

Delaware River Channel Deepening Project
2010 Sediment Core Analysis
Reach B Contract (Station 137+000 to Station 176+000)

I. Scope of Work

The work under this contract includes collecting sediment cores from 21 locations within Reach B of the Delaware River Philadelphia to the Sea navigation channel and conducting bulk sediment analyses of 21 sediment samples collected from the cores and modified elutriate analyses of two sediment samples collected from the cores to evaluate the extent of any sediment contamination. Sediment samples will be collected between the Marcus Hook Range and the Cherry Island Range. Seventeen cores will be collected in the navigation channel, two cores will be collected in a Sun Marcus Hook berthing area and two sediment cores will be collected in a ConocoPhillips berthing area. Sample location coordinates are provided in Table 1. In addition to sediment cores, a sufficient quantity of water will be collected from the sampling site to prepare filtered and unfiltered modified elutriate samples for two sediment samples. The modified elutriate analysis will be conducted on sediment samples collected from cores DRV-93 and DRV-101. Sediments will be collected and appropriately preserved in the field and delivered to a laboratory for analyses. Bulk sediment samples will be analyzed for grain size (Folk, 1980), TAL inorganics, total mercury, TCL pesticides, organo phosphorus pesticides, PCB congeners/dioxin and furans, TCL volatile organic compounds, TCL semi-volatile organic compounds, cyanide, particulate organic carbon, dissolved organic carbon and total organic carbon. The filtered and unfiltered modified elutriate samples and the Delaware River water used to prepare the elutriates will be analyzed for TAL inorganics, total mercury, TCL pesticides, organo phosphorus pesticides, PCB congeners/dioxin and furans, TCL semi-volatile organic compounds, cyanide, particulate organic carbon, dissolved organic carbon, and total suspended sediment. Table 2 provides a list of the number and type of samples to be analyzed for each contaminant group. Sediment sampling and analysis will commence immediately after a Delivery Order is issued for this work. The contractor will prepare a brief report that documents the sampling procedures, sample preparation techniques, laboratory methods, QA/QC, test results and data analysis.

II. Corps Point of Contact

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III. Sediment Sample Collection

The work consists of furnishing all plant, labor, materials, supplies, and accessories required to accomplish the investigation, together with all other operations incidental to the work, in strict accordance with these specifications. Core sample locations and the approximate water depth are provided in Table 1. Changes necessitated by field conditions shall be approved by the Government over the telephone immediately prior to vibrational coring.

The U.S. Coast Guard, River Pilots Association and the Maritime Exchange for the Delaware River and Bay will have to be contacted for notification of working in and near the channel and for channel closing or ship traffic diversion that may be required as the vibracoring work proceeds. The vibracoring shall be performed during daylight hours and the floating plant shall be removed from the immediate channel area to a safe location for overnight mooring. The Sun Marcus Hook and ConocoPhillips facilities will also be contacted to schedule the work in their berthing areas.

All equipment and supplies, as specified herein, are subject to approval by the Contracting Officer prior to issuing notice to proceed. The vibracore subcontractor shall have a copy of these specifications on the drill rig and shall be familiar with the applicable provisions of the specifications governing the contract work at all times.

The work under this contract includes collecting sediment samples from twenty one (21) vibrational core borings. The work shall be performed in the Delaware River channel off the coast of Pennsylvania and Delaware. The areas to be sampled range in depth from approximately forty (40) to fifty (50) feet below MLLW. All vibrational cores are to be fifteen (15) feet long, with continuous samples obtained to the termination depth of the vibracores for geotechnical analysis and contaminant testing.

The order in which the vibrational cores are drilled will be at the Contractor's discretion, unless otherwise stated by the Government.

Positioning – The vibrational cores will be collected from locations furnished by the Government. These locations are or shall be designated by New Jersey state plane coordinates (NAD 83). The positioning of the vessel relative to the sample site locations shall meet standards outlined in the Corps of Engineers Hydrographic Surveying Manual, EM 1110-2-1003, dated 1 April 2004 for Class 1 surveys. Differential GPS positioning shall be determined immediately prior to the start of the vibrational core boring at each location. Positioning systems used for sediment sampling activities must have an accuracy of ± 1 meter. Before each core is taken, the exact position and the water depth at that point shall be recorded. Depths shall be corrected for tide and converted to North American Vertical Datum (NAVD 88) elevations and presented in depth below MLLW depth on the vibracores logs.

Vibrational core borings – Vibrational cores comprising the surface of the bottom and sub-bottom sediments shall be obtained by pneumatic- or hydraulically-activated borings

having a minimum diameter of three (3) inches. The continuous samples recovered shall be representative of the relative position of the bottom and sub-bottom strata. A transparent plastic rigid tube of appropriate size that permits visual inspection of the cored material will be placed inside the coring tool prior to operation. The same tube shall be removed from the coring tool when the operation at a site has been completed. If a core is to be transported to another location for processing, the plastic tube with the sample shall be cut into five (5) foot length segments for ease of handling and appropriately marked as to the sequence of segments and sample location. If necessary, packing will be inserted at both ends of the tube to prevent disturbance of the core during handling and shipping. Both ends of the segments will be sealed with plastic caps and plastic pressure sensitive tape. The plastic segments will be identified as to top and bottom of each core segment. The depth of the top and bottom shall be printed on the top and bottom of each five (5) foot core segment. The core identification number and the sample designation shall also be recorded on each core segment. This information shall be clearly and coherently marked on each five (5) foot core segment with a water-proof marking pen.

When located over a boring site, the Contractor shall make every reasonable effort to reach the required depth or to reach penetration refusal. Penetration refusal shall be completed when less than one tenth of one foot of advance is accomplished after one (1) minute of vibration with vibrating-type coring tool. Sample penetration of less than ten (10) feet will not be accepted as a complete sample. The depth of penetration shall be determined by measuring the depth to the vibrating core head and comparing it to the depth to the sediment surface. An estimate of the penetration resistance will be determined by timing the rate of penetration of the vibracore sampler during sampler penetration into the sediment. Depth of penetration beneath the surface of the bottom must be known to within plus or minus 0.5 feet of actual penetration. The desired depth of penetration is fifteen (15) feet. It is recognized, however, that maximum penetration may not be achieved at all sample locations. When refusal is met at less than ten (10) feet, the Contractor will remove the sampled portion from the pipe, and a new liner will be inserted into the core pipe. One retry will be accomplished in an attempt to reach at least ten (10) feet of penetration. The coring device shall recover a minimum of eighty (80) percent of the unconsolidated strata through which it has penetrated. The percent recovery will be measured by placing a tape measure, with a weighted end, down the top of the retrieved core to measure the distance to the top of the sediment in the core. This value will be compared to the measured depth of penetration to calculate percent recovery.

Upon completion of the sample collection activities, the Contractor shall provide a table that lists the individual sediment core sample identification numbers, the actual latitude/longitude and New Jersey State Plane coordinates where the individual sediment core samples were collected, and the actual water and sampling depths (i.e. core lengths) for each individual sediment core sample. This table shall also list any strata identified within each core sample. The Contractor shall also provide figures that identify the actual locations where the individual core samples were collected.

Upon retrieval, each sediment core will be inspected and a core log will be prepared. This work will be done by a geologist provided by the Government. When the core log is completed a sediment sample for contaminant testing will be collected. The sample will represent the portion of the core from the surface of the bottom to a depth of -46 feet below Mean Low Water (MLW). The geologist will determine the location of any distinct sediment strata greater than two feet in length within the core. If distinct sediment strata greater than two feet exist within the portion of the core representing the surface of the bottom to -46 feet MLW, the Corps Point of Contact will be contacted by telephone before any sample is collected. For cost estimating purposes, it is assumed that only one sediment sample will be analyzed in the laboratory per core. Material will be removed from the core(s) with stainless steel knives and spoons, thoroughly homogenized in a stainless steel container and stored in appropriate containers. All utensils and containers shall be appropriately washed to minimize any sample contamination. Enough sediment will be collected to provide sufficient material for later chemical and geotechnical analysis. **Samples for analysis of volatile and semi-volatile organic contaminants will be collected with as little disturbance to the sediments as possible (ie. prior to homogenization).**

When collecting individual sediment core samples, the Contractor must ensure that a sufficient volume of sediment is collected to conduct all of the tests specified in Table 2. This may entail the collection of additional individual core samples co-located with those specified in Table 1. If this occurs, the Contractor shall document how and where these additional core samples were collected.

Individual sediment core samples shall be homogenized consistent with the requirements specified in Chapter III-Section D(2)d & e of NJDEP *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (1997) and USEPA *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual EPA-823-B-01-002* (2001). Provided they are of uniform consistency (i.e. apparent grain size distribution and visual characteristics), individual sediment core samples six (6) feet or less in length may be homogenized. Individual sediment core samples greater than six (6) feet in length may be homogenized unless there are distinct visual strata in grain size and composition which are at least two (2) feet in depth. For those individual sediment core samples that show grain size stratification, each strata with a depth of two (2) feet or greater must be tested and analyzed separately (i.e. the entire core must not be homogenized for testing purposes if distinct strata are present). For cost estimating purposes, it is assumed that only one sediment sample will be analyzed in the laboratory per core. If distinct sediment strata greater than 2 feet occur in a core the Contractor will contact the Corps Point of Contact prior to compositing a sample.

For DRV-102, DRV-103, DRV-104 and DRV-105 the bottom six (6) inches of the sediment core sample shall be visually inspected to determine if the sediments are predominantly sand, gravel, silt or clay. If the bottom six (6) inch sample of a core is similar in grain size and visual characteristics to the overlying material in that core, these

sediments may then be homogenized with the rest of the sediment core (or appropriate core strata) sample for analysis. Otherwise, unless the bottom six (6) inch sample is greater than 90% sand (as determined by grain size analysis), Bulk Sediment Chemistry (only) analysis of the bottom six (6) inch sample shall be required. For cost estimating purposes, it is assumed that the bottom 6 inch samples from these cores will be similar in grain size to the overlying material.

The sample preservation requirements and holding times for each analysis, as specified in the analytical methods, must be adhered to. The sample handling, preservation, and storage procedures (including achieved holding times) shall be summarized in a report. Chain of custody documentation for each individual sediment core sample shall be maintained and provided to the Corps. Storage and preservation procedures for sediment samples are provided as Appendix A. These procedures are from: *The Management and Regulation of Dredging Activities and Dredged Material in New Jersey's Tidal Waters* (NJDEP, 1997). All analyses shall be conducted within the specified holding times. Samples to be analyzed for metals should not come in contact with metal sampling equipment, and samples to be analyzed for organic compounds should not come into contact with plastics. All sample containers should be appropriately cleaned: acid-rinsed (10% nitric acid) for metal analysis, and solvent-rinsed (acetone is preferred; however, other approved solvents such as methanol and hexane can be used as well) for organic analysis. When equipment will be used to take samples for both metal and organic compound analysis, the acid rinse must be conducted first, and the solvent rinse second. Samples should completely fill the storage container, leaving no head space, except for expansion volume required for potential freezing. Samples should be refrigerated or frozen with dry ice immediately after sample collection.

One equipment blank will be created in the field by pouring laboratory grade water over all sampling equipment that comes in contact with the sediments (e.g., stainless steel spoons, knives and bowls). The equipment blank will be created in the middle of the sampling, immediately after the equipment has been cleaned. The water will be collected into an appropriate sample container after it has been poured over the equipment, and analyzed for TAL inorganics, total mercury, TCL pesticides, organo phosphours pesticides, PCB congeners/dioxin and furans, TCL semi-volatiles, TCL volatiles and cyanide.

IV. Sediment and Water Sample Analysis

The Contractor shall prepare a table for the Methods Section of the report that provides the following:

- Target Analyte List (TAL)
- Analytical Method (for each target analyte)
- Analyte Method Detection Limits (MDL; for each target analyte)
- Required sample volume/mass
- Identify the certified laboratory that will perform the test

Sediment and water samples will be analyzed using the following procedures: TAL inorganics (SW846-6020), total mercury (USEPA Method 1631E), total cyanide (SW846-9012A), TCL pesticides (Low Level SW846-8081A), organo phosphorus pesticides (SW846-8141A), TCL volatiles (SW846-8260B) and TCL semi-volatile organic compounds including PAHs (Low Level SW846-8270C). Samples will also be analyzed for particulate organic carbon (POC) using USEPA Method 440.0, dissolved organic carbon (DOC) using USEPA Method 415.2 and total organic carbon analyses will follow the procedure provided as Attachment 2 to Appendix B. Total organic carbon will not be analyzed on the four elutriate water samples and one Delaware River water sample. These parameters are essential for partitioning calculations. In addition, sediment and water samples will be analyzed for PCB congeners using USEPA Method 1668A and a dioxin/furan analysis (USEPA Method 1613). Table 2 provides a complete summary of the analyses to be conducted for each of the tests. Attachment 1 of Appendix B provides a complete list of contaminant parameters and the required detection limits for sediment samples. The 21 bulk sediment samples will also be analyzed for grain size following the methods described by Folk (1980).

The modified elutriate test procedure can be found on the web at: www.wes.army.mil/el/dots/eedptn.html#section4 Environmental Effects of Dredging Technical Note EEDP-04-2. A slurry concentration of 150 grams of sediment per liter of river water (dry weight basis) will be used. After aeration the samples will be allowed to settle for 24 hours. Both filtered and unfiltered water samples will be obtained and analyzed from the modified elutriate test. In addition, the Delaware River water sample collected for preparation of the elutriates will be analyzed to represent background river conditions. All five samples will be analyzed for total suspended sediment.

All analytical laboratory procedures must be conducted by a laboratory certified by the New Jersey Department of Environmental Protection to conduct that procedure pursuant to the Regulations Governing the Certification of Laboratories and Environmental Measurements (NJ.A.C. 7:18) or the National Environmental Laboratory Accreditation Program (NELAP). Analytical detection limits for all target analytes must be below the applicable regulatory criteria or the analytical detection limit must not exceed the PQL.

To minimize, or otherwise address, analytical errors in the data to be collected, a Quality Assurance/Quality Control (QA/QC) Program shall be implemented in support of all sediment sample collection, testing, and analytical activities. The goal of this QA/QC program is to ensure the collection and analysis of sediment samples to provide accurate and high quality data. This will include the use of state-of-the art analytical methods for all sediment analyses. Analytical laboratories must follow all of the required QA/QC procedures specified in the analytical methods used. Any deviations from these procedures must be documented and justified. The sample preservation requirements and holding times for each analysis, as specified in the analytical method, must be adhered to.

The following Quality Control (QC) samples and procedures are required for the collection and chemical analysis of sediment and water samples:

- Field Blanks: one (1) with every batch of 1-20 samples.
Method Blanks: one (1) with every batch of 1-20 samples or every twelve (12) hours, whichever is less.
- Matrix Spike/Duplicate: one (1) with every batch of 1-20 samples.
- Surrogate Spike Recovery: each sample (organic compounds only).
- Duplicate/Split Samples: analyses to be conducted as per method requirements.
- Minimum Detection Limit (MDL) Verification: within last two (2) years, to be submitted to the NJDEP upon request.

The accuracy of the analytical methods used must be within ± 20 per cent compared to a certified reference material, standard reference material, or another standard with a known concentration. The precision of analytical laboratory replicates must be within ± 20 per cent (20%), and the precision of field duplicate samples within ± 30 per cent (30%), calculated using relative per cent difference.

All routine procedures associated with the sampling, handling, transport, storage, preservation, and analysis of the sediments must be specified in Standard Operating Procedure (SOP) documents maintained by the parties actually collecting and/or analyzing the sediments. These SOPs must be followed when collecting, handling, transporting, storing, preserving, and analyzing the sediments. Any deviations from these SOPs must be documented and justified. These SOPs must be available for review, if so requested.

Sample handling, storage, preservation, and transport procedures must be implemented so as to maintain sample integrity and minimize potential contamination from external sources. Samples must be preserved immediately after collection, homogenization, and/or compositing.

Sample chain of custody must be documented, beginning with sample collection and continuing through all sample handling, transport, preservation, and storage activities, ending with laboratory analysis of the sample. The sample custody documentation must identify the condition of the sample (temperature/preservation status, etc.). The NJDEP Field Sampling Procedures Manual (NJDEP, 2005) presents and discusses the NJDEP's sample chain of custody requirements (<http://www.state.nj.us/dep/srp/guidance/fspm/>).

V. Report Format and Content

The Contractor shall provide electronic copies of the summary analytical data reports prepared by the analytical laboratories. These reports will include, at a minimum, the following information:

- Laboratory certification number for all performed analyses;
- A summary of all sample handling, storage, preparation, and analysis methods.
- A summary of sample preservation methods, sample conditions, and actual sample holding times (before and after extraction);
- Sample analytical results for all required parameters and target analytes, including method detection limits and practical quantitation levels (PQLs), and laboratory QA flags;
- QA/QC program summary;
- A discussion of any analytical problems, corrective actions taken, and potential effects on interpretation of the sample data.

Laboratory reports shall be provided as they become available from the laboratory rather than after completion of a report.

A brief report will be prepared that describes all methodologies and the data obtained. The intent of the report is to document the sampling procedures, sample preparation techniques, laboratory methods, QA/QC and test results. The report will include figures that show the core sample locations. The report will also include tables that display the concentrations of detected parameters. The bulk sediment tables will display New Jersey Department of Environmental Protection residential and non-residential soil remediation criteria. The bulk sediment tables will include the particulate organic carbon, dissolved organic carbon and total organic carbon data. The report shall include all laboratory data sheets. Grain size curves will also be provided for each of the 21 sediment samples.

Bulk sediment data collected from the berthing areas of Sun Oil Marcus Hook and ConocoPhillips and the navigation channel will be analyzed for the potential impact on Delaware River water quality during deepening of these berthing areas from 40 to 45 feet using the methodology provided in: *Greene, Rick 2010. An Evaluation of Toxic Contaminants in the Sediments of the Tidal Delaware River and Potential Impacts Resulting from Deepening the Main Navigation Channel in Reach C. Delaware DNREC Division of Water Resources, Watershed Assessment Branch. Dover, DE.* Both data analysis and report preparation will follow the approach presented in the referenced report. Analyses will include inorganics, pesticides, semi-volatile organics and PCBs. Three separate analyses will be prepared (Sun Oil Marcus Hook, ConocoPhillips, and the navigation channel).

VI. Period of Performance

One electronic copy of a draft report will be submitted to the Corps within three months of issuing a Delivery Order for this work. The draft report must be a polished product and an accurate representation of the content of the final report. The draft must be clean-

typed, complete with all figures, tables and sections of the report. All graphics will appear in the same format and general location in the draft report as they will in the final report.

The Corps will provide the Contractor with comments on the draft report. The Contractor will then have an additional week to revise and submit the final report. The Contractor shall submit five bound copies and a pdf file of the final report. When the Corps accepts the final report the Delivery Order will be complete.

This Delivery Order must be complete by 31 October 2010.

VII. Inspection

All work will be conducted under the general discretion of the Contracting Officer and shall be subject to inspection by his appointed inspectors to insure strict compliance with the terms of the contract. The presence of the inspector shall not relieve the Contractor of responsibility for the proper execution of the work in accordance with the above specifications.

**Delaware River Channel Deepening Project
2010 Sediment Core Analysis
Reach B Contract (Station 137+000 to Station 176+000)**

Table 1. Sediment Core Sample Location Coordinates.

| <u>Core</u> | <u>State</u> | <u>Northing</u> | <u>Easting</u> | <u>Approximate Water Depth (- feet MLW)</u> |
|-------------|--------------|-----------------|----------------|---|
| DRV-89 | NJ | 350184 | 225899 | -41.9 |
| DRV-90 | NJ | 349355 | 224811 | -42.2 |
| DRV-91 | NJ | 348713 | 223791 | -42.2 |
| DRV-92 | NJ | 347561 | 222208 | -43.1 |
| DRV-93 | NJ | 346425 | 222588 | -43.0 |
| DRV-94 | NJ | 346336 | 221248 | -42.3 |
| DRV-95 | NJ | 345904 | 220877 | -42.9 |
| DRV-96 | NJ | 345021 | 220246 | -41.9 |
| DRV-97 | NJ | 341553 | 217688 | -44.1 |
| DRV-98 | NJ | 337428 | 214662 | -41.1 |
| DRV-99 | NJ | 335212 | 213974 | -44.4 |
| DRV-100 | NJ | 332673 | 212125 | -42.4 |
| DRV-101 | NJ | 332416 | 211400 | -41.7 |
| DRV-102 | NJ | 325407 | 209195 | -39.2 |
| DRV-103 | NJ | 323987 | 208790 | -39.7 |
| DRV-104 | NJ | 322519 | 208920 | -40.6 |
| DRV-105 | NJ | 321951 | 208047 | -41.2 |
| SMH-1 | DE | 658031.15 | 655862.92 | NA |
| SMH-2 | DE | 657048.40 | 654627.98 | NA |
| CP-1 | DE | 660044.80 | 659001.20 | NA |
| CP-2 | DE | 659705.09 | 658677.90 | NA |

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Table 2. Summary Matrix of Required Testing.

| | <u>Laboratory Method</u> | <u>Bulk Sediment</u> | <u>Modified Elutriate*</u> | <u>Equipment Blank</u> | <u>Total</u> |
|------------------------------|--------------------------|----------------------|----------------------------|------------------------|--------------|
| TAL Inorganics | SW846-6020 | 21 | 5 | 1 | 27 |
| Mercury | USEPA Method 1631E | 21 | 5 | 1 | 27 |
| TCL Pesticides | Low Level SW846-8081A | 21 | 5 | 1 | 27 |
| Organo-Phosphorus Pesticides | SW846-8141A | 21 | 5 | 1 | 27 |
| TCL Semi-Volatiles | Low Level SW846-8270C | 21 | 5 | 1 | 27 |
| TCL Volatiles | SW846-8260B | 21 | 0 | 1 | 22 |
| PCB Congeners | USEPA Method 1668A | 21 | 5 | 1 | 27 |
| Dioxin and Furans | USEPA Method 1613 | 21 | 5 | 1 | 27 |
| Cyanide | SW846-9012A | 21 | 5 | 1 | 27 |
| Total Organic Carbon | Appendix B Attachment 2 | 21 | 0 | 0 | 21 |
| Particulate Organic Carbon | USEPA Method 440.0 | 21 | 5 | 0 | 26 |
| Dissolved Organic Carbon | USEPA Method 415.2 | 21 | 5 | 0 | 26 |
| Grain Size | Folk 1980 | 21 | 0 | 0 | 21 |
| Total Suspended Sediment | Filter Method | 0 | 5 | 0 | 5 |

* Includes one sample of Delaware River water used to prepare the elutriates.

APPENDIX A
SUMMARY OF RECOMMENDED PROCEDURES FOR
SAMPLE COLLECTION, PRESERVATION AND
STORAGE

Attachment 1

SUMMARY OF RECOMMENDED PROCEDURES FOR SAMPLE COLLECTION, PRESERVATION, AND STORAGE

| Analyses | Collection Method ^a | Sample Volume ^b | Container ^c | Preservation Technique | Storage Conditions | Holding Times ^d |
|--|--------------------------------|----------------------------------|--|--|-------------------------|--|
| Sediment | | | | | | |
| Chemical/Physical Analyses | | | | | | |
| Metals | Grab/corer | 100 g | Precleaned polyethylene jar ^e | Dry ice ^f or freezer storage for extended storages; otherwise refrigerate | ≤ 4°C | Hg - 28 days Others - 6 months ^g |
| Organic compounds (e.g., PCBs, pesticides, polycyclic aromatic hydrocarbons) | Grab/corer | 250 g | Solvent-rinsed glass jar with Teflon ^h lid ⁱ | Dry ice ^f or freezer storage for extended storage; otherwise refrigerate | ≤ 4°C/dark ^j | 14 days ^o |
| Particle size | Grab/corer | 100 g | Whirl-pac bag ^k | Refrigerate | < 4°C | Undetermined |
| Total organic carbon | Grab/corer | 50 g | Heat treated glass vial with Teflon ^h -lined lid ⁱ | Dry ice ^f or freezer storage for extended storage; otherwise refrigerate | ≤ 4°C ^o | 14 days |
| Total solids/specific gravity | Grab/corer | 50 g | Whirl-pac bag | Refrigerate | < 4°C | Undetermined |
| Miscellaneous | Grab/corer | ≥ 50 g | Whirl-pac bag | Refrigerate | < 4°C | Undetermined |
| Sediment from which elutriate is prepared | Grab/corer | Depends on tests being performed | Glass with Teflon ^h -lined lid | Completely fill and refrigerate | 4°C/dark/airtight | 14 days |
| Biological Tests | | | | | | |
| Dredged material | Grab/corer | 12-15 L per sample | Plastic bag or container ^r | Completely fill and refrigerate; sieve | 4°C/dark/airtight | 14 days ^s |
| Reference sediment | Grab/corer | 45-50 L per test | Plastic bag or container ^r | Completely fill and refrigerate; sieve | 4°C/dark/airtight | 14 days ^s |
| Control sediment | Grab/corer | 21-25 L per test | Plastic bag or container ^r | Completely fill and refrigerate; sieve | 4°C/dark/airtight | 14 days ^s |

| Analyses | Collection Method ^a | Sample Volume ^b | Container ^c | Preservation Technique | Storage Conditions | Holding Times ^d |
|---|--------------------------------|----------------------------|--|--|--------------------|---|
| Water and Effluents | | | | | | |
| Chemical/Physical Analyses | | | | | | |
| Particulate analysis | Discrete sampler or pump | 500-2,000 mL | Plastic or glass | Lugols solution and refrigerate | 4°C | Undetermined |
| Metals | Discrete sampler or pump | 1 L | Acid-rinsed polyethylene or glass jar ^e | pH < 2 with HNO ₃ ; refrigerate ^f | 4°C | Hg - 14 days Others - 6 months ^g |
| Total Kjeldahl nitrogen | Discrete sampler or pump | 100-200 mL | Plastic or glass ^h | H ₂ SO ₄ to pH < 2; refrigerate | 4°C ⁱ | 24 h ^j |
| Chemical oxygen demand | Discrete sampler or pump | 200 mL | Plastic or glass ^h | H ₂ SO ₄ to pH < 2; refrigerate | 4°C ⁱ | 7 days ^k |
| Total organic carbon | Discrete sampler or pump | 100 mL | Plastic or glass ^h | H ₂ SO ₄ to pH < 2; refrigerate | 4°C ⁱ | <48 hours ^l |
| Total inorganic carbon | Discrete sampler or pump | 100 mL | Plastic or glass ^h | Airtight seal; refrigerate ^m | 4°C ⁱ | 6 months ⁿ |
| Phenolic compounds | Discrete sampler or pump | 1 L | Glass ^h | 0.1-1.0 g CuSO ₄ ; H ₂ SO ₄ to pH < 2; refrigerate | 4°C ⁱ | 24 hours ^o |
| Soluble reactive phosphates | Discrete sampler or pump | -- | Plastic or glass ^h | Filter; refrigerate ^p | 4°C ⁱ | 24 hours ^o |
| Extractable organic compounds (e.g., semi-volatile compounds) | Discrete sampler or pump | 4 L | Amber glass bottle ^q | pH < 2, 6N HCl; airtight seal; refrigerate | 4°C ⁱ | 7 days for extraction; 40 days for sample extract analyses ^r |
| Volatile organic compounds | Discrete sampler or pump | 80 mL | Glass vial ^r | pH < 2 with 1:1 HCl; refrigerate in airtight, completely filled container ^s | 4°C ⁱ | 14 days for sample analysis, if preserved ^t |
| Total phosphorus | Discrete sampler or pump | -- | Plastic or glass ^h | H ₂ SO ₄ to pH < 2; refrigerate | 4°C ⁱ | 7 days ^u |

| Analyses | Collection Method* | Sample Volume ^b | Container ^c | Preservation Technique | Storage Conditions | Holding Times ^d |
|---------------------------------|--|----------------------------------|---|---|---|--|
| Total solids | Discrete sampler or pump | 200 mL | Plastic or glass ^e | Refrigerate | 4°C ^f | 7 days ^g |
| Volatile solids | Discrete sampler or pump | 200 mL | Plastic or glass ^e | Refrigerate | 4°C ^f | 7 days ^g |
| Sulfides | Discrete sampler or pump | " | Plastic or glass ^e | pH > 9 NaOH (ZnAc); refrigerate ^h | 4°C ^f | 24 hours ^g |
| Biological Tests | | | | | | |
| Site water | Grab | Depends on tests being performed | Plastic carboy | Refrigerate | < 4°C | 14 days |
| Dilution water | Grab or makeup | Depends on tests being performed | Plastic carboy | Refrigerate | < 4°C | 14 days |
| Tissue | | | | | | |
| Metals | Trawl/Teflon ^o -coated grab | 5-10 g | Double Ziploc ^o | Handle with non-metallic forceps; plastic gloves; dry ice ⁱ | ≤ -20°C ^j or freezer storage | Hg - 28 days Others - 6 months ^m |
| PCBs and chlorinated pesticides | Trawl/Teflon ^o -coated grab | 10-25 g | Hexane-rinsed double aluminum foil and double Ziploc ^o | Handle with hexane-rinsed stainless steel forceps; dry ice ⁱ | ≤ -20°C ^j or freezer storage | 14 days ^g |
| Volatile organic compounds | Trawl/Teflon ^o -coated grab | 10-25 g | Heat-cleaned aluminum foil and water-light plastic bag ^l | Covered ice chest ^l | ≤ -20°C ^m or freezer storage | 14 days ^g |
| Semivolatile organic compounds | Trawl/Teflon ^o -coated grab | 10-25 g | Hexane-rinsed double aluminum foil and double Ziploc ^o | Handle with hexane-rinsed stainless steel forceps; dry ice ⁱ | ≤ -20°C ^j or freezer storage | 14 days ^g |
| Lipids | Trawl/Teflon ^o -coated grab | Part of organic analyses | Hexane-rinsed aluminum foil | Handle with hexane-rinsed stainless steel forceps; quick freeze | ≤ -20°C ^j or freezer storage | 14 days ^g |

ote: This table contains only a summary of collection, preservation, and storage procedures for samples. The cited references should be consulted for a more detailed description of these procedures.

PCB · polychlorinated biphenyl

· Collection method should include appropriate liners.

· Amount of sample required by the laboratory to perform the analysis (wet weight or volume provided, as appropriate). Miscellaneous sample size for sediment should be increased if auxiliary analytes that cannot be included as part of the organic or metal analyses are added to the list. The amounts shown are not intended as firm values. More or less tissue may be required depending on the analytes, matrices, detection limits, and particular analytical laboratory.

· All containers should be certified as clean according to U.S. EPA (1990c).

· These holding times are for sediment, water, and tissue based on guidance that is sometimes administrative rather than technical in nature. There are no promulgated, scientifically based holding time criteria for sediments, tissues, or elutriates. References should be consulted if holding times for sample extracts are desired. Holding times are from the time of sample collection.

NOAA (1989).

Tetra Tech (1986a).

Sample may be held for up to 1 year if $\leq -20^{\circ}\text{C}$.

Polypropylene should be used if phthalate bioaccumulation is of concern.

Two weeks is recommended; sediments must not be held for longer than 8 weeks prior to biological testing.

U.S. EPA (1987a); 40 CFR Part 136, Table III.

Plumb (1981).

If samples are not preserved to $\text{pH} < 2$, then aromatic compounds must be analyzed within 7 days.

· Tetra Tech (1986b).

Excerpted from pp. 54-57 of the USEPA "QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations", Office of Water (EPA 823-B-95-0001, April 1995).

APPENDIX B

ATTACHMENT 1

Exhibit C -- Section 1
Medium Level CRQLs

TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

1.0 Aqueous and Soil Medium Level CRQLs

Target Analyte Metals and Cyanide for Medium Level Aqueous Samples and Soil/Sediment/Solid Samples

| Analyte | Medium Level Contract Required Quantitation Limits ^(1,2) | |
|-----------|---|--------------------------------|
| | Water (µg/L) | Soil ⁽³⁾ (mg/Kg) |
| Aluminum | 200 | 40 |
| Antimony | 60 | 12 |
| Arsenic | 10 | 2 |
| Barium | 200 | 40 |
| Beryllium | 5 | 1 |
| Cadmium | 5 | 1 |
| Calcium | 5000 | 1000 |
| Chromium | 10 | 2 |
| Cobalt | 50 | 10 |
| Copper | 25 | 5 |
| Iron | 100 | 20 |
| Lead | 3 | 0.6 |
| Magnesium | 5000 | 1000 |
| Manganese | 15 | 3 |
| Mercury | 0.2 | 0.1 |
| Nickel | 40 | 8 |
| Potassium | 5000 | 1000 |
| Selenium | 5 | 1 |
| Silver | 10 | 2 |
| Sodium | 5000 | 1000 |
| Thallium | 10 | 2 |
| Vanadium | 50 | 10 |
| Zinc | 20 | 4 |
| Cyanide | 10 | 2.5 |

- (1) Method detection limits must be less than or equal to one-third of the Contract Required Quantitation Limits (CRQLs).
- (2) Subject to the restrictions specified in Exhibits D and E, any analytical method specified in the REAP Inorganic SOW, Exhibit D, may be utilized as long as the documented method detection limits achieve the CRQL requirements (MDLs must be less than or equal to one-third of the CRQLs). Higher detection limits may only be used in the following circumstance:

If the sample concentration exceeds five times the method detection limit, the value may be reported even though the method detection limit may not meet the Contract Required Quantitation Limit requirement. This is illustrated in the example below:



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Pesticides/Aroclors Target Compound List and Corresponding CRQLs

| COMPOUND | CONTRACT REQUIRED QUANTITATION LIMITS | | |
|---------------------|---------------------------------------|----------------------------|----------------------------|
| | OLC03.2 Water (ug/L) | OLM04.2 Water (ug/L) | OLM04.2 Soil (ug/Kg) |
| alpha-BHC | 0.01 | 0.05 | 1.7 |
| beta-BHC | 0.01 | 0.05 | 1.7 |
| delta-BHC | 0.01 | 0.05 | 1.7 |
| gamma-BHC (Lindane) | 0.01 | 0.05 | 1.7 |
| Heptachlor | 0.01 | 0.05 | 1.7 |
| Aldrin | 0.01 | 0.05 | 1.7 |
| Heptachlor epoxide | 0.01 | 0.05 | 1.7 |
| Endosulfan I | 0.01 | 0.05 | 1.7 |
| Dieldrin | 0.02 | 0.10 | 3.3 |
| 4,4'-DDE | 0.02 | 0.10 | 3.3 |
| Endrin | 0.02 | 0.10 | 3.3 |
| Endosulfan II | 0.02 | 0.10 | 3.3 |
| 4,4'-DDD | 0.02 | 0.10 | 3.3 |
| Endosulfan sulfate | 0.02 | 0.10 | 3.3 |
| 4,4'-DDT | 0.02 | 0.10 | 3.3 |
| Methoxychlor | 0.10 | 0.50 | 17.0 |
| Endrin ketone | 0.02 | 0.10 | 3.3 |
| Endrin aldehyde | 0.02 | 0.10 | 3.3 |
| alpha-Chlordane | 0.01 | 0.05 | 1.7 |
| gamma-Chlordane | 0.01 | 0.05 | 1.7 |
| Toxaphene | 1.0 | 5.0 | 170.0 |
| Aroclor-1016 | 0.20 | 1.0 | 33.0 |
| Aroclor-1221 | 0.40 | 2.0 | 67.0 |

| | | | |
|--|------|-----|------|
| Aroclor-1232  | 0.20 | 1.0 | 33.0 |
| Aroclor-1242  | 0.20 | 1.0 | 33.0 |
| Aroclor-1248  | 0.20 | 1.0 | 33.0 |
| Aroclor-1254  | 0.20 | 1.0 | 33.0 |
| Aroclor-1260  | 0.20 | 1.0 | 33.0 |

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Volatile Target Compound List and Corresponding CRQLs

| COMPOUND | CONTRACT REQUIRED QUANTITATION LIMITS | | | |
|---|---------------------------------------|----------------------|--------------------------|-----------------------------|
| | OLC03.2 Water (ug/L) | OLM04.2 Water (ug/L) | OLM04.2 Low Soil (ug/Kg) | OLM04.2 Medium Soil (ug/Kg) |
| Dichlorodifluoromethane | 0.5 | 10 | 10 | 1,200 |
| Chloromethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Bromomethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Vinyl Chloride EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Chloroethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Trichlorofluoromethane | 0.5 | 10 | 10 | 1,200 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 0.5 | 10 | 10 | 1,200 |
| Methylene Chloride EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Acetone EXIT disclaimer > | 5 | 10 | 10 | 1,200 |
| Carbon Disulfide | 0.5 | 10 | 10 | 1,200 |
| Methyl Acetate | 0.5 | 10 | 10 | 1,200 |
| 1,1-Dichloroethene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,1-Dichloroethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| cis-1,2-Dichloroethene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| trans-1,2-Dichloroethene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Methyl tert-Butyl Ether | 0.5 | 10 | 10 | 1,200 |
| Chloroform EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,2-Dichloroethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 2-Butanone EXIT disclaimer > | 5 | 10 | 10 | 1,200 |
| Bromochloromethane | 0.5 | NA | NA | NA |

| | | | | |
|---|-----|----|----|-------|
| 1,1,1-Trichloroethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Cyclohexane | 0.5 | 10 | 10 | 1,200 |
| Carbon Tetrachloride EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Bromodichloromethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,2-Dichloropropane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| cis-1,3-Dichloropropene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Trichloroethene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Methylcyclohexane | 0.5 | 10 | 10 | 1,200 |
| Dibromochloromethane | 0.5 | 10 | 10 | 1,200 |
| 1,1,2-Trichloroethane | 0.5 | 10 | 10 | 1,200 |
| Benzene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| trans-1,3-Dichloropropene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Bromoform EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Isopropylbenzene | 0.5 | 10 | 10 | 1,200 |
| 4-Methyl-2-pentanone | 5 | 10 | 10 | 1,200 |
| 2-Hexanone EXIT disclaimer > | 5 | 10 | 10 | 1,200 |
| Tetrachloroethene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,2-Dibromoethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Toluene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,1,2,2-Tetrachloroethane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Chlorobenzene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Ethylbenzene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Styrene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| Xylenes (Total) EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,2-Dibromo-3-chloropropane EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,3-Dichlorobenzene | 0.5 | 10 | 10 | 1,200 |
| 1,4-Dichlorobenzene EXIT disclaimer > | 0.5 | 10 | 10 | 1,200 |
| 1,2-Dichlorobenzene | 0.5 | 10 | 10 | 1,200 |
| 1,2,3-Trichlorobenzene | 0.5 | NA | NA | NA |
| 1,2,4-Trichlorobenzene | 0.5 | 10 | 10 | 1,200 |



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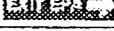
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Semivolatile Target Compound List and Corresponding CRQLs

| COMPOUND | CONTRACT REQUIRED QUANTITATION LIMITS | | | |
|------------------------------|---------------------------------------|----------------------------|--------------------------------|-----------------------------------|
| | OLC03.2 Water (ug/L) | OLM04.2 Water (ug/L) | OLM04.2 Low Soil (ug/Kg) | OLM04.2 Medium Soil (ug/Kg) |
| Benzaldehyde | 5 | 10 | 330 | 10,000 |
| Phenol | 5 | 10 | 330 | 10,000 |
| bis(2-Chloroethyl) ether | 5 | 10 | 330 | 10,000 |
| 2-Chlorophenol | 5 | 10 | 330 | 10,000 |
| 2-Methylphenol | 5 | 10 | 330 | 10,000 |
| 2,2'-oxybis(1-Chloropropane) | 5 | 10 | 330 | 10,000 |
| Acetaphenone | 5 | 10 | 330 | 10,000 |
| 4-Methylphenol | 5 | 10 | 330 | 10,000 |
| N-Nitroso-di-n-propylamine | 5 | 10 | 330 | 10,000 |
| Hexachloroethane | 5 | 10 | 330 | 10,000 |
| Nitrobenzene | 5 | 10 | 330 | 10,000 |
| Isophorone | 5 | 10 | 330 | 10,000 |
| 2-Nitrophenol | 5 | 10 | 330 | 10,000 |
| 2,4-Dimethylphenol | 5 | 10 | 330 | 10,000 |
| bis(2-Chloroethoxy) methane | 5 | 10 | 330 | 10,000 |
| 2,4-Dichlorophenol | 5 | 10 | 330 | 10,000 |
| Naphthalene | 5 | 10 | 330 | 10,000 |
| 4-Chloroaniline | 5 | 10 | 330 | 10,000 |
| Hexachlorobutadiene | 5 | 10 | 330 | 10,000 |
| Caprolactam | 5 | 10 | 330 | 10,000 |
| 4-Chloro-3-methylphenol | 5 | 10 | 330 | 10,000 |
| 2-Methylnaphthalene | 5 | 10 | 330 | 10,000 |
| | | | | |

| | | | | |
|-----------------------------|----|----|-----|--------|
| Hexachlorocyclopentadiene | 5 | 10 | 330 | 10,000 |
| 2,4,6-Trichlorophenol | 5 | 10 | 330 | 10,000 |
| 2,4,5-Trichlorophenol | 20 | 25 | 830 | 25,000 |
| 1,1' Biphenyl | 5 | 10 | 330 | 10,000 |
| 2-Chloronaphthalene | 5 | 10 | 330 | 10,000 |
| 2-Nitroaniline | 20 | 25 | 830 | 25,000 |
| Dimethylphthalate | 5 | 10 | 330 | 10,000 |
| Acenaphthylene | 5 | 10 | 330 | 10,000 |
| 2,6-Dinitrotoluene | 5 | 10 | 330 | 10,000 |
| 3-Nitroaniline | 20 | 25 | 830 | 25,000 |
| Acenaphthene | 5 | 10 | 330 | 10,000 |
| 2,4-Dinitrophenol | 20 | 25 | 830 | 25,000 |
| 4-Nitrophenol | 20 | 25 | 830 | 25,000 |
| Dibenzofuran | 5 | 10 | 330 | 10,000 |
| 2,4-Dinitrotoluene | 5 | 10 | 330 | 10,000 |
| Diethylphthalate | 5 | 10 | 330 | 10,000 |
| 4-Chlorophenyl-phenylether | 5 | 10 | 330 | 10,000 |
| Fluorene | 5 | 10 | 330 | 10,000 |
| 4-Nitroaniline | 20 | 25 | 830 | 25,000 |
| 4,6 Dinitro-2-methylphenol | 20 | 25 | 830 | 25,000 |
| N-Nitrosodiphenylamine | 5 | 10 | 330 | 10,000 |
| 4-Bromophenyl-phenylether | 5 | 10 | 330 | 10,000 |
| Hexachlorobenzene | 5 | 10 | 330 | 10,000 |
| Atrazine | 5 | 10 | 330 | 10,000 |
| Pentachlorophenol | 5 | 25 | 830 | 25,000 |
| Phenanthrene | 5 | 10 | 330 | 10,000 |
| Anthracene | 5 | 10 | 330 | 10,000 |
| Carbazole | NA | 10 | 330 | 10,000 |
| Di-n-butylphthalate | 5 | 10 | 330 | 10,000 |
| Fluoranthene | 5 | 10 | 330 | 10,000 |
| Pyrene | 5 | 10 | 330 | 10,000 |
| Butylbenzylphthalate | 5 | 10 | 330 | 10,000 |
| 3,3'-Dichlorobenzidine | 5 | 10 | 330 | 10,000 |
| Benzo(a)anthracene | 5 | 10 | 330 | 10,000 |
| Chrysene | 5 | 10 | 330 | 10,000 |
| bis-(2-Ethylhexyl)phthalate | 5 | 10 | 330 | 10,000 |

| | | | | |
|--|---|----|-----|--------|
| Di-n-octylphthalate | 5 | 10 | 330 | 10,000 |
| Benzo(b)fluoranthene  | 5 | 10 | 330 | 10,000 |
| Benzo(k)fluoranthene  | 5 | 10 | 330 | 10,000 |
| Benzo(a)pyrene  | 5 | 10 | 330 | 10,000 |
| Indeno(1,2,3-cd)pyrene  | 5 | 10 | 330 | 10,000 |
| Dibenzo(a,h)anthracene  | 5 | 10 | 330 | 10,000 |
| Benzo(g,h,i)perylene  | 5 | 10 | 330 | 10,000 |
| 1,2,4,5-Tetrachlorobenzene | 5 | NA | NA | NA |

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Attachment 2

DETERMINATION OF TOTAL ORGANIC CARBON

1.0 APPLICATION AND SCOPE

This method, developed by the U.S. Environmental Protection Agency, Region II, Environmental Services Division laboratory in Edison, New Jersey, describes protocols for the determination of organic carbon in ocean sediments. Although the detection limit may vary with procedure or instrument, a minimum reporting value of 100 mg/kg will be required for the ocean dumping/dredging program. Several types of determinations, which are considered equivalent, are presented in this procedure. However, wet combustion methods are not considered to be equivalent to the pyrolytic methods described.

In this method, inorganic carbon from carbonates and bicarbonates is removed by acid treatment. The organic compounds are decomposed by pyrolysis in the presence of oxygen or air. The carbon dioxide that is formed is determined by direct nondispersive infrared detection, flame ionization gas chromatography after catalytic conversion of the carbon dioxide to methane; thermal conductivity gas chromatography, differential thermal conductivity detection by sequential removal of water and carbon dioxide; or thermal conductivity detection following removal of water with magnesium perchlorate.

Water content is determined on a separate portion of sediment and data are reported in mg/kg on a dry weight basis.

2.0 DEFINITIONS

The following terms and acronyms are associated with this procedure:

| | |
|-----|------------------------|
| LRB | Laboratory record book |
| TOC | Total organic carbon |

3.0 PROCEDURE

3.1 Sample collection

Collect sediments in glass jars with lids lined with Teflon or aluminum foil. Cool samples and maintain at 4°C. Analyze samples within 14 days. If unrepresentative material is to be removed from the sample, it should be removed in the field under the supervision of the chief scientist and noted in the LRB on the field log sheet.

3.2 Apparatus and Reagents

- Drying oven maintained at 103° to 105°C.
- Analytical instrument. No specific TOC analyzer is recommended as superior. The following listing is for information on instrument options only, and is not intended to restrict the use of other unlisted instruments capable of analyzing TOC. The instrument to be used must meet the following specifications:
 - A combustion boat that is heated in a stream of oxygen or air in a resistance or induction-type furnace to completely convert organic substances to CO₂ and water.
 - A means to physically or by measurement technique to separate water and other interferants from CO₂.
 - A means to quantitatively determine CO₂ with adequate sensitivity (100 mg/kg), and precision (25% at the 95% confidence level as demonstrated by repetitive measurements of a well-mixed ocean sediment sample).
 - A strip chart or other permanent recording device to document the analysis.
- (1.) Perkin Elmer Model 240C Elemental Analyzer or equivalent. In this instrument, the sample from Section 3.5 is pyrolyzed under pure oxygen, water is removed by magnesium perchlorate and the carbon dioxide is removed by ascarite. The decrease in signal obtained by differential thermal conductivity detectors placed between the combustion gas stream before and after the ascarite tube is a measure of the organic carbon content.
- (2.) Carlo Erba Model 1106 CHN Analyzer, or equivalent. In this apparatus, the sample is pyrolyzed in an induction-type furnace, and the resultant carbon dioxide is chromatographically separated and analyzed by a differential thermal conductivity

detector.

- (3.) LECO Models WR12, WR112, or CR-12 carbon determinators, or Models 600 or 800 CHN analyzers. In the LECO WR-12, the sample is burned in high frequency induction furnace, and the carbon dioxide is selectively absorbed at room temperature in a molecular sieve. It is subsequently released by heating and is measured by a thermal conductivity detector. The WR-112 is an upgraded WR-12 employing microprocessor electronics and a printer to replace the electronic digital voltmeter.

In the LECO CR-12 carbon determinator, the sample is combusted in oxygen, moisture and dust are removed by appropriate traps, and the carbon dioxide is measured by a selective, solid state, infrared detector. The signal from the detector is then processed by a microprocessor and the carbon content is displayed on a digital readout and recorded on an integral printer.

In the LECO CHN-600 and CHN-800 elemental analyzers, the sample is burned under oxygen in a resistance furnace and the carbon dioxide is measured by a selective infrared detector.

- (4.) Dohrman Model DC85 Digital High Temperature TOC Analyzer. In this instrument, the sample is burned in resistance furnace under oxygen, the interfering gases are removed by a sparger/scrubber system, and the carbon dioxide is measured by a non-dispersive infrared detector and shown on a digital display in concentration units.

• Reagents

- (1.) Distilled water used in preparation of standards and for dilution of samples should be ultrapure to reduce the carbon concentration of the blank.
- (2.) Potassium hydrogen phthalate, stock solution, 1000 mg carbon/L: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 mL.

NOTE: Sodium oxalate and acetic acid are not recommended as stock solutions.

- (3.) Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

- (4.) Phosphoric acid solution, 1:1 by volume.

3.3 Interferences

- 3.3.1 Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.
- 3.3.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4 °C, analyzing within the specified holding time, and analyzing the wet sample.

3.4 Sample Preparation

- 3.4.1 Allow frozen samples to warm to room temperature. Homogenize each sample mechanically, incorporating any overlying water.
- 3.4.2 Weigh the well-mixed sample (up to 500 mg) into the combustion boat or cup. Add 1:1 phosphoric acid dropwise until effervescence stops. Heat to 75°C.

NOTE: This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample.

3.5 Sample Analysis

Analyze the residue according to the instrument manufacturer's instructions.

3.6 Percent Residue Determination

Determine percent residue on a separate sample aliquot as follows:

- 3.6.1 Heat a clean 25-mL beaker at 103° to 105°C for 1 h. Cool in a desiccator, weigh to

the nearest mg, and store in desiccator until use.

3.6.2 Add 1 g, weighed to the nearest mg, of an aliquot of the well-mixed sample .

3.6.3 Dry and heat in the 103° to 105°C oven for 1 h. Cool in a desiccator. Weigh to the nearest mg.

3.7 Calibration

- Follow instrument manufacturer's instructions for calibration. Prepare a calibration curve by plotting mg carbon vs. instrument response using four standards and a blank, covering the analytical range of interest.

3.8 Data Recording

Record all data and sample information in LRBs or on project-specific data forms.

All transfers of data to forms and data reductions (e.g., concentration calculations, means, standard deviations) should be checked by the analyst and approved by a lab manager, project manager, or principal investigator. Hard copies of sample data and spreadsheet reports should be kept in the testing laboratory's central files.

3.9 QA/QC Procedures

3.9.1 Precision and Accuracy The precision and accuracy will differ with the various instruments and matrices, and must be determined by the laboratories reporting data. A representative sample of well-mixed, meshed, sediment should be analyzed in quadruplicate for 4 days to determine the analytical precision.

3.9.2 It is critical that each sample be thoroughly homogenized in the laboratory before a subsample is taken for analysis. Laboratory homogenization should be conducted even if samples were homogenized in the field.

3.9.3 Dried samples should be cooled in a desiccator and held there until they are weighed. If a desiccator is not used, the sediment will accumulate ambient moisture and the sample weight will be overestimated. A color-indicating desiccant is recommended so that spent desiccant can be detected easily. Also, the seal on the desiccator should be checked periodically and, if necessary, the ground glass rims should be greased or the "O" rings replaced.

4.0 DATA REDUCTION, DOCUMENTATION, AND REPORTING

4.1 Data Reduction

Data analysis and calculations will be performed whenever possible on computers using commercial spreadsheet software such as Lotus 1-2-3, Quattro Pro, or Microsoft Excel.

4.2 Documentation

Keep all laboratory records, test results, measurements, other and supporting documentation for each sediment test in a LRB or project file dedicated to that purpose.

4.3 Reporting

A report should be prepared including, but not limited to, the following information:

- Sources of samples
- Description of methods
- Summary of sample analysis results
- Summary of any deviations from the project test plan
- Copies raw data, observations, or data forms

Total organic carbon should be reported as a percentage of the dry weight of the unacidified sample to the nearest 0.1 unit. The laboratory should report the results of all samples (including QC replicates, method blanks, and standard reference measurements) and should note any problems that may have influenced sample quality. The laboratory should also provide a summary of the calibration procedure and results (e.g., range covered, regression equation, coefficient of determination).

A.4

Source: U.S. Army Corps of Engineers - New York District and Environmental Protection Agency -Region II, 1992, "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal," Draft-18 Dec 1992.