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Standard Practice for Examination of Water-Formed Deposits by Chemical Microscopy¹

This standard is issued under the fixed designation D1245; 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 a procedure for the examination of water-formed deposits by means of chemical microscopy. This practice may be used to complement other methods of examination of water-formed deposits as recommended in Practices D2331 or it may be used alone when no other instrumentation is available or when the sample size is very small.

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

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

D887 Practices for Sampling Water-Formed Deposits

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2331 Practices for Preparation and Preliminary Testing of Water-Formed Deposits

D2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence

D3483 Test Methods for Accumulated Deposition in a Steam Generator Tube

3. Terminology

3.1 *Definitions*—For definitions of terms in this practice relating specifically to water and water-formed deposits, refer to Terminology D1129.

3.2 *Descriptions of Terms Specific to This Standard*—Certain terms in this practice that relate specifically to chemical microscopy are described as follows:

3.2.1 *anisotropic, adj*—having different optical properties in different optical planes.

3.2.1.1 *Discussion*—These planes are referred to as the alpha, beta, and omega axes.

3.2.2 *Becke line, n*—a faint, halo-like line that surrounds a crystal when the crystal is mounted in an oil of different refractive index. ¶

3.2.2.1 *Discussion*—The Becke line increases in intensity as the difference in the refractive index between the crystal and the oil increases.

3.2.3 *dispersion* dispersion, n—the variation of index of refraction with wavelength.

3.2.4 *dispersion staining, n*—the color effects produced when a transparent object, immersed in a liquid having a refractive index near that of the object, is viewed under the microscope by transmitted white light and precise aperture control.

3.2.5 *extinction angle* extinction angle, n—the angle between the extinction position and some plane, edge, or line in a crystal.

3.2.6 *extinction position* extinction position, n—the position in which an anisotropic crystal, between crossed polars, exhibits complete darkness.

3.2.7 *index of refraction* index of refraction, n—the numerical expression of the ratio of the velocity of light in a vacuum to the velocity of light in a substance.

3.2.8 *isotropic* isotropic, adj—having the same optical properties in all directions.

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling 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.

3.2.9 *petrographic, adj*—pertaining to the description of rocks or rocklike substances.

3.2.9.1 *Discussion*—Such description is usually in terms of morphology and optical properties.

3.2.10 ~~*solid solutions*~~ *solid solution, n*—a homogeneous mixture of two or more components, in the solid state, retaining substantially the structure of one of the components.

4. Summary of Practice

4.1 The practice is essentially chemical microscopical, supplemented by optical data obtained by the petrographic method. The identification of compounds is made by observing, under the microscope, characteristic reactions and precipitates resulting from the action of specific reagents on the solid sample or solutions thereof, and by measuring the optical properties.

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5. Significance and Use

5.1 Chemical composition of water-formed deposits is a major indicator of proper or improper chemical treatment of process water, and is often an indicator of operational parameters as well, for example, temperature control. This practice allows for rapid determination of constituents present in these deposits, particularly those indications of improper water treatment, since they usually have very distinctive and easily recognized optical properties.

5.2 This practice, where applicable, eliminates the need for detailed chemical analysis, which is time-consuming, and which does not always reveal how cations and anions are mutually bound.

5.3 Qualitative use of this practice should be limited to those deposits whose control is generally known or predictable, based on treatment and feedwater mineral content, and whose constituents are crystalline, or in other ways optically or morphologically distinctive. If these criteria are not met, other techniques of analysis should be used, such as Practice D2332 or Test Methods D3483, or both.

5.4 Quantitative use of this practice should be limited to estimates only. For more precise quantitative results, other methods should be used (see 5.3).

6. Interferences

6.1 Organic material may interfere with both the petrographic and the chemical procedures. Organics can usually be removed by solvent extraction as recommended in Practices D2331.

6.2 Deposits containing solid solutions present a complication in that optical data vary throughout such a system, and unless the presence of this complication is known, the data may be misinterpreted.

6.3 Extremely fine material and opaque material are difficult to identify. When present in appreciable amounts they may cloud over and obscure details of otherwise recognizable particles.

6.4 Interference with the chemical tests will be discussed in the procedures.

7. Apparatus

7.1 *Beakers*, of borosilicate glass, 30-mL.

7.2 *Cover Glasses*, No. 1 or No. 1½ thickness, round or square cover glasses.

7.3 *Glass Rods*, 150 by 5-mm, for transferring drops, and 75 by 1-mm, for stirring and leading reagent drops on the slides.

7.4 *Hotplate*.

7.5 *Light Source*—Microscope lamp with concentrated filament bulb and a focusing lens.

7.6 *Micro Gas Burner*.

7.7 *Micro Spatula*.

7.8 *Microscope Slides*, of selected grade, 25.4 by 76.2-mm or 25.4 by 50.8-mm.

7.9 *Mortar and Pestle*, of tool steel, mullite, or aluminum oxide.

7.10 *Petrographic Microscope*—A microscope equipped with a circular rotating stage, graduated in degrees. The optical system shall include two polarizing devices, one mounted below the condenser and the other just above the objective; 4×, 10×, and 45× objectives; and 5× and 10× eyepieces fitted with crosshairs. The optic axis of the microscope shall be adjustable so that it can be brought into coincidence with the center of rotation of the revolving stage. A Bertrand-Amici lens equipped with an iris diaphragm, or a sliding stop ocular, shall be used for viewing interference figures. A quartz wedge, gypsum plate, and standard mica plate are necessary external accessories. Aperture stops are necessary for observing the color effects of dispersion, that is, dispersion staining. A cardboard “washer” in the objective and a cover glass with a centered dried drop of India ink are sufficient; however, a device is available commercially.

7.11 *Porcelain Crucibles*, No. 0.

7.12 *Reagent Bottles for Immersion Liquids*—Glass dropping bottles of 30-mL capacity. These bottles shall be equipped with groundglass stoppers with dropping rods integral with the stoppers. Inert plastic bulbs and caps may be used, but dropping bottles with rubber bulbs are unsatisfactory because of the effect of some of the immersion liquids on the rubber. It is essential that the bottles be marked with the refractive index of the contained liquid. Commercially available liquids come in dropping bottles which are acceptable.

7.13 *Refractometer*, for measuring the refractive index of immersion liquids.

7.14 *Sample Vials*, 45 by 15-mm.

7.15 *Sieve*, No. 100 (149 μm).

7.16 *Small Alloy Magnet*.

8. Reagents

8.1 *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