

Designation: D3976 - 22

# Standard Practice for Preparation of Sediment Samples for Chemical Analysis<sup>1</sup>

This standard is issued under the fixed designation D3976; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This practice describes standard procedures for preparation of test samples (including the removal of occluded water and moisture) of field samples collected from locations such as streams, rivers, ponds, lakes, and oceans.
- 1.2 These procedures are applicable to the determination of volatile, semivolatile, and nonvolatile constituents of sediments.
- 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. For a specific precautionary statement, see Note 3.
- 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:<sup>2</sup>

D596 Guide for Reporting Results of Analysis of Water

D1129 Terminology Relating to Water

D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits (Withdrawn 2003)<sup>3</sup>

D3370 Practices for Sampling Water from Flowing Process Streams

## D4410 Terminology for Fluvial Sediment

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminologies D1129 and D4410.

## 4. Summary of Practice

- 4.1 Samples collected (see Practice D3370 and Guide D1192) in the field are screened to remove foreign objects prior to homogenization for chemical examination and analysis. Large objects are mechanically removed and small ones are eliminated by sieving the sample through a 10-mesh (2 mm openings) sieve.
- 4.2 Wet, sieved samples are mixed for preliminary homogenization, then allowed to settle to remove most of the occluded water.
- 4.3 Moisture determinations are made on separate samples from those analyzed for volatile or semivolatile constituents.
- 4.4 Analyses for volatile constituents are made using wet, settled samples from which supernatant liquid has been removed by decantation. The results are corrected to those that would have been obtained on samples dried to constant weight at 105 °C  $\pm$  2 °C, on the basis of a moisture determination using a separate sample.
- 4.5 Analyses for semivolatile constituents (for example, mercury) are made on samples previously dried at a temperature found to be adequate for the purpose, and specified in the corresponding analytical procedure.
- 4.6 Analyses for nonvolatile constituents are made on samples previously dried to constant weight at  $105 \, ^{\circ}\text{C} \pm 2 \, ^{\circ}\text{C}$ .
- 4.7 A flow diagram, outlining typical procedures, is shown in Fig. 1.

## 5. Significance and Use

- 5.1 The chemical analysis of sediments, collected from such locations as streams, rivers, ponds, lakes, and oceans can provide information of environmental significance.
- 5.2 Sediment samples are inherently heterogeneous in that they contain occluded water in varying and unpredictable amounts and may contain foreign objects or material not ordinarily considered as sediment, the inclusion of which would result in inaccurate analysis.

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> 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

<sup>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

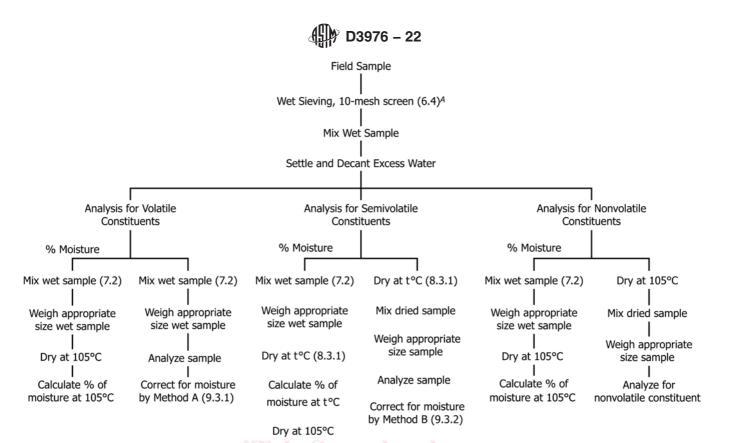


FIG. 1 Flow Diagram for Sediment-Sample Treatment

Calculate % of moisture at 105°C

- 5.3 Standard methods for separating foreign objects to facilitate homogenization will minimize errors due to poor mixing and inclusion of extraneous material.
- 5.4 Standardized procedures for drying provide a means for reporting analytical values to a common dry weight basis.

## 6. Preliminary Treatment of Field Samples

6.1 The analytical sample is arbitrarily defined as that which passes a 10-mesh (approximately 2 mm openings) sieve. The purpose of this is to provide a basis for discrimination of sediment and foreign objects or materials. Stainless steel or nylon sieves may be used when inorganic constituents are to be determined. Stainless steel or brass sieves are suitable for use when organic substances are to be determined.

Note 1—For inorganic analyses, stainless steel sieves are acceptable provided the mesh is not soldered or welded to the frame. For organic analyses, organic materials such as rubber or plastics should not be used in the storage or handling of samples.

- 6.2 Sieve dry samples without further pretreatment and follow the procedures given in 7.3, 7.4, or 7.5, or a combination thereof, as appropriate.
- 6.3 Vigorously stir wet field samples, which may have settled during transit, to incorporate as much field water as possible, thereby facilitating subsequent wet sieving.

Note 2—Do not add additional laboratory water since this may extract constituents or otherwise change the composition of the sediment. However, it is permissible to slurry the sediment with a minimum quantity

of field water collected with the sample, when necessary, to facilitate wet-sieving.

- 6.4 Pass the wet sample, preferably as a slurry, through the sieve (plastic or stainless steel). The bottom of an appropriate size Erlenmeyer flask may be used to gently press the sediment through the sieve, as necessary.
- 6.4.1 Manually remove foreign objects such as stones, twigs, leaves, trash, etc., which would obviously not pass through the sieve and which may interfere with the sieving operation.
- 6.5 Mix the sieved material by stirring and allow it to settle for subsequent removal of supernatant liquid.
- 6.5.1 Store the material, as prepared above, in contact with its supernatant liquid, until time of use for chemical examination.

Note 3—**Precaution:** Samples intended for both organic and inorganic compound analysis may undergo changes in composition during storage. The analytical method should specify the conditions necessary to assure requisite stability. In the absence of specific instructions, storage at a temperature of 4 °C or lower for a period of time not to exceed 1 week is recommended, although it is known that microbiological activity does not cease under these conditions.

## 7. Preparation of Analytical Samples

7.1 Decant the supernatant liquid from the settled sediment prepared in accordance with Section 6. Save the supernatant liquid for separate analysis of any suspended material, as necessary.