Guidance for Sediment Assessment in the State of Wisconsin

Wisconsin Department of Natural Resources
Bureau of Water Resources

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FORWARD

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THE WISCONSIN COASTAL MANAGEMENT PROGRAM, part of the Wisconsin Department of Administration, and overseen by the **WISCONSIN COASTAL MANAGEMENT COUNCIL**, was established in 1978 to preserve, protect and manage the resources of the Lake Michigan and Lake Superior coastline for this and future generations.

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I. INTRODUCTION

Contaminated sediments are a chronic and sometimes toxic problem affecting some of Wisconsin's inland lakes and streams and Great Lakes coastal areas, especially where industrial and other human activity has been most intense. Contaminated sediments can become a storage sink and possible resource for contaminants to be released to the bioactive environment. Suspended contaminated sediments can move through a water system to be deposited, sometimes over a wide area, in previously clean waters. Contaminants from sediments have been shown to move into the food chain, and thus be available for consumption by fish, wildlife and humans. Contaminants in sediments can also affect the base of the food chain by affecting existing resident biota communities.

Specific consequences of contaminated sediments in the state of Wisconsin include for example: contaminated fish and the need for advisories on the consumption of these fish; reduced vitality and diversity of species in an ecosystem; restrictions on dredging activities and costly handling of dredge spoils; expense to businesses and the taxpayers for the remediation of contaminated sites; unpleasant aesthetics and reduced recreational useability of some water bodies.

Although there are many adequate methods for sampling sediments, the intent of this guidance is to present the few most desirable methods to be used for sediment work performed for or by the Wisconsin Department of Natural Resources. The methods presented herein are, at this time, well standardized (i.e., EPA), reliable, effective, repeatable, and deemed most economical. Standardization of sediment assessment procedures should help increase the reliability and quality of the sediment data generated for Wisconsin waters, and will consequently benefit those who must use the data for decision making.

This guidance presents information generally in the order in which one would approach a sediment assessment project. However, later stages of a project including sampling and analysis must be collectively considered in the initial planning stage to insure that all conditions for the assessment and data quality will be met throughout the study. A well planned assessment is most likely to produce complete and reliable data.

II. PLANNING AND BACKGROUND INFORMATION

A. Planning a Sediment Survey

1. Scope

The goal of this section is to provide an understanding of the Department's sediment assessment activities as well as a very basic framework (including where to go for additional information) for designing and planning the types of sediment quality assessments most often carried out by the Department. In addition to what is presented here, much literature exists that describes in detail principles that should be followed during the planning of any enviromental study. This literature as well as Sediment Management and Remediation Techniques program (SMART Program) staff should be consulted when planning a sediment assessment project to assure a well planned and effective study that provides quality data.

2. Sediment Assessment Activities within WDNR

There are generally three types of Department activities under which sediment assessment studies are conducted by the Department:

a. *Condition monitoring* - Condition monitoring of sediments are conducted within the framework of the Department's annual water resources monitoring plan and basin assessment cycle whenever possible and appropriate. Monitoring strategy activities include: 1) the discovery of suspected and previously unknown contaminated sediment sites; 2) confirmation and definition of the extent of contamination and ecological and/or human health threat at contaminated sites; and 3) monitoring to evaluate effectiveness of a remediation project.

b. *Special projects linked to basin assessments* - These are special projects usually designed to fully evaluate the spatial and chemical extent of sediment contamination including the possible ecological and human health impacts. These projects are implemented through the annual surface water monitoring plan in conjunction with the basin assessment cycle.

c. *Special projects* - Any special project for research or other purposes that does not naturally fall within the auspices of the annual surface water monitoring plan and basin assessment cycle.

3. Principles of Study Design

"The specific design of a sediment assessment depends on the objectives of the study. Therefore, it is essential that the study objectives be sufficiently detailed to adequately guide a sediment toxicity evaluation (or invertebrate survey or chemical survey)." Because the study design governs the successfull outcome of the study, improper study design can nullify the best field collection, laboratory, and data analysis methods (EPA 1994).

There are basic aspects of any sediment assessment that need to be considered at the planning stage:

a. Study objectives
b. Statistical needs

c. Site selection
d. Sampling design (#s of samples, locations, replication, quantitative vs qualitative)
e. Quality assurance/quality control
f. Possible decisions or actions resulting from study
g. Safety plan

If these items are not considered during project design, the risk exists that the study data will not be of the highest quality possible and that the purpose of the study is not fully met.

The following are ten principles of study design taken directly from Green (1979). They describe the different aspects of study design that should and must be adhered to to assure a resultant quality data set that answers the questions posed in the study.

a. "Be able to state concisely to someone else what question you are asking. Your results will be as coherent and as comprehensible as your initial conception of the problem."

Seek statistical help during the design of the experiment, before any samples are taken. This also helps to ensure that all collected data is used to a meaningful end and that raw data is not collected without a purpose in mind. Saves money by not wasting field time and analysis dollars.

b. "Take replicate samples within each combination of time, location, and any other controlled variable. Differences among can only be demonstrated by comparison to differences within."

c. "Take an equal number of randomly allocated replicate samples for each combination of controlled variables. Putting samples in "representative" or "typical"places is not random sampling."

d. "To test whether a condition has an effect, collect samples both where the condition is present and where the condition is absent but all else is the same. An effect can only be demonstrated by comparison with a control."

e. "Carry out some preliminary sampling to provide a basis for evaluation of sampling design and statistical analysis options. Those who skip this step because they do not have enough time usually end up losing time."

f. "Verify that your sammpling device or method is sampling the population you think you are sampling, and with equal and adequate efficiency over the ectire range of sampling conditions to encountered. Variation in efficiency of sampling from area to area biases among-area comparisons."

g. "If the area to be subsampled has a large-scale environmental pattern, break the area up into relatively homogeneous subareas and allocate samples to each in proportion to the size of the subarea. If it is an esrimate of total abundance over the entire area that is desired, make the allocation proportional to the number of organisms in the subarea."

h. "Verify that your sample unit size is appropriate to the sizes, densitiesm and spatial distrributions of the organisms you are sampling. Then estimate the number of replicate samples required to obtain the precision you want."

i. "Test your data to determine whether the error variation is homogeneous, notmally distributed, and independent of the mean. If it is not, as will be the case for most field data, then (a) appropriately transform the data, (b) use a distribution-free (nonparametric) procedure, (c) use an appropriate sequential sampling design, or (d) test against simulated H_0 data."

j. "Having chosen the best statistical methods to test your hypothesis, stick with the result. An unexpected or undesired result isnot a valid reason for rejecting the method and hunting for a "better" one."

2. References

Baudo, R., Giesy, J., and H. Muntau, (Eds.). 1990. *Sediments: Chemistry and Toxicity of In-Place Pollutants*. Lewis Publishers, Boca Raton, FL.

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EPA. 1994. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates.

Green, Roger H. 1979. Sampling design and statistical methods for environmental biologists. John Wiley & Sons. New York. 257 pp.

Klemm, D.J., P.A. Lewis, F. Fulk, and J.M. Lazorchak. 1990. Macroinvertebrate field and laboratory methods for evaluating the biological integrity of surface waters. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH 45268. EPA/600/4-90/030.

WDNR. 1993 (draft). Field Procedures Manual. Office of Technical Services, Bureau of Water Resources Management.

WDNR. 1990 (draft). Quality Assurance Guidance for Inplace Pollutant Monitoring Activities. Unpublished document on file at Office of Technical Services, Bureau of Water Resources Management.

B. Reviewing Background Information

1. Scope

A review of existing background information from all reasonably available sources for a site or study area should be the first step in collecting data for a sediment quality assessment. The information obtained in a review of a site's historical (industrial and other uses) and existing sediment data costs relatively little and can provide information about the likelihood and types of contamination that may be present. Historical information can help guide study plans and may reduce the amount of field work and analysis needed to accomplish information goals. Background information should be used to help make study design decisions about, for example, site selection, target contaminants, and level of necessary preliminary sampling effort.

This section attempts to discuss the types of information that should and can be sought for a site background review, and lists suggested sources for obtaining historical uses and more recent sediment contaminant and other data information.

2. Types of information

a. Historical Information - Historical information is useful in trying to find out what permanent contaminants may have been introduced to the water body historically, and can indicate specific contaminants that should be targeted either in a preliminary or full scale sediment survey.

1) Land use - agricultural, industrial, residential, recreational

2) Water usage - industrial, municipal wastewater treatment plants, power plants, municipal water intakes, shipping

3) Dredging activity

4) River, lake, harbor morphology and bathymetry

b. Recent information - < 10 years old

1) Precise description of designated uses

2) Quantity and quality of potential and known inputs:

a) Point sources - locations of outfalls from industrial discharges, storm sewers, etc.

b) Non-point sources

3) Any previous sampling and chemical analysis data

4) Sediment (bathymetric) maps - Many harbors have up-to-date bathymetric maps of the harbor area. The local harbor authority, U.S. Army Corps of Engineers (USACE), U.S. Coast Guard, or National Oceanic and Atmospheric Association (NOAA) should be able to provide that information.

3. Factors affecting contaminant source pathways

1) bathymetry

2) Water current patterns

3) tributary flows

4) watershed hydrology and land uses

5) sediment and soil types

6) sediment deposition rates

4. Information Sources

1) WDNR Case files (WDNR - field offices and all Bureaus) - these are files containing information and reports on previous and ongoing DNR projects. EPA studies and information about superfund and RCRA sites may also be contained in the WDNR case files.

2) Wisconsin DNR published and unpublished reports - some studies are carried out by the WDNR that is written down in report form, but never formally published. These reports may contain valuable information about sediment sites.

3) WDNR Sediment and Fish Contaminant Database (under development). For more information, contact Jim Amhrein at (608) 266-0164 or Linda Talbot at (608) 266-8148 in Water Resources.

4) Local government or academic related research: Fish and Wildlife Service in Green Bay and LaCrosse. EPA-Duluth. University of Wisconsin campuses: UW-LaCrosse River Studies Center, UW-Green Bay, UW-Superior, UW-Milwaukee, UW-Stevens Point, UW-Madison departments of water chemistry and limnology. Wisconsin State Laboratory of Hygiene in Madison.

5) STORET - is a database maintained by WDNR and EPA to store and make available data on many water quality parameters, including contaminant concentrations in sediment and fish. For more information, contact Carol Tiegs in Water Resources at (608) 267-7659.

6) Published scientific research - A search of the published literature (mostly journals) should be conducted for any research that has been conducted at the study site. The WDNR library is very helpful in a literature search, and given the appropriate information will conduct the search and help obtain the literature for you.

7) WDNR Remedial Action Projects (RAPs), basin plans, etc...

8) NPDES and WPDES permit records

9) Selected chemical spill reporting system (EPA) - Information is available from the WDNR Spill Response Coordinator for each district and directly from the EPA.

10) Pesticide spill reporting system (EPA)

11) Report of pollution caused fish kills (EPA) - Information available from WDNR Water Resources Staff (Joe Ball-WR\2) and district/area Fish Managers

12) Pollution incident reporting system (U.S. Coast Guard)

13) Identification of In-Place Pollutants and Priorities for removal (EPA)

14) Hazardous waste sites and Management facilities reports (EPA)

15) USACE studies of sediment pollution and sediments

16) Water and sediment data on major tributaries (U.S. Geological Survey)

5. References

Report to the Great Lakes Water Quality Board. 1985. "Guidance on characterization of toxic substances problems in areas of concern in the Great Lakes Basin".

WDNR - Water Resources Water Quality Operations Handbook

EPA/USACE. 1994. Evaluation of dredged material proposed for discharge in waters of the U.S. - Testing Manual (draft). Office of Water, Washington D.C. EPA-823-B-94-002.

III. SAFETY

1. Scope

The reason for managing contaminated sediments is to protect living things (including humans) from the risks of exposure to sediment borne contaminants. It would be counter productive for the people who must physically handle the sediment to put themselves at unnecessary risks by not attempting to protect themselves from avoidable exposure to contaminants in the sediments. A Standard Operating Procedures (SOP) draft document for sediment sampling follows this introduction. This document is still in the draft stage and will be revised in the future. It is highly recommended that all personnel involved in sediment sampling use common sense in limiting exposures to contaminated sediments since the contaminants in sediments and the resultant short and long term health effects from exposure are often unknown.

In addition to the dangers of chemical exposure during sediment handling, safety precautions to prevent injury while in the field should be followed from the Safety SOPs related to open water monitoring and boating. These Safety SOPs should be available from the Department Safety Office and the District Water Resource Managers.

The following Sediment Safety SOP includes procedures and cautions to be taken under different circumstances. The safety procedures written may be too rigid for many sites that would be encountered in Wisconsin and may be too lax for some contaminated sites in Wisconsin. The point is to use your best judgement and always try to err on the side of caution or overprotection. It is important to report any exposure, especially if anyone experiences <u>any</u> symptoms during or after the field work or sediment exposure. This is partly for the benefit of the staff person doing the field work, but also so the DNR Safety office can begin to gather information about contaminated site exposures and resultant health problems. The information you give the Safety office may be helpful to other staff in the future and can be used to decide on the level of safety equipment needed to work at a particular site.

Many of the recommendations in this SOP regarding sediment exposure are derived from EPA safety regulations and are less rigorous than what is required of EPA staff. Therefore, the strongest recommendation to follow is to minimize exposure to contaminants in the sediment and water whenever possible.

Sediment Sampling and Monitoring
Standard Safety Operating Procedures

Bureau of Water Resources Management

Objective: Sample sediments or a component of sediments from contaminated or unknown sites.

Physical: Setting: Open water, ice covered water body, wetlands, rivers and streams, near dams, urban or city waterways.

Possible Hazards: Boating related accidents; thin ice; equipment related injuries; extreme weather exposure; exposure to known and unknown hazardous chemicals or infectious agents through direct contact, inhalation and/or ingestion of contaminated sediment or water.

Prohibited Activities: Eating, drinking or smoking is prohibited while at a site or without taking proper sanitary precautions after leaving a site; see related SOPs.

Site Safety Plan:

Projects of any duration where more than two people are involved in the sampling effort and the sites are known to be contaminated should have a set of project specific safety guidelines written down and passed on to all staff involved. (These guidelines need not be formal, just informative. This is to make sure everyone involved has thought about and understands any site related safety issues.) The written guidelines should contain information about:

1) Contaminants suspected or known to exist at the site
2) Protective gear necessary for each major task while sampling (taking notes may not require the same gear as pulling cores)
3) A list of the personnel on project with specialized safety training (to be consulted by other staff if questions arise)
4) A list of any other known hazards of the site.

Recommended Training: Depending on circumstances and site: Forty-hour (EPA) or other personal safety and hazardous materials training is recommended for at least one member of sampling team (see references below); First aid; CPR.

Equipment: Protective clothing suitable to the site and weather conditions and type of sampling effort. These should include: sturdy gloves of adequate length; rubber boots, hip boots or waders; protective clothing such as rain gear or coveralls to prevent skin contact or contamination of personal clothing; and goggles or face shield. means to wash skin in case of contact; First aid kit including a first aid guide booklet (M.C. 9182.2); area emergency phone numbers.

Support Staff: For help with specific project safety plans, the department safety office should be consulted at: (608) 267-4580 in Madison.

Medical Monitoring: Refer to M.C.9180.7on Employee Medical Surveillance. Baseline and yearly if personnel make significant contact (greater than 30 field days/year at known contaminated sites) with contaminated sediment/water. All incidents of contact with contaminated sediments that produce symptoms should be reported to a supervisor and to the Department Safety Office.

Related SOPs: Sampling from boat; Extreme weather hazards; Monitoring during ice cover; Open water monitoring; Personnel safety.

Procedures to Follow:

Before going into the field

1) Review site location and any contamination or discharger history, or prior sampling efforts to familiarize yourself with possible contaminants present.

2) Review other safety SOPs applicable to your sampling effort.

3) Make sure you know how to operate all equipment before getting to the field. This will increase sampling accuracy as well as safety.

4) Assume an unknown site to be contaminated unless you have good reason (i.e., chemical evidence) to believe otherwise, and plan accordingly.

5) Be aware of where the nearest emergency medical facility is to sampling sites. Just knowing this detail could save time in case of an emergency.

6) Carry a phone credit card and/or mobile phone while travelling or in the field.

While sampling at a site:

1) Protect yourself (skin, eyes) from exposure to contaminated sediment, water by wearing adequate protective clothing. Sturdy (and chemical resistant as contaminants dictate) gloves should be worn anytime you will be working with sediments of known or unknown contamination. Waders or rubber boots, rain pants and rain coat or coveralls protect the wearer's clothing and skin from exposure. Goggles or a face shield should be worn if splashing could occur while sampling. Shorts are not recommended for sediment sampling at contaminated sites unless covered with protective clothing.

2) Clean all equipment and protective clothing as well as possible before leaving the site or as soon as possible. This will protect the field vehicle from becoming contaminated and will help prevent cross-contamination between sites and samples; especially if you are collecting samples for chemical analysis.

3) Carry waterproof first aid kit including: eye wash, wash soap and clean water to site. Any sediment splashed on the skin or in eyes should be washed immediately or as soon as possible, but do not scrub the skin hard as this may increase absorption into the skin. Certain sediment contaminants can produce a skin rash within minutes (especially if exposed to the sun) while others (some possible carcinogens) may be absorbed through the skin unnoticed by the field person.

4) Break for clean-up and lunch in a non-contaminated area. Do not eat, drink or smoke while at a contaminated site or without proper clean-up after leaving the site.

5) No sampling effort is worth risking your health! If a site is obviously contaminated, and produces adverse symptoms such as headache, nausea, lightheadedness or lung, skin or eye irritation, leave the site immediately and do not return until proper steps are taken to insure the site can be sampled safely by staff.

6) Although Water Resources staff will rarely encounter a site so contaminated as to pose a real inhalation hazard, respirators may be necessary at some hazardous sites with volatile chemicals. Respirator training is <u>required</u> to assure proper respirator selection and use. The DNR Safety Office in Madison (see below) has information about training for use of respirators.

7) Maintain awareness of potential physical and chemical hazards of a site.

8) Shower as soon as possible at the end of the day.

<u>Handling contaminated samples and chemicals used in sampling</u>

1) Adequately label all samples and chemicals.

2) Excess sediment should be disposed of at the site where it came from.

3) Used chemicals and chemical containers should be properly stored and carried from site for proper disposal. (Refer to SOP for Chemical Handling).

4) Alert the laboratory receiving your samples of any suspected or known "high level" contamination in the samples, either by calling ahead if the samples are sent by mail (so they know about it before they open it), or as an attached note describing the contaminants as best possible.

Further information about maintaining safety at a site or for a particular project can be obtained by contacting the Department Safety Office in Madison (PS/G3) at (608) 267-4580.

IV. Field Procedures

A. Field Positioning Methods

1. Scope:

In order to effectively implement a sediment assessment, it is necessary to be able to accurately describe the field position of a site as well as accurately relocate the site for further studies. The precision necessary in site location depends on the goals of the activity being conducted at the site. Sediment deposits are often heterogeneous in composition and contaminant loading and can cover as little as a few square feet to as much as a many square miles. Many sediment "sites" are sampled or monitored over a period of time, and require being relocated several times, sometimes by different people. This section describes some methods for finding and recording accurate location descriptions for sediment sites including the degree of accuracy each method can deliver.

2. Finding and recording site positions using maps

a. Maps

1) U.S. Geological Survey (USGS) 7.5 minute topographic maps. Scale = 1/24,000. Any point or line on the map is accurate to $\pm$40 feet. These maps show sections, townships, ranges, latitude, longitude, roads, buildings and other permanent structures such as water and radio towers. Be aware of the date of the latest revision for any particular map. Maps covering the entire state of Wisconsin are available at the DNR library in Central Office.

2) National Oceanographic and Atmospheric Administration (NOAA) Navigational Charts. These are charts of navigable waterways, and so would only be available for larger water bodies such as the Great Lakes and major rivers. Scale varies depending on the chart. They are generally available from a map store or marina near the selected water body or directly from NOAA.

3) Army Corps of Engineers - Provides project maps for commercial navigation (dredging projects). Maps are available for many or most harbors and ports. Maps are highly detailed and very large scale, often containing good bathymetry information.

4) County plat maps - Shows ownership land boundaries and roads.

5) Federal Emergency Management Administration maps (FEMA) - These are maps created and used for planning in flood plain areas.

b. Public Land Survey System - section, township, range

The section, township, range system for describing locations and parcels of land can be used and is often requested (STORET) as a site position description. Townships are approximately six miles by six miles square and labeled by township numbers running north and south and range numbers running east and west. Each township is divided into 36 numbered sections, each approximately one mile by one mile square. Smaller than one mile square parcels of land are described using 1/4 sections labeled NE, NW, SW, and SE. Each 1/4 section ($\sim$160 acres) can be further divided into quarters (= 1/16 of a section) and so on. Detailed descriptions of this system can be found in many Land Atlas and Plat Books available for Wisconsin counties.

This information, along with latitude and longitude, is requested and should be given when establishing or looking for a STORET station (see *STORET stations* in section IV.C.3.).

c. Latitude/Longitude

Latitude and longitude can be measured electronically (see below) or read from a map with the aid of a ruler and pencil. Accuracy and precision will depend on the scale and accuracy of the map as well as the skills of the map reader. Latitude and longitude readings to 0.1 second are requested when establishing or looking for an existing STORET station number (see *STORET stations* in section IV.C.3.).as well as for entering sampling points into the GIS system.

3. Equipment and methods for obtaining or finding a precise location

a. Compass and rope

A compass and rope can be used to locate a point fairly accurately by measuring the distance and bearing from an arbitrary permanent landmark on shore. Accuracy depends on conditions, instruments, and the skills of the field persons, but can be as good as within one foot under ideal conditions. Generally, a landmark is chosen on shore that is visible and reachable from the water. Then, the distance and compass bearing from the landmark to the site is measured with rope and/or tape measure and a compass. This method becomes more difficult as sites are located farther from shore.

b. Surveying Equipment

Sediment mapping team staff at Central Office can assist in the location and instructions on use of surveying equipment for site location and sediment mapping purposes. For information or help, call the Planning Section in the Bureau of Water Resources Management (Dale Patterson at (608) 266-0155).

c. Electronic location devices

1) Loran-C - For a full description and procedures for use, refer to the "Field Procedures Manual" (WDNR, 1993).

2) Global Positioning System (GPS)

a) Hand held Magellan GPS units are used in some districts, and are typically accurate within a 30 meter radius of the point location, and will provide a longitude and latitude reading to 0.1 second. Location data are generated on the spot and can be collected with only one person.

b) A differential GPS system can be used to obtain sub-meter accuracy. Two units and two people are necessary. Position data from the two units may need to be adjusted after the readings are taken to obtain the true and accurate position.

3) Surveying Equipment - See b. Surveying equipment above.

B. Field Observations and Measurements

1. Recording field observations

Field notes are an important and sometimes critical addition to the data collected during a sediment assessment study. These written notes are used to record exact locations, measurements, dates, times, events and conditions encountered during field work that could affect the results of sampling effort and analysis. They should be recorded in a field notebook with waterproof ink or pencil in addition to field sheets or directly on field sheets that are maintained together so none can be lost. The field information is important both for people looking at the data who are not familiar with the sites, as well as those who know the sites. Nobody can remember all specific details about a field trip and sites. The written information also provides a picture in time or historical record, and may prove to be very useful later on to help interpret results. Specific information that should or might be recorded includes:

1) *Site and sample locations* with enough detail to be able to relocate the sampled site (see section **IV.A.Field Positioning Methods**). Information may include major waterbody and county, nearby bridges and roads, GPS or Loran-C readings, compass readings to permanent landmarks (triangulation), distances from shore and relation to permanent landmarks, a hand drawn map of the immediate area.

2) *Date and time* each sample was taken.

3) *Types of samples* being taken and for what purpose.

4) *Observations for each sample* including: depth of sediment sample, water depth, sediment texture, odor, color, presence/absence of invertebrates, presence of detritus.

5) *Observations for each site* including: flow direction and velocity; shade; submerged macrophytes; shoreline proximity and characteristics; debris such as wood chips, coal pieces, detritus, etc.

6) *Field measurements* - see **2. Field Measurements** below.

7) *Equipment* being used.

8) *Name(s) of collector(s).*

9) Any other detailed information that is important for understanding or interpreting the data.

Notetaking can be time consuming, but too little information is a far worse situation than too much information.

2. Field measurements

Field measurements should be taken in the field whenever sediments are being sampled. The project plan and type of sampling will influence which field measurements are necessary to complete the data for each site. Generally, if samples are collected only for bulk chemistry analysis, sediment depth and site location are probably sufficient. Any sampling for a biologically based test or survey such as invertebrate community, toxicity or bioaccumulation should include additional field measurements. These measurements may include: water depth, pH, water temperature (at depth and surface), dissolved oxygen, light attenuation, turbidity, conductivity and flow. Measurements may need to be taken close to the sediment surface, at the water surface or mid-depth, depending on the study.

All meters should be calibrated and tested following the manufacturers' instructions prior to departure into the field so faults can be discovered and fixed before the meter is needed. Write down dates of calibrations and tests performed on instruments in your field notebook so field measurements are not questioned later.

For detailed instructions on taking field measurements and use of appropriate equipment, refer to the following sections in the Field Procedures Manual (WDNR, 1993).

2001 FIELD MEASUREMENT - pH METERS
2101 DISSOLVED OXYGEN METERS
2102 WINKLER TITRATION FOR DISSOLVED OXYGEN
2201 FIELD MEASUREMENT - CONDUCTIVITY METERS
2301 OPEN CHANNEL FLOW MEASUREMENT
2501 TEMPERATURE - THERMOMETRIC
2502 TEMPERATURE - ELECTRONIC
2802 LIGHT ATTENUATION, SECCHI DISK

3.

4. References

WDNR. 1993 (draft). Field Procedures Manual. Office of Technical Services, Bureau of Water Resources Management.

WDNR. 1990 (draft). Quality Assurance Guidance for Inplace Pollutant Monitoring Activities. Unpublished document on file at Office of Technical Services, Bureau of Water Resources Management.

C. General Sediment Sampling Equipment and Procedures

1. Scope:

The goal of this section is to describe proper general procedures for sediment sampling and the use of common sediment sampling equipment. Sound sediment sampling techniques that are followed for all sampling efforts throughout the state will improve the quality of data received from sediment surveys in the State of Wisconsin. Effective and proper use and cleaning of sampling equipment is important to the safety of field staff and quality assurance and control of samples. Study goals may require that additional or alternate equipment or procedures be used than are discussed here. Any procedure changes should be based on sound scientific and practical reasons and should ultimately help further the goals of the study, without the loss of quality assurance and control.

2. Equipment and Supplies:

Below is a suggested list of equipment needed for most sediment sampling efforts. This list suggests equipment that may be necessary for your project and should not be considered exhaustive. Equipment that is specific to a specialized type of sampling may be included only in the section describing the particular type of sampling.

a. Equipment Checklist

1) Boat, anchor, motor, gas tank, tow vehicle
2) Extra vehicle keys
3) Protective clothing: boots, waders, gloves, rain gear, etc.
4) First aid kit
5) Mobile phone
6) Credit card for gas and emergencies
7) Maps: road and site maps
8) Compass and measuring equipment
9) Electronic location device (Loran or GPS)
10) Field notebook and field sheets
11) Waterproof pens and pencils
12) Field measurement equipment (temperature, dissolved oxygen, etc.)
13) Sample containers
14) Sample labelling tape or paper and permanent marker
15) Sediment pole for measuring depth
16) Coring device and dredge or grab with adequate rope and extension poles (grab is backup for corer in sandy sediments), including extension poles.
17) Slide hammer for corer
18) Pliers, wrenches, etc. for adjusting equipment
19) Mixing bowl and spoon
20) Cleaning (decontamination) supplies (non-ionic detergent, tub, brushes, etc.)
21) Wash bottles
22) Ice chest and ice for cooling samples
23) Extra rope
24) Extra sense of humor in case yours or your coworkers gets lost.

b. Equipment suitability for chemical analysis:

All equipment or sample containers that will come into contact with a sediment sample for chemical analysis should be constructed of materials that will not affect the concentration of contaminants in the sediment sample. In general, sediment samples to be analyzed for metals should not touch metallic surfaces (other than stainless steel), and samples for organic analysis should not contact materials that can react with organic substances. The level of care that needs to be taken with the materials used will depend on the level and types of contaminants associated with the sediment and the quality assurance needs and study goals.

1) For **organic analysis**, equipment and containers should be constructed of: *glass, teflon, polycarbonate, nylon, aluminum, galvanized steel, stainless steel or porcelain* (WDNR 1990). *Acrylic* core tubes are also acceptable for almost all sampling needs.

2) For **inorganic analysis**, equipment and sample containers should be constructed of: *glass, teflon, polyethylene polycarbonate,* (WDNR 1990), *stainless steel* or *acrylic*.

3. Basic Sediment Sampling Procedures

a. Preparation

Sampling Plan - Sampling strategy decisions should be made well before going into the field, and should be designed to collect quality data that will best answer the questions or meet the goals of the study or monitoring program. Decisions should be made ahead of time about what sites, how many replicates at each site (sampling strategy), and what chemical analyses will performed on the samples (see section **II.A. Planning a Sediment Survey** and WDNR 1990). This will help ensure that appropriate and quality samples are collected.

STORET stations - Every site that is sampled in Wisconsin should have a STORET station number assigned to it so that chemical data about the site will automatically be added into the STORET database. After site locations are decided upon, each site should be checked to see if a STORET station number exists for it. This number should continue to be used if the sites are really the same. A STORET number must be assigned (before samples are sent to the lab for processing) to each new site in a project. Location information including section, township, range and longitude and latitude will be needed to establish the station (see **IV.A. Field Positioning Methods**). To establish a STORET station, call Carol Tiegs at (608) 267-7659.

Field Staff - All field staff working at a site should understand the basic goals of the study the samples are for and the basic methods to be used to assure that quality samples are collected.

Safety - All field staff should be aware of and fully understand the possible physical and chemical safety hazards posed by any site they will be working at. Precautions should be taken to prevent exposure to contaminated sediments (see **III. Safety**).

Equipment - Make all the preparations necessary to obtain suitable collecting equipment, protective clothing, vehicle and boat if necessary. Test and calibrate any equipment (according to manufacturers instructions) that will be used to take field measurements, etc. This way, a malfunctional pH meter can be detected in time to be repaired or replaced. Always check that the batteries are good and take extras! Record in the field notebook information about the instrument tests and calibrations including: dates, results and person testing the equipment. It may help to label sample containers for each site

ahead of time. Field conditions can sometimes make scribbling on jars difficult, and wet conditions make tape not sticky and permanent markers not so permanent.

Cleaning Equipment - All equipment should be cleaned before going into the field and between sites to prevent contaminating sediment samples. Equipment should be washed with clean scrub brushes using a non-phosphate detergent that leaves no residue when rinsed such as Alconox® powdered or Liqui-nox liquid detergent (Liqui-nox is the EPA standard detergent for sampling apparatus). To properly clean equipment, wash apparatus thoroughly with detergent, then rinse 5-6 times with tap water and 3 times with deionized/distilled water if it is available. Rinse the apparatus with site water before taking the first sediment sample.

Field Observations - Take turbidity or Secchi readings first, before the sediment is stirred up. **Record all field measurements and observations.**

b. General procedures in the field

1) Turn on any equipment that needs to warm up (like a DO meter) first or before reaching the site.

2) Make sure all equipment is clean and ready to use.

3) When working from a boat, two or three anchors or spuds driven into the sediment in shallow water will help stabilize boat in breezy, open water conditions.

4) Each grab or core attempt, whether for a composite sample or replicates, should be taken from undisturbed sediment at the site. Avoid disturbing sediments with a boat motor or by walking on the site. Approach sites from downstream to avoid suspending sediment into the water column over the site.

5) Have container ready to accept entire sample quickly upon retrieval.

6) Label every sample container with a permanent marker on labelling tape on the side of the jar or wherever the label will not come off accidentally. Information on the label should include: **STORET #, District code** (e.g., SED, WD, NWD, etc...), **Field #, replicate #, date, collector name** and **analysis type** (organic, inorganic).

7) Record all site information in a field notebook or on fieldsheets before leaving site. Information usually includes: field measurements, time and date, persons collecting samples, number and types of samples taken including field blanks, etc., labels assigned to each sample, and any general observations. Keep records of all samples, how they were labelled and any blanks or controls that are submitted for analysis.

c. Collecting Composite Samples

Composite samples are generally used to estimate the average concentration of the individual samples that make up the composite. Multiple grabs or cores for a composite sample should be taken from a relatively homogeneous sediment deposit (i.e., all grabs should be of similar sand/silt content). In some cases, composite samples are needed to generate sufficient sample volume for all analyses. It is best to know the rough boundaries of the sediment deposit or "site" before sampling.

Place each grab or core into a single mixing bowl (made of suitable material), remove any large objects such as sticks, leaves or stones, etc.. and stir thoroughly with a spoon to homogenize. A single grab or core should be mixed at least two minutes. Multiple grab or core samples should be mixed five minutes or longer if necessary.

Fill sample jars with the sediment mixture by placing one spoonful sequentially into each jar until the jars are full (see section on sample containers). This subsampling system assures that each sample container contains a sample as similar as possible to the other containers.

d. Collecting Replicate Samples

Replicate samples can be obtained at different stages of the sampling for different purposes depending on the objectives of the study. A study plan should describe where and how much replication is necessary. The procedures described here are for collecting distinct field replicate samples where the object is to determine the variability within a deposit and compare one field site to another.

When collecting replicate samples to statistically compare sediment deposits, sample sites within each deposit should be randomly located for statistical comparisons to be valid.

Be sure each sample is taken from an undisturbed area of sediment

If the replicate samples are fairly similar, the equipment need only be rinsed with site water between samples. But, if the replicates are not similar, and some contain significantly more fines than others, than the core tube or dredge may need to be washed with a non-ionic detergent (see equipment) and rinsed in between samples to prevent cross-contamination and to keep replicate samples independent for valid statistical analysis of the data. Use a tub of water in the boat to wash equipment to prevent getting detergent in the site water while sampling.

4. Procedures for Core and Grab Sampling Devices

Sediment samples are most commonly collected using a coring device or dredge or grab. The type of collecting equipment chosen will depend on sediment texture, site location (depth and current velocity), analyses to be performed and study goals. See References for more detailed discussion of the pros and cons of various sampling devices.

a. Piston Corer

Preparation and Scope

A corer allows excellent quantitative and qualitative sampling to a specified sediment depth with little disturbance of the sediment water interface. Samples can be separated or stratified by depth or color/texture to analyze distinct layers of sediment, although the sediment along the side of the core may smear as the core penetrates, slightly distorting the stratification of the sediment.

A corer may not be able to penetrate and/or retain very sandy substrates. Coring in high clay-content sediments where grabs won't work is possible if the water is not too deep, but may be difficult with a push corer and may require the use of a slide hammer or vibrating corer.

A large bore corer will provide a larger volume of sediment per attempt. This is important if discreet sample replicates are desired, and enough sample must be collected for a specific analysis or test. Even with the large bore core tube, samples may need to be composited to obtain enough sediment volume for the required analyses and/or tests.

A hand-operated, 3 inch diameter core sampler with an optional piston and extensions for deeper water can be effectively used in soft sediments with some silt/clay content in water up to ~30 ft deep (see Appendix A for a diagram of the coring device and its use). Core samplers may not be able to penetrate or retain very sandy sediments. Each district should have one of these coring devices with an acrylic core tube. A stainless steel core tube is also available for use from Central Office (call Tom Janisch 266-9268).

Collection Procedure

This procedure can be used for a push corer with or without a piston. A piston may not be necessary in high clay sediments. Disregard directions for use of the piston if piston will not be used.

1) Assemble the corer. Adjust the piston (the nut on the bottom adjusts piston diameter) so that it just fits snugly. If the piston is too loose, it will not stay in place until the corer reaches the sediment. If too tight, the piston will not move sufficiently when the corer is being pushed into the sediment, and compaction of the sediment core may occur.

2) Position the piston at the bottom of the core tube (open end), with the rope attached and threaded through the core head.

3) With the piston in place, let the core tube fill with water from the top, then lower the corer slowly and vertically to the sediment. If the piston falls out the bottom or moves up the core tube before reaching the sediment, tighten piston slightly and try again.

4) With the bottom edge of the corer and the piston in contact with the sediment in a vertical position, push the core tube into the sediment while maintaining some tension on the piston rope. The piston should remain at the sediment surface while the core tube moves into the sediment. In difficult sediments, it may be necessary to actually pull on the rope as the corer is pushed into the sediment. The object however is to maintain the piston in a fixed position <u>at</u> the sediment-water interface without compacting the sediment.

5) In hard or clay sediments where it is difficult to push the corer into the sediment by hand, a slide hammer designed specifically for the core sampler should be used. Do not pound on the core head or extension tubes with a hammer or anything else as this could break or damage the core head or other parts, and is generally less effective than the slide hammer.

6) After core is pushed to desired depth, pull up the corer slowly while maintaining the position of the piston by holding the piston rope in place. Even with the piston, some sediment may be lost from the bottom of the corer if the sediment is sandy. To help prevent sample loss, bring the corer into a horizontal position as it reaches the surface. A plug can also be inserted into the bottom of the sampler before removal from the water.

7) Place the corer on the work surface (boat or ice) over the receiving container. The sediment core can be extruded from the top or bottom of the core tube, depending on the purpose of the sample and study goals. Generally, cores collected for macroinvertebrate work should be extruded out the bottom, and cores collected for chemical analysis should be extruded out the top of the core tube if only part of the segment is needed to reduce contamination of the sample segment from other layers.

8) To extrude through the bottom, remove the sampler head, insert a pole through the top and push down on the piston eyebolt. Extrude the core into a waste container until the desired length of core remains, then extrude the remaining sediment into the sample container. To extrude through the top, remove the sampler head and place an extrusion pole and rubber plug at the bottom of the sampler and push sediment out through the top slowly. A premarked acrylic or polycarbonate (clear) core tube is helpful for measuring core lengths.

b. Grab Samplers

Preparation and Scope

Grab samplers rely on their own weight and gravity to penetrate the sediment as well as the leverage from the closing of the jaws. For this reason, they are not as efficient in water flowing over one meter per second. They normally take a discreet "bite" of sediment to a fairly consistent and measurable depth. Grabs often cause a shock wave upon descent which may disturb very fine sediment at the sediment-water interface.

Many grabs and dredges such as the petite Ponar and Ekman dredge are available and used by DNR staff. These two can be hand operated from a suitably sized boat, preferably flat-bottomed. The Ponar is better suited to sampling hard or sandy sediments because of the greater ability to penetrate. The Ekman is more suited to sampling in soft sediments in low flow waters. Neither grab will effectively sample hard clays where a coring device or shovel such as a sharpshooter spade can be used.

Have a sample tub ready to receive sediment that is large enough to receive the entire contents of the sampler.

Understand and be careful of the closing mechanism and moving parts on a sampler. It is easy to pick up a grab the wrong way and pinch fingers.

Collection Procedure

1) Set closing mechanism and lower grab slowly to substrate, being careful to avoid a shock wave caused by too rapid of a descent near the sediment.

2) Initiate closure mechanism of grab. This is usually a messenger sent down the rope or a sharp pull on the rope.

3) When it feels like the grab has closed and contains sediment, raise grab at a steady rate and immediately position over large bucket. If jaws are not completely closed due to obstructions, discard entire grab contents away from sampling area and try again. Make sure to move the sampling site at least several feet away from the previous attempt(s) to avoid sampling a disturbed area.

4) If the study dictates careful sampling for metals analysis, the middle portion of the sample not touching the metal grab can be collected with a teflon or plastic spoon, and the rest of the sample discarded.

5) Empty entire contents of grab into mixing bowl if sample needs to be mixed.

6) Place appropriate volume of sediment into sample container.

5. Quality Control Measures

Sediment samples should be collected from the reference or control sites first whenever possible to reduce the chances of cross-contamination from other sites.

All samples in a study should be handled identically, including using the same sampling equipment, stirring times, etc.

When collecting samples for chemical or toxicity tests, take appropriate measures to prevent contamination from other sources such as vehicle and boat motor exhaust or associated contaminants and other contaminated sites. The person operating the boat motor should either not handle sediment samples or make sure to put on clean gloves to prevent contamination from the motor.

6. References

Baudo, R., Giesy, J., and H. Muntau, (Eds.). 1990. *Sediments: Chemistry and Toxicity of In-Place Pollutants*. Lewis Publishers, Boca Raton, FL.

EPA. 1992. Sediment classification methods compendium. Office of Water, Washington, DC. EPA 823-R-92-006.

EPA. 1985. Sediment sampling quality assurance user's guide. Environmental Monitoring Systems Laboratory. Las Vegas, Nevada. EPA/600/4-85/048.

EPA. 1994. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates.

Green, Roger H. 1979. Sampling design and statistical methods for environmental biologists. John Wiley & Sons. New York. 257 pp.

Klemm, D.J., P.A. Lewis, F. Fulk, and J.M. Lazorchak. 1990. Macroinvertebrate field and laboratory methods for evaluating the biological integrity of surface waters. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH 45268. EPA/600/4-90/030.

WDNR. 1993 (draft). Field Procedures Manual. Office of Technical Services, Bureau of Water Resources Management.

WDNR. 1990 (draft). Quality Assurance Guidance for Inplace Pollutant Monitoring Activities. Unpublished document on file at Office of Technical Services, Bureau of Water Resources Management.

D. Collecting and Processing Sediment Samples for Chemical and Physical Analysis

1. Scope:

Analytical data are produced for some kind of decision-making whether it is regulatory or monitoring. Data that are closely controlled (quality assurance) provide reliable evidence for use in court or to make assessments of pollution in the environment.

In the collection, storage and transport of samples intended for organic analysis, the field personnel should be aware of possible contamination and/or degradation that can occur in organic samples. The following information will help eliminate problems with sample analysis.

Quality data can only be obtained from environmental samples that are properly collected, preserved and promptly shipped to the laboratory for analysis. The procedures involved in this process include: 1) collecting the samples using appropriate sampling techniques; 2) selecting proper sample containers; 3) preserving the samples immediately after collection either chemically or by cooling to 4 °C, whichever is appropriate; 4) clearly identifying the samples and completing the corresponding laboratory sheets; and 5) carefully packaging and promptly shipping the samples to the laboratory for analysis.

Sediments for organic and inorganic chemical analyses are most often collected using grab, dredge or core methods. The chosen method should target the goals of the study plan and complement any other biological tests that may be conducted at the site or with sediments from the site. Samples slated for different types of physical and chemical analysis may need to be collected and handled in slightly different ways. The level of precautions that must be taken to prevent contamination of samples will depend on the type of analysis to be performed and the study objectives.

2. Equipment

Refer to section **C. General Sediment Sampling Equipment and Procedures** for suggested equipment list and suitability as well as procedures for collection equipment.

a. Sample Containers

Samples for organic analysis and inorganic (metals) analysis must be in separate containers. Containers are prepared by and should be obtained from the Wisconsin State Laboratory of Hygiene (SLOH) if it is the laboratory doing the analyses. Other laboratories may provide their own containers and should be contacted to find out their requirements for appropriate sample jars. The following information pertains to analyses to be performed by SLOH.

1) *Sample Containers for Inorganic Analysis*

Sediment samples should be submitted to the laboratory in a container appropriate for the analyses requested.

Metals - Samples that require metals analyses should be submitted either in 250 mL "metals" bottles or a glass quart mason jar with teflon lid. One 250 mL "metals" bottle (same as for water) provides enough sample to perform all of the routine metals analyses and solids analyses.

Nutrients - Samples that require nitrogen, phosphorus and solids analyses should be submitted in 250 mL "nutrient" bottles or a glass quart mason jar with teflon lid.

Oil & Grease - Samples for Oil & Grease are analyzed by the inorganic section and must be in a glass quart jar with a teflon lined lid. Fill jar 3/4 full or more. Separate containers for metals or nutrients are not necessary if the glass quart jar is used.

If additional analyses or information about special preservation and handling procedures for SLOH are required, contact the Inorganic Chemistry Unit for instructions:

The Wisconsin State Laboratory of Hygiene
Inorganic Chemistry Unit
(608-262-3458)

2) *Sample Containers for Organic Analysis*

Soil and sediment samples should be submitted to the laboratory in a container appropriate for the analyses requested. All containers are prepared by and should be obtained from the State Laboratory of Hygiene.

Organics (PCBs, PAHs, etc.) - Samples for all regular organics analysis should be contained in glass quart jars with teflon lined lids. Jars should be 3/4 full or more. If analyzing for semi- or volatile organics fill jar completely so no air space exists.

Volatile Organic Carbon (VOC) and Gasoline Range Organics (GRO) - A 60 milliliter glass vial with a septum top should be used for soil and sediment samples that are to be analyzed for VOC and GRO. The laboratory will provide three preweighed sample vials for each sample site. The vials should be filled with sediment to the "Fill to here---" label (approx. 25g) found on the side of each vial. A water and methanol "trip blank" will be included in each sample mailer.

Diesel Range Organics (DRO) - A 60 milliliter glass vial should be used for soil samples that are to be analyzed for DRO. The laboratory will provide three preweighed sample vials for each sample site. The vials should be filled with soil to the "Fill to here---" label (approx. 25g) found on the side of each vial.

A portable balance for measuring GRO and DRO samples is available from the Central Office. Contact Tom Janisch at (608) 266-9268 for availability information.

If additional analyses or information about special preservation and handling procedures are required from the State Lab of Hygiene, contact the Inorganic Chemistry Unit for instructions:

The Wisconsin State Laboratory of Hygiene
Organic Chemistry Unit
(608) 262-2797

3) *Samples for Bioassays and Chemical and Physical analyses*

If chemical and/or physical analyses are required on sediment samples also slated for toxicity or bioaccumulation tests at the Aquatic Life Toxicity Testing Laboratory (A.K.A. Biomonitoring Lab), the lab can perform the sediment homogenization and fill sample jars for the chemical analyses from the same sediment that will be used for the bioassays. Clean, five gallon, polyethylene buckets are available from the Biomonitoring lab for holding and transport of sediment samples for toxicity and bioaccumulation tests (see section on Bioassays). Small buckets are also available if only toxicity tests and chemical and physical analyses are planned. If a laboratory other than the Biomonitoring Lab is used, the testing lab should be contacted for information on appropriate sample containers and procedures.

Aquatic Life Toxicity Testing Laboratory
977 Jonathan Drive
(608-265-4023)

4) *Samples for Particle size analysis*

Quart-size plastic bags (from the store) can be used for particle size samples. Double bag the sample and fill 1/2-3/4 full. Label both bags in permanent marker with STORET #, District code, Field #, date and collector's name. There is no separate lab slip, but the "particle size analysis" box should be checked on the new sediment lab slip in order to keep the data tracked together. Particle size analysis is usually contracted for every chemical analysis sample, so make sure to collect sediment for this analysis. The Biomonitoring Lab will also subsample sediments for particle size analysis if toxicity tests are being conducted.

5) *Quality Control of Sample Containers*

Quality control audits are conducted on representative portions of all sample bottles from the State Lab of Hygiene for chemical analysis to verify that they are free from contaminants. These audits are performed before any bottles are approved for use. Because of the considerable effort expended in assuring the quality of sample bottles, it is important that they be used only for the parameters specified on the label.

Information should be obtained about the quality control of sample containers obtained from analysis laboratories other than the State Lab of Hygiene when the contract for service is being set up to make sure appropriate procedures are used to prevent contamination.

The sample container cleaning procedures and details regarding the Quality Assurance audit protocols are described in the "Organic Chemistry Manual", and section 5 and 6 of the State Laboratory of Hygiene's Quality Assurance Manual prepared by the Water Chemistry Unit. Labs other than SLOH should be contacted for QA protocols.

3. Sediment collection:

See Section **IV.C. General Sediment Sampling Equipment and Procedures** for a description of equipment and procedures for collecting sediment.

The following steps for **cleaning** new or used sediment sampling equipment and containers are recommended by EPA (1994):

1. Soak 15 min in tap water, and scrub with detergent.

2. Rinse twice with tap water.

3. Rinse once with fresh, dilute (10% V:V) hydrochloric or nitric acid. To prepare a 10% solution of acid, add 10 ml of concentrated acid to 90 ml of deionized water.

4. Rinse twice with deionized water.

5. Rinse once with full-strength, pesticide-grade acetone (use a fume hood or canopy).

6. Rinse three times with deionized water.

7. Rinse field collection equipment with site water immediately before use. Lab equipment should be rinsed with test dilution water immediately before use in a test.

Clean equipment can be protected from contamination during transport (i.e., exhaust, pickup beds, boat motors, etc.) by wrapping in aluminum foil.

Request and obtain appropriate sample containers ahead of time (see above).

Quality control procedures to be followed at the sites should be written down for all field staff if a formal QAPP is not written.

4. Sample Preservation

All sediment samples for chemical analysis should be preserved as soon as possible after collection by cooling to and **maintaining** a temperature of ~4°C (ice cold) by putting samples on ice in a cooler.

Keep samples shaded from sunlight to prevent breakdown of chemicals by UV light.

The SLH provides "blue ice packs" in each sample kit designed for VOC, GRO and DRO analysis, although samples should first be cooled to 4 °C on ice. Plastic bottles can also be filled with water, frozen, and placed in the shipping container. Samples should be pre-chilled if these cooling materials are used for shipping.

For soil or sediment samples to be analyzed for GRO, it is required to add 25 ml of premeasured methanol to two of the sample vials at the time of collection. (Vials of methanol are available at the DNR district office.) The third vial is used for determining moisture of the sample.

For soil samples to be analyzed for VOCs, the collector should consult the individual program needs for the appropriate preservation requirements which may include methanol preservation.

Contact the contracted laboratory for additional preservative requirements for specific parameter requests.

5. Packaging and Shipping

a. Cooling Samples

When cooling is required during shipping, the samples should be pre-cooled in an ice chest, and later placed in a field pack with a suitable quantity of ice or "Blue Ice". Ice should not be placed in the field pack loose. It should be securely sealed in a heavy plastic bag to prevent leakage during shipment. Plastic quart sample bottles or plain 250 mL bacteria sample bottles filled with water and frozen, may be used in place of ice cubes (they leak less and may be reused). However, if sample bottles are used, clearly label them as "ICE". DO NOT USE metals bottles, nutrient bottles, or bottles designated for specific tests as ice containers. A great deal of Quality Assurance effort goes into preparing these bottles before they are approved for use.

b. Packing Samples

Properly packaging sediment samples for shipping is important for maintaining sample quality and safety of persons contacting the samples.

After collection, check each sample to make sure the container lid is securely closed and the sample is properly preserved. The exterior of each sample container should be wiped clean with a wet cloth.

Check all samples for secure, correct and complete labels that match the accompanying lab sheets (see below).

Styrofoam field packs for shipping environmental samples are available from the State Laboratory of Hygiene. A whirl-pak (plastic bag) is included in the pack and should be used to protect the laboratory sheets from moisture damage during shipment. Dividers, included in the packs, help protect the sample bottles during shipment and should be used whenever possible. When sealing the field packs, secure all four sides of the lid by wrapping with reinforced tape. The tape should be completely wrapped around the pack to make sure that the lid is secure. When more than one field pack is needed to ship various sample portions from a single sampling site, tape the field packs together. This will prevent sample sorting errors and will allow the lab to match the bottles with the correct laboratory sheets.

A cooler lined with a polyethylene bag can be used instead of the foam pack if necessary, but be sure to pack sample jars to avoid breakage during shipping and handling.

c. Laboratory Sheets

Different laboratories may have their own lab sheets that should accompany all samples. Lab sheets for the State Lab of Hygiene are available from DNR Supplies and Forms - PS/DR, Madison (Winston Piotrzkowski at (608) 246-7981), sediment staff at Central Office and Technical Services.

The laboratory sheet is an important link between the laboratory and field personnel. The laboratory relies on the sheet to obtain the information necessary to prepare and analyze the sample properly.

The sample collector must select a laboratory sheet appropriate for the sampling program, and complete it with a waterproof pen. The completed sheet should contain:

1) STORET number , 2) a sample description, 3) the sampling program, 4) name and address of the person to whom the report should be sent, 5) Last name of the sample collector, 6) primary station number and/or county code number, 6) field information, 7) tests (parameters) requested.

New computerized sample and location information entry will be available early FY '96. This system will also generate the lab slip to accompany the sample to the lab

d. Shipping Samples

If storage time limitations are recommended for the sample parameters, coordinate with the laboratory before collecting samples to let them know the sampling schedule.

Alert the receiving laboratory of any samples that are known or believed to contain high levels of specific contaminants, including an estimated concentration if possible. This can be done either over the phone before the samples arrive or with an enclosed written warning. The advanced notice allows the lab to handle highly contaminated samples in a way to prevent human exposure as well as cross-contamination of samples in the lab. Additionally, the lab will be able to process and analyze the samples more quickly if they know before analysis that the contaminant concentration is high.

Samples should be shipped with an "overnight" mail service or personally delivered to the laboratory or shipped or delivered to the Central Office for temporary storage so that the samples arrive before all of the ice melts in the shipping container. Monday, Tuesday or Wednesday are the best days to ship samples to assure they do not sit in a mail room with no refrigeration over the weekend. Even "overnight mail" can take longer than 24 hours, so Thursdays can be risky. DO NOT send samples on Fridays unless you have made previous arrangements with the lab and/or Central Office.

All sediment samples for the DNR (unless other arrangements for a special project have been arranged) should be shipped with the lab slips inside the shipping container to the Central Office care of Linda Talbot:

Department of Natural Resources
C/O Linda Talbot
101 S. Webster St.
Box 7921
Madison, WI 53707

Phone: (608) 266-8148

NOTE: Samples are logged in and assigned to the appropriate lab account numbers as well as checked against the lab contract for parameters and numbers of samples. Additionally, sampling information may need to be keyed into the computer from the lab slip by Central Office before samples are sent to the lab, after which lab slips are gone forever. Because sediment samples are run under special contracts with the lab and not under the Basic Agreement like the surface water samples, this sample transfer and tracking is necessary. This process allows the DNR to best utilize the analysis dollars.

Call the Madison office or laboratory before shipping samples to make sure someone will be available to receive the samples and take care of them. If using a lab other than SLOH or you have made arrangements to deliver samples directly to the lab, coordinate with Linda Talbot for the appropriate log in of samples and lab slip information.

e. <u>Shipping Safety</u>

If a sample bottle seal is questionable and no additional bottles are available, place the entire bottle in a whirl-pak (250 mL bottles only). This will contain the sample and prevent any preservative from contaminating other samples in the field pack.

The outside of the sample containers should be completely free of contaminated material before the samples are shipped. If this is not possible, the laboratory should be made aware of these samples before shipment.

If the submitter believes a sample contains a Department of Transportation (DOT) regulated material or hazardous material, refer to the "Sample Shipping Requirements" section in the Field Procedures Manual (DNR, 1993).

6. References

Baudo, R., Giesy, J., and H. Muntau, (Eds.). 1990. *Sediments: Chemistry and Toxicity of In-Place Pollutants*. Lewis Publishers, Boca Raton, FL.

EPA. 1985. Sediment sampling quality assurance user's guide. Environmental Monitoring Systems Laboratory. Las Vegas, Nevada. EPA/600/4-85/048.

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State Laboratory of Hygiene. 1994. Organic Chemistry Manual.

State Laboratory of Hygiene. 1992. Inorganic Chemistry Manual.

E. Benthic Invertebrate Surveys - Benthic Samples

1. Scope:

> "Benthic invertebrates [are]... the most appropriate biological indicators [of *in situ* toxic effects] because they are most directly associated with contaminants in sediments through their feeding and behavioral activities." (Reynoldson et al, draft).

Benthic invertebrate communities (living in and directly on the surface layers of sediment) are directly affected by the chemical and physical integrity of the sediment and overlying water. Invertebrates living in the water column are often not directly affected by contaminants in sediment deposits accept when contaminants leach into the water column or invertebrates are exposed to suspended sediments moved from the deposit by some event. For this reason, the most direct way to measure the effects of sediment associated contaminants is to survey the invertebrates inhabiting the top 10-15 cm of soft sediment.

Sampling the benthos of surface waters involves many considerations, many of which are not within the scope of this manual. There is a considerable body of scientific literature dealing with the theoretical basis of benthic population distribution and proper study design, other sampling techniques, taxonomy, and statistical analysis of benthic samples. The intent of this section is not to condense the scientific literature but to present collection techniques commonly used in Department investigations. It is assumed that the investigator already has a working knowledge of fresh water benthos and has developed a sound study design prior to collecting samples in the field.

The most basic consideration in developing a plan of study is to decide on the type of data needed, either quantitative or qualitative, which then determines the type of sampling method used. Quantitative techniques are defined as those that estimate population densities, or numbers of organisms per unit area of substrate, and usually require many samples per site to produce reliable data. Qualitative techniques cannot generate density information but require fewer samples per site and can provide estimates of relative abundances; water quality and diversity indices; and measures of similarity between stations if based on measures not requiring similar sampling effort and effectiveness (see Klemm 1990 and WDNR 1990). once a study design is formulated, the sampling method can be chosen.

2. Safety:

Formalin (formaldehyde) is a known human carcinogen and should be handled with caution. Wear gloves whenever handling formalin and always choose a well ventilated area to work in; upwind of the samples if outside or under a ventilated hood if processed inside. Samples containing formaldehyde should not be sent through the mail. Any samples containing formalin should be carefully handled and packed during transport to avoid breakage, leaks and spills.

3. Equipment:

<u>Equipment Checklist</u> - Benthic samples

1. Container for temporary sample storage. Container must be large enough to be able to empty a grab sampler without losing any of the sample, and should have no gaps where small invertebrates could get caught.
2. White enamel or plastic sorting tray.

3. Sieve bucket with U.S. Standard #60 or #30 mesh screened bottom.
4. Suitable size sample storage containers.
5. Labels for both inside and outside of sample jars. Inside labels should be water proof. A quality bond paper works well.
6. Pencils or permanent marking pen.
7. Preservative: 95% ethanol (EtOH) or Formaldehyde.
8. Wash bottles for water and preservative solution.
9. Forceps for picking invertebrates.
10. Field sheets and/or invertebrate bench sheets (Forms 3200-81, one per sample site.
11. Aquatic invertebrate identification guide.

A fairly complete discussion of the various factors that should be considered in selecting the proper sediment collection equipment for invertebrate sampling is presented in EPA (1992), Klemm et al (1990), Weber (1973), and Chapter 4 of Downing (1984).

a. Corers

Corers are preferred for collecting quantitative benthic invertebrate samples because they tend to disturb the sample area less than a grab and they provide greater accuracy in determining depth of sample collected. Core samples also allow viewing and separation of stratified sediment layers. Disadvantages of a core sampler include the inability to collect very sandy or course sediment samples because of sample loss from the core tube during retrieval. Sampling very deep sites may also be impossible depending on the corer being used. See section **C. General Sediment Sampling Equipment and Procedures** for core use procedures.

b. Grabs

Grab samplers may also be used to sample soft sediments and are considered quantitative when properly used, although the accuracy and precision of depth of penetration and sediment volume collected with each attempt is lower than with a corer.

4. Preparation

Preservative - Prepare the proper strength preservative ahead of time. Be aware that the unpreserved sample will contain some water which will dilute the preservative to some extent.

NOTE: Formalin is the name used for the commonly sold ~40% (by weight) formaldehyde solution. So, 40% formaldehyde = 100% formalin. 10% formalin = 4% (w/w) formaldehyde.

Label all containers with contents (such as distilled water, 10% formalin, 80% ethanol, etc.) to avoid confusion and accidents while in the field.

Refer to preparation in section **C. General Sediment Sampling Equipment and Procedures**

5. Sample Processing

The following is derived from Klemm (1990) and the Field Procedures Manual (1993).

Collection procedures will vary, depending upon the type of collecting device used. Follow procedures described in section **C. General Sediment Sampling Equipment and Procedures** for the collection of the sediment sample. Below is described the procedures for processing a benthic invertebrate sample once the sediment is obtained.

a. Sieving Samples

Sieving invertebrate samples reduces the volume of sediment that must be sorted through in the lab. A #60 sieve (250 μm openings) is recommended for most all new projects in Wisconsin because the smaller invertebrates will be retained by the #60 sieve and should yield more complete invertebrate community data for a site. Number 30 (500 μm) sieves should be used for continuing projects where data using the #30 size sieve already exists.

1) After collecting a sample, the sample should be placed into a #60 or #30 mesh (250 μm and 500 μm openings, respectively) sieve bucket. Any large debris should be cleaned (remove invertebrates and add them to the sample) and removed from the sample. The sample is then washed through the sieve over the side of the boat or in a tub with site water until no more fine sediment washes through the mesh. *Take care not to allow site water into the bucket from the top as this could allow non-sample organisms to contaminate the sample.* Washing can be accomplished by adding small portions of the sample at a time to the sieve or the whole sample at once. Many invertebrates are fragile, so this should be done as gently as possible while still getting the job done. If the sample clogs the mesh so water does not drain out, submerge the bucket half way into the water and lower and raise the bucket with enough thrust to push water in from the bottom and suspend the clogging sediment. Performing this action while also sliding the bucket sideways should clean a sample relatively quickly without much damage to the invertebrates. Sometimes the sample will contain chunks of clay that must be carefully broken up with your gloved hands (remember, most toxic contaminants are associated with fine sediments). Another method of cleaning the sample is to use a gloved hand to swish the water while the bucket is partially submerged to resuspend the sample and allow fine sediment to fall out.

2) After no more fine sediment will wash through the sieve, place sample in sample jars. To move the sample to a jar, tip the sieve bucket so a bottom corner is lowest and wash the remaining sample off the screen and into the bottom corner of the bucket. Wash this into a flat light colored wash pan or directly into the sample jar if possible (a wash bottle and forceps are handy for this). Make sure not to use so much wash water that the final preservative concentration is too dilute.

3) Quart-size sample jars made of glass (possible breakage and sample loss problem) or plastic make good sample containers. Whirl-pacs or other well sealed bags can also be used, but invertebrates become even more fragile after being preserved, so the container should protect the integrity of the sample during handling and transport.

b. Sample Preservation and Processing

1) Add 95% ethanol to the sieved sediment sample to a final concentration of ~ 70-80% ethanol. Ethanol is most commonly used to preserve benthic invertebrate samples. If identification of oligochaetes is desired, samples should be preserved in 10% formalin (final concentration) for 10

minutes or longer before transferring them into 70-80% ethanol (Klemm et al, 1990). Samples do not need to be transferred to alcohol if the laboratory doing the identification will accept formalin. If time and space is available in the field, it may be easiest to preserve the samples in formalin in the sorting pan (keep formalin downwind, see safety section), then decant formalin (into appropriate container) and rinse any remaining formalin out with sieved site water (unsieved site water may introduce additional invertebrates).

2) To exchange ethanol for formalin in sample jars: 1) drain the formalin into a suitable container using a funnel if necessary. This can be accomplished with a #30 or #60 mesh screen placed over the sample jar or by carefully dumping the sample back into the sieve bucket. 2) Rinse out any remaining formalin with sieved site water or tap water. 3) Move sample to sample jar and add ethanol to a final 70-80% concentration.

3) After addition of the preservative, gently invert sample jar to mix. Invertebrates can become fragile after preservation. Do not shake sample.

4) Label each sample with tape and permanent marker on the outside of the container. Also place a water resistant label made of part bond paper written in pencil on the inside. Information should include the date, time, site identification, name of person taking sample and replicate number/total samples at the site (sometimes the order the samples were taken in can make a difference).

c. Sorting

The specific sorting procedure may vary for each study, depending upon the needs of the investigation. In general, sorting of samples obtained with grabs and cores is similar to those obtained with other devices that collect both substrate material and organisms. Sorting is usually accomplished by placing sample material in a shallow, white pan and picking invertebrates from the sample debris with the aid of a low power magnification device (binocular scope or scanning lens). Sorting is made considerably easier when the invertebrates are stained a bright red color from ETOH with Rose Bengal dye added. However, ask the laboratory performing the identifications before using Rose Bengal to make sure it will not interfere with their identification procedures. Prior knowledge of the taxonomic references to be used in identifications is essential before staining, since staining may obscure color patterns sometimes used as diagnostic features for identification.

The following procedures may be done by the contracted laboratory or by the investigator.

1) To remove the alcohol or formalin preservative prior to processing, place the sample material on a U.S. Standard #30 or #60 mesh screen or sieve, and wash with water. Be careful to remove all invertebrates from the screen after washing.

2) Small amounts of sample material are then placed into the sorting pan and searched for invertebrates as noted above.

3) Invertebrates are removed with a forceps and placed into suitably sized, labeled storage containers with 70-80% ETOH for long-term storage.

d. Subsampling

When sorting samples with large amounts of sample material or organisms, sorting and analysis time can be considerably reduced by subsampling.

For routine investigations and when sample sizes are too large to sort 100%, subsampling is accomplished by the following method (Weber, 1973):

1) Thoroughly mix and distribute the entire sample evenly over the bottom of a shallow, white tray.

2) Place a divider in the tray which delineates quarter sections.

3) Sort the two opposite quarters delineated by the divider, or one randomly selected quarter in extremely large samples.

4) Combine the two remaining quarters and store as reference material or discard.

5) Follow the same procedure for individual taxonomic groups, if present in excessively large numbers, to reduce analysis time.

A similar procedure is followed for sorting Hilsenhoff Biotic Index (HBI) samples, except a gridded sorting tray is used and only 100 arthropods are subsampled. Refer to Hilsenhoff (1987) for a complete description.

Other subsampling methods are possible, depending upon the needs of the study. A method to statistically check on the validity of the subsamples withdrawn and to predict upper and lower confidence limits for the estimated total is presented in Elliott (1977).

e. Taxonomic Identification

The taxonomic level to which organisms are identified may vary with the objective of the study and should be discussed in each project's Plan of Study, Quality Assurance Project Plan, or similar document. Benthic invertebrate samples are normally sent out to a laboratory for sorting and identification (see below). Only someone with training in the field of benthic invertebrate taxonomy should perform sample identifications. Follow procedures below if samples are to be sorted before being sent to a lab.

There are two laboratories that the Department currently uses for invertebrate sample analysis (see below). Contracts for invertebrate sample analysis are coordinated by Joe Ball-WR/2 at Central Office. Each lab can only process a limited number of samples in a year.

1) Dr. Stanley Szczytko's laboratory at the University of Wisconsin-Stevens Point. This laboratory currently processes most or all of the Department's basin assessment and HBI invertebrate samples. They do not perform oligochaete identification beyond family.

Address: Dr. Stanley Szczytko
College of Natural Resources
UW-Stevens Point
Stevens Point, WI 54481

2) Dr. Kurt Schmude directs the invertebrate laboratory at the University of Wisconsin-Superior's Lake Superior Research Institute. This laboratory was used by the DNR for the first time in 1993 with better than satisfactory service. Because of the location of this lab, they may be or become more experienced with the identification of Great Lakes benthic invertebrates (Especially Lake Superior). Services offered by this lab may in the future include data analysis for each sample and project. Special services such as specific identifications or data analysis must be negotiated.

Address: Dr. Kurt Schmude
Lake Superior Research Institute
Hawkes Hall, Rm 153
1800 Grand Avenue
Superior, WI 54880

6. Documentation:

Information about the sample site and collection procedures are normally recorded on Department Form 3200-81, Macroinvertebrate Field and Bench Sheet, and sent with the samples to the lab doing the taxonomy work. They are compatible with the DNR invertebrate (BUG) computer program utilized by Dr. Stan Szczytko's lab. But, because these bench sheets were designed for stream locations and not specifically for soft sediment deposits, and they must be sent with the samples to the lab, you may want to develop a separate field sheet for a specific project. Whichever field sheet is used, it is imperative that all pertinent site information is written down. It is also very important to label the sample container(s) from each site with the corresponding Sample ID number that appears on Form 3200-81 for that site.

7. Quality Assurance:

The main concern with quality assurance of benthic core or grab samples is to prevent carryover of organisms from one sample to the next. Carryover is prevented by careful washing and inspection of collection devices and sieve screens after each sample collection.

Another concern is disturbance of sample sites prior to and during sample collection. Anchoring a boat over the sample site must be done carefully to avoid physical disturbance of the area to be sampled. Whenever possible, anchor the boat upstream at least several feet and drift to the site or anchor a few feet away and gently paddle the boat to the sample site. In windy conditions, two anchors may be necessary on different sides of the boat for stabilization. Mobile invertebrates may leave the area or others may be carried off-site by water currents generated by anchoring, causing sample error. A similar problem can occur from the core or grab impacting the substrate.

If the jaws of a grab don't close successfully on retrieval, another sample must be attempted at the same site. It is very important to move successive sampling attempts several feet away from all preceding attempts to avoid sampling previously disturbed substrates.

Invertebrates become brittle in preservative. Samples should be handled as gently as possible to avoid breaking up individual invertebrates.

Refer to EPA 1992, Klemm 1990 and the Quality Assurance Guidance for In-place Pollutant Monitoring Activities (WDNR, 1990) for more detailed quality assurance procedures.

8. References:

See Section IV.F.9.below for references pertaining to macroinvertebrate sampling.

F. Benthic Invertebrate Survey - Artificial Substrates

1. Scope:

Artificial substrates are useful for collecting macroinvertebrates associated with the water column and can be used to demonstrate water quality and effects of contaminated sediments on the overlying or downstream water column. They can be used in almost any stream or lake habitat, either suspended in the water column or anchored to the bottom. They are left in the water for relatively long time periods (2-8 weeks) to be colonized by drifting or swimming aquatic organisms.

The **main advantage** of artificial substrates is the minimization of the effects of physical variables between sites such as: substrate type, depth, and light penetration. Data comparisons between stations are, therefore, simplified.

Some **disadvantages** include: large effort and time to results (two separate field trips, 2-8 weeks to colonize), loss of sample because of weather or vandalism, and unquantifiable loss of invertebrates if samplers are accidentally disturbed.

For sediment assessments, the most significant attribute is the bias for aquatic rather than benthic invertebrates. Invertebrate colonies from artificial substrates generally reflect water quality rather than sediment quality unless the sediment is directly affecting the quality of the water above it. So, artificial substrates should be used to demonstrate a change in water quality near or downstream from a sediment and in conjunction with other sediment data, but should not be used as a direct measure of sediment quality alone. Discussions of both the advantages and disadvantages of artificial substrate samplers appear in, Klemm et al (1990), Rosenberg and Resh (1982), Weber (1973) and Chapter 5 of Downing (1984).

2. Equipment:

Several different designs of artificial substrates are available, but only two common types will be described here. Other types are described in the scientific literature (Klemm et al, 1990; Rosenberg and Resh, 1982).

a. Multi-plate Samplers

Multi-plate artificial substrate samplers consist of a series of square or circular hardboard discs. separated by spacers and fastened together through their centers to a threaded eyebolt. The standardized, reproducible and easily measured substrate surface areas allow for uniform replicates and the collection of quantitative data. The substrates can either be suspended or anchored a predetermined distance from the water or sediment surface.

The Fullner (1971) modification to the Hester-Dendy (1962) multiple-plate design is widely used. The modified Hester-Dendy sampler is constructed of 0.3 cm (0.125 in) thick tempered hardboard with 7.6 cm (3 in) diameter round plates and 2.5 cm (1 in) round spacers that have 5/8 in center-drilled holes. The plates are separated by spacers on a 1/4 in diameter eyebolt, held in place by a nut at the top and bottom. A total of 14 large plates and 24 spacers are used. The top nine plates are each separated by a single spacer, plates 9 and 10 are separated by two spacers, plates 11 and 12 are separated by three spacers, and plates 13 and 14 are separated by four spacers. The hardboard sampler is about 14 cm (5.5 in) long and has a surface area of about 1,160 cm^2 (0.116 m^2). The surface area and dimensions will change from absorption of water. *Hardboard samplers exposed to toxicants, oils and/or preservatives (alcohol, formalin) cannot be reused.*

b. Rock Baskets

The other common type of artificial substrate is the rock basket sampler most commonly composed of a cylindrical, chrome plated basket (barbecue basket) filled with 1 to 3-inch diameter rocks or rock-like material. See Mason et al. (1967) or Klemm et al (1990) for a more detailed description of a sampler and suspension system. It is more difficult to calculate surface area for rock basket samplers, but the rocks simulate rubble type natural substrates better than multi-plate samplers.

c. Alternative substrate materials

Alternative substrate materials that have been used in samplers include: 3M plastic mesh (conservative webbing, 3M corporation, St. Paul, MN), natural rubble, natural leaf litter or any other suitable substrate for colonization of invertebrates. Samplers constructed from rotisserie baskets and 3M mesh have been described by Stauffer et al. (1976) and Swift (1985).

Equipment Checklist - Artificial Substrates

1) Artificial substrates.
2) Anchoring or suspension equipment for artificial substrates.
3) Dip net or net bag for retrieval of artificial substrates.
4) Shallow, white pan for cleaning artificial substrates.
5) Wash bottle and forceps.
6) Labeled sample containers large enough to enclose assembled, multi-plate artificial substrates, or
7) Labeled sample containers large enough to contain contents of rock basket type artificial substrates.
8) Labels for inside sample jars.
9) Preservative - 95% EtOH, formalin.
10) Tools for assembly and deployment or retrieval (cutting tool, pliers, etc.)
11) Permanent marker and pencils.
12) Field sheets and/or Forms 3200-81, one per sample site.

3. Collection Procedure:

a. Setting the samplers

Artificial substrates are anchored or suspended at the sample site to be colonized by drifting and swimming invertebrates. Placement within the water column depends upon the objectives of the study. For sediment assessments, the samplers should usually be placed close to the sediment, but samplers from all sites should be at similar depths. For water quality assessment, samplers are normally placed in the euphotic zone (1-3 feet from water surface) to collect the highest diversity and abundance of invertebrates possible (Mason et al. (1973) in: Klemm et al. (1990)).

Care should be taken to prevent silt from building up and thus reducing the colonizable surface area of the substrate.

To prevent vandalism, place samplers out of the view of passers by and away from boat traffic and heavily used fishing areas. If water levels at the site will fluctuate, place the sampler so it will be midway in the water column at low-flow or at least will not ever be exposed to the air.

It is recommended to place at least three samplers at each site. There is no exact standard for exposure time, but six weeks is recommended by many authorities (Klemm et al, 1990, American Public Health Association, 1985, and Weber, 1973). Whatever the exposure time, data should not be compared between samplers exposed for different lengths of time.

The best times for sampling invertebrates is spring/early summer, and fall. This is when there is the most activity and the highest abundance of invertebrates can be obtained. Remember that invertebrate assemblages will change through the seasons, so the time of year will affect the numbers and species of invertebrates obtained.

b. Retrieving Samplers

The method used to retrieve an artificial substrate is critical to the resulting quality of data. Some invertebrates will abandon the substrate as soon as the sampler is disturbed. For this reason, samplers should be retrieved with great care to reduce the loss of invertebrates from the sample. Ideally, the sampler is approached from downstream and enclosed with a U.S. Standard #30 or finer mesh bag or dip net before it is moved and brought to the surface. SCUBA or a remote enclosure system can be used in deep water. But, if these are not viable options, samplers should be pulled up with the least amount of disturbance and netted before reaching the surface.

The same procedures to deploy samplers should be used for all samples and sites that will be compared (e.g.,depth, exposure period, current velocity, sunlight exposure, habitat type).

4. Sample Processing:

After retrieval, multi-plate samplers can be placed directly into containers large enough to hold the sampler plates (one quart, large mouth jars or sturdy plastic bags) and preserved with 70-80% ethanol until processing; or they can be cleaned (organisms removed) in the field. Rock baskets are almost always cleaned in the field. The contracting laboratories will normally clean samplers if asked, but the work must be included in the contract, and will cost slightly more per sample.

Remember: If stored in preservative, substrates absorb the preservative and are considered unsuitable for reuse.

Alternatively, samplers can be stored in sieved, refrigerated water from the sampling site. Sorting and preservation should proceed within 24 hours of storage.

With all artificial substrate types, the enclosing net used during retrieval should be inspected for invertebrates and the recovered invertebrates included in the sample.

Always place a water resistant sample label inside the bag or jar as well as on the outside. Information should include: Site ID, location, habitat type, time, date and names of collectors.

a. Cleaning samplers

1) Disassemble the sampler and place plates, spacers or rocks in a tub of water (sieved to prevent introducing non-sample organisms if site water is used) and gently scrub plates or rocks into the tub with a soft bristled brush to remove all invertebrates.

2) Concentrate the resulting sample by pouring the tub of water through an appropriate sized sieve, and then place into sample containers with the appropriate preservative (10% formalin or 70-80% ethanol). With multi-plate samplers, the sampler is disassembled and each plate and spacer is inspected with the aid of a low power magnifier. All target invertebrates are then removed for preserved storage in 70-80% ETOH. The ETOH remaining within the sample containers is then poured through a #30 or #60 mesh sieve and all retained macroinvertebrates are included in the sample. The inside of the emptied sample container should also be inspected for invertebrates to be included in the sample.

3) For rock basket samplers, rocks stored in preservative are treated similarly to the plates in the above example. If only the organisms and associated materials from the rock surfaces were retained in ETOH, the ETOH/organism mixture is passed through a #30 or #60 mesh sieve. The retained organisms and debris are then transferred to a white pan for sorting, with the aid of a low power magnification device.

For quantitative work, the surface area of the artificial substrate sampler must be determined to allow for calculation of organism density per unit surface area.

b. Sorting and Subsampling

Refer to the sorting and subsampling parts in section **IV.E.Benthic Invertebrate Surveys - Benthic Samples.**

c. Taxonomic identification

Refer to the taxonomic identification part in section **IV.E.Benthic Invertebrate Surveys - Benthic Samples** above for procedures and a list of invertebrate taxonomy laboratories.

5. Documentation:

Information about the sample site and collection procedures are recorded on Department Form 3200-81, Macroinvertebrate Field and Bench Sheet. It is very important to label the specimen container(s) from each site with the corresponding Sample ID number that appears on Form 3200-81 for that site.

6. Quality Assurance:

To ensure quality data from artificial substrate samples, the investigator should be aware of the following:

a. Samplers should be retrieved very carefully to prevent the loss of organisms. Samplers should not be disturbed at all prior to retrieval (approach site from downstream) and should be enclosed during retrieval with a net having a mesh size at least as fine as U.S. Standard #30 or #60 or small enough to retain target organisms.

b. Multi-plate samplers or others made from porous materials should not be reused after exposure to oils/toxins in water or storage in preservatives.

c. Samplers to be reused should be inspected carefully to remove all organisms to prevent carryover to successive sample sites or exposure periods.

d. Artificial substrates may not sample all organisms found on natural substrates with equal efficiency. Use caution when interpreting data from artificial substrate samples. Refer to Chapter 5 of Downing (1984) for a more complete discussion.

e. Sampler to sampler variability can be estimated from results for replicate samplers placed at each location. Samplers should be placed so that they do not interfere with each other by changing water flow, light, siltation, etc.

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Many more taxonomic and other references are available in the scientific literature. An excellent summary of the literature for benthos is published by the North American Benthological Society in their annually published series "Current and Selected Bibliographies on Benthic Biology".

H. Laboratory Toxicity and Bioaccumulation Sediment Tests

1. Scope:

Toxicity and bioaccumulation tests on whole sediment are generally conducted to assess whether the sediment might adversely affect benthic and aquatic biota *in situ*. Sediments collected in the field are placed into exposure vessels with test organisms under controlled laboratory conditions. Various endpoints such as mortality, growth and reproduction are used to measure toxicity of the sediment. Toxicity test data can be evaluated in conjunction with resident benthic invertebrate community structure and bulk chemistry data to provide an integrative assessment of sediment quality. Chemical analysis is often performed on the sediment sample to provide a chemical data to compare with the toxicity and any other test results, although toxicity tests and chemical data alone should cannot reliably predict the contaminants responsible for observed effects.

Toxicity and bioaccumulation tests on sediment for the DNR are predominantly conducted at the Wisconsin State Laboratory of Hygiene - Aquatic Life Toxicity Testing Laboratory (ALTTL), also known as the Biomonitoring Lab.

2. Equipment:

a. Sample Containers

Clean, five-gallon plastic (polyethylene) pails with tight fitting lids are generally used to contain, transport and store sediments for toxicity tests. Clean pails are available from the Biomonitoring lab. These pails are used because enough sediment must be collected at each site for all replicates of toxicity tests and chemical and physical analyses. The sediment will be subsampled for the chemical and physical analyses at the Biomonitoring laboratory, so separate sample containers are not necessary.

The following steps for **cleaning** new or used sediment sample containers are recommended by EPA (1994):

1. Soak 15 min in tap water, and scrub with detergent.

2. Rinse twice with tap water.

3. Rinse once with fresh, dilute (10% V:V) hydrochloric or nitric acid. To prepare a 10% solution of acid, add 10 ml of concentrated acid to 90 ml of deionized water.

4. Rinse twice with deionized water.

5. Rinse once with full-strength, pesticide-grade acetone (use a fume hood or canopy).

6. Rinse three times with deionized water.

7. Rinse field collection equipment with site water immediately before use. Lab equipment should be rinsed with test dilution water immediately before use in a test.

b. Equipment List

1) Boat, waders, boots
2) Corer or dredge
3) Clean 5 gallon buckets with lids, or other sample containers
4) Permanent Marker for labelling buckets
5) Field sheets
6) Locating equipment, maps, GPS, compass, etc.

3. Sediment Collection:

a. Preparation

1) The quality of the samples collected is directly related to the quality and reliability of the resulting toxicity test data. A sampling plan and quality control measures should be decided upon and written down before sampling begins.

2) Toxicity and bioaccumulation tests must be scheduled ahead of time with the Biomonitoring lab so that sediment samples will not be held for longer than two weeks prior to the beginning of the tests. Because a finite number of tests can be run during any given week, the schedule of the lab and the collecting schedule should be coordinated.

3) After the tests are scheduled, the amount of sediment necessary to run all tests should be calculated, and sampling strategies for collecting adequate amounts of sediment should be determined. Remember, core or grab samples may need to be composited to obtain enough sediment from each site for the selected tests and analyses.

b. Collecting sediment samples

Collection procedures described in section **IV.C. General Sediment Sampling Equipment and Procedures** should be followed with the following exceptions and reminders:

1) Remember to collect enough sediment for all tests and analyses such as particle size, organic carbon content, moisture content, and chemical analysis.

2) The depth and type of sediment to be collected should be kept in mind when selecting sampling equipment. Usually the top 2-15 cm are the biologically active sediment layers of interest for toxicity and bioaccumulation tests, but deeper sediments may also be tested depending on the objectives of the study.

3) Sediment can be collected by any method that targets the study objectives, including the use of a grab or corer as described in section IV.C. More than one grab or core full of sediment may be necessary to obtain enough sediment for all tests and analyses. When this is the case, be careful to sample only undisturbed sediment with each grab or core attempt.

4) When possible, sites should be sampled in order of increasing contamination to reduce cross-contamination. All sampling equipment contacting the samples should be cleaned between discreet samples with a non-ionic detergent and distilled, deionized water if in the field. The following cleaning procedure is recommended by ASTM (1990), and may be used where if appropriate waste

handling containers and techniques can be followed: "1) soap and water wash, 2) distilled water rinse, 3) methanol rinse, 4) methylene chloride rinse, and 5) site water rinse." Waste solvents should be collected in labelled hazardous waste containers, not dumped at the site or on the ground.

5) If there is concern about oxygen-sediment interaction during transport, fill the sample containers to the top so no air space exists.

c. Chemical analysis of sediment and tissue samples

1) The biomonitoring lab will subsample sediments for chemical and physical analysis when the sediment is homogenized for the biological tests. Appropriate sample containers for each analysis must be provided to the Biomonitoring lab (see section **IV.C. General Sediment Sampling Equipment and Procedures**), and the DNR is responsible for getting sediment and tissue samples to the appropriate laboratory for chemical or physical analyses.

4. Description of toxicity and bioaccumulation tests

The DNR primarily uses the State Lab of Hygiene - Biomonitoring Laboratory to conduct toxicity and bioaccumulation tests on sediments. It is recommended that greater than three replicates per test be conducted since some statistical tests (Steel's Many Rank and Wilcoxon) show only borderline significance at 100% survival at the reference site and 0% survival at a study site. Available tests for sediment toxicity include:

a. 48-hour acute sediment toxicity test with *Daphnia magna*

Sediment volume* = 200ml/replicate x 3 replicates/site

b. 48-hour acute sediment toxicity test with *Ceriodaphnia dubia*

Sediment volume* = 5 ml/replicate x 3 replicates/site

c. 10-day survival sediment toxicity test with *Hyallela azteca*

Sediment volume* = 100 ml/ replicate x 4+ replicates/site

d. 10-day chronic sediment toxicity test with *Daphnia magna*

Sediment volume* = 200 ml/replicate x 3 replicates/site

e. 10-day survival and growth sediment toxicity test with *Chironomus tentans*

Sediment volume* = 100 ml/replicate x 4+ replicates/sample

f. Sediment Bioaccumulation Test with *Pimephales promelas* or *Lumbriculus variegaetis*

Sediment volume* = 2.4 L/replicate x 3 replicates/site

*** Additional sediment volume equal to one replicate is needed in each test for sediment chemistries.**

5. Quality Assurance:

The *in situ* toxicity of a sediment may be unavoidably altered by the manipulations of collecting, handling and storing sediment samples. Sediments *in situ* have a biological, chemical and physical integrity which affects the availability of contaminants and the subsequent toxicity to organisms. Thus, manipulation of sediments may increase or decrease the toxicity of sediment samples in the laboratory tests.

> "Subsampling, compositing, or homogenization of sediment samples is often necessary and the optimal methods will depend on study objectives. Important considerations include: loss of sediment integrity and depth profile; changes in chemical speciation by means of oxidation and reduction or other chemical interactions; chemical equilibrium disruption resulting in volatilization, sorption, or desorption; changes in biological activity; completeness of mixing; and sampler container contamination." (ASTM E 1391)

6. Documentation:

See Section **F.1.** on field notes.

All measurements and observations occurring during the laboratory tests will be documented by staff at the Biomonitoring lab and will accompany the test results.

7. Preservation and Shipping:

For preservation, sediment samples must be placed on ice as soon as possible after collection and maintained on ice or refrigerated at ~4°c until tests are begun. Sediment suspected of containing volatile organic chemicals should be packed into sample containers so that no air space exists. This should reduce the chances of oxidation of the sediment.

If samples are of suitable size for shipping, pack them to prevent breakage and with enough ice or cold packs to maintain the preservation temperature of ~ 4 °c. Make sure melting ice cannot leak during shipment.

If hazardous samples are to be transported or shipped, consult 49 CFR 100-177 for current Department of Transportation regulations.

8. Data Reporting and Analysis:

The following data are reported by the Biomonitoring Lab for each site and/or replicate depending on the test:

a. Initial and Final Chemistries and Measurements

Dissolved Oxygen (DO), pH, Conductivity, Alkalinity, Hardness, Total Ammonia, Un-ionized Ammonia, temperature, total suspended solids, **percent survival, mean length and weight, # of young produced.**

b. Statistical and Biological Significance

The replicate data are used to statistically test for significant differences between results from reference and test sites. At least 70% of the control organisms must live for the test to be valid. If too many control organisms die, this indicates that a factor other than toxicity of the test sediments is contributing to organism mortality and something has gone wrong with the test. The test is technically invalid, and use of the data should only be used with discretion.

Questions about results or testing procedures should be directed to Steve Geis at the Biomonitoring Lab. Phone: (608) 265-4023.

9. References

ASTM. 1991. Standard guide for collection, storage, characterization, and manipulation of sediments for toxicological testing. Method E1391-90. In: Annual Book of ASTM Standards, Water and Environmental Technology, Vol. 11.04. American Society for Testing and Materials, Philadelphia, PA.

EPA. 1985. Sediment Sampling Quality Assurance User's Guide. 600/4/85/048. Environmental Monitoring and support lab, Las Vegas, NV.

EPA. 1994, (In-Press). QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations. Phase I - Chemical Evaluations. U.S. Environmental Protection Agency, Office of Water, Washington DC.

WDNR. DRAFT 1990. Quality Assurance Guidance for Inplace Pollutant Monitoring Activities. Unpublished document on file at Office of Technical Services, Bureau of Water Resources Management.

H. In Situ Bioaccumulation using Caged Fish

1. Scope

In situ bioaccumulation tests are used to assess if sediment borne contaminants are potentially bioavailable to aquatic biota under the conditions in the field. An *in situ* test bypasses the problem of possibly skewed results caused by sediment manipulation during collection and laboratory testing. Disrupting the physical and chemical integrity of the sediment can change the bioavailability of contaminants. *In situ* tests cannot, however, control for biouptake of contaminants that might come from water or food, or other factors such as temperature that might affect bioavailability and bioaccumulation.

Many different methods can be used to test for bioaccumulation in situ. This section offers one method utilizing caged fathead minnows, and should provide the first-time field person with a guide to plan ahead and avoid many pitfalls that might compromise the quality of the study results. With common sense and an understanding of the principles, this method can be modified to fit different sites and study objectives. Variations include the use of other fish or invertebrate species and suspending the cages in the water column rather than resting on the sediment.

2. Equipment

Equipment List

1) Fish cages with weights
2) Cable ties or wire to close cages
3) Suspension or anchoring materials
4) Buoy and rope for marking cage locations
5) Live, healthy, adult fathead minnows
6) Apparatus to keep fish cool and aerated until placement
7) Wire cutters and/or knife and scissors
8) Field measurement equipment
9) Fish nets
10) Large tub for water
11) Pump or bucket for filling tub
12) Gloves
13) Aluminum foil
14) Dry ice and cooler
15) Permanent marker and labeling tape
16) Cleaning equipment and solutions
17) Field sheets and/or field notebook and pencils
18) First aid kit

3. Method

a. Preparation and Considerations

1) The most commonly used exposure times used for bioaccumulation in fathead minnows are 10 and 28 (or 30 days) from the time of placement to the time of collection. For most bioavailable

contaminants, equilibrium will not be achieved by 30 days, so tissue concentrations will increase throughout the test period.

2) Twenty grams (wet weight) of fish tissue are needed for sample to be analyzed for chemical contaminants. Most adult fathead minnows weigh 1-3 grams. At least twice as many fish as are needed should be ordered for each site. This leaves some room for fish mortality.

3) A source of uncontaminated, healthy fish is essential to the ability to discern significant biouptake of contaminants, especially if the sediment contaminant levels are moderate. Finding a reliable source of uncontaminated fish can be difficult, but is necessary. The concentration of PCBs in fish from a single hatchery can change throughout the year. PCBs have also been detected in laboratory raised fish and fish food used at hatcheries. Contamination with metals (esp. mercury) has occurred during a two week holding period while being fed frozen brine shrimp that itself contained mercury. The level of mercury in these fish was significant and possibly occluded or shadowed any bioaccumulation of mercury that might have originated in the sediment.

4) When planning the analyses and budget, it is a **requirement** that enough analysis dollars to analyze "blank" (control samples, time = 0) and reference site tissue samples be available. Without a reliable "blank" sample, there is no way to know if fish tissue concentrations increase during the testing period from the time of placement to (time=0) to the time of collection (usually time=10 or 28 days). Fish tissue should be collected for analysis immediately before the rest of the fish are placed at the sediment sites to determine the pre-exposure contaminant concentrations. Remember that any other containers or food introduced to the fish can be a source of contamination.

5) Make sure enough minnow cages are available for the sampling period.

6) Make sure the cages are sufficiently weighted or anchored to remain in position during rough water or currents.

7) Arrange for the most convenient time and place for the delivery of the fish to reduce the necessary holding time and stress. A method to keep fish cool and the water aerated will reduce fish stress and loss.

8) A placement and sampling plan including quality control measures should be written down to avoid pitfalls and unreliable data.

9) Choose sites containing soft sediments. Unless the study objectives specifically dictate otherwise, sites for *in situ* bioaccumulation tests should contain fairly soft sediments to allow the fish to interact directly with the sediment through the mesh of the cage. Bioaccumulable contaminants are most often associated with fine sediments, and unless they partition readily into the water column, must make direct contact with the test organism to be bioaccumulated.

10) Choose sites where there will be adequate water to completely submerge the cages even in low flow. Also, some sites in silty rivers or streams may lead to a mortal buildup of silt in and around the cage.

11) Sites should not be located near a known toxics discharger if a measure of bioaccumulation from this point source is not desired.

b. Placing Cages

1) If direct interaction between the fish and the sediment is desired, cages should be placed on a soft sediment deposit rather than hard sand or rubble.

2) Anchor the boat so that the sediment where the cages will be is not disturbed, and the boat is directly over the cage sites.

3) Upon arrival at the site, begin acclimating fish to the site water (both chemically and thermally) by adding site water every few minutes to the fish, until they are in at least 50% site water. The more difference there is between the fish water and site water temperatures, the more gradual should be the process.

4) Fill a tub large enough to hold a cage with water, and place a cage in it, so that the fish will be in water while the cage is being filled.

5) When the fish are acclimated to the site water, fill the cage with the appropriate number of fish, and secure the lid with cable ties or wire.

6) Record the number of fish placed and any observations.

7) Attach sufficient line to the cage and lower to the bottom gently so that the bottom of the cage or one full side is lying directly on the bottom. The line can be attached to a stationary object or to a buoy for retrieval of the cages. Leave enough extra line from the cages to the surface float so wind and wave action will not move the cages. A separate anchor for the buoy with a line slightly shorter than the cage lines should help prevent this.

8) Repeat with any addition cages for that site. Replicate cages should be at least a few feet apart from each other and should not affect the flow of water around the other cages.

c. Collecting Fish

1) Be aware of all possible routes of sample contamination especially during the collection procedures. All equipment and gloved or bare hands that touch the fish should be totally clean. Be aware of a possible petroleum slick from the boat motor; approach site and anchor boat so motor is downwind and/or downstream from the cages and work area. Ideally, the person operating the motor should not contact the sampling equipment or fish unless some cleansing procedure is used. All equipment should be washed or very well rinsed between sites, especially if some sites are obviously contaminated or an oil sheen develops when the sediment is disturbed.

2) Take and record any field measurements before raising the cages and disturbing the site.

3) Raise a cage and place into a tub of clean, fresh (same temperature) site water. If subsampling and some fish will remain at the site until a later time, fish should be held only as long as necessary to minimize stress.

4) Make observations about the general health or appearance of the fish and the number dead and alive and record. The overall well-being of the fish at a site as well as mortality may be a clue to some toxicity.

5) If subsampling, count and collect the necessary live fish in as random fashion as possible with a clean net, and place the fish in fresh aluminum foil and double wrap. Dead fish and dropped fish should not be included as part of the sample. Remember to leave enough fish for the final or subsequent samples with the possibility of some additional mortality.

6) Tape the package closed and label appropriately: Site ID, contents (# and species), date, collector's name, analysis type (inorganic or organic).

7) Place packaged fish on dry ice as soon as possible to freeze and preserve fish for later analysis. Fish tissue must be kept frozen until delivered to the lab for analysis.

4. Documentation

a. Laboratory Sheets - Laboratory form #'s 3200-82 and 3200-83 are specifically for fish tissue and must be delivered with the fish tissue samples to the State Lab of Hygiene. Record and keep a copy of sample information (number of samples, sites, field IDs, blanks, etc.) in addition to the lab sheets that will be sent with the samples to the lab. The lab sheets will not be sent back with the data, and the information will aid in tracking samples at the lab to see if analyses have been completed or not. This information will be keyed into the new Fish/Sediment Contaminant data base.

b. Field Measurements - It is important to measure and record parameters at each site that can affect bioaccumulation at the site. These might include: water temperature near the sediment, dissolved oxygen near the sediment, pH, water depth, conductivity, light attenuation, current velocity. Knowing these parameter can provide further information along with the data about the potential for bioaccumulation *in situ.*

5. Selected References

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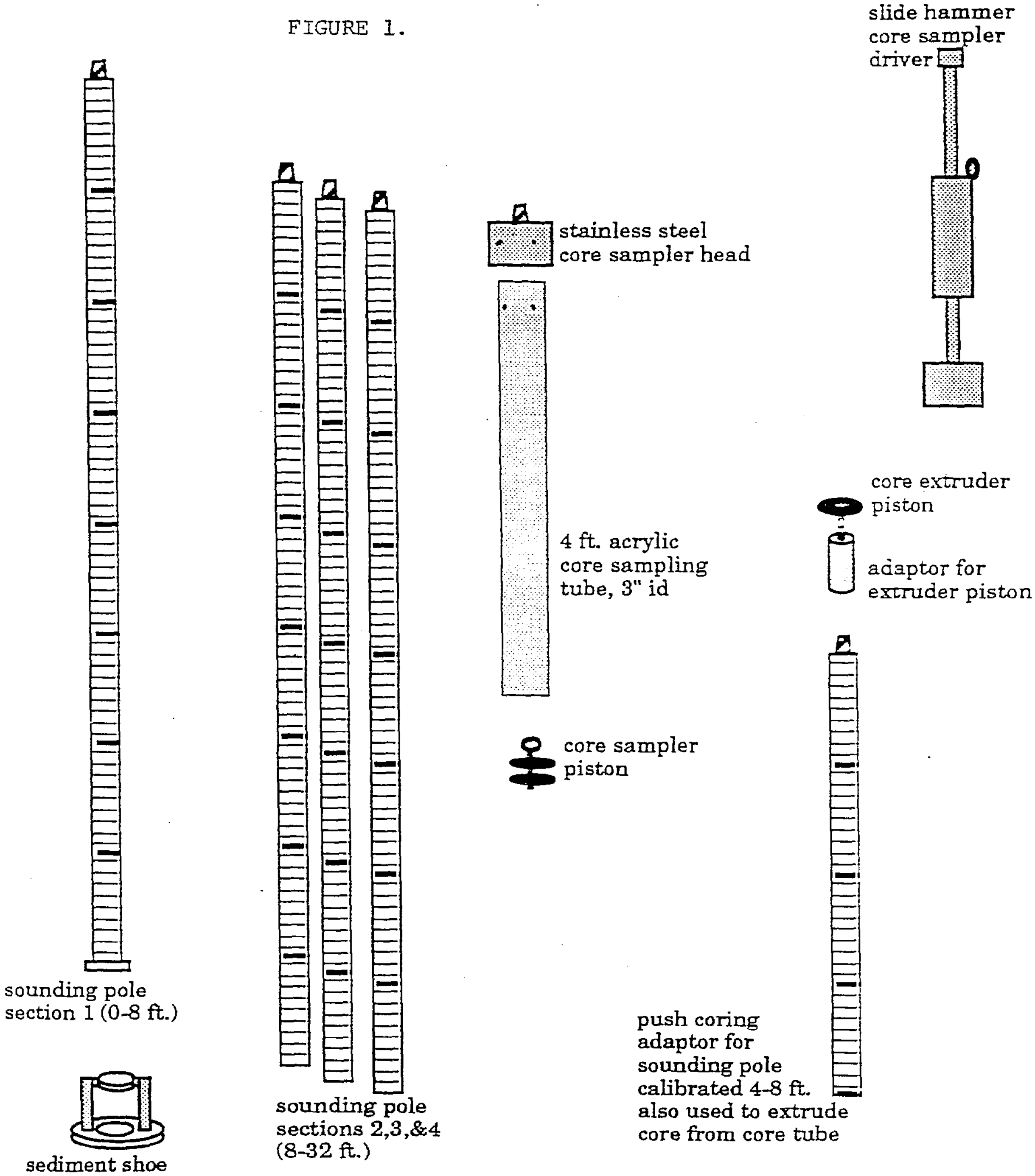
EPA. 1989. Guidance manual: bedded sediment bioaccumulation tests. ERL-N Pacific Ecosystems Branch. EPA/600/x-89/302.

Mac, M.J. and C.J. Schmitt. 1992. Sediment bioaccumulation testing with fish. *In* Allen B.G. (Ed.), Sediment Toxicity Assessment. Lewis Publishers. Boca Raton, Florida. pp 295-312.

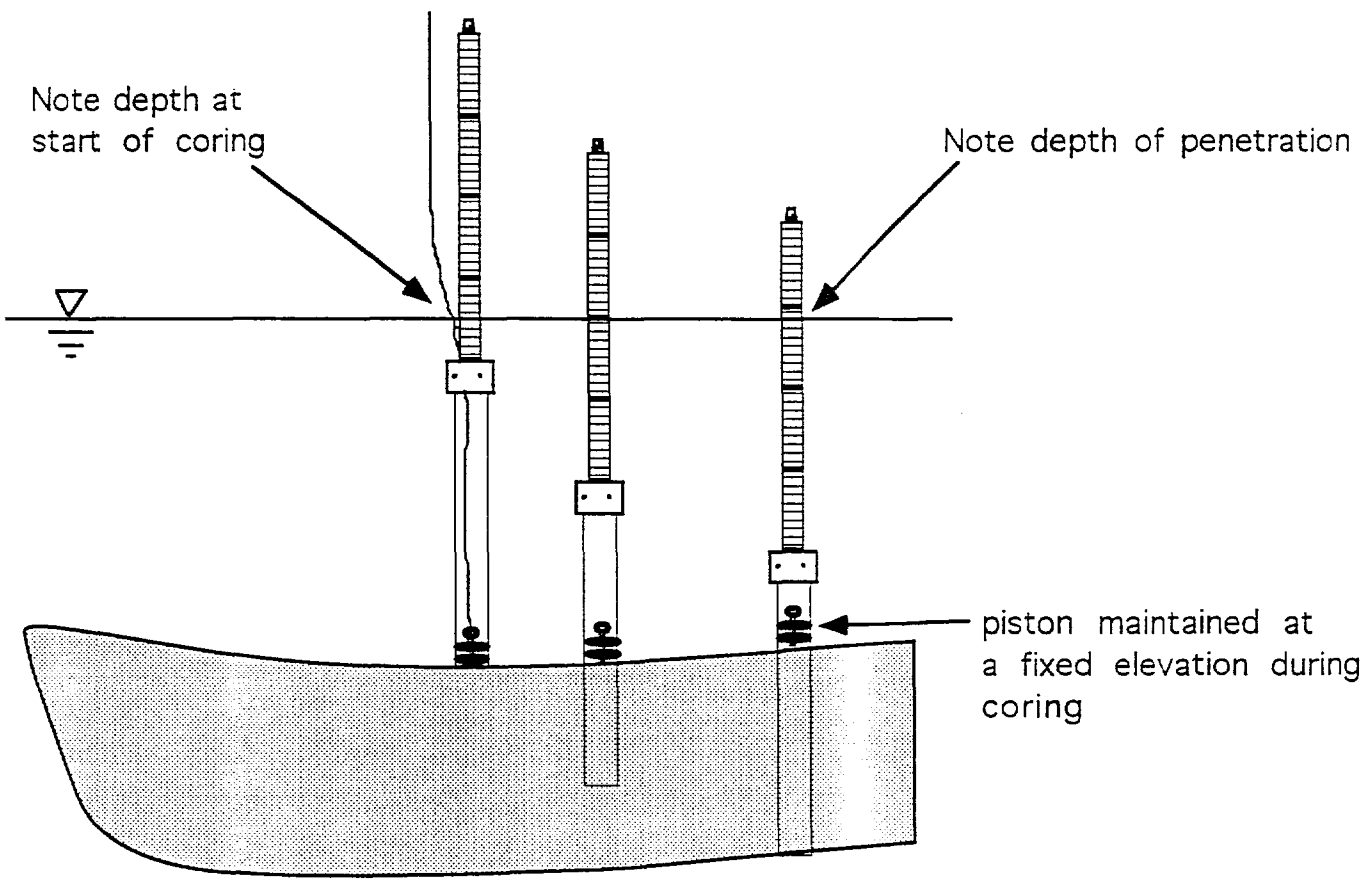
Appendix A

FIGURE 1.



SMART SEDIMENT SOUNDING POLE/CORE SAMPLER

FIGURE 2.



1) Recovery ratio (R_r) = recovered core length/total penetration of corer
2) Smooth and consistent driving force
3) Avoid bow-wave disturbance-slowly approach sediment surface
4) Do not overdrive corer; maintain headspace above piston
5) Maintain piston at a fixed elevation during driving