OSAC 2022-S-0023
Standard Practice for the Forensic Analysis of Explosives By Polarized Light Microscopy

Ignitable Liquids, Explosives, and Gunshot Residue Subcommittee
Chemistry: Trace Evidence Scientific Area Committee (SAC)
Organization of Scientific Area Committees (OSAC) for Forensic Science

Draft OSAC Proposed Standard

OSAC 2022-S-0023

Standard Practice for the Forensic Analysis of Explosives By Polarized Light Microscopy

Prepared by
Ignitable Liquids, Explosives, and Gunshot Residue Subcommittee
Version: 1.0
February 2021

Disclaimer:

This OSAC Proposed Standard was written by the (subcommittee) of the Organization of Scientific Area Committees (OSAC) for Forensic Science following a process that includes an open comment period. This Proposed Standard will be submitted to a standards developing organization and is subject to change.

There may be references in an OSAC Proposed Standard to other publications under development by OSAC. The information in the Proposed Standard, and underlying concepts and methodologies, may be used by the forensic-science community before the completion of such companion publications.

Any identification of commercial equipment, instruments, or materials in the Proposed Standard is not a recommendation or endorsement by the U.S. Government and does not imply that the equipment, instruments, or materials are necessarily the best available for the purpose.

To be placed on the OSAC Registry, certain types of standards first must be reviewed by a Scientific and Technical Review Panel (STRP). The STRP process is vital to OSAC's mission of generating and recognizing scientifically sound standards for producing and interpreting forensic science results. The STRP shall provide critical and knowledgeable reviews of draft standards or of proposed revisions of standards previously published by standards developing organizations (SDOs) to ensure that the published methods that practitioners employ are scientifically valid, and the resulting claims are trustworthy.

The STRP panel will consist of an independent and diverse panel, including subject matter experts, human factors scientists, quality assurance personnel, and legal experts, which will be tasked with evaluating the proposed standard based on a comprehensive list of science-based criteria.

For more information about this important process, please visit our website at: https://www.nist.gov/topics/organization-scientific-area-committees-forensic-science/scientific-technical-review-panels.

Standard Practice for the Forensic Analysis of Explosives By Polarized Light Microscopy

---

1. Scope

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

1.2 This practice 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 practice does not purport to address all of 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

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 Related to Forensic Science
E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Program
WK56998 Terminology Relating to the Examination of Explosives
WK67862 Practice for Establishing an Examination Scheme for Intact Explosives

2.2 Other Resources

Technical/Scientific Working Group for Fire and Explosion Analysis (T/SWGFEX) Suggested Guide for Explosive Analysis Training (https://www.nist.gov/system/files/documents/2018/09/21/twgfex_suggest_guide_for_explosive_analysis_training.pdf)

3. Terminology

3.1 Definitions - For definitions of terms used in this guide other than those listed in 3.2, see E1732 and WK56998.

3.2 Definitions of Terms Specific to This Standard:

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

anomalous interference colors, n – atypical polarization colors which occur due to variation with wavelength of the refractive index; very few substances display this characteristic.

Becke line, n – a halo observed near the boundary of a transparent particle when mounted in a medium that differs from its refractive index.

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.

Discussion--The Becke line always moves toward the higher refractive index medium (particle or mountant) when the focus is raised (stage is lowered) ,and towards the lower refractive index medium when the focus is lowered (stage is raised). 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]).

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

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.

birefringence, n - the numerical difference between the maximum and minimum refractive indices of anisotropic substances: $\varepsilon - \omega$ for uniaxial crystals and $\gamma - \alpha$ for biaxial crystals

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.

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

euhedral, adj - well-formed crystals bounded by plane faces

eutectic, n - the lowest melting mixture of two or more substances miscible in the melt state.

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.

extinction, oblique, n - a type of extinction in which the vibration directions are oblique to the long direction of the crystal

extinction, parallel, n - a type of extinction in which the vibration directions are parallel and perpendicular to the long direction of the crystal

extinction, symmetrical, n - a type of extinction in which the vibration directions bisect a prominent interfacial angle of the crystal

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

interference figure, n – pattern observed during conoscopic examination 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.

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.

Discussion—The procedure frequently employs a temperature controlled hot stage capable of at least 300^{O} Celsius placed on the stage of a polarizing light microscope.

omega (ω) - Any vibration direction in the plane of the a axes for uniaxial crystals

optic axial angle (2V), n - the acute angle between two optic axes of a biaxial crystal.

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.

optic sign, n - determined by the relationship of the refractive indices of a material; for uniaxial crystals: if $\varepsilon > \omega$, the crystal is positive (+); if $\omega > \varepsilon$, 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 (-)

polymorphism, n - crystallization into two or more chemically identical but crystallographically distinct forms.

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.

subhedral, adj - crystals with imperfectly developed faces

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 present in explosives 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 the various explosive components, 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 mounting in a refractive index liquid, by recrystallizing the component from a small drop of solvent, or recrystallizing from its melt, i.e., 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 electron spectroscopy (SEM-EDS).

4.3 Water-soluble components present in explosives, such as inorganic salts used as oxidizers, are dissolved in 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 a compound and microchemical testing for its ions are independent techniques that provide complementary information. A combination of tests that include 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 are used in conjunction with other independent techniques such as FTIR or SEM-EDS to identify chemical components present in explosives. Refer to ASTM WK67892 for the requirements for the identification of commonly encountered explosives and chemical components present in explosive materials.

5. Significance and Use

5.1 This practice is designed to assist the analyst using polarized light microscopy for the identification of explosives.

5.2 Intact explosives and post-blast residues that contain unconsumed explosive components are suitable for this practice.

5.3 Polarized light microscopy is not appropriate for the examination of smokeless powder.

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

6. Apparatus

6.1 Polarized light microscope - A compound microscope with a rotating stage, polarizing filters, condenser and compensator slot. Most commercially available polarized light microscopes should be adequate for the forensic analysis of explosives.
6.2 Objectives - Strain-free 10X, 20X and 40X. A 40X 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.
6.3 Compensator - A full wave compensator for use with the microscope.
6.2 Hot stage - a temperature controlled hot stage for use with a microscope with a range of approximately 30ºC to 300ºC.
6.5 The best practice for documenting some microscopical observations, such as crystal formations, is to take a digital photograph. It is advised that the minimum equipment required also has the capability of digital photography.
6.6 Alcohol lamp
6.7 Glass ring with a diameter of approximately 10-12 mm and 5-10 mm in height

7. Reagents and Materials

7.1 Microscope slides and cover slips
7.2 Refractive index oils
7.3 Reagents for microcrystal tests: chloroplatinic acid, zinc/uranyl acetate, ammonium molybdate, squaric acid, potassium iodide, nitron sulfate, strychnine sulfate, methylene blue
7.4 Reference materials, including reagent grade chemicals and explosive materials, can be obtained from commercial and retail sources or directly from the distributor or manufacturer.

7.5 Distilled or deionized water
7.6 Toluene or methylene chloride, reagent grade
7.7 Sodium hydroxide, reagent grade
7.8 Glass rod with a tapering tip of approximately 1 mm or less in diameter
7.9 Glass ring with an approximate diameter of 10-12 mm
7.10 Tungsten needles
7.11 589 nm light filter

8. Initial Microscopical Examination

8.1. If preliminary examinations, such as stereomicroscopical observations, ignition susceptibility test (IST), and chemical spot tests indicate that the specimen is an explosive mixture or a component thereof, 1-3 particles or grains (approximately 1 mg or less) are mounted on a glass microscope slide with a cover slip and dispersed in a refractive index oil. Any refractive index oil in the 1.400 to 1.660 range can be used, however, if preliminary examinations indicate a particular explosive material, e.g., black powder, ammonium nitrate-fuel oil (ANFO) or Composition C-4, the examiner can select a refractive index oil that will provide characteristic optical crystallographic data based upon Tables 1 and 2.

8.2 Examine the mounted sample using a polarized light microscope using a 10X objective. Increase magnification as needed.

8.3 Charcoal, if present, will appear as irregularly shaped, black and opaque particles, and as brown flakes often with cellular features such as pits or cell walls.

8.4 Most common explosives and oxidizers are colorless when viewed with a transmitted light microscope. Using crossed polarizers, determine if the crystals are isotropic or anisotropic. Assess the interference colors to determine if the birefringence is low (less than 0.01), moderate (0.01-0.10) or high (0.10 and above).

8.5 Determine relative refractive indices of crystals present in the sample using the Becke line method.

8.6 The morphology, relative refractive indices and interference colors can indicate the presence of an explosive ingredient or chemical component. If explosive related components are indicated, additional microscopical analysis is performed to further characterize the components via optical crystallography. If inorganic salts are indicated, additional microscopical analysis can also be performed by microcrystal tests.

9. Optical Crystallography by Solvent Recrystallization

9.1 Solvent recrystallization allows for a more detailed optical crystallographic characterization of a compound as it recrystallizes from a small drop of suitable solvent.

9.2 Water recrystallization for inorganic oxidizers and salts

9.2.1 Add 1-3 particles or grains (approximately 1 mg or less) of the specimen to a small drop (5-7 mm in diameter) of distilled or deionized water on a microscope slide. It may be necessary to crush and disperse the sample with the tip of a glass rod. Alternatively, a portion of the specimen can be mixed with water and filtered to remove water insoluble components such as carbon, aluminum, and sulfur; a small drop of the filtered water extract is then placed on a microscope slide.

9.2.2 Examine the test drop using the PLM. Crystals typically form at the edge of the drop first. Distorted or subhedral crystals, if formed, can be pushed back into the center of the drop using a glass rod or metal probe to produce characteristic euhedral crystals. Multiple water-soluble components can be present in the sample and, depending on their concentration and solubility, can recrystallize at different times as the drop evaporates.

9.2.3 Examine the crystals using plane-polarized and crossed-polarized light. Note their crystal habit, relief, and whether they are isotropic or anisotropic.

9.2.4 For anisotropic crystals, determine if extinction is parallel, symmetrical, or oblique.

9.2.5 Assess the interference colors to determine if the birefringence is low (less than 0.01), moderate (0.01-0.10), or high (0.10 and above).

9.2.6 If observable, use interference figures to determine if the crystal is uniaxial or biaxial and the optic sign. Interference figures can also be used to relate morphology of the crystal to its optical orientation, and, if the crystal is biaxial, estimate 2V.

9.2.7 Compare formed crystals to known controls or published references. Table 1 lists optical crystallographic properties for eleven common oxidizers. Document the crystals by description, sketch, or photomicrographs.

9.2.8 Dried crystals can be mounted in refractive index oils for refractive index determination, analyzed via microcrystal tests or other instrumental techniques (WK 67862). If the crystals cannot be further analyzed while on the glass slide, use a tungsten needle to transfer the crystals from the glass slide to another substrate, such as a potassium bromide pellet, for additional analysis.

9.3 Organic solvent recrystallization for sulfur

9.3.1 Add 1-3 particles or grains (approximately 1 mg or less) of sample to a small drop (5-7 mm in diameter) of a suitable solvent such as toluene or methylene chloride. Sulfur, if present, will recrystallize as highly refractive, birefringent dipyramids (diamond-shaped) or form liquid drops that crystallize when touched with a tungsten needle.

9.3.2 Document the crystals by description, sketch, or photomicrograph.

9.3.3 Compare formed crystals to known controls or published references. Table 1 lists optical crystallographic properties for sulfur.

9.3.4 Dried crystals can be further analyzed by other analytical techniques such as Raman or SEM-EDS. If the crystals cannot be further analyzed while on the glass slide, use a tungsten needle to transfer crystals from the glass slide to another substrate, such as carbon tape, for additional analysis.

10. Optical Crystallography Using Refractive Index Liquids

10.1 Organic and inorganic compounds present in explosives are characterized by their optical crystallographic properties by mounting intact material in a liquid of known refractive index.

10.2 Mount 1-3 particles or grains (approximately 1 mg or less) of the specimen, or dried crystals obtained during recrystallization, as described in Section 9, in a refractive index oil. If previous examinations indicate a particular compound, mount in a refractive index oil to confirm one or more known refractive indices of that compound.

10.3 Examine using PLM to determine the optical crystallographic properties such as refractive indices, birefringence, extinction type (parallel, symmetrical or oblique), optic sign, and optic axial angle (2V).

10.4 Refractive index may be determined using the Becke line method or dispersion staining.

10.5 Compare the optical crystallographic properties of the specimen to known reference materials or published references (6, 8, 12, 13, 22). Table 1 lists optical crystallographic properties for sulfur, ascorbic acid, sucrose and twelve common oxidizers. Table 2 lists optical crystallographic properties for four organic high explosives.

Table 1. Optical Crystallographic Properties of Common Fuels & Oxidizers

Compound	Crystal System	Refractive Indices/ Optic Angle	Habit	Comments
S (**12**)	orthorhombic	1.958 2.038 2.245 2V = 68^{O}(+)	dipyramids	High relief in solvent. Larger crystals appear yellowish in color.
Ascorbic Acid (**22**)	monoclinic	1.474 1.595 1.746 2V = 88^{O}(+)		High order interference colors
Sucrose (**12, 22**)	monoclinic	1.540 1.567 1.572 2V = 48^{O}(-)		
$NaClO_3$ (**8**)	cubic	1.518	squares, rectangles	
$Ba(NO_3)_2$ (**8**)	cubic	1.571	cubes octahedra	
$Sr(NO_3)_2$ (**6**)	cubic	1.586	octahedra	
$Pb(NO_3)_2$ (**8**)	cubic	1.781	octahedra cubes	High relief in water
$KClO_4$ (**8**)	orthorhombic	1.4731 1.4737 1.4769 2V = 50^{O}(+)	rectangular prisms, rhombs	Low order interference colors
NH_4ClO_4 (**8**)	orthorhombic	1.4818 1.4833 1.4881 2V = 70^{O}(+)	six-sided prisms	Low order interference colors. Crystals lying on a {110} face will exhibit an off-centered biaxial interference figure.
$NaClO_4$ (**8**)	orthorhombic	1.4606 1.4617 1.4730 2V = 69^{O}(+)	See $NaClO_4.2H_20$	Sodium chlorate recrystallizes as the hydrate. Optical data is for the anhydrous.
$NaClO_4.2H_20$ (**8**)	monoclinic		diamonds, rhombs	Low relief in water. Some orientations exhibit oblique extinction. Refractive indices not known.
$KClO_3$ (**8**)	monoclinic	1.415 1.517 1.523,	diamond-shaped tablets, often truncated	Moderate order interference colors, symmetrical extinction

		$2V = 28^{O}(-)$		
KNO_3 (**8**)	hexagonal & orthorhombic	1.3346 1.5056 1.5064 $2V = 7^{O}(-)$	rhombohedra & chevrons early; prisms late	The hexagonal forms with high order interference colors appear first at the edge of the drop. Using a glass rod to move them to the middle of the drop causes the stable orthorhombic forms to appear.
NH_4NO_3 (**8, 9**)	orthorhombic	1.413 1.611 1.637 $2V = 35^{O}(-)$	prisms, as blades & rods	Crystal formation is slow due to the high solubility of ammonium nitrate. The resulting crystals exhibit high order interference colors. As a fusion preparation cools, three to four polymorphs are observed. MP = 169
$NaNO_3$ (**8**)	hexagonal	1.5874 (ω) 1.3361 (ε)	rhombohedra	High order interference colors and symmetrical extinction.

Table 2. Crystallographic and Optical Properties of High Explosives

Compound	Crystal System	Optical Properties	Habit	Comments
RDX (**13**)	orthorhombic	1.578 1.597 1.602 $2V = 53^{O}(-)$		Exhibits anomalous interference colors. MP = 204-206
HMX (**13**)	monoclinic	1.589 1.594 1.73 $2V = 20^{O}(+)$	dipyramids & pinacoids, rare acicular	Typically exhibits high order interference colors. Oblique extinction
TNT (**13**)	orthorhombic	1.543 1.674 1.717 2V = 60 (-)		MP = 81
PETN (**13**)	tetragonal	ω = 1.556 ε = 1.551		MP = 141.5-142

11. Microcrystal tests

11.1 Microcrystal tests are chemical precipitation reactions where the target substance precipitates with the reagent. The crystals formed are examined using a polarized light microscope. Microcrystal tests are used in conjunction with other independent techniques to identify explosive compounds.

11.2 Microcrystal tests are used to test for anions and cations of oxidizers and other substances, such as aluminum, that are present in explosives. Many microcrystal tests are available, but those commonly used in explosives analysis are listed in Table 3.

11.3 Prior to casework, the reagents used for microcrystal tests shall be tested for reliability using the appropriate chemical standard and negative controls. Once it is determined that the reagents are producing the expected response, the reagents can be used for testing case specimens.

11.4 There are four routine methods for conducting microcrystal tests (**4**). Test drops and reagent drops used for microcrystal tests should be approximately 5-7 mm in diameter or smaller. The reactions normally take place within one minute.

11.4.1 Method 1

11.4.1.1 Place a drop of deionized water on a clean microscope slide and add one or more particles of the sample with stirring, crushing and dispersing as needed. Keep this test drop small and from spreading on the slide.

11.4.1.2 A drop of reagent is placed near, but not touching, the test drop. Depending on the microcrystal test being conducted some reagent solutions are pre-made and a drop added to the slide. Other reagent drops are created on the slide by placing a drop of water near the test drop and adding 1-5 particles of the reagent to the water droplet.

11.4.1.3 Place the slide on the microscope stage.

11.4.1.4 Using a clean glass rod, draw one drop into the other drop and immediately examine the preparation microscopically using a 10X objective. Higher magnification may be used but care must be taken so the objective does not contact the test drop.

11.4.1.7 Observe the entire preparation including the mixing zone and within each drop. Note the presence of characteristic microcrystals or the lack of a reaction.

11.4.1.8 The formation of microcrystals corresponding to those obtained with the chemical standard is indicative for that anion or cation.

11.4.2 Method 2

11.4.2.1 Follow 11.4.1 except add the reagent drop into the middle of the test drop.

11.4.2.2 Observe the preparation microscopically as described in Method 1. Note the presence of characteristic microcrystals or the lack of a reaction.

11.4.2.3 The formation of microcrystals corresponding to those obtained with the chemical standard is indicative for that anion or cation.

11.4.3 Method 3

11.4.3.1 Follow 11.4.1 except add reagent particles into the middle of the test drop.

11.4.3.2 Observe the preparation microscopically as described in Method 1. Note the presence of characteristic microcrystals or the lack of a reaction.

11.4.3.3The formation of microcrystals corresponding to those obtained with the chemical standard is indicative for that anion or cation.

11.4.4 Method 4

11.4.4.1. Place a small drop of deionized water on a clean microscope slide and add one or more particles of the sample with stirring, crushing, and dispersing as needed. Keep this test drop small and from spreading on slide.

11.4.4.2 Place a glass ring with an internal diameter of about 10-12 mm around the test drop so the drop is in the center of the ring.

11.4.4.3 Place a small drop of prepared reagent on a coverslip.

11.4.4.5 Place the slide on a microscope stage making sure there is sufficient working distance between the top of the glass ring and the objective.

11.4.4.6 Add a small drop of dilute (approximately 10-30%) sodium hydroxide to the test drop inside the center of the ring.
11.4.4.7 Immediately place the coverslip onto the glass ring so the reagent drop is suspended in the center of the ring above the sample.
11.4.4.8 Observe the reagent drop for the presence of microcrystals or the lack of reaction.
11.4.4.9 The formation of microcrystals corresponding to those obtained with the chemical standard is indicative for that anion or cation.

11.5 Table 3 lists twelve tests for anions and cations common to low explosives along with the reagent, literature reference, and method as described in 11.4. Microcrystal tests for additional ions can be found in Chamot & Mason (**4**), Benedetti-Pichler (**21**), Wills (**19**), and Hollifield (**20**).

Table 3. Microcrystal Tests for Ions

Cation/anion tested	Microcrystal test & reference	Method
Potassium	Chloroplatinic acid (**4**)	1, if positive, repeat using Method 4, if potassium present and ammonium ion absent method 4 test will be negative
Ammonium	Chloroplatinic acid (**4**)	4
Sodium	zinc/uranyl acetate (**4**)	4
Aluminum	ammonium molybdate (**4**)	2
Magnesium	Squaric acid (**19, 20**)	2
Barium	Squaric acid (**19, 20**)	2
Strontium	Squaric acid (**19, 20**)	2
Lead	Potassium iodide (**4**)	1
Nitrate	Nitron sulfate (1, **4**)	1
Perchlorate	Strychnine sulfate (**4**)	1, 3
Perchlorate	Methylene Blue (**7**)	1
Chlorate	Methylene Blue (**7**)	1

12. Fusion Methods

12.1 Fusion methods (10) are techniques that allow for the characterization of crystals by determining the melting point and observing optical crystallographic properties as the sample recrystallizes from the melt. This method is most useful for organic high explosives (11, 14, 15, 16) but can also be applied to some inorganic explosives (9).

12.2 The solid sample is placed on a microscope slide with a cover slip, or on a cover slip (according to manufacturer's instructions), placed in the hot stage, and brought into the field of view. The temperature in the hot stage is raised as the sample is observed using the polarized light microscope. The melting point is documented and the temperature is lowered slowly while observing the recrystallization of the sample from the melt.

12.3 Note the optical crystallographic properties such as morphology, crystal front shape, polymorphism, interference colors, anomalous interference colors, extinction characteristics, interference figure, and optic sign for each solid state as appropriate.

12.4 Most inorganic oxidizers found in low explosives have melting points above 300°C. These higher melting oxidizers are heated on a microscope slide using an alcohol lamp (9). An accurate melting point is not determined, but the sample is observed through the microscope as the slide cools to obtain various optical crystallographic properties, as described in 12.3.

12.5 Compare the optical crystallographic properties of the specimen to published reference descriptions (9-11, 14-16) or a known reference material.

12.6 Mixed fusions

12.6.1 If an explosive compound is indicated using optical crystallography, microcrystal tests, or other techniques, a mixed fusion using a known reference material of the suspected compound can confirm the identity of the unknown (9, 10).

12.6.2 A small amount of material from the specimen is placed near the edge of a coverslip, melted over an alcohol lamp, and allowed to flow under part of the coverslip. Limit the amount of specimen so that the entire area under the coverslip is not filled with the specimen.

12.6.3 Once the unknown specimen has cooled and recrystallized, the known reference material is placed near the opposite edge of the coverslip and melted over an alcohol lamp such that the melted standard flows under the coverslip and contacts the previously cooled unknown specimen. The portion of the unknown specimen that contacts the reference material will also melt during this process.

12.6.4 Using the PLM, view the zone of mixing as the preparation cools and recrystallizes. If the unknown compound recrystallizes into the reference material without differences in velocity, appearance, or the presence of a eutectic, this is strong evidence that the two compounds are the same.

12.6.5 Once cooled, the mixed fusion preparation can be inserted into the hot stage at a temperature approximately 10 degrees lower than the melting point of the reference material. While observing the zone of mixing, slowly raise the temperature (1-3 degrees/minute) of the hot stage to the melting point of the reference material If the two compounds are the same, the melting point will be the same across the zone of mixing. Note: this can only be performed if the sample melts below the upper temperature range of the hot stage.

13. Documentation

13.1 All details necessary to support the interpretation made from the examinations are documented.

13.2 Examination notes are reviewed to ensure they reflect the particular characteristics used in the microscopical examination including measured, observed, or calculated values, descriptions, sketches, or photomicrographs.

13.3 Examination documentation is reviewed to ensure they contain sufficient detail to support the conclusions such that another qualified examiner could independently reach the same conclusion based on the notes or documentation.

14. References

1. Lawrence, G., and Fink, M., "The Evaluation of Nitron Sulfate as a Microchemical Test for Some Common Oxidizers," *The Microscope*, Vol 55, No. 2, 2007, pp 55-58.

2. Parker, R. G., Stephenson, M. O., McOwen, J. M., and Cherolis J. A., “Analysis of Explosives and Explosive Residues. Part 1: Chemical Tests, ” *Journal of Forensic Science*, Vol 20, No. 1, January 1975.

3. Chamot E. M., and Mason C. W., *Handbook of Chemical Microscopy*, *Volume 1* John Wiley & Sons, Inc., New York, NY, 1958, p. 51.

4. Chamot, E., and Mason, C., *Handbook of Chemical Microscopy, Volume II*, John Wiley & Sons, Inc., New York, NY, 1940.

5.Hopen, T. J., “Tricks of the Trade, Extraction and Identification of Sulfur,” *The Microscope*, Vol 48, No. 1, 2000, p. 51.

6. Winchell A. N., *The Microscopic Characters of Artificial Inorganic Solid Substances or Artificial Minerals*, John Wiley & Sons, Inc., New York, NY, 1931.

7. Hopen T. J., and Crippin J. B., “Methylene Blue Microchemical for the Detection and Identification of Chlorates and Perchlorates,” *The Microscope*, Vol 49, 2001.

8. Hopen T.J., and Kilbourn J. H., “Characterization and Identification of Water Soluble Explosives,” *The Microscope*, Vol 33, No. 1, 1985.

9. Kilbourn J. H., and McCrone W. C., “Fusion Methods Identification of Inorganic Explosives,” *The Microscope*, Vol 33, No. 2, 1985.

10. McCrone W. C., *Fusion Methods in Chemical Microscopy: A Textbook and Laboratory Manual*, Interscience Publishers, Inc., New York, NY, 1957.

11. McCrone W. C., “The Identification of High Explosives by Microscopic Fusion Methods,” *Microchemical Journal*, Vol 3, 1959, pp 479-490.

12. McCrone W. C., and Delly J. G., *The Particle Atlas, Edition 2, Volumes I, II & IV*, Ann Arbor Science Publishers, Inc., Ann Arbor, MI, 1973.

13. McCrone W. C., Delly J. G., and Palenik S.J., *The Particle Atlas, Edition 2, Volume V*, Ann Arbor Science Publishers, Inc., Ann Arbor, MI, 1979.

14. McCrone W. C., Andreen J. H., and Tsang S. M. “Identification of Organic High Explosives,” *The Microscope*, Vol 41, 1993, pp 161-182.

15. McCrone W. C., Andreen J. H., and Tsang S. M. “Identification of Organic High Explosives, II,” *The Microscope*, Vol 42, 1994, pp 61-73.

16. McCrone W. C., Andreen J. H., and Tsang S. M. “Identification of Organic High Explosives III,” *The Microscope*, Vol 42, 1999, pp 183-200.

17. McCrone, W.C., “Solubility, Recrystallization and Microchemical Tests on Nanogram Single Particles,” *The Microscope*, V34 N2, 1986, pp 107-118.

18. Delly J. G., *Essentials of Polarized Light Microscopy and Ancillary Techniques*, Hooke College of Applied Sciences, Westmont, IL, 2017.

19. Wills WF, Jr., “Squaric Acid Revisited,” *The Microscope*, Vol 38, 1990.

20. Hollifield, J.M., “Characterization of Squaric Acid Precipitates,” *The Microscope*, V51 N2, 2003, pp 81-103.

21. Benedetti-Pichler, A.A., “Identification of Materials via Physical Properties, Chemical Tests, and Microscopy”, Academic Press Inc., New York, NY, 1964.

22. Winchell A. N., *The Optical Properties of Organic Compounds*, McCrone Research Institute, Chicago, IL, 1987 (reprint of 1942 edition, Academic Press, Inc)

23. New York Microscopical Society, Glossary of Microscopical Terms and Definitions, Second Edition, McCrone Research Institute, Chicago, IL, 1989.