Protocols For Collecting NRDA Samples — INTERTIDAL SEDIMENTS

Sampling Objectives

- To determine the concentration and source of oil compounds in the sediments collected.
- To measure sediment characteristics for interpreting chemical and biological results.
- To maintain the integrity the sample(s) during sampling, transport, and storage.

Sample Volume by Analytical Method *(see back page for description and suggested detection limits)*

THC by GC/FID	500 mL; or 1 pint; or 16 oz
PAH by GC/MS-SIM	500 mL; or 1 pint; or 16 oz
TOC	10 g; or less than 10 mL
Grain size	100 g; or 4 oz

Sampling Equipment/Containers

- To collect subsurface samples in coarse sediments (sand and gravel), it is easiest to use a shovel to dig a small trench and collect the desired sediment intervals from the exposed wall in the trench.
- To collect subsurface samples in fine sediments (mud), use a shovel to expose the sediments at the desired depth. Collect the sample from the natural break side, rather than the shovel side.
- Coring tubes can be used in muddy sediments when the sampling intervals have not been determined. Plastic tubes (polycarbonate or polyethylene is okay) should be 5 cm (2 in) in diameter, with a wall thickness of about 3 mm.
- Sediment samples for THC and PAH should be placed in glass containers, certified-clean to be organic-free (solvent rinsed), with teflon- or aluminum-lined lids. For TOC, they can be placed in soap-cleaned glass or plastic containers. For grain size, Ziploc or Whirl-Pak bags can be used.

Sample Collection Methods

- Decon sampling equipment and supplies initially and between samples, if re-using them. First wash with laboratory-grade detergent and clean water, with a triple clean water rinse. Use a clean water source for rinsing (distilled water from a local store is OK). Then rinse with methanol or acetone, followed by methylene chloride or hexane (Capillary GC Pesticide Residue Grade or equivalent). Do not work with solvents downwind of exhaust or other airborne hydrocarbon source. Collect waste/rinsate solvents for proper disposal.
- To reduce the need for field decon, use pre-cleaned, disposable utensils (e.g., stainless steel blade or wooden spatula), single-use core tubes, etc. The only equipment to be used between sites is a shovel.
- Discrete samples from a single sample point may be collected to represent a specific condition, such as a tarball for fingerprinting and source identification.
- Composite samples (of at least three subsamples) are recommended for characterization of a sampling site, such as contaminant content in marsh sediments.
- Photograph the sampling site prior to sample collection to document the site conditions.
- For surface sediments, use a wooden spatula or stainless steel blade to accurately collect the top 2 cm, avoiding contact with disturbed sediments.
- For subsurface sediments, expose a fresh surface at the desired interval, then remove the sediments which were in contact with the shovel prior to sampling.
- On each trip, try to sample control and least oiled areas first, then the most contaminated areas.
- Record the sample no. on both the label and lid. Record the following on the field log sheet:
  -sample no.; date/time; station location; tidal elevation
  -description of oiling conditions, using standard shoreline assessment terminology
  -sediment characteristics: texture, color, biota, vegetation, debris, odor, etc.
- Make a quick sketch in a field log book or sketch form showing the sampling locations in enough detail that the location could be re-occupied by someone else.

Preservation/Holding Times

- Immediately place all sediment samples in a cooler and keep at 4°C . Freeze samples for chemical analysis by the end of each day. Refrigerate samples for TOC and grain size (do not freeze).
- Use packing material, such as bubble wrap, around containers to prevent breakage.
- Sediment samples can be held frozen in the dark for several years without loss of sample integrity. Sediment extracts can be held at 4°C in the dark for 40 days without loss of sample integrity.

Analytical Methods

Total hydrocarbons (THC). Often referred to as total petroleum hydrocarbons, but most methods do not differentiate among petroleum, pyrogenic, and biogenic hydrocarbons. THC by GC-FID (total area of FID gas chromatogram of combined f_1 and f_2 fractions after column chromatography) is often the preferred method because of the low detection limit (2 ppm versus 100-1000 ppm for other THC methods) and the direct measurement of hydrocarbons. This method does not detect low boiling compounds (below n-C_8). For NRDA, THC analyses generally will not provide the data needed to support calculation of toxic effects from PAH exposure, and will have to be corrected to equivalent PAHs. The THC results, however, can be used to track oil weathering and map extent of exposure of intertidal resources. Detection limits are usually higher than those needed for intertidal injury assessment.

- **Polynuclear aromatic hydrocarbons** (PAH). Since most of the toxicity in oil is due to the PAHs, it is often the preferred analysis for NRDA. The analytes must include the alkyl-substituted PAH homologs, in addition to the standard PAH "priority pollutants". This method is referred to as Modified EPA Method 8270, because the list of PAHs is expanded to include the alkylated homologs, using GC/MS in the selected ion monitoring mode. Detection levels should be 1 ppb for individual PAHs to support injury assessment using toxicity thresholds.

Other Considerations

- Be aware of the potential for contamination of the site from oil on boots and shovels.
- Keep a detailed photo log so that each photograph can be labeled.
- Collect background samples from clean sites representative of pre-oiling conditions, as well as areas not yet oiled but in the potential path of the oil.
- It is very important to have a defined sampling strategy prior to conducting field work. Intertidal sediments are difficult to sample because of their inherent heterogeneity over space, depth, and time. Truly quantitative samples have to be collected in a very precise manner and be related to a surface area or meaningful volume metric. Grab samples are not very useful to injury quantification. Documenting exposure of intertidal areas is best accomplished with photography, video, and good field notes and sketches using standard shoreline assessment methods. Samples may be needed for fingerprinting or monitoring weathering, to correlate a degree of oiling term with oil loading, to confirm the presence of oil, or for bioassay purposes.

Key References

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