



Standard Guide for Forensic Analysis of Explosives By Polarized Light Microscopy¹

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1. Scope

1.1 This guide addresses the use of polarized light microscopy (PLM) to identify explosive-related compounds from intact explosives and post-blast residues containing unconsumed explosive compounds or their solid reaction products, and to isolate them for further analysis.

1.2 This guide 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 (refer to the T/SWGFEX Suggested Guide for Explosives Analysis Training).

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

1.4 *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

- E620 Practice for Reporting Opinions of Scientific or Technical Experts
- E860 Practice for Examining and Preparing Items That Are or May Become Involved in Criminal or Civil Litigation
- E1732 Terminology Relating to Forensic Science
- E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E3196 Terminology Relating to the Examination of Explosives

E3253 Practice for Establishing an Examination Scheme for Intact Explosives

E3255 Practice for Quality Assurance of Forensic Science Service Providers Performing Forensic Chemical Analysis

E3329 Practice for Establishing an Examination Scheme for Explosive Residues

2.2 Other Resources

T/SWGFEX Suggested Guide for Explosive Analysis Training³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide other than those listed in 3.2, see Terminologies E1732 and E3196.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *alpha (α)*—symbol representing the lowest of the three principal refractive indices of a biaxial crystal.

3.2.2 *anomalous interference colors, n*—atypical polarization colors which occur due to variation with wavelength of the refractive index (very few substances display this characteristic).

3.2.3 *Becke line, n*—a halo observed near the boundary of a transparent particle that is mounted in a medium that differs from the particle's refractive index.

3.2.4 *Becke line method, n*—method for determining the refractive index of a transparent particle relative to its mountant by noting the direction in which the Becke line moves when the focus is changed.

3.2.4.1 *Discussion*—The Becke line always moves toward the higher refractive index medium (particle or mountant) when the focus is raised, and towards the lower refractive index medium when the focus is lowered. At the point where the index of the particle matches the index of the mounting medium, the Becke line is no longer visible. The Becke line is generally viewed at a wavelength of 589 nm (the D line of sodium [n_D]).

³ Available from the Technical/Scientific Working Group for Fire and Explosion Analysis (T/SWGFEX), https://www.nist.gov/system/files/documents/2018/09/21/twgfex_suggest_guide_for_explosive_analysis_training.pdf.

3.2.5 *beta* (β)—symbol representing the intermediate principal refractive index of a biaxial crystal.

3.2.6 *biaxial, adj*—an anisotropic crystal in the orthorhombic, monoclinic, or triclinic system with three principal refractive index directions (α , β , γ) and two optic axes that are isotropic.

3.2.7 *birefringence, n*—the numerical difference between the maximum and minimum refractive indices of anisotropic substances.

3.2.8 *crystal system, n*—crystals are classified according to their crystallographic axes length and the angles between them. There are six crystal systems: cubic, tetragonal, hexagonal, orthorhombic, monoclinic, and triclinic. All crystal systems are anisotropic except for the cubic system, which is isotropic.

3.2.9 *epsilon* (ϵ)—any vibration direction in the plane of the c axis for uniaxial crystals.

3.2.10 *euhedral, adj*—well-formed crystals bounded by plane faces.

3.2.11 *extinction, n*—condition when an anisotropic substance appears dark when observed between crossed polarizers and occurs when the vibration directions of the specimen are parallel to the vibration directions of the polarizer and analyzer.

3.2.12 *extinction, oblique, n*—a type of extinction in which the vibration directions are inclined at an angle relative to a crystal face.

3.2.13 *extinction, parallel, n*—a type of extinction in which the vibration directions are parallel to the crystal faces.

3.2.14 *extinction, symmetrical, n*—a type of extinction in which the vibration directions bisect a prominent interfacial angle of the crystal.

3.2.15 *fusion methods, n*—A process in which compounds are heated on a microscope slide and observed via PLM during heating, melting, and, upon cooling, recrystallization of the melt.

3.2.15.1 *Discussion*—Fusion methods frequently employ a temperature controlled hot stage capable of at least 300 °C placed on the stage of a polarizing light microscope.

3.2.16 *gamma* (γ)—symbol representing the highest of the three principal refractive indices of a biaxial crystal.

3.2.17 *interference figure, n*—pattern observed during conoscopic observation of an anisotropic material which consists of a combination of extinction positions and interference colors corresponding to the full cone of directions by which the sample is illuminated.

3.2.17.1 *Discussion*—Conoscopic observations are typically made by inserting a Bertrand lens into the body tube or by removing an eyepiece and viewing down the body tube.

3.2.18 *omega* (ω)—any vibration direction in the plane of the a axis for uniaxial crystals.

3.2.19 *optic axial angle* ($2V$), *n*—the acute angle between two optic axes of a biaxial crystal.

3.2.20 *optic axis, n*—a direction of isotropic light propagation in an anisotropic crystal. Uniaxial crystals have one optic axis; biaxial crystals have two optic axes.

3.2.21 *optic sign, n*—determined by the relationship of the refractive indices of a material. For uniaxial crystals, if $\epsilon > \omega$, the crystal is positive (+); if $\omega > \epsilon$, the crystal is negative (-). For biaxial crystals, if $\gamma - \beta > \beta - \alpha$, the crystal is positive (+); if $\gamma - \beta < \beta - \alpha$, the crystal is negative (-).

3.2.22 *polymorphism, n*—crystallization into two or more chemically identical but crystallographically distinct forms.

3.2.23 *relief, n*—contrast between a particle or crystal and its media due to the difference between their refractive indices. The greater the numerical difference in refractive indices, the greater the relief.

3.2.24 *subhedral, adj*—crystals with imperfectly developed faces.

3.2.25 *uniaxial, adj*—an anisotropic crystal in the tetragonal or hexagonal system having one optic axis (isotropic direction) and either two (tetragonal) or three (hexagonal) directions which are alike and perpendicular to the direction of the optic axis.

4. Summary of Practice

4.1 Solid chemical components in samples are analyzed using a polarized light microscope (PLM). The optical crystallographic properties such as refractive index, birefringence, and morphology are determined. Determination of these properties, which are different for various explosive components and other chemical compounds, can aid in the identification of explosives and explosive residues submitted to the forensic laboratory.

4.2 Optical crystallographic properties of an unknown component can be determined by either mounting the component in a refractive index liquid, recrystallizing the component from a small drop of solvent, or recrystallizing the component from its melt, that is, recrystallization occurring when a melted compound is cooled. Recrystallized components can be isolated for further analysis using other analytical techniques such as Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, or scanning electron microscopy-energy dispersive x-ray spectroscopy (SEM-EDS).

4.3 Water-soluble components, such as inorganic salts used as oxidizers, are dissolved in a drop of water and tested using reagents to form characteristic microcrystals. Microcrystal tests can aid in the determination of anions and cations present in oxidizers and other components of explosives.

4.4 The optical crystallographic properties of an inorganic compound and microcrystal testing for its ions are independent techniques that provide complementary information. A combination of tests that includes the optical crystallographic characterization of a compound and microchemical tests to confirm the anion and cation are used to identify oxidizers and other salts present in explosives.

4.5 A combination of tests that include optical crystallographic characterization or microchemical tests in conjunction with other independent techniques are used to identify chemical components present in explosives. Refer to Practices [E3253](#) and [E3329](#) for the requirements for the identification of

commonly encountered explosives and chemical components present in explosive materials.

5. Significance and Use

5.1 This guide is designed to assist the analyst using polarized light microscopy for the examination of test samples for the presence of explosives.

5.2 Intact explosives and post-blast residues that contain unconsumed explosive components or their solid reaction products are suitable for this guide (Practices [E3253](#) and [E3329](#)). Particles only a fraction of a millimeter in size can be examined using this guide.

5.3 This guide is not suitable for the examination of smokeless powder or liquid explosives.

5.4 Some of the microscopical techniques described in this guide allow for the recovery of the specimen for subsequent analysis, but others, such as microcrystal tests, are destructive.

5.5 Identifications of explosive-related components based on these properties shall be part of an analytical scheme as described in Practices [E3253](#) and [E3329](#).

6. Apparatus

6.1 *Polarized Light Microscope*—A compound microscope with a rotating stage, polarizing filters, substage condenser, and compensator slot.

NOTE 1—Most commercially available polarized light microscopes should be adequate for the forensic analysis of explosives and appropriate for use following this guide.

6.2 *Objectives*—Strain-free typically 10×, 20×, and 40×.

6.2.1 A 40× objective with a numerical aperture of 0.65 or higher is recommended to provide the widest view of the back focal plane for conoscopic observations.

6.2.2 A 10× dispersion staining objective is required if performing dispersion staining.

6.3 *Compensator*—A full wave compensator for use with the microscope. Optional compensators include quarter-wave plate and quartz wedge.

6.4 *Hot Stage*—A temperature controlled hot stage for use with the microscope with a range of approximately 30 °C to 300 °C.

6.5 *Alcohol Lamp*.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. It is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. 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.⁴

7.2 *10 % Platinum Chloride* (H_2PtCl_6) in water.

7.3 *Zinc Uranyl Acetate*.

7.4 *Ammonium Molybdate* $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O})$.

7.5 *Squaric Acid*.

7.6 *Potassium Iodide* (KI).

7.7 *Nitron Sulfate*.

7.8 *Strychnine Sulfate*.

7.9 *0.3 % Methylene Blue* in deionized water (Methylene Blue Reagent).

7.10 *20 % Potassium Nitrate* (KNO_3) in deionized water.

7.11 *Saturated Zinc Sulfate* (ZnSO_4) in deionized water.

7.12 *Toluene or Methylene Chloride*.

7.13 *Sodium Hydroxide*.

7.14 *Deionized water*.

7.15 *Reference Materials*, including reagent grade chemicals and explosive materials, can be obtained from commercial and retail sources or directly from the distributor or manufacturer.

7.16 *Refractive Index Liquids* with known temperature coefficients (dn/dt) and tolerance at n_D .

7.17 *Microscope Slides and Coverslips*.

7.18 *Glass Rod* with a tapering tip of approximately 1 mm or less in diameter.

7.19 *Wood Toothpicks*.

7.20 *Glass Ring* with an approximate diameter of 10 mm to 12 mm and 5 mm to 10 mm in height.

7.21 *Tungsten Needles*.

7.22 *589 nm Light Filter*.

8. Initial Microscopical Examination

8.1 Preliminary assessment of the samples using visual and stereo microscopical examinations are conducted following Practices [E3253](#) and [E3329](#). If preliminary examinations indicate that the sample is an explosive mixture or a component thereof, a portion of the sample is examined by PLM in order to obtain further information regarding the composition of the sample.

⁴ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.