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Designation: E3163 – 18

Standard Guide for Selection and Application of Analytical Methods and Procedures Used during Sediment Corrective Action[1]

This standard is issued under the fixed designation E3163; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This guide discusses the selection and application of analytical methods and test procedures used during sediment programs. This guide provides a flexible, defensible framework for selection and application of analytical methods and testing protocols used in wide range of sediment programs. This guide is structured as a tool kit to support a tiered approach with procedures of increasing complexity as the user proceeds through the process. This guide provides information on key decision criteria and best practices to support the user in achieving intended sediment program objectives.

There are numerous technical decisions that must be made in the selection and application of analytical methods and testing protocols used during a sediment programs. It is not the intent of this guide to define appropriate technical decisions, but rather to provide technical support within existing decision frameworks. This guide is not intended to replace existing regulatory requirements or guidance, but rather to complement these programs. This guide encourages user-led collaboration with stakeholders, including analytical laboratories and testing service providers, in the selection and application of analytical methods and testing protocols used to support project-specific decision criteria for a particular sediment program. This guide recognizes the complexity and diversity of sediment programs and project objectives and provides appendixes to support a range of project applications. The appendixes are provided for additional information and are not mandatory for the use of this guide. ASTM standard guides are not regulations; they are consensus based standard guides that may be followed voluntarily.

1. Scope

1.1 This is a guide for the selection and application of a range of analytical methods and testing procedures that may be used during sediment programs, including physical properties testing, chemical analytical methods, passive sampling procedures, bioassays and toxicity testing, environmental forensics methods and procedures, and methods development procedures for sediment programs.

1.2 Sediment programs vary greatly in terms of environmental complexity, physical, chemical and biological characteristics, human health and ecological risk concerns, and geographic and regulatory context. This guide provides information for the selection and application of analytical methods and testing protocols applicable to a wide range of sediment programs.

1.3 This guide describes widely accepted considerations and best practices used in the selection and application of analytical procedures used during sediment programs. This guide supports and complements existing regulations and technical guidance.

1.4 This guide is designed for general application to a wide range of sediment programs performed under international, federal, state and local environmental programs. This guide describes the selection and application of analytical methods and test procedures, not the requirements for specific regulatory jurisdictions. This guide compliments but does not replace regulatory agency requirements.

1.5 This guide may be used for a wide range of sediment programs, including programs with overlapping regulatory jurisdictions, programs without a clearly established regulatory framework, voluntary programs, Brownfield programs, and international programs. The users of this guide should be aware of the appropriate regulatory requirements that apply to sediment programs. The user should consult applicable regulatory agency requirements to identify appropriate technical decision criteria and seek regulatory approvals, as necessary, prior to

[1] This guide is under the jurisdiction of ASTM Committee E50 on Environmental Assessment, Risk Management and Corrective Action and is the direct responsibility of Subcommittee E50.04 on Corrective Action.

Current edition approved Sept. 1, 2018. Published November 2018. DOI: 10.1520/E3163–18

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selection and application of analytical methods and test procedures to sediment programs.

1.6 This guide supports the collaboration of stakeholders, including project sponsors, regulators, laboratory service providers, and others, on the selection and application of analytical procedures to sediment programs. This guide highlights key considerations for designing sediment program data acquisition plans, including applicability and use limitations of analytical methods and test procedures, and data usability considerations. This guide recognizes the challenges inherent in selection and application of analytical methods and test procedures for sediment systems, as well as the challenges inherent in generating analytical data of sufficient sensitivity to meet regulatory criteria applied to sediment programs.

1.7 ASTM standard guides are not regulations; they are consensus standard guides that may be followed voluntarily to support applicable regulatory requirements.

1.8 Test methods, procedures, and guidelines published by ASTM, USEPA, and other U.S. and international agencies are used for sediment programs, many of which are referenced by this guide. However, these documents do not provide guidance on the selection and application of analytical methods and test procedures for sediment programs. This guide was developed for that purpose.

1.9 This guide may be used in conjunction with other ASTM guides developed for sediment programs.

1.10 The user of this guide should review existing information and data available for a sediment project to determine the most appropriate entry point into and use of this guide.

1.11 *Table of Contents:*

	Section
Introduction	
Scope	1
Referenced Documents	2
Terminology	3
Significance and Use	4
Physical Property Test Methods	5
Chemistry Analytical Methods	6
Passive Sampling Methods	7
Biological Test Methods	8
Environmental Forensics Analytical Methods	9
Analytical Method Development	10
Key Differences in Physical Properties of Sediment and Soil	Appendix X1
Guidelines for Collection of Sediment Samples for Physical Properties Testing	Appendix X2
Key Concepts in Sediment Stratigraphy for Physical Properties Testing	Appendix X3
Quick Reference Guide for Sediment Chemistry Analytical Method Selection	Appendix X4
Sampling Reference Guide for Sediment Chemistry Analytical Methods	Appendix X5
Critical Success Factors for Sediment Chemistry Analytical Programs	Appendix X6
Quick Reference Guide for Passive Sampling Method Selection	Appendix X7
Advantages and Limitations of Passive Sampler Types for Organic Compounds	Appendix X8
Methodologies and Equations for Determining Aqueous Chemical Concentrations from Passive Sampler Results	Appendix X9
Pros and Cons Evaluation of Biological Test Methods	Appendix X10
Decision Tree for Biological Testing Selection	Appendix X11
Species List for Biological Testing	Appendix X12
Daubert Criteria to Guide the Selection and Application of Analytical Test Methods Used for Environmental Sediment Forensics	Appendix X13
References	
Bibliography	

1.12 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.13 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D1498 Test Method for Oxidation-Reduction Potential of Water

D1586 Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D2217 Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants

D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D2488 Practice for Description and Identification of Soils (Visual-Manual Procedures)

D2937 Test Method for Density of Soil in Place by the Drive-Cylinder Method

D2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils

D3213 Practices for Handling, Storing, and Preparing Soft Intact Marine Soil

D4220/D4220M Practices for Preserving and Transporting Soil Samples

D4318 Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

D4464 Test Method for Particle Size Distribution of Catalytic Materials by Laser Light Scattering

D4643 Test Method for Determination of Water Content of Soil and Rock by Microwave Oven Heating

D5084 Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter

D5739 Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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D6169/D6169M Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations
D6913/D6913M Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis
D7263 Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens
D7363 Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode
D7928 Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis
E1367 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates
E1391 Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing and for Selection of Samplers Used to Collect Benthic Invertebrates
E1525 Guide for Designing Biological Tests with Sediments
E1611 Guide for Conducting Sediment Toxicity Tests with Polychaetous Annelids
E1688 Guide for Determination of the Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates
E1689 Guide for Developing Conceptual Site Models for Contaminated Sites
E1706 Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates
E1850 Guide for Selection of Resident Species as Test Organisms for Aquatic and Sediment Toxicity Tests
E2081 Guide for Risk-Based Corrective Action
E2122 Guide for Conducting In-situ Field Bioassays With Caged Bivalves
E2205/E2205M Guide for Risk-Based Corrective Action for Protection of Ecological Resources

2.2 *API Document:*[3]

API RP40 Recommended Practices for Core Analysis, 1998

2.3 *EPA Documents:*[4]

USEPA Method 608 Organochlorine Pesticides and PCBs, SW-846 Compendium, 1984
USEPA Method 680 Determination of Pesticides and PCBs in Water and Soil/Sediment by GC/MS, SW846 Compendium, 1985
USEPA Method 1613 Revision B Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, SW-846 Compendium, 1994
USEPA Method 1664 Revision B n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, SW-846 Compendium, 2010
USEPA Method 1668 Revision A Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGS/HRMS, SW-846 Compendium, 2003
USEPA Method 1668 Revision B Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGS/HRMS, SW-846 Compendium, 2008
USEPA Method 8082 Revision A Polychlorinated Biphenyls (PCBs) by GC, SW-846 Compendium, 2007
USEPA Method 8260 Revision B Volatile Organic Compounds by GC/MS, SW-846 Compendium, 1996
USEPA Method 8270 Revision C Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Compendium, 1996
USEPA Method 8290 Revision A Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDF) by HRGC/HRMS, SW-846 Compendium, 2007

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 The reader should review the definitions presented below prior to proceeding with use of the guide. This guide assumes a basic working knowledge of analytical procedures and test methods applicable to sediment programs. Where possible and applicable, the terms included in this guide have meanings consistent with published regulatory definitions widely used within existing international, federal, state, and local programs. The following terms are being defined to reflect their specific use in this guide. These definitions do not replace existing regulatory definitions.

3.1.2 *sediment, n*—a matrix of pore water and particles including gravel, sand, silt, clay and other natural and anthropogenic substances that have settled at the bottom of a tidal or non-tidal body of water.

4. Significance and Use

4.1 This guide should be used to support existing decision frameworks for the selection and application of analytical procedures to sediment programs.

4.2 Activities described in this guide should be conducted by persons familiar with current sediment site characterization and remediation techniques, sediment remediation science and technology, toxicology concepts, risk and exposure assessment methodologies, and ecological evaluation protocols.

4.3 This guide may be used by various parties involved in sediment programs, including regulatory agencies, project sponsors, environmental consultants, toxicologists, risk assessors, site remediation professionals, environmental contractors, analytical testing laboratories, data validators, data reviewers and users, and other stakeholders, which may include, but are not limited to, owners, buyers, developers, lenders, insurers, government agencies, and community members and groups.

4.4 This guide is not intended to replace or supersede federal, state, local or international regulatory requirements. Instead this guide may be used to complement and support such requirements.

[3] Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, http://www.api.org.

[4] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

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4.5 This guide provides a decision framework based on over-arching features and elements that should be customized by the user based on site-specific conditions, regulatory context, and sediment program objectives for a particular site. This guide should not be used alone as a prescriptive checklist.

4.6 The selection and application of analytical methods and test procedures for sediment programs is an evolving science. This guide provides a systematic but flexible decision framework to accommodate variations in approaches by regulatory agency and by user based on project objectives, site complexity, unique site features, programmatic and regulatory requirements, newly developed guidance, newly published scientific research, use of alternative scientifically-based methods and procedures, changes in regulatory criteria, advances in scientific knowledge and technical capability, multiple lines of evidence approach, and unforeseen circumstances.

4.7 The user of this guide should review the overall structure and components of this guide before proceeding with use, including: Section 1 - Scope; Section 2 - References; Section 3 - Terminology; Section 4 - Significance and Use. The remainder of this guide is organized as a tool kit to support the selection and application of a range of test methods and procedures that may be used at various stages of a sediment program, including: Section 5 - Physical Property Test Methods; Section 6 - Chemistry Analytical Methods; Section 7 - Passive Sampling Methods; Section 8 - Biological Test Methods; Section 9 - Environmental Forensics Analytical Methods; and Section 10 - Analytical Methods Development. Nonmandatory Appendix X1 – Appendix X13 provide users of this guide with additional information. A list of References and a Bibliography are provided at the end of this guide.

4.8 *Project Scoping and Planning*—This guide supports that systematic planning process for selection and application of analytical procedures used for sediment programs. The use of this guide compliments applicable existing guidance used to develop a Quality Assurance Project Plan (QAPP) and to establish data quality objectives (DQO) necessary to meet project goals and to fully understand data quality. This process encourages planners to identify and focus on the key issues that must be addressed and resolved for successful, cost-effective, and defensible project outcomes.

4.9 The use of this guide also supports the development and refinement of a Conceptual Site Model (CSM) as part of the planning process for site activities that involve gathering environmental data.

4.10 Implementation of the guide is site-specific. The user of this guide may choose to customize the implementation of the guide for particular types and/or phases of sediment programs.

4.11 This guide may be initiated at any time during a sediment program, including: site characterization, assessment, remedy selection, remedial design, remedial implementation, remedial operation and maintenance, baseline and long-term monitoring, remedy optimization, and corrective action.

4.12 Use of this guide supports the use of systematic project planning, dynamic work strategies, use of innovative sampling and analytical technologies, and application of best management practices and guiding principles as applied to contaminated sediment programs.

4.13 Use of this guide supports a multiple lines of evidence approach, including a weight of evidence approach, for assessment, remediation, and monitoring of contaminated sediments.

4.14 Use of this guide is consistent with the Sediment-RBCA process which guides the user to acquire and evaluate additional data, obtain the appropriate data and refine goals, objectives, receptors, exposure pathways, and the site conceptual model. As the Sediment-RBCA process proceeds, data and conclusions reached at each tier help focus subsequent tiered evaluation. This integrative process results in efficient, cost-effective decision-making and timely, appropriate response actions for contaminated sediment programs.

4.15 *Planning Framework*—When applying this guide, the user should undertake a systematic project planning and scoping process to collect information to assist in making site-specific, user-defined decisions for a particular project. Planning activities should include the following factors: *(a)* Assemble an experienced team of project professionals; *(b)* Engage stakeholders early and often in the planning process; *(c)* Define, agree on, and document clearly stated project objectives and intended outcomes; *(d)* Recognize that sediment programs are complex, uncertainty is high, that an appropriate projectspecific approach may be developed with the investment of time and effort, and that compromise and uncertainty are inherent in the process; *(e)* Identify the applicable regulatory program(s); *(f)* Compile existing site data; and *(g)* Establish a plan for documenting and reporting key decisions and results. These project planning and scoping activities should be carried forward as the project progresses.

4.16 *Experience and Expertise*—The users of this guide should consider assembling a team of experienced project professionals with appropriate expertise to scope, plan and execute a sediment data acquisition program. The team may include: regulatory agencies, project sponsors, environmental consultants, toxicologists, risk assessors, site remediation professionals, environmental contractors, analytical testing laboratories, and data reviewers, data validators, data users, and other stakeholders.

4.17 *Stakeholders*—The users of this guide are encouraged to engage key stakeholders early and often in the project planning and scoping process, especially regulators, project sponsors, and service providers including analytical testing laboratories. A concerted ongoing effort should be made by the guide user to continuously engage stakeholders as the project progresses in order to gain insight, technical support and input for resolving technical issues and challenges that may arise during project implementation.

4.18 *Documentation*—The users of this guide should establish a plan for documenting and reporting the results of the project planning process, including: key challenges, options considered, decisions taken, data acquisition approach, data results, and project outcomes relative to project objectives.

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Project documentation may include: Project Work Plans, Sampling and Analysis Plans (SAP), Quality Assurance Project Plans (QAPP), Technical Memos, and Project Reports. The user must ensure that the test methods used meet the analytical rigor required by the regulatory agency or agencies having oversight authority for the project.

4.19 The users of this guide are encouraged to continuously update and refine the project Conceptual Site Model (CSM), Work Plans and Reports used to describe the physical properties, chemical composition and occurrence, biologic features, and environmental conditions of the sediment project.

4.20 *Key Considerations*—This guide supports users in the identification of key considerations for designing and implementing sediment program data acquisition plans, including discussion of applicability and use limitations of analytical methods and testing procedures.

4.21 *Challenges*—This guide is designed to assist the user in more fully understanding and navigating the challenges inherent in the selection and application of analytical methods and test procedures for use in sediment programs, specifically challenges in generating analytical data of sufficient sensitivity to support the stringent regulatory screening levels applied to sediment programs. USEPA (2005a) (**1**)[5] has long recognized the challenges associated with sediment programs, as summarized below:

4.21.1 Sources may be various, large, ongoing, and/or difficult to control,

4.21.2 Impacts may be diffuse, large, and diverse,

4.21.3 Environment may be dynamic, increasing the difficulty in understanding effects of natural forces and man-made events on sediment movement and stability and contaminant fate and transport,

4.21.4 Cleanup work often involves engineering challenges and higher costs than for other media,

4.21.5 Mixed land uses and numerous property owners and communities with differing views, opinions, and impacts often complicate cleanup efforts, and

4.21.6 Ecologically valuable resources and/or legislatively protected species or habitats may be present.

5. Physical Property Test Methods

5.1 This section of the guide discusses the selection and application of physical property test methods to characterize sediment sites for the evaluation of risk. This section of the guide contains a general discussion of tools and common methods separated via stratigraphic and internal scale of analysis.

5.2 The guide is intended to provide technical direction with respect to the fundamental physical properties to enable an independent and comprehensive description of the sediment to support general environmental investigations. Physical property measurements provide quantitative understanding of the size, composition, and consolidation of the particles composing the sediment and the capacity for water to move through the sediment. Although the chemical composition and chemistry of the porewater may influence these fundamental properties to a degree, these are generally minor influences and do not affect the selection and use of physical property tests.

5.3 Sediment physical properties have significant impacts on how sediment functions in the environment and are therefore important components of the conceptual site model. Knowledge of physical properties has many uses during every phase of sediment programs, from initial assessment through corrective actions and finally closure. For example, measurements of sediment physical properties provide information on sediment behaviors such as deposition, erosion, and resuspension, habitat types for plants and animals, groundwater to surface water seepage, and assessment of gas generation and ebullition facilitated NAPL transport. The physical properties, such as density, water, hydraulic conductivity, and plasticity, also provide useful information for remedial purposes including the ability of the sediments to be dredged, dewatered, and handled once removed from the water, and the ability of the sediments to support caps.

5.4 This guide does not provide a comprehensive discussion of the analyses that may be warranted in remedial selection or design, and it is not intended to provide guidance for geotechnical testing or analysis of sediments. However, many of the physical property analyses presented would be needed in evaluating remedial actions.

5.5 *Overview:*

5.5.1 The physical properties of sediment are utilized throughout the investigation including development of the Conceptual Site Model (CSM), site characterization, risk evaluation, and monitoring processes. Since the results of these analyses are applied throughout the environmental process, it is critical that the analyses conducted are appropriately performed. This section of the guide documents the key parameters that can describe the physical characteristics of sediments, the methods that are applied to quantify the parameters, and the sampling protocols for physical parameters testing. One of the challenges faced by practitioners working with sediments is accurately defining and interpreting physical properties of sediments

5.5.2 Many of the inferences relied upon in land-based programs, may not be appropriate to apply in sediments. Sediments may exhibit significant differences compared to soil for several key physical properties. In particular, sediments typically have higher porosities and higher moisture contents than land-based soils. As a result, sediment bulk densities are significantly lower and more variable than soils.

5.5.3 Due to the differences in variability in porosity and bulk density between land-based soils and sediments, similar values of common parameters such as concentration and saturation cannot be assumed to be consistent between these media. For example, the concentration of a chemical compound in soils and sediment is reported on a dry weight basis (that is, mg/Kg). Due to the variability and significant differences in bulk densities of sediments relative to soils, direct comparison of concentration values between soils and sediments may not be technically valid. A two to four-fold

[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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difference may occur between the Bulk Density values between sediments and soils, which then produces a two to four-fold mass per volume difference between these media. This difference as well as many others relative to soils can affect perspectives on risk, remedial mass, and porewater volume. Hence, the recognition of the importance of measuring sediment physical properties is critical throughout the sediment evaluation, corrective action, and monitoring phases. Further information on the differences in soil and sediment is further discussed in Appendix X1.

5.5.4 In the sediment environment, physical parameters may vary vertically and laterally within the sediment profile. For example, in many cases, the bulk density of the sediment increases with depth as consolidation of the sediment column occurs. This reduces porosity and the sediment moisture content. Hence, a comprehensive sampling program is needed to fully characterize the sedimentary environment.

5.5.5 Physical properties are important in numerous facets of the evaluation, remediation, and monitoring of contaminated sediments. Physical properties aid in defining such key CSM components as *(1)* the depositional energy of the site, *(2)* the rate and form of sedimentation, *(3)* the stratigraphy of the sediment column, *(4)* the nature of the sediment pore structure, and *(5)* the movement of fluids within the sediment.

5.5.6 Moreover, the physical properties may provide key information as to the origin and source of contamination, the form and distribution of the contamination, and the mobility of the contamination, which are critical elements in evaluating risk.

5.6 *Sediment Collection:*

5.6.1 Sediment collection for physical properties testing may be performed by the general methods of grab samples, cores, and borings. Grab samples disturb the sediment structure and include sediment collected from ponar, box or similar methods. Grab samples may also include sediment from cores where hand samples are collected. Core samples refer to sediment collected within a continuous core tube driven or vibrated into the sediment. The sediment is retained within the container and, if handled and capped appropriately, provides a representative sample of the sediment column. Borings are generally used to obtain a relatively undisturbed sample, which is critical to obtaining accurate measurement values for many physical properties. Borings are drilled into the sediment using equipment similar to land borings and relatively undisturbed sediment samples are collected at discrete depth intervals.

5.6.2 Methods for collecting sediment samples for physical testing are described in several of the ASTM methods in Section 2 including Test Method D1586, as well as in EPRI (2008) (**2**), ITRC (2014) (**3**), National Research Council (2007) (**4**), NAVFAC (2003) (**5**), NAVFAC/SPAWAR (2010) (**6**), USACE (1998, 2008) (**7, 8**), and USEPA (2001, 2005b, 2014) (**9-11**), as well as state regulatory agency documents.

5.6.3 Some of the analyses performed for sediment physical properties (including bulk density, porosity, saturation, and permeability) require an intact, relatively undisturbed sample, which can be collected from borings.

5.6.4 In finer grained sediments, vibracore, Geoprobe, or use of thin-walled tubes, or a combination thereof, in borings may be more practicable methods to collect an intact sample. However, each drilling technique will have positive and negative attributes. In particular, the disturbance of the sediment structure by these methods will require an independent, site-specific assessment.

5.6.5 Other physical properties analyses (for example, grain size, water content, Atterberg limits) can be performed on disturbed samples, which can be collected from grab or core samples. Appendix X2 discusses the collection of sediment samples for physical property analyses conducted for characterization purposes. Discussion of the collection of sediment samples for higher level geotechnical testing for engineering design applications (including hydraulic conductivity, consolidation, and shear strength) is beyond the scope of this document .

5.7 *Sediment Characterization:*

5.7.1 Since sediments are deposited and are generally composed of a series of layers that have accumulated through time, scale becomes an important component in the characterization process. In particular, the sample(s) obtained from a sediment core should be representative of a select sediment interval.

5.7.2 In general, the characterization process should be conducted whereby the stratigraphic characteristics of the sediment are described first, followed by subsequent investigation of the internal structure of the sediment.

5.7.3 This may require more than one co-located core, where intact, non-disturbed samples are needed. The following analyses are discussed with respect to the scale evaluated.

5.8 *Stratigraphic Physical Analyses:*

5.8.1 Upon retrieval, sediment cores utilized for descriptive observations may be handled in a similar manner as land-based cores.

5.8.2 Specifically, the sediment is extracted from the core tube and is examined visually with characteristics of color, texture, moisture, and stratification being recorded. Visual observations of the particles and their arrangement provide the basis to discern a representative sample from a defined interval. As such, a hand lens should be used to describe the size and shape of the particles considering such characteristics as roundness, sphericity, and mineralogy. The distribution of the particle sizes should also be described and the relative consolidation of the sediment. Shallow sediments, in particular, may act as a fluid due to their high water content and lack of consolidation. From these descriptions, the macroscale components of the sediment, such as stratification and the character and nature of contacts, are identified. These features ultimately define representative intervals from which samples can be collected to further refine the visual observations.

5.8.3 When appropriate, observations and measurements associated with organic or other constituents can be made. If safe conditions exist, odors should be documented and described. Organic vapor measurements can be made using Photo Ionization Detection (PID) equipment and identification of separate phase organic liquids may be identified by applying UV light.

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5.8.4 By defining the elevation of the water surface and the depth of the water during the collection process, the elevation (relative to a benchmark) of the measurements and samples can be determined.

5.8.5 The collected data is then presented in a sediment core log. A comprehensive description of the sediment is the fundamental basis from which sediment profile is discerned and is the technical document to which laboratory results, both physical and chemical, are ultimately referenced.

5.8.6 The collection and description of several cores will produce an understanding of the sediment stratigraphy. The stratigraphy will document changes in sediment texture laterally and vertically and the nature of the contact between the different sediment layers.

5.8.7 These features will provide an understanding of the depositional conditions during the past. If the contamination was derived from sedimentation, then the stratigraphy will document the physical conditions that were occurring during the time period of contamination. Appendix X3 provides further information regarding sediment stratigraphy.

5.9 *Physical Property Analyses:*

5.9.1 Characterization of the internal nature of the sediment entails measurement of numerous properties to evaluate the particle size and distribution and the fluids contained within the pore structure. In general, with depth sediments become compacted, which changes the physical properties of the sediment.

5.9.2 The following discusses the measurement of physical properties that are commonly applied to characterize sediments. The applicable Method Reference, preferred sample volume/size, preservation requirements, and holding time constraints are summarized in Table 1.

5.9.3 *Grain Size*—Grain size is one of the most fundamental sediment characteristics. Test Methods D6913/D6913M Sieve analysis, Test Method D4464 Laser Diffraction analysis, and Test Method D7928 Hydrometer analysis provide information on sediment consisting primarily of clay to sand size particles. In these analyses, particles sizes larger than 75 microns (µm) are measured by sieves and particles less than 75 µm by hydrometer. In addition to these methods, Test Method D4464 Laser Diffraction also provides high resolution measurement of

TABLE 1 Summary of Typical Sample Volume, Preservation and Holding Time Requirements

Test Description	Method Reference	Typical Sample Volume/Size[A]	Preservation	Holding Time[B]
GRAIN SIZE[C]				
Grain Size Analysis: Dry Sieve Only	Test Methods D6913/D6913M	200-500 g; core, bag or jar	[D]	—
Grain Size Analysis: Laser Diffraction	Test Method D4464	50-100 g; core, bag or jar	[D]	—
Particle Size Analysis - Water: Laser Diffraction	Test Method D4464	1-2 L of water	[D,E]	[F]
Grain Size Analysis: Dry Sieve + Laser Diffraction "Combined analysis"	Test Methods D6913/D6913M-17	200-500 g; core, bag or jar	[D]	—
Grain Size Analysis: Dry Sieve + Hydrometer	Test Methods D6913/D6913M-17	200-500 g; core, bag or jar	[D]	—
Particle-Size Distribution (Gradation) of Fine-Grained Soils using the Sedimentation (Hydrometer) Analysis	Test Method D7928-16	200-500 g; core, bag or jar	[D]	
BULK DENSITY				
Bulk Density (dry unit weight)	Test Method D2937 / Test Methods D7263 or API RP40	2.5 in. diam. × 6 in. long sleeve core	[D,G,H]	—
WATER CONTENT				
Water Content	Test Methods D2216	50-100 g; core, bag or jar	[D,G]	—
POROSITY				
Porosity: Total	Test Methods D854—14 / API RP40	2.5 in. diam. × 6 in. long sleeve core	[D,G,H]	—
Porosity: Air or Water-Filled	Test Methods D854-14 / API RP40	2.5 in. diam. × 6 in. long sleeve core	[D,G,H]	—
HYDRAULIC CONDUCTIVITY				
Hydraulic Conductivity: Saturated; flexible wall triaxial	API RP40, EPA9100, Test MethodsD5084	2.5 in. diam. × 6 in. long sleeve core	[D,G,H]	—
ATTERBERG LIMITS AND CLASSIFICATION				
Atterberg Limits; Plastic & Liquid Limits	Test MethodsD4318	100-500 g; core, bag or jar	[D,G]	—
Classification: Engineering USCS	Practice D2487	Requires Grain Size + Atterberg	[D,G]	—
Classification: Visual / Manual	Practice D2488	100-500 g; core, bag or jar	[D,G]	—
MINERALOGY				
Bulk and Clay Mineralogy	USGS Open-File Report	200-500 g	[D]	—

[A] Typical sample size is the volume or core type needed to obtain undisturbed or minimally disturbed material for testing in laboratory instruments or apparatus, or both. The requested core sample size is a 2.5 in. diameter. × 6 in. long brass sleeved core or similar volume. Core types suitable for submittal are brass or stainless steel rings/sleeves, acetate sleeved core (from direct-push or continuous coring methods) Shelby tubes, PVC sleeves, etc.

[B] There are no recognized holding times for many conventional physical properties analyses or core analysis tests. Indefinite holding times may be appropriate if it can be demonstrated that the test results are not adversely affected from preservation or storage. Selection of core size/containers, preservation techniques and applicable holding times should be based on the stated project-specific data quality objectives. Samples should be analyzed as soon as possible to provide data measured at as-received conditions.

[C] Reported volumes are provided as a general guide. The sample volume depends on particle size of the sediment. When particles are up to 75 mm in size, sample volumes up to 5 kilograms may be required.

[D] Keep sealed.

[E] Keep cool, chilled at 4 °C is required.

[F] Water samples should be analyzed within 7 days of sample receipt.

[G] Keep cool, chilled at 4 °C is recommended.

[H] Minimally disturbed samples recommended.

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the sand through fine clay particle fractions (2 mm to submicron range). This analysis may be needed for very fine grained sediments. Additional methods for the measurement of fine grain sediments that have been utilized include the Puget Sound Estuary Protocols (PSEP) (USEPA, 1986, 1997, 2015a) (**12, 13**), which utilizes a pipette approach, and the Coulter counter, which utilizes electric currents in electrolyte solutions. The selection of the test method employed for a given sediment investigation needs to consider many factors including project data quality objectives, intended data uses, cost, and technical acceptance.

5.9.4 *Bulk Density*—Bulk Density can be measured on a dry or wet basis. In Test Methods D7263 discusses the measurement of bulk density on a dry basis. The density of the sediment is expressed as the mass of the sediment divided by the volume of sediment, and is usually reported in grams per cubic centimeter (g/cm^3). This is based on dry unit weight. Bulk density depends on mineral composition and degree of compaction. Sediment dry-bulk density values are used in mass accumulation rate calculations.

NOTE 1—Shallow sediments, in particular, may act as a fluid due to the high water content and lack of compaction; special sample preparation methods such as freezing subsamples may need to be employed in the laboratory for certain analyses. In Test Method D2937 the measurement of bulk density on a wet basis is described. The density of the sediment is expressed as the wet mass of sediment divided by the volume and is reported in pounds per cubic foot.

5.9.4.1 Dry or wet unit weights can be calculated using formulas given in soil mechanics references such as Design Manual 7.1, Soil Mechanics (NAVFAC, 1986) (**14**). For saturated sediment, unit weights can be calculated based on the water content, specific gravity of dry solid particles and the density of water.

5.9.5 *Water Content*—In Test Methods D2216 the measurement of water content of the sediment is described. The geotechnical method for calculating moisture content on a dry-weight basis is not comparable to the chemical method for calculating percent solids or total solids on a total-weight basis or percent water (refer to Section 6). Water content determined by Test Methods D2216 is calculated as a percent of the weight of the water divided by the weight of the dry sediment, which can result in moisture contents greater than 100 %.

5.9.5.1 Percent solids are used to report chemical analytical results on a dry weight basis (refer to Section 6) . Percent solids analyzed using SM 2540G or USEPA Method 160.3 is calculated as the weight of the dry sediment divided by the weight of the wet sediment (refer to Section 6). An explanation of the different ways of reporting solids concentration and the relationship between Water Content, percent solids by weight, and dry unit weight for saturated sediment or soils is discussed in Chapter 3 of USACE (1987) (**15**).

5.9.6 *Total Porosity*—Total porosity is defined as the sediment pore volume as a percent relative to the total sediment volume. Method API RP40 describes the measurement of total porosity. Total porosity values may vary in sediments depending upon depth and particle size, as well as roundness and packing of particles. Porosity is an important parameter in characterizing void space in a material. Porosity may be determined by calculation from Specific Gravity, Bulk Density and Moisture Content results (see formulas in Test Methods D7263 and NAVFAC 1986 (**14**)).

5.9.7 *Specific Gravity*—Specific gravity is the density of soil divided by the density of water. Specific gravity is the ratio between density of the soil or sediment particles and the density of water at 4 °C. The specific gravity is used to correlate the mass and volume of a material. Test Methods D854 describe the measurement of specific gravity.

5.9.8 *Hydraulic Conductivity (Permeability)*—Hydraulic conductivity is the rate of discharge of water under laminar flow conditions through a unit cross-sectional area of porous medium under a unit hydraulic gradient and standard temperature conditions (20 °C). Test Methods D5084 describes the measurement of hydraulic conductivity of water-saturated porous materials with a flexible wall permeameter at temperatures between 15 and 30 °C. The method describes six methods to measure the hydraulic conductivity.

5.9.9 *Atterberg Limits*—The Atterberg limits are a basic measure of the plasticity of a fine-grained (that is, silt and clay) sediment. The analysis determines the moisture content where the material begins to act in a plastic state which is defined as the plastic limit and the moisture content where the material begins to act in a liquid state which is defined as the liquid limit. The changes in behavior and consistency are characterized as fine grained sediment takes on increasing amounts of water. Depending on the water content of the sediment, the sediment may appear in four states: solid, semi-solid, plastic and liquid. In Test Methods D4318 the measurement of the Atterberg limits is described.

5.9.10 *Particle Form and Mineralogy*—The form and type of minerals present in the sediment are an important characteristic that influences both the physical and chemical conditions of the sediment. Properties influenced by the sediment mineralogy include sorption, bulk density, porosity, hydraulic conductivity, water content, and Atterberg limits. Due to their generally sheet-like structure and electrical surface charges, clay minerals are particularly important in affecting the physical and chemical conditions within a sediment. Moreover, the composition of the clay mineral suite provides unique information regarding the upland source conditions. The USGS Open File Report 01-041 (USGS, 2001) (**16**) describes the application of x-ray diffraction (XRD) to determine the mineralogy of clay minerals. For larger particle sizes (that is, sand) the shape of the particles can be described. Two common characteristics have been widely investigated roundness, the shape of the corners of a particle, and sphericity, a measure of a particle shape relative to a sphere. These characteristics reflect the original source rock as well as the type and distance the particle has been transported. The roundness and sphericity will affect the consolidation and movement of fluids within the sediment. In general, platy minerals such as clays and micas will to be deposited in a horizontal orientation, which enhances consolidation and lowers the permeability of the sediment relative to spherical-shaped particles that produce a more open porous sediment framework. Quantitative methods describing the shape of particles are described in Pettijohn (1975) (**17**) and Lewis and McConchie (1994) (**18**).

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6. Chemistry Analytical Methods

6.1 This section of the guide supports users on the selection and application of analytical procedures used for chemical determination of compounds of potential concern (COPC) for sediment programs.

6.2 *Project Planning Considerations*—Chemical determination of COPCs for sediment programs presents unique challenges due to the significant differences that exist between upland and sediment programs. The project decision framework applied to upland programs may not be applicable for use in sediment programs. Sediment chemistry analytical programs typically involve method modifications, performance enhancements, and additional procedures to achieve project criteria. A general project planning guide for sediment chemistry analytical programs is provided in Table 2.

6.3 *Selection and Application of Chemical Analytical Methods:*

6.3.1 *Overview*—The determination and quantitation of chemicals of potential concern is important throughout the characterization, evaluation, corrective action, and monitoring phases of a sediment program. The selection and application of chemical analytical procedures for sediment programs should include a review of available analytical chemistry approaches and best practices discussed in the sections below. Many of the sediment chemistry analytical methods discussed in this guide are based on USEPA's Test Methods for Evaluating Solid Waste: Physical/Chemical Methods Compendium SW-846 (USEPA, 2017) (**20**). Appendix X4 provides a Quick Reference Guide to Sediment Chemistry Analytical Methods. Appendix X5 provides a Sediment Chemistry Sampling Reference Guide. Appendix X6 provides a discussion of critical success factors for sediment chemistry analytical programs. Sediment chemistry analytical laboratory selection should include review of laboratory's accreditation issued by the appropriate regulatory entity, as well as engagement of the laboratory in the development of the Quality Assurance Project Plan (QAPP) and review of project data quality objectives (DQOs) and project specific criteria (for guidance refer to USEPA, 2000 (**21**); USEPA, 2001 (**22**); USEPA, 2002ab (**23, 24**)).

6.3.2 *Sample Media and Matrix Considerations:*

6.3.2.1 Samples collected in support of sediments programs may consist of multiple media and matrix types including: *(a)* sediment, *(b)* pore water, *(c)* surface water, *(d)* groundwater, and *(e)* biota.

6.3.2.2 Sample media and matrix complexities present unique analytical chemistry challenges, such as elevated sediment moisture levels which directly impact analytical reporting limits and extraction efficiencies. Also sediments may contain elevated concentrations of target and/or non-target compounds and other matrix interferences, necessitating a customized analytical approach to improve data quality and usability outcomes.

6.3.2.3 Considerations unique to sediment programs require detailed planning and coordination with the laboratory to develop customized analytical approaches to achieve project-specific objectives.

6.4 *Trace Organic Compound Analysis:*

6.4.1 *Overview*—Trace organic compound analytical methods consist of three separate stages: *(a)* the preparative stage to isolate (extract) and concentrate target analytes of interest, *(b)* the extract clean- up stage to remove potential matrix interferences, followed by *(c)* the determinative stage where the sample extract is analyzed for target analytes of interest by specific analytical instrumentation. The objective of the sample preparation step is to maximize the analyte of interest concentration, minimize potential interferences, and enhance the level of sensitivity (that is, Reporting Limit) required for the project. The analytical method dictates the specific instrumentation used for a given analytical parameter at the level of sensitivity which is required for the project. For projects requiring a QAPP, the analytical method reference for each of these three steps must be documented in the QAPP. A general discussion follows for the most commonly used analytical methods for sediment programs.

6.4.2 *PCB Aroclors, Homologs and Congeners:*

6.4.2.1 Polychlorinated Biphenyls (PCBs) are a class of 209 compounds called congeners with groups called homologs. PCB Aroclors are complex mixtures of congeners produced to support various commercial uses. PCB Aroclors 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262 and 1268 identify nine commercial products produced and used from 1929 until 1979 in the United States.

6.4.2.2 Screening level investigations often begin with PCB Aroclor analysis by USEPA Method 8082 with gas chromatography with electron capture detection (GC-ECD) instrumentation. PCB Aroclor analysis is qualitative in nature due to the fact that chromatographic patterns and peak ratios are subject to change in the environment due to "weathering". However, PCB Aroclors may be reported as not detected (ND) due to a lack of pattern recognition even though PCB congeners may be present. Determination of Total PCB concentration using the PCB Aroclor approach presents challenges as individual Aroclors represent mixtures of PCB congeners, thus there is a possibility that the "double counting" of PCBs may occur. Additional information is provided in Section 9 of this guide.

6.4.2.3 Sediment programs may use USEPA Method 1668 based on high resolution gas chromatography with high resolution mass spectrometry (HRGC/HRMS) to determine low level PCB congeners and homologs to eliminate uncertainties associated with the PCB Aroclor analysis approach. USEPA Method 1668 is also extremely sensitive, thus it is used for ambient water quality studies, TMDL determinations, forensic PCB investigations, and sediment and tissue programs. This procedure can determine all 209 PCB congeners, or a subset of congeners (that is, WHO, NOAA or custom list), and homolog groups. USEPA Method 8270-SIM-modified GC/LRMS for PCB congener and homolog analysis provides an option for determination of all 209 PCB congeners with less sensitivity (higher RL). Additional information is provided in Section 9 of this guide.

6.4.3 *Semivolatile Compounds, Polynuclear Aromatic Hydrocarbons (PAHs), Alkylated PAH Homologs:*

6.4.3.1 Analysis of acid and base-neutral extractable compounds also known as semivolatile organic compounds (SVOCs) for the full list of target analytes that may be

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TABLE 2 Project Planning Guide for Sediment Chemistry Analytical Programs

Considerations	Upland Programs	Sediment Programs
Laboratory Certifications	Laboratory certification required by regulatory entity for conventional analytical protocols (that is, published analytical methods)	Laboratory certifications may be required by regulatory entity for specialized extraction, extract cleanup, digestion, and instrument analysis methods, as well as non-standard analytical protocols (such as literature references, laboratory SOPs, etc.)
Regulatory Criteria	Standard analytical approaches used to meet regulatory criteria for upland sites	Specialized analytical approach may be needed to meet project specific criteria (such as low level Ecological Screening Levels)
Project Plans	Standardized, routine regulatory program approach	Non-standard approaches detailed in project workplan and QAPP
Program/Project Goals	Site characterization, delineation, risk evaluation, corrective action, monitoring	Site characterization, delineation, risk assessment, corrective action, monitoring, ecological restoration
Matrices	Soil, groundwater, surface water	Sediment, pore water, surface water, biota
Target Analytes	Broad screening approach typically based on Target Compound List (TCL) Organics, Target Analyte List (TAL) Metals, and General Chemistry parameters	Focused approach typically based on Parent PAHs, Alkyl PAHs, PCB Congeners, Dioxins/Furans, Select Pesticides, Select Metals, Mercury, AVS-SEM, Total Organic Carbon (TOC), Black Carbon, Grain Size, etc.
Analytical Methods	Routine analytical approach with conventional method-defined reporting limits (that is, TCL SVOCs by GC/MS in full scan mode; PCBs aroclors by GC/ECD, etc.)	Specialized analytical approach with non-routine sample extraction, extract cleanup, and trace instrumentation to achieve low level reporting limits (that is, Alkyl PAHs by EPA GC/MS-SIM; PCB 209 congeners by HRGC/HRMS, etc.)
Sample Volume	Conventional sampling approach yields suitable volume/mass for method-defined reporting limits	Need to calculate sample mass/volume required for target RLs to meet project specific criteria; may be a challenge for biota and pore water matrices; limited volumes may increase RLs; prioritization may be required
Sample Handling	Conventional sample handling is suitable for standard analytical approach (that is, methoddefined reporting limits)	Customized laboratory sample handling and processing may be needed (such as glove box to maintain anaerobic conditions; centrifuge to generate pore water; etc.)
Sample Compositing / Homogenization	Conventional sample handling approaches are suitable (that is, method-defined approach, “as is” condition)	Specialized sample handling procedures, if needed, should be well documented in project work plans and during implementation
Reporting Limits	Routine analytical approach provides suitable reporting limits (that is, methoddefined approach)	Sediment programs often require specialized analytical approach to meet project specific criteria (such as ecological screening levels)
Contaminant Concentrations	High concentrations of target and/or nontarget analytes (relative to instrument calibration range) may require sample dilution which will elevate standard reporting limits potentially exceeding standard Regulatory Criteria	High concentrations of target and/or non-target analytes (relative to instrument calibration range and sensitivity) may require sample dilution which will elevate low level reporting limits potentially exceeding project specific criteria (such as ecological screening levels)
Moisture Levels	Solid data reported on a dry weight basis with results corrected for moisture content; typically low soil moisture levels (that is, 5-10 %) may not impact reporting limits relative to standard Regulatory Criteria	Solid data reported on a dry weight basis with results corrected for moisture content; elevated sediment moisture levels (that is, 30-70 + %) result in elevated reporting limits and lower extraction efficiencies
Organic Extraction	Routine extraction approach of isolate, extract, and concentrate target analytes of concern to exploit solubility difference offers suitable results (that is, conventional method-defined approach)	Sediment sample extraction may result in sensitivity and method performance issues as high organic content typical of sediment and biota matrices is extracted along with target analytes of concern (that is, “like dissolves like” plus 1000× concentration factor)
Mass Extracted	Standardized approach to sample aliquot (mass) extracted (that is, 5 grams), then concentrated to a method defined final extract volume (that is, conventional method-defined approach)	Sample mass may need to be increased and sample extract volume may need to be decreased to achieve targeted detection limits to meet project specific criteria
Interferences	Extraction step to concentrate target analytes will magnify interferences, which may require dilutions, which will elevate Reporting Limits, which may impact data usability relative to standard Regulatory Criteria	Extraction step to concentrate target analytes will also magnify non-target analytes and matrix interferences, which often requires dilutions, which will elevate Reporting Limits, which will impact data usability relative to project specific criteria (such as ecological screening levels)
Organic Extract Cleanups	Extract cleanups in soils are often not required due to limited matrix interferences (that is, conventional method-defined analytical approach is suitable)	Extract cleanups may be required to support low level analytical approach (such as gel permeation chromatography (GPC), silica gel, alumina, florisil, copper, sulfuric acid, etc.) to minimize interferences common in sediment and biota sample matrices
Organic Extract Concentration	Standard analytical approach is suitable (that is, conventional method-defined analytical approach)	Targeting lower final extract volumes can provide lower reporting limits for some but not all targeted parameters; alternate methods may be needed to achieve project specific criteria
Metals Digestion and Analysis	Standard analytical approach is suitable (that is, conventional method-defined analytical approach)	Specialized prep and analytical approaches may be needed (such as trace metals by ICP-MS, AVS-SEM, low level mercury, methylmercury, sea water prep for trace metals analysis by chelation extraction and hydride generation atomic fluorescence, etc.) to meet project specific criteria
Non-Detects	ND = not detected at Reporting Limit; viewed as “inconclusive”	ND may be viewed as inconclusive, or as = RL value, or as = ½ RL value (USEPA, 2015a (**13**); Rouhani and van Geel, 2017 (**19**))

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determined by USEPA Method 8270 may not be amenable to extract clean up techniques like other trace organic methods. Laboratories use USEPA Method 8270 in both full scan and selected ion monitoring (SIM) modes to maximize sensitivity for the full list of 70 SVOC analytes, 17 parent PAHs, or 34 Alkyl PAHs in sediment samples. USEPA Method 8272 or Test Method D7363 are used for determination of parent and alkylated PAHs in sediment pore water using solid phase microextraction (SPME) and GC/MS in SIM mode. Additional information is provided in Section 9 of this guide.

6.4.3.2 Parent PAHs are two or more fused conjugated rings. Alkylated PAHs are the parent PAHs with various alkyl groups attached. Alkylated PAHs are categorized by the total number of alkyl carbon atoms present. A homolog group includes all of the isomers with the same number of carbon atoms. Analysis of alkyl PAHs in combination with the parent compounds provides a more complete estimation of total PAH exposure. Thousands of alkylated forms of PAHs are unaccounted for in conventional analytical methods. Refer to Section 7 and Section 9 for additional information.

6.4.4 *Organochlorine Pesticides:*

6.4.4.1 Analysis of organochlorine pesticides for sediment programs typically uses USEPA Method 8081 by gas chromatography with electron capture detection (GC-ECD) instrumentation. However, the presence of high concentrations of PCBs in samples can potentially cause false positive pesticide detections with this analytical approach.

6.4.4.2 An alternative approach for determination of organochlorine pesticides which addresses sample matrix issues is USEPA Method 1699 using an HRGC/HRMS instrument.

6.4.5 *Hydrocarbons:*

6.4.5.1 The hydrocarbon category encompasses a wide range of petroleum and coal based hydrocarbon products with a variety of chemical analytical options that may be applicable to sediment programs.

6.4.5.2 A screening approach may begin with determination of total petroleum hydrocarbons (TPH) by USEPA Method 8015 modified using gas chromatography flame ionization detection (GC-FID) to determine *(a)* gasoline range organics (GRO) by a purge and trap sample introduction approach, and *(b)* diesel range organics (DRO) by an extraction approach.

6.4.5.3 Another analytical approach is based on separation of aliphatic and aromatic fractions, then quantitation of specific hydrocarbon ranges within each fraction using regulatory agency defined analytical methods. Analytical protocols vary for fractionated extractable hydrocarbon analysis.

6.4.5.4 Another analytical approach is based on a GC-FID hydrocarbon "fingerprint." Laboratory protocols based on USEPA Method 8015 vary for sample extraction, GC column types, temperature rates, chromatographic run-times, and instrument sensitivity. Differences occur between a high resolution output generated by a laboratory that specializes in hydrocarbon analysis, and a low resolution result with a shorter run time that relies on matching to known reference materials. Additional information is provided in Section 9 of this guide.

6.4.6 *Dioxins and Furans:*

6.4.6.1 Dioxins and furans are common names for polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF).

6.4.6.2 Analysis of dioxins and furans for sediment programs is typically based on USEPA Method 8290 or 1613 using gas chromatography / high resolution mass spectrometry (GC/HRMS) to determine seven (7) polychlorinated dibenzopdioxins and ten (10) polychlorinated dibenzofurans.

6.4.6.3 Dioxin/furan sample analytical results are reported along with toxicity equivalency factors (TEFs) relative to 2,3,7,8-TCDD using the World Health Organization (WHO) internationally accepted framework or other TEFs based on the site location or defined by the applicable regulatory program.

6.4.7 *Volatile Organic Compounds (VOCs):*

6.4.7.1 Determination of VOCs for sediment programs typically uses USEPA Method 8260 with purge and trap sample introduction and GC/MS instrumentation.

6.4.7.2 Regulatory entities may require solid VOC sample collection by USEPA Method 5035. However, this approach this presents challenges for sediment programs due to sample matrix issues such as elevated moisture levels and sample collection and handling issues. The laboratory should be consulted to evaluate sediment VOC sample collection and analysis options.

6.4.8 *Other Organics Analyses*—Other organic tests associated with a specific historic use of a chemical at a sediment site may be available upon special request. For example, laboratory analysis of butyl tins, an antifouling agent used in boat paint, requires development of specialized analytical protocols based on a literature review. Additional information is provided in Section 10 of this guide.

6.5 *Trace Metals, Inorganic, and Other Analysis:*

6.5.1 *Overview*—Trace metals and inorganic compound analytical methods consist of two separate stages: *(a)* the preparative stage to digest (isolate) and concentrate target analytes of interest, followed by *(b)* the determinative stage where the digested sample is analyzed for target analytes of interest by specific analytical instrumentation. The objective of the sample preparation step is to maximize the solubility of the analyte of interest, minimize potential interferences, and enhance the level of sensitivity (that is, Reporting Limit) required for the project. The analytical method determines the instrumentation used for an analytical parameter at the level of sensitivity needed to support the project. The analytical method reference for each of these steps should be documented in the QAPP. A general discussion follows for the most commonly used analytical methods to support sediment programs.

6.5.2 *Sample Preparation*—Sample preparation methods are selected based on analyte of interest and sample media and matrix types. Trace metals sample preparation by hot acid digestion is typically used for sediment and aqueous matrices. Biota matrices are subjected to a robust digestion approach to completely break down the organic matrix and solubilize the metals for instrument analysis.

6.5.3 *Specialized Preparation*—Determining trace metals concentrations in saline, brackish and sea water sample matrices presents special challenges, due to high levels of total dissolved solids present. These may interfere with trace metals

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analyses yielding false positive, false negative or biased results. These interferences may be minimized by sample dilution, with reporting limits elevated accordingly. Specialized sample preparatory protocols, including chelation extraction and hydride generation, may be used to mitigate saline sample matrix effects while providing the level of sensitivity needed to support sediment programs.

6.5.4 *Metals Instrument Analysis*—Trace metals analyses for sediment applications may specify USEPA Method 6020 using inductively coupled plasma / mass spectrometry (ICP/MS) with collision / reaction cell technology. Certain analytes have 'same mass' interferences, which in standard ICP/MS without the collision /reaction cell may result in false positives or biased results for trace metals analysis. Metals analysis can be supported by USEPA Method 6010 using inductively coupled plasma atomic emission spectroscopy (ICP/AES) for applications requiring less sensitivity.

6.5.5 *Mercury Analysis*—Mercury analysis for sediment programs is typically based on the cold vapor atomic absorption (CVAA) approach by USEPA Method 7471 with target sensitivity in the 3-30 µg/kg range. If lower sensitivity is required, USEPA Method 7474 or 1631 modified for sediment and biota can support target sensitivities in the 1-2 µg/kg range. USEPA Method 7473 Direct Mercury Analyzer is also used for biota analysis for target sensitivity in the 20 µg/kg range.

6.5.6 *Methyl Mercury*—Methyl mercury is determined by USEPA Method 1630 with modified sample preparation procedures for sediment and biota matrices for targeted level of sensitivity of 0.1 µg/kg.

6.5.7 *Acid Volatile Sulfide / Simultaneously Extracted Metals (AVS/SEM)*—Measurements of AVS/SEM in sediments can be used to evaluate the toxicity of metals to indigenous benthic organisms. AVS is volatilized from sediments by the addition of acid. SEM are metals simultaneously extracted during the AVS procedure. Toxicity is evaluated by calculating the sum of the SEM (µmol/g dry weight) divided by the AVS concentration in the same units: a ratio less than or equal to 1 indicates that the sediment metals are not bioavailable while a ratio > 1 would indicate that the metals may be bioavailable. SEM metals typically include cadmium, copper, lead, mercury, nickel and zinc, but it should be recognized that other binding compounds (for example, iron and manganese oxyhydroxides) can also influence bioavailability. Method guidance (USEPA, 1991, 1992) (**25**, **26**) is predicated on maintaining anoxic conditions required to sustain metal sulfide concentrations. Exposure of sediment samples to oxygen during sample collection and analysis may result in false positive assertions of metal bioavailability. This method is written as guidance with ongoing research documenting the impacts of sample handling on the representativeness of this procedure. Sample collection and sample handling protocols should be discussed with the laboratory, as well as laboratory protocols for maximizing representativeness.

6.5.8 *Organic Carbon*—Organic carbon has been shown to influence contaminant bioavailability. Organic Carbon Methods include determination of: *(a)* Total Organic Carbon (TOC), *(b)* Black Carbon (also called Soot), *(c)* Particulate Organic Carbon (POC), and (c) Fraction Organic Carbon (FOC). Total organic carbon (TOC) analysis is conducted by USEPA Method 9060 in water, while the USEPA Lloyd Kahn protocol is used for sediment (USEPA, 1988) (**27**). Black carbon analysis has no published USEPA methods, thus laboratories follow procedures based on the literature. Laboratories typically perform both TOC and black carbon analysis in duplicate (at a minimum) due to the inherent variability due to small sample aliquots [>50 mg] utilized. On request, laboratories can perform these analyses in triplicate or quadruplicate to improve the level of certainty. There is no accepted definition of or published analytical method for fraction of organic carbon (FOC), referred to as the portion of the organic matter available to adsorb organic contaminants of concern. In general, the higher the organic carbon content, the more organic chemicals may be adsorbed to solids and the less of those chemicals will be available to leach to water. Laboratories rely on the literature for method protocol references.

6.5.9 *Lipids Determination*—Percent lipids analysis is often requested in order to normalize the results of lipophilic contaminants present in biota samples. There are no published analytical methods for lipids determination, so discussions with the laboratory to agree on suitable method references to meet project objectives should be part of the project planning process.

7. Passive Sampling Methods

7.1 The freely dissolved concentration (C_{free}) of chemicals in sediment porewater is the driving force for transport to the water column and bioaccumulation in benthic organisms. The freely dissolved chemicals in the surface water control chemical uptake by the pelagic organisms, such as phytoplankton and zooplankton, as well as higher trophic level organisms such as fish (fish uptake is controlled by both trophic transfer from prey and C_{free}). Potential risks of adverse biological effects from chemicals are most directly related to a chemical's C_{free}, which can be measured through passive sampling methods (PSM). Passive sampling measurements can be used for various purposes: to better understand contaminant bioavailability and associated risk; inform design specifications of a remediation technology; and to monitor the efficacy of a remedy. For example, understanding the concentration of contaminants available to benthic organisms can inform the assessment of benthic bioaccumulation and benthic toxicity, which ultimately supports improved risk assessments and the development of more accurate sediment quality evaluations. Also, measurements of the C_{free} in a sediment cap provides chemical gradient concentrations from the porewater to the surface water, which can be useful in evaluating the effectiveness of a sediment cap.

7.2 The use of PSM provides the following benefits:

7.2.1 Measures the C_{free} in porewater and surface water, which provides a more relevant measurement of exposure for evaluating bioaccumulation and risk as compared to traditional methods that are based on total extraction.

7.2.2 Can provide a time-averaged response.

7.2.3 Regulators are increasingly accepting the use of PSMs as a reliable and accurate tool for measuring C_{free}.

7.3 The following drawbacks of PSM should be considered:

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7.3.1 Trained personnel are needed for the preparation, deployment and retrieval of the samplers.

7.3.2 Obtaining results through PSM can take more time than standard sampling methods, because the passive sampler needs to be in contact with the environmental media until equilibrium or partial equilibrium conditions are achieved. Sampler preparation, if performance reference compounds (PRCs) are used, added effort is required for sampler preparation and data analysis to interpret results.

7.3.3 There may be additional costs associated with the use of PRCs, especially when using deuterated and ^{13}C labelled compounds (see 7.5 and 7.5.8).

7.3.4 Proper calibration and validation of PSM are needed, including values for polymer-water partition coefficients (K_{PS}), determination of equilibrium, and non-depletive sampling conditions.

7.4 The selection of the passive sampler material (that is, the polymer) and the selection between *in situ* and *ex situ* deployments are dependent on several site-specific goals and considerations as well as the target contaminant of interest. These key considerations and the associated sampling requirements are further discussed in 7.5 for organic constituents and 7.6 for inorganic constituents. Appendix X7 Quick Reference Guide for Passive Sampling with a matrix of potential polymer types for different chemicals of interest; and Appendix X8 Summary of Advantages and Limitations of Passive Sampler Types for Organics; and Appendix X9 Methodologies and Equations for Determining C_{free} from Passive Sampler Results. A list of References and a Bibliography are provided at the end of this guide.

7.5 *Organic Constituents:*

7.5.1 The C_{free} of a hydrophobic organic constituent (HOC) can be estimated with passive samplers by two operational methods: *(1)* an equilibrium method, where sufficient time is allowed for the target analyte to reach equilibrium between the sampler and water or sediment, and *(2)* a pre-equilibrium method that targets a time-specific concentration that must be corrected to equilibrium condition (Lydy et al. 2014, Mayer et al. 2014) (**28, 29**).

7.5.2 It is important to understand how rapidly the target contaminant(s) reaches equilibrium with the passive sampler, sediments, and other environmental media. This kinetic state depends on exposure time, passive sampler characteristics, such as construction material, thickness, and dimensions, and the target contaminant's physicochemical properties (Vrana et al. 2005, Apell et al. 2015) (**30, 31**). Passive samplers with polymer thickness of a few micrometers are available for equilibrium sampling of the most hydrophobic chemicals within a practical timeframe. If equilibrium is not expected to be achieved during the sampling time span, passive samplers are "spiked" with known concentrations of PRCs prior to deployment. PRCs are analytically noninterfering chemicals that are embedded in the passive sampler before environmental exposure and *(1)* allow precise measurement of its loss, *(2)* follow the same kinetics as the target analyte, and *(3)* do not exist in the target environment (Ghosh et al. 2014) (**32**). The depletion rate of a PRC during sampler deployment reflects the uptake rates of a contaminant, assuming isotropic exchange kinetics occur (Ghosh et al. 2014) (**32**). In principle, PRCs experience the same mass transfer limitations while diffusing out of the sampler as the target analytes that are diffusing into the sampler, therefore the measured losses of the PRCs can be used to infer the level of equilibrium of the target analyte, as discussed in more detail in Section 7.5.8 (Fernandez et al. 2009, Gschwend 2010, Apell and Gschwend, 2014) (**33-35**).

7.5.3 *Types of Organic Passive Samplers:*

7.5.3.1 The three types of passive sampler polymers typically used for estimating the C_{free} of HOCs include polyoxymethylene (POM), polyethylene (PE), and the silicone polydimethylsiloxane (PDMS). Each of these passive samplers can be deployed in the field and laboratory. PE and POM are deployed as sheets, can easily be deployed in both surface water and sediment, and if chosen with high surface area to volume ratio, have the advantage of lower analytical detection limits. PDMS typically serves as the passive sampler polymer used on Solid Phase Micro Extraction (SPME) fibers. SPMEs are fiber-optic cables with an inner core of glass, which is insulated by PDMS. The PDMS coating can vary in thickness (typically 10 to 100 µm) and the length of coated fiber is adjustable. SPMEs are more suitable for sediment deployment, rather than surface water, due to limited polymer mass on the fiber. Also, the fibers are fragile, and need to be protected during deployment in sediment.

7.5.3.2 The selection of the appropriate passive sampler material should align with the overall assessment objectives, after taking into consideration pros and cons of each polymer (that is, sampler performance, cost, durability, required detection limits, polymer-water partition, sampler fouling, material availability, etc.), as outlined in Gschwend et al. 2011 (**36**), Ghosh et al. 2014 (**32**), USEPA 2012a (**37**), USEPA et al. 2017 (**38**), and summarized in Appendix X8. While K_{PS} have not been determined for all organic compounds and for all passive sampler material, the list of K_{PS} continues to expand (USEPA 2012a (**37**), USEPA 2012b (**39**); Ghosh et al. 2014 (**32**), USEPA et al. 2017 (**38**)). This matter can inform a decision on selecting the appropriate passive sampler or the need for determining K_{PS} for a specific study.

7.5.4 *Pre-Deployment:*

7.5.4.1 Prior to sampler exposure, it is necessary to pre-extract the passive sampler device to confirm it is free of background contamination. The selection of pre-extraction solvents varies by material type and target analytes (Ghosh et al. 2014) (**32**). If the sampling program has decided to use PRCs to account for nonequilibrium conditions, these compounds must be loaded into the samplers prior to deployment. This process includes soaking the sampler in a volume of water or a methanol water solution that has been "spiked" with the PRCs and allowing for adequate equilibration time (Booij et al. 2002 (**40**), Fernandez et al. 2009 (**33**), USEPA et al. 2017 (**38**)). Equilibration times are sampler and analyte dependent, but typically range from 7 to 30 days. Following cleaning and PRC loading (if applicable), care should be taken to avoid exposing samplers to possible sources of contamination by wrapping the samplers in clean aluminium foil, placing in a sealable plastic bag and storing frozen (-4 °C) until deployment (USEPA 2012a) (**37**). It is important to retain a dose of replicate

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samplers to serve as an initial PRC concentration (C_{PRCi}) for completing later calculations.

7.5.5 *Deployment:*

7.5.5.1 Passive samplers can be deployed both in the field (*in situ*, water column or sediment) or used in the laboratory with field-collected samples (*ex situ*). Each deployment method has its own advantages and disadvantages as further discussed below and in other documents (USEPA 2012a (**37**), Ghosh et al. 2014 (**32**), USEPA, 2016 / Thompson et al. 2016 (**41**), Schmidt et al. 2017 (**42**), USEPA et al. 2017 (**38**)). Overall, the selection of *ex situ* versus *in situ* deployment depends on the objectives of the assessment weighed against logistical and financial constraints. Certain situations, such as the evaluation of the performance of in-place remedial alternatives (that is, capping and treatment remedies), will require *in situ* measurements to accurately capture the full suite of site specific processes while other objectives (that is, bench scale comparison of treatment alternatives) can be addressed with *ex situ* methods (Thompson 2016) (**43**).

7.5.5.2 Benefits of *ex situ* methods include being less labor intensive, less expensive, and providing more ability to control environmental variables (that is, temperature, light, addition of biocides, etc.) compared to *in situ* methods. *Ex situ* sediment exposures are performed in closed systems in a controlled environment, which typically require little or no sampler protection. *Ex situ* deployments of polymer sheets (PE and POM) can be added directly to these systems. During *ex situ* equilibration, the sediment can be agitated to enhance the exchange of chemicals between the sediment and sampler, greatly reducing the time required to reach equilibrium, while avoiding the need for PRCs (Ghosh et al 2014) (**32**). Equilibration times are dictated by target contaminants and polymer characteristics, such as material and surface area to volume ratio. Equilibrium can typically be achieved within 1-4 weeks (Mayer et al. 2003 (**44**), Ghosh et al. 2014 (**32**)), after which the samplers are recovered. A disadvantage of *ex situ* deployments is that *ex situ* conditions may not reflect site-specific conditions at contaminated sites (USEPA 2012a) (**37**). When conducting *ex situ* sediment exposures appropriate ratios of polymer mass to sediment organic carbon mass (1:100) should be used so that the relative amount of the target constituent transferred from the sediment to the sampler results in a negligible loss (<1 % depletion) (USEPA et al. 2017) (**38**).

7.5.5.3 *In situ* methods generally result in a better characterization of true field exposures. This approach may be preferable when it is important to capture local site processes, such as groundwater intrusion, currents, bioturbation, vertical contaminant porewater concentration profiles, and sediment-water column gradients and fluxes (Ghosh et al. 2014) (**32**). The success of an *in situ* passive sampling program depends in large part on the success of deployment and retrieval of the samples, which may require an experienced diver depending on the depth of the water column, the desired depth of the sampler in the sediment bed, and the required accuracy of the placement of the sampler (Thompson et al. 2016) (**43**). During *in situ* deployment in water and sediment, polymer sheets are typically attached to rigid, metal (that is, aluminium, stainless steel) frames or in metallic mesh (that is, aluminium, copper, stainless steel) that is suited to fully expose the sampler surface to its environmental surroundings while protecting it from damage (USEPA et al. 2017) (**38**). The thinner SPME fibers are not as robust as the relatively simple passive sampling polymer sheets and are often deployed in sediment *in situ* and *ex situ* in a protected form (that is, metal mesh, copper or stainless steel sheath or tubing) to avoid loss or breakage (USEPA et al. 2017) (**38**). Shielded, modified push point type samplers with perforations are especially useful for housing SPME fibers and providing sufficient protection for insertion into sediments (Reible and Lotufo 2012) (**45**). Depending on site conditions, such as deep water settings, a diver may be required for the insertion of the sampler into the sediment and for retrieval at the end of the deployment period. The GPS location of each sampler needs to be carefully recorded and marked with a buoy or other readily located marker.

7.5.5.4 Generally, *in situ* samplers are left in place for a week up to a month or more. It is important to choose an equilibrium time that is a balance between using short times, to minimize sample disturbance or vandalism, and the time required to achieve a significant fraction of equilibrium for highly hydrophobic contaminants (USEPA et al. 2017) (**38**). This balance often means that chemical equilibrium is not reached for all target analytes, requiring the use of PRCs. The use of PRCs adds additional effort and potential for additional errors that affect both the precision and accuracy of derived C_{free} values (Mayer et al 2014) (**29**). Additionally, the use of *in situ* deployments requires adequate spatial coverage to address the extent and heterogeneity of contamination, given that these devices sample microscale environments and chemical concentrations in sediments are generally not homogeneous (Ghosh et al. 2014) (**32**).

7.5.6 *Retrieval:*

7.5.6.1 After deploying the *in situ* passive samplers for an appropriate length of time, they are then removed from the environment being sampled. Recovery will require similar personnel as deployment activities (that is, if divers were needed for deployment, they will generally be required for retrieval as well). Following the retrieval of the sampler and support device, it is important to remove adhering sediment or surface growths with water or damp laboratory tissues (do not use solvents to clean), as these materials can interfere with analytical procedures and data analysis (USEPA et al. 2017) (**38**). Once the sampler is clean, it can be processed as defined in the assessment plan (that is, sectioned to evaluate depth profiles) and placed in clean glass vials in a freezer at –4 °C, in the dark, until they are analyzed (USEPA et al. 2017) (**38**).

7.5.6.2 Processing of passive sampler devices should be performed as quickly as possible following retrieval to minimize loss of more volatile analytes (that is, naphthalene and other low molecular weight compounds) during exposure to air (Thomas et al. 2014 (**46**), USEPA et al. 2017 (**38**)). PDMS samplers have been extensively used due to their rapid uptake kinetics, but these properties also lead to faster loss of volatile analytes (Thomas et al. 2014) (**46**).

7.5.7 *Quality Assurance and Quality Control (QA/QC):*

7.5.7.1 Appropriate QA/QC guidelines should be followed when implementing a passive sampler monitoring program.

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Guidelines specific to passive samplers are described in Ghosh et al. (2014) (**32**) and USEPA et al. (2017) (**38**). At a minimum, blanks (trip blanks and field blanks) are an important measure to implement to account for residual contamination in sampler or storage materials as well as contamination introduced to the sampler from ambient site air. Proper replication of blanks will allow for reliable corrections of sampler data. Additionally, replication of deployed passive samplers at select locations can help the data user understand field specific variability. Additional considerations for passive sampler specific QA/QC measures (that is, field solvent blanks, field control samplers, field internal standards, etc.) are discussed in USEPA et al. 2017 (**38**). In addition, time series measurements or parallel sampling with multiple coating thicknesses can be used as an approach to confirm the attainment of equilibrium and the absence of sampling artefacts as part of the passive sampling measurement (Mayer et al. 2003 (**44**), Jahnke et al. 2012 (**47**), Schmidt et al. 2017 (**42**)). The quality of chemical analytical methods should be quantified by using internal calibration standards (Ghosh et al. 2014) (**32**).

7.5.8 *Data Conversions and Corrections for Field Conditions and Site-Specific Equilibrium Levels:*

7.5.8.1 Results from the passive samplers typically provide two basic types of information: The concentrations of target analytes in the passive sampler (C_{PS}) and 2) C_{free} of target analytes in the aqueous phase around the sampler (USEPA 2012a). The C_{free} in the aqueous phase around passive samplers can be calculated based on the amount of contaminant per mass or volume of passive samplers (C_{PS}). Therefore, it is very important to measure the sampler mass or dimensions accurately. The steps to converting the C_{PS} to C_{free} will depend on whether equilibrium or non-equilibrium conditions were attained. When equilibrium has been achieved and confirmed, C_{free} can be obtained as ratio of C_{PS} and appropriate K_{PS} value.

7.5.8.2 To correct for non-equilibrium conditions, the use of PRCs can be used to quantify the level of equilibrium reached). Correction approaches for non-equilibrium using PRCs are based on the first order exchange kinetics and Fickian diffusion (Huckins et al. 2006 (**48**), Fernandez et al. 2009 (**33**)). The methodologies and equations for determining the C_{free} in the aqueous phase when PRCs are used, are presented in Appendix X9.

7.5.8.3 The methodologies and equations for calculating C_{free} in the aqueous phase based on concentrations in the sampler are provided in Appendix X9 for both equilibrium and pre-equilibrium cases.

7.6 *Inorganic Constituents:*

7.6.1 This section describes the passive samplers typically used for estimating C_{free} of inorganic constituents, including dialysis samplers, diffusive gradient in thin flims (DGT) and diffusive equilibrium thin films (DET). In addition to discussion below, Appendix X7, the quick reference guide for passive sampling, also provides a matrix of samplers types for inorganic constituents.

7.6.2 *Dialysis Samplers:*

7.6.2.1 Dialysis samplers, commonly called peepers, are one of the original passive sampling techniques that are used to obtain samples for analysis of sediment porewater concentrations (Hesslein 1976, 1977) (**49, 50**). Dialysis samplers rely on the equilibration of a compartmentalized volume of pure water with the constituents in the surrounding water (water column or sediment interstitial water) via diffusion of the dissolved constituents across a semi-permeable dialysis membrane.

7.6.2.2 The most common application of dialysis samplers is for collection of porewater samples for analysis of inorganic constituents. Of particular interest are porewater trace metals and other inorganic constituents (and some organic constituents) that may have a bearing on metal bioavailability to benthic organisms exposed via sediment porewater. The total porewater concentration may consist of a variety of forms, including ionic C_{free}, inorganic and organic complexes, and a fraction bound to colloidal organic matter that may be present in the porewater (C_{doc}). The degree to which a particular type of dialysis sampler measures each of these forms will be design-specific.

7.6.2.3 The use of peepers requires consideration of a number of important factors. These include but are not limited to equilibration time, sample volume requirement, type of membrane used, whether oxidation reduction conditions affect the constituents, and sample resolution. In view of these considerations the specific procedure to be followed will be design- and site-dependent. Several of these considerations are discussed briefly below, with further details provided in the cited references.

7.6.2.4 *Commercial/Customized Dialysis Samplers:*

(1) Numerous designs of dialysis samplers have been used to measure sediment porewater concentrations. Three fundamentally different designs will be described to illustrate some of the advantages and disadvantages of each. They are the classic multi-cell - multi-level peeper (for example, Hesslein 1976, 1977; Carignan et al. 1985) (**49-51**), a two-sided peeper with several cells at each vertical sampling horizon (Brandl and Hanselmann, 1991) (**52**), and a relatively simple single-cell peeper (Brumbaugh et al., 2009, 2013) (**53, 54**). The first two designs are appropriate for obtaining high resolution measurements of vertical concentration gradients in sediments, while the third is amenable for use during laboratory toxicity and bioaccumulation tests with homogenized sediment samples. Some standard design peepers may be commercially available, fabricated in-house without a need for specialized equipment, or custom designed, and ordered to specification.

(2) The classic multi-cell high resolution design consists of a rectangular sheet of acrylic (for example, Plexiglas or Lucite) having a series of parallel chambers (peeper cells) machined into one side at a uniform spacing. Prior to use, the compartments are filled with deoxygenated deionized water, covered with the dialysis membrane, and a cover plate is securely fastened to the body with nylon machine screws. The peeper is deployed by insertion vertically into the sediment and left to equilibrate for the desired period of time. Upon retrieval it is rinsed and the cells are sampled through the membrane via a syringe.

(3) The second design is a variation of the Hesslein design. In this instance, cylindrical holes are drilled completely through a faceplate-body-faceplate assembly. There is a membrane and faceplate on each side of the acrylic body, enabling

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the membrane surface area available for transfer into the peeper cell. The other notable design difference is that multiple cells are drilled into the body at each depth horizon, allowing for multiple discrete samples to be collected at each vertical horizon. The design is in other respects comparable to the multi-cell design described above.

(4) The third type of peeper has a relatively simple single-cell design that is well-suited for use in either the laboratory or field. Brumbaugh (2009) (**53**) describes two sizes that have been developed for use in the laboratory (2.9 mL, or 2.5 mL nominal) or in the field (50 mL). The smaller of these (the "mini-peeper") is well suited for burial in a homogenized sediment sample to monitor porewater chemistry during a sediment toxicity test (Brumbaugh, 2009; Brumbaugh et al., 2013) (**53, 54**). The mini-peepers are made from acid-cleaned, 2.9 mL polyethylene snap-cap vials (1.2-cm inner diameter) with a 6-mm diameter hole drilled through the center of the cap. Appropriately cleaned vials are submerged in deoxygenated deionized water and a 25-mm diameter polyether-sulfone (PES) membrane (0.45 µm pore size) is positioned over the opening and the perforated cap is then closed to seal the vial and secure the membrane in place. Further details regarding the design and implementation of this device are available in Brumbaugh (2009) and Brumbaugh et al. (2013) (**53, 54**). Another alternative design that is also appropriate for use in monitoring sediment toxicity tests is the multi-celled mini-peeper described by Doig and Liber (2000) (**55**). Note that for any of the preceding designs, deployment duration should be sufficient to approach equilibrium conditions, or steps should be taken to adjust for incomplete equilibration (see Section 7.6.2.8).

7.6.2.5 *Pre-Deployment and Equipment Preparation:*

(1) It is imperative that peepers be properly prepared prior to deployment to prevent cross-contamination of trace metals and to remove any source of oxygen that might alter the redox state in the sediment (Carignan et al., 1994; Teasdale et al., 1995; Peijnenburg et al., 2014) (**56-58**). The peeper body and cover plate should first be cleaned with a detergent and well rinsed with deionized water to remove any residual oil or grease that may have originated during fabrication. They should then be bathed in a nitric acid solution for at least two days to remove any trace metals. Finally, the equipment should be thoroughly rinsed with deionized water and deoxygenated by placement in a water bath through which N_2 gas is bubbled.

(2) Peepers are often fabricated from polymethacrylate (such as Acrylic, Plexiglas) because of its low cost, durability and workability. However, Carignan et al. (1994) (**56**) has shown that these and other plastics may store significant amounts of oxygen which may alter *in situ* porewater concentrations that are measured in anoxic sediments. High density polyethylene (HDPE) and polyvinylidene difluoride (PVDF) were found to be most suitable for use, primarily due to the low solubility of oxygen in those materials. Regardless of material used, it was recommended that peeper preconditioning include at least a 30-day immersion in deionized water that is continuously purged with N_2 gas prior to assembly of the peeper. This should be sufficient to reduce the stored oxygen (5.7 day half-life for release) to levels that avoid problems due to oxygen. Finally, following final assembly, and just prior to deployment an additional 48 hour immersion is recommended (Carignan et al., 1994; Teasdale et al., 1995) (**56, 57**).

7.6.2.6 *Deployment:*

(1) Dialysis samplers can be deployed either *in situ* (field deployments) or *ex situ* (laboratory deployments) to sample the water column, sediment interstitial water, or both. *In situ* methods, when properly applied, are generally considered to provide the most direct measure of exposure to porewater constituents under field conditions. *In situ* measurements will also properly reflect factors that might lead to a non-equilibrium condition between the sediment and the porewater, as might occur if groundwater seepage is ongoing at a site. *Ex situ* laboratory measurements are often performed to characterize porewater exposure concentrations during a toxicity or bioaccumulation test. Laboratory measurements of porewater concentrations may not be representative of field concentrations, but *ex situ* measurements are thought to provide a more direct characterization of exposure conditions to benthic organisms as compared to *in situ* measurements.

(2) Deployment times for *ex situ* and *in situ* samplers for inorganics may range from a few days to a few weeks or longer, depending on the type of device used. Several methods have been proposed to predict the equilibration time needed during deployment and to provide a correction to adjust the results when equilibrium has not been achieved (Brandl and Hanselmann, 1991 (**52**); Holcombe et al., 2001 (**59**); Thomas and Arthur, 2010 (**60**)). (Further details are provided in Appendix X9 of this guide.)

(3) In deep water settings, a diver will be required for both the initial insertion into the sediment and for retrieval at the end of the deployment period. The devices should be located in areas where the potential for disturbance is minimized and, if possible, in proximity to a physical landmark. The GPS location of each sampler needs to be carefully recorded and marked with a buoy or other readily located marker.

7.6.2.7 *Recovery / Retrieval:*

(1) The exact timing of retrieval is not likely to be a critical factor as estimates of equilibration time are not precise, and corrections for this can often be made to reflect the actual deployment duration. The devices should be removed from the sediment with reasonable care, using divers as required, to limit disturbance of the sediment, and to ensure that the device is not damaged, particularly the exposed membranes. Once removed from the water the peeper should immediately be rinsed with DI water to remove any particles adhering to the surface. The sample water should be removed from each peeper cell using a pipette tip (or syringe) to pierce the membrane. The sample water should immediately be transferred to a previously labelled, clean plastic vial or other suitable container and capped. Exposure to the atmosphere should be minimized as much as possible. The samples should then be preserved and stored on ice until they are analyzed.

7.6.2.8 *Quality Assurance and Quality Control (QA/QC):*

(1) QA/QC guidelines that are specific for dialysis samplers typically include use of trip blanks and field blanks, with replicates, to quantify the degree of residual contamination that may be present. Replicate devices should be deployed at a

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subset of the sample locations. This will provide an indication of sampling variability. If the peeper design includes multiple cells at a fixed depth, a similar form of replication can be achieved by repeating the same analyses on subsets of samples from the same sampling horizon.

(2) If the peeper deployment duration was sufficient for equilibrium conditions to have been achieved, then concentrations measured on the peeper cell water can be used directly. If deployment times were not considered to be sufficient to achieve equilibrium conditions, then either of two approaches should be considered for making a correction. One approach for adjusting measured results to equilibrium conditions is to fit time series data with a 2-parameter kinetic equation (Brandl and Hanselmann, 1991) (**52**). An alternative approach for correcting for non-equilibrium in the peeper cells is to use one or more performance reference compounds, or PRCs (Thomas & Arthur, 2010) (**60**). These approaches are explained in further detail in Appendix X9.

7.6.3 *Diffusive Gradient and Diffusive Equilibrium in Thin Film:*

7.6.3.1 Diffusive gradient in gel thin-film (DGT) passive samplers are used to measure chemical porewater concentrations based on chemical diffusion into the sampler and the measured flux over the sampling period. DGT samplers are used to measure cations, including trace metals. Common target analytes include arsenic, cadmium, calcium, copper, lead, iron, nickel, and zinc, but these samplers have been used for dozens of cations.

7.6.3.2 The DGT sampler consists of a resin gel, a diffusion gel, and a filter. The diffusion rate into the sampler is controlled by the diffusion gel. The resin gel can hold a much larger amount of analyte than is contained in the surrounding porewater. The porewater concentration can be calculated from the mass accumulated, the diffusion length, the diffusion coefficient, the area exposed, and the time exposed. The diffusion length and area exposed are physical parameters of the samplers, while the diffusion coefficient is a chemical parameter of the target analyte. The diffusion coefficient through water can be used as an approximation or the site specific diffusion coefficient can be found experimentally (Chess 2010) (**61**).

7.6.3.3 DGT samplers rely on several assumptions to calculate the concentration of the analyte being measured. The main assumptions include the following:

(1) The geometric values of exposure area and diffusion length are well known,

(2) Analyte interaction with the diffuse layer and filter are negligible,

(3) Analyte binds to the resin layer at the surface instantaneously,

(4) Time to steady-state diffusion is negligible relative to deployment time,

(5) Colloid-associated analyte species contribute negligibly to DGT uptake (Davison and Zhang 2012) (**62**).

7.6.3.4 The kinetics of the analyte binding with the resin layer is relatively fast, on the order of minutes, and is negligible for deployment times greater than two hours. The assumption that there is negligible impact on DGT uptake by colloid-bound species is not completely clear, nor is the effect of solution speciation on DGT uptake. If analyte complexes readily disassociate or have the same diffusion coefficient as the freely dissolved species, they will be taken up by the DGT samplers and included in the measured concentration. These complexes are considered labile. For example, metal complexes with inorganics such as carbonates, hydroxides, sulfates, and chlorides all follow this behavior (Davison and Zhang 2012) (**62**). However, the interaction between DGT samplers and metals complexed with organic ligands is more complicated. The total analyte uptake will be the sum of all diffusive fluxes (Zhang and Davison 2000) (**63**), but not all complexes are either fully labile or non-labile. These semi-labile complexes will partially disassociate and contribute to the diffusive uptake into the sampler, but not all of the complex will be available. The partial disassociation can be modelled, but requires a complete analysis of complexing ligands for an accurate calculation. Semi-labile complexes tend to disassociate more within the diffusion gel than they do in solution, which could lead to overestimation of solution concentrations (Mongin et al. 2011) (**64**).

7.6.3.5 Diffusive Equilibrium in Thin Film (DET) is a similar passive sampling technique which measures porewater concentration by coming into equilibrium with the porewater as opposed to the flux measurement of a DGT. The DET samplers consist of a similar resin gel to DGT but without a diffuse gel. The resin gel comes into equilibrium with the porewater and the known partitioning coefficient is used to calculate that concentration. These samplers do not concentrate analytes in the same way as DGT and they are not as useful for trace metal sampling. Instead, they are more commonly used for higher concentration analytes such as iron and manganese.

7.6.3.6 *Types of DGT and DET Samplers:*

(1) DGT and DET samplers are categorized by the target analyte to be measured and by the sampling matrix. For a target analyte, the resin gel must strongly adsorb the analyte. The type of resin gel used will depend on the analyte of interest. For DGT, the diffuse resin must not strongly interact with the analyte. Two sampler designs are available for sediment porewater. The first sampler design is long enough to give a depth profile of porewater concentrations in the sediment, typically 10-15 cm. The second sampler design is a smaller sampler that provides a concentration for one depth, typically the surface sediment. The second sampler is also used for surface water measurements. The physical design of the DGT or DET samplers can be changed to match sampling needs. DGT and DET passive samplers are not yet widely used outside of research applications. Samplers and resin gels are available for purchase from very few commercial sources, and samplers for all analytes are not available.

7.6.3.7 *Pre-Deployment and Equipment Preparation:*

(1) It is imperative that DGT and DET be properly prepared prior to deployment to ensure that any source of oxygen that might alter the redox state in the sediment be removed. Prior to use, the DGT samplers are de-oxygenated in 10 mmol/L sodium nitrate with bubbling N_2 gas for 12-24 hours. This step removes oxygen from the samplers which can be introduced into the sediment when the DGT samplers are

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deployed. The sodium nitrate is necessary to raise the ionic strength of the diffusion gel and increases the reproducibility of metal ion diffusion (Zhang 1999) (**65**). DGT samplers should not be exposed to oxygen prior to deployment. If the DGT samplers need to be transported prior to deployment, the samplers should be transferred to double-bagged plastic bags in an anoxic atmosphere. The de-oxygenation step should be completed as close to deployment as possible as oxygen can diffuse back into the samplers over time. DET samplers should also be de-oxygenated with N_2 gas for 12-24 hours prior to deployment. However, since DET samplers have no diffusion gel, it is not necessary for the sodium nitrate to be added to the de-oxygenation solution.

7.6.3.8 *Deployment:*

(1) The samplers are then deployed in the media to be measured. If they are being deployed in water, the samplers must be in the liquid and have the gel face exposed. In laboratory experiments, slow mixing is recommended to reduce any diffusion boundary layers at the surface of the DGT samplers. For sediment, the samplers are pushed into the sediment. Piston samplers are placed at the surface of the sediment and can be sealed with putty to ensure that the gel face remains in contact with the porewater.

(2) The DGT samplers are left exposed in the media for a known amount of time. In sediments, this time is typically 1-7 days and for water samples this time is typically 1-21 days. The deployment time will vary depending on the range of concentrations that are being measured. Lower concentrations require a longer deployment to allow the samplers to accumulate a measurable amount of analyte. Higher concentrations should not be deployed too long, as the sampler can accumulate enough analyte to disrupt the linear concentration gradient across the gel causing non-linear uptake. In addition, in mixinglimited systems, long deployment times can deplete concentrations in the vicinity of the sampler and limit the applicability of a constant flux in the analysis. When the DGT samplers are removed, they should be thoroughly rinsed with deionized water. For sediment sampling, all residual solids need to be cleaned from the exterior of the sampler as they can contaminate the gels during processing.

(3) DET samplers are deployed in the same manner for water and sediments. However, since the DET samplers are equilibrium samplers, they should be deployed for longer times. The deployment times vary depending on the system and analyte to be measured.

7.6.3.9 *Recovery / Retrieval:*

(1) The DGT and DET samplers can be processed in similar manners. The gels are cut out from the sampler body using a TFE-fluorocarbon coated razor blade. For DGT, the filter and diffusion gel can be discarded. If any solids penetrated the filter layer and make direct contact with the resin gel, the resin gel should be discarded. Direct contact with the sediment will contaminate the resin gel and result in overestimation of porewater concentrations. For both DGT and DET, the resin gel is then sectioned. For depth profiler samplers, the resin gel is sectioned over depth to give porewater concentrations by depth. The depth profilers are typically sectioned at 0.5 or 1.0 cm resolution but sections have been taken as low as 0.1 cm. As long as there is a detectable amount of analyte in each section, the area chosen will not affect the estimated porewater concentration.

(2) For elution, each resin section is eluted in 3 mL of concentrated trace-metal grade hydrochloric or nitric acid for 24 hours. Resin gels should not be left in the acid for longer than 24 hours as the gel will break down and can interfere with analysis. Once the acid is removed from the gel, it is ready for analysis. A subsample of the acid is diluted for analysis and digested for at least 24 hours. The samples are then analyzed according to standard analytical methods, such as USEPA Method 1631 Revision E. The volume of acid used to dilute should be chosen in order for the samples to fall within the calibration range of the analytical instrument. The samples should then be preserved and stored on ice until they are analyzed.

7.6.3.10 *Quality Assurance and Quality Control (QA/QC):*

(1) It is important to analyze quality control checks during use of both DGT and DET samplers. There are several types of QC samples that are run with every sampler batch. The first sample is a laboratory blank. A laboratory blank is a check on the contamination mass contained in the resin before it is assembled into a sampler. Contamination in the resin can come from chemical contamination, especially the thiol resin beads, glassware, or storage solutions. A laboratory blank is taken from each set of resin gels cast for a sampling event. The second QC sample is a de-oxygenation blank. The de-oxygenation blank is taken after the samplers are de-oxygenated, before they are taken into the field. The deoxygenation solution may contain a small amount of the target analyte from the ambient atmosphere and chemicals used, and samplers may accumulate a small amount of this analyte during the deoxygenation process. The de-oxygenation blank will check that this contamination is not significant compared to the analyte accumulated during sampling. Field blanks are also taken as a QC sample. The field blanks are samplers which are de-oxygenated and then transported along with all deployed samplers. The field blanks will measure any contamination accumulated during transportation, storage, and field use of the samplers.

(2) The detection limit is an important factor when considering what sampling technique to employ. For DGT, the detection limit is often controlled by the level of background analyte in the resin gels. It is not possible to clean resins prior to deployment as the chemicals required to remove the analytes from the resin also destroy the resin. The detection limit will also be a function of the physical dimensions of the sampler and deployment time for DGT samplers. Lower detection limits are possible in controlled laboratory settings where contamination is lower.

7.7 *Current Regulatory Perspective and Next Steps in Passive Sampling:*

7.7.1 Research and case studies have demonstrated PSMs as an effective tool for measuring C_{free} in surface water and sediment porewater. In addition, current guidance documents identify PSMs as a means for further understanding contaminant migration, bioavailability, and associated risk in sediment

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sites (ITRC 2011 (66), USEPA 2012b (39)). However, there are additional improvements to PSMs that would further advance the quality and confidence of data collected. Such improvements may include the following:

7.7.1.1 Development of laboratory testing procedures to verify the precision of the methods across different laboratories and technicians when prescribed field and analytical procedures are employed;

7.7.1.2 Development of laboratory standard reference material to allow for method accuracy checks (Ghosh et al. 2014) (32);

7.7.1.3 Development of non-equilibrium passive sampling methods for *in situ* deployments in order to validate PRC use in static sediment environments, and identify ways to improve the accuracy of PRC correction methods (Ghosh et al. 2014) (32).

7.7.2 The application of PSMs is a relatively new practice as compared to other sediment analytical techniques, and the state of the practice for PSMs continues to evolve. It is expected additional standard methods and guidance documents will be developed, further improving and standardizing PSMs.

8. Biological Test Methods

8.1 An understanding of the potential impacts to biological resources is useful for the assessment, management, and remediation of contaminated sediments given that these impacts are often an important endpoint of sediment investigations and corrective actions. This biological test methods section addresses the selection and application of biological test methods for sediment sites. These methods incorporate the assessment of biota tissue contaminant concentrations and direct effects of sediment exposure to biota.

8.2 The assessment of sediment-associated tissue chemistry is necessary to understand both chemical bioavailability and the magnitude of chemical concentrations in tissues at a site. The evaluation of tissue chemistry can provide understanding of exposure and uptake through the analysis of native biota tissue chemistry and bioaccumulation testing (*in situ* and *ex situ*). Conversely, the assessment of sediment direct effects is key to evaluate if sediment exposure is causing adverse impacts to biota. Sediment direct effects to biota can be evaluated through *ex situ* toxicity testing, community surveys, and histopathology.

8.3 The assessment of bioavailability and toxicity (that is, capacity to directly affect biota) may be useful in baseline monitoring, risk assessment, site investigations, remedial design, and remedy performance evaluations. These test methods can also support additional sediment related investigations, such as dredge material management, but this is not the focus of this document as specific guidance for these programs exist (for example, USACE, 1998) (7).

8.4 Physical and chemical characteristics of the sediment may enhance or reduce the inherent potential for bioaccumulation in tissues and contaminant direct effects to biota. Given this relationship, confounding factors will be addressed for their influence on biological test results within this section.

8.5 *Overview of Approaches:*

8.5.1 *Applicable Organisms*—This biological test methods section will discuss benthic invertebrates, fish, wildlife, vegetation, and microorganisms, with a particular focus on benthic invertebrates and fish as these organisms are most closely associated with sediments. Benthic invertebrates interact directly with sediment by living and feeding on or in the sediment. Due to their direct exposure and small home ranges, benthic invertebrates tend to show good correlation between biological test results and sediment contamination. Fish represent the effects of sediment-associated contaminants on higher trophic levels that are exposed to sediment flux in overlying water, bioaccumulation and biomagnification through consumption of diet items, and incidental sediment ingestion during bottom foraging (ITRC 2011) (66). Fish typically have a much larger home range than benthic invertebrates, so biological test results, such as concentrations of chemicals in tissues, are often not as well correlated to sediment contamination as benthic invertebrates. Fish also represent a common regulatory driver of human health and wildlife exposure due to ingestion (USEPA 2017) (67). Wildlife, vegetation, and microorganisms are sampled at sediment sites with less frequency, but are still useful organisms for biological analyses. Many of the analytical methods and techniques described within this section can also be applied to pelagic invertebrates as they represent a broader range of exposure pathways and assessment endpoints. However, because these organisms are exposed to contaminants predominantly via the water column, they may be influenced by non-sediment or non-site related contaminants. Given their more direct exposure, benthic invertebrates offer a conservative surrogate for the assessment of sediment associated contaminant impacts to pelagic invertebrates. Terrestrial invertebrates are not considered within the scope of this document due their lack of exposure to sediments.

8.5.2 *Sample Matrices*—Biological testing at sediment sites generally includes analyzing tissue samples that have been either field collected or exposed to site sediments, or completing biological testing or surveying of organisms for toxicological responses. The end results of biological testing include analytical chemistry data and quantitative/semi-quantitative direct effect response data.

8.5.3 *Sample Volume/Mass/Container Considerations*—Biological testing includes a breadth of experimental and analytical techniques. Given the variability in the methods and the biota studied, it is recommended that site assessors work with an accredited analytical laboratory to determine sample size requirements, containers, and preservation methods for the given study objectives. The sample collection and handling requirements will be influenced by the selected organisms, sediment-associated contaminants of interest, and application of the analytical tests presented within this section.

8.5.4 *Quick Reference Guide*—Several analytical tests can be utilized to evaluate sediment-associated tissue chemistry and direct effects to biota. The evaluation of impacts to biota is nuanced and the experimental selection and design must be site-specific in order to meet required study objectives. Table 3 provides an overview of six biological test methods to assist in test method selection. Table 3 lists six biological test methods that are commonly employed when evaluating sediments,

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TABLE 3 Overview of Biological Test Methods

	Native Biota Tissue Chemistry (8.6)	In situ Bioaccumulation Testing (8.7.6)	Ex situ Bioaccumulation Testing (8.7.7)	Ex situ Toxicity Testing (8.8)	Community Assessment (8.9)	Histopathology (8.10)
Method						
Tissue Chemistry Assessment	•	•	•			
Direct Effects to Biota Assessment				•	•	•
In situ	•	•			•	•
Ex situ			•	•		•
Steady State Testing	•	•	•			
Primary Species Class						
Benthos	•	•	•	•	•	•
Fish	•	•				•
Wildlife	•					•
Vegetation	•	•	•	•		
Microorganisms				•	•	
Native Biota	•				•	•
Laboratory Raised		•	•	•		•
Experimental Qualities						
Strong Representativeness of Field Conditions	•	•			•	•
High Experimental Control		•	•	•		•
Known Contaminant Exposure		•	•	•		•
Ease of Collection of Sufficient Sample Mass		•	•	•		•
Recommended Testing of Reference Sites	•	•		•	•	•
Existence of Common Standard Operating Procedures for Test Method			•	•		

as well as the basic concepts of these methods. The dots in Table 3 indicate that the methodolody (column header) meets the criteria (row header).

8.5.5 *Applicability*—The biological test methods section applies to the testing for sediment-associated chemicals in the tissues of biota and the testing of adverse effects to biota that are exposed to sediments. These methods enable evaluation of potential for impacts to biota at multiple trophic levels including microorganisms, vegetation, benthos, pelagic invertebrates, benthic/invertivorous fish, piscivorous fish, and wildlife. The application of this section includes data usage in risk assessment to evaluate potential for adverse effects, site investigations to understand the distribution of effects throughout a system, and evaluation of remedial efficacy. Additionally, this data can be used toward ecological foodweb modeling of contaminants to estimate the exposure and potential for adverse effects to wildlife and humans that consume fish, invertebrates, vegetation, and other wildlife, though details on these models are not within the scope of this section.

8.5.6 *Methods/Approaches*—The methods described within the biological test method section should be selected with consideration of the study objectives and site of interest. Appendix X10 provides a pros and cons evaluation of the various experimental and analytical techniques to support sediment practitioners in planning for biological testing. Appendix X11 provides a decision tree for biological test method selection. Appendix X12 provides species lists for *ex situ* bioaccumulation testing and sediment toxicity testing.

8.6 *Native Biota Tissue Chemistry:*

8.6.1 Concentrations of chemicals in tissues inform sediment practitioners on the bioavailability of sediment associated chemicals (that is, the fraction of chemicals that is available for uptake into the tissues of biota) and the magnitude of chemical tissue accumulation through multiple exposure pathways such as sediment ingestion and porewater contact. The bioavailability of chemicals is often a key metric used in site investigations, exposure modeling for non-target organisms, and assessment of remedies designed to reduce the bioavailable chemical fraction in sediment (for example, activated carbon amendment caps). Measurements of exposure (that is, concentrations of chemicals in tissues) are similarly important for assessing the potential for effects, screening level risk assessments, or exposure modeling for higher trophic level organisms. Chemical concentrations in the tissue of benthos, fish, wildlife, vegetation, and other ecological receptors can be used as an indicator of sediment-associated chemical exposure. However, tissue concentrations are not evaluated in microorganisms due to matrix limitations. Tissue chemistry analyses can be conducted on organisms collected from the site (native biota) or from either field collected or laboratory reared organisms that have been exposed to site sediments *in situ* or *ex situ* for an appropriate length of time to accumulate contaminants in tissues (that is, bioaccumulation testing, Section 8.7 of this guide). Design of tissue chemistry testing requires selection of supporting data and adherence to quality assurance and quality control protocols.

8.6.2 *Experimental and Sampling Design Considerations:*

8.6.2.1 Sampling design for all tissue chemistry assessments should include data quality objectives such that the data can be verified to be reliable and useable. General quality assurance/quality control (QA/QC) considerations include collection of sufficient sample volumes to provide mass for field duplicate samples to measure analytical variability; field, equipment, and method blank samples to identify other sources of chemical contamination of samples; statistical power and sampling design; appropriate documentation of field procedures and laboratory internal QA/QC protocols. Specifically, when measuring tissue concentrations of contaminants, additional QA/QC procedures should include sufficient replicates of exposure groups or organisms to characterize variability in

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