should be allowed come to this temperature prior to use.

The sample to be extracted shall be examined. The sample identification and any other pertinent information shall be recorded on the data sheet. The sediment for pore water extraction shall be placed in the nitrogen chamber along with centrifuge jars, spoons, a balance, etc. and the chamber sealed and filled with nitrogen. After about 30 minutes the oxygen level shall be checked using the hand held oxygen meter. Allow about 5 minutes for the instrument reading to stabilize. Because sulfides which are often present in marine sediments interfere with the instrument, subsequent oxygen readings may be erroneous. The centrifuge jars should be opened to allow them to come to equilibrium with the chamber. The oxygen level in the chamber shall be less than 1% before any work in the chamber is started.

Once the chamber is ready, the sediment jars may be opened and the sediment spooned into labeled centrifuge bottles. Any free liquid on the top of the sediment may be poured into the centrifuge bottle. The bottles will be balanced to within 0.1 g. Once a set of four

Pore water extraction for TBT

bottles is ready, they are removed from the chamber via the air lock and placed in the pre-cooled centrifuge. All samples for TBT analysis are centrifuged at 3,000 rpm for 30 minutes. They are then placed back in the nitrogen chamber, decanted into another PC bottle, balanced with another bottle containing DI water, and were placed in another pre-cooled centrifuge (4°C) and spun at 7,000 rpm for 30 minutes.

Following the second “clean up” spin, the bottles are replaced in the nitrogen chamber and the water is poured into pre-preserved PC bottles. For TBT analysis, the preservative will be a few drops of HCl. The pore water samples are then placed in the refrigerator until they are transferred to the Organic Extractions lab or Sample receiving.

4.0 SAFETY

4.1 Keep all laboratory areas clean and follow all safety requirements.

5.0 REFERENCE

5.1 Clarification paper for the TBT-interstitial water extraction protocol, USA Corps of Engineers web site, <http://www.nps.usace.army.mil/dmno/smarm98/papers.htm>.