



Designation: D 2331 – 80 (Reapproved 1999)

Standard Practices for Preparation and Preliminary Testing of Water-Formed Deposits¹

This standard is issued under the fixed designation D 2331; 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 These practices provide directions for the preparation of the sample for analysis, the preliminary examination of the sample, and methods for dissolving the analytical sample or selectively separating constituents of concern.

1.2 The general practices given here can be applied to analysis of samples from a variety of surfaces that are subject to water-formed deposits. However, the investigator must resort to individual experience and judgement in applying these procedures to specific problems.

1.3 The practices include the following:

	Sections
Preparation of the Analytical Sample	8
Preliminary Testing of the Analytical Sample	9
Dissolving the Analytical Sample	10

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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific warning statement, see Note 2.

2. Referenced Documents

2.1 ASTM Standards:

- D 887 Practices for Sampling Water-Formed Deposits²
- D 932 Test Method for Iron Bacteria in Water and Water-Formed Deposits²
- D 933 Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits²
- D 934 Practices for Identification of Crystalline Compounds in Water-Formed Deposits by X-Ray Diffraction²
- D 993 Test Method for Sulfate-Reducing Bacteria in Water and Water-Formed Deposits³
- D 1128 Method for Identification of Types of Microorganisms and Microscopic Matter in Water and Waste Water⁴

- D 1129 Terminology Relating to Water⁵
- D 1193 Specification for Reagent Water⁵
- D 1245 Practice for Examination of Water-Formed Deposits by Chemical Microscopy²
- D 2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence²
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁶

3. Terminology

3.1 *Definitions*—For definitions of terms used in these practices, refer to Terminology D 1129.

4. Significance and Use

4.1 Deposits in piping from aqueous process streams serve as an indicator of fouling, corrosion or scaling. Rapid techniques of analysis are useful in identifying the nature of the deposit so that the reason for deposition can be ascertained.

4.2 Possible treatment schemes can be devised to prevent deposition from reoccurring.

4.3 Deposits formed from or by water in all its phases may be further classified as scale, sludge, corrosion products or biological deposits. The overall composition of a deposit or some part of a deposit may be determined by chemical or spectrographic analysis; the constituents actually present as chemical substances may be identified by microscope or X ray.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of

¹ These practices are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Surveillance of Water, and Flow Measurement of Water. Current edition approved July 3, 1980. Published September 1980. Originally published as D 2331 – 65 T. Last previous edition D 2331–73(1979).

² *Annual Book of ASTM Standards*, Vol 11.02.

³ Discontinued—see 1987 *Annual Book of ASTM Standards*, Vol 11.01.

⁴ Discontinued—see 1981 *Annual Book of ASTM Standards*, Part 31.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water conforming to Specification D 1193.

6. Sampling

6.1 Collect and preserve the sample in accordance with Practices D 887.

7. Preparation of Analytical Sample

7.1 *Preliminary Examination*—Examine the sample as collected, using a microscope if available, for structure, color, odor, oily matter, appearance of mother liquor if any, and other characteristics of note (for example, attraction to magnet). Record results for future reference.

7.1.1 Filtration and other steps in the preparation of the analytical sample may frequently be bypassed; for example, a moist sample that contains no separated water shall be started in accordance with 7.3.1, and a dry sample shall be started in accordance with 7.4, 7.5, or 7.6. Partitioning, 7.4, is not always practical or even desirable. Solvent extraction, 7.5, is unnecessary if the sample contains no oily or greasy matter.

7.2 *Filtration of Sample* (see Note 1)—If the sample includes an appreciable quantity of separated water, remove the solid material by filtration. Save the filtrate, undiluted, pending decision as to whether or not its chemical examination is required. Transfer all of the solid portion to the filter, using the filtrate to rinse the sample container if necessary. Air-drying or partial air-drying of the filter is frequently helpful toward effecting a clean separation of the deposit.

NOTE 1—If the sample obviously contains oily matter, its extraction with a suitable solvent (see 7.5) is essential before filtration or air-drying is attempted. Likewise, if the sample is suspected to contain easily oxidizable materials, such as sulfide, analysis for these materials should be completed before air-drying.

7.3 *Air-Drying*—Remove the drained solid sample from the filter, being careful to avoid gross contamination with filter paper.

7.3.1 Air-dry the entire quantity of solid, spread in a thin layer on a nonreactive, impervious surface. A record of the loss of weight during air-drying is often used.

7.4 *Partitioning the Sample*—Many samples are obviously heterogeneous. If useful to explain the occurrence of the water-formed deposit, separate clearly defined layers or components, and approximate the relative percentages.

7.4.1 Retain the individual air-dried fractions for separate analysis, preferably storing over an effective desiccant such as anhydrite.

7.5 *Solvent Extraction*—This step is essential only if the air-dried sample smears or agglomerates when tested for pulverization (smears caused by graphite are possible but rare with water-formed deposits).

7.5.1 Weigh no more than 10 g of air-dried sample and place this, wrapped in fine-textured filter paper, in a prepared (extracted and dried) Soxhlet thimble. Paper clips are useful for preventing unfolding of the paper. Weigh the thimble and its contents and extract in a Soxhlet apparatus until the solvent (chloroform) in the extraction chamber is colorless. Record the

loss in weight of the thimble and contents, dried at 105°C, as chloroform-extracted matter. If important to the solution of the problem, evaporate the solvent, and examine the residue.

7.5.2 The extraction may be repeated with other volatile organic solvents if exploratory tests warrant such procedure.

7.6 *Pulverizing*—Whether the sample is dry as received, air-dried or air-dried extracted, it must be pulverized to adequate homogeneity. Grind the entire sample, or enough of it to be representative of the whole, to pass a No. 100 (150- μ m) sieve, as specified in Specification E 11. Continue the grinding until all the material passes through the sieve, except for fragments such as splinters of fiber, wood, and metal.

7.6.1 Identify fragments separated from the sample during grinding by standard methods if this information is valuable.

7.6.2 Mix the sieved material thoroughly by tumbling in a closed dry container that is no more than two thirds full.

7.6.3 Transfer 5 to 10 g of the thoroughly mixed material to a weighing bottle. This is the analytical sample. Unless the determinations are to be made on an air-dried basis, dry at 105°C and store in a desiccator.

8. Preliminary Testing of Analytical Sample

8.1 This section outlines methods for the preliminary examination of samples of water-formed deposits. Use one or more of these methods to disclose the component elements of the sample and whether the concentrations are major, minor, or trace, an essential guide to planning the analysis. This preliminary testing frequently also provides important guidance toward defining technological problems associated with the occurrence of the deposits. The methods include spectrography, atomic absorption spectrophotometry, X-ray diffraction, X-ray fluorescence, microscopy, and ordinary qualitative analysis.

8.2 *Spectrography*—Make the spectrographic analysis by a suitable method, for example, as outlined in 8.2.2 to 8.2.7.

8.2.1 Although superior results are obtainable with a spectrograph and associated equipment, data of lesser degree of accuracy can frequently be obtained with less formal equipment such as a visual-arc spectroscope.

8.2.2 For best results use a spectrograph having a suitable reciprocal linear dispersion, associated adjuncts and optics, a microphotometer for measuring the transmittances of spectral-line images, and associated equipment for determining intensity ratios.

8.2.3 Mix 50 mg of the pulverized sample, obtained in accordance with 7.6.2, with 900 mg of graphite powder and 250 mg of lithium carbonate. Pack the mixture into graphite-cup electrodes.

8.2.4 Record the spectra obtained upon excitation with a d-c arc.

8.2.5 Measure the transmittances of the analytical and lithium lines (internal standards other than lithium are preferred by some operators). Determine intensity ratios from these data.

8.2.6 Use the intensity ratios to estimate concentrations from standard analytical curves.

8.2.7 The metallic constituents can frequently be determined within 20 % of their content in the deposit, which is sufficiently close for classification as major, minor, or trace.