TESTING OF DREDGED MATERIAL FOR MARINE DISPOSAL

GEO REPORT No. 83

EVS Environment Consultants

GEOTECHNICAL ENGINEERING OFFICE
CIVIL ENGINEERING DEPARTMENT
THE GOVERNMENT OF THE HONG KONG SPECIAL ADMINISTRATIVE REGION

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PREFACE

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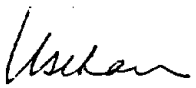

R.K.S. Chan
Principal Government Geotechnical Engineer
January 1999

FOREWORD

This report provides guidance on environmental laboratory procedures for chemical analyses and biological testing of dredged material.

The report was prepared by EVS Environment Consultants of Canada for Geotechnical Engineering Office (GEO) of the Civil Engineering Department under Agreement No. CE 68/94. Dr. K.C.Ng of GEO coordinated the study and reviewed the report.

D.C.H. Chang
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LIST OF ACRONYMS

AAS	Atomic Absorption Spectroscopy
AFD	(Hong Kong) Agriculture and Fisheries Department
AOAC	Association of Official Analytical Chemists
APHA	American Public Health Association
ASTM	American Society for Testing and Materials
AVS	Acid Volatile Sulphide
CAEAL	Canadian Association of Environmental Analytical Laboratories
CED	(Hong Kong) Civil Engineering Department
COC	Contaminant of Concern
CRM	Certified Reference Material
CSA	Canadian Standards Association
CV	Coefficient of Variation
CVAAS	Cold Vapour Atomic Absorption Spectroscopy
CVAF	Cold Vapour Atomic Fluorescence
DDT	1,1-bis(4-Chlorophenyl)-2,2,2-trichloroethane
DL	Detection Limit
DQO	Data Quality Objective
EPA	(US) Environmental Protection Agency
EPD	(Hong Kong) Environmental Protection Department
ESC	East Sha Chau
FAAS	Flame Atomic Absorption Spectroscopy
GC	Gas Chromatograph
GC/ECD	Gas Chromatography/Electron Capture Detection
GC/ECD-PID	Gas Chromatography/Electron Capture Detection/Photoionization Detection
GC/FPD	Gas Chromatography/Flame Photometric Detection
GC/MS	Gas Chromatography/Mass Spectrometry
GFAAS	Graphite Furnace Atomic Absorption Spectroscopy
GPC	Gel Permeation Chromatography
HOKLAS	Hong Kong Laboratory Accreditation Scheme
HPAH	High Molecular Weight PAH
HPLC	High Performance Liquid Chromatography
HRMS	High Resolution Mass Spectrometry

ICP-OES	Inductively Coupled Argon Plasma Spectrometry/Optical Emission Spectroscopy
ICP-MS	Inductively Coupled Argon Plasma Spectrometry/Mass Spectrometry
ISQV	Interim Sediment Quality Value
LPAH	Low Molecular Weight PAH
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology
NOAA	National Oceanic and Atmospheric Administration
NRCC	National Research Council of Canada
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PSDDA	Puget Sound Dredged Disposal Analysis
PSEP	Puget Sound Estuary Program
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percent Difference
SAD	Strong Acid Digestion
SD	Standard Deviation
SOP	Standard Operating Procedure
SEM	Simultaneously Extracted Metals
SRM	Standard Reference Material
SW-846	EPA Test Methods for Evaluating Solid Waste (SW-846 3rd edition)
TAD	Total Acid Digestion
TBT	Tributyltin
TOC	Total Organic Carbon
TTS	Tiered Testing System
USACE	US Army Corps of Engineers

GLOSSARY

Note: Explanations of glossary terms are provided here for guidance and may not be universally definitive.

Abnormality — A deviation from the normal; in bioassays, a difference in gross morphology from control animals.

Accuracy — The agreement between an analytical result and the true value.

Acute — With reference to toxicity, having a sudden onset, lasting a short time (usually within 4 to 7 days for fish). Of a stimulus, severe enough to induce a response rapidly. Can be used to define either the exposure or the response to an exposure (effect). The duration of an acute aquatic toxicity test is generally 4 days or less for water, and 10 days or less for sediments, and mortality is the response measured. An acute effect could also be a mild or sublethal one.

Amphipods — Small shrimp-like crustaceans (e.g., sand fleas). Many live on the bottom, feed on algae and detritus, and serve as food for marine species. Amphipods are commonly used in laboratory bioassays to determine the toxicity of sediments because they are relatively sensitive to chemical toxicity.

Analyte — That which is identified and quantified in the process of analyzing samples.

Analytical Spike — A duplicate aliquot of a prepared sample, fortified with the analyte of interest and analyzed exactly the same as, and immediately after, the unspiked sample.

Aqua Regia — One part of nitric acid and three parts of hydrochloric acid; used chiefly to dissolve metals.

Aroclors — Commercial mixtures of polychlorinated biphenyls (PCBs), each containing a large number of isomers and identified by numbers reflecting the average degree of chlorination.

Batch — The number of samples that are prepared or analyzed with associated laboratory QC samples at one time. A typical batch size is 20 samples.

Benthic — Referring to organisms living in or on the sediments of aquatic/marine habitats.

Bias — The systematic or persistent distortion of a measurement process which causes errors in one direction.

Bioaccumulation A general term, meaning that an organism stores within its body a higher concentration of a substance than is found in its environment. Includes uptake of substances from water (=bioconcentration) and from food. This phenomenon is not necessarily harmful. For example, freshwater fish must bioaccumulate common salt if they are to live because the water in which they swim dissolves the salts out of their bodies. Many toxicants, such as arsenic, can be excreted by aquatic organisms, and are not included among the bioaccumulative substances (e.g., certain chemicals in food eaten by a fish tend to accumulate in its liver or other tissues).

Bioassay The use of an organism or part of an organism as a method for measuring or assessing the presence or biological effects of one or more substances under defined conditions. A bioassay test is used to measure a degree of response (e.g., growth, death, contaminant uptake) produced by exposure to physical, chemical or biological variables. Bioassays include toxicity tests and bioaccumulation studies.

Bioavailability The degree to which the total chemical in the surrounding environment can be taken up by organisms. The environment may include water, sediment, suspended particles, and food items.

Bivalve A class of molluscs that have a shell composed of two hinged valves; includes clams, mussels, oysters.

Calibration The systematic standardization of the response of instruments used for measurements.

Certified Reference Material A reference material accompanied by, or traceable to, a certificate stating the concentration of chemicals contained in the material. The certificate is issued by a public or private organization that routinely certifies such material (e.g., NRCC, NIST).

Chain of Custody An unbroken trail of accountability that ensures the physical security of samples, data, and records.

Check Standard A QC sample prepared independently of calibrated standards, analysed exactly like the samples, and used to estimate analytical precision and indicate bias due to calibration.

Chlorinated Hydrocarbons A class of organic compounds composed of carbon, hydrogen and chlorine. Specific toxicities depend on the number and location of chlorine atoms in the molecule.

Chlorophenols	Compounds with the basic phenol structure but with one (chlorophenol) to five (pentachlorophenol) chlorine atoms substituted for hydrogens on this structure. Chlorophenol compounds are commonly used as wood preservatives, and are produced as a by-product of the wood pulping process.
Chronic	Involving a stimulus that is lingering or continues for a long time; often signifies periods from several weeks to years, depending on the reproductive life cycle of the aquatic species. Can be used to define either the exposure or the response to an exposure (effect). Chronic exposure typically induces a biological response of relatively slow progress and long continuance. A chronic aquatic toxicity test is used to study the effects of continuous, long-term exposure of a chemical or other potentially toxic material on aquatic organisms.
Cold Vapour Atomic Absorption Spectroscopy	A technique for the analysis of mercury, whereby mercury is selectively chemically reduced to an elemental state and aerated from solution in a closed system. Absorption of the vapour at a given wavelength is a measure of the concentration of mercury in the sample.
Coefficient of Variation	The standard deviation expressed as a percentage of the mean.
Comparability	The confidence with which one data set can be evaluated in relation to another data set.
Completeness	A measure of the amount of data that is determined to be valid in proportion to the amount of data collected.
Contaminant of Concern	A chemical at a site that may cause risks to exposed organisms.
Control	A treatment in a toxicity test that duplicates all the conditions of the exposure treatments but contains no test material. The control is used to determine the absence of toxicity in the basic test conditions (e.g., health of test organisms, quality of dilution water).
Control Limit(s)	A value or range of values against which results of QC sample analyses are compared in order to determine whether the performance of a system or method is acceptable. Control limits are typically statistically derived. When QC results exceed established control limits, appropriate corrective action should be taken to adjust the performance of the system or method.

Control Sediment Clean sediment, taken from the site where the test organisms are collected, and intended for use in the 10-day test with amphipods. This sediment must contain no test material, and must enable ≥90% survival of the test organisms during the 10-day period of exposure. It is usually a sample of sieved sediment, collected at the time that the test organisms are obtained. The use of control sediment provides a basis for interpreting data derived using test sediment(s).

Data Quality Objectives Qualitative and quantitative statements that define the appropriate type and quality of data needed to support the objective of a given project.

Dioxins A group of approximately 75 chemicals of the chlorinated dibenzodioxin family. 2,3,7,8-TCDD is considered the most toxic form.

Duplicate A homogenous sample is split either in the field or in the laboratory with the duplicate presented to the analyst as an additional sample to check for precision. If more than two splits are analyzed, the term replicate is normally used.

Duplicate Analysis Analysis performed on a second subsample in the same manner as the initial analysis, used to provide an indication of measurement precision.

Echinoderms Includes organisms from the phylum Echinodermata (e.g., sea urchins, sand dollars, sea cucumbers, starfish). These organisms are marine and are characterized by having radial symmetry, an endoskeleton with external spines and a water-vascular system.

Ecotoxicology The science that deals with toxins and their effects in natural ecosystems and on living organisms.

Effect Significant and meaningful difference measured in an environmental variable between an exposed and reference area, or operational vs. baseline conditions.

Elutriate A standard test used to predict the release of contaminants in sediment to a water column resulting from open water disposal of the sediment.

Endpoint The variable(s) (i.e., time, reaction to the organisms, etc.) that indicate(s) the termination of a test, and also means the measurement(s) or value(s) derived that characterizes the results of the test (e.g. EC50, LC50).

Flame Atomic Absorption Spectroscopy	A technique for metals analysis in which a sample is aspirated and atomized in a flame through which light of a prescribed wavelength is directed. The amount of light from the light beam that is absorbed by the flame is a measure of the concentration of metal in the sample.
Furans	A group of many chlorinated dibenzofurans. The most common furan in pulp mill effluents is 2,3,7,8-TCDF. A furan contains one less oxygen molecule than does a dioxin.
Graphite Furnace Atomic Absorption Spectroscopy	A technique for metals analysis in which a sample is atomized in a graphite tube in a furnace, and the resulting vapour placed in a beam of radiation containing excited molecules of the element to be measured. Attenuation of the transmitted radiation is a measure of the concentration of that element in the sample.
Holding Time	The storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are used.
Inductively Coupled Argon Plasma Mass Spectrometry	A technique for multi-element analysis at relatively low concentrations. When nebulized samples are introduced into a radio frequency inductively coupled argon plasma, ions are produced. The positively charged ions are transmitted by a quadrapole mass filter, based upon selected mass to charge ratios, and isotope-specific mass spectra are produced.
Inductively Coupled Argon Plasma Optical Emission Spectroscopy	A technique for simultaneous or rapid sequential analysis for many elements in a short time. Element-specific atomic-emission line spectra of nebulized samples are produced by radio-frequency inductively coupled plasma.
Interstitial Water	The water within a wet sediment that surrounds the sediment particles (sometimes referred to as porewater).
Lethal	Causing death, or sufficient to cause death.
Matrix	The sample material in which the analytes of interest are found (e.g., water, sediment, tissue).
Matrix Spike	A QC sample created by adding known amounts of analytes of interest to an actual sample, usually prior to extraction or digestion. The matrix spike is analysed using normal analytical procedures. The result is then corrected for the analyte concentration determined in the unspiked sample, and expressed as percent recovery. This provides an indication of the sample matrix effect on the recovery of target analytes.

Method Blank — A QC sample intended to determine the response at zero concentration of analyte and assess the positive contribution from sample analysis procedures to the final result. A clean matrix (generally water) known to be free of target analytes that is processed through the analytical procedure in the same manner as associated samples.

Method Detection Limit — The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero; determined from analysis of a sample in a given matrix containing the element.

Phenols — A group of acidic organic compounds analogous to phenol (C_6H_5OH) and regarded as hydroxylated (containing the -OH group) derivatives of aromatic hydrocarbons.

Polychaetes — Annelid worms of the class Polychaeta including mostly marine forms with well developed parapodia (e.g., clam worm *Nereis (Neanthes)*).

Polychlorinated Biphenyl — A class of about 70 different persistent, man-made, organic chemicals (consisting of carbon, hydrogen and chlorine) which tend to bioaccumulate through the food chain, cause reproductive failure and cancer. A family of chemically inert compounds, having the properties of low flammability, volatility, water insolubility, and high electrical insulation quality. Past applications include use as hydraulic fluids, heat exchange, dielectric fluids, and plasticizers for plastics. They were banned in 1980, except for continued use in existing electrical equipment.

Polycyclic Aromatic Hydrocarbon — A class of complex organic compounds, some of which are persistent and carcinogenic. These compounds are formed from the combustion of organic material and are ubiquitous in the environment. PAHs are found in fossil fuels such as coal and oil and are formed by incomplete combustion of organic fuels like gasoline, wood and oil. They are commonly formed by forest fires, wood stoves, and internal combustion engines. They often reach the aquatic environment through atmospheric fallout, highway runoff and oil discharge.

Precision — The statistical agreement among independent measurements determined from repeated applications of a method under specified conditions.

Quality Assurance	An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvements to ensure that a process, item or service is of the type and quality needed and expected by the customer.
Quality Control	The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process. Quality control is an element of quality assurance. QC samples and auditing/assessment are common quality control activities.
Recovery	The percentage difference between two measurements, before and after spiking, relative to the concentration spiked, or the percentage difference between a measured value and a true value, as in the case of a reference material or check standard.
Reference Material	A material of known analyte composition which can be used for comparison of analytical results but for which the reported analyte concentrations have not been certified (see Certified Reference Material).
Reference Sediment	A field-collected sample of sediment, taken from a site thought to be relatively free of contaminants (i.e., "clean" sediment), and intended for use in sediment bioassays. It is often collected from a site within the general vicinity of a test sediment, and is frequently selected for biological testing because of its geochemical similarity (e.g., particle size, compactness, total organic content) to the test sediment(s).
Reference Toxicant	A chemical of quantified toxicity used to test organisms, to gauge fitness, health and sensitivity of a batch of test organisms.
Relative Percent Difference	Difference of two measurements, x_1 and x_2, divided by the mean of the measurements, multiplied by 100.
Replicate	One of several identical experiments, procedures, or samples. Duplicate is a special case of replicates consisting of two samples or measurements.
Representative-ness	The degree to which data accurately and precisely represent an environmental condition.
Semivolatile Organic Compounds	Gas chromatographable organic compounds with moderate or low vapour pressures that can be extracted from samples using organic solvents.
Spike	The addition of a known amount of a substance to a sample or a blank.

Standard	A substance or material, the properties of which are believed to be known with sufficient accuracy to permit its use to evaluate the same property of a sample. In chemical measurements, a standard often describes a solution of analytes used to calibrate an instrument, commonly prepared by the analyst, to establish a calibration curve or the analytical response function of an instrument.
Standard Reference Material	A material with known properties produced and distributed by the National Institute of Standards and Technology (NIST).
Sublethal	Involving a stimulus/concentration below the level that causes death. Exposure to sublethal concentrations of a material may produce less obvious effects on behaviour, biochemical and/or physiological function, and histology of organisms.
Surrogate Spike Compound	A compound that has characteristics similar to that of a compound of interest, is not expected to be found in environmental samples, and is added to a sample prior to extraction. The surrogate compound can be used to estimate the recovery of chemicals in the sample.
Tiered Testing System	A testing procedure in which all testing is not done synoptically, or even concurrently. Initial testing is done to determine areas for in-depth study, which may (or may not) involve a more thorough step.
Toxicity	The inherent potential or capacity of a material to cause adverse effects (lethal or sublethal) in a living organism. Toxic effects are a result of concentration and exposure time, and are modified by variables such as temperature, chemical form and availability.
Toxicity Test	A determination of the effect of a material on a group of selected organisms of a single species under defined conditions. An aquatic toxicity test usually measures either (a) the proportions of organisms affected (quantal) or (b) the degree of effect shown (graded or quantitative), after exposure to a specific test material (e.g., a sample of sediment).
Trace Metals	Elements which are present in a matrix at trace concentrations (e.g., trace metals in sea water include arsenic, cadmium, chromium, copper, lead, nickel, and silver).

Validation	Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Can refer to a process whereby environmental data are determined by an independent entity to be complete and final (i.e., subject to no further change), and to have their value for the intended use described by both qualitative and quantitative statements.
Volatile Organic Compounds	Organic compounds with high vapour pressures that tend to evaporate readily from a sample.
Warning Limit	A value either above or below which data returned by a laboratory are subjected to qualification before inclusion in a regional database. The principle is identical to that of a control limit, but is less stringent and serves as a warning that the system or method may become out of control.

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EXECUTIVE SUMMARY

As part of the tiered testing system (TTS) which has been proposed for the Classification of Dredged Material for Marine Disposal, sediment sampling for chemical analyses and possible biological testing will be required. The ability to quantify chemical and toxicology information in a reliable and technically defensible manner is key to sound environmental management and protection programs.

The purpose of this document is to provide guidance on environmental laboratory procedures consistent with the chemical and biological analyses being recommended as part of the TTS. Although guidance is provided for several priority contaminants of concern (COCs) identified in Hong Kong, the document focuses on the subset of priority COCs for which interim sediment quality values (ISQVs) have been developed. Specific objectives were to:

- Review current capabilities of environmental laboratories in Hong Kong.
- Identify and reference recognized and acceptable chemistry methodologies and bioassay protocols for marine sediments.
- Provide guidance for assessing quality and acceptability of environmental laboratory data.

Information on the analytical capabilities, capacity and relative costs of specific sediment chemical analyses was obtained from government and commercial laboratories in Hong Kong that are currently conducting environmental testing. Although the laboratories generally met the requirements of the TTS for metals analysis, their current capabilities may not support the TTS requirements (e.g., analytical detection limit above lowest ISQV concentrations) for organic analysis.

Methods for performing chemical analyses for priority COCs identified for Hong Kong sediments were described. These included analyses for metals, several categories of organic parameters, and conventional parameters. Methods for performing biological screening tests (sediment toxicity and bioaccumulation tests) were described. Suggested techniques for further biological assessment were also outlined. There are currently no commercial laboratories in Hong Kong that perform sediment bioassay tests, so these tests will have to be performed elsewhere until capabilities can be developed locally.

1.0 INTRODUCTION

1.1 BACKGROUND

To allow decision-making as part of the tiered testing system (TTS) described in EVS (1996a), sampling of dredged sediment and reference sediment will be required for chemical analyses (i.e., priority contaminants of concern [COCs]) and possibly biological testing (i.e., toxicity tests and bioaccumulation studies). The framework for the TTS is shown in Figure 1. Environmental laboratories perform analyses which enable the chemical concentrations and toxicity of samples to be quantified. This quantification allows the classification of the dredged materials by: comparing measured sediment contaminant concentrations to interim sediment quality values (ISQVs; EVS, 1996a) which demarcate concentrations unlikely or very likely to cause adverse biological effects; and by assessing specific toxicity (i.e., lethal and sublethal effects) or bioaccumulation potential related to a given sediment sample. Based on results of the tiered evaluations, dredged materials are classified as Class 1 - suitable for marine disposal, Class 2 - requiring confined marine disposal (i.e., contaminated mud disposal facility at East Sha Chau [ESC]), or Class 3 - non-marine disposal. Therefore, the ability to quantify chemical and toxicological information in a reliable and technically defensible manner is key to sound environmental management and protection programs.

1.2 PURPOSE AND OBJECTIVES

The purpose of this document is to provide guidance on the identification of environmental laboratory procedures consistent with chemical and biological testing recommended as part of the TTS for evaluation of dredged material for marine disposal. Although guidance is provided for several priority COCs identified in Hong Kong, the document focuses on the subset of priority COCs for which ISQVs have been developed (i.e., ISQV contaminants) (Table 1; EVS, 1996a). Specific objectives are to:

1. Review the current capabilities of environmental laboratories in Hong Kong.

2. Identify and reference recognized and acceptable analytical chemistry testing methodologies for priority COCs identified for Hong Kong sediments (EVS, 1996a), as well as for other classes of contaminants which may also be of interest.

3. Identify and describe recognized and acceptable bioassay protocols for testing marine sediments.

4. Provide guidance on assessing the quality and acceptability of data generated by a testing laboratory.

1.3 APPROACH

The review of present environmental testing capabilities in Hong Kong (for both chemistry and bioassay testing) involved obtaining information on laboratory services from the Hong Kong Laboratory Accreditation Scheme (HOKLAS) and directly from selected laboratories in Hong Kong. The HOKLAS technical criteria for laboratory accreditation were also reviewed for requirements for demonstrating technical competence. These criteria tended to be geared towards the overall laboratory quality system, rather than project-specific performance and deliverables. A list of contacts who supplied information presented in this chapter is provided in Table 2. The identification and review of various analytical and bioassay methodologies involved the compilation of accepted and proven methodologies from commercial and government laboratories in Canada, the United States, and Hong Kong. Guidance for assessing the quality or acceptability of chemistry or toxicity data generated by a testing laboratory was based primarily on EPA (1995) and PSEP (1995; 1996a,b,c), which provided recent reviews of methods for evaluation of marine samples required by regulatory programs in North America.

The guidance provided in this document focusses on performance of laboratory analyses. Recommendations for design of sampling programs, sample collection methods, and decision criteria for evaluation and interpretation of biological testing results are outside the scope of this document and have not been included.

2.0
CURRENT ENVIRONMENTAL LABORATORY CAPABILITIES

2.1 ANALYTICAL CHEMISTRY

Information on the analytical capabilities of laboratories in Hong Kong was obtained from HOKLAS (1996). To supplement the information contained in HOKLAS (1996), questionnaires were sent to various Hong Kong laboratories (see below). The purpose of the questionnaire was to identify additional specific information with regard to testing capabilities for the priority COCs recommended in EVS (1996a) and more specifically ISQV contaminants. Note that testing capabilities for soil are generally the same as for sediments.

HOKLAS is a voluntary laboratory accreditation scheme, open to any Hong Kong laboratory that performs objective testing falling within the scope of the scheme and meeting the HOKLAS criteria of competence. This competence is measured relative to ISO/IEC Guide 25 (ISO, 1990) which contains all the quality system elements of ISO 9000 which are relevant to laboratory operation for calibration and testing activities. ISO (1990) is also used as the foundation for Canada's national accreditation program for environmental laboratories (CSA, 1995; CAEAL, 1995).

HOKLAS (1996) covers many areas of testing services, but this document focusses on laboratories listed under the category of environmental testing as specifically conducting analysis of sediment, soil and biota samples. The Government Laboratory and three commercial laboratories (Inchcape Testing Services Hong Kong Limited; MateriaLab Limited; and ALS Technichem (HK) Pty Ltd.) were identified by HOKLAS as accredited Hong Kong laboratories having these capabilities. Hong Kong laboratories described in this document are identified according to their HOKLAS accreditation number.

Specific information with respect to analysis of the ISQV contaminants has been summarized in Tables 3, 4, and 5. These tables show the detection limits of the laboratories compared to available ISQV-low concentrations (i.e., minimum concentrations which should be detectable; EVS, 1996a), the analytical methods used, and the costs for the analyses. Methods listed in Table 4 may include some which are not currently HOKLAS accredited. Analytical costs were per sample, based on a batch of 10 samples, and do not include shipping. For illustrative purposes, two international laboratories (ASL Analytical Service Laboratories Ltd., Vancouver, Canada and Analytical Resources Incorporated, Seattle, USA) have been included in these tables. Both of these laboratories are accredited by the Washington State Department of Ecology (USA), and ASL is accredited in Canada by CAEAL. General information on the capabilities of each of the four Hong Kong laboratories is outlined below, including specific information with respect to ISQV contaminants. It is important to note that this

information was current at the time this document was published, but that laboratory capabilities are subject to change, especially as instrumentation and expertise improve.

Government Laboratory (HOKLAS No. 1) — The laboratory is a government funded organization that provides testing and advisory capabilities to government departments free of charge. Testing services are not normally available to the general public. The laboratory has state-of-the-art instrumentation including: ion coupled plasma/mass spectrometry (ICP/MS), ion coupled plasma (ICP), flame atomic absorption spectrometry (FAAS), hydride generation atomic absorption spectrometry (HGAAS), cold vapour atomic absorption spectrometry (CVAAS), and graphite furnace atomic absorption spectrometry (GFAAS) for metals; and gas chromatography/mass spectrometry (GC/MS), high resolution mass spectrometry (HRMS), gas chromatography (GC), and high performance liquid chromatography (HPLC) for trace organic analyses. Capabilities include a full suite of metal and organic parameters for sediments/soils and biota using APHA, EPA, and in-house methods. In 1995, the laboratory carried out 220,000 analyses of water, sediment, sludge, waste and air samples for the EPD.

The Government Laboratory has equipment and testing capabilities to measure the ISQV contaminants using acceptable detection limits (Tables 3 and 4), with the possible exception of PAHs. Specific comments and recommendations with respect to the grouping of analytical test categories are as follows:

Metals and Metalloids	Adequate detection limits and a wide range of available instrumentation.
Organics - PAHs	The suite of PAH parameters does not currently cover all the PAH compounds for which ISQVs have been developed, although detection limits are adequate for those being analysed. Based on HOKLAS (1996), the laboratory is using an HPLC method for these parameters. This method has generally been replaced by GC/MS based methods, in particular by EPA Method 8270C.
Organics - non-PAHs	Adequate detection limits for PCBs and pesticides and a wide range of available instrumentation. Tributyltin (TBT) analyses are performed.

Services of the Government Laboratory are not currently available on a commercial basis, so costs for sample analyses are not provided.

Inchcape Testing Services Hong Kong Limited (HOKLAS No. 5) — The laboratory is part of a worldwide network of analytical laboratories. Laboratory instrumentation includes: ICP, FAAS, CVAAS, GFAAS, and HGAAS for metals; and

GC, GC/MS, HPLC for trace organic analyses. Capabilities include a wide range of metal and organic parameters from sediments/soils using APHA, EPA, and in-house methods. Inchcape-Hong Kong typically analyses about 45 to 50 sediment samples each week (i.e., approximately 2500 samples per year).

In general, the capabilities of Inchcape Testing Services do not currently meet requirements for the analysis of the ISQV contaminants (Tables 3 and 4). Specific comments and recommendations with respect to the grouping of analytical test categories are as follows:

Metals and Metalloids	Adequate detection limits, although detection limits for cadmium, mercury and silver are close to or equal to ISQV-low concentrations. Methodology using ICP, AAS, HGAAS, and CVAAS is sound.
Organics - PAHs	Inadequate detection limits for most of the individual PAH compounds. The laboratory is currently using EPA Method 8270A (GC/MS) which has since been replaced by EPA Method 8270C (GC/MS). Capabilities should be upgraded to use newer methods.
Organics - non PAHs	Inadequate detection limits for PCBs and pesticides, despite the fact that the laboratory is using EPA Method 8081 (GC/ECD-capillary column). Analyte extraction and clean-up methods may need to be improved. This laboratory does not currently perform TBT analyses.

The cost for the analysis of a sediment sample for the ISQV contaminants (excluding TBT) is US$786 per sample (Table 5).

MateriaLab Limited (HOKLAS No. 15) — Laboratory instrumentation includes: AAS, CVAAS, and HGAAS for metals; and GC and GC/MS for trace organic analyses. Materia Lab typically analyses about 1100 sediment and/or biota samples per year for heavy metals and about 40 sediment and/or biota samples per year for organic parameters.

In general, the capabilities of MateriaLab do not currently meet the requirements for analysis of the ISQV contaminants (Tables 3 and 4). Specific comments and recommendations with respect to the grouping of analytical test categories are as follows:

Metals and Metalloids	Adequate detection limits. Detection methods are acceptable (AAS, HGAAS, CVAAS), but standardized methods (e.g., EPA, APHA) rather than in-house methods should be used. ICP instrumentation could be considered where appropriate.
Organics - PAHs	Inadequate detection limits for some PAH compounds, despite the fact that the laboratory is currently using EPA Method 8270 (GC/MS). Analyte extraction and clean up methods may need to be improved.
Organics - non-PAHs	Inadequate detection limits for PCBs, pesticides and TBT. The laboratory is using EPA Method 8080 (GC/ECD - packed column) rather than EPA Method 8081/8082 which uses a capillary column instead of a packed column. The latter method enables better separation, resolution, and detection of compounds.

The cost for the analysis of a sediment sample for the ISQV contaminants is US$995 per sample (Table 5).

ALS Technichem (HK) Pty Ltd. (HOKLAS No. 66) — The laboratory is part of the largest environmental analytical group in Australasia and has the broadest range of HOKLAS accreditation for environmental laboratories in Hong Kong. Laboratory instrumentation includes: ICP/MS, AAS, HGAAS for metals; and GC/MS and GC for trace organic analyses. Capabilities include the analyses of a wide range of metal and organic parameters using APHA, EPA, and in-house methods. ALS Technichem (HK) typically analyzes about 120 sediment samples per week (i.e., approximately 6,000 per year) for metals and organics.

In general, the capabilities of ALS Technichem do not currently meet the requirements for analysis of the ISQV contaminants (Tables 3 and 4). Specific comments and recommendations with respect to the grouping of analytical test categories are as follows:

Metals and Metalloids	Excellent detection limits produced by using EPA Method 6020 (ICP/MS) and CVAAS techniques.
Organics - PAHs	From the information supplied, the detection limits are inadequate for most of the PAH compounds. The laboratory currently uses EPA Method 8270 (GC/MS) which should be able to provide adequate detection limits for the range of compounds necessary.

Organics - non-PAHs	Inadequate detection limits for PCBs and pesticides. The laboratory currently uses EPA Method 8270 (GC/MS) which is less sensitive for chlorinated compounds than EPA Method 8081/8082 (GC/ECD - capillary column). Detection limits for TBT are adequate.

The cost for the analysis of a sediment sample for the ISQV contaminants is US$645 per sample (Table 5).

2.2 SEDIMENT BIOASSAY TESTING

There are presently no regulatory requirements in Hong Kong for conducting bioassay testing on marine sediments. Although the Agriculture and Fisheries Department (AFD) has experience conducting aquatic toxicity tests with oil dispersants, none of the government or commercial laboratories are equipped to perform sediment bioassays at present. While the equipment needed to conduct bioassays is not as sophisticated or expensive as that required for chemical analyses, setting up a bioassay testing facility is more complex because of the need for a supply of clean, uncontaminated seawater; collection, culture and maintenance of test organisms; and photoperiod and temperature control during testing. Some work is being done by university researchers to develop suitable bioassays for use with Hong Kong species (R. Wu, 1996, pers. comm.). However, these methods have not been sufficiently tested to be used in a routine or regulatory capacity. For the present, bioassay tests are being performed by laboratories outside Hong Kong. Guidance is provided in this document to assist in selecting suitable test protocols for Hong Kong and evaluating the quality of the testing services.

3.0
RECOMMENDED METHODS - ANALYTICAL CHEMISTRY

Recommended methods for the analysis of sediment parameters (particularly the ISQV contaminants) identified in EVS (1996a) are summarized below. These methods are also applicable to soil samples. Guidance on selection of appropriate parameters and analytical methods is based primarily on EPA (1995) and PSEP (1996a,b,c), which represent recent reviews of procedures for evaluating dredged material for regulatory programs in North America. Other methods, not included in this document, may also be appropriate provided they meet specific study objectives and the data quality requirements in this document. In conducting these analyses, it is important that laboratories comply with all environmental laws and safety requirements (e.g., sample disposal) relevant to their jurisdiction.

The list of parameters for analysis is usually defined from a review of available historical data, and by identifying any specific inputs to the area in question (e.g., pesticides from agricultural runoff, tributyltin in shipyards). The proposed Hong Kong TTS identifies the minimum ISQV contaminants for inclusion and at a maximum, 22 Category 1 COCs (individual compounds or classes of compounds) may be included. The option also exists for regulatory bodies to include other contaminants on a situation (e.g., spill) and/or site specific basis. From the list of parameters developed, appropriate methods for sample preparation and analysis should be selected that will provide adequate detection limits (e.g., for comparison to ISQV-low concentrations). For example, PSEP (1996c) recommends that detection limits should be at least three times lower than the project-specific detection requirements. This may not always be possible, but the usefulness of data is increased if it is not subject to the inherent analytical variability associated with concentrations measured near the detection limit.

PSEP (1996bc) recommend the use of EPA methods in general, preferably methods based on the most recent update of SW-846 (EPA, 1986). This is a methods manual that is updated as new data and advances in analytical techniques occur, by incorporation as new or revised methods. Where a suitable EPA method is not available, then a validated standard method from another recognized source, such as APHA, is recommended. Where no standard method is available, the method chosen must be a written method and the laboratory must document method performance and the ability to meet data quality objectives (PSEP, 1996b).

3.1 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

Each of the analytical methods referred to in this document has an established set of quality assurance/quality control (QA/QC) measures that should be followed in order to obtain reliable data that is technically defensible. It is important that laboratories establish

and maintain a formal QA program, such as is required under the HOKLAS accreditation program, to increase the quality of the data being generated. If analyses are to be performed by laboratories outside Hong Kong, they should be able to demonstrate competence equivalent to HOKLAS accreditation criteria.

Before performing a chemical analysis, the laboratory needs to establish its own limits for performance of a particular method (i.e., method validation, establishing detection limits). This is usually done by analysis of QC samples. EPA (1995) provides guidance on this, summarized below, which is consistent with and in some cases more stringent than HOKLAS criteria. Some laboratories establish limits (e.g., control charts) for their own measurement systems, and these limits need to be evaluated to ensure that they meet generally accepted guidelines or that there are acceptable reasons for having a less stringent limit. Also if a laboratory has consistently demonstrated better performance than indicated by general guidelines, then those limits should be used to determine whether a problem is present. Exceedance of warning limits indicates that the QC sample data need some sort of qualification before they can be accepted. These limits serve as a warning that some component of the analytical system may not be performing normally and that data should be qualified as "estimated" before using the results for technical analysis; the standard value for warning limits is ± 2SD. Control limits are limits placed on the acceptability of QC sample data. Exceeding the control limits indicates that the analytical system or instrument is performing abnormally and needs to be corrected. Data that exceed control limits are often rejected and excluded from a project database. The standard value for control limits is ± 3SD.

Instrument calibration is always required because it is the means by which instrument responses are properly translated into chemical concentrations (EPA, 1995). Calibration is performed prior to sample analysis and repeated during sample analysis at intervals specific for each method. In addition to performing the instrument calibrations, the acceptability of these calibrations should be evaluated.

Information related to the QA/QC measures that accompany each batch of samples should be included in each laboratory report. This includes the results obtained from the analysis of calibration and method QC samples which are designed to demonstrate the ongoing control of contamination and to define the precision and accuracy of the method for the parameter and type of sample under investigation. These QC samples should be analyzed concurrently with "environmental samples". Types of QC samples include:

Method Blank — a blank which undergoes processing identical to that carried out for samples (CSA, 1995). Method Blank results are used to assess contamination and/or provide background correction.

Reference Material — a material or substance, one or more properties of which are sufficiently well established for it to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials (AOAC, 1991;

CSA, 1995). Reference material results are used to assess the accuracy and precision of a method. Types of reference materials include:

- Certified Reference Material - a material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to certified or other documentation issued by a certifying body;

- Standard Reference Material - a reference material distributed and certified by the U.S. National Institute for Standards and Technology (NIST), formerly the National Bureau of Standards. SRMs are certified for specific chemical or physical properties and are issued with certificates that report the results of the characterization and indicate the use of the material;

- Other Reference Material - a reference material other than a CRM or SRM, not certified but considered by the laboratory to be useable as a reference material if no suitable CRM or SRM is available. It may come from an external supplier or be prepared in-house.

Method Analyte Spike — sample, clean matrix, or reagents which are fortified with a known quantity of the analyte(s) of interest prior to undergoing sample processing identical to that carried out for samples. Method Analyte Spike results are used to determine effects of the matrix on recovery and losses incurred during sample preparation.

Sample Replicates — two or more independently subsampled portions of the same sample, separately prepared and analyzed by the same method. Sample Replicate results are used to assess the precision of a method.

A method blank, reference material, and sample replicate should be included with each batch of samples as the minimum requirement. Batches should not be larger than 20 samples; EPA (1995) recommends that QC samples be analysed with every 10-20 samples. Many of the GC methods also require that a method analyte spike be added. Information on sources and types of CRMs available for marine sediment (and tissue) is provided in Table 6.

3.2 DATA QUALITY OBJECTIVES (DQOS)

Data Quality Objectives (DQOs) are narrative statements or numerical values used to define performance-based goals for data accuracy, representativeness, comparability, completeness, and sensitivity (i.e., adequate detection limits) for chemical measurements (EPA, 1995). Numerical DQOs can be summarized in a table for convenient reference. An example is provided in Table 7. The following guidance on DQOs is summarized from EPA (1995) and PSEP (1996bc).

Once the project objectives and intended use of the data have been established, then the DQOs should be defined. The laboratory that will perform the analyses should be involved in the project planning process and understand the project requirements for data quality and reporting before samples are collected and received. In particular, this includes ensuring that adequate sample volumes are collected, especially if TBT is a potential COC. Once the DQOs have been defined and documented in a statement of work, they should be adhered to throughout the duration of the project to ensure generation of reliable, defensible data. This involves incorporating QC (Section 3.1) into all aspects of the project, from sample collection, through analysis to data evaluation and reporting.

All measurements should be made so that results are representative of the matrix (e.g., water, sediment, tissue) being measured. For example, results from method blanks should be reported in units that allow direct comparison to concentrations being measured in the samples (e.g., mg/kg for sediments).

DQOs for precision and bias should be based on past experience with a particular measurement system, method validation studies, and the requirements of the specific project. Precision of approximately 30 -50% relative percent difference between measurements (determined from laboratory replicates) and bias of 50 - 150% of the true value (determined from reference materials and spikes) are generally adequate.

Once the analyses are complete, the data should be reviewed for completeness and quality by comparison to the DQOs. This will require knowledge of sample holding times and conditions, the types of analyses requested, and the form in which data were to be delivered by the laboratory.

3.3 GENERAL CONSIDERATIONS

In addition to the QA/QC guidance provided in the previous section, there are a number of general considerations which apply to all the analyses described here. These include prevention of contamination, cleaning procedures, and potential interferences. Guidance pertaining to these issues is provided below (EPA, 1995; PSEP, 1996abc).

Sample Storage and Preservation

Sediment samples should be cooled in the field to about 4°C, and kept cool during shipment to the laboratory. Use of ice chests and artificial ice packs should be sufficient, even for international shipment, provided overnight courier delivery service is used. Tissue samples (i.e., for TBT analysis) should be frozen immediately after collection and kept frozen during shipment. This can be achieved using dry ice, however this will require consideration with respect to international transportation (i.e., airline restrictions on dry ice quantities). No preservatives should be added to sediment or tissue samples.

All samples should be packed securely to avoid breakage, and should be accompanied by chain-of-custody documentation.

Contamination and Low Level Work

Precautions need to be taken to prevent contamination at each stage of sample collection, handling, storage, preparation and analysis. The best way to control contamination is to avoid any exposure by performing operations in areas known to be free from contamination (e.g., a clean room or a clean, nonmetal, laminar flow fumehood). It is also important that dilution water and reagents used in sample preparation and analysis be of appropriately high purity (e.g., deionized water, analytical grade chemicals). All field equipment and labware must be carefully cleaned and cleaning methods monitored and verified using field and laboratory blanks. Laboratories generating trace level data should conduct trace level work on an ongoing basis so that procedures and facilities are proven. The laboratory's QC program should include regular procedural and equipment blanks to update knowledge about background information in the sample processing environment.

Cleaning Methods for Labware

All labware used for sample preparation and analysis must be free from contamination, and ideally it should be dedicated according to sample type and anticipated concentrations of analytes. For metals analyses, all labware should be thoroughly cleaned with a detergent solution, rinsed with metal-free water, and soaked overnight, or longer, in a covered acid bath containing dilute nitric acid prepared from reagent grade nitric acid. The laboratory should have written procedures for labware cleaning methods, and should routinely verify their effectiveness by analysing blanks.

Interferences

Analysis of marine sediment samples is a significant challenge because of the presence of salt, which can interfere with sample analysis. Analytical methods needs to identify the steps taken to control salt interference. For metals, sediment digestates contain high concentration of dissolved solids, from both interstitial seawater salts and salts resulting from sample digestion. For organic analyses, some instrumental methods are more affected by interference than others. Certain target analytes may become interferences (e.g., high concentrations of PCBs can interfere with pesticides in the same sample). Extraction solvents and other common laboratory chemicals can cause interference. Methylene chloride and acetone interfere with volatiles analysis and bis(2-ethylhexyl)phthalate interferes with semivolatile analyses. Another type of interference is specific to the matrix. Marine sediments may contain high concentrations of organic materials that coextract with analytes of interest. Even after extensive cleanup, these interferences may still be present. For example, the presence of sulphur will interfere with the analysis of pesticides and PCBs.

3.4 METALS AND OTHER INORGANIC PARAMETERS

For metals analyses, PSEP (1996b) recommends that any overlying water present on top of the sediment should be stirred back into the sample prior to removing subsamples for digestion and analysis. The sample should also be thoroughly homogenized to ensure that the subsamples are representative. The sediments can be analysed wet, air dried at room temperature, oven dried at 60°C or freeze-dried. Analysis of wet or freeze-dried samples is preferred (PSEP, 1996b) if retaining the original particle size distribution of the sample is important. Concentrations of metals in sediments should be reported on a dry weight basis, to allow data comparability.

There are two methods that can be used for sample digestion for metals analysis. The differences between these methods can have a significant impact on the results obtained for the same sample, so selection of the appropriate digestion method needs to consider the project objectives. Total Acid Digestion (TAD) uses hydrofluoric and other strong acids to completely dissolve the silicate matrix and allow for complete recovery of the elements (PSEP, 1996b). Strong Acid Digestion (SAD) uses nitric and hydrochloric acids and hydrogen peroxide to dissolve nearly all the heavy metals in fine-grained sediments (e.g., cadmium, copper, lead, mercury, silver, and zinc), but not the all the minerals. Recoveries of iron, aluminum, manganese, chromium and nickel are not complete. According to EPA (1995), TAD is not necessary for dredged material evaluations, and digestion with *aqua regia* (nitric and hydrochloric acids) yields acceptable results. PSEP (1996b) have documented several reasons for recommending SAD rather than TAD relating to technical issues (interferences from high dissolved solids requiring dilution and higher detection limits), safety, disposal and cost. ISQVs are based on sediment chemistry data analysed using SAD. For the foregoing reasons, SAD is the method recommended for Hong Kong.

Sample preparation should preferably be carried out using a SAD technique such as EPA Method 3050/3051 which involves a digestion using nitric acid and hydrochloric acid (e.g., 1:1 ratio), along with hotplate or microwave heating. Digestions using peroxide are not recommended as they typically result in an incomplete extraction of the metals from the sediments.

There are a relatively small number of instrumental methods for metals analyses; methodologies developed by EPA (e.g., EPA, 1986) and APHA (e.g., APHA, 1992, 1995) are presented below. These instrumental methods include: flame atomic absorption spectrophotometry (FAAS), inductively coupled argon plasma spectrophotometry (ICP), graphite furnace atomic absorption spectrophotometry (GFAAS), inductively coupled argon plasma mass spectrometry (ICP-MS), cold vapour atomic absorption spectrophotometry (CVAAS) or Cold Vapour Atomic Fluorescence (CVAF). The methods presented in Table 4 (e.g., APHA 3111B [APHA, 1992] and EPA Method 7000/7130 [AAS]; EPA Method 6100 [ICP/OES]; EPA Method 6010 [ICP/AES]; EPA Method 6020 [ICP/MS]) are appropriate for most analyses provided that detection limits

can be achieved and the potential interferences are minimized. The choice of instrumental method will be determined by metals concentrations in the samples and detection limit requirements. CVAAS or CVAF are generally the only options for mercury analysis (e.g., EPA Method 7471). ICP is the most efficient method for measuring many analytes when low detection limits are not required.

For the metalloid, arsenic, the preferred analytical method is usually hydride generation atomic absorption spectrophotometry (HGAAS) using EPA Method 7061 or APHA 3114E (APHA, 1992).

3.5 ORGANIC PARAMETERS

Methods described in this section apply to the ISQV contaminants as well as several other priority COCs identified in Hong Kong sediments. PSEP (1996c) recommends that any overlying water be decanted before the sediment samples are prepared (note this differs from Section 3.4), and that EPA Method 3540 (Soxhlet Extraction) be used for extraction of organic compounds from marine sediments. Note that if TBT tissue analysis is required, any water associated with the tissues should be included as part of the sample.

Organic compounds should be analysed using capillary-column gas chromatography (GC); gas chromatography/mass spectrometry (GC/MS) techniques for semivolatile and volatile priority pollutants, and dual column gas chromatography/electron-capture detection (GC/ECD) or GC/MS with selective ion monitoring for pesticides and PCBs (EPA, 1995). Concentrations of organic parameters in sediments should be reported on a dry weight basis, to allow data comparability. For TBT tissue analyses, concentrations should be reported on a wet weight basis.

3.5.1 PAH Compounds

There are 12 individual PAH compounds which have ISQVs and should be included in the analysis, at a minimum (Table 1), plus an additional four which are also recommended. These compounds can be divided into two categories: low molecular weight PAHs (LPAHs) and high molecular weight PAHs (HPAHs). The LPAHs are acenaphthene, acenaphthylene, anthracene, fluorene, naphthalene and phenanthrene. The HPAHs are benzo(a)anthracene, benzo(a)pyrene, dibenzo(a,h)anthracene, chrysene, fluoranthene, pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, indeno(1,2,3-c,d)pyrene and benzo(g,h,i)perylene.

EPA Method 8270C (GC/MS - capillary column) is the most appropriate technique for PAH analysis. This is also the detection method that EVS (1996d) recommended for the analysis of PAHs in marine tissue samples. Analytes should be extracted from the sediments using dichloromethane with the soxhlet extraction technique (EPA Method 3540) and cleaned up by using silica gel chromatography (EPA Method 3630). This

procedure should effectively remove the aliphatic and heterocyclic hydrocarbons which could potentially interfere with the analyses, and provide the required performance and detection limits.

In addition to reporting results for the individual compounds, the concentrations of LPAH, HPAH and total PAH should also be reported (especially if data will be compared to ISQVs). LPAH is calculated as the sum of the detectable amounts of the LPAHs. HPAH is calculated as the sum of the detectable amounts of the HPAHs. Total PAH is the sum of LPAH and HPAH.

3.5.2 Non-PAH Compounds

In addition to the non-PAH ISQV contaminants (e.g., PCBs), there are a number of classes of organic parameters for which analytical methods are recommended.

Monocyclic Aromatic Hydrocarbons and Chlorinated Hydrocarbons

Volatile Organic Compounds — These compounds should be analysed using EPA Methods 5030 and 8240. This involves a purge and trap extraction, followed by GC/MS-capillary column for separation and detection.

Chlorinated Benzenes — These compounds are analysed in the same manner as PAHs; they are extracted with the base-neutral fraction.

Congener PCBs — These compounds should be analysed using EPA Methods 3540 and 8082/8070. This analysis involves soxhlet extraction by using hexane/acetone, sample clean up, and GC/ECD and MS - capillary column separation and detection. EPA (1995) recommends GC/ECD methods for PCB analysis.

EPA (1995) recommends that all PCB analyses be made using congener-specific methods rather than the historical Aroclor methods. The concentrations of specific congeners are an appropriate measure of total PCBs. Congener-specific analyses also provide data that can be used for specialized risk assessments that reflect the widely varying toxicity of different PCB congeners.

Dioxins and Furans — Dioxins and furans can be analysed according to methods described in Environment Canada (1992a,b). EPA (1995) also recommends a number of methods for analysis of these compounds, including EPA Method 1613 which was developed for analysis of water, soil, sediment, sludge and tissue.

Phenolic Compounds

Chlorinated and Non-Chlorinated Phenolics — These compounds should be analysed using EPA Methods 3540, 8270 and 8040 (PSEP, 1996c). This analysis involves

soxhlet extraction of the sample with dichloromethane, solvent exchange to hexane and derivatization, and GC/MS-capillary column and GC/nitrogen phosphorus (NP) for separation and detection.

Pesticides

Organochlorine Pesticides — These compounds, which include various forms of DDT, DDD and DDE, should be analysed using EPA Methods 3540, 3610 and 8081(EPA, 1995; PSEP, 1996c). This analysis involves soxhlet extraction with dichloromethane, solvent exchange to hexane and alumina column clean-up, and GC/ECD separation and detection. The use of GC/MS (EPA Method 8070C) is also possible, but may not provide the same sensitivity.

Organophosphate Pesticides — These compounds should be analysed using EPA Methods 3540 and 8140. This analysis involves soxhlet extraction and GC-capillary with thermionic detection.

Carbamates — These compounds can be analysed in the same manner as PAHs; they are extracted with the base-neutral fraction.

Herbicides — Compounds such as 2,4-D can be analysed using EPA Method 8151A. This procedure involves extraction with acidified acetone and diethyl ether and analysis by capillary column GC/MS.

Organometallic and Miscellaneous Organic Parameters

Tributyltin — Analysis of TBT is a special case under the TTS for evaluation of dredged material (EVS, 1996a). While analysis of all other parameters is performed using whole sediment, TBT analysis needs to be performed on the interstitial water (IW) fraction as part of the chemical screening. It is important that the sediment volume collected be large enough to allow collection of enough IW to meet analytical detection limit requirements. The recommended method for IW collection is to centrifuge the sediment and then collect the resulting overlying water. Depending on moisture content, a 200-mL sediment volume may yield 10 - 50 mL of IW. If Tier III biological screening is also required for TBT, then TBT concentrations in tissues will also need to be measured at the end of the bioaccumulation study (see Section 4.6). PSEP (1996a) provides guidance on methods for analysis of TBT in marine water, sediment and tissue. Methods by Krone et al., (1989) are recommended and the analysis should be performed by GC/MS.

Phthalates — These compounds can be analysed in the same manner as PAHs; they are extracted with the base-neutral fraction.

3.6 CONVENTIONALS AND OTHER PARAMETERS

There are several conventional parameters which should always be included in sediment chemical analyses, as they can provide important information regarding the potential impacts of these sediments on the aquatic environment. These include measurement of moisture content, grain size and total organic carbon (TOC). In addition, EVS (1996a) recommends that the samples also be analysed for acid volatile sulphide (AVS) and simultaneously extracted metals (SEM). Although guidance interpreting AVS and SEM measurements is still being developed, these measurements can provide useful information on the bioavailability of metals in sediments.

Moisture Content — The moisture content of each sample is needed so that chemistry results can be reported on a dry weight basis. This is determined gravimetrically by drying a portion of the sample at 105°C to constant weight.

Grain Size — The grain size distribution of the sediment samples is important because contaminants tend to be more closely associated with fine-grained material (e.g., silt and clay) than with course sand and gravel. Grain size can be measured by drying a portion of sample and then using standard sieves to determine the gravel, sand and silt fractions, and a pipette method for the clay fraction.

Total Organic Carbon (TOC) — TOC should always be measured, especially if hydrophobic organic compounds are being analyzed (EPA, 1995). The TOC content is a measure of the total amount of oxidizable organic material in a sample and also affects contaminant bioaccumulation by, and effects to, organisms. TOC analyses should be based on high-temperature combustion rather than on chemical oxidation, because some classes of organic compounds are not fully degraded by chemical/ultraviolet techniques.

Acid Volatile Sulphide/Simultaneous Extracted Metals (AVS/SEM) — These compounds can be analysed according to Battelle (1990). Analysis for AVS involves adding HCl to the sediment, purging hydrogen sulphide (H_2S) from the sample with nitrogen, trapping H_2S in a solution of zinc acetate solution, and then analyzing the solution colourimetrically for sulphide. Analysis for SEM involves adding HCl to the sediment, purging hydrogen sulfide (H_2S) from the sample with nitrogen, and analyzing the extract for simultaneous extracted metals (SEM).

3.7 REPORTING REQUIREMENTS

The information provided in an analytical laboratory report should be sufficiently complete to allow an independent evaluation of the quality of the data, in addition to reporting the test values. Deliverable requirements and deadlines should be established prior to beginning the study. Guidance on the type of information to include in laboratory

reports is provided in various methodology documents (e.g., EPA, 1995; PSEP, 1996abc). These references recommend that laboratory reports should include the following:

- name and location of the testing laboratory, and investigator(s)
- source of samples, method of collection, handling, shipping and storage, dates and times of sample collection and receipt at the testing laboratory
- dates of extraction and analysis
- summary of extraction or digestion procedures and analytical methods
- detection limits and quantification limits
- sample identification codes (if the lab has its own identification system)
- explanation of all data qualifier symbols
- tabulated sample results with units, including reporting basis (e.g., wet, dry, TOC normalized)
- summary of results and control limits for all QC analyses, such as blanks, spikes, surrogates, duplicates and CRMs
- explanations for all data quantifications
- tentatively identified compounds (if requested) and methods of quantification
- explanations for all departures from the analytical protocols and discussion of possible effects on the data
- reference methods
- copies of completed chain-of-custody records and sample analysis request forms.

In addition to the information provided in the laboratory report, records of supporting backup should be maintained on file by the laboratory, including: calibration results; method blanks; sample sizes and dilution factors; replicates and spikes; amounts spiked; control or reference samples; chromatograms; GC/MS tuning documentation; GC/MS supporting spectra; chain of custody and sampling records; and any anomalies in instrument performance or unusual instrument adjustments.

4.0
RECOMMENDED METHODS - SEDIMENT BIOASSAY TESTING

Biological screening (Tier III of the TTS) involves performing lethal and sublethal toxicity tests by exposing standard numbers of individual organisms of one or more benthic species to samples of marine sediments. Note that there is one exception to this; when TBT is identified as requiring biological screening then the recommended procedure is bioaccumulation testing. At present, such sediment testing procedures have not been developed for regulatory use with marine species native to Hong Kong (Section 2.2). Practically speaking, commercial toxicity testing capabilities are not yet available in Hong Kong. For regulatory objectives to be met with some confidence, toxicity tests need to be conducted using established "benchmark" test species of known sensitivity, for which standard test procedures have been developed and effects on the test organisms documented for a wide range of contaminants.

Protocols or guidance documents for conducting such tests have been published by the following agencies:

- American Society for Testing and Materials (ASTM)
- Environment Canada
- Puget Sound Estuary Program (PSEP)
- U.S. Army Corps of Engineers (USACE)
- U.S. Environmental Protection Agency (EPA).

ASTM publishes guidance documents for various toxicity tests. These documents outline a series of options or instructions to offer guidance, based on a consensus of viewpoints, rather than recommending specific courses of action by an established fixed procedure. Guidelines are intended to increase the awareness of the user to the available techniques and to provide information from which subsequent evaluation and standardization can be derived. Environment Canada publishes biological test method documents which describe recommended methods for guidance to facilitate the use of consistent, appropriate, and comprehensive procedures for obtaining data on the toxic effects of samples (Environment Canada, 1992c). These methods also provide a foundation for very explicit instructions (as might be required in a regulatory protocol or standard reference method). PSEP (1995) developed testing guidelines to encourage investigators to use standardized methods whenever possible, so that data from different studies would be directly comparable and could be integrated into a database for Puget Sound. EPA has published protocols for sediment toxicity tests which are used for regulatory testing (e.g., EPA, 1994). EPA and USACE have developed tiered testing systems for evaluating dredged material for disposal (e.g., EPA/USACE, 1991, 1994). These documents provide recommendations for test species and methodology selection and overviews of the test methods. However, they do not include detailed instructions for conducting the various bioassays.

4.1 RECOMMENDED INTERIM BIOASSAY METHODS AND PROCEDURES

Recommendations for selecting appropriate test protocols, as well as guidance on conducting the tests and evaluating the results, are described below. The following sediment bioassays are recommended for interim use until region-specific tests are developed and implemented (EVS, 1996a; Section 2.5):

Lethal Effects

- 10-d amphipod sediment test (using *Eohaustorius estuarius*)

Sublethal Effects

- 20-d juvenile polychaete sediment test (using *Neanthes arenaceodentata*)
- 48-h larval development sediment test (using either bivalve [*Crassostrea gigas* or *Mytilus edulis]* or echinoderm [*Strongylocentrotus* sp. or *Dendraster excentricus*] species)

Bioaccumulation (special case for TBT only)

- 28-d bivalve bioaccumulation study (using *Macoma nasuta*)

As with the methods described in Section 3.0, it is important that laboratories conducting these tests comply with all environmental laws and safety requirements (e.g., sample disposal) relevant to their jurisdiction.

4.2 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

Implementation of a clearly defined QA/QC program is an integral part of conducting laboratory toxicity tests. A thorough and effective QA Program is the principal means of maintaining the accuracy and precision of field and laboratory analyses to assure scientific credibility. It ensures complete documentation and also standardizes and minimizes possible errors in computation and reporting of results. It is important that standard laboratory procedures be followed for all testing and that any unusual observations or deviations from established procedures be documented. The following general QA/QC guidelines (PSEP, 1995) apply to all sediment toxicity tests.

Negative Controls — All tests must be conducted using well-established negative (clean) controls. For every toxicity test, one series of test chambers must only contain clean diluent water (or clean diluent water and clean sediment). The complete test series

must be repeated if the mean control response does not meet the acceptability criteria for that test method.

Positive Controls (Reference Toxicants) — All toxicity tests must contain positive (toxic) controls which are conducted with well-established standard reference toxicants. Reference toxicants are used to provide insight into mortalities or changes in sensitivity that may occur as a result of acclimation, disease, loading density or handling stress. For organisms obtained from outside sources (either purchased or field-collected), a reference toxicant test must be run for each new batch of organisms obtained. For organisms obtained from in-house laboratory cultures, reference toxicant tests can be performed on a monthly basis. Control charts should be constructed for each species and reference toxicant used and the cumulative mean value and upper and lower control limits (± 2SD) should be plotted on each chart. If the results of a reference toxicant test fall outside the control chart limits, the test procedures and health/source of the test organisms should be reviewed; subject to those findings, the test may have to be repeated.

Reference Samples — Reference samples are usually required for sediment toxicity tests and are used to separate toxicant effects from unrelated effects such as sediment grain size. Reference sediments should be collected from an area documented to be free of chemical contamination and should represent the range of important physical characteristics (e.g., grain size, TOC) found in the test sediments.

Test Organisms — Only healthy organisms of similar size and life history stage are used for toxicity tests. Taxonomic identifications should be confirmed by the qualified taxonomist. All test organisms used for a batch of tests must be from the same source. Records of collection, shipping and acclimation should be maintained for all species obtained from outside of the laboratory.

Blind Testing — All treatment containers should be randomized during testing, and samples should be coded so that laboratory personnel do not know the sample identities.

Replication — The number of replicates required varies from one test protocol to another, but should always be sufficient to account for variability in test organism response. Unless otherwise specified in the experimental design, each treatment must begin with the same number of replicates.

Instrument Calibration — Calibration of instruments is required to ensure that accurate measurements are made throughout a test and to ensure the equipment is operating correctly. Water quality instruments (dissolved oxygen, pH and conductivity meters, refractometers) must be calibrated at the start of each day (and any time the environmental conditions are changed), according to the manufacturer's instructions. Each piece of equipment should have a logbook for daily recording of calibration information, repairs, replacement, etc.

Water Quality Measurement/Maintenance — Toxicity tests involving exposure of organisms in aqueous media require that the media be uncontaminated and that proper water quality conditions be maintained to ensure the survival of the organisms, and to ensure that undue stress is not exerted on the organisms, unrelated to the test materials. Appropriate water quality parameters must be measured at the start and end of a test as a minimum (every 24 h is more appropriate). If acceptable limits are exceeded at any time, the data should be reviewed to determine whether the test should continue.

Standard Laboratory Procedures — Standard laboratory procedures must be followed in all testing. These include use of established methods, proper documentation, proper cleaning, avoidance of contamination and maintenance of appropriate test conditions. All unusual observations or deviations from established procedures must be documented.

4.3 DATA QUALITY OBJECTIVES (DQOS)

Data quality objectives (DQOs) for toxicity tests serve a similar function to the DQOs for chemical analyses (Section 3.2). DQOs are narrative statements or numerical values used to define performance-based goals for data accuracy, representativeness, comparability, completeness, and sensitivity. The laboratory that will perform the toxicity tests should be involved in defining the DQOs as part of development of the study plan. Example DQOs for sediment toxicity tests are provided in Table 8. When testing is complete, the data should be reviewed to ensure that they meed the DQOs.

4.4 GENERAL CONSIDERATIONS

In addition to the QA/QC guidelines described in the previous section, there are a number of general procedures which apply to all the bioassays described here. These include sample collection, sample storage and holding time, cleaning procedures, and preparation of bioassay seawater. Guidance pertaining to these procedures is provided below.

Sample Storage and Holding Time

Samples should be cooled in the field to about 4°C. It is important that samples also be kept cool (but never frozen) during shipment to the laboratory, and that they be packed securely to avoid breakage. All shipments should be accompanied by chain-of-custody documentation. Fresh, whole sediments should be stored at 4°C in the dark after field collection. They must not be frozen and no preservatives need to be added. At present, the effects of storage on sediment toxicity have not been thoroughly evaluated (PSEP, 1995; EPA, 1993). The holding time between collection and test initiation should be as short as possible (preferably no longer than two weeks). If a tiered testing approach is

being used (where chemical analyses are conducted before the toxicity tests), then storing sediments for a maximum of either six weeks (Environment Canada, 1994) or eight weeks is considered acceptable (PSDDA, 1989; EPA/USACE, 1994). If samples are to be stored longer than two weeks, they should be stored under a nitrogen atmosphere.

Cleaning Procedures

Prevention of contamination of samples is critical to the successful performance of any laboratory analysis. It is important that all equipment that might come in contact with the sediment samples or test organisms be cleaned properly prior to use. In general, this involves washing with detergent, following by rinses with acetone and dilute acid (10% nitric or hydrochloric acid) followed by repeated rinsing with distilled or deionized water.

Bioassay Seawater

Clean seawater from an uncontaminated source is required for use as the overlying water in the toxicity tests, and for use in the reference toxicant tests. This water can be either natural or reconstituted (use of natural seawater is generally preferred). Seawater should not be collected from areas where algal blooms have occurred. The seawater should be filtered (0.45-μm pore size), aerated to ensure that the dissolved oxygen content is 90 - 100% saturation, and used within two days of collection. Depending on the species being tested, it may be necessary to adjust the salinity, by addition of either distilled water or hypersaline brine.

Water Quality Measurements

The type and frequency of water quality measurements required during sediment bioassays varies depending upon test protocols and study objectives. Temperature must be measured at least once a day. The other parameters that should be measured are salinity, pH and dissolved oxygen. Some protocols specify that these should be measured in every replicate but only at the start and end of the test, while others specify daily measurement but only in one or two replicates per treatment. It is important that water quality parameters be monitored frequently enough to be able to document exposure conditions. Two other parameters which have received increased attention in recent years are ammonia and sulfide. Measurement of these parameters at a minimum in the overlying water at the start of a test is recommended. If ammonia is suspected to be the cause of toxicity, it may be desirable to measure it more frequently or to modify the experimental design to allow for removal of interstitial ammonia prior to test initiation (EPA, 1994). It is important to consider what measurements to perform as part of the experimental design.

4.5 SEDIMENT TOXICITY TESTS

4.5.1 Amphipod Toxicity Test (Lethal Effects)

Sediment toxicity tests with marine amphipods were first developed in the early 1980s (e.g., Swartz et al., 1985), and are now considered one of the most important "benchmark" species in sediment toxicity testing programs. Procedures for conducting this 10-d static acute toxicity test with the amphipod, *Eohaustorius estuarius*, were developed by DeWitt et al. (1989). *E. estuarius* is an effective test species because it is sensitive to a wide range of chemicals, but is relatively insensitive to non-toxic parameters, such as grain size. It is able to tolerate salinities ranging from 2 - 28 ppt, and can be used for both marine and estuarine sediment samples. Published documents describing this test (ASTM, 1995a; Environment Canada (1992c); PSEP (1995), are very similar in their requirements. For testing in Hong Kong, the PSEP (1995) protocol is recommended.

Recommended test conditions are summarized in Table 9. The test containers are 1-L glass jars, each containing a 2-cm layer of sediment and 800 mL of filtered seawater. Depending on the interstitial salinity of test sediments, the salinity of the overlying water may need to be adjusted to match it. The negative control consists of sediment from the amphipod collection site and filtered seawater. There are five replicates per treatment, each containing 20 amphipods. A sixth replicate is used for daily monitoring of water quality parameters (temperature, pH, dissolved oxygen, salinity). The test is conducted at 15 ± 1 °C, under continuous illumination, with gentle aeration provided. Test duration is 10 days. Sediments are prepared the day before test initiation and allowed to settle overnight, and amphipods are added the following day (Day 0). Water quality and emergence are measured daily. The number of living, dead and missing amphipods is determined after 10 days' exposure for each replicate. Response criteria include mortality, emergence from sediment and ability to rebury in clean sediment after a 10-d exposure.

For the 10-d amphipod toxicity test to be considered acceptable, mean survival in the negative control must be ≥90%, and survival in each control replicate must be ≥80%. Water quality parameters should be within the recommended test conditions.

4.5.2 Polychaete Worm Toxicity Test (Sublethal Effects)

The 20-d static-renewal growth test with the juvenile polychaete, *Neanthes arenaceodentata*, was developed for use in Puget Sound (Washington State) because of a regulatory requirement for assessment of sublethal effects (Johns et al., 1990). Methods for conducting this test have been published by ASTM (1995b) and PSEP (1995). For testing in Hong Kong, use of the PSEP (1995) protocol is recommended.

Recommended test conditions are summarized in Table 10. The test uses juvenile (2-3 weeks post-emergence) *Neanthes*. Test containers are 1-L glass jars, each containing a

2-cm layer of sediment and 800 mL of filtered seawater. Sediments are prepared the day before test initiation and allowed to settle overnight, and worms are added the following day (Day 0). The negative control consists of sediment from an uncontaminated site (such as an amphipod collection site) and filtered seawater. There are five replicates per treatment, each containing five worms. The test is conducted at 20 ± 1°C, under continuous light. Approximately one-third of the overlying water in each test container is replaced every third day, and water quality parameters are measured at that time. Worms are fed ground TetraMarin (40 mg/jar) every second day. On Day 20, worms are sieved from the sediments and the number of living, dead and missing worms is recorded for each replicate. Surviving worms are rinsed and dried overnight. The average dry weight obtained for each treatment is compared to the initial dry weight measured on Day 0, to determine the increase in dry weight.

For the test to be considered valid, mean survival in the negative control must be ≥90%. Water quality parameters should be maintained within acceptable limits.

4.5.3 Larval Development Toxicity Test (Sublethal Effects)

The larval development toxicity test is used to provide information on the toxicity of marine and estuarine sediments to the embryos and larvae of either bivalves or echinoderms. Test species include Pacific oysters (*Crassostrea gigas*), the blue mussel (*Mytilus edulis*), purple sea urchins (*Strongylocentrotus purpuratus*) and the sand dollar (*Dendraster excentricus*). Using bivalve larvae to test sediment toxicity was first developed by Chapman and Morgan (1983), using a modification of procedures described in ASTM (1995c). Methods for testing bivalves or echinoderms are described in PSEP (1995).

Recommended test conditions are summarized in Tables 11 and 12. Mature adult bivalves are stimulated to spawn by thermal and biological stimulation. Eggs and sperm are examined under a microscope to check their viability, and combined to allow fertilization. While spawning and fertilization are in progress, sediment treatments are prepared. The test containers are clean 1-L polyethylene jars. Five replicates are prepared for each treatment, each containing 18 g of sediment and 900 mL of clean seawater. Sediments are shaken for 10 sec and then allowed to settle for 4 h before inoculation with the newly fertilized embryos. The negative control consists of five replicates of clean seawater only. Within 4 h of embryo fertilization, each container is inoculated with a sufficient number of embryos to provide a density of 20 - 40 embryos/mL. Subsamples are collected from "extra" control replicates immediately after inoculation to confirm the initial embryo density; this value is used for calculating larval mortality at 48 h. The test containers are allowed to incubate at test temperature (20 ± 1°C for oysters; 16 ± 1°C for mussels) for 48 ± 4 h. After 48 h, the water in each test container is carefully decanted and then mixed to resuspend the larvae. Multiple 10-mL aliquots of solution are removed by pipette, transferred to vials, and preserved with 5% buffered formalin. The larvae are examined under a compound microscope to determine the number of normal and

abnormal larvae in each sample. Larvae which fail to transform to the fully-shelled, D-shaped Prodissoconch I (for bivalves) or echinopluteus (for echinoderms) stage are considered abnormal. The test endpoints are based on larval development and survival.

For the test to be considered valid, mean normal development in the negative control must be ≥90%, and mean survival must be ≥70%. Water quality parameters should also have been maintained within acceptable limits.

4.6 SEDIMENT BIOACCUMULATION

Under the proposed TTS for evaluation of dredged material, if interstitial water concentrations of TBT exceed the ISQV-low, then the recommended biological screening tool is a laboratory bioaccumulation study, rather than the sediment toxicity tests described in Section 4.5. In Tier III of the proposed TTS, this is the only occasion where bioaccumulation studies apply. Note that if concentrations of other contaminants are high enough, then toxicity tests would also be required. The recommended method is a 28-d bioaccumulation test with the clam (*Macoma nasuta*), using procedures described in EPA (1993) and EPA/USACE (1991, 1994).

Recommended test conditions are summarized in Table 13. The bioaccumulation tests should be conducted using a flow-through seawater system, which allows the container volume to be replaced at least once every 4 h. Test sediments, reference and control sediments should first be sieved to remove any animals that might be present. Each of the test containers is prepared by placing a 30-40 mm layer of sediment in the bottom and then filling the container with clean seawater. There are five replicates for each treatment. The following day (Day 0), the clams are hand sorted and damaged, dead or unhealthy animals are discarded. Care is taken to ensure that the largest animal used is no more than twice the length of the smallest animal. Each replicate is seeded with the same number of clams; typically 20 or 25 animals are used to provide eough tissue for analysis. At the same time, an additional subsample of clams are rinsed and placed in clean seawater for 24 h to void their digestive tracts, and then frozen for analysis of background chemical levels.

The test duration is 28 d. Water chemistry parameters (pH, temperature, dissolved oxygen, salinity) and observations of mortality and burrowing behaviour should be recorded daily. Dead organisms are removed daily. After 28 d, the clams are sieved from the sediments and the number of survivors is recorded. Surviving animals from each replicate are rinsed in clean seawater to remove any sediment, placed in separate trays containing clean, flowing seawater (no sediment) to void their digestive tracts for 24 h, rinsed again in clean seawater and either dissected from their shells immediately and then frozen, or frozen immediately and then subsequently dissected from their shells (while still frozen) just prior to analysis. Animals from each replicate are pooled together to provide sufficient tissue for chemical analysis.

Criteria for test acceptability for a bioaccumulation study differ from those for toxicity tests. There is no specific requirement for control survival, just that there be enough tissue available for the required chemical analyses to be performed.

4.7 DATA ANALYSIS

Decision criteria for evaluating Tier III biological screening results have not yet been established, however some general guidance on data analysis is provided. For sediment toxicity tests, similar data analysis methods can be used for each test. For each endpoint, the mean and standard deviation should be calculated for each treatment. Statistical comparisons should be made by performing pair-wise comparisons (e.g., two-sample *t*-tests) to compare each treatment with its respective negative control and reference sediment. For TBT, the mean tissue concentration measured for each treatment will be compared to a tissue screening level (EVS, 1996a).

4.8 REPORTING REQUIREMENTS

The information provided in a toxicity testing report should be sufficiently complete to allow independent evaluation of the quality of the data and interpretation of the results. Deliverable requirements and deadlines should be established prior to beginning the study. The protocol documents cited for these test methods (e.g., ASTM, 1995a,b,c; Environment Canada, 1992c; PSEP, 1995; EPA, 1994) each provide guidance about records that should be kept and details to be included in test reports. Although this guidance differs in some details of required content from document to document, they all require that the following information should be included in the report:

- Type of test, test species, start and end dates of the test.
- Name and location of the testing laboratory, and investigator(s).
- Source of control or test sediment, method of collection, handling, shipping and storage, dates and times of sample collection and receipt at the testing facility.
- Source and characteristics of control seawater, including any pretreatment.
- Source, history and age of test organisms, including: date and location of collection (or purchase), taxonomic identification, age or life-stage used, size, holding/acclimation procedures, and any unusual observations.

- Source and composition of food (if applicable), procedure used to prepare food, feeding methods, frequency and ration.

- Description of the experimental design, test chambers, depth and volume of sediment and overlying water in the chambers, lighting and temperature control, numbers of replicates and test organisms, water quality measurements.

- Description of the procedures used to prepare the test treatments, and to introduce the test organisms into the test chambers.

- Methods used for physical and chemical characterization of the sediments (if applicable).

- Definition of the effects used to determine the biological endpoint(s) of the test, and a description of general observations of other effects.

- A table of the biological data for each replicate in each treatment, including the negative control, in sufficient detail to allow independent statistical analysis.

- Water quality measurements during testing (i.e., dissolved oxygen, temperature, salinity, pH, and possibly ammonia and sulfide).

- Interstitial water salinity for control, reference, and test sediments.

- Methods used for statistical analysis of the data.

- Criteria for test acceptability.

- Results for the concurrent reference toxicant test, with a comparison to the laboratory's control chart (mean ± 2SD) for that species-method-reference toxicant combination (results for metallic compounds should be reported in terms of the metal ion rather than as the weight of the whole metal salt).

- Summary of general observations, include anything unusual about the test, any deviation from standard procedures, and any other relevant information which may have affected data quality.

In addition to the information provided in the laboratory report, supporting backup records should also be maintained on file by the laboratory, including: organism culture and receipt records; original data sheets and testing logbooks; reagent preparation records; instrument calibration records and documentation of any anomalies with the tests or equipment.

5.0
OTHER BIOLOGICAL ASSESSMENT TECHNIQUES

Tier IV of the proposed TTS provides for further site-specific or situation-specific biological assessment to determine disposal requirements for dredged material. Tier IV is intended as an option which may be implemented if the findings from Tier II and III investigations indicate that the material is not suitable for any form of marine disposal. Depending on the findings from Tier IV, the dredged material may be designated as being suitable for confined marine disposal.

The biological assessment techniques which can be used in Tier IV will need to selected with specific objectives in mind and will likely differ from study to study. As such, provision of detailed method descriptions and recommendations is not appropriate at this time. Suggested techniques include, but are not limited to, the following: additional sediment bioassays, either *in situ*, using different species or endpoints, or of longer duration; bioaccumulation tests to assess steady state using field exposures; ecological risk assessment; or benthic community analysis. Additional information is provided in EVS (1996a).

6.0
CONCLUSIONS

Based on the information available for preparation of this laboratory guidance document, we present the following conclusions.

1. Environmental laboratories in Hong Kong do not currently have capabilities to support the complete analytical chemistry requirements associated with all the ISQV contaminants (EVS, 1996a). Current analytical capabilities may be summarized as follows:

   - trace metal analyses - adequate performance, technology, and methods.
   - trace organic analyses - inadequate performance, adequate technology, although some limited experience with acceptable methods.

   These capabilities provide a foundation that may be enhanced to provide the performance necessary to support analysis for priority contaminants in Hong Kong sediments.

2. Enhancement of the capabilities of environmental analytical laboratories with respect to analytical parameters and detection limits needs to occur during a reasonable period of time. Laboratories will need time to implement recommended analytical protocols for the ISQV contaminants (Section 3.0) and demonstrate their proficiency with each sample preparation and determinative method combination, by generating data of acceptable accuracy and precision for target analytes at the required detection limits. Given that the laboratories already have some degree of experience with analysis of most of the parameters, a period of six months should be reasonable for upgrading these capabilities. This is consistent with the amount of time generally allotted for achieving HOKLAS accreditation.

3. The commercial scale capabilities in Hong Kong related to the testing of sediments for toxicity do not exist in either the government or private laboratories.

4. The lack of facilities and current expertise related to toxicity testing indicates that, in the short term, Hong Kong will have to rely on laboratories outside Hong Kong to provide this information.

5. The motivation for the environmental analytical laboratories in Hong Kong to improve their analytical capabilities will be driven by market and regulatory requirements.

7.0
RECOMMENDATIONS

Based on the information available for preparation of this report, we propose the following recommendations:

1. Analyses should only be performed by laboratories which are accredited by HOKLAS or by a comparable accreditation program applicable to their jurisdiction (i.e., for laboratories outside Hong Kong). Confirmation of the requirements of such an accreditation program may need to be provided.

2. The ability of the laboratories to generate reliable, technically defensible data should be monitored by an agency such as HOKLAS.

3. Laboratories should be informed of the required performance capabilities (i.e., meeting detection limits for ISQV contaminants) before they are implemented so that they have a chance to respond. A program for the laboratories to demonstrate their proficiency with the new analytical requirements should also be implemented, perhaps as part of HOKLAS accreditation. Each HOKLAS test certificate should be accompanied by a detection limit and method outline describing the acceptable analytical technique, and the results from QA/QC samples should be included in each laboratory report.

4. As sediment testing procedures have not currently been developed for marine species native to Hong Kong, tests using internationally established species for which methods have been standardized for a wide range of contaminants are recommended.

5. Technical assistance with the development of biological testing capabilities is recommended to ensure that meaningful data can be obtained.

8.0
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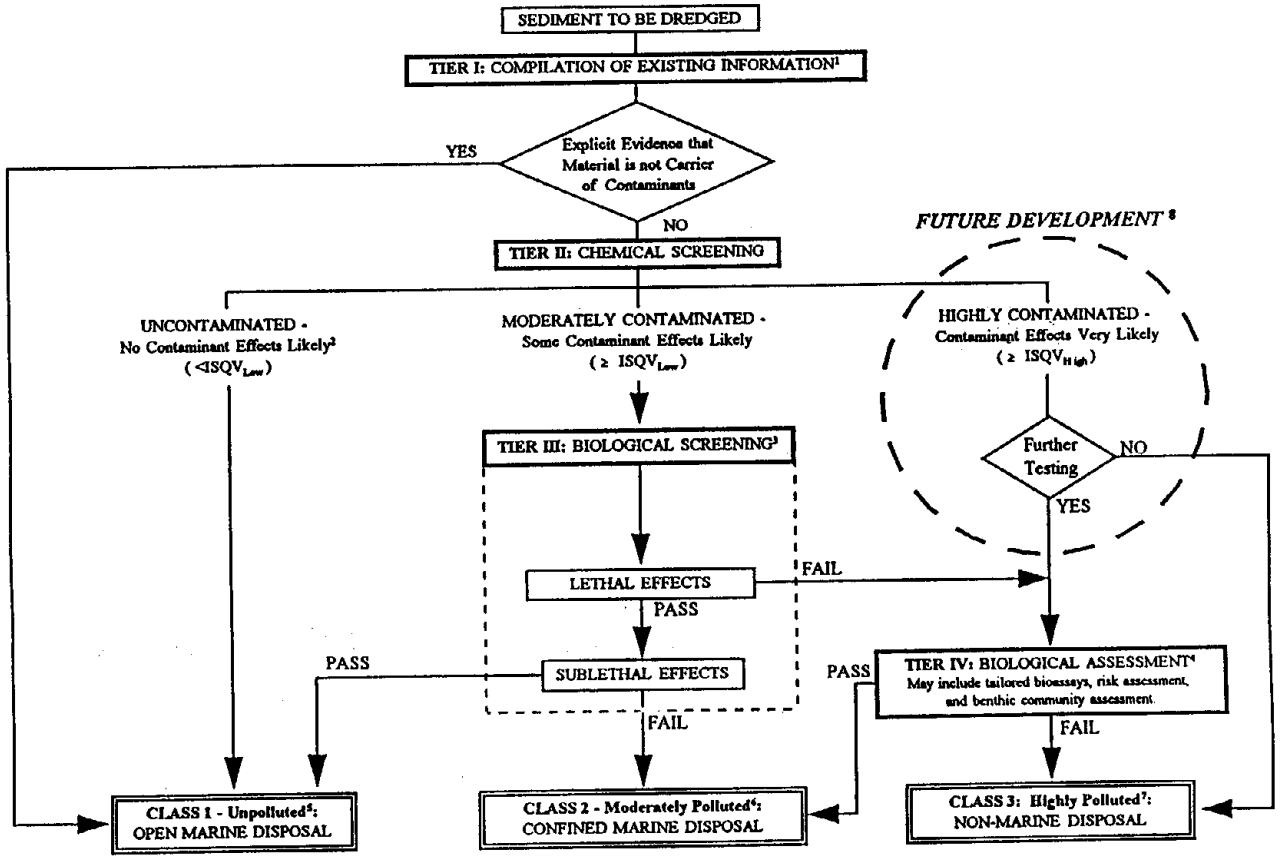
Swartz, R.C., W.A. DeBen, J.K. Phillips, J.O. Lamberson and F.A. Cole. 1985. Phoxocephalid amphipod bioassay for marine sediment toxicity. R.D. Cardwell, R. Purdy and R.C. Bahner (eds). In: Aquatic Toxicology and Hazard Assessment: Proceedings of the Seventh Annual Symposium ASTM STP 854, American Society for Testing and Materials, Philadelphia, PA.

Wu, R. 1996. Personal Communication (conversation with C. McPherson, EVS Environment Consultants, North Vancouver, BC.) July 1996. Professor, City Polytechnic University Hong Kong.

FIGURES

Figure 1. Tiered Testing System (TTS) to be applied for management of dredged material (EVS, 1996a).



1 Although not shown, in exceptional cases, where there is reason to believe that the material is polluted, the option exists to waive chemical testing and proceed directly to biological screening.
2 Although not shown and not expected to be routinely implemented, the option (regulator's option) exists for conducting confirmatory biological screening.
3 Generic bioassays include lethal and sublethal sediment toxicity tests; bioaccumulation tests are used as a special case for TBT (see text).
4 At a minimum, lethal and sublethal effects and bioaccumulation tests. Extent and type of assessment to be determined in consultation with EPD.
5 Unpolluted: uncontaminated, and/or no biological effects related to contaminants.
6 Moderately Polluted: moderately contaminated, and/or limited biological effects related to contaminants.
7 Highly Polluted: highly contaminated, and/or unacceptable biological effects related to contaminants.
8 Pending adequate effects data to establish ISQV$_{High}$ for Hong Kong.

TABLES

Table 1. List of priority COCs for which ISQVs have been developed.

CONTAMINANT
METALS
Cadmium (Cd)
Chromium (Cr)
Copper (Cu)
Mercury (Hg)
Nickel (Ni)
Lead (Pb)
Silver (Ag)
Zinc (Zn)
METALLOIDS
Arsenic (As)
ORGANICS-PAHs
Acenaphthene
Acenaphthylene
Anthracene
Fluorene
Naphthalene
Phenanthrene
Low Molecular Weight PAHs
Benzo[a]anthracene
Benzo[a]pyrene
Chrysene
Dibenzo[a,h]anthracene
Fluoranthene
Pyrene
High Molecular Weight PAHs
Total PAHs
ORGANICS-non-PAHs
Total PCBs
Total DDT
p,p'-DDE (4,4'-DDE)
ORGANOMETALLICS
Tributyltin (TBT)

Table 2. List of contacts providing information regarding testing capabilities.

CONTACT	POSITION	AFFILIATION	LOCATION
Dr. L.H. Ng	Executive Administrator	HOKLAS	Hong Kong
Mr. K.L. Tsang	Technical Services Manager	Hong Kong Productivity Council	Hong Kong
Mr. S.T. Chan	Assistant Government Chemist	Hong Kong Government Laboratory	Hong Kong
Mr. H.W. Yeung	Regional Vice President	Inchape Testing Services Hong Kong Limited	Hong Kong
Mr. P. Hamilton	General Manager	MateriaLab Limited Hong Kong	Hong Kong
Mr. C.R. Stace	General Manager	ALS Technichem (HK) Pty Ltd.	Hong Kong
Dr. R. Wu	Professor	City Polytechnic University Hong Kong	Hong Kong
Mr. S.V. Jones	Project Director	Mouchel Asia Ltd.	Hong Kong
Mr. A. Maynard	Partner	ASL Analytical Services Laboratory Ltd.	Vancouver, Canada
Mr. M. Hugdahl	Manager Organics	ASL Analytical Services Laboratory Ltd.	Vancouver, Canada
Mr. R. Jornitz	Manager Inorganics	CanTest Laboratories Ltd.	Vancouver, Canada
Dr. C. Hamilton	President	AXYS Analytical Services	Victoria, Canada
Mr. J. Hicks	Marketing Manager	Analytical Resources Inc.	Seattle, USA

Table 3. Comparison of laboratory detection limits for ISQV contaminants.

CONTAMINANT	ISQV-LOW	GOVERNMENT HOKLAS No. 1 (HONG KONG)	INCHCAPE HOKLAS No. 5 (HONG KONG)	MATERIALAB HOKLAS No. 15 (HONG KONG)	ALS (HK) HOKLAS No. 66 (HONG KONG)	ASL (CANADA)	ARI (USA)
METALS (mg/kg dry wt.)							
Cadmium	1.5	0.05	1	0.2	0.01	0.1	0.02
Chromium	80	0.1	1	1	0.05	2	0.5
Copper	65	0.1	1	1	0.05	1	0.2
Mercury	0.15	-[1]	0.1	0.05	0.02	0.005	0.005
Nickel	40	0.1	1	1	0.05	2	1.0
Lead	75	0.1	1	1	0.05	50	2.0
Silver	1.0	0.1	1	1	0.05	0.1	0.3
Zinc	200	1	1	1	0.05	1	0.4
METALLOIDS (mg/kg dry wt.)							
Arsenic	8.2	0.1	0.1	0.1	-	0.05	0.1
ORGANICS - PAHs (ug/kg dry wt.)							
Acenaphthene	16	-	330	100	500	10	20
Acenaphthylene	44	-	330	100	500	10	20
Anthracene	85.3	0.5	330	100	500	10	20
Fluorene	19	8	330	100	500	10	20
Naphthalene	160	-	330	100	500	10	20
Phenanthrene	240	4	330	100	500	10	20

Table 3. continued.

CONTAMINANT	ISQV-LOW	GOVERNMENT HOKLAS No. 1 (HONG KONG)	INCHCAPE HOKLAS No. 5 (HONG KONG)	MATERIALAB HOKLAS No. 15 (HONG KONG)	ALS (HK) HOKLAS No. 66 (HONG KONG)	ASL (CANADA)	ARI (USA)
Benzo[a]anthracene	261	-	330	100	500	10	20
Benzo[a]pyrene	430	0.1	330	100	500	10	20
Dibenzo[a,h] anthracene	6304	-	330	100	500	10	20
Chrysene	384	2	330	100	500	10	20
Fluoranthene	600	1	330	100	500	10	20
Pyrene	665	3	330	100	500	10	20
ORGANICS - non PAH (ug/kg dry wt.)							
Total PCBs	22.7	5	40	50	100	2	4
Total DDT	1.58	0.5	50	50	50	1-2	0.2
p,p-DDE (4,4-DDE)		0.5	50	50	50	1	0.2
ORGANOMETALLICS (μg TBT/L in Interstitial Water)							
TBT	0.15	-	Not Done	-	-	0.002	0.3

[1] Dashes (-) indicate that information was not available from the questionnaires or HOKLAS (1996). Comparisons to ISQV-low concentrations provided where possible.

Table 4. Comparison of laboratory methods and instrumentation used to determine concentrations of ISQV contaminants.

CONTAMINANT	GOVERNMENT HOKLAS No. 1 (HONG KONG)	INCHCAPE HOKLAS No.5 (HONG KONG)	MATERIALAB HOKLAS No.15 (HONG KONG)	ALS(HK) HOKLAS No.66 (HONG KONG)	ASL (CANADA)	ARI (USA)
METALS						
Digestion	ASTM D-3974-81 (SAD) In House Method	ASTM D-3974-81 (SAD) EPA 3050 In House Method	Not Reported	In House Method - microwave EPA 3051 (SAD)	EPA 3050 (SAD)	EPA 3050 (SAD)
Cadmium	APHA 17e 3120-ICP/AES APHA 17e 3113-AAS EPA 6020-ICPMS	APHA 18e 3111B-AAS APHA 18e 3120-ICP	In House Method-AAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 7130-AAS	EPA 7000-AAS
Chromium	APHA 17e 3120-ICP/AES APHA 17e 3113-AAS EPA 6020-ICPMS	APHA 18e 3111B-AAS APHA 18e 3120-ICP	In House Method-AAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 6100-ICP/OES	EPA 6010-ICP/AES
Copper	APHA 17e 3120-ICP/AES APHA 17e 3113-AAS EPA 6020-ICPMS	APHA 18e 3111B-AAS APHA 18e 3120-ICP	In House Method-AAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 6100-ICP/OES	EPA 6010-ICP/AES
Mercury	InHouse Method-ICPMS & CVAAS APHA 17e 3112B-CVAAS	In House Method-CVAAS	In House Method-CVAAS	In House Method - CVAAS APHA 3112-CVAAS	EPA 7000 - CVAAS	EPA 7000-CVAAS
Nickel	APHA 17e 3120-ICP/AES APHA 17e 3113-AAS EPA 6020-ICPMS	APHA 18e 3111B-AAS APHA 18e 3210-ICP	In House Method-AAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 6100-ICP/OES	EPA 6010-ICP/AES
Lead	APHA 17e 3120-ICP/AES APHA 17e 3113-AAS EPA 6020-ICPMS	APHA 18e 3111B-AAS APHA 18e 3120-ICP	In House Method-AAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 6100-ICP/OES	EPA 6010-ICP/AES
Silver	EPA 6020-ICPMS	EPA 7760-AAS	Not Reported	In House Method - ICPMS	EPA 7000-FAAS	EPA 6010-ICP/AES
Zinc	APHA 17e 3120-ICP/AES EPA 6020-ICPMS	APHA 18e 3111B-AAS APHA 18e 3120-ICP	In House Method-AAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 6100-ICP/OES	EPA 6010 -ICP/AES

Table 4. continued.

CONTAMINANT	GOVERNMENT HOKLAS No. 1 (HONG KONG)	INCHCAPE HOKLAS No.5 (HONG KONG)	MATERIALAB HOKLAS No.15 (HONG KONG)	ALS(HK) HOKLAS No.66 (HONG KONG)	ASL (CANADA)	ARI (USA)
METALLOIDS						
Arsenic	APHA 17e 3114-HGAAS EPA 6020-ICPMS	APHA 18e 3114e-HGAAS	In House Method-HGAAS APHA 3114B-HGAAS	In House Method - ICPMS EPA 6020-ICPMS	EPA 7061-HGAAS	EPA 7061-HGAAS
ORGANICS - PAHs[1]						
Total PAHs	InHouse Method-HPLC	EPA 8270A - GC/MS	EPA 8270-GC/MS	In House Method - GC/MS EPA 8270 - GC/MS	EPA 8270C-GC/MS	EPA 8270-GC/MS
ORGANICS non-PAHs						
Total PCBs	InHouse Method-GC/ECD	EPA 8081-GC/ECD	EPA 8080-GC/ECD	In House Method - GC/MS EPA 8270-GC/MS	EPA8081/8082-GC/ECD	EPA 8081-GC/ ECD
Total DDT[2]	In House Method-GC/ECD	EPA 8081-GC/ECD	EPA 8080-GC/MS	In House Method - GC/MS EPA 8270-GC/MS	EPA 8081-GC/ECD	EPA 8081-GC/ ECD
ORGANOMETALLICS (In Interstitial Water)						
TBT	In House Method - GC/FPD	Not Done	In House Method - GC/MS	In House Method - GC/MS	Krone et al., (1989) - GC/MS	Krone et al., (1989) - GC/MS

[1] Total PAHs: individual PAH compounds identified in Table 1.
[2] Methods for individual DDT metabolites are included under Total DDT.

Table 5. Comparison of laboratory costs for ISQV contaminants.

CONTAMINANT	INCHCAPE (HONG KONG)	MATERIALAB (HONG KONG)	ALS (HK) (HONG KONG)	ASL (CANADA)	ARI (USA)
METALS [1]	$167	$220	$95	$80	$165
METALLOIDS					
Arsenic	$64	$60	-	included above	included above
ORGANICS - PAHs [2]					
Total PAHs	$185	$190	$120	$115	$200
ORGANICS - non PAHs					
Total PCBs	$185	$160	$120	$120	$150
Total DDT	$185	$180	$100	$195	$250
ORGANMETALLICS					
TBT	Not Done	$185	$210	$500	$230
Total Cost/Sample[3]	**$786**	**$995**	**$645**	**$995**	**$995**

1 Metals analysis includes sample digestion and analysis of eight metals: cadmium, chromium, copper, mercury, nickel, lead, silver and zinc.
2 Total PAHs: individual PAH compounds identified in Table 1.
3 Costs are provided as unit prices per sample, in U.S. dollars, based on a batch of 10 samples. Cost estimates are provided for illustrative purposes only, based on available information, and are subject to change. Unit costs not provided for Government Laboratory as its services are not available on a commercial basis. Sample shipment costs have not been included.

Table 6. Sources and types of certified reference materials (CRMs).

PCBs			
	National Research Council of Canada	Marine sediment	HS-1 and HS-2
PAHs			
	National Research Council of Canada	Marine sediment	HS-3, HS-4, HS-5, HS-6
	National Institute for Standards and Technology	Sediment	SRM #1647 and SRM #1597
Metals			
	National Bureau of Standards	Estuarine sediment	SRM #1646
	National Research Council of Canada	Marine sediment	MESS-1, BCSS-1, PACS-1
		Dogfish liver	DOLT-1
		Dogfish muscle	DORM-1
		Lobster hepatopancreas	TORT-1
	International Atomic Energy Agency	Marine sediment	SD-N-1/2(TM)
		Fish flesh	MA-A-2(TM)
		Mussel tissue	MAL-1(TM)

Standard reference materials (SRMs) may be obtained from the following organizations:

Organic Constituents

U.S. Department of Commerce
National Institute for Standards and Technology
Office of Standard Reference Materials
Room B3111 Chemistry Building
Gaithersburg, Maryland 20899
Telephone: (301)975-6776

Marine Analytical Chemistry Standards Program
National Research Council of Canada
Atlantic Research Laboratory
1411 Oxford Street
Halifax, NS Canada B3H 3Z1
Telephone: (902) 426-8280

Inorganic Constituents

U.S. Department of Commerce
National Institute for Standards and Technology
Office of Standard Reference Materials
Room B311 Chemistry Building
Gaithersburg, Maryland 20899
Telephone: (301) 975-6776

Marine Analytical Chemistry Standards Program
National Research Council of Canada
Division of Chemistry
Montreal Road
Ottawa, ON Canada K1A 0R9
Telephone: (613) 993-2359

Table 7. Examples of Data Quality Objectives (DQOs) for sediment chemical analysis.

VARIABLE	MATRIX	REPORTING UNITS	METHOD DETECTION LIMIT	SAMPLE SIZE[1]	PRECISION	ACCURACY	COMPLETENESS	METHOD	REFERENCE	HOLDING TIME
Metals	Sediment	mg/kg (dry weight)	0.005-30[2]	50g	±25%	±25%	95%	ICP/HVAAS/ CVAAS	EPA[3]Methods 6010/7000 Series	6 mths (Hg 28 days)
PAHs	Sediment	mg/kg (dry weight)	0.02	200g	±50%	±50%	95%	GC/MS	EPA[3]Methods 3540/8270 Series	14 days
PCBs/ Pesticides	Sediment	mg/kg (dry weight)	0.2	200g	±50%	±50%	95%	GC/ECD	EPA[3]Methods 3540/8080 Series	14 days
Percent Moisture	Sediment	%	0.1	50g	±20%	±20%	95%	Gravimetric	PSEP (1996b)	6 mths
Particle Size	Sediment	mg/kg (dry weight)	0.01	100g	±20%	±20%	95%	Sieve/ Pipette	Walton (1978)	6 mths
TOC	Sediment	mg/kg (dry weight)	0.05	20g	±20%	±20%	95%	Combustion	EPA[3]Methods 9060A	6 mths

[1] Recommended sample size in wet weight for one laboratory analysis.
[2] Detection limits vary for individual metals.
[3] EPA SW-846.

Table 8. Examples of Data Quality Objectives (DQOs) for sediment toxicity tests.

Test Type	Matrix	Endpoint(s)	Sample Size	Replicates	Negative Control	Reference Toxicant	Reference	Holding Time
10-d amphipod (*Eohaustorius estuarius*)	Sediment	Survival Avoidance Reburial	2L	5	≥90% Survival	±2SD	PSEP (1995)	2 weeks preferred; 8 weeks maximum
20-d polychaete *(Neanthes arenaceodentata)*	Sediment	Survival Dry Weight	2L	5	≥90% Survival	±2SD	PSEP (1995)	2 weeks preferred; 8 weeks maximum
48-h larval development	Sediment	Survival Normality	1L	5	≥70% Survival ≥90% Normality	±2SD	PSEP (1995)	2 weeks preferred; 8 weeks maximum

Table 9. Recommended test conditions for the amphipod toxicity test.

TEST PARAMETER	TEST CONDITION
Test species	*Eohaustorius estuarius*
Test type	Static, non-renewal
Test duration	10 days
Temperature (°C)	15 ± 1°C
Salinity (ppt)	28 ± 1 ppt
Light quality	Ambient laboratory light
Photoperiod	Continuous light
Test vessel size	1-L glass jar or beaker
Test material volumes	175 mL (2-cm layer) of sediment; 800 mL of overlying seawater
Replicates/treatment	5 plus water quality jar
Test organisms/replicate	20
Size/age of test organisms	3 - 5 mm long immature adults
Feeding	None
Renewal of overlying water	None
Aeration	Gentle bubbling to maintain DO above 60% saturation
Overlying water	Natural or reconstituted seawater (filtered, UV-sterilized)
Effects measured	Survival, avoidance, reburial
Test acceptability criteria	≥90% mean control survival

Table 10. Recommended test conditions for the polychaete worm toxicity test.

TEST PARAMETER	TEST CONDITION
Test species	*Neanthes arenaceodentata*
Test type	Static-renewal
Test duration	20 days
Temperature (°C)	20 ± 1°C
Salinity (ppt)	28 ± 1 ppt
Light quality	Ambient laboratory light
Photoperiod	Continuous light
Test vessel size	1-L glass jar or beaker
Test material volumes	175 mL (2-cm layer) of sediment; 800 mL of overlying seawater
Replicates/treatment	5 plus water quality jar
Test organisms/replicate	5
Size/age of test organisms	2 - 3 weeks post-emergence (0.5 - 1.0 mg dry weight) juveniles
Feeding	40 mg crushed TetraMarin/jar every second day
Renewal of overlying water	One-third of overlying water every third day
Aeration	Gentle bubbling to maintain DO above 60% saturation
Overlying water	Natural or reconstituted seawater (filtered, UV-sterilized)
Effects measured	Survival, dry weight, reburial
Test acceptability criteria	≥90% mean control survival

Table 11. Recommended test conditions for the bivalve larvae toxicity test.

TEST PARAMETER	TEST CONDITION
Test species	Mussels (*Mytilus edulis*) Oysters (*Crassostrea gigas*)
Test type	Static, non-renewal
Test duration	48 hours
Temperature (°C)	Mussels: 16 ± 1°C Oysters: 20 ± 1°C
Salinity (ppt)	28 ± 1 ppt
Light quality	Ambient laboratory light
Photoperiod	14:10 h light:darkness
Test vessel size	1-L jar or beaker
Test material volumes	18 g of sediment; 900 mL of overlying seawater
Replicates/treatment	5 plus water quality jar
Test organisms/replicate	20,000 - 40,000/L
Size/age of test organisms	<4 h post-fertilization embryos
Feeding	None
Renewal of overlying water	None
Aeration	None, unless specified
Overlying water	Natural or reconstituted seawater (filtered, UV-sterilized)
Effects measured	Survival, larval development
Test acceptability criteria	≥90% mean control normal development; ≥70% mean control survival

Table 12. Recommended test conditions for the echinoderm larvae toxicity test.

TEST PARAMETER	TEST CONDITION
Test species	Sand dollar (*Dendraster excentricus*) Sea urchin (*Strongylocentrotus*)
Test type	Static, non-renewal
Test duration	48 hours
Temperature (°C)	15 ± 1°C
Salinity (ppt)	28 ± 1 ppt
Light quality	Ambient laboratory light
Photoperiod	14:10 h light:darkness
Test vessel size	1-L jar or beaker
Test material volumes	18 g of sediment; 900 mL of overlying seawater
Replicates/treatment	5 plus water quality jar
Test organisms/replicate	20,000 - 30,000/L
Size/age of test organisms	<4 h post-fertilization embryos
Feeding	None
Renewal of overlying water	None
Aeration	None, unless specified
Overlying water	Natural or reconstituted seawater (filtered, UV-sterilized)
Effects measured	Survival, larval development
Test acceptability criteria	≥90% mean control normal development; ≥70% mean control survival

Table 13. Recommended test conditions for the clam bioaccumulation test.

TEST PARAMETER	TEST CONDITION
Test species	*Macoma nasuta*
Test type	Flow-through
Test duration	28 days
Temperature (°C)	15 ± 2°C
Salinity (ppt)	28 ± 1 ppt
Light quality	Ambient laboratory light
Photoperiod	14:10 h light:darkness
Test vessel size	10-L glass aquarium (minimum)
Test material volumes	3-4-cm layer of sediment; 5-10 cm of overlying seawater
Replicates/treatment	5
Test organisms/replicate	20
Size/age of test organisms	immature adults
Feeding	None
Renewal of overlying water	Continuous, flow-through
Aeration	Gentle bubbling to maintain dissolved oxygen at 60% saturation
Overlying water	Natural or reconstituted seawater (filtered, UV-sterilized)
Effects measured	Bioaccumulation
Test acceptability criteria	Performance-based