

METHODS MANUAL FOR BOTTOM SEDIMENT SAMPLE COLLECTION

**by**

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## INTRODUCTION

### Great Lakes Harbor and River Sediment Sampling Program

Toxic substances are being introduced into the environment from many sources. Secondary compounds from these toxicants are often formed in the environment. Some of these secondary compounds are more hazardous than the primary chemicals, from which they came.

Sediments serve as a sink as well as a potential source for toxic and conventional pollutants. Even if discharges of pollutants are completely eliminated; contaminated sediments can serve as a continuing *source* of pollution to aquatic life, the Great Lakes, and the populations using the water bodies for drinking water supplies. If one names the toxic substance problem areas around the Great Lakes - Waukegan, Indiana Harbor Canal, Ashtabula, Saginaw River and Bay, Sheboygan River, Green Bay, Milwaukee, Buffalo and Niagara River, the "problem" is invariably linked with toxics in the Sediments.

Some 10 million cubic meters of sediments are dredged annually to maintain navigation-in Great Lakes' ports. Many of these ports contain sediment contaminated with toxic substances. Environmentally safe dredging and disposal is necessary to protect the lakes, wildlife, and the public while maintaining the economic viability of water borne commerce,

A large data-base on levels of sediment contamination by conventional pollutants (volatile solids, nutrients, metals) in Great Lakes exists from past USEPA monitoring and Corps of Engineers' monitoring of federal navigation channels. This data base has allowed the development of guidelines for determining the relative level of contamination in sediments and has been used to evaluate the suitability of sediments for open lake disposal. This data base may be the largest consistent (sampling methods and analytical methods) sediment contamination data

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base in the country, representing over 100 harbors and rivers. The data has been widely used for dredge and fill review, ranking of problem areas, reporting on environmental status of areas, etc. Data gathered in the program has helped detect or verify toxic substance problems at Waukegan (PCBs) Marinette-Menominee (arsenic), Ashtabula (PCBs, HCBs), Detroit River and Lake St. Clair (mercury), Sacketts Harbor (mercury), Green Bay (mercury), etc. In a number of cases the information stimulated remedial actions.

Due to the relatively recent identification of in-place pollutants as major remaining sources of contaminants and the availability of the analytical capability to allow the measurement of toxic organics, only a very limited and disjointed data base presently exists for organic contaminant levels in sediments. To fill the void, Great Lakes National Program Office (GLNPO) is working to determine the degree of contamination of Great Lakes river and harbor sediments by toxic substances. Sampling priorities are being determined by examining fish flesh contaminant data, locations of likely industrial sources, and by review of USEPA and other agency data.

The information generated by this program will be used in making regulatory decisions on dredging and disposal and to identify environmental "hot spots" requiring further remedial activity including identification and control of sources. Chemicals monitored in the sediments will form a new information base for the Great Lakes. Selected samples will be scanned for organics and metals using best available methods. The organic scans involve acid, base, and neutral extraction of volatile and non-volatile substances. Quantification is routinely done by gas chromatograph electron capture technology (GC/EC) for PCBs and 30 some pesticides.

The basic tests in current use for the pollutional evaluation of bottom sediments include:

1. The field observation of the sample for color, texture, odor, oil, benthic organisms, detritus, etc.
2. Bulk sediment chemical analysis of the sediment.
3. Sediment size distribution.
4. An elutriate test of the sample mixed with water from the dredging site.
5. Macroinvertebrate identification and enumeration.
6. Sediment bioassay using sediment together with macroinvertebrates, periphyton, and fish to determine relative toxicity.

## 2. Purpose

The purpose of this Methods Manual is to provide detailed procedures for survey planning, sample collection, document preparation and quality assurance for sediment sampling surveys conducted by the Great Lakes National Program Office. The data base will be utilized, if need be, to institute a remedial action program in accordance with USEPA policies and procedures guidelines.

In order to remain current, this manual will be reviewed and updated on an annual basis.

## 3. SAMPLE SITE SELECTION

Each site survey is designed by determining and plotting on a large scale map the location of sewage treatment plant discharges, combined sewer discharges (particularly those carrying industrial waste), industrial discharges, and any other feature that might result in contaminated sediments. To this is added any data on sedimentation patterns that may exist from dredging records, and

existing data on sediment quality: This information is used to identify locations where contaminated sediments are most likely to be found.

Two categories of sampling sites are selected. Primary sites are sites that are most likely to be contaminated and all primary sites samples will be scanned and run for specific compounds which are known to be used in the area or have been found in fish from the area. Secondary sites are sites which will be run if the primary sites indicate a significant problem exists and will be used to define the extent of the problem. Secondary samples would only be analyzed for the specific compounds indicated as problems at primary sites. Compounds for specific analysis at primary sites will be selected based on data from fish and from data on industrial and agricultural usage in the area.

In general, the finer and more polluted sediments will deposit along the edges of a navigation channel, on the inside edge of a curve in a river, and on the down drift side of the littoral drift beach zone. Samples should, therefore, generally be collected in these areas rather than mid-channel. Sounding charts are extremely helpful for sample site selection since they show the areas requiring the most dredging and, therefore, where the shoal material is depositing. On a straight channel, lacking sounding information, a good approach is to select sites on alternating sides of the channel.

Areas likely to show the pollutional effects of man's activity should be sampled. Therefore, where applicable, sample sites should be located in the vicinity of marinas, loading docks, industrial or municipal outfalls, etc. If only a limited number of PCB and pesticide samples are to be collected, they should be collected at these sites.

If an open water disposal site may be used for the dredged sediment, sample sites in the disposal site may be appropriate.

If a sediment bioassay is to be performed, sediment samples from "unaffected areas" in the lake should be collected for use as "control" sediments in the toxicity testing.

#### 4. SAMPLING EQUIPMENT

Where the bottom material is of recent origin, such as in areas dredged recently, surface grab samples are usually sufficient. Where there is likely to be stratification of different sediment types with varying pollutional levels, such as in areas where dredging has not taken place for a long time, core sampling is recommended. The length of the core should approximate the depth of the unconsolidated deposits., The final choice of sampler to be used will depend upon the known or suspected characteristics of the area to be sampled. Currently, a 2 inch diameter stainless steel corer with a CAB plastic liner is used for all samples whenever possible.

Surface grab samples are collected when the coring device is ineffective (sand or gravel), or when large quantities of surface material are required (benthos collection or bioassay).

Macroinvertebrates are separated from the sediment by washing on a U.S. standard No. 30 sieve.

##### 4.1 Core Sampler

The stainless steel corer utilized by GLNPO is approximately 4 ft in length and has a 2 inch inside diameter. A weighted free fall through the water generates the force to penetrate the bottom sediment. A bronze nose flutter valve prevents the material collected from dropping out. Thus once the sediment is in the corer it will remain in the corer, unless it is very watery (non-cohesive).

If the sample is brought back in its liner, the core liners should be labelled with a sample tag (Figure 6) including date, time, sample location, the number of the core if multiple cores are collected at the same site, and the person collecting the sample. The liners should be labelled to indicate which end is the top of the core since this may not be obvious from the appearance of the core.

The core diameter and the vertical resolution desired (i.e., 2 inch vertical layers, 1 foot vertical layers, etc.) will determine the number of cores required at a sample site to provide sufficient sample material to perform the desired chemical tests.

Core samples are usually best collected and stored using a core liner which is capped at both ends for transportation and storage. Metallic liners (stainless steel preferred) or Teflon liners should be used if PCB and pesticides analysis is to be performed on the sample.

Experience with plastic liners, to date has revealed no contamination problems with regard to PCBs and pesticides, but before extensive use of a particular material, that material should be ground up, mixed with sediment and the results compared with the results from the sediment prior to mixing.

Core samples are usually best left in the core liner for processing at the laboratory. If this is not possible due to the type of corer used or the excessive length of the cores, the cores may have to be extruded in the field with a solid extruding rod (Figure 8), cut in about 30 cm, or less lengths and placed in sample jars with Teflon lined caps. In this case, upon being extruded, the

samples are handled in the same manner as discussed below for surface grab samples. Particular attention must be paid to properly labelling the sample jars that will contain the various core vertical sections. The labels should include the sample site, the number of 'the core if multiple cores will be taken at the same site, and the vertical section of the core (i.e., 0-6 inches, 6-12 inches, etc.).

#### 4.2 Ponar Dredge

The Ponar dredge is an aluminum/steel device also used in collecting sediment samples whenever a corer cannot be utilized. The jaws of the Ponar dredge, which close on the bottom, provide a sharp cutting action. The wide jaws functions to prevent stones from jamming the shutting mechanism. The screen on the top serves to reduce the sediment loss due to the pressure-wave in front of the dredge upon descent.

Upon retrieval, the sample should be emptied into either a metal (bioassay, PCB or pesticides sample) or polyethylene (all other samples) tub. The field observations should be recorded as discussed later in "Field Observations". Additional pertinent information that should be recorded includes the number of grabs used if a composite sample was necessary to obtain sufficient sample material and a separate description of any grab in the composite which differs markedly from the other grabs in the same composite (see figure 2).

Care should be taken to assure that a representative sample is taken. Therefore, it is recommended that the sample be thoroughly mixed in the tub prior to transfer to a sample jar.

Bioassay samples should be processed using a stainless steel spoon or spatula. Multiple grabs will likely be required to obtain sufficient sample volume. The sample should be appropriately labelled and put in an ice chest with ice (see "Preservation" section).

#### 4.3 Macroinvertebrate samples

Samples must be washed through a U.S. Standard No. 30 sieve. The material that is retained on the sieve after thorough washing should be spooned or transferred with tweezers to the sample jar. Care must be taken to avoid injury to the benthic organisms when transferring them to the sample jar.

The appropriate label should be filled out and affixed to the sample jar. If more than one jar is necessary for a sample, all tags should include a notation such as "1 of 3, 2 of 3, . . .". The jars do not have to be placed in an ice chest but they may be for convenience.

#### 4.4 Safety Equipment

The field personnel will follow all the safety procedures to assure an accident free working environment. They will utilize floatation devices, wear rubber gloves, and steel-toe shoes.

The equipment needed on sediment surveys will vary, depending on the area and the purpose, but a check list such as Figure 1 should be used to assure that needed equipment/supplies are not over-looked.

## 5.0 Sample Containers

Any container material can be criticized for some reason, legitimate or not, unless adequate quality assurance, in conjunction with the sampling is used to show that contamination of the sample and loss or degradation of the sample parameters is insignificant. For all sediment samples glass jars with teflon lined lids, are generally considered satisfactory. The major disadvantage of glass is that freezing samples in the jars will usually break the jar. The suitability of various plastics for use as containers for PCB, pesticides, and bioassay samples has not been adequately studied to warrant their use. Polyethylene and polypropylene are generally considered acceptable for most inorganic chemical, physical and biological parameters. Teflon is generally considered satisfactory for virtually every parameter, but the extreme cost of Teflon makes its use limited.

For organics samples, the sample should be transferred from the metal tub to the glass sample jar using a metal spoon or spatula. The glass jar should be pre-washed as outlined below. An aluminum foil square should be placed over the mouth of the jar (overlapping about 2 inches) or a Teflon liner inserted in the cap before sealing with the cap. The sample jar should be labelled (see Figure 6) and placed in an ice chest (see "Preservation" section).

In some cases, such as bioassay and elutriate water, quality assurance studies for sample Containers have not been performed. GLNPO advocates the use of either glass or polyethylene for elutriate water and glass or stainless steel for bioassay samples.

Aluminum foil has been used for lining caps for sediment samples in the past, apparently with no adverse effects. Beginning with the October 1982 survey, teflon lined closures will be used exclusively for all sediment samples.

Occasionally glass sediment sampling jars have been prewashed with soap and water or with hexane, but for the most part, new unwashed jars have been used, with at most a thorough rinsing with water from the sampling site. Since no adverse effects have been noted from this procedure, it is now the standard operating procedure. In summary, at present new (unused) jars are pre-rinsed with water from the sampling site and Teflon lined paper backed lids are used for closures.

#### 6.0 SAMPLE COLLECTION

If samples are to be collected from a small boat, the location of the sampling can be defined by compass point to nearby landmarks or navigation buoys, rangefinder readings to nearby landmarks, fathometer readers, Loran C readings, visual estimates or various combinations of these.

Depending on wind velocity or difficulty of obtaining a sample it may or may not be necessary to put down an anchor to maintain position while sampling. The core sampler may be allowed to free-fall from a certain distance above bottom or it may be lowered fairly rapidly. The advantage of a fairly rapid versus lowering free-fall, on the present winch, at least, is that it reduces the incidence of tangled cable. It may also reduce the incidence of smashed or bent nosepieces.

As indicated under equipment, coring is the preferred method of sampling for priority pollutants.

If elutriate tests will be performed on sediments, water samples for the elutriate test should be collected at a depth of 1 meter from the bottom in an area representative of that which will be dredged. A non-metallic sampler should be used. The sample bottle should be placed in an ice chest for storage (see "Preservation" section).

## 7.0 FIELD OBSERVATIONS

Observations on a sediment sample in the field form an important part of the pollutional evaluation. Comprehensive field observations are a very valuable aspect of the survey for sediments.

Field observations that should always be recorded include sampling date and time; sampling site significance and location; sampling depth; color, texture, odor, presence of oil in the sediments; and the absence or presence of benthic organisms, as well as their identification, if possible. Figure 2 & 3 show an example of a properly completed observation sheet. The field logbook is also utilized (Figure 4).

At surface sampling sites where macroinvertebrate samples will not be retained for further analysis, it is useful to sieve a portion of the sediment sample through a U.S. Standard No. 30 sieve as an aid to completing the entries for observed benthic organisms on the field observations sheet.

Notations of any unusual conditions encountered should also be recorded on the field observation sheet. This can include, for example, adverse weather conditions, equipment malfunctions, frequent passage of shipping traffic, etc.

Further notations to be made depend upon the sampling equipment used or the type of samples being collected are discussed in the appropriate sections elsewhere.

In order to achieve consistency in recording such data, guides to classifying sediment types and odors are given in Tables 1 and 2. In the case of odors, the abbreviated codes given in Table 2 can be used for a kind of shorthand notation. Additional pertinent information that should be recorded includes the number of attempts required to obtain a useable core, the approximate penetration depth of the corer, loss of sample through top or bottom of sampler upon retrieval, etc.

#### 8.0 Preservation of Sample

Except for macroinvertebrates samples, the samples should be placed in an ice chest with wet ice for storage. The chest should be refilled with wet ice as necessary to keep the chest contents cold, at approximately 4°C to prevent biological activity and chemical reaction.

The appropriate label should be filled out and affixed to the sample jar. If more than one jar is necessary for a sample, all tags should include a notation such as "1 of 3, 2 of 3, . . .".

For macroinvertebrates samples, the preferred preservative is 70% ethanol. A 70% ethanol solution is approximated by filling the jar, containing the sample, half full and a small amount of rinse water, with 95% ethanol.

If formalin is used for the macroinvertebrates, the jar should be filled about one-half full with sample. Sufficient rinse water should be added (or already present) to assure that the sample is suspended in water. Formalin should then be added to 5% by volume, i.e., add about 25 ml formalin per pint of sample (including rinse water).

Once preserved as above, the samples do not have to be kept cold. For convenience, they may be kept in the same ice chest used for the samples discussed above. However, the macroinvertebrate samples should not be frozen.

The required field preservations can be summarized as follows:

<u>Preservation</u>	<u>Sample types</u>
Iced, at about 4°C, but not frozen	Bulk sediment chemical analysis Elutriate test sediment samples PCB and pesticides samples Water for the elutriate test Bioassay samples
70% ethanol or 5% formalin	Macroinvertebrate samples

## 9.0 SAMPLE HANDLING AND CUSTODY PROCEDURES

### 9.1 Introduction

As in any other litigation, the USEPA must be able to prove that any analytical data offered into evidence in a court of law accurately represents environmental conditions existing at the time of sample collection. This implies that it can be clearly demonstrated that none of the involved samples could possibly have been tampered with during collection, transfer, storage or analysis. Therefore, an accurate written record must be maintained to trace the possession of each sample from the moment of it's collection through it's introduction into evidence. Samples for which this accurate documentation is maintained are called custody samples.

Since the USEPA, Region V, performs the same basic sample handling operations on both known litigation and non-litigation samples all sediment samples collected by the SRS will be collected and handled according to the standard custody procedures. This does not apply to GLNPO's lake surveillance water samples.

## 9.2 Sample control/holding procedures

A sample is physical evidence collected from a facility and/or from the adjacent environment. An essential part of all enforcement investigations is that evidence collected be controlled. To accomplish this, standard operation procedures for sample handling and chain-of-custody have been developed. Accordingly, these procedures shall be utilized for all sampling situations and sample types (parameters and preservative types) carried out by the SRS staff on sediment surveys.

Environmental samples may represent several media/ matrices or mixed media/matrices types; e.g., water, fish, sediment, air, soil, oil, and water, etc. Some of the desired measurement parameters may be completed in the field (e.g., pH, temperature, flow measurement, etc.). All field measurements will be recorded (in the field at the time of measurement) directly in serialized Field Logbooks or on field data record forms. (Field Data Record Sheet and logbook format are shown in Figures 2 & 4.)

Samples other than the in-situ and in-field measurements will be identified by the National Standard format tags (see Figure 6) with all information filled out as appropriate and indicated.

These samples are removed from the sample location and transported to a laboratory or other location for analysis under proper preservation and ship-

ping procedures. Sample tags shall be completed for each sample, using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather. The information recorded on the sample tag would include:

- Project Code - A number assigned by Environmental Services Division (ESD) and also serves as the Document Control number for the survey.
- Station Number - A number assigned by the Project Leader using the, CRL Log Number system.
- Date - A number, indicating the year, month, and day of collection.
- Time A four digit number (XXXX) indicating the military time of collection-for example: 0954
- Station Location- The sampling station description, as specified in the project plan.
- Samplers - Each sampler signs.
- Tag Number - A unique serial number is stamped on each tag.
- Remarks - The samplers record, pertinent observations and sample type; i.e., water, sediment, fish, etc.

Referencing the example sheet of the Field Logbook, (see Figure 4) the responsible field staff will enter both formatted information (required entries) and observational information (judgemental data). The Field Logbook will be serially numbered and unique to each survey/project.

During collection, separation, identification, and preservation, all samples will be maintained under Chain-of Custody procedures discussed later. If the composite or grab sample is to be split, it is aliquoted into similar sample containers. Identical sample tags are completed and attached to each replicate

and marked with a "A" in the sample number. The tag identifies the replicate sample for the appropriate government agency; facility, laboratory, or company. In a similar fashion, all tags on blank or duplicate samples will be marked with an "R" or a "D", respectively. An explanation of the numbering system follows.

The sample type letter is used to identify quality assurance and other sample types. The following letters are fixed and are to be used only as specified:

- S = Sample
- D = Duplicate Sample (two samples collected)
- A = Duplicate Analysis (one sample split)
- L = Laboratory Control Standard
- R = Reagent Blank (Field)
- B** = Reagent Blank (Laboratory)

All other letters may be used as the Project Officer wishes, after clearing with the CRL Sample Custodian.

The sample numbers should be assigned in numerical order to all samples collected during the specified survey. If more than 99 samples are collected during a given survey, a new survey number should be used as required to uniquely identify all samples. Quality Assurance samples should receive unique numbers with duplicates being always for the preceding sample.

Additional examples are given below to further explain the system.

Sample Number "GP01S01"

WHERE: G = Great Lakes National Program Office  
P = Marvin Palmer  
01 = Palmer's first survey in FY 82  
S = Sample  
01 = First sample collected for project 01

Sample Number "GP01D06"

WHERE: O = Great Lakes National Program Office  
P = Marvin Palmer  
01 = Palmer's First Survey in FY-82  
D = A Duplicate Sample of Sample Number GP 01S05  
06 = The Sixth Sample in Project 01

During the time that the environmental samples are collected, the proper aliquots are prepared and properly preserved, an analysis request sheet is completed, commensurate with the desired parameters for each discrete aliquot. The analysis request forms should have parameters listed that match with the parameters checked off on each sample tag, on each sample aliquot. (In the future, the CRL will generate the analysis request sheet by computer.) Most sample data will be entered onto these analysis request sheets by the CRL and returned to the data user.

All field collected samples requiring shipment from the field to an USEPA laboratory or to a centralized location, and/or shipment to a contractor's laboratory will be shipped in compliance with all applicable D.O.T. regulations, preservation requirements, and USEPA safety requirements. An overview of these procedures are listed below.

#### 9. 2. 1 PROCEDURE FOR HAZARDOUS AND NON-HAZARDOUS WATER AND SEDIMENT SAMPLES

These samples generally are collected in one (1) pint, one (1) quart, one (1) gallon, or two and one-half (2 1/2) gallon glass containers.

Forty-eight (48) or seventy-six (76) quart capacity plastic picnic coolers should be used to ship the samples.

Samples are placed in the picnic cooler in an upright position and separated by Styrofoam sheets of 1" to 3" thickness. Alternately, cardboard sections are placed in a manner so as to keep the glass sample bottles from "banging up" against each other, both sideways and from the top and bottom.

After the Styrofoam or cardboard is placed in the picnic cooler, additional packaging material consisting of "peanut", "popcorn" absorbents or "bubble" plastic sheets are used to further cushion and compact the cooler so that movement is minimized.

Volatile organic samples (40 ml VOA vials) are wrapped in the "bubble" plastic sheets and placed in one corner of the picnic cooler to prevent breakage and leakage. Paperwork to be shipped with the samples is placed in a plastic ziplock bag and sealed with tape. Liberal portions of ice, crushed or cubed, are added to fill the cooler and a cardboard sheet placed over the ice and the picnic cooler is sealed. The cardboard serves to prevent breakage if the cooler is dropped, either in an upright or upside down position. D. O. T. regulations require packaging to withstand a four foot (4') fall. The above packaging methods achieve this requirement. The picnic cooler is sealed with filament tape completely around all edges and the custody seal is placed on both sides of the cooler and taped once so that when the picnic cooler is received, the receiver can readily check to see if the seal has been tampered with.

On the outside of the chest, a sticker indicating "THIS SIDE UP", "WATER SAMPLES", "FLAMMABLE" or "HAZARDOUS MATERIALS", "GLASS", or "FRAGILE" will be

attached to the sides and top of the cooler (to assure that any warning notice can be clearly recognized by the courier):

The custody seals should be covered with clear plastic and tape to decrease the chance of accidental breakage during transfer.

When shipping hazardous samples, a "HAZARDOUS MATERIALS SHIPPERS CERTIFICATE" and an address label must be attached to the top of the picnic cooler (these latter procedures apply to Federal Express shipments only). All of the former procedures mentioned above apply to Purolator Courier and Federal Express Courier. (The CRL presently does not use United Parcel Service.)

#### 9.2.2 SAMPLE HANDLING - CONTRACT LABORATORIES AND CONTRACT FIELD STAFF

When practicable, all Regional laboratory and field contracts managed by the GLNPO will require the Contractor(s) to follow identical sample handling procedures as described above - any justifiable exception to the above shall require the approval of the GLNPO Director. However, it should be noted - with particularity - that the Agency has several national contracts for field investigation/sample collection and laboratory analysis and each of these contracts has it's own specific handling protocol; accordingly, these specific protocols must be followed attendant to the contractual agreement. In addition, all of Region Y's handling requirements must also be met. Prior to establishing analytical or field investigation/sampling contracts, CRL staff person(s) responsible for coordinating contract analytical work should be contracted for specifics. General guidelines follow:

Both USEPA and Contractors will use the National Standard Sample Tag and

the National Standard Custody form - other specific field and tracking forms and as required by the contract.

All samples to be shipped will follow the Standard Regional / D.O.T. requirements as defined under Shipping Procedures.

All samples shipped to Region V contracted laboratories for analyses will be accompanied by the tracking form, with distribution made as shown on the form.

Samples to be collected by the FIT's Contractor or USEPA Region V staff for analysis by the VIAR Contractor will follow specific protocol. All forms and procedures must follow the required order and sequences or the associated samples will be discarded.

### 9.3 CHAIN OF CUSTODY PROCEDURES

Due to the known or potential evidential nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings- it shall be the policy in USEPA's Region V GLNPO to collect all sediment samples under the standard custody procedures.

Sample custody is initiated at the time of sample collection by fixing a numbered custody seal to each sample taken or by placing the sample in a locked container or into a container which is sealed with a custody seal. The custody form is also, immediately filled out and signed by the person collecting the sample. It is the responsibility of the sampler to ensure that the sample and sample descriptive forms are in custody (locked or properly sealed to prevent

tampering) and that all descriptive information is accurate and complete. Each individual who subsequently signs the custody form has a similar responsibility and, in addition, must ensure that all information added to the sample descriptive forms is also complete and accurate. This process is documented by the use of the Standard National Chain of Custody Record form (see Figure 7). The Chain of Custody Record forms are serially numbered (forms are accountable) and provides an original for accompanying the associated samples and a copy for the field records.

#### 9.3.1 SAMPLE CUSTODY

By definition, a sample is under custody if:

- A. It is in your possession or
- B. It is in your view, after being in your possession or
- C. It was in your possession and then you locked it up to prevent tampering or
- D. It is in a designated secure area.

#### 9.3.2 FIELD CUSTODY PROCEDURES

9.3.2.1 In collecting samples for evidence, collect only that number which provides a good representation of the media being sampled. To the extent possible, the quality and types of samples and sample locations are determined prior to the actual field work. As few people as possible should handle the samples.

9.3.2.2 The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly.

9.3.2.3 Sample tags shall be completed for each sample using water proof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used since a pen would not function in freezing weather.

9.3.2.4 The Project Leader determines **whether** proper custody procedures were followed during the field work and decides if additional samples are required.

### 9.3.3 TRANSFER OF CUSTODY AND SHIPMENT

9.3.3.1 Samples are accompanied by a Chain-of Custody Record. When transferring the possession of samples, the individuals relinquishing and 'receiving will sign, date, and note the time on the record. This record documents samples custody transfer from the sampler, often through other persons, to the analyst and subsequently, sample storage in a custody room ( If appropriate).

9.3.3.2 Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis with a separate custody record accompanying each shipment (each ice chest). Shipping containers will be pad-locked or sealed for shipment to the laboratory. The method of shipment, courier name(s), and other pertinent information, is entered in the "Remarks" section. (The general and specific procedures for shipping are described under "Shipping Procedures" of the document.)

9.3.3.3 Whenever samples are split (replicated) with a source or government agency, it is noted in the "Remarks" section of the Custody form and in the Field Logbook. The note indicates with whom the samples are being split and signed by both **the** sampler and the recipient. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" section.

9.3.3.4 All shipments will be accompanied by the Chain-of-Custody

Record identifying its contents. The original record will accompany the shipment, and a copy will be retained by the Project Coordinator.

9.3.3.5 If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a Government Bill of lading will be used. Air Freight shipments are sent collect. Freight bills, post office receipts and Bills of Lading will be retained as part of the permanent documentation (see Shipping Procedures Section).

#### 9.3.4 FIELD CUSTODY - CONTRACTORS

All Regional field contractors managed by the GLNPO will follow the above custody procedure when practical and appropriate.

All justifiable cases requiring a variance from the above shall be reviewed by GLNPO for approval. This review shall occur before the fact in normal operational procedures or as soon as possible after the fact for emergencies or other controlled situations. NOTE: The National Standard Tag and Custody Sheets are always used for all sampling protocols.

#### Chain of Custody Record

Project Number (data set number)

YYAANN

YY - last two digits of calendar year in which samples are collected

A - first alphanumeric code for sampling office - N for GLNPO

A - second alphanumeric - code for survey leader

NN - survey number for survey leader

Project Name - Rivermouth, harbor, industry, etc.

Samplers (signature) - signatures of all survey members.

Station numbers: So1, SOL2, D03, B04, S05 etc.

#### 9.4 FIELD LOGBOOK

In addition to the sample tags, field sheets, analysis request sheets, custody sheets, and/or other contractor required forms, a serially numbered bound field logbook must be maintained by the Survey Leader and/or other Field Team members (as needed) to provide a daily record of significant events. In order to accommodate surveys of different magnitudes filed logbooks will be ordered in different sizes.

#### 9.5 LABORATORY CHAIN-OF-CUSTODY PROCEDURE

Due to the evidentiary nature of enforcement type samples collected and analyzed in the laboratory during enforcement investigations (active and potential), possession must be traceable from the time received by the laboratory until they are introduced as evidence in legal proceedings or ultimately disposed. These samples include all samples collected by GLNPO staff and all samples received and/or analyzed by the CRL. To maintain and document sample possession in the laboratory, the following Chain-of-Custody Procedure shall be followed:

A sample is under custody if:

1. It is in your possession; or
2. It is in your view, after being in your possession; or
3. It is in a secure area.

The actual procedure shall be as follows:

There shall be designated, a Sample Custodian and Alternate Sample Custodian(s). The Custodian accepts custody of the shipped or brought-in samples and verifies that the seal is intact and has not been tampered with, opens the case, reviews and verifies that the information on the tags are appropriate and that they match the information on the Field Chain-of-Custody Record. Other

pertinent information, as to shipment pickup, courier, etc., is entered in the "Remarks" section of the Chain-of-Custody Record. The Chain-of-Custody Record form will remain in the official transfer registry within the laboratory. All appropriate transfers will be entered on that form. The samples will then be placed in a secured area, preferably, a dedicated area whose only access is to the Custodian or Alternate Custodian (Custody Sample Storage Room).

The Custodian will assign samples from and to his/her custody to the appropriate analysts. The names and signature of individuals who receive samples are recorded on the Chain-of-Custody Record. Laboratory personnel are responsible for the care and custody of samples from the time they are received until they are returned to the Custodian. The sample is considered in the Custody of the Analyst when it is in their possession, site, or in a secure area. The sample will be returned to the Sample Custodian when the Analyst completes the procedure.

When sample analyses and necessary quality assurance checks have been completed for identified enforcement cases, all identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation file. Residual (unused) sample quantities will be retained in secure storage after the completion of analytical determinations until formal notice is given from GLNPO that the samples can be disposed of.

The final date(s) of disposal will be recorded on the Chain-of-Custody Record by memorandum. For all samples not identified as enforcement cases, the file management will be the same as above except that all attendant samples shall be disposed of after three (3) months of storage. Disposal date(s) shall be documented to the documentation file by memorandum from the Sample Custodian.

It should be noted - with particularity - that all of the National Standard Tags associated with samples must be filed respectively in the appropriate survey documentation files, at the time the sample is disposed of or at the time of laboratory analysis, if all the sample volume is used during the analytical phases. This requirement means that tags associated with active samples are never discarded by the bench analysts or field staff -- tags can only be discarded by procedures governing the documentation Control File.

## 10.0 DOCUMENTATION CONTROL PROGRAM

### 10.1 INTRODUCTION

The goal of the Region V Document Control Program is to assure that all project documents issued to and/or generated by Region V staff will be accountable (in legal connotation) when the project is completed. The system involves locating all cases affecting information in officially located documented files. These files shall be capable of consolidation into one central file or to stand alone containing a unique single document control number system, per case/survey, a document inventory procedure, and an evidentiary filing system all operated and managed by respective document control managers.

### 10.2 REGION V DOCUMENTATION CONTROL PROGRAM DESCRIPTION

Region V shall operate it's Documentation Control Program at *two* levels, as described below.

The District Offices will maintain a Document Control Program file with an appointed document control file manager--will receive all required field office investigation and laboratory data. Active enforcement Document Control files will be forwarded to the appropriate enforcement office for maintaining Tracking of this numbering system will be done by use of a computer program.

Program files for a period of at least five years ...this file will be designated as the D.O. Branch Documentation Control File.

The Central Regional Laboratory (CRL) will maintain a Document Control Program file with an appointed document control file manager--will receive all required laboratory records and analytical data records. Active enforcement Document Control files will be forwarded directly to the District Office(s)... this file will be designated as the CRL Branch Documentation Control File.

The above two level systems will allow the Region V to handle all surveys and analytical analyses in the custody mode with little additional resources usage. This procedure will allow survey data to be used in Court, if needed, without resampling.

### 10.3 DOCUMENT CONTROL NUMBER

The CRL has been assigned a sequential data set number to each survey of samples received at the laboratory--this number is preceded by an identification code which identifies the office collecting the sample. As an example, EDO for Eastern District Office, CDO for Central District Office, GLNPO for Great Lakes National Program Office etc. This data set numbering system will be used to generate the Document Control Program numbers for each field investigation study. This system of assigning Document Control numbers will apply to both sampling and non-sampling surveys alike- in essence, every field investigation situation and/or any CRL reception of samples from other Divisions or other sources must result in the assignment of a document control number. The CRL Document Control Program manager will assign the document numbers to the data users' Document Control Program managers in blocks of series. Tracking of this numbering system will be done by use of a computer program.

#### 10.4 SERIALIZED DOCUMENTS

The CRL has responsibility for the procurement of all forms and logbooks. Typically, several months supply of these numerically serialized materials will be assigned to each office conducting field surveys or collecting samples. The series of numbers on the documentation materials will be recorded by the CRL Documentation Control Program manager. The field investigation offices' Documentation Control Program managers will assign the subject serialized materials to each project coordinator, who will in turn assign the subject serialized material to the appropriate field investigation team or to the participating samplers-- the project coordinator has complete responsibility for insuring and documenting documenting in his or her logbook, the sufficient and proper distribution of of documentation control documents. Policy Note: All serialized documentation control documents will have a document control number affixed to it, prior to use on any survey.

#### 10.5 PROJECT LOGBOOKS

Generally, logbook entries should be dated, legible and contain accurate and inclusive documentation of an individuals's project activities. Because the logbook forms the basis for the subsequent written reports, it must contain only facts and observations. Language should be objective, factual and free of personal feelings or other terminology which might prove inappropriate. Entries made by individuals other than the person to whom the logbook was assigned are dated and signed by the individual making the entry.

All project logbooks are the property of Region V and are to be returned to the Project Leader when a survey assignment has been concluded.  
logbook.

The organization of the logbook would be such that a format is available to prevent overlooking required entries while at the same time it should allow entry of unusual events. This can be accomplished either by using the first two or three pages of the field logbook to describe the required entries or each page can be formatted or a combination of the two.

#### 10.6 FIELD DATA RECORDS - REGION V FIELD RECORD FORMS

Where appropriate, serialized Field Data Records (FDR) (in the form of Region V Field Record Forms or bound logbooks with affixed document control numbers) are maintained for each survey sampling station or location and the project code and station number are usually recorded on each page. The project leader also numbers the FDR covers with the appropriate project code and station number. All in-situ measurements and field observations are recorded in the FDR's with all pertinent information necessary to explain and reconstruct sampling operations. Each page of a Field Data Record is dated and signed by all individuals making entries on that page. The Project Leader and the field team on duty are responsible for ensuring that FDRs are present during all monitoring activities and are stored safely to avoid possible tampering. Any lost, damaged or voided FDRs are reported to the Project Leader.

#### 10.7 SAMPLE IDENTIFICATION DOCUMENTS

All necessary serialized sample tags (with affixed document control numbers) are distributed to field personnel by the Project Leader (or designated project participant) and the serial numbers are recorded in the Project Leader's logbook and each involved inspector's logbook. Individuals are accountable for each tag assigned to them. A tag is considered in their possession until it has been

filled out, attached to a sample, and transferred to another individual with the corresponding Chain-of-Custody Record. At no time are any sample tags to be discarded and if any tags are lost, voided, or damaged, this is noted in the appropriate or logbook immediately upon discovery and the Project Leader is notified. At the completion of the field investigation activities, all unused sample tags are returned to the designated individual who checks them against the list of assigned tag serial numbers. Tags attached to those samples split with the source or another government agency are accounted for.

#### 10.8 CHAIN-OF-CUSTODY RECORDS

Serialized Chain-of-Custody Records (with affixed document control numbers) are assigned and accounted for in a manner similar to that used for sample tags. Double copy forms of the Custody Sheets are filled out in the field, according to the Region V Custody Procedures Manual. All field staff having charge (legal custody) of a sample(s) must sign the respective Custody Sheet(s). The Project Leader or a designated field custodian transmits the samples and properly signed Custody Sheets to the respective office custodian locally or may mail samples from the field directly to another U.S. EPA or contractor laboratory(s) -- the various options involving samples exchanges and Custody Sheet management are listed below:

1. Samples transported from the field by field staff and turned in to the laboratory or office custodian -- Double copied Custody Sheets are signed by all field staff legally responsible for custody and turned over to the laboratory or office custodian.

2. Samples mailed from the field directly to the CRL -- Samplers and/or inspectors sign the double copied Custody Sheet(s) and forward original

(top sheet) with the samples (Note: A copy of the Custody Sheet must be packaged with each package of samples shipped. The second Custody Sheet copy is turned over to the shipping office's sample custodian, either by the Project Leader or designated staff-- a record of this transaction is entered into the Project Leader's field logbook.

3. Samples mailed from the field directly to a contractor's laboratory-- Follow the protocol prescribed for the specific contract, such as VIAR and the additional Regional protocol(s) as specified by the Project Officer.

4. For all samples to be tracked by the local field laboratory(s) -- No CRL responsibility -- the original of the Custody Sheet will be mailed to the contract laboratory stamped, "Sign and return to Sample Custodian". Generally, the contract will state that contract laboratory shall make and retain a copy of the subject Custody Sheet, prior to returning it. (Note: It is expected to be rare cases where samples will be sent to contract by S&AD Offices and the CRL will not track the analyses.)

#### 10.9 PHOTOGRAPHS

When movies, slides or photographs are taken which visually show the effluent or emission source and/or any monitoring locations, they are numbered to correspond to logbook entries. The name of the photographer, date, time, site location, and site description are entered sequentially in the logbook as photos are taken. Chain-of-Custody procedures depend upon the type of film and the processing it requires. Once developed, the slides or Photographic prints shall be serially numbered corresponding to the logbook description, labeled and forwarded with the logbook in the logbook photo page format.

## 10.10 CORRECTIONS TO DOCUMENTATION

As previously noted, unless prohibited by weather conditions, all original data recorded in logbooks, sample tags, and other data sheet entries are written with waterproof ink. None of the accountable serialized documents listed above are to be destroyed or thrown away, even if they are illegible or contain inaccuracies which required a replacement document.

if an error is made on an accountable document assigned to one individual, the individual may make contemporaneous corrections simply by crossing a line through the error and entering the the correct information. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

It a sample tag is lost in shipment, or a tag was never prepared for a sample(s), or a properly tagged sample was not transferred with a formal Chain-of-Custody tag, the following procedure applies. A written statement is prepared detailing how the sample was collected, air-dispatched or hand-transferred to the field or ERL laboratory. The statement should include all pertinent information, such as entries in field logbooks regarding the sample, whether the sample was in the sample collector's physical possession or in a locked compartment until hand-transferred to the laboratory, etc. Copies of the statement are distributed to the Project Leader and the appropriate office Custody Files.

## II. QUALITY ASSURANCE

### 11.1 Introduction

U.S. Environmental Protection Agency (USEPA) Policy, enunciated in memoranda of May 30 and June 14, 1979, requires participation in a centrally managed Quality Assurance (QA) program by all USEPA Regional Offices, Program Offices, USEPA Laboratories and the States.

This includes those monitoring and measurement efforts mandated or supported by USEPA through regulations, grants, contracts, or other formalized agreements. The QA programs for the States in Region V will be cooperatively developed with them; and implemented through the Regional Office.

The Office of Research and Development (ORD) has been given responsibility for developing, coordinating, and directing the implementation of the Agency QA program. In addition, an Agency QA Advisory Committee, chaired by ORD and with representatives from the Program Offices, Regional Offices, Staff Offices, and the States, has been established to coordinate this effort.

As an initial step, in implementing this policy, QA Plans (Programs) must be prepared by all USEPA-supported or - required environmental monitoring and measurement activities per the specifications of USEPA's guidance document QAMS-004/00.

Such a program has been documented by the Quality Assurance Office (QAO) to Report No. EPA - 905/4-80-001, title "QA program, Guidelines and Specifications, Criteria, and Procedures, Region V". The purpose of this report (manual) is to describe the QA program for Region V, U.S. EPA that will produce a numerical estimate of the reliability of all data values reported or used by the Region.

## 11.2 POLICY AND OBJECTIVES

QA is necessary at each organizational level to ensure high quality data. Each organization should have a written QA policy.

The primary goal of the Region V, QA program is to define and improve the reliability (accuracy and precision) of data generated and used by the Region, per Headquarters' mandate and Agency regulations. There must be a mechanism for so doing. In order to measure or estimate changes in data quality, the quality must be expressed in measurable (numerical) terms. Therefore, the first

priority in the Region V QA program is to establish and implement a method to define and quantitate the program product - data quality.

Other QA program objectives are listed in Section 3 of Report No. EPA - 905/4-80-001.

The Agency's QA policy statement specifies that the Regional Administrator is responsible for the implementation and coordination of the mandatory QA activities within Region V.

Specific details of this responsibility are delineated in Section 4 of the Report No. EPA - 905/4-80-001.

Elements to be addressed for the preparation of QA programs are contained in Section 8 of Report No. EPA - 905/4-80-001.

### 11.3 Quality Assurance Procedure

Basically Quality Control consists of:

1. A mechanism for promoting consistent operation, and
2. Verification of adequacy of this operation.

In order to achieve consistent operation, methods and procedures must be documented to an extent that no decisions are available or necessary. The purpose of this manual is to provide a mechanism for achieving consistent operation.

Verification of the adequacy of this operation will be monitored by the use of blanks and replicate samples. Since the utility of replicates with respect to analyses for parameters that are normally present at undetectable levels is questionable, duplicates should be selected from the areas that are most likely to be polluted. Sediment blanks will be empty jars covered at the

sampling site with lined caps and carried along with the other samples. Replicate samples are, as described elsewhere, either a split of a collected sample ("A" designation), or a second collected sample at the same site at nearly the same time ("D" designation). Since most of the information available from split samples can be achieved by splitting the sample in the laboratory, standard operating procedures is for all field duplicates to be duplicate samples ("D" designation).

Approximately, for every fifteen analyses, one blank and one field duplicate will be included. In view of the cost of duplicate sampling compared to cost of analyses, it may be advisable to collect duplicates at every site so that analysis of duplicate samples can be chosen on the basis of analytical results.

At least one blank and one field duplicate will be collected for every fifteen samples.

Figure 1  
Equipment and Supply Checklist

BOAT	
DINGHY	
MOTOR	
FUEL/OIL	
BATTERIES	
IGNITION KEYS	
OAR	
BOAT HOOK	
ANCHOR/LINE	
TOOLBOX	
WORKVESTS	
FOUL WEATHER GEAR	
WADERS	
SAFETY SHOES	
GLOVES	
TAPE/RULER	
WATER PROOF TAPE	
FLASHLIGHT	
COMPASS	
RANGEFINDER	
LORAN C	
RADIO/ANTERRA	
BOTTOM SOUNDER	
WINCH/DAVIT/CABLE	
SAFETY LINE	
CORER	
LINERS	
END CAPS	
NOSE CONES	
CORE CATCHER	
COLLAR	
HOLDER	
SAW	
PONAR	
PAN	
SPATULA	
SCREEN	
FOIL	
SAMPLE JARS	
S.J. LINERS	
COOLERS	
PAPER TOWELS	
WATER SAMPLER/LINE	
FIRST AID KIT	
THERMOMETER	
MACROINVERTEBRATE PRESERVATIVE	
SECCHI DISK	
BOAT BUMPERS	
FLARE GUN & FLARES	

SEDIMENT MANUAL	
SURVEY PLAN	
LOGBOOK	
CHARTS	
LAKE SURVEY	
STATION LOCATION	
LORAN C	
STATION LIST	
FIELD SHEETS	
ADHESIVE TAGS	
CHAIN OF CUSTODY	
RECORDS	
TAGS	
SEALS	
WATER PROOF PENS	
ANALYSIS REQUEST FORM	
SPARE PROPELLER FOR MOTOR	
SPARE TIRE FOR BOAT FRAILER	
FIRE EXTINGUISHER	
JUMPER CABLES	
BINOCULARS	





## Figure 4

### Logbook Format

Page Heading (Harbor or periodic entries)

Harbor/River// Date // Survey members

Weather conditions ( fair, rain, drizzle, overcast)

Wind (0.5, 5/10, 10/20, >20)

Water (calm, 0/6 in., 6 in to 1 ft., 1 ft to 2 ft, 2 ft. to 4 ft.,  
4 ft. to 8 ft.)

Station entries

STATION NUMBER/time

Location description (landmarks, compass reading, Loran C reading, etc.)

Water appearance (very turbid, turbid, moderately turbid, clear)

Average stream depth, breadth and velocity if possible to estimate

Type of sampler used, number of tries

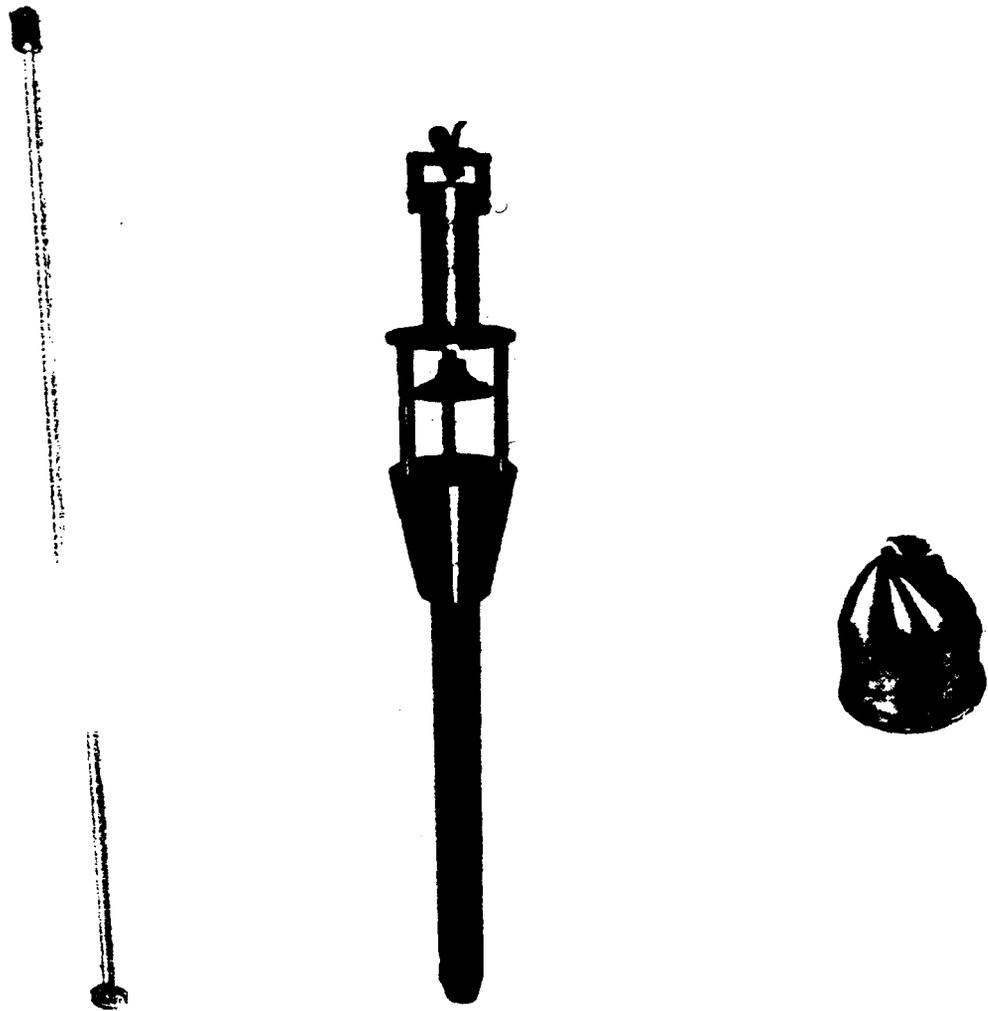
Number of containers & Chain of Custody tag numbers

Chain of Custody record number

Benthics (if screened so indicate)



Figure 8



Extruding rod

Core Sampler

Core Sampler

TABLE 1. CATEGORIES FOR FIELD EVALUATION OF SEDIMENT CHARACTERISTICS\*

TYPE	<u>SIZE OR CHARACTERISTIC</u>
<b>Inorganic Components</b>	
<b>Bed rock or solid rock</b>	
Boulders	>256 mm (10 in.) in diameter
Rubble	64 to 256 mm (2-1/2 to 10 in.) in diameter
Gravel	2 to 64 mm (1/12 to 2-1/2 in.) in diameter
Sand	0.06 to 2.0 mm in diameter; gritty texture when rubbed between fingers
Silt	0.004 to 0.06 mm in diameter
Clay	(0.004 mm in diameter; smooth, slick feeling when rubbed between fingers
Marl	Calcium carbonate; usually gray; often contains fragments of mollusc shells or CHARA; effervesces freely with hydrochloric acid
<b>Organic: Components</b>	
Detritus	Accumulated wood, sticks, and other undecayed coarse plant materials
Fibrous Peat	Partially decomposed plant remains; parts of plants readily distinguishable
Pulpy Peat	Very finely divided plant remains; parts of plants not distinguishable; varies in color from green to brown; varies greatly in consistence-often being semi-fluid
Muck	Black, finely divided organic matter; completely decomposed

\*Biological Field and Laboratory Methods for Measuring the Quality of Surface Waters and Effluents, EPA-670/4-73-001, July 1973, USEPA, NERC, Cincinnati, Ohio.

TABLE 2. QUALITATIVE DESCRIPTIONS OF ORDORS\*

<u>CODE</u>	<u>NATURE OF ODOR</u>	<u>DESCRIPTION</u> (Such as Odors of:)
A	Aromatic (spicy)	Camphor, cloves, lavender, lemon
Ac	cucumber	SYNURA
B	Balsamic (flowery)	Geranium, violet, vanilla
Bg	geranium	ASTERIONELLA
Bh	nasturtium	APHANIZOMENON
Bs	sweetish	COELOSPHAERIUM
Bv	violet	MALLOMONAS
C	Chemical	Industrial wastes or treatment
CC	chlorinous	chemicals
Ch	hydrocarbon	free chlorine
Cm	medicinal	oil refinery wastes
Cs	sulfuretted	phenol and iodoform
D	Disagreeable	hydrogen sulfide
Df	+ shy	(Pronounced, unpleasant)
Dp	pi gpen	UROGLENOPSIS, DINOBYRON
Ds	septic	ANABAENA
E	Earthy	stale sewage
EP	peaty	Damp earth
G	Grassy	peat
M	Musty	Crushed grass
Mh	Moldy	Decomposing straw
V	Vegetable	damp cellar
		Root vegetables

\*Standard Methods for the Examination of Water and Wastewater, 12th Edition, 1965, APHA, AWWA, WPCF, New York.

**TECHNICAL REPORT DATA**

*(Please read Instructions on the reverse before completing)*

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7. AUTHOR(S) Marvin Palmer		6. PERFORMING ORGANIZATION CODE 5GL	
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Environmental Protection Agency Great Lakes National Program Office Chicago, Illinois 60605		8. PERFORMING ORGANIZATION REPORT NO.	
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15. SUPPLEMENTARY NOTES			
16. ABSTRACT The Methods Manual is detailed procedures for survey planning, sample collection, document preparation and quality assurance for sediment sampling surveys conducted by the Great Lakes National Program Office. The data base will be utilized to institute a remedial action program in accordance with USEPA policies and procedures guidelines.			
17. KEY WORDS AND DOCUMENT ANALYSIS			
a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Toxic substances Sediment bioassay Core samples Volatile organic samples Contamination Harbor sediment			
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