

SOP 7 – Sediment Total Organic Carbon (TOC) Sampling and Analysis

Version 1.0 (August, 2007)

The following table lists all changes that have been made to this Standard Operating Procedure since the original publication date. Any recommended or required changes added to the log must be complete and concise and promptly brought to the attention of the Project Leader. The Project Leader will review and incorporate all changes, officially complete the revision history log, and change the date and version number on the title page. For complete instructions, please refer to Instructions for Recording Revisions in SOP 13.

Revision History Log:

New Version #	Previous Version #	Revision Date	Author (full name, title, affiliation)	Location in Document and Description of Change	Reason for Change

Introduction

This SOP covers the methods for collecting surface sediments from NER coastal park estuaries and analyzing them for total organic carbon (TOC). Sediment TOC is measured every fifth year of the monitoring program. The same probability-based sampling design is applied to TOC sampling as is used for spatial water-quality monitoring. This includes the requirement that all sampling be completed during a four-week index period during mid-to-late summer (July-August). For logistical simplicity, it is recommended that TOC sampling be incorporated into the fieldwork for water-quality monitoring. All aspects of this method including data quality objectives and quality control procedures follow the methods prescribed for the U.S. EPA National Coastal Assessment (NCA). It is highly recommended that NPS Vital Signs personnel familiarize themselves with the EPA NCA Field Operations Manual (U.S. EPA 2001a) and Quality Assurance Project Plan (U.S. EPA 2001b) as they relate to this SOP. Note that the above two EPA documents differ in their recommendations on sample storage. For Vital Signs monitoring, NPS is advised to follow the guidance from the latter EPA document and store and ship samples frozen at -20°C. Another important difference between this SOP and the NCA method is that the NCA protocol includes additional benthic measures and requires larger volumes of sediment. NPS sediment sampling for TOC requires only 100 ml of surface sediment (the top 2-3 cm) and may be accomplished with substantially lighter gear (as allowed by NCA).

Collection and Field Handling of Samples

Station locations

Every five years, NER estuarine monitoring shall include sediment sampling for TOC. Sediment samples are collected using the same probability based spatial design applied to water-quality monitoring. At each park, 30 stations are occupied during a four-week late summer index period, and these stations shall be the same ones used for water-quality monitoring for that sampling year. For logistical efficiency, it may be desirable to incorporate sediment sampling into water-quality survey cruises, but this is not required. Nor is it necessary to repeat the sampling sediments at trend stations during each week of the index period. Only one composite sample is required for each station during the index period.

Collection gear

The standard sediment grab specified for NCA collection is a 1/25 (0.04) m², stainless steel, Young-modified Van Veen Grab; however, other gear is also acceptable (U.S. EPA 2001a). Since NPS Vital Signs Monitoring requires a relatively small amount of sample (100 ml) from a relatively shallow depth (2 cm), alternative coring devices are likely to be more efficient. Whichever collection gear is used, it must be capable of gathering a sample that is 7 cm deep with an undisturbed surface layer. In order to collect the required volume of sediment within 3 cores, the device should have a diameter no less than 5 cm. In addition to the sediment sampler, this SOP requires the following.

- 500 ml vessel with lid for homogenizing composite samples
- spatula or scoop
- wide-mouth glass sample jars (250 ml)
- ice and insulated chest
- cm ruler

While the NCA protocol calls for stainless steel or Teflon sampling devices, the constraint is not necessary for this SOP since organic contaminants are not being measured. Similarly, the requirement to wash all equipment with Alconox between stations may be omitted. Coring devices and utensils should, however, be rinsed clean of any residual sediment between stations.

Sample collection

At each site, three sediment grabs will be collected and the surficial sediment layer (top 2 cm) will be collected by spatula or scoop. The surficial sediments from these three grabs will be combined in a clean vessel. Once three grabs have been added to the vessel, the composite sample shall be well blended to ensure a homogenous mixture. Approximately 100 ml of the composite sample shall be placed in each of two clean, pre-labeled glass jars and held on wet ice until back at the laboratory, at which point the samples shall be frozen at -20°C until further analysis. It is recommended for this SOP that jars with a capacity of at least 250 ml be used since full jars tend to break when frozen.

It is imperative that only “successful” core or grab samples be included in the composite. A successful sample is one having a level, intact sediment surface over the entire area of the grab, and a total depth of at least 7 cm. Grabs containing no sediments, or grabs with shelly substrates or grossly slumped surfaces are unacceptable. Grabs with washed-out surfaces, or ones

completely filled to the top (where it is not possible to confirm an intact sediment surface) are also unacceptable.

Storing, Shipping and Analysis of Samples

TOC in sediment samples is analyzed by the method described in U.S. EPA 1995. Samples are dried and then acidified to remove any inorganic (carbonate) sources of carbon. A sub-sample is then combusted in a TOC analyzer using infrared detection of carbon dioxide gas. It is acceptable for this analysis to be conducted in-house, but in most cases a contract laboratory will be used. In either case, it is expected that the analysis will follow the NCA method (U.S. EPA 1995) as well as the quality assurance procedures described in the NCA Quality Assurance Project Plan (U.S. EPA 2001b). TOC is run in batches of 20-25 samples, and QC samples included with each batch are as follows: method blank, at least one duplicated sample, and a sample certified marine reference material from the National research council of Canada's Marine Analytical Chemistry Standards Program (e.g. BCSS-1, MESS-2 or PACS-1). QC criteria and reporting requirements are provided in U.S. EPA 2001b.

Until analysis, samples should be stored frozen at -20 °C. At this temperature, they can be held for up to one year. In most cases samples will be shipped to a contract laboratory. Prior to shipping, confirm that the receiving laboratory has appropriate sample storage facilities and alert them to expect a shipment requiring immediate attention upon arrival. Express ship all samples on dry ice using overnight service following applicable federal, state and local shipping regulations for hazardous materials (dry ice). FedEx will currently ship samples overnight packaged with dry ice at no additional charge over their standard rates. If a Styrofoam insulated box is used, it must be encased in an outer cardboard container. Small insulated coolers (with rigid shell) are also acceptable. All shipments containing dry ice must carry an appropriate hazardous material label (provided by FedEx).

References

- U.S. EPA. 1995. Environmental Monitoring and Assessment Program (EMAP): Laboratory Methods Manual-Estuaries, Volume 1: Biological and Physical Analyses. U.S. Environmental Protection Agency, Office of Research and Development, Narragansett, RI EPA/620/R-95/008.
- U.S. EPA. 2001a. National Coastal Assessment: Field Operations Manual. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA620/R-01/003. 72 pp.
- U.S. EPA. 2001b. Environmental Monitoring and Assessment Program (EMAP): National Coastal Assessment Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA/620/R-01/002. 198 pp.