Designation: D 1245 – 84 (Reapproved 1999)

Standard Practice for Examination of Water-Formed Deposits by Chemical Microscopy[1]

This standard is issued under the fixed designation D 1245; 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 D 2331 or it may be used alone when no other instrumentation is available or when the sample size is very small.

1.2 *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:*

D 887 Practices for Sampling Water-Formed Deposits [2]
D 1129 Terminology Relating to Water [3]
D 1193 Specification for Reagent Water [3]
D 2331 Practices for Preparation and Preliminary Testing of Water-Formed Deposits [2]
D 2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence [2]
D 3483 Test Methods for Accumulated Deposition in a Steam Generator Tube [2]

3. Terminology

3.1 *Definitions*—For definitions of terms in this practice relating specifically to water and water-formed deposits, refer to Terminology D 1129.

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*—having different optical properties in different optical planes. These planes are referred to as the alpha, beta, and omega axes.

3.2.2 *Becke line*—a faint, halo-like line that surrounds a crystal when the crystal is mounted in an oil of different refractive index. It increases in intensity as the difference in the refractive index between the crystal and the oil increases.

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

3.2.4 *dispersion staining*—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*—the angle between the extinction position and some plane, edge, or line in a crystal.

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

3.2.7 *index of refraction*—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*—having the same optical properties in all directions.

3.2.9 *petrographic*—pertaining to the description of rocks or rocklike substances. Such description is usually in terms of morphology and optical properties.

3.2.10 *solid solution*—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.

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

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[1] This practice is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Surveillance of Water, and Flow Measurement of Water.

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

[3] *Annual Book of ASTM Standards*, Vol 11.01.

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 D 2332 or Test Methods D 3483, 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 Practice D 2331.

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*, 30-mL, borosilicate glass.

7.2 *Cover Glasses*, No. 1 or No. 1 ½ 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 or 25.4 by 50.8 mm (1 by 3 or 1 by 2 in.).

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 such specifications are available. [4] 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.

8.1.1 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

8.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

8.3 *Ammonium Molybdate Solution* (100 g/L)—Dissolve 1 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) in water, add 35 mL of nitric acid HNO_3(sp gr 1.42) and dilute to 1 L with water.

8.4 *Ammonium Persulfate* —($(NH_4)_2S_2O_8$), crystalline.

8.5 *Barium Chloride Solution* (100 g/L)—Dissolve 100 g of barium chloride ($BaCl_2\cdot 2H_2O$) in water and dilute to 1 L.

8.6 *Cesium Sulfate*—Cs_2SO_4 crystals, 10 to 20-mesh.

8.7 *Chloroform*.

8.8 *Chloroplatinic Acid Solution*—Dissolve 1 g of chloroplatinic acid $H_2PtCl_6\cdot 6H_2O$ in 5 mL of water and add 0.5 mL of HCl (sp gr 1.19).

8.9 *Diammonium Phosphate Solution* (100 g/L)—Dissolve 100 g of diammonium phosphate $(NH_4)_2HPO_4$ in water and dilute to 1 L.

8.10 *Dimethylglyoxime*, crystalline.

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[4] *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.

8.11 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

8.12 *Hydrochloric Acid* (1+4)—Mix 1 volume of HCl (sp gr 1.19) with 4 volumes of water.

8.13 *Lead Acetate Test Paper*.

8.14 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

8.15 *Nitric Acid* (1+19)—Mix 1 volume of HNO_3(sp gr 1.42) with 10 volumes of water.

8.16 *Phenolphthalein Indicator Solution*.

8.17 *Potassium Ferricyanide* [$K_3Fe(CN)_6$], crystalline.

8.18 *Potassium Iodide* (KI), crystalline.

8.19 *Potassium Mercuric Thiocyanate Solution* (100 g/L)—Prepare freshly precipitated mercuric thiocyanate $Hg(CNS)_2$ by adding a concentrated solution of mercuric nitrate $Hg(NO_3)_2$ to a concentrated solution of potassium thiocyanate KCNS. Filter and air-dry the precipitate. To one part $Hg(CNS)_2$ add three parts KCNS, dissolve in a minimum quantity of water, and evaporate in a desiccator. Collect the first crop of tabular crystals of potassium mercuric thiocyanate $K_2Hg(CNS)_4$, wash with alcohol, and dry. Dissolve 10 g of the crystals in water and dilute to 100 mL.

8.20 *Refractive Index Standards*—A set of liquids having refractive indices ranging from 1.40 to 1.74 in steps of 0.01. In the range from 1.45 to 1.65, it is desirable to have liquids available in steps of 0.005. Commercially available liquids are recommended; however directions for the preparation of suitable liquids are given in U. S. Geological Survey Bulletin No. 848 **(1)** [5] or *Elements of Optical Mineralogy* **(2)**. The index of refraction of these liquids must be checked prior to their use, as they may change from loss of more volatile constituents.

8.21 *Silver Nitrate Solution* (50 g/L)—Dissolve 50 g of silver nitrate $AgNO_3$ in water, add 20 mL of HNO_3(sp gr 1.42), and dilute to 1 L with water.

8.22 *Sodium Bismuthate*—Powdered $NaBiO_3$.

8.23 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

8.24 *Sulfuric Acid* (1+19)—Add 1 volume of H_2SO_4(sp gr 1.84) slowly and with stirring to 19 volumes of water.

8.25 *Zinc Dust*—Powdered zinc metal.

8.26 *Zinc Uranyl Acetate Solution*—Dissolve 1 g of uranyl acetate $UO_2(C_2H_3O_2)_2 \cdot 2H_2O$ and 0.1 mL of glacial acetic acid in 5 mL of water. Dissolve 3 g of zinc acetate $Zn(C_2H_3O_2)_2 \cdot 2H_2O$ and 0.1 mL of glacial acetic acid in 5 mL of water. Warm if necessary to complete solution. Mix the two solutions and store in a chemically resistant glass bottle. If precipitation occurs, filter the solution before use.

9. Sampling

9.1 Collect the sample in accordance with Practice D 887.

10. Laboratory Preparation of Samples

10.1 Prepare the sample in accordance with Practice D 2331.

10.2 Place a portion of the ground sample (approximately 0.1 g or less) in a porcelain crucible, add 4 drops of HNO_3(sp gr 1.42), and evaporate to dryness over the microburner. Add 1 mL of water, warm, and stir with a glass rod. Allow the insoluble material to settle. Withdraw portions of the supernatant liquid, henceforth referred to as the test solution, on the end of a glass rod and transfer to a slide for carrying out certain of the tests described in Section 11.

11. Chemical Procedures

11.1 The tests in this section are intended as an aid to the petrographic section of this practice. The sensitivity of these tests varies so that the operator should become familiar with each test to be able to judge semiquantitatively the amount of each constituent present based on the amount of sample used and the strength of the reaction observed. Some of these tests may not be necessary if spectrographic or X-ray diffraction equipment or both are available. For a more detailed discussion of these tests refer to Chamot and Mason **(3)** or to Feigl **(6).**

11.2 *Evolution of Gas with Dilute Acid*—Place a portion of the ground deposit on a slide and allow a drop of HCl (1+4) to flow into it. Observe macroscopically or under the 4× objective for evolution of gas bubbles which indicates that presence of carbonates, sulfites, sulfides, nitrites, or metals. Effereve-scense due to carbonates is usually violent and of short duration. The gas evolution due to sulfites, nitrites, and sulfides is usually less vigorous and there is a characteristic odor of the gas. Evolution of hydrogen gas from a metal is usually of considerable duration. Dry and examine the slide used for this test. If sodium salts are present, cubic crystals of sodium chloride will be formed. If appreciable amounts of calcium and sulfate ions were present, characteristic clumps of $CaSO_4 \cdot 2H_2O$ needles will be formed.

11.3 *Magnetic Material*—Place some of the ground sample on a slide and bring the magnet under the slide. As the magnet moves under the slide, any magnetic material in the sample will respond to the magnetic field.

NOTE 1—A coating of magnetite on nonmagnetic particles may give an erroneous indication of the total amount of magnetic material actually present.

11.4 *Water-Soluble Components*—Add a drop of water to a portion of ground sample on a microscope slide and warm over the microburner. Set aside to evaporate. If water-soluble components are present they will crystallize at the edge of the drop.

11.5 *Alkaline Material*—Spread some of the ground sample on a slide and cover with a drop of water. Allow a drop of phenolphthalein solution to flow into the drop. The presence of alkaline material will be indicated by the pink solution surrounding the alkaline components of the deposit.

11.6 *Combustible Matter*—Gently heat a portion of the ground sample in a porcelain crucible and note the odor. Heat more strongly and note the type of combustion which takes place and the volume of material that is lost. Rapid, spontaneous ignition may indicate nitrates. A glowing indicates carbon or organic matter. Substances like cotton, wool, rubber, sulfur, or sulfites can be identified by their odor. A steady luminous flame may indicate oil or asphalt.

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[5] The boldface numbers in parentheses refer to the references listed at the end of this practice.