



Designation: ~~D 2332–84 (Reapproved 2003)~~ Designation: D 2332 – 08

Standard Practice for Analysis of Water-Formed Deposits by Wavelength- Dispersive X-Ray Fluorescence¹

This standard is issued under the fixed designation D 2332; 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 X-ray spectrochemical analysis of water-formed deposits.

1.2 The practice is applicable to the determination of elements of atomic number 11 or higher that are present in significant quantity in the sample (usually above 0.1 %).

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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D 887 Practices for Sampling Water-Formed Deposits

~~D 1129 Terminology Relating to Water~~ 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Terminology

3.1 ~~Definitions—~~For definitions of terms used in this practice, refer to Terminology D 1129.

4. Summary of Practice

4.1 The sample or its fusion with a suitable flux is powdered and the powder is compacted (mounted). The mount is then irradiated by an X-ray beam of short wavelength (high energy). The characteristic X rays of the atom that are emitted or fluoresced upon absorption of the primary or incident X rays are dispersed, and intensities at selected wavelengths are measured by sensitive detectors. Detector output is related to concentration by calibration curves or charts.

4.2 The K spectral lines are used for elements of atomic numbers 11 to 50. Whether the K or L lines are used for the elements numbered 51 or higher depends on the available instrumentation.

5. Significance and Use

5.1 Certain elements present in water-formed deposits are identified. Concentration levels of the elements are estimated.

5.2 Deposit analysis assists in providing proper water conditioning.

5.3 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 diffraction studies. Organisms may be identified by microscopical or biological methods.

6. Apparatus

6.1 *Sample Preparation Equipment:*

6.1.1 *Fusion Crucibles*, prepared from 25-mm (1-in.) commercial-grade graphite rods. The dimensions shall be 29 mm (1 $\frac{1}{8}$ in.) high, an inside diameter of 19 mm ($\frac{3}{4}$ in.), and a cavity 22 mm ($\frac{7}{8}$ in.) deep.

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

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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.

6.1.2 *Pulverizers*, including an agate or mullite mortar and pestle, minimum capacity 25 ml.

6.1.3 *Sieves*—No. 100 (150- μm) and No. 270 (53- μm) as specified in Specification E 11.

6.1.4 *Compactors*—A press, equipped with a gauge enabling reproducible pressure, is recommended.

6.2 *Excitation Source (~~X-ray Tube~~—X-ray Tube)*:

6.2.1 *Stable Electrical Power Supply* ($\pm 1\%$).

6.2.2 Source of high-intensity, short-wave-length X rays.

6.3 *Sample Housing (Turret)*.

6.4 *Spectrometer*—Best resolution of the spectrometer and best sensitivity are not simultaneously attainable; a compromise is effected to give adequate values for each.

6.4.1 *Collimating System*.

6.4.2 *Spectrogoniometer*.

6.4.3 *Analyzing Crystal and Holder*—The choice of the analyzing crystal is made on the basis of what elements must be determined; for example, a gypsum or an ammonium dihydrogen phosphate crystal can be used for determining magnesium but lithium fluoride is much superior for copper and iron (high-intensity diffracted secondary rays and consequently greater sensitivity and potential precision). A salt, sodium chloride (NaCl), crystal is frequently employed for general use, being applicable over a broad range and producing intense lines and medium broadening.

6.4.4 *Counter-Tube Support*.

6.5 *Evacuating or Flushing System*.

6.6 *Measuring System*:

6.6.1 *Detector* (of which the principal types are the Geiger counter, scintillation counter, and flow-proportional counter).

6.6.2 *Amplifiers (Including Preamplifier), Rate Meter, Recorder, Scaler, and Printout*.

6.6.3 *Zeroing, Gain, and Sequence Controls*.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193 Type II.—reference to water shall be understood to mean water that meets or exceeds the quantitative specifications for Type I or Type II reagent water conforming to Specification D 1193, Section 1.1.

7.3 *Detector Gas*, usual composition 90 % argon, 10 % methane (other compositions are used); usually used with flow-proportional counter for lines of longer wavelength (0.2 nm or greater).

7.4 *Gallium Oxide*, spectrographic grade (frequently used as a convenient internal standard).

7.5 *Helium Gas*, commercial grade, for the spectrometer flushing system, when vacuum or air paths are not used. 32-08

7.6 *Sodium and Lithium Borates*—($\text{Na}_2\text{B}_4\text{O}_7$ and $\text{Li}_2\text{B}_4\text{O}_7$), commonly used as fluxes for the sample.

8. Sampling

8.1 Collect the sample in accordance with Practices D 887.

9. Preparation of Sample

9.1 Reduce the entire sample of deposits to about 100 g (drying, degreasing, and crushing if necessary) and grind this subsample to a powder that will pass a No. 100 (150- μm) sieve.

9.2 Mix the powdered sample thoroughly and remove about 10 g for X-ray fluorescence testing (Note 1). Fuse a weighed amount with a weighed amount of a suitable flux (2 to 10 g of flux/g of sample) to present a reproducible surface composition to the X-ray beam.

NOTE 1—At least semiquantitative results can be obtained more quickly by compacting (mounting) the test portion (9.3 and 9.4) and proceeding in accordance with Sections 10, 11, and 12. The decrease in sample preparation will actually result in improved accuracy in some instances.

9.3 Grind not more than 10 g of the material prepared for X-ray analysis (sample or fusion) to pass a No. 270 (53- μm) sieve).

9.4 Make duplicate wafers (or suitable mounts for the particular equipment that will be used) by compacting the powdered sample (precision and accuracy are improved by briquetting). An internal standard is frequently added by fusion to the material to be compacted. Some samples may require a binder (generally organic and added in minimum concentration) for reproducible packing and a smooth surface.

³ *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 *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.