

DREDGE
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ISBN 0-7778-3293-3

EVALUATING CONSTRUCTION ACTIVITIES

IMPACTING ON WATER RESOURCES

PART III C

HANDBOOK FOR DREDGING AND DREDGED MATERIAL

DISPOSAL IN ONTARIO - SEDIMENT

SAMPLING AND LABORATORY ANALYSIS

Report prepared by:

Standards Development Branch
Ontario Ministry of the Environment and Energy

FEBRUARY 1991
Revised February 1994

Cette publication technique
n'est disponible qu'en anglais

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Log 87-2231-001
PIBS 1711E03

FOREWORD

In 1976, the Ontario Ministry of Environment (OMOE) published the guideline "Evaluating Construction Activities Impacting on Water Resources" as an aid in the assessment of the environmental impact of construction activities. Information gained since 1976 now warrants a revision of the original document.

The revised guidelines have been divided into five parts, as follows:

- Part I: Guidelines for construction of hydrocarbon transmission and distribution pipelines crossing water courses (March 1984)
- Part II: Guidelines for construction of highways and bridges (March 1984)
- Part III: Handbook for dredging and dredged material disposal in Ontario A,B,C
 - A - Legislation, Policies, Sediment Classification & Disposal
 - B - Dredging, Transport and Monitoring
 - C - Sediment Sampling and Laboratory Analysis(November 1990, Revised January 1994)
- Part IV: Guidelines for marine construction projects (April 1986)
- Part V: Guidelines for small-scale waterfront projects (April 1986)

This handbook (Dredging and Dredged Material Disposal - Part III) has been prepared to assist dredging project proponents, OMOE staff and staff of other regulatory agencies in the selection of safe and appropriate management methods based on dredged material characteristics and current OMOE legislation. This document is intended to be a reference handbook of dredging-disposal activities, the details of which may not be required on a routine basis. The current revision incorporates the new sediment evaluation procedures from the Provincial Sediment Quality Guidelines (Persaud *et al.* 1992).

Mention of trade names and commercial products in this handbook does not constitute endorsement.

ACKNOWLEDGEMENTS

The preliminary report was prepared under contract by Beak Consultants and Ocean Chem Group.

This report underwent review and consequently many modifications were made based on valuable comments from the following people: Dredged Material Management Working Group, Steve Maude, Gerry Myslik, Archie McLarty, Duncan Boyd, Deo Persaud, Wolfgang Scheider, John Ralston, Tammy Lomas, Don King, Ian Carter and Elizabeth Pastorek from the Ontario Ministry of the Environment, and Ian Orchard, Laurie Sarazin, John Marsden, Susan Humphrey, Simon Llewellyn, Alfred Chau, Peter Fowlie and Bill Lee from Environment Canada.

Tammy Lomas and Stephen Petro of the Water Resources Branch, Ontario Ministry of the Environment coordinated editorial revisions to the report.

Special thanks to Rose-Marie Gonsalves of the Water Resources Branch, Ontario Ministry of the Environment for her endurance in typing the many editorial revisions.

Volume III of the Ontario Ministry of Environment's Report-Evaluating Construction Activities Impacting on Water Resources was supported in part by funds received from Environment Canada under terms of the Canada-Ontario Agreement (COA) on Great Lakes Water Quality. Activities are coordinated with those of the Federal Government under the guidance of the COA Polluted Sediments Committee.

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INTRODUCTION

Environmentally sound marine construction practice requires that every effort be made to preserve the physical and biological integrity of Ontario's waterbodies in accordance with the provincial goals - "To ensure that the surface waters of the Province are of a quality which is satisfactory for aquatic life and recreation" (Ontario Ministry of the Environment, 1978).

The aim of this Ministry of Environment handbook is to provide an overview of the management options for the handling of dredged material in the Province of Ontario. These guidelines were developed to protect the receiving environment according to the physical, chemical and biological quality of the material being dredged. Recognition is given, where appropriate, to the potential re-use of certain materials.

Dredging, for the purposes of this handbook, is identified as the planned, mechanical movement of material located below the surface of a waterbody, or at the land/water interface. These guidelines apply to all forms of dredging.

The following sections review sediment sampling techniques, analytical methodologies used for analysis of sediment quality and quality assurance/control considerations necessary to achieve acceptable data results.

1.0 SEDIMENT SAMPLING, HANDLING AND ANALYSIS

1.1 Sampling

1.1.1 Survey Design

Before undertaking a collection of samples, the proponent should prepare a properly designed program and consult with the regional offices of Ontario Ministry of the Environment (OMOE). The survey design consists of three phases:

1. project and data review,
2. design strategy, and
3. consultation with OMOE.

1.1.1.1 Project and Data Review

The objective of project and data review is to draw together all the necessary requirements and available information in order to design a sampling survey which will generate the required information with minimal effort and time. The main emphasis of a sampling survey is to define the nature of the material to be dredged.

The proponent should first define the project by obtaining an up-to-date bathymetry map of the area and, in combination with the areal extent of the project, determine the volume of material to be dredged.

The second task is to review the regulatory requirements of the legislation as outlined in Part III A of this document, i.e., determine what information will be required in the regulatory review process. Monitoring requirements for the dredging or disposal operations should be identified in order that background data may be collected.

A careful review of historical data should be made before a sampling program is designed. The data review should consider the following:

1. Does the information meet regulatory requirements?
 - Are there results for all parameters of concern for that specific area?

- Are analytical methods and detection limits appropriate and adequate?
 - Have the data been generated with adequate quality assurance and quality control practices in place?
2. Does the information adequately define the nature of the material to be dredged and disposed of?
- Were an adequate number of samples taken?
 - Do the samples represent surficial sediment or provide a complete depth profile of the material to be dredged?
 - Were the samples collected and handled appropriately?
3. Are there any long-term temporal trends in the data which indicate a change in the degree of contamination in the project area?

There are a variety of data sources to be considered:

- **Site-Specific or Harbour-Specific Data:** This includes data most relevant to the project review. Data collection will likely have been undertaken by OMOE, Environment Canada or a proponent such as Canada Department of Public Works (PWC). The data may be available only in the files of the regulatory agencies, of proponents (PWC) or of funding agencies (e.g., Harbour Commissions, Transport Canada). Some data may have been published in the form of data or technical reports published by government departments, e.g., Thomas and Mudroch (1979), Persaud *et al.* (1985).
- **Area or Regional data:** This includes data collected on a regional basis and may include the harbour under consideration, but is not specific to it. Such data may be used to predict ranges of concentrations to be expected as a minimum in the harbour, but it must be remembered that harbours are typically contaminant "sinks" and will generally be found to have higher concentrations. An example would be Thomas and Mudroch (1979), where the chapters describing each lake clearly show the differences between the lakes. Typically, area or regional data has been collected by such groups as OMOE, Environment Canada or Fisheries and Oceans Canada as part of a general description of the environment of the Great Lakes.

Thus, not all relevant parameters may have been measured.

- **Point-Sources and Outfall data:** This would include monitoring data for various effluents into a harbour. Data may be available from OMOE or Environment Canada, or possibly a municipality. Data should be reviewed in the context of identifying a potential problem, e.g., elevated concentrations of chromium in the area of a metal plating industry.
- **Other Data Sources:** This includes data from a variety of sources where the data can be used to predict or indicate the nature of the material to be dredged. For example, geotechnical borehole logs may indicate layers of black organic-rich silt on a sand layer; the silt layer may be contaminated. Such information may be useful in the prediction of problems or hot-spots. The data will generally be in the files of agencies responsible for harbour construction projects.

Having collected all available data, the proponent should collate the available historical data, making predictions of concentrations to be expected in the sediments proposed to be dredged. The collation of the data will serve to identify the gaps in available information. The filling of these gaps will assist in defining the sampling program to be undertaken.

One important aspect which is often overlooked by a proponent is an inspection of the site to ensure that the historical information is still accurate. This site inspection should include identification of new sources of contaminant input (industries, outfalls); discussion with municipal planning agencies; and discussion with district fish and wildlife agencies. This site inspection will enable the proponent to obtain a better understanding of public perceptions and concerns and identify any difficulties which could hinder the sampling operation.

At this stage, the proponent has sufficient information to plan a sampling program. However, it is highly recommended that the proponent first consult with the appropriate OMOE regional office. This will ensure that all information has been obtained and that any special requirements are identified in advance of sampling.

1.1.1.2 Sampling Design Strategy

The objectives of the sampling program are as follows:

- to ensure that the information package meets the most recent regulatory requirements;
- to fill the gaps identified by the information collection/collation exercise; and
- to provide the basis for review of disposal alternatives.

A sampling pattern should be designed to most efficiently and with minimum effort collect the information desired that will adequately describe the nature of the material to be dredged. To adequately define the nature of the sediment, the concentrations of the parameters in the sediment must be evaluated in accordance with the OMOE Provincial Sediment Quality Guidelines (see Part IIIA; Appendix A). The sampling design, specifically number of samples, should describe the sediment in terms of mean measured chemical concentration with an associated 95% confidence limit. That is, the proponent should be able to state that the data will fall within the 95% confidence interval (e.g., ± 0.2 mg/g). For example, the 0-15 cm interval of sediment contains 0.8 ± 0.2 mg/g cadmium having collected and analyzed adequate samples to be able to achieve 95% confidence in the data.

A sampling pattern can be devised knowing only the depth of material to be removed. However, knowing the detailed bathymetry of the site, or the particle size distribution, or the distribution of contaminants or the sources of contaminants to the site area, assists in refining the sampling design.

(Note, preference should always be given to collecting more samples and replicates from the deeper parts of material to be dredged).

1.1.1.3 Sample Collection

Sampling Devices

There are a variety of sediment sampling devices, which can be commonly grouped into grab samplers and core samplers. Grab samplers are jaw-like devices designed primarily to collect surficial sediments. Their ability to collect a sample is a function of degree of penetration, angle of penetration, depth of water and lateral motion of the boat during collection. Unless sealed, there is also a tendency for "washout" of fine-grained materials during retrieval. Core samplers are usually tube-shaped devices which can penetrate the sediment by gravity (free-fall), vibration or hydraulic pressure (water

or oil). Table 1.2 taken from Sly (1969) describes the various types of samplers, both corers and grabs, and their advantages and limitations. Figures 1.1 and 1.2 illustrate the various types of grab samplers and core samplers respectively.

The distribution of contaminants in sediments varies both horizontally and vertically. Horizontal variation can be assessed by the collection of samples from select sites throughout the project area. Typically, the concentration of contaminants varies with depth in sediments. If information on horizontal variability is required, it is recommended that sediment samples be collected using either a coring device from a boat or having a diver collect a core. A grab sample is recommended if information on surficial sediment is required.

The typical coring device is a length of pipe with a weighted head of 50 to 200 kg. Inside is the plastic liner (polybutylacrylic plastic is recommended). At one end is a metal core cutter which assists the coring device to penetrate the sediment and a core catcher to retain the sediment in the liner. At the top end is a ball-valve or piston which retains the sediment in the liner when the device is pulled back out of the sediment.

There are three major drawbacks to the gravity core sampler:

1. There is a "shock wave" ahead of the sampler before it penetrates the sediment. This may displace the very unconsolidated top layer of sediment;
2. The gravity action tends to compress the sediment during penetration, thereby compressing the vertical profile of the contaminants (Baxter *et al.* 1981); and
3. The use of a small sampling boat necessitates the use of a small core sampler with a small head weight. The small barrel diameter of the sampler can cause gross disturbance of the sediment profile during penetration, potentially destroying the vertical profile of the contaminants. The small weight may lead to insufficient penetration.

Table 1.1
OPERATIONAL EVALUATION OF SAMPLING DEVICES
(from Sly, 1969)

<u>GRAB SAMPLERS</u>	<u>Characteristics</u>
Franklin-Anderson Grab	Suitable for obtaining material for bulk sample analysis. Works best in soft clays, muds, silts and sands. Will occasionally obtain a good gravel sample. Material of no use for structural or other specific analyses.
Dietz-LaFond Grab	Can be used for general sampling but not recommended for any particular use. Of all the samplers tested, this pattern proved to be the least suitable.
Birge-Ekman Dredge	Suitable for soft clays, muds, silts and silty sands. This sampler should be used under calm water conditions, typically in small lakes or restricted areas. The lack of sample disturbance, square cross section and moderate penetration make this sampler suitable for detailed studies (i.e., biological and geochemical) of the top 2 to 3 cm of bottom sediment. Because of its light weight and easy handling, it is well suited to small boat operations.
Petersen Grab	This sampler, like the Franklin-Anderson, is suitable for taking bulk sample material in most types of sediment. It is quite unsuited for studies of detailed and specific sediment properties, though it is perhaps a little more successful in taking gravel samples. Either of these two samplers (Petersen or Franklin- Anderson) will do well as a general purpose bulk sampler.
Ponar Grab	An excellent general purpose bottom sampler. In practice it operates better than either the Petersen or Franklin-Anderson over the full range of bottom types. It can also obtain samples with little or no disturbance and with the protecting screens removed or folded back, direct access can be had to the sediment surface of the sample. Such access to undisturbed samples makes it suitable for geochemical, sedimentological, biological and structural studies. Because of the large sample volume and its relatively undisturbed state, this sampler is very suitable for population studies of the bottom sediment fauna.
Shipek Grab	An excellent general purpose sampler, though perhaps a little heavy for small boat operation. This sampler is capable of working with almost equal success on all types of bottom materials. It provides a sample even less disturbed than the Ponar, making it the most suitable sampler (under test) for detailed geological studies of the sediment surface. The sampler volume is significantly less than that of the Ponar, and the quantity of material sampled at maximum cutting depth is also less than the Ponar. These two points may, therefore, favour the Ponar for certain biological (population)

studies. On the other hand, the rapid rotation of the Shipek bucket, as opposed to the much slower closure of the Ponar's jaws, may make it more suitable for sampling sediment containing a significant population of non-sessile forms.

Grab Trigger System Reliability

Franklin Anderson Grab	Good, but perhaps too sensitive on hard sand and gravel bottom.
Dietz-LaFond Grab	Poor, unless area of trigger foot is increased to at least 50 cm ² . Triggering may often be impossible in very soft mud unless the foot has been modified.
Birge-Ekman Dredge	Good. Triggered by messenger weight dropped from surface, normally consistent but can be affected on soft bottoms if sampler is allowed to settle for too long before dropping the messenger.
Petersen Grab	Fair to good, though tends to be a little over-sensitive on hard sand and gravel bottoms.
Ponar Grab	Good, though like the Petersen, tends to be a little over-sensitive on gravel bottoms.
Shipek Grab	Good, though some slight settlement may occur before triggering on very soft materials. Sampler may fail to trigger when lowered gently on soft bottoms. By lifting and dropping the trigger weight a few centimetres after bottom contact, abortive casts may be avoided. The slight movement of the inertial trigger weight has no other affect on the sampler.

Grab Jaw Shape, Design and Cut

Franklin-Anderson Grab	Poor. During the first stages of closure and when under the greatest pressure of springs and weight, the jaw shape loosely follows the arc of cut. However, the degree of fit becomes progressively worse as the closing pressure is reduced. Because each jaw is semi-cylindrical in shape, sample displacement is necessary within it if anything near maximum capacity is to be achieved.
Dietz-LaFond Grab	Poor. As for Franklin-Anderson.
Birge-Ekman Dredge	Excellent. Jaw shape exactly follows arc of cut and almost no sample displacement occurs.
Petersen Grab	Poor. Comments as for Franklin-Anderson, except that instead of the reduction in closure pressure being produced by slackening of tensional springs, the same result is effected by reduced leverage on the scissor arms mounted across the hinge line.
Ponar Grab	Excellent. Jaw shape exactly follows arc of cut and almost no sample displacement occurs.

Shipek Grab	Excellent. As for Ponar. In addition, the rotation of the bucket is extremely rapid. In most cases, the rotational shear is far greater than the sediment shear strength, thus the cutting action is very clean (producing minimal disturbance), particularly in soft clays, muds, silts and sands.
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Preservation and Protection from Washout in Grabs

Franklin-Anderson Grab	Fair, but the tightness of closure is largely dependent upon the lack of grains trapped between the edges of the jaws. Providing tight fit between the two jaws is obtained, the sample is well shielded against washout. If the jaws are kept open by material trapped between the jaws, washout can be severe or total.
Dietz-LaFond Grab	Fair. Comments as for Franklin-Anderson.
Birge-Ekman Dredge	Good, except when the sampler is used in very coarse or shelly sediment. Under these conditions, material may be trapped between the jaws, preventing their closure. In this case, washout may be severe. The jaws are so designed that they slightly overlap one another, thus a slight imperfection of closure can be tolerated.
Petersen Grab	Good. Comments as for Birge-Ekman.
Ponar Grab	Good. Comments as for Birge-Ekman. In addition to the overlap jaws, this sampler has a pair of metal side plates, mounted close to the moving side faces of the jaws. These plates further reduce the possibility of washout.
Shipek Grab	Excellent. The great advantage of the Shipek, over all of the other samples described, is that the bucket closes with its separation plane aligned in the horizontal rather than in the vertical. Good samples can be retrieved even when bucket closure is prevented by pebbles or similar material, even 2 to 5 cm across. With the bucket properly rotated, washout is completely avoided.

Stability

Franklin-Anderson Grab	Fair. Despite the weight of this grab, it tends to "stream" at an inclined angle under conditions of rapid ship drift or fast water flow. Provided lowering conditions are calm and stable, the sampler will hold upright during the initial sampling process; if, however, the line is allowed to slack, the sampler will fall over.
Dietz-LaFond Grab	Poor. This sampler is very sensitive to "streaming" and will rarely operate in the vertical position unless used in ideal conditions. Its tendency to maintain an inclined attitude during descent sometimes results in a failure to trigger.
Birge-Ekman Dredge	Fair. Despite the light weight of this sampler and its tendency to

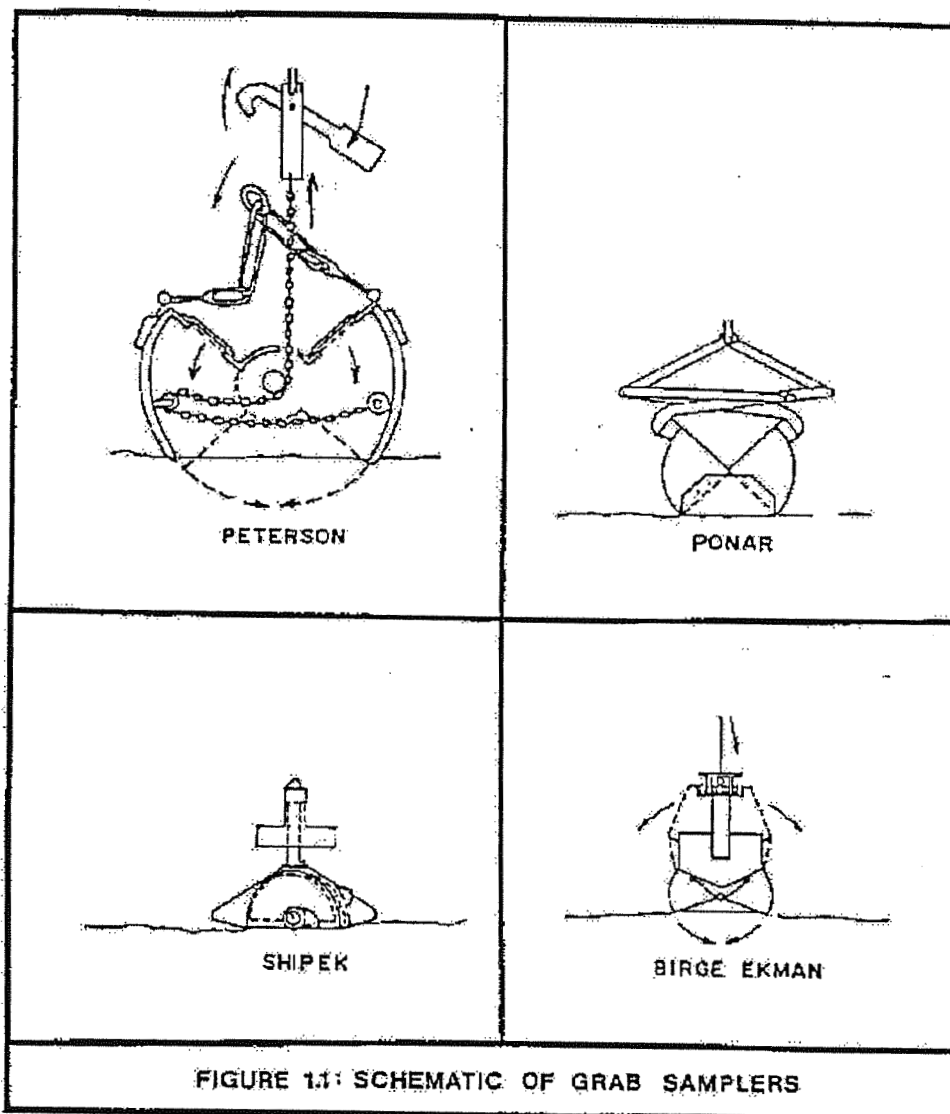
"stream", its wide base gives good stability and stance once it has come to rest on the sediment floor. Under poor sampling conditions, however, it becomes impossible to operate because: (a) the sampler, due to its light weight, is continually being lifted and dropped and "streamed" along the bottom and (b) any slack in the line, particularly near the sampler, is likely to impede the proper function of the triggers' messenger weight. It tends to roll over after triggering on all but soft bottoms.

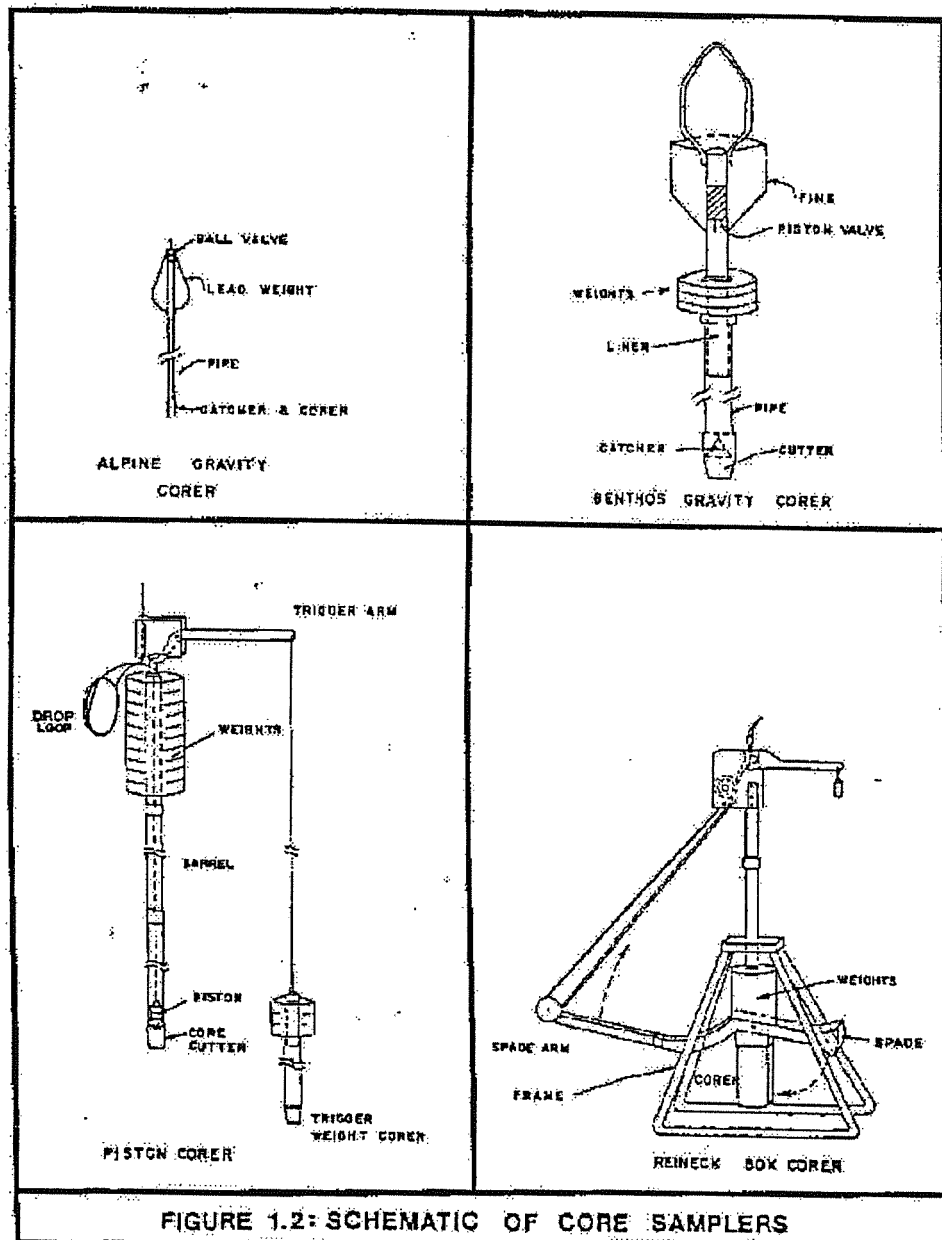
Petersen Grab	Good. This is a heavy sampler with a wide base line (when the jaws are open). It maintains a near vertical descent under all conditions, but after sampling it tends to fall over (unless on a soft bottom).
Ponar Grab	Very good. Comments as for Petersen; because of its weight and wide baseline (when jaws are open), this grab has a good vertical descent under most conditions and has a stable stance on the bottom. The presence of the fixed side plates prevents the grab from falling over after jaw closure and helps in preserving a near perfect bottom sample.
Shipek Grab	Excellent. Despite the large size of this Grab sampler, its weight ensures a near perfect vertical descent even under conditions of rapid drift or fast water flow. The sampler is also very stable even on bottom slopes 20 degrees or more. This stability ensures the minimum possible disturbance of the sample material.

CORE SAMPLERS

Characteristics

Benthos Gravity Corer	Cores of 3 m or less in soft clays, muds or sandy silts, particularly suitable for studies of the sediment/water interface, for studies on depositional sediment structures.
Alpine Gravity Corer	Cores of 2 m or less in almost all sediment types. The rugged nature of this corer lends itself to general usage. For studies involving sediment structure or large volumes of material, the corer is unsuitable; for studies of a pilot nature, or to prove the suitability of an area for piston coring, this gravity corer is excellent.
Phleger Corer	Cores of 0.5 m or less, in almost all sediment types, particularly suited to bottom materials containing a high percentage of fibrous organic material. The low cutter angle, the narrow wall thickness and high point loading, and the extremely sharp cutter, make it very suitable for sampling shallow lacustrine and estuarine deposits, marsh deposits and thin peat beds.
Multiple Corers	Still under investigation but early results indicate are very effective in soft sediments where a number of replicates are required or spatial heterogeneity is high.





A diver-collected core is preferred over a core collected by a free-falling coring device. The diver is able to carefully insert the liner in the sediment, minimizing the disturbance of the surficial sediment, virtually eliminating the compression problem and is able to use a relatively wide diameter liner. The use of a diver also permits an observation of the general nature of the bottom and the presence of aquatic biota. A limitation of the diver-collected core is that the retained length is typically less than 1 m.

In very specialized cases, where it is important to preserve the fine structure of the sediments, a large box-corer or hydraulic corer is recommended. The box-corer, because of its size, can collect a sediment sample with the centre undisturbed. A major drawback to the device is that it is large and complex to operate and collects a tubular core similar to the gravity corer. One advantage is the slow penetration which reduces the compression effects noted with the gravity corer.

The core and grab samplers described above are best used in fine sands or muds. Collection of grabs of coarse sand or cobble requires a large and adequately weighted grab (larger than 0.5 sq m capacity). A vibra-corer is required to collect a core in coarse or compacted sand. This device is similar to the gravity corer, but a vibration source is attached which vibrates the barrel down into the sand. Such a corer may require a specialized sampling boat, due to the weight of the equipment and the power requirements.

Positioning

Sample site locations, both field and correct positioning on a map, are to be submitted with the data for evaluation to the OMOE regional office. Positioning is especially important if the sites are to be re-sampled at a later date.

The sample sites can be determined using land marks, actual measurements, distance estimator or electronic positioning equipment.

A very important feature of the application for environmental review is the submission of a detailed plan of the project site, delineating the site boundaries and the location of the sample sites. A chart scale of 1:500 or 1:1,000 is recommended.

Field Notes

The information submitted by a proponent for evaluation should include field notes covering the

following points:

- weather conditions;
- time and date of collection;
- positioning information;
- type of sampler used;
- name of sampling personnel;
- notation of odd or unusual events which occurred during sampling (e.g., "corer returned only a few rocks");
- field description of samples:
 - odour,
 - approximate particle size,
 - colour,
 - presence of non-decomposed organics (e.g., wood fibres),
 - presence of oil and grease,
 - presence of distinct layering as given by changes in colour or particle size,
 - presence and type (to broad groupings) of aquatic biota, and
 - length of retained core;
- brief description of handling procedures and types of containers used;
- notation where there was a deviation from standard handling and splitting procedures; and
- laboratory to which samples were delivered and the date of delivery.

If the proponent is routinely having samples collected, a standardized form covering the field information is suggested in Table 1.3.

Physical Analysis

Before a sample is mixed and split in the field, the odour and colour should be noted and the pH and redox potential measured. Odour can be divided into four categories:

- Odourless
- Chemical
 - chlorine
 - petroleum
 - medicinal - phenol, iodine

- sulfurous
- Decaying Organic - manure
 - sewage
- Natural
 - earthy
 - peat
 - grassy
 - mouldy

Colour can be best determined by comparison of the sediment to the Munson colour code system. If that is not available, each colour zone or depth of core should be described. Colours will range from reddish-brown to jet black.

The pH and redox conditions should be measured with appropriate electrodes which have been properly calibrated. The electrodes should be rinsed with clean water between measurements and stored in appropriate containers. Accuracy of measurement should be ± 0.1 pH units; ± 10 mv for redox potential.

1.1.2 Sample Handling and Preliminary Processing

Field Storage and Handling (Grab Samples)

If redox and pH measurements are required, then the probes should be inserted into the sediments (3-5 cm), as soon as the grab is on-board. If possible, the probes should be inserted and samples removed through top-access doors rather than transferring (and thereby mixing) the sample into a pan.

Observations should also be made at this time: presence of oxidized surface layer, colour and smell of underlayer, approximate particle size description and presence of obvious oil or grease or non-decomposed organics (e.g., wood fibres).

The top 3-5 cm of the grab sample should be transferred into a clean pan and thoroughly mixed using a large, clean teflon or ceramic spoon. Subsamples should be handled as follows:

- I. For metals/particle size/carbon/phosphorus/total Kjeldahl nitrogen/loss on ignition, place in clean plastic or glass containers; and

TABLE 1.3 FIELD OBSERVATIONS

Sampling Location _____		Date and Time Sampled _____	
Field Sample Number (Separate sheet for each sample) _____			
(NEI: In the case of a core, submit sheet for each sub sample noting depth from top of section (in cm) _____			
Weather Conditions _____		Sampler Used _____	
Sampling Personnel _____			
Position of Samples (Attach separate sheet if necessary) (Also Note on Maps) _____			
Special Remarks (note problems with sampling) _____			
Sediment Type _____			
Odour _____			
(Indicate with "X")			
Odourless Chemical	•	chlorine	
	•	petroleum	
	•	oil	
	•	gasoline	
	•	medicinal - phenol, iodine	
	•	sulfurous	
Decomposing Organics	•	manure	
	•	sewage	
Natural	•	earthy	
	•	peat	
	•	grassy	
	•	slimy	
Approximate Particle Size Distribution:			
% gravel	% sand	% silt	% clay
_____	_____	_____	_____
Presence of coarse cobble _____			
Type _____	Colour _____ (visual or Munsell colour code)		
	•	clean sampler	
	•	wood bits	
	•	dead leaves and grasses (from shore) (detritus)	
	•	dead aquatic plants and roots	
	•	shells	
	•	obvious animal greases	
	•	petroleum greases	
	•	oil sheen on water or sediment	
Approximate Depths of Layers (in cores) (cm from top) _____			

2. For trace organics/oil/grease, place in clean solvent-rinsed glass bottles with clean aluminum foil cover caps. Amber-coloured bottles are preferred.

The amount of sediment required for analyses should be determined in consultation with the analytical laboratory. The samples must be kept at 4°C and out of sunlight. Samples should be shipped to the laboratory as soon as possible after collection. Sample containers should be carefully labelled with indelible ink pens. Labels should contain the following information:

- date and time of collection,
- identification of collector, and
- site identification (including harbour name).

This information should correspond to information recorded in the field notes.

Field Storage and Handling (Cores)

With the bottom end of the liner securely capped, the excess water should be carefully decanted or siphoned. The core may need to stand for some time to permit settling out of disturbed material before decanting.

The length of retained material should be measured in centimetres. Excess core liner should be cut off and the top of the liner capped. The core should be retained upright and carefully labelled. It is suggested that the label be placed only on the top end of the liner, to ensure that the core is not inadvertently turned over during transit or storage. The core should be handled in such a way as to prevent "sloshing" of the material.

As with the grab samples, the core samples should be stored at 4°C. As the cores may be long and cumbersome, it may be convenient to split the cores in the field. (This is best done on shore). Before extrusion, the core should be examined and the depths where redox discontinuities occur should be noted. The cores should be extruded from the bottom end (the firmer end). The core sample may be sectioned in one of two ways. The first way is to section the sample according to the different layers if the colours are obvious. Otherwise, samples may be sectioned into top, middle and bottom sections. The actual amount should be determined in consultation with the analytical laboratory. Each section should then be treated as a separate sample and handled as described above. This will include measurements of pH and redox, noting colour, odour, redox discontinuities, approximate particle size

and presence of oil or organic matter; non-decomposed organics etc.

Field Storage and Handling where samples will be subject to Partial Geochemical Leaching

It is critical that the samples not be exposed to air, as geochemical changes may occur. As soon as the grab or core sample is brought on board, the sediment should be placed in a nitrogen-filled bag or glove-box. All sub-samples should be purged with nitrogen and maintained at 4°C. The samples should not be allowed to dry out or be frozen.

Laboratory Handling and Storage

There should be sufficient facilities in the analytical laboratory to store all of the samples at the appropriate storage temperatures. As soon as the samples enter the laboratory, they should be logged into the laboratory sample management system and labelled with the laboratory control number. It is expected that the laboratory undertaking the work has shown evidence of good laboratory practices (i.e. adequate quality control and quality assurance). Care must be taken to ensure that the samples are not contaminated by other samples in the laboratory.

Archive and Duplicate Samples

Sediments may be heterogenous and therefore must be thoroughly mixed before they are sub-sampled. Each container should be mixed and sub-samples taken for the required analysis. Remaining material should be combined into one container and this preserved frozen as an archive sample. This should be retained for at least one year or until the dredging operation is completed.

The purpose of the archive sample is to permit subsequent re-analysis for a particular constituent or external audit analysis. For field and laboratory quality control and quality assurance, the following duplicates must be taken. For example, if 5 samples are to be taken, 1 additional sample is to be taken as a field duplicate. The laboratory views these as 6 unknowns and therefore there would be 6 laboratory samples and 1 laboratory duplicate for a total of 7 samples for analysis.

Sample Splitting Report

To assist the Ontario Ministry of the Environment in their review, the proponent is requested to submit a

sample splitting report. This report should contain the following information:

- pH and redox potential measurements (if requested);
- odour;
- visual description of particle size;
- presence of redox discontinuities (e.g., oxidized surface layer), if core, note the depth of discontinuity layers;
- presence of oil and grease;
- presence of non-decomposed organic matter (e.g., wood fibres);
- length of retained core;
- who collected, handled and split the samples; and
- deviations from splitting and handling routine as outlined above.

1.2 Sample Analysis

There are several manuals available which detail methods of analysis for sediments. The recommended manual is OMOE (1983), Handbook of Analytical Methods for Environmental Samples (2 Vol.), Ontario Ministry of the Environment, Toronto. Other acceptable manuals are:

- Plumb, R. H. Jr., 1981. Procedures for Handling and Chemical Analysis of Sediment and Water Samples. EPA/CE Tech. Rept. 81-1. U. S. Army Corps Engineers, Waterways Experiment Station, Vicksburg, Miss.
- Walton, A. (ed.) 1978. Methods for Sampling and Analysis of Marine Sediments and Dredged Materials. Ocean Dumping Report #1.
- Loring, D.H. and R.T.T. Rantala. 1977. Geochemical Analysis of Marine Sediments and Suspended Particulate Matter. Fisheries and Marine Service Tech. Rept. 700. Environment Canada, Ottawa.
- Carver, R.E. (ed.) 1971. Procedures in Sedimentary Petrology. Wiley-Interscience. Toronto.

1.2.1 Particle Size Analysis

The detailed particle size of the sample should be determined for the size range of -4 phi (16mm) (Wentworth Scale) to +9 phi (0.002mm). The determination between -4 and +9 phi may be made using a variety of techniques, these include: sieve, pipette and microtrac (used by OMOE). Percent gravel, sand, silt and clay should be calculated from the detailed particle size analyses.

1.2.2 Trace Inorganic Chemical Analysis

Nutrients

The Provincial Sediment Quality Guidelines require the determination of total organic carbon, total phosphorus and total Kjeldahl nitrogen. The methods of analysis are outlined in Plumb (1981) and OMOE (1983).

Trace Metals ("Bulk Metal")

In order to determine suitability for open water disposal, the Provincial Sediment Quality Guidelines (PSQGs) specifically include the following metals: mercury, lead, zinc, iron, chromium, copper, arsenic, cadmium, and nickel. In addition, analyses may be requested for cobalt and silver on a site specific basis. The metals are analyzed using dissolution in aqua-regia (i.e., hydrochloric : nitric, 3:1 acid digestion). The PSQGs were developed with metal concentrations that were obtained by using the aqua-regia acid digestion. All of these are routinely determined by atomic absorption spectroscopy or plasma emission spectroscopy with due consideration for chemical interferences and detection limit requirements and capabilities.

Trace Metals (Partial Leaching)

The use of bulk metal analysis simply characterizes the metal composition of the sample, but does not differentiate the geochemical distribution within the various sediment phases with which the metals are associated. The technique of partial chemical leaching can be used to describe the geochemical phase distribution of the metal(s) and has been used to provide an estimate of the bioavailability of the metal. The techniques developed are strictly method dependent and the results operationally defined. The methods are typically applied to cadmium, copper, lead, zinc, chromium, nickel and cobalt. The following procedure has been used extensively by OMOE and for comparability it is very important to follow the procedure as outlined and not other sets of sequential leaching procedures. Steps 1-3 must be done in a nitrogen-glove box or bag (under inert gas):

- Step 1 • **Interstitial Water** - Centrifuge the wet sediment at 15000 rpm. for 10 min. Decant and analyze supernatant. Sieve the material through a 64 micron sieve (i.e., only the silt-clay fraction).

- Step 2 ● **Cation and Adsorbed** - Shake the wet equivalent of 0.5 g dry weight of sieved material from stage 1 with 1M ammonium acetate solution for 2 hr. Centrifuge at 15000 rpm. for 10 min. Decant and wash sediment with distilled-deionized water; centrifuge and add rinse water to original solution. Acidify to pH 2 and store for subsequent analysis.
- Step 3 ● **Easily Reducible** - the residual sediment from stage 2 is shaken for 2 hr. at room temperature with 20 ml of 0.1M hydroxylamine hydrochloride at pH 2, centrifuged at 15000 rpm. for 10 min. and decanted. The sediment is rinsed with distilled-deionized water, centrifuged and the rinse added to the original decanted solution. Store for later analysis.
- Step 4 ● **Organic Complexed** - The residue from stage 3 is shaken for 1 hr. at room temperature with 20 ml of hydrogen peroxide acidified to pH 2 with nitric acid, then for 5 hr. at 95°C. Then 10 ml of 1M ammonium acetate (pH 2) is added and the extraction continued for 1 hr. The suspension is centrifuged at 15000 rpm. for 10 min. and decanted. The sediment is rinsed with distilled-deionized water, centrifuged and the rinse water added to the original decanted solution. Acidify the solution to pH 2 and store for subsequent analyses.
- Step 5 ● **Moderately Reducible** - The residue from stage 4 is shaken for 2 hr. at 95°C with 1M hydroxylamine hydrochloride/glacial acetic acid (1:1 volumes) solution. The suspension is centrifuged at 15000 rpm. for 10 min. and decanted. The sediment is rinsed with distilled-deionized water, centrifuged and the rinse water added to the original decanted solution. Store for subsequent analysis.
- Step 6 ● **Residual (Lattice)** - 1.0 g of dried material from the above is dissolved in 3:1 aqua regia following the method for "total" metal.

Calculations should be made of the relative percentage of total metal with each phase.

Other Inorganic Compounds

While the PSQGs do not include cyanide, analysis may be required on a site specific basis. The analytical methodology is a water extraction of the sediment, with distillation of the extract, followed by a colourimetric determination using a pyridine-barbaturic acid reagent. The method is outlined in OMOE (1983).

Other inorganic compounds (e.g., organo-leads) may be required to be determined as a result of OMOE Regional Office review. Appropriate methods of determination will be provided by OMOE at that time.

1.2.3 Trace Organic Chemical Analysis

There is an extremely large number of trace organic compounds that are of environmental concern. Many of these compounds may bioaccumulate and are suspected or known carcinogens or carcinogenic precursors. The open water disposal guidelines specifically identify PCB with an acceptable limit of 0.05 µg/g in sediment. However, with the increasing evidence that sediments in many areas of the Great Lakes and the Inter-connecting Channels are heavily contaminated with a wide variety of trace organics (e.g., Report of the Niagara River Toxics Committee, October 1984), the numerical guidelines for the evaluation of open water disposal are being expanded to include other substances.

PCB and Organochlorine Pesticides

The sample is extracted with distilled-in-glass acetone and dichloromethane, concentrated, and the PCB fraction separated from the pesticide fraction by liquid chromatography using for example, a Florisil column. The extracts are reduced in volume, cleaned with solvent-washed mercury to remove sulphur compounds and analyzed using gas chromatography with an electron capture detector. Calibration should be made using standards of the pure compounds. Quality assurance should be monitored by testing the extraction efficiency, the separation of the Florisil and the use of standard reference materials. The method is detailed in OMOE (1983). The procedure can be used to determine: PCB, BHC isomers, Chlordane (both isomers), DDD, DDT, DDE, Dieldrin, Endosulfan (isomers I, II and sulphate), Endrin, HCB, Heptachlor, Heptachlor Epoxide, Lindane and Methoxychlor.

Polyaromatic Hydrocarbons (PAHs)

This is a particular sub-group of the aromatic fraction of petroleum hydrocarbons. These compounds are produced by the incomplete combustion of coal, coke, petroleum hydrocarbons and various aromatic compounds. These compounds are generally considered to be carcinogenic or carcinogenic precursors.

Although not yet included in the guidelines, the proponent may be requested to determine the

concentrations of the following PAHs in sediments:

acenaphthylene	acenaphthene
benzo (a) anthracene	benzo (a) pyrene
benzo (b) fluoranthene	benzo (e) pyrene
benzo (ghi) perylene	benzo (k) fluoranthene
chrysene	dibenz (ah) anthracene
fluorene	fluoranthene
indeno (1,2,3,cd) pyrene	naphthalene
pyrene	phenanthrene
anthracene	

The sample is solvent extracted in a Soxhlet system, reduced in volume, cleaned up and analyzed by high pressure liquid chromatography with fluorescence and UV-Vis detectors. Calibration is made against appropriate standards of the pure chemicals. The National Research Council of Canada will shortly be offering for sale four marine sediment reference materials, each spiked with selected PAHs which together will provide analysts with a range of concentrations for the 16 PAHs listed above.

Other compounds

Additional compounds may be required to be determined after OMOE review. The methods of determination will be provided at that time by OMOE.

1.2.4 Miscellaneous Analyses

Organic/Inorganic Carbon

This category is often labelled as "loss of weight on ignition" or total organic matter. There are two principal techniques used: combustion in a muffle furnace at fixed temperatures ("loss of weight on ignition"); and, combustion in a high temperature furnace in a stream of oxygen with determination of the resultant carbon dioxide (furnace method). In both techniques, the dried sample is heated to a specific temperature and the resultant weight or carbon dioxide generation can be quantified to represent total carbon. The other sub-sample is treated with hydrochloric acid to remove the carbonate carbon, and the treated sub-sample analyzed as above with this fraction defined as the organic carbon. The difference is taken as the inorganic carbon fraction. Both procedures are detailed in OMOE (1983).

Oil and Grease

This is a catch-all category which includes petroleum hydrocarbons but mainly hydrocarbons of a biogenic origin, such as fats, waxes and greases. The wet sample is extracted with dichloromethane and the resultant extract is solvent evaporated and the residue weighed (gravimetric method). A detailed procedure is given in OMOE (1983).

A major drawback is that the method is non-specific. Most non-halogenated hydrocarbons are extracted and the detection method does not offer any means of discriminating between the various hydrocarbon types. The present guidelines specify a limit of 1500 µg/g dry weight. Typically this is taken as total extractable matter. Obviously, significant quantities of non-petroleum hydrocarbons (e.g., sewage) will give a "high" value which cannot be directly correlated with lethal or sub-lethal effects due to petroleum hydrocarbons which might be present.

Analyses for chemical determinations (metals, nutrients, organics) should be made using a method that achieves the lowest detection limit.

2.0 QUALITY ASSURANCE/QUALITY CONTROL CONSIDERATIONS

2.1 Introduction

The "quality" of data is a function of the uncertainty of the data compared to its end-use requirements. Thus, data can be acceptable under one requirement and be unacceptable under another. Quality assurance is the mechanism by which data are produced to meet a defined standard of quality with a stated degree of confidence. Quality assurance quantifies the uncertainty associated with reported data. Quality control is the series of activities used to obtain and maintain that standard.

Quality assurance and quality control (QA/QC) are important elements in all facets of a project. They are mechanisms whereby the proponent can monitor the performance and results of his staff or contractors, and they permit the regulatory agency to determine the quality of data submitted as part of a project review. The complexity of environmental data and the need for comparability has led to requirements for quality assurance and control in the analytical laboratory without necessarily recognizing that quality assurance and control must be applied throughout the program. For example, poor sampling or sample handling practices can obviate the most careful laboratory analyses.

QA/QC should not be considered as just another requirement by the proponent; it should be recognized that it is in the proponent's best interests to provide quality data, since it is that data which will be used to determine the restrictions on the dredging/disposal components of the project.

There are five basic elements in QA/QC:

- completeness of the data set;
- representativeness of the data;
- comparability of data;
- validity of identification; and
- accuracy and reproducibility of quantification.

Completeness of the measurement can be defined as obtaining the amount of data that OMOE expected to be obtained to meet the project objectives.

The representativeness of the data is the degree to which data accurately and precisely represent the concentrations of the constituents or the characteristics of the material to be dredged. For example, a grab sampler samples the top 10 to 15 cm of sediment. If a 1 m dredge cut is to be made, then the sample represents only the top 10 to 15% of the material to be dredged.

The comparability of data is defined as the degree of confidence with which one data set can be compared to another or to guideline concentrations. This requires that proponents use consistent and documented methods throughout the data collection. It is the purpose of these guidelines to ensure that data generated be obtained by a common set of procedures. Recognizing that sampling and analytical methods are constantly being changed and improved, the Ministry may accept non-standard procedures after consultation with the proponent.

The validity of identification is important for environmental samples where the analyst is asked to determine low concentrations of contaminants in complex matrices. This is particularly true for trace organic compounds (e.g., PCB).

The accuracy and reproducibility of quantification are the elements which most people use to define QA/QC. Quantitative measurements are only estimates with a stated degree of probability of uncertainty. Measurements must be made in a sufficient number and way so as to provide a statistically acceptable definition of the degree of confidence. This will require the analysis of a number of replicate samples by the analytical laboratory and the analysis of standard reference

materials. The results of these additional analyses should be included with the application.

2.2 Elements of QA/QC Program

The basic elements of a QA/QC program include:

- technical competence of staff;
- suitability of facilities and equipment;
- good measurement practices;
- standard operating procedures;
- special operating procedures; and
- good documentation.

Good measurement practices can be defined as those procedures which have been tested and refined so that the results are consistently accurate and precise. Such practices can range from ensuring that equipment is routinely maintained and calibrated to procedures for the cleaning of sample containers. Typically such procedures are not documented but are assumed; unfortunately because they are not documented, such practices are variable even within a department.

Standard operating procedures are those procedures which specify how samples are to be collected, handled and analyzed. Such procedures should include the necessary information so that the techniques used can be repeated by another group or laboratory. Similar to the good measurement practices, standard operating procedures are typically not documented except where a laboratory is required to use a method published in a laboratory manual. Such operating procedures must include the procedures necessary to calibrate the techniques or to position the sampling equipment.

Most laboratory procedures are documented and would therefore be classified as standard operating procedures. However, sampling and sample handling procedures are often not documented and therefore special operating procedures must be developed and documented. These procedures should be developed with the assistance of well-qualified staff to ensure that all relevant points are included.

In each stage of the QA/QC, documentation of the procedures and techniques is important. It is in the proponent's best interests to ensure that all procedures are carefully and thoroughly documented, including obtaining relevant documentation from the laboratory. Full documentation will also ensure that the same techniques are used each time samples are collected and analyzed, thus permitting

comparison of the data.

2.3 Implementation of QA/QC Program

2.3.1 Sampling

This component is the most difficult to monitor for QA/QC. The proponent must ensure that samples are collected using acceptable equipment and at the specified locations. A check on equipment can be made by the collection of samples from a relatively non-contaminated area. Equipment must also be maintained in good condition and kept clean of obvious contaminants such as rust and oil and grease. Sample site positioning is very important, particularly when sampling at close intervals or if the site is to be re-sampled at a future date. Specialized positioning and survey equipment may be required.

Suitability of facilities and equipment includes the sampling equipment, the vessel, the positioning equipment, the sample handling equipment and the laboratory facilities. The guidelines require the use of a grab or core sampler of suitable size and design to collect an adequate sample. Due to the weight of such equipment, winches and a boom of suitable capacity will likely be required.

2.3.2 Sample Handling and Processing

The proponent must ensure that the samples are transferred from the collection equipment to the laboratory while maintaining the structure of the sample, limiting contamination and ensuring that there is minimal loss of constituents. It is recommended that samples be transferred as soon as possible from the sampling device to transfer containers. These containers must be clean. Plastic containers should be used for sub-samples intended for trace metal, particle size and miscellaneous components; glass containers should be used for sub-samples intended for trace organic analyses. One container of each type and set should be retained and tested as a sample container blank.

If samples cannot be processed and analysis started within 48 hours of collection, they may be stored at 4°C for longer periods.

2.3.3 Laboratory Analysis

In addition to the requirements for suitable facilities and personnel, specific components in laboratory analysis include:

- sample receipt;
- sample processing;
- control charts;
- standard reference materials;
- non-standard reference materials;
- replication; and
- external auditing.

Sample receipt should include maintaining proper documentation, chain- of-custody and ensuring that the specific analytical requirements are defined by the client. A proper logging-in system with some type of laboratory identification is a prerequisite.

Sample processing can introduce errors due to the heterogeneity of sediment. Samples should be well homogenized before sub-sampling. To assess sample processing quality, split samples should be taken from 10% of the samples and handled as additional unknowns. An archival sub-sample should always be taken and maintained frozen for at least one year. A suitable area should be maintained for sample processing and great care taken to avoid cross-contamination.

Control charts are critical to assessing the performance of the analysis stage. Control charts should be maintained for calibration functions, extraction/digestion efficiencies, reagent blanks and for determining the reproducibility of a measurement process. Control charts can be constructed by the use of repetitive analysis over a period of time of a particular sample or set of samples. In many cases, standard reference materials are used; however, any well-homogenized sediment can be used. While the latter type of materials cannot be used to monitor bias, the initial concern should be to maintain consistency and reproducibility. Each chart should consist of the mean and difference of duplicate determinations with warning and control limits set at two and three standard deviations respectively. The use of control charts should be an ongoing task and should be monitored by the supervisory analyst to ensure that a group of analyses does not go "out of control".

The concurrent analysis of standard reference materials provides both a monitor on bias in the analysis and provides a source of data for the control charts. A variety of sediment reference materials are now available from both the National Research Council of Canada and the U.S. National Bureau of Standards. A limited number of standard reference materials for other matrices (e.g., biota) are also

now becoming available. The major drawback is that most of these materials are for trace metals; very few are available for trace organics. It is recommended that two reference materials be concurrently analyzed with each set of twenty samples. Results of analyses should be included with the application.

The proponent may also want to audit the analysis by the use of non-standard reference materials. However, care must be taken to ensure that:

- the materials are similar in matrix and concentration range to the unknowns; and
- the samples are submitted as unknowns to the analyst, so that they are truly "blinds".

There are a number of sources of non-standard reference materials; however, their major drawback is often poor characterization. Replication is an easy method of maintaining control charts and monitoring QA/QC. It is recommended that 10% of the samples be concurrently analyzed in duplicate and that this data be included in the application.

External auditing is also useful as a monitor of QA/QC. This can include participation in inter-laboratory comparisons and participation in contracted analyses of reference materials. Use of outside experts (e.g., university research scientists, National Water Research Institute) to review operating procedures and control charts is also useful.

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GLOSSARY

OMOE - Ontario Ministry of Environment.

IJC - International Joint Commission.

APHA - American Public Health Association.

ASTM - American Society for Testing and Materials

PWC - Public Works Canada.

EPS - Environmental Protection Service.

ECPS - Environmental Conservation and Protection Service.

NWRI - National Water Research Institute.

CDF - Confined disposal facility.

QA/QC - Quality control/quality assurance.

LC50 - Concentration lethal to 50% of the exposed population.

EC50 - Concentration producing an effect in 50% of the exposed population.

OECD - Organization for Economic and Co-operative Development.

EPA - U.S. Environmental Protection Agency.

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