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LITERATURE REVIEW AND SUMMARY REPORT: MEASURING SOFT SEDIMENT FLUX IN LAKE JESUP AND OTHER SHALLOW EUTROPHIC AND HYPEREUTROPHIC LAKES

LITERATURE REVIEW AND SUMMARY REPORT: MEASURING SOFT SEDIMENT FLUX IN LAKE JESUP AND OTHER SHALLOW EUTROPHIC AND HYPEREUTROPHIC LAKES

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ABSTRACT

Methods of measurement of nutrient flux from the sediment in aquatic environment are presented. The methods implemented by various researchers for measuring nutrient fluxes in sediment water-column interface are briefly described. The methods described include *in situ* benthic chambers, sediment-core incubations, pore water profiles, and pore water equilibration. A list of nutrient flux measurement case studies in shallow lakes is provided and a peer review of Lake Jesup nutrient flux study report is presented.

1.0 A REVIEW OF METHODS TO MEASURE NUTRIENT FLUX IN SEDIMENT WATER-COLUMN INTERFACE

1.1 BACKGROUND

Resuspension is an important aquatic process with regard to particle cycling and sedimentation, which has a great importance to limnological ecosystems (Bloesch, 1994). During resuspension, a particle can be exposed to biogeochemical processes and travel long distances and is, therefore, important in the cycling of nutrients and pollutants (Sanford, 1992). Resuspension affects a lake directly by increasing the amount of particles in the water column and indirectly by increasing the internal nutrient loading. Nutrient loading enhances phytoplankton growth which, in turn, increases the light attenuation. Wind, currents, and lake morphometry are the most important forces behind resuspension in shallow lakes (Bloesch, 1995). In such lakes, resuspension occurs frequently and over large areas. Bottom shear stress and cohesion of sediments play an important role in the resuspension process. Important factors that affect resuspension include sediment composition and macrophyte biomass (Schallenberg and Burns, 2004). Evans (1994) describes the resuspension theory and the relevant variables such as lake depth and wind stress, and the importance of the process to various water systems. Past studies, for example, Hilton *et al*. (1986) and Bengtsson *et al*. (1990), describe the basic processes and driving forces of resuspension and sediment transport in lakes. Bloesch (1994) describes various methods to measure sediment resuspension in lakes.

Flux is defined as the rate that fluid, chemicals, particles, or energy flow through a surface. There are several mechanisms which contribute to nutrient release and exchange in the bottom of lakes (D'Angelo and Reddy, 1994). Such mechanisms include resuspension, recycling, and diffusion. Resuspension is the remixing of sediment particles and pollutants back into the water by storms, currents, organisms, and human activities, such as dredging (EPA, 1994). Internal recycling represents the transport of dissolved chemical species across the solid-liquid interface at the bottom of aquatic systems (Kuwabara *et al*., 1999). Internal cycling of nutrients in the sediment and water column can be an important contribution to the total nutrient load of aquatic ecosystems (Malecki *et al*., 2004). Diffusion is the movement of individual molecules through a

material because of concentration gradients. Diffusion is an important mechanism for nutrient transport from the sediment to the water column (D'Angelo and Reddy, 1994).

The flux of solutes can be either positive (into the water column from the sediment) or negative (out of the water column into the sediment) and can vary over multiple temporal and spatial scales (Kuwabara *et al*., 1999). Nutrient concentrations gradients near the sediment-water interface may show water column concentration increasing near the sediment-water interface and continuing to increase in the sediment pore waters. This gradient can physically drive the release of dissolved chemical species from the sediment to the overlying water, generating a positive flux. During negative flux, the sediment consumes a substance, represented by dissolved oxygen where microbial respiration may create a sediment demand for oxygen.

This report presents commonly used methods to measure sediment resuspension and the methods for estimating nutrient fluxes. The primary focus of this report is to present the general approaches used in measuring nutrient flux between the sediment-water interfaces.

1.2 SEDIMENT RESUSPENSION MEASUREMENT METHODS

Sediment resuspension can be an important contributor to nutrient flux. Various methods of measuring sediment resuspension are provided in Table 1.

Table 1. Summary of Methodologies to Measure Sediment Resuspension

Method	Reference
Beam transmissometers and nephlometers	Pierson and Weyhenmeyer, 1994
Time-laps or video cameras	Davies, 1985
High frequency echosounders	Wright *et al*., 1986; Bokuniewicz *et al*., 1991
Infra-red sensors	Erlingsson, 1991

Method	Reference
Instantaneous multiple point water samplers	Rosa *et al*., 1983
Sediment traps	Rosa *et al*., 1983; Rosa, 1985; Hakanson *et al*., 1989; Bloesch, 1995
Radionuclides: Cs^{137} and Be^{7} dating	Cornett *et al*., 1994
Modeling	Lick, 1982; Aalderink *et al*., 1994; Evans and Hakason, 1992; Hakanson, 1994

1.3 NUTRIENT FLUX MEASUREMENT METHODS

The results of the literature review indicated there are four basic methods for measuring nutrient fluxes between the sediment–water interfaces. These methods include:

1. *In situ* benthic chambers (Miller-Way *et al*., 1994; Berelson *et al*., 1998; Forja and Gomez-Parra, 1998; Clavero *et al*., 2000; Fisher and Reddy, 2001).
2. Sediment-core incubations (Cowan *et al*., 1996; Moore *et al*., 1998; Fisher and Reddy, 2001; Qu *et al*., 2005).
3. Pore water equilibration (D'Angelo and Reddy, 1994; Moore *et al*., 1998; Fisher and Reddy, 2001).
4. Pore water profiles (Moore *et al*., 1998; Qu *et al*., 2005).

These methods are briefly described in the following sections.

1.3.1 BENTHIC CHAMBERS

Benthic chambers enclose an area of sediment *in situ* in a controlled volume of water. The variations in nutrient concentrations in the overlying water within the chamber are measured and used to infer nutrient flux.

Miller-Way *et al*. (1994) determined *in situ* rates of sediment oxygen demand and nutrient exchange using benthic chambers at a shallow site on the Louisiana continental shelf. The authors used benthic chambers that contained a water volume of 7 liters and covered an area of 0.09 square meter (m^2). A stirring motor circulated the water in each closed chamber system and maintained flow over an oxygen probe. Oxygen probes and meters were enclosed with a data logger in a glass sphere. The flux chambers were gently placed on the sediments by a SCUBA diver after being manually flushed with bottom water. Collars on the outside of each chamber assured a precise penetration depth into the sediment. The chambers were left in place for approximately 10 hours and water samples from the chambers were sampled at 0-, 4.3-, and 9.4-hours. Flux rates were calculated as the slope of the concentration differences between successive samplings and corrected for the volume of water and the areal coverage of each chamber.

Berelsen *et al*. (1998) measured nutrient fluxes in Port Phillip Bay, Australia, using benthic chambers. Benthic chambers captured approximately 7 liters of water in contact with 0.073 m^2 of sea bed. Three samples of 110 milliliters (mL) and three of 250 mL were removed during the incubation, which typically lasted 12 to 24 hours. The chambers were stirred by a rotating paddle. The oxygen concentration within the chamber was monitored by a pulsed electrode and readings were made every 6 minutes from within the chamber and from the ambient surrounding water. At each station, one or more chambers were lowered slowly by hand line from a research vessel to the sea floor. Visual observation by divers confirmed that the chambers were seated squarely in the mud. Analyses of chamber water samples were completed after filtering. Ammonium, nitrate, nitrite, phosphate, and silicate were analyzed. Sediment samples were recovered by diver-emplaced cores and the top 2 centimeters (cm) of a core was subsampled and incubated in a glass jar with 50 to 100 mL of bay water. Fluxes into and out of the chamber were calculated as the product of the slope of concentration versus incubation time and chamber height.

Forja and Gomez-Perra (1998) conducted a study in a temperate, shallow-water coastal ecosystem in the Bay of Cadiz in Spain. The authors measured the fluxes across the

sediment-water interface using benthic chambers installed on the bottom. Benthic chambers were constructed of opaque Plexiglas and were ellipsoid in shape, with a circular base which covered a sediment area of 0.385 m^2. The volume of each chamber varied between 59.1 and 89.8 liters. The choice of chamber size to be used at each site was made on the basis of the expected magnitude of the fluxes. Water sampling from inside the chamber was conducted continuously by means of a peristaltic pump situated on the bottom surface. At pre-set intervals, usually every 5 minutes, a fraction collector took a sample of 5 mL from the outflow for nutrient analysis. The observed changes in nutrient concentrations were used to estimate the nutrient flux.

Clavero *et al*. (2000) conducted *in situ* benthic flux experiments in the Palmones River Estuary, southern Spain. Authors measured net fluxes of dissolved phosphate, ammonium, and dissolved oxygen between the sediment and overlying water using three opaque PVC chambers (volume 30 liters, section 0.2 m^2) at low tide. Water samples (10 mL each for phosphate and ammonium) were collected at 20-minute intervals for 4 hours. At the same time, dissolved oxygen was measured. Water samples were carefully withdrawn from the chambers, avoiding the generation of turbidity using syringes. Samples were analyzed and the net dissolved oxygen, phosphate, and ammonium fluxes (J) across the sediment-water interface were calculated using:

$$J = MA^{-1}T^{-1} \text{ where } M = \sum V_t (C_t - C_{t-1})$$

Where: J = the oxygen, phosphate or ammonium flux (µmol m^{-2} d^{-1}).

V_t = the total volume of overlying water at time t in the chamber (in liters).

C_t and C_{t-1} = the dissolved oxygen, phosphate, or ammonium concentrations at times t and $t-1$, respectively, in the water (µmol l^{-1}) and A is the surface area of the sediment enclosed by the flux chamber (m^2).

Fisher and Reddy (2001) used benthic chambers in a study conducted in Water Conservation Area 2A in the northern Florida everglades. The authors placed benthic

chambers at the sediment–water interface to determine *in situ* fluxes of dissolved oxygen, nitrate, sulfate, and phosphorus. The chambers were constructed of 6.35-millimeters (mm) (0.25 inch)-thick acrylic and enclosed a sediment surface area of 0.5 m^2. Each of the chambers was equipped with a recirculation pump and a port for a dissolved oxygen electrode. The approximate water volume enclosed by the chamber was 80 liters.

Dissolved oxygen, nitrate, sulfate, temperature, and pH were measured for a period of 24 hours. Ambient temperature, dissolved oxygen, and pH were also recorded concurrent with the benthic chamber measurements. The recirculation pump was operated for approximately 1 minute prior to each oxygen determination in order to have sufficient water velocity for the oxygen electrode. While operating the pump, it was important not to disturb the bottom sediment in order to avoid sediment resuspension. Visual observations were made to ensure that sediment resuspension did not occur during pump operation. Phosphorus flux was calculated by determining the slope of the concentration versus time curve through linear regression, then multiplying by the flood water volume to soil surface area ratio:

$$J_i = \left(\frac{dC}{dt}\right)\left(\frac{V}{A}\right)$$

Where: J_i = flux of component *i* ($mg/m^2/day$).
C = component concentration in water (milligrams per liter [mg/L]).
V = water volume (liter).
A = sediment surface area (m^2).
t = time (days).

1.3.2 INTACT CORE INCUBATION

The second method for measuring nutrient flux is incubating sediment core and measuring the nutrients that are released over the incubation time. Sediment core incubations rely on careful sediment extraction, specific volume of overlying water, and nutrient concentration changes in the overlying water.

Cowan *et al*. (1996) measured sediment oxygen consumption and nutrient fluxes in Mobile Bay, Alabama, using three intact undisturbed sediment cores overlain with about 2,000 mL of water collected in cylindrical Plexiglas chambers. The cores were sealed air tight at both ends by Plexiglas plates. The top plate had one portal for sampling and replacement water tubing, and a floating Teflon stir bar mounted to it for gentle mixing of the overlying water. Approximately 90 liters of bottom-water were also collected in cubitainers. An additional flux chamber, filled with ambient bottom-water only, was used as a water-column control. Immediately prior to beginning flux measurements at the lab, bottom-water overlying the cores and in the water-column control was replaced with bottom-water from the cubitainers to ensure that water quality conditions at the start of the experiment closely resembled *in situ* conditions. To do so, tubing drained water from the cubitainers into the existing water in the cores at a rate of approximately 0.95 liters per minute for about 20 minutes, which was slow enough to prevent both aeration of the water and resuspension of the sediment. The cores were then placed in a darkened water-filled incubator maintained at ambient temperature by a circulating constant temperature bath, and the sampling tubes were inserted. The total time that elapsed between collection of the cores and the start of the incubation was between 3 and 4 hours.

Dissolved oxygen and dissolved inorganic nutrient samples were taken five times during a 6-hour period. As a sample was extracted, an equal amount of ambient bottom-water drained in through the replacement tube from a darkened insulated cubitainer. Less than 5 percent of the overlying water was replaced during the course of the incubation period. Nutrient samples were filtered and frozen for later analysis of ammonium, nitrate, nitrite, phosphate, and silicate concentrations. Dissolved oxygen samples were processed immediately. Oxygen and nutrient fluxes were estimated by calculating the mean rate of change in concentration during the incubation period by regression analysis.

Moore *et al*. (1998) conducted a study in Lake Okeechobee, Florida, to measure phosphorus flux from sediment using intact core incubation method. The authors utilized SCUBA divers to collect intact sediment cores of 6.35 cm internal diameter from eight

different locations in the lake using 1-meter-long Plexiglas tubes. The tubes were slowly inserted into the sediments, generally to a depth of 60 cm. At sites where the substrate was peaty or sandy, tubes were manually driven into sediments using a mallet. The tubes were then sealed at both ends with rubber stoppers. Upon return to the laboratory, depth of the overlying water in the cores was adjusted so that each core contained 1 L of lake water. The water column was aerated with aquarium pumps via Tygon tubing. The cores were wrapped in aluminum foil to exclude light and were incubated at 22degrees Celsius (°C). Flux measurements were made on three cores from each station for a total period of about 30 days. At 2- to 4-day intervals, a 20 mL sample was removed from the water column, filtered through a 0.45 micron (µm) membrane filter, acidified to pH 2.0 with sulfuric acid, and analyzed for dissolved reactive phosphorus (DRP). The anaerobic phosphorus flux from all eight stations was determined by purging the water column of previously aerobically incubated cores with nitrogen gas for about 15 days. During this period, water samples were obtained periodically, filtered, and analyzed as described above. At the end of the anaerobic period, the water column was purged with air for an additional 15 days.

Flux calculations were based on the increase in water column DRP of the cores incubated in the dark at 22°C. The amount of phosphorus removed during sampling was accounted for, as was the amount of phosphorus in the refill water. These gradients were then used to estimate diffusive phosphorus flux. Calculations were based on the linear portion of the curve (representing maximum slope) where the direction of the phosphorus exchange was constant. Dissolved phosphorus gradients were calculated from the sediment pore water phosphorus data using simple linear regression. These gradients were then used to estimate diffusive phosphorus flux. Calculations were based on the linear portion of the profile at the sediment/water interface. This gradient was then substituted into Fick's first law, which states that flux is proportional to the concentration gradient or,

$$J = -8.64x10^5 \varphi D_s \left(\frac{\delta C}{\delta z} \right)$$

Where:

J = diffusive flux (milligram per square meter per day ($mg/m^2/day$).

C = DRP concentration in water (microgram per cubic meter [$\mu g/cm^3$]).

φ = soil porosity.

D_s = whole-sediment diffusion coefficient (square centimeter per second (cm^2/s). Mass flux due to diffusion within the sediment particle is "whole-sediment diffusion coefficient (D_s)".

z = depth (cm).

$8.64x10^5$ = conversion factor.

Qu *et al*. (2005) collected intact sediment cores in Lake Illawarra, Australia with Plexiglas tubes (350 mm long, 70 mm wide) that are directly inserted into the sediment. The cores were sealed with a spherical valve and withdrawn from the surrounding sediment. A 200-mm-high water column was left above the sediment core in order to obtain a sufficient oxygen and nutrient reservoir. In the laboratory, the sediment cores were placed in an open tank filled with air-saturated lake water and maintained at temperatures similar to field conditions. About 100 liters of the lake water was aerated by bubbling with air, which had passed through an ammonium trap. A Teflon-coated stirring magnet was suspended 50 mm above the sediment surface. Momentum for rotation of the small magnets was provided by a large external magnet rotating at 60 revolutions per minute (rpm) to ensure a homogenous mixing of the water column.

Cores were equilibrated for at least 12 hours before flux determinations. Measurements of fluxes across the sediment–water interface were initiated by placing transparent polycarbonate lids on the sediment cores. Three replicate cores were incubated under either light or dark (double wrapped in aluminum foil) conditions for 1 to 4 hours,

depending on the oxygen consumption/production rates. Water samples were collected from the water column at regular time intervals during the incubation period.

Fluxes of ammonia, nitrogen dioxide, nitrate, and phosphorus across the sediment–water interface were calculated as:

$$J_i = \alpha\left(\frac{V}{A}\right)$$

Where: α = is the slope of regression line obtained by plotting the concentration of relevant species as a function of incubation time.

V = is the volume of the water column (liter).

A = is the sediment surface area (m^2).

1.3.3 PORE WATER EQUILIBRATION

A third method of measuring nutrient flux is the pore water equilibration method. The operation of a sampler in the pore water equilibration process is based on the equilibration of a contained quantity of water with the surrounding water through a dialysis membrane. The contained water is then removed from the system and container for analysis (Hesslein, 1976).

D'Angelo and Reddy (1994) used pore water equilibrators according to the methods of Hesslein (1976) to obtain ammonium and soluble phosphorus concentrations in the sediment-water column of Lake Apopka, located in central Florida. Pore water equilibrators consisted of Plexiglas sheets that have been machined to provide 8-cm^3 cells at 1-cm intervals. Equilibrators were 35- or 60-cm in length, and were placed above the native sediment surface. Prior to emplacing the equilibrators, all cells were filled with deoxygenated and deionized water and covered with a 0.2-µm polycarbonate membrane plus a protective 1-µm Nitex mesh cover. Equilibrators were then placed in Plexiglas storage containers containing deoxygenated and deionized water, and maintained under oxygen-free conditions until installed in the field.

Upon placement in the bottom soil, overlaying water and pore water constituents were allowed to equilibrate for a period of 2 weeks. Equilibrators then were removed from the sediment, rinsed with water, and 5-mL samples were immediately withdrawn from each cell with a syringe for transfer to sample vials. Samples were acidified and stored for a maximum of 2 weeks at 4^{o}C before analysis for selected nutrients (ammonium and soluble phosphorus).

Determination of diffusive flux of ammonium and soluble phosphorus utilized Fick's first law of diffusion modified for unconsolidated sediments:

$$J_i = -\varphi D_s \left(\frac{\delta C_i}{\delta z} \right) \approx -\varphi^3 D_o \left(\frac{\delta C_i}{\delta z} \right)$$

Where:

J_i = diffusive flux of component *i* (mass per unit area per unit time).

φ = soil porosity (volume water per volume soil).

D_s = whole-sediment diffusion coefficient (area per unit time).

D_o = free-solution diffusion coefficient (area per unit time). The diffusion in the solution between sediment particles is "free-solution diffusion coefficient (D_o)."

$\frac{\delta C_i}{\delta z}$ = concentration gradient of component *i* in the vertical direction (concentration per unit distance).

Pore water equilibration method has been used in other geographical regions. For example, Moore *et al*. (1998) determined phosphorus flux between sediment and overlying water in Lake Okeechobee. Fisher and Reddy (2001) conducted a study in the Water Conservation Area 2A in the northern Florida everglades to determine phosphorus flux.

1.3.4 PORE WATER PROFILES

The fourth method of determining the nutrient flux is by measuring pore water concentration gradients. Nutrient concentration gradients across the sediment–water interface can be obtained by sampling sediment pore waters at discrete depths. This concentration–depth data may then be used to calculate nutrient flux on the basis of Fick's law of diffusion (i.e., that flux is proportional to the slope of the concentration gradient). The concentration gradient can be obtained and calculated from the pore water chemistry within the sediment cores (nutrient profiles) using conventional techniques including *in situ* multiple sipper and coring/centrifugation (Kuwae *et al*., 1998), and a high resolution microelectrode (Mortimer *et al*., 1999).

Qu *et al*. (2005) collected intact sediment cores in Lake Illawarra, Australia with Plexiglas tubes as described in Section 1.3.2. In addition to using some of the cores for the incubation, one set of sediment was extruded from the cores and sliced into 2 cm segments in a nitrogen glove box. The samples were centrifuged to extract the pore water (5,000 rpm, 20 minutes) from each segment, which was then passed through 0.45 µm cellulose acetate filters into 30 ml polyethylene vials. The authors calculated the fluxes of solutes diffusing from sediments into overlying water from Fick's first law of diffusion:

$$J_s = -\varphi D_s \left(\frac{\delta C}{\delta z} \right)_{z=0}$$

Where:

J_s = flux of a solute with concentration C at depth z (millimole per square meter per hour [mmol $m^{-2}h^{-1}$]).

φ = porosity of the sediment (volume/volume).

D_s = whole-sediment diffusion coefficient (m^2/s).

$\left(\frac{\delta C}{\delta z} \right)_{z=0}$ = concentration gradient at the sediment–water interface (millimole per liter per meter [mmol L-1 m^{-1}]).

The whole-sediment diffusion coefficient was estimated using the molecular diffusion coefficient (D_o) after temperature correction and tortuosity.

$$D_s = \frac{D_o}{\left(1 - \ln(\varphi^2)\right)}$$

The porosity of the sediment was determined as

Porosity, φ = *ww* – *dw* / *vol*

Where: *ww* = the wet weight of sediment sample.

vol = the volume of sample (= *ww/d*).

d = density.

Dry weight (dw) is determined after drying the sediment at 105°C until constant weight.

The nutrient flux measurement methods and references used in this report are summarized in Table 2. The table also summarizes the results and sources of errors.

Table 2. Summary of Methodologies for Nutrient Flux Measurement

Method	Description	Study Area	Reference	Result(s)	Source(s) of Errors
Benthic Chambers	Benthic chambers enclose an area of sediment *in situ* in a controlled volume of water. The variations in nutrient concentrations in the overlying water within the chamber are measured and used to infer nutrient flux.	Louisiana continental shelf, USA	Miller-Way *et al*., 1994	*In situ* flux measurements were no different than remote methodologies.	• Disturbance during sampling • Prolonged incubation time • The uncertainty in the concentration vs time slope • Reduction of oxygen inside the chamber • Longer duration of measurement periods • The quality of organic matter • Electrode malfunctioning • Water circulation within the chamber
		Port Phillip Bay, Australia	Berelson *et al*., 1998	Most flux measurements at a given site were consistent within 50 percent for chambers deployed side-by-side and deployed 1 year apart.	
		The Bay of Cadiz, Spain	Forja and Gomez-Parra, 1998	Experimental data were used to validate the model prediction.	
		The Palmones River Estuary, Spain	Clavero *et al*., 2000	Diffusive nutrient fluxes measured from the pore water gradient were lower than those measured *in situ*, and nutrient fluxes obtained *in situ* were similar to values obtained by other researchers.	
		Everglades, USA	Fisher and Reddy, 2001	Phosphorus flux measured with the intact soil core and *in situ* benthic chambers were similar, and the water column redox conditions and water-level drawdown can significantly influence phosphorus flux to the water column.	

Table 2. Summary of Methodologies for Nutrient Flux Measurement

Method	Description	Study Area	Reference	Result(s)	Source(s) of Errors
Sediment Core Incubation	This method consists of incubating sediment core and measuring the nutrients that are released over the incubation time. Sediment core incubations rely on careful sediment extraction, specific volume of overlying water, and nutrient concentration changes in the overlying.	Mobile Bay, USA	Cowan *et al*., 1996	Nutrient release from the sediment depends on the availability of labile organic matter.	• Disturbance during sampling • Aeration of water • Resuspension of sediment • Electrode malfunctioning • Lab conditions not replicating lake bottoms • Variabilities in lab accuracies
		Lake Okeechobee, USA	Moore *et al*., 1998	Phosphorus fluxes from lake sediments were very sensitive to oxygen status of the overlying water, and anaerobic conditions resulting in high rates of phosphorus release.	
		Everglades, USA	Fisher and Reddy, 2001	Phosphorus flux measured with the intact soil core and *in situ* benthic chambers were similar.	
		Lake Illawarra, Australia	Qu *et al*., 2005	Measured nutrient fluxes using sediment core incubation were higher than the calculated diffusive fluxes.	
Pore Water Equilibration	The operation of a sampler in the pore water equilibration process is based on the equilibration of a contained quantity of water with the surrounding water through a dialysis membrane.	Lake Apopka, USA	D'Angelo and Reddy, 1994	Initial flooding of agricultural soil resulted in high nutrient concentration, diffusion was found to be the most important mechanism for transport of nutrients from the soil to the water column, and anaerobic conditions in both floc sediment and peat soil layers had significant effects on nutrient retention and release in the soil-water column.	• Disturbance during installation • Disturbance during sampling • Electrode malfunctioning • Water circulation within the chamber

Table 2. Summary of Methodologies for Nutrient Flux Measurement

Method	Description	Study Area	Reference	Result(s)	Source(s) of Errors
		Lake Okeechobee, USA	Moore *et al*., 1998	Phosphorus fluxes from lake sediments were very sensitive to oxygen status of the overlying water, and anaerobic conditions resulting in high rates of phosphorus release.	
		Everglades, USA	Fisher and Reddy, 2001	Flux measured with porewater equilibrators was approximately an order of magnitude lower than results observed from *in situ* benthic chambers and intact soil cores.	
<u>Pore Water Profiles</u>	In this method, nutrient concentration gradients across the sediment–water interface are obtained by sampling sediment pore waters at discrete depths. This concentration–depth data then is used to calculate nutrient flux on the basis of Fick's law of diffusion.	Lake Okeechobee, USA	Moore *et al*., 1998	Phosphorus fluxes from lake sediments were very sensitive to oxygen status of the overlying water, and anaerobic conditions resulting in high rates of phosphorus release.	• Disturbance during sampling • Disturbance during sampling
		Lake Illawarra, Australia	Qu *et al*., 2005	The diffusive fluxes determined from pore water profiles were generally lower than the measured nutrient fluxes using sediment core incubation.	

Source: ECT, 2007.

1.4 DISCUSSION

In each of the nutrient flux measurement methods, concentrations are measured. These concentrations are then converted to fluxes. Positive flux represents upward flux from sediment to water-column. Cumulative flux from entire lake system can be estimated by multiplying the average diffusive flux by the lake area (Malecki *et al*., 2004). As the fluxes are derived from the concentration gradients, these fluxes are "net" fluxes.

Despite the large number of studies in which benthic chambers have been used, no uniformity in their design has been achieved (Forja and Gomez-Parra, 1998). The shape, size, material of construction, method by which or degree to which the incubated water is agitated, and procedures used for chamber anchorage and to collect samples from them, differ sharply (Malan and McLachlan, 1991). Benthic chambers are time consuming and expensive to construct. By virtue of their high cost, they do not usually allow for assessment of a large number of sites. They also require logistically elaborate application with significant occupational hazard, which frequently requires installation by divers. Various forms of benthic chambers (rigid or flexible) have been used for *in situ* benthic nutrient flux measurements (Qu, 2004). Fluxes estimated by chambers include the effects of both diffusion and bioturbation; therefore this technique provides a more realistic approach than other techniques (Santschi et al., 1990). The results from chamber experiments have been proved to be very close representations of the natural fluxes by many experiments (Nicholson *et al*., 1999).

Due to the heterogeneous benthic environment in aquatic systems, high variabilities in flux measurements using incubated cores have often been reported in previous studies (Berelson, 1998; Eyre and Ferguson, 2002; Qu *et al*., 2003). Sediment core incubations do perturb the sediment system and hence introduce uncertainty; with care, however, they can provide cost-effective data at a large number of sites. The incubated core technique requires the recovery of intact or undisturbed cores, which can either be incubated in the laboratory in a bench mode or in a continuous flow-through system under *in situ* conditions (Elderfield *et al*., 1981).

In a comparative study using benthic chambers, intact soil cores, and pore water equilibrators to estimate phosphorus flux from the soil to the overlying water column, Fisher and Reddy (2001) reported that the phosphorus flux measured with the intact soil cores and the *in situ* benthic chambers gave similar results. Flux measured with the pore water equilibrators was approximately an order of magnitude lower than results observed from the other two techniques. Qu *et al*. (2005) reported no significant difference between the magnitudes of the nutrient fluxes estimated using incubated cores and pore water profile methods.

1.5 CONCLUSION

The primary methods of estimating nutrient fluxes between the sediment–water interfaces include *in situ* benthic chambers, sediment-core incubations, pore water profiles, and pore water equilibration. All of these methods use concentration gradients between the sediment and water-column to determine fluxes of nutrients.

Benthic chambers are considered the best method for estimating nutrient flux since they involve few assumptions and minimal perturbation to the sediment– water interface. The sediment core incubation method is relatively cheaper and less complex compared with the *in situ* benthic chamber technique, and incubation and experimental conditions can be easily regulated. Even though, pore water equilibrators are easy to construct and use, a previous study showed that the nutrient flux measured with these equipments was approximately an order of magnitude lower than results observed from benthic chambers and sediment core incubation techniques.

DISCLAIMER

Although ECT tried to be as complete as possible in reviewing sediment flux measurement techniques, it is always possible that other techniques exist. Any omission of such techniques is not intentional and neither ECT nor the SJRWMD endorse any method over another for commercial purposes.

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2.0 NUTRIENT FLUX: CASE STUDIES

Many past studies on nutrient flux from the sediments are reported for estuaries, and deep water lakes. However, there exists limited number of such studies on shallow eutrophic and hypereutrophic lakes in the US and elsewhere. A selected list of shallow lake nutrient flux studies are provided below.

1. Aquatic Bioassay and Consulting Laboratories, 2007. Lake Machado Nutrient Flux Study. A draft report submitted to The Southern California Coastal Water Research Project. [Available online at: http://www.swrcb.ca.gov/rwqcb4/html/meetings/tmdl/DominguezChannel/07_0712/ML_core%20Flux%20draft%20report.pdf]

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5. Noges, P, Jarvet, A., Tuvikene, L., and Noges, T. 1997. The Budgets of Nitrogen and Phosphorus in Shallow Eutrophic Lake Vortsjarv (Estonia). Hydrobiologia, 363:219-227.

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7. Qu, W., Morrison, R.J., West, R.J., and Su, C. 2005. Diagenetic Stoichiometry and Benthic Nutrient Fluxes at the Sediment–Water Interface of Lake Illawarra, Australia. Hydrobiologia, 537:249–264.

8. Reddy, K.R., Fisher, M.M., and Ivanoff, D. 1996. Resuspension and Diffusive Flux of Nitrogen and Phosphorus in a Hypereutrophic Lake. Journal of Environmental Quality, 25: 363-371.

9. Sen, S., Haggard, B., Chaubey, I., Brye, K., Costello, T., and Matlock, M. 2007. Sediment Phosphorus Release at Beaver Reservoir, Northwest Arkansas, USA, 2002-2003: A Preliminary Investigation. Water, Air, and Soil Pollution, 179:67-77.

10. Sondergaard, M., Kristensen, P., and Jeppesen, E. 1992. Phosphorus Release from Resuspended Sediment in a Shallow and Wind-Exposed Lake Arreso, Denmark. Hydrobiologia, 228: 91-99.

3.0 REVIEW AND CRITIQUE ON "LAKE JESUP, SEMINOLE COUNTY, FLORIDA: *IN SITU* ASSESSMENT OF SEDIMENT NUTRIENT FLUX ASSESSMENT AND SEDIMENT OXYGEN DEMAND"

$HydrO_2$, Inc. ($HydrO_2$) used the methodology described in Murphy and Hicks (1986) to assess sediment nutrient flux and sediment oxygen demand (SOD) for Lake Jesup in Seminole County, Florida. The Lake Jesup report presents field methodologies, sampling procedure, sediment nutrient flux and SOD rates based on the data collected in Lake Jesup during September 25-29, 2006.

A review of Lake Jesup report is summarized as follows:

- *In situ* sediment nutrient flux assessment using methodology developed by Murphy and Hicks (1986) appears to be a promising method for measuring SOD and nutrient fluxes in rivers (e.g., Rathbun *et al*., 1996; Rounds and Doyle, 1997); and in lakes (e.g., Kuwabara *et al*., 1999). Even though $HydrO_2$ stated the study objectives and approach, the methodology is not described in detail in the Lake Jesup report and is not clear.
- Three out of five sampling locations within Lake Jesup used *in situ* benthic chambers, whereas the other two locations used aluminum "tubes". Ratio of chamber volume to sediment area is different from the ratio of "tube" volume to sediment area. As these ratios are used for calculating nutrient fluxes within the chambers and the "tubes", flux estimation may be biased and the "tube" volumes were not presented.
- The sample extractions from the chambers and "tubes" were carried out at different heights above the sediment-water interface (i.e., 10 inches in chambers versus 20 to 23 inches in "tubes). If the samples were not properly mixed during the sampling, it may not represent appropriate nutrient concentrations in those samples. Chambers were not mechanically stirred. It is not clear in the report how samples were stirred in the "tubes" prior to extraction of samples. However, $HydrO_2$ was concerned that stirring or mixing the chamber would undermine their ability to accurately estimate sediment nutrient fluxes and SODs because of the flocculent material.

- It is not clearly mentioned as to how the summary of mean flux rates provided in Table 2 was derived. Even though the lake average flux rates for ammonia and dissolved phosphorus for Stations 1, 3, and 5 (where chambers were used) seems correctly presented, lake average of nutrient flux from all stations is misleading as it averages the fluxes determined from two different devices. A similar statement can be made for the nutrient flux to water column. The calculations were checked and appear to be correct. The "blank" values were subtracted from the test results prior to calculating he fluxes. Minor discrepancies were found because of roundoff error and because the exact volumes in the sediment "tubes" were not reported, so ECT used estimates of volumes to cross check the calculations.
- SOD data and regressions presented in Appendix A are appropriately presented.
- There is concern that in many cases (see Site No. 5) that the ammonia flux was positive and the total Kjeldahl nitrogen flux was negative. Since total Kjeldahl nitrogen equals ammonia plus organic nitrogen, it follows that increases in ammonia would result in an increase in total Kjeldahl nitrogen unless there are large changes in organic nitrogen.
- There is an excellent presentation on SOD, but little discussion on nutrient flux. In some cases, the volume of the chambers/"tubes" was not provided, so the calculations could not be checked.
- The difficulty of attempting these measurements within/through the flocculent layer is recognized and will probably introduce error. There is concern about the variability in some of the initial conditions. For example, looking at one of the worst cases (Site No. 2), the initial conditions for ammonia in the three replicates are 0.24, 0.06, and 0.21 mg/L. Since these are replicates, ideally the initial conditions would be similar. The maximum difference of 0.18 mg/L at the initiation of the test is greater than the maximum of 0.163 mg/L observed during the incubation period.
- The difficulty of attempting these measurements with the existing flocculent layer, the variability in results within replicates and between stations, and

the observation of both positive and negative fluxes of different nutrients at the same site illustrates the difficulty and reliability of the measurements. Further extrapolating the results over the entire lake within a variety of substrate types leads to questionable results.

- According to a previous study conducted in Lake Jesup by Gao (2005), the lake drains a watershed of about 136.4 mi^2 to the St. Johns River on the northeast side of the Middle St. Johns Basin. As the lake is low-lying, during lower local rainfall than regional rainfall (particularly to the south), the river rises, and water flows from the St. Johns River into the lake (Keesecker, 1992). Also, surface runoff discharges into Lake Jesup primarily through three tributaries—Howell Creek, Gee Creek, and Soldier Creek, which are located to the south and southwest of the lake. There are a number of potential sources of nutrients to Lake Jesup including ground water, tributaries, and the St. Johns River itself. On an annual average basis, about 128 and 16 tons of total nitrogen and total phosphorus are discharged into Lake Jesup through surface runoff, respectively. Similarly ground water contribution of total nitrogen and total phosphorus to Lake Jesup are approximately 4 and 8 tons annually (Gao, 2005). A comparison of known nutrient input values might have provided more insight to their results.
- The nutrient fluxes presented in the report are based on three benthic chambers (e.g., 15,675 pounds per day [lbs/day] for ammonia and 722 lbs/day for dissolved phosphorus) may not truly represent the overall lake flux because of the few numbers of sampling locations (three for a 25 mi^2 lake!). Also, flux measurements using "tube" methodology may not represent appropriate values. If the value of 15,675 lbs/day is correct, that would mean 5,721,375 pounds per year; what would this do to the concentration in the lake?

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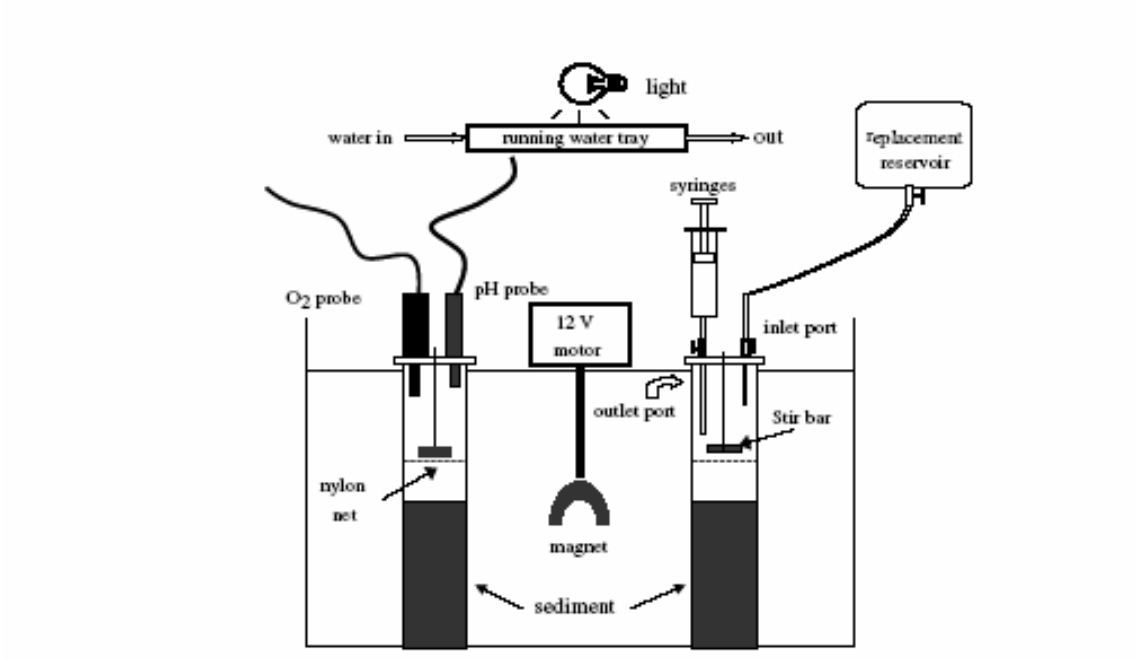
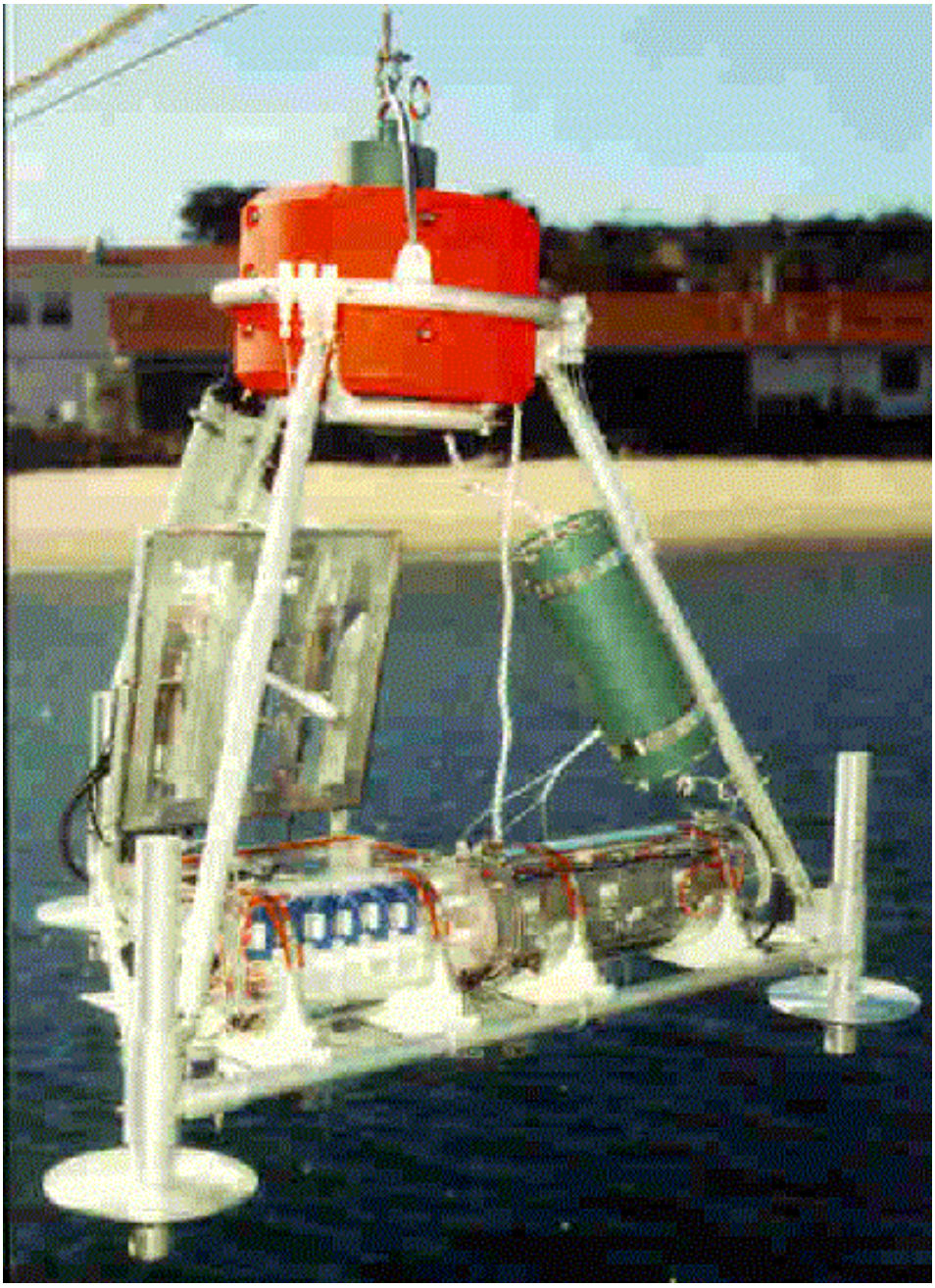
APPENDIX

SCHEMATICS AND PHOTOGRAPHS OF VARIOUS NUTRIENT FLUX AND SOD MEASURING DEVICES

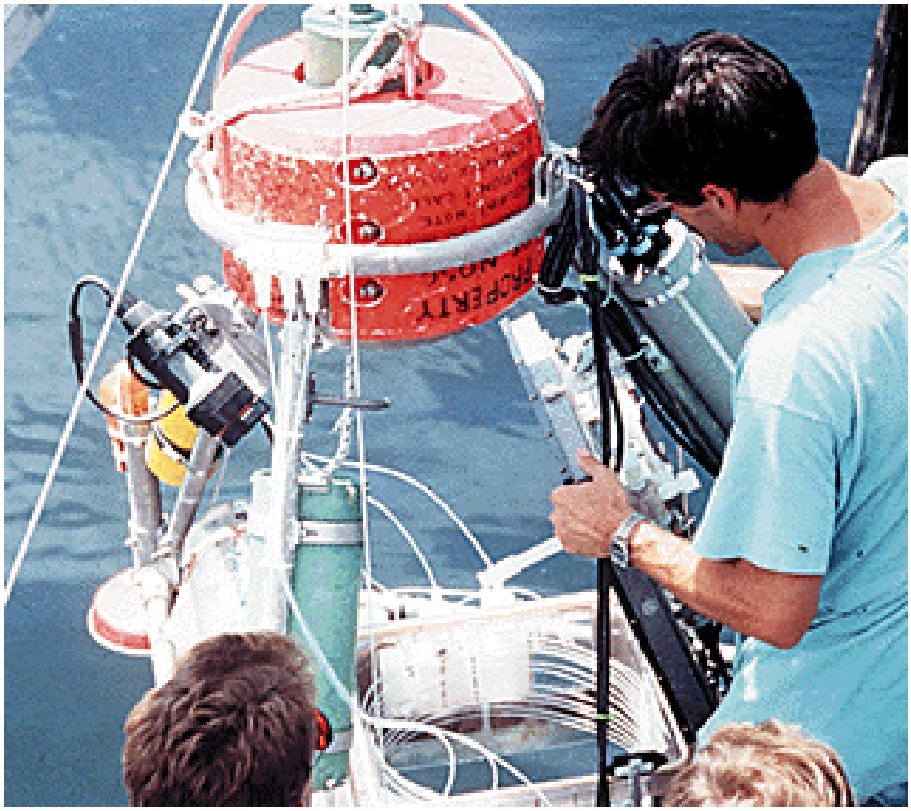


Schematic diagram of the batch incubation system for benthic flux measurement (Qu *et al*., 2005)



Benthic Flux Sampling Device

(Source: http://www.spawar.navy.mil/sti/publications/pubs/td/2790/index.html)



Deployment of the Benthic Flux Sampling Device at the Puget Sound Naval Shipyard (PSNS) in Sinclair Inlet, Washington

(Source: http://www.spawar.navy.mil/sti/publications/pubs/td/2790/index.html)

In situ Flux Chamber

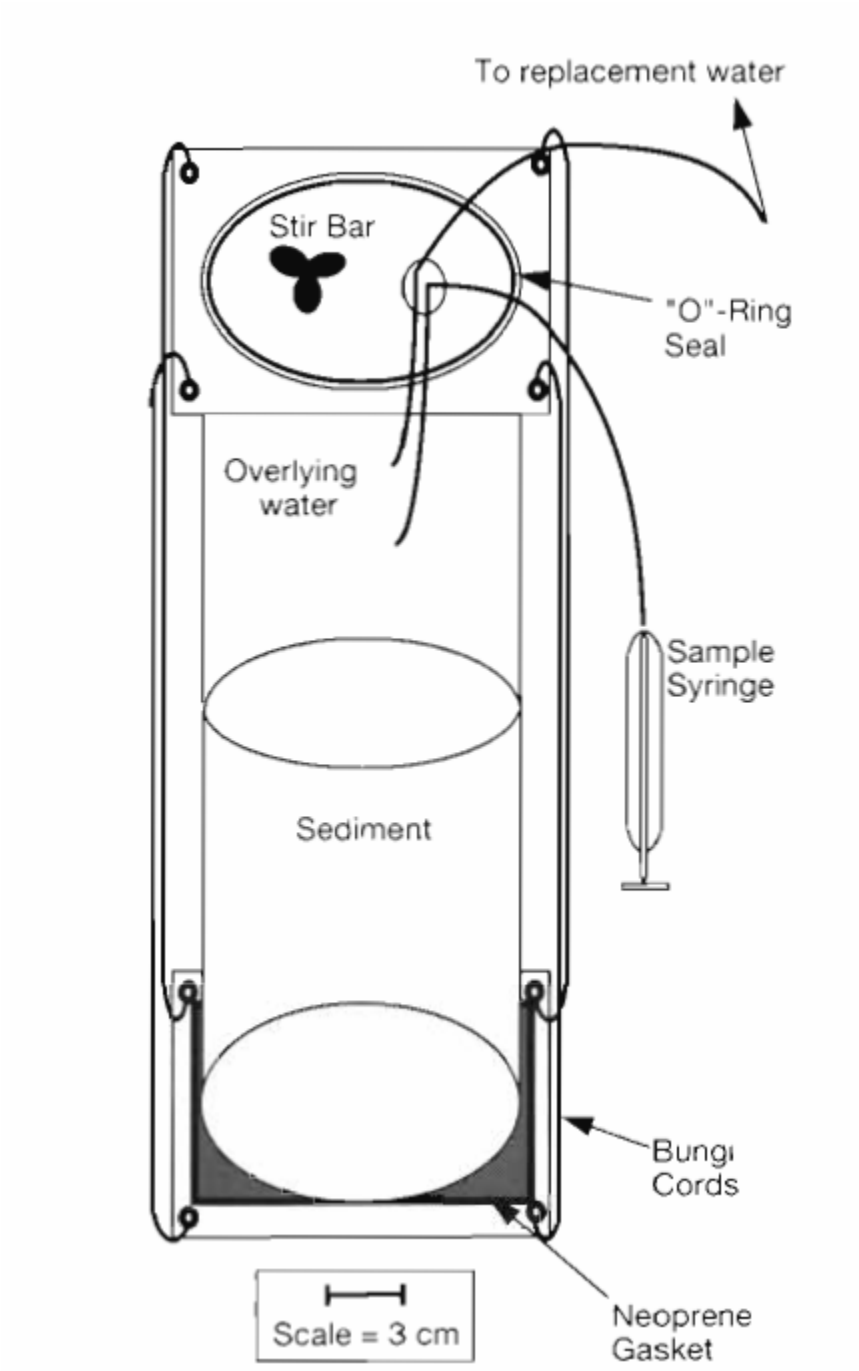
(Source: http://pubs.usgs.gov/wri/wri004132/pdf/WRIR-00-4132.pdf)

Incubation Core

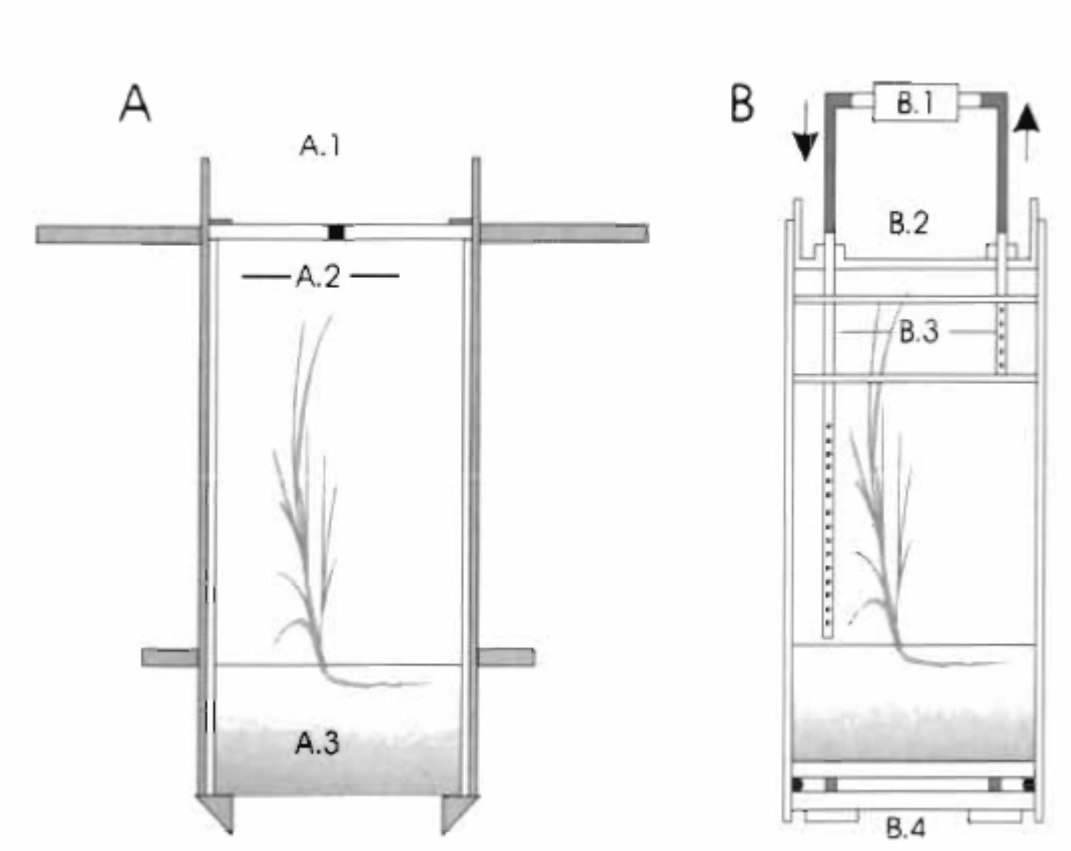
(Source: http://pubs.usgs.gov/wri/wri004132/pdf/WRIR-00-4132.pdf)

Coring Operation

(Source: http://pubs.usgs.gov/wri/wri004132/pdf/WRIR-00-4132.pdf)

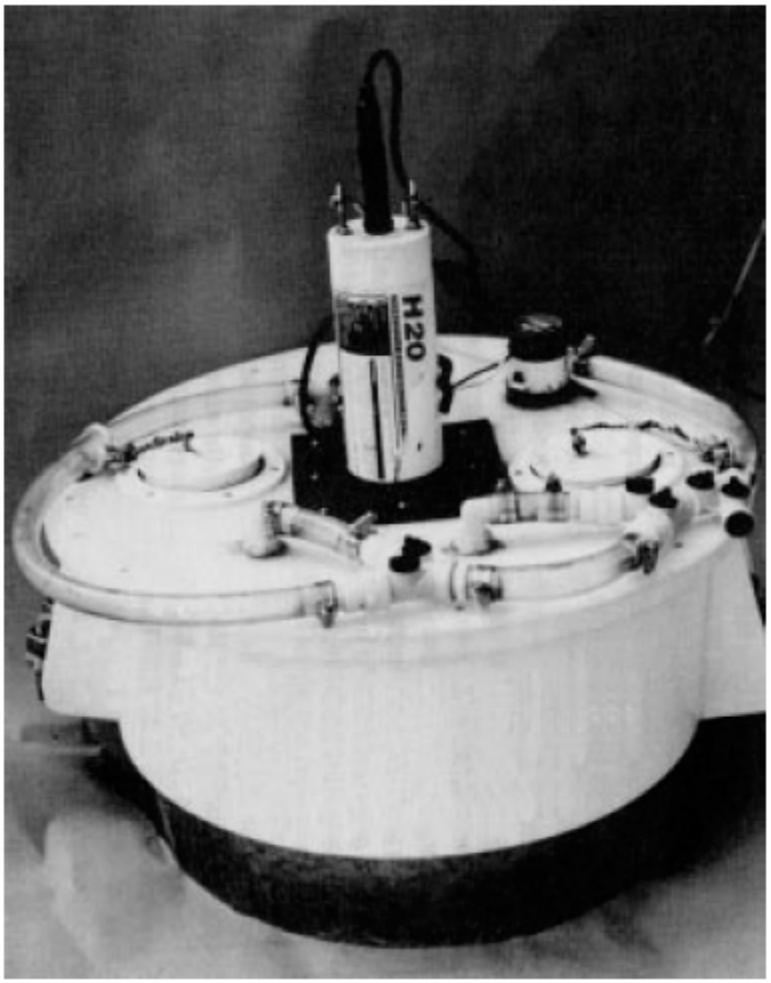


Schematic diagram of the incubation chamber used in making measurements of net sediment-water nutrient and oxygen fluxes (Cowan *et al*., 1996)

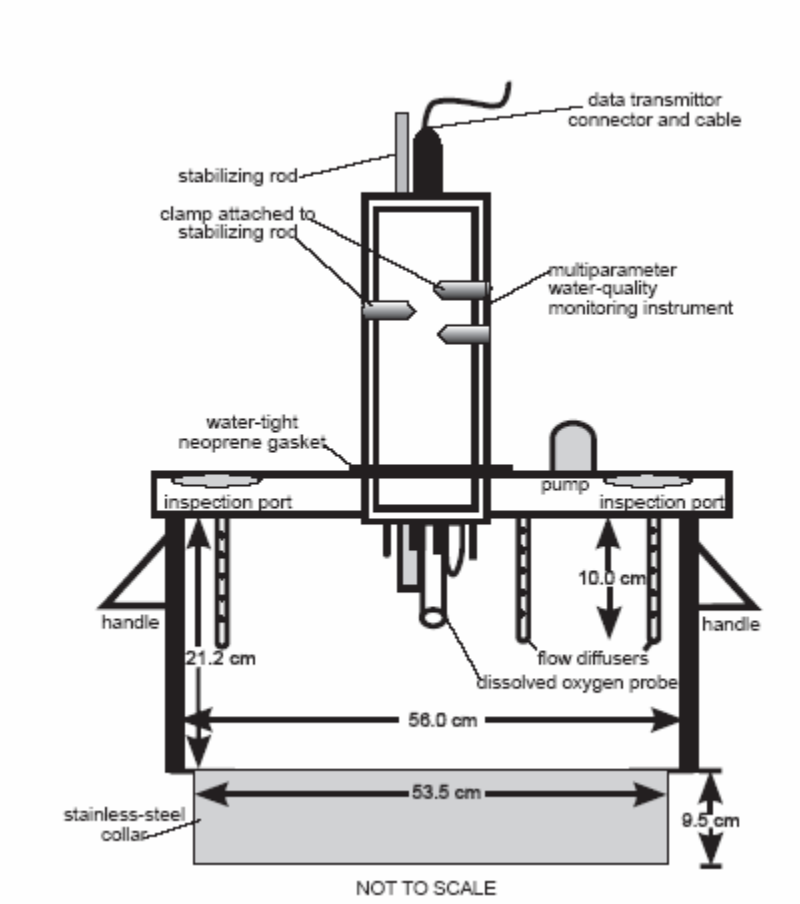


Core Sampler and Incubation System (Risgaard-Petersen and Ottosen, 2000)

(A) Core sampler: lid (A.1), Plexiglas tube (A.2), sediment core (A.3).
(B) Incubation system: water pump (B.1), transparent floating Plexiglas lid (B.2), perforated acrylic tubes (B.3), an elastic rubber ring squeezed between two PVC discs with bolts (B.4)

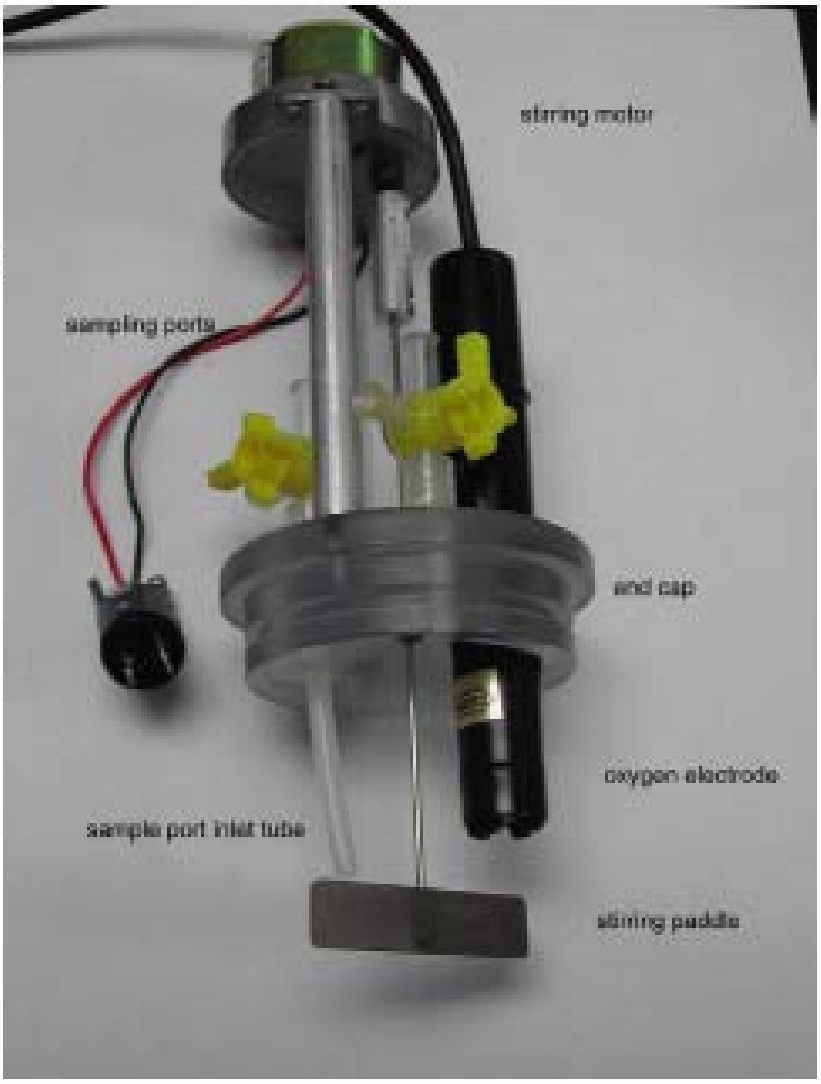


Sediment Oxygen Demand Chamber (Caldwell and Doyle, 1995)



Schematic of Sediment Oxygen Demand Chamber (Caldwell and Doyle, 1995)

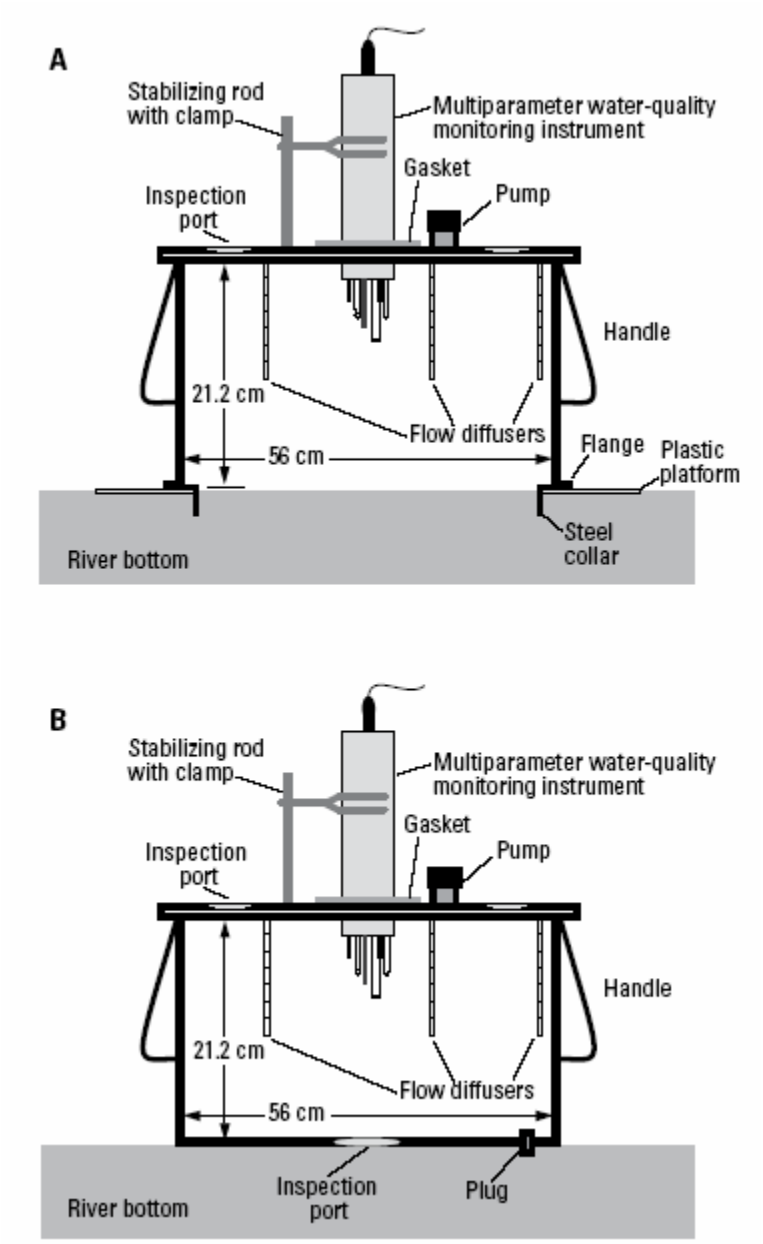
Core Incubator: end cap showing oxygen electrode, stirring motor, stirring paddle, sampling ports, and sample withdrawal tube (Val Klump *et al.*, 2004)



Core Incubator: core with end cap in place prior to initiation of experiment

(Val Klump *et al*., 2004)

Core Incubator: setup for simultaneous run of nine cores. Cores are covered with aluminum foil during incubation. Also shown are oxygen electrodes (Val Klump *et al*., 2004).



Sediment Oxygen Chamber: (A) sediment oxygen demand measurement chamber and (B) control chamber (Doyle and Lynch, 2005).

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