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Standard Practice for Preparation of Sediment Samples for Chemical Analysis¹

This standard is issued under the fixed designation D 3976; 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 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 *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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see Note 3.

2. Referenced Documents

2.1 ASTM Standards:

D 596 Practice of Reporting Results of Analysis of Water²

D 1129 Terminology Relating to Water²

D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits²

D 3370 Practices for Sampling Water from Closed Conduits²

D 4410 Terminology for Fluvial Sediment²

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminologies D 1129 and D 4410.

4. Summary of Practices

4.1 Samples collected (see Practices D 3370 and Specification D 1192) 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 $105 \pm 2^\circ\text{C}$, 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 $105 \pm 2^\circ\text{C}$.

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.

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

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² *Annual Book of ASTM Standards*, Vol 11.01.

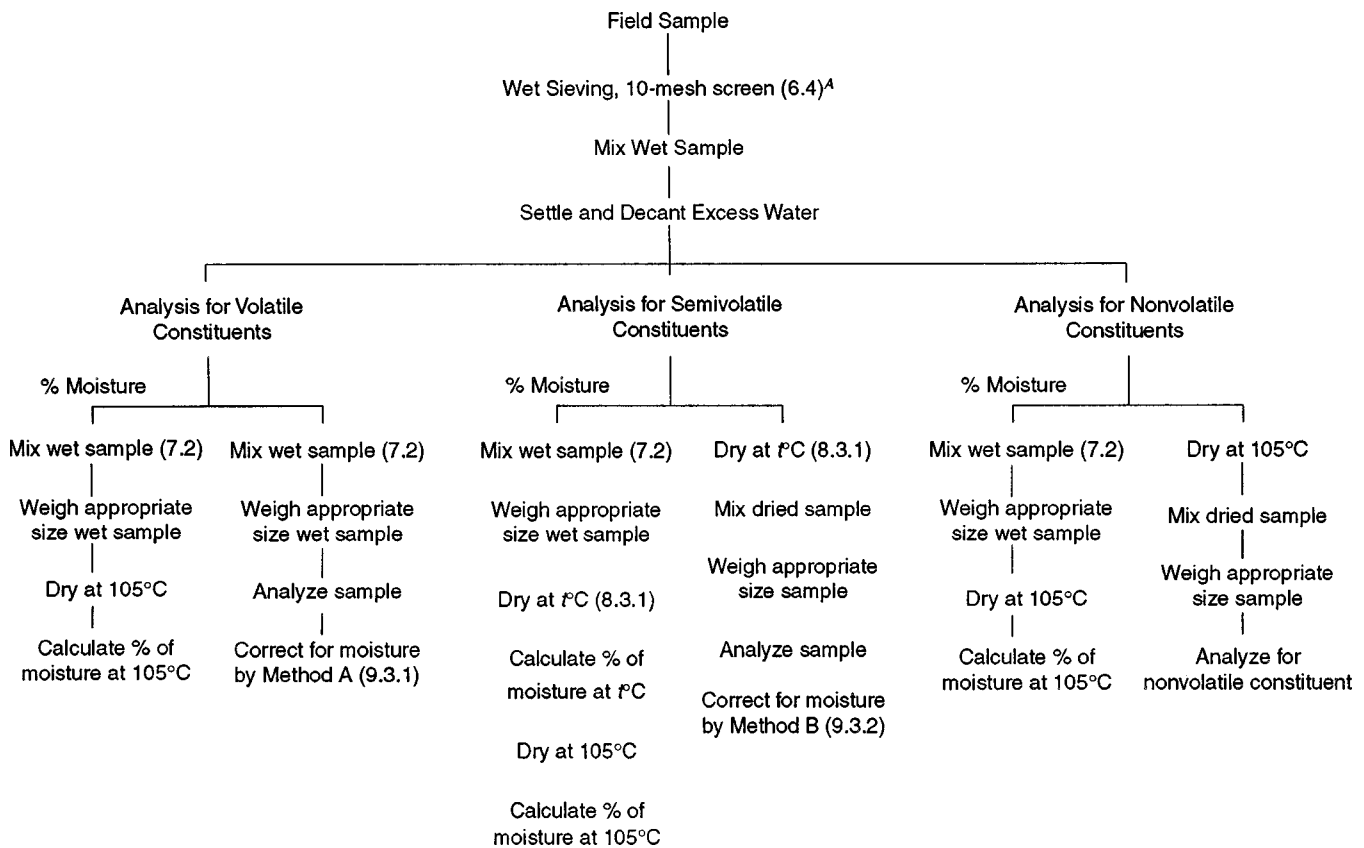


FIG. 1 Flow diagram for Sediment-Sample Treatment

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.

7.2 Mix the sediment, using a glass rod or porcelain spatula, to minimize stratification effects due to differential rates of settling.

7.3 Remove a number of small portions (at least ten) from random locations in the sample container and composite them to obtain a representative sample of size suitable for determination of moisture (see Section 9), or for drying to prepare material for analysis of semivolatile or nonvolatile constituents.

7.4 Determine volatile constituents, using wet samples prepared in accordance with 7.3.

7.5 Determine semivolatile and nonvolatile constituents, using samples prepared in accordance with 7.3 and Section 8.

7.5.1 Disaggregate the dried material by gently crushing any lumps or clumps in a mortar. Mix the disaggregated material and prepare a composite sample resulting from removal of a number of smaller portions as indicated in 7.3. The use of freeze drying facilitates sample disaggregation.

8. Drying Procedure

8.1 Use a sample prepared in accordance with directions given in Section 7.

8.2 Accurately weigh 5 to 10 g (±1 mg) or 10 to 25 g (±10 mg) of the sediment into a previously tared porcelain dish, weighed with the same accuracy.

8.2.1 When a limited amount of sample is available, the moisture may be determined on 1- to 2-g samples, weighed with an accuracy of ± 0.1 mg. The use of samples smaller than 1 g is not recommended for moisture determination.

8.3 Transfer the dishes containing the weighed sediment to an oven and dry for 2 h as follows:

8.3.1 For determination of semivolatile constituents, use the temperature ($t^{\circ}\text{C}$) specified in the analytical procedure for that constituent. For determination of nonvolatile constituents use $105 \pm 2^{\circ}\text{C}$.

8.4 Cool in a desiccator, then weigh the dried samples with the same accuracy as the wet sample.

8.5 Repeat drying at hourly intervals, to a constant weight.

9. Moisture Correction and Calculations

9.1 The possibility of loss of volatile constituents dictates the drying procedure that should be used, prior to chemical analysis. Volatile constituents are usually determined using undried samples. Semivolatile constituents must be determined using samples dried at a temperature at which no significant losses occur. Nonvolatile constituents are analyzed using samples dried at $105 \pm 2^{\circ}\text{C}$.

9.2 Report all analytical values (see Method D 596), regardless of how measured, on the basis of a sample dried at $105 \pm 2^{\circ}\text{C}$ to facilitate correlation of data.

9.3 The following equations are useful to correct analytical results, C (weight percent, $\mu\text{g/g}$, etc.), obtained on an undried sample or one partially dried at $t^{\circ}\text{C}$, to the basis of one dried at $105 \pm 2^{\circ}\text{C}$.

9.3.1 *Test Method A*—Calculate volatile constituents as follows:

$$C_v = C_{vl} / (100 - \% \text{moisture}_{105})$$

where:

C_v = dry basis, and

C_{vl} = wet basis.

9.3.2 *Test Method B*—Calculate semivolatile constituents as follows:

$$C_{sv} = C_{svl} (100 - \% \text{moisture}) / (100 - \% \text{moisture}_{105})$$

where:

C_{sv} = dry basis, and

C_{svl} = dried at $t^{\circ}\text{C}$.

10. Keywords

10.1 chemical analysis; nonvolatile constituents; preparation; sediment samples; semivolatile; volatile

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