

Naval Command,
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Surveillance Center RDT&E Division

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August 1993

Benthic Flux Sampling Device

Prototype Design,
Development, and Evaluation

D. B. Chadwick
S. D. Stanley

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**NAVAL COMMAND, CONTROL AND
OCEAN SURVEILLANCE CENTER
RDT&E DIVISION
San Diego, California 92152-5001**

**J. D. FONTANA, CAPT, USN
Commanding Officer**

**R. T. SHEARER
Executive Director**

ADMINISTRATIVE INFORMATION

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Released by
J. G. Grovhoug, Head
Marine Environment Branch
Division

Under authority of
P. F. Seligman, Head
Environmental Sciences

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The design, development, and testing of the BFSD was achieved in collaboration with Clare Reimers and Matt Christianson at Scripps Institution of Oceanography. Trace metal chemistry was performed by John Andrews and Lora Kear at our NRaD laboratory and by Eric Crecelius at Battelle Marine Sciences Laboratory; nutrients, alkalinity, and CO₂ were analyzed by Clare Reimers. Chuck Katz, Brad Davidson, and Andy Patterson of Computer Sciences Corporation assisted with preparation and deployment of the flux chamber.

EXECUTIVE SUMMARY

As part of the Navy's cleanup program under the Defense Environmental Restoration Act (DERA), we are conducting research to provide more effective assessment, remediation, and restoration strategies for sites that contain sediments contaminated with toxic compounds. Toward this goal, we have developed a remote instrument for *in situ* measurement of toxicant flux rates from contaminated 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 an increase or decrease in toxicant concentration. The device that performs this task is an autonomous sampling instrument, consisting of an open-bottomed chamber mounted in a tripod-shaped framework with associated sampling gear, sensors, control system, power supply, and deployment/retrieval equipment. It is used in coastal and inland waters to depths of 50 m, with a maximum deployment time of about 4 days, based on available battery capacity. The instrument is easily deployed from a small boat by lowering it to a position just above the sediment and then allowing it to free fall to the bottom. All sampling, data logging, and control functions are then carried out automatically, based on user-programmable experimental parameters. Upon completion, the system is retrieved using an acoustically triggered buoy that carries a line to the surface for lifting the device back on board.

Results from a series of test deployments indicate that the system can quantify flux rates of contaminants and other biogeochemical compounds at realistic levels for coastal and inshore sediments using a sample period of 2–4 days. The resulting flux rates will be useful in evaluating the risks posed by in-place sediment contamination from several aspects, including

- Source quantification for comparison to other sources and input to models.
- As an indicator of bioavailability, since many studies indicate that resolubilized contaminants are more readily available for uptake.
- Determining the cleansing rate of a contaminated sediment site due to natural biogeochemical cycling of the in-place contaminants.
- Providing a nonintrusive monitoring tool for sites capped or sealed to minimize biological exposure.
- As a scientific tool for a realistically testing and validating hypotheses and models for predicting the response of marine sediments to various contaminants.

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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 a significant potential for affecting aquatic environments. Historically, contamination has occurred directly through industrial discharge, chemical spills, improper disposal of shipyard and shipboard waste, and indirectly through urban runoff and ground-water 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 for submission to the laboratory for chemical and biological assays. These methods are very costly in terms of sample collection and analysis, and they 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 for submission to a laboratory environment may also represent an unrealistic departure from natural conditions. Neither of these techniques addresses 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 usually 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 cleanup program (the Installation Restoration (IR) program), methods are being 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 toxicant mobility 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 an increase or decrease in toxicant concentration. Increasing concentrations indicate that the toxicant is fluxing out of the sediment. Decreasing concentrations indicate a sediment uptake. 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 can measure a variety of contaminant and nutrient fluxes.

INSTRUMENTATION

DESIGN PARAMETERS

During the design of the Benthic Flux Sampling Device (BFSD), two major categories of design constraints were identified. The first category included all the requirements needed to perform the basic sampling operations. These requirements are similar to those of previous chamber designs (Berelson et al., 1987) and include the following:

- *Sediment Disturbance.* The BFSD and associated landing gear must be emplaced with minimum disturbance of the sediment surface.
- *Isolation of Chamber Volume.* The chamber design must provide an adequate seal at the sediment-water interface to isolate the chamber volume during the experiment.
- *Chamber Mixing.* The water inside the chamber must be mixed artificially to avoid its stagnation and stratification within the chamber and to ensure the samples collected represent the water at the interface.
- *Sampling Effects.* Chamber-induced effects, such as oxygen depletion, must be minimized to ensure that changes in one parameter do not affect the exchange rates of the target contaminants.

The second category included additional constraints defined by (1) the environment in which the chamber would be utilized and (2) the need to monitor pollutants as well as nutrients. The BFSD system was designed primarily for use in industrialized coastal bays and estuaries where sediment contamination is prevalent. These areas present significant operational challenges in addition to the known hardships imposed upon scientific instrumentation by the marine environment. Shipping traffic, strong currents, bottom debris, low visibility, and vandalism are all major hazards when deploying systems at such sites. We developed the following design parameters to address these issues:

- *Operation Time.* The device must be capable of continuous, unattended operation for a minimum of 72 hours. Based on previous measurements performed in laboratory settings (Hunt & Smith, 1983) and analytical detection limits for target contaminants, we estimated this period of time would suffice for detecting release rates at significant levels.
- *Operation Depth.* A depth capability of 50 m is sufficient to perform studies in most U.S. bays and estuaries.
- *Deployment and Recovery.* The system must be capable of deployment and recovery from a small craft using light-duty handling equipment. Operations must be done without diver assistance to minimize costs and scheduling constraints associated with diver emplacement and retrieval.
- *Autonomous Operation.* The device must operate in a completely autonomous (untethered) mode after it has been placed at the site. This is essential to minimize exposure to navigation hazards and vandalism.

- *Sample Size.* Samples collected by the system must have sufficient volume to facilitate analysis for trace levels of organic and metal compounds.
- *Construction.* All materials used in the system must be suitable for use and prolonged exposure in the marine environment. All materials that contact the sample water must be noncontaminating with respect to trace-level measurement of organic and metal compounds.
- *Environmental Considerations.* The system must be operational under a wide range of environmental conditions, and the device should be stable in bottom currents up to 5 knots. In poor visibility conditions, the system must be deployable without diver assistance. In addition, the system must provide an adequate seal and supportive footprint for sediments ranging from coarse sand to soft organic ooze.

BFSD PROTOTYPE SYSTEM DESCRIPTION

The BFSD prototype system shown in figure 1 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 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. Mounted on the vertical members are the acquisition and control unit, the oxygen supply bottle, a video camera, and the retrieval line canister. The upper frame houses an acoustic release 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 4 days, based on available battery capacity. Descriptions of the major system components follow. Appendix A contains construction drawings and details.

Chamber Enclosure

The chamber (figure 2) is a bottomless box approximately 40 cm square by 25 cm tall and isolates 32.7 l of seawater. As samples are drawn from this volume, bottom water is allowed to replace it via a length of 4-mm Teflon tubing. The volume was chosen to allow for a maximum overall dilution of 10 percent due to sampling withdrawal and subsequent replacement of six samples of 500 ml each. The chamber is constructed of clear polycarbonate to avoid disrupting any exchanges that may be biologically driven and, thus, light sensitive. To prevent stagnation in the corners of the chamber, triangular blocks of polycarbonate occupy the 90-degree angles.

Sediment disturbance must be minimized, since the surface sediment may be quite flocculent, and a bow wave propagating in front of the descending chamber could remove the most reactive material. To minimize such a disturbance, the lid of the chamber is hinged and left open during deployment. Once the chamber is in place, the computer control system closes the lid. A gasket around the perimeter of the chamber ensures a positive seal between the chamber and the lid. Exact alignment is not required, because the lid is slightly larger than the sealing perimeter of the gasket and pivots about two sets of hinges. The lid is held closed by rows of magnets placed along the chamber perimeter. The bottom of the chamber forms a knife edge; and a flange, circling it 5 cm above the base, provides a positive seal between the chamber and the sediment.

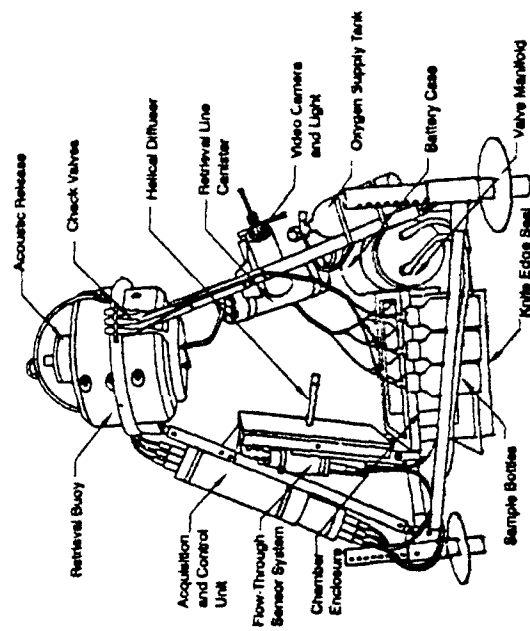
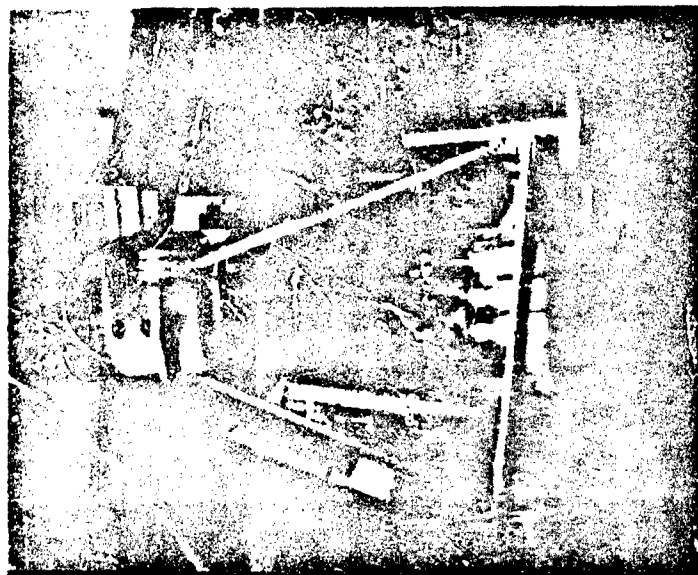


Figure 1. Benthic Flux Sampling Device (BFSD).

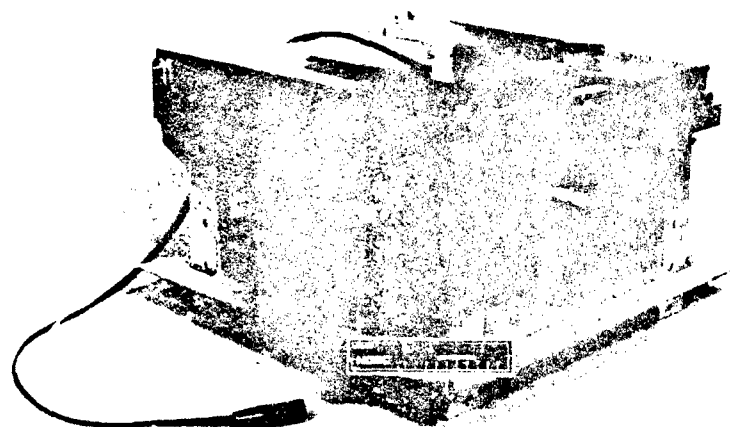


Figure 2. Chamber enclosure.

Acquisition and Control System

The acquisition and control unit (figure 3) is a Seabird Electronics, Model SBE-19 Seacat Profiler, modified to allow control of the BFSD. It consists of a data logger that acquires and stores data from sensors, and a control unit that regulates sampling and other functions of the BFSD. The data logger collects data from a suite of sensors mounted in a flow-through loop on the lid of the chamber; these data include temperature, oxygen, pH, and salinity. The control unit, an integrated part of the data logger, performs several functions; for example, it closes the lid, activates the flow-through/mixing pump, and opens the sampling valves.

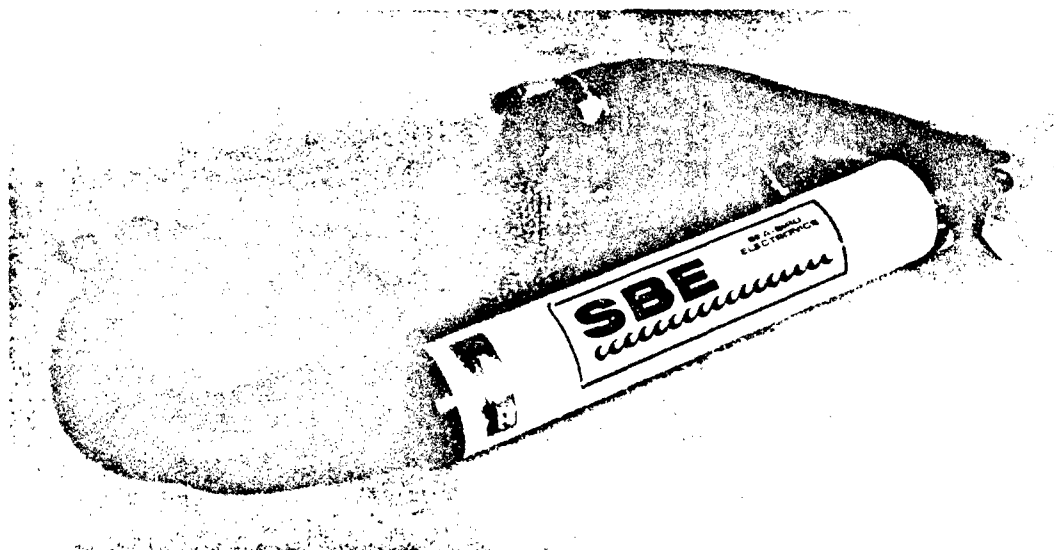


Figure 3. Acquisition and control unit.

Sampling System

The sampling system is shown schematically in figure 4. Discrete samples are obtained using a hydrostatic collection system consisting of sample containers, fill and vent lines, a check valve on the vent line, and a water-tight solenoid control valve on the fill line (figures 5 and 6). Off-the-shelf collection bottles are modified to allow filling and venting through the cap. Sampling containers of any volume, material, or shape may be used—provided the cap can be modified to accept the fill and vent line connection, the bottle walls are strong enough to withstand the sampling depth pressure, and the cap seal is watertight at the sampling depth pressure. Glass, Teflon, and polycarbonate bottles have been tested and used successfully with this system. All valves, fittings, and tubes are made of Teflon to minimize potential contamination of samples and to facilitate cleaning. Samples are drawn from the chamber through a 4-mm Teflon tube connected to a manifold of valves and into the air-filled sampling bottles. If necessary, prior to deployment, preservatives may be added to the sample containers. Sampling is initiated by the control system that opens the valves at preprogrammed intervals. Hydrostatic pressure then causes the bottles to fill while venting through check valves mounted at the top of the frame. The head difference between the chamber level and the vent level is sufficient to open the check valves. Once the bottle and vent line have filled, the head difference equalizes, the check valve closes, and the sample volume is sealed.

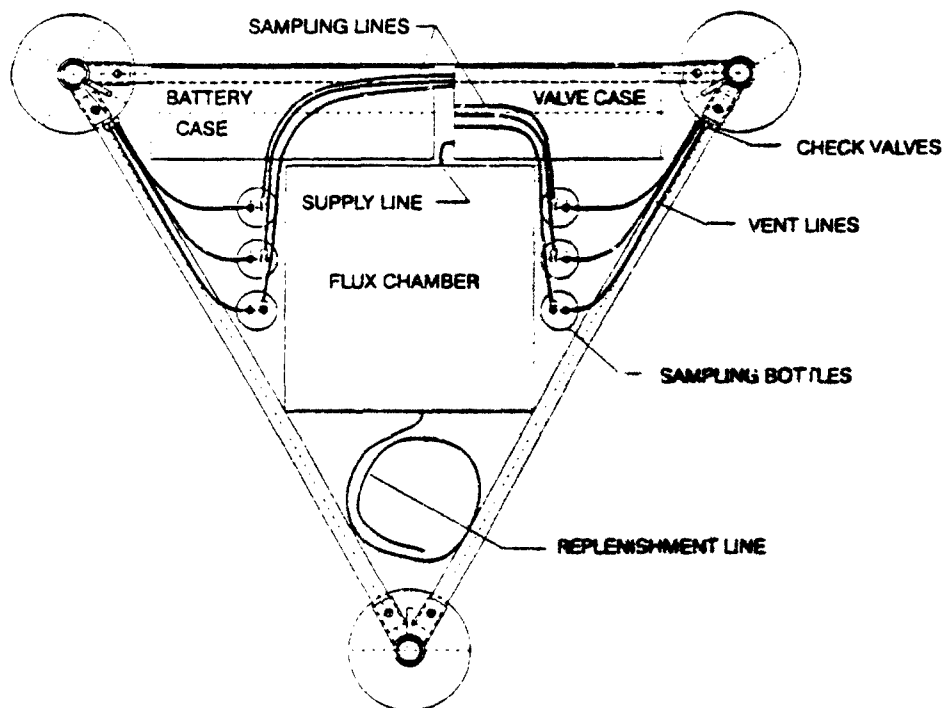


Figure 4. Sampling system schematic.

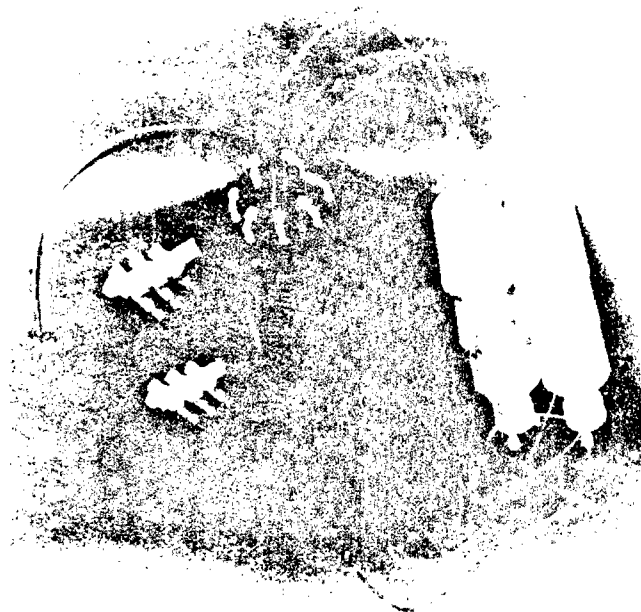


Figure 5. Sampling system components.

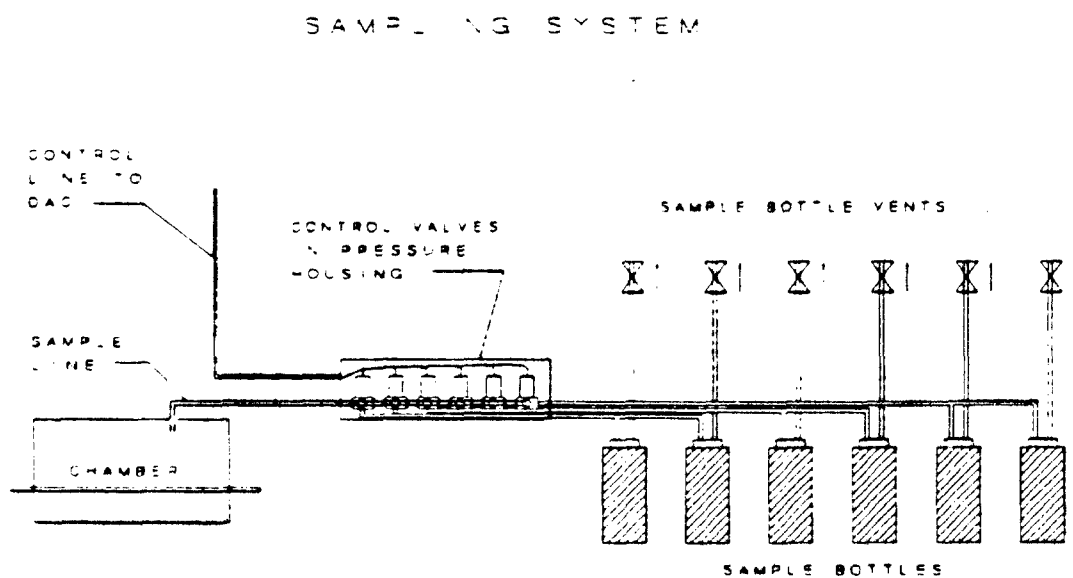


Figure 6. Hydrostatic collection system schematic.

Flow-Through Sensor System

Sensors, manufactured by Seabird Electronics, are mounted on the chamber lid (figure 7) and used for monitoring conditions within the chamber, including temperature, salinity, pH, and dissolved oxygen. The temperature sensor, Model SBE 3, is a pressure-protected, shock- and vibration-resistant, aged thermistor. Salinity is measured using a Model SBE-4 Conductivity Meter containing a two-terminal, three-electrode (platinum), flow-through sensing element. A Model SBE-18 pH Sensor measures the pH with a combination probe, using a pressure-balanced Teflon-junction Ag/Ag-Cl reference electrode. The Dissolved Oxygen Sensor, Model SBE 13, is a "Beckman" polarographic sensor that produces an oxygen-dependent electrical current. Circulation in the flow-through sensor system is maintained using a Model SBE-5 Submersible Pump with a flow rate of 90 mls/sec.

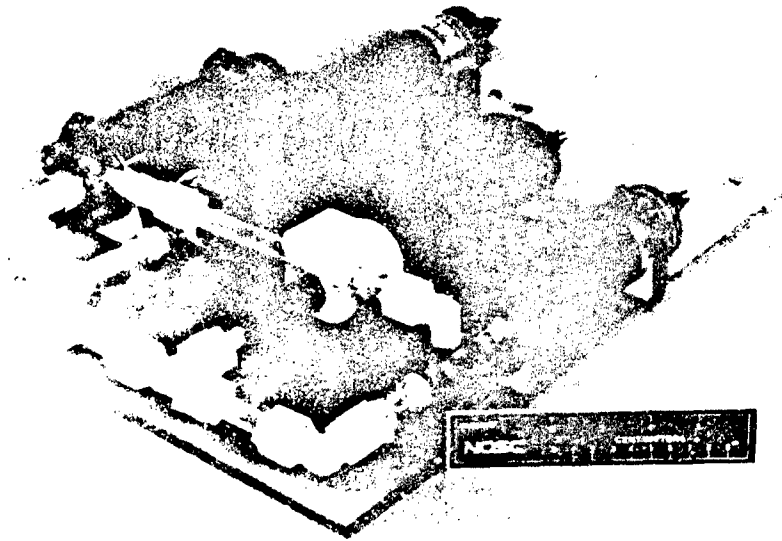


Figure 7. Flow-through sensor system.

Stirring System

In the experimental chamber, the process in question is the exchange of chemical toxicants at the sediment-water interface. The hydrodynamics inside the chamber must adequately simulate movement of water from near bottom currents outside the chamber. For this purpose, a helical diffuser mounted vertically on the central axis of the chamber is used to mix the enclosed volume.

The diffuser system was tested by constructing a mock chamber and performing a series of mixing experiments with varying geometries and flow rates. A stirring-bar configuration was also tested using two orthogonal 1/4-inch diameter glass rods rotating on a vertical shaft at the

center of the chamber. Based on these experiments, we found the diffuser provided a uniform, gentle mixing action that effectively dispersed dye injected into the chamber—without disturbing the sediment layer on the chamber bottom. At rotation rates sufficient to provide adequate mixing, the stirring bar system tended to suspend light sediments from the chamber bottom.

The final diffuser system consisted of a standard Model SBE-5 Submersible Pump outfitted with a custom polycarbonate head to minimize potential contamination (see Appendix A). The pump circulates water from an inlet on the lid of the chamber, over the flow-through sensors, and back into the chamber via a rigid Teflon pipe 0.25 m long. The vertically mounted pipe is capped at the discharge end and has eight 5-mm holes drilled in a helix pattern along its length. The tests showed that within 30 to 60 seconds, this method visually dispersed a dye injection of Rhodamine WT.

Oxygen Control System

The oxygen regulating system consists of a supply tank, pressure regulator, control valve, diffusion coil, and oxygen sensor, and control hardware and software (figure 8). The supply tank is a 13-cubic foot aluminum diving tank equipped with a first-stage regulator that allows adjustment of output pressure to the system. The control valve is a 12-volt, latching-solenoid valve housed within a watertight pressure case with connections through bulkhead fittings on the end cap. The diffusion coil is thin-walled, 4-mm, oxygen-permeable, Teflon tubing approximately 15 m long. Oxygen is monitored using the oxygen sensor in the flow-through system described previously. The oxygen control system is incorporated into the control system of the BFSD.

During an experiment, when the flux chamber is initially deployed, the ambient oxygen level is recorded by averaging a user-specified number of samples from the oxygen sensor. The control system then establishes maximum and minimum allowable oxygen levels based on a user-specified range about the average. Once the chamber is sealed, the oxygen level inside the chamber is monitored continuously. If the level drops below the allowable minimum, the control valve is opened, the diffusion coil is pressurized, and the oxygen level in the chamber begins to increase. When the oxygen level reaches the maximum allowable level, the control valve is closed. This sequence is repeated continuously during the deployment, maintaining the oxygen level in the chamber closely to that of the ambient level.

Deployment and Retrieval Systems

During deployment and prior to landing, the test site is surveyed for obstacles. This is done using a SeaCam-2000 video camera and SeaLite, manufactured by DeepSea Power & Light, on board the BFSD. The BFSD is also equipped with an Endeco Type-900 Acoustic Release, encased in a retrieval buoy (figure 9). Upon completion of the experiment, the release is triggered by the Deck Command Unit, the retrieval buoy surfaces, and the BFSD is recovered.

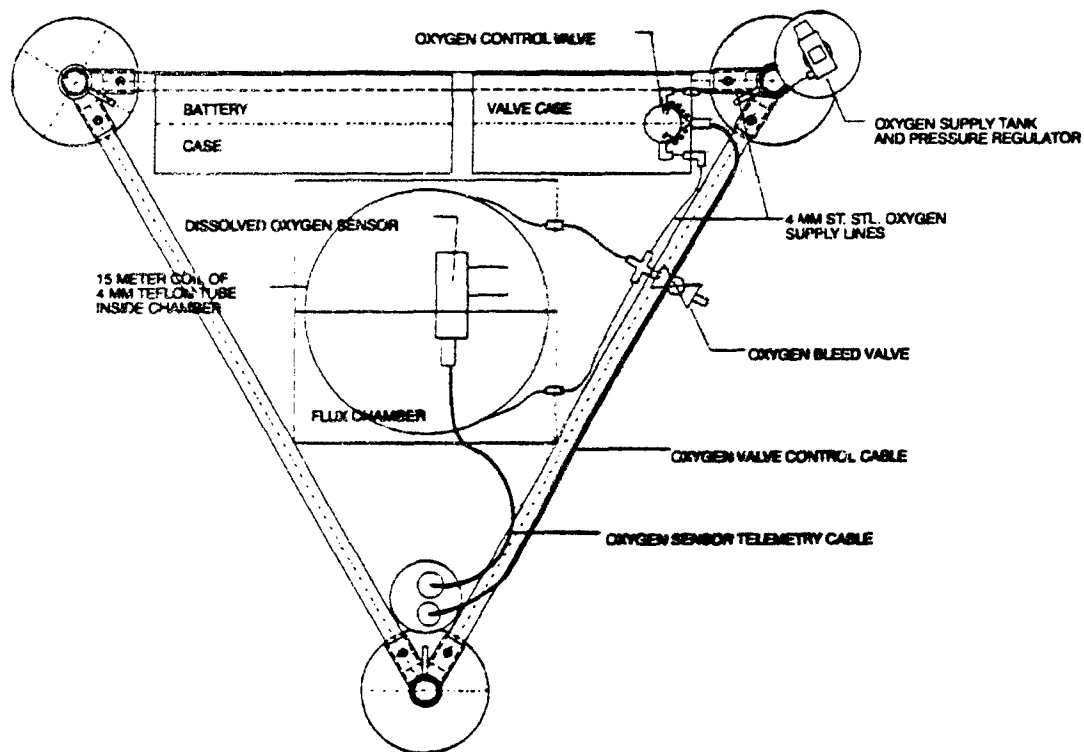


Figure 8. Oxygen control system.

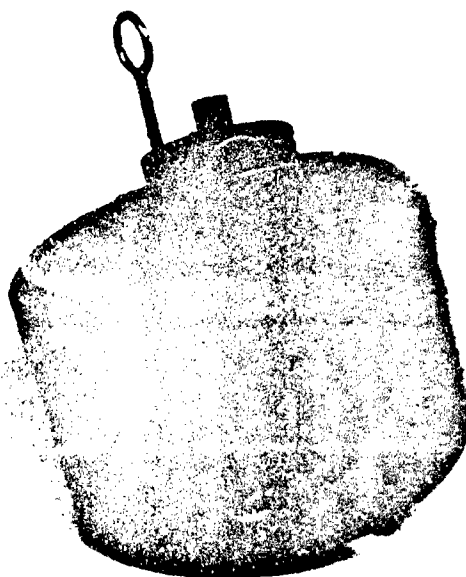


Figure 9. Acoustic release and retrieval buoy.

METHODS

This chapter briefly describes the methods for conducting experiments with the Benthic Flux Sampling Device (BFSD). Detailed procedures for BFSD preparation, deployment, sample collection, and sensor-data collection are described by Chadwick and Stanley, 1993.

PREPARATION

Prior to deployment, several steps must be performed to prepare the BFSD for operation and to ensure the integrity of the samples the device will collect. The entire system, including the flow-through sensor system, all plumbing lines, and sample bottles, must be cleaned with solutions appropriate for the analyses to be performed on the collected samples. Batteries must be charged, and the acoustic release and oxygen systems must be checked to ensure successful deployment, experimentation, and retrieval.

DEPLOYMENT

Once all the cables have been connected, and proper systems operation is verified, the experiment software is run to set up the actual sampling intervals and to input other information needed for experimentation (Chadwick & Stanley, 1993). Final adjustments are completed, such as opening the lid of the chamber and the oxygen supply tank, and the BFSD is lowered into the water (figure 10). When the sea floor becomes visible, the video camera on board the BFSD is

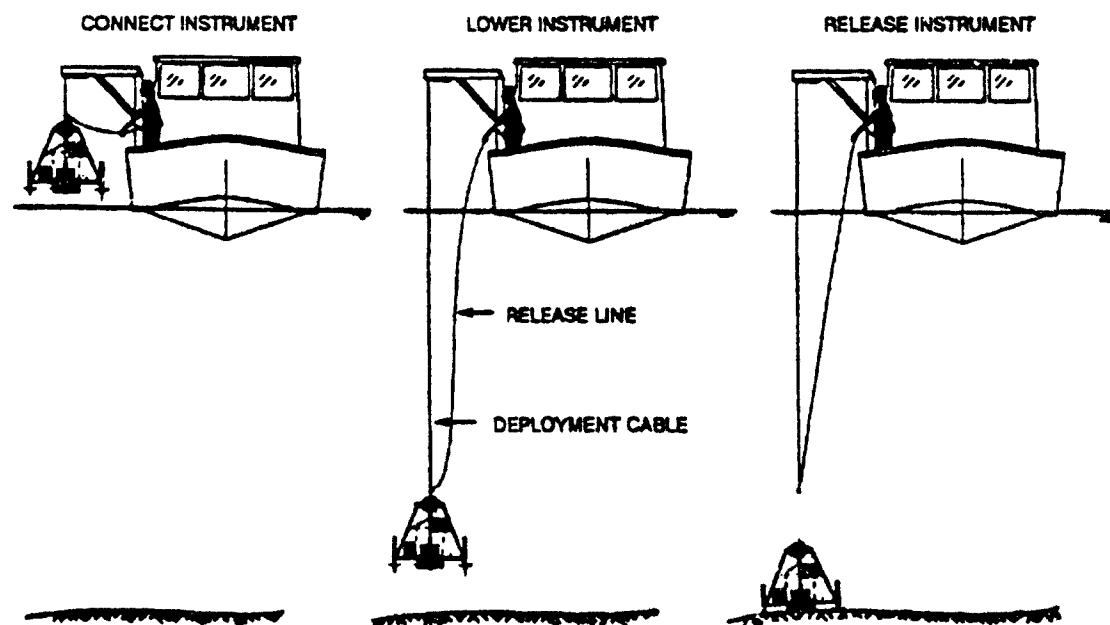


Figure 10. BFSD deployment.

used to survey the area for an appropriate landing site. Once a clear site is established, the instrument is raised 2 to 5 m above the sediment and allowed to free fall to the bottom. The weight of the system and its downward momentum bury the knife-edge seal of the sampling chamber into the sediment. To assure the system is functioning correctly, the initial functions, such as lid closure and pumping, are monitored by the computer and video camera aboard the deployment vessel. The cables are then detached, plugged, and thrown overboard for the remainder of the experiment.

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.

During deployment, time-series water samples are collected by the BFSD at preprogrammed time intervals. Initial (t_0) water samples are taken from outside the BFSD using the Teflon pumping system aboard the survey vessel. Samples for metals analysis are collected in acid-washed, 500-ml Teflon (TFE) sampling bottles, while precleaned borosilicate glass sampling bottles are used for collecting PAH/PCB samples. These water samples are then refrigerated until they are analyzed. Prior to processing, split samples for silica analysis are taken from time-series and t_0 samples.

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

RETRIEVAL

After returning to the approximate deployment location, a hydrophone is lowered into the water, and the acoustic release is triggered by the Deck Command Unit. After 56 seconds, coding is complete, and the buoy should appear on the water's surface (figure 11). The BFSD is then retrieved, and sample bottles are labeled before being transferred from their holders to a refrigerator or cooler. Sensor data is retrieved by reconnecting the cables and again running the experiment software. The sensors on board the BFSD are prepared for storage, and the frame is thoroughly cleaned and dried.

SAMPLE PROCESSING AND ANALYSIS

Trace Metal Samples

In the lab, 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 APDC chelation/MIBK extraction. The extracts are then analyzed by Graphite Furnace Atomic Absorption (GFAA), using the

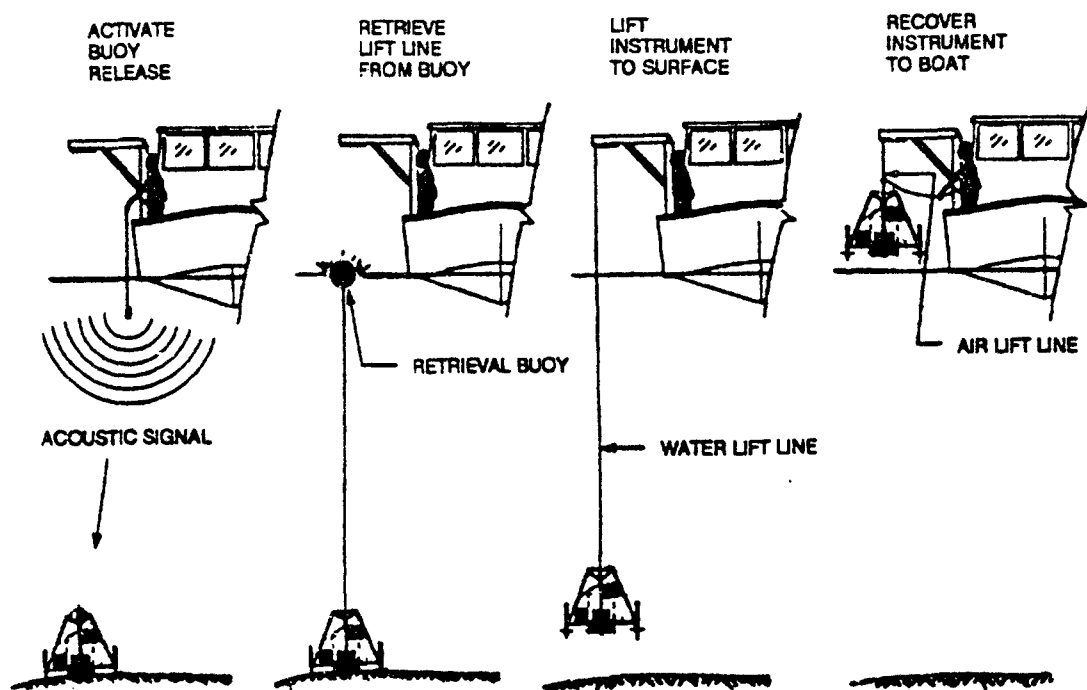


Figure 11. BFSD retrieval.

method of standard additions to develop a standard curve. Additional water samples, including replicates and t_0 samples, are similarly analyzed. 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 just described, for all elements of interest, except arsenic (As) and mercury (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 prior to acidification. Samples are refrigerated until analyzed. The analysis follows the standard colorimetric method for determining reactive silicate in seawater (Strickland & Parsons, 1968).

QA/QC PROCEDURES

TRACE METAL SAMPLES

Method Blanks

Throughout the analyses, method blanks are employed to verify 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—and 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 analyzing the digestates. Initial calibration is verified by subsequently measuring 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 also validate this 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 1 in 20 field samples.

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 range around 90 percent. Internal standards are also used as part of the GC/MS (Selected Ion Monitoring Gas Chromatography—Mass Spectrometry) procedures that 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 typically agree 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 percent relative standard deviation (%RSD) values for its target compounds. Typical median %RSD values are approximately 15 percent for water and sediment.

Chadwick et al. (1993) contains a complete record of sample collection, processing, and QA/QC procedures for an actual deployment.

DATA ANALYSIS

CALCULATION OF FLUX RATES

Flux rates from BFS D time-series samples are estimated using a linear regression model. Before running the regression, sample concentrations are corrected for dilution effects caused by taking in 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 dissolved silica during the experiment indicates a "problem-free" deployment. Additionally, deployments are evaluated by reviewing oxygen data from the sensors on board the BFS D. 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,

$$\text{Flux Rate} = \frac{mV}{A}$$

where m is the slope of regression, V is the chamber volume, and A is the chamber area. Typically, flux rates are calculated using a standard spreadsheet similar to the one shown in figure 12.

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 an upper limit of the 80% CI less than zero, then the flux is designated an *uptake*. The 80% CI is chosen to be conservative, i.e., it does not eliminate potential release rates (as an indicator of environmental impact), unless confidence in them is quite low.

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'

GPS lat: not recorded
long: not recorded
Depth: 4.6'

lab data		BFSO data		outlier correct		dil. correction		from reg		Regressions		Flux rates	
sample id	[Conc] (ppb)	sample	elapsed time	[Conc] (ppb)	elapsed time	[Conc] (ppb)	[Conc] (ppb)	# of dilutions	[Conc] (ppb)	Constant	Std Err of Y Est	Regression Output	Chamber Volume = Chamber Area = Flux rate =
Copper													
T061992-0	2.60	T0	0.00	2.60	0.00	2.13	2.13	0.00	2.33	2.33	0.45	2.33	Chamber Volume = 40.65 liters Chamber Area = 1600.40 cm ²
T061992-b	2.13	#1	14.00	2.13	14.00	2.84	2.84	1.00	2.17	2.17	0.36	0.45	Flux rate = 70 ug/m ² /day
T061992-a	2.84	#2	28.00	2.84	28.00	1.72	1.72	2.00	2.01	2.01	6.00	0.36	80% CI (low) = -142 ug/m ² /day (high) = 1 ug/m ² /day
T061992-f	1.72	#3	42.00	1.72	42.00	1.58	1.58	3.00	1.85	1.85	4.00	6.00	
T061992-i	1.58	#4	56.00	1.58	56.00	1.99	1.99	4.00	1.69	1.69		4.00	
T061992-c	1.99	#5	70.00	1.99	70.00	1.81	1.81	5.00	1.53	1.53	-0.0115		
T061992-h	1.81	#6									0.0076		
Cadmium													
T061992-0	0.072	T0	0.00	0.072	0.00	0.059	0.059	0.00	0.055	0.055	0.06	0.06	Chamber Volume = 40.65 liters Chamber Area = 1600.40 cm ²
T061992-b	0.059	#1	14.00	0.059	14.00	0.057	0.057	1.00	0.057	0.057	0.00	0.06	Flux rate = 1 ug/m ² /day
T061992-a	0.057	#2	28.00	0.057	28.00	0.059	0.059	2.00	0.060	0.060	0.44	0.44	80% CI (low) = 0 ug/m ² /day (high) = 2 ug/m ² /day
T061992-f	0.059	#3	42.00	0.059	42.00	0.059	0.059	3.00	0.062	0.062	6.00	6.00	
T061992-i	0.059	#4	56.00	0.059	56.00	0.074	0.074	4.00	0.064	0.064	4.00	4.00	
T061992-c	0.074	#5	70.00	0.074	70.00	0.070	0.070	5.00	0.068	0.068	0.0001		
T061992-h	0.070	#6									0.0001		
Nickel													
T061992-0	0.37	T0	0.00	0.37	0.00	0.37	0.37	0.00	0.59	0.59	0.59	0.59	Chamber Volume = 40.65 liters Chamber Area = 1600.40 cm ²
T061992-b	0.35	#1	14.00	0.35	14.00	0.69	0.69	1.00	0.67	0.67	0.17	0.17	Flux rate = 33 ug/m ² /day
T061992-a	0.69	#2	28.00	0.69	28.00	0.67	0.67	2.00	0.74	0.74	0.47	0.47	80% CI (low) = 4 ug/m ² /day (high) = 62 ug/m ² /day
T061992-f	0.67	#3	42.00	0.67	42.00	0.91	0.91	3.00	0.82	0.82	6.00	6.00	
T061992-i	0.91	#4	56.00	0.91	56.00	1.16	1.16	4.00	0.90	0.90	4.00	4.00	
T061992-c	1.16	#5	70.00	1.16	70.00	0.79	0.79	5.00	0.97	0.97	0.0055		
T061992-h	0.79	#6									0.0029		
Zinc													
T061992-0	7.67	T0	0.00	7.67	0.00	7.67	7.67	0.00	7.04	7.04	7.04	7.04	Chamber Volume = 40.65 liters Chamber Area = 1600.40 cm ²
T061992-b	6.63	#1	14.00	6.63	14.00	10.05	10.05	1.00	9.91	9.91	0.47	0.47	Flux rate = 1250 ug/m ² /day
T061992-a	10.05	#2	28.00	10.05	28.00	13.68	13.68	2.00	12.78	12.78	0.99	0.99	80% CI (low) = 1175 ug/m ² /day (high) = 1324 ug/m ² /day
T061992-f	13.68	#3	42.00	13.68	42.00	15.75	15.75	3.00	15.65	15.65	6.00	6.00	
T061992-i	15.75	#4	56.00	15.75	56.00	19.17	19.17	4.00	18.52	18.52	4.00	4.00	
T061992-c	19.17	#5	70.00	19.17	70.00	21.56	21.56	5.00	21.39	21.39	0.2050		
T061992-h	21.56	#6									0.0010		

Figure 12. Flux rate calculation spreadsheet.

RESULTS

The BFSD has undergone a series of test deployments to determine the effectiveness of the system to quantify sediment-water contaminant exchange rates. A blank test was performed by sealing the chamber bottom with a polycarbonate panel and conducting a standard experiment over 50 hours. Additional blank results were obtained from deployments at clean sites in San Diego Bay and Sinclair Inlet, WA. Mean chamber concentration, %RSD, calculated blank fluxes, and their standard errors (S.E.) are summarized in table 1 and presented graphically in figure 13. These results set a lower limit on the flux rates that can be resolved using the BFSD system.

Table 1. Blank chamber results.

Compound	n	Mean Conc. ($\mu\text{g/l}$)	%RSD	Flux $\pm$ S.E. ($\mu\text{g/m}^2/\text{day}$)
¹ Cadmium	7	0.52	14	6 ± 7
¹ Copper	7	3.4	14	-71 ± 62
³ Iron	—	—	—	—
¹ Lead	7	0.39	16	-4 ± 8
³ Manganese	—	—	—	—
² Nickel	6	1.5	27	65 ± 69
² Zinc	6	2.1	25	-227 ± 65

¹From blank chamber.

²From clean reference sites.

³No blank data available.

Two experiments were conducted at Shelter Island yacht basin in San Diego Bay (figure 14) from 6/19–6/21/92 and 6/25–6/28/92. Previous studies have shown elevated water, sediment, and mussel-tissue levels of a number of trace metals at this site (Salazar and Chadwick, 1991). Table 2 summarizes bulk sediment characteristics at this site from samples collected during the deployments. These data suggest elevated concentrations of cadmium, copper, lead, nickel, and zinc at 2- to 25-times background levels at reference sites.

Table 2. Bulk sediment characteristics at the Shelter Island test site.

Metal Concentrations		¹ Other Characteristics	
Cadium	0.26 $\mu\text{g/g}$	Sand	5%
Copper	161 $\mu\text{g/g}$	Silt	65%
Iron	4.02 $\mu\text{g/g}$	Clay	30%
Lead	42.8 $\mu\text{g/g}$	TOC	2.4%
Manganese	414 $\mu\text{g/g}$		
Nickel	16.2 $\mu\text{g/g}$		
Zinc	199 $\mu\text{g/g}$		

¹From Kram et al. (1989).

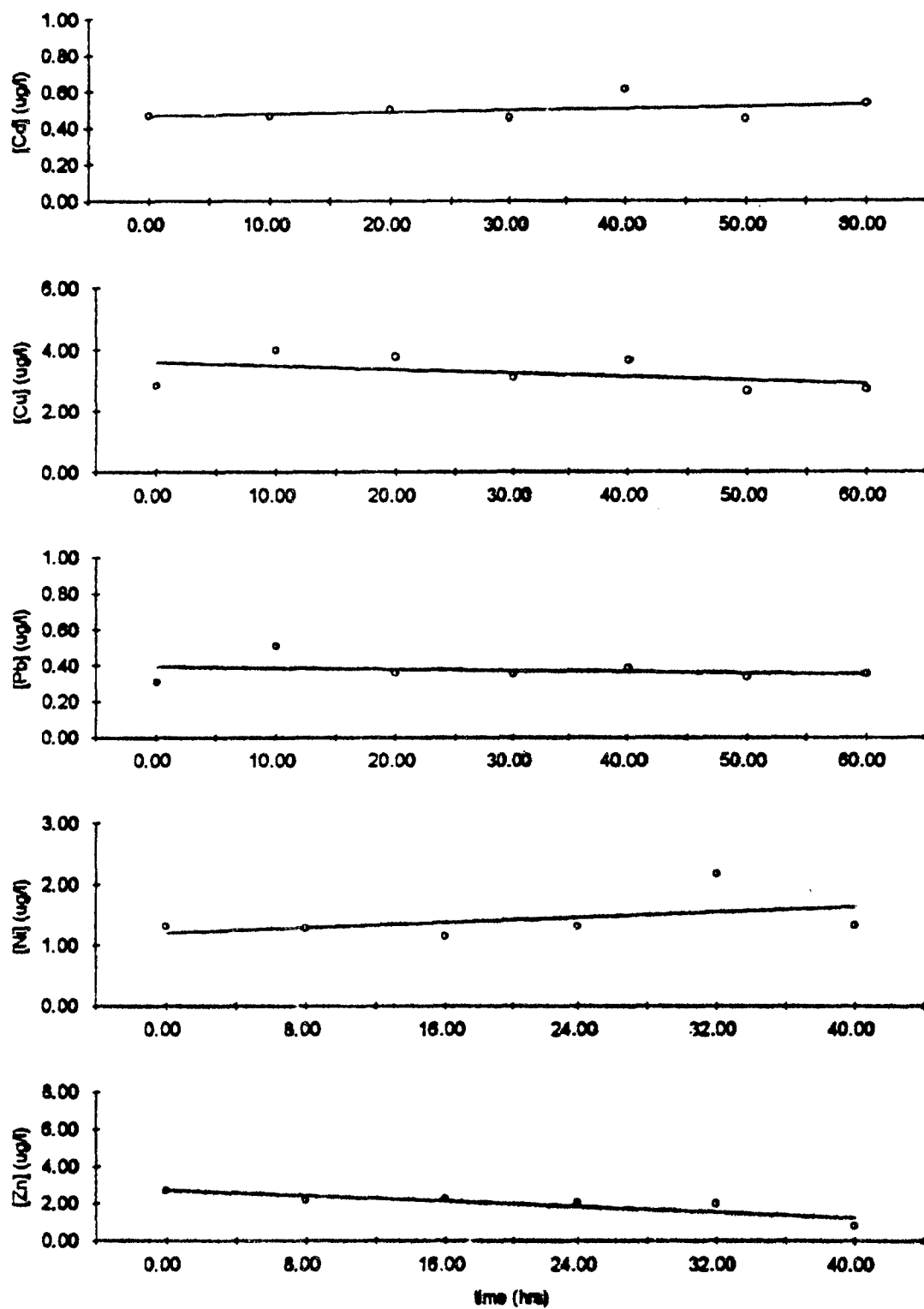


Figure 13. Time-series results from blank tests of the BFSD.

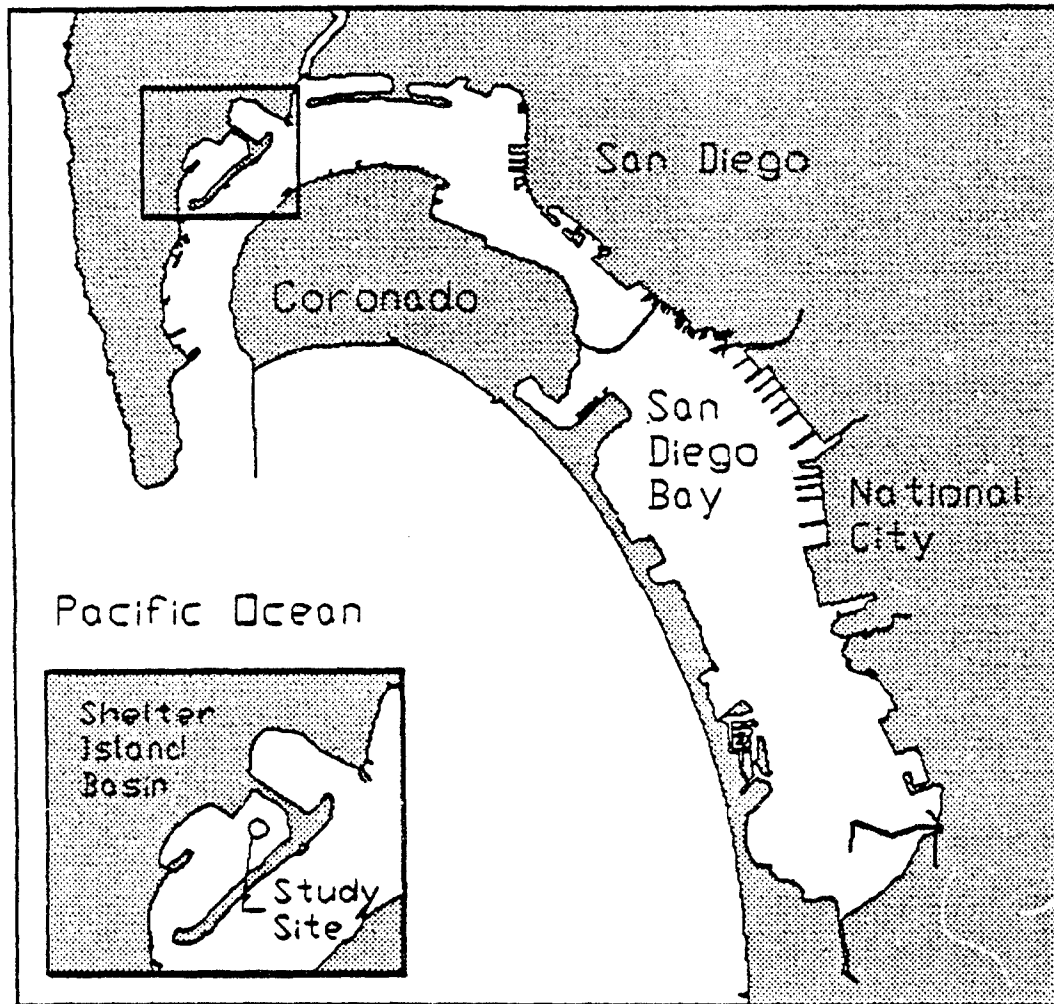


Figure 14. Shelter Island yacht basin.

Typical time-series traces from the on-board oxygen, pH, temperature, and salinity sensors are shown in figure 15 for the 6/19 deployment. The traces show that oxygen was maintained between 130–200 μM during the experiment with an initial decrease due to the response time of the feedback control system. The pH decreased from an initial value of about 8.1 to 7.5 at the end of the experiment, presumably due to sediment respiration and consequent production of CO_2 (figure 16). The shallow depth of the site (4 m) is reflected in the diel variation of the temperature signal that also followed a longer-term decreasing trend of about 1°C over the 75-hour deployment. Salinity showed a slight, monotonic decrease during the deployment.

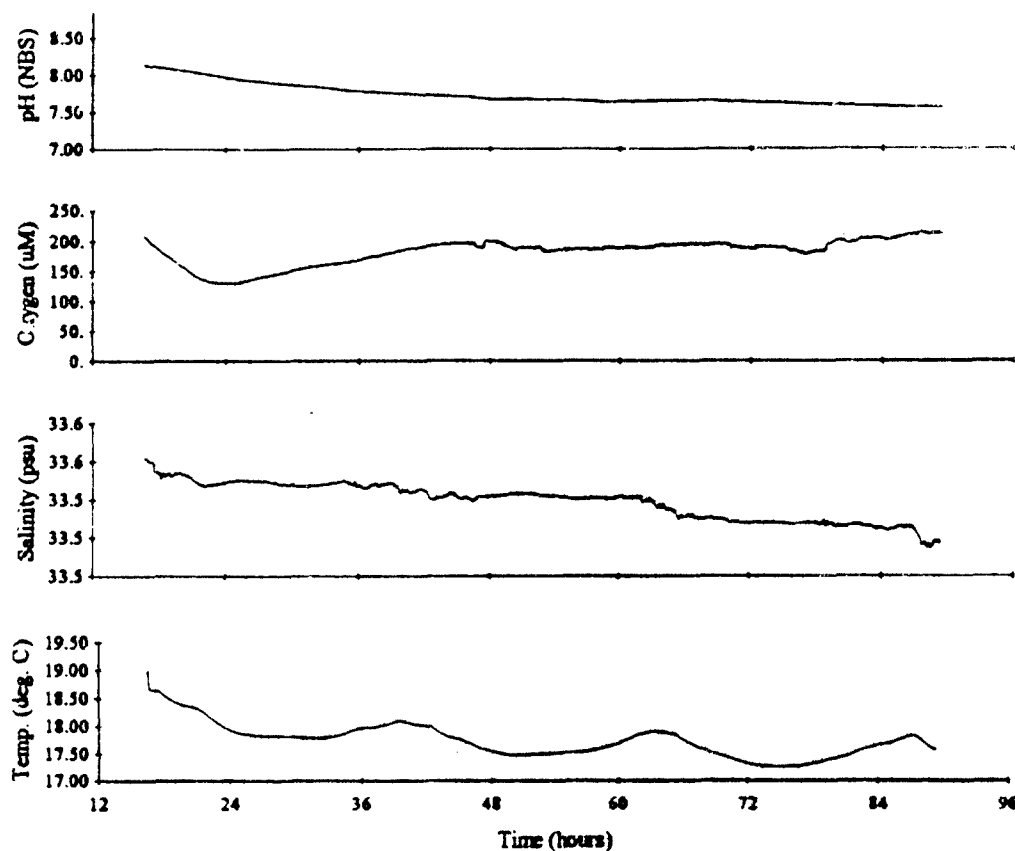


Figure 15. Continuous time-series traces for chamber conditions during the 6/19/92 deployment.

Flux data time-series plots for the two deployments are shown in figure 16 for a number of trace metals and nutrients, as well as for alkalinity and CO_2 . The consistent flux of silica is used as a performance check on the seal and sampling integrity of the deployment. Based on this criteria, both deployments appear to have been successful. While previous deployments during which the seal was violated or the samples were compromised showed erratic silica concentrations, the time series from these deployments were linear and consistent between deployments. Trace metal flux rates were analyzed for cadmium, copper, nickel, iron, manganese, zinc, silver, and lead. Of these, lead and silver were not present at detectable levels ($<0.26 \mu\text{g/l}$ and $<0.001 \mu\text{g/l}$ respectively), so no results are reported. Manganese and zinc showed strong increasing trends during both experiments, while nickel, cadmium, and iron showed no significant flux; and copper generally indicated a slight sediment uptake. Phosphate increased during the 6/19 experiment, but was inconsistent during the 6/25 experiment (figure 17), with an initial large increase followed by a more gradual decrease. Alkalinity and total CO_2 both increased steadily throughout the experiments. Flux rates calculated from these time series are summarized in table 3 and compared to results from other deployments of the BFS and measurements by other research-

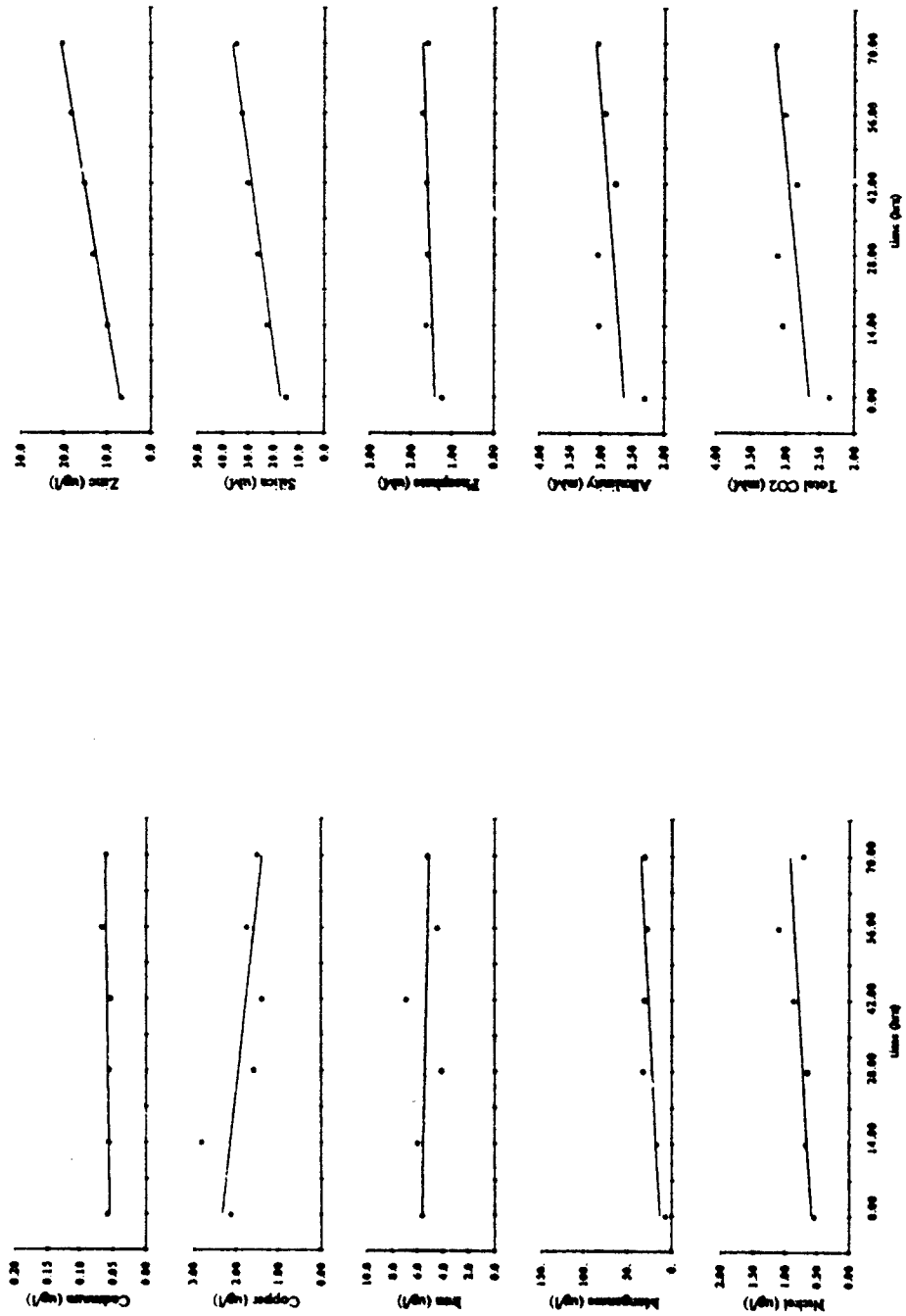


Figure 16. Time-series evolution of trace metals, nutrients, alkalinity, and total CO₂ in the flux chamber during the 6/19/92 deployment at Shelter Island yacht basin.

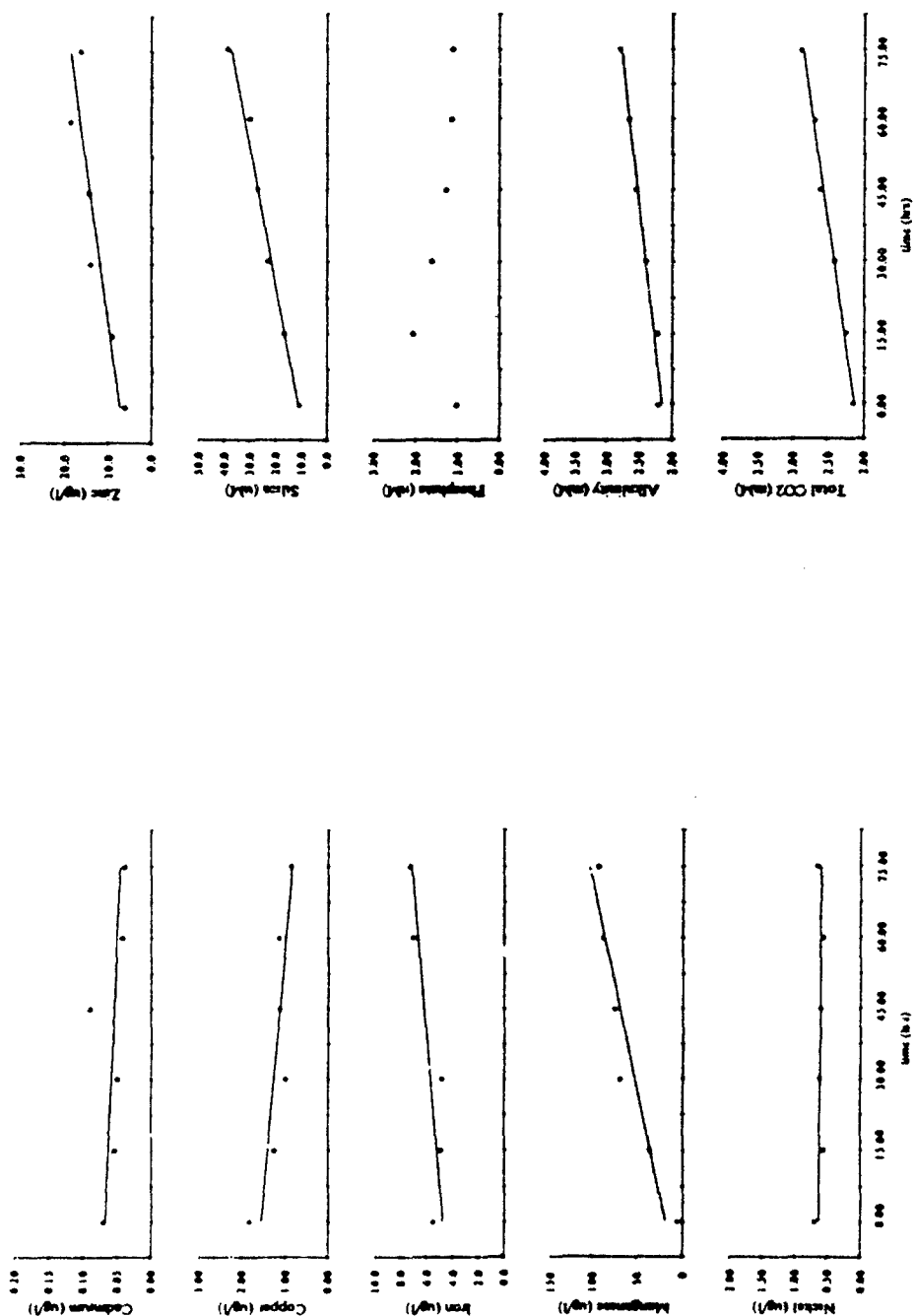


Figure 17. Time-series evolution of trace metals, nutrients, alkalinity, and total CO₂ in the flux chamber during the 6/25/92 deployment at Shelter Island yacht basin.

Table 3. Summary of flux rates from the 6/19 and 6/25 deployments compared to results from other studies.
Trace metal flux rates are in $\mu\text{g}/\text{m}^2/\text{day}$. Units for Si, PO_4 , and CO_2 are $\text{mmoles}/\text{m}^2/\text{day}$.

Study Site	Cd	Cu	Fe	Mn	Ni	Pb	Zn	Si	PO_4	CO_2
Shelter Island, San Diego Bay 6/19/93 (this study)	0.6	-81	-47	1820	29	—	1179	1.6	0.04	44
Shelter Island, San Diego Bay 6/25/93 (this study)	-2	-58	193	6847	-4	—	920	2.1	—	53
Paleta Creek, San Diego Bay 3/22/93 (Chadwick et al., 1993)	20	-87	760	11730	128	2	3553	—	—	—
PACO Term., San Diego Bay 3/15/93 (Chadwick et al., 1993)	3	103	3	2519	34	31	1557	—	—	—
Naragansett Bay (Hunt and Smith, 1983)	—	67	—	—	—	5	—	—	—	—
Naragansett Bay (McCaffrey et al., 1980)	—	—	—	—	—	—	—	12	0.7	20
Gullmarsfjorden, Sweden (Westerlund et al., 1986)	0.6	3.4	—	1208	—	5.8	42	—	—	—
Gullmarsfjorden, Sweden (Ruigers van der Loeff et al., 1984)	—	—	—	—	—	—	—	0.17-6.26	—	—
Long Island Sound (Aller, 1980)	—	—	1-500	10-4000	—	—	—	—	—	—
Puget Sound (Murray and Gill, 1978)	—	—	13	—	—	—	—	—	—	—
Whites Point, Los Angeles (Berelson and Johnson, 1991)	—	—	—	—	—	—	—	1.9	0.44	—

CONCLUSIONS

An autonomous system has been developed to monitor the exchange rates of contaminants and other biogeochemical compounds across the sediment water interface. Results from a series of test deployments indicate that the system can quantify these exchange rates at realistic levels for coastal and inshore sediments using a 2-4 day sampling period. The resulting flux rates will be useful in evaluating the risks posed by in-place sediment contamination, from several aspects, including

- Source quantification for comparison to other sources and input to models.
- As an indicator of bioavailability, since many studies indicate that resolubilized contaminants are more readily available for uptake.
- To determine the cleansing rate of a contaminated sediment site due to natural biogeochemical cycling of the in-place contaminants.
- To provide a nonintrusive monitoring tool for sites that have been capped or sealed to minimize biological exposure.
- As a scientific tool, to provide realistic testing and validation of hypotheses and models for predicting the response of marine sediments to various contaminants.

Future efforts include adapting the BFS system to allow feedback control of pH, an increased number of samples, in-place filtration and preservation of samples, increased depth rating, and standardization for constructing multiple units. Methodologies are also being developed to allow continuous flux measurements of contaminants using integrated, *in-situ* sensor technology.

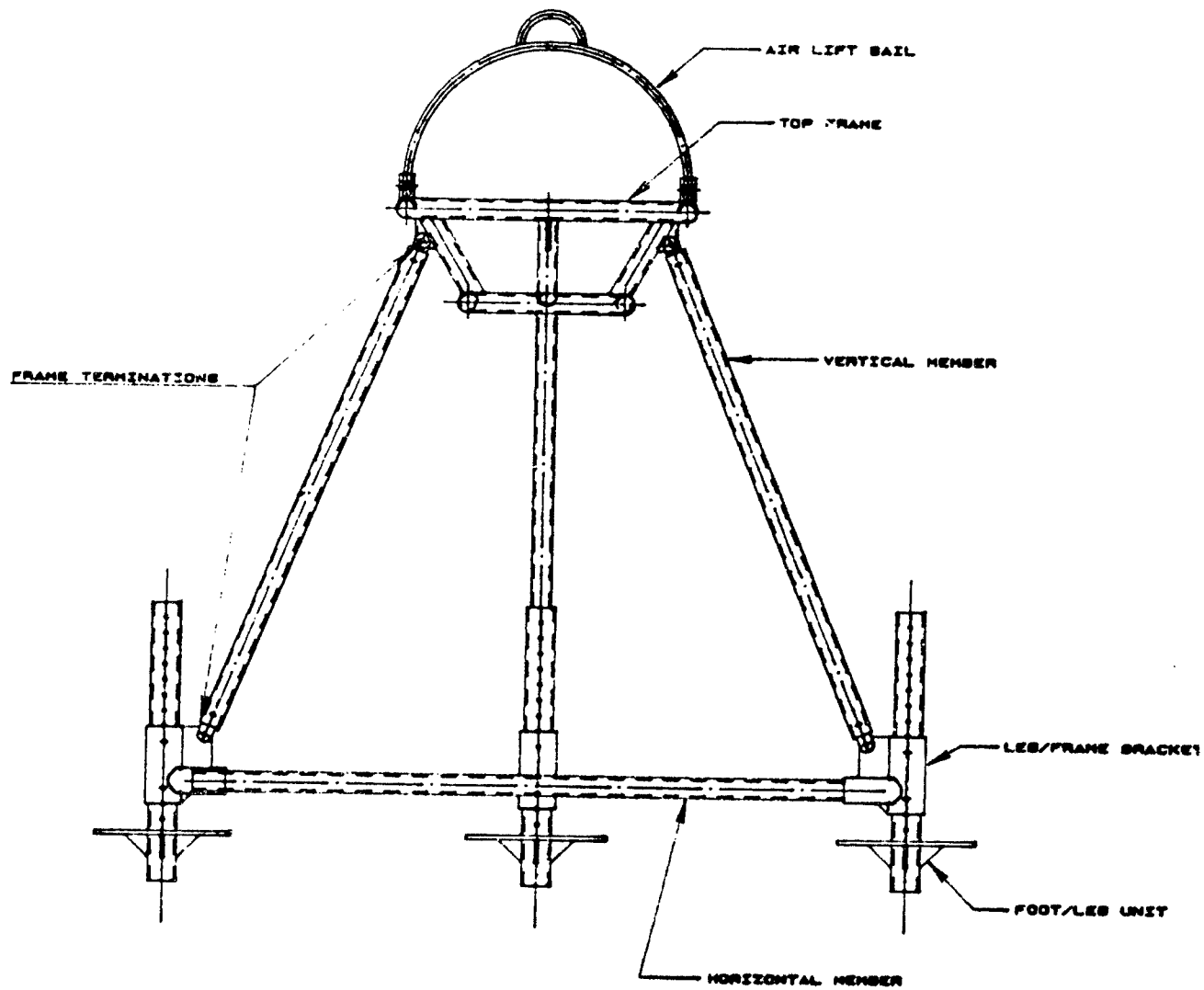
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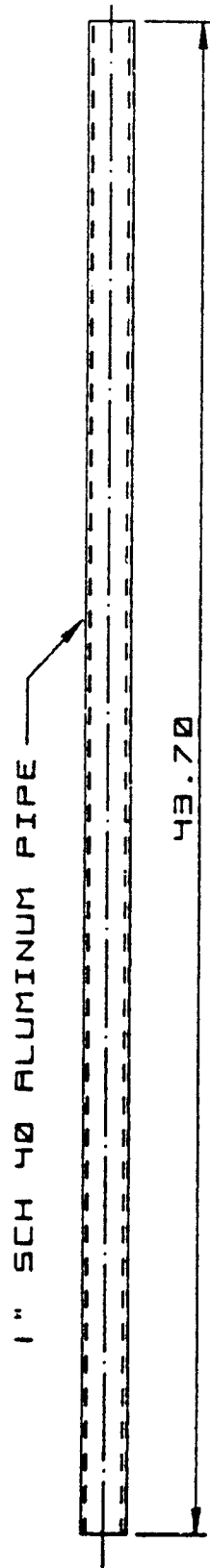
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Appendix A

Drawings

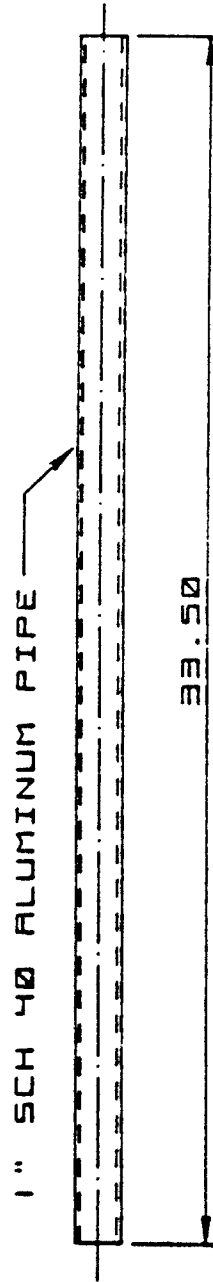


TRIPOD ASSEMBLY



HORIZONTAL MEMBER

(3 REQUIRED)

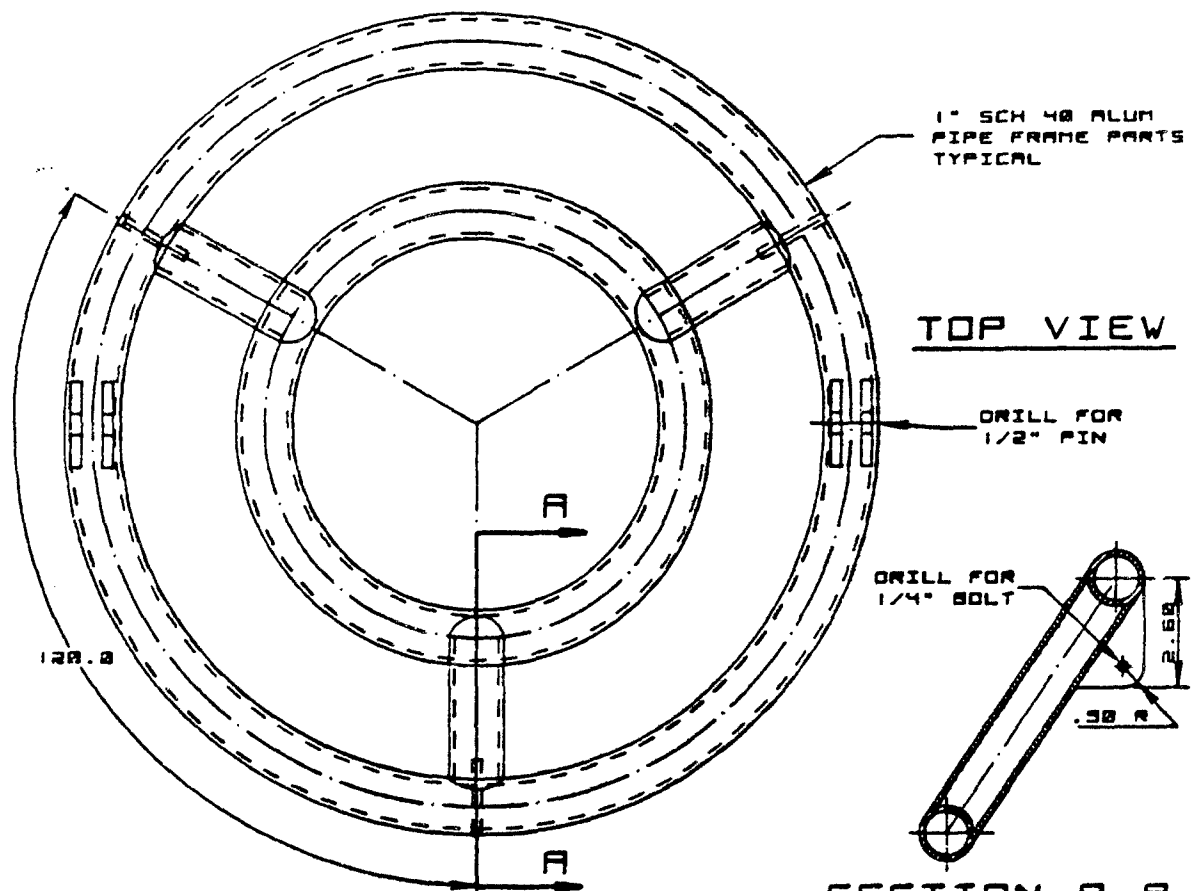


VERTICAL MEMBER

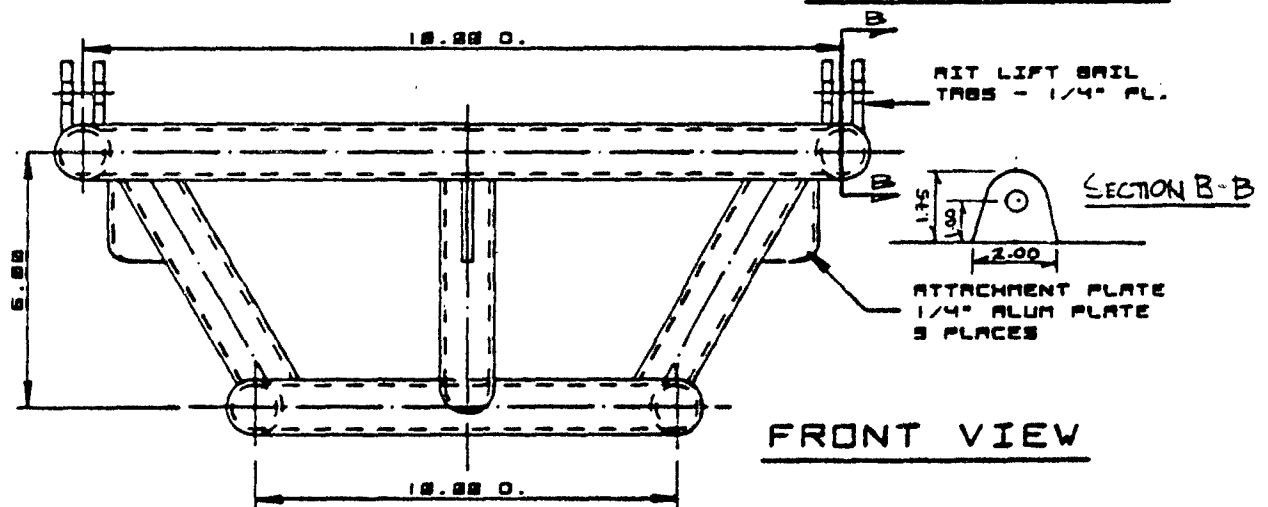
(3 REQUIRED)

FRAME MEMBERS

SCALE: 1/15



SECTION A-A

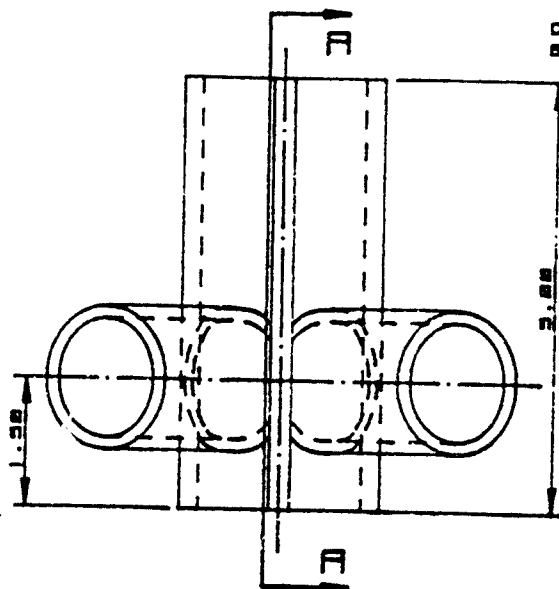


TOP FRAME & RELEASE/FLOAT CAGE

SCALE: 1:1

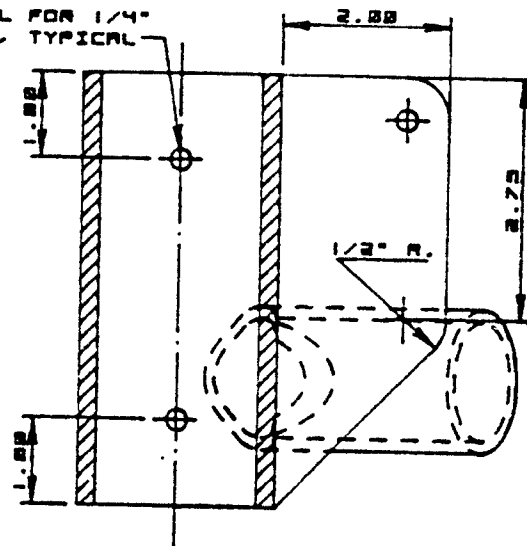
1 REQUIRED

A-5

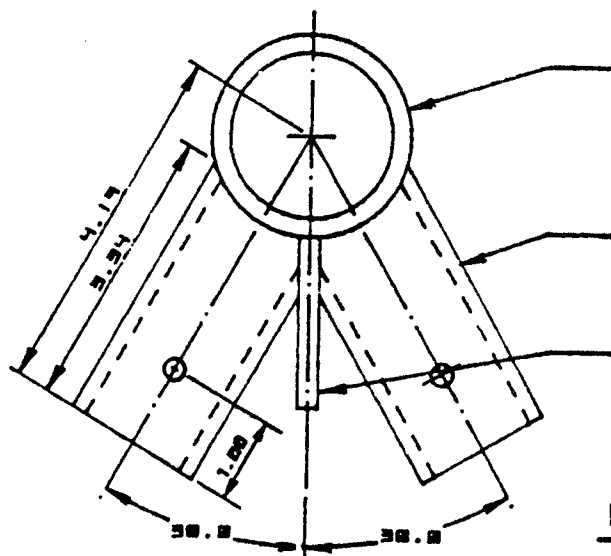


FRONT VIEW

DRILL FOR 1/4"
BOLT, TYPICAL



SECTION A-A



TOP VIEW

LEG SOCKET
2" SCH. 80
ALUM. PIPE

FRAME SOCKETS
1 1/4" SCH. 40
ALUM. PIPE

FRAME ATTACHMENT
FLANGE 1/4" PLATE
ALUMINUM

LEG/FRAME BRACKET

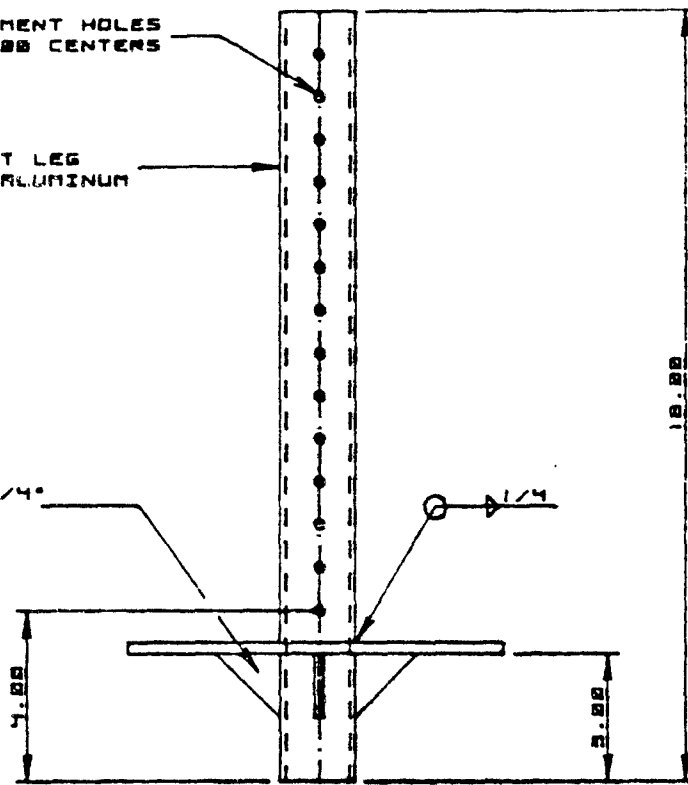
SCALE: 1:2

UNITS REQUIRED = 3

VERTICAL ADJUSTMENT HOLES
0.25 DIA. ON 1.00 CENTERS

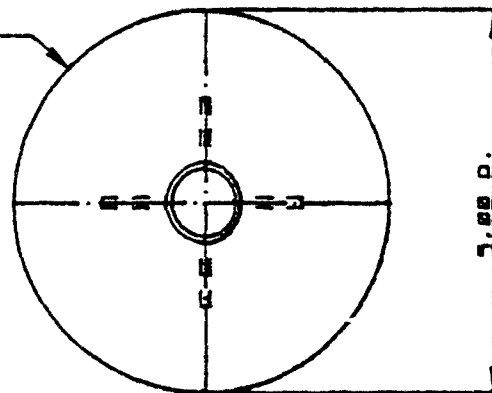
VERTICAL SUPPORT LEG
1 1/2" SCH. 40 ALUMINUM

FOOT/LEG GUSSET
1.00 X 1.00 X 1/4"
PLATE ALUMINUM
(4 PLACES)



SIDE VIEW

FOOT PLATE 1/4" PLATE
ALUMINUM

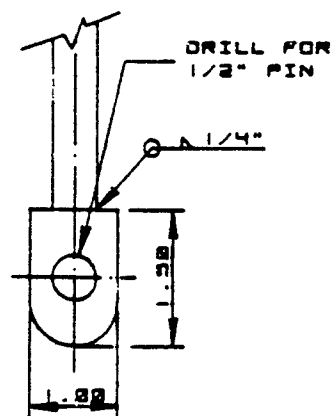


PLAN VIEW

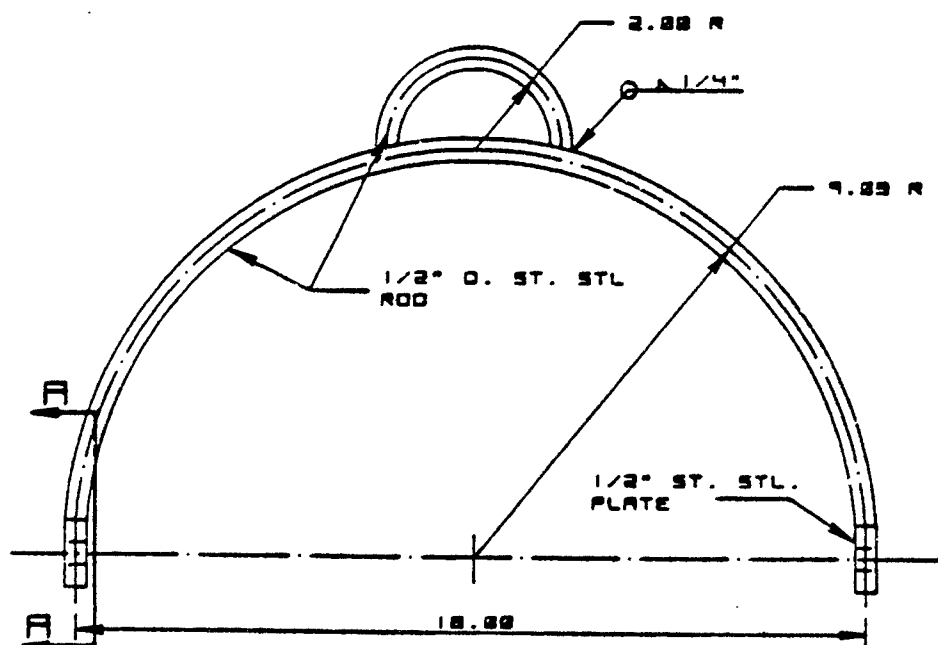
FOOT/LEG UNIT

SCALE: 1/4"

UNITS REQUIRED = 2

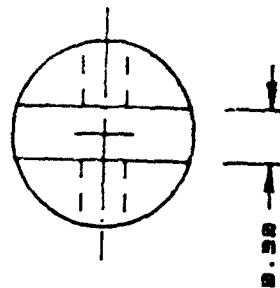


SECTION A-A
SCALE: 1:2

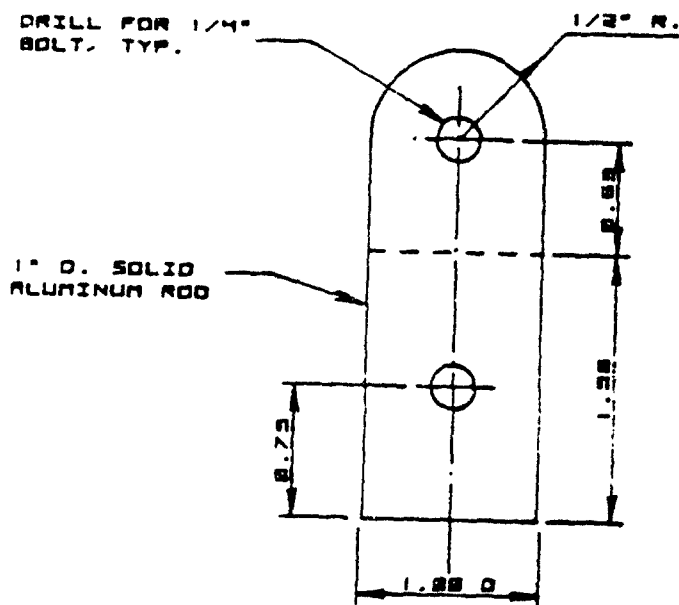


FRONT VIEW
SCALE: 1:4

AIR LIFT BAIL



TOP VIEW



FRONT VIEW

FRAME TERMINATION

SCALE: 1:1

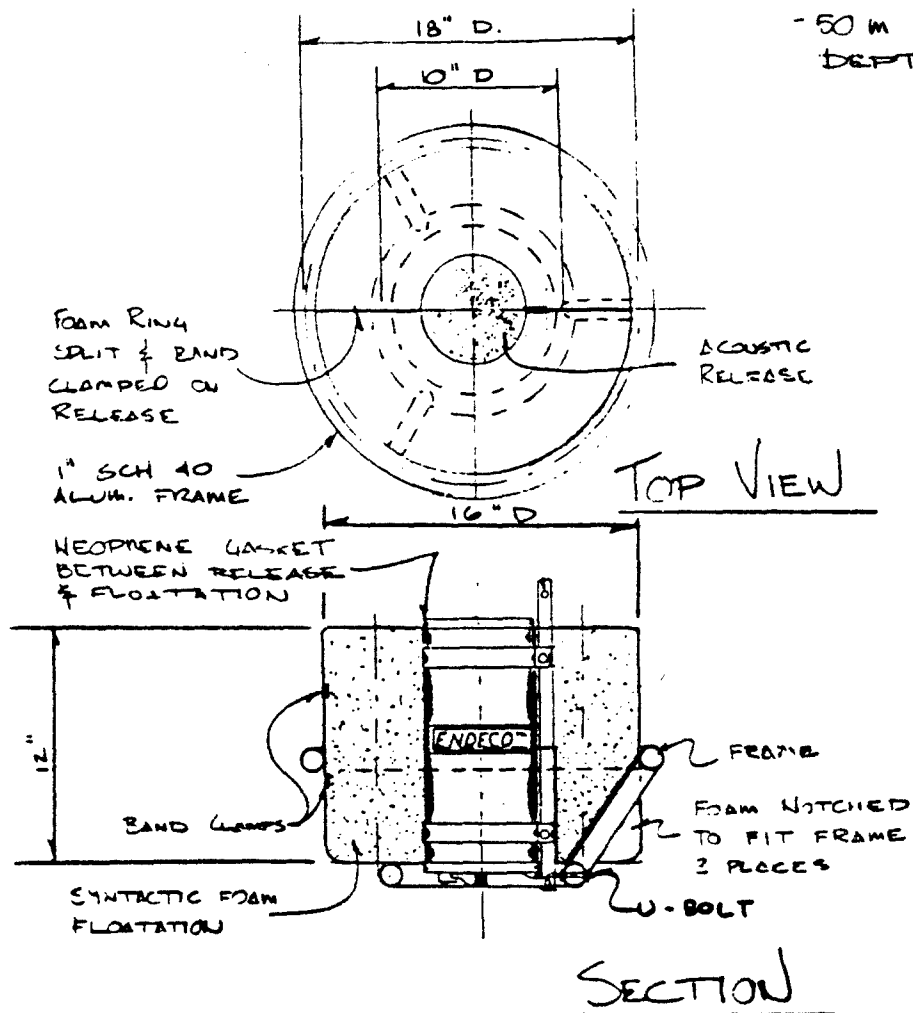
UNITS REQUIRED = 12

FLOATATION: -16" O.D.

-12" HIGH

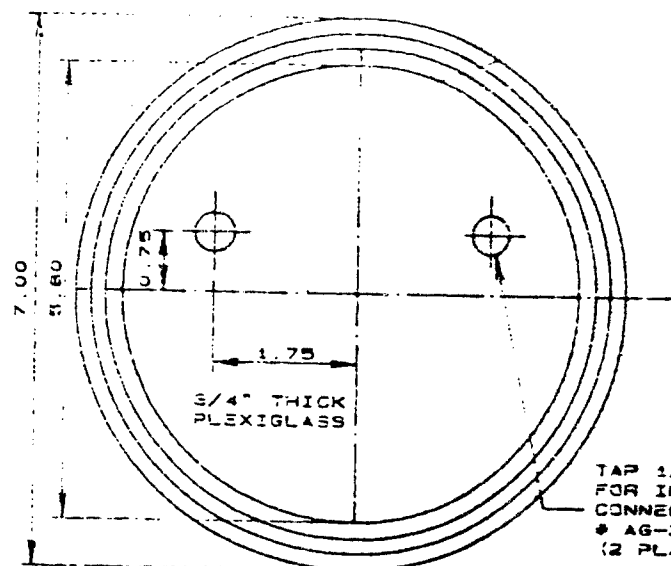
-I.D. TO SUIT

-50 M WORKING
DEPTH

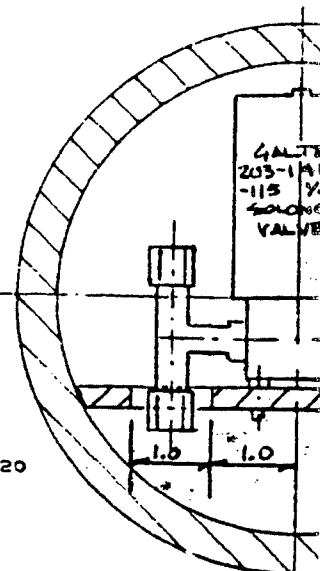


ACOUSTIC RELEASE
INTERFACE & FLOATATION

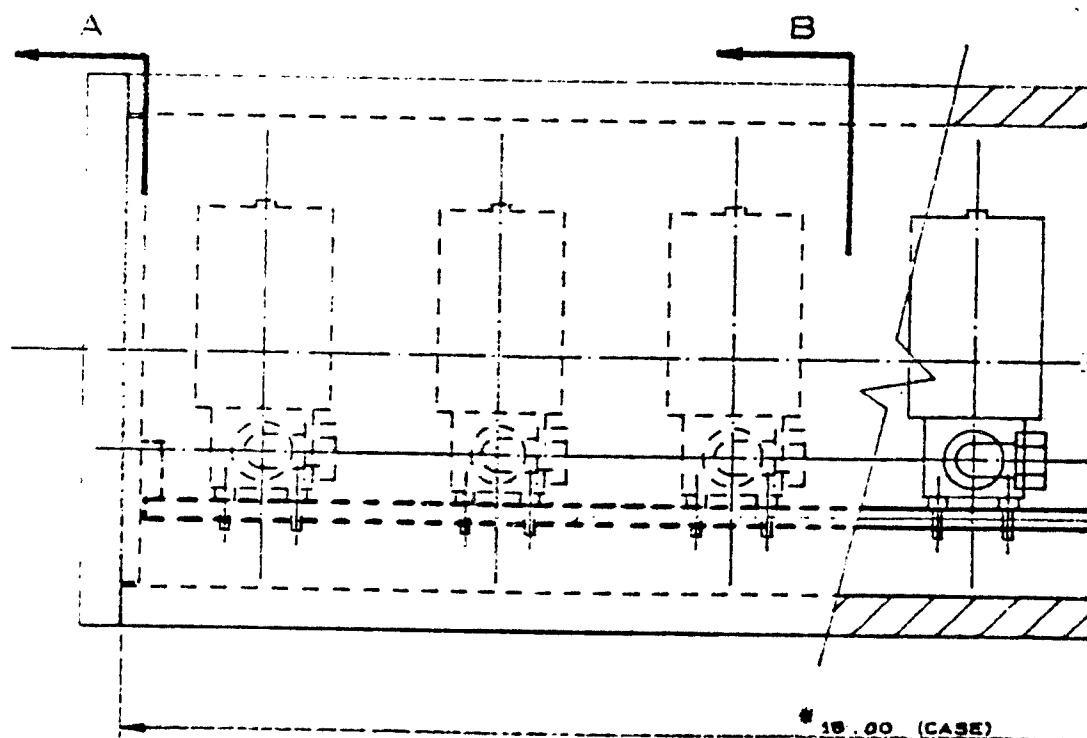
SCALE: 1 1/2" = 1'-0"



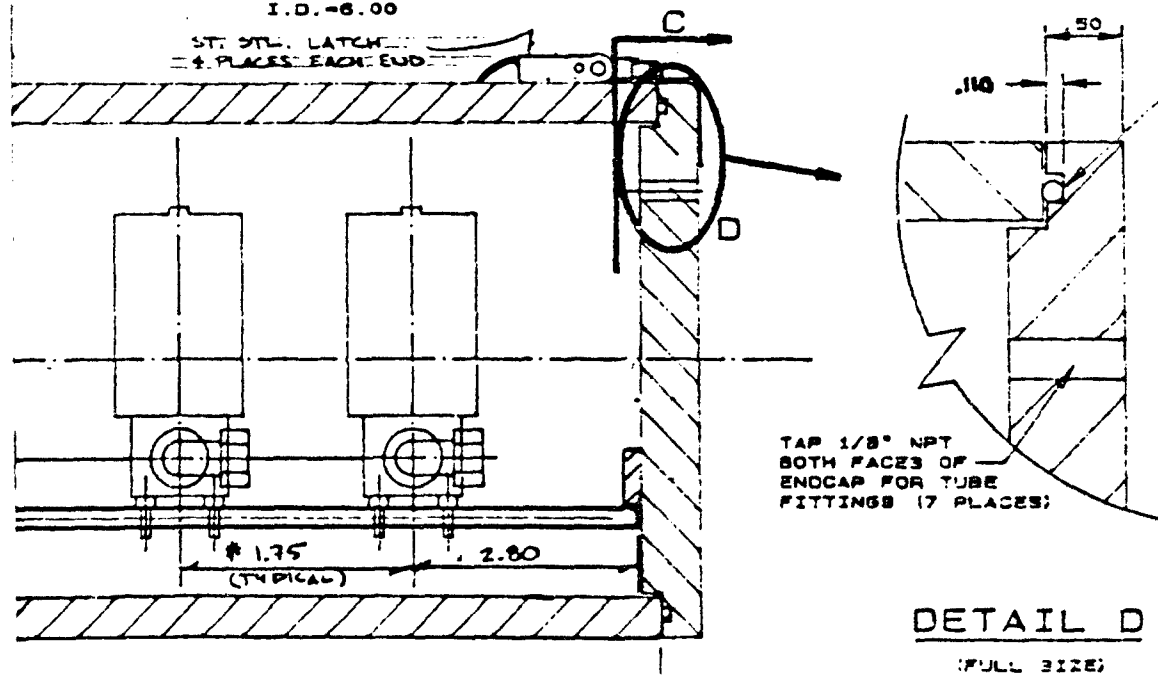
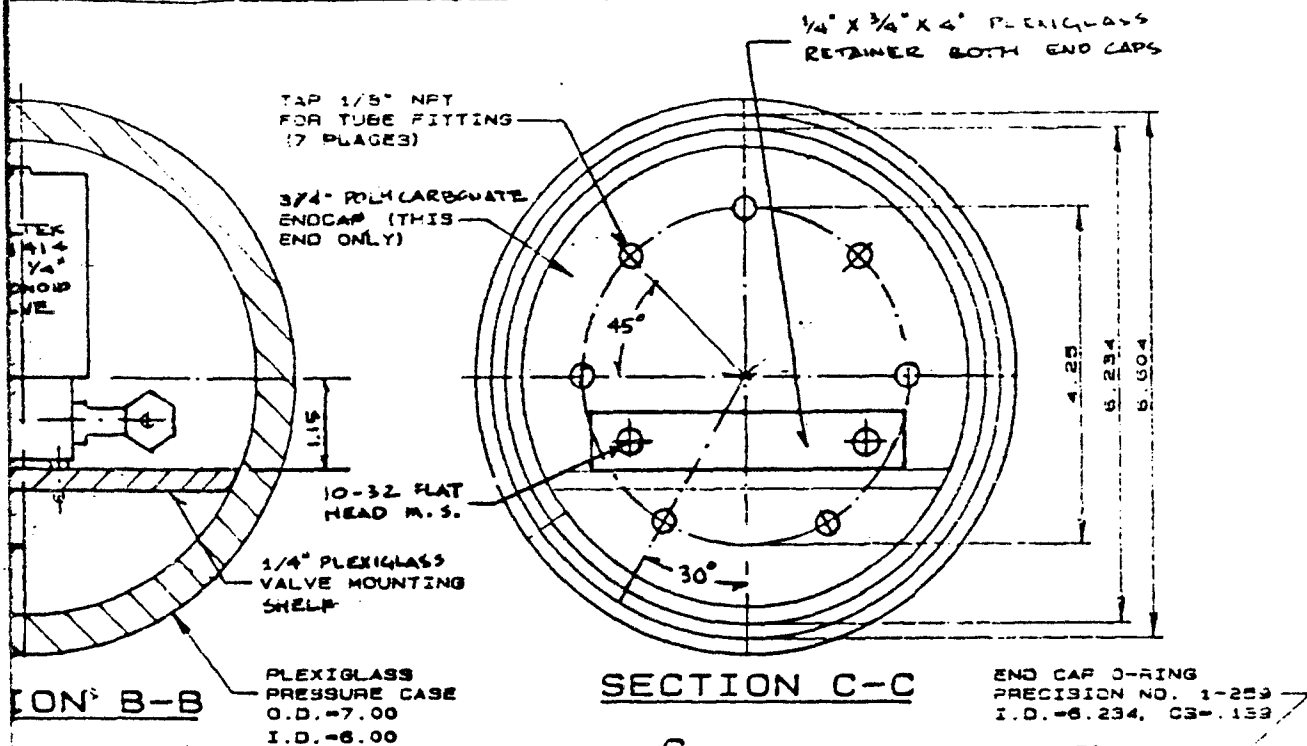
SECTION A-A



SECTION B-B

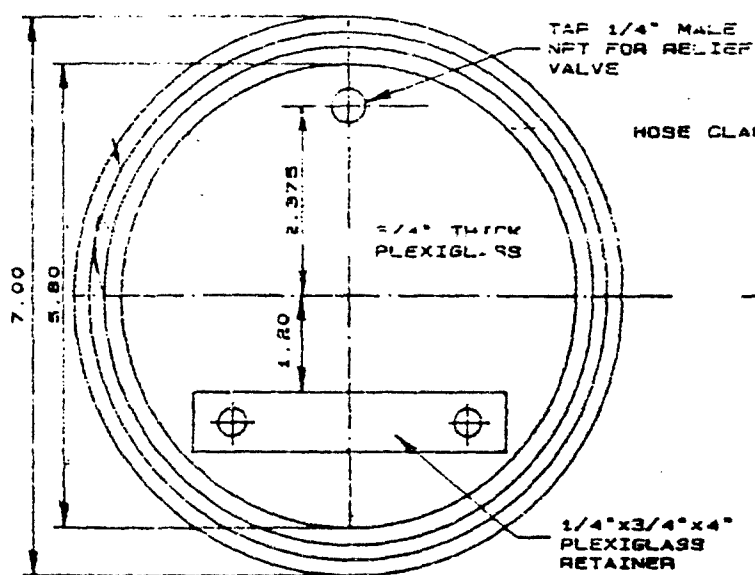


SIDE VIEW

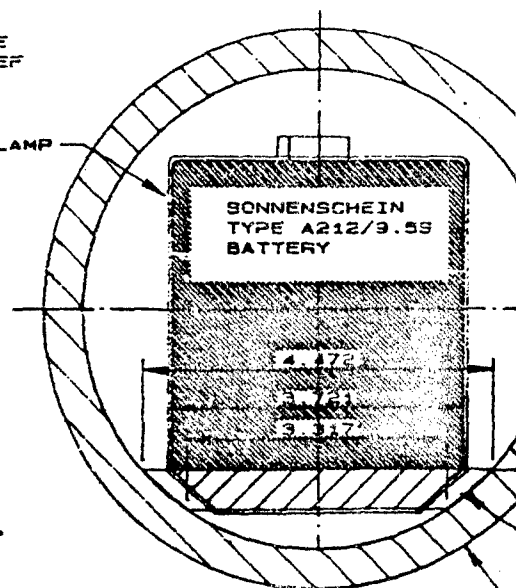


VALVE CASE
SCALE = HALF SIZE

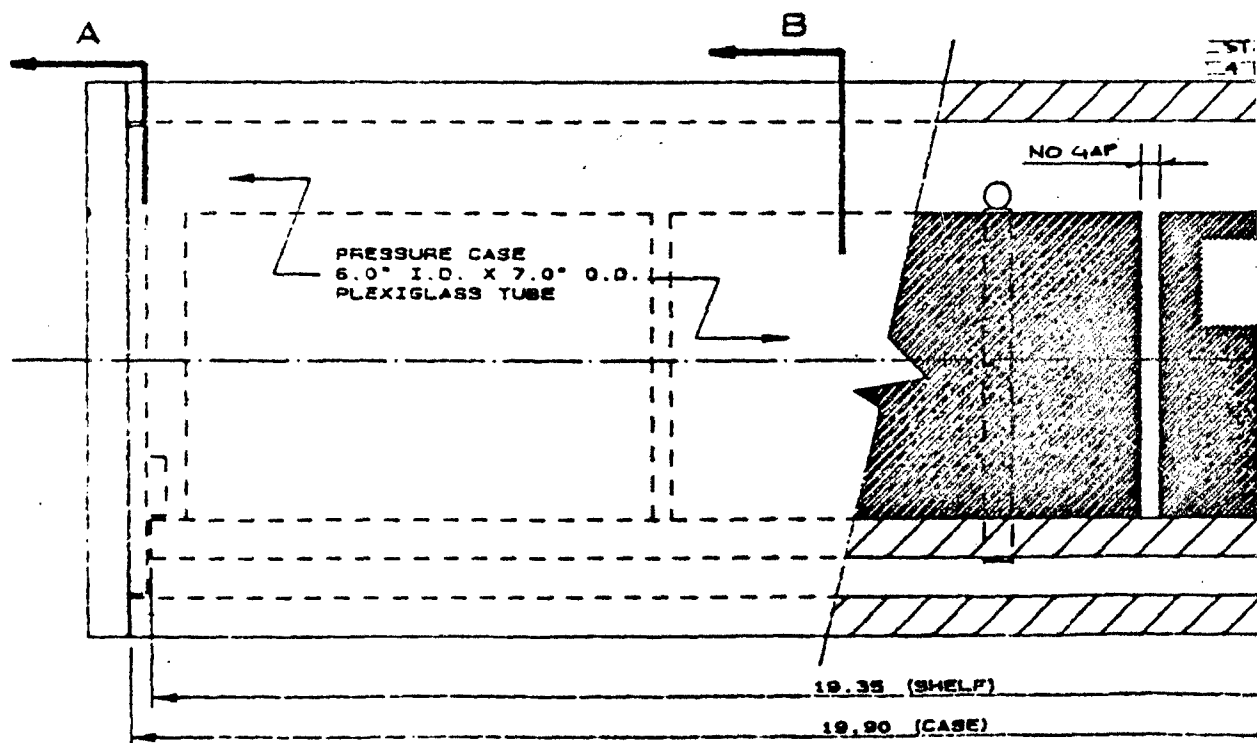
* NOT TO SCALE



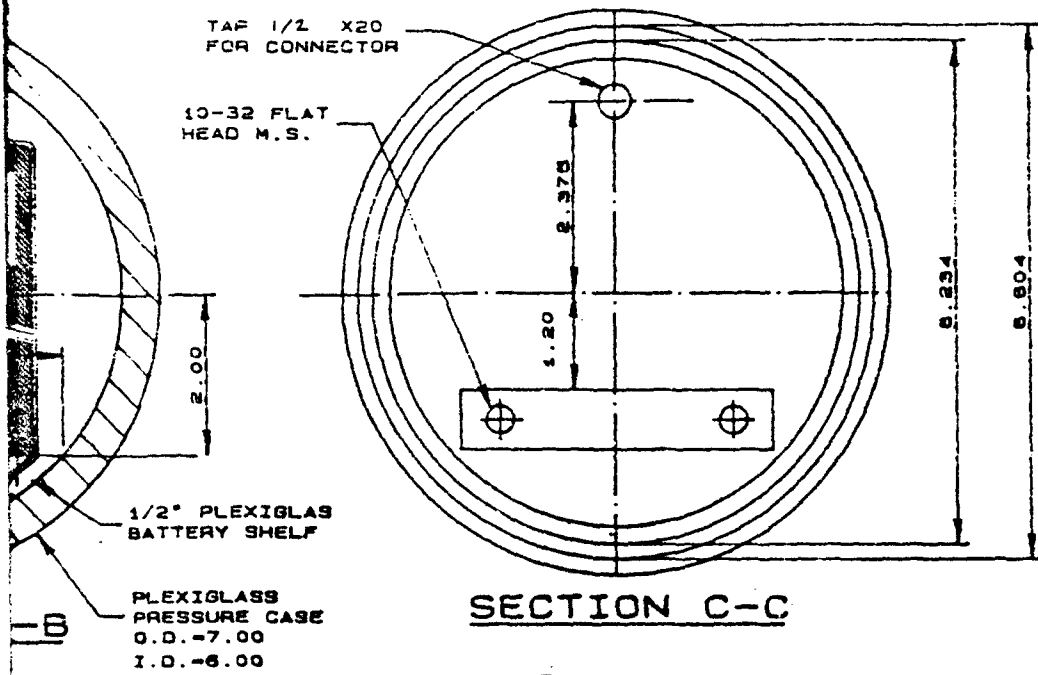
SECTION A-A



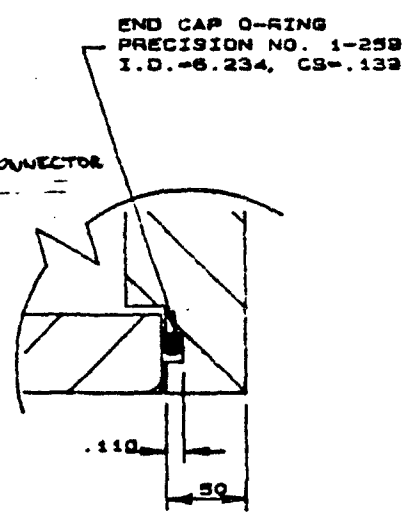
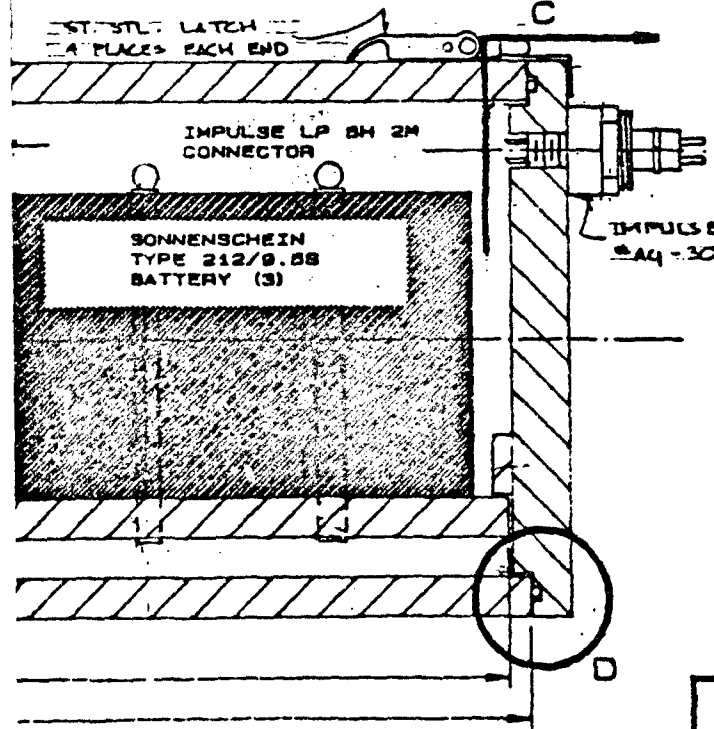
SECTION B-B



SIDE VIEW



SECTION C-C



DETAIL D
(FULL SIZE)

BATTERY CASE
SCALE = HALF SIZE

1.5" X 1.5" X 1/8" AL. ANGLE
LID STIFFENERS

1.5" X 1.5" X 1/8" AL. ANGLE
LID HINGE

18.125 (LID OVERALL)

8.5

1/4" POLYCARBONATE
CHAMBER SIDE PANELS

SKIRT 2

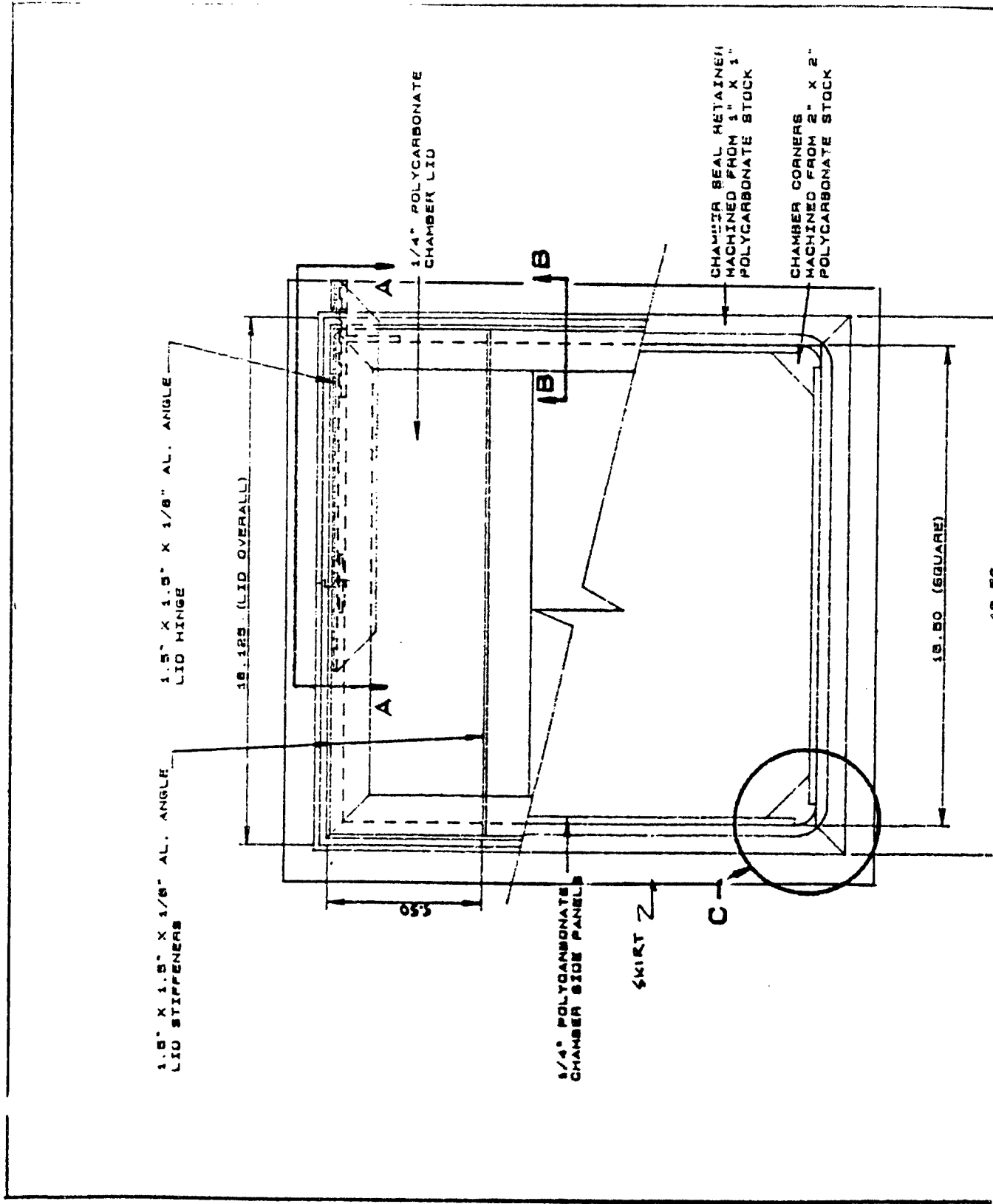
C

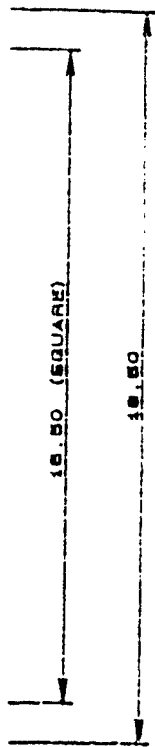
1/4" POLYCARBONATE
CHAMBER LID

CHAMBER SEAL RETAINER
MACHINED FROM 1" X 1"
POLYCARBONATE STOCK

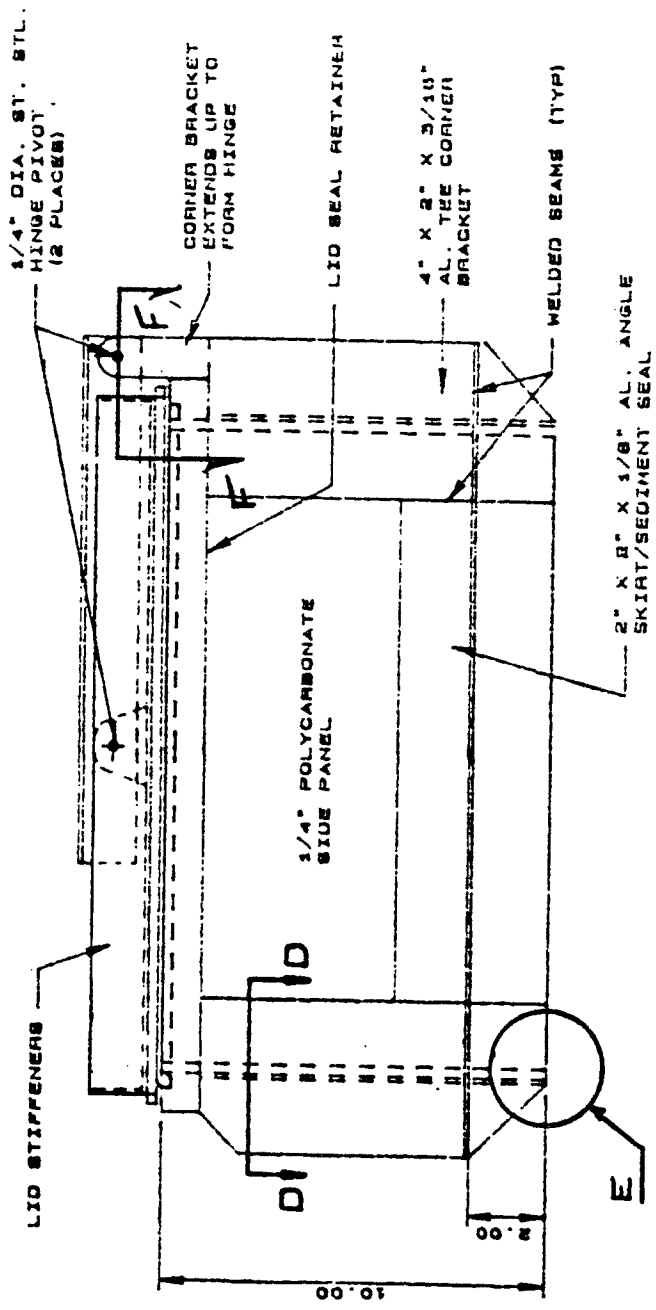
CHAMBER CORNERS
MACHINED FROM 2" X 2"
POLYCARBONATE STOCK

18.50 (SQUARE)



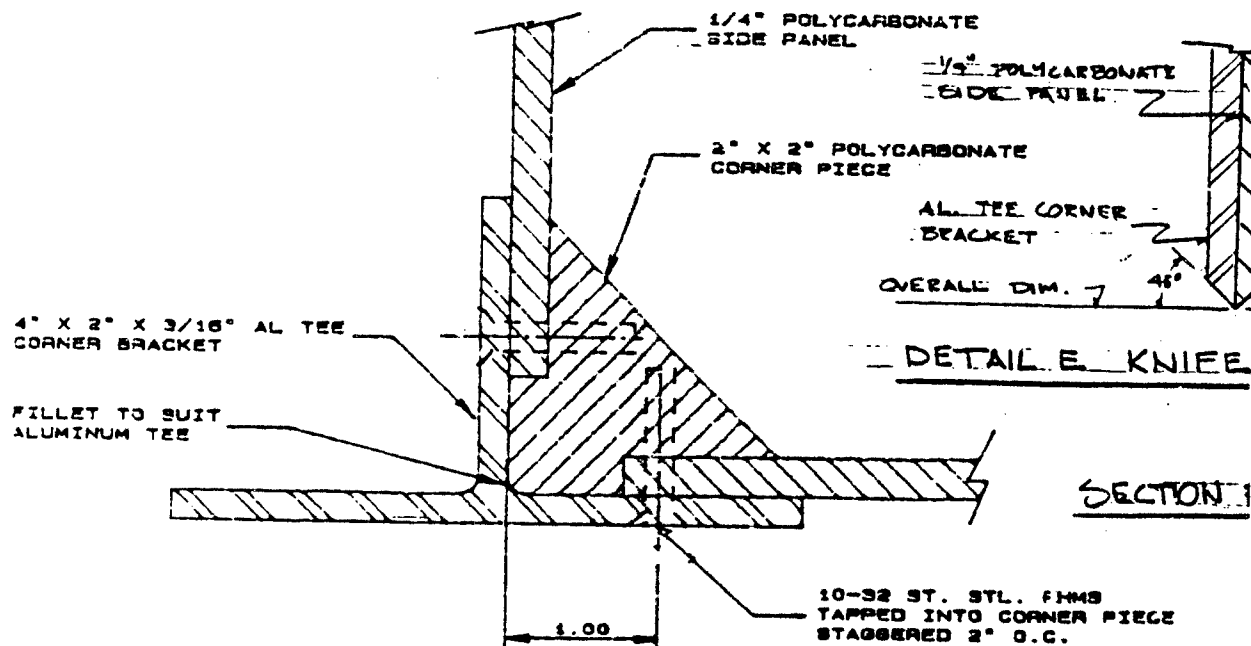
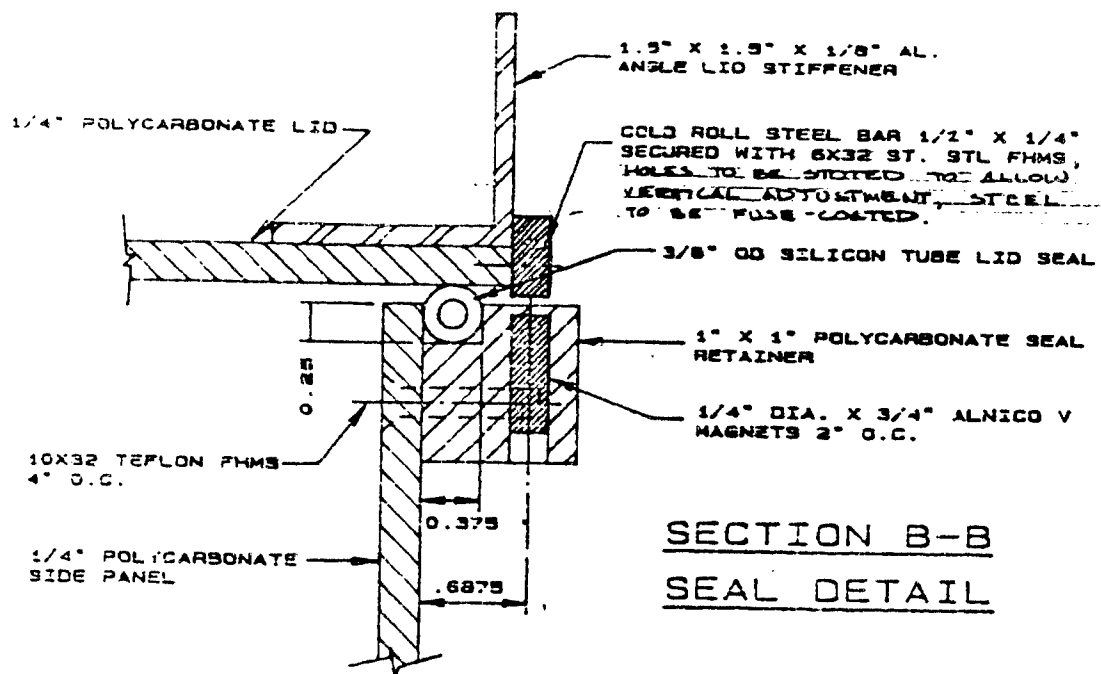


TOP VIEW

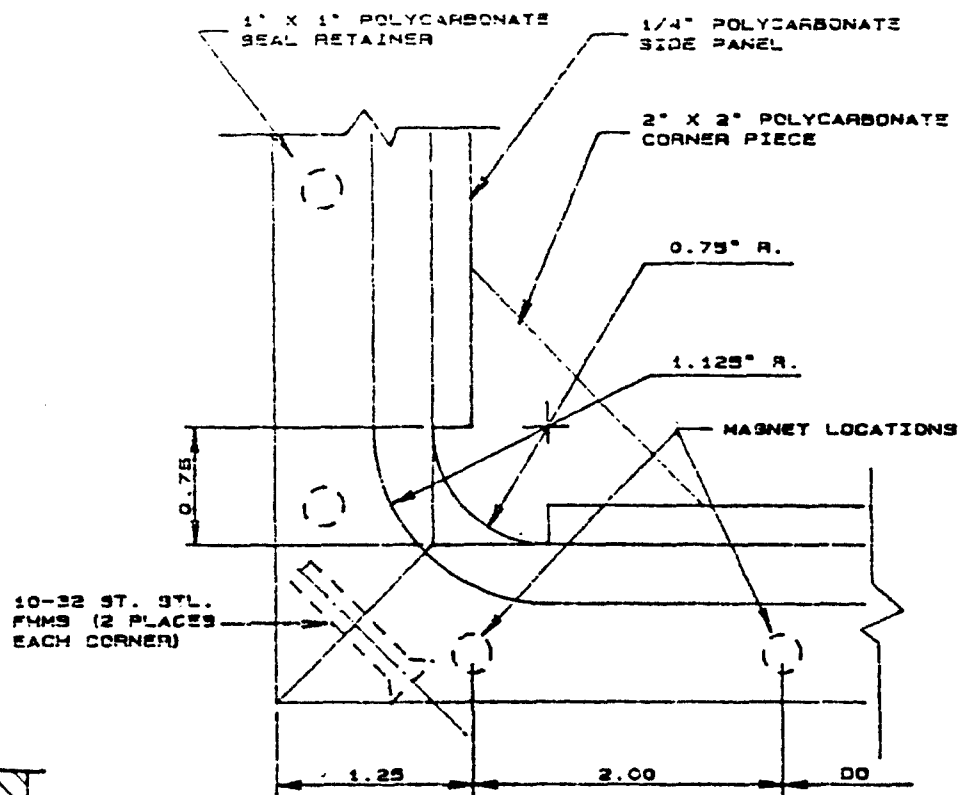


SIDE VIEW

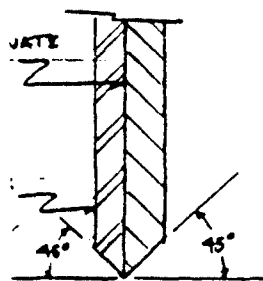
CHAMBER
SCALE: 1:4



SECTION D-D CHAMBER CORNER

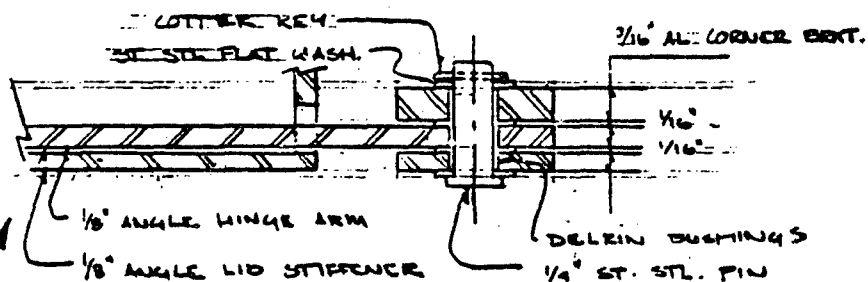


DETAIL C LID SEAL CORNER



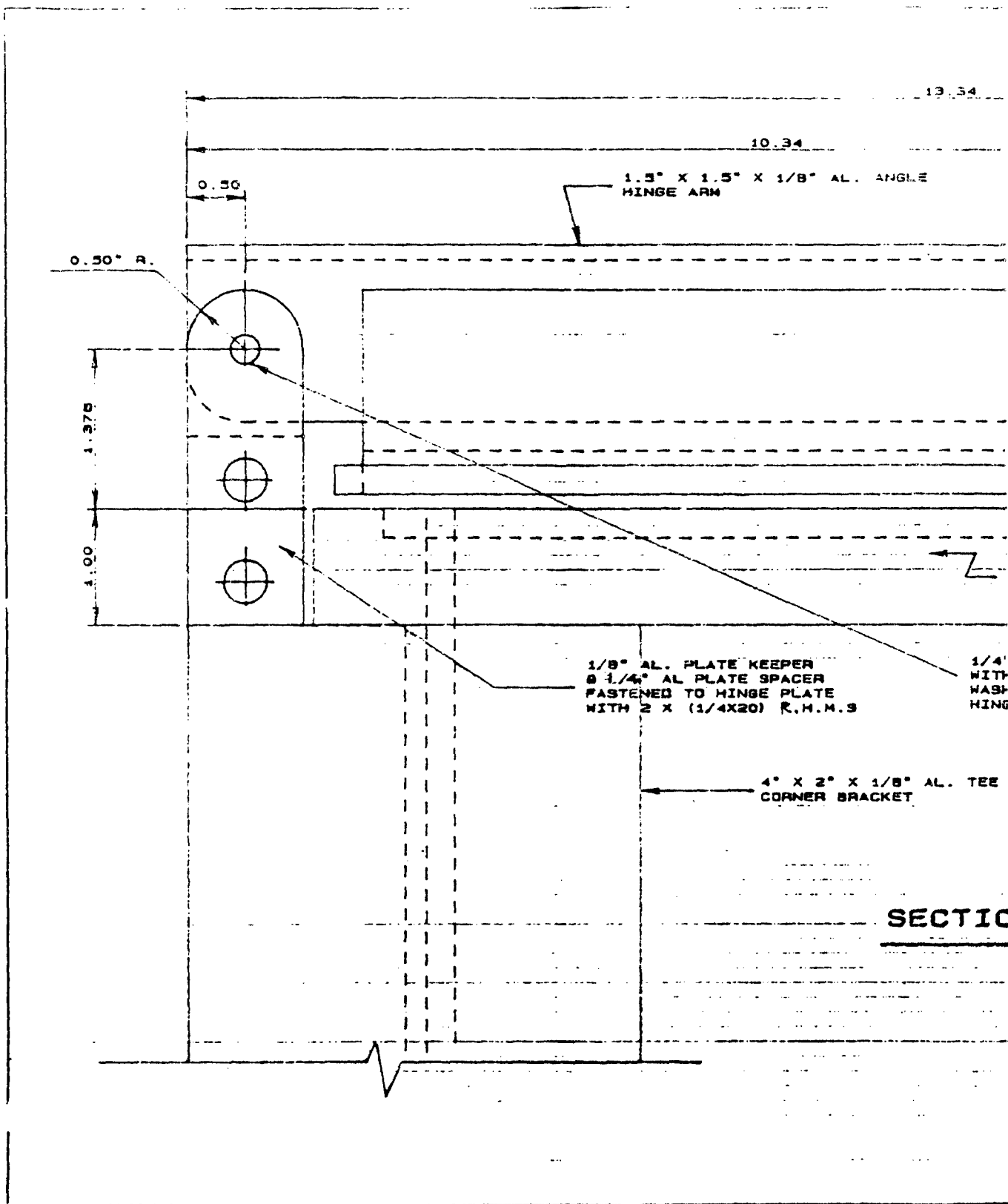
KNIFE EDGE

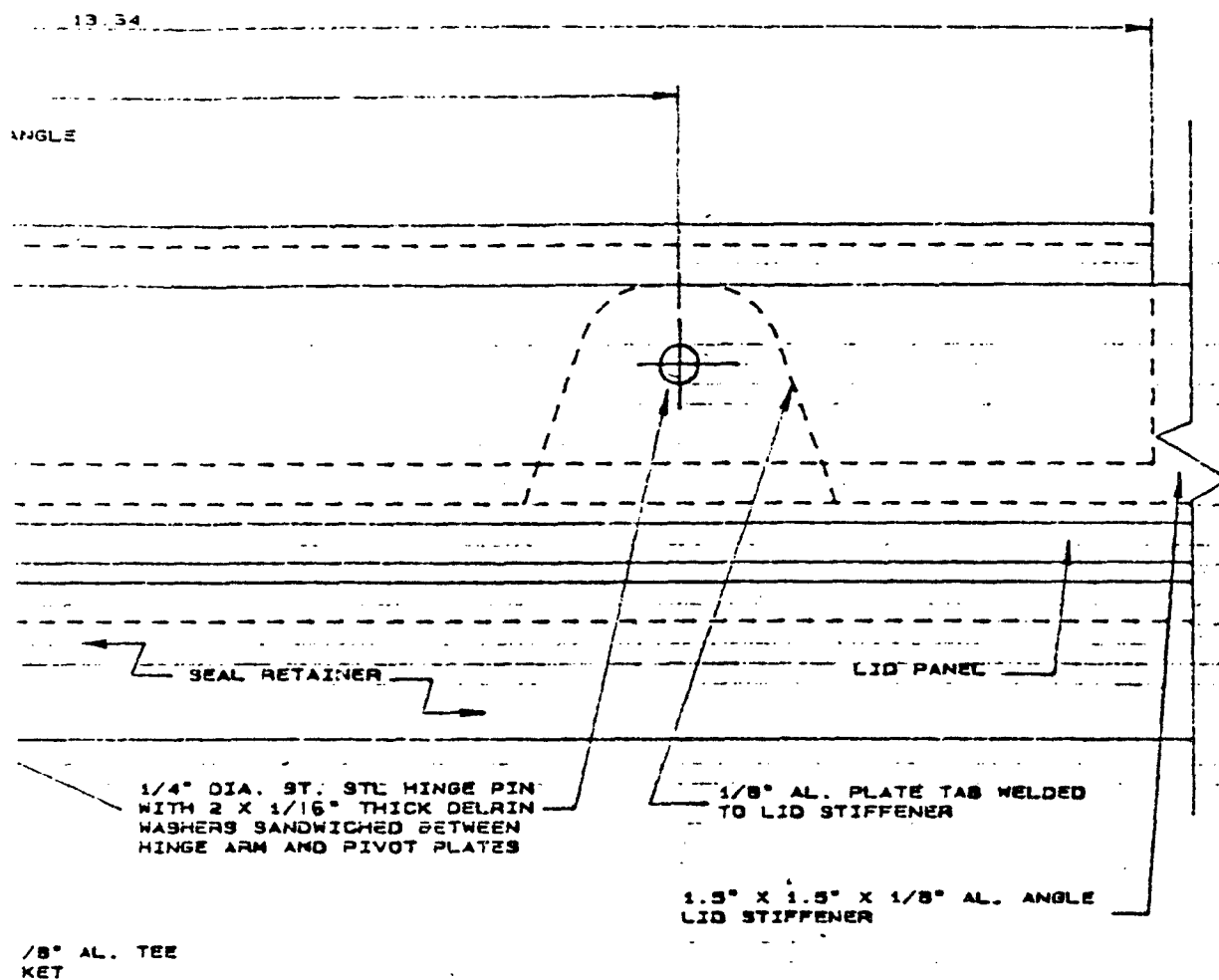
SECTION F-F HINGE PIN



CHAMBER DETAILS

SCALE: FULL SIZE

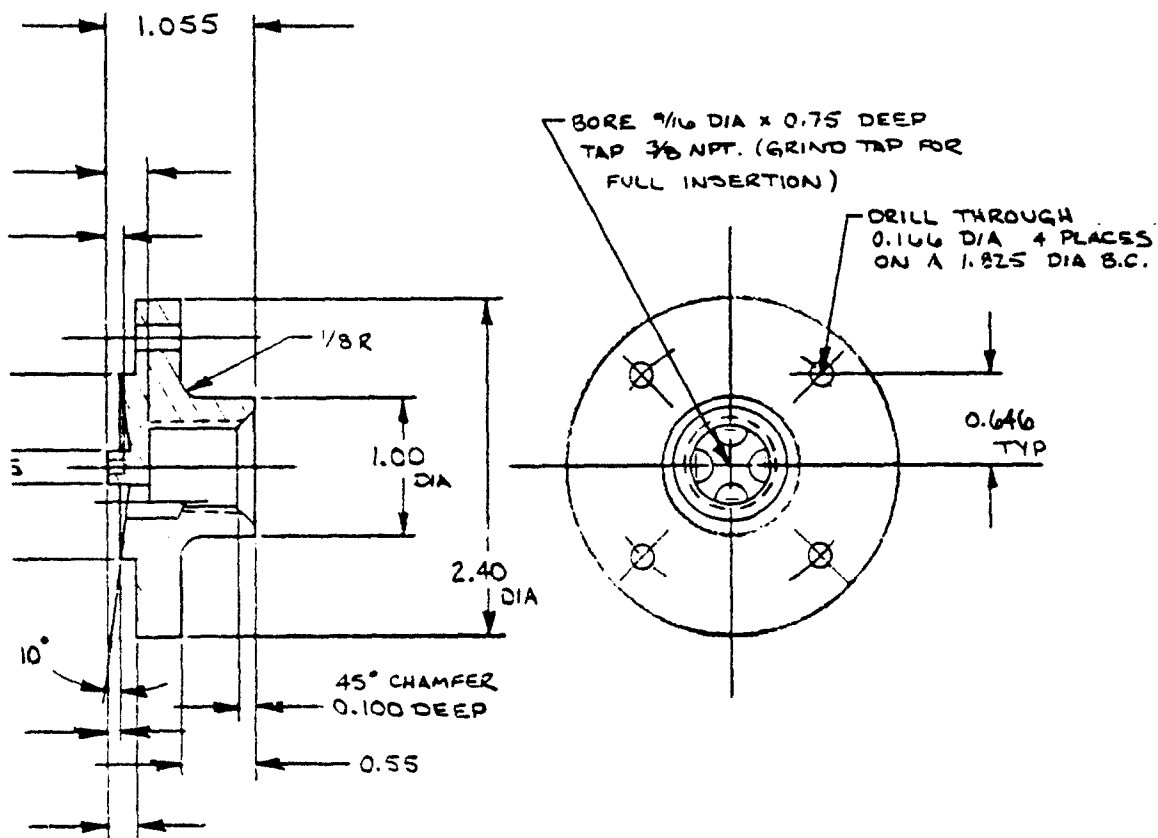




SECTION A-A HINGE DETAIL

CHAMBER DETAILS

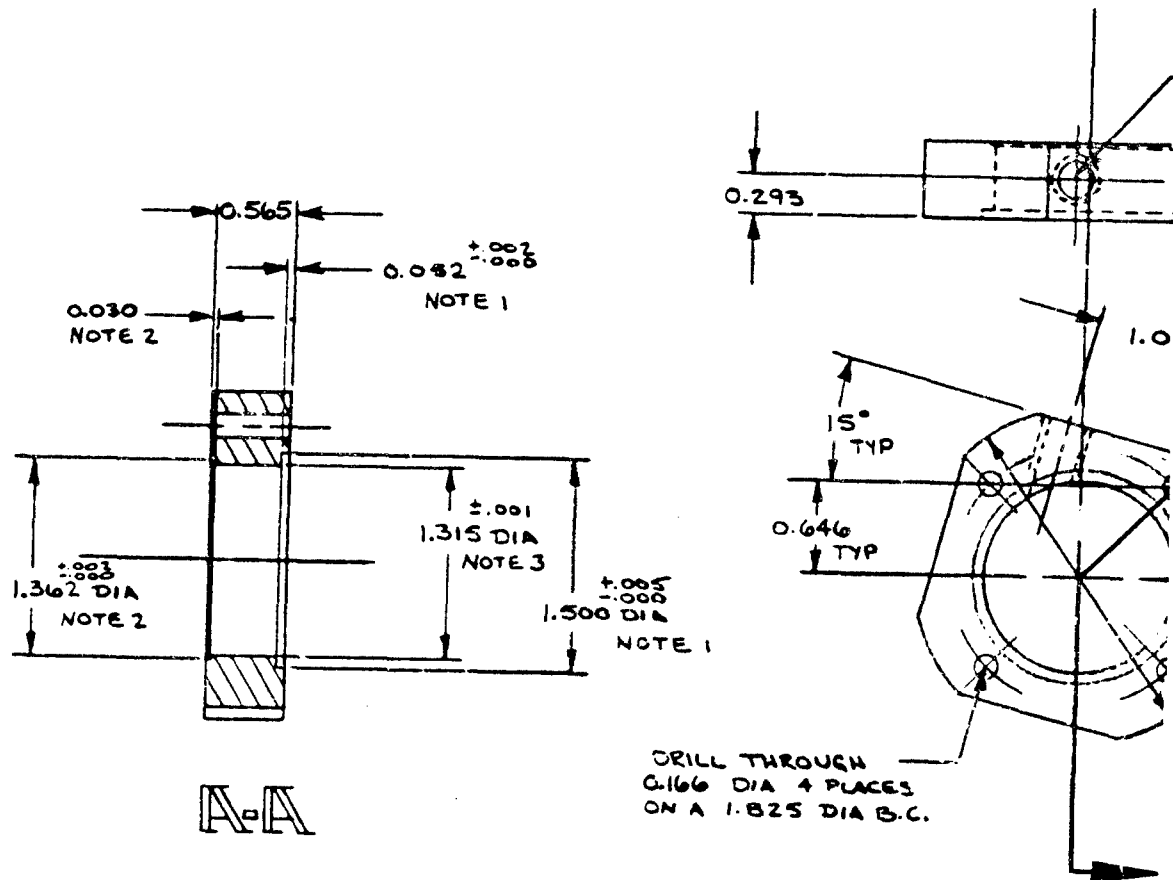
SCALE: FULL SIZE



MOULDER 2-027
 L SHARP EDGES
 L SURFACES

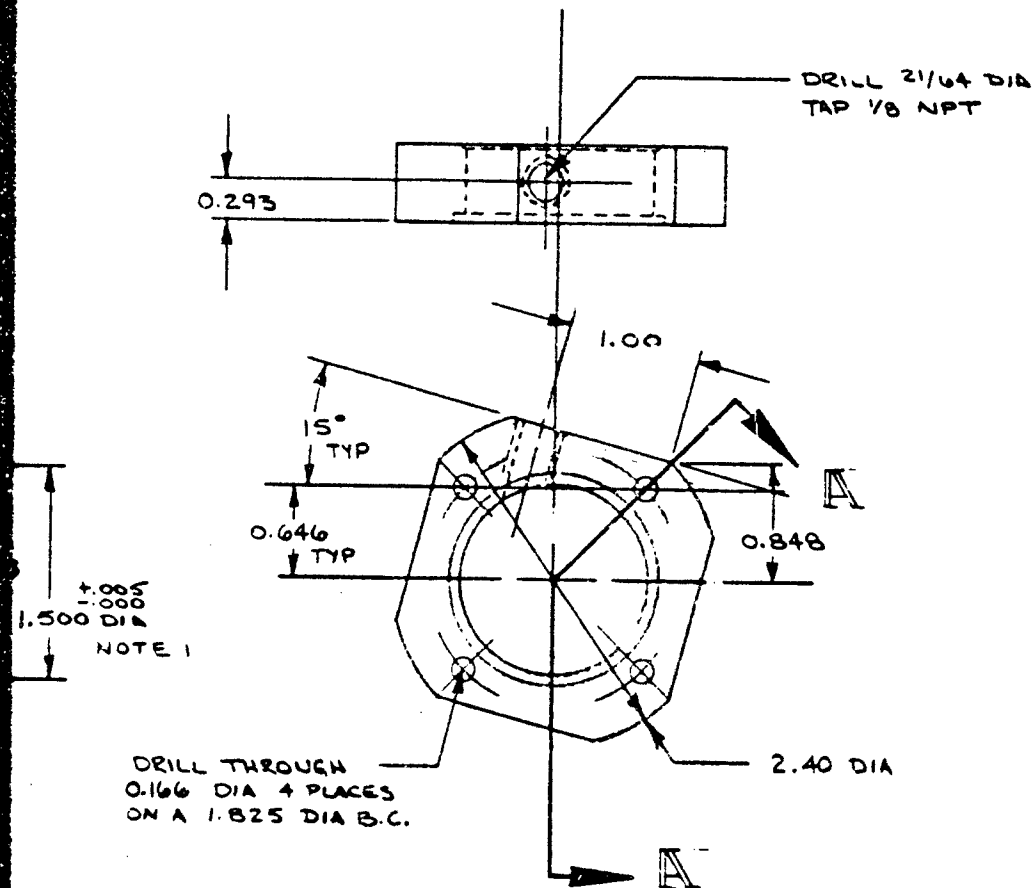
MAT'L
 POLYCARBONATE ROD

CDD			
FRONT BULKHEAD SUBMERSIBLE PUMP HEAD			DRW# CDD/NOBC B-001
DRW BY MBC	REV	DATE 6 JUN 89	SCALE 1:1



NOTE 2

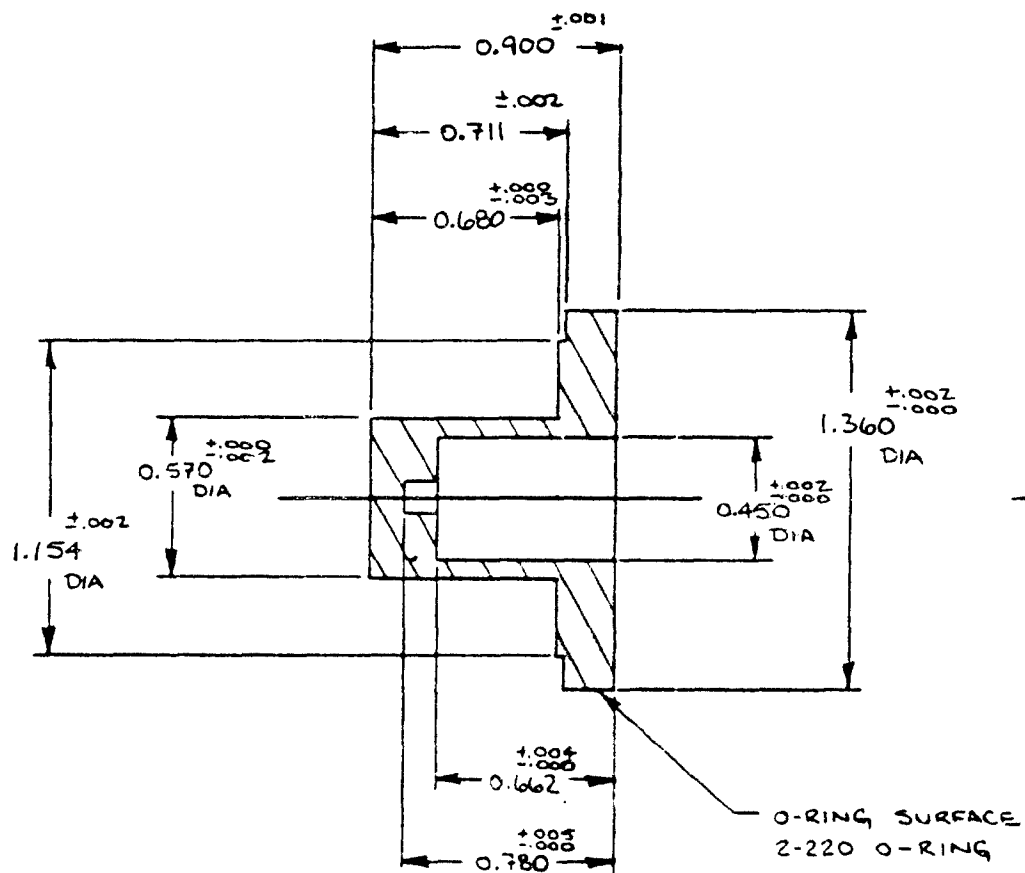
- 1) GROOVE FOR 2-027 O-RING SEAL
- 2) PILOT DIA FOR REAR BULKHEAD
- 3) PUMP BORE, POLISH THIS SURFACE
- 4) BREAK ALL SHARP EDGES
- 5) POLISH ALL SURFACES



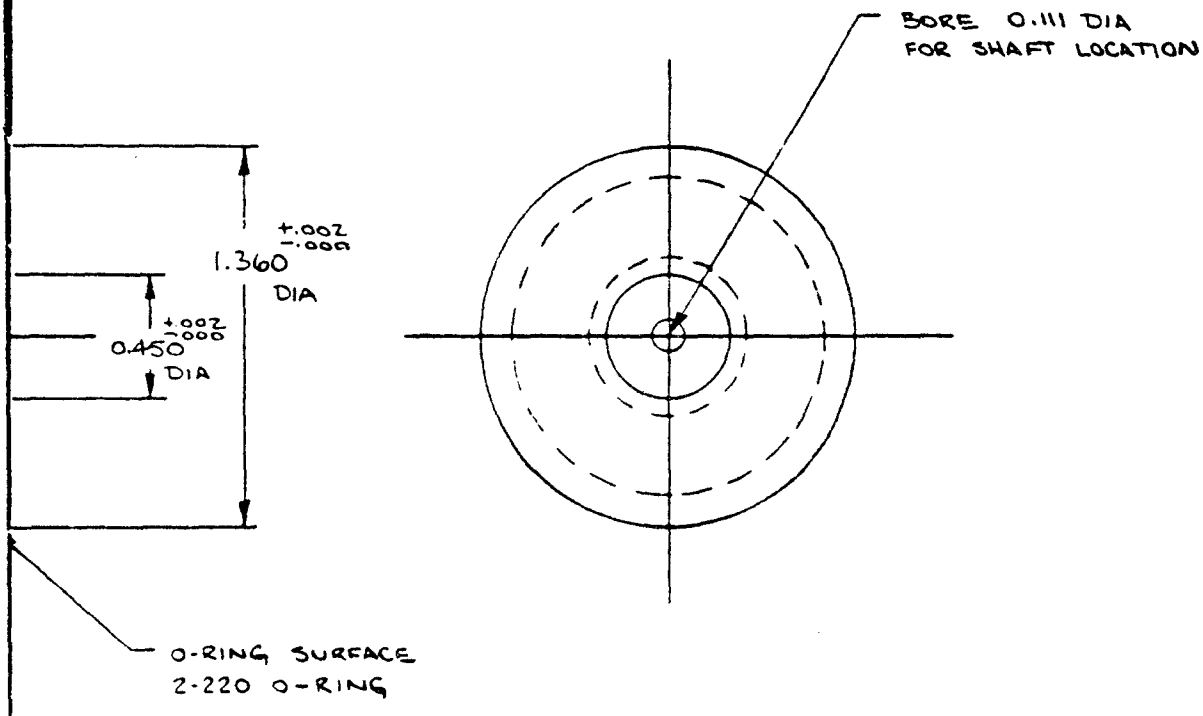
DR'FACE

MAT'L
2 1/2' POLYCARBONATE ROD
1 PER ASSEMBLY

CDD			
PUMP BODY SUBMERSIBLE PUMP HEAD			DESIGN # CDD/WOSC B-002
DESIGNED BY MBC	REV.	DATE 6 JUN 89	SCALE 1:1



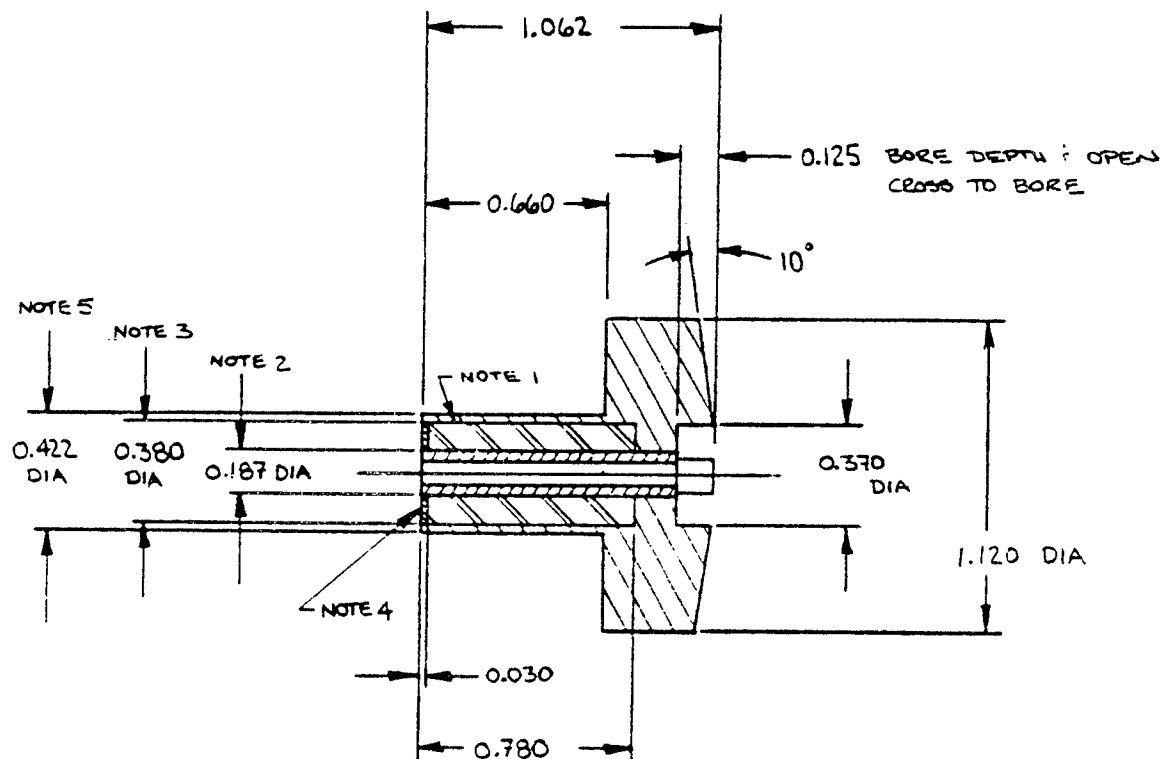
POLISH ALL SURFACES
BREAK ALL SHARP EDGES



LISH ALL SURFACES
EAK ALL SHARP EDGES

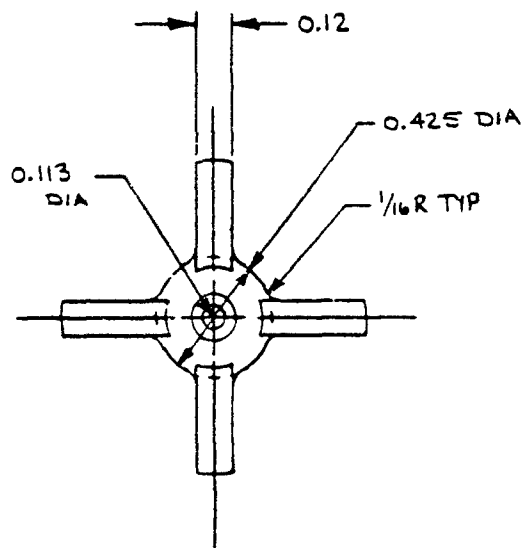
MAT'L
POLYCARBONATE ROD STOCK

CDD			
REAR BULKHEAD SUBMERSIBLE PUMP HEAD			DRW# CDD/NOBC B-003
DRW BY MSC	REV	DATE 6 JUN 89	SCALE 2:1



NOTE :

- 1) REMOVE MAGNET FROM MARCH MANUF. ROTOR #115-008.01
+ GRIND TO 0.380 OUTER DIAMETER
- 2) LIGHT PRESS FIT ONTO TEFLON JOURNAL WITH EPOXY
- 3) LIGHT PRESS FIT INTO IMPELLER BODY BLANK WITH EPOXY
- 4) EPOXY INTO PLACE A TEFLON WASHER TO COVER BACK END OF MAGNET
- 5) TURN IMPELLER BODY TO FINAL DIAMETER + MILL CROSS



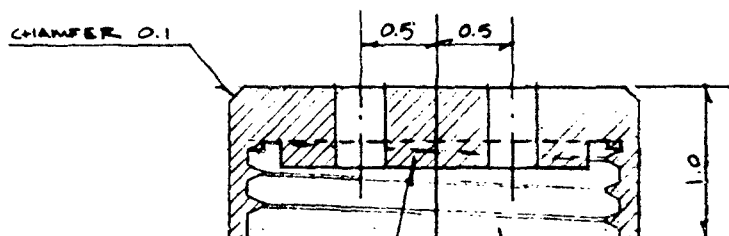
1 EPOXY
K WITH EPOXY
OVER BACK END OF MAGNET
R 1 MILL CROSS

CDD			DRW TH CDD/NO3C B-004
TEFLON ROTOR ASSEMBLY SUBMERSIBLE PUMP HEAD			
DRW BY MSC	REV	DATE 6 JUN 89	SCALE 2:1

SCALE: FULL SIZE

CAP DETAIL

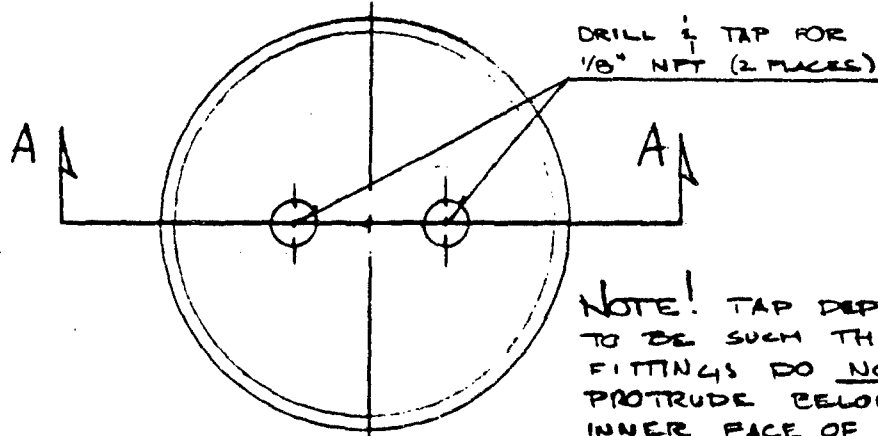
Date 4/10/90



CENTER TO BE SOLID
WHERE EXISTING LAP
IS RECESSED

INSIDE OF CAP INCLUDING
THREADS & O-RING GROOVE
TO BE SAME AS EXISTING CAP

SECTION A-A



TOP VIEW

MATERIAL: POLY CARBONATE

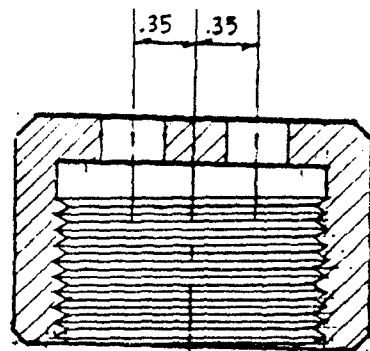
REQUIRED: 7

6/19/89

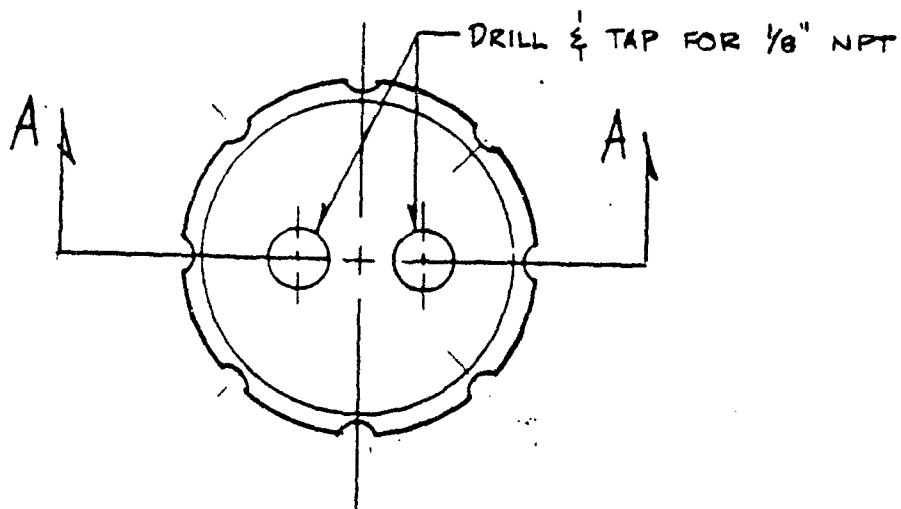
LAP DETAIL

SCALE: FULL SIZE

REQUIRED: 6

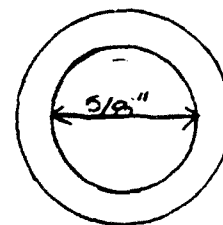


SECTION A-A



TOP VIEW

GAP DETAIL FOR GLASS BOTTLES

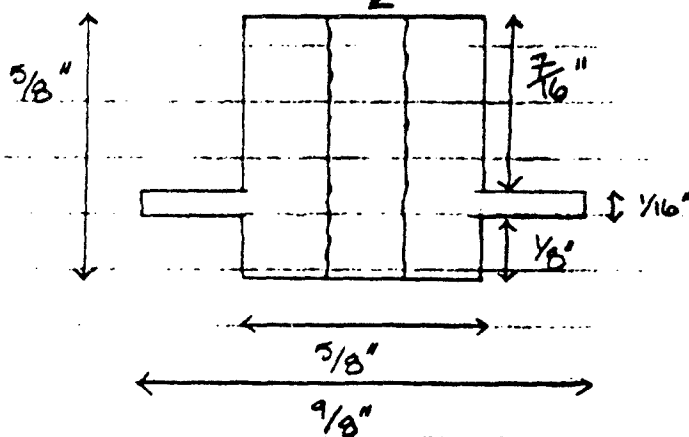


SIDE VIEW

TOP VIEW of Cap

TEFLON INSERT

Drill + Tap for 1/4" NPT



SIDE VIEW

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
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21a. NAME OF RESPONSIBLE INDIVIDUAL D. B. Chadwick	21b. TELEPHONE (include Area Code) (619) 553-5333	21c. OFFICE SYMBOL Code 522

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