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APPENDIX 7-A

SAMPLE HANDLING PROCEDURES

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Listed below are details concerning the sample handling procedures outlined in Chapter 7. All sample handling procedures should be specified in the sampling and analysis plan.

Decontamination Procedures

It is also recommended that all sampling equipment and utensils, such as spoons, mixing bowls, extrusion devices, sampling tubes and cutter heads, etc., be made of non-contaminating materials and be thoroughly cleaned prior to use. The intention is to avoid contaminating the sediments to be tested, since this could possibly result in dredged material, which would otherwise be found acceptable for open-water disposal, being found unacceptable. While not strictly required, an adequate decontamination procedure is highly recommended. The dredging proponent assumes a higher risk of sample contamination by not following an established protocol. The following procedure has been used successfully for other dredging projects:

- √ Wash with brush and Alconox soap.
- √ Double rinse with distilled water.
- √ Rinse with nitric acid.
- √ Rinse with metal-free water.
- √ Rinse with methanol.

While methylene chloride has been used extensively in the past as an organic solvent, and is recommended by PSEP, its use is discouraged by the dredging regulatory agencies because of its status as a potential carcinogen and its impact on the ozone layer.

After decontamination, sampling equipment should be protected from recontamination. Any sampling equipment suspected of contamination should be decontaminated again or rejected. If core sampling is being conducted, extra sampling tubes should be available on-site to prevent interruption of operations should a sampling tube become contaminated. Sampling utensils should be decontaminated again after all sampling has been conducted for a DMMU to prevent cross-contamination. Disposable gloves are typically used and decontaminated or disposed of between DMMUs.

Volatiles and Sulfides Sub-sampling

The volatiles and sulfides sub-samples should be taken immediately upon extrusion of cores or immediately after accepting a grab sample for use. For composited samples, one core section or grab sample should be selected for the volatiles and sulfides sampling. Sediments which are directly in contact with core liners or the sides of the grab sampler should not be used.

Two separate 4-ounce containers should be completely filled with sample sediment for volatiles. No headspace should be allowed to remain in either container. Two samples are collected to ensure that an acceptable sample with no headspace is submitted to the laboratory for analysis. The containers, screw caps, and cap septa (silicone vapor barriers) should be washed with detergent, rinsed once with tap water, rinsed at least twice with distilled water, and dried at >105/ C. A solvent rinse should not be used because it may interfere with the analysis.

To avoid leaving headspace in the containers, sample containers can be filled in one of two ways. If there is adequate water in the sediment, the vial should be filled to overflowing so that a convex meniscus forms at the top. Once sealed, the bottle should be inverted to verify the seal by demonstrating the absence of air bubbles. If there is little or no water in the sediment, jars should be filled as tightly as possible, eliminating obvious air pockets. With the cap liner's PTFE side down, the cap should be carefully placed on the opening of the vial, displacing any excess material.

For sulfides sampling, 5 mls of 2 Normal zinc acetate per 30-g of sediment should be placed in a 4-ounce sampling jar. The sulfides sample should be placed in the jar, covered, and shaken vigorously to completely expose the sediment to the zinc acetate.

The volatiles and sulfides sampling jars should be clearly labeled with the project name, sample/composite identification, type of analysis to be performed, date and time, and initials of person(s) preparing the sample, and referenced by entry into the log book. The sulfides sampling jars should indicate that zinc acetate has been added as a preservative.

Sampling Logs

As samples are collected, and after the volatiles and sulfides sub-samples have been taken, logs and field notes of all samples should be taken and correlated to the sampling location map. The following should be included in this log:

Date and time of collection of each sediment sample.

Names of field supervisors and person(s) collecting and logging in the sample.

The sample station number and individual designation numbers assigned for individual core sections.

Quantitative notation of apparent resistance of sediment column to coring.

The water depth at each sampling station. This depth should then be referenced to mean lower low water (MLLW NAD 83) through the use of an on-site tide gage.

Length, depth interval (referenced to the sediment/water interface) and percent recovery of core sections.

Weather conditions

Physical sediment description, including type, density, color, consistency, odor, stratification, vegetation, debris, biological activity, presence of an oil sheen or any other distinguishing characteristics or features.

Any deviation from the approved sampling plan.

Extrusion, Compositing and Sub-sampling

Depending on the sampling methodology and procedure proposed, sample extrusion, compositing and subsampling may take place at different times and locations. If core sampling is conducted, these activities can either occur at the sampling site (e.g., on board the sampling vessel) or at a remote facility. Grab samples will be processed immediately upon sampling. If cores are to be transported to a remote facility for processing, they should be stored at 4/C onboard the sampling vessel and during transport. The cores should be sealed in such a way as to prevent leakage and contamination. If the cores will be sectioned at a later time, thought needs to be given to core integrity during transport and storage to prevent loss of stratification. For cores or split-spoon sampling, the extrusion method should include procedures to prevent contamination.

For composited samples, representative volumes of sediment should be removed from each core section or grab sample comprising a composite. The composited sediment should be mixed until homogenized to a uniform color and consistency, and should continue to be stirred while individual samples are taken of the homogenate. This will ensure that the mixture remains homogenous and that settling of coarse-grained sediments does not occur.

At least 6 liters of homogenized sample needs to be prepared to provide adequate volume for physical, chemical and biological laboratory analyses. Bioassays require approximately 4 liters while chemical testing requires approximately 1 liter of sediment. Both chemistry and bioassay samples should be taken from the same homogenate. Portions of each composite sample will be placed in appropriate containers obtained from the chemical and biological laboratories. See Table 7-1 for container and sample size information. In high-ranked areas, the sample taken

from the foot beyond the dredging overdepth should be placed in a 250 ml glass jar and frozen for possible future analysis.

After compositing and subsampling are performed, the sample containers should be refrigerated or stored on ice until delivered to the analytical laboratory. The samples reserved for bioassays should be stored at 4/C in a nitrogen atmosphere, i.e., nitrogen gas in the container headspace, for up to 56 days pending initiation of any required biological testing. Each sample container should be clearly labeled with the project name, sample/ composite identification, type of analysis to be performed, date and time, and initials of person(s) preparing the sample, and referenced by entry into the log book..

Sample Transport and Chain-of-Custody Procedures

Sample transport and chain of custody procedures should follow the PSEP protocols, which include the following guidelines:

If sediment cores are taken in the field and transported to a remote site for extrusion and compositing, chain of custody procedures should commence in the field for the core sections and should track the compositing and subsequent transfer of composited samples to the analytical laboratory. If compositing occurs in the field, chain-of-custody procedures should commence in the field for the composites and should track transfer of the composited samples to the analytical laboratory.

- √ Samples should be packaged and shipped in accordance with U.S. Department of Transportation regulations as specified in 49 CFR 173.6 and 49 CFR 173.24.
- √ Individual sample containers should be packed to prevent breakage and transported in a sealed ice chest or other suitable container.
- √ Ice should be placed in separate plastic bags and sealed, or blue ice used.
- √ Each cooler or container containing sediment samples for analysis should be delivered to the laboratory within 24 hours of being sealed.
- √ A sealed envelope containing chain-of-custody forms should be enclosed in a plastic bag and taped to the inside lid of the cooler.
- √ Signed and dated chain-of-custody seals should be placed on all coolers prior to shipping.

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- √ The shipping containers should be clearly labeled with sufficient information (name of project, time and date container was sealed, person sealing the container and consultant's office name and address) to enable positive identification.
- √ Upon transfer of sample possession to the analytical laboratory, the chain-of-custody form should be signed by the persons transferring custody of the sample containers. The shipping container seal should be broken and the condition of the samples should be recorded by the receiver.
- √ Chain-of-custody forms should be used internally in the lab to track sample handling and final disposition.