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| **Region 4**<br>**U.S. Environmental Protection Agency**<br>**Science and Ecosystem Support Division**<br>**Athens, Georgia** |
| OPERATING PROCEDURE |
| Title: **Sediment Oxygen Demand** |

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| **Effective Date:** November 1, 2007 | **Number:** SESDPROC-507-R1 |

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## Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Field Quality Manager.

| History | Effective Date |
|---|---|
| SESDPROC-507-R1, *Sediment Oxygen Demand*, replaces SESDPROC-507-R0.<br><br>**General**<br>Corrected any typographical, grammatical, and/or editorial errors.<br><br>**Title Page**<br>Changed title for Bill Cosgrove from Acting Chief to Chief.<br><br>**Table of Contents**<br>Revised with new sections added.<br><br>**Section 1.2**<br>Added SESD procedure.<br><br>**Section 1.3**<br>Updated information to reflect that procedure is located on the H: drive of the LAN. In addition, text has been revised in this section.<br><br>**Section 1.5**<br>Renamed Section. Revised to be alphabetical, and added references.<br><br>**Section 2**<br>Added SESD procedures.<br><br>**Section 2.1**<br>Deleted section heading.<br>B. Deleted sentence: Measurement of bottom velocities, if possible, may be useful for modeling purposes. | November 1, 2007 |
| SESDPROC-507-R0, Sediment Oxygen Demand, Original Issue | February 05, 2007 |

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## FIGURES

# 1 General Information

## 1.1 Purpose

The purpose of this procedure is to document both general and specific procedures, methods and considerations to be used and observed when conducting sediment oxygen demand (SOD) and nutrient exchange (NUTX) studies.

## 1.2 Scope/Application

This document describes specific methods to be used by field investigators when conducting SOD and NUTX measurements. On the occasion that field investigators determine any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain the desired data, the alternative procedure will be documented in the field log book, along with a description of the circumstances requiring its use. All documentation in the field logbook will follow SESD Operating Procedure SESDPROC-010, Logbooks.

## 1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the H: drive of the SESD local area network. The Field Quality Manager (FQM) is responsible for ensuring the most recent version of the procedure is placed on the H: drive and for maintaining records of review conducted prior to its issuance.

## 1.4 General Background and Considerations

Sediment oxygen demand measurements are typically conducted utilizing aluminum chambers approximately 2 feet in diameter (Figure 1). Typically, when conducting SODs from a boat, two replicates for water column respiration and four replicates for sediment respiration rates are employed. The boat must be on a secure 3 or 4 point anchorage to avoid pulling the chambers off the bottom during the course of the study. When conducting SODs in small streams, where equipment must be carried in and placed by hand, typically one water column respiration chamber and two sediment contact chambers are utilized.

Chambers are carefully placed on the bottom, sealed and a dissolved oxygen (DO) probe is placed inside the chamber. The DO concentration within the chamber is monitored and recorded every 5 to 15 minutes in order to document the rate of utilization of oxygen by the sediments. Twelve to twenty-four readings over a period of 1.5 to 2 hours are generally sufficient to develop a regression to determine the SOD rate of the sediments.

## 1.5 References

American Public Health Association (APHA), American Waterworks Association (AWWA), and the Water Environment Federation (WEF). 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition. Washington, D.C.

Murphy, Philip J. and Delbert B. Hicks. 1986. In-Situ Method for Measuring Sediment Oxygen Demand. U.S. Environmental Protection Agency, Athens, GA. pp. 307-322. *In* Kathryn J. Hatcher (ed.). Sediment Oxygen Demand, Processes, Modeling and Measurement. University of Georgia Institute of Natural Resources, Athens, GA.

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version.

SESD Operating Procedure for Field Sampling Quality Control, (SESDPROC-011), Most Recent Version.

SESD Operating Procedure for Field Specific Conductance Measurement, (SESDPROC-101), Most Recent Version.

SESD Operating Procedures for Field Turbidity Measurement, (SESDPROC-103), Most Recent Version.

SESD Operating Procedure for Measurement of Dissolved Oxygen, (SESDPROC-106), Most Recent Version.

SESD Operating Procedure for *in situ* Water Quality Monitoring, SESDPROC-111, Most Recent Version.

United States Environmental Protection Agency (USEPA). 2000. Diving Safety Manual. v 1.1. US Environmental Protection Agency, Washington, DC.

USEPA. 2002. Region 4 Ecological Assessment Standard Operating Procedures and Quality Assurance Manual. Science and Ecosystem Support Division, Region 4. Athens, GA.

USEPA. 2007. Analytical Branch Laboratory Operations and Quality Assurance Manual. Region 4, Science and Ecosystem Support Division, Athens, GA.

## 1.6 General Precautions

### *1.6.1 Safety*

Sediment oxygen demand measurements may be conducted in a variety of water bodies from small fresh-water streams to large estuarine systems. Due to the physical size of the measurement chambers, water depths need to be a least two feet to insure that the chambers are adequately submerged. In small wadeable streams, chambers may be carried out by two people and set in place. Great care must be taken not to disturb the sediment surface to be covered by the chambers during this process. Once the water depth becomes too deep to avoid submersion of the field investigator's face, then dive equipment must be utilized to set the chambers. Measurements of SOD may be conducted in water up to 50 feet deep or deeper if there is little current and adequate substrate for setting chambers. Typical depths are 15 to 30 feet deep. An appointed Environmental Protection Agency (EPA) divemaster will prepare a dive safety plan prior to the anticipated diving operation. All diving must be conducted in accordance with EPA's Diving Safety Manual vs. 1.1 (USEPA 2000).

Depending upon suspected or known contaminants in the water, adequate personal protection must be employed such as dry suits, full face AGA masks or Superlight diving helmets. Diver-to-diver and diver-to-surface communications should be employed if the divemaster on site deems it necessary.

In addition to contaminated water, other hazards exist that divers should be aware of and take appropriate safety precautions. These include, but are not limited to, biological hazards such as alligators, snakes, sharks and jellyfish. Other site hazards include strong currents, little or no visibility, hypothermia, heat stroke or heat exhaustion, and the possibility of entanglement.

Due to the size of chambers, the number of people required (typically 3 or 4), diving and other gear associated with collecting measurements, a large boat (typically 24 – 26 feet) is required. In large estuaries, wave heights of 2 feet are common and up to 4 feet are not uncommon. Therefore, an experienced operator capable of safely handling a vessel of this size in conditions that may be hazardous must be on board.

### *1.6.2 Procedural Precautions*

The following precautions should be considered when conducting SOD measurements or when collecting NUTX water samples.

- When filling the water column respiration (blank) chambers, care must be taken to fill the chambers with the same bottom water that is in the contact chambers without entraining suspended sediment into the chambers.
- Care must be taken to ensure that there are no sticks, rocks or other obstructions beneath the sealing lips of the contact chambers that would allow the entrance of ambient water from outside the chambers.
- When conducting SOD measurements, a description of the observed sediment type or characteristics should be included in the field log book.

## 2 Sediment Oxygen Demand Measurement

Four 64-liter opaque cylindrical sediment contact chambers with open bottoms (Figure 1) are deployed into the sediment and a DO probe is inserted into each chamber and the chamber is sealed off. The rate of decline of the DO within the chamber is then measured over a 1.5 to 2 hour period. In order to measure water column respiration, two completely sealed "blank" chambers (i.e. no sediment/water contact) are filled with ambient bottom water, a DO probe is inserted and the rate of DO decline is measured in these chambers. The water column respiration rate is then subtracted from the rates in the sediment contact chambers.

The rate of oxygen demand for the sediments is determined by utilizing a logging multiparameter sonde in each chamber. The sondes are programmed to log a DO reading every five minutes for approximately one to two hours. Data are backed up to a notebook computer approximately every 20 minutes. This not only insures against data loss, but also enables monitoring of any problems associated with the data collection such as a pump not running or a leaking seal on a chamber. If necessary, DO measurements may also be obtained using other approved DO meters (SESDPROC-106) with data recorded manually or stored in a logger.

Following is the procedure utilized for conducting field measurements of SOD using the previously described equipment.

A. Calibrate DO, and conductivity probes on the instruments, in accordance with SESD Operating Procedure for *in situ* Water Quality Monitoring, SESDPROC-111, and SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101. Conductivity must be calibrated in order for the sonde to determine salinity in estuarine environments because DO is salinity dependent. In soft substrates, it is useful to have turbidity measurements as well, in order to determine if sediments have been disturbed or suspended within the chambers. If turbidity is to be measured, follow SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103. Maintain a record of calibrations. If using the Clark Cell type DO probe, a stirring device is required in order to maintain appropriate water velocities across the membrane of the probe. Probes utilizing either the pulse technology or luminescent technology consume very little or no oxygen during measurements, therefore when using these types of DO probes, a stirrer is not required. A pump within the chamber is required in order to continually circulate water over the sediment within the chamber. The chamber pump maintains approximately a 0.1 feet/second velocity within the chamber.

B. Measure vertical profiles of DO, and temperature. Salinity should be measured in an estuarine environment to determine bottom salinities. If using a DO meter that does not also have a conductivity/salinity probe, the salinity measurement must be manually entered into the DO meter. Where nearbottom concentrations of DO are less than 2 milligrams per liter (mg/1), measurement of SOD rates must be done with care or the DO may be depleted within too short a period of time for effective assessment of SOD rates.

C. Check delivery of power and operation of the circulation pumps.

D. Deploy chambers. Gently lower the chambers with a line from the boat. If the SOD is being conducted in a stream or river that is too shallow for boat operation, then the chambers must be carried in from shore. If carrying in from shore, place the down stream chambers first and work upstream so as not to disturb the sediment where the chambers will be placed. Place the blank chambers last or in such a manner as to not introduce suspended sediment into the blank chambers during the purging process. If deploying from a boat, deploy the blank chambers first and purge with ambient bottom water. The bottom stoppers must be placed in the blank chambers prior to lowering to the bottom in order to avoid entraining sediment into the chambers. Purging of the blank chambers is accomplished by unplugging one of the circulating hoses and turning on the circulating pump. Deploy the remaining contact chambers while purging the blank chambers and filling with bottom water, again being careful not to disturb sediment which might be drawn into the blank chambers during the purging process.

E. Allow approximately 20 to 30 minutes for settlement of material that might have been suspended during deployment of the chambers and for the purging of the blank chambers. Install the monitoring probes and engage the circulation pumps. Terminate purging of the blank chambers when the monitoring probes are installed. A purging period of 20 minutes allows the chamber contents to turn over approximately three times.

F. Lower an ambient probe to chamber level; approximately one foot above the bottom.

G. Record initial monitoring data.

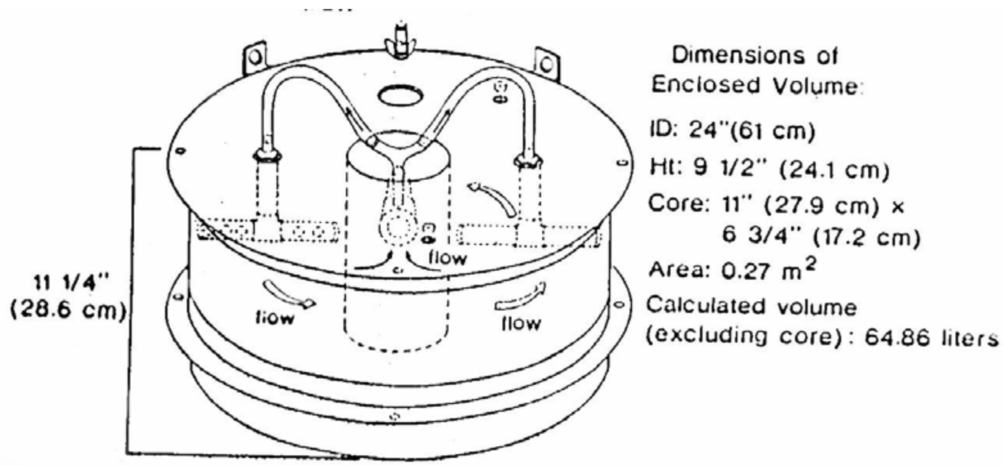
H. If desired, dark bottles filled with ambient bottom water using a pump or VanDorn sampler, may be deployed alongside the blank chambers for incubation during the course of the SOD experiments. Use water column respiration values obtained from the dark bottles as a back up to blank chamber experiments in case of chamber failure. General procedures for conducting the light-dark bottle tests are provided in Standard Methods (APHA *et al*., 1998).

I. Record monitoring data every 5 minutes if logging to a sonde or every 15 minutes if manually recording from a standard DO meter.

J. Continue the experiment for approximately 2 hours or longer, if necessary, when using a non-logging DO meter. Oxygen readings at 15-minute intervals generally provide sufficient data points for determining the slope of the DO curve. If a logging DO meter or sonde recording at 5 minute intervals is utilized, 1.5 hours is generally sufficient.

K. When sufficient data points have been recorded, remove monitoring probes from the chambers and retrieve equipment. Check meter calibrations and record in the log book.

**Figure 1: SOD Chamber**




(Notes: ID = inner diameter, Ht = height, " = inches, cm = centimeter, m = meter)

# 3 Nutrient Exchange (NUTX)

Water samples may also be collected from the SOD chambers in order to determine the rate of nutrient flux (or nutrient exchange) at the sediment water interface.

Through isolation of a known volume of water over a known area of sediment, nutrient flux can be measured by periodic extraction of water samples from each chamber and analytically determining the change of concentration of selected nutrients over an extended incubation period. Sampling of water from the chamber may occur only at the beginning and end of the deployment period. Nutrient Flux assessments are usually conducted in conjunction with SOD measurement. Accordingly, the deployment and DO monitoring techniques are essentially the same, with minor modification of the DO monitoring due to the extended deployment times associated with nutrient flux assessments. In a similar fashion to SOD, nutrient samples should also be collected from the blank chamber(s) to account for water column changes in the NUTX calculation.

## 3.1 Nutrient Exchange Equipment

Following is a description of the supplies and techniques utilized for collecting nutrient samples from the SOD chambers:

- Narrow-mouth glass, 500 milliliter (ml) bottles: Number of bottles required is based upon the number of stations, number of chambers, and the sampling regime (i.e., initial plus final plus any intermediate samples) as defined in a Quality Assurance Project Plan. Glass bottles, as opposed to plastic or Nalgene®, are essential since the underwater sampling procedure depends upon a siphoning effect for the water sample to replace the air within the sampling container. Plastic bottle walls will compensate (collapse) for the water pressure and, thus, will not fill properly.

- Sample collection siphon for extracting water sample from the chamber into the glass sample bottle: The siphoning device consists of a two-hole stopper sized properly to fit into the mouth of the glass sample bottle. The stopper is fitted with a stainless steel tube in each hole. One tube serves as the inlet for siphoning the sample into the bottle. The inlet tube should extend into the bottle almost to the bottom. The second tube is shorter and serves as an outlet to allow the air to escape from the bottle during sampling. This outlet tube is cut flush with the bottom of the stopper to ensure that all air is purged from the sample bottle. Each tube should extend approximately one inch above the top of the stopper.

Tygon® tubing, equipped with clamps, is attached to the outboard end of each tube. The Tygon® tubing attached to the stainless inlet tube should be approximately 15 to 18 inches in length. A one-hole stopper, containing a piece of stainless tubing is connected to the distal end of the tubing. The stopper is sized appropriately to fit the sampling port in the chamber lid. The portion of the stainless tube that is inserted into the chamber should be approximately 6 inches in length while the section extending from the top of the stopper outside the chamber need only to be approximately one inch in length in order to attach the Tygon® tubing.

- The suite of analytes for NUTX typically includes ammonia nitrogen, nitrate-nitrite nitrogen, total Kjeldahl nitrogen, total phosphorus, and total dissolved phosphorus. Nutrient samples are preserved with 10% sulfuric acid to a pH less than 2.

## 3.2 Nutrient Exchange Procedures

Deployment procedures for chambers used for (NUTX) measurements are the same as those outlined for SOD measurements. As stated previously, NUTX measurements can be initiated concurrently with SOD. However, incubation times (chamber deployment) must be considerably longer for NUTX than for SOD. Measurement of nutrient flux rates can be conducted under aerobic or anaerobic conditions. However, the strategy for such measurements must be developed prior to initiation of field activities and include an assessment of available oxygen resources immediately prior to deployment of chambers. This approach is required since accomplishment of NUTX measurements must be initiated and completed without transitioning from aerobic to anaerobic conditions, or vice versa. Such a change in the status of oxygen resources of the water column entrapped within the chamber will yield inconsistent results not reflective of ambient conditions. Accordingly, chamber incubation times (the time period from collection of the initial sample to the final sample) must be estimated by using the observed SOD rate in consideration of the chamber DO concentrations. Following is the sampling approach for collection of samples for determination of sediment nutrient flux rates.

Procedurally, water samples from each chamber are extracted as follows:

- Attach the siphoning device to a pre-cleaned sampling bottle and confirm that the tubing clamps are tight and the stopper is secure in the bottle mouth.

- The diver, or wader, inverts and submerges the bottle with siphon to chamber depth and attaches the siphon to the chamber by removing the solid (size 1) rubber stopper from the chamber lid and inserting the inlet tube stopper into the chamber lid. All tubing clamps remain closed. The bottle remains inverted in order to prevent the trapped air within the bottle from loosening the stopper and allowing ambient water to get into the bottle. If ambient water enters the sample bottle, it must be replaced with a clean bottle before collecting the sample.

- With the attached bottle held next to the chamber and inverted, the clamp on the inlet tube is released.

- Due to the pressure differential between the sample bottle and chamber, water will be forced into the bottle from the chamber until the pressure equalizes. The deeper the chamber, the greater the pressure differential and more water will be forced into the bottle. Once water quits flowing into the bottle from the chamber, move the bottle to an upright position on top of the chamber and release the clamp on the outlet tube. The bottle will still be positively buoyant so it must be held in place in order to prevent it from floating away. Care must be taken in order not to accidentally loosen or remove the inlet tube's stopper from the chamber lid. Because the air in the bottle is compressed due to the depth, it will begin flowing out of the outlet tube as soon as the clamp is released. As air flows out, water from the chamber will be siphoned into the bottle, eventually displacing all of the air.

- A second (size 1) stopper is positioned on the chamber lid across from the location where the inlet tube has been plugged into the chamber. This second stopper should be immediately removed when the siphoning begins to allow recruitment into the chamber of the sample volume removed during sampling. This is necessary because it is advisable to recruit from the water column rather than from sediment interstitial water.

- Observe or listen for air bubbles being purged from the sample bottle as it fills. When the bubbling ceases, the bottle is full. Secure clamps on both the exhaust and inlet port tubes, remove the siphon stopper from the chamber lid and replace the #1 rubber stoppers back into the sampling and recruitment ports.

- Return the sample to the surface and process according to procedures appropriate for sample handling and preservation based upon the target analyses (USEPA 2007).

- A final sample at the end of the incubation period is required for NUTX rate determinations. The final sample is acquired in the same manner as the initial sample. Again, it is important to state that the DO concentration within the chamber must be monitored, and recorded, to make sure that if the experiment within the chamber was initiated under aerobic conditions, then it must be completed under aerobic conditions. The same requirement applies to experiments conducted under anaerobic (anoxic) conditions.

- After the final sample is collected from each chamber, and pump circulation has been checked, the activity is complete and the chambers can be retrieved.