Guidelines for Collecting Ephemeral Data in the Arctic: INTERTIDAL SEDIMENTS

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Note: These guidelines are limited data collection aides that do not necessarily consider all possible scenarios under which samples may be collected. Use best professional judgment to modify these guidelines according to area-specific field conditions.

Guideline Objectives

The primary objective of this document is to provide guidelines on collection of intertidal sediment samples for chemical analysis during the early stages of an oil spill in the Arctic to support Natural Resource Damage Assessment (NRDA) exposure and injury evaluations. See other guidelines for collection of biological samples from different types of intertidal habitats.

Sampling Objectives

Characterize oil

- Determine the concentration and composition of oil compounds in intertidal sediments compared to background concentrations
- Determine the source of contamination via chemical fingerprinting analysis and characterize oil weathering and fate
- Characterize other sources of oil or hydrocarbons in the environment

Describe habitat

- Estimate the areal extent and degree of intertidal sediment oiling
- Measure sediment characteristics for interpreting chemical and biological results
- Support oil environmental transport modeling by documenting where oil stranded onshore

Study exposure

- Document exposure of sediment dwelling organisms to oil compounds
- Support exposure modeling

Quality assurance/quality control

- Ensure the integrity of the sample(s) throughout sampling, transport, and storage
- Ensure the reliability of chemical characterizations

Collaboration

- Support other assessment efforts (see Subtidal Sediment, Stranded Oil, Water, Snow, Ice, Shellfish Tissue, and/or Sand Beach Infauna guidelines)

Before Field Sampling

- Assure that all personnel have required safety training and protective equipment for Arctic field work (not described in this guideline).
- Arctic weather conditions (e.g., wind direction and speed) are variable within a short timeframe. Be prepared for changing weather conditions, be aware of your surroundings, and take precautions to ensure the safety of the sampling team.

Study design

- It is important to have a defined sampling strategy prior to conducting fieldwork. Intertidal sediments are difficult to sample because of the inherent heterogeneity of oil distribution over space, depth, and time.
- The following terminology is used to define general to specific sampling geographies:
  - Area = general area of uniform characteristics, such as degree of oil exposure, physical setting, habitat types present, etc.
  - Location = a specific location that is representative of the area and contains the type of habitat to be sampled, such as intertidal habitats or lagoons
  - Transect = a line through a site along which samples are collected or observations are made
  - Site = a specific point at which samples are collected or observations are made
- Plan ahead the number of locations and number of sites per location, taking into account level of effort, potential logistical limitations, weather conditions, and other issues that may compromise sample integrity.
- Review the guideline and resolve any area-specific issues. Area-specific modification of the guideline may be needed based on environmental conditions, geography, access to remote areas, and shipping capabilities.
- Use a computer or conceptual model of the extent of intertidal sediment contamination or an appropriate power analysis to estimate the number of sampling locations and number of samples per site needed to respond to the sampling objectives.
- A stratified random sampling approach, which divides the sampling location into non-overlapping zones (strata) from which random samples are collected, is recommended if no other sampling strategy has been developed. This type of sampling improves the representative quality of samples by reducing sampling error (variability).
- Unreplicated grab samples are not very useful to injury quantification. Samples should be quantitative relative to a surface area metric.
- Contact the laboratories that will be receiving field samples for analysis and assure that they have the capacity to receive and analyze samples from the study. Follow relevant guidelines from the laboratory and consult with them about necessary modifications.
- Shoreline visualization tools (e.g., ESI maps, satellite images, ShoreZone) should be used to develop a sampling strategy and estimate distances, number of sampling transects, transect spacing, intertidal zone width, etc. before going into the field.
- The sampling strategy should have flexibility to be adjusted based on conditions in the field.
- Consult appropriate guidelines for the collection of other environmental media and biota concurrent with intertidal sediment sampling. Tarballs, sheens or other oil residues can be collected opportunistically for chemical analysis and fingerprinting.

Equipment

- Review the list of sampling equipment/containers, make adjustments as needed, and assure that all essential field materials are ready to be taken to the field.
- If not all sampling equipment is available, consult the alternative equipment guidelines or determine if other appropriate options are available.
- Consider area-specific conditions for remote Arctic regions and make adjustments in methodology and equipment as necessary.
- It may be necessary to coordinate with the laboratory that will receive the samples to assure that acceptable materials and conditions are used for sampling and sample storage and shipping.
- Do as much material preparation prior to field deployment, including: labeling sample jars using permanent markers or laboratory labels (e.g., peel and stick waterproof labels); solvent rinsing of jars

and aluminum foil for total hydrocarbons (THC) and polycyclic aromatic hydrocarbons (PAH) analyses, etc.
- Make sure that all essential equipment is in working order and operational under Arctic field conditions, and that spare equipment and/materials are available.
- Store solvents carefully to prevent spillage. Follow regulations regarding the shipment and storage of chemicals.

Sampling Areas and Timing

- Follow a sampling plan/work plan if one is available.
- If a sampling plan is not available for ephemeral data collection immediately after a spill, data collection should focus on collecting samples from a range of unoiled, likely to be oiled, and already oiled areas.
- It is important to obtain reliable information and analytical chemistry data that account for spatial and temporal variations of oil impacts.
- When sampling in remote areas with limited shipping capabilities, plan ahead to make sure that the integrity of samples is not compromised by ensuring that the processing laboratory receives the samples within their recommended holding time. Remember that it may take multiple days for shipments from remote areas to reach a laboratory facility. This last stage is the most important and requires due diligence until the samples are safely delivered.
- The number of locations and number of sites per location need to be considered accordingly, making sure that there is enough space in the coolers to accommodate all samples without sacrificing their integrity.
- Plan all sampling strategies within daylight hours; sampling in the dark, even with headlamps, is not recommended. This guideline may not apply during winter or much of the fall.
- The challenges of collecting samples in remote areas, particularly during winter, are great and require adequate planning and careful field implementation to attain the data quality required to meet the objectives of the sampling plan.

Area selection

- Areas for intertidal sediment sampling are coastal areas with relatively fine sediments (e.g., clay, silt, sand, granules, or small pebbles). Sediments from inundated tundra and peat shorelines can also be sampled though some modifications to the guidelines may be necessary. Quantification metrics may differ and the receiving analytical laboratory will need to be informed of the nature of the sample. See the Gravel Beach and Rocky Beach guidelines for information about sampling intertidal habitats with larger sediment sizes.
- Sampling locations should be representative of areas that have been or may be oiled by the spill and unoiled reference areas.
- Use trajectory models, conceptual models, overflight information, SCAT data or other tools to determine what location have been oiled and which ones are likely to be oiled.
- Samples should also be collected from locations known or suspected to be impacted by other natural or anthropogenic sources of contamination (e.g., oil seeps, coal, peat, mining, combustion engines), as these will be important to differentiate background sources and levels of contamination.
- It may be necessary to prioritize sampling locations. In this case, highest priority samples are to be collected from oiled intertidal areas that are sensitive habitats, biologically productive, or highly relevant for human use. Collecting pre-oiled intertidal sediments from sensitive/productive areas that are likely to be oiled by the spill in the near future is also a priority. Sampling at unoiled “control” areas and sampling other sources of contamination should be prioritized based on the ephemerality of the data and relative importance to developing a NRDA case.

- Sediment samples should be collected pre-oiling, if possible, as soon as practical after oiling, and periodically thereafter. Sampling frequency is a function of oil persistence, biological community, habitat importance, and resource availability and should be defined in the study design.
- The number of locations and number of sites per location should be defined in the study design. A minimum guideline for collecting sediment samples is at least three samples per intertidal zone per location of relatively uniform oiling exposure. If relevant data are available, a power analysis or other modeling approaches should be used to determine the number of samples needed before going into the field.
- Sample along exposure gradients, starting in the cleanest zone, at regular intervals proportional to the exposure area.

Collaboration

- Sediment samples can be collected in conjunction with nearshore water sampling, stranded oil sampling, and sand beach infauna surveys.
- Close collaboration and coordination with other ongoing ephemeral sampling efforts is important.

Field Sampling Methods

Sampling Equipment/Containers

Note: The amount of equipment required depends on the sampling plan, desired sample volumes, and logistics. Analytical laboratories may provide required sampling and sample storage and transport materials – contact the receiving lab before preparing to collect samples in the field.

- Coolers – for sample storage and transport
- Ice packs/collapsible jugs – for storage temperature regulation (if ambient temperature exceeds 4°C)
- Thermometer or temperature logger (1 per cooler)
- Disposable nitrile gloves (preferred), insulated nitrile-coated gloves (less ideal)
- Insulated shoulder-length rubberized gloves preferred for sediment sampling under extreme cold conditions
- Sampling jars – 4 or 8 oz certified organic-clean jars with Teflon-lined lids and labels – for TPH/PAH/biomarkers; for fine sediments
- Aluminum foil: the dull side should be pre-cleaned with acetone, methanol, or hexane (Capillary GC Pesticide Residue Grade or equivalent) to wrap samples of larger gravel, with the dull side in contact with the sample
- 10 mL soap-cleaned glass or plastic containers – for total organic carbon, TOC
- Ziploc or Whirl-Pak bags – for grain size
- Ziploc bags and additional sampling jars – for non-viscous tarballs or oil residues
- Shovel
- Stainless steel spatulas and spoons – organic clean, wrapped in foil and sealed for transport
- Disposable aluminum pans – for composite samples
- Field Sample Forms (template in Appendix A)
- Chain of Custody forms (see Chain of Custody guideline)
- Field notebook, evidence tape (see Chain of Custody guidelines)
- GPS, camera (with spare batteries), and photo scales
- Tape measure and ruler
- Packaging materials for glass jars (e.g., bubble wrap, sorbent pads, tape) – may be provided by the analytic laboratory
- Suitable disposal bags for oiled PPE and disposable sampling materials

Optional (if single-use sampling equipment is not available):

- Sufficient quantities of pre-cleaned or disposable equipment, single-use equipment are preferable. If equipment will be reused in the field, decontamination is necessary and will require the following materials:
  - Reusable sampling equipment
  - Laboratory-grade detergent (Liquinox or similar)
  - Solvents for cleaning sampling equipment – acetone, methanol, or hexane (Capillary GC Pesticide Residue Grade or equivalent) – consider shipping/airline regulations for solvents
  - Teflon solvent squirt bottles
  - Laboratory-grade, certified-clean distilled water (preferred), store-bought distilled water (less ideal); laboratory-grade detergent
  - Approved, sealed container for collecting solvent rinsate for disposal

Quality Assurance/Control

- Obtaining an adequate number of quality control samples is essential. At a minimum, a trip blank (accounts for contamination introduced during shipping and handling) and field blank (accounts for contamination introduced during sampling) should be maintained for each sampling effort and generally be collected at a rate of 5% and 10%, respectively, of all samples.
- A trip blank is an unopened sampling jar and should be transported with the samples and remain sealed in the cooler during sampling activities.
- A field blank should be collected at approximately every third sampling site, or at least at an "unoiled" and "oiled" site, by leaving the field blank sample jar open for the duration of the sampling period at that site. Record the site where field blanks were taken on the field sample form.
- Ideally, kiln-fired sand supplied by the laboratory can be transferred (poured or scooped) from one jar to another and returned to the lab as a field blank, but if this is not possible, use the open-jar technique (empty jar).
- Duplicate samples should be collected at every third sampling site or following the specifications of the work plan. A duplicate sample is collected from the same location and following the same steps as the preceding sample. This is not the same as collecting replicates from each site/depth. Duplicates should account for 10% of all samples, but consideration should be given to sample storage capacity. Do not split samples unless specified in the work plan.
- Rinsate blanks should be collected if there is a risk of cross contamination from reuse of sampling equipment. After cleaning the equipment in accordance with the procedures described in this method, rinse the clean equipment with solvent or cleaning solution and collect the rinsate in a sample jar. Note on the field sample form where and how rinsate blanks were collected.

Good Sampling Practices and Decontamination

- Good field practices and the development of a consistent sampling routine will help provide for the integrity of the samples and their validity in environmental assessments.
- Disposable nitrile gloves should be worn when sampling and changed between each sample collected or as necessary to prevent cross contamination.
- Disposable nitrile gloves can be worn over low-profile insulated gloves (e.g., neoprene gloves) in cold conditions and should be changed between samples to prevent cross contamination if they become contaminated or damaged. If nitrile gloves are not available or will not fit over insulated gloves in cold conditions, insulated nitrile-coated gloves may be an alternative, but extra precautions will have to be taken to prevent sample contamination; gloves will need to be cleaned with soap and clean water between samples and should not come in contact with the sample or with the surfaces of glassware or tools that will be in direct contact with the sample. Similar precautions should be taken when using insulated shoulder-length rubberized gloves.
- To reduce the need for field decontamination, use pre-cleaned and/or disposable equipment and tools (e.g., pre-cleaned stainless steel spoons).

- The only equipment to be used between sites is a shovel, which should be cleaned with soap and clean water. Repeated digging in clean sediments can be a last resort for cleaning the shovel if soap or clean water are not available. Alternatively, use a clean dry towel or other dry material to clean the shovel before its next use. Additional cleaning may be required when working at oiled sites (see below).
- Sediment samples for THC and PAH analysis should be placed in certified organic-clean (solvent rinsed) glass containers with Teflon- or aluminum foil-lined lids. Samples for TOC can be placed in soap-cleaned glass or plastic containers. For grain size, Ziploc or Whirl-Pak bags can be used.
- If disposable sampling equipment are not available, reusable sampling equipment MUST be decontaminated between samples:
  - Wash sampling utensil with laboratory-grade detergent and clean with a triple clean-water rinse. Cleaning with laboratory-grade water is preferred, though store-bought distilled water is a less ideal alternative and, as a last resort, "background" water from an up-current clean area can be used
  - Rinse with methanol or acetone, followed by hexane (Capillary GC Pesticide Residue Grade or equivalent). Collect solvent rinsate for proper disposal or shipment to the lab as a rinsate blank. Allow solvents to evaporate from equipment before use. Do not work with solvents downwind of exhaust or other airborne hydrocarbon source. If solvents are not available, use a diluted detergent solution and fresh water, followed by a distilled water rinse. If transporting solvents is not feasible, use single-use sampling material
- Take precautions to avoid cross-contamination of the site from oil on personal equipment. Sampling unoiled areas first, then lightly oiled areas and finally heavily oiled areas can minimize cross-contamination. Personal equipment should be exchanged or cleaned between sites if it becomes contaminated.
- Potential sources of contamination while sampling from vessels (exhaust fumes, oily surfaces) are a concern. Work up-wind of any exhausts and designate clean areas for handling samples. Segregate dirty/clean areas. Layout clean surfaces to work on and replace frequently.

Study Design Implementation

- If no other sampling strategy has been developed, use a stratified random sampling approach by randomly selecting the first transect starting point and expanding the sampling site (other transects) systematically into a grid from that initial point. Randomly select sampling sites within each intertidal zone intervals.
- Based on the study design and/or sampling strategy outlined before going into the field, establish a minimum of three transects spaced at least 30 m apart (recommended) (Figure 1). Transects should be perpendicular to the shoreline and encompass the entire intertidal zone. Run transects within two hours of low tide (before or after). In the Chukchi and Beaufort seas the tide range is small (<30 cm); wind-driven storm surge is the most determinant factor in water height and should be considered for sampling. Tides are only a consideration south of the Bering Strait where the tidal range is greater than 1 m.
  - Record the transect location using a GPS and accurately plot the transect location on a map or aerial photograph
  - If possible, permanently mark the transect location using "front" and "back" stakes that line up with the transect. Consider placement carefully to minimize loss due to vandalism, erosion, ice scouring, etc.
  - Record the transect angle with a compass so it can be re-surveyed at a later date even if one of the stakes is lost; note whether the angle is magnetic or true north

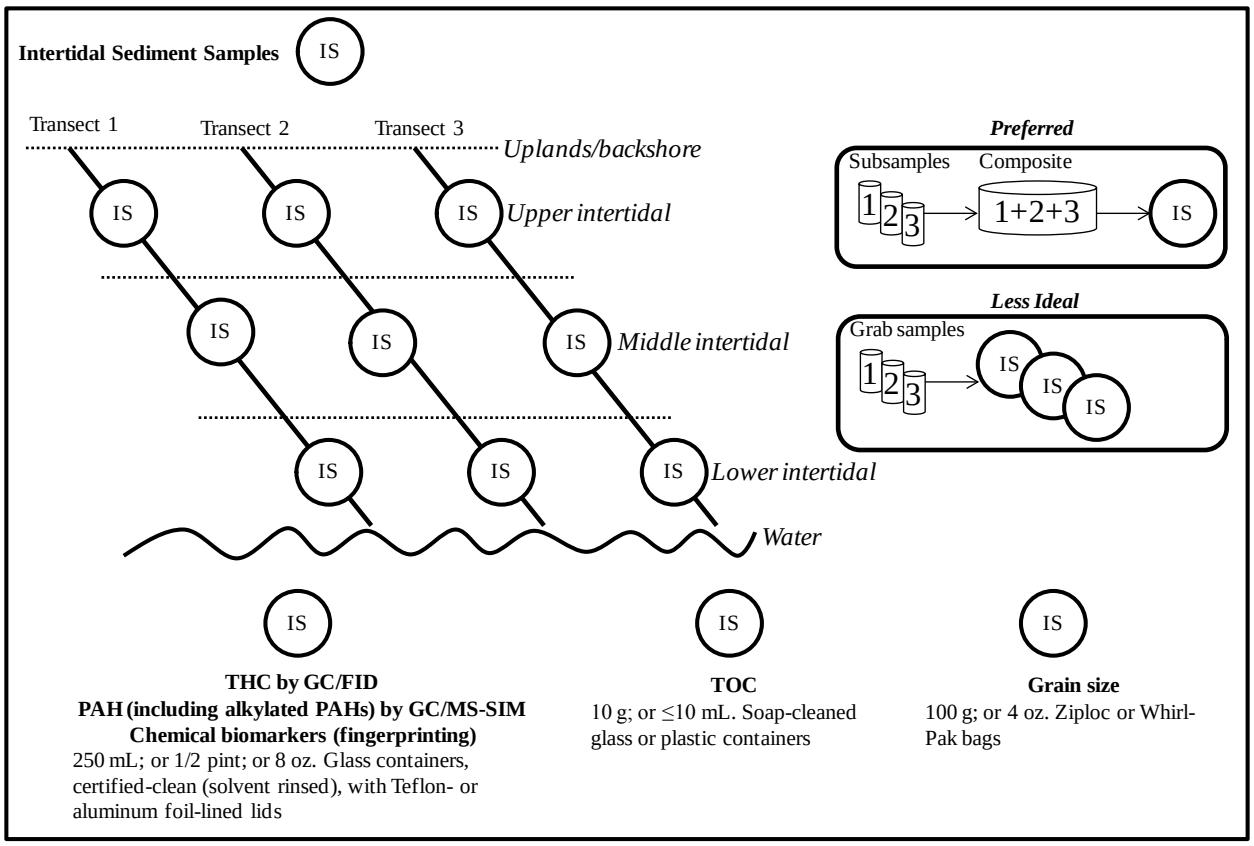

Figure 1. Schematic representation of the recommended intertidal sediment sampling strategy, including transects and sampling sites. Dashed lines represent approximate tidal zones. IS= intertidal sample. Area-specific modifications may be needed. For example at sites with very narrow intertidal zones, only one or two sampling intervals may be used.

- Divide each transects into sampling intervals based on the intertidal zones: upper intertidal, middle intertidal and lower intertidal.
- On some high-Arctic shorelines, the intertidal zone may be very narrow. If an intertidal transect is too narrow to have three sampling intervals on, consider running the transect from the supratidal or storm surge line (usually demarcated by a line of logs or debris) to the lower intertidal. Alternately, two sampling intervals can be defined on each transect, in the upper (or storm surge) and lower intertidal zone, or just one station if the intertidal zone is very narrow.
- For each transect, record:
  - Date, time, weather conditions (e.g., wind direction and speed), and tide level
  - Physical setting (shoreline orientation, exposure, etc.)
  - Length of the transect (in meters) and of the sampling zone
  - Sediment type
  - Extent and degree of visible shoreline oiling (use SCAT guidelines for a more detailed assessment of shoreline oiling if needed)
  - Extent and degree of shoreline cleanup, if already performed (particularly note sediment disturbance or removal, flushing of oil and/or sediments into down-slope areas, etc.)
  - Presence of snow or ice along shoreline
  - Presence of biological resources or other relevant information
- Collect samples from each intertidal zone (described below) and record the distance along the transect and GPS coordinates of each sampling site.

- If specified in the work plan, collect separate splits of homogenized samples for infauna or toxicity testing, so they can be correlated with chemical results.
- Take pictures of the transect before and after sampling and pictures of each sampling site.

Sample Collection Methods

- Use field data forms included in the work plan, if one is available. Otherwise, use forms in Appendix A. Coordinate data form development/modification with the data management group.
- Because GPS units will be used to record locations and times, make sure that all units are using the same coordinate system, datum, reporting units, and correct time. Follow the recommended GPS datum of the study plan, if one is available. Alternatively, set the default to WGS84.
- Record GPS coordinates for each sample site.
- Photograph the sampling site prior to sample collection to document the site conditions, as well as the sample collected. Make sure each photograph or series can be later associated with the corresponding sampling locations (e.g., through use of GPS Photo link software or by keeping a detailed photo log with waypoints and/or lat/long). Do not delete or alter any photographs. The numbering sequence of photographs uploaded from your camera must not have any gaps (see Field Photography guideline).
- If intertidal sediments are covered by snow or ice, carefully remove the snow/ice without mixing or disturbing the sediment underneath and proceed with sediment sampling. Note snow and ice conditions on the field data sheet. If snow or ice in the intertidal zone are impacted by oil (as opposed to deposited on top of oiled sediments), it may be desirable to collect snow/ice samples in addition to sediments for chemical analysis (see the Ice guideline).
- Special consideration should be given to sampling frozen sediments. Sediments frozen *in situ* (<-7°C) must be maintained frozen in appropriate containers and sub-sampled as soon as practical without thawing. Consider using a sturdy shovel, rock pick, or other tool to loosen sediments for sampling. Collection of frozen samples should be noted in the field logbook.
- Carefully remove gravel, sticks, and other debris on the sediment without mixing or disturbing the sediment underneath. For surface sediments, use a clean spatula to accurately collect the top 2 cm.
- Composite samples (of at least 3 subsamples) are preferred for characterization of a sampling site. Subsamples should be collected at random within a 5 m radius and within the same tidal zone or sampling interval (see Figure 1). Mix and homogenize composite samples before placing in the jar(s) using a disposable aluminum pan. Collect a minimum of 3 replicates per site. A less preferable alternative, if it is not feasible to composite samples in the field, is to collect a minimum of 3 grab samples at each sample site (interval on a transect).
- To collect surface samples from inundated tundra, use a metal spatula or shovel to cut the vegetative mat and collect a sample of the top layer of vegetation and sediments. Consider surveying the extent and degree of oiling and/or collecting stranded oil samples (see Stranded Oil guideline) if it is not possible to collect quantitative inundated tundra intertidal sediment surface samples. To collect subsurface samples in coarse sediments (sand and gravel), it is easiest to use a shovel to dig a small trench/pit and collect the desired sediment intervals from the exposed wall in the trench/pit.

- To collect subsurface samples in fine sediments (e.g., clay, silt, mud) use a shovel to dig out a slice of sediments to the desired depth. Collect the sample of the entire oiled layer from the natural break side of the shovel-full of sediments (see photograph). For example, if the oil penetrated from the surface to 10 cm, collect a representative sample of this entire layer; if the oiled layer was at a depth of 5-10 cm, collect a representative sample of this entire layer.

- Though oil is unlikely to penetrate inundated tundra shorelines, subsurface sediments samples can be collected in tundra by digging a small pit and collecting the desired sediment intervals from the exposed wall in the pit.
- For subsurface samples, record the depth of each sediment interval, total depth of the pit, details about the vertical profile including distinct layers, type of material and presence of biological structures, degree of sample disturbance, and the presence of oil, oily sheen and/or the smell of oil.
- In addition to collecting samples for chemical analysis, take samples for TOC (placed in soap-cleaned glass or plastic containers) and grain size (placed in Ziploc or Whirl-Pak bags) analysis.
- Collect representative non-viscous tarball or oil residues samples, which can be scooped into sampling jars using a stainless steel spatula or spoon, or wrapped in aluminum foil and double bagged in Ziploc bags with sample ID label placed between the inner and outer bags.
- Photograph the sampling pit (see Field Photography guideline).
- Clean the spade of the shovel with soap and water between each sampling pit to prevent cross contamination.
- Discrete samples from a single sample point may be collected to represent a specific condition, such as a tarball for fingerprinting and source identification (see Stranded Oil guideline).

Sample Labeling and Record Keeping

- Verify that all samples are properly labeled, and that field sample forms are properly filled out.
- Follow chain of custody procedures for securing samples and complete chain of custody forms (See Chain of Custody guidelines).
- Complete the Chain of Custody form, noting where each intertidal sample was collected, sampling equipment used, time/date of collection, size and container type, and sampler name.
- Make notation on the Chain of Custody form about any problems or observations during sampling.
- Maintain strict chain of custody during sample storage and transportation.
- Record the sample number on both the sample jar label and lid. Record the following on the field sample form:
  - Sample collection site (NRDA sample grid ID and GPS coordinates)
  - Sample matrix (sediment, soil, tundra)
  - Sample #, date/time
  - Sampling method (grab, core, composite) and sample collection depth
  - Note if sample is for QA/QC (field blank, trip blank, rinsate blank) or if it is a spilt or duplicate sample
  - Sediment oiling conditions (using standard shoreline assessment terminology), tidal elevation, weather conditions (e.g., wind direction and speed), sediment characteristics, vertical changes in sediment characteristics, presence of biota, vegetation or debris, odors and other relevant information on the field data sheet
  - Sediment characteristics: grain size, texture, color, biota, vegetation, debris, odor, etc.; vertical changes in sediment characteristics
- All sample numbers must be unique. Use the sample number convention provided by data management if available. Otherwise, the sample number should consist of a sample team ID and sequential numbers. For example AKA-0001, AKA-0002, etc.
- If sample volume is split between two jars, both jars should receive the same sample ID and be recorded on a single line of the Chain of Custody form.
- Documenting oil distribution in intertidal areas is best accomplished with photography, video, and good field notes and sketches using standard shoreline assessment methods. These data may be collected by SCAT teams and available to support environmental assessments. 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.

- Make a quick sketch in a field logbook or sketch form showing the sampling locations in enough detail that the location could be re-occupied by someone else.
- Keep a detailed photo log so that each photograph can be labeled.
- Note any deviations from the recommended guidelines in the field book.

Sample Preservation, Recommended Holding Times and Shipping

- Follow chain of custody procedures for sample storage and shipping.
- Immediately place all sediment samples for chemical analysis in a cooler and keep at approximately 4°C. Use frozen gel packs to maintain the temperature if ambient temperatures are above freezing. In below freezing temperatures, collapsible water jugs filled with warm water can be used to maintain the temperature if heated storage space is not available. A programmable temperature logger or thermometer should be placed in each cooler to maintain a record of storage temperatures.
- Refrigerate (do not freeze) samples for TOC. Samples for grain size do not require refrigeration.
- Protect samples for chemical analysis from direct sun exposure (e.g., UV radiation).
- Tape lids on sample bottles so that they do not accidentally come off.
- If possible, store samples for chemical analysis from unoiled locations in one set of coolers, with oiled samples in a separate set of coolers.
- Samples for TOC and grain size can be stored separately from samples for chemical analysis.
- Use packing material, such as bubble wrap or sorbent pads, around glass jars to prevent breakage during transport and shipping. Take special care with gravel sediments because individual pieces can rattle around during shipping and break the glass jars.
- Freeze samples for chemical analysis as soon as practical or by the end of each day if samples are not going to be analyzed within 7 days of collection.
- If sampling sediments frozen in situ (<-7°C), samples MUST be maintained frozen in appropriate containers and sub-sampled as soon as practical without thawing.
- 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.
- Ship samples directly to the lab as soon as practical with complete chain of custody forms. If necessary, samples can be stored under specified conditions and with complete chain of custody until they can be shipped. Assure that samples are packaged to protect them from breakage, shipping containers are sealed, and use ice packs or dry ice to maintain storage temperatures during shipment.
- Ship highly oil-contaminated samples separate from non-contaminated or low-contaminated samples to reduce risk of cross contamination.
- NEVER discard any samples even if these have exceeded their recommended holding times or storage temperatures.

Sample Volume and Requirements

Analytical Method	Sample Volume	Minimum Detection Levels [a]	Recommended Holding Time	Minimum No. of Samples per Location
Total Hydrocarbons (THC) by GC/FID	250 mL; or 1/2 pint; or 8 oz. Filled ¾ full [c]	15 µg/kg	7 days at 4°C [b] or several years frozen[c]	1 per site
PAH (including alkylated PAHs) by GC/MS-SIM		0.001-0.01 µg/kg		
Petroleum biomarkers (fingerprinting)		0.001-0.01 µg/kg		
Total Organic Carbon (TOC)	10 g; or ≤10 mL Filled ¾ full	0.01%	28 days	1 per site
Grain size	100 g; or 4 oz	NA	NA	1 per site

[a] µg/L= ppb; [b] Store at 4°C in the dark; [c] Stored frozen in the dark; [d] Several analyses can be made from a single sample.

Analytical Methods

- **Total hydrocarbons** (THC). Often referred to as total petroleum hydrocarbons (TPH), but most methods do not differentiate among petroleum, petrogenic, and biogenic hydrocarbons. THC by GC-FID (total area of FID gas chromatogram of combined f_1 and f_2 fractions after column chromatography; e.g., EPA Method 8015 Modified) is often the preferred method because of the low detection limit (compared to other THC methods) and the direct measurement of hydrocarbons. This method does not detect low boiling point compounds (below n-C_8). 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 document changes in oil weathering and map extent of exposure of water column resources, if meaningful detection limits are used. THC results can be used as a screening tool to estimate the presence and amount of hydrocarbons and provide an indication of which samples should receive highest priority for more extensive analyses.
- **Polycyclic aromatic hydrocarbons** (PAH). Because most of the toxicity in oil is due to the PAHs, it is often the preferred analysis. However, PAHs are expensive and require special laboratory skills. If PAHs are to be measured, it is important that the analytes 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 (SIM) mode. Detection levels should be at least 0.1 ppb for individual PAHs to support injury assessment using toxicity thresholds. Have the lab also run the source oil with each batch.
- **Petroleum biomarkers**. These chemicals are the most important hydrocarbon groups used for chemical fingerprinting allowing a quantitative identification of the source oil. Because biomarkers are more resistant to weathering and biodegradation than other hydrocarbons, these can also be used to quantify the degree of oil weathering. Biomarkers include steranes/triterpanes, which are "fossil" compounds unique to the oil formation and compounds that provide a secondary and confirming line of evidence in forensic oil identification. Chemical biomarkers are typically analyzed by GC-FID (e.g., EPA Method 8015 Modified). These chemicals are typically analyzed concurrently with THCs.
- Analyses may also include:
  - **Sediment grain size**. There are different methods for measuring sediment grain size and may include sieve analysis of gravel to sand fractions, pipette analysis for muddy sediments, or more sophisticated methods that use rapid sediment analyzer equipment
  - **Water content**. Water content is typically measured in conjunction with chemical and TOC analyses by drying a specified mass of sediment in an oven at constant temperature (typically 100-110 °C) and calculating the mass difference between wet and dry sediments
  - **Total organic carbon**. There are different methods for measuring TOC in sediments, and may include high-temperature combustion methods or more sophisticated methods that use total organic carbon analyzers

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Appendix A
Supporting Documentation - Field Data Form Examples

Unique field data forms may be included in the work plan if one has been developed, otherwise, use the attached form.

- Print the form on weather-resistant paper (if available). Make more than enough copies of the form before going into the field.
- Fill out forms with waterproof pen or permanent marker. Do not use pencil or biro (erasable) ink.
- Make any additional notes that do not fit on the form in a field notebook and indicate the presence of associated additional notes on the field data form.
- Fill in blanks with "N/A" if data are not applicable or not available. Avoid leaving blank values on data forms.
- Do not erase or black out erroneous entries on the field data forms. Errors should be corrected by crossing out the entry with a single line and initialing and dating the strike-through.
- Electronic versions of field data forms are available. Coordinate data entry with NRDA data management personnel.

Attached form:
- Soil/Sediment Sample Collection Form

Sample Collection Form - SOIL/SEDIMENT										
Lead Sampler's Name/Phone								**Sampler Team Code**		
Lead Sampler's Affiliation								**Resource Group**		
NRDA Contact/Phone								**Resource Group Leader**		
Incident Name								**Habitat (e.g., sand beach)**		
General Location Description								**Sample date (mm/dd/yyyy)**		
Location Code	**Matrix**	**Sample Number**	**Sample Time**	**Sampling Method**	**Sample Collection Depth**	**Depth units**	**Sample QA/QC Type**	**Latitude**	**Longitude**	**Sample Notes**
NRDA Sample Grid ID	*(S)ediment, Soil (L), Tundra (T)*	*Sample # (Team ID – sequential number)*	*(24-hr clock, local time)*	*Method of sampling (i.e., core, grab or composite)*	*Include upper and lower collection depth. Use 0 for surface samples.*	*Units for depth values*	*Normal sample or Field QA/QC type*	*Latitude in DD XX.XXXXXX*	*Longitude in DD-YYY.YYYYYY*	*Description of sample, equipment used, including estimated volume, photo numbers, etc.*

Survey Notes - (weather, wildlife, field team composition, sampling design changes, photos, etc.)

Samples Relinquished by:				Received by:			
Date	**Time**	**Signature - Field Sampler**	**Print Name- Field Sampler**	**Date**	**Time**	**Signature - Sample Runner/ Command Post**	**Print Name - Sample Runner/ Command Post**

Matrix	Sample methods and descriptions		Sample Area Sketch
Sediment or Soil	**Sampling Method**	**Depth units**	
(S)ediment	(GR)ab	(c)m	
Soil (L)	(CO)re	(m)	
(T)undra	Co(MP)osite	(i)nches	
Blan(K) Water		(f)eet	
Oil, Tarball or Water	**Sampling Method**	**Sample Position/Depth**	
(O)il	(GR)ab	(FLOAT)ing	
Tarball (B)	(SC)rape	(SUB)merged	
(W)ater	(OT)her	(STRAND)ed	
Blan(K) Water		(COV)ering	
Other (H)		0 - (Surf)ace	
		<depth in meters> m	
Tissue or Wrack	**Tissue Type**	**Tissue Type (Continued)**	
(T)issue	(WH)ole body	(MU)scle	
Wrack (R)	Whole body w/o shell (WNS)	Yolk	
Blan(K) Water	Chorioallantoic Membrane (CAM)	NA <for Wrack only>	
	Egg (EM)bryo		
	Fillet with skin (FS)	**Species**	
	Fillet without skin (FWOS)	<enter species>	
	Gall Bladder (GB)	NA <for Wrack only>	
	Leaves (LEV)		
	Leaves and stems (LVS)		
	(LI)ver		
Sample Identifier system			
Sample IDs : Team ID-Sequential Numbers (ex. AKA-0001)			
QA/QC types:		Other sample types:	
Field Blank (FB)	Rinsate Blank (RB)	(S)plit	
Trip Blank (TB)	(D)uplicate		