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An American National Standard

Standard Guide for Forensic Analysis of Geological Materials by Powder X-Ray Diffraction[1]

This standard is issued under the fixed designation E3294; 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 guide covers techniques and procedures for the use of powder X-ray diffraction (XRD) in the forensic analysis of geological materials (to include soils, rocks, sediments, and materials derived from them such as concrete), to enable non-consumptive identification of solid crystalline materials present as single components or multi-component mixtures.

1.2 This guide makes recommendations for the preparation of geological materials for powder XRD analysis with adaptations for samples of limited quantity, instrumental configuration to generate high-quality XRD data, identification of crystalline materials by comparison to published diffraction data, and forensic comparison of XRD patterns from two or more samples of geological materials to support criminal investigations.

1.3 *Units*—The values stated in SI units are to be regarded as standard. Other units are avoided, in general, but there is a long-standing tradition of expressing X-ray wavelengths and lattice spacing in units of Ångströms (Å). One Ångström = 10^{-10} meter (m) = 0.1 nanometer (nm).

1.4 This standard is intended for use by competent forensic science practitioners with the requisite formal education, discipline-specific training (see Practice E2917), and demonstrated proficiency to perform forensic casework.

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 establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1365 Test Method for Determination of the Proportion of Phases in Portland Cement and Portland-Cement Clinker Using X-Ray Powder Diffraction Analysis

D934 Practices for Identification of Crystalline Compounds in Water-Formed Deposits By X-Ray Diffraction

E620 Practice for Reporting Opinions of Scientific or Technical Experts

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

E3272 Guide for Collection of Soils and Other Geological Evidence for Criminal Forensic Applications

2.2 *ISO Standard:*[3]

ISO/IEC 17025:2017 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Bragg equation* or *Bragg's law, n*—describes the physical phenomenon of X-ray scattering from a crystallographic three-dimensional lattice plane as $n\lambda=2d\sin\theta$, in which n is any integer, λ is the wavelength of the X-ray, d is the crystal plane separation, also known as d-spacing, and θ is the angle between the crystal plane and the diffracted beam, also known as the *Bragg Angle*.

3.1.2 *crystal, n*—a homogeneous, solid body of a chemical element or compound, having a regularly repeating atomic arrangement that can be outwardly expressed by plane faces (adapted from Ref (**1**)).[4]

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.15 on Trace.

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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] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

3.1.3 *crystal lattice, n*—the three-dimensional regularly repeating set of points that represent the translational periodicity of a crystal structure.

3.1.3.1 *Discussion*—Each lattice point has identical surroundings. Lattice is the abstract pattern used to describe the internal geometric structure of crystals. Lattice and structure are not synonymous, as structure refers to the real mineral material (adapted from Ref (**1**)).

3.1.4 *crystalline, adj*—having a crystal structure or a regular arrangement of atoms in a crystal lattice.

3.1.5 *d-spacing, n*—in diffraction of X-rays by a crystal, the distance or separation between successive and identical parallel planes in the crystal lattice; *d*-spacing is expressed as *d* in the Bragg equation (adapted from Ref (**1**)).

3.1.6 *diffractometer, n*—an instrument that records either powder or single-crystal X-ray diffraction patterns.

3.1.7 *known sample, n*—known samples of geological material are intentionally collected, typically from crime scene or alternate locations, for comparison to a questioned sample.

3.1.7.1 *Discussion*—Geological materials are typically more heterogeneous than manufactured materials, so a greater number of known samples of geological material are needed to represent the range of variation (see Guide E3272). *Reference sample* and *control sample* are synonyms of *known sample*.

3.1.8 *mineral, n*—a naturally occurring inorganic element or compound having an orderly internal structure and characteristic chemical composition, crystal form(s), and physical properties, or an element or chemical compound that is crystalline and that has formed as a result of geological or pedogenic (soil-formed) processes (adapted from Ref (**1**)).

3.1.8.1 *Discussion*—Artificial and biogenic crystalline materials are not minerals but can occur in geological materials (for example, cement powder, lime, lye, biogenic calcite, biogenic hydroxyapatite, bricks) and can be detected by XRD.

3.1.9 *phase, n*—a part of a chemical system that is homogeneous, physically distinct and at least hypothetically separable, and which has single or continuously variable chemical and mechanical properties (adapted from Ref (**1**)).

3.1.10 *provenance, n*—a place of origin; specifically, the area from which the constituent materials of a sedimentary rock or facies are derived (adapted from Ref (**1**)).

3.1.10.1 *Discussion*—In the context of forensic provenance analysis, geological material is analyzed and interpreted to estimate or limit the geographic or environmental conditions of the source of this material to provide an investigative lead. For example, soil on a shovel can be examined to aid in the search for a clandestine grave, typically by comparison of observations to reference data. *Geographic attribution* is an alternative term for *provenance*.

3.1.11 *questioned sample, n*—geological evidence of unknown origin, or a *questioned sample*, typically consists of debris adhering to an evidentiary object (for example, tire, wheel well, garment, shoe, digging tool); exogenous soil left at a crime scene (transferred from a shoe/tire, or adhering to a re-buried body/object); or debris recovered from within a body (nasal, stomach, or lung contents).

3.1.12 *Rietveld refinement, n*—a procedure for carrying out a crystal-structure refinement using X-ray or neutron powder diffraction data, in which an entire powder pattern is simulated for a trial structure(s) and matched against the observed powder pattern; atomic parameters and other variables are modified to achieve an acceptable fit between the calculated and observed powder patterns (adapted from Ref (**1**)).

3.1.13 *unit cell, n*—the smallest group of atoms of a crystal lattice that has the overall symmetry of a crystal of that substance, and from which the entire lattice can be built up by repetition in three dimensions.

3.1.14 *X-ray diffraction (XRD), n*—the diffraction of a beam of X-rays, usually by the three-dimensional periodic array of atoms in a crystal that has periodic repeat distances (lattice dimensions) of the same order of magnitude as the wavelength of the X-rays (**1**).

3.1.15 *X-ray diffraction pattern* or *diffractogram, n*—the characteristic interference pattern obtained when X-rays are diffracted by a crystalline substance; the geometry of the pattern is a function of the repeat distances (lattice dimensions) of the periodic array of atoms in the crystals; the intensities of the diffracted beams give information about the atomic arrangement, and unit-cell dimensions (adapted from Ref (**1**)).

3.1.16 *X-ray powder diffraction, n*—diffraction of a beam of X-rays by planes of atoms in a powdered crystalline sample; the powders are prepared so they ideally represent all possible crystal orientations to the X-ray beam (adapted from Ref (**1**)).

4. Summary of Guide

4.1 Powder X-ray diffraction produces results related to the crystal structure(s) of one or more crystalline components of the material being analyzed that can allow phase identification.

4.2 This guide recommends specific techniques and procedures for XRD analysis of geological materials in forensic casework, including XRD analysis of minimally modified materials, small quantities of material (a common limitation in forensic casework), and in situ XRD of material adhering to a substrate.

4.3 XRD patterns are compared to reference databases as means of identifying the crystalline constituents of a sample.

4.4 XRD can be used to determine the crystal structure of a material, but this is not described in this guide.

4.5 XRD patterns from various samples are compared to each other for forensic comparison and provenance purposes.

5. Significance and Use

5.1 The overarching goals of the forensic analysis of geological materials include (*A*) identification of an unknown material (see 11.3), (*B*) analysis of soils, sediments, or rocks to restrict their possible geographic origins as part of a provenance analysis (see 11.4), and (*C*) comparison of two or more samples to assess if they could have originated from the same source or to exclude a common source based on observation of exclusionary differences (see 11.5). XRD is only one analytical method that can be applied to the evidentiary samples in