



Designation: D934 – 08

Standard Practices for Identification of Crystalline Compounds in Water-Formed Deposits By X-Ray Diffraction¹

This standard is issued under the fixed designation D934; 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 for X-ray diffraction analysis of powdered crystalline compounds in water-formed deposits. Two are given as follows:

	Sections
Practice A—Camera	12 to 21
Practice B—Diffractometer	22 to 30

1.2 Both practices yield qualitative identification of crystalline components of water-formed deposits for which X-ray diffraction data are available or can be obtained. Greater difficulty is encountered in identification when the number of crystalline components increases.

1.3 Amorphous phases cannot be identified without special treatment. Oils, greases, and most organic decomposition products are not identifiable.

1.4 The sensitivity for a given component varies with a combination of such factors as density, degree of crystallization, particle size, coincidence of strong lines of components and the kind and arrangement of the atoms of the components. Minimum percentages for identification may therefore range from 1 to 40 %.

1.5 *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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8 and Note 20.

¹ These practices are under the jurisdiction of ASTM Committee D19 on Water and are 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.

Current edition approved Oct. 1, 2008. Published November 2008. Originally approved in 1947. Last previous edition approved in 2003 as D934 – 80 (2003). DOI: 10.1520/D0934-08.

2. Referenced Documents

- 2.1 *ASTM Standards*:²
 - D887 Practices for Sampling Water-Formed Deposits
 - D933 Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits
 - D1129 Terminology Relating to Water
 - D1193 Specification for Reagent Water
 - D2331 Practices for Preparation and Preliminary Testing of Water-Formed Deposits
 - E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

- 3.1 For definitions of terms used in these practices, refer to Terminology D1129.

4. Summary of Practices

4.1 Powdered samples are irradiated with a monochromatic X-ray beam of short wavelength (from about 0.05 to 0.25 nm). The X rays interact with the atoms in the crystal and are scattered in a unique diffraction pattern which produces a fingerprint of the crystal's atomic or molecular structure. The analytical instrumentation used in X-ray diffraction includes the powder camera and the diffractometer (**1-5**).³

5. Significance and Use

5.1 The identification of the crystalline structures in water-formed deposits assists in the determination of the deposit

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 The boldface numbers in parentheses refer to the references listed at the end of these practices.

sources and mode of deposition. This information may lead to measures for the elimination or reduction of the water-formed deposits.

6. Purity of Reagents

6.1 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

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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.

6.2 *Purity of Water*—Unless otherwise indicated, 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 **D1193**, Section 1.1.

7. Sampling

7.1 Collect the sample in accordance with Practices **D887**.

7.2 A suitable amount of sample should be obtained so that it is representative of the deposit under investigation.

7.3 Deposits shall be removed and protected in such a way that they remain as nearly as possible in their original states.

8. Safety Precautions

8.1 The potential danger of high-voltage and X-ray radiation makes it mandatory for anyone operating X-ray apparatus to be thoroughly familiar with basic safety precautions.

8.2 Place colorful signs displaying the international radiation symbol near X-ray equipment.

8.3 When X-ray equipment is producing radiation, illuminate a conspicuous light. There should be no X rays if the bulb burns out. Equipment without this feature can be modified.

8.4 Use a portable counter periodically to test for leakage of X rays from equipment. Lead or lead glass shielding is sometimes needed. X rays of shorter wavelength require more caution.

8.5 Film badges, dosimeters, or other monitoring devices shall be worn by personnel who regularly work with X-ray equipment.

9. Preliminary Testing of Analytical Sample

9.1 It may be advantageous and even necessary to perform other analytical investigative methods to aid in the rapid identification of crystalline components in water-formed deposits. For other testing methods refer to Practices **D2331**.

10. Preparation of Sample

10.1 *Apparatus*—The apparatus used for preparing the sample is as follows:

10.1.1 *Mullite or Agate Mortar*.

10.1.2 *Sieves*—A series of sieves from No. 100 mesh (150- μm) to No. 325 mesh (45- μm) as specified in Specification **E11**.

10.1.3 *Soxhlet Extractor*.

10.2 *Procedure*—The following procedure is to be used in preparing the sample:

10.2.1 Air-dry moist samples before grinding. If there is special need to preserve the nature or composition of the original deposit, special handling must be observed. Handle deliquescent deposits in a dry-box atmosphere. Handle samples subject to oxidation in an inert atmosphere.

10.2.2 If samples contain oil or grease, prepare a chloroform-insoluble fraction by first drying the specimen for 1 h at 105°C and then extracting for 2 h using chloroform in a Soxhlet extracting apparatus. Air-dry to remove solvent from specimen.

10.2.3 Grind the sample in a mullite or agate (mechanical or hand) mortar until approximately 98 % passes a No. 325 mesh (45- μm) sieve (see **Note 1**). Remove fragments of fiber, wood, and metal. If the specimen is not sufficiently brittle at ordinary temperatures to be ground to a fine powder or if it is suspected that certain crystallites may be plastically deformed during the grinding, the deposit can be subjected to dry-ice temperatures and then ground immediately. Grind hydrated samples under alcohol, if indicated, to prevent structural damage.

NOTE 1—Most materials found in water-formed deposits are sufficiently brittle to be reduced to 45 μm and this crystallite dimension will generally give good identifiable diffraction patterns. However, it may not always be practical or possible to reduce certain materials to 45 μm . Often good diffraction results can be obtained from larger crystallite sizes (No. 200 mesh (75- μm) to No. 270 mesh (53- μm)). The only practical test for proper grain size is in reproducibility of diffraction line intensities. The ideal grain size may be in the subsieve range as small as 1 μm , but reduction to this size may be impractical.

11. Selective Segregation of Analytical Sample

11.1 *Chemical and Physical Treatment of Samples*—Depending on the contents of the sample, it may or may not be necessary to concentrate or segregate components by chemical or physical treatment (see **Note 2**). Many crystalline materials produce sharp diffraction patterns and they are identifiable when present to 1 or 2 % in a mixture. Other substances that can be readily identified alone are difficult to detect in mixtures even when they are present to the extent of 50%. Separation of phases by density, acid solubility, or magnetic properties followed by diffraction analysis of the separated phase(s) may help to identify various deposit components. Separation treatment is also helpful in resolving line coincidence in complex mixtures. If concentration or segregation is not deemed necessary, disregard any treatment and proceed in accordance with Section 10. When treatment is necessary, use one or more of the following chemical or physical treatments described in **11.2** to **11.7**. It must be pointed out that the treatments provide no absolute separation, but serve only to concentrate or partially segregate specific components.

NOTE 2—It should be emphasized that water-formed deposits often occur in clearly defined layers and that physical separation at the time of sampling is more advantageous than later treatment.

11.2 *Water-Insoluble Fraction*—This treatment removes the water soluble from the water insoluble components. Soluble constituents would include most sodium, potassium, and lithium compounds (see **Note 3**).

11.2.1 Weigh 0.5 g or more of sample that has been ground and passed through a No. 100 mesh (150- μm) sieve. Add 100 mL of water to a beaker containing the powdered specimen.

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 *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.

Heat to boiling and then cool. Allow 30 min of reaction time, filter through a 45- μ m membrane filter, wash, and air-dry the residue. Regrind to pass through a No. 325 mesh (45- μ m) sieve.

NOTE 3—The filtrate may be evaporated and the residue examined by diffraction. Although the crystalline structure may have changed from the original sample, it is often helpful in identifying simplified variations of the original crystals. Sodium phosphate compounds found dispersed in boiler deposits are often noncrystalline or are so complex that they are not easily identified. The water soluble residue from these deposits after evaporation is often more easily identified.

11.3 Hydrochloric Acid-Insoluble Fraction—This treatment removes carbonates, phosphates, and hydroxides. Partial decomposition occurs to some silicates such as serpentine, xonotlite, and analcite. Components such as anhydrite undergo only partial dissolution.

11.3.1 Reagent—The reagent used for this treatment is as follows:

11.3.1.1 Hydrochloric Acid (1 + 6)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 6 volumes of water.

11.3.2 Weigh approximately 0.5 g of the sample that has been ground and passed through a No. 100 mesh (150- μ m) sieve. Add 100 ml of HCl (1 + 6) to a beaker containing the powdered specimen. Allow 30 min of reaction time, filter through a membrane filter, wash and air-dry the residue. Regrind to pass through a No. 325 mesh (45- μ m) sieve.

11.4 Nitric Acid-Insoluble Fraction—This treatment removes all the components indicated in **11.3** in addition to copper and most copper compounds.

11.4.1 Reagent—The reagent used for this treatment is as follows:

11.4.1.1 Nitric Acid (1 + 13)—Mix 1 volume of concentrated HNO_3 (sp gr 1.42) with 13 volumes of water).

11.4.2 Weigh 0.5 g or more of the sample which has been ground and passed through a No. 100 mesh (150- μ m) sieve. Add 100 ml of HNO_3 (1 + 13) to a beaker containing the powdered specimen. Allow 30 min of reaction time, filter through a membrane filter, wash and air-dry the residue. Regrind to pass through a No. 325 mesh (45- μ m) sieve.

11.5 Density Fraction—This treatment separates compounds in water-formed deposits which differ appreciably in density such as copper oxide and calcite.

11.5.1 Weigh 0.5 g or more of sample that has been ground and passed through a 100 mesh (150- μ m) sieve. Add 100 mL of water to a beaker containing the powdered specimen. Stir the liquid with a mechanical stirrer, but do not use a magnetic stirrer (**Note 4**). The denser particles will settle to the bottom and the less dense particles will remain suspended. With continued stirring, withdraw sufficient liquid and filter through a membrane filter. Varying the speed of the stirrer will put more or less of the powdered sample into suspension. Air-dry the residue and regrind to pass through a No. 325 mesh (45- μ m) sieve.

NOTE 4—A magnetic stirrer would attract any particle that was magnetic and might prevent segregation of particles of different densities.

11.6 Magnetic and Nonmagnetic Fraction—This treatment separates the magnetic components from the nonmagnetic components such as magnetite from hydroxyapatite.

11.6.1 Weigh 0.5 g or more of sample that has been ground and passed through a No. 325 mesh (45- μ m) sieve. Add 100 mL of water to a beaker containing the powdered specimen. Stir with a mechanical stirrer for several minutes and then attach a magnet to the outside of the beaker (see **Note 4**). While stirring continues, magnetic components will be attracted to the area in front of the magnet, while the nonmagnetic components will remain in suspension. After 30 min, pour off the liquid while the magnet remains in place and filter. The residue should be essentially nonmagnetic. Remove the magnet from the side of the beaker, rinse the magnetic portion down the side of the beaker, swirl, and filter. Air-dry the residues and regrind to pass through a No. 325 mesh (45- μ m) sieve.

11.7 Brittle Fraction—This treatment concentrates the more brittle or friable components from those that are less brittle or less friable such as calcite from silica.

11.7.1 Apparatus—The apparatus necessary for this treatment is as follows:

11.7.1.1 Electrical Mechanical Sieve Shaker.

11.7.2 Weigh 0.5 g or more of sample and hand grind in a mullite mortar until the largest particle size is approximately 1 mm in diameter. Set up the sieve-shaker apparatus with four different size sieves; No. 100 mesh (150- μ m), No. 140 mesh (105- μ m), No. 200 mesh (75- μ m) and No. 325 mesh (45- μ m). Shake the sample for several minutes and then collect the various fractions from each sieve. The most friable portion of the deposit should have passed to the bottom-most sieve. Each fraction may be examined separately after regrinding to pass a No. 325 mesh (45- μ m) sieve.

PRACTICE A—CAMERA

12. Scope

12.1 This method covers the qualitative X-ray diffraction analysis of powdered crystalline substances using photographic film as the detector. The film-camera technique has the advantage of being less expensive initially and less costly to maintain. The camera practice permits the use of smaller samples. Film cameras do not require the well-stabilized power supply as electronic detection techniques do. Often faint diffraction lines are more readily detected with film.

13. Summary of Practice A

13.1 Practice A utilizes the Debye-Scherrer type camera. In this method a powdered sample is placed in the center of a camera cylinder and a narrow film is wrapped around the inner wall. A monochromatic X-ray beam is directed upon the sample and the randomly oriented crystallites diffract the incident beam into a set of concentric cones in accordance with Bragg's Law ($n\lambda = 2d \sin \theta$). The interceptions of the diffraction cones on the positioned film strip result in curved lines on that film. The spacings of these lines are used to identify the crystalline material.

14. Interferences

14.1 Large particles in the powder mount give spotty or discontinuous diffraction lines. A particle size that is too small will cause broadening of the diffraction lines.

14.2 For certain elements the absorption of the X rays is relatively high. This is especially true for the heavier elements when longer wavelength X rays are used (see **Note 5**). Therefore, larger samples of such elements can actually give rise to fainter diffraction lines than would be obtained from a smaller sample of the same element. Excessively large mounts should be avoided if the elemental composition of the sample has high absorption for a selected radiation.

NOTE 5—Values for absorption by different elements with various commonly-used X-ray sources can be found in the International Crystallographic Tables.

14.3 The proper filters should be used to remove beta radiation and to ensure essentially monochromatic radiation from the target.

14.4 Preferred orientation of crystallites will produce diffraction lines that are not of uniform intensity around the entire circumference.

14.5 Fluorescent radiation should be avoided if possible since it will obscure certain diffraction lines. This radiation may be minimized by selecting the incident-beam wavelength as far removed as possible from the *K* and *L* absorption edges of metallic elements expected in the deposit. If a prominent metallic element exists in the sample under investigation, a target of the same metal should be used if possible.

NOTE 6—Copper radiation is not as satisfactory as other recommended radiations for specimens containing considerable iron or iron bearing compounds because of excessive fluorescence, which will produce fogging on the film. However, copper targets are by far the most widely used and most generally applicable.

15. Apparatus

15.1 *Beta Filters*—Free metals or metal oxides filters of vanadium, manganese, iron, nickel, and zirconium. For the choice of filter refer to the following list (see **Note 7**):

X-ray Target Material	Filter
Chromium	Vanadium
Iron	Manganese
Cobalt	Iron
Copper	Nickel
Molybdenum	Zirconium

NOTE 7—The filter shall be of sufficient thickness to reduce the intensity of the K_{β} radiation to 1/100 of the K_{α} radiation. Monochromator crystal attachments are available for many cameras which will eliminate the use of beta filters and produce clean diffraction lines corresponding to a single X-ray wavelength with low background.

15.2 *Camera*, Debye-Scherrer type usually 57.3 or 114.6 mm in diameter so that 1 mm of film equals 2 or 1 degs, respectively. Features of the camera should include: (1) a device to hold film flush against the cylinder, (2) a sample holder with provision to center the mount, (3) a device for rotating or oscillating sample, (4) a collimating system to produce a narrow and nondivergent beam, and (5) a beam stop or exit tube that prohibits the direct beam from exposing the film or escaping into the room.⁴

15.3 X-ray Equipment⁵:

⁵ The equipment listed is generally sold as a package by most manufacturers of X-ray diffractometers. The International Union of Crystallography (I.C.Cr.) publishes an Index of Crystallographic Supplies and lists various suppliers.

15.3.1 *X-ray Generator*, with stable high-voltage electrical supply.

15.3.2 *X-ray Generator Control Unit*, containing the following: (1) a milliammeter connected across the secondary circuit to indicate current flow through the X-ray tube, (2) a warning light in full view that indicates that X rays are being produced, (3) an overload relay to trip the circuits when the tube current exceeds normal operating values, (4) a pressure switch in the water line to trip the circuits if the tube receives an insufficient flow of cooling water, (5) an electric timer that will shut off the unit at the end of a preset time, (6) a time totalizer that indicates the total time the generator has been on, and (7) interlocking safety switches that turn off the high voltage circuit if the panels of the unit are opened.

15.4 *Filtered Cooling Water System*—Installed prior to X-ray equipment.

15.5 *X-ray Source*—A number of X-ray targets are available that produce K_{α} radiation of various desired wavelengths. They include: Targets of chromium, iron, cobalt, copper, and molybdenum.

16. Reagents and Material

16.1 *Capillary Tubes*—Diameter 0.1 to 1.0 mm thin-walled, nonabsorbing glass or quartz. Also, fine glass fibers 0.1 to 0.5-mm diameter.

16.2 *Collodion*, gum tragacanth, or other suitable amorphous binders.

16.3 *Film*—X-ray film with the following characteristics: (1) fine-grained texture and homogenous emulsion, (2) high sensitivity for the wavelength of the selected radiation, (3) low fogging and low expansion due to changes in temperature and humidity. The film should be of the screenless type (see **Note 8**). All film should be handled and processed in accordance with manufacturer's recommendations.

NOTE 8—If molybdenum radiation is used, a screen-type film with the fluorazure type intensifying screen may be used.

17. Preparation of Test Specimen

17.1 Prepare the powdered specimens in such a shape or form as required by the camera used.

17.2 Common Mounts:

17.2.1 *Rod Mount*—A few milligrams of the powdered sample are mixed with an amorphous binder such as collodion or gum tragacanth and rolled into a rod 0.1 to 1.0-mm diameter.

17.2.2 *Coated Glass Fiber Mount*—A fine glass fiber is dipped in collodion or some amorphous glue and rolled in the powdered sample to coat the outer cylindrical surface. Photographs made with glass fibers usually show one or two diffused halos at about 0.42 nm (see **Note 9**).

17.2.3 *Capillary Mount*—Small diametered nonabsorbing glass tubes can be filled with the powdered sample and then compacted by dropping through a 1 to 2-ft (0.3 to 0.6-m) length of small diameter glass tubing. The capillary can be sealed with wax if desired to prevent hydration, oxidation, or carbonation (see **Note 9**).

17.2.4 *Ball Mount*—A small amount of powdered sample is mixed with collodion and rolled into a ball and then glued onto a small glass fiber.

17.2.5 *Wedge Mount*—The powdered sample is pressed into a wedge-shaped cavity which can be supported without a binder.

NOTE 9—Glass fibers, and thin-walled tubes contribute scattering to the diffraction pattern. Wall thicknesses of the capillary tubes and diameters of fibers should be kept to minimum. Amorphous substances contribute blackening of the film at low Bragg angles.

18. Calibration and Standardization

18.1 Prepare standard X-ray diffraction film patterns from pure compounds and mixtures which are likely to occur in water-formed deposits. These patterns can be compared with standard films which are available from various companies. An alternative and more precise identification method is one in which the d-spacings are obtained from carefully measured diffraction angles and compared with published authenticated data of interplanar spacings and relative intensities (see [Note 10](#) and [Note 11](#)). [Table 1](#) lists some of the more common crystalline compounds found in water-formed deposits, and the relative intensities of the three strongest lines.

NOTE 10—The International Centre for Diffraction Data has established a Powder Diffraction File which operates on an international basis and cooperates closely with the Data Commission of the International Union of Crystallography and ASTM International. New data for over 1000 compounds are issued yearly.⁶

NOTE 11—A method of indexing the standard patterns is described by Hanawalt, Rinn, and Frevel.⁷

19. Procedure for Taking Patterns

19.1 Place the mounted specimen in the camera as recommended by the apparatus manufacturer. Line up the specimen so that the diffracted beam will emerge from the specimen at the axial center of the camera. Specimen centering devices are available through the manufacturer.

19.2 Select the proper X-ray tube for the desired radiation (see [Note 12](#), [Note 13](#), and [Section 12.5](#)). Do not touch the tube windows.

NOTE 12—If costs prohibit the purchase of more than one X-ray tube, one with a copper target will generally give the best results for the majority of compounds.

NOTE 13—The proper care of X-ray tubes includes regular use to prevent a gassy condition, cleaning the cooling water screens to prevent flow blockage, carefully cleaning electrical contacts to make a good electrical contact, and gentle handling.

19.3 Load with film and mount the camera on the X-ray unit in front of the X-ray tube window in such a manner as to produce a strong, uniform beam through the collimating system. Shield the other tube windows to protect the operator and prevent stray radiation from fogging the film during exposure.

⁶ The data are available on cards, tapes, or in book form and can be purchased from the International Centre for Diffraction Data, 1601 Park Lane, Swarthmore, PA 19081.

⁷ Hanawalt, J. D., Rinn, H., and Fevel, L. K., “Chemical Analysis by X-Ray Diffraction,” *Industrial Engineering Chemistry*, Analytical Edition, IECHA, Vol 10, 1939.

NOTE 14—There are several types of film loading in Debye-Scherrer cameras, including Bradley-Jay, Van Arkle, Straumanis, and Wilson. The Straumanis method, which is the most popular, contains mounting holes for both the beam catcher and collimator.

19.4 Be sure all requirements specified by the manufacturer have been fulfilled and that all safety precautions have been taken before activating the X-ray tube. Apply the proper power to the tube as specified by the manufacturer.

19.5 Expose the film by setting the timer for suitable period of time. Exposure time depends upon several factors: (1) crystal structure of deposit, (2) characteristic wavelength of X-ray tube target, (3) the shape of the sample and type of mount, (4) elemental composition of the material, (5) absorption coefficient for a given radiation, and (6) type of film.

19.5.1 Deposits with imperfect crystal structures usually require longer exposure times. A denser phase of a compound generally requires shorter exposure time.

19.6 After proper exposure, shut off apparatus power, remove camera and process the film in accordance with the film manufacturer’s instructions.

NOTE 15—The exposure and development time of the film shall be such that maximum contrast is obtained between pattern lines and film background when using a good diffractor such as sodium chloride.

20. Identification of Patterns⁸

20.1 Place the pattern of the test specimen beside that of a standard on a viewing light so that the corresponding edges of the patterns come together and the centers of diffraction ([Note 16](#)) lie on the same vertical line. Visually scan the specimen pattern for the strong lines occurring in the standard pattern. Find by repeated trial the standard pattern whose strong lines match strong lines of the specimen pattern when compared in the above manner. The substance represented by the standard pattern shall be considered to be a component of the test specimen if at least the five strongest lines ([Note 17](#)) of the standard pattern occur in the specimen pattern in the corresponding positions and with the correct relative intensities. When making this comparison, due regard must be given to the possibility of increased intensity of certain lines because of the superposition of lines of more than one component. Considering those lines of the specimen pattern not accounted for by the above match, including those of greater than normal intensity, repeat the matching with standard patterns until all the lines of the specimen pattern are accounted for.

NOTE 16—The center of diffraction of the undeviated beam is found by locating the mid-point between corresponding lines on either side of the central spot.

NOTE 17—When other evidence, such as chemical analysis, spectrographic analysis, microscopic or petrographic examination, supports the finding of a component in a specimen, fewer lines may be considered to constitute an identification.

20.2 When there are lines in the test specimen pattern that cannot be matched by the available standard patterns, determine their interplanar spacings (see [Note 18](#)) and relative

⁸ A more thorough and comprehensive approach to pattern identification can be found in *X-ray Diffraction Procedures*, by Klug and Alexander, 1954, pp. 396-402 (see [Reference 3](#)).