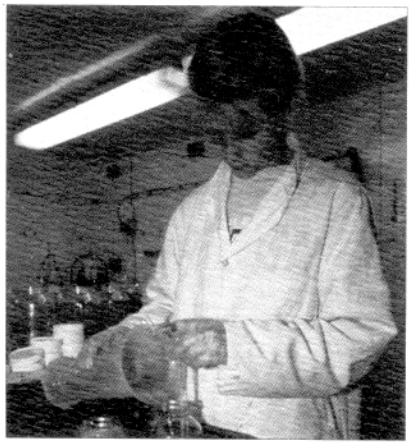
Environmental Protection Series



Guidance Document on Collection and Preparation of Sediments for Physicochemical Characterization and Biological Testing

Report EPS 1/RM/29
December, 1994

Canada

Environment Canada Environnement Canada

Guidance Document on Collection and Preparation of Sediments for Physicochemical Characterization and Biological Testing

Method Development and Applications Section
Environmental Technology Centre
Environment Canada
Ottawa, Canada

Report EPS 1/RM/29
December 1994

CANADIAN CATALOGUING IN PUBLICATION DATA

Main entry under title:

Guidance document on collection and preparation of sediments for physicochemical characterization and biological testing

(Report ; EPS 1/RM/29)
ISBN 0-660-16297-0
Cat. no. En49-24/1-29E

1. Marine sediments – Analysis.
2. Toxicity testing – Methodology.
I. Canada. Technology Development Directorate.
II. Canada. Environment Canada.
III. Series: Report (Canada. Environment Canada);
EPS 1/RM/29.

QD79.S4G84 1995 551.4'6083 C95-980001-0

Catalogue No. En 49-24/1-29E
ISBN 0-660-16297-0

Readers' Comments

Comments regarding the content of this report should be addressed to:

Richard Scroggins
Method Development and Applications Section
Environmental Technology Centre
Environment Canada
335 River Road
Gloucester, Ontario
K1A 0H3

Cette publication est disponsible aussi en français. Pour l'obtenir, s'addresse à:

Publications de la Protection de l'environnement
Direction générale de l'avancement des technologies environnementales
Environnement Canada
Ottawa (Ontario)
K1A 0H3

This report was edited by the Publications Unit of the Environmental Technology Advancement Directorate, Environment Canada.

Abstract

This document describes methods recommended by Environment Canada (EC) for the selection of sampling stations within a study site, and the collection, handling, storage, transportation, and manipulation of samples of whole sediments from marine, estuarine, and freshwater environments, for the purposes of physicochemical characterization and/or biological assessment using whole sediments, pore waters, or sediment elutriates.

General methods and procedures are outlined for two types of undertakings:

- *monitoring and assessment studies (Section 2), and*

- *studies specified in permit requirements for open-water disposal of dredged materials (Section 3).*

Included in the general procedures are recommended methods of collection for test, control, reference sediment, in-situ *collection of pore water, sample handling, transportation, storage methods or conditions, and whole-sediment sample preparation for bioaccumulation tests, physiochemical characterization and/or to toxicity testing. Methods are also recommended for the laboratory collection of pore water and elutriate from field-collected whole-sediment samples. Additional procedures or conditions specific to the various aquatic environments are also addressed.*

Résumé

Le présent document décrit les méthodes recommandées par Environnement Canada pour le choix de points d'échantillonnage sur les lieux d'une étude ainsi que pour le prélèvement, la manutention, le stockage, le transport et la manipulation d'échantillons de sédiments entiers provenant de milieux marins, estuarins et d'eau douce. On utilise ces échantillons entiers, l'eau de porosité qu'ils contiennent ou des élutriats produits à partir de ces sédiments à des fins de caractérisation physiocochimique et d'évaluation biologique.

Des méthodes et des modes opératoires généraux sont décrits pour deux types d'études:

- *les études de surveillance et d'évaluation (section 2);*

- *les études requises pour l'obtention des permis d'immersion en eau libre de matières draguées (section 3).*

Les modes opératoires généraux comprennent des méthodes recommandées pour le prélèvement de sédiments d'essai, de contrôle et de référence, pour la collecte in situ *de l'eau de porosité, pour la manutention, le transport et le stockage des échantillons de sédiments entiers en vue d'essais de bioaccumulation, de caractérisation physicochimique et de toxicité. On y trouve aussi des méthodes recommandées pour la collecte de l'eau de porosité et pour l'obtention d'élutriats en laboratoire, à partir d'échantillons de sédiments entiers prélevés sur le terrain. Le document contient également des modes opératoires particuliers tenant compte des conditions propres à chacun des milieux aquatiques visés.*

Foreword

A series of guidance manuals and recommended testing methods for measuring and assessing the biological effects of toxic substances in freshwater, estuarine, and marine environments has been developed by Environment Canada.

Recommended guidance methods for those which have been evaluated by the Environmental Protection Service (EPS) and Environmental Conservation Service (ECS) and are favoured:

- *for use in EPS or ECS aquatic and sediment toxicity laboratories;*
- *for testing which is contracted out by Environment Canada or requested from outside agencies or industry;*
- *in the absence of more specific instructions, such as are contained in regulations; and,*
- *as a foundation for the provision of very explicit instructions as may be required in a regulatory protocol or standard reference method.*

These reports are intended to provide guidance and to facilitate the use of consistent, appropriate, and comprehensive procedures for obtaining data on toxic effects of samples of chemical, effluent, elutriate, leachate, receiving water, or sediment. This report is to serve as a companion document to the biological testing methods, in the Environmental Protection Series, that describe toxicity or bioaccumulation tests with whole sediment, pore water, or elutriates of sediment. The methods described within this guidance manual for the collection, handling, transportation, storage, and manipulation of whole sediment, and pore water, and sediment-elutriate samples, are germane to the acceptability and success of the recommended test methods involving sediment toxicity evaluation. Although considerable guidance is provided within, key original references should be consulted for details.

Mention of trade names in this report does not constitute endorsement by Environment Canada; other products (e.g., materials and equipment) of equal value are available.

Table of Contents

List of Tables

List of Figures

List of Abbreviations and Chemical Formulae

Ag . . . . . silver
Al . . . . . aluminium
AVS . . . . . Acid volatile sulphide
Ba . . . . . barium
Be . . . . . beryllium
Ca . . . . . calcium
Cd . . . . . cadmium
CEC . . . . . cation exchange capacity
Cl . . . . . chloride
cm . . . . . centimetre(s)
cm^3 . . . . . cubic centimetre(s)
Co . . . . . cobalt
CO_2 . . . . . carbon dioxide
Cr . . . . . chromium
Cu . . . . . copper
$CuSO_4$ . . . . . copper sulphate
DDW . . . . . distilled deionized water
DIC . . . . . dissolved inorganic carbon
DO . . . . . dissolved oxygen (concentration)
DOC . . . . . dissolved organic carbon
DQO . . . . . data quality objectives
EC . . . . . Environment Canada
EDTA . . . . . ethylenediamine tetraacetate
Eh . . . . . oxidation-reduction potential
Fe . . . . . iron
F . . . . . fluoride
g . . . . . gram(s)
G . . . . . unit of acceleration (= 9.8 m/s^2)
GPS . . . . . **g**lobal **p**ositional **s**ystem
h . . . . . hour(s)
H_2O . . . . . water
H_2SO_4 . . . . . sulphuric acid
HCl . . . . . hydrochloric acid
Hg . . . . . mercury
HNO_3 . . . . . nitric acid
I.D . . . . . inner diameter
K . . . . . potassium
kg . . . . . kilogram(s)
kHz . . . . . kilohertz
km . . . . . kilometre(s)
L . . . . . litre(s)
Li . . . . . lithium
LOI . . . . . loss on ignition
LORAN . . . **LO**ng-range **RA**dio **N**avigation
M . . . . . molar
m . . . . . metre(s)
m^2 . . . . . square metre(s)
mg . . . . . milligram(s)
Mg . . . . . magnesium
min . . . . . minute(s)
mL . . . . . millilitre(s)
mm . . . . . millimetre(s)
Mn . . . . . manganese
Mo . . . . . molybdenum
MPS . . . . . minimum performance standard
Na . . . . . sodium
NH_4-N . . . . . ammonium nitrogen
Ni . . . . . nickel
NO_2-N . . . . . nitrite nitrogen
NO_3-N . . . . . nitrate nitrogen
O_2 . . . . . oxygen
P . . . . . phosphorus
PAH . . . polynuclear(polycyclic)armomatic hydrocarbons
Pb . . . . . lead
PCB . . . . . polychlorinated biphenyls
QA . . . . . quality assurance
QA/QC . . . quality assurance/quality control
QC . . . . . quality control
® . . . . . Registered trade name
RADAR . . . **RA**dio **D**etection **A**nd **R**anging
s . . . . . second(s)
S . . . . . sulphur
SATNAV . . . . . **SAT**ellite **NAV**igation
Sb . . . . . antimony
SD . . . . . standard deviation
Si . . . . . silicon
SiO_2 . . . . . silica or silicon dioxide
SI . . . . . International System of Units
SO_4 . . . . . sulphate
Sr . . . . . strontium
TC . . . . . total carbon

TKN . . . . . total Kjeldahl nitrogen
TM (™) . . . . . Trade Mark
TOC . . . . . total organic carbon
TVS . . . . . total volatile solid(s)
Va . . . . . vanadium
× G . . . . . times 9.8 m/s^2
Zn . . . . . zinc
± . . . . . plus or minus
< . . . . . less than
> . . . . . greater than
≤ . . . . . less than or equal to
≥ . . . . . greater than or equal to
% . . . . . percentage or percent
‰ . . . . . parts per thousand
° . . . . . degree(s)
° C . . . . . degree(s) Celsius
µg . . . . . microgram(s)
µm . . . . . micrometre(s)

Terminology

Note: All definitions are given in the context of the procedures in this report, and may not be appropriate in another context.

Grammatical Terms

Must is used to express an absolute requirement.

Should is used to state that the specified condition or procedure is recommended and ought to be met if possible.

May be used to mean "is (are) allowed to".

Can is used to mean "is (are) able to".

Might is used to express the possibility that something could exist or happen.

General Technical Terms

Acoustic mapping is the conversion of acoustic signal information by electronic techniques to form a representative physical image.

Acoustic survey is the practical application of sound energy for probing solid media. Instrumentation technologies include sound and vibration generators, sound and vibration sensors, recording systems, and data analyzers.

Artifact is an undesirable, detectable feature (e.g., chemical or physical change) in a substrate, that has resulted from the activities or manipulations of those substrates.

Assessment studies are projects that undertake the systematic gathering of information for the purpose of identifying and describing a specific condition in an ecosystem or environment.

Bathymetry is the science of measuring the depths of a body of water or the information resulting from such measurements.

Benthic is an adjective used to describe organisms, samples, or bottom material related to, living in, or associated with the benthos.

Benthos refers to a region at the bottom of a body of water (e.g., the sediment-water interface) or the organisms living in this region.

Chain-of-continuity is the documentation that establishes the control of a sample between the time it is collected and the time it is analyzed. It usually applies to legal samples to

demonstrate that there was no tampering with or contamination of the sample during this time. It is synonymous with chain-of-custody.

Clean sediment is sediment that contains no substances at concentrations that cause discernible distress to the test organisms or reduce their survival, growth, or reproduction.

Clean water is marine, estuarine, or fresh water that contains no substances at concentrations that cause discernible distress to the test organisms or reduce their survival, growth, or reproduction.

Composite sample is a sample that is formed by combining material from more than one sample or subsample.

Constitutent(s) is (are) the chemical substance(s), solid(s), dissolved or particulate organic matter, and organisms associated with or contained in or on sediments.

Contaminant(s) refers to any undesirable agent, or substance, or material that is present in sediments or water.

Control sediment is a field-collected or artificially prepared (e.g., formulated), clean sediment (i.e., essentially free of contaminants) of known physicochemical composition, and of consistent quality. This sediment must not contain concentrations of contaminants that affect the test organisms in any way. The physical characteristics of the control sediment should be within the tolerance thresholds of the test organisms. The control sediment should be free of organisms which might interfere with the test organisms. The use of control sediment in tests provides a basis for interpreting data derived from toxicity and bioaccumulation tests using test sediment, and it can also be used in reference tests to monitor the health of test organisms, the relative sensitivity of test organisms over time, and the "performance" of laboratories.

Core sample is a sample of sediment that has been collected using a core sampler.

Core sampler is a devise used to collect a column of sediment (e.g., core sample) which when analyzed represents the historical or vertical distribution of the physical and chemical characteristics of the sediment.

Data Quality Objectives (DQOs) are pre-defined criteria for the quality of data generated or used in a particular study so as to ensure that the data are of acceptable quality to meet the needs of the program.

Deionized water is fresh water that has been purified to remove ions from solution by passing it through resin columns or a reverse osmosis system.

Disposal site refers to a site or area within which disposal of a substance or material at sea (Section 3) is permitted in accordance with the terms and conditions of a valid ocean dumping permit.

Distance line is a graduated line or wire that is stretched at a right angle, between the shore and a vessel or person positioned at the sampling station, to measure the distance. The location of the shore station must be recorded, or marked, if relocation of the sampling station is desired.

Distilled water is water that has been passed through a distillation apparatus of borosilicate glass or other material to remove impurities.

Dredge, as a verb, is the act of excavating a quantity of sediment; as a noun, it is a barge equipped to excavate sediment.

Dredge sampler is a type of device used to collect sediments specifically for the assessment of benthic organisms.

Dredge site is the area to be dredged.

Dredged material is material excavated or dredged from waters.

Dumping is the action of the disposal of substances at sea, or in fresh or estuarine waters.

Echosounder is an instrument that generates acoustic pulses (e.g., ultrasonic pulse) and receives the reflected signal. It is synonymous with fathometer, or sonic depth finder.

Elutriate is an aqueous solution obtained after adding water to a solid substance or loose material (e.g., sediment, tailings, drilling mud, dredge spoil), shaking the mixture, then centrifuging or filtering it or decanting the supernatant.

Epibenthic is an adjective used to describe organisms that have regular contact with sediment and live just above the sediment/water interface, to a depth of about 100 fathoms which is approximately 183 m (when rounded to a metric equivalent ~ 200 m).

Extract is the solution obtained from batched procedures, column leaching, or Soxhlet extraction methods, after adding an extractant or extractants (e.g., acids and/or solvents) to sediments. Extraction procedures may also include a single or multiple extraction with one chemical, a mixture of chemicals, or sequential extractions with more than one organic extractant.

Fathometer is synonymous with echosounder; it is a device that is used to determine the depth of water by measuring the time interval between a transmission of an acoustic impulse and the reception of its bottom echo (reflection).

Fine-grained sediment is sediment comprised of predominantly particles $\leq$63 µm (i.e., silts and clays).

Geochemistry is the study of the chemical elements, their isotopes and related processes, with respect to their abundance and distribution within solid rocks, consolidated and unconsolidated sediment, and interstitial water.

GPS (Global Positional System) refers to a relatively new navigation system that relies on satellite information. It was developed by the U.S. military and can give continuous position reports. This system is considered to be the navigational system of the future and it is likely to become widely used.

Grab sample is a sample of sediment that has been collected using a grab sampler.

Grab sampler is a mechanical device used to collect sediment. It generally consists either of a set of jaws that close when lowered to the surface of the bottom sediment, or it has a bucket that rotates into the sediment upon reaching the bottom.

Holding time is the period of time during which a sample of sediment can be stored after collection. Changes that occur in sediments with respect to the physical, chemical, or biological characteristics should be minimal during this period, and the integrity of the sample should not be compromised to any substantial degree. The sediment should be analyzed or used in a biological test preferably within this time period or immediately thereafter.

Hydrographic survey is a survey of the physical characteristics of a body of water including currents, depth, bottom topography; it may also include chemical and physical properties of water.

Interstitial water is the water occupying space between sediment particles. The amount of interstitial water in sediment is calculated and expressed as the percentage ratio of the weight of water in the sediment to the weight of the whole sediment including the interstitial water. Interstitial water is synonomous with pore water. It can be recovered from sediment *in situ*, usually by means of a dialyzer, or it can be recovered from field-collected samples of sediment by methods such as squeezing, centrifugation, or suction.

Lacustrine is an adjective that describes a lake or reservoir environment or sediments in a lake.

Legal sample is a sample that is collected with a view to prosecution (i.e., the analytical results might be admissible in court). A legal sample is considered to be representative of the substance or material being sampled and must be free of contamination by foreign substances during or after sampling. The origin of the sample, time and method of collection must be identified and the chain-of-continuity (chain-of-custody) clearly documented. Legal samples are transported in labelled containers with a seal, stored in a secure and locked place, and processed as soon as possible after collection.

Line-of-sight is a straight line between two points (e.g., from a sighting or surveying instrument to the object being sighted). In radar/radio applications, it refers to the direct, unobstructed straight path between the source and the target.

LORAN (**LO**ng-ranged **RA**dio **N**avigation) is a widely used radio system used for navigation. The system determines the position of hyperbolic lines by measuring the difference of time in receiving signals from fixed, synchronized transmitters.

LORAN-C is the LORAN navigation system that operates in the frequency band of 100–110 kHz.

Macrofauna refers to benthic organisms that can easily be seen with the naked eye.

Meter wheel is a measuring block over which a measuring line passes. The measuring line is both graduated and weighted, and it is used to measure the depth of water from the surface of the sediment to the surface of the water. It is accurate to within ± 0.2 m, and is readily available in different sizes from most companies that sell sampling equipment.

Microfauna refers to benthic organisms that can not easily be seen with the naked eye.

Monitoring studies are projects that undertake to measure or evaluate the conditions in a particular ecosystem or environment on a repetitive basis.

Pelorus is a device for measuring relative horizontal bearings of observed objects in degrees.

Penetration depth refers to the depth of sediment that the sampling device should penetrate to collect a sample of sediment. It is generally greater than the desired sampling depth.

Physicochemical characterization refers to the analysis of sediment or interstitial water to determine physical and chemical properties or constituents (such as: pH, particle size distribution, major ion concentrations, cation exchange capacity, redox potential, salinity, ammonia, total organic carbon, and total volatile sulphides).

Pore water is synonomous with interstitial water.

Pressure sieving is the mechanical pressing of sediment particles through a sieve of a particular mesh size.

Quality Assurance (QA) refers to the management and technical practices (e.g., planning, control, assessment, reporting, remedial action) designed to ensure an end product of known or reliable quality.

Quality Control (QC) refers to the techniques and procedures used to measure and assess data quality and the remedial actions to be taken when data quality objectives are not realized.

*RADAR (**RA**dio **D**etection **A**nd **R**anging)* is a system of navigation that uses reflected electromagnetic radiation to determine the velocity and location of a targeted object.

Random is synonymous with stochastic. It means there is, statistically, the same probability of occurrence throughout some distribution of sample variables (e.g., temporally, spatially, or both).

Reference sediment is a field-collected sediment, thought to be relatively free of contaminants (i.e., “clean sediment”). It is often collected from a site within the general vicinity of a test sediment (i.e., same body of water), and is frequently selected for biological testing because of

its geochemical similarity (e.g., particle size, total organic content) to the test sediment(s). A reference sediment may be used as an experimental control in addition to a control sediment in a sediment toxicity test.

Reference toxicant is a chemical (reagent grade) used to measure the sensitivity of the toxicity test organisms to establish confidence in the toxicity data obtained for a test substance or material. In most instances, a toxicity test with a reference toxicant is performed to assess the sensitivity of the organisms at the time the test substance or material is evaluated, and to determine the precision of results obtained by the laboratory for that chemical.

Replicate samples of sediment are collected from a sampling station to provide an estimate of the sampling error or to improve the precision of estimation. A single sediment sample (i.e., one core or grab sample) from a sampling station is treated as one replicate. Additional grab or core samples are considered to be additional replicate samples when they are treated identically but stored in separate sample containers (i.e., not composited).

Rinsates are water or solvent samples processed through the sediment or pore water collection device(s) in the field just before sampling, to assess equipment contamination and demonstrate the efficacy of washing procedures.

Riverine is an adjective used to describe a river or other lotic environment.

Sample(s) is a representative part of a larger whole that is studied to gain information about the characteristics and infer properties of the whole (e.g., sediment, pore water). It also refers to a subset of a population (e.g., benthos).

Sample container is a container into which a field-collected sample is placed directly from the sampler.

Sample size is the actual volume (L or m^3), weight (g), or dimensions (e.g., diameter and length of the core, I.D. = 2.5 cm, 1.2 m long) of sample of sediment.

Sample volume is the volume (m^3, L) of a sample.

Sampler refers to the device used to collect samples or subsamples.

Sampling is the act of collecting samples.

Sampling depth is the depth of sediment from which the sample is collected. It is generally less than the depth of sediment to which the sampler penetrates in order to collect the sample.

Sampling station(s) is the location where samples are collected within the study site.

*SATNAV (**SA**Tellite **NAV**igation)* is a relatively low-cost system of navigation that provides worldwide, but discontinuous coverage. The system relies on the integration of satellite fixes and other data sources to provide a position.

Sediment is a natural particulate material that has been transported and deposited, and is normally found below the water level. The term can also describe a substrate that has been experimentally prepared and within which test organisms can successfully burrow, survive, grow, and reproduce.

Sextant is a double-reflecting optical instrument designed to determine latitude and longitude by measuring angular distance usually, relative to the ocean horizon.

Sieved sediment is a sediment that has been sieved through a screen using pressure or water previously adjusted to the desired air saturation, temperature, and salinity (if marine or estuarine test). The sieved sediment and dilution water mixture should be allowed to settle for a minimum of 12 h (i.e., overnight) before the dilution water is decanted and discarded.

*Sonar (**SO**und **N**avigation **A**nd **R**anging*) is a system that uses underwater acoustic reflection/transmission energy for communication or to determine the location of objects.

Spiked control sediment is a control sediment to which a specific amount of reference toxicant has been added to achieve a specific concentration of reference toxicant in the sediment. This sediment serves as a positive control used to ensure that test organisms respond in a consistent manner over time to a specific concentration of a reference toxicant in sediment.

Spiked sediment is any sediment to which a test substance or material such as a chemical, a mixture of chemicals, drilling mud, contaminated dredge spoil, or sludge has been added, and mixed thoroughly, for experimental purposes.

Spiking refers to the addition of a known amount of test substance or material to a sample of sediment. After the addition of the chemical, which might involve a solvent carrier, the sediment is mixed thoroughly to evenly distribute the test chemical throughout the sediment.

Split sample is a sample that has been partitioned into two equal, or unequal, parts with, or without, prior homogenization of the sample with the intention of producing representative subsamples.

Stochastic is used to describe a sampling strategy with no specific patter to the location of sampling stations, because each station has an equal probability of being selected. It is synonymous with random and the opposite of systematic.

Storage container is a container that is used to store field-collected samples. It may or may not be the sample container.

Study area refers to the study site and surrounding area (i.e., that which might influence the study site) that are to be monitored or assessed.

Study site is the body of water and its associated sediments to be monitored and/or assessed.

Subsample is a representative part of a sample that is studied in order to gain information about characteristics and infer properties of the sample.

Subsampling refers to the act of collecting subsamples.

Substance is a particular kind of material having more or less uniform properties.

Taut wire is synonymous with distance line.

Test sediment is a field-collected sample of sediment, taken from a site though to be contaminated with one or more chemicals, and intended for use in laboratory testing. In some instances, the term may also apply to any sediment sample (including control and reference sediment) used in the test; however, this practice is strongly discouraged.

Theodilite is a telescope used in surveying that accurately measures horizontal and vertical angles.

Topography refers to the vertical, as well as horizontal positions of surface features (e.g., relief).

Toxicity is the inherent potential or capacity of a substance or material to cause adverse effects toward the exposed organism.

Toxicity test is an experiment designed to determine the effect of a material or substance on a population of a given test species (e.g., *Rhepoxynius abronius*) under defined conditions. A toxicity test usually measures either (a) the proportion of organisms affected (quantal) or (b) the degree of effect shown (graded or quantitative), after exposure to a specific test substance (e.g., a sample of sediment, pore water, or elutriate).

Transport container is a container that is used to transport field-collected samples. It may or may not be the storage or sample container.

Travel blank refers to a randomly selected sample container (e.g., bottle, tube, or tube liner) that has been treated and handled identically to those contains to which a sample was added. The empty container is filled with clean water (or sediment) and submitted with the field-collected samples for which it serves as a travel blank for either the chemical or toxicological analyses. The purpose of a travel blank is to assess any variation (or effects) that may be attributed to the actual transportation of the samples to the laboratory.

Wet sieving refers to a procedure of washing sediment particles through a sieve, using a particular mesh size, and a small volume of water.

Whole sediment or solid-phase sediment is the whole, intact sediment that has had minimal manipulation following collection or formulation. It is not a form or derivative of the sediment such as an elutriate or a resuspended sediment.

Zone is an area with relatively consistent characteristics such that it differs substantially from the characteristics of adjacent zones.

Zones of deposition are areas where fine-grained sediments (i.e., silt and clay particles with grain sizes ≤ 63 μm) accumulate, or are deposited continuously, such that the resulting deposits are comparatively loose (e.g., unconsolidated), and characterized by a relatively high water and organic content.

Acknowledgements

This report was written by Gladys L. Stephenson (Ecological Services for Planning Ltd., Guelph, Ontario). It is based on existing guidance documents and reports that describe methods and technologies for similar endeavours in the Atlantic, Pacific-Yukon and Arctic regions of Canada, the United States, and Australia, as well as the most recent scientific data reported in recent publications and provided by members of the scientific community who are actively involved in research relevant to this report. L. Porebski and R. Scroggins (Environmental Protection Service, Environment Canada) acted as Scientific Authorities and provided technical advice and guidance throughout this endeavour. The contributions of P. Mudroch (EC, Ottawa), A. Mudroch (EC, Burlington), K.E. Day (EC, Burlington), and J. Osborne (EC, Hull), as members of the *ad-hoc* Technical Sub-committee, are also greatly appreciated.

Members of the Inter-Governmental Aquatic Toxicity Group (IGATG - Appendix A), Ocean Disposal RODAC Committee, the Laboratory Manager's Committee (LMC) of Environment Canada, and the representatives from the regional offices (Appendix B), who actively participated in the review of the initial and final draft, are gratefully acknowledged. Particularly useful guidance and technical expertise were provided by:

D. Gauthier and C. Gobeil (Department of Fisheries and Oceans, Mont-Joli, PQ),
D. Loring (Department of Fisheries and Oceans, Dartmouth, NS),
C. Duerden and K. Tay (Atlantic Region, EC, Dartmouth, NS),
D. Goyette, D. Brothers, and S. Yee (Pacific & Yukon Region, EC, West Vancouver, BC),
A. Chevrier (Environment Canada, Hull)

Members of the Technical Sub-committee who actively participated in the development and review of this report are thanked accordingly for their comments and suggestions. Special acknowledgment is made of the recommendations, many technical contributions, and information provided by:

G. Ankley (USEPA, Duluth, MN),
G. Atkinson (Applied Statistics Division, EC, Hull),
S. Bay (Southern California Coastal, Long Beach, CA),
G.A. Burton (Wright State University, Dayton, OH),
R. Burgess (USEPA, Narragansett, RI),
G. Brun (Environmental Quality Laboratories, Moncton, NB),
A. Chevrier (Environmental Protection Service, Environment Canada, Ottawa, ON),
J. Ciborowki (University of Windsor, ON),
K. Doe (Environmental Conservation Service, Atlantic Region, EC, Dartmouth, NS),
B. Eadie (NOAA, Great Lakes Environmental Research Laboratory, Ann Arbor, MI),
J. Gearing (Department of Fisheries and Oceans, Mont-Joli, PQ),
J. Giesy (Michigan State University, East Lansing, MI),
R. Hesselin (Department of Fisheries and Oceans, Winnipeg, MB),
C. Hickey (NIWA Ecosystems, Hamilton, New Zealand),
K. Ho (USEPA, Narragansett, RI)

R. Hoke (SAIC, Hakensack, NJ),
P. Landrum (NOAA, Great Lakes Environmental Laboratory, Ann Arbor, MI),
C. Ingersoll and M. Nelson (U.S. National Biological Survey, Columbia, MO),
K. Keenleyside (Evaluation and Interpretation Branch, EC, Hull),
G. Krantzberg (Water Resources Branch, OMOEE, Toronto, ON),
J. Lazorchak (USEPA, Cincinnati, OH),
K. Liber (University of Wisconsin, Superior, WI),
S. Lorenzato (State Water Resources Control Board, Sacramento, CA),
B. McGee (University of Maryland, Queenstown, MA),
D. McLeay (McLeay Environmental Ltd., West Vancouver, BC),
D. Pascoe (University of Wales, Cardiff, Wales),
W. Peltier (USEPA, Athens, GA),
J. Percival (Natural Resources Canada, Ottawa, ON),
F. Philbert (Inland Waters Directorate, Canada Centre for Inland Waters, Burlington, ONT),
E. Power (EVS Consultants, North Vancouver, BC),
M. Redmond, J. Jones, D. Schultz, and M. Vance (AScI Corporation, Newport, OR),
L. Rosman (U.S. Army Corp. of Engineers, New York, NY),
J. Rodgers (University of Mississippi, University, Mississippi),
D. Rossel (Polytechnical School of Lausanne, Orbe, Switzerland),
J. Skei (Norwegian Institute of Water Research, Oslo, Norway),
P. Sly (Rawson Academy of Aquatic Science, Ottawa, Ontario),
B. Suedel (EA Engineering, Science and Technology, Hunt Valley, MD),
D. Sutherland (B.C. Environment, Prince George, BC),
K. Taberski (California Regional Water Quality Control Board, Oakland, CA),
A. Tessier (INRS-Eau/University of Quebec, Sainte-Foy, QUE),
R. Vezeau (Environment Canada, Montreal, QUE),
P. Winger (U.S. National Biological Survey, Athens, GA),
G. Winters and I. Hardy (Natural Resources Canada/GSC, Dartmouth, NS).

Funding and technical guidance that enabled the development of this guidance document were provided by the Marine Environment Division of the Office of Waste Management, Environment Canada, Hull, through L. Porebski and J. Osborne, and the Technology Development Directorate, Environmental Technology Centre, Environment Canada, Gloucester, through Rick Scroggins.

Introduction

1.1 Background

Sediments are an integral component of aquatic ecosystems. They originate from the differential settling of both suspended terrigenous particles that have been introduced into aquatic ecosystems and precipitates that have resulted from chemical and biological processes within aquatic systems. Suspended particles entering the aquatic system may already contain contaminants. Alternately, non-contaminated particles suspended in water may accumulate soluble contaminants present in the waters of aquatic systems. Precipitation processes are also capable of scavenging contaminants. As a result, sediments are often viewed as either a reservoir (e.g., source) or a sink for contaminants in aquatic systems.

Contaminants from agricultural, municipal, and industrial sources have been accumulating over the years in sediments of rivers, lakes, estuaries, and oceans. In some areas, concentrations of contaminants have reached levels that are detrimental to benthic and epibenthic communities. The extent of the problem of contaminated sediments in Canada is generally unknown; however, the concerns of scientists and regulators have culminated in a number of programs designed to ultimately protect the integrity and health of aquatic ecosystems. These programs fall into three general categories which include monitoring and assessment, disposal of soil/sediment into open water, and remediation. Germane to all of these studies are the methods and procedures for the collection of pore water and sediment for physiocochemical characterization, bioaccumulation tests, and/or toxicity evaluation. Since the selection of sampling stations and methods of sample collection, storage, transport, and manipulation can potentially influence both the characterization of the chemical and physical properties and the toxicity or bioaccumulation tests, it was considered prudent to provide guidance for these procedures and methods, to complement the existing biological and analytical test methods and standards (EC, 1990, a,b,c; EC, 1992 a,b,c,d,e,f,g; EC, 1996, a,b). Guidance will facilitate the use of consistent, appropriate, and comprehensive procedures for obtaining data on the bioaccumulation and toxic effects of contaminants in sediments.

1.2 Significance and Use of Guidance Document

The need for guidance on the methods for selecting sampling stations and the collection, transport, handling, storage, and manipulation of sediment samples has resulted from the knowledge that these variables can affect the results of the physiocochemical characterization of the sediment, bioaccumulation tests, and toxicity tests, or influence the interpretation of test results. The lack of standard guidance has resulted in the use of a myriad of methods and procedures that make it very difficult to understand and compare the results from different studies. Standard guidance for collection, storage, characterization, and manipulation of sediments was first provided by ASTM (1992a). The information in this document served as a foundation for the guidance contained herein. An attempt has

been made to harmonize the guidance in this document with the guidance provided by ASTM in the revised guide (ASTM, 1995a).

Although methods for toxicity assessment of sediments are in their infancies, development is progressing at a phenomenal rate. In recent years a number of methods and protocols for sediment toxicity testing have emerged (EC, 1992 at to g; 1996 a,b; USEPA, 1994 a,b; ASTM, 1995), as well as guidance on the collection , storage, and manipulation of sediments (ASTM, 1995). These documents reflect the current "state-of-the-science" and are subject to revision. As more information becomes available, the recommendations contained herein will be re-evaluated and, where necessary, revised. The toxicity of contaminated sediments is a complex subject influenced by physical, geochemical, and biological factors and, despite the concentrated efforts of many scientists, data gaps and unanswered questions remain.

This guidance manual has been divided into sections to reflect two different approaches to assessing or evaluating contaminated sediments: Procedures for Monitoring and Assessment Studies; and Procedures associated with the Open-water Disposal of Dredged Materials.

The recommended options, methods, or procedures for monitoring and assessment and open-water disposal studies are presented in Sections 2 and 3, respectively. The sections differ with respect to the level of detail presented. The procedures recommended in one section may differ from those in another section to accommodate different goals and objectives. Users of this guidance must decide what approach best fits their particular project and consult the appropriate sections. The recommended options or procedures are presented in bullet form, with bold text, at the beginning of each subsection, and these recommendations are also summarized at the end of each section for quick referencing. An overview of the application of the guidance is presented in Figure 1.

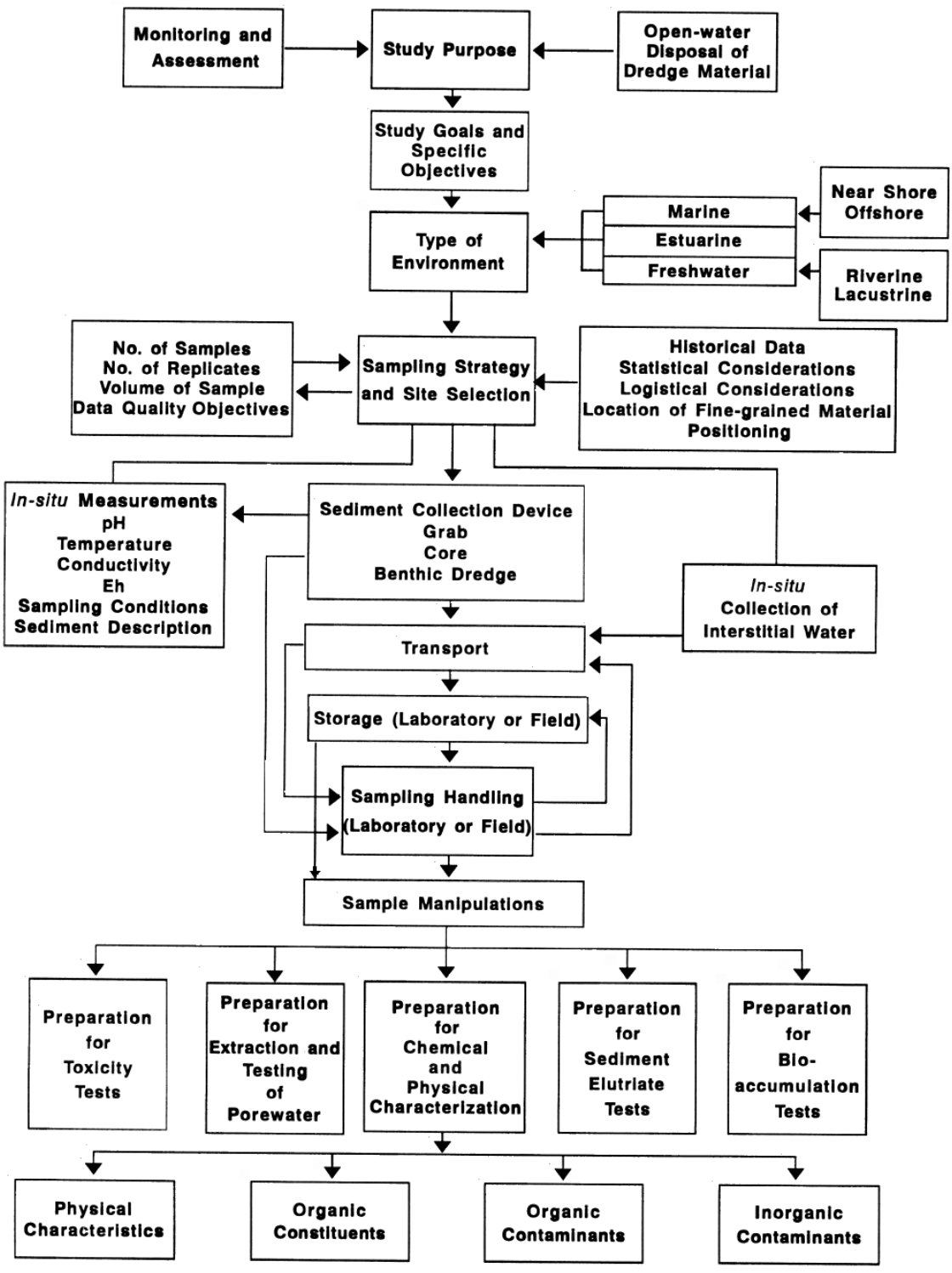


Figure 1 Flowchart Describing the Guidance Provided in this Document

Procedures for Monitoring and Assessment Studies

2.1 Overview

In the early 1960s, sediment contamination was identified as a major concern in many Canadian aquatic environments. In the 1970s, it became apparent that some contaminants in sediments were readily bioavailable and could re-enter aquatic food-webs. It also became apparent that the contamination of sediments from point and non-point sources such as stormwater discharges, industrial effluents, accidental or deliberate spills, the disposal of dredged material and sewage sludges, agricultural runoff, urban drainage, atmospheric deposition, and resuspension of sediments was relatively widespread. As a result, programs were developed to monitor and assess the nature, magnitude, and extent of contamination of sediments in freshwater, estuarine, and marine environments.

The following sections and subsections provide guidance on methods for selection of sampling stations, collection of sediment and pore water, handling, transport, and storage methods and conditions for whole-sediment and water samples, and methods for the preparation of test samples for the physical and geochemical characterization and toxicity assessment specific to monitoring and/or assessment studies. These recommendations are contingent upon the prevailing scientific information.

2.2 Study Purpose and Objective(s)

Recommendation

- Clearly state the purpose, objective(s) and/or hypothesis of the study, and integrate them as an integral component of the study plan.

Monitoring and assessment studies cover a broad spectrum of activities and are by their very nature, variable. Most studies are designed to monitor and assess sediments in terms of:

a) historical contamination;

b) the impact of point-source pollution;

c) geochemical surveys for oil, gas, or mining exploration;

d) the impact of construction or development (e.g., bridges, dams, wharfs, docks, etc.); or

e) habitat suitability (i.e., to support biological communities).

Monitoring and assessment studies are also used to determine the extent to which sediments act as either sources or sinks for contaminants and to determine the presence of temporal and/or spatial distributions of selected contaminants in sediments. These studies can have either regulatory implications (e.g., dredging, disposal, or remediation) or research implications (e.g., geochemistry, biological and physicochemical processes, or validation of transport and deposition models). Ultimately, the data must be used to assess risk to human health and/or the environment from the accumulation or redistribution of contaminants in sediments.

The most important aspect of monitoring and/or assessment study is the study plan, which consists of the goals and objectives of the study, the methods and strategies for data and sample collection, and the procedures required to ensure that all data-quality objectives (DQO) are satisfied.

2.3 Study Plan

Recommended Procedures

- Define the study area and the study site, and outline them on a recent hydrographic chart or topographic map. A physical inspection of the study area and proposed study site should be undertaken.

- Identify potential sources of contamination and plot their locations on the chart or map.

- Consult local expert(s) on site conditions.

- Access, review, and evaluate all available historical data relevant to the study.

- Determine the location of fine-grained sediments that are usually associated with low energy zones in water.

- Select a method for determining the location of the sampling stations.

- Decide on a positioning method that is most appropriate to the site and study.

- Determine the sample size (e.g., weight or volume) required to satisfy the analytical methods and QA/QC program for all of the intended analytical tests.

- Decide on the level of confidence and the acceptable size of effect required from the sample data.

- Determine the frequency of sampling (i.e., how often or when samples should be collected) that is required to meet the study goals and objectives. If there is a seasonal component to the study, it should be included in the study plan. Consideration of sedimentation rates is critical when determining sampling frequency.

- Determine the number of samples required to achieve the field sampling objectives and the QA/QC program.

- If replicate samples from each sampling station are required, a minimum of five replicate samples per sampling station is recommended. However, the number of replicate samples may be determined *a priori* from preliminary sample collection and analyses.

- The study plan, including the sampling design (i.e., the frequency, number, and location of field-collected samples), should be discussed with a statistician or other qualified professional, before the monitoring study begins.

- Disposal of wastes from the study should be addressed in the study plan.

2.3.1 Definition of the Study Area and Study Site

The study area refers to the body of water that contains the study site to be monitored and/or assessed, as well as adjacent areas (e.g., land or water) that might affect or influence the conditions of the study site. The study site refers to the body of water and associated sediments to be monitored and/or assessed. The boundaries of both of these areas should be clearly defined and outlined on a chart or map of the study area.

All known and potential sources of contamination should be identified and their locations also accurately denoted.

2.3.2 Historical Data and Identification of Potential Sources and Present Conditions

After clearly stating the problem, objective(s), or hypothesis(es) of the study, and defining the study area and study site, the next step is to select the location of sampling stations by initially accessing, reviewing, and evaluating any available historical information relevant to the study. Contaminants in sediments could reflect continuous or episodic (e.g., accidental spills) inputs; hence, historical data from newspaper files, government agencies, municipal archives, hydrographic surveys, harbour commission records, past geochemical surveys, and bathymetric maps provide important information for developing a sampling program. Local experts on site conditions should be consulted. Potential sources of contamination must be identified and their locations placed on a map or chart of the proposed study area. Reports or records with information on the nature of the contaminants or degree of contamination in the study area should be considered, where possible, when selecting the location of sampling stations. A comprehensive review of the types of historical and practical considerations for locating sampling stations is provided by Mudroch and MacKnight (1991). A summary of the historical and practical considerations for sampling sediments has been compiled in Table 1.

A physical inspection of the site is strongly recommended for all study plans, in order to assess the completeness and the validity of the collected historical data, and to identify any significant changes that might have occurred at the site or in the study area (Murdoch and MacKnight, 1991).

2.3.3 Determination of Deposition Zones

Recommendations

- There is no one method for locating fine-grained sediments that is applicable to every site. Therefore, a process that combines the available historical, bathymetric, and hydrographic data with the technology most appropriate for the site is recommended to locate the probable zones of deposition. These zones should also be validated by either site inspection (e.g., diver or electronic surveillance) or the collection of preliminary, surficial-sediment samples.

For monitoring and assessment studies, the location of fine-grained sediments is often a priority. Fine-grained sediments are generally located in zones of deposition, have higher organic carbon content than other sediment particle size fractions, and are usually associated with higher levels of contaminants than other particle size fractions (e.g., sand and gravel) (IJC, 1988; Persaud *et al.,* 1988; Baudo *et al.,* 1990; Suedel and Rogers, 1991; Power and Chapman, 1992; Holland *et al.,* 1993). Information on bottom topography, water depth, particle size distribution, and zones of erosion, transportation, and accumulation or deposition at the study site are instrumental in identifying areas with fine-grained sediments. Samples collected from depositional zones generally provide a "worst-case" scenario in terms of sediment contamination or toxicity.

There are various non-disruptive technologies available to assist in the location of fine-grained sediments (van

Table 1 Historical and Practical Considerations for Selection of Sampling Stations in a Monitoring and Assessment Study

Hydrologic Information

- quality and quantity of runoff
- potential erosional inputs of total suspended solids
- up-wellings
- seepage patterns

Bathymetric maps and hydrographic charts

- water depth
- zones of erosion, transport, and deposition
- bottom topography
- distribution, thickness, and type of sediment
- velocity and direction of currents
- sedimentation rates

Anthropogenic considerations

- location of urban centres
- historical changes in land uses
- types, densities, and sizes of industries
- location of waste disposal sites
- location of sewage treatment facilities
- location, quantity, and quality of effluents
- urban drainage (e.g., storm water runoff)
- previous monitoring and assessment or geochemical surveys
- location of dredging and open-water disposal sites
- location of historical spills of substances or materials

Geochemical considerations

- type of bedrock and soil/sediment chemistry
- physical and chemical properties of water

Climatic conditions

- prevailing winds
- seasonal changes in temperature, precipitation, solar radiation, etc.
- tides, seiches
- seasonal changes in anthropogenic and natural loadings

Woerden *et al.,* 1988; Schock *et al.,* 1989; Guigné *et al.,*1991). These techniques are not universally suitable to all aquatic environments. For example, acoustic survey techniques (e.g., echo sounding, seismic reflections etc.), can be used to characterize the type (e.g., sand gravel, silt, or clay) of surficial and subsurface sediment (Mudroch and MacKnight, 1991); however, these techniques cannot be used effectively in shallow waters.

A side-scan sonar used with a sub-bottom profiler provides a permanent, continuous graphic or digital record of the surface topography and the sub-bottom layers. Contaminated sediment in depositional zones resulting from on-shore industrial discharges have been successfully mapped using these methods, and rock, sand, and fine-grained substrates can be differentiated. Although high-resolution, side-scan sonars have been used with success in some studies (Wright *et al.,*1987; Mudroch, 1992), they have also failed to impart the degree of resolution desired in other studies (Giesy, 1992). The limitations of the technology to date, apart from the high cost of the equipment, are that gas bubbles in water or ebulating from bottom sediments interfere with the sub-bottom profiling which is also sensitive to both the tilt of, and density variations within, the sediment layers. The success of an acoustic survey also depends, to a large extent, on the level of technical expertise available for data interpretation.

Aerial reconnaissance with, or without, satellite imagery can also assist in visually identifying depositional zones where clear water conditions exist. They are not reliable, however, if waters are turbid. Grab sampling, inspection by divers, or photography using an underwater television camera or remotely controlled underwater vehicle (ROV) are other methods that can also be used to locate fine-grained sediments (Nishimura, 1984; Lawless and Padan, 1986; Schneider *et al.,* 1987; Burton, 1992).

2.3.4 Selection of Sampling Stations

The selection of sampling stations in any monitoring or assessment study depends on the problem that has been identified and the objective(s) to be achieved. The problem must be clearly defined, as in any study plan, and objective(s) or hypothesis(es) clearly stated. All participants in the program should understand the significance of achieving the objective(s).

Recommended Options

- The use of historical data, when available, should be an integral component of the sampling station selection process.

- A physical inspection of the study area and proposed site must be undertaken.

- If the objective of the survey is to identify sites of toxic and/or contaminated sediments on a quantitative spatial and/or temporal basis, a systematic or regular grid-sampling is the most appropriate sampling plan (Green, 1979; Atkinson, 1985).

- If the monitoring objective is to determine sediment contamination originating from a point source, the sampling pattern could be based on the assumption that concentrations decrease with distance from the source, thus, factors affecting dispersion of substances or materials from the point source (e.g., currents) must be considered. Therefore, in a river where the point source is an outfall, sampling stations should be located in zones of accumulation at fixed distances

downstream, following a geometric progression (i.e., ×, 2×, 4×, 8×, etc.) (Mudroch and MacKnight, 1991). Samples are usually collected concurrently from upstream locations that serve as reference or control sites.

- If the point source is within a waterbody where dispersion is not unidirectional, the sampling stations should be located in a concentric pattern at the intersection of fixed geometric distances and a specified angle or bearing.

- Stratified random sampling should be used where historical, sediment-mapping data are available and there are well-defined zones of different sediment types (Thomas *et al.,* 1976; Cahill, 1981; Håkanson and Jansson, 1983; Burton, 1992).

- A positioning method with the appropriate degree of resolution should be used to identify the location of the sampling stations.

- Each sampling station in the study site should be preassigned a name or number to identify the sampling location.

The selection of sampling stations is probably one of the most critical steps in monitoring and assessment studies. Unfortunately, too little time is usually devoted to sample-station selection and, too often, statistical power is sacrificed to reduce sampling effort in the expectation of reducing costs. A well-prepared sampling scheme makes data analyses and interpretation of laboratory results easier and more meaningful. The rationale for the selection of sampling stations is seldom reported, but statements regarding such rationale should be an integral component of any data report.

A number of methods, summarized in Figure 2, can be used to select the location of sampling stations within a study site. Most statistical methods are based on a systematic grid and can be modified to accommodate equal-area grids that fall within the aquatic habitat. The method of choice depends, to a large extent, on the objective(s) of the study, the size of the study site, the desired degree of statistical resolution, and the available resources. No one method can be recommended for all monitoring and assessment studies. Each sampling station within the study site should be preassigned a name or number to identify the sampling location. This will facilitate the recording of field notes, labelling, and tracking of samples.

2.3.5 Methods for Positioning of Sampling Stations

Recommended Options and Precautions

- Regular calibration of the positioning system by at least two methods is required to ensure accuracy.

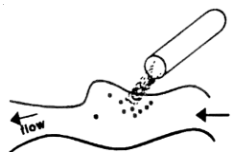
- Trained and experienced personnel should be responsible for positioning.

- For monitoring and assessment studies of large areas (e.g., Great Lakes and offshore marine environments) where an accuracy of ±100 m is sufficient, the LORAN or GPS system is recommended. Differential GPS is recommended for a high accuracy in positioning.

- For near-shore areas of marine environments and large freshwater rivers, where visible or suitable and permanent targets are available, RADAR is recommended for a required accuracy of 10 to 100 m and a Trisponder system is

Method Selection Criteria		Reference
NON-STATISTICAL:		
• Haphazard	selected sampling stations are the easiest to sample or have the easiest access	Mudroch and Macknight, 1991
• Deterministic or Judgemental	sampling stations are those most likely to contain contaminated sediments (i.e., depositional areas with fine-grained sediments)	Wright *et al*., 1987; Nishimura, 1984 Schneider *et al*., 1987; Baudo *et al.*, 1990

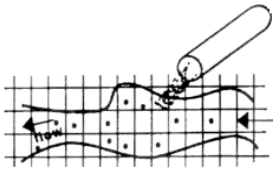
e.g., Riverine



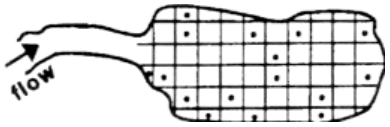
e.g., Lacustrine

STATISTICAL: 1. Stochastic Random	study site is divided into numbered blocks or triangles and random numbers (tables or electronically generated) are used to select the sampling stations according to the number of samples required. The location usually is the centre or at the intersection of the grid	Burton, 1992 Keith, 1992

e.g., Riverine

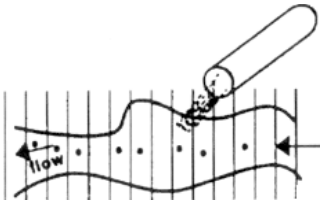


e.g., Lacustrine



Stratified Random	study site is divided into a number of blocks according to similar substrates or the bathemetry of the site, and the blocks are divided into subunits that are then randomly selected for sampling	Keith, 1992

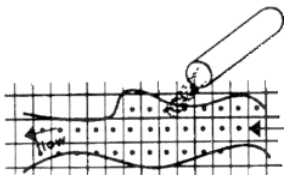
e.g., Riverine



e.g., Lacustrine

2. Systematic Regular	study site is divided into a number of blocks and each unit is sampled at regular intervals so that the distance between sampling stations is constant	Keith, 1992

e.g., Riverine



e.g., Lacustrine

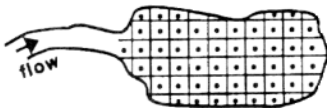


Figure 2 Sampling Strategies for Monitoring and Assessment of Contaminated Sediments (• the location of a sampling station)

recommended for a required accuracy of 1 to 10 m.

- For small areas where the sampling stations are numerous and located relatively close together, a high accuracy in positioning is required and either the Miniranger, Trisponder, or Differential GPS is recommended.

- For small rivers, lakes or ponds, and urban water fronts, visual angular measurements (e.g., sextant) by an experienced operator should provide accurate and precise positioning. Alternatively, a distance line or taut wire can be used.

The most important function of positioning technology is to define the location of the sampling station (e.g., latitude and longitude), so that the user can return to the same position. There are a variety of navigation and/or position-fixing systems available. The criteria for selecting a positioning system primarily depends on the purpose and objective(s) of the study, the physical conditions, topography, and size of the study area, equipment availability, the distance between sampling stations, site accessibility, station reoccupation, the desired degree of precision and accuracy, and financial and technical constraints (USEPA, 1987). Normally, monitoring surveys over large study areas do not require the highly accurate or precise positioning required by smaller site-specific surveys where stations are monitored repeatedly. Each of the available methods has an associated absolute and relative accuracy, and availability might be simply a function of geographical location. For example, LORAN-C is not available in all geographic locations in Canada.

The characteristics, advantages, and disadvantages of the various positioning methods (e.g., line-of-sight, optical, or electronic) are discussed in Mudroch and MacKnight (1991), and summarized with additional information (EC, 1985; Tetra Tech, 1986; USEPA, 1987) in Table 2. The appropriate positioning technique for any study plan should be planned in advance. When selecting a new system, it is recommended that assistance be solicited from the appropriate government agencies, from a local surveying company, and/or local marina. Often the different systems that are available are supported locally by either a government or industrial organization (e.g., acoustic transponders may be installed for positioning) and the use of these systems may reduce the total cost of positioning. Regardless of the type of system selected, calibration of the system is recommended by using at least two of these methods to ensure accuracy. The positioning equipment must be properly set up and calibrated, and operated by trained and experienced personnel familiar with the standard operating procedures of both the primary and the backup method. At each sampling station, a fathometer or meter wheel can be used to determine the sampling depth. This will ensure that the water is the desired depth and the bottom is sufficiently horizontal for proper operation of the sampling equipment.

2.3.6 Sample Size, Number of Samples, and Replicate Samples

The volume or weight of sediment per sample (e.g., sample size), the number of stations to be sampled at a study site and the number of replicate samples per station will be specific to each study. This will involve, in most cases, a compromise between logistical and practical constraints (e.g., time

Table 2 Methods for Positioning Used to Locate Sampling Stations (adapted from EC, 1985; Tetra Tech, 1986; USEPA, 1987)

Category	Accuracy	Range	Advantages	Disadvantages
Optical or Line-of-Methods				
Theodolite	10 to 30 s ± 1 m and up	200 m to 5 km	• traditional method that measures horizontal angles between known targets; • inexpensive; • high accuracy; successfully applied	• triangulation between two manned shore stations or targets is required; • simultaneous measurements are required; limits on intersection angles; area coverage is limited because it requires good visibility; sampling platform must be stationary
EDMI	1.5 to 3.0 cm	3 km without multiple prisms	• extremely accurate; usable for other surveying projects; • relatively inexpensive; compact; portable; rugged	• motion and directionality of reflectors; • line-of-sight good visibility is necessary unless microwave unit available; two shore stations are required; ground wave reflection causes errors
Total stations	5 to 7 cm	< 5 km	• single onshore station; other uses; minimum logistics	• reflector movement and directionality; prism costs; line of sight; optical or infrared range limitations
Sextant*	± 10 s ± 3 to 5 m but variable	200 m to 5 km	• portable, handheld device which can be highly accurate with experienced operator; rapid; easy to implement; common equipment; low cost; no shore party is necessary; high accuracy when used close to shore	• triangulation requires two targets; good target visibility required; orientation of target affects accuracy; simultaneous measurement of two angles are required; good target visibility is required; location and maintenance of targets is required for relocation of station; line-of-sight method; best in calm conditions; limits on acceptable angles
Pelorus	variable	< 5 km	• rapid; easy to implement; common equipment; low cost; no shore party necessary; high accuracy when used close to shore	• simultaneous measurements of two angles and good target visibility are required; location and maintenance of targets is required for relocation of station; line-of- sight method; best in calm conditions; limits on acceptable angles
RADAR	variable	30 to 50 km	• standard equipment on ships; easy to operate; yields range and relative bearing to targets	• not portable; requires a suitable target (i.e., one that reflects microwave signals)
Autotape	± 0.5 m	limited	• highly accurate, very precise; portable	• very expensive (e.g., > $50 000)

Table 2 Methods for Positioning Used to Locate Sampling Stations (adapted from EC, 1985; Tetra Tech, 1986; USEPA, 1987) **(Cont'd)**

Category	Accuracy	Range	Advantages	Disadvantages
Electronic Positioning Systems				
Microwave navigation systems (e.g., Miniranger, Trisponder, Rascal Microfi, Del Norte)	± 1 to 3 m	25 to 80 km (depends on height of transceiver units)	• no visibility restrictions; multiple users; highly accurate; radio line of sight; portable; easy to operate	• moderately expensive; multiple onshore stations required; logistics and security of the necessary shore units increases cost; signal reflective nulls are a potential source of error; limited range because of low-powered shore units
Shoran	± 10 m	< 80 km (short range)	• highly accurate	• limited in range; requires two shore transmitters
LORAN-C	± 15 m and up	up to 2800 km (long range)	• no visibility or range restrictions, no additional personnel; low cost; existing equipment	• interference in some areas; used only for repositioning, except in limited areas; need to locate station initially with another system; coverage in Canadian arctic is rare
Decca HIFIX/6	± 1 m	up to 300 km (medium range)	• high accuracy and precision	• expensive; multiple shore stations are required
Variable range	± 0.5˚	16 to 72 km	• no visibility restrictions; no additional personnel; low cost; existing equipment	• line-of-sight method; relies on map accuracies of targets; accuracy decreases with range scale
Decca Minifix	± 2 m	up to 70 km	• high accuracy and precision; light weight equipment	• expensive
Range-azimuth	0.02˚ and 0.5 m	< 5 km (optical) 30 km (elec)	• high accuracy; single station; circular coverage	• single user; high cost; line-of-sight method; signal reflective nulls are a potential source of error
Satellite Positioning Systems				
SATNAV	1 to 10 m	no limit on the range	• high accuracy; single minimum logistics; can be used in restricted/congested areas; no shore stations are required; it integrates satellite fixes with other data sources	• continuous coverage is not provided initial development cost was high; local and atmospheric effects can cause error; distortion of signal paths over polar ice caps
GPS or Navstar	± 100 m (0.1 to 1 m for differential GPS)	no limit on the range	• continuous position reports available worldwide	• relatively new therefore cost is likely to decrease and its use is expected to increase greatly in the next few years; military scrambling can be a site-specific problem

* Accuracies greater than ± 20 m are not common farther than 1 km from shore under normal operating conditions

[1] expensive = > $50, 000; moderately expensive = $5 000 to $50 000; inexpensive = < $5 000

and cost) and statistical considerations. The total number of samples collected during a study is normally the product of the number of stations to be sampled times the number of replicate samples at each station.

Sample Size

Recommended Minimum Sample Size

- The recommended minimum volume or weight of sediment required for each end use is summarized in Table 3. Accordingly, the sample size should be determined on a case-by-case basis.

Before commencing a sampling program, the type and number of analyses and tests should be determined, and the required volume or weight of sediment per sample calculated. Each physicochemical and biological test requires a specific amount of sediment which, for chemical analyses, depends on the detection limits attainable by the procedure and, for biological testing, depends on the test organisms and test method. For example, the amount of sediment required in a bioaccumulation test depends on the amount of tissue required for the analyses which, in turn, dictates the number of animals required for each treatment in a test. The loading rate is generally species specific so the number of animals determines the amount of sediment required. Therefore, the testing laboratory responsible for the analysis of samples should be consulted to confirm the amount of sediment required for each sample. Generally, a volume of 1 L of whole sediment is sufficient to satisfy the sample-size requirements for physicochemical characterization and contaminant analyses. However, if sediments are to be analyzed for oil and grease (generally 50 g dry weight of sediment will satisfy the petroleum hydrocarbon analytical requirements) then an additional 1 L of sediment should be collected. The preparation of sediment elutriate requires at least 1 L of sediment. Although test-dependent, biological toxicity test methods require at least 1 to 3 L of whole sediment for each sample and a bioaccumulation test requires 3 L for each sample, assuming no replication. A bioaccumulation test with five replicates would require 15 L of sediment. Therefore, physicochemical characterization, contaminant analyses, and biological testing normally requires 5 to 7 L of whole sediment per sample. After the sample size is determined, it is important to compare the sample size required (Table 3) with the capacity of the sampler to deliver the desired amount of sediment (Table 5), and reassess the number of replicate samples per station (see Subsection 2.3.6) consider the QA/QC sampling program and whether or not samples are to be archived. If the core sampler cannot deliver the sample size required for biological toxicity testing, collect additional samples and composite the samples or subsamples (Garner *et al.,* 1988). Alternatively, replicate sediment samples can be incorporated into the test design.

The dimensions and/or volumes of the various sediment collection devices are indicated in Subsection 2.5.1. The volume or weight requirements might dictate further sample handling such as subsampling, compositing, or sample splitting.

Number of Samples.

Recommendations

- The number of samples collected is usually determined by: the size of the study site, the objective(s) of the study,

Table 3 Minimum Volume or Weight of Sediment Required for a Specific End Use

End Use	Volume (mL)	Weight[1] (g dry weight)	(g wet weight)
Physical/Chemical Analyses:			
Inorganic Contaminants	90	10	100
Organic Contaminants	230	50	250
Other Chemical Constituents (e.g., TOC, moisture content)	300	60	330
Particle Size	230	50	250
Petroleum Hydrocarbons[2]	50 to 1000	50 to 200	275 to 1100
Biological Testing			
Toxicity Tests[3]	1000 to 3000	200 to 600	1100 to 3300
Bioaccumulation Tests[4]	3000	600	3300
Porewater Extraction[5]	2000	NA	3200
Preparation of Elutriate	1000	200	1100

[1] Based on a specific gravity of 2.0, a moderate organic matter content, and a water content of 90%.

[2] The maximum volume (e.g., 1 L) is required only for oil and grease analysis; otherwise 250 mL is sufficient.

[3] Based on the average requirement for three tests.

[4] Based on an average of 3 L of sediment per sample; an additional 3 L is required for each replicate.

[5] Based on a specific gravity of 2.0, a moderate organic matter content, and a water content of 40%.

NA Not applicable.

the type and distribution of the contaminants being measured, the characteristics and homogeneity of the sediment, the concentrations of contaminants likely to be found in the sediments, the analytical requirements (e.g., sample volume or weight), and the desired level of statistical resolution. Accordingly, sample requirements should be determined on a case-by-case basis. In most monitoring and assessment studies, the number of samples to be collected usually results from a compromise between the ideal and the practical. The major practical constraints are the costs of analyses and logistics of sample collection.

There are a number of approaches that can be used to determine the number of samples required to achieve a minimum detectable difference at a specific confidence level and power (Cohen, 1977; Guenther, 1981; 1982; Friedman, 1982; Alldredge, 1987; Green, 1989; EC, 1991; 1995). The USEPA (1994) presents a concise discussion of the relationships of these statistical considerations. Traditionally, acceptable coefficients of variation vary from 10 to 35%, the power from 80 to 95%, the confidence level from 80 to 99%, the minimum detectable relative difference from 5 to 40% (Barth and Starks, 1985). Sediments can be both relatively homogeneous or very heterogeneous, depending on the criterion used to assess variability. This variability can be the result of the differential distribution of different chemicals in the sediment, which might be specific to each station. Therefore, an acceptable size of effect or expected difference must be predetermined.

To determine the number of samples required, the following questions should be answered (Alldredge, 1987):

1. What is the null hypothesis?

2. What are the alternative hypotheses? What is being compared?

3. Is the significance criterion directional (one-tailed test)?

4. What is the level of significance between the expected and actual value of the criterion being measured?

5. How large a difference is acceptable between the expected and actual value of the criterion being measured, and with what level of probability?

6. What variability is expected in the data?

Once these questions have been answered, then appropriate methods can be used to determine the required number of samples. These methods are described in most books on statistics (e.g., Steel and Torrie, 1980) and have been discussed in a number of reviews (Henriksen and Wright, 1977; Kratochvil and Taylor, 1981; Håkanson, 1984; EC, 1985; Alldredge, 1987; Lesht, 1988; Green, 1989; Baudo, 1990; EC, 1991; Keith, 1992). A summary of the statistical formulae generally used to determine an appropriate number of samples is presented in Appendix C. These formulae are a useful guide; however, under some circumstances site-specific information may preclude their use. The major limitation to this approach is that it either entails a preliminary survey or relies on the availability of historical data.

Replicate Samples

Recommended Minimum Number of Replicate Samples

- If replicate samples from a sampling station are required, the collection of a minimum of five replicate samples within a sampling station is recommended unless determined otherwise from preliminary sampling and analysis.

- The collection of replicate samples should be mandatory as part of the QA/QC requirements of any good sampling program and should comply with the DQO.

- The number of replicate samples should be higher at stations located close to a source of contamination (Skei, 1992).

Most monitoring and assessment studies have traditionally collected only one sample from each sampling station. However, a single sediment sample from a sampling station will impart little information on the variability in the sediment at the station. The objective of collecting separate replicate samples at each station is to allow for quantitative statistical comparison within and among different stations (Holland *et al.,* 1993). Separate subsamples from the same grab, core, or box-core sample might be used to measure the variation within a sample but not necessarily within the station. The collection of separate samples within a sampling station will impart valuable information on the spatial distribution of contaminants at the station and on the heterogeneity of the sediments within the site.

Traditionally, one to five sediment samples from each station have been collected to assess the heterogeneous nature of most sediments (Sly, 1978; Håkanson and Jansson, 1983; Hilton *et al.,*1986; Smith *et al.,* 1986; Bennet, 1987; Downing and Rath, 1988; Holland *et al.,* 1993). These samples are kept separate and considered replicates. The number of replicates required per station is a function of the need for sensitivity or statistical power. Typically, the smallest deviation from the null hypothesis that is considered scientifically or environmentally important to detect must be decided *a priori,* together with the power of the test that is desired for the specific alternative (Green, 1989). Statistical power depends on the standard deviation, the number of samples, size of effect or difference that is to be detected, the probability of false negatives (i.e., type 2 error - not rejecting the null hypothesis when it is false), and the probability of false positives (i.e., type 1 error - rejecting the null hypothesis when it is true).

The major costs associated with the collection of sediment samples are those for travel to the site and the processing of the samples (e.g., chemical and biological analyses). The actual costs of sampling "on-site" are minimal by comparison. Consequently, a number of monitoring studies investigating the physicochemical characteristics of sediments adopt the approach whereby either a number of replicate samples from each sampling station, or samples from other sampling stations, are collected in excess of the minimal number required for the desired end use, and a subset equal to the minimum is selected for analysis. The precision of the estimate of the variable of interest is then determined, and if the coefficient of variation associated with the mean estimate exceeds some predetermined criterion (e.g., 20% of the mean), then additional archived replicate samples can be analyzed until the criterion for precision is achieved. The archived replicate samples can also be used to replace lost or misprepared samples for the independent testing of *a posteriori* hypotheses that may arise from screening the initial data. This approach is not desirable for studies involving biological assessments of sediment.

Replicate samples collected from a sampling station have been kept separate and treated as true replicate samples or they have either been combined and homogenized, or homogenized, then subsampled and the subsamples combined, to generate a composite sample. A composite sample from a sampling station is treated as a single sample. Compositing of sediment samples within a habitat location might be desirable when the objective of the monitoring study includes relating invertebrate populations and tissue contaminant information (e.g., body residues) to the chemistry of the sediment in which they are likely to spend most of their

life span. Compositing samples or subsamples might provide a more ecologically relevant exposure scenario and reduce the variability, and therefore, the precision of relating sediment chemistry to biological indices and/or whole-body contaminant levels. Compositing replicate samples within a sanpling station is one way to ensure that the sample is representative of the sediments from that particular station and that the sample contains enough material to satisfy the requirements of the analyses (Garner *et al.,* 1988).

2.3.7 Preparations for Field Sampling

Preparations for the execution of a sampling program must include:

A. Preparation of a written protocol for logistical considerations including information on:

- accessing the study site (e.g., road access or air access only, type of vehicle to be used, shipping of equipment, type and size of vessel, route to the site, time to get to the site);
- qualified personnel for the safe operation of the vessel;
- methods of locating designated sampling stations and maintaining the position at each sampling station during sampling (e.g., positioning equipment operated by qualified personnel who are also responsible for the calibration and maintenance of the positioning equipment, anchor system);
- accessing the sampling stations (e.g., time to actually collect and process the samples at each sampling station, the time required to travel between sampling stations);
- adequate space on the vessel or sampling platform to accommodate collection of the samples, the recording of *in-situ* field measurements, or on field-collected samples;
- handling the retrieved sample, and temporary field storage of the collected samples;
- a communication system (and backup system) to monitor weather conditions and report position and progress to a land base;
- route and method of access to temporary field storage;
- emergency plans in case of an accident;
- time to adequately deal with unforeseeable circumstances should be considered when preparing the sampling schedule; and
- deposition of sampling plan and schedule with the appropriate authorities.

B. Preparation of separate checklists for the necessary equipment and/or reagents associated with:

- the collection of various types of samples (e.g., grab samplers and corers, core liners, core valves, ropes, caps, tool box, etc.);
- the handling of samples (e.g., spatulas, scoops, pans for homogenization, containers for samples, reagents for preservation);

- cleaning and repairing or maintaining sampling equipment;
- field measurements (e.g., pH, DO, temperature, and conductivity meters, waterproof pens, kimwipes, thermometer, optical refractometer);
- the storage of samples (e.g., ice, ice boxes, refrigerators, freezers);
- the transport of samples (e.g., shipping containers, insulation material to stabilize sample containers);
- the necessities for personnel accommodations (e.g., food, lodging, water);
- maps, navigation, and communication equipment for location of sampling stations and study site; and
- transportation to the study site (e.g., vehicle and/or vessel).

Each checklist should be the responsibility of an experienced field technician or assistant. The sampling plan and projected time schedule should be posted so that all personnel are aware of what is expected and when. The names, addresses, and telephone numbers of all participants involved with both the preparation and actual execution of the sampling program should be available to all participants, and the duties and responsibilities of each participant clearly documented. The study director or manager is ultimately responsible for all aspects of the study and should, therefore, ensure that the appropriate personnel clearly understand their role and are capable of carrying out their assigned responsibilities and duties. Contingency planning should address the need for backup personnel in the event of accident or illness. Sampling should always be conducted by two or more technicians. The reagents for cleaning, operating or calibrating equipment, collecting, preserving and/or processing samples should be handled by appropriately qualified personnel and the appropriate data for health and safety (e.g., Material Safety Data Sheets) should be available.

Written approved protocols and standard operating procedures (including QA/QC requirements) should be readily accessible at all times, to ensure proper and safe operation of equipment. Data forms and log books should be prepared in advance so that field notes and data can be quickly and efficiently recorded. Extra forms should be available in the event of a mishap or loss. These forms and books should be waterproof and tear resistant. Under certain circumstances audio or audio/video recordings might prove valuable.

All equipment used to collect and handle samples must be cleaned and all parts examined to ensure proper functioning (e.g., on-site assembly or operation) before going into the field. A repair kit should accompany each major piece of equipment in case of equipment failure or loss of removable parts. Backup equipment and sampling gear should be available.

Storage, transport, and sample containers, including extra containers in the event of loss or breakage, should be pre-treated and labelled appropriately (i.e., with a waterproof adhesive level to which the appropriate data can be added, with an indelible ink pen capable of writing on wet surfaces). The containers must have lids that are fastened securely, and if the samples

are for legal purposes, they should be transported to the field in a locked container.

A sample-inventory log and a sample-tracking log should be prepared in advance of sampling. The responsibility for these logs should be assigned to one individual who will be required to monitor the samples from the time they are collected until they area analyzed and disposed of, or archived.

Safety precautions and equipment that apply to the operation of the vessel, the sampling equipment, and sample handling methodologies should be observed and they should be the responsibility of the health and safety field-officer. Quality assurance and quality control should be incorporated into all aspects of the sampling program as required.

Careful preparation and the detailed planning of the sampling program can obviate, or at least minimize, many of the frustrations that might be encountered in field sampling. A weather check must be performed before the sampling crew sets out in a boat. One of the key components of a successful field trip is to use experienced field personnel, who understand their duties and responsibilities, and who know and understand the standard operating procedures and approved protocols and yet are flexible enough to deal with the unexpected.

2.4 Field Measurements and Observations

Field measurements and observations are critical to any sediment collection study and they should be documented clearly in the field notebook. The following information (Mudroch and MacKnight, 1991) should be recorded in the field notebook at the time each sediment sample is collected from a sampling station:

- sample number, replicate number, station number, site identification (e.g., name);
- time and date of the collection of the sample;
- ambient weather conditions, including wind speed and direction, wave action, current, tide, vessel traffic, temperature of both the air and water, thickness of ice if present;
- station location (e.g., positioning information) and location of each replicate sample;
- type of vessel used (e.g., size, power, type of engine);
- type of sediment collection device and any modifications made during sampling;
- the water depth at each sampling station and the sediment sampling depth;
- name of personnel collecting the samples;
- details pertaining to unusual events which might have occurred during the operation of the sampler (e.g., possible sample contamination, equipment failure, unusual appearance of sediment integrity, control of vertical descent of the sampler, etc.);
- description of the sediment including texture and consistency, colour, odour, presence of biota, estimate of quantity of recovered sediment by a grab sampler, or

length and appearance of recovered cores (photographs provide a good permanent record of a retrieved sample); and

- deviations from SOPs

Field measurements should include the following:

- temperature and pH of the sediment at the sediment-water interface;
- redox potential of the surficial sediments which define oxic or anoxic conditions;
- the concentration of dissolved oxygen of the surficial sediments to determine if the sediments are oxic or anoxic, or the depth of the interface between these conditions in the sediments; and
- temperature and salinity or conductivity of the overlying water.

These measurements could be useful for the interpretation of the analytical results.

2.5 Collection of Whole Sediments

Recommended Options and Procedures

- The devices recommended for the collection of sediments in freshwater, estuarine, and marine environments are presented in Figures 3, 4, and 5, respectively.
- A minimum penetration depth of 6 to 8 cm is recommended for surficial sediment sampling; however, a depth of 10 to 15 cm is preferred.
- Appropriate winching systems are required to control the rate of ascent and descent of the samplers.
- The sampling vessel or platform should be stationary, and as stable as possible.
- Field notes and/or measurements must accompany each sample.
- If sediments adhere to the outside of the sampler, the external surface of the sampler should be carefully hosed with clean water upon retrieval, before the sample is transferred to a storage container.
- The sampler should be rinsed thoroughly with water at the sampling station between within-station samples, and rinsed with water from the next sampling station before collecting a sample. Equipment used in the handling of sediment must also be washed thoroughly between samples.
- A sample must meet the criteria of acceptability (e.g., see Subsection 2.5.4) before it is considered adequate.
- *In-situ* collection of pore water is recommended for geochemical investigations but, for routine toxicity testing, pore water may be extracted in the laboratory using centrifugation of collected sediments.
- Samples must be collected to satisfy the sampling QA/QC program and DQO:
  - only personnel experienced and trained with respect to the standard operating procedures of the equipment should execute the sampling plan;
  - sampling equipment must be maintained and calibrated, and consistently operated throughout the study;

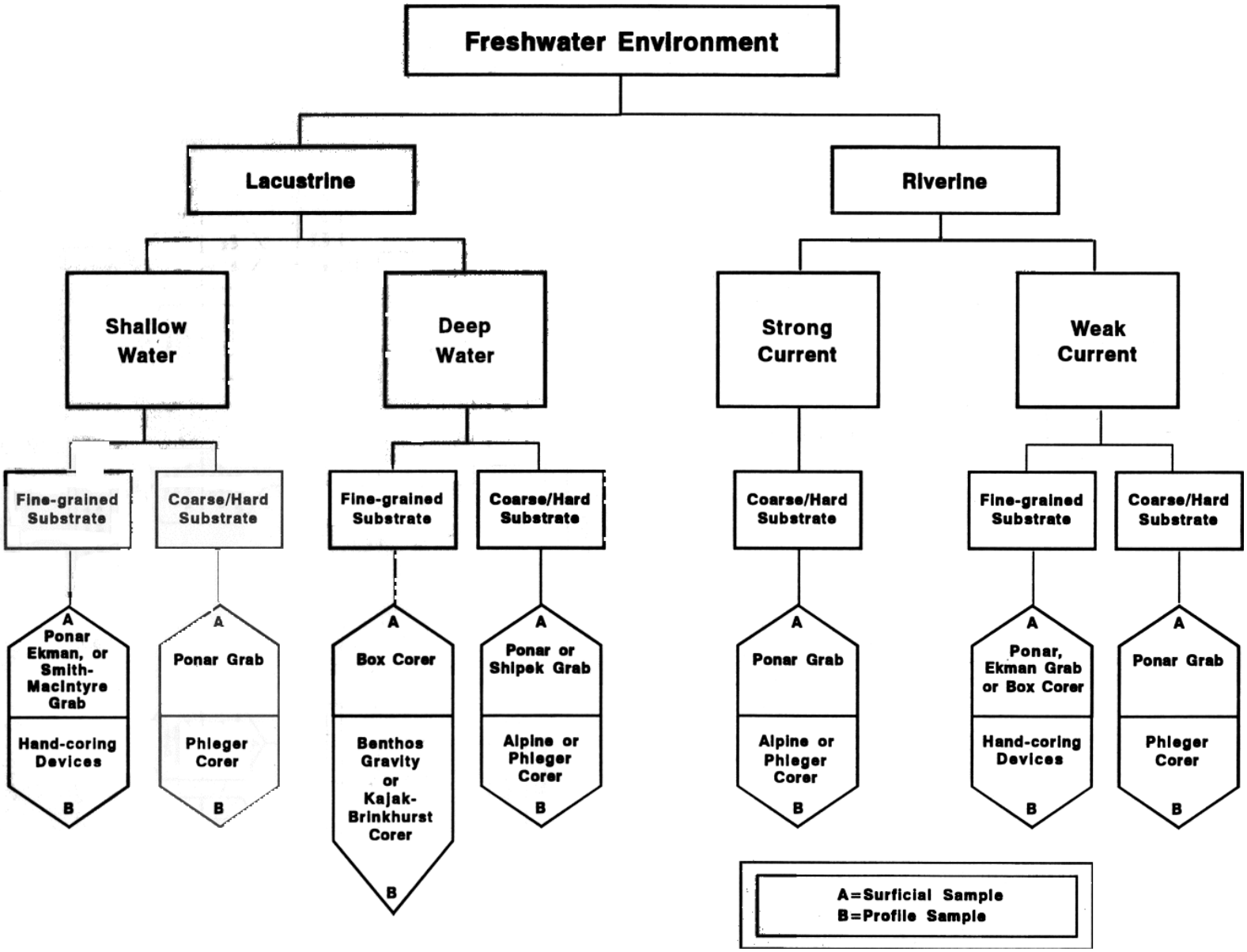


Figure 3 **Recommended Samplers for Different Types of Freshwater Environments**

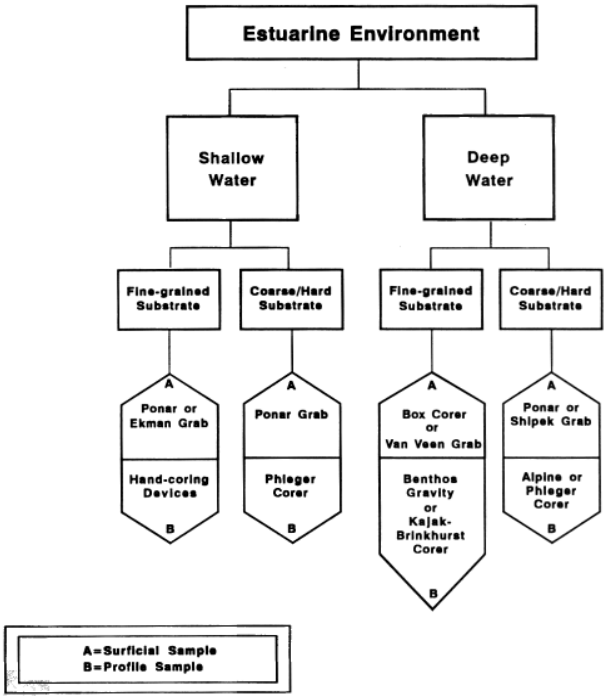


Figure 4 Recommended Samplers for Different Types of Estuarine Environments

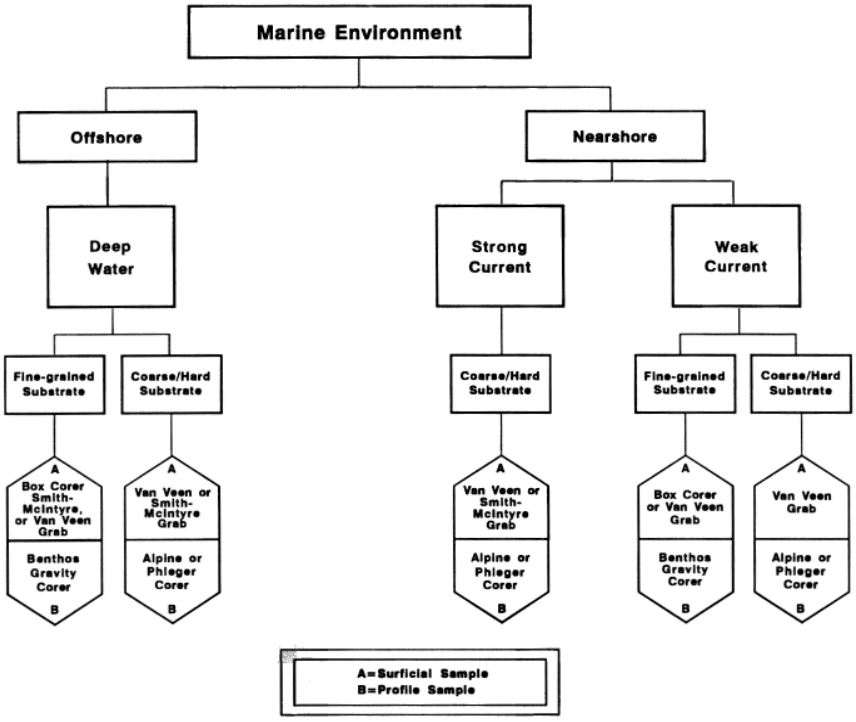


Figure 5 Recommended Samplers for Different Types of Marine Environments

- written standard operating procedures must be available to personnel at all times during the field study;

- collection procedures and methods must be documented and available;

- replicate samples within stations should be collected.

2.5.1 Criteria and Considerations for Selecting a Collection Device

There are numerous methods and procedures reported in the literature that describe how to collect various types of sediment samples in different types of environments (for reviews see Baudo *et al.,* 1990; Mudroch and MacKnight, 1991; ASTM, 1992a; Burton (1992). Baudo *et al.,* (1990) suggest several factors that should be considered when collecting sediments and most of these factors address concerns with station selection and collection devices (Table 4). Obviously, there is no one sampler that will satisfy all of the considerations.

Most sediment samplers are designed to consistently isolate and retrieve a volume of sediment, to a required depth below the sediment surface, with minimum disruption to the integrity of sample, and no contamination of the sample. Maintaining the integrity of the collected sediments is of primary concern in most studies, since disrupting the structure of the sediment changes the physicochemical and biological characteristics which in turn could influence the partitioning, complexation, speciation, and bioavailability of the toxicants, and thus the potential toxicity of the sediments. It is difficult to collect a sediment sample with most samples devices without some degree of disruption; however, and in some specific instances (e.g., ocean disposal of dredged material), maintaining the integrity of a sediment sample may be less important than the consistent, well-documented collection of samples and proper operation of the sampling device.

There are three main types of sediment samplers that either grab, core, or dredge sediments. Grab samplers are used to collect surficial sediments for the determination and assessment of the horizontal distribution of sediment characteristics. Core samplers are used to collect sediment profiles for the determination of the vertical distribution of sediment characteristics. Dredge samplers are used primarily to collect benthos. The advantages and disadvantages of the various collection devises or methods have been summarized in Table 5 and discussed briefly in the following, and in detail elsewhere (de Groot and Zschuppe, 1981; Baudo *et al.,* 1990; ASTM, 1992a; Burton, 1992; Sly and Christie, 1992).

Gas samplers have the advantage of being easy to handle and operate, readily available, moderately priced, versatile in terms of substrate type, and they can collect either small volumes (0 to 10 cm deep; e.g., Birge-Ekman, Ponar, mini-Ponar, or mini-Shipek) or large volumes (0 to 30 cm deep; e.g., Van Veen, Smith-McIntyre, Petersen). Careful use of sampling devices can avoid most of the problems associated with unpredictable penetration of sediment, loss of sediment from tilting or washout upon ascent, mixing of sediment layers at impact, loss of fine-grained surface sediments from the bow wave during ascent, and susceptibility to the influences of waves and currents (Plumb, 1979; Golterman *et al.,* 1983; Blomqvist, 1990; Baudo *et al.,* 1990; ASTM, 1992a).

Table 4 Factors that Should be Considered for Selection of Sediment Sampler and Sampling Location (after Baudo, 1990; Håkanson and Jansson, 1983)

The ideal sediment sampler should for the most part:

- permit free water passage during descent, to avoid a pressure wave
- the cutting surface should be sharp-edged, have a small edge angle, smooth inside surface, small wall thickness to minimize disturbance
- close tightly for the ascent
- allow subsampling
- have the capability of adjusting weight for penetration of different substrates
- be able to retrieve a volume of sediment large enough to meet the analytical test requirements
- effectively and consistently retrieve sediments from various water depths
- effectively and consistently retrieve sediments from the desired sampling depth
- not contaminated or influenced by the nature of the sediment
- require a minimum of supportive equipment
- be easy and safe to operate and not require extensive training of personnel
- be easily transported to and assembled at the sampling site

Core samplers are generally preferred where maintaining the integrity of the sediment profile is essential because they are considered to be the least disruptive. Core samplers should be used where it is important to maintain an oxygen-free environment, for there is less of a chance of oxidation to occur.

Sediment coring devices are variable and include hand-coring devices (50 to 120 cm core tubes), single gravity corers for sediment samples that are 0 to 50 cm deep (e.g., Kajak-Brinkhurst, Phleger), or 0 to 2 m deep (e.g., Benthos and Alpine), multiple corers (0 to 50 cm deep, 2 to 4 barrels), box corers (0 to 50 cm deep), and piston corers for sediment samples deeper than 2m. Boomerang corers (0 to 1.2 m deep) are used to collect samples from the seafloor and percussion corers or vibrating corers with a stationary piston (0 to 13 m deep) are used for hard clays, shales, and calcareous sandstones (Mudroch and MacKnight, 1991). A portable vibrating corer is available for the collection of sediments from water depths up to 18 m (Lanesky *et al.,* 1979). Although core samplers have the advantage of collecting minimally disturbed, intact sediment samples from both surficial sediments (upper 15 to 30 cm) and deep sediments (30 cm deep), there are few that function efficiently in substrates with sand, gravel, clay, or till. The major limitation to core samplers is that although the core tubes can vary in diameter (3.5 to 10 cm I.D.), the volume of the upper surficial horizon (0 to 5 cm deep) is relatively small, and repetitive, time-consuming sampling may be required to obtain the desired quantity of material. Loss of the highly porous layer at the sediment-water interface due to material displacement during the initial contact, compression during penetration, over penetration, tilting, and loss of sediments during ascent are commonly encountered problems that must be overcome or minimized by ensuring that the proper techniques for collecting the sample are followed.

Table 5 Advantages and Limitations of the Different Sediment Collection Devices Most Commonly Used to Collect Sediments (ASTM, 1992a; Mudroch and MacKnight, 1991)

Device/ Dimensions	Use	Sediment Depth Sampled (cm)	Volume of Sediment Sample (cm^3)	Advantages	Disadvantages
GRAB SAMPLERS					
Orange-Peel Grab, Smith-McIntyre Grab	Deep lakes, rivers and estuaries	0 to 30	10 000 to 20 000	Designed for sampling hard substrates	Loss of fine-grained sediment; heavy relative to other grabs; may require winch; possible metal contamination
*Birge-Ekman Small	Lakes and marine areas. Soft sediments, silt and sand	0 to 10	≤ 3 400	Same as Ekman	Same as Ekman
*Birge-Ekman Large	Lakes and marine areas. Soft sediments, silt and sand	0 to 30	≤ 13 300	Same as Ekman	Restricted to low current conditions; penetration depth exceeded by weight of sampler
*PONAR Grab Standard	Deep lakes, rivers and estuaries. Useful on sand, silt or clay	0 to 10	7 250	Most universal grab sampler; adequate on most substrates; large sample obtained intact, permitting subsampling; good for coarse and firm bottom sediments	Shock wave from descent may disturb fine-grained sediment; possible incomplete closure of jaws results in sample loss; possible contamination from metal frame construction.
PONAR Grab Mini	Deep lakes, rivers and estuaries. Useful on sand, silt or clay	0 to 10	1 000	Adequate for most substrates that are not compacted	Smaller volume does not minimize disturbance to sample
* Van Veen	Deep lakes, rivers and estuaries; useful on sand, silt or clay; effective in marine environment in deep water and strong currents	0 to 30	18 000 to 75 000 18 to 75 L	Adequate on most substrates; large sample obtained intact, permitting subsampling; available in stainless steel	Shock wave from descent may disturb fine-grained sediment; possible incomplete closure of jaws results in sample loss; premature closing in rough waters; possible contamination from metal frame construction
*Petersen Grab Sampler	Deep lakes, rivers and estuaries. Useful on most substrates.	0 to 30	9 450	Large sample can penetrate most substrates	Heavy, likely requires winch; no cover lid to permit subsampling; all other disadvantages of Ekman and Ponar

Table 5 **Advantages and Limitations of the Different Sediment Collection Devices Most Commonly Used to Collect Sediments** (ASTM, 1992a; Mudroch and MacKnight, 1991) **(Cont'd)**

Device/ Dimensions	Use	Sediment Depth Sampled (cm)	Volume of Sediment Sample (cm^3)	Advantages	Disadvantages
*Shipek Grab Sampler Standard	Used primarily in marine waters and large inland lakes and reservoirs; not useful for compacted sandy clay or till substrates	0 to 10	3 000	Sample bucket may be opened to permit subsampling; retains fine-grained sediments effectively	Possible contamination from metal construction; heavy, may required winch
Mini Shipek	Lakes, useful for most substrates that are soft	0 to 3	500	Easily operated by hand from most platforms	Requires vertical penetration; small volume; washout of fine-grained sediment; premature closing
CORE SAMPLERS					
Fluorocarbon plastic or glass tube 3.5 to 7.5 cm I.D.; ≤ 120 cm long	Shallow wadeable waters or deep waters if SCUBA available. Soft or semi- consolidated deposits	0 to 10	96 to 442	Preserves layering and permits historical study of sediment deposition; rapid; samples immediately ready for laboratory shipment; minimal risk of contamination	Small sample size requires repetitive sampling
*Hand corer with removable fluorocarbon plastic or glass liners 3.5 to 7.5 cm I.D. ≤ 120 cm long	Same as above except more consolidated sediments can be obtained	0 to 10	96 to 442	Handles provide for greater ease of substrate penetration; above advantages apply	Careful handling necessary to prevent spillage; requires removal of liners before repetitive sampling; slight risk of metal contamination from barrel and core cutter
* Box corer	Same as above but the depth of the unconsolidated sediment must be at least 1 m	0 to 50	≤ 30 000	Collection of large sample, undisturbed, allowing for subsampling	Hard to handle; heavy machinery required
* Gravity corers: Phleger Corer 3.5 to I.D ≥ 50 cm long	Deep lakes and rivers. Semi-consolidated sediments	0 to 50	≤ 481	Low risk of sample contamination; maintains sediment integrity relatively well; high point loading with sharp cutting edge	Careful handling necessary necessary to avoid sediment spillage; small sample requires repetitive operation and removal of liners. Time consuming

Table 5 Advantages and Limitations of the Different Sediment Collection Devices Most Commonly Used to Collect Sediments (ASTM, 1992a; Mudroch and MacKnight, 1991) **(Cont'd)**

Device/ Dimensions	Use	Sediment Depth Sampled (cm)	Volume of Sediment Sample (cm^3)	Advantages	Disadvantages
* Kajak-Brinkhurst Corer 5 cm I.D. ≤ 70 cm long	Deep lakes and rivers. Soft fine-grained sediments	0 to 70	≤ 1 374	Greater volume than the Phleger Corer	Same as Phleger Corer
* Benthos Gravity Corer 6.6, 7.1 cm I.D. ≤ 3m long	Soft, fine-grained, sediments	0 to 3 m	≤ 10 263	No loss of sample from tube because the valve is fitted to the core liner; fins promote vertical penetration	Weights required for deep penetration so the required lifting capacity is 750 to 1000 kg; vertical penetration is required; sediment compaction
* Alpine Gravity Corer 3.5 cm I.D.	Soft, fine-grained, semi-consolidated substrates	≤ 2 m	≤ 1 924	Interchangeable steel barrel for different penetration depths	Requires a vertical penetration but has no stabilizing fines; penetration is often non-vertical and incomplete and requires a lifting capacity of 2 000 kg; sheared laminae and disturbed sediment integrity; compaction of sediment
*Piston Corers	Ocean floor and large deep lakes. Most substrates	3 to 20 m	?	Typically recovers a relatively undisturbed sediment core in deep waters	Requires lifting capacity of > 2 000 kg; non-activation of piston and piston positioning at penetration; disturbance of the surface (0 to 0.5 m) layer
BMH-53 Piston Corer	Waters of ≤ 2m ft deep when used with extension rod. Soft to semi-consolidated deposits	> 2 m	?	Piston provides for greater samples retention	Cores must be extruded onsite to other containers; metal barrels introduce risk of metal contamination
Boomerang Corer 6.7 cm I.D.	Ocean floor (up to 9 000 m deep)	1 m	3 525	Minimal shipboard equipment so small vessels could be used	Only 1.2 m penetration; recovery requires calm water; 10 to 20% loss rate
Vibratory Corer 5 to 7.5 cm I.D.	Continental shelf of oceans, large lakes. Sand, silty sand, gravelly sand substrates	3 to 6 m	5 890 to 13 253	For deep profiles it effectively samples sandy substrates with minimum disturbance; can be operated from small vessels (e.g., 10 m long)	Labour intensive; and assembly and disassembly might require divers; disturbance of the surface (0 to 0.5 m) layer

Table 5 Advantages and Limitations of the Different Sediment Collection Devices Most Commonly Used to Collect Sediments (ASTM, 1992a; Mudroch and MacKnight, 1991) **(Cont'd)**

Device/ Dimensions	Use	Sediment Depth Sampled (cm)	Volume of Sediment Sample (cm^3)	Advantages	Disadvantages
DREDGE SAMPLERS					
Young Grab (fluorocarbon) plastic- or kynar-lined modified 0.1 m^3 van Veen)	Lakes and marine areas	0 to 30	≤ 18 000	Eliminates metal contamination; reduced bow wake	Expensive; requires winch
*Ekman or Box Dredge	Soft to semi-soft sediments. Can be used from boat, bridge or pier in waters of various depths	0 to 10	≤ 4 000	Obtains a larger sample than coring tubes; can be subsampled through box lid	Possible incomplete jaw closure and sample loss; possible shock wave which may disturb the fine-grained metal construction may introduce contaminants; possible loss of fine-grained sediment on retrieval; difficult to use in current
Scoops, Drag Buckets	Various environments depending on depth and substrate	Variable	10 000 to 20 000	Inexpensive, easy to handle	Loss of fine-grained sediment on retrieval through water column

This list is representative of the types of sediment collection devices which are available, but is not exhaustive. The selection criteria used to derive the recommendations were applied only to those devices designated by an asterisk (*).

Dredges are used primarily for the collection of benthos, for they are usually equipped with net sides designed to filter out fine-grained sediments and retain coarse sediments and fauna. It is virtually impossible to accurately measure the surface area covered by the dredge sampler or judge the depth to which the sediment sample has been collected. In addition, sediment integrity is disrupted, pore water excluded, volatile organic compounds lost, and fine-grained sediments lost during ascent using dredge samplers. For these reasons, grab and core samplers are recommended. The ASTM (1992 a,b) also concur that grab or core collection devices should be used rather than dredges.

2.5.2 Penetration Depth

The desired depth of sediment penetration is a decision that depends upon the objective(s) of the study, the type of sampling device, the nature of the sediment, and the volume of sediment required. The actual depth of penetration depends primarily on the type of sampling device and the nature of the

sediment. For most monitoring and assessment studies where historical contamination is not a priority, the upper 0 to 5 cm of sediment is the horizon of interest for chemical characterization and toxicity evaluation. Generally, the most recently introduced contaminants of concern and most infaunal organisms are found in the upper 2 cm, and epifaunal organisms have access to this horizon (Burton, 1992). Therefore, a preferred penetration depth of 10 to 15 cm and a minimum penetration depth of 6 to 8 cm are recommended to ensure minimum disturbance of the upper layer during sampling. Both core and grab sampling devices can be used to collect surficial sediments. For collection of sediments to greater depths of penetration, core samplers designed for this purpose are available and are recommended (see Table 5).

2.5.3 Sampler Operation

Grab Sampler. When collecting bottom sediments with grab samplers, the speed of descent of the sampling device should be controlled and it should not be permitted to "free fall". To minimize twisting during the descent, a ball-bearing swivel should be used to attach the sampler to the cable. The sampler should contact the substrate or be positioned just above it and only its weight or piston mechanism should be used to force it into the sediment. The winching system should be in place to control both the ascent and descent of the sampling device. After the sample is contained, the sampling device should be lifted slowly off the bottom then steadily raised to the surface at about 30 cm/s. When the sampler is brought to the surface, the outside of the sampler should be carefully hosed with water from the sampling station to remove material that could potentially contaminate the sample during transfer, and inspected to see that the sampler has closed properly. Maintaining a hydraulic head by using a submerged bucket which can accommodate the grab sampler will reduce the loss of soft sediment during withdrawal from the water. The bucket and grab sampler are withdrawn together (Hesslein, 1993). The standard operating procedures specific for each grab sampler should be followed to ensure proper operation of the sampler.

Core Sampler. Collecting core samples with hand-coring devices should be executed with care to minimize disturbance and/or avoid compression during collection or capping. Rutledge and Fleeger (1988) demonstrated that sediments collected by divers using hand-coring devices can mix in the core tube during transport to the surface of the water. To minimize this disruption of the sediment, the sample must be kept vertical and as stationary and vibration-free as possible during transport.

Coring devices are extremely variable in terms of their construction, assembly, and operation; therefore, it is very important that the standard operating procedures specific for each coring device be followed, to ensure proper operation of the sampler. The speed of descent should be controlled, especially during the initial penetration of the sediment, to avoid disturbance of the surface by the bow wave and to minimize compression due to the frictional drag from the sides of the core liner. Winches should be used, where appropriate (i.e., where the weight or type of coring device dictates the need), to minimize twisting and tilting and to control both the rate of descent and ascent. Careful removal and retrieval of the sampler from the sediment is required to minimize loss of sediment and disturbance of the core. The core sampler should be raised to the surface at a steady rate, similar

to that described for grab samples. Where core caps are required, it is crucial to quickly and securely cap the core samples at the appropriate time. If a messenger is used to trigger a release mechanism, it is important to keep all cables free and as straight as possible. When the sampler is brought to the surface and the sample capped correctly, the sampler should be hosed, if necessary, to remove material adhering to the sampler which might interfere with the further handling of the sediment core, such as the removal of the core liner. The liner from the core sampler should be carefully removed, kept in a vertical position, and the core visually inspected for acceptability (Subsection 2.5.4), and its physical appearance should be described.

Regardless of the type of samplers used, standard operating procedures for each device must be immediately accessible, and all personnel involved with the collection of samples should be familiar with these procedures. The sampling vessel or platform should be stationary, and sufficiently stable to permit inspection and handling of the retrieved sample. Field notes must accompany each sample that is collected (see Section 2.4). The sampling device must be cleaned thoroughly between sampling stations and between within-station samples by dipping the sampler into and out of the water at a rapid speed to wash the sediment off. Alternatively, a hose can be used to wash the sediment off of the sampler with lake, river or ocean water, depending on the study environment. The sampler should be rinsed with water from the next sampling station before collecting a sample.

2.5.4 Criteria of Acceptability

All samples should be visually inspected to ensure that:

- the overlying water (if present) is clear or not excessively turbid;
- sediment-water interface is intact with no sign of channelling, sample washout, or over-penetration;
- the desired depth of penetration has been achieved;
- there is no evidence of incomplete closure of the grab sampler, or that the grab or core sampler was inserted on an angle or tilted upon retrieval (i.e., loss of sediment);
- the core is complete with no air space at the top of the liner before capping (i.e., no loss of sediment; and,
- the length of the core is within the range stipulated in the sampling protocol.

If the collected sample fails any of these criteria, then the sample should be rejected and another sample collected at the site. The location of consecutive attempts should be as close to the original attempt as possible and, where the direction of the current is known, consecutive attempts should be located in the opposite direction of the current or "upstream". Rejected sediment samples should be discarded in a manner that will not affect subsequent samples at that station or other possible sampling locations.

2.5.5 Collection of Control and Reference Sediment

The collection, transport, and storage of reference sediments and naturally occurring control sediments should follow the procedures recommended in Subsections 3.4.2 and 3.4.3, respectively.

2.6 In-situ *Collection of Pore Water*

Pore water may be recovered either from sediments *in-situ* or from field-collected samples of sediment. This section briefly describes various methods for the *in-situ* collection of pore water; whereas, methods for the recovery of pore water from field-collected samples of sediment are described in Subsection 2.9.3.

Fine-grained surface sediments in lakes typically contain 90 to 95% water (Adams, 1991). Some of this water is bound to the crystalline lattice of minerals in the sediments, but most of the water simply occupies the space between sediment particles. This water is referred to as pore or interstitial water. The intimate association of this water with the surface of sediment particles results in reactions between the particles and the water approach equilibrium. The partitioning of contaminants in sediments between the particulate and water phases depends to a large extent on the amount of organic carbon, sediment particle size, the chemical form of the contaminants, and the physicochemical environment (e.g., pH, temperature, redox potential, sorption/desorption properties of sediments, or the equilibrium between the solid and liquid phases). The dynamics of these processes are not well understood (Giesy and Hoke, 1990); however, it is generally assumed that concentrations of most substances in the pore water approach equilibrium with the solid phase and its associated contaminants. Consequently, pore water has been collected for toxicity testing to approximate the relative toxicity of contaminated sediments, and/or to assess contaminant levels. Small quantities of pore water are sufficient for geochemical analyses; however, it is difficult to collect sufficient quantities for biological toxicity tests by *in-situ* sampling. Most assessments of this nature have been conducted to date with water recovered from sediments by squeezing, centrifugation, or suction (see Subsection 2.9.3).

The *in-situ* sampling of pore water has been achieved by various methods (Barnes, 1973; Sayles *et al.,* 1973; 1976; Hesslein 1976; Mayer, 1976; Murray and Grundmanis, 1980; Brinkman *et al.,*1982; Whiticar, 1982; Bottomley and Bayly, 1984; Howes *et al.,*1985; Jahnke, 1988; Belzile *et al.,*1989; Buddensiek *et al.,*1990; Carignan and Lean, 1991). There are too few comparative studies available to recommend one method over another. Most *in-situ* porewater samplers have been designed to sample pore water close to the sediment-water interface by either diffusion through a membrane sandwiched between acrylic plates (e.g., dialyzer or peeper), or by suction and filtering. These methods have evolved in response to the realization that separating pore water from collected sediments in the laboratory produces results that do not always represent the nature or chemistry of the water (Manheim, 1976; Kriukov and Manheim, 1982; Howes *et al.,*1985; Adams, 1991). These inconsistencies (e.g., artifacts) result primarily from changes in temperature (Bischoff *et al.,* 1970); pressure (e.g., CO_2 degassing) (Simon *et al.,* 1985), and the exposure of both the liquid and particulate phases of sediment to oxygen (Bray *et al.,* 1973; Sayles *et al.,* 1973; Hesslein, 1976; Mayer, 1976; Lyons *et al.,* 1979; Lee and Jones, 1984).

The various methods for collection of *in-situ* pore water and their respective attributes and limitations are summarized in Table 6. For more details consult Appendix D and the original citations. It is important to be aware that all of the techniques use some type of

Table 6 A Summary of the Various Methods for the Collection of Pore Water, *In-Situ*

Method of Collection	Advantages	Disadvantages	Reference
Long-needled syringes	· closed system minimizes the possibility of contamination	· restricted to use in shallow waters (e.g. , <10 m); small volumes (5 to 10 mL) can be collected from a given depth	Bauer *et al.,* 1988 Brinkman *et al*., 1982
Modified syringes	· absence of contamination from overlying water	· effectively sampled <350 µL	Knezovich and Harrison, 1987
Probe system	· repeated sampling at specific depths (0 to 35 cm; 1-cm increments); low cost, easily constructed; stable unit which could be operated throughout the year	· requires a 2-week period for sediment stabilization; potential problem with mixing within adjoining horizons; requires adjustment of the zero level because of shifting sediment surfaces	Buddensiek *et al.,* 1990
Hydropressure or vacuum filtration	· closed system minimizes the possibility of contamination; can be used in shallow (< 10 m) or deep waters (20 to 1800 m); displacement with inert gas maintains anoxic conditions and minimizes artifacts; can be used for estuarine sediments, rivers, lakes, and marshes, deeper marine waters or shallow subtidal marine waters.	· although volumes were slightly larger (50 to 75 mL) the depth from which the interstitial water is retrieved is uncertain (e.g., limited depth resolution)	Montgomery *et al.,* 1979; 1981 Hertkorn-Obst *et al.,* 1982 Howes *et al.,*1985 Sayles *et al.,* 1976 van der Loeff, 1980
Dialyzers or peepers	· use of specific membranes can confer some solute selectivity; relatively free of temperature, pressure, and oxidation artifacts; simple and less costly to construct and operate than most other methods.	· cellulose-based membranes are susceptible to microbial attack; equilibrator and system must be degassed with nitrogen to ensure deoxygenation; equilibration chamber is required; equilibration time must be determined experimentally as it is variable and may be lengthy; oxidation of interstitial water during careless transfer from dialyzer; development of electrical potential across membrane.	Adams, 1991 Martens and Klump, 1980 Hesslein, 1976 Kelly *et al.,* 1984 Carignan and Tessier, 1985 Tessier *et al.,* 1989 Simon *et al*., 1985 Carignan, 1984

filter, membrane, or plastic material such that sorption of organic contaminants can become a significant problem. Additionally, these techniques usually generate only small volumes of pore water and so both chemical analysis and biological testing for organic contaminants might be problematic. Finally, for those techniques that use dialysis, organic contaminants may not approach equilibrium because of the limits of diffusion within sediments and the large capacity of the membranes for absorbing organic contaminants.

2.7 Handling of Collected Samples

Recommended Procedures

- All samples must be handled in a manner that satisfies the QA/QC program and DQO.

- If samples are designated as legal samples, then the appropriate procedures (see Subsection 2.8.3) and chain-of-continuity documentation (Appendix H) must be followed.

- Sediment might contain a mixture of hazardous substances or materials, so it is prudent to avoid skin contact with these sediments by wearing protective clothing and equipment (e.g., gloves, boots, lab coats or aprons, safety glasses, and respirator) during sampling, sample handling, and preparation of test substances or sediments.

- Handling of samples should be performed in a well-ventilated area (e.g., outside, in a fume hood, or in an enclosed glove box) to minimize the inhalation of sediment gases.

- Work surfaces should be covered with Teflon® sheets, high-density polyethylene trays, or other impervious or disposable, similarly inert material.

- A spill control protocol should be in place in the laboratory or sampling vessel, and participants in the project should be familiar with all Standard Operating Procedures and recommendations.

- Disposal of all hazardous waste should adhere to the existing applicable by-laws, guidelines, or regulations.

2.7.1 Core Samples

Recommended Procedures

- Sediment core samples in tubes or liners must be capped tightly with air excluded, secured in an upright position, labelled appropriately, and placed into an insulated transport container with ice. If the sediment has been retrieved from a water depth 10 m and has a high organic content and high concentrations of methane and carbon dioxide, it may be necessary to remove the overlying water within a minute of retrieval, to minimize disturbance to the core by the formation of bubbles.

- Subsampling, if necessary, should be done using extrusion procedures within 24 h of collection and should be restricted to parts of the sediment sample that have had no direct contact with the sampler.

- Box-core samples should be subsampled using clean, non-reactive (e.g., Teflon®-line scoop or hand-coring device) implements for the top 0 to 5 cm and a hand-corer for greater depths; subsampling should be restricted to sediments that have had no direct contact with the sampler.

- If compositing samples or subsamples is required, it can be done in the field or in the laboratory. Before a composite sample or subsample may be analyzed or used in a test it should be homogenized with a mechanical mixer, or by hand, until uniform in colour and texture, or for a specified period of time that has been determined experimentally.

- If the maintenance of an oxygen-free environment for sediment handling is part of the study plan, handling of anoxic sediments should be done in a glove box in the presence of an inert gas.

If the retrieved sediments are core samples to be transported directly to the laboratory intact, the following procedures and precautions should be followed.

- The core liners should be capped or stoppered and taped closed immediately upon retrieval to prevent loss of sediment.

- A visual inspection of the intact core should be recorded in field notes, describing the length of the sediment core; the thickness of the various sediment units; and descriptions of the core including colour, consistency (e.g., packed, loose, consolidated, unconsolidated), texture (e.g., gravel, sand, silt, clay), and the presence of shells, organic matter, vegetation, organisms, oil, and noticeable odour (e.g., sulphur, chlorine, sewage, petrol). Because odours from contaminated sediments could be potentially hazardous, care should be taken to avoid smelling them directly.

- The intact core samples (liners) should be secured in an upright position (e.g., rack) and labels should be applied with the appropriate information (sampling site location, sample number, and/or identification, time and date of collection, method of collection, name or initials of the collector) to ensure accurate sample identification.

- Air should be excluded from the liners and the volume of overlying water should be minimized to reduce the potential for resuspension of the surface sediments during transport. Particular care should be taken to retain the surficial floc overlying the core. The core tubes should be placed into either a transport container (e.g., insulated box or cooler) with ice packs or into a refrigerated unit that can maintain a preferred temperature of $4 \pm 2°$ C which can be continuously monitored during transport.

- A travel blank should be a part of the QA/QC program.

If the transport container cannot accommodate the dimensions of the long core samples (core liners > 1 m), then the core samples can be cut before transporting into 1-m lengths, and the ends securely capped such that there is no air trapped inside the liners. It must be demonstrated that the methods and equipment used to cut or section the core cause minimal disruption of the integrity of the sediment within the section, does not contaminate the sample, and results in no loss of the sediment continuum. Various methods have been used to cut, split, or section sediment cores (Mudie *et al.,*1984; see Mudroch and MacKnight, 1991, for review). Freezing before transporting or sectioning is not recommended because freezing changes the sediment volume, which varies with water content, and it permanently changes the structure of the sediment (de Groot and Zschuppe, 1981; Rutledge and Fleeger, 1988; Mudroch and MacKnight, 1991).

Impregnating unconsolidated sediment cores with epoxy or polyester resins will preserve sediment structure and texture (Ginsburg *et al.,* 1966; Crevello *et al.,*1981) but not the chemical characteristics, so this procedure is not recommended for samples destined for chemical characterization or biological testing.

Subsampling and/or Compositing Core Samples The decision to subsample (i.e., section) and/or composite sediment samples collected from the same sampling station depends on the purpose and the objective(s) of the study, the nature and heterogeneity of the sediments, the volume of sediment required for analytical and/or toxicity assessment, the degree of statistical resolution that is acceptable, and the cost/benefit analysis of not performing these procedures.

Subsampling sediment core samples is usually done to focus the assessment on a particular sediment horizon or horizons. Most monitoring and assessment studies of contaminated surface sediments (i.e., the upper 10 to 15 cm) are concerned with the horizontal distribution of recent deposited contaminants and/or their effects. However, other monitoring and assessment studies might require the collection of deeper sediment samples to evaluate historical changes (i.e., zones of contamination, sedimentation rates, geochemical differences with depth of sediment.

If subsampling of the retrieved sediment core is required, it should be done as soon as possible (i.e., within 24 h). This can be accomplished in the field if the appropriate facilities, space, and equipment are available, or in the laboratory after transport.

Various methods have been developed to cut, section, split and/or subsample sediment. These methods have gradually evolved to accommodate different study objectives, types of sediments, and different aquatic environments. The most common method of subsampling sediment cores is by upward piston or pressure extrusion of measured aliquots of sediment (e.g., 1 or 2 cm in length) which are sectioned with a metal, nylon, or Teflon® cutter after the overlying water is siphoned off and discarded. The method of extrusion should permit subsampling of only the inner area of the core and the subsequent exclusion of peripheral sediment (i.e., that which is in direct contact with the core liner). The surficial layer (e.g., upper 0 to 2 or 0 to 5 cm) may be subsampled more effectively with flat scoops or wide-bore syringes (e.g., modified pipette) made of non-reactive material. Each sediment subsample should be placed in a labelled, clean, and chemically inert container (Section 2.8). The size of the container should be as close to the volume of the sample as possible, to minimize the head space in the container. Ideally, all oxygen in the container should be removed or displaced with an inert gas (e.g., nitrogen or argon). Although the depth increment most frequently subsampled is 0 to 2 cm, increments of 0 to 5 cm or 0 to 10 cm are not uncommon. The depth increment for core sectioning can be variable and depends on the objective(s) of the study, the nature of the substrate, sedimentation rates at the site, and the type of environment from which it originated. If it is desirable to maintain an oxygen-free environment during subsampling, then all handling or manipulations should take place in a glove box or bag filled with an inert gas and modified to accommodate the core liner through an opening (Mudroch and MacKnight, 1991).

Sediments from box-core samples can be effectively subsampled with a small hand

corer after the overlying water has been carefully siphoned off and discarded. If sediment is in suspension in the overlying water, as indicated by turbidity, it should be allowed to settle before subsampling. Hand corers with small inner diameters less than 3 cm tend to compact sediments, so they must be used with care. Scoops have also been used to subsample surface sediments from a box corer.

If **compositing** subsamples from the core samples is required to meet the study objectives (see Subsection 2.3.6) the quality of the core sample must be acceptable and only sediments depths with similar stratigraphy should be combined. There might be occasions when it is desirable to composite incremental core depths; however, it is recommended that only horizons with similar stratigraphy be composited. Before it is used in a test, the composite sediment sample should be homogenized with a mechanical mixer or by hand until uniform in colour and texture, or for a specified period of time that has been defined experimentally. If the sediments are anoxic, homogenization should occur only in an oxygen-free atmosphere to minimize oxidation reactions during handling and storage.

2.7.2 Grab Samples

If a retrieved grab sample of sediment is to be transported to the laboratory intact, it is usually released carefully, but directly, into a labelled sample container that is the same shape as the sampler and made of a chemically inert material (Section 2.8). The container must be large enough to accommodate the sediment sample and should be tightly sealed with the air excluded.

Subsampling and/or Compositing Grab Samples. If the retrieved grab sample is to be **subsampled,** then access to the surface of the sample without a loss of water or fine-grained sediment is a prerequisite for selection of the sampler. The non-turbid overlying water, if present, must be gently siphoned off before the sediment is subsampled with a flat, clean, scoop (e.g., Teflon® or a similarly inert, non-contaminating, non-reactive material) or a suitable hand-coring device. The sediment should be collected to a depth of 5 cm; however, it could be subsampled to a greater depth, depending on the size of the sampler and the objectives of the study. Subsampling the top two centimetres is a common practice when most recent sediment deposits are of concern; the depth depends on the objectives of the study. Ideally, each subsample should be placed into clean, separate, prelabelled containers made of non-contaminating material (Section 2.8). The labelled sample container must be sealed and the air excluded.

In the event that the collection device does not allow access to the surface, the following procedures should be followed. Upon the retrieval of the sample, the contents must be carefully deposited into a clean, inert container that is the same shape as the sampler. The sampler is placed into the container and the jaws opened slowly to allow the sample to be deposited into the container with as little disturbance as possible. Once the sample is in the container, subsamples can be collected from the sample with a hand corer or scoop. The edges of the sample where the sediments may be disturbed during removal from the sampler should be excluded during subsampling.

It is difficult to subsample a grab sample under oxygen-free conditions. Therefore, the use of core samplers is encouraged where this is a priority [i.e., where chemical forms of trace metals or volatile constituents

(e.g., AVS) are of interest]. Nevertheless, subsampling of a grab sample of sediment in an oxygen-free atmosphere is possible, though not very practical, using a glove box, or bag, which as been filled with a constant, controlled volume of inert gas and modified to accommodate an undisturbed grab sample that has been released into a container. It is much more practical to subsample a grab sample with a hand-coring device. Place the core into a glove box or bag, and extrude the core under oxygen-free conditions. Except for the surface layers (0 to 5 cm), sediments are generally anoxic and will rapidly oxidize when exposed to air.

Collection devices made of unprotected metallic material can potentially affect the concentration of trace or major elements in the sediment samples and should be avoided where metal contamination of sediment is suspected. Collection devices should not be made of copper, zinc, brass, or galvanized material. Likewise, plastic devices should be avoided where contamination with organic compounds is suspected. If this is not possible then, when subsampling, exclude the sediment which is in direct contact with the sides of the sampler. The subsampled sediment must be transferred to clean containers comprised of inert material that will neither contaminate nor influence the characteristics of the sediment sample. The container should be tightly sealed and air should be excluded.

If the objective(s) of the study dictate(s) **compositing** subsamples from separate grabs within a sampling station, the subsamples may be placed into one clean, sample container and, when full, sealed without trapped air. Compositing of sediment samples or subsamples may also be performed in the laboratory.

2.8 Transport and Storage of Field-collected Sediments and Pore Water

Sediments collected in grab samplers are usually transferred from the sampler to sample containers which may or may not serve as the storage container as well. The containers might be either stored temporarily in the field, before being transported to and stored at the laboratory, or transported immediately to the laboratory for storage. If sediment core samples are not sectioned or subsampled in the field, they may be stored temporarily in the field before they are transported intact, in the core liner, directly to the laboratory. If sectioning or subsampling takes place in the field then the subsamples or sections may also be transferred to sample containers and stored temporarily. The sample containers with the field-collected sediments are then placed into a transport container and shipped to the laboratory. Pore water collected *in-situ* must be transferred under anoxic conditions (e.g., in a glovebox) directly into a clean storage container with inert head-gas seals, frozen or cooled to 4 ± 2° C, and transferred to the laboratory as soon as possible. Pore water for use in biological tests should not be frozen during transit or temporary field storage.

The following subsections describe the methods and conditions for temporary storage in the field, and the transport to and storage of field-collected sediments and pore water in the laboratory. Chain-of-continuity documents (Appendix H) must accompany all legal samples to demonstrate that control of a sample can be established continuously between the time it is collected and the time it is analyzed.

2.8.1 Storage Containers and Conditions

Recommendations

The recommended type of container and storage conditions (which usually depends on the required analysis) for samples of sediment and pore water are summarized in Table 7. For additional information consult Appendix E.

A whole-sediment sample may be transferred directly from a grab sampler into a clean, large volume (e.g., 1 L), pretreated, high-density polyethylene or Teflon® container. These materials are relatively inert and optimal for samples contaminated with a mixture of both organic and inorganic chemicals. If smaller volumes of sediment are collected or subsampled, then clean Teflon®, borosilicate glass, or high-density polyethylene containers with wide mouths and Teflon®-lined lids are recommended for volumes ranging from 250 to 1000 mL. If the contaminant(s) of interest is (are) known to be organic, then wide-mouthed amber glass bottles (250 to 1000 mL) are recommended. However, glass containers are susceptible to breakage, weigh more, and generally take up more space. When the contaminant(s) of interest is (are) known, then Table 7 should be consulted for more specific instructions regarding type of sample container and storage times. Samples of pore water collected *in situ* should be injected from the sampling syringes into clean, pretreated, amber glass bottles, or into vials (10 to 40 mL) with Teflon®-lined septum caps. All sample containers should be pretreated before receiving a field sample (EC, 1983; 1989). New glass and most plastics must be pretreated to remove residues, and/or leachable compounds, and to minimize potential sites of adsorption. Pretreatment includes the sequence of activities detailed in Table 8. For example, the rinses of organic solvent will remove most of the adsorbed organic compounds. Solvent rinses have been demonstrated to be as effective as combustion at 350° C for the removal of organic compounds (EC, 1989). The acid bath will leach trace metals (e.g., Cu, Fe, Mo, Ni, Zn) from plastics. The triple rinse with distilled water is necessary because the acid treatment can activate adsorption sites on polymers which are then capable of binding trace metals in the field sample.

Ideally, if samples are to be stored at 4° C then sample containers should be filled to the rim and air excluded during capping. If samples are to be frozen for storage then glass containers should not be filled completely. A space of approximately 2.5 cm should be left to accommodate expansion of the sample when frozen. The headspace in the container should be purged with nitrogen before capping tightly. Clear glass containers are often wrapped tightly with an opaque material (e.g., clean aluminum foil) to eliminate light and reduce accidental breakage.

Each sample container must be properly labelled and stabilized in an upright position in the transport container. Labelling of each sample container must include, as a minimum, the site, station location or identification, the sample type, the method of collection, the name of the collector, and the date and time of collection.

2.8.2 Storage of Samples in the Field

There are three ways to temporarily store field-collected samples, before they are transported to the laboratory. They can either be stored in refrigerated units on board the sampling vessel, placed into insulated containers containing ice or frozen

Table 7 Type of Container and Conditions Recommended for Storing Samples of Sediment or Pore Water

End Use	Container Type	Wet Weight or Volume of Sample	Storage Conditions	
			Temperature	Holding Time
SEDIMENT				
Particle Size Distribution	• Teflon® • Glass • High-density polyethylene containers or bags	250 g	4 to 40° C Do not freeze	≤ 6 mo
Major ions and elements: Al, C, Ca, Cl, Cr, Fe, Fl, H, K, Mn, Mg, Na, P, S, Si, Ti, (oxides and total)	• Teflon® • High-density polyethylene containers or bags	250 g	4 ± 2° C	≤ 2 wk
Nutrients: NH_4-N, NO_3-N, TKN, TC, TOC	• Teflon® • Glass with Teflon® Polyethylene-lined cap	100 g	4 ± 2° C	≤ 48 h
Trace elements: Ag, Ba, Be, Cd, Co, Cr, Cu, Hg, Li, Mn, Mo, Ni, Pb, Sb, Sr, Va, Zn	• Teflon® • High-density polyethylene containers or bags	250 to 500 g	4 ± 2° C or -20° C	≤ 2 wk ≤ 6 mo
Organic contaminants	• Stainless steel canisters • Aluminum canisters • Amber glass with aluminum-lined cap	250 to 500 g	4 ± 2° C or -20° C	≤ 2 wk ≤ 6 mo
Sediments for toxicity tests where the suspected contaminants are metals	• Teflon® • Glass • High-density polyethylene bags or containers	1 to 3 L	4 ± 2° C	≤ 6 wk preferably ≤ 2 wk
Sediments for toxicity tests where the suspected contaminants are organic(s)	• Glass with A1- or polyethylene-lined caps • Teflon® • Stainless Steel • High-density polyethylene bags or container	1 to 3 L	4 ± 2° C	≤ 6 wk preferably ≤ 2 wk
Control and reference sediment for toxicity tests	• Teflon® • Glass • High-density polyethylene bags or containers	>15 L	4 ± 2° C	≤ 12 mo[1]

Table 7 Type of Container and Conditions Recommended for Storing Samples of Sediment or Pore Water (Cont'd)

End Use	Container Type	Wet Weight or Volume of Sample	Storage Conditions	
			Temperature	Holding Time
PORE WATER				
Major ions and elements: Ca, Mg, Cl Si, Fl, Na, SO_4, K, Al, Fe, acidity, alkalinity	• Teflon® • Amber glass with Teflon®-lined lids • High-density polyethylene containers	40 mL	- 20° C	≤ 6 wk
Nutrients in pore water: NH_4-N, NO_2-N,NO_3-N, C (Total organic), P (soluble reactive), DIC, DOC, P (total)	• Amber glass with Teflon®-lined lids	40 mL	- 20° C or	≤ 6 mo
	• Amber glass with Teflon®-lined lids	40 mL	- 20° C or 4 ± 2° C with 1 mL of 30% H_2SO_4 per 100 mL	≤ 6 wk ≤ 2 wk
Trace Elements (total) in pore water: Ba, Be, Cd, Cr, Cu, Co, Li, Mn, Mo, Ni, Pb, Sb. Sr, Va, Zn	• Teflon® • Polyethylene	10 to 250 mL	- 20° C or 4 ± 2° C with 2 mL of 1 *M* HNO_3 per 1000 mL pore water	≤ 6 mo ≤ 6 wk
Ag	• Amber Polyethylene	250 mL	4 ± 2° C with 1 g Na_2--EDTA per 250 mL pore water	≤ 6 wk
Hg	• Teflon® • Glass (Soviral/Wheaton)	100 mL	4 ± 2° C with 1 mL H_2SO_4 per 100 mL of pore water	≤ 6 wk
Organic contaminants in pore water[2]:	• Amber glass with A1-lined caps • Amber glass with Teflon®-lined caps	1000 mL	- 20° C or 4 ± 2° C acidified with H_2SO_4 or with the addition of 10 g Na_2SO_4 per L of pore water	≤ 6 mo ≤ 6 wk

Table 7 Type of Container and Conditions Recommended for Storing Samples of Sediment or Pore Water (Cont'd)

End Use	Container Type	Wet Weight or Volume of Sample	Storage Conditions	
			Temperature	Holding Time
Organochlorine and PCBs	• Amber glass with A1-lined caps • Amber glass with Teflon®-lined caps	1000 mL	- 20° C or 4 ± 2° C	≤ 6 mo ≤ 6 wk
Organophosphates	• Amber glass with A1-lined caps • Amber glass with Teflon®-lined caps	1000 mL	- 20° C or 4 ± 2° C acidified with HCl to pH 4.4	≤ 6 mo ≤ 6 wk
PCP	• Amber glass with A1-lined caps • Amber glass with Teflon®-lined caps	1000 mL	-20° C or 4 ± 2° C acidified with H_2SO_4 to pH <4 or preserved with 0.5 g $CuSO_4$ per litre of pore water	≤ 6 mo ≤ 6 wk
Phenoxy acid hebicides	• Amber glass with A1-lined caps • Amber glass with Teflon®-lined caps	1000 mL	-20° C or 4 ± 2° C with acidification to pH <2 with H_2SO_4	≤ 6 mo ≤ 6 wk
PAHs	• Amber glass with Al-lined caps • Amber glass with Teflon®-lined caps	1000 mL	-20° C or 4 ± 2° C	≤ 6 mo ≤ 6 wk
Pore water[3] or Elutriate for toxicity tests	• Amber glass with Teflon®-lined caps	1 to 3 L	4 ± 2˚ C	≤ 72 h

[1] These sediments should be monitored over this period of time to ensure that changes that might occur to the physicochemical characteristics are acceptable.

[2] It is very difficult to collect sufficient pore water for analyses of volatile organic compounds and aromatic organic compounds.

[3] It is very difficult to collect sufficient pore water for standard toxicity testing; however, smaller, quantities will suffice if the experimental design of the test accommodates extraction of successive samples of sediment and/or compositing of within-station replicate samples. It should be recognized that once pore water that has been collected *in situ* is exposed to oxygen (e.g., air) it becomes geochemically distinct (Mudroch, 1992).

Table 8 Recommendations for the Pretreatment of Containers for Samples of Sediment or Pore Water[1]

SEDIMENT SAMPLES:

Inorganic Contaminants
- Scrub with phosphate-free soap and hot water
- Rinse with high-pressure hot water
- Subject to a 72-h acid bath with 8 *M* HNO_3 per litre)
- Rinse four (4) times with hot water
- Rinse three (3) times with DDW
- Wash bottle Teflon® or Teflon®-lined caps with soap and hot water, and rinse with DDW

Organic Contaminants
- Scrub with phosphate-free soap and hot water
- Rinse with high-pressure hot water
- Subject to a 72-h acid bath with 8 *M* HNO_3 (50 mL of HNO_3 per litre)
- Rinse four times with hot tap water
- Rinse three times with DDW
- Rinse twice with acetone[2] (pesticide grade)
- Rinse twice with petroleum ether[2]
- Evaporate solvents in fumehood[2]
- Rinse aluminum foil (or Teflon® lining) twice with acetone, and twice and with petroleum ether, and let dry in fumehood
- Wash bottle caps with soap and hot water, and rinse with DDW
- Cut aluminum foil (or Teflon® lining) with acetone-washed scissors
- Use cleaned alumnium foil (or Teflon® lining) between cap and bottle

PORE WATER SAMPLES

Inorganic Contaminants/Trace Metals
- Wash lids and bottles with hot water, phosphate-free soap, and scrub brush
- Rinse twice with hot water
- Triple-rinse with distilled deionized water (DDW)
- 72-h acid bath in 8 *M* HNO_3 (50 mL of HNO_3 per litre)
- Triple-rinse with DDW

Non-volatile Organic Contaminants
- Scrub with phosphate-free soap and hot water
- Rinse with high-pressure hot water
- Rinse three (3) times with DDW
- Rinse twice (2) with acetone[2] (pesticide grade)
- Rinse twice (2) with petroleum ether[2]
- Evaporate solvents in fumehood[2]
- Rinse aluminum foil (or Teflon® lining) twice with acetone and twice with petroleum ether and let dry in fumehood
- Wash bottle caps with soap and hot water, and rinse with DDW

- Cut aluminum foil (or Teflon® lining) to size with acetone-washed scissors
- Use cleaned alumnimum foil (or Teflon® lining) between cap and bottle

Volatile Organic Contaminants

- Scrub with phosphate-free soap and hot water
- Rinse with high-pressure hot water
- Rinse three (3) times with DDW
- Rinse twice (2) with acetone[2] (pesticide grade)
- Rinse twice (2) with hexane[2] (pesticide grade)
- Evaporate solvents in a fumehood[2]
- Wash Teflon® septum with soapy water
- Rinse thoroughly with hot water
- Rinse four to five times with DDW
- Replace cleaned septum after each use
- Ensure that the Teflon® side (white) of the septum is face down

---

[1] Adapted from EC (1989)

[2] Alternatively, heat-resistant bottles and lids can be baked at 350° C to combust residues of organic solvent

ice-packs, or taken immediately to a local storage facility where they can be either frozen in a freezer or placed into a refrigerator. The storage conditions recommended in Table 7 apply to temporary storage as well. However, where these conditions cannot be met due to operational constraints, then the temporary storage method and conditions adopted should strive to compromise the integrity of sample as little as possible (Mudroch and MacKnight, 1991). If samples are to be frozen and a freezer is not readily available, the use of dry ice is recommended as long as its efficacy is known and the user is aware of regulations that accompany the transportation of samples stored in this manner. Sediment samples for toxicity testing must not be frozen. While in transit to a temporary storage facility or to the laboratory, frozen samples should not be thawed and, the temperature of unfrozen samples should not exceed 7° C nor fall below 1° C. Sediment samples that were collected for legal purposes should be accompanied by the appropriate chain-of-continuity documents.

2.8.3 Transport Conditions and Regulatory Considerations

Recommended Procedures and Conditions

- The transport container should be refrigerated to 4 ± 2° C or contain ice or frozen gel packs that will keep the field samples between 1 and 7° C during transport to the laboratory.

- If field-collected samples are warm (e.g., 7° C), they should be cooled between 1 and 7° C with ice prior to placement in the transport container.

- Samples must not freeze during transport.

- A maximum/minimum thermometer or a continuous temperature recorder should be placed inside the transport container and the container sealed. Deviations in temperature should be reported.

- Light should be excluded from the transport container.

- All field-collected samples that require further processing before storage must be transported to the laboratory within 72 h, preferably within 24 h, of collection.

In addition to this information, the following constraints apply to the handling, storage, and transport of «legal samples»:

- Procedures for preparing the sample containers should be documented, together with the name of the person responsible.

- All transport containers must remain locked during transport to and from the site, and never left unattended.

- Field notes documenting the handling and sampling procedures for each sample, and additional relevant data (e.g., location and description of sampling station, water temperature, level or flow rate, weather, sample collection or handling technique, photographs, identification numbers, etc.), must be made and initialled by the person responsible for collection.

- The name and signature of the person who collected the sample must be placed on each sample container and witnessed (co-signature).

- The label should be securely fastened to the bottle after the sample has been placed into the container and the lid tightly secured.

- For samples that have legal implications, the appropriate completed chain-of-continuity papers and Ministry of Transport documents must be completed and accompany the transport containers with the field samples. Each field-sample container must have a tape seal to demonstrate that it has not been opened during transport, and the transport container must be locked during pickup, transit, and delivery.

- A description of the nature and origin of the sample, and its designated end use, should be included in the shipment, and a separate copy sent to the laboratory.

- The transport container should be labelled properly; including a description of the contents, the destination, any special handling instructions, and phone numbers to call on arrival or in the case of an emergency.

- The appropriate documentation for the legal transfer of the sample(s) must accompany the transport container and chain-of-continuity documented (i.e., every time the package changes hands the transfer of responsibilities must be documented with names and signatures).

- A file for all documentation, including signed package slips, registration slips and waybills, and photographs of the transport container before transport and upon arrival at the laboratory should be established.

- Laboratory notes must be made regarding the condition of the samples.

- Samples must be kept in a locked area with restricted access.

- All documentation of the analytical procedures and results should be kept on file and in the control of the QA/QC officer.

- All archived samples must be stored appropriately in sealed containers in a locked area with restricted access.

2.9 Manipulation of Collected Samples

One of the major goals associated with the collection and storage of sediment and pore water is to maintain the integrity of the samples until their chemical characteristics and/or toxicity are evaluated, by minimizing changes to the physical, chemical, and biological properties of the samples and subsamples that might result from transportation and storage. Evaluation methods usually require the preparation of test samples form stored field samples. All manipulations and procedures used to prepare the test samples can potentially alter the nature of the field samples. Manipulation procedures, therefore, are intended to prepare or process field samples so that they remain as representative as possible of the sediment at the site of collection, in terms of its chemistry and toxicity, while making the sample more amenable for biological testing or characterization (Nelson *et al.,*1991; 1992).

Sample preparation may involve physical operations such as sieving, drying, freeze-drying, crushing, grinding and/or chemical treatment steps such as dissolution, extraction, digestion, fractionation, derivatization of some of the organic contaminants, pH adjustment, and the addition of reagents (Keith, 1992). These sample preparations, although necessary, are also potential sources of bias, variance, contamination, chemical alteration, and sediment loss. Therefore, sample preparation must be included in the study plan and fully documented to give a complete history of the transformation from a field-collected sediment to a test sediment. Schematics for preparation of test sediment are presented in Figures 6 and 7, together with the major procedures or manipulations.

2.9.1 Preparation of Sediment Sample for Biological Tests (Toxicity or Bioaccumulation)

Field-collected sediment samples may be delivered to the laboratory as intact core or grab sediment samples, or samples that have already been subsampled, sectioned, composited and/or homogenized, or some combination of these, depending on the study plan. Whole-sediment samples that have been collected and placed directly into field-sample containers and transported to the laboratory should be prepared for use in toxicity tests, as soon as possible following their arrival. The manipulations that apply to whole sediments to be used in biological tests are described in Figure 6.

Methods for the Removal of Indigenous Organisms

Recommendation

- Removal of indigenous macrofauna from sediments can be achieved by hand picking or pressure sieving freshwater, estuarine, and marine sediments.

- The mesh size should be chosen in consideration of the toxicity test and test organisms, potential predators and/or competitors present, and the nature of the sample (e.g., particle size, quantity, and size of debris).

It may be desirable to remove indigenous organisms from the best sediments that interfere directly (e.g., predators) or indirectly (e.g., competitors) with the test organisms (Redmond and Scott, 1989; Ingersoll and Nelson, 1990). For example, amphipod or chironomid test results using freshwater sediments containing tubificid

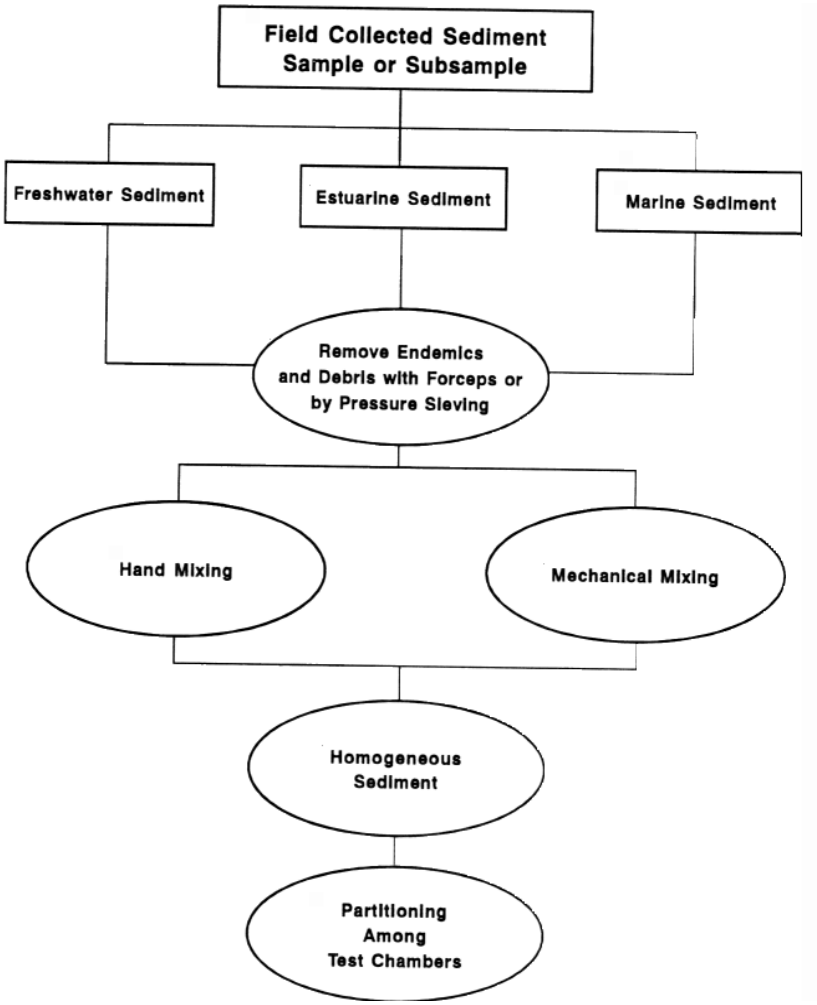


Figure 6 Preparation of Test Sample for Whole-sediment Solid-phase Toxicity Testing (manipulations are circled)

worms, or marine sediments containing polychaete worms, will differ significantly from test results using sediments free of worms (Redmond and Scott, 1989; Reynoldson *et al.,*1994). A number of removal methods (Table 9) have recently been investigated (Day *et al.,*1995); however, the effects of these methods on the physiochemical and biological characteristics of the sediment are largely unknown. The methods are presented in the table in the order that they are recommended (i.e., most desirable to least desirable based on the little data available).

Freezing has been used to kill indigenous organisms in sediments and inhibit microbial activity; however, dead organisms must be removed to prevent the sediments from becoming anoxic. Chemosterilants (e.g., antibiotics, formalin, methylmercury, mercuric chlorides, sodium azide) have been added to the sediments to remove, inhibit, or kill biota. Autoclaving sediments and gamma irradiation have also been used to kill biota naturally occurring in sediments. Sieves with a mesh size of 0.25 mm will remove the macrofauna from freshwater sediments (Day *et al.,*1995); however, the

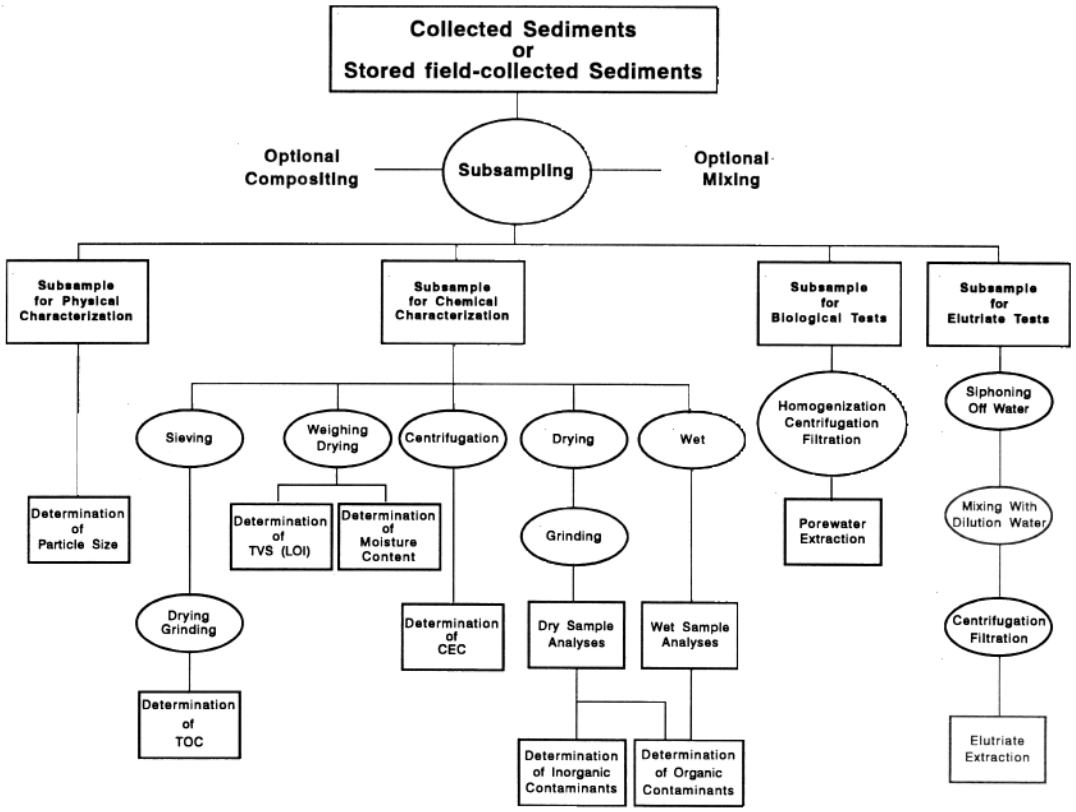


Figure 7 Preparation of Test Sediments for Physicochemical Characterization and for Pore Water and Elutriate Extraction (manipulations are circled).

microfauna will persist. Sieves with a mesh size of 0.5 mm will remove most of the immature amphipods of *Rhepoxynius abronius* and *Corophium spinicorna* from marine sediments (Swartz *et al.,* 1990). Gamma irradiation is probably the most promising method because evidence to date suggests that it causes the least alteration in both the physical and chemical characteristics of the sediment (Day *et al.,* 1995). However, there is little published information on the effects of gamma irradiation on the toxicity of contaminants in sediments. Picking with forceps (i.e., "hand picking") is the recommended technique for removing organisms from sediments that are to be used in toxicity tests. Pressure sieving without the addition of water can be used when "hand picking" is not practical. The other procedures presented should not be used for the removal of indigenous organisms from sediments that are to be used in toxicity tests.

Methods for Sieving Sediments

Recommended Procedures

- Before sieving, the sediment sample should be placed onto a sorting tray made of an appropriately inert material, at this time, large rocks and other debris should be removed by hand.

- If the surficial sediments are anoxic, and it is desirable to retain this condition, all preparation of the sediments should be

Table 9 Methods Used to Sterilize Sediments, Remove, Inhibit Growth, and/or Kill Organisms in Sediments

Method	Comment	Reference
Handpicking	• most common method to remove organisms that are visible and easily captured by pipette or tweezers	
Sieving	• promotes homogenization of sample • 1.0-mm mesh will remove most of the adult amphipods; • 0.25-mm mesh will remove most of the immature amphiphods and most macrofauna; however, microfauna will persist	Landrum *et al.,* 1992 Robinson *et al.,* 1988 Day *et al.,* 1995
Gamma irradiation	• unknown effects on most contaminants • efficacy yet to be demonstrated adequately • insufficient data • appears to have minimal disruption to the physical and chemical characteristics of the sediment • *H. azteca* cannot survive in irradiated sediments • alters the "structure" of the sediment	Day *et al.,* 1995
Freezing	• kills organisms and inhibits microbial activity • dead organisms must be removed; decomposition processes can result in anoxic conditions • alters the "structure" of the sediment	Day *et al.,* 1995
Autoclaving	• common method though variable and not standardized • loss of volatile substances • disrupts both the physical and chemical characteristics of the sediment • *H. azteca* cannot survive in autoclaved sediments	ASTM, 1992a Hood and Ness, 9182 Burton, 1992 Day *et al.,*1995
Chemosterilants: mercuric chloride sodium azide	• mercuric chloride is more effective as a bacteriocide than sodium azide • interference with contaminants in sediments	ASTM, 1992a
Antibiotics: streptomycin ampicillin	• they readily bind to organic matter • labile • light sensitive	Burton *et al*., 1987

done in a glove box. In some cases modifications to the glove box may be necessary to accommodate these procedures.

- If it is necessary to sieve marine, estuarine, or freshwater sediments (sieving should be avoided if possible), the mesh size should be chosen in consideration of the toxicity test and test organism, potential predators and/or competitors present, and the nature of the sample (e.g., particle size, quantity and size of debris).

- The same criteria for selecting sample containers (Section 2.8) should be considered when choosing a device for sediment sieving, to mitigate or minimize contamination and adsorption. The use of brass sieves is discouraged.

- All pressure sieving of sediments without the addition of water should be performed in a well-ventilated area (e.g, fumehood). Only the liquid that has been separated from the sample during transport and storage should be used during the sieving process; no water should be used.

- If sediments are sieved, it is recommended that the physicochemical properties of the sediment be determined before and after sieving to document changes attributable to sieving. In some cases, comparative toxicity tests using sieved and unsieved sediment might be necessary to discern the effect of sieving on the toxicity of a sediment.

Sediments to be used in toxicity evaluations should be microscopically examined for the presence of interfering endemic species and sieved only when it is necessary (i.e., when hand-picking methods are either impossible or ineffective). The investigator should be aware that sieving could alter the concentration or bioavailability of contaminants in the sediment by removing (e.g., coarse and medium sand particles) or concentrating substances, disrupting chemical equilibrium (e.g., increased volatilization, sorption, and desorption), or by changing the biological activity within the sediment. Therefore, every effort should be made to pick out organisms and large particles (e.g., rocks and debris) using tweezers before resorting to sieving. If sediments are sieved, it is recommended that the physicochemical properties of the sediment be documented before and after sieving.

When sieving sediments is inevitable, pressure sieving is preferable to wet sieving. Pressure sieving involves the mechanical pressing of sediment particles through a sieve with a specified mesh size (Giesy *et al.,* 1990; Johns *et al.,* 1991). It is an acceptable method for sieving sediments. However, it is recognized that pressure sieving sediments with debris, vegetation, or a high clay content through a single mesh size less than 1 mm is very difficult. Therefore, a series of sieves might be required.

Wet sieving has often been used to remove from sediment indigenous organisms whose presence may compromise test results (Pastorok and Becker, 1990; Stemmer *et al.,* 1990b); however, this practice is recommended only when pressure sieving is not possible. Wet sieving involves agitating or swirling the sieve with the sediment in water that has separated from the sediment during storage or transport so that the particles smaller than the selected mesh size are washed through the sieve into a dish. The liquid must be remixed with the sieved sample. To facilitate the process, the sieve

can be agitated (e.g., mechanical shaker) or the sediments on the screen stirred with a nylon brush (Mudroch and MacKnight, 1991). No new water should be added to the sample in the process of sieving a sediment. Although the use of wet-sieved sediments is strongly discouraged, it might, in some instances, be the method that is least disruptive to the toxicity evaluation process (Day, 1993). Until additional research on the usefulness of alternative methods is available, pressure sieving, or wet sieving where sediments cannot be pressure sieved, are the methods recommended only when removal of endemic species with forceps is either impossible or ineffective.

Sediments used in toxicity tests have been sieved with stainless steel, brass, or plastic woven polymer sieves (e.g., polyethylene, polypropylene, nylon, and Teflon®) with mesh sizes that vary from 0.24 to 2.0 mm (Keilty *et al.,* 1998a,b,c; Giesey *et al.,*1990; Lydy *et al.,*1990; Pastorok and Becker, 990; Stemmer *et al.,*1990a,b; Johns *et al.,*1991; Landrum and Faust, 1991). The most frequently used mesh size is 1.0 mm; however, no comparative data were found that demonstrated: a) the effect of the different types of mesh available; or b) that 1.0-mm mesh was the optimum mesh size. Recent comparative toxicity data have demonstrated that the optimal mesh size for removing indigenous organisms from freshwater sediments was 0.25 mm (Day *et al.,*, 1995) which effectively removed most of the macrofauna from the sediments and promoted homogenization. Swartz *et al.,* 1990) demonstrated that, for marine sediments used in amphipod toxicity tests with *Rhepoxynius abronius* (Barnard), an adequate mesh size was 0.5 mm. If sediments cannot be sieved by pressure, wet sieving may suffice; however, the effect of potentially ameliorating toxicity should be recognized.

Methods for Homogenizing Sediments

Recommended Procedures

- Mixing by hand or mechanical mixing may be used to achieve homogeneity of colour, texture, and moisture; however, the efficacy of the method must be demonstrated, *a priori,* and the mixing time standardized and minimized to ensure consistency and to minimize changes to the size distribution of sediment particles, respectively.

- Mixing of sediments should take place in the sample/storage container or transfer sample to a clean mixing container.

- Coning or caking and quartering is the recommended technique for partitioning the sediment for distribution among test containers. If a sediment splitter is used, its efficacy must be demonstrated and documented and it must be made of an appropriately inert material.

Following hand-picking or sieving to remove debris and organisms, the sediment must be mechanically homogenized until uniform in colour and texture. This can be accomplished with a mechanical mixer (Ditsworth *et al.,*1990; Stemmer *et al.,* 1990b; Kemble *et al.,*1993), although hand mixing is more common (Malueg *et al.,* 1986; Clark *et al.,*1987; Burton *et al*., 1989; Ingersoll and Nelson, 1990; Pastorok and Becker, 1990; Carr and Chapman, 1992; Johns *et al*., 1991). Hand mixing can be performed by blending with a spatula, rolling the sediment out flat on a sheet of plastic or pre-combusted foil and tumbling

by raising each corner of the sheet in succession, or by coning or caking, and quartering (Mudroch and MacKnight, 1991). Regardless of the mixing method (i.e., mechanical or by hand) the efficacy of the method should be demonstrated and the mixing time standardized to ensure consistency (Ditsworth *et al.,*1990; Stemmer *et al.,*1990a,b). It is recommended that mixing of the sample take place in the sample or storage container (Ditsworth *et al.,*1990). However, if this is not possible, the sample should be transferred to an appropriate mixing container, selected according to Table 7, and mixed thoroughly. The mixing time required to achieve a homogeneous mixture should be minimized because prolonged mechanical mixing can alter the particle-size distribution in a sample. Oxidation of the sediments can also occur with prolonged mixing. Sandy sediments with significant amounts of macrofauna have gone markedly anoxic in test containers, whereas unmixed sediments remained oxic (Hickey, 1994). This was attributed to the physical breakup of organisms and their subsequent degradation. The problem was overcome by either hand-picking or sieving the organisms from the sediments before mechanical mixing.

After the field-collected sediment has been homogenized, it must be partitioned among the test containers. The volume of sediment to be placed in each test container is dictated by the test protocol. The coning or caking, and quartering technique is recommended for partitioning the test sediment. This could involve placing the sediment in a conical or cake shape in the centre of a pan or rolling sheet. The sediment cone or cake is then either quartered with a special quartering tool or flattened into a circular shape (e.g., cake) with uniform dimensions and divided into four quarters. The opposite quarters are removed and combined. The split sample is then repeatedly mixed, coned or caked, and quartered until the desired volume of test sediment is achieved. The sediment is then randomly allocated to the test containers. For sediments with a high water content, it may be more appropriate to use a mechanical splitter (e.g., splitter box) specifically designed to generate consistent subsamples of equal volume.

Ideally, the volume of homogenized sediment from a single grab sample should be sufficient to execute a toxicity test. However, depending on the objectives of the study and method of sediment collection, this may not always be the case. If within-site variability is not a concern, the sediments from multiple grabs may be hand-picked, combined either during pressure sieving, if sieving is desirable, or during homogenization, and placed into the test containers. However, if within-site variability is of concern, then the sediments from each grab sample must be kept separate, processed appropriately, and then placed into separate replicate test containers.

2.9.2 Preparation of Test Sample for Chemical Characterization

Ideally, sediment characteristics that are unstable (e.g, pH, oxidation-reduction potential) or changed by conditions of transit and storage (e.g., temperature) should be measured in the field to help characterize the sample. In the laboratory, each sample of field collected sediment should be thoroughly mixed (Section 5.3), and representative subsamples taken for physicochemical characterization. The sample should then be characterized by analyzing the subsamples for at least the following (USEPA, 1994a):

whole-sediment particle size distribution (percentage of sand, silt, and clay), percent water content, and total organic carbon content; and pore water pH and ammonia. Other analyses could include (USEPA, 1994a): total inorganic carbon, total volatile solids, biochemical oxygen demand, chemical oxygen demand, cation exchange capacity, acid volatile sulphides, metals, synthetic organic compounds, oil and grease, petroleum hydrocarbons, and porewater analyses for various physicochemical characteristics. Unless indicated otherwise, identical chemical, physical, and toxicity analyses should be performed with subsamples representative of each replicate sample of field-collected sediment (including reference sediment) taken for a particular survey of sediment quality, together with a subsample of control sediment.

The contaminants to be monitored in sediments will depend to a large extent on the objectives of the study and on the current and historical information regarding potential source(s) of contaminants in the study area. The most common constituents measured in sediment are silicates, nitrates, phosphates, major ions, total organic carbon, and ammonium. The most common inorganic contaminants measured in sediment include total and ionic forms of arsenic, cadmium, mercury, copper, chromium, nickel, lead, zinc, cobalt, iron, cyanide, and aluminum. The most common organic contaminants include both volatile and semivolatile compounds containing hydrocarbons with, and without, halogen substituents (e.g., trihalomethanes, benzene, toluene, ethylbenzene, xylene, and environmentally persistent pesticides such as phenoxy acids, organochlorines, cyclodienes), chlorinated, neutral, base and acid extractables [(including polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs)], dioxins, furans, phenols and total petroleum hydrocarbons (e.g., oil and grease). The contaminants to be analyzed should be chosen in light of the available historical data.

The selection criteria to determine the list of analytical parameters, the instrumental requirements, and analytical methods should be decided *a priori* and included in the study plan. Evaluation of the historical data and potential sources of contaminants could provide adequate insight into the levels and types of contaminants present. However, where these data are lacking, or are inadequate, it could be necessary to perform an initial full-analyte scan to focus attention on target contaminants which will then be analyzed routinely and quantified. It is important to analyze replicate samples to determine the variance within a sediment sample and the variance associated with the analytical method. Preparation procedures for replicate samples must be identical.

An analytical QA/QC program must address the variance in both sediment characteristics and analytical methods. Environment Canada promotes the use of a data quality objectives (DQO) approach to sediment contaminant analysis. These DQO are statements of the level of uncertainty that is acceptable in results derived from environmental data, especially when the results are going to be used in a regulatory or program decision. Environment Canada also promotes the use of Minimum Performance Standards (MPS) and, as long as the analytical methodology satisfies the DQO and the MPS, it is acceptable.

Sediment Drying. Drying a sediment subsample might be a prerequisite to, or part

of, chemical characterization (e.g., TOC determination). Sediment samples can be air-, oven-, or freeze-dried. Oven drying at low temperatures (40 to 60° C) or freeze-drying sediment samples are the recommended methods for samples of sediment. The sediment subsamples are placed in a laboratory drying oven in clean, inert crucibles or trays (e.g., porcelain, aluminum, pyrex glass) that have been pretreated (e.g., baked) to remove carbon. The oven must be preset to the desired constant temperature, and have good air circulation and accurate temperature control with automatic shut-off temperatures. The samples must be dried to a constant weight. Drying times and temperatures which depend primarily on the volume of the sediment sample have ranged from 4 to 48 h and 50 to 110° C, respectively (Plumb, 1981; EC, 1985; Loring and Rantala, 1992). The objective is to dry the sample of sediment as rapidly as possible without contaminating the sample and minimizing biological and chemical oxidation losses. Oven drying sediment subsamples at 60° C for 16 h is recommended (Centre St-Laurent, 1992). It should be noted that drying sediment before analyses can result in losses of organic compounds and reduced extraction efficiencies (Windsor and Hites, 1979; Haddock *et al.,*1983). Both drying methods can, in marine sediments, result in the retention of soluble sea salts that interfere with some major element analyses and prevent some measurements of physical structure (Loring and Rantala, 1992).

Sediment Crushing/Grinding. Crushing or grinding a sediment subsample might be necessary for chemical characterization. These manipulations are performed to disaggregate the dried sample and ensure homogenization. Crushing is generally achieved by use of a mortar and pestel. Commercially available ball and pebble mills are recommended for fine-grinding small volumes of sediment (Mudroch and MacKnight, 1991); however, it should be noted that grinding could change the chemistry of the material.

2.9.3 Preparation of Test Sample for Collection of Pore Water

This section deals with the separation of pore water from collected sediments; the recovery of pore water with *in-situ* sampling devices has been previously addressed in Section 2.6.

Recommended Procedures

- Centrifugation is the recommended method for the collection of pore water.

- It is important to maintain an oxygen-free atmosphere during the extraction phase. This task is much simpler using centrifuge tubes or bottles rather than sediment-squeezing devices. The centrifugation procedure is relatively simple, although it has the disadvantage of collecting only 20 to 30% of the water present in the interstitial spaces depending on the type of sediment.

- Teflon®, Corex glass, polycarbonate, or stainless steel (where organic chemicals are the contaminants of concern) centrifuge tubes should be used.

- Extraction of pore water by centrifugation methods should be completed as soon as possible (i.e., preferably within 24 h of sample collection).

- To prepare the sample for extraction of pore water, place 1-L subsamples from the homogenized sediment sample into centrifuge bottles. Generally 1 L of sediment will yield about 400 mL of pore

water. If smaller volumes of pore water are required then smaller volumes of sediment may be extracted.

- Water from the interstitial spaces might accumulate on the surface of the sediment sample during storage. This overlying water should be mixed into the sediment before it is partitioned among the centrifuge bottles.

- Centrifuge at 10 000 × G and 4° C for 30 minutes using a large-capacity centrifuge equipped with refrigeration.

- Carefully decant the supernatant (i.e., the pore water) and place in a clean glass container.

- The pore water should be analyzed or used in biological tests immediately, or as soon as possible after extraction. It should be stored at 4 ± 2° C for not longer than 24 h, unless the test method dictates otherwise.

- Low-pressure filtration at low speeds might be required for some tests. Filtering pore water is strongly discouraged because it might reduce pore-water toxicity. Double centrifugation, first at a low speed then at a high speed may remove particles from pore water in place of filtering. If it is absolutely necessary (i.e., stipulated in the biological test method) to filter the pore water, treated filters should be used and filter blanks must be included to assess adsorption and desorption of potential toxicants to and from the filter, respectively (Lam, 1967; Levi and Novicki, 1972; Novicki *et al.,*1979; Carr and Chapman, 1995).

- If analysis of pore water for trace metals cannot be done immediately, the samples may be acidified and stored for up to six weeks (Table 7).

Squeezing and centrifugation are the two most common techniques for collecting pore water, from sediment samples in the laboratory. Other methods to collect pore water include desiccation, vacuum filtration, leaching, and displacement (Adams, 1991). The advantages and disadvantages of three porewater collection methods (centrifugation, vacuum suction, and pressurized squeezing) are summarized and discussed in detail by Carr and Chapman (1995). Squeezing and centrifugation methods are generally preferred when large volumes of pore water are required (ASTM, 1992a). Centrifugation generally yields greater volumes of pore water than squeezing (Saager *et al*., 1990). Details regarding these manipulations are presented in Appendix F.

2.9.4 Preparation of Test Sample for Collection of Elutriate

Recommendation

- The sediment sample (which has been stored at 4° C in air-tight containers) for collection of an elutriate, must be homogenized before transferring subsamples to centrifuge bottles. This will ensure that the overlying water is mixed into the sediment.

- Determine the mean weight of replicate (e.g., ten) centrifuge bottles.

- Transfer subsamples of sediment (50 to 200 g) to the clean centrifuge bottles (250 or 1000 mL capacity, respectively) and add dilution water in a ratio of approximately 1:4 sediment to water.

- The quantities of sediment and water must be measured individually in tared bottles to ensure that the weights of the bottles are equal for centrifuging.

- Place the centrifuge bottles, containing the appropriate weights of water and sediment, in an elutriate-mixing apparatus, and rotate end-over-end for 30 minutes at 12 rpm at 4° C (Cleveland *et al*., 1995).

- Weight the bottles before placement into the centrifuge to ensure the weight of all of the bottles is within ± 0.20 g of each other. If necessary add sufficient water to equilibrate the weights.

- Centrifuge the bottles at 10 000 × G for 10 minutes at 4° C.

- Remove the elutriate carefully from each bottle, filter only if necessary (see below), and either pool then subsample and transfer to appropriate containers, or simply transfer elutriate directly to test vessels without compositing.

- The elutriate should be analyzed or used in biological tests immediately, or as soon as possible thereafter. It should be stored at 4° C for not longer than 24 h, unless the test method dictates otherwise.

- Filtering the elutriate is discouraged; however, it may be required for some test methods. If the elutriate is filtered then only pre-treated filters should be used and discard the first 10 to 15 mL of elutriate though the filter. An assessment should be included to determine the extent of analyte adsorption or desorption to, or from, the filter.

The elutriate method was initially developed to assess the effects of dredging operations on water quality (USEPA, 1979), but it is applicable to any situation where the resuspension of sediment-bound toxicants is of concern (ASTM, 1992a). Elutriate tests are not designed to measure the toxicity of pore waters or bedded sediments. Elutriates have been found to be both more toxic (Hoke *et al*., 1990), as toxic (Flegal *et al.*, 1994), and less toxic (Ankley *et al*., 1991c) than pore water, primarily because of differences in toxicant bioavailability in two types of media (Harkey *et al*., 1994). The aqueous extracts of whole sediment might not accurately represent the exposure observed in whole sediment (Harkey *et al.*, 1994); therefore, sediment elutriate is not universally accepted as an appropriate test fraction to assess the toxicity of sediments. It is not considered acceptable as an assessment of bedded sediment toxicity but it may be indicative of toxicity during disposal of dredged material or where there is a frequent resuspension of very fine sediments due to strong winds (e.g., San Francisco Bay).

2.10 Summary Recommendations for Monitoring and Assessment Studies

Study Plan

- Clearly state the purpose, objective(s), or hypothesis of the study, and integrate them as an integral component of the study plan.

- Define the study area and the study site, and outline them on a hydrographic chart or topographic map. A physical inspection of the study area and proposed study site must be undertaken, and local expert(s) on site conditions should be consulted.

- Identify potential sources of contamination and plot their locations on a chart or map.

- Access, review, and evaluate all available historical data relevant to the study (Subsection 2.3.2).

- Determine (e.g., acoustic survey technique) and validate (e.g., diver or electronic surveillance, or with a preliminary sampling survey) the location of fine-grained sediments.

- Select a method for determining the location of the sampling stations (e.g., judgmental, random, stratified random, systematic, etc.). If the objective of the survey is to identify sites of toxic and/or contaminated sediments on a quantitative spatial and/or temporal basis, a systematic or regular grid-sampling strategy is the most appropriate sampling plan (Green, 1979; Atkinson, 1985).

- If the monitoring objective is to determine sediment contamination originating from a point source, the sampling pattern could be based on the assumption that concentrations decrease with distance from the source; thus, factors affecting dispersion of substances or materials from the point source (e.g., currents) must be considered. Therefore, in a river where the point source is an outfall, sampling stations should be located in zones of accumulation at fixed distances downstream, following a geometric progression (i.e., ×, 2×, 4×, 8× etc.) (Mudroch and MacKnight, 1991). Samples are usually collected concurrently from upstream locations that serve as reference or control sites.

- If the point source is within a waterbody where dispersion is not unidirectional, the sampling stations should be located in a concentric pattern at the intersection of fixed geometric distances and a specified angle or bearing.

- Decide on a positioning method that is most appropriate to the site and study objectives (Subsection 2.3.5).

- Decide on the number and the nature of the sample analyses (e.g., type of analytical tests).

- Determine the sample volume or weight required to satisfy the analytical methods and QA/QC program for all of the intended analytical tests (i.e., physical and chemical characterization, biological tests, etc.).

- Determine the frequency of sampling (i.e., how often or when samples should be collected) that is required to meet the study goals and objectives. If there is a seasonal component to the study, it should be included in the study plan. Consideration of sedimentation rates is critical when determining sampling frequency.

- Decide on the level of confidence and the acceptable size of effect required from the sample data, and determine the number of samples required to achieve these criteria as well as the field-sampling and the data-quality objectives of the QA/QC program (Subsection 2.3.6; Appendix C).

- If replicate samples from each sampling station are required, a minimum of five, replicate samples per station is recommended. However, the number of replicate samples may be determined *a priori* from preliminary sample collection and analyses (Subsection 2.3.6).

- The study plan, including the sampling design (i.e., the frequency, number, and location of field-collected samples, field measurements and observations), should be discussed with a statistician or other qualified professional, before the monitoring study begins.

- Disposal of wastes from the study should be addressed in the study plan.

Preparations for Field Sampling

- Careful preparation and planning should precede the sampling study and should include written protocols for logistical considerations and equipment checklists for each sampling activity. For considerations see Subsection 2.3.7.

- The sampling plan and projected time schedule should be posted so that all personnel are aware of what is expected and when. The study director or manager and should ensure that the appropriate personnel clearly understand their role and are capable of carrying out their assigned responsibilities and duties, and that the names, addresses, and telephone numbers of all participants involved with both the preparation and actual execution of the sampling program are available to all participants. Contingency planning should address backup personnel and equipment.

- The reagents for cleaning, operating or calibrating equipment, collecting, preserving and/or processing samples (e.g., handling and manipulation), should be the responsibility of the person in charge of health and safety and the appropriate Material Safety Data Sheets should be available.

- Waterproof and tear-resistant data forms and log books should be prepared in advance so that field notes and data can be quickly and efficiently recorded.

- All equipment used to collect and handle samples must be cleaned and all parts examined to ensure proper functioning (e.g., on-site assembly or operation) before going into the field. Repair kits and backup equipment and sampling gear should be taken into the field.

- Sample containers (Table 7) should be clean and/or pretreated, depending on the end use, before being taken to the field (see Table 8, Subsection 2.8.1). Extra containers should be prepared in the event of accident or breakage.

- During transport to the field, container lids should be secure and a label attached with a sample identification number and space to add the mandatory information (see Subsection 2.8.1).

- Travel blanks should be included as part of the QA/QC program (see Section 3.7).

Collection of Field Data, Sediment, and Pore Water Samples

Field Data

- sample number, replicate number, station number, site identification (e.g., name);

- time and date of the collection of the sample;

- ambient weather conditions, including wind speed and direction, wave action, current, tide, vessel traffic, temperature of both the air and water, thickness of ice if present;

- station location (e.g., positioning information) and location of each replicate sample;

- type of vessel used (e.g., size, power, type of engine);

- type of sediment collection device and any modifications made during sampling;
- the water depth at each sampling station and the sediment sampling depth name of personnel collecting the samples;
- details pertaining to unusual events which occurred during the operation of the sampler (e.g., possible sample contamination, equipment failure, unusual appearance of sediment integrity, etc.);
- description of the sediment including texture and consistency, colour, odour, presence of biota, estimate of quantity of recovered sediment by a grab sampler, or length and appearance of recovered cores;
- deviations from SOPs must be reported;
- temperature and pH of the sediment at the sediment-water interface;
- redox potential of the surficial sediments which define oxic and anoxic conditions;
- the concentration of dissolved oxygen of the surficial sediments to determine if the sediments are oxic or anoxic, or the depth of the interface between these conditions in the sediments; and
- salinity and/or conductivity of the overlying water.

Sediment

- The devices recommended for the collection of sediments in freshwater, estuarine, and marine environments are presented in Figures 3, 4, and 5, respectively (Subsection 2.5).
- A minimum penetration depth of 6 to 8 cm is recommended for surficial sediment sampling; however, a depth of 10 to 15 cm is preferred (Subsection 2.5.2).
- Appropriate winching systems are required to control the rate of ascent and descent of the samplers (e.g., see Subsection 2.5.3).
- The sampling vessel or platform should be stationary, and as stable as possible.
- If sediments adhere to the outside of the sampler, the external surface of the sampler should be carefully hosed with clean water upon retrieval, before the sample is transferred to a storage container.
- The sampler and sampling equipment should be rinsed thoroughly with water at the sampling station between within-station samples, and rinsed with water from the next sampling station before collecting a sample. Equipment used in the handling of sediment must also be washed thoroughly between samples.
- A sample should meet the criteria of acceptability (e.g., see Subsection 2.5.4) before it is considered adequate.

Pore Water

- *In-situ* collection of pore water is recommended for geochemical investigations (Section 2.6) but, for routine toxicity testing, pore water can be extracted from sediment samples in the laboratory using centrifugation (Subsection 2.9.3).

Sample Handling

- All samples must be handled in a manner which satisfies the QA/QC program and DQO.

- If samples are designated as legal samples, then the appropriate procedures (see Subsection 2.8.3) and chain-of-continuity documentation (Appendix H) must be followed.

- Sediment might contain a mixture of hazardous substances, so it is only prudent to avoid all skin contact with these sediments by wearing protective clothing and equipment (e.g., gloves, boots, lab coats or aprons, safety glasses, and respirator) while sampling, handling, and preparing test substances or sediments.

- A spill control protocol should be in place in the laboratory, or sampling vessel, and participants in the project should be familiar with the procedures and recommendations.

- Disposal of all hazardous waste should adhere to the existing by-laws or regulations.

- Handling of samples should be performed in a well-ventilated area (e.g., outside, in a fume hood, or in an enclosed glove box) to minimize the inhalation and contact of sediment gases.

- Work surfaces should be covered with either Teflon® sheets, high-density polyethylene trays, or other impervious or disposable, similarly inert material.

- If the core sample is to be transported to the laboratory intact in the liner, then the overlying water should be minimal, a visual inspection and description of the core should be reported in the field notes, and the liners must be tightly capped with air excluded and secured in an upright position in the insulated transport container with ice. If methane or carbon dioxide gas bubbles form to disturb the sediment, remove the overlying water within a minute of retrieving the core.

- All subsampling of sediment samples should, if necessary, be done within 24 h of collection and should be restricted to parts of the sediment sample which have had no direct contact with the sampler.

- Core samples should be subsampled using extrusion procedures, other sediment samples should be subsampled using clean, non-reactive (e.g., Teflon®-lined scoop) implements.

- Box-core samples should be subsampled using a hand-coring device, or a scoop for the top 0 to 5 cm and a hand-corer for subsequent depths.

- Compositing samples or subsamples depends on the objectives of the study. If compositing samples or subsamples is required, it can be done in the field or in the laboratory. Before a composite sample or subsample may be analyzed or used in a test it should be homogenized with a mechanical mixer, or by hand, until uniform in colour and texture, or for a specified period of time that has been determined experimentally.

- If the maintenance of an oxygen-free environment for sediment handling is part of the study plan, handling of anoxic sediments should be done in a glove box in the presence of an inert gas.

Sample Containers

- Teflon® or Teflon®-lined containers are recommended for storage of whole sediments, regardless or the nature of the contaminants of concern.

- Amber glass and stainless steel wide-mouth containers with Teflon®-lined lids are suitable for storage of whole sediments with suspected organic contaminants (Table 7).

- Wide-mouth high-density polyethylene or borosilicate glass containers are suitable for storage of whole sediments with suspected inorganic contaminants (Table 7).

- Amber glass with Teflon®-lined septum caps should be used for interstitial or pore water to be analyzed for volatile, aromatic or halogenated organics (Table 7).

- Samples to be analyzed for metals should not come into contact with PVC, nylon, soda or flint glass, or metal materials, including stainless steel.

- Samples to be analyzed for organic compounds including pesticides should not come into contact with plexiglass, rubber, neoprene, polystyrene, painted surfaces, or low-density plastics.

- Sample containers must be pretreated before receiving a field sample (Table 8) and pretreatment of all containers must be completed by experienced individuals.

- If samples are to be stored at 4° C, sample containers should be filled to the rim, and air excluded before capping. If samples are to be frozen then containers which cannot accommodate expansion of the sample during freezing should not be filled completely. A headspace of about 2.5 cm should be left to accommodate expansion. An inert gas should occupy the headspace.

- Labelling of each sample container must include the site identification, sampling station location, the sample type, the method of collection, the name of the collector, and the date and time of collection.

- For samples with regulatory or legal implications, the sample container must have a tape seal and appropriate accompanying documentation (see Subsection 2.8.3).

Sample Transport

- The transport container should be refrigerated to 4 ± 2° C or contain ice or frozen gel packs that will keep the field samples between 1 and 7° C during transport to the laboratory.

- If field-collected samples are warm (e.g., >7° C), they should be cooled to between 1 and 7° C with ice before being placed in the transport container.

- Samples must not freeze during transport.

- A maximum/minimum thermometer or a continuous temperature recorder should be placed inside the transport container and the container sealed. Deviations in temperature should be reported.

- Light should be excluded from the transport container.

- For samples that have legal implications, the appropriate completed chain-of-continuity papers and Ministry of Transport documents must be completed and accompany the transport containers with the field samples. Each field-sample container must have a tape seal to demonstrate that it has not been opened during transport and the transport container must be locked during transit (see Subsection 2.8.3).

- All field-collected samples that require further processing before storage must be transported to the laboratory within 72 h, preferably within 24 h, of collection.

Sample Storage

- Storage times should be minimized.

- Whole sediments to be used in biological tests must be refrigerated in the dark at 4 ± 2° C, in tightly sealed storage containers without preservation reagents, for a preferred maximum storage time of two weeks, and maximum permissible storage time of six weeks.

- Sediments destined for chemical analyses only may be stored in the dark at 4 ± 2° C, in sealed inert containers, with or without preservation reagents, and should be analyzed within two weeks of collection. If sample analyses within two weeks of collection is not possible, then samples may be frozen at -20° C and stored for no longer than 6 months.

- Pore water samples should be anlayzed or used in biological tests immediately.

- Continuous monitoring of the storage conditions must be part of the QA/QC program.

- Temporary storage of samples in the field can be achieved by using refrigerated units on board vessels, freezers or refrigerators at a local land facility or insulated containers filled with ice or ice packs.

Test Sample Preparation

- Preparation of anoxic test sediments should be done in a glove box in the presence of a controlled flow of an inert gas, if it is desirable to maintain these anoxic conditions.

- Preparation of test samples should take place in a well-ventilated area (e.g., fumehood) and the appropriate health and safety precautions should be followed.

Test sample preparation might require the following manipulations:

a. Removal of indigenous organisms with forceps, pressure sieving, or wet sieving

Hand Picking (Subsection 2.9.1)

- Removal of indigenous macrofauna from sediments can be achieved by hand picking with forceps or pressure sieving freshwater, estuarine, and marine sediments.

Pressure or Wet Sieving (Subsection 2.9.1)

- Prior to sieving, the sediment sample should be placed onto a sorting tray made of an appropriately inert material and rocks and other debris removed with forceps.

- If it is necessary to sieve marine, estuarine, or freshwater sediments (sieving should be avoided if possible), the mesh size should be chosen in consideration of the toxicity test and test organisms, potential predators and/or competitors present, and the nature of the sample (e.g., particle size, quantity and size of debris).

- The same criteria for selecting sample containers (Subsection 2.8.1) should be considered when choosing a device for sediment sieving, to mitigate or minimize

contamination and adsorption. The use of brass sieves is discouraged. No new water should be used in the process of sieving the sediment.

- If sediments are sieved, it is recommended that the physicochemical properties of the sediment be determined before and after sieving to document changes attributable to sieving. Comparative toxicity tests using sieved and unsieved sediments might be necessary to discern the effect of sieving toxicity.

b. Homogenizing allocating sediment to test containers (Subsection 2.9.1)

Homogenizing

- Mixing by hand or mechanical mixing may be used to achieve homogeneity of colour, texture, and moisture; however, the efficacy of the method must be demonstrated, *a priori,* and the mixing time standardized and minimized to ensure consistency and to reduce alterations in the size distribution of sediment particles, respectively.

- Mixing of sediments should take place in the sample/storage container, or in a clean mixing container.

Partitioning

- Coning or caking and quartering is the recommended technique for partitioning the sediment for distribution among test containers. If a sediment splitter is used, its efficacy must be demonstrated and documented and it must be made of an appropriately inert material.

c. Drying (Subsection 2.9.2)

Oven drying sediment subsamples (1 to 5 g of wet sediment) at low temperatures (40 to 60° C) until a constant weight is reached or freeze drying sediment subsamples are the recommended methods for drying sediment. It should be noted that drying sediment before analyses can result in losses of organic compounds and reduced extraction efficiencies (Windsor and Hites, 1979; Haddock *et al.,* 1983).

d. Crushing/Grinding (Subsection 2.9.2)

Commercially available ball and pebble mills are recommended for fine-grinding small volumes of sediment (Mudroch and MacKnight, 1991); however, it should be noted that grinding could change the chemistry of the material. Crushing can usually be achieved with a mortar and pestel.

e. Dewatering (Subsection 2.9.3)

Centrifugation with subsequent decanting of the supernatant is the recommended method for dewatering sediment samples. The centrifugation speed depends on the sample size (e.g., sediment weight or volume).

Procedures for Open-water Disposal Studies

3.1 Introduction

The government of Canada regulates the disposal of substances at sea through a system of permits and inspections administered by Environment Canada pursuant to the provisions of Part VI of the *Canadian Environmental Protection Act* (CEPA) which became law in June, 1988. As such, CEPA, Part VI (formerly the *Ocean Dumping Control Act),* implements domestically the provisions of the London Convention, an international convention on the prevention of marine pollution by dumping wastes and other matter.

To receive an Ocean Disposal Permit, a proponent must, *inter alia,* complete an application in the form stipulated in the Ocean Disposal Regulations (see permit application, Appendix H). The information required for the Ocean Disposal Permit generally includes a description of the physical, chemical, and biological characteristics of the material intended for disposal.

The disposal site must be assessed if the proposed disposal site is new, or there is no recent characterization data for the area. Proponents applying for an Ocean Disposal Permit could be required to provide information about the sediments and their associated benthic communities at both the disposal and dredge sites. Collection and analyses of sediments are required for use in the physicochemical and biological assessments (Figure 8).

Environment Canada has provided the following guidance for the collection, storage, and handling of sediment samples to be used in support of an ocean-disposal application. Guidance is necessary because the manner in which sediment is handled, from the time of collection through to analysis, has a significant effect on its physical, chemical, and toxicological properties, and to improve the overall comparability, consistency, and reliability of these data across Canada. In addition to the guidance provided herein, applicants should also consult the "User's Guide to the Application Form for Ocean Disposal" (EC, 1995a) before undertaking any field work.

Section 3 of this document provides guidance on the procedures and methods for the selection of sampling stations, collection, handling, transport, storage and manipulation of sediments which are to be characterized physically and chemically, tested for toxicity, and/or assessed for the potential bioaccumulation of contaminants, to satisfy the ocean-disposal application requirements. The recommendations in this section may be supplemented by information presented in Section 2 or the Appendices. These sections, subsections, and appendices are cross-referenced in the text where appropriate.

3.2 Study Purpose and Objective(s)

Open-water disposal studies are designed to fulfill the minimum information requirements for ocean disposal permit applications (Appendix H). The purpose and objectives of the study should be clearly defined and integrated with the sampling program as part of the study plan.

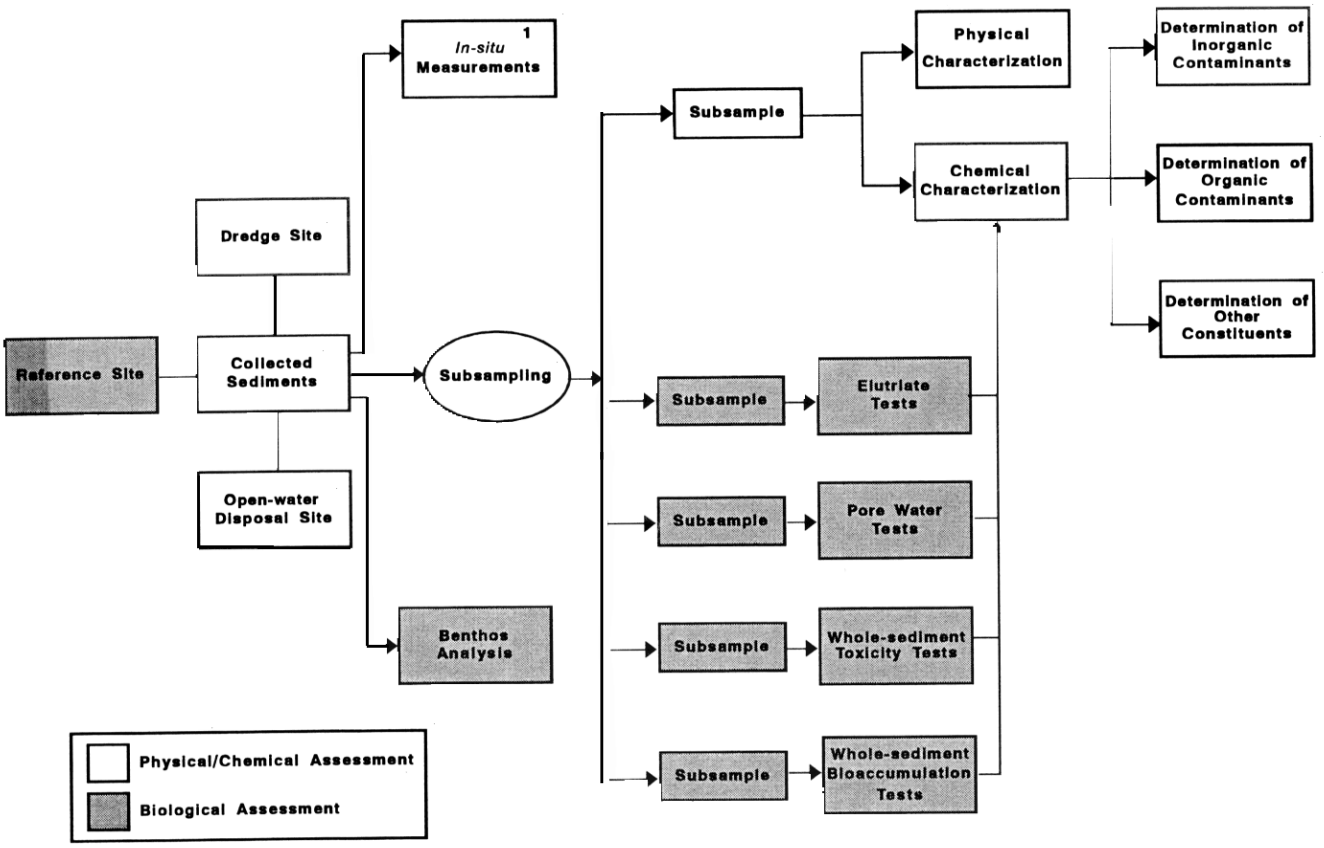


Figure 8 **Potential End Uses for Field-collected Sediments from Dredge, Disposal, and Reference Sites ([1] measurements are detailed in Section 2.4)**

for ocean disposal permit applications (Appendix H). The purpose and objectives of the study should be clearly defined and integrated with the sampling program as part of the study plan.

3.3 Definition of the Study Area and Study Site

The study area refers to both the study site (e.g., the area to be dredged), and the adjacent areas (e.g., land or water) that might affect or influence the conditions of the study site. The study site could refer to either the body of water in which dredging is to occur (e.g., dredge site), or the body of water that will receive the dredged material (e.g., disposal site). The boundaries of the study area and the study site(s) should be clearly defined and outlined on a suitable map or chart (see Parts B and F of the Permit Application in Appendix H for additional details).

3.3.1 Sampling Plan

Recommendation

- The sampling program should be completed in consultation with the Ocean Disposal Office **before** any action is taken.

The sampling plan is an integral component of the study plan. The recommended procedures for developing a study plan are presented in Section 2.3 and, additional information concerning the sampling plan for dredging projects is presented in the following.

A sampling plan must be designed to accommodate the specific objectives of the project. This information will be required in Part D of the permit application for ocean dumping (Appendix H). If recent data are available (within the last four years), it may be used in Part E of the application form (Appendix H). The plan should address the following:

- historical data review;
- the number, location, and description of the sampling stations;
- the types of samples to be collected;
- the number of samples to be collected;
- the number of replicate samples per sampling station;
- the volume or weight of sediment per sample to be collected, and the desired sampling and penetration depth;
- the method of collection, type of collection device, and type of storage containers;
- the type of analyses;
- the sample preservation methods;
- the method of storage and storage conditions of samples in the field and during transportation of samples to the laboratory;
- the method of sample storage in the laboratory; and,
- the QA/QC programs and DQO (CCME, 1993; USEPA, 1991).

In addition, the following operational requirements should be included:

- the health and safety plan for field collection of samples;
- the designation of personnel and their responsibilities;
- the logistics of the actual operation (e.g., vehicles, manpower, equipment, timing of events, distance from collection site to laboratory);
- the contingency plans in the event of equipment loss/failure;
- the backup personnel (e.g., in the event of sea sickness);
- the environmental conditions (e.g., currents, tides, substrate variation, recent bathymetry); and,
- photograph(s) of the study site(s) and area.

3.3.2 Historical Data and Identification of Potential Sources and Present Conditions

In order to define the sediment-collection stations for a sampling plan specific to a dredge or disposal site, all available historical data should be evaluated. In addition to the information discussed in Subsection 2.3.2, historical data for dredge or disposal sites should include municipal or harbour commission archives, reports from government agencies, hydrographic surveys, bathymetric maps, and previous submissions

for ocean-disposal permits. Basically, the study site should be stratified using any available information (i.e., potential sources of contaminants, areas of marine activity, geotechnical, geochemical, or hydrographic data) which helps in identifying areas where contaminants are likely to be found, where contaminants were found in the past, or where fine-grained particles are likely to accumulate. Contaminants of concern in dredged material include inorganic contaminants (e.g., metals and metalloids) and organic contaminants such as petroleum hydrocarbons, PCBs, PAHs, as well as pesticides and solvents; therefore, all potential sources should be considered.

These parameters form part of the Prohibited Substance List which appears in Schedule III, Part I of CEPA. Consult CEPA Schedule III, Parts I, II, and III for other prohibited and restricted substances, and other factors to be considered. Minimum information requirements are identified in the permit application form (Appendix H).

3.3.3 Methods for Locating Sampling Stations

General methods for locating sampling stations have been defined in Subsections 2.3.3 and 2.3.4. The same procedures apply here with some requirements specific to dredging projects. The goal is to collect sediment samples that will be representative of the study site and area.

Dredge Site. The characteristics of the dredge site will influence, to a considerable extent, the distribution of the sampling stations located within the study site. A dredge site can be treated as a single zone (i.e., a zone within which there is a uniform distribution of contaminants). Alternatively, dredge sites can often be divided into different strata according to the historical (or suspected distribution of contaminants, geographical configurations, or substrate type. In addition, surface sediments might be considered district from subsurface sediments, if vertical stratification of contamination is expected.

Many dredge sites exhibit considerable spatial variation in the sediment concentrations of contaminants. Such differences are often associated with sources of contamination and characteristics of the site. It usually, therefore, desirable to stratify dredge sites according to possible sources of contamination, historical information on contaminant levels, and/or sediment characteristics. For very small projects stratification may not be warranted unless there are clear indications of large differences in probable contaminant levels. Large projects should always be stratified. For projects that are divided into separate areas, each such area should be treated as a stratum.

The total number of samples required per stratum (or for a small project that is not stratified) is given in Appendix G. It is suggested that strata be classified as “likely contaminated”, “suspect”, or “probably clean”. When good historical information on sediment chemistry supporting this classification is available, the sample sizes in the “likely contaminated” and “probably clean” strata may be reduced. Past data will be considered adequate to support this reduction in number of samples if sampling was done not more than four years previously, if the number of samples used in previous sampling meets the current requirements, if there has been no change in the site characteristics (e.g., no new pollution sources, no changes in use patterns). If these requirements are met the number of samples in the “likely contaminated” and “probably

clean" strata may be one-half the number given in Appendix G.

If good historical data on sediment chemistry are lacking then each stratum should have the number of samples specified in Appendix G; stratification would then be based on indirect information (e.g, potential sources of contaminants, areas of marine activity, geotechnical or hydrographic data, shape of the area or harbour, etc.).

If the results (e.g., average concentration for a specific analyte) in a "likely contaminated" stratum are found to fall below the limits for Ocean Disposal, additional samples up to the number given in Appendix G may be required before a decision is made. Similarly, if the results in a "probably clean" stratum are found to fall above the limits for Ocean Disposal, additional samples may be required before a decision is made.

When a dredging project consists of material of very different particle sizes it may also be desirable to stratify according to particle size (silt, sand, and gravel). If a stratum of gravel is identified the gravel need not be sampled; however, the stratum should be explored for the occurrence of "pockets" of finer materials. If such "pockets" are found they may be treated as an additional stratum and sampled as such.

A random approach is recommended for the location of sampling stations in unstratified projects or in each individual stratum. To assist in the random selection of sampling stations, the project (or stratum) is divided into square blocks. There should be at least five times as many blocks as the number of sampling stations required. Dividing the project area into blocks in this way is practical method of providing a "frame" for the selection of a random sample. The number of sampling stations (assuming one sediment sample per station) required for the project or stratum volume is given in Appendix G. No project (or stratum) should have fewer than six sampling stations (except when the "one-half rule" applies). In a project (or stratum) where contamination is suspected it is very desirable to collect more than the minimum number of samples, hence the number of sampling stations would increase (Skei, 1992).

Additional guidance for locating sampling stations for three types of dredging projects are provided in the following case studies.

CASE 1: Contamination Pattern Unknown - No Stratification

Figure 9 shows the blocks constructed for a relatively simple unstratified project. The project has about 20 000 m^3 of sediment to be dredged so eight samples are required (Appendix G) and the project is divided into at least 40 blocks. The blocks are constructed to cover the project completely. A block is included in the selection process if at last half the area of the block falls within the dredge site. Each block is assigned a number and the blocks to be sampled are randomly selected (using random number tables or a random number generator) and usually one sediment sample is collected from the centre of each of these blocks. The minimum number of blocks for this project is 40 but this was increased to 44 since this gave a convenient coverage of the area to be dredged.

CASE 2: Suspected Areas of Contamination with no Historical Data - Simple Stratification

Figure 10 illustrates a simple case where stratification is desirable. The volume to be

dredged is 40 000 m^3 divided into two units, an inner basin of 30 000 m^3 and the outer harbour of 10 000 m^3. The inner harbour is suspected to be contaminated because of the presence of docks, an outfall, and its enclosed nature. The outer harbour may be less contaminated but is suspect because of mooring activities at the public wharf, the presence of a marine railway, and the presence of a skidway. For a project of this size, nine samples for the Inner Harbour and six samples of the outer Harbour are suggested (Appendix G). The Inner Harbour should be divided into at least 45 blocks and the Outer Harbour into at least 30 blocks. From each area, the blocks are selected randomly and the sampling stations located in the centre of each of these blocks.

CASE 3: Suspected Areas of Contamination with Historical Data - "Likely contaminated" and "Probably clean" Areas

Figure 11 shows a situation with "likely contaminated" and "probably clean" areas. There is a harbour area which, aside from obvious marine activity, includes other sources of contamination. The approach change has no clear source of contaminants. Historical data indicate unacceptably high concentrations (i.e., those that exceed the Ocean Disposal limits) in the "likely contaminated" area, lower concentrations (i.e., those below but near the Ocean Disposal limits) in the "suspect" area, and very low

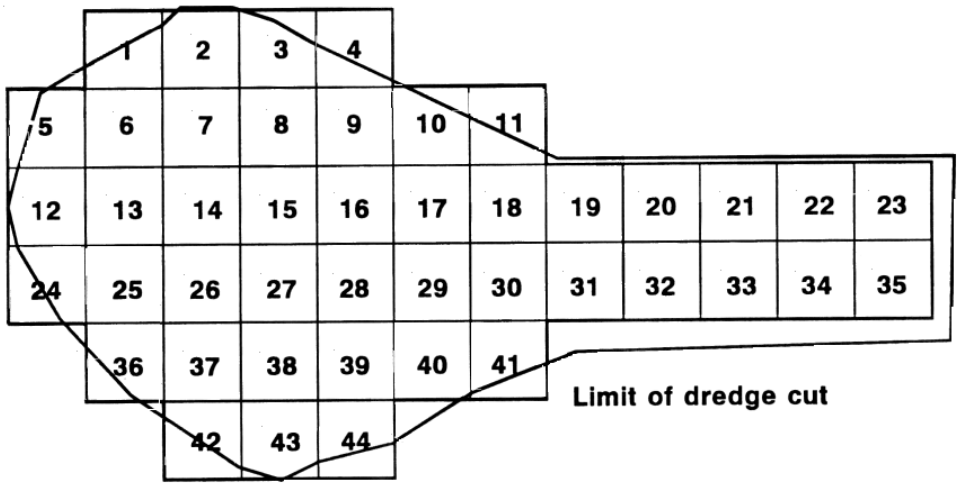


Figure 9 Block Construction for Selecting Sampling Stations in a Project without Stratification

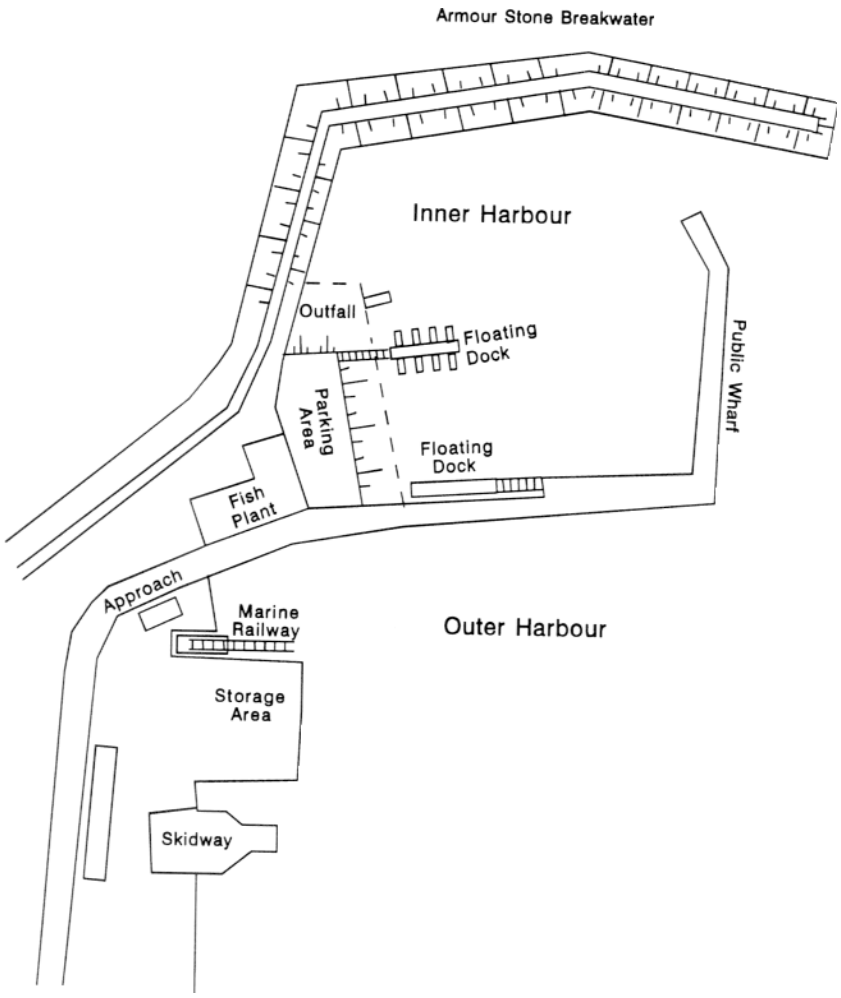


Figure 10 A Dredging Project with Suspected Areas of Contamination.

levels of all contaminants in the "probably clean" area. This situation suggests constructing three strata, the inner harbour, the suspect area, and the remainder of the approach channel. The volume of material to be dredged (i.e., the project size) is about 200 000 m^3 with 90 000 m^3 in the inner harbour, 36 000 m^3 in the suspect area, and 74 000 m^3 in the approach channel.

Since there is good historical information available, the results in the Harbour ("likely contaminated") and Channel ("probably clean") will be viewed as confirmatory; therefore, the final number of samples to be collected is one-half the number suggested in Appendix G (Table 10). To determine the location of each sampling station, the Harbour should be divided into at least 45 blocks, the Suspect Area into at least 50 blocks, and the Channel into at least 40 blocks. The blocks to be sampled are then chosen at random within each stratum, and the sampling stations located in the centre of each block.

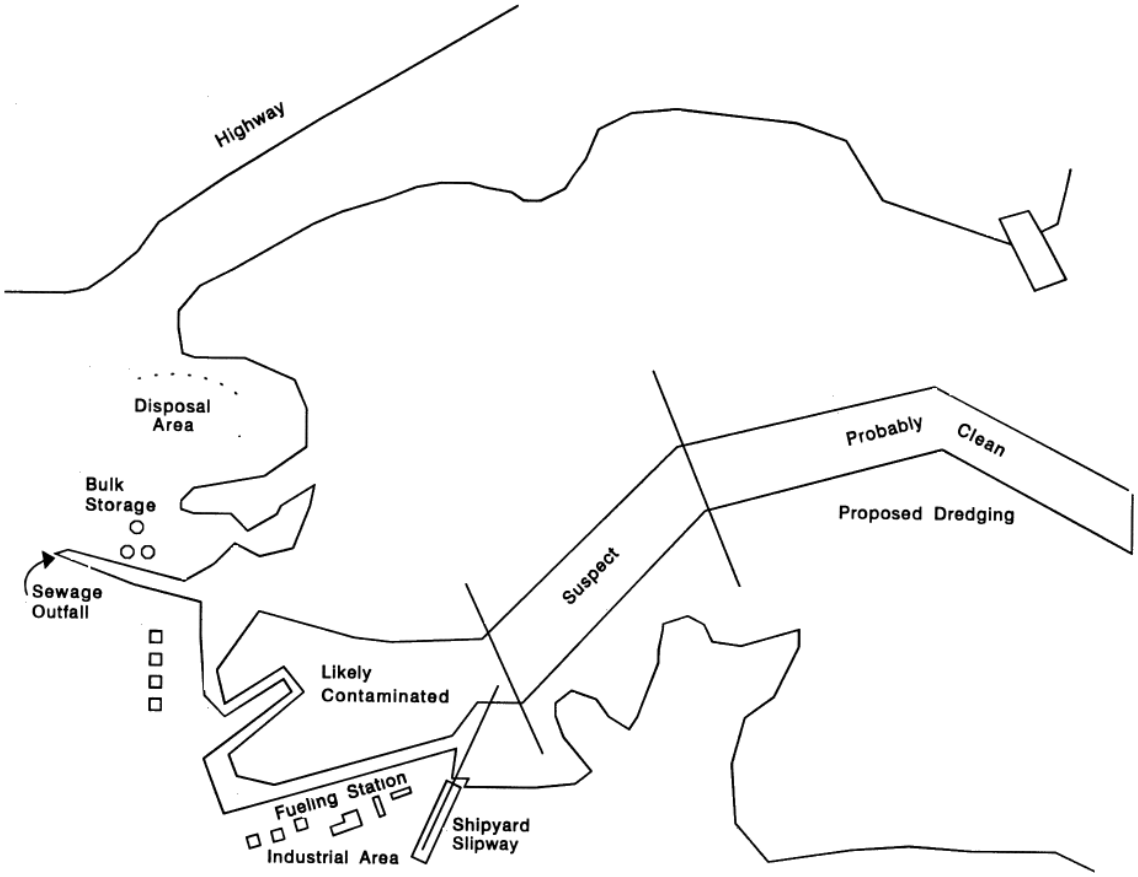


Figure 11 **A Dredging Project which Combines Stratification with Historical Data to Determine the Number and Location of Sampling Stations**

Table 10 **Number of Blocks to be Sampled in a Dredging Project Stratified on the Basis of Historical Data and Areas of Suspected Contamination**

Stratum	Volume of Sediment in Each Stratum (m^3)	Basic Number* of Samples per Stratum (Appendix G)	Final Number* of Samples Per Stratum
Harbour	90 000	17	9
Suspect	36 000	10	10
Channel	74 000	15	8
Total	200 000	42	27

* One sediment is collected at each sampling station; therefore, the number of samples also equals the number of sampling stations

This example illustrates the way stratification may be used where general knowledge of the dredge-site conditions can be combined with historical data. Historical data can often provide an objective basis for drawing the strata boundaries to provide more precise estimate of the contaminant distribution.

Disposal Site. Minimum information requirements related to the selection of new disposal sites are found in the application form for ocean disposal of dredged materials (Appendix H). Post-disposal monitoring is addressed in the Interim Monitoring Guidelines for Ocean Disposal (EC, 1993). In essence, the selection of a new disposal site involves describing and characterizing the proposed site in consideration of its dispersive characteristics that are required to predict the area of deposition. Sediment transport models may be used to assist with this process. The information required for the selection of an acceptable disposal site also provides baseline information for comparison to post-disposal monitoring results. Guidance provided in this subsection specifically addresses the minimum information requirements related to the selection of new disposal sites.

The location of the sampling stations within the area depends, to a large extent, on the dispersal characteristics of the area and should reflect the areas most likely to be affected by the disposal of the dredged material. Once dumped, material can be transported according to the combined effects of wind-generated currents, waves and tides, bathymetry, water depth, and time-varying current velocities. Therefore, adequate information is required on each of these factors. Information on salinity will also be required. The Department of Fisheries and Oceans (DFO), the Ocean Disposal Office of the Department of the Environment, Canadian Hydrographic Service and Coastal Oceanography, Natural Resources Canada (NRCan), and local sources can be consulted for both historical and contemporary data. Dispersion models (e.g., Automated Dredging and Disposal Alternatives Management System - ADDAMS) are also useful for predicting the concentrations of dissolved and suspended constituents of dredged material in the water column, the length of time it will take the material to each the bottom, and the radius and direction of the dredge plume (USEPA, 1991). The key to the effectiveness of these models is to provide the most accurate data possible which will describe the area. Further guidance on use of sediment transport models can be found in the Draft Technical Guidance on Physical Monitoring (EC, 1994a).

The number of samples and the location of sampling stations may be determined on a site-specific basis, using information from preliminary sampling studies, available historical data, and statistical power analysis. Additional information to be considered are described in Part F of the application form (Appendix H). If preliminary sampling (e.g., pilot-scale study) cannot be undertaken, procedures described in Subsection 3.3.3 can be applied to the disposal site. Sediment transport models might be useful in predicting the initial area of deposition from which the disposal-site boundaries can be established (EC, 1993; 1994a).

The location of all sampling stations must be identified with accurate coordinates on a map containing the disposal site relative to adjacent landmarks (e.g., study area) in order that they can be sampled at the monitoring stage. Other information required for the map is identified in Appendix H.

3.3.4 Methods for Positioning

The location of each field-collected sample (e.g., sediment, water, or benthos) must be accurate and precise; therefore, only those methods with an accuracy of 1 to 10 m are recommended. An accurate description of the location of a "hot spot" in a dredging operation is critical for any additional sampling that might be subsequently required. Any of the electronic positioning systems listed in Table 2, with an accuracy of 1 to 10 m, may be used for positioning.

3.3.5 Sample Size, Number of Samples and Replication

Sample Size

Recommended Procedure for Determining Sample Size

- Determine the sample analytical requirements for each dredging project (Figure 8), on a case-by-case basis, and calculate the required volume or weight of sediment per sample.

- The recommended minimum sample size to be collected for the various types of analyses is presented in Table 3. Compare the quantity of sediment required (Table 3) with the capacity of the sampler to deliver the desired amount (Table 5).

- Consider the QA/QC sampling program and whether or not samples are to be archived. If the core sampler cannot deliver the volume required for toxicity testing and/or assessment of bioaccumulation, collect additional samples and composite appropriately (Subsection 2.7.1). Alternatively, collect replicate sediment samples and incorporate them into the test design.

At present, an application to load or dump dredged material requires physical and chemical characterization of the sediments from the dredge site. Biological testing is becoming an increasingly important component of sediment characterization and will likely be required routinely in future years. Therefore, in the interest of saving both time and money, it is recommended that enough sediment be collected during the sampling program to accommodate both the physical/chemical and biological assessment.

For example, if the chemical analysis indicates a need for bioassessment, the bioassessment must be performed using sediments from the same batch as the chemical analyses. If the proponent has not collected sufficient quantities of sediments or a sufficient number of samples of sediment from the study site, then additional sampling will be required. Since the additional samples of sediment will constitute a second batch of sediments, the physicochemical characterization will have be repeated for the new batch of sediments. Therefore, it is advantageous to design a sampling program to collect both enough sediment and a sufficient number of samples to accommodate a biological assessment as well as a physicochemical assessment.

Number of Samples

Recommended Minimum Number of Samples

- The minimum number of sampling stations to be sampled for dredging projects or strata of different sizes are presented in Appendix G.

- It is recommended that additional samples of sediment be collected in areas most likely to be affected by urban centres, agricultural activities, industry (i.e., in the vicinity of sewage, industrial, or municipal effluent outfalls, navigational routes, loading and unloading areas, refuelling points, chemical transhipment areas, catchments for drainage basins, booming grounds, log pockets, etc.).

The number of samples collected should depend on the size of the disposal site or area to be dredged, the type of contaminants, the volume of sediment required, the size of the collection device, and the desired level of statistical resolution.

Although the goal is to obtain sufficient information to evaluate the potential environmental impact of dredging and ocean disposal, operational constraints are acknowledged. Possible responses to such constraints (e.g., funding, time, etc.) are reducing the number of replicates at each sampling station and using stratification based on known (or suspected) contaminant levels to increase precision of the estimate of the contaminant concentrations.

It is strongly recommended that the sampling plan be optimal for the project and, if constraints are to be applied to the analytical plan, they should be approved by the Ocean Disposal Office. In addition to the collection of sediment samples from the area to be dredged, or from the disposal site, a minimum of three "uncontaminated" reference sediment samples from at least one site (preferably two) should be collected (see Subsection 3.4.2).

Replication

Recommended Minimum Number of Replicate Samples and Procedures

- The collection of replicate samples within a sampling station is not usually necessary for dredging projects.

- For quality control purposes a limited amount of replicate sampling (10% of the sampling stations) may be used.

- Replicate samples should be kept separate (i.e., separate sample and/or storage containers).

The collection of replicate samples at a given sampling station is not usually necessary for dredging projects because the objective is to obtain the best possible estimate of the mean concentration within the project area or within strata. In almost all cases this will be achieved by sampling as many stations as possible (subject to cost constraints) with a single observation at each station.

The collection of replicate sediment samples from a particular station could be dictated by the QA/QC program established to satisfy the DQO of the Ocean Disposal Office. Because of the large surface area and the minimum disruption to the sediment during collection, subsamples collected from a box-core sampler may be considered acceptable as replicate samples for QA/QC purposes. If replication within stations is used, samples should be true replicates. Replicate core samples should be collected using a multiple core sampler. For grab samplers, multiple samples should be collected. Replicate samples should be kept separate (i.e., separate sample and/or storage containers). Samples obtained by divers

using hand-corers should be collected as close as possible to the first sample to be considered replicate samples. If a current is detected, subsequent samples should be taken at points countercurrent to the previous sample.

If the quantity of a single sample is insufficient to meet the sample-size requirements for analyses, it may be necessary to sample repeatedly, at the same station, and composite the material. Compositing samples from the same station is usually performed in the field.

In general, samples from different stations should not be composited since this will lead to loss of information on the area being sampled. For analyses that are very expensive (e.g., dioxin and furan analysis) limited compositing of samples or subsamples may be used. If this is done, an attempt should be made to group samples for compositing according to suspected levels of contamination.

3.4 Collection of Whole Sediments

A proponent intending to submit an application for an Ocean Disposal Permit might have to collect samples of sediment from the dredge site and/or from the proposed disposal site (Figure 8). Sediments from a reference site and a control site could also be required as part of the disposal-site assessment (see Subsection 3.4.2).

Sediment samples from the reference site should be collected concurrently with the test sediments, or within 48 h (Subsection 3.4.2); however, the samples of field-collected control sediment should be collected or acquired concurrently with the collection or acquisition of the field-collected test organisms (see Subsection 3.4.3), in the event that they are not cultured in the laboratory. Formulated or artificial control sediment may be used as an alternative to field-collected control sediment.

There is a recognized need for the development of "artificial" or "formulated" control sediments for marine environments and, as such, a number of research initiatives are currently underway (EC, 1995b). For marine environments, the control sediments to date have been comprised of beach sand collected from an appropriately clean site and manipulated and prepared for testing by either washing, wet sieving, and oven drying the sand, or modifying the sand by placing it in a muffle oven at a high temperature for a fixed period of time (Yee *et al.,* 1992; Burgess *et al.,* 1994a). If an artificial or formulated control sediment is used in biological assessments for dredging projects, it should meet the following criteria:

- support the survival, growth, or reproduction of a variety of benthic invertebrates;
- provide consistent acceptable biological endpoints for a variety of species;
- be characterized by a consistency in terms of both the individual constituents in the formulation, as well as, its performance in a test;
- be comprised of constituents that are readily available to all individuals and facilities;
- be free from concentrations of contaminants that might cause adverse effects to test organisms (i.e., only trace levels permitted) (ASTM, 1994).

Guidance on the use of formulated sediments as control sediments is provided by Environment Canada (1995b).

The following subsections provide guidance for the collection of whole sediments from dredge and/or disposal sites, as well as sediments from reference and control sites. Collected sediments may be required for chemical assessment and/or biological assessment.

3.4.1 Collection of Sediments from Dredge or Disposal Sites

Recommended Options and Procedures for a Chemical Assessment Only

- Core samples should be collected, where possible, and the penetration depth should extend to the depth of the intended dredge cut for each sampling station.
- The coring devices recommended for the open-water disposal site are presented in Figure 5; whereas, those recommended for the dredge site are presented in either Figures 3, 4, or 5, depending on the type of environment.
- Where core samplers cannot be used to collect sediments, a surficial sample of sediment may be collected with a grab-sampling device, as recommended in either Figures 3, 4, or 5, depending on the type of environment.
- The penetration depth of the grab sampler should be ≥ 15 cm.
- All samples should be collected from the sampling stations designated and approved in the sampling plan.
- Guidance on sampler operation is presented in Subsection 2.5.3.
- The minimum volume of sediment per sample typically required for physicochemical characterization is 1.0 L of wet sediment assuming a water content of > 90%. If analyses includes petroleum hydrocarbons (e.g., oil and grease) an additional 1 L of sediment might be required.
- The QA/QC procedures for collection of sediment from dredge, disposal, or reference sites as outlined in Section 3.7 should be followed.
- The on-site data forms for sediment collection (Appendix H) must be completed as part of the permit application.
- Safety precautions associated with the collection of potentially hazardous material should be observed.
- A minimum of three, preferably five, samples must be collected from one (preferably two) sampling stations at the reference site (see Subsection 3.4.2) .

Recommended Options and Procedures for a Chemical and Biological Assessment

The following applies to projects where both chemical and biological assessments are imminent.

- Core samples should be collected, where possible, and the penetration depth should extend to the depth of the intended dredge cut for each sampling station.

- Box-core samplers are recommended for the collection of fine-grained surficial sediments at both dredge and disposal sites. In shall waters (e.g., ≤10 m depth) hand-coring devices are recommended for shallow dredge cuts. When box-core or hand-coring devices cannot be used, either a PONAR, Smith-McIntyre, or Van Veen grab sampler may be used depending on substrate type, water depth, and conditions (see Section 2.5, Figures 3, 4, or 5). A penetration depth of ≥15 cm is desirable. Alpine or Phleger gravity-core samplers are recommended for the collection of sediments at dredge sites where the dredge cut is less than 2 m or where a vertical profile of subsurface sediments is required. Piston-core samplers are recommended for sediment samples deeper than 2 m. The core liner should be glass, polyethylene, or polypropylene with an inner diameter of 100 ± 25 mm for cores ≤1.0 m and 75 ± 25 mm for those >1.0 m.

- Guidance on sampler operation is presented in Subsection 2.5.3.

- All samples should be collected from the sampling stations designated and approved in the sampling plan.

- The volume of sediment collected should be sufficient to support physicochemical characterization (1 to 2 L) and biological assessment (Table 3). A minimum of 3 L of sediment per sample is recommended where the biological assessment includes whole-sediment toxicity tests only. A minimum of 7 L per sediment sample is recommended if the biological assessment includes a bioaccumulation test with no replication.

- If replicate samples of sediment are required, a minimum of three, preferably five, replicate samples should be collected at a sampling station.

- A minimum of three, preferably five, samples must be collected from one preferably two, sampling stations at the reference site (see Subsection 3.4.2) .

- The QA/QC procedures for collection of sediment from dredge, disposal, or reference sites as outlined in Section 3.7 should be followed.

- The on-site data forms for sediment collection (Appendix H) must be completed as part of the permit application.

- Safety precautions associated with the collection of potentially hazardous material should be observed.

- An analysis of the benthic community might be required as part of the disposal-site assessment and, therefore, additional subsampling (e.g., box-core sample) or sampling (e.g., hand-corers, grabs) of the surficial sediments (e.g., 0 to 5 cm deep) may be required.

- Collection of sediments from control sites together with test organisms, may be required as part of the biological assessment (see Subsection 3.4.3) .

A box-core sampler is recommended for the collection of fine-grained surficial sediments < 1 m deep. Although a box-core sampler is costly and cumbersome to use, it is recommended for sampling surficial sediments at all stations because it is ideal for the collection of an undisturbed, soft

sediment which has a relatively large quantity of material for a given horizon, a reflection of the large cross-sectional area of the device. Although they are usually constructed of metal (e.g., steel or anodized aluminum), plastic or Teflon® liners are available (EC, 1985). Box-core samplers can effectively sample benthic organisms and the large-volume core can be easily subsampled with hand-coring devices. A single box-core sample will deliver enough sediment to meet the biological and chemical analytical requirements.

In shallow waters (e.g., ≤10 m depth), hand-coring devices, operated remotely or by divers, are recommended for shallow dredge cuts (i.e., ≤0.5 m) in soft sediments; however, repeated sampling might be necessary to obtain a sufficient quantity of sediment.

When the substrate type is not conducive to the use of a box-core or hand-core sampler, either a PONAR, Smith-McIntyre, or van Veen grab sampler, depending on the substrate type, water depth, and conditions, should be used to collect surficial marine sediments (Figure 5). Grab samplers are also suitable for the collection of sediments that are known to be vertically homogeneous.

The sediments in these areas (e.g., navigation channels with frequent ship travel, annual dredging projects, etc.) experience resuspension and turbulence which prevent stratification; therefore, a surficial grab sample is likely to be representative of deeper sediments. The depth of penetration must be at least 15 to 20 cm. These grab samplers can effectively sample the benthic organisms.

For the collection of subsurface sediments, gravity (e.g., Alpine or Phleger) (1 to 2 m sediment depth) and piston core (> 2 m sediment depth) samplers are recommended. The inner diameter of the core tube or liner should be 100 ± 25 mm for cores ≤1.0 m in length, and 75 ± 25 mm for cores > 1.0 m, to retrieve a sufficient volume of sediment. The core liner should be either high-density polyethylene or polypropylene. The penetration depth should be equal to the depth of the dredge cut.

The advantages and disadvantages of the different coring devices that currently receive the greatest use are detailed in Section 2.5 and summarized in Table 5. Effective sampler operation and cleaning instructions are discussed in Subsection 2.5.3 of this document, and criteria of acceptability are outlined in Subsection 2.5.4. The make and model number of the collecting device should be included in the study plan, as well as a brief description of its standard operating and cleaning procedures, dimensions, weight, and capacity.

3.4.2 Collection of Reference Sediments

Recommended Options or Procedures

- The location of the references site (i.e., the site within which sampling stations are located for the collection of reference sediments) should be in the vicinity of the study site, within the same body of water but relatively free of the influence of the sources of contamination.

- The reference sediment should be generally comparable to the test sediment in terms of characteristics such as particle size distribution, organic carbon content, pH, and cation exchange capacity.

- The sampler used to collect sediment from the reference site should be identical to that used to collect surficial test sediments (e.g., disposal-site sediment).

- The volume of sediment collected should be sufficient to support both physicochemical (1 to 2 L) and biological assessment (3 L for the toxicity test, 3 L for the bioaccumulation test); therefore, a minimum volume of 7 L per sample is recommended.

- A minimum of three, preferably five, replicate samples of reference sediment should be collected.

- Sediment samples from the reference station should be collected concurrently, or within 48 h of the test sediments.

- The sediment collection forms (Appendix H) must be completed as part of the permit application.

- Although sediments from the reference sites are considered to be "clean", collection and handling procedures should be the same as those for the test sediments.

- The QA/QC procedures for collection of test sediment as outlined in Section 3.7 should be followed.

- Reference sites for which analytical data are already available should be used, where possible, and it is recommended that the proponent consult the regional office of Environment Canada (Appendix B) for advice, as needed.

A reference sediment is a field-collected sample of relatively "uncontaminated" and "non-toxic" sediment that is selected as a comparative sediment for biological testing because of its geochemical similarity to a specific test sediment. The primary purpose for collecting reference sediments in a dredging study is to provide a geochemically similar sediment to measure the effects, which are not contaminant-related (e.g., physical characteristics), of the test sediment on the test organism. There are currently no sites in Canada that have been designated as sites for reference sediments.

In addition to these recommendations, the collection, handling, transport and storage of reference sediments should follow the procedures recommended in Sections 2.5, 2.7, and 2.8, where applicable. The sample and/or storage containers and handling equipment (e.g., mixers, sieves, subsampling devices, etc.) must be made of either new materials, or have been used previously to handle or contain "uncontaminated" materials only and cleaned thoroughly before use. All equipment including the collection device should be cleaned thoroughly before use (Section 2.8) and all equipment used to handle, store, and manipulate control or reference sediments should be kept separate from those used to handle, store, and manipulate test sediments. Recommendations relevant to preparations for field sampling are provided in Subsection 2.3.7.

3.4.3 Collection of Control Sediments for Biological Assessment

Recommended Options and Procedures

- Control sediments may be collected from aquatic environments, or they may be artificially prepared. Control sediments

for dredging studies should be collected from the same site(s) as the test organisms. If the test organisms are cultured in the laboratory with a formulated sediment, the formulated sediment may serve as a control sediment.

- A proponent should consult the permit-issuing office for information on the location of sites for the collection of test organisms.

- The volume of sediment collected should be sufficient to support holding the organisms in the laboratory, as well as physicochemical characterization, and biological assessment. A minimum volume of 15 L per sample is recommended.

- The sediment and test-organism collection forms (Appendix H) must be completed and submitted as part of the permit application.

- The QA/QC procedures for the collection of test organisms, as outlined in the biological test method, should be followed.

- The QA/QC procedures for collection of test sediment as outlined in Section 3.7 should be followed.

The primary purpose for collecting a control sediment in a dredging study is to provide a sediment with characteristics conducive to the survival, growth, and reproduction of the test organisms. It may or may not have characteristics similar to those of the test sediment. The control sediment serves as a substrate for monitoring the health of the test organisms over the duration of a test, or over the course of a series of tests in the laboratory. Sufficient sediment should be collected at the site to support the holding of the test organisms in the laboratory, the bioassessment (e.g., toxicity tests), and the physical/chemical analyses.

Although the volume of sediment required is species specific, and depends on the type of toxicity test, a minimum of 15 L of sediment will typically be sufficient to support a test program. Additional sediment should be collected when additional tests are anticipated. The use of formulated sediments as a control sediment might obviate the collection of a natural control sediment (Burgess 1994a,b; Suedel and Rodgers, 1994) if it possesses the attributes of both types of sediment.

In addition to these recommendations, the collection, handling, transport, and storage of naturally occurring control sediments should follow the procedures recommended in Sections 2.5, 2.7, and 2.8, where applicable. The sample and/or storage containers and handling equipment (e.g., mixers, sieves, subsampling devices, etc.) must be made of either new materials, or have been used previously to handle or contain “uncontaminated” materials only. All equipment including the collection device should be cleaned thoroughly before use (Section 2.8) and all equipment used to handle, store, and manipulate control sediments should be kept separate from those used to handle, store, and manipulate test sediments.

3.5 Handling of Collected Samples

3.5.1 Dredge Core Samples

Handling of sediment samples has been discussed in Section 2.7 and Subsection

2.7.1. It is important to reiterate the need to avoid unnecessary contact with glassware and utensils. Samples for trace metal analysis and biological testing should not contact metal surfaces. Samples for organic analyses should not contact plastic surfaces. The appropriate handling precautions should be taken to avoid contamination of samples. The procedures recommended in Subsection 2.7.1 apply to core samples collected from dredge sites.

Subsampling of core samples should be part of the preparation of whole-sediment test samples for physical and chemical characterization, bioaccumulation tests, and toxicity tests. A permanent record of the identification and appearance of the core can be made by photographing the core in the transparent tube or liner with its sample identification number visible. the recommended procedures for subsampling core samples from dredge or disposal sites follow.

Subsampling Core Samples from Dredge or Disposal Sites The recommended procedure for subsampling sediment cores, other than box-core samples is as follows:

- Record the appearance of the core (e.g, photography, or description of sediment type, colour, structure, fauna, length) .

- Siphon off any remaining surface water, leaving the surface fines undisturbed.

- Mechanically extrude the top 5 cm, excluding the sediment that is in direct contact with the core liner (see Subsection 2.7.1), and place into the appropriate type of container (see Section 2.8, Table 7).

- Mechanically extrude the sections of the core that have been identified by visual inspection and best professional judgement as being distinct sediment horizons and place them in separate containers.

- The sediment in each container should be homogenized (see Subsection 2.9.1) until uniform in colour and texture.

- Collect, using a Teflon® or Teflon®-lined scoop, subsamples of sediment for metal and organic contaminant analysis, followed by subsamples for other chemical and physical characterization.

- The subsample volumes will depend on the analytical test requirements. Since analyses might be performed on a dry-weight basis, and the water content of sediments is variable (e.g., <30 to 80%), collect at least 2 or 3 times the dry weight of sediment required for analyses.

- If maintenance of an oxygen-free environment for sediment handling is part of the study plan, extrusion of cores should take place in a modified glove box in the presence of an inert gas (e.g., Subsection 2.7.1).

3.5.2 Box-core or Grab Surficial Samples from Dredge, Disposal, Reference, or Control Sites

The handling of sediment samples collected with box-core samplers, or grab samplers is discussed in Subsections 2.7.1 and 2.7.2, respectively. These recommended procedures apply to sediments collected from dredge, disposal, reference, and control sites.

Subsampling Grab or Box Core Samples.

The recommended procedures for subsampling the surficial sediments collected with a grab sampler are as follows:

- Carefully hose with water, any sediment adhering to the outside of the sampler which could potentially contaminate the sample.
- Place the sampler on a platform (e.g., table) that is stable and readily accessible.
- Let the sample "stand" for a few minutes.
- Open the top (or side) of the sampler and examine the sample. Record the appearance of the sample (see section 2.4). If it is acceptable (see Subsection 2.5.4), then carefully siphon-off the overlying water, if present, leaving the fine-grained surface sediment undisturbed, and discard.
- Field-measurements may be made on the sample prior to subsampling (see Section 2.4).
- Using a Teflon®-lined, scoop or hand-coring device, carefully remove the top 0 to 5 cm and place into a sample container filling to the brim.
- Cap the container after topping with a thin layer of deoxygenated overlying water, if necessary, to assist with the exclusion of air.
- Subsampling in an oxygen-free environment may be desirable depending on the objectives of the study and procedures are described in Subsection 2.7.2.

For a box-core sample, follow the first four procedures as previously described, then:

- Using a hand-coring device, collect subsamples according to the sediment horizons that have been identified visually. A recommended diameter of the hand-corer is 12 cm, which would yield a volume of 565 cm^3 for a 0 to 5 cm depth.
- The number of subsamples will depend on the volume requirements of the analytical methods.
- Do not subsample sediment that has been in direct contact with the box-core sampler.
- Subsamples may be composited to get sufficient material for the surface layer.
- The subsamples should be carefully hand picked or pressure sieved (Subsection 2.9.1) through an appropriately sized sieve, to remove the indigenous organisms that could interfere with the test organisms. These subsamples are suitable for toxicity tests and chemical analyses.
- If benthos data are required, separate subsamples should be wet-sieved through a sieve with 0.5 mm or other suitable mesh (Subsection 2.9.1).
- The material retained by the sieve can be sorted immediately to enumerate and identify the benthos, or placed in salt water, in a glass of nalgene container, to be sorted within 24 h of collection. If this is not possible, an appropriate preservative may be used and the sample sorted at a later date.
- All subsamples must be placed into the appropriate type of container (see Section 2.8, Table 7) and placed into the

transport container after labelling correctly (see Subsection 2.8.1).

- If further subsampling in the laboratory is required, homogenize the contents in the storage container (see Subsection 2.9.1) before subsampling.

3.6 Transport and Storage of Field-collected Sediments

The recommended procedures for transporting and storing field-collected samples are described in Section 2.8.

3.7 Manipulation of Field-collected Samples

The preparation of test samples for whole-sediment toxicity or bioaccumulation tests, physicochemical characterization, contaminant analyses (inorganic and organic), and extraction of sediment elutriate and pore water is detailed in Section 2.9. These procedures and methods apply to sediments from the disposal, dredge, and reference sites.

3.7.1 Preparation of Test Sample for Whole-sediment Toxicity Tests

The preparation of whole sediment samples for use in toxicity tests has been described in detail in Subsection 2.9.1. The following summarized procedures apply to sediments from dredge, disposal, and reference sites.

- Remove large rocks, debris, and interfering endemic organisms from the sample with instruments made of inert material.

- If sieving is necessary (see Subsection 2.9.1), pressure-sieve the sediments through sieves with the appropriate-sized mesh (e.g., 0.5 mm).

- Place the sediment into its storage container (see Subsection 2.8.1, Tables 7 and 8) and homogenize with a mechanical mixture until uniform in colour or texture (see Subsection 2.9.1).

- Stored samples of sieved sediment must be homogenized again before partitioning among the test chambers.

- Partition by coning or caking, and quartering, and randomly allocate samples of sediment to the test containers (see Subsection 2.9.1). A sediment splitter box may be used for sediments with a high water content.

3.7.2 Preparation of Test Sample for Chemical Characterization

Preparation procedures are generally specific to the type of test being performed (i.e., the analytical method dictates sample preparation); therefore, the analytical methods should be determined before sample collection and the manipulation of field-collected samples should be part of the study plan. The standard analytical manual for methods and procedures used by the analytical laboratory should be consulted to ascertain preparation procedures (e.g., Loring and Rantala, 1992).

The minimum information requirements for dredged-material disposal are source of material, levels of cadmium, mercury, PCBs, total PAHs, high and low molecular weight PAHs total organic carbon, and particle size. The minimum information requirements for disposal at new disposal sites are: bathymetry, sediment transport, salinity, current flows, and sediment chemistry which

must include levels of cadmium, mercury, PCBs, total PAHs, and high and low molecular weight PAHs (see the Permit Application in Appendix H).

3.7.3 Preparation of Test Sample for Collection of Pore Water

To prepare the sediment sample for separation of pore water follow the procedures in Subsection 2.9.3.

3.8 Sampling QA/QC for Open-water Disposal of Dredged Material

- DQO should be established before execution of the sampling program and should be a part of the study plan.
- A complete record of all field procedures, including field preparations, should be maintained. Data sheets must include locations of the study site and collection stations, sampling and sample handling methods, preservation techniques and reagents (if used), and storage procedures with the dates and times of collection, the name of the collector, and the type of vessel used for the collection. Examples of field-data sheets are presented in Appendix H.
- Written SOPs for field procedures and operation of equipment must be available at all times.
- A procedure for tracking samples from collection through to disposal of all samples must be in place (e.g., chain-of-continuity, Appendix H).
- Sample inventories must be maintained.
- The sediment sample should meet the criteria for acceptability.
- Travel and filter blanks (2) should be part of the sampling QA/QC program.
- A minimum of 1% of the field collected samples should be split samples, in order to assess the variation associated with sample handling.
- Replicate subsamples (5%) should be analyzed to assess subsampling variability.
- If replicate field-samples are composited (i.e., combining more than one sample per station), due to analytical cost constraints, then at least 5% of the stations, should not be composited in order to assess heterogeneity within the sampling stations.
- All sampling devices and instruments used to collect data in the field must be regularly maintained and calibrated; these activities should be documented.
- Only experienced personnel, familiar with all aspects of the study plan and sampling plan, should participate in the collection of field samples and data.
- Deviations from standard operating procedures, as well as those described in this document for the collection, handling, transport, storage, or manipulation of sediment samples must be described and reported.
- Detailed information on additional QA/QC procedures are presented by CCME (1993), USEPA (1992), and Keith (1990; 1992).

3.9 Summary of Recommendations for Open-water Disposal Studies

3.9.1 Study Plan

- The purpose and objective(s) of the dredging project must be stated clearly.
- The objectives of the sampling plan must be stated clearly.
- Accurately define the dreding area, the reference site, and the disposal site, and map them with coordinates.
- Access, review, and evaluate all available historical data (see Subsection 2.3.2 and 3.3.2), particularly previous submissions for Ocean Disposal Permits.
- Identify potential sources of contamination and plot their locations on the map.
- Identify sensitive areas and plot their locations on the map.
- Determine (e.g., acoustic survey technique) and validate (e.g., diver or electronic surveillance, or with a preliminary sampling survey) the location of the fine-grained sediments.
- Define the sediments at the dredge and/or disposal sites in terms of substrate type.
- Determine the depth of dredge cut(s) over the entire area.
- The sampling program should be completed in consultation with the Ocean Disposal Office before any action is taken.
- Select a positioning method with an accuracy of 1 to 10 m.
- Identify the types of analyses required for all field-collected samples.
- Determine the sample volume or weight necessary to satisfy the analytical requirements (i.e., physicochemical characterization, contaminant analyses, toxicity test methods, etc.) and the QA/QC program.
- The minimum volume of sediment per sample typically required for physicochemical characterization and biological assessment is 7 L of wet sediment assuming a water content of <90%.
- Determine the minimum number of stations to be sampled for each dredging project using the size of the dredged project as the main criterion (Appendix G) and use the availability and quality of historical data (e.g., with or without stratification) to determine the location of the sampling stations within the dredge site (see Subsection 3.3.3). Generally, one sediment sample is collected at each sampling station. Alternatively, the number of samples may be determined to achieve a minimum detectable difference at a specific confidence level and power (see Subsection 2.3.6 and Appendix C) and to meet the DQO of the QA/QC program (see Subsection 3.7).
- It is recommended that additional samples of test sediment be collected in areas most likely to be affected by urban centres, agricultural activities or industry (i.e., in the vicinity of sewage, industrial,

or municipal effluent outfalls, navigational routes, loading and unloading areas, such as refuelling points, and chemical transhipment areas, and catchments for drainage basins, etc.).

- The location of the sampling stations should be plotted with the appropriate co-ordinates on the map.

- The method of disposal must be defined in the study plan, including type of vessel, type of discharge (e.g., continuous, instantaneous, single hopper, stationary versus non-stationary), time of disposal, frequency of dumping, navigational route to the proposed disposal site, and speed of vessel during disposal.

Preparations for Field Sampling

- Careful preparation and planning should precede the sampling study and should include written protocols for logistical considerations and equipment checklists for each sampling activity. Consult Subsection 2.3.7 for detailed considerations or the summary in Section 2.10.

- Travel blanks should be included as part of the QA/QC program (Section 3.7) .

3.9.2 Sample and Field Data Collection

All Samples

- Detailed field notes and measurements must be recorded (see Section 2.10 for summary and the permit application in Appendix H) and the on-site data forms for sediment collection (i.e., sediment from dredge and reference sites) must be completed as part of the permit application (Appendix H).

- The QA/QC procedures for collection of sediment from dredge, disposal, or reference sites as outlined in Section 3.7 should be followed.

- All samples should be collected from the sampling stations designated in the sampling plan.

- Safety precautions associated with the collection of potentially hazardous material should be observed.

- Guidance on sampler operation is presented in Subsection 2.5.3.

- Where core samplers cannot be used to collect sediments, a surficial sample of sediment may be collected with a grab-sampling device.

- If surficial sediments are to be compared among sites (dredge, disposal, and reference), they should be collected with the same type of sampling device, where possible.

- Recommended surficial sediment collection devices, in order of preference, are box-core, hand-core, PONAR, Van Veen grab, or Smith-McIntyre samplers.

- The penetration depth of the grab sampler should be ≥ 15 cm.

Chemical Assessment

- Core samples should be collected, where possible, and the penetration depth should extend to the depth of the intended dredge cut for each sampling station.

- The coring devices recommended for the open-water disposal site are presented in Figure 5; whereas , those recommended for the dredge site are presented in either Figures 3, 4, or 5, depending on the type of environment.

- Recommended core samplers for subsurface sediments are gravity (1 to 2 m depths) and piston (> 2 m) corers.

Chemical and Biological Assessment

- Core samples should be collected, where possible, and the penetration depth should extend to the depth of the intended dredge cut for each sampling station.

- Box-core samples are recommended for the collection of fine-grained surficial sediments at both dredge and disposal sites. In shallow waters (e.g, $\leq$10 m depth) hand-coring devices are recommended for shallow dredge cuts. When box-core or hand-coring devices cannot be used to collect surficial sediments, either a PONAR, Smith-McIntyre, or Van Veen grab sampler may be used depending on substrate types, water depth, and conditions (see Section 2.5, Figures 3, 4, or 5). A penetration depth of $\geq$15 cm is desirable. Alpine or Phleger gravity-core or piston-core samplers are recommended for the collection of sediments at dredge sites where the dredge cut is greater than 0.5 m or where a vertical profile of subsurface sediment is required. The core liner should be glass, polyethylene, or polypropylene with an inner diameter of 100 ± 25 mm for cores $\leq$1.0 m in length and 75 ± 25 mm for those greater than 1.0 m in length.

- The volume of sediment collected should be sufficient to support physicochemical characterization and biological assessment (Table 3). A minimum of 7 L of sediment per sample is recommended where the biological assessment includes sediment toxicity and bioaccumulation tests.

- An analysis of the benthic community might be required as part of the disposal-site assessment and, therefore, additional subsampling (e.g., box-core sample) or sampling (e.g., hand-corers, grabs) of the surficial sediments may be required.

- Collection of sediments from natural control sites, together with test organisms, may be required as part of the biological assessment (see Subsection 3.4.3).

Reference Sediment

- Reference sites for which analytical data are already available should be used, where possible, and it is recommended that the proponent consult the regional office of Environment Canada (Appendix B) for advice, as needed. Otherwise the location of the reference site should be in the vicinity of the study site, within the same body of water, but at a location where the sediment is relatively free of the influence of the sources of contamination.

- The reference sediment should be generally comparable to the test sediment in terms of particle size distribution, organic carbon content, pH, and cation exchange capacity.

- The sampler used to collect sediment from the reference site should be identical to that used to collect surficial (0 to 5 cm) test sediments (e.g., disposal-site sediment) and handling procedures should be identical to those for the test sediments.

- The volume of sediment collected should be sufficient to support both chemical and biological assessment; therefore, a minimum volume of 7 L per sample is recommended.

- Sediment samples from the reference site should be collected concurrently, (e.g., within 48 h of the test sediments).

- The sediment collection forms must be completed as part of the permit application (Appendix H).

- All subsamples must be placed into the appropriate type of container (see Section 2.8, Table 7) and placed into the transport container after labelling correctly (see Subsection 2.8.1).

Control Sediment

- Control sediments for dredging studies should be collected from the same site(s) as the test organisms. If the test organisms are cultured in the laboratory with a formulated sediment, the formulated sediment can serve as a control sediment.

- A proponent should consult the permit-issuing office for information on the location of sites for the collection of test organisms to be used in toxicity tests.

- The volume of sediment collected should be sufficient to support holding the organisms in the laboratory, as well as physicochemical characterization and biological assessment. A minimum volume of 15 L per site is recommended.

- The sediment and test-organism collection forms must be completed and submitted as part of the permit application (Appendix H).

- The QA/QC procedures for the collection of test organisms, as outlined in the biological test method, should be followed.

- The QA/QC procedures for collection of control sediment as outlined in Section 3.7 should be followed.

3.9.3 Sample Handling

The procedures recommended in Section 2.7 also apply to dredge and disposal projects, with the following additions and/or qualifications:

All Samples

- Samples must be handled in a manner that satisfies the QA/QC program and DQO.

- Samples for trace metal analysis and biological testing should not contact metal surfaces.

- Samples for organic analyses should not contact plastics, other than high-density polyethylene.

- Protective clothing and equipment should always be worn or used when collecting and handling sediments.

- All sample handling should occur outside or in a fumehood vented to the outside of the building away from air intakes.

- A spill control protocol should be in place in the laboratory, or sampling vessel, and personnel must understand its contents.

- Disposal of hazardous dredged material must comply with existing bylaws and regulations.

Subsampling Core Samples

- Siphon off any remaining surface water, leaving the surface fines undisturbed.

- Mechanically extrude the top 5 cm, excluding the sediment that is in direct contact with the core liner (see Subsection 3.5.1), and place into the appropriate type of container (see Section 2.8, Table 7).

- Mechanically extrude the sections of core that contain visually distinct sediment horizons and place them in separate containers.

- The sediment in each container should be homogenized (see Subsection 2.9.1) until uniform in colour and texture.

- Collect, using at Teflon® or Teflon®-lined scoop, subsamples of sediment for metal and organic contaminant analysis, followed by subsamples for other chemical or physical characterization.

- The subsample volumes will depend on the analytical test requirements. Since analyses might be performed on a dry-weight basis, and the water content of sediments is variable (e.g., < 30 to 80%), collect at least 2 or 3 times the dry weight of sediment required for analyses.

- If maintenance of an oxygen-free environment for sediment handling is part of the study plan, extrusion of cores should take place in a modified glove box in the presence of an inert gas (e.g., Subsection 2.7.1).

Box-Core Samples

- Using a hand-coring device, collect subsamples to a depth of 0 to 5 cm and then from each visually distinct sediment horizon. A recommended diameter of the hand-corer is 12 cm, which would yield a volume of 565 cm^3 for a 0 to 5 cm depth.

- The number of subsamples will depend on the volume requirements of the analytical methods.

- Do not subsample sediment that has been in direct contact with the box-core sampler.

- Subsamples may be composited to get sufficient material for the surface layer.

- At least three subsamples of the 0 to 5 cm depth should be carefully hand-picked (Subsection 2.9.1) or pressure sieved (Subsection 2.9.1) through an appropriately sized sieve to remove the indigenous organisms that might interfere with the test organism. These subsamples are suitable for toxicity tests and chemical analyses.

- If benthos data are required, separate subsamples should be wet-sieved through a sieve with 0.5 mm or other suitable mesh.

- The material retained by the sieve can be sorted immediately to enumerate and identify the benthos, or placed in salt water in a glass or nalgene container, to be sorted with 24 h of collection. If this is not possible, an appropriate preservative may be used and the sample sorted at a later date.

- All subsamples must be placed into the appropriate type of container (see Section 2.8, Table 7) and placed into the transport container after labelling correctly (see Subsection 2.8.1).

- If further subsampling in the laboratory for the designated end uses is required, homogenize the contents in the storage container (see Subsection 2.7.1) before subsampling.

Grab Samples

- Carefully hose with water, any sediment adhering to the outside of the sampler which could potentially comtaminate the sample during subsampling.

- Place the sampler on a platform (e.g., table) that is stable and readily accessible.

- Let the sample "stand" for a few minutes.

- Open the top (or side) of the sampler and examine the sample. Record the appearance of the sample (see Section 2.4). If it is acceptable (see Subsection 2.5.4), then carefully siphon-off the overlying water, if present, leaving the fine-grained surface sediment undisturbed, and discard.

- Field-measurements may be made on the sample before subsampling (see Section 2.4).

- Using a Teflon®-lined scoop or hand-coring device, carefully remove the top 0 to 5 cm and place into a sample container filling to the brim.

- Cap after topping with a thin layer of de-oxygenated water, if necessary, to assist with the exclusion of air.

- Repeat the sectioning procedure according to the sediment horizons.

- If the sampler does not allow access to the surface, the contents must be carefully deposited into a container with as little disturbance as possible. Subsamples can then be collected from the centre of the sample with a hand corer or scoop.

- Subsampling in an oxygen-free environment may be desirable depending on the objectives of the study, and procedures are described in Subsection 2.7.2.

3.9.4 Sample Containers

- Follow the procedures recommended in Subsection 2.8.1 and summarized in Section 2.10.

3.9.5 Sample Transport

- Follow the procedures recommended in Subsection 2.8.3, and summarized in Section 2.10.

3.9.6 Sample Storage

Follow the procedures recommended in Subsection 2.8.2 and summarized in Section 2.10 where applicable, except for the following.

- The elapsed time between collection and analysis should be as short as possible. Sediments destined for organic and inorganic contaminant analysis should be prepared and tested within 14 days (preferably 7) of collection. Sediments destined for biological testing should be hand picked to remove indigenous organisms that might interfere with the test organism, as soon as possible after collection, and prepared and used in a test within two weeks of collection. At all times the samples of sediment should be kept refrigerated at 4 ± 2° C, tightly sealed, and in the dark. Where hand picking is ineffective, pressure sieve the sediments. However, sieving of sediments is to be avoided if possible.

- A maximum permissible cold-storage time for sediments destined for toxicity tests is six weeks.

- Dredged material destined for chemical analysis may be stored at -20 ± 2° C for up to 60 days.

3.9.7 Test Sample Preparation

The recommendations presented in Section 2.9, which are summarized in Section 2.10 and Subsections 3.7.1 or 3.7.2, also apply to dredge and disposal projects.

Test sample preparation can include removal of indigenous organisms by hand picking or pressure sieving (Subsection 2.9.1), homogenizing and partitioning a sediment sample for distribution to test containers (Subsection 2.9.1), drying, crushing/grinding (Subsection 2.9.2), wet sieving (Subsection 2.9.1), or dewatering (Subsection 2.9.3) sediments.

References

Adams, D.D., "Sampling Sediment Pore Water", pp. 203–202, *in: CRC Handbook of Techniques for Aquatic Sediments Sampling,* A. Mudroch and S.D. MacKnight (eds.) CRC Press, Inc. Boca Raton, FL, 210 p. (1991).

Adams, D.D., D.A. Darby, and R.J. Young, "Selected Analytical Techniques for Characterizing the Metal Chemistry and Geology of Fine-grained Sediments and Interstitial Water", p. 3, *in: Contaminants and Sediments,* R.A. Baker (ed.), Ann Arbor Science Publishers, MI (1980).

Adams, W.U., R.A. Kimerle, and R.G. Mosher, "Aquatic Safety Assessment of Chemicals Sorbed to Sediments", pp. 429–453, *in: Aquatic Toxicology and Hazard Assessment,* R.D. Cardwell, R. Purdy, and R.C. Bahner (eds.), American Society for Testing and Materials, Philadelphia, PA (1985).

Allen, H.E., G. Fu, and B. Deng, "Analysis of Acid-volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM) for the Estimation of Potential Toxicity in Aquatic Sediments", *Environ. Toxicol. Chem., 12:*1441–1453 (1993).

Alldredge, J.R., "Sample Size for Monitoring of Toxic Chemical Sites", *Environ. Monitor. Assess.,* 9(2):143–154 (1987).

Anderson, J., W. Birge, J. Gentile, J. Lake, J. Rodgers, Jr., and R. Swartz, "Biological Effects, Bioaccumulation and Ecotoxicology of Sediment-associated Chemicals", pp. 267–296, *in: Fate and Effects of Sediment Bound Chemicals in Aquatic Systems: Proceedings of the 6th Pellsonton Workshop,* Florrisant, CO, Aug. 12–17, 1984, Pergamon Press, New York, NY (1987).

Ankley, G.T., V.R. Mattson, E.N. Leonard, C.W. West, and J.L. Bennett, "Predicting the Acute Toxicity of Copper in Freshwater Sediments: Evaluation of the Role of Acid-volatile Sulfide", *Environ. Toxicol. Chem., 12:* 315–320 (1993).

Ankley, G.T., G.L. Phipps, E.N. Leonard, D.A. Benoit, V.R. Mattson, P.A. Kosian, A.M. Cotter, J.R. Dierkes, D.J. Hansen, and J.D. Mahony, "Acid Volatile Sulfide as a Factor Mediating Cadmium and Nickel Bioavailability in Contaminated Sediments", *Environ. Toxicol. Chem., 10:*1299–1307 (1991a).

Ankley, G.T., M.K. Schubauer-Berigan, J.R. Dierkes, and M.T. Lukasewycz, "Sediment Toxicity Identification Evaluation: Phase I (Characterization), Phase II (Identification) and Phase III (Confirmation)", Modifications of Effluent Procedures, National Effluent Toxicity Assessment Centre Technical Report, 68-91, Draft VII, 12 p. (1991b).

Ankley, G.T., M.K. Schubauer-Berigan, and J.R. Dierkes, "Predicting the Toxicity of Bulk Sediments to Aquatic Organisms with Aqueous Test Fractions: Pore Water Versus Elutriate", *Environ. Toxicol. Chem., 10:*1359–1366 (1991c).

ASTM (American Society for Testing and Materials) E 1391-90, Standard Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing, pp. 1134–1153,

in: 1992 Annual Book of ASTM Standards, Vol. 11.04, Section 11, Phliadelphia, PA (1992a).

ASTM (American Society for Testing and Materials) E 1383-92, *Standard Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates,* Annual Book of ASTM Standards, Vol. 11.04, Section 11, Philadelphia, PA (1992b).

ASTM (American Society for Testing and Materials), "Standard Test Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates", Draft 2/4/94, prepared for ASTM Committee E47, ASTM, Philadelphia, PA, 157+ p. (1995).

Atkinson, G., *"Ocean Dumping Control Act"* Sampling: Project U390, Ocean Dumping Program, Environmental Protection Service, Environment Canada, Ottawa, Ontario, 17 p. (1985).

Atkinson, G., "Guidance Document on Sampling", a report prepared for the Open-water Disposal Program of the Office of Waste Management, Environmental Protection Service, Environment Canada, Ottawa, Ontario (1994).

Barnes, R.O., "An *in-situ* Interstitial Water Sampler for Use in Unconsolidated Sediments", *Deep Sea Res., 20:*1125–1128 (1973).

Barth, D.E. and T. Starks, "Sediment Sampling Quality Assurance User's Guide", prepared for Environmental Monitoring Systems Laboratory, United States Environmental Protection Agency, 99 p. (1985).

Baudo, R., "Sediment Sampling, Mapping, and Data Analysis", pp.15–60, *in: Sediments: Chemistry and Toxicity of In-place Pollutants,* R. Baudo, J.P. Giesy, and H. Muntau (eds.), Lewis Publishers, Inc., Chelsea, MI (1990).

Baudo, R., J.P. Giesy, and H. Muntau (eds.), *Sediments: Chemistry and Toxicity of In-place Pollutants,* Lewis Publishers, Inc., Chelsea, MI, 405 p. (1990).

Bauer, J.E., P.A. Montagna, R.B. Spies, M.C. Prieto, and D. Hardin, "Microbial Biogeochemistry and Heterotrophy in Sediments of a Hydrocarbon Seep", *Limnol. Oceanogr., 33:*1493–1513 (1988).

Belzile, N., R.R. De Vitre, and A. Tessier, *"In-situ* Collection of Diagenetic Iron and Manganese Oxyhydroxides from Natural Sediments", *Nature, 340* (6232):376–377 (1989).

Bender, M.B., W. Martin, J. Hess, F. Sayles, L. Ball, and C. Lambert, "A Whole-core Squeezer for Interfacial Pore-water Sampling", *Limnol. Oceanogr., 32(6):*1214–1225 (1987).

Bennett, J.R., "The Physics of Sediment Transport, Resuspension, and Deposition", *Hydrobiologia, 149:*5–12 (1987).

Bischoff, J.L., R.E. Green, and A.O. Luistro, "Composition of Interstitial Waters of Marine Sediments: Temperature and Squeezing Effects", *Science, 167:*1245–1246 (1970).

Blau, K. and G.S. King (eds.), "Acylation", pp. 104–151, *in: Handbook of Derivatives for Chromatography,* Hayden, London (1977).

Blomqvist, S., "Sampling Performance of Ekman Grabs - *In-situ* Observations and Design Improvements", *Hydrobiologia, 206:*245–254 (1990).

Bottomley, E.Z. and I.L. Bayly, "A Sediment Porewater Sampler Used in Root Zone Studies of the Submerged Macrophyte, *Myriophyllum spicatum*", *Limnol. Oceanogr., 29:*671–673 (1984).

Bray, J.T., O.P. Bricker, and B.N. Troup, "Phosphate in Interstitial Waters of Anoxic Sediments: Oxidation Effects During Sampling Procedures", *Science, 180:*1362–1364 (1973).

Brinkman, A.G., W. van Raaphorst, and L. Lijklema, "*In-situ* Sampling of Interstitial Water from Lake Sediments", *Hydrobiologia, 92:*659–663 (1982).

Brouwer, H., T. Murphy, and L. McArdle, "A Sediment-contact Bioassay with *Photobacterium phosphoreum*", *Environ. Toxicol. Chem., 9:*1353–1358 (1990).

Bryden, G.W. and L.R. Smith, "Sampling for Environmental Analysis Part 1: Planning and Preparation", *Amer. Lab.,* 2*/(7):*30–39 (1989).

Buddensiek, V., H. Engel, S. Fleischauer-Rössing, S. Olbrich, and K. Wächtler, "Studies on the Chemistry of Interstitial Water taken from Defined Horizons in the Fine Sediments of Bivalve Habitats in Several Northern German Lowland Waters", I: Sampling Techniques", *Arch. Hydrobiol., 119(1):* 55–64 (1990).

Burgess, R.M., B.A. Rogers, S.A. Rego, J.M. Corbin, and G.E. Morrison, "Sand spiked with Copper as a Reference Toxicant Material for Sediment Toxicity Testing: A Preliminary Evaluation", *Arch. Environ. Contam. Toxicol., 26:*163–168 (1994a).

Burgess, R.M. and G.E. Morrison, "A Short-exposure, Sublethal, Sediment Toxicity Test Using the Marine Bivalve *Mulinia lateralis:* Statistical Design and Comparative Sensitivity", *Environ. Toxicol. Chem., 13 (*4):571–580 (1994b).

Burton, G.A. Jr. (ed.), *Sediment Toxicity Assessment,* Lewis Publishers, Inc. Chelsea, MI, 457 p. (1992).

Burton, G.A. Jr., J.M. Lazorchak, W.T. Waller, and G.R. Lanza, "Arsenic Toxicity Changes in the Presence of Sediment", *Bull. Environ. Contam. Toxicol. Chem., 38:*491–499 (1987).

Burton, G.A. Jr., B.L. Stemmer, K.L. Winks, P.E. Ross, and L.C. Burnett, "A Multitrophic Level Evaluation of Sediment Toxicity in Waukegan and Indiana Harbors", *Environ. Toxicol. Chem., 8*:1057–1066 (1989).

Cahill, R.A., "Geochemistry of Recent Lake Michigan Sediments", Illinois State Geological Survey, Circular 517, Champaign, IL, 94 p. (1981).

Cairns, M.A., A.V. Nebeker, J.H. Gakstatter, and W.L. Griffis, "Toxicity of Copper-spiked Sediments to Freshwater Invertebrates", *Environ. Toxicol. Chem., 3:*435–445 (1984).

Carignan, R., "Intersititial Water Sampling by Dialysis: Methodological Notes", *Limnol. Oceanogr., 29:*667–670 (1984).

Carignan, R. and D.R.S. Lean, "Regeneration of Dissolved Substances in

a Seasonally Anoxic Lake: the Relative Importance of Processes Occurring in the Water Column and in the Sediments", *Limnol. Oceanogr., 36*(43):683–707 (1991).

Carignan, R. and A. Tessier, "Zinc Deposition in Acid Lakes: The Role of Diffusion", *Science, 228:*1524–1528 (1985).

Carignan, R., F. Rapin, and A. Tessier, "Sediment Porewater Sampling for Metal Analysis: A Comparison of Techniques", *Geochim. et Cosmochim. Acta, 49:*2493–2497 (1985).

Carignan, R., S. St-Pierre, and R. Gächter, "Use of Diffusion Samplers in Oligotrophic Lake Sediments: Effect of Free Oxygen in Sampler Material", *Limnol. Oceanogr., 39*(2):468–474 (1994).

Carr, R.S. and D.C. Chapman, "Comparison of Methods for Conducting Marine and Estuarine Sediment Porewater Toxicity Tests-extraction, Storage, and Handling Techniques", *Arch. Environ. Contam. Toxicol., 28:*69–77 (1995).

Carr, R.S. and D.C. Chapman, "Comparison of Solid-phase and Pore-water Approaches for Assessing the Quality of Marine and Estuarine Sediments", *Chem. Ecol., 7:*19–30 (1992).

Carr, R.S., J.W. Williams, and C.T.B. Fragata, "Development and Evaluation of a Novel Marine Sediment Pore Water Toxicity Test with the Polychaete *Dinophilus gyrociliatus*", *Environ. Toxicol. Chem., 8:*533–543 (1989).

Casas, A.M. and E.A Crecelius, "Relationship Between Acid Volatile Sulfide and the Toxicity of Zinc, Lead and Copper in Marine Sediments", *Environ. Toxicol. Chem., 13*(3):529–536 (1994).

CCME (Canadian Council of Ministers of the Environment), "Guidance Manual on Sampling, Analysis, and Data Management for Contaminated Sites, Volume I: Main Report", National Contaminated Sites Remediation Program Report EPC-NCS62E, CCME, Ottawa, Ontario, 79 p. (1993).

Centre St.-Laurent, "Methods Manual for Sediment Characerization", Conservation and Protection, Environment Canada - St. Lawrence Centre and the Quebec Ministry of Environment, Longueuil, Quebec, 144 p. (1992).

Chapman, P.M., "Marine Sediment Toxicity Tests", pp. 391–402, in: *Chemical and Biological Characterization of Sludges, Sediments, Dredge Spoils and Drilling Muds,* J.J. Lichtenberg, F.A. Winter, C.I. Weber, and L. Fradkin (eds.), STP 976, American Society for Testing and Materials, Philadelphia, PA (1988).

Clark, J.R., J.M. Patrick, Jr., J.C. Moore, and E.M. Lores, "Waterborne and Sediment-source Toxicities of Six Organic Chemicals to Grass Shrimp (*Palaemonetes pugio*) and Amphioxus (*Branchiostoma caribaeum)", Arch. Environ. Toxicol., 16:*401–407 (1987).

Cleveland, L., J.J. Coyle, C.G. Ingersoll, and E.A. Crecelius, "Great Lake Areas of Concern: Sediment Pore-water and Elutriate Chemistry and Toxicity of Elutriates to *Daphnia magna* and *Photobacterium phosphoreum* (Microtox)", *Environ. Toxicol. Chem., (1995).*

Cohen, J. (ed.), *Statistical Power Analysis*

for the Behavioural Sciences, Academic Press, New York, NY, pp. 52–68 (1977).

Crevello, P.D., J.M. Rine, and D.E. Lanesky, "A Method for Impregnating Unconsolidated Cores and Slabs of Calcareous and Terrigenous Muds", *J. Sed. Petrol., 51:*658–660 (1981).

de Groot, A.J. and K.H. Zschuppe, "Contribution to the Standardization of the Methods of Analysis for Heavy Metals in Sediments", *Rapp, P.-v. Reun. cons. Int. Explor. Mer., 181:*111–122 (1981).

Day, K.E., personal communication, Research Scientist, Inland Waters Directorate, Environment Canada, National Water Rsearch Institute, Burlington, Ontario (1993).

Day, K.E., R. Scott Kirby, and T.B. Reynoldson, "The Effect of Manipulations of Freshwater Sediments on Responses of Benthic Invertebrates in Whole-sediment Toxicity Tests", *Environ. Toxicol. Chem.,* (in press) (1995).

DeWitt, T.H., R.C. Swartz, and J.O. Lamberson, "Measuring the Acute Toxicity of Estuarine Sediments", *Environ. Toxicol. Chem., 8:*1035–1048 (1989).

Di Toro, D.M., "A Review of the Data Supporting the Equilibrium Partitioning Approach to Establishing Sediment Quality Criteria", report to the National Research Council (as cited in Baudo 1990) (1989).

Di Toro, D.M., J.D. Mahony, D.J. Hansen, K.J. Scott, M.B. Hicks, S.M. Mayr, and M.S. Richmond, "Toxicity of Cadmium in Sediments: the Role of Acid Volatile Sulfide", *Environ. Toxicol. Chem., 9:*1487–1502 (1990).

Di Toro, D.M., J.D. Mahony, D.J. Hansen, K.J. Scott, A.R. Carlson, and G.T. Ankley, "Acid Volatile Sulfide Predicts the Acute Toxicity of Cadmium and Nickel in Sediments", *Environ. Sci. Technol., 26:*96–101 (1992).

Ditsworth, G.R., D.W. Schults, and J.K.P. Jones, "Preparation of Benthic Substrates for Sediment Toxicity Testing", *Environ. Toxicol. Chem., 9:*1523–1529 (1990).

Downing, J.A. and L.C. Rath, "Spatial Patchiness in the Lacustrine Sedimentary Environment", *Limnol. Oceanogr., 33:*447–458 (1988).

EC (Environment Canada), "Sampling for Water Quality", Water Quality Branch, Inland Waters Directorate, EC, Ottawa, Ontario, 55 p. (1983).

EC (Environment Canada), "Bottle Washing Procedures", National Water Quality Laboratory, Inland Waters Direcgtorate, EC, National Research Institute, Burlington, Ontario (1989).

EC (Environment Canada), "Biological Test Method: Acute Lethality Test Using Rainbow Trout", Environmental Protection Series Report, EPS 1/RM/9, Ottawa, Ontario, 51 p. (1990a).

EC (Environment Canada), "Biological Test Method: Acute Lethality Test Using Threespine Stickleback Gasterosteus aculeatus", Environmental Protection Series Report, EPS 1/RM/10, Ottawa, Ontario, 45 p. (1990b).

EC (Environment Canada), "Biological Test

Method: Acute Lethality Test Using *Daphnia* spp.", Environmental Protection Series Report, EPS 1/RM/11, Ottawa, Ontario, 57 p. (1990c).

EC (Environment Canada), "Technical Guidance Manual for Aquatic Environmental Effects Monitoring at Pulp and Paper Mills: Volume 2 Methodology", Environment Canada and Department of Fisheries and Oceans, Ottawa, Ontario, 199 p. (Draft Report) (1991).

EC (Environment Canada), "Biological Test Method: Test of Reproduction and Survival Using the Cladocean *Ceriodaphnia dubia*", Environmental Protection Series Report, EPS 1/RM/21, Ottawa, Ontario, 72 p. (1992a).

EC (Environment Canada), "Biological Test Method: Test of Larval Growth and Survival Using Fathead Minnows", Environmental Protection Series Report, EPS 1/RM/22, Ottawa, Ontario, 70 p. (1992b).

EC (Environment Canada), "Biological Test Method: Fertilization Assay Using Echinoids (Sea Urchins and Sand Dollars)", Environmental Protection Series Report, EPS 1/RM/27, Ottawa, Ontario, 97 p. (1992c).

EC (Environment Canada), "Biological Test Method: Acute Test for Sediment Toxicity Test Using Marine or Estuarine Amphipods", Environmental Protection Series Report, EPS 1/RM/26, Ottawa, Ontario, 83 p. (1992d).

EC (Environment Canada), "Biological Test Method: Toxicity Test Using Luminescent Bacteria (*Photobacterium phosphoreum*)", Environmental Protection Series Report, EPS 1/RM/24, Ottawa, Ontario, 61 p. (1992e).

EC (Environment Canada), "Biological Test Method: Growth Inhibition Test Using the Freshwater Alga *Selenastrum capriocornutum*", Environmental Protection Series Report, EPS 1/RM/25, Ottawa, Ontario, 41 p. (1992f).

EC (Environment Canada), "Biological Test Method: Toxicity Tests Using Early Life Stages of Salmonid Fish (Rainbow Trout, Coho Salmon, or Atlantic Salmon)", Environmental Protection Series Report, EPS 1/RM/28, Ottawa, Ontario, 91 p. (1992g).

EC (Environment Canada), "Interim Monitoring Guidelines for Ocean Disposal", Marine Environment Division, Environment Canada, Ottawa, Ontario, Draft (July) 34 p. (1993).

EC (Environment Canada), "User's Guide to the Application Form for Ocean Disposal", Internal Report prepared by Marine Environment Division, EPS, 121 p. (1995a).

EC (Environment Canada), "Guidance Document on Measurement of Toxicity Test Precision Using a Control Sediment Spiked with a Reference Toxicant", Conservation and Protection, Ottawa, Ontario, (draft) (1995b).

EC (Environment Canada), Draft Technical Guidance on Physical Monitoring, prepared by Seaconsult Marine Research Ltd., Coastal and Ocean Resources Inc. and Office of Waste Management, Environment Canada, Ottawa, Canada (1994a).

EC (Environment Canada), *Sampling and*

Analysis in the Arctic Marine Benthic Environment, Volume I. Review of Methods, Environmental Protection Service, Ottawa, Ontario (1985).

EC (Environment Canada), "Test for Growth and Survival in Sediment Using the Freshwater Amphipod *Hyalella azteca*", Environmental Protection Service, Ottawa, Ontario (1996a).

EC (Environment Canada), "Test for Growth and Survival in Sediment Using Larvae of Freshwater Midges (*Chironomus tentans, Chironomus riparius)*", Environmental Protection Service, Ottawa, Ontario (1996b).

Edmunds, W.M. and A.H.Bath, "Centrifuge Extraction and Chemical Analysis of Interstitial Waters", *Environ. Sci. Tech., 10*(5):467–492 (1976).

Emerson, S., R. Jahnke, M. Bender, P. Froelich, G. Klinkhammer, C. Bowser, and G. Setlock, "Early Diagenesis in Sediments from the Eastern Equatorial Pacific, I. Pore Water Nutrient and Carbonate Results", *Earth Planet. Sci. Lett., 49:*57 (1980) (as cited in Adams, 1991).

Flegal, A.R., R.W. Risebrough, B. Anderson, J. Hunt, S. Anderson, J. Oliver, M. Stephenson, and R. Pickard, "San Francisco Estuary Pilot Regional Monitoring Program: Sediment Studies", San Francisco Bay Regional Water Quality Control Board/State Water Resources Control Board, Oakland, CA (1994).

Friedman, H., "Simplified Determinations of Power, Magnitude of Effect and Research Sample Sizes", *Educ. Psych. Meas., 42:*521–527 (1982).

Froelich, P.N., G.P. Klinkhammer, M.L. Bender, N.A. Luedtke, G.R. Heath, D. Cullen, P. Dauphin, D. Hammond, B. Hartmann, and V. Maynard, "Early Oxidation of Organic Matter in Pelagic Sediments of the Eastern Equatorial Atlantic: Suboxic Diagenesis", *Geochim. Cosmochim. Acta, 43:*1075–1090 (1979).

Garner, F.C., M.A. Stapanian, and L.R. Williams, "Composite Sampling for Environmental Sampling", *in: Principles of Environmental Sampling,* L.H. Keith (ed.), American Chemical Society, Washington DC, 363 p. (1988).

Giam, C.S. and M.K. Wong, "Problems of Background Contamination in the Analysis of Open Ocean Biota for Chlorinated Hydrocarbons", *J. Chromatogr., 72:*283–292 (1972)

Giesy, J.P., personal communication, Department of Fisheries and Wildlife, Michigan State University, East Lansing, MI (1992).

Giesy, J.P., and R.A. Hoke, "Freshwater Sediment Quality Criteria: Toxicity Bioassessment", pp. 265–348, *in: Sediments: Chemistry and Toxicity of In-place Pollutants,* R. Baudo, J.P. Giesy and H. Muntau (eds.), Lewis Publishers, Inc. Chelsea, MI, 405 p. (1990).

Giesy, J.P., C.J. Rosiu, R.L. Graney, and M.G. Henry, "Benthic Invertebrate Bioassays with Toxic Sediment and Pore Water", *Environ. Toxicol. Chem., 9:*233–248 (1990).

Ginsburg, R.N., H.A., Bernard, R.A. Moody, and E.E. Daigle, "The Shell Method of Impregnating Cores of Unconsolidated Sediments", *J. Sed. Petrol, 36:*1118–1125 (1966).

Glass, G.E. and J.E. Poldoski, "Interstitial Water Components and Exchange Across the Water Sediment Interface in Lake Superior", *Berh. Int. Verhin. Limnol., 19:*405–420 (1975).

Golterman, H.L., P.G. Sly, and R.L. Thomas, *Study of the Relationship Between Water Quality and Sediment Transport,* published by the United Nations Educational, Scientific, and Cultural Organization (UNESCO), Mayenne, France, 231 p. (1983).

Green, R.H., *Sampling Design and Statistical Methods for Environmental Biologists,* Wiley Interscience, New York, NY, 257 p. (1979).

Green, R.H., "Power Analysis and Practical Strategies for Environmental Monitoring", *Environ. Res., 50:*195–205 (1989).

Guenther, W.C., "Sample Size Formulas for Normal Theory, T-tests", *Amer. Stat., 35:*243–244 (1981).

Guenther, W.C., "Normal Theory Sample Size Formulas for Some Non-normal Distributions", *Commun. Stat., B11:*727–732 (1982).

Guigné, J.Y., N. Rukavina, P.H. Hunt, and J.S. Ford, "An Acoustic Parametric Array for Measuring the Thickness and Stratigraphy of Contaminated Sediments", *J. Great Lakes Res., 17*(1):120–131 (1991).

Haddock, J.D., P.F. Landrum and J.P. Giesy, "Extraction Efficiency of Anthracene from Sediments", *Anal. Chem., 55:*1197–1200 (1983).

Håkanson, L., "Sediment Sampling in Different Aquatic Environments: Statistical Aspects", *Water Resour. Res., 20:*41–46 (1984).

Håkanson, L. and M. Jansson, *Principles of Lake Sedimentology,* Springer-Verlag, Berlin, GD, 316 p., (1983).

Hardy, J.T., S. Kiesser, L. Antrim, A. Stubin, R. Kocan, and J. Strand, "The Sea-surface Microlayer of Puget Sound: Part I. Toxic Effects on Fish Eggs and Larvae", *Marine Environ. Res., 23:*227–249 (1987).

Harkey, G.A., P.F. Landrum, and S.J. Kaine, "Comparison of Whole-sediment, Elutriate, and Pore-water Exposures for Use in Assessing Sediment-associated Organic Contaminants in Bioassays", *Environ. Toxicol. Chem., 13*(8):1315–1329 (1994).

Henrikson, A. and R.F. Wright, "Effects of Acid Precipitation on a Small Acid Lake in Southern Norway", *Nordic Hydrol., 8:*1–10 (1977).

Hertkorn-Obst, U., H. Wendeler, T. Feurerstein, and W. Schmitz, "A Device for Sampling Interstitial Water Out of River and Lake Beds", *Environ. Technol. Lett. 3:*263 (1982).

Hesslein, R.H., "An *in-situ* Sampler for Close Interval Pore Water Studies", *Limnol. Oceanogr., 21:*912–914 (1976).

Hesslein, R.H., personal communication, Department of Fisheries and Oceans, Winnipeg, Manitoba (1993).

Hickey, C.W., personal communication, NIWA Ecosystems, National Institute of

Water and Atmospheric Research, Hamilton, New Zealand (1994).

Hilton, J., J.P. Lishman, and P.V. Allen, "The Dominant Processes of Sediment Distribution and Focusing in a Small, Eutrophic, Monomictic Lake", *Limnol. Oceanogr., 31:*125–133 (1986).

Hines, M.E., S.L. Knollmeyer, and J.B. Tugel, "Sulfate Reduction and Other Sedimentary Biogeochemistry in a Northern New England Salt Marsh", *Limnol. Oceanogr., 34:*578–584 (1989).

Hoke, R.A., J.P. Giesy, G.R. Ankley, J.L. Newsted, and J. Adams, "Toxicity of Sediments from Western Lake Erie and Maumee River at Toledo, Ohio, 1987: Implications for Current Dredged Material Disposal Practicies", *J. Great Lakes Res., 16* (3):457–470 (1990).

Holland, P.T., C.W. Hickey, D.S. Roper, and T.M. Trower, "Variability of Organic Contaminants in Inter-tidal Sandflat Sediments from Manukau Harbour, New Zealand", *Arch. Environ. Contam. Toxicol., 25:*456–463 (1993).

Hood, M.A. and G.E. Ness, "Survival of *Vibrio cholerae* and *Escherichia coli* on Esturaine Waters and Sediments", *Appl. Environ. Microbiol., 43:*578–584 (1982).

Howes, B.L., J.W.H. Dacey, and S.G. Wakeham, "Effects of Sampling Technique on Measurements of Porewater Constituents in Salt Marsh Sediments", *Limnol. Oceanogr., 30(1):*221–227 (1985).

Hurlbert, M.H. and M.P. Brindle, "Effects of Sample Handling on the Composition of Marine Sedimentary Pore Water", *Geol. Soc. of Amer. Bull., 86:*109–110 (1975).

IJC (International Joint Commission), Procedures for the Assessment of Contaminated Sediment Problems in the Great Lakes, Sediment Subcommittee and its Assessment Work Group, 140 p. (1988).

Ingersoll, C.G. and M.K. Nelson, Testing Sediment Toxicity with *Hyalella azteca* (Amphipoda) and *Chironomus riparius* (Diptera), pp. 93–109, *in: Aquatic Toxicology and Risk Assessment, Volume 13,* W.G. Landis and W.H. van der Schalie (eds.) ASTM STP 1096, American Society for Testing and Materials, Philadelphia, PA (1990).

Jahnke, R.A., "A Simple, Reliable, and Inexpensive Pore-water Sampler", *Limnol. Oceanogr., 33:*483–487 (1988).

Jenne, E.A. and J.M. Zachara, "Factors Influencing the Sorption of Metals", pp. 83-98, *in: Fate and Effects of Sediment-bound Chemicals in Aquatic Systems: Proceedings of the 6th Pellston Workshop,* Florissant, CO, Aug. 12–17, 1984, Pergamon Press, New York, NY (1987).

Johns, D.M., R.A. Pastorok, and T.C. Ginn, "A Sublethal Sediment Toxicity Test Using Juvenile *Neanthes* sp. (Polychaeta: Nereidae)", pp. 280–293, *in: Aquatic Toxicology and Risk Assessment, Volume 14,* M.A. Mayes and M.G. Barron (eds.), ASTM STP 1124, American Society for Testing and Materials, Philadelphia, PA, 383 p. (1991).

Keilty, T.J., D.S. White, and P.F. Landrum, "Sublethal Responses to Endrin in Sediment by *Limnodrilius hoffmeisteri* (Tubificidae) and in Mixed-culture with

Stylodrilius heringianus (Lumbriculidae)", *Aquat. Toxicol., 13:*241–270 (1998a).

Keilty,T.J., D.S. White, and P.F. Landrum, Sublethal Responses to Endrin in Sediment by *Stylodriliuis heringianus* (Lumbriculidae) as Measured by a ^{137}Celsium Marker Layer Technique, *Aquat. Toxicol., 13:*227–250 (1988b).

Keilty, T.J., D.S. White, and P.F. Landrum, "Short-term Lethality and Sediment Avoidance Assays with Endrin-contaminated Sediment and Two Oligochaetes from Lake Michigan", *Arch. Environ. Toxicol., 17:*95–101 (1988c).

Keith, L.H., "Environmental Sampling: A Summary", *Environ. Sci. Technol., 24:*610–617 (1990).

Keith, L.H., *Environmental Sampling and Analysis: A Practical Guide,* Lewis Publishers, Inc., Chelsea, MI, 143 p., (1992).

Kelly, C.A., J.W.M. Rudd, A. Furutani, and D.W. Schindler, "Effects of Lake Acidification on Rates of Organic Matter Decomposition in Sediments", *Limnol. Oceanogr., 29:*687–694 (1984).

Kemble, N.E., J.M. Besserr, W.G. Brumbaugh, E.L. Brunson, T.J. Canfield, J.J. Coyle, F.J. Dwyer, J.F. Fairchild, C.G. Ingersoll, T.W. La Point, J.C. Meadows, D.P. Mondo, B.C. Poulton, D.F. Woodward, and J.L. Zäjicek, "Sediment Toxicology", pp. 2-1 to 2-100, *in: Effects of Metal-contaminated Sediment, Water, and Diet on Aquatic Organisms,* C.A. Ingersol, W.G. Brumbaugh, A.M. Farag, T.W. La Point, and D.F. Woodward (eds.), May 10, 1993, U.S. Environmental Protection Agency, Helena, MT (1993).

Klinkhammer, G.P., "Early Diagenesis in Sediments from the Equatorial Pacific. II. Pore Water Metal Results", *Earth Planet, Sci. Lett., 49:*81–101 (1980).

Knezovich, J.P. and F.L. Harrison, "A New Method for Determining the Concentrations of Volatile Organic Compounds in Sediment Interstitial Water", *Bull. Environ. Contam. Toxicol., 38:*937–940 (1987).

Knezovich, J.P. and F.L. Harrison, "The Bioavailability of Sediment-sorbed Chlorobenzenes to Larvae of the Midge, *Chironomous decorus*", *Ecotoxicol. Environ. Safety, 15:*226–241 (1988).

Knezovich, J.P., F.L. Harrison, and R.G. Wilhelm, "The Bioavailability of Sediment-sorbed Organic Chemicals: A Review", *Water Air Soil Pollut., 32*:233–245 (1987).

Kratochvil, B. and J.K. Taylor, "Sampling for Chemical Analysis", *Anal. Chem., 53*:924A–938A (1981).

Kriukov, P.A. and F.T. Manheim, "Extraction and Investigative Techniques for Study of Interstitial Waters of Unconsolidated Sediments: A Review", pp. 3–26, *in: The Dynamic Environment of the Ocean Floor,* K.A. Fanning and F.T. Manheim (eds.), Lexington Books, Washington, DC (1982).

Kupper, L.L. and K.B. Hafner, "How Appropriate are Popular Sample Size Formulas?", *Amer. Statist., 43:*101–105 (1989).

Lam, J., "Phthalates in filter paper", *Chem. Ind. Eng., 43*:1837 (1967).

Lamberson, J.O. and R.C. Swartz, "Use of Bioassays in Determining the Toxicity of Sediment to Benthic Organisms", pp. 257–279, *in: Toxic Contaminants and Ecosystem Health: A Great Lakes Focus*. M.S. Evans (ed.), John Wiley and Sons, New York, NY (1988).

Landrum, P.F. and W.R. Faust, "Effect of Variation in Sediment Composition on the Uptake Rate Coefficient for Selected PCB and PAH Congeners by the Amphipod, *Diporeia* sp.", pp. 263–279, *in: Aquatic Toxicology and Risk Assessment,* M.A. Mayes and M.G. Barron (eds.), STP 1124, American Association for Testing and Materials, Philadelphia, PA (1991).

Landrum, P.F., B.J. Eadie, and W.R. Faust, "Variation in the Bioavailability of Polycyclic Aromatic Hydrocarbons to the Amphipod *Diporeia* (spp.) with Sediment Aging", *Environ. Toxicol. Chem., 11:*1197–1208 (1992).

Lanesky, D.E., B.W. Logan, R.G. Brown, and A.C. Hine, "A New Approach to Portable Vibracoring Underwater and on Land", *J. Sed. Petrol, 49:*654–647 (1979).

Lawless, J.P. and J.W. Padan, "Developing Deep Seabed Minerals", *Sea Technol., 27:* 1–45 (1986).

Lee, G.F. and R.A. Jones, "Water Quality Significance of Contaminants Associated with Sediments: An Overview", pp. 1–34, *in: Fate and Effects of Sediment-bound Chemicals in Aquatic Systems,* K.L. Dickson, A.W. Maki, and W.A. Brungs (eds.), Pergamon Press, New York, NY (1984).

Lesht, B.M., "Non-parametric Evaluating of the Size of Limnological Sampling Networks: Application to the Design Survey of Green Bay", *J. Great Lakes Res., 14:*325–337 (1988).

Levi, I. and T.W. Novicki, "Interfering GLC Peaks from Materials and Chemicals in Pesticide Residue Analysis", *Bull. Environ. Contam. Toxicol., 7*:193–199 (1972).

Lonneman, W.A., J.J. Bufilini, R.L. Kuntz, and S.A. Meeks, "Contamination from Fluorocarbon Films", *Environ. Sci. Technol., 15*:99–103 (1981).

Loring, D.H. and R.T.T. Rantala, "Manual for the Geochemical Analyses of Marine Sediments and Suspended Particulate Matter", *Earth-Sci. Rev., 32*:235–283 (1992).

Lydy, M.J., K.A. Bruner, K.A. Fry, and S.W. Fisher, "Effects of Sediment and the Route of Exposure on the Toxicity and Accumulation of Neutral Lipophilic and Moderately Water Soluble Metabolizable Compounds in the Midge, *Chironomus riparius*", pp. 140–164, *in: Aquatic Toxicology and Risk Assessment, Volume 13,* W.G. Landis, and W.H. van der Schalie (eds.), ASTM STP 1096, American Society for Testing and Materials, Philadelphia, PA (1990).

Lyons, W.B., H.E. Gaudette, and G.M. Smith, "Pore Water Sampling in Anoxic Carbonate Sediments: Oxidation Artifacts", *Nature, 277:*48–49 (1979).

Malcolm, S.J., P.J. Kershaw, M.B. Lovett, and B.R. Harvey, "The Interstitial Water Chemistry of ^{239,240} Pu and ^{241}Am in the Sediments of the North-East Irish Sea", *Geochim. Cosmochim. Acta., 54*:29–35 (1990).

Malueg, K.W., G.S. Schuytema, and D.F. Krawczyk, "Effects of Sample Storage on a Copper-spiked Freshwater Sediment", *Environ. Toxicol. Chem., 5*:245–253 (1986).

Manheim, F.T., "Interstitial Waters of Marine Sediment", p. 115, *in: Chem. Oceanogr., Volume 6,* J.P. Riley and R. Chester (eds.), Academic Press, New York, NY (1976).

Martens, C.S. and J.V. Klump, "Biochemical Cycling in an Organic-rich Coastal Marine Basin. I. Methane Sediment-water Exchange Processes", *Geochim. Cosmochim. Acta, 44:*471–490 (1980).

Mayer, L.M., "Chemical Water Sampling in Lakes and Sediments with Dialysis Bags", *Limnol. Oceanogr., 21:*909–911 (1976).

Milton, J.S., J.J. Corbet, and P.M. McTeer, *Introduction to Statistics,* D.C Heath and Company, Toronto, Ontario, 517 p. (1986).

Montgomery, J.R., M.T. Price, J. Holt, and C. Zimmermann, "A Close-interval Sampler for Collection of Sediment Pore Waters for Nutrient Analyses", *Estuaries, 4*:75–77 (1981).

Montgomery, J.R., C.F. Zimmerman, and M.T. Price, "The Collection, Analysis and Variation of Nutrients in Estuarine Pore Water", *Estuarine Coastal Mar. Sci., 9:*203–214 (1979).

Moody, J.R. and R.M. Lindstrom , "Selection and Cleaning of Plastic Containers for Storage of Trace Element Samples", *Anal. Chem., 49*(14):2264–2267 (1977).

Mudie, P.J., D.J.W. Piper, K. Rideout, K.R. Robertson, C.T. Schafer, G. Vilks, and I.A. Hardy, "Standard Methods for Collecting, Describing, and Sampling Quaternary Sediments at the Atlantic Geoscience Centre", Geological Survey of Canada, Bedford Institute of Oceanography, Dartmouth, NS, 38+ p. (1984).

Mudroch, A., personal communication, Research Scientist, Inland Waters Directorate, Environment Canada, National Water Research Institute, Burlington, Ontario (1992).

Mudroch, A. and S.D. MacKnight, *CRC, Handbook of Techniques for Aquatic Sediments Sampling*, CRC Press, Boca Raton, FL 210 p. (1991).

Muir, D.C.G., N.P. Grift, B.E. Townsend, D.A. Metner, and W.L. Lockhart, "Comparison of the Uptake and Bioconcentration of Fluridone and Terbutryn by Rainbow Trout and *Chironomus tentans* in Sediment and Water Systems", *Arch. Environ. Contam. Toxicol., 11:*595–602 (1982).

Muir, D.C.G., G.P. Rawn, B.E. Townsend, W.L. Lockhart, and R. Greenhalgh, "Bioconcentration of Cypermethrin, Deltamethrin, Fenvalerate and Permethrin by *Chironomus tentans* Larvae in Sediment and Water", *Environ. Toxicol. Chem., 4*:51–61 (1985).

Murray, J.W. and V. Grundmanis, "Oxygen Consumption in Pelagic Marine Sediments", *Science, 209*:1527–1530 (1980).

Nath, B.N., V.S. Rajaraman, and A.V. Mudholkar, "Modified Interstitial Water Squeezer for Trace Metal Analysis", *Indian J. Marine Sci., 17*:71–72 (1988).

Nebeker, A.V., M.A. Cairns, J.H. Gakstatter, K.W. Malueg, G.S. Schuytema, and D.R. Krawczyk, "Biological Methods for Determining Toxicity of Contaminated Freshwater Sediments to Invertebrates", *Environ. Toxicol. Chem., 3*:617–630 (1984).

Nelson, M.K., J.J. Coyle, and G.A. Burton, "Testing the Toxicity of Field Collected Freshwater Sediments", *Midwest Pollution Control Biologists Meeting*, United States Environmental Protection Agency, Region 5 (1991).

Nelson, M.K., J.J. Coyle, L.B. King, N.E. Kemble, E.A. Crecelius, and L.E. Greer, "Whole Sediment Toxicity Tests. Biological Assessment of Contaminated Great Lakes Sediment", United States Environmental Protection Agency, Final Report, Assessment and Remediation of Contaminanted Sediments, Great Lakes National Program Office, Chicago, IL (1992).

Nishimura, A., "Bottom Sampling and Photographing on the Southeastern Offshore of the Boso Peninsula", *Cruise Rep. Geol. Surv. Jap., 19*:54–66 (1984).

Novicki, H.G., C.A. Kieda, R.F. Devine, V. Current, and T.H. Schaefors, "Identification of Organic Compounds Extracted from Paper and glass Soxhlet Thimbles", *Anal. Lett., A12*:769–776 (1979).

Othoudt, R.A.., J.P. Geisy, K.R. Grzyb, D.A. Verbrugge, R.A. Hoke, J.B. Drake, and D. Anderson, "Evaluation of the Effects of Storage Time on the Toxicity of Sediments", *Chemosphere, 22(9-10):*801–807 (1991).

Pastorok, R.A. and D.S. Becker, "Comparative Sensitivity of Sediment Toxicity Bioassays at Three Superfund Sites in Puget Sound", pp. 123–139, *in: Aquatic Toxicology and Risk Assessment: Thirteenth Volume*, W.G. Landis and W.H. van der Schalie (eds.), ASTM STP 1096, American Society for Testing and Materials, Philadelphia, PA (1990).

Patrick, W.H. Jr., R.P. Gambrell, and R.A. Khalid, "Physicochemical Factors Regulating Solubility and Bioavailability of Toxic Heavy Metals in Contaminated Dredged Sediment", *J. Environ. Sci. Health, A12*: 475–492 (1977).

Persaud, D., T.D. Lomas, and A. Hayton, The in-place Pollutants Program. Volume III. Phase I studies. Aquatic Biology Section, Water Resources Branch, Ontario Ministry of the Environment, Toronto, Ontario, 94 p. (1988).

Plumb, R.H., Jr., "Sampling, Preservation, and Analysis of Sediment Samples: State-of-the-art Limitations", presented at *The Fifth US/Japan Experts Meeting in the Management of Sediments Containing Toxic Substances,* New Orleans, LA (November 26–28, 1979).

Plumb, R.H., Jr., Procedures for Handling and Chemical Analysis of Sediment and Water Samples, Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material, Contract EPA-4805572010, EPA/CE-81-1, 478 p. (1981).

Power, E.A. and P.M. Chapman, "Assessing Sediment Quality", pp. 1–18, *in: Sediment Toxicity Assessment*, G.A.

Burton, Jr. (ed.), Lewis Publishers, Inc., Chelsea, MI, 457 p. (1992).

Rapin, F., A. Tessier, P.G.C. Campbell, and R. Carignan, "Potential Artifacts in the Determination of Metal Partitioning in Sediments by a Sequential Extraction Procedure", *Environ. Sci. Technol., 20*:836–840 (1986).

Redmond, M.S. and K.J. Scott, "Amphipod Predation by the Infaunal Polychaete, *Nephtys incis*", *Estuaries, 12*:205–207 (1989).

Reeburgh, W.S., "An Improved Interstitial Water Sampler", *Limnol. Oceanogr., 12*:163–165 (1967).

Reynoldson, T.B., K.E. Day, C. Clarke, and D. Milani, "Effect of Indigenous Animals on Chronic End Points in Freshwater Sediment Toxicity Tests", *Environ. Toxicol. Chem., 13*:973–977 (1994).

Robinson, A.M., J.O. Lamberson, F.A. Cole, and R.C. Swartz, "Effects on Culture Conditions on the Sensitivity of a Phoxocephalid Amphipod, *Rhepoxynius abronius*, to Cadmium in Sediment", *Environ. Toxicol. Chem., 7*:953–959 (1988).

Rochon, R. and M. Chevalier, Sediment Sampling and Preservation Methods for Dredging Projects, Conservation and Protection, Environment Canada, Ottawa, Canada (1987).

Rutledge, P.A. and J. Fleeger, Laboratory Studies on Core Sampling with Application to Subtidal Meiobenthos Collection, *Limnol. Oceanogr., 33*:274–280 (1988).

Saager, P.M., J-P. Sweerts, and H.J. Ellermeijer, "A Simple Pore-water Sampler for Coarse, Sandy Sediments of Low Porosity", *Limnol. Oceanogr., 35*(3):747–751 (1990).

Sayles, F.L. and F.T. Manheim, "Interstitial Solutions and Diagenesis in Deeply Buried Marine Sediments: Results from the Deep Sea Drilling Project", *Geochim. Cosmochim. Acta, 39*:103–127 (1975).

Sayles, F.L., P.C. Mangelsdorf, Jr., T.R.S. Wilson, and D.N. Hume, "A Sampler for the *in-situ* Collection of Marine Sedimentary Pore Waters", *Deep Sea Res., 23*:259–264 (1976).

Sayles, F.L., T.R.S. Wilson, D.N. Hume, and P.C. Mangelsdorf, Jr., *"In-situ* Sampler for Marine Sedimentary Pore Waters: Evidence for Potassium Depletion and Calcium Enrichment", *Science, 181:*154–156 (1973).

Schneider, D.C., J.M. Gagnon, and K.D. Gilkinson, "Patchiness of Epibenthic Megafauna on the Outer Grand Banks off Newfoundland", *Mar. Ecol. Prog. Ser., 39*:1–13 (1987).

Schock, S.G., L.R. LeBlanc, and L.A. Mayer, "Chirp Subbottom Profiler for Quantitative Sediment Analysis", *Geophysics, 54(4):*445–450 (1989).

Schuytema, G.S., P.O. Nelson, K.W. Malueg, A.V. Nebeker, D.F. Krawczyk, A.K. Ratcliff, and J.H. Gakstatter, "Toxicity of Cadmium in Water and Sediment to *Daphnia magna*", *Environ. Toxicol. Chem., 3*:293–308 (1984).

Shaner, S.W. and A.W. Knight, "The role of Alkalinity in the Mortality of *Daphnia*

magna in Bioassays of Sediment-bound Copper", *Comp. Biochem. Physiol., 82C*:273–277 (1985).

Simon, N.S., M.M. Kennedy, and C.S. Massoni, "Evaluation and Use of a Diffusion-controlled Sampler for Determining Chemical and Dissolved Oxygen Gradients at the Sediment-water Interface", *Hydrobiologia 126*:135–141 (1985).

Skei, J.M., "A Review of Assessment and Remediation Strategies for Hot Spot Sediments", *Hydrobiologia, 235/236*:629–638 (1992).

Sly, P.G., "Sedimentary Processes in Lakes", pp. 65-89, *in: Lakes: Chemistry Geology Physics*, A. Lerman (ed.), Springer-Verlag, New York, NY (1978).

Sly, P.G. and W.J. Christie, "Factors Influencing Densities and Distributions of *Pontoporeia hoyi* in Lake Ontario", *Hydrobiolgia, 235/236*:321–352 (1992).

Smith, J.N., B.P. Boudreau, and V. Noshkin, "Plutonium and ^{210}Pb Distributions in Northeast Atlantic Sediments: Subsurface Anomalies Caused by Non-local Mixing", *Earth Planet. Sci. Lett., 81*:15–28 (1986).

Steel, R.G.D. and J.H. Torrie, *Principles and Procedures of Statistics*, McGraw-Hill, New York, NY (1980).

Stemmer, B.L., G.A. Burton, Jr., and S. Leibfritz-Frederick, "Effects of Sediment Test Variables on Selenium Toxicity to *Daphnia magna*", *Environ. Toxicol. Chem., 9*:381–389 (1990a).

Stemmer, B.L., G.A. Burton, Jr., and G. Sasson-Brickson, "Effect of Sediment Spatial Variance and Collection Method on Cladoceran Toxicity and Indigenous Microbial Activity Determinations", *Environ. Toxicol. Chem., 9*:1035–1044 (1990b).

Suedel, B.C. and J.H. Rodgers, "Variability of Bottom Sediment Characteristics of the Continental United States", *Wat. Res. Bull., 27*(1):101–109 (1991).

Suedel, B.C. and J.H. Rodgers, "Development of Formulated Reference Sediment for Freshwater and Estuarine Sediment Testing" , *Environ. Toxicol. Chem., 13*:1163–1175 (1994).

Swartz, R.S., W.A. DeBen, J.K.P. Jones, J.O. Lamberson, and F.A. Cole, "Phoxocephalid Amphipod Bioassay for Marine Sediment Toxicity", pp. 284–307, *in: Aquatic Toxicology and Hazard Assessment: Volume 7*, R.D. Cardwell, R. Purdy, and R.C. Bahner (eds.), American Society for Testing and Materials, Philadelphia, PA (1985).

Swartz, R.S., D.W. Schults, T.H. DeWitt, G.R. Ditsworth, and J.O. Lambertson, "Toxicity of Fluoranthene in Sediment to Marine Amphipods: A Test of the Equilibrium Partitioning Approach to Sediment Quality Criteria", *Environ. Toxicol. Chem., 9*:1071–1080 (1990).

Tessier, A., R. Carignan, B. Dubreuil, and F. Rapin: "Partitioning of Zinc Between the Water Column and the Oxic Sediments in Lakes", *Geochim. Cosmochim. Acta, 53*:1511–1522 (1989).

Tetra Tech, "Recommended Protocols for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples", prepared for Office of Puget Sound

Region 10, United States Environmental Protection Agency, Seattle, Washington (as cited in USEPA, 1987) (1986).

Thomas, R.L., J-M. Jaquet, A.L.W. Kemp, and C.F.M. Lewis, "Surficial Sediments of Lake Erie", *J. Fish. Res. Board. Can., 33*:385–403 (1976).

USEPA (United States Environmental Protection Agency), "Chemistry Laboratory Manual for Bottom Sediments and Elutriate Testing", EPA-905-4-79-014, U.S. Environmental Protection Agency/Corps of Engineers, Region V, Chicago, IL (1979).

USEPA (United States Environmental Protection Agency), "Guidance for Sampling of and Analyzing for Organic Contaminants in Sediments", Office of Water, Regulations, and Standards, Criteria and Standards Division, USEPA, Washington, DC, 49 p. (1987).

USEPA (United States Environmental Protection Agency), "Evaluation of Dredged Material Proposed for Ocean Disposal: Testing Manual", EPA-503/8-91/001, Office of Water (WH-556F), Vicksburg, MS (1991).

USEPA (United States Environmental Protection Agency), "Sediment Classification Methods Compendium, Chapter 2: Quality Assurance/Quality Control, Sampling, and Analytical Considerations", EPA 823-R-92-006, Office of Water, USEPA, Vicksburg, MS (1992).

USEPA (United States Environmental Protection Agency), "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates", Draft 2/4/94, Office of Science and Technology, United States Protection Agency, Washington, D.C. 245+ p. (1994a; 1994b).

van de Guchte, C. and J.L. Maas-Diepeveen, "Screening Sediments for Toxicity: A Water-concentration Related Problem", *in: Proceedings of the 14th Annual Aquatic Toxicity Workshop,* November 1–4, 1987, Toronto, Ontario, Canada, *Can. Tech. Rep. Fish. Aquat. Sci., 1607:*81–91 (1988).

van der Loeff, R.M.M., "Time Variation in Interstitial Nutrient Concentrations at an Exposed Subtidal Station in the Dutch Wadden Sea", *Neth, J. Sea Res., 14:*123–143 (1980).

van Raaphorst, W. and A.G. Brinkman, "The Calculation of Transport Coefficients of Phosphate and Calcium Fluxes Across the Sediment-water Interface, from Experiments with Undisturbed Sediment Cores", *Water Sci. Technol., 17:*941–951 (1985).

van Woerden, J.A., J.A. Hellema, and M. Tielman, "Hydrographic Echosounding Combined with Subbottom Profiling and Nuclear Density Measurements", *Hydrogr. J., 50:*5–10 (1988).

Viel, M., A. Barbanti, L. Langone, G. Buffoni, D. Paltrinieri, and G. Rosso, "Nutrient Profiles in the Pore Water of Deltaic Lagoon: Methodological Considerations and Evaluation of Benthic Fluxes", *Estuar. Coastal Shelf Sci., 33:*361–382 (1991).

Watson, P.G., P. Frickers and C. Goodchild, "Spatial and Seasonal Variations in the

Chemistry of Sediment Interstitial Waters in the Tamar Estuary", *Estuar. Coastal Shelf Sci., 21:*105–119 (1985).

Whiticar, M.J., "Determination of Interstitial Gases and Fluids in Sediment Collected with an *in-situ* Sample", *Anal. Chem., 54:*1796–1798 (1982).

Windsor, J.G. and R.A. Hites, "Polycyclic Aromatic Hydrocarbons in Gulf of Main Sediment and Nova Scotia Soils", *Geochim. Cosmochim. Acta., 43:*27–33 (1979).

Word, J.Q., J.A. Ward, L.M. Franklin, V.I. Cullinan, and S.L. Kiesser, "Evaluation of the Equilibrium Partitioning Theory for Estimating the Toxicity of the Non-polar Organic Compound DDT to the Sediment Dwelling Amphipod *Rhepoxynius abronius*", Final report submitted by Battelle Washington Environmental Protection Agency, Criteria and Standards Divison, 60 p., (1987).

Wright, L.D., D.B. Prior, C.H. Hobbs, R.J. Byrne, J.D. Boon, L.C. Schaffner, and M.O. Green, "Spatial Variability of Bottom Types in the Lower Chesapeake Bay and Adjoining Estuaries and Inner Shelf", *Estuar. Coastal Shelf Sc., 24:*765–784 (1987).

Yee, S., M. VanRikxoort, and D. McLeay, "The Effect of Holding time on *Eohaustorius washingtonianus* During Ten-day Sediment Bioassays and Reference Toxicant Tests", prepared for Environment Canada and the Intergovernmental Aquatic Toxicity Group, Ottawa, Ontario, 44 p. (appendices) (1992).

Appendix A

Members of the Inter-Governmental Aquatic Toxicity Group*

Federal, Environment Canada

P.G. Wells (Chairperson)
Dalhousie Unviersity
Halifax, N.S.

K.G. Doe
Atlantic Region
Dartmouth, N.S.

W.R. Parker
Atlantic Region
Dartmouth, N.S.

S.J. Wade
Atlantic Region
Dartmouth, N.S.

J.D.A. Vaughan
Atlantic Region
Dartmouth, N.S.

N. Bermingham
Centre St. Laurent
Longueuil, Quebec

C. Blaise
Centre St. Laruent
Longueuil, Quebec

G. Elliott
Environmental Protection
Edmonton, Alberta

R.G. Watts
Pacific Region
North Vancouver, B.C.

S.E. Yee
Pacific Region
North Vancouver, B.C.

J. Miller
Technology Development
Directorate
Ottawa, Ont.

D. Moul
Pacific Region
North Vancouver, B.C.

K.E. Day
National Water Research Inst.
Burlington, Ont.

B. Dutka
National Water Research Inst.
Burlington, Ont.

C. Boutin
Canadian Wildlife Service
Hull, Quebec

D. MacGregor
Technology Development
Directorate
Ottawa, Ont.

R.P. Scroggins
Technology Development
Directorate
Ottawa, Ont.

G.A. Sergy
Technology Development
Directorate
Edmonton, Alberta

Federal, Fisheries & Oceans

R. Stevens
Oceanography & Contaminants
Branch
Ottawa, Ont.

Federal, Atomic Energy Control Board

P. Thompson
Environmental Protection
Ottawa, Ont.

Federal, Natural Resources Canada

D. Rodigque
CANMET, Mineral Sciences
Ottawa, Ont.

Provincial

C. Bastien
Que. Ministry of the Environment
Ste. Foy, Quebec

S.G. Abernethy
Ont. Ministry of Environment
Rexdale, Ont.

C.M. Neville
Ont. Ministry of Environment
Rexdale, Ont.

D.G. Poirier
Ont. Ministry of Environment
Rexdale, Ont.

I.R.Smith
Ont. Ministry of Environment
Rexdale, Ont.

G.F. Westlake
Ont. Ministry of Environment
Rexdale, Ont.

B. Bayer
Manitoba Environment
Workplace, Safety & Health
Winnipeg, Manitoba

J. Somers
Alberta Environment
Edmonton, Alberta

K. Smiley
Alberta Environment
Vegreville, Alberta

S.H. Horvath
B.C. Ministry of Environment
Vancouver, B.C.

G. van Aggelen
B.C. Ministry of Environment
North Vancouver, B.C.

* as of December, 1994

Environmental Protection Service, Regional and Headquarters Offices

Headquarters
351 St. Joseph Boulevard
Place Vincent Massey
Hull, Quebec
K1A 0H3

Ontario Region
4905 Dufferin St.,
2nd Floor
Downsview, Ontario
M3H 5T4

Atlantic Region
15th Floor, Queen Square
45 Alderney Drive
Dartmouth, Nova Scotia
B2Y 2N6

Western and Northern Region
Room 210, Twin Atria #2
4999 - 98 Avenue
Edmonton, Alberta
T6B 2X3

Quebec Region
1141 Route de L'Eglise
P.O. Box 10100
Sainte Foy, Quebec
G1V 4H5

Pacific and Yukon Region
401 Burrard Street
Vancouver, British Columbia
V6C 3S5

Statistical Formulae for Determining Number of Samples

A Summary of Several Formulae used to Statistically Determine the Number of Samples to be Collected for Environmental Monitoring and Assessment

Objective	Formulae	Reference
To determine the sample size required to detect an effect in an impacted area versus a control area over time:		
a) resampling same sites before and after impact and testing if the mean change in the control area is the same as that in the impacted area	$n = 2\,(t_\alpha + t_\beta)^2\,(S/\Delta)^2$	Green, 1989
b) sampling different sites before and after impact and testing if there is no interaction between area effect and time effect	$n = 4\,(t_\alpha + t_\beta)^2\,(S/\Delta)^2$ where: n = number of samples for each of the control and impact areas S = standard deviation Δ = magnitude of change required to be a real effect with specified power (1-ß) t_α = t statistic given a Type I[1] error probability $t_ß$ = t statistic given a Type II[2] error probability	Green, 1989
To determine if the mean value for and impacted area: a) differs significantly from a standard value (e.g., sediment quality criterion)	$n \geq \dfrac{(Z_\alpha + Z_\beta)^2\,(S/\Delta)^2}{d^2} + 0.5Z_\alpha^{\,2}$ where: n = sample size Z_α = Z statistic for Type 1 error probability (e.g., $a = 0.05$) $Z_ß$ = Z statistic for Type II error probability (e.g., ß - 0.90) d = magnitude of the difference to be detected (i.e., effect level)	Alldredge, 1987

b) differs significantly from the mean of a control site

$$n \geq \frac{2\,(Z_\alpha + Z_\beta)^2}{d^2} + 0.25Z_\alpha{}^2$$

Alldredge, 1987

where:
- n = sample size
- Z_α = Z statistic for Type I error probability (e.g., $\alpha = 0.05$)
- Z_β = Z statistic for Type II error probability (e.g., β - 0.90)
- d = magnitude of the difference to be detected (i.e., effect level)

To determine the number of samples required to estimate a mean value (representative of the area) with a given statistical certainty

$$y\bar{x} = t_c \left[\frac{S_x}{(n-1)^{1/2}} \right]$$

Håkanson, 1984

where:
- y = accepted error in the percent of the mean value(e.g., y = 10%)
- $\bar{x}$ = mean value of X_i ($i = {}_{1...n)}$
- S_x = standard deviation
- t_c = confidence coefficient (e.g., 90% or $t_{0.95}$)
- n = number of samples

To determine the number of samples required to estimate a mean

$$n = \frac{(Z_{\alpha/2})^2\,\sigma^2}{d^2}$$

Milton *et al.,* 1986

where:
- n = number of samples
- Z = Z statistic (standard normal curve)
- σ^2 = variance
- $\alpha/2$ = probability of a 95% confidence level
- d = the distance between the centre of the lower confidence and the upper confidence bound

---

To determine the number of samples required for a particular power for: a normal distribution (i.e., $\bar{x} > s^2$)

$$n = \frac{10^4 (t^2\ s^2)}{(R^2 \bar{x}^{\ 2})}$$

Kratochvil and Taylor, 1981

where:
- n = number of samples
- t = t statistic for a desired confidence level
- $\bar{x}$ = mean value from preliminary sampling or historical data
- s = standard deviation of mean
- R^2 = percentage coefficient of variation
- K = index of clumping

1

2 Type I (*a*) error is the probability of rejecting the hypothesis being tested when it is true
Type II (ß) error is the probability of rejecting the hypothesis being tested when it is false

The formulae presented in this appendix apply to four different situations:

1. Estimation of a mean (Kratochvil and Taylor, 1981; Håkanson, 1984; Milton *et al.,* 1986).

2. Comparison of a mean with a standard (Alldredge (a), 1987).

3. Comparison of two means (Alldredge (b), 1987; Green (a), 1989).

4. Interaction between area and time effects (Green (b), 1989).

Estimation of the Mean. The formula of Milton *et al.* (1986) is frequently used. Unfortunately it is known that it seriously underestimates the necessary number of samples. The extent of this underestimation is discussed by Kupper and Hafner (1989) who provide a means of correcting for this underestimation. The formulae of Håkanson (1984) and Kratochvil and Taylor (1981) are variations of the same basic estimation and are subject to the same underestimation of the number of samples. Because they use the t-distribution, however, the underestimation problem may not be as great but they require an iterative solution. In the formula of Håkanson the quantity "*y*" must be expressed as a proportion (or the righ-hand-side multiplied by 100), and the use of "*n*-1" in the forumla leads to a slighly larger estimate than that of Kratochvil and Taylor (1981).

Comparison of a Mean with a Standard. One of the most common comparisons is that of a mean value with a standard value which essentially tests the hypothesis H_O: $\mu_o = \mu_A$ against the alternative H_A: $\mu_o \mu_A$. The significance level is generally α. The detectable difference that is desirable is generally defined as Δ, with a probability β such that P (Reject H_o when $\mu_A - \mu_o > \Delta) \geq 1 - \beta$ ($1-\beta$ represents the power of the test). The estimate of the sample size, *n*, required to satisfy these conditions is:

$$n = \frac{Z_\alpha + Z_\beta}{\Delta^2} + 2$$

(with *n* rounded up to the nearest integer).

If the hypothesis is two-tailed (i.e., H_A: $\mu_o \neq \mu_A$), Z_α is replaced by $Z_{\alpha/2}$ in the preceding formula. The formula presented by Alldredge (1987) has the term $0.5\ Z_\alpha^2$ which provides an adjustment for the uncertainty in the estimate of the variance. This addition is probably a good estimator, perhaps a little liberal.

Comparison of Two Means. The formulae (b) and (a) provided, respectively, by both Alldredge (1987) and Green (1989) agree closely; however, because iteration is required by the latter, the formula by Alldredge may be slightly more desirable.

Interaction Between Area and Time Effects. This formula should be used when the objective of the project is to examine differences in contaminant levels at a site relative to a control site over time. The method requires iteration.

In-situ Collection of Pore Water

In shallow waters (e.g., <10 m deep), pore water has been collected by divers directly from fine-grained sediments using long-needled syringes (Bauer *et al.,* 1988) or a tube, either by hydrostatic pressure or vacuum, after passage through a fliter (Brinkman *et al.*, 1982; Buddensiek *et al.*, 1990). Only relatively small volumes (5 to 10 mL) can be collected at one time from a given depth. In shallow estuarine waters (0.5 m deep), a water sampler was developed to repeatedly collect anoxic pore water from estuarine sediments (Montgomery *et al.,* 1979; 1981). Sample volumes between 50 and 75 mL were collected by vacuum in an inert atmosphere (provided by an inert gas which periodically displaced the collected sample). A similar system was designed for use in rivers and lakes (Hertkorn-Obst *et al.,* 1982). In deeper marine waters (20 to 1800 m), a stainless-steel tube sampler which functioned like a giant syringe was developed to collect pore water simultaneously at several depths (Sayles and Manheim, 1975). This design was modified to collect pore water from shallow, subtidal marine environments (van der Loeff, 1980). Howes *et al.* (1985) collected pore water in shallow marshes from different sediment depths by modifying the design of Montgomery *et al.* (1979), but only 5 to 10 mL could be sampled at one time from the depth profile.

All of these methods for collecting pore water rely on water suction or displacement with inert gases, and filtering. Most other methods rely on diffusion principles. The dialysis samplers or "peepers" have membranes that differ with respect to their composition and pore size, which confer some solute selectivity (e.g., 0.2-µm polysulfone or polycarbonate membranes, 0.45-µm PVC membranes, 3.0-µm porous Teflon) (Adams, 1991). Cellulose-based dialysis membranes are susceptible to microbial attack and should not be used (Martens and Klump, 1980). Degassing of PVC membranes is desirable before use in peepers (Carignan *et al*., 1994). The dialysis sampler is usually deoxygenated in distilled, or demineralized water that has been purged with nitrogen (Hesselin, 1976; Kelly *et al.*, 1984; Carignan and Tessier, 1985; Tessier *et al*., 1989; Viel *et al*., 1991), or a nitrogen-purged solution of sodium chloride (Simon *et al*., 1985). Degassing with nitrogen for 24 to 48 h in an equilibration chamber is generally used to deoxygeneate the dialysis sampler. The sampler must be placed directly from the equilibration chamber into the sediment (usually by a diver), where it is allowed to equilibrate for a period of time (3 to 20 days) before the pore water is collected from the recovered dialysis sampler (i.e., either by syringe or attached collecting tubes). The equilibration time must be experimentally determined, *a priori* by daily sampling and analyses of the pore water (Carignan, 1984).

Experimental artifacts can result from contamination of the pore water by the sampling equipment, incomplete equilibration between the sediment pore water and the water in the dialyzer, membrane disruption, interference with diffusion of ions from the development of an electrical potential across the membrane, or oxidation of the pore water during collection from the dialyzer and transfer to the sample

container (Carignan *et al*., 1985). These artifacts can be minimized by:

a) using only clean chemically inert material in the construction of the dialyzer (i.e., no rubber, neoprene, PVC, polystyrene, metallic or galvanized metallic materials);

b) constructing the dialyzers carefully with intact, sealed porous Teflon®, polycarbonate membranes, or biological inert polysulfone;

c) using only a purified oxygen-purged equilibrator in the chamber;

d) ensuring that the equilibration time is adequate;

e) using non-metallic syringes for extraction of pore water;

f) using clean, pretreated storage or sample containers of the appropriate material (Section 2.8); and;

g) transferring the pore water from the dialyzer to the sample container in an oxygen-free atmosphere (i.e., glove box with argon or nitrogen gas).

Viel *et al*., (1991) compared the iron and nutrient ammonia, phosphate, silica (NH_3, PO_4, SiO_2) concentrations in pore water, collected by centrifugation at 5000 rpm for 20 min., with the concentrations in pore water collected *in situ* by dialysis. They found that the concentrations of PO_4 and Si were comparable in the two types of water, but that concentrations of iron and ammonia differed, probably because of manipulation of the core samples during transfer to the centrifugation tubes, and/or spatial heterogeneity.

Knezovich and Harrison (1987) compared the concentration of chlorobenzene in pore water collected, *in situ*, from the surficial layers of sediment (e.g., 0 to 8 mm), by a modified syringe, with that found in pore water collected by compression methods and contrifugation. The loss of the volatile compound was greatly reduced by the *in situ* extraction method.

Dialyzers have the advantage of collecting pore water samples free of temperature, pressure, and oxidation artifacts, but require a relatively long equilibration period. The suction samplers can recover samples that are also relatively free from the same artifacts, but are more time consuming to operate, more expensive to construct, and have limited depth resolution. Since the two methods of sampling pore water *in situ* (e.g., water suction versus dialyzers) have, only recently, been directly compared, one method cannot be recommended over another.

The concentrations of contaminants in pore water may be more highly correlated with toxicity to aquatic organisms (Cairns *et al.*, 1984; Nebeker *et al*., 1984; Schuytema *et al*., 1984; Knezovich and Harrison, 1988; Giesy and Hoke, 1990) than those in bulk sediments (Patrick *et al*., 1977; Adams *et al.*, 1985; Shaner and Knight, 1985; van de Guchte and Mass-Diepeveen, 1988; Di Toro, 1989; Ankley *et al*., 1991c; Carr and Chapman, 1992). The relative importance of pore water and whole sediments as sources of toxic contaminants to aquatic organisms appears to depend on the species of test organism and the type of contaminant (Knezovich *et al*., 1987; Giesy and Hoke, 1990; Harkey *et al*., 1994).

Transport and Storage of Field-collected Samples

Sample Containers

Any material that is in contact with a field sample has the potential to contaminate the sample or adsorb components from the sample. The use of appropriate materials can minimize or mitigate these interferences. For example, polyvinyl chloride (PVC) material is high in zinc, iron, antimony, and copper, and should not be used to store or contain sediments where metal contamination is of concern. Nylon is high in leachable cobalt. Soda glass, flint glass, and metal materials, including stainless steel, can contaminant samples with metallic elements (e.g., chromium, iron, nickel, molybdenum). Plexiglass readily adsorbs organic elements or compounds; whereas, rubber and neoprene materials might contaminate samples with organic compounds. Substances that should not have direct contact with field-collected samples include PVC, rubber, nylon, talcum powder, polystyrene, galvanized metal, brass fittings, metal materials, soda glass, paper tissues, and painted surfaces.

Polytetrafluoroethylene (PTFE) or Teflon®, borosilicate glass and stainless steel are considered relatively chemically inert in terms of adsorption and desorption processes (Moody and Lindstrom, 1977; Bryden and Smith, 1989). However, Teflon® has been shown to both emit organic impurities (Giam and Wong, 1972; Lonneman *et al*., 1981) and to absorb compounds (Blau and King, 1977). Borosilicate glass can contribute and adsorb inorganic salts such as sodium, fluoride, and boron, and it breaks relatively easily. High-density polyethylene materials minimally (i.e., relative to other materials) adsorb and desorb organic compounds, are lightweight, resilient to physical stresses and relatively inexpensive. Teflon® does not contaminate water or sediment samples, but it must be handled with care. Stainless steel containers should not be used for sediments that are to be analyzed for inorganic contaminants. Therefore, the preferred sample containers recommended for whole sediments should be made of, or lined with Teflon®. Both borosilicate and high-density polyethylene containers are acceptable, however, if they have been properly pretreated and samples are stored for fourteen days or less. Stainless steel and glass containers are suitable for sediments that are to be analyzed for organic contaminants.

Storage Containers and Conditions

If field-collected samples are also processed in the field (e.g., subsampled, sectioned, filtered, homogenized, sieved, etc.), the appropriate type of container for storing samples must be selected. Sediments that are to be used for toxicity tests must not be stored in a dried, frozen, or freeze-dried condition (Malueg *et al*., 1986; Swartz *et al.*, 1985; Anderson *et al*., 1987; ASTM, 1992a). They should be refrigerated in the dark, in tightly sealed storage containers, without preservation reagents, at 4 ± 2° C to minimize the physical, chemical, and biological changes that are inevitable over time. Sediments that are destined for only chemical analyses can be stored at 4 ± 2° C, with or without the addition of reagents (e.g., preservatives), or -20° C. Sediments have been fast-frozen and stored frozen until

analyzed (Malueg *et al*., 1986; Cairns *et al.*, 1984; Jenne and Zachara, 1987; Muir *et al.*, 1982, 1985; Rapin *et al*., 1986; Rochon and Chevalier, 1987); however, freezing or fast-freezing of whole sediments is discouraged because it alters the structure of the sediment, distorts the sediment profile, and fails to preserve the chemical integrity of the sediment (Swartz *et al*., 1985; Chapman, 1988; Lamberson and Swartz, 1988). Samples of sediment which is contaminated with volatile organic contaminants are commonly stored in at -20° C to minimize losses during storage. Freezing of sediment samples has been a common practice in monitoring and reconnaissance studies; however, the effects of freezing on the physicochemical properties of the sediment should be discerned if the samples are to be stored in this manner. Drying of the sediment (e.g., freeze-drying or oven-drying) during storage should be avoided (Rapin *et al*., 1986).

Storage Durations

The "shelf life" of a sediment sample in cold storage (e.g., 4° C) depends on the nature of the sediment, the degree of contamination, and the type of contaminant(s). Studies have shown significant changes in sediment toxicity after storage periods of less than seven days and as long as twelve months (DeWitt *et al*., 1989; Brouwer *et al.,*1990; Stemmer *et al*., 1990b; ASTM, 1992a; Othoudt *et al*.,1991; Landrum *et al*., 1992). Carr and Chapman (1995) compared at approximately one-week intervals over a 29-d period the toxicity of pore water stored at 4° C. The toxicity of pore water to the sea urchin (*Arbacia punctulata*) exhibited substantial short-term (e.g., days, weeks) changes; however, the direction and magnitude of change in toxicity was unpredictable. The weight of evidence suggests that storage times should be minimized (ASTM, 1992a; Othoudt *et al*., 1991). Toxicity testing of sediment samples should be conducted as soon as possible after collection, preferably within six weeks of collection (Chapman, 1988). A maximum permissible storage time of six weeks has been used in sediment toxicity test methods of Environment Canada (1992 c; d; and e) to accommodate logistical considerations including the time required if initial chemical analyses are to be performed. However, it is recommended that toxicity tests should be conducted within two weeks of sample collection and ideally within one week (i.e., the sooner, the better). Sediments may be stored for longer periods (i.e., up to six weeks) in monitoring and assessment studies, if it can be demonstrated that significant changes in toxicity or chemistry do not occur, or have not occurred, over the storage period (Othoudt *et al.*, 1991).

Samples of pore water are particularly susceptible to changes in chemistry during storage. Significant changes were observed within 24 h for samples stored at 4° C (Hurlbert and Brindle, 1975; Watson *et al.,* 1985; Kemble *et al*., 1993). Storage times for samples of pore water can be lengthened by preparing the samples for analysis immediately upon collection. Preparation methods such as filter sterilizing and/or use of preservative reagents will extend the "shelf life" of pore water. However, it must be recognized that filtering could ameliorate toxicity. Freezing of prepared pore water did not affect toxicity in some studies (Hardy *et al*., 1987; Carr *et al*., 1989), whereas studies with wastewaters have shown that freezing and thawing can cause marked effects on toxicity. Therefore, toxicity testing with pore water should occur immediately after collection.

Freezing and thawing of porewater samples extracted by pressurized squeezing had no significant effect on toxicity of the pore water to the sea urchin *Arbacia punctulata* (Carr and Chapman, 1995). However, pore water collected from sediment by centrifugation or suction (e.g., vacuum) methods and frozen without additional removal of suspended particulate by centrifugation exhibited increased toxicity to sea urchins compared to the unfrozen sample (Carr and Chapman, 1995). Therefore, they recommend that, regardless of the method used to collect the pore water, the porewater sample should be centrifuged before testing or freezing to reduce the effects of the suspended particulate on the ability of sperm to either locate or fertilize the eggs used in this particular assay.

Recovery of Pore Water from Samples of Sediment

Manipulations for Extraction of Pore Water

Squeezing of sediments to extract pore water can be done by high- or low-pressure mechanical squeezing, or low-pressure gas squeezing. The squeezing devices generally consist of a series of clamps, filters, and membranes in conjunction with collection devices for retaining pore water. The composition of the materials used in the construction of a squeezing device varies and may include Teflon®, stainless steel, brass, and nylon. A comprehensive description of a large number of squeezers including the composition of their parts, the pressure used, the sample volume processed, and the volume of pore water obtained is provided in Adams (1991).

Factors that could influence the composition of pore water obtained using the squeezing technique include contamination from component materials of the squeezing device, operational conditions, and chemical fractionation of the sample. The materials from which the components of the squeezing device are made can affect the chemistry of the pore water. Devices made of stainless steel are suitable for collecting pore water for the determination of anions, alkali metals, and alkaline earths, but not for iron and other transition elements. These elements are subject to contamination from stainless steel (Adams, 1991).

Using a mechanical squeezer, Manheim (1976) observed that squeezing pressure did not significantly affect the composition of the extracted pore water. A problem that may be associated with squeezing is the formation of an impermeable cake at the bottom of the squeezing device, preventing the passage of pore water. Stepwise increases in pressure over a 30-minute period can minimize this problem (Reeburgh, 1967). Nath *et al.* (1988) modified the Manheim squeezer to reduce the collection time from between 60 and 90 min to collect 20 mL of pore water to less than 20 min. The new design included a Teflon® liner, which prevented exposure of the sediment to the atmosphere, thus limiting oxidation effects. Jahnke (1988) also developed a simple, reliable, and inexpensive whole-core squeezer to collect the pore water in gas-tight syringes connected to the core tube to minimize oxidation effects.

Klinkhammer (1980) observed high concentrations of trace metals, particularly iron, in pore water, and suggested that iron-rich, colloidal-sized particles were not retained by the filters in squeezing devices. Hines *et al*., (1989) found that sulphide concentrations were lower in squeezed samples when compared to samples of pore water collected *in-situ*. This occurred even though a strict oxygen-deficient atmosphere was maintained during squeezing. Malcolm *et al*. (1990) found that concentrations of reduced Pu (a transuranium nuclide) increased on exposure to air and they recommend that sampling and extraction of interstitial waters from anoxic sediment be conducted in a glove box to minimize compositional changes.

Chemical fractionation of a pore water sample can also occur during squeezing. Significantly higher ammonium concentrations were reported in pore water

in the first three aliquots (Emerson *et al.*, 1980). Froelich *et al.* (1979) compared two types of squeezers and found that, for both squeezers, when calcium carbonate ($CaCO_3$-rich sediments were squeezed through filters that had been acid-washed, pore water carbon dioxide (CO_2) levels were higher in the first aliquot obtained than in subsequent aliquots.

It is accepted that some pore water constituents are affected by the collection method. Dissolved organic carbon and dimethylsulphide are affected, while the effects on ammonia and sulphide (given that oxidation is prevented) are equivocal (Burton, 1992). Bender *et al*. (1987) concluded that the composition of pore water collected by a whole-core squeezing device was very susceptible to rapid alteration by solid-solution reactions, and that the method was unsuitable for determining pore-water profiles of trace metals and other particle-reactive chemicals.

Centrifugation has been compared to an *in-situ* dialysis method and was found to give similar results. The general procedure for centrifuging a sediment core sample is as follows. A horizon of a core of sediment is cut, or displaced, in an oxygen-free environment (e.g., glove box, glove bag, etc.). The sediment is loaded into acid-washed polycarbonate centrifuge tubes. The tubes are tightly capped and centrifuged at a high speed while under refrigeration. The supernatant is then siphoned off (in the oxygen-free environment) and only filtered if necessary (i.e., as stipulated in the test method). The supernatant or filtrate is then stored in a polystyrene vial that has been previously acidified with hydrogen chloride (HCl) or nitric acid (HNO_3).

The variable aspects of the centrifugation process are the centrifuge speed and the composition and pore size of the filter used. Adams *et al*. (1980) examined the effect of centrifuge speeds (7 000 to 19 000 rpm) on pore water composition. Little change was found in concentrations of calcium (Ca), iron (Fe), manganese (Mn), and zinc (Zn), but PO_4 concentrations doubled. Another concern is that the centrifuge speed may not be high enough to remove dispersible clays. Trace metals concentrate on solids and this might have a significant effect on sorption studies (Jenne and Zachara, 1987).

Processes such as adsorption of potential toxicants in the pore water to the filters and desorption of potential toxicants from the filter require that filters be pretreated and that filter banks be used to assess the extent to which these processes might occur (Levi and Novicki, 1972; Novicki *et al*., 1979). Filtration with glass fibre or plastic filters has been shown to remove non-polar organics. Certain dissolved hydrophobic contaminants might be adsorbed to glass fibre filters (Word *et al*., 1987). Other studies recommend the use of polycarbonate filters (Knezovich and Harrison, 1987). Carr and Chapman (1995) compared the toxicity of pore water collected from subsamples of contaminated sediment with a Teflon® extractor and centrifugation. Five types of filters (e.g., fluorocarbon, nylon, polyester, polycarbonate, glass fibre) were used and testing demonstrated that pretreatment which involved soaking the filters in deionized water for 24 h did not adequately remove likable toxic compounds from the nylon filters in 24 h and that the glass fibre filters could not withstand the soaking. The problem with the nylon filters was overcome by simply extending the soaking period to 48 h. Different types of

filters used in squeezing devices adsorbed soluble contaminants to varying degrees. Recent studies indicate that filtering pore water will substantially reduce toxicity regardless of the method of pore water extraction (e.g., centrifugation, pressure extraction, syringe extraction, or dialysis) (Ankley *et al*., 1991b). Saager *et al*. (1990) used a centrifuge tube specially designed for collection of pore water.

Carignan *et al*. (1985) compared pore water recovered from field-collected sediments by centrifugation and filtration with that collected from sediment *in-situ* by dialysis. Centrifugation was conducted at two speeds (5 000 and 11 000 rpm), followed by filtration with pore sizes of filters being 0.45, 0.2, and 0.03 µm. The concentration of calcium (Ca), magnesium (Mg), iron (Fe), manganese (Mn), chromium (Cr), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), cadmium (Cd), and organic carbon were measured and compared. Centrifuging at 5 000 rpm and filtering through a pre-washed 0.45 µm filter resulted in pore water levels of Co, Ni, Cr, Fe, and Mn comparable to those in the pore water obtained by the dialysis method. However, Cu, Zn, and organic carbon levels were significantly higher in pore water obtained by the centrifuge method. Centrifuging at 11 000 rpm and filtering through 0.2 or 0.03 µm filters produced pore water levels of Cd, Co, Cr, Ni, and organic carbon equivalent to those observed in the dialysis pore water. It would appear that centrifuging compares well with the *in-situ* dialysis method, at least for the elements examined in this study.

The effects of five pore water extraction methods on sediment toxicity were evaluated using sediments that were heavily contaminated with metals, and/or oil and grease, PAHs, and NH_3 (Ankley *et al.,*1991b). The extraction methods included centrifugation at low and high speeds (e.g., 250 × G and 10 000 × G, respectively) at 4° C, pressure extraction, syringe extraction, and dialysis. The pore water samples were collected with and without filtration. The comparative results indicate that centrifugation speed had little effect on sediment toxicity. They recommended, however, that the higher speed was more appropriate because it isolated only the bioavailable metal fraction whereas the lower speed resulted in the presence of unavailable metals in pore water. Pore water collected by centrifugation was generally as toxic or more toxic than pore water collected by the other methods. Filtering pore water decreased toxicity by between 40 and 85% (Ankley *et al*., 1991b).

The effects of three porewater extraction methods (e.g., centrifugation, vacuum suction, and pressurized squeezing) on toxicity of contaminated sediments were evaluated (Carr and Chapman, 1995). They concluded that: the toxicity of porewater samples is greatly influenced by the type of collection method; centrifugation should be used when the primary contaminants of concern are highly hydrophobic organic compounds to maximize the sensitivity of the egg fertilization test; and, regardless of the method used for the initial extraction, the sample should be (re-) centrifuged prior to testing or freezing to remove suspended particulate which compromise test results.

Chemical fractionation of the pore water sample is a concern for the centrifuge method as well. Edmunds and Bath (1976) studied the potential fractionation during centrifugation of five cations (Na^+, K^+, Ca^{2+}, Sr^{2+}, Mg^{2+}) from the pore water of Cretaceous rock. Cation concentrations were measured at four times during pore

water removal. Sodium and potassium showed a progressive depletion as a greater portion of the pore water was extracted, with concentrations levelling off during the extraction of the final 10 to 20% of the sample. Concentration of Ca^{2+} decreased until extraction was complete. No apparent fractionation of Sr^{2+} or Mg^{2+} was observed.

Other methods for extraction of pore water from samples of sediments include desiccation procedures (Adams, 1991), vacuum filtration (Glass and Poldoski, 1975), and displacement techniques (Adams, 1991). The displacement technique involves the use of an immiscible liquid to displace the pore water. Extraction is made with filter press after the liquid is poured over the sediment and then pressurized (Adams, 1991). Vacuum filtration with a Buchner funnel is used to extract pore water (Glass and Poldoski, 1975). This is a tedious process, especially with fine-grained sediments where evaporation of the pore water becomes a major problem (Adams, 1991). The potential errors involved with this technique are the solubilization of solids and the changes that occur in the sediment-water equilibria.

van Raaphorst and Brinkman (1985) used polyethylene tubing containing cotton threads to extract pore water from a sample of cored sediment. Under vacuum, 5 mL of pore water was collected over two or three days. Phosphate in the pore water did not interact with the cotton. This procedure would not be appropriate for samples where analyses for organic contaminants are desired.

Appendix G

Number of Sediment Samples[1] to be Collected for Dredging Projects (or strata) of Different Sizes (Atkinson, 1994)

Volume to be Dredged (m^3)			Number of Samples
Greater than		Less than or Equal to	
0	to	10 000	6
10 000		17 000	7
17 000		23 000	8
23 000		30 000	9
30 000		37 000	10
37 000		43 000	11
43 000		50 000	12
50 000		58 000	13
58 000		67 000	14
67 000		75 000	15
75 000		83 000	16
83 000		92 000	17
92 000		100 000	18
100 000		141 000	19
141 000		182 000	20
182 000		223 000	21
223 000		264 000	22
264 000		305 000	23
305 000		346 000	24
346 000		386 000	25
386 000		427 000	26
427 000		468 000	27
468 000		509 000	28
509 000		591 000	29
519 000		632 000	30
632 000		673 000	31
673 000		714 000	32
714 000		755 000	33
755 000		795 000	34
795 000		836 000	35
836 000		877 000	36
877 000		918 000	37
918 000		959 000	38
959 000		1 000 000	39

For projects > 1 000 000 m^3, round off the result of:
40 + (volume to be dredged - 1 000 000) / 75 000 samples

[1] The number of sampling stations is equal to the number of samples collected because there is one sample collected per station. In the event that more than one sample is collected at a sampling station, the additional samples should be considered as extra samples.

Appendix H

Information Forms: On-site Data Required for Organism and Sediment Collection, Chain-of-continuity Form and an Ocean-dumping Permit

INFORMATION FORM–ON-SITE FOR ORGANISM COLLECTION

(please complete one form per collection site)

DATE:

SPECIES:

SAMPLING STATION IDENTIFICATION/LOCATION:

LAT/LONG:

DESCRIPTION OF STATION (Please include photographs of area (landmarks) and/or detailed map, input sources of fresh water; point sources of pollution)

rock, sand, silt, clay

intertidal/subtidal (approximate depth):

high energy/low energy

SEAWATER QUALITY: (quality of seawater organisms were exposed to at the collection site)

temperature (˚ C)

sanlinity (‰)

pH:

oxygen:

SITE RATING/EASE OF COLLECTION: [Is this a good station to return to? i.e., are organisms abundant (density), is station easy to get to and collect from, is the presence of other benthic species likely to pose a problem, etc.]

INFORMATION FORM–ON-SITE DATA FOR ORGANISM COLLECTION (continued)

APPROXIMATE NUMBER OF _____(Volume)
"SCOOPS/SHOVELS/GRABS/CORES" NEEDED:

APPROXIMATE NUMBER OF ORGANISMS COLLECTED AT THIS STATION:

APPROXIMATE NUMBER OF HOURS FOR COLLECTION OF ORGANISMS:
(This will give an estimate of the density)

APPEARANCE AND BEHAVIOUR OF ORGANISMS [take a photo and/or describe colour, shape and approximate size of organism (e.g., amphiphod) any distinguishing features, and their behaviour when sieved from sediment (e.g., float, swim, curl into ball) and returned to sediment (e.g., burrow or stay on surface)]

INFORMATION FORM–ON-SITE DATA FOR ORGANISM COLLECTION (continued)

GENERAL PROCEDURES

EQUIPMENT USED:

Boat [if used, also describe sampler (grab, core, etc.)]:

Sampling Equipment (types of containers for holding organisms, spatulas/shovels, sieves, etc.)

Other Relevant Equipment:

SAMPLING PROCEDURE (please describe the sampling methods used and any precautions taken for QA/QC).

LABELLING, STORING, AND SHIPPING PROCEDURES (please include times and conditions between collection and shipping, indicate whether temperature and or dissolved oxygen was regulated during holding and shipping).

NAMES OF ALL FIELD PERSONNEL:

_____ _____

Signature Date completed

INFORMATION FORM–ON-SITE DATA FOR SEDIMENT COLLECTION
(please complete one form per collection site)

DATE/TIME:

SAMPLING STATION IDENTIFICATION/LOCATION:

LAT./LONG.:

DESCRIPTION OF STATION (please include landmarks, weather, prevailing wind/currents, intertidal position if not subtidal, water depth, etc.).

ESTIMATED VOLUME AND WEIGHT OF SAMPLE:

NUMBER OF GRABS/CORES REQUIRED TO COLLECT SAMPLE:

ESTIMATE POSSIBLE DRIFT BETWEEN GRABS/CORES (to give an approximation of the size of the area being represented–please consider depth, length of anchor, winds/currents)

DESCRIBE APPEARANCE OF EACH GRAB/CORE (to estimate initial homogeneity of site–include description of colour and odour of sample, coarseness of grains, note any signs of life in sediment, and indicate if a photographic record was made)

INFORMATION FORM–ON-SITE DATA FOR SEDIMENT COLLECTION (continued)

GENERAL PROCEDURES

EQUIPMENT USED:

Sample (e.g., Ponar, Box-Core, etc.):

Type, size, and number of containers for sample:

Other apparatus contacting sample:

Type of boat and number of anchors if applicable:

Other relevant equipment (e.g., winch, depth sounder):

SAMPLING PROCEDURE
(please describe the sampling methods used including sediment penetration depth and any precautions taken for QA/QC).

SAMPLE LABELLING, STORING AND SHIPPING
(please describe the labelling, storing, and shipping procedures and conditions).

Signature _____ Date completed _____

CHAIN-OF-CONTINUITY RECORD FORM

Canadian Environmental Protection Act

Name of Person Taking Sample: _____

Signature of Person Taking Sample: _____

Name of Inspectors(s): _____
_____

Identification Number(s): _____
_____

Sample Number(s):

_____
_____

Date of Sample(s) Taken: _____ Time Sample(s) Taken: _____

Place Sample(s) Taken:

_____

Required Analysis

_____

Chain-of-Continuity

Relinquished by: _____ Date: _____

Received by: _____ Time: _____

Relinquished by: _____ Date: _____

Received by: _____ Time: _____

Relinquished by: _____ Date: _____

Received by: _____ Time: _____

Relinquished by: _____ Date: _____

Received by: _____ Time: _____

Relinquished by: _____ Date: _____

Received by: _____ Time: _____

Environment Canada
Conservation and Protection

Environnement Canada
Conservation et Protection

PERMIT APPLICATION (OCEAN DUMPING)*

Application Identification
(OFFICE USE)
Name:
Number:

Permits are issued in accordance with Part VI of the *Canadian Environmental Protection Act.* Dumping is defined in section 66 of the Act.
Information provided on this form will be used to evaluate the application for a permit. The following activities are covered by this application (indicate those activities that apply to you): ☐ 1. Loading for the purpose of dumping. ☐ 2. Dumping of any substance ☐ 3. Disposal on ice ☐ 4. Disposal of a ship, aircraft, platform or other structure. ☐ 5. Incineration or other thermal degradation.

PART A - IDENTIFICATION

SUBSTANCE TO BE DUMPED

APPLICANT INFORMATION

1. NAME OF APPLICANT	2. TELEPHONE NO.	3. FAX NO.

4. ADDRESS	5. TYPE OF BUSINESS

6. PREVIOUS PERMITS - List the permit numbers of your previous permits, if any, relevant to this application.	Permit No.	Expiry Date (year/month)

7. NAME OF INDIVIDUAL(S) RESPONSIBLE FOR PROPOSED ACTIVITY	8. TELEPHONE NO.	9. FAX NO.

10. NAME OF TECHNICAL CONTACT(S) FOR PROPOSED ACTIVITY	11. TELEPHONE NO.	12. FAX NO.

PART B - GENERAL INFORMATION

13. DESCRIPTION OF THE ACTIVITY - Give a general description of the proposed activity and its purpose.

* Ce formulaire est disponible en français.

14. SUBSTANCE TO BE DUMPED - Indicate the substance to be dumped. See the applicable item in Part I or II of Appendix I for details of the information that must be included in your application.

15. TOTAL QUANTITY (m^3 or t)

16. PROPOSED TERM OF PERMIT
(maximum 1 year)

from year | month | day

to year | month | day

17. LOAD SITES(S)

NAME AND ADDRESS OF SITE	LATITUDE	LONGITUDE	QUANTITY TO BE LOADED (m^3 or t)

18. DUMP SITES(S)

DUMP SITE NAME (if any)	LATITUDE	LONGITUDE	DEPTH (m)	QUANTITY TO BE DUMPED(m^3 or t)

Provide an estimate of the movement and dispersion in the water columns and on the seafloor of the substance dumped. In the case of disposal at a new dump site or disposal on ice, see the applicable item in Appendix II for details of additional information that must be included in your application.

NUMBER OF PAGES ATTACHED ☐

19. ROUTE FROM LOAD SITE TO DUMP SITE - Attach a map, chart or good reproducible set of drawings that show the location of each load site and each dump site. If the route is not direct, provide reasons and show the intended route on the map, chart or drawing.

NUMBER OF DOCUMENTS ATTACHED ☐

20. EQUIPMENT AND METHODS - Describe the equipment and methods to be used at each load site and dump site. For other thermal degradation, see the applicable item in Part II of Appendix I for details of additional information that must be included in your application.

21. METHODS OF PACKAGING AND CONTAINMENT

DUMPING SPECIFICATIONS

22. MAXIMUM QUANTITY PER DUMP (m^3 or t)

23. RATE (where applicable) (m^3 /h or t/h)

24. FREQUENCY (dumps per day, week or month)

25. SPEED DURING DUMPING

26. TIME REQUIRED FOR DISCHARGE (sinking) (min)

27. TRACK FOLLOWED DURING DUMPING

CARRIER INFORMATION

28. NAME AND ADDRESS OF CARRIER

29. TELEPHONE NO.

30. NAME, TITLE, AND ADDRESS OF THE OWNER OF THE SHIP, AIRCRAFT, PLATFORM OR STRUCTURE USED TO CARRY OUT THE DUMPING

31. TELEPHONE NO.

32. NAME OF INDIVIDUALS RESPONSIBLE FOR LOADING OR DUMPING ON BEHALF OF THE APPLICANT (including the master)

33. TELEPHONE NO.

34. NAME OR NUMBER OF SHIP, AIRCRAFT, PLATFORM OR STRUCTURE USED TO CARRY OUT THE DUMPING

35. APPROVALS - List all permits, licences and reviews, including environmental impact assessments, required by any federal, provincial, territorial, municipal or local agency for the activity described in this application to be carried out.

ISSUING AGENCY	TYPE OF APPROVAL	ID NO.	DATE OF APPLICATION	DATE OF APPROVAL	DATE OF REFUSAL

36. NOTICE OF APPLICATION - Attach proof that notice of this application was published in a newspaper of general circulation in the vicinity of the loading, dumping or disposal described in the application.

NEWSPAPER CLIPPING ATTACHED ☐

NAME OF NEWSPAPER	PLACE OF PUBLICATION (CITY AND PROVINCE)	DATE OF PUBLICATION

PART C - INFORMATION ON ALTERNATIVES TO DUMPING AT SEA

37. WASTE AUDIT - List all steps taken to **REDUCE, REUSE, RECYCLE, AND RECOVER** the substance to be dumped

NUMBER OF PAGES ATTACHED ☐

38. ALTERNATIVES - Provide a comparative assessment of dumping at sea and the practicable alternatives (including treatment, land-based disposal, etc.) Indicating the following:

Environmental impact
Risk to human health
Hazards (including accidents) associated with treatment, packaging, transport and disposal
Economics (including energy costs)
Conflicting use of resources (potential and actual)

NUMBER OF PAGES ATTACHED ☐

PART D - HISTORICAL DATA

39. PREVIOUS DISPOSAL METHODS - Describe the methods, if any, other than dumping at sea, that you have previously used to dispose of this type of substance. Indicates dates and locations.

40. LOAD SITE HISTORY - For dredged or excavated material, indicate how each dredging or excavation site was used during the last 10 years.

NUMBER OF PAGES ATTACHED ☐

PART E - CHEMICAL, BIOLOGICAL AND PHYSICAL INFORMATION

41. CHEMICAL INFORMATION - Provide a chemical characterization of the substance. Attach detailed data and methods, and the quality assurance and control data and methods, where possible. If no data are provided, explain why. See the applicable item in Part I or II of Appendix I for details of additional information that must be included in your application.

NUMBER OF PAGES ATTACHED ☐

42. BIOLOGICAL INFORMATION - Provide an assessment of the potential effects of the substance, including toxicity, on living marine resources. Attach detailed bioassessment data and methods, an the quality assurance and control data and methods, where possible. If no data are provided, explain why.

NUMBER OF PAGES ATTACHED ☐

43. PHYSICAL INFORMATION - Provide an assessment of the potential of the substances, once dumped, to cause long-term physical effects. Attach detailed physical data and methods, and the quality assurance and control data and methods, where possible. If no data are provided, explain why. See the applicable item in Part I of Appendix I for details of additional information that must be included in your application.

NUMBER OF PAGES ATTACHED ☐

PART F - PROXIMITY AND MITIGATION

44. PROXIMITY TO THE FACILITIES - For dredged or excavated material, provide a map for each load site that shows, by means of the symbols indicated below, the location of the major operating and historical facilities in the vicinity of the site. Indicate you sources of information and attach a copy of the information where possible. Where the source is a person, provide the source's name, address and telephone number.

FACILITIES	SYMBOL		SOURCE OF INFORMATION
	OPERATING	HISTORICAL	
– Oil refineries	(O)	(O*)	
– Mills (give type)	(M)	(M*)	
– Mines (give type)	(N)	(N*)	
– Sewage outfalls	(S)	(S*)	
– Storm drains/pipes	(P)	(P*)	
– Shipping docks	(D)	(D*)	
– Other industries (specify)	(I)	(I*)	
– Other source of pollution and contamination (specify)	(C)	(C*)	

45. PROXIMITY TO SENSITIVE AREAS - For a new dump site, provide a map that shows, by means of the symbols indicated below, the location of all sensitive areas in the vicinity of the dump site. Indicate your sources of information and attach a copy of the information where possible. Where the source is a person, provide the source's name, address and telephone number.

SENSITIVE AREAS	SYMBOL	SOURCE OF INFORMATION
– Recreational areas	(RA)	
– Spawning and nursery areas	(SN)	
– Known migration routes or living marine resources	(MR)	
– Sport and commercial fishing areas	(FA)	
– Areas of natural beauty or cultural or historical importance	(BH)	
– Areas of special biological importance	(IS)	
– Mariculture	(MC)	
– Shipping lanes	(SL)	
– Areas of the seafloor having engineering uses (Mining, cables, desalination or energy conversion sites)	(EU)	
– Other areas (describe use)	(XZ)	

NUMBER OF PAGES ATTACHED ☐

46. MITIGATION - Indicate measures intended to minimize the environmental, health, navigational and aesthetic impacts during loading, transport and dumping. See the applicable item in Part II of Appendix I for details of additional information that must be included in your application.

47. TIME RESTRICTIONS - If the load site or dump site will be in the vicinity of spawning areas, migration routes or fishing areas, list the major species involved and the periods during which they are the most sensitive (active time of year).

Application is hereby made for a permit authorizing the activity described in this application. I certify that I have reviewed the information provided in this application and that, to the best of my knowledge and belief, the information is true, complete and accurate. I further certify that I am authorized to undertake the activity or am acting as a duly authorized agent of the applicant.

Date	Name (print)	Signature

Telephone No.	Fax No.

Send the completed permit application, together with all documents to be attached, to one of the following addresses.

For an application made from within Canada:

Regional Director
Atlantic Region
Conservation and Protection
Department of the Environment
Queen Square, 15th Floor
45 Alderney Drive
Dartmouth, Nova Scotia

Regional Director
Pacific and Yukon Region
Conservation and Protection
Department of the Environment
224 West Esplanade
North Vancouver, British Columbia
V7M 5V3

District Manager
Newfoundland District Office
Conservation and Protection
Department of the Environment
P. O. Box 5037
St. John's, Newfoundland
A1C 5V3

Regional Director
Quebec Region
Conservation and Protection
Department of the Environment
1179 de Bleury Street, 2nd Floor
Montreal, Quebec
H3B 3H9

District Manager
Northwest Territories District Office
Conservation and Protection
Department of the Environment
9th Floor, Bellanca Building
P. O. Box 370
Yellowknife, Northwest Territories
X1A 2N3

For an application made from outside Canada:

Director, Office Waste Management
Conservation and Protection
Department of the Environment
Ottawa, Ontario
CANADA
K1A 0H3

Appendix I

Part I

MINIMUM INFORMATION REQUIREMENTS‡ (BY TYPE OF SUBSTANCE) FOR DISPOSAL BY MEANS OTHER THAN INCINERATION OR OTHER THERMAL DEGRADATION

Each type of substance requires different information. Provide the required information on the form in the square indicated. Attach additional pages as needed. For incineration or other thermal degradation at sea, see Part II.

DREDGED MATERIAL AND EXCAVATED MATERIAL

14. Substance to be Dumped

Soil or sediment
Other components (e.g., wood waste)

41. Chemical Information

Chemistry of soil or sediment in respect of the following parameters:

- cadmium
- mercury
- polychlorinated biphenyls (PCBs)
- total polycyclic aromatic hydrocarbons (PAHs)
- low molecular weight PAHs
- high molecular weight PAHs
- total organic carbon

43. Physical Information

Grain size of soil or sediment

FISHERIES WASTE

14. Substance to Be Dumped

Species
Type of waste (e.g., shells, offal)
Source of waste

---

‡ The Minister may, pursuant to paragraph 71(1)(*b*) of the *Canadian Environmental Protection Act,* require further information for the purpose of taking into account any factor referred to in subsection 72(1) of that Act.

SHIPS, AIRCRAFT, PLATFORMS AND OTHER ANTHROPOGENIC STRUCTURES

14. Substance to Be Dumped

Name, if applicable
Location of registry
Model or official number
Dimensions
Weight (dead weight tonnage)
Principal materials of construction
Name and address of owner
State of seaworthiness, if applicable

41. Chemical Information

Cargo, fuel and hazardous materials, including chemicals, left on board

43. Physical Information

Last cargo
Type of engine, if left on board

SCRAP METAL AND OTHER BULKY ITEMS

14. Substance to Be Dumped

Principal components (composition) of substance
Dimensions
Weight (t)

41. Chemical Information

Contamination by hazardous materials including chemicals

OTHER SUBSTANCES

14. Substance to Be Dumped

Principal components (composition) of substance
Origin of substance and process giving rise to substance

Appendix I

Part II

MINIMUM INFORMATION REQUIREMENTS‡ FOR INCINERATION OR OTHER THERMAL DEGRADATION

Provide the required information on the form in the square indicated. Attach additional pages as needed.
For an activity other than incineration or other thermal degradation at sea, see Part I.

ALL SUBSTANCES

14. Substance to Be Dumped

Principal components (composition) of substance
Description of the products of combustion and the rate of their production
Origin of substance and process giving rise to substance

20. Equipment and Methods

Description of incineration equipment
Description of air pollution control equipment
Description of monitoring and control systems in place
Stack dimensions
Combustion temperature
Retention time
Combustion and destruction efficiency
Proposed method of loading and storage
Capability of meeting *Operating and Emission Guidelines for Municipal Solid Waste Incinerators,* CCME-TS/WM-TRE003, as amended from time to time, published by the Canadian Council of Ministers of the Environment
Capability of meeting the Regulations for the control of Incineration of Wastes and Other Matter at Sea, as amended from time to time, set forth in Annex I of the *Convention on the Prevention of Marine Pollution by Dumping of Wastes and Other Matter,* entered into force on August 30, 1975

41. Chemical Information

Results of the latest tests on stack emissions (for particulate matter, hydrogen chloride (HCl), carbon monoxide (CO), dioxins and furans)

46. Mitigation

Methods of complying with applicable noise by-laws
Methods of managing ash and minimizing fugitive emissions
Methods of managing wastewater to comply with provincial or municipal discharge limits
Methods of preventing hazards to other vessels
Methods of spill response and contingency plans in the event of a spill
Methods of emergency shutdown
Qualifications of the operating personnel

---

‡ The Minister may, pursuant to paragraph 71(1)(*b*) of the *Canadian Environmental Protection Act,* require information for the purpose of taking into account any factor referred to in subsection 72(1) of that Act.

Appendix II

MINIMUM INFORMATION REQUIREMENTS‡ FOR DISPOSAL AT NEW DUMP SITES AND ON ICE

Provide the information on the form in the square indicated. Attach additional pages as needed. Contact your regional ocean dumping control office prior to collecting data on a new dump site, as some of the information may already be on file.

DISPOSAL AT A NEW DUMP SITE

18. Dump Site(s)

Bathymetry
Sediment transport
Salinity
Current flows
Sediment chemistry in respect of the following parameters:
- cadmium
- mercury
- polychlorinated biphenyls (PCBs)
- total polycyclic aromatic hydrocarbons (PAHs)
- low molecular weight PAHs
- high molecular weight PAHs

DISPOSAL ON ICE

18. Dump Site(s)

Area of ice to be used as the dump site
Thickness of ice at the proposed dump site (m)
Estimated date of ice breakup (year/month/day)
Estimated location of ice breakup (lat./long.)
Estimated time from breakup to melting (days)
Estimated depth of water at dump site (m)

---

‡ The Minister may, pursuant to paragraph 71(1)(*b)* of the *Canadian Environmental Protection Act,* require further information for the purpose of taking into account any factor referred to in subsection 72(1) of that Act.