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Naval Command,
Control and Ocean
Surveillance Center RDT&E Division

San Diego, CA
92152-5001

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Technical Document 2387
February 1993

Benthic Flux Sampling Device

**Operations, Methods,
and Procedures**

D. B. Chadwick
S. D. Stanley

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**NAVAL COMMAND, CONTROL AND
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ADMINISTRATIVE INFORMATION

This document was prepared by members of Codes 522 and 571 of the RDT&E Division of the Naval Command, Control and Ocean Surveillance Center.

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INTRODUCTION

Sediments in many U.S. bays, harbors, and coastal waters are contaminated with potentially harmful metal and organic compounds. The United States Environmental Protection Agency (EPA) (1988) reports 134 toxic hot spots where in-place pollutants are a serious problem. A review of Navy hazardous waste sites (Johnston, et al., 1988) found 367 sites at 58 Navy and Marine Corps activities with significant potential for impact on aquatic environments. Historically, contamination has occurred directly through industrial discharge, chemical spills, improper disposal of shipyard and ship-board waste, and indirectly through urban runoff and groundwater exchange with land sites. These pollutants pose a threat directly to benthic organisms via pore water and particulate-bound contaminate exchange, and indirectly to aquatic organisms through leaching and resuspension.

Difficulties in assessing sediment contamination have led to a myriad of approaches to sediment quality assessment and criteria (Giesy & Hoke, 1990). Many of the disadvantages cited for various approaches relate to removal of the contaminated material to the laboratory for chemical and biological assays. These methods are costly in sample collection and analysis, and also represent an unrealistic departure from natural conditions. In many instances, identification of chemical contamination in sediments based on bulk concentrations has led to extensive assessment and remedial measures. However, the bulk concentration of a toxic substance in sediment is not always a good measure for predicting biological risk (Di Toro, 1989). Bioassay methods in which indicator species are exposed to sediment removed from the site to a laboratory environment may also represent an unrealistic departure from natural conditions. Neither of these techniques address the potential for sediments to act as a source to the water column through leaching of toxicants.

Previous studies indicate that biological uptake, accumulation, and toxicity result primarily from the fraction of the toxicant pool that is readily solubilized (Anderson & Morel, 1982). In surface sediments, the production of this soluble fraction will cause it to migrate through the pore water and across the sediment-water interface. For these reasons, benthic toxicant fluxes can provide a unique *in situ* measure of the source potential of contaminated sediments as well as an indication of bioavailability. In concert with traditional monitoring and assessment techniques, these flux measurements can lead to a better understanding of the environmental significance of historically contaminated sediments.

As part of the Navy's clean up program, the Installation Restoration (IR) Program, methods are evaluated to better assess suitable remediation and restoration strategies for sites that contain sediments contaminated with toxic compounds. Toward this goal, we have developed a Benthic Flux Sampling Device (BFSD) to quantify mobility of toxicants from contaminated sediments. The BFSD is a remote instrument for *in situ* measurement of toxicant flux rates from sediments. A flux out of or into the sediment is measured by isolating a volume of water above the sediment, drawing off samples from this volume over time, and analyzing these samples for increase or decrease in toxicant concentration. Increasing concentrations indicate that the toxicant is fluxing

out of the sediment. Decreasing concentrations indicate that the toxicant is fluxing into the sediment. Initial tests carried out in conjunction with Scripps Institution of Oceanography and the Environmental Protection Agency's Environmental Research Laboratory (Newport, OR) show that the system is capable of measuring a variety of contaminant and nutrient fluxes (Chadwick, et al., 1993).

BFSD SYSTEM DESCRIPTION

Figure 1 shows the prototype BFSD system developed for measurement of toxicant flux rates from sediment. The system consists of an open-bottomed chamber mounted in a tripod-shaped framework with associated sampling gear, sensors, control system, power supply, and deployment/retrieval equipment. The entire device is approximately 1.2 by 1.2 m from leg to leg. The lower part of the framework contains the chamber, sampling valves, sampling bottles, and batteries. The chamber is a bottomless box, approximately 40-cm square by 25-cm tall, and constructed of polycarbonate. The top of the chamber is hinged at one edge so it may be left open during deployment to minimize sediment disturbance. The bottom of the chamber forms a knife edge with a flange 5 cm above the base providing a positive seal between the box and the sediment. Samples are drawn off through a 4-mm Teflon tube to a manifold of valves and into air-filled Teflon sampling bottles (500 ml). Sampling is initiated by the control system that opens the valves at preprogrammed intervals. The bottles then fill by hydrostatic pressure while venting through a check valve mounted at the top of the frame. Sensors for monitoring conditions within the chamber including temperature, salinity, pH, and dissolved oxygen, are mounted on the chamber lid. A small pump maintains circulation in the flow-through system to the sensors and is also used to mix the chamber volume by a helical diffuser mounted vertically on the central axis of the box. The acquisition and control unit, the oxygen supply bottle, a video camera, and the retrieval line canister are mounted on the vertical members. The oxygen system is used to maintain oxic conditions within the chamber by diffusing oxygen at a constant rate through a coil of thin-walled Teflon tubing. The upper frame houses an acoustic release that is embedded in a syntactic foam retrieval buoy. The BFSD is designed for use in coastal and inland waters to depths of 50 m. Maximum deployment time is approximately 7 days based on available battery capacity. Chadwick and Stanley (1993) give a description of the BFSD and its components.

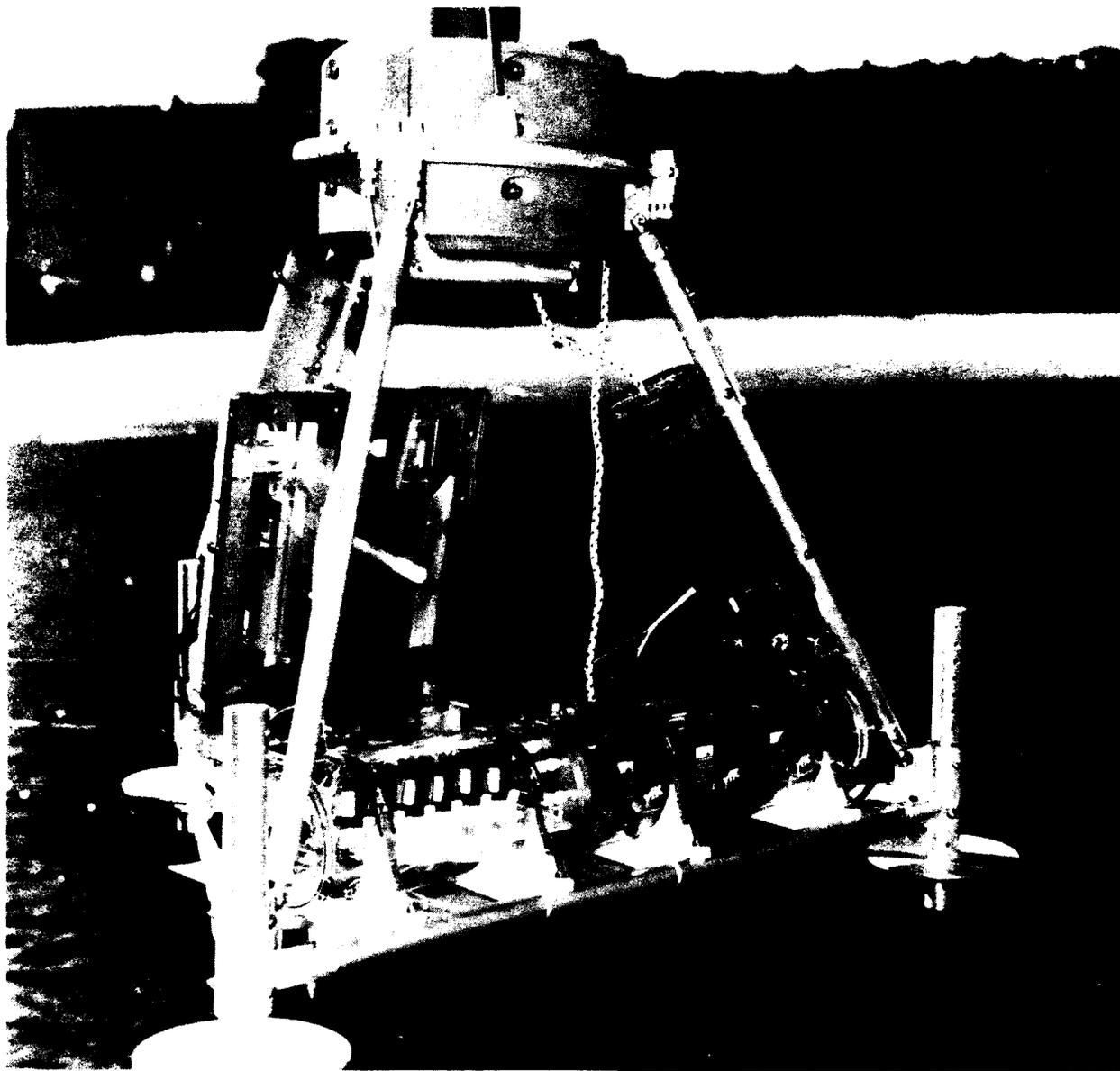


Figure 1. Benthic Flux Sampling Device (BFSD).

PREPARATION

This section gives step-by-step instructions for preparation for deployment of the BFSB.

CLEAN SAMPLE BOTTLES

Clean bottles, lids with plumbing, ferrules, and plugs. Methods of cleaning will differ depending on the analysis to be performed. For standard 500-ml Teflon bottles, the cleaning procedure is as follows: Begin with a deionized (D.I.) water rinse, wash and soak in RBS (a liquid detergent) overnight, soak in nitric acid (HNO_3) overnight, then rinse with D.I. water. When in doubt, consult with the chemist for proper cleaning protocols.

CHARGE BATTERIES

Remove the batteries from the pressure case (figure 2). Remove the connecting wires at the terminal strip on the end cap. The black wire is ground. Attach the battery charger (gel cell type, 12 V) and charge the batteries on the fast setting; move the setting to float for overnight. It should take approximately 1 day to recharge the batteries. However, they should be checked with the voltmeter to ensure that they are fully charged. The batteries should reach a potential of 12.3 to 12.6 volts when charged to full capacity. Replace batteries and then the end cap on the pressure case using silicon grease to ensure a watertight seal.

CHECK THE ACOUSTIC RELEASE

Remove the syntactic foam float (figure 3) by unbolting the six clamping bolts. Open the pressure case and unthread the bolts that secure the two stainless-steel bands. Remove the bands and the stainless-steel rod. The bolts attaching the release bracket to the endcap should not be removed to preserve the threads in the plastic material. Remove the vacuum plug next to the hydrophone, and the base by evenly loosening the three screws. Check the batteries. Set the code and the timer (usually disabled, see Endeco manual). Replace the base, bands, and stainless-steel rod.

PREPARE FLUSHING SOLUTIONS

The solutions used for cleaning the BFSB will differ according to the type of analysis being performed. The system will be flushed with methanol to remove organic compounds and with nitric acid to remove metals. The nitric acid must be diluted to a concentration of 1 N. This is approximately a 1:18 dilution by volume with 18-M Ω D.I. water. Conduct a final flush with approximately 20 l of 18-M Ω D.I. water.

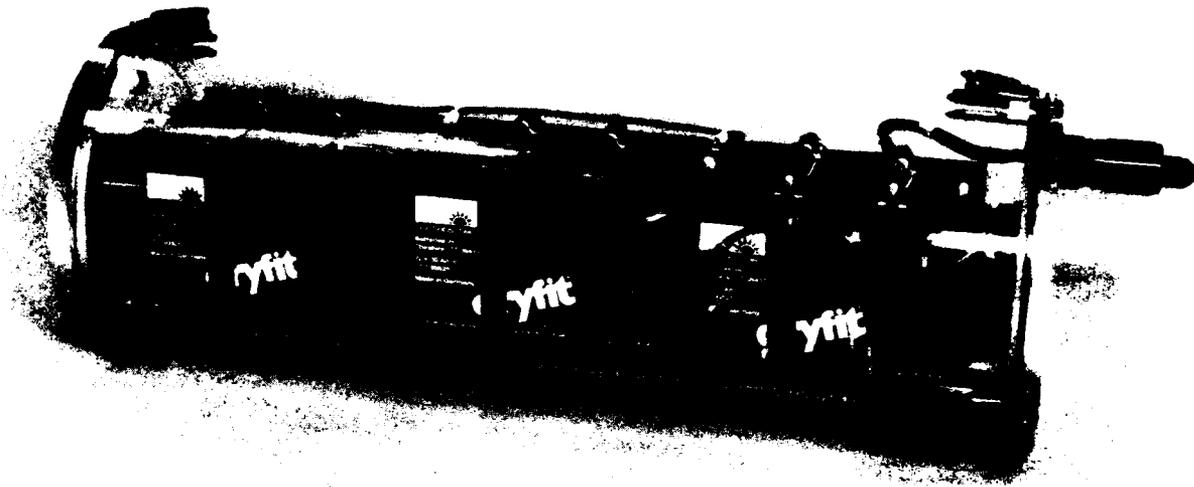


Figure 2. Battery pack pressure case.



Figure 3. Acoustic release float.

CLEAN PLUMBING ASSEMBLY

Remove the clear T-cap from the oxygen sensor and the W-shaped tubing from the chamber lid (figure 4). Remove the check valves from the frame and unthread the ferrules. Disassemble check valves by unscrewing the top portion, taking care to retrieve spring and valve with o-ring. Clean all parts with appropriate solution, a brush, and disposable cotton swabs. Rinse with D.I. water.

Reassemble the check valves. Apply a thin film of silicon grease to the o-ring to ensure a good seal. Place the o-ring part into the valve fitting with plastic tweezers. Replace the Teflon spring. Thread the top fitting until hand tight; secure with a 3/4 turn using a plastic wrench.

CLEAN PLUMBING LINES

Obtain the vacuum pump and two filter flasks. The two flasks will be placed between the pump and the lines to be cleaned. The flask closest to the pump should contain calcium sulfate to prevent liquid entry into the pump.

To clean the lines from the chamber to the valves to the sample bottles, begin by unthreading the ferrule at the fitting on the chamber wall (the line leading to the pressure case valves, figure 5). Attach the pump to the line from the chamber and place sample lines into a container holding the cleaning solution. Attach the laptop computer to the BFS computer. Use an external 12-V power source so the battery will not be drained unnecessarily. Close the chamber lid and begin the flushing program by typing *cd seasoft* <enter> followed by *bfcterm* <enter>:

- F1 read the experiment file *clean.exp*
- F2 check parameters; valves should be set to remain open for 3 minutes (vod=00:03:00)
- F3 download experiment to logger
- F4 program will prompt for status filename and experiment filename: *clean.sta* and *clean.exp*
- F5 set time and date to approximately 1 minute prior to start time from F2
- F6 terminal mode: type *st* to set time and date (F5), then type *gl* (go logger) to begin the program

Lift each line out of the solution for the last 20 seconds of its vod to remove liquid. To exit the program press the F10 key, then press F7 to download data into a dummy file. Turn off the laptop and disconnect the power supply.

To clean the vent lines from sample bottles to check valves, attach the pump to the top end of the line and place the opposite end into the cleaning solution. Flush the six lines for 2 minutes each, then draw water out of the lines using the vacuum pump. It is imperative that the sample lines be free of liquid to ensure proper sample collection during deployment. After cleaning, secure all tube ends in ziploc bags until final assembly to prevent contamination.

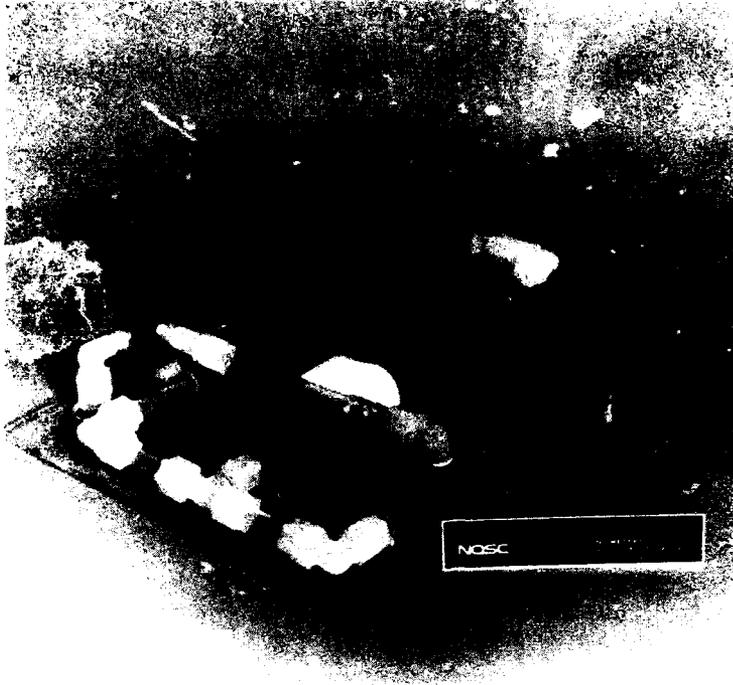


Figure 4. Flow-through sensor system.



Figure 5. Sampling system components.

To clean the flow-through system on the chamber lid, attach the pump to the temperature sensor and flush for 3 minutes. Then attach the pump to the conductivity cell (use extreme care, the end is glass) and flush for 3 minutes. Empty the filter flasks and store the pump.

REASSEMBLE CHECK VALVES AND LINES

Place the valve in the bracket and tighten the set screw until it is held firmly in place. Remove the ferrule from the valve and place it on the line past the groove. Apply silicon grease to the groove, push the line into the bottom of the valve and tighten the ferrule. Lightly tug on the line to feel the groove catch. Tighten 1/2 turn with a plastic wrench. Repeat for all lines.

REASSEMBLE FLOW-THROUGH SYSTEM

Remove the water-filled Tygon tubing from the conductivity sensor, and insert the tubing from the sensor into the tube in the center of the lid that drains into the chamber. Secure the Tygon connection with cable ties.

Remove the buffer bottle from the pH electrode taking care to retrieve the o-ring.

Replace the red cap from the oxygen sensor with the clear T-cap. Be sure that only the red cap is moving (do not unthread other parts of the sensor) and the o-ring is in place. Attach a small section of Tygon tubing from the conductivity sensor to the T-cap on the oxygen sensor and secure with cable ties.

Replace the W-shaped tubing; the short leg attaches to the temperature sensor, the center leg attaches to the pH electrode, and the long leg attaches to the T-cap on the oxygen sensor (figure 4). Secure all connections with cable ties and check to see that the pH sensor is protruding far enough into the flow.

Tighten the metal straps on the oxygen/pH sensor. Rubber strips should be placed between the metal and the sensor. Tighten the ferrule on the pH electrode.

ATTACH SAMPLE BOTTLES

Before leaving the lab (or dust-free environment) and using gloves to reduce contamination, attach the caps with plumbing to the bottles. Be sure o-rings are in place.

Loosen the nuts on the bottle holders of the BFSB and insert the bottles. Attach the lines from the pressure case valves to the elbow fitting; follow the same procedure as with the check valves. Attach the vent lines to the straight fitting. Take care not to kink the Teflon tubing.

INSTALL FLOAT

Secure float onto the top of the BFSB. Repack the retrieval line in the canister. Hold release bar in place while rotating locking cam to secure it. This is done by activating the reed switch on end cap of unit with magnet.

CHECK OXYGEN SYSTEM

Check all fittings to ensure they are tight. Check the bottle pressure with the high-pressure gauge and fill if the pressure is below 1000 psi. Check that the control valve is closed for start of the experiment. Open the vent valve and turn on the oxygen supply. If oxygen comes out of the vent then the control valve is open and must be closed by applying power to the tee end cap connector.

SAMPLE COLLECTION AND HANDLING

Sampling procedures have been developed for metals, polychlorinated biphenyls, (PCBs) and polynuclear aromatic hydrocarbons (PAHs). Additional samples are taken for silica as a performance indicator, and bulk sediment samples are taken to determine the condition of the sediment by traditional methods.

METAL FLUX SAMPLES

Time series water samples for metals analysis are collected in acid-washed, 500-m^l Teflon (TFE) sampling bottles aboard the BFSD. After each deployment, blank ferrules are fitted in place of the sampling lines and the samples are transported to the clean lab for processing. In the lab, samples are filtered and acidified immediately upon receipt. Initial (t_0) water samples are collected from outside the BFSD into precleaned 500-m^l polyethylene bottles using the Teflon pumping system aboard the survey vessel. These samples are refrigerated until the BFSD deployment is finished, and then processed in the lab with the corresponding time series samples. Split samples for silica analysis are taken from time series and t_0 samples prior to processing.

PAH/PCB FLUX SAMPLES

Time series water samples for PAH/PCB analysis are collected in precleaned, 500-m^l borosilicate glass sampling bottles aboard the BFSD. After each deployment, blank ferrules are fitted in place of the sampling lines and the samples are brought to the lab for initial processing (split samples and preservation). Initial (t_0) water samples are collected from outside the BFSD into precleaned 500-m^l glass bottles using the Teflon pumping system aboard the survey boat. These samples are handled in the same manner as the time series samples.

BULK SEDIMENT SAMPLES

Bulk sediment grab samples are acquired at the end of the deployment using a modified Van Veen grab. Sediment samples for metals analysis are transferred from the grab into precleaned, 500-m^l wide-mouth polyethylene jars using a precleaned plastic scoop. Samples for PAH/PCB analysis are collected using a precleaned, stainless-steel scoop into precleaned 500-m^l, wide-mouth glass jars. Samples are transported to the lab and stored frozen prior to analysis.

BFSD DEPLOYMENT PROCEDURES

This section gives instructions for deployment and retrieval of the BFSD. The experiment software is explained as well as sample retrieval, data retrieval, and BFSD preparation for storage.

DEPLOYMENT

Attach davit line with release shackle to the lift bail at the top of BFSD tripod frame. Lift BFSD on to boat (figure 6).

Connect all cables to the appropriate devices, and verify all systems are operating properly. Cables from the BFSD to the boat include

- Camera power supply
- Video input BNC
- RS232 connector for laptop computer
- Light power supply

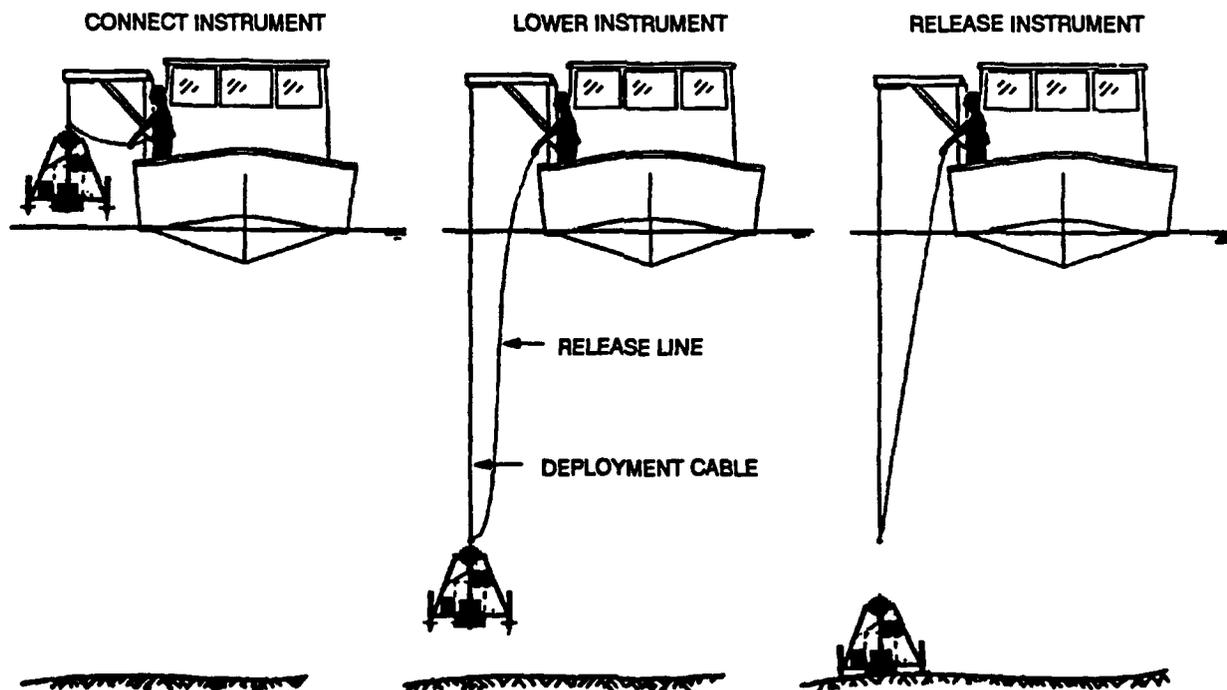


Figure 6. BFSD deployment.

Experiment Software

Close lid; solenoid will be activated when the experiment is downloaded.

At C:\ type *cd seasoft* <enter>

Run the experiment program by typing *bfcterm* <enter>. The following menu will be displayed:

F1:	read in experiment file
F2:	modify experiment variables
F3:	download experiment to logger
F4:	store experiment file and logger status
F5:	set logger date and time
F6:	terminal mode
F7:	upload logged data
F10:	exit to DOS

Select F1 to load an experiment file (____.exp). If one does not exist, select F2 and create one. When F2 is selected, or you have loaded an experiment file, the following menu will be displayed:

F1:	t_0 , time of lid closure	MM/DD/YY HH:MM:SS
F2:	lcd, lid closure duration	HH:MM:SS
F3:	sl, stirring latency	+--HH:MM:SS
F4:	sd, stirring duration	DD HH:MM:SS
F5:	vl, valve latency	+--HH:MM:SS
F6:	vod, valve open duration	HH:MM:SS
F7:	vsp, valve sample period	HH:MM:SS
F8:	dal, data acquisition latency	+--HH:MM:SS
F9:	dsp, data sample period	HH:MM:SS
Ctrl-F1:	oxygen sensor current M =	
Ctrl-F2:	oxygen sensor current B =	
Ctrl-F3:	number of oxygen samples to average	n
Ctrl-F4:	oxygen half window (m amps)	
F10:	return to main menu	

Modify desired variables by selecting the appropriate keys. See figure 7 for a pictorial representation of the acquisition timing sequence. See table 1 for most frequently used experimental parameters. Some notes on selected experimental variables follow:

- lcd corresponds to the length of time solenoid for the lid latch is pulsed.
- vsp corresponds to the amount of time from the beginning of one bottle sample and the beginning of the next sample (valve opening to valve opening).

- Ctrl-F1 through Ctrl-F4 variables are for the oxygen feedback loop. The software will not run without these variables. Oxygen sensor currents M and B can be found in the oxygen sensor calibration data (CTD Data Acquisition Manual). The oxygen half window sets the range of allowable oxygen concentrations in milliamps, and is equal to plus or minus the average of the first n (Ctrl-F3) samples.
- F3, F5, and F8 are values with reference to t_0 (F1), time of lid closure. For example, if F3 sl, is set to +00:10:00, the stirring will begin 10 minutes after lid closure.

Table 1. Frequently used experimental parameters.

Function key	Parameter	Description	Value
F1	t_0	time of lid closure	date/time
F2	lcd	lid closure duration	00:00:10
F3	sl	stirring latency	00:10:00
F4	sd	stirring duration	03 00:00:00
F5	vi	valve latency	00:20:00
F6	vod	valve open duration	00:40:00
F7	vsp	valve sample period	08:00:00
F8	dal	data acquisition latency	00:00:00
F9	dsp	data sample period	00:05:00
Ctrl-F1	M	oxygen sensor (current cal.)	
Ctrl-F2	B	oxygen sensor (current cal.)	
Ctrl-F3	n	number of oxygen samples to average	10
Ctrl-F4	m amps	oxygen half window	0.3

When variables are satisfactory, select F10 to return to the main menu.

Select F3 of the main menu to download the experiment to the logger.

Select F4. After the computer establishes communications with the logger, type a carriage return. You will be prompted for the filename for uploaded logger status. The name can be from one to eight characters from the following list:

Letters A through Z, upper or lowercase

Numerals 0 through 9

These symbols: !@#\$%^&()-_{}-'"

The extension on the status file should be *sta*. Next you will be prompted for the experiment file name. The extension for this file should be *exp*.

Select F5 to set the logger date and time. Usually 20 to 30 minutes prior to time of lid closure (F1 from loading experiment). This is approximately the time necessary to complete the rest of the experimental preparation and allow the sediment plume caused by the chamber upon landing to settle.

Select F6, and type a carriage return.

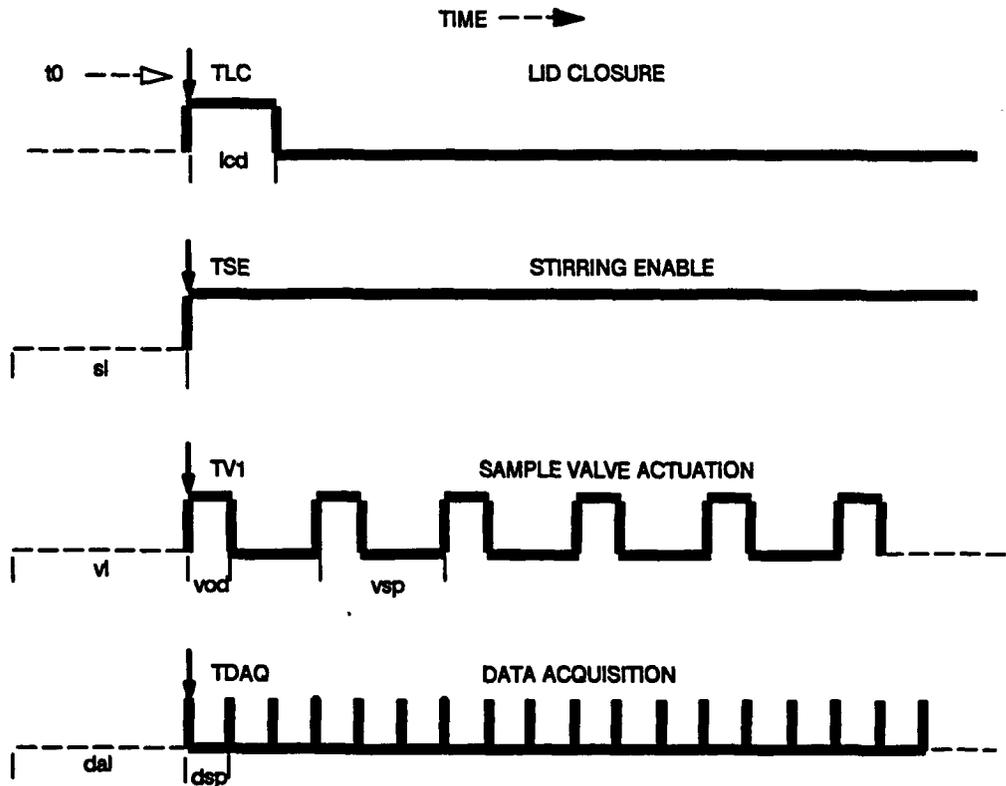


Figure 7. BFS control and acquisition timing sequence.

At the S> prompt type *st* (set time). You will be prompted for the date and time, these will be the same as the time set with the F5 command. If you have forgotten the F5 time, it is displayed at the middle of the terminal screen.

Once the logger time has been set, type *gl* (go logger) followed by a carriage return to begin the experiment. Answer the prompts to confirm the start (~Y means to press the CTRL and Y keys simultaneously).

Once the experiment has started, "W" will appear once per second until it is time for the first event. After the first event, "." will be displayed once per second along with the following codes to indicate the current status of the experiment:

s	sample data
a	all valves and lid off
b	lid on
c	valve 1 ON
d	valve 2 ON
e	valve 3 ON
f	valve 4 ON
g	valve 5 ON
h	valve 6 ON
A	pump ON
B	lid and pump ON
C	valve 1 and pump ON
D	valve 2 and pump ON
E	valve 3 and pump ON
F	valve 4 and pump ON
G	valve 5 and pump ON
H	valve 6 and pump ON

While the experiment is running, you may exit terminal mode by selecting F10. Do not press F3, F4, F5, or F7 while in the main menu or the experiment will be halted. The experiment will also be halted if power is removed from the logger, or the HOME or CTRL+Z keys are pressed. You may return to terminal mode to monitor the logger's progress by selecting F6 again.

Open lid and secure the lid latch on the frame of the BFS D.

Turn the oxygen tank to the OPEN position.

Attach the release line to the release shackle (end of davit line at BFS D lift bail).

Using the davit, lower the BFS D into the water until the chamber lid is just below the surface when in the open position. Remove the three ferule plugs in the plumbing and push water through at these points with a priming bulb to evacuate air trapped in the plumbing on the flow-through lid.

Continue lowering the BFS D until the bottom becomes visible with the camera equipment. Survey the area for an appropriate landing site free from rocks or other items that would hinder the operation.

Raise the BFS D until it is approximately 2 m off the floor. With the aid of the hydraulics, allow the BFS D to fall quickly (controlled free fall) until it rests on the ocean floor. The 2 meters of rapid fall will ensure a good seal with the sediment.

Once the BFS D lands, check with the camera (on the video screen) to make sure it is buried correctly. Check the fluorescent tape in the corner opposite the camera to

make sure it has gone far enough into the sediment and all sides are evenly buried. Also, check the angle on the inclinometer so that it is not too steep for the lid to fall.

Monitor the progress of the experiment on the laptop computer under terminal mode. Once the latch solenoid pulses, watch the video screen to make sure the lid closes.

After the experiment starts and the BFSD is secure, insert dummy plugs into the ends of the cable and throw it over making sure it does not land on top of the BFSD. Disconnect and retrieve the deployment wire by pulling on the shackle release line.

RETRIEVAL

Chamber Retrieval

When the experiment is complete, return to the approximate deployment location.

Connect the Endeco hydrophone to the transmitter deck unit and lower the hydrophone into the water below the boat's keel.

Turn the deck unit power ON. The pilot lamp should glow. Dial the CODE switch to the channel matching the target receiver. Depress the TRANSMIT button momentarily. The pilot lamp will flash and a tone sounds each time a pulse is transmitted. Coding is completed in 56 seconds. Look overboard for the buoy, and repeat pulse activations with the TRANSMIT button periodically until it is sighted on the surface.

Retrieve the float and pull the BFSD to the surface using the dacron retrieval line. With the top of the BFSD at the surface, connect the wire retrieval line to the stainless-steel retrieval bail and lift the unit onto the boat (figure 8).

After retrieving the BFSD, haul in the hydrophone and disconnect it from the transmitter unit. Turn the power OFF.

Turn the oxygen tank to closed and bleed oxygen tubing.

Sample Retrieval

After the BFSD has been retrieved, rinse the loose sediment off the chamber, inside the chamber itself, and around the sample bottles. Label all bottles while plumbing is still attached; a sequence number is taped on each bottle's corresponding line leading from the valve pressure case.

Obtain clean ferrule plugs. To avoid contamination, use gloves to unthread ferrules on each bottle and replace with plug immediately. You may want to wipe off the exterior of the bottle prior to detaching it from the lines. Place bottles in refrigerator or cooler.

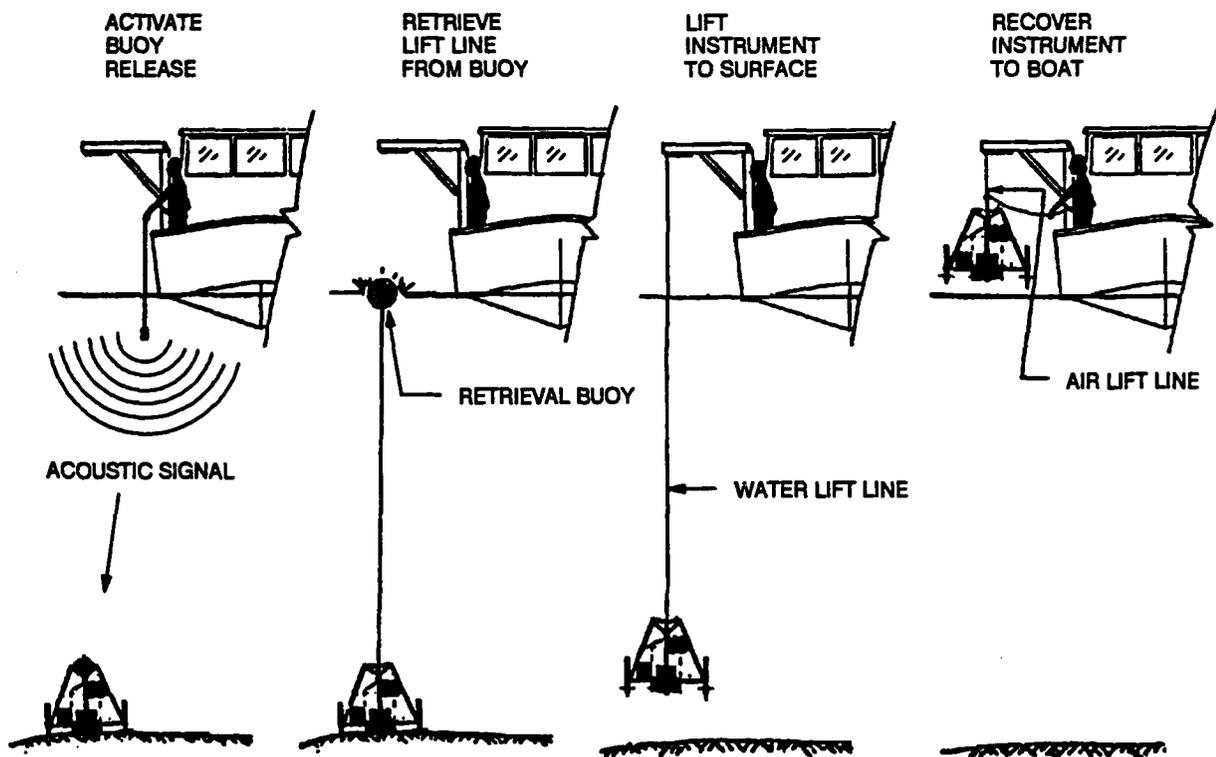


Figure 8. BFGD retrieval.

Data Retrieval

Connect the laptop computer to BFGD logger. At C:\ type `cd seasoft` <enter>. Return to the experiment program by typing `bfcterm` <enter>. The same menu will be displayed. Select F7 to upload the recorded HEX ASCII data. You will be prompted for the data file name which should be the same as the experimental file with a HEX extension (____.hex).

Before any manipulation of the data is attempted it is imperative that all .cfg files that will be used to manipulate the data, be updated or confirmed so they reflect the data sampling period (dsp) that was used in the experiment. We suggest that changes be made to `seasoft.cfg`, `oxy.cfg`, and `tps.cfg` to avoid confusion as to which .cfg will be used, and which will not.

To make these changes, run `seacon`, `seacon-coxy`, and `seacon-ctps`. Select item 1 to change instrument type or instrument configuration. Every prompt may be answered with a carriage return until you are asked for the sample interval in seconds. If you sampled once every minute this value would be 60. Before making changes, copy old .cfg files under an appropriate name.

When uploading of data is complete, select F10 to exit to DOS. The uploaded HEX data are converted to a .dat file by running `cnvhex`. Data may now be plotted as desired, see Sensor Data Review and Plotting. Disconnect laptop from BFGD logger and replace dummy plug.

PREPARATION FOR STORAGE

The BFSD should be thoroughly cleaned and dried before storage.

The flow-through system must be disassembled:

- Remove all plumbing that was attached during preparation.
- Attach a length of Tygon tubing to one end of the conductivity sensor and fill with 18-M Ω D.I. water. Secure the free end of the tubing to the other end of the conductivity sensor.
- Fill the red oxygen sensor cap with distilled water. Place o-ring on the oxygen sensor and screw cap into place.
- Fill the pH buffer bottle with buffer solution (pH 4, KCl sat.). Slide cap and followed by the o-ring over pH sensor. Screw the bottle into the cap, making sure that the pH electrode is completely submersed.

SAMPLE PROCESSING AND QA/QC PROCEDURES

SAMPLE PROCESSING

Trace Metal Samples

Once in the laboratory, the samples are immediately filtered through precleaned 0.45- μ cellulose nitrate membrane filter units and acidified to pH 2 with high-purity nitric acid. Constituent metals of interest are separated from the seawater matrix and concentrated by ammonium pyrrolidone dithiocarbonate chelation (APDC)/methyl isobutyl ketone (MIBK) extraction. The extracts are then analyzed by Graphite Furnace Atomic Absorption (GFAA) using the method of standard additions to develop a standard curve. Additional water samples including replicates and t_0 samples are analyzed in a similar fashion. Bulk sediment samples are acid digested by a standard microwave assisted digestion technique (EPA Method 3051). Following digestion, the digestate is analyzed by GFAA following the procedures described above for all elements of interest except As and Hg. Sample aliquots are digested separately and analyzed for As and Hg by a cold-vapor technique (EPA 7471A). A detailed description of these procedures may be found in the standard methods cited.

PAH/PCB Samples

Water samples from the BFSD are liquid-liquid extracted immediately after collection. Sediment samples from surficial sediments are extracted by sonication with acetonitrile and cleaned using C-18 solid-phase sorbent. The concentrations of selected PAH compounds and PCB congeners in these two matrices are then determined using high performance gas chromatography and mass spectrometry. Total PCB concentrations are then estimated from an Aroclor 1254 standard based on PCB congener 110. A detailed description of these protocols may be found in Young, et al., 1991.

Silica Samples

Silica samples are split into 50-ml plastic vials from the BFSD time series samples and t_0 samples following filtration and before acidification. Samples are refrigerated until analyzed. The analysis follows the standard colorimetric method for determination of reactive silicate in seawater (Strickland & Parsons, 1968).

QA/QC PROCEDURES

Trace Metal Samples

Method Blanks. Throughout the analyses, method blanks are employed as a means of verifying contamination-free preparation and reagents. Each batch of extracted and digested samples is accompanied by a blank that is analyzed in parallel with the rest of the samples, being carried through the entire preparation and analysis procedure.

Instrument Calibration. Instruments are calibrated at the start of each analytical batch. With water samples and extracted water samples, the method of standard

additions is used to generate each calibration curve. Successive dilution of a standard is used to generate standard curves for the analysis of the digestates. Initial calibration is verified by subsequent measurement of an independently prepared standard. The calibration is confirmed at regular intervals during an analytical batch.

Method Accuracy and Precision. Standard reference sediments are digested and analyzed periodically as a check on general method accuracy. Additionally, spiked replicates of field samples are processed with each analytical batch to validate method accuracy within the context of varying matrices. With water and extracted water samples that are analyzed by the method of standard additions, spiked samples are not used. Analytical precision and method detection limits are determined by replicate storage, preparation, and analysis of standard seawater. Further verification of precision is achieved by splitting one in twenty field samples. Typical results for selected metals based on these split samples are shown as percent relative standard deviation (%RSD) in table 2.

Table 2. Typical percent Relative Standard Deviation (%RSD) values.

Element	% RSD
Cu	21
Cd	10
Ni	6
Pb	4
Zn	10

PAH/PCB Samples

Accuracy of PAH/PCB Concentrations. For water samples, relatively clean representative matrix samples are spiked with target PAH compounds. Typical recovery efficiencies for the compounds are in the 90-percent range. Internal standards are also used as part of the GC/MS (Selected Ion Monitoring Gas Chromatography-Mass Spectrometry) procedures which automatically correct for recovery efficiency, to the first order. For sediment analyses, standard reference sediments containing known PCB and PAH concentrations are analyzed in duplicate, and are typically in agreement within 15 percent.

Precision of PAH/PCB Analyses. Duplicate procedural blanks are analyzed, as well as triplicate water samples. For sediments, replicate aliquots are taken from within one sample container. In addition, the precision of the GC/MS injection step is measured by periodically programming a sample to be injected three times to determine the %RSD values for its target compounds. Typical median %RSD values are approximately 15 percent for water and sediment.

A complete record of sample collection, processing, and QA/QC procedures for an actual deployment may be found in Chadwick, et al., 1993.

DATA ANALYSIS

CALCULATION OF FLUX RATES

Flux rates from BFSD time series samples are estimated using a linear regression model. Prior to running the regression, sample concentrations are corrected for dilution effects caused by the intake of outside water as sample water is removed from the chamber. The concentration of the diluting water is based on the t_0 sample. The corrected concentration is calculated from the equation,

$$[C_n] = [s_n] - \frac{v}{V} \left(\sum_{i=1}^n s_{n-i} + (n-1)[t_0] \right)$$

where $[C]$ is the corrected concentration, $[s]$ is the measured sample concentration, n is the sample number (1 through 6), v is the sample volume, and V is the chamber volume.

Some samples may be dropped from the regression based on performance criteria for dissolved silica and oxygen. A steady increase in the dissolved silica over the time of the experiment is an indicator of a "problem-free" deployment. Additionally, deployments are evaluated by reviewing oxygen data from the sensors onboard the BFSD. Since during deployment, oxygen is controlled within the chamber, anoxic conditions, or large oxygen fluctuations indicate possible problems with the experiment.

Following the regression of the time series concentrations, the flux is calculated from the equation,

$$FluxRate = \frac{mV}{A}$$

where m is the slope of regression, V is the chamber volume, and A is the chamber area.

An 80-percent confidence interval (80% CI) is then assigned to the flux rate based on a two-sided T-test ($t_{0.05(2),n-2}$), and the standard error of the regression coefficient. If the mean flux is positive and the lower limit of the 80% CI is greater than zero, then the flux is designated a *release rate* with magnitude of mean \pm 80% CI. Similarly, if the flux rate is negative with upper limit of the 80% CI less than zero, then the flux is designated an *uptake*. The 80% CI is chosen to be conservative in the sense that it does not eliminate potential release rates (as an indicator of environmental impact) unless confidence in them is quite low.

Typically, flux rates are calculated using a standard spreadsheet similar to the one shown in figure 9. A graphical representation for Zinc is presented as figure 10.

SENSOR DATA REVIEW AND PLOTTING

This section explains methods for reviewing and plotting the data obtained from the BFSD deployment.

Data review and plotting may be accomplished by running *binplot* or *seasave*. Both methods are outlined below.

Plotting Data Using *Binplot*

To run *binplot*, the program *binavg* must be run first to create a summary (*.sum*) file that will be referred to while establishing plot parameters.

Type *binavg* <enter>. You will be asked to select the bin type.

SELECT ONE OF THE FOLLOWING:

- 1 pressure bins
- 2 depth bins
- 3 scan number bins
- 4 time (seconds) bins
- 5 time (hours) bins

bin type = 5 new value =

For BFS data, select 5 for plotting ease.

bin size (hours) = 0.0

If you desire that the data be left in original form, and not averaged, make the bin size equal to the sampling period. If you sampled once every minute while collecting the data (F9 of the experiment file variable menu in *bfcterm*) and selected the bin type to be time (hours), number 5 in the above menu, then the appropriate bin size would be 0.01667 (1/60), which is 1 minute in units of hours. If you had selected the bin type to be time (seconds), the bin size would be 60.

Shelter Island Release Rate Study

Site: Shelter Island
 Date: 6/19-6/22/92
 Start time: 16:35:00 6/19/92
 Interval: 14:00:00
 End time: 16:15:00 6/22/92
 GPS lat: not recorded
 long: not recorded
 Depth: 4.6 m
 Lor lat: not recorded
 long: not recorded
 Depth: 4.6 m

lab data	BFSD data			outlier correct	dli. correction	from reg	Regressions		Flux rates
	[Conc] (ppb)	elapsed time	[Conc] (ppb)				[Conc] (ppb)	# of dilutions	
Copper						yes			
T061992-0	2.60	0.00	2.60	2.60	0.00	2.33	0.45	2.33	40.65 liters
T061992-b	2.13	14.00	2.13	2.13	1.00	2.17	0.36	2.17	1600.40 cm ²
T061992-a	2.88	28.00	2.88	2.88	2.00	2.01	6.00	2.01	Flux rate = -70 ug/m ² /day
T061992-f	1.72	42.00	1.72	1.72	3.00	1.85	4.00	1.85	80% CI (low) = -1.42 ug/m ² /day
T061992-l	1.58	56.00	1.58	1.58	4.00	1.69		1.69	(high) = 1 ug/m ² /day
T061992-c	1.99	70.00	1.99	1.85	5.00	1.53		1.53	
T061992-h	1.81		1.81	1.63					
Cadmium						yes			
T061992-0	0.072	0.00	0.072	0.072	0.00	0.055	0.08	0.055	40.65 liters
T061992-b	0.059	14.00	0.059	0.059	1.00	0.057	0.00	0.057	1600.40 cm ²
T061992-a	0.057	28.00	0.057	0.057	2.00	0.060	0.44	0.060	Flux rate = 1 ug/m ² /day
T061992-f	0.059	42.00	0.059	0.057	3.00	0.062	6.00	0.062	80% CI (low) = 0 ug/m ² /day
T061992-l	0.059	56.00	0.059	0.056	4.00	0.064	4.00	0.064	(high) = 2 ug/m ² /day
T061992-c	0.074	70.00	0.074	0.070	5.00	0.068		0.068	
T061992-h	0.070		0.070	0.065					
Nickel						yes			
T061992-0	0.37	0.00	0.37	0.37	0.00	0.59	0.59	0.59	40.65 liters
T061992-b	0.55	14.00	0.55	0.55	1.00	0.67	0.17	0.67	1600.40 cm ²
T061992-a	0.69	28.00	0.69	0.68	2.00	0.74	0.47	0.74	Flux rate = 33 ug/m ² /day
T061992-f	0.67	42.00	0.67	0.66	3.00	0.82	6.00	0.82	80% CI (low) = 4 ug/m ² /day
T061992-l	0.91	56.00	0.91	0.89	4.00	0.90	4.00	0.90	(high) = 62 ug/m ² /day
T061992-c	1.16	70.00	1.16	1.14	5.00	0.97		0.97	
T061992-h	0.79		0.79	0.76					
Zinc						yes			
T061992-0	7.67	0.00	7.67	7.67	0.00	7.04	7.04	7.04	40.65 liters
T061992-b	6.63	14.00	6.63	6.63	1.00	9.91	0.47	9.91	1600.40 cm ²
T061992-a	10.05	28.00	10.05	9.95	2.00	12.78	0.99	12.78	Flux rate = 1250 ug/m ² /day
T061992-f	13.68	42.00	13.68	13.47	3.00	15.65	6.00	15.65	80% CI (low) = 1175 ug/m ² /day
T061992-l	15.75	56.00	15.75	15.44	4.00	18.52	4.00	18.52	(high) = 1324 ug/m ² /day
T061992-c	19.17	70.00	19.17	18.75	5.00	21.39		21.39	
T061992-h	21.56		21.56	21.04					

Figure 9. Flux rate calculation spreadsheet.

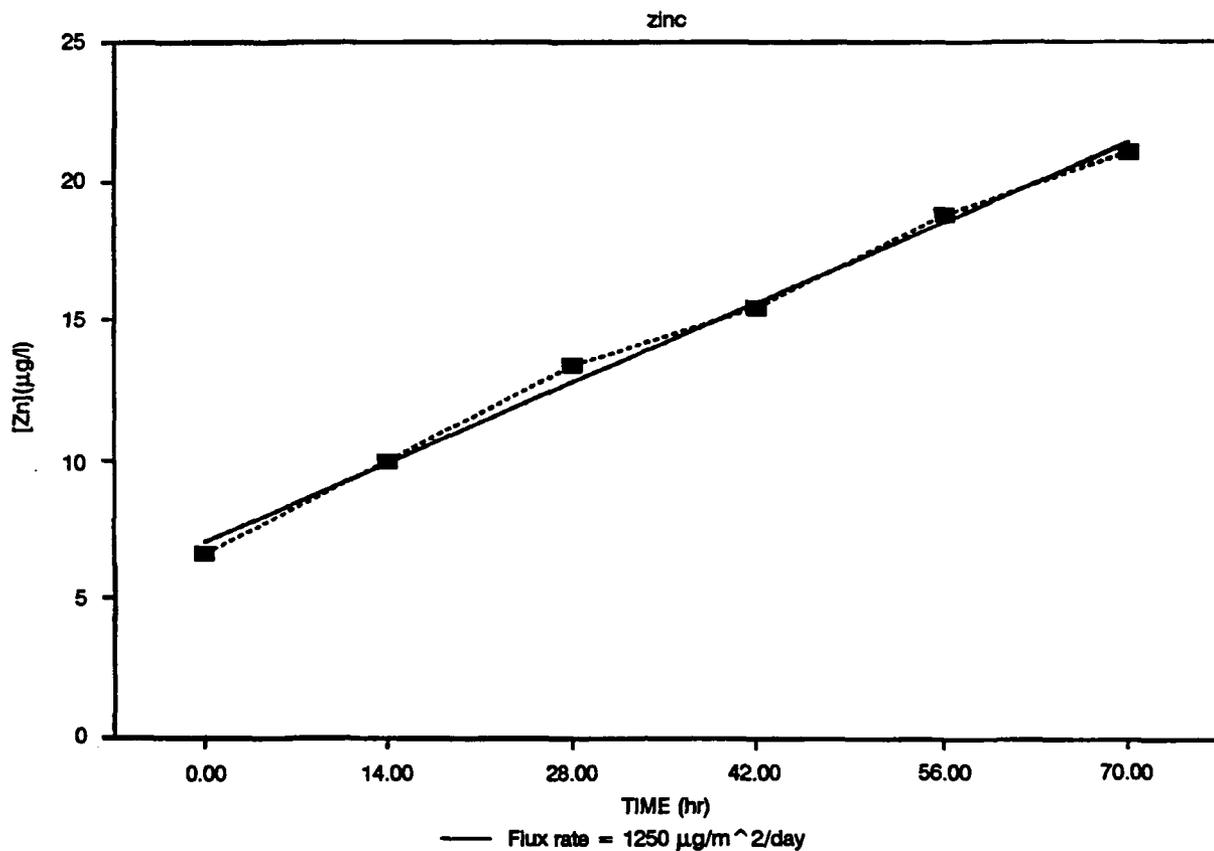


Figure 10. Flux rate data and regression.

include all scans or use increasing pressure filter (a/p)? =

-->include all scans new value =

you will want to include all scans

include number of scans per bin in last column (y/n)? = y new value =

answer y

change data output format (y/n)? = n new value =

answer y to check and/or change the format.

Here you will be given the following:

TABULAR DATA FORMAT:

column	variable
1	temperature (deg C)
2	pH
3	salinity (ppt)
4	oxygen current (microamps)
5	oxygen temperature (deg C)
6	oxygen (ml/l)
7	time (hours)

ok (y/n)? = yes new value =

If the list is not satisfactory, you may edit it by answering *n* to the prompt. For the BFS-D, the above list is correct. If a modification is necessary, follow the prompts and consult the CTD Data Acquisition Software Manual provided by Sea-Bird Electronics, Inc., if necessary.

Type a carriage return and files with the extension *dat* will be displayed. These are files that were created from raw _____*.hex* (the blank represents your experiment name) files earlier by running *cnvhex* (see Data Retrieval). You will be asked for the archived data file name. Type in your selection from this list.

You will then be prompted:

number of scans to skip over = 0 new value =
answer 0

process to end of file (y/n)? = y new value =
answer y

When the program is finished, it will ask if you would like to start again to convert other files for plotting. Answer accordingly. You are now ready to plot files.

Before running *binplot*, copy *tps.pp* to _____*.pp*. This will set up the plotting parameters of temperature, pH, and salinity for the first set of BFS-D plots, so that the only input needed by the program will be the maximum and minimum values. If the file *tps.pp* is not copied, the plotting parameters will need to be typed in each time a plot is required.

Type *binplot* <enter>. Select the appropriate data file. Note that the files are *.avg* files created by running *binavg*.

change plotting parameters (y/n)? = n new value =
answer y

plot data on HP plotter (y/n)? = n new value =
answer n unless a plotter is to be used

number of x axis variables (1, 2, or 3) = 3 new value =
select the number of variables, or side-by-side plots that you wish to be displayed

X-Y plot label = , new label =
type in the desired plot label

plot with connected lines or dots (c/d)? = connected new value =
answer as desired, dots take less time

The following display will then be given. This is the *.sum* (summary) file referred to earlier.

column	minval	maxval	variable
1			temperature (deg C)
2			pH
3			salinity (ppt)
4			oxygen current (microamps)
5			oxygen temperature (deg C)
6			oxygen (ml/l)
7			time (hours)

The minval and maxval columns will be filled with the minimum and maximum values of the data in the file you are attempting to plot. This will be displayed in the top half of your computer screen while various prompts regarding plotting will appear in the lower half. After all plotting ranges have been defined you will be prompted with the following:

number of scans to skip over = 0 new value =
answer 0

process to end of file (y/n)? = y new value =
answer y unless only a portion of the data is to be plotted

X-Y plot label = , new label =
type in the desired plot label

Plots similar to figure 11 will appear on the screen. If a printout is desired press CTRL and F9 simultaneously. To exit, press CTRL C.

To create the second set of BFSD plots, containing oxygen current, oxygen temperature, and oxygen, copy *oxy.pp* to _____.pp. Then run *binplot* as before.

Plotting Data Using *Seasave*

Data may also be plotted by running *seasave*. This program displays data in three format types, one of which is an X-Y plot. The advantage of running *binplot* to create X-Y plots instead of *seasave* is that *seasave* offers no display of maximum and minimum values to establish plotting ranges. Recall that in *binplot* the *.sum* (summary) file was displayed while establishing plotting parameters. However, *seasave* actually requires less steps. If you would like to refer to the *.sum* file while establishing plotting parameters, run *binavg* following the directions above. Then type the summary file out by typing *type _____ .sum*. You may use print screen to obtain a hard copy of this file for reference.

To run *seasave*, type *seasave -ctps <enter>* at the C:\SEASOFT prompt. The *-ctps* tells *seasave* to run the program using the *tps.cfg* file instead of the default *seasoft.cfg*. When the program is run with the *tps.cfg* file, the plotting parameters for temperature, pH, and salinity are set up automatically. You will only have to enter maximum and

minimum values from the *.sum* file. To obtain the second set of BFS-D plots, containing oxygen data, type *seasave -coxy*. You will then be prompted for the following:

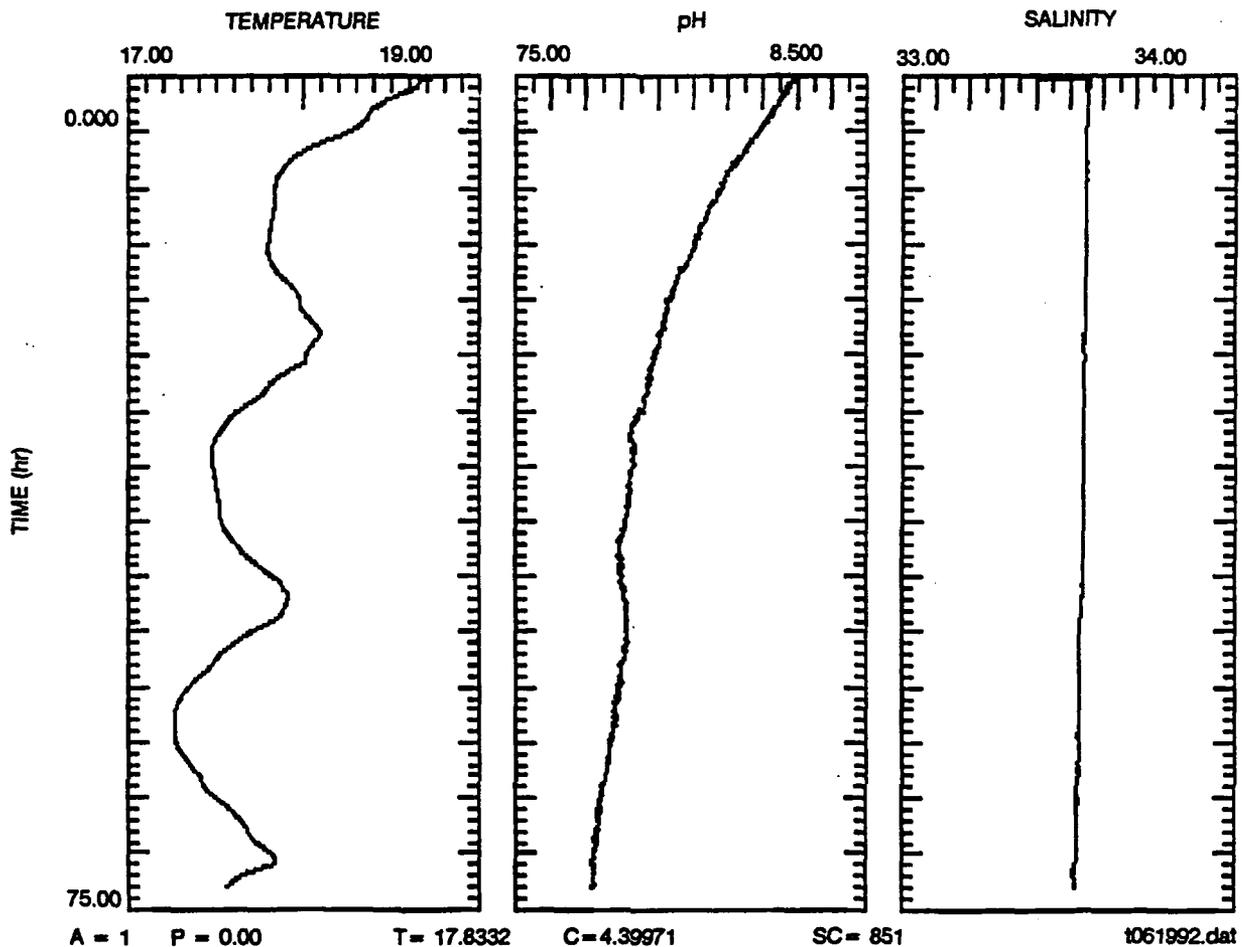


Figure 11. BFS-D sensor data.

change data acquisition or display parameters (y/n)? = n new value =
answer y for yes.

change CRT display parameters (y/n)? = n new value =
answer y again

Next you will be given the following choice of display types:

1. X-Y plots
2. fixed display
3. scrolled display

If the program is being run with *tps.cfg* or *oxy.cfg*, the default display will be X-Y plots, (1). The remaining prompts may be answered with a carriage return, unless changes are required. You will need to alter the maximum and minimum values while

plotting parameters are asked for. Please consult the CTD Data Acquisition Software Manual provided by Sea-Bird Electronics, Inc., if a detailed explanation of the prompts is desired.

To see a fixed display, choose the fixed display format (2) and follow prompts accordingly.

If the scrolled display format (3) is chosen an explanation of the display format will be listed as follows:

TABULAR DATA FORMAT

- | | |
|---|----------------------------|
| 1 | temperature (deg C) |
| 2 | salinity (ppt) |
| 3 | oxygen current (microamps) |
| 4 | oxygen temperature (deg C) |
| 5 | pH |
| 6 | oxygen (mℓ/ℓ) |
| 7 | time (hours) |

ok (y/n)? = yes new value =

If the list is not satisfactory, you may edit it by answering *n* to the prompt. For the BFS, the above list is correct. If a modification is necessary, answer the prompts accordingly and see the CTD Data Acquisition Software Manual provided by Sea-Bird Electronics, Inc., if necessary.

Answer the next three prompts with a carriage return unless changes are necessary.

display on HP plotter (y/n) ? = no new value =

realtime or archived data = archived new value =

number of points to skip between computations = 0 new value =

You will next be asked to select one of the following:

1. update *seasoft.cfg* and exit to DOS
2. exit to DOS, do not update *seasoft.cfg*
3. restart
4. update *seasoft.cfg* and continue

choice = 4, new choice =

Type a carriage return and files with the extension *dat* will be displayed. These are files that were created from raw *.hex* files earlier by running *cnvhex*. You will be asked for the archived data file name. Type in your selection from this list.

Depending on the length of data you would like displayed, answer the following prompts accordingly:

number of scans to skip over = 0 new value =

process to end of file (y/n) ? = yes new value =

data file start time 0 00:00:00.000 ok (y/n) ? = yes new value =

The data will begin scrolling. These values are generated by *seasave* by taking data from the *.dat* file and manipulating it into appropriate units using sensor calibration constants that have been entered into the *seasoft.cfg* file.

As a review:

After deployment and retrieval of data that has been saved as _____.*hex* (fill in the blank with your experiment file name):

TO PLOT USING *binplot*

- (1) run *cnvhex* to create _____.*dat*
- (2) run *binavg* to create _____.*sum*
- (3) copy *tps.pp* to _____.*pp*
- (4) run *binplot* to plot first set of plots
- (5) CTRL C to stop
- (6) copy *oxy.pp* to _____.*pp*.
- (7) run *binplot* to plot second set of plots

TO PLOT USING *seasave*

- (1) run *cnvhex* to create _____.*dat*
- (2) run *binavg* to create _____.*sum*
- (3) type _____.*sum* and print out
- (4) run *seasave -ctps* to plot first set of plots
- (5) CTRL C to stop
- (6) run *seasave -coxy* to plot second set of plots

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