Designation: D5074 – 90 (Reapproved 2022)

Standard Practice for Preparation of Natural-Matrix Sediment Reference Samples for Major and Trace Inorganic Constituents Analysis by Partial Extraction Procedures[1]

This standard is issued under the fixed designation D5074; 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.

1. Scope

1.1 This practice covers uniform procedures to develop, select, collect, prepare, and use oxidized, relatively unpolluted, aquatic natural-matrix bed-sediment reference samples for the collaborative testing of chemical methods of analysis for sediments and similar materials. Reference samples prepared using this practice are intended for use as natural sediments, analyzable for major, minor, and trace elements, and general physical/organic analyses only. The samples are not designed or tested for environmental pollutants such as trace organic compounds.

1.2 Few, if any, aquatic sediment reference materials have been certified, defined, or are even available for developing or evaluating partial and sequential extraction procedures. This practice describes factors and considerations in site selection, sample characteristics, collection, and subsequent raw sample treatment needed to prepare natural-matrix bed-material sediments for use as partial or sequential extraction procedure reference test samples. The user of this practice is cautioned that in light of the many variables that may affect natural materials, neither the list of factors included for evaluation nor preparation of natural-matrix reference samples should be considered as all inclusive. It is the user's responsibility to ensure the validity and applicability of these practices for preparing specific-matrix samples appropriate for testing the constituents of interest and the operationally defined extraction procedures utilized.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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]

D1129 Terminology Relating to Water

D3974 Practices for Extraction of Trace Elements from Sediments

D3975 Practice for Development and Use (Preparation) of Samples for Collaborative Testing of Methods for Analysis of Sediments

D3976 Practice for Preparation of Sediment Samples for Chemical Analysis

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *natural-matrix sediment, n*—granular rock or earthy material that has been naturally deposited in or by a water body, in which the finer grained material encloses or fills the interstices between the larger grains or particles of sediment.

4. Summary of Practice

4.1 Natural-matrix sediment reference samples of adequately defined composition and homogeneity are required for evaluating the accuracy and precision of partial or sequential

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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[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.

sediment leachate analyses and test methods. Reference samples should be typical in all respects to the sample for which the test method is applicable. Practically, this is difficult to achieve because of the heterogeneity and compositional variability of natural sediments. However, natural sediments collected from diverse sources can be used to prepare reference samples similar or typical in many respects to the samples for which the test methods are to be applicable. For a minimal sediment quality assurance testing effort, and to evaluate the linearity of test methods, reference samples should be available to provide at least three levels of concentration for each measured parameter. Mixtures of samples of known composition may also be used.

5. Significance and Use

5.1 The objective of this practice is to provide guidelines for the preparation of stable, representative, oxidized, relatively unpolluted, aquatic natural-matrix bed-sediment reference test samples. When prepared as described, such test samples should be useful for collaborative methods testing, to evaluate the precision and bias of test methods, and to evaluate test methods performance during their development.

5.2 The availability of defined representative natural-matrix reference or test samples, closely approximating a variety of typical environmental samples, is a key requirement for the effective collaborative methods evaluation and development of test methods, and quality assurance testing. When the composition of the reference or test samples has been determined, either for operationally defined "total recoverable" leaching techniques, or for "total analysis" determined by total dissolution, the defined samples should also be suitable for analytical quality assurance testing.

5.3 Certified analyses of most rock, sediment, sludge, and soil reference samples are typically based on the total amount of each constituent of interest in the entire sample. "Total" chemical analysis of these samples generally requires complete decomposition or dissolution of the standard material. These are the only feasible analytical approaches if knowledge of finite concentrations for each element of interest in the entire sample is required. Certain instrumental methods, such as X-ray fluorescence or neutron activation analysis, may provide information as to the total constituent composition without sample destruction.

5.4 Partial chemical extraction of sediments, or "total recoverable" analyses (operationally defined procedures) for selecting constituents, frequently are useful for defining "available" constituent concentrations. In addition, partial chemical extractions may also provide data on partitioning, phase associations, or on how trace elements are entrained. Operationally defined extractable trace constituent concentrations are generally best obtained by using very specific reagent mixtures and extraction procedures, including method of mixing, vessel size and shape, extraction time, temperature, and so forth.

5.5 The various iron and manganese oxides and hydroxides, clay minerals, and organic solutes and particulates, that commonly occur as coatings on most oxidized sediment particles, are generally recognized as the controls governing the concentrations and distribution of most trace metals in natural water-sediment hydrologic environments. Anthropogenic sources clearly dominate in the number of sources and in total loading to most systems, although other factors may also be important.[3] Under reducing conditions the iron and manganese oxide coatings, organic components, and associated trace metals may be resolubilized and remobilized. Migration of the reduced solubilized species, with possible subsequent formation of sulfides and so forth, and reoxidation and redeposition at some new location, may then occur. Analysis of extractable trace constituent concentrations in leachates obtained from reduced sediments thus will probably not be indicative of the trace constituent concentrations initially associated with the oxidized and coated sediment grains.

6. Sampling

6.1 Realistic natural-matrix aquatic bed-sediment test samples needed for test methods development and testing purposes, ideally require samples closely resembling the materials for which the test method is designed. Collection and preparation of a realistic test sample necessitates consideration of a number of factors in addition to the presence or absence of certain characteristics in the raw sample material collected. These include but are not limited to the following: sampling logistics, water chemistry, and the availability of adequate quantities of sediment with the appropriate particle sizes.

6.2 The sampling site should provide easy access to fresh water with a pH of 6 to 8 and a specific conductance not exceeding 3000 µS/cm. Samples collected from higher conductivity areas should be washed to remove excessive salts. Normally, flow velocities in the collection area should be sufficiently low to allow deposition of the fine grained materials desired in the bed material to be sampled. The sample collection site should also be suitable for launching any necessary sample collection craft, or have close access to boat launching facilities.

6.3 Sufficient quantities of raw sample material should be available to obtain desired quantities of oxidized sediment. This should consist primarily of light colored quartz and silicate minerals, deposited in an aerobic environment. The raw sediment should contain only minimal quantities of particulate organic material or total organic constituents (TOC) (no more than 2 to 3 %), to minimize bacterial growth and the development of reducing conditions. The sediment collected should be free of detectable levels of reduced iron and manganese species, have no perceptible sulfide odor, and exhibit no observable methane generation. Readily soluble materials such as ore minerals, carbonates, chlorides, and sulfates should also be absent. Inclusion of relatively soluble mineral species in the sediment reference sample will result in increasing concentrations of associated major and trace parameters as a function of increased sample digestion times, until those phases are completely dissolved. "Total" digestion analyses are generally more appropriate for samples containing ore minerals and

[3] Jennett, J. C., Effler, S. W., and Wixson, B. G., "Mobilization and Toxicological Aspects of Sedimentary Contaminants," *Contaminants and Sediments*, ed., Baker, R. A., Ann Arbor Science Publishers, Inc., Ann Arbor, MI, 1990.