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## Standard Practice for Examination of Water-Formed Deposits by Chemical Microscopy<sup>1</sup>

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 ( $\epsilon$ ) 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<sup>2</sup>
- D 1129 Terminology Relating to Water<sup>3</sup>
- D 1193 Specification for Reagent Water<sup>3</sup>
- D 2331 Practices for Preparation and Preliminary Testing of Water-Formed Deposits<sup>2</sup>
- D 2332 Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence<sup>2</sup>
- D 3483 Test Methods for Accumulated Deposition in a Steam Generator Tube<sup>2</sup>

### 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

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.02.

<sup>3</sup> *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 1/2 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.<sup>4</sup> 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<sub>4</sub>OH).

8.3 *Ammonium Molybdate Solution* (100 g/L)—Dissolve 1 g of ammonium molybdate ((NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O) in water, add 35 mL of nitric acid HNO<sub>3</sub>(sp gr 1.42) and dilute to 1 L with water.

8.4 *Ammonium Persulfate* —((NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>), crystalline.

8.5 *Barium Chloride Solution* (100 g/L)—Dissolve 100 g of barium chloride (BaCl<sub>2</sub>·2H<sub>2</sub>O) in water and dilute to 1 L.

8.6 *Cesium Sulfate*—Cs<sub>2</sub>SO<sub>4</sub> crystals, 10 to 20-mesh.

8.7 *Chloroform*.

8.8 *Chloroplatinic Acid Solution*—Dissolve 1 g of chloroplatinic acid H<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O 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<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> in water and dilute to 1 L.

8.10 *Dimethylglyoxime*, crystalline.

<sup>4</sup> *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<sub>3</sub>).

8.15 *Nitric Acid* (1+19)—Mix 1 volume of HNO<sub>3</sub> (sp gr 1.42) with 10 volumes of water.

8.16 *Phenolphthalein Indicator Solution*.

8.17 *Potassium Ferricyanide* [K<sub>3</sub>Fe(CN)<sub>6</sub>], crystalline.

8.18 *Potassium Iodide* (KI), crystalline.

8.19 *Potassium Mercuric Thiocyanate Solution* (100 g/L)—Prepare freshly precipitated mercuric thiocyanate Hg(CNS)<sub>2</sub> by adding a concentrated solution of mercuric nitrate Hg(NO<sub>3</sub>)<sub>2</sub> to a concentrated solution of potassium thiocyanate KCNS. Filter and air-dry the precipitate. To one part Hg(CNS)<sub>2</sub> 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<sub>2</sub>Hg(CNS)<sub>4</sub>, 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)<sup>5</sup> 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<sub>3</sub> in water, add 20 mL of HNO<sub>3</sub> (sp gr 1.42), and dilute to 1 L with water.

8.22 *Sodium Bismuthate*—Powdered NaBiO<sub>3</sub>.

8.23 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>).

8.24 *Sulfuric Acid* (1+19)—Add 1 volume of H<sub>2</sub>SO<sub>4</sub> (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<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>·2H<sub>2</sub>O and 0.1 mL of glacial acetic acid in 5 mL of water. Dissolve 3 g of zinc acetate Zn(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>·2H<sub>2</sub>O 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<sub>3</sub> (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. Efferevescence 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<sub>4</sub>·2H<sub>2</sub>O 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.

<sup>5</sup> The boldface numbers in parentheses refer to the references listed at the end of this practice.

11.7 *Metallic Elements*—To a portion of the ground sample on a slide, add a drop of  $\text{AgNO}_3$  solution and examine under low power. The presence of metallic iron, copper, or other metals will be confirmed by the formation of feathery crystals of metallic silver.

11.8 *Silicates*—To a portion of the ground sample on the slide, add HCl (sp gr 1.19) and warm. The presence of silicates which are attacked by acid will be indicated by the formation of a gelatinous residue on the surface of the test drop.

NOTE 2—The presence of large amounts of organic matter may obscure this test. Gentle ignition may permit removal of organic matter.

11.9 *Calcium*—Add a drop of  $\text{H}_2\text{SO}_4(1+19)$  to the preceding test drop. The presence of calcium will be confirmed by the formation of masses of radiating needles of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ .

11.10 *Orthophosphates*—To a portion of the ground sample add several drops of HCl (1+4) and take to dryness on the hotplate. Add several drops of  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$  solution and heat on the hotplate. Orthophosphates yield a lemon-yellow precipitate which is visible to the naked eye.

11.11 *Magnesium*—Place a drop of test solution on a slide, add a drop of  $\text{NH}_4\text{OH}$  (sp gr 0.90), and evaporate to dryness. Add a drop of  $\text{NH}_4\text{OH}$  to the residue and warm gently. Without disturbing the residue, draw the clear liquid to a clean area and evaporate to dryness. Then add a drop of water, allow to stand a few moments, and add a small drop of  $(\text{NH}_4)_2\text{HPO}_4$  and a drop of  $\text{NH}_4\text{OH}$  (sp gr 0.90). The presence of magnesium will be confirmed by the formation of large feathery stars and crosses which, on standing, develop into plates or rectangular prisms. If the amount of magnesium is low, a period of 1 min or more may be required for the development of the crystals.

11.12 *Aluminum*—Place a small portion of the ground sample on the end of a slide and add a small drop of  $\text{H}_2\text{SO}_4$  (sp gr 1.84). Evaporate to dryness over the microburner, cool, and add a drop of water and warm gently. Lead the solution to a clean portion of the slide; then add a crystal of  $\text{Cs}_2\text{SO}_4$ . If aluminum is present, crystals of cesium “alum” will separate in large, well-formed, colorless octahedra.

11.13 *Ferric Iron, Zinc, Copper, and Cobalt*—Place a drop of the test solution on a slide. Place a drop of  $\text{K}_2\text{Hg}(\text{CNS})_4$  solution beside the test drop and, by means of a glass rod, draw the drop of reagent into the test drop in such a manner as to form a narrow channel of liquid between the two drops. If the test solution does not turn blood red, ferric iron is absent. Examine the preparation under the  $10\times$  objective. The presence of zinc will be confirmed by the formation of white feathery crosses which appear black by transmitted light. The presence of copper will cause formation of greenish-yellow, mossy dendrites, or boat-shaped crystals, or both. Cobalt will give rise to deep blue orthorhombic prisms. If the test drop turns red, indicating the presence of ferric iron, take a test drop and evaporate to dryness with 1 drop of  $\text{NH}_4\text{OH}$  (sp gr 0.90). Then cover the residue with a drop of  $\text{NH}_4\text{OH}$  (sp gr 0.90) and warm gently. Without disturbing the residue, draw the drop to a clean area and evaporate to dryness. Then cover the residue with a drop of water, allow to stand for a few moments, and, with a glass rod, lead the solution to a clean area of the slide.

Add a drop of  $\text{K}_2\text{Hg}(\text{SCN})_4$  solution and examine under the  $10\times$  objective for zinc, copper, and cobalt as previously described.

11.14 *Sodium*—Evaporate a drop of test solution to dryness. Place a drop of water on the residue and then lead it to a clean area of the slide. Add a drop of zinc uranyl acetate solution. The presence of sodium is indicated by the formation of monoclinic crystals. These crystals are usually colorless but, if large, may be faintly yellow.

11.15 *Potassium*—Evaporate a drop of test solution to dryness and treat with a drop of water, after which transfer it to a clean area of the slide. Add a drop of  $\text{H}_2\text{PtCl}_6$  solution and observe under the  $10\times$  objective. Deep yellow octahedra are formed if potassium is present. The crystals may have a hexagonal aspect under certain conditions. If ammonium compounds are present, ignite a portion of the sample to drive off  $\text{NH}_3$  compounds before testing for potassium.

11.16 *Manganese*—Add 2 drops of HCl (sp gr 1.19) to a small portion of the ground sample on a slide and evaporate to dryness. Add 2 drops of  $\text{HNO}_3(1+19)$ , warm, and allow to cool. Add a small amount of powdered  $\text{NaBiO}_3$  and stir with a glass rod. The development of a magenta color indicates the presence of manganese.

11.17 *Lead*—To a drop of test solution, add a crystal of KI. The presence of lead will be indicated by the formation of bright yellow, thin scales and hexagonal plates that may appear greenish, brownish, or even gray by transmitted light.

11.18 *Nickel*—To a large drop of test solution add  $\text{NH}_4\text{OH}$  (sp gr 0.90) and warm gently. Draw the solution from the insoluble material by means of a glass rod to a clean slide and evaporate to dryness. Treat the residue with additional  $\text{NH}_4\text{OH}$  and add to the original drop. In a clean area of the slide prepare a dimethylglyoxime solution by dissolving several crystals in a drop of water. Allow the two drops to run together. A rose-pink or magenta-colored precipitate will be formed if nickel is present. Under high power the precipitate will be shown to consist of fine needles.

11.19 *Ferrous Iron*—Add several drops of HCl (1+4) to a small portion of the ground sample on a slide. Place the slide on the hotplate and take to dryness. Add several drops of HCl (1+4), heat, then add several crystals of  $\text{K}_3\text{Fe}(\text{CN})_6$ . A dark blue color indicates the presence of ferrous iron. Strong reducing agents may interfere with this test.

11.20 *Sulfate*—Place a small portion of the ground sample in a 30-mL beaker, add several mL of HCl (1+4) and heat on the hotplate for 15 to 20 min adding HCl (1+4) to prevent drying. Filter into a clean beaker, add 10 drops of  $\text{BaCl}_2$  solution, and heat on the hotplate. A fine white precipitate indicates the presence of sulfate.

11.21 *Sulfide*—Place a small portion of the ground sample in a 30-mL beaker and cover with an equal amount of zinc dust. Place a moistened strip of lead acetate paper over the mouth of the beaker and add several drops of HCl (1+4). A darkening of the test paper indicates the presence of sulfide. Sulfite will be reduced by zinc dust and will give a positive test.

11.22 *Chloride*—Place several millilitres of the test solution in a 30-mL beaker and dilute to about 10 mL with water. Add several drops of AgNO<sub>3</sub> reagent. A white precipitate indicates the presence of chloride.

11.23 *Organic Phosphate*—To a portion of the ground sample on a slide add several drops of HCl (1+4) and several small crystals of ammonium persulfate and take to dryness on the hotplate. Organic phosphates will be oxidized to orthophosphate which is tested for as described in this method. Since orthophosphate will be included in this test its presence should be determined first.

## 12. Petrographic Procedure

NOTE 3—Detailed procedures for petrographic examination are described in the literature (1, 2, 3, 4, 5, 7, 8).

12.1 *Determination of Crystalline Habit*—Place a drop of immersion liquid (index of refraction,  $N = 1.57$ ) on a slide and mark the index of refraction of the liquid on the end of the slide. Sprinkle some of the ground sample over this liquid and cover with a cover glass. Examine under 300× to 500×. Observe and record any unique crystal habit or cleavage. Insert the upper polar and rotate the stage. Record whether isotropic or anisotropic crystals, or both, are present. Isotropic crystals, and anisotropic crystals oriented with an optic axis parallel to that of the microscope, will remain dark in all positions of rotation. Anisotropic crystals will be alternately bright and dark on rotation of the stage and will exhibit either two or three indices of refraction.

12.2 *Determination of Extinction Angle*—If any of the crystals is elongated, determine the extinction angle. The extinction angle is observed between the extinction position and some recognizable crystallographic direction such as a cleavage plane. Measure the angle by means of the graduated scale on the rotating stage. Position the crystal carefully so that the intersection of the cross hairs intersects the cleavage plane.

12.3 *Determination of Index of Refraction*—Use the Becke line test to determine whether the particle under examination has an index above or below that of the liquid in which the crystal is immersed. The slide prepared in accordance with 13.1 may be used for the preliminary examination.

12.3.1 Choose a crystal near the center of the field and focus the microscope. Partially close the diaphragm of the substage condenser in order to obtain central illumination. Raise the tube or lower the stage of the microscope by means of the fine adjustment and observe a narrow white line which will appear just inside or outside the boundary of the fragment with the surrounding liquid. If, on raising the tube, or lowering the stage, the line moves into the crystal, the index of refraction of the crystal is greater than that of the liquid. If the index of refraction of the crystal is less than that of the liquid, the line will move into the liquid. If the indices of the crystal and the liquid are identical, the boundary of the crystal will become very difficult to see. Due to the dispersion of the liquid, that is, the variation of index of refraction of the liquid with wave length, the boundary will not disappear. Complete disappearance can occur only if monochromatic light is used.

NOTE 4—The determination of refractive indices of any component in a mixture is complicated by the necessity of recognizing the component in

the series of liquid mounts. The material must be sufficiently characteristic in appearance to be recognized from mount to mount without reasonable doubt.

12.3.2 Choose a recognizable component of the deposit for determination of index of refraction. Choose an appropriate liquid and prepare a second slide with the higher or lower liquid, whichever is indicated by the preliminary test. By this procedure a liquid will be found that most nearly matches the index of the crystal. If the crystal, on rotation of the stage, alternately darkens and becomes bright, the crystal is anisotropic and may exhibit two or three indices of refraction. Choose a particle that shows a high order interference color. Turn the stage to extinction, remove the upper polar, and apply the Becke line test. Rotate the stage 90 deg and repeat the test. Prepare additional slides with different liquids until the highest and lowest indices of the component are determined. The highest and lowest indices of the crystal will not be exhibited unless the crystal is exactly oriented relative to the plane of the lower polar. The crystals which exhibit the highest order of interference color between crossed polars are most nearly oriented in the proper position to exhibit the highest and lowest indices of refraction. An anisotropic crystal which remains dark, or nearly so, on rotation of the stage will exhibit the beta index of refraction for a biaxial crystal and the omega index of refraction for a uniaxial crystal.

12.4 *Sign of Elongation*—If the crystals are elongated, record the sign of elongation. The sign of elongation is positive in those crystals in which the higher index is exhibited parallel to the axis of elongation and negative in those in which the lower index is exhibited in this position.

12.5 *Dispersion Staining*—Use the dispersion staining device and carefully adjust axial illumination; examine the slides prepared for the determination of refractive index. Components of the deposit will display varying colors dependent upon the refractive index of the liquid, refractive index of the crystals, and use of either central or annular screening. If a refractive index of one of the components matches or nearly matches that of the immersion liquid and the slide is viewed using annular screening, that component will show a color that corresponds to the wavelength at which the dispersion curves of the liquid and the crystal intersect; other components on the slide will show other colors or remain uncolored. If central screening is used, the colors seen will be complementary to those seen with annular screening. By this method it is possible to differentiate components with similar morphological and optical properties. Dispersion staining also aids in estimating amounts of the components present, in recognizing constituents present in trace quantities, in identifying very small particles, and in searching for suspected components.

## 13. Interpretation of Data

13.1 Identification of crystals is made by comparing the determined optical data with that which is published in the literature or with data determined from pedigreed samples. The refractive indices are the most important of the optical constants, and often identification can be made by this determination alone. Comparison with pedigreed samples from the user's own system is strongly recommended. The analyst should be aware of such samples when they are encountered. They should



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be retained, and properly labelled with date, location, significant operating parameters, and constituents of interest. Confirmatory information, from other optical data and from chemical, spectrographic, and X-ray diffraction results make the identification more positive.

### 14. Keywords

14.1 crystallography; deposits; microchemistry; microscopy; qualitative analysis; refractive index

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