OSAC 2025-S-0019
Standard Practice for Characterization of Solid Oxidizer and Fuel Explosive(s)

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

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Draft OSAC Proposed Standard

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Ignitable Liquids, Explosives, and Gunshot Residue
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Standard Practice for
Characterization of Solid Oxidizer and Fuel Explosive(s)

1. Scope

1.1 This practice describes procedures for the analysis and identification of explosive mixtures containing fuels and solid oxidizers in powder specimens or residues, when visible material is present.

1.2 This practice describes the analysis of improvised fuel and oxidizer mixtures and commercial products such as black powder, black powder substitutes, pyrotechnic formulations, and ammonium nitrate mixtures.

1.3 *This standard involves handling of energetic materials. Training in the storage and safe handling of energetic materials and familiarity with the properties and hazards of explosives is required.*

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 (refer to the T/SWGFEX Suggested Guide for Explosives Analysis Training).*

1.5 *This standard does not purport to address all of the possible 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 requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:

E620 Practice for Reporting Opinions of Scientific or Technical Experts

E1386 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

E1388 Practice for Sampling of Headspace Vapors from Fire Debris Samples

E1412 Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration with Activated Charcoal

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

E1618 Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

E1732 Terminology Relating to Forensic Science

E2881 Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry

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

E3196 Terminology Relating to 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 Residue

WK73484 Practice for Reporting Results and Opinions of Explosives Analysis

WK86785 Standard Guide for the Forensic Analysis of Explosives by Polarized Light Microscopy

3. Terminology

3.1 *Definitions:*

For definitions of terms that can assist in interpreting this Practice, refer to Terminologies E3196 and E1732.

4. Summary of Practice

4.1 This practice establishes the analytical approach to characterize solid oxidizer and fuel explosives, to include black powder, black powder substitutes, pyrotechnic compositions, and improvised fuel and oxidizer mixtures.

4.2 Multiple techniques and methods are used to identify these mixtures including visual examinations, ignition susceptibility testing, spectroscopic techniques, chromatographic techniques, and spectrometric techniques.

5. Significance and Use

5.1 The minimum requirements for identifying intact materials are physical characteristics and the identification of an oxidizer and fuel. Refer to Practice E3253 for an overarching examination scheme for intact explosives.

NOTE – Some improvised mixtures can vary in visual and physical characteristics.

5.2 Morphology of some commercial products can be a distinct characteristic used for classification and identification purposes.

5.3 Analysis and identification of ignitable liquids used as the fuel for these mixtures is covered in Practices E1386, E1388, and E1412, and Test Methods E1618 and E2881. Use of these standards could require additional training.

5.4 Identification of solid oxidizer or fuel residues in the absence of visible material is beyond the scope of this practice.

5.5 Identification of commercial blasting agents (except ammonium nitrate and fuel oil) and mixtures containing hydrogen peroxide (or other liquid oxidizers) are beyond the scope of this practice.

5.6 Identification criteria for individual compounds by instrumental methods are beyond the scope of this practice.

5.7 The requirements to associate an unknown material to a unique product by brand name or to compare two or more questioned mixtures are beyond the scope of this practice, except as noted.

6. Apparatus and Supplies

6.1 Magnifying lamp.

6.2 Stereo light microscope.

6.3 Polarized light microscope (PLM) —Specifics for this apparatus can be found in WK86785.

6.4 Fourier transform infrared spectrometer (FTIR)—An FTIR capable of acquiring spectra in the mid-infrared region.

6.4.1 FTIR Supplies—Salt plates, mortar and pestle, pellet press.

6.5 Raman spectrometer.

6.6 X-ray powder diffractometer (XRD).

6.6.1 XRD Supplies—Mortar and pestle, XRD specimen holders.

6.7 Scanning electron microscope with energy dispersive X-ray spectroscopy (SEM-EDS).

6.7.1 SEM-EDS supplies—Specimen mounts, specimen holders, SEM stubs.

6.8 X-ray fluorescence analyzer (XRF).

6.8.1 XRF supplies—Specimen mounts, specimen holders, conductive adhesive tabs.

6.9 Gas chromatograph-mass spectrometer—A gas chromatograph (GC) coupled to either a mass spectrometer (MS) or tandem MS.

6.10 GC with flame ionization detector (FID), thermal energy analyzer (TEA), or electron capture detector (ECD).

6.11 Liquid chromatograph (LC)-MS—An LC coupled to either an MS or tandem MS.

6.12 LC with an ultraviolet (UV) or photodiode array (PDA) detector.

6.13 Ion chromatograph (IC)—An IC coupled with a conductivity detector or an MS or a combination thereof.

6.14 Capillary electrophoresis (CE) – A CE coupled with a UV-visible detector, a conductivity detector, or an MS.

6.15 Glassware and Other Supplies—Disposable test tubes, pipettes, beakers, autosampler vials, weigh boats, weigh paper, watch glasses, filtration apparatus, sieves, centrifuge.

7. Reagents and Materials

7.1 Purity of Solvents and Reagents—Reagent grade or better chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades can 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.

7.2 Reference Materials—Reference materials, including single compounds and explosives, can be obtained from commercial and retail sources or directly from the distributor or manufacturer.

7.3 Chromatography Carrier Gas— Helium or hydrogen of purity 99.995 % or higher.

7.4 Deionized Water—18.2 megohms or better (ultrapure).

8. General Solid Oxidizer and Fuel Mixture Information [1-5]

8.1 Solid oxidizers include nitrate salts (e.g., potassium nitrate, ammonium nitrate), chlorate and perchlorate salts (e.g., potassium chlorate, potassium perchlorate), hypochlorite salts (e.g., calcium hypochlorite), and potassium permanganate.

NOTE – Halogenated oxidizers include polytetrafluoroethylene, perfluoropolyether, and hexachloroethane.

8.2 Solid fuels include organic fuels (e.g., sugar, sawdust, terephthalic acid), metals (e.g., aluminum, magnesium, magnalium), and other inorganic fuels (e.g., sulfur, antimony trisulfide).

8.2.1 Solid fuel and oxidizer mixtures include improvised mixtures and commercial products such as black powder, black powder substitutes, and pyrotechnic mixtures.

8.3 Semi-solid fuels include petroleum jelly and waxes.

8.3.1 Semi-solid fuel and solid oxidizer mixtures include those based on potassium chlorate or perchlorate (e.g., with petroleum jelly or a wax material).

8.4 Liquid fuels include petroleum-based fuels, nitromethane, and vegetable oil.

8.4.1 Liquid fuel and solid oxidizer mixtures include those based on ammonium nitrate (e.g., ammonium nitrate and fuel oil, ammonium nitrate and nitromethane), and potassium chlorate (e.g., potassium chlorate and vegetable oil).

9. Sampling Handling

9.1 The applicable procedures for handling and documentation of all submitted specimens are described in Practices E1492, E1618, E3253, E3255, and E3329.

WARNING – Explosive materials can be initiated by external stimuli such as heat, friction, and electrostatic discharge.[1-5]

9.2 Remove a portion of the specimen for analysis. Follow safety regulations for storing the remaining bulk powders.

10. Visual Examinations and Ignition Susceptibility Tests

10.1 Using a stereo light microscope, observe and record physical characteristics of the specimen, including morphology and color. If available, photograph the material using a digital camera microscope attachment.

10.1.1 Commercial black powder is commonly a graphite-glazed grain with a smooth appearance. Some commercial sources of black powder do not appear granular (e.g., fuses, model rocket engines).

10.1.2 Pyrodex® and Triple Seven® powders (trademarked by Hodgdon Powder Co., Inc., in Shawnee, KS) are visually indistinguishable. Both are grey textured heterogeneous grains containing white crystalline material. These products are also manufactured as pellets.

10.1.3 Ascorbic acid-based black powder substitutes are commonly brown textured homogenous grains. Some products can be manufactured as pellets (e.g., White Hots®).

10.1.4 Comparison of size distributions to reference materials can be performed if sufficient material is present.

10.1.5 Pyrotechnic mixtures can be found in a wide variety of colors and morphologies. Metal-containing mixtures, such as flash powders, are commonly grey. Pyrotechnic mixtures can also contain plant-based materials (e.g., wood meal, grain hulls).[4-5]

10.1.5.1 Pyrotechnic stars can contain complex layered mixtures in spherical and cylindrical shapes.[4-5]

10.1.6 Other commercial and improvised mixtures can have varied appearances and different levels of homogeneity.

10.1.6.1 Commonly encountered oxidizers are white crystalline materials.

10.1.6.2 When mixed with fuels, the appearance of the mixture can change. For example, a mixture of potassium perchlorate and sugar is white, while a mixture of potassium perchlorate and aluminum is grey.

10.2 If sufficient material is present, an ignition susceptibility test (IST) can aid in determining the sensitivity and energetic properties of the mixture.

10.2.1 Remove a small amount (e.g., matchhead-size) of the mixture for testing. Introduce the specimen to a flame and record observations (e.g., smoke, color, sound, odor, residues).

10.2.2 If no reaction occurs, subject the specimen to prolonged contact with the flame and record observations.

10.2.3 Some energetic mixtures will not burn unconfined in an open flame. Mixtures such as these can be lightly confined inside a piece of tissue-like paper and then introduced to a flame.

10.2.4 Some mixtures will not ignite when subjected to a flame, even if confined.

10.3 The results of the IST can be used to guide specimen preparation and analysis.

11. General Specimen Preparation [5-7]

11.1 Separate visually distinct particles, grains, or stars in a specimen unless the mixture is agglomerated, too finely combined, or otherwise inseparable.

11.2 If the item is suspected to contain an ignitable liquid, perform an ignitable liquid extraction and analysis on a portion of the material prior to continuing with the analysis for oxidizers and other fuels. Refer to Practices E1386, E1388, E1412, and Test Method E1618. If the item is suspected of containing vegetable oil, refer to Test Method E2881.

WARNING – Exceeding auto-ignition temperatures of suspected materials during headspace extraction could result in a fire.

11.3 If other non-solid hydrocarbon-based fuels are suspected, perform a solvent extraction using a suitable non-polar solvent (e.g., pentane or hexane).

11.4 Utilize negative controls when performing extractions or concentration of extractions by using equivalent solvent volumes and concentration means.

11.5 Chemical and instrumental analyses are performed in any order after specimen preparation.

11.5.1 Demonstrate instrument performance for analysis prior to the analysis of specimens. The criteria for instrument performance will vary but can include expected resolution, expected retention/elution times, and expected mass spectra.

NOTE – The size of the sample and the use of destructive techniques can affect the order of analyses.

11.6 Not all listed analyses are required for identification. Refer to Practice E3253 for minimum requirements.

12. Analysis by Chemical Spot Tests

12.1 Chemical spot tests are presumptive and used in conjunction with other techniques.

NOTE - There are a wide variety of chemical spot tests (or test strips) available for oxidizers and fuels. Refer to spot test references[8-9] for further information.

12.2 Prepare and concurrently analyze positive and negative controls in the same manner as the specimen for any spot test.

12.3 Record (to include photographs) resulting visual data from the spot test.

13. Analysis by PLM

13.1 Determine optical crystallographic properties to identify oxidizers and solid fuels. Refer to WK86785.

14. Analysis by SEM-EDS or XRF [10-11]

14.1 Conduct elemental analysis using either SEM-EDS or XRF.

14.1.1 Specimens can be ground prior to analysis or analyzed as received, depending on the type and amount of material submitted.

WARNING - Vigorous grinding can result in the initiation of some mixtures.

14.1.2 Extraction and separation techniques can be used to isolate components of a mixture.

14.2 Prepare specimens for SEM-EDS or XRF analysis using supplies recommended by the instrument manufacturer that do not interfere with anticipated elements.

14.2.1 Acceptable mounting mediums for SEM-EDS include adhesive conductive tapes, metal stubs, and carbon planchets.

14.2.2 Acceptable mounting mediums for XRF include adhesive conductive tapes, carbon planchets, and Mylar® support mounts.

14.3 Evaluate the resulting spectra to determine that the peak resolution and signal-to-noise ratios are sufficient for elemental identification.

14.4 Identify elements by their characteristic X-ray energies.

14.5 Record any relevant morphological features of specimen (e.g., cellular structures for wood/charcoal, flake v. spherical metal fuels).

15. Analysis by XRD [12-13]

15.1 Prepare specimens for XRD analysis by carefully grinding a portion using a mortar and pestle. Some fine powders do not require grinding.

WARNING - Vigorous grinding can result in the initiation of some mixtures.

15.2 Evaluate the resulting pattern to determine that the peak resolution and signal-to-noise ratios are sufficient for identification.

15.2.1 Complex mixtures can result in peak overlap. Extraction and separation techniques can simplify diffraction patterns and aid in identification.

15.2.2 Extract sulfur with toluene, or other solvents of similar polarity, and dry before further analysis.

15.2.3 To isolate water insoluble materials, rinse the specimen with distilled or deionized water and dry the remaining material before further analysis. Warm or hot water can facilitate the separation of the water soluble material from the water insoluble material.

15.3 Compare specimen to patterns of known materials or reference library patterns for the purpose of identification.

16. Analysis by FTIR or Raman Spectroscopy [10, 14-19]

16.1 Analyze specimens spectroscopically by FTIR or Raman.

WARNING - When using Raman spectroscopy, heat is generated within a specimen, notably with dark-colored materials or particles, and can cause specimen initiation.

16.1.1 Mixtures can result in peak overlap. Extraction (e.g., using water to remove inorganic oxidizers) and separation techniques (e.g., sieves) can simplify spectra and aid in identification.

16.2 Spectral information can be limited by instrumental conditions (e.g., scan range based on IR window material and ATR crystal) or chemical composition (e.g., distinguishing some nitrate salts).

16.3 Evaluate the resulting spectra to determine that the peak resolution and signal-to-noise ratios are sufficient for identification.

16.4 Compare specimen to spectra of known materials or reference library spectra for the purpose of identification.

17. Analysis by Chromatography Techniques

17.1 Prepare and concurrently analyze negative controls in the same manner as the specimen for any analysis utilizing chromatography with an extraction.

17.2 Prepare and concurrently analyze reference materials for any analysis utilizing chromatography.

17.3 Evaluate resulting chromatograms to determine if retention/elution times, peak resolution, and signal-to-noise ratios are sufficient for identification.

17.4 Compare specimen to chromatograms/mass spectra of reference material or reference library spectra.

17.5 Analysis by GC or GC-MS [16]

17.5.1 If indicated visually or by other analyses, extract sulfur with toluene or other solvents of similar polarity and filter or centrifuge before further analysis.[20]

17.5.2 Extract additives such as sodium benzoate, dicyandiamide, and ascorbic acid with methanol or ethanol and filter or centrifuge before further analysis.[21] Some organic fuels (e.g., sugars) require derivatization prior to analysis by GC.[22]

17.5.3 Use a high temperature GC for extracts that could contain semi-solid fuels or liquids with a high boiling point.

17.6 Analysis by LC or LC-MS [16, 23-24]

17.6.1 Extract additives such as sodium benzoate, dicyandiamide, ascorbic acid, and some organic fuels (e.g., sugars) with deionized water and filter or centrifuge before further analysis.

NOTE: Some ions will degrade in water. For example, ascorbic acid will degrade into threonic acid, monohydrated diketogulonic acid, and oxalic acid.

17.6.2 Do not analyze semi-solid fuels and ignitable liquids by LC.

17.7 Analysis by IC or IC-MS or CE [25-28]

17.7.1 Extract oxidizer salts, additives such as sodium benzoate, ascorbic acid, and some organic fuels (e.g., sugars) with deionized water and filter or centrifuge before further analysis. Analyze the resulting extract for cations and anions.

NOTE: Some ions will degrade in water. For example, ascorbic acid will degrade into threonic acid, monohydrated diketogulonic acid, and oxalic acid.

17.7.1.1 Benzoate ions are detected in products such as Pyrodex® and Triple Seven®.

17.7.2 Do not analyze semi-solid fuels and ignitable liquids by IC or CE.

18. Identification Criteria

18.1 Refer to Practice E3253 for the identification requirements.

19. Documentation

19.1 Retain all notes and supporting analytical data used for an identification in accordance with Practices E620 and E3255. Examples of such data include chromatograms/spectra, photographs/photocopies of results, and detailed descriptions.

19.2 Record case notes in sufficient detail such that an independent analyst could understand and evaluate all the work performed, interpret the data, and form an opinion.

19.3 Report results in accordance with Practice E620 and WK73484.

20. Keywords

Explosives; fuel and oxidizer mixtures

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