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Standard Practice for Characterization and Classification of Smokeless Powder [1]

This standard is issued under the fixed designation E2998; 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 practice describes procedures for characterization and analysis of smokeless powders recovered from explosives incidents (**1**, **2**),[2] materials or objects containing gunshot residue (**3**) when visible grains are present, or bulk samples of powder.

1.2 Smokeless powder is characterized by shape, color, texture, manufacturing toolmarks, markers, dimensional measurements, and chemical composition (**4-6**).

1.3 Smokeless powder is an energetic material classified as a low explosive or propellant. Smokeless powder can be further classified as single-base, double-base, or triple-base.

1.4 Analysis of post-blast debris and items containing gunshot residue when visible grains of smokeless powder are not present is beyond the scope of this practice.

1.5 This practice will provide guidelines for the analysis of organic components of smokeless powders using various instrumental techniques, such as gas chromatography-mass spectrometry, liquid chromatography, and Fourier transform infrared spectroscopy.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *This standard involves handling of low explosives and potentially other energetic materials. It is strongly suggested that an analyst be trained in the storage and safe handling of energetic materials and be familiar with the properties and hazards of explosives.*

1.8 *This standard cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E620 Practice for Reporting Opinions of Scientific or Technical Experts

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ball powders, n*—a class of smokeless powders produced by a process where the final grain morphologies are spherical, flattened-ball, or flake.

3.1.2 *double-base, n*—propellant containing nitrocellulose and nitroglycerin.

3.1.3 *deterrent, n*—a compound to slow the burning rate of a powder.

3.1.4 *energetic, n*—an explosive compound used to enhance the burning rate of a powder.

3.1.5 *extruded powders, n*—a class of smokeless powders produced by an extrusion process where the final grain morphologies are disc or cylinder.

3.1.6 *grain, n*—an individual particle of smokeless powder.

3.1.7 *marker, n*—a colored grain of smokeless powder to assist in the visual identification of a bulk reloading smokeless powder.

3.1.8 *perforation, n*—a hole in a disc powder or one or more holes running through the length of a cylinder powder created during the manufacturing process in extruded powders.

3.1.9 *single-base, n*—propellant containing nitrocellulose as the major energetic material.

[1] This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

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

[3] 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.

3.1.10 *smokeless powder, n*—a propellant and low explosive composed of nitrocellulose and other organic and inorganic compounds.

3.1.11 *stabilizer, n*—a compound to prevent or slow down self-decomposition.

3.1.12 *triple-base, n*—propellant containing nitrocellulose, nitroglycerin, and nitroguanidine.

4. Summary of Practice

4.1 The physical properties of smokeless powder grains are recorded by visual examination using a stereo light microscope, micrometer, manual measuring device, digital measurement and recording device, or camera.

4.2 The significant physical properties of smokeless powders to measure are diameter, length, and thickness. The significant physical properties to record are shape, color, perforations, texture, striations (manufacturing toolmarks), and markers.

4.3 Techniques are described for the extraction of organic components of smokeless powders for instrumental analysis.

4.4 The chemical properties and composition of smokeless powders can be determined by a combination of techniques which may include burn testing, gas chromatography, liquid chromatography, capillary electrophoresis, mass spectrometry, and Fourier transform infrared spectroscopy (**7-12**).

5. Significance and Use

5.1 This practice establishes guidelines for the characterization of smokeless powder which can be used as an explosive for improvised explosive devices or as a propellant, such as for small arms ammunition and for military ordnance.

5.2 This practice establishes the minimum criteria necessary to classify smokeless powders.

5.3 The morphology of smokeless powder is a distinct characteristic used for classification and identification purposes.

5.4 The identification of a questioned sample as smokeless powder (that is, it is a propellant or low explosive) does not require the identification of chemical components of a smokeless powder other than nitrocellulose.

5.5 Identification of organic compounds associated with smokeless powders is a requirement to classify a smokeless powder sample as single-base, double-base, or triple-base.

5.6 Additional analytical techniques may be available that are not mentioned within this document that are acceptable for the characterization and analysis of smokeless powders.

5.7 The requirements to associate a questioned smokeless powder to a unique smokeless powder product by brand name or intercomparison of two or more questioned powders are beyond the scope of this document (**13-15**).

5.8 The identification of smokeless powder residue in the absence of whole or partial grains is beyond the scope of this document.

6. Apparatus

6.1 *Stereo light microscope* with an appropriate light source.

6.2 *Magnifying lamp* with at least 3 diopter magnification.

6.3 *Gas chromatograph-mass spectrometer*—A gas chromatograph (GC) capable of using capillary columns and being interfaced to a mass spectrometer (MS) operating in electron ionization (EI) mode.

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

6.4.1 *Micro-FTIR.*

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

6.6 *Capillary electrophoresis (CE) system.*

6.7 *Liquid chromatograph (LC).*

6.8 *LC-MS.*

6.9 *Digital imaging system and computer.*

6.10 *Digital camera* that can attach to or be used in conjunction with a stereo light microscope.

7. Chemicals, Reagents, and Reference Materials

7.1 *Purity of 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.[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.

7.2 *Solvents*—Methylene chloride and acetone (ACS reagent grade or better) or other appropriate solvents of equal quality are acceptable.

7.3 *Test Mixture*—The test mixture should consist of nitroglycerin, diphenylamine, ethyl centralite, and 2,6-dinitrotoluene. The final test solution is prepared by diluting the above mixture such that the concentration of each component is no greater than 0.005 % weight/volume (0.05 mg/mL) in the chosen solvent (see 7.2). Additional compounds commonly found in smokeless powders may also be included in the test mixture, such as methyl centralite, 2,4-dinitrotoluene, 2-nitrodiphenylamine, 4-nitrodiphenylamine, diethylphthalate, and dibutylphthalate.

7.3.1 Appropriate concentrations of individual reference materials or standards of these compounds may be used in addition to or instead of a test mixture.

NOTE 1—In addition to component identification, appropriate concentrations of the test mixture (or standards) can be used to evaluate overall instrument performance or sensitivity (for example, column resolution, instrument detection limits).

[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

7.4 *Internal Standard*—An appropriate internal standard (for example, undecane, decane) may be used in the extraction solvent for GC and LC analyses.

7.5 *Reference Smokeless Powders*—Reference smokeless powders can be obtained as bulk reloading powders from commercial and retail sources or directly from the distributor or manufacturer.

7.5.1 Reference smokeless powders may be analyzed as positive controls or comparison samples following the same procedure for questioned samples.

7.6 *GC Carrier Gas*—Helium or hydrogen of purity 99.995 % or higher.

7.7 *Deionized Water*—18 megohms or better.

7.8 *Polystyrene Film Standard.*

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

7.10 *Glassware and Other Supplies*—Disposable test tubes, pipettes, beakers, autosampler vials, weigh boats, weigh paper, watch glasses.

8. Sample Handling

8.1 Observe the appropriate procedures for handling and documentation of all submitted samples as described in Practice E1492.

8.2 Open and examine the item in order to determine that it is consistent with its description.

8.3 If the item is suspected of containing residues of an ignitable liquid, perform an ignitable liquid extraction and analysis on the item (or sample of bulk powder) prior to continuing with the analysis for smokeless powder. Refer to Test Method E1618.

8.3.1 **Warning—**Headspace extraction techniques for ignitable liquids should be performed at temperatures below 40°C on specimens.

8.4 On a clean surface, conduct a visual examination of the item.

8.4.1 An examination lamp with an optical magnifier or a stereo light microscope can be used to enhance the detection of small-grain powders.

8.5 Photograph observed grains in situ on debris samples (if possible and probative).

8.6 Physically remove a representative sample of suspected smokeless powder from debris samples and transfer to a suitable sample holder.

8.7 For bulk powders, a representative sample should be separated from bulk powders for analysis as a safety consideration. Store the remaining bulk powders per laboratory policy and local regulations.

8.8 Separate smokeless powder grains from any other materials in the sample, using a stereo light microscope if necessary.

8.8.1 Other energetic materials, such as black powder and flash powder, are sometimes combined with smokeless powder in explosives casework.

8.9 When two or more smokeless powders are obviously present, separate them into similar morphological groups and examine separately if necessary.

9. Analysis Plan for the Characterization of Smokeless Powders

9.1 Characterization of a smokeless powder involves identifying the unique physical characteristics of the powder along with chemical analysis (**4-6**) to identify the nitrocellulose (which is common to all smokeless powders) and other organic components present in propellants.

9.2 *Analysis Plan Summary:*

9.2.1 Visual examination and recording of physical characteristics

9.2.2 Extractions and analysis of organic components

9.2.3 Burn test (if sufficient sample is available)

9.3 *Visual Examination and Recording of Physical Characteristics:*

9.3.1 Use a stereo light microscope if necessary to observe and record the shape, color, markers, perforations, toolmarks, irregular shapes, and any unique physical characteristics.

9.3.1.1 If feasible, capture a scaled image of the powder for comparison to similar known powders.

9.3.2 Record the shape (morphology) of the powder grain:

9.3.2.1 *Disc*—a flat circular grain (coins), either solid or containing a perforation, of varying thickness typically under 0.35 mm.

9.3.2.2 *Cylinder (or rod)*—a short rod-like grain either solid or containing one or more perforations.

NOTE 2—Most cylinder powders used in small arms ammunition have a single perforation which is often difficult to observe because of the graphite coating on the grains or from effects caused by the mechanical cutting of the grain. Large cylindrical powders having multiple perforations are characteristic of military propellants.

9.3.2.3 *Sphere (or ball)*—a round grain with no flat surfaces.

9.3.2.4 *Flattened-ball*—a spherical grain that is flattened top and bottom (some extremely flattened) and may exhibit radial stress fractures.

9.3.2.5 *Flake*—a flat irregularly shaped grain usually with a rough non-uniform surface.

9.3.2.6 *Agglomerate*—multiple small spherical grains adhering together (such as grapes on a vine).

9.3.2.7 *Lamel*—a thin square or parallelogram grain (made from a sheet manufacturing process).

9.3.2.8 *Irregular*—a highly modified grain lacking any particular shape with no consistently measurable dimension such as length or diameter.

9.3.3 Coarse dimensional measurements of the diameter, length, or width of powder grains in the specimen can be determined through a side by side comparison to reference smokeless powders of similar morphology or by use of measurement tools.

9.3.3.1 Precise measurement of diameter, length, or thickness is not required for class identification of smokeless powders.

9.3.4 A specimen containing powder of one or more different types of morphology may be further differentiated and

treated as separate specimens based on differing physical characteristics noted in the microscopical analysis.

9.3.4.1 Further differentiation is not required for class identification of smokeless powder but may be useful for future intercomparison between two or more powders or associating a questioned powder with a manufacturer or by brand name.

9.4 *Extraction and Analysis of Organic Compounds:*

9.4.1 A solvent extraction is followed by an instrumental technique or combination of techniques that is capable of identifying nitroglycerin and other organic compounds present in smokeless powders.

9.4.1.1 Two different solvent extraction schemes using methylene chloride or acetone are described in this section for the initial extraction or solvation of smokeless powders. An analyst may choose to use either or both extraction schemes as necessary depending upon the desired results.

9.4.1.2 Other solvents, such as methanol or chloroform, are suitable for the extraction of smokeless powders but are not covered by this practice. The solubility of nitrocellulose versus the other organic additives in a solvent is an important factor in choosing a suitable extraction solvent.

9.4.1.3 GC-MS and LC-MS are acceptable techniques to identify nitroglycerin and other organic compounds **(4, 7-9)**.

9.4.1.4 FTIR is an acceptable technique to identify nitroglycerin and other organic compounds unless significant amounts of co-extractable compounds are present in which case an additional orthogonal technique (such as those listed in 9.4.1.5) may be necessary.

9.4.1.5 A combination of two or more orthogonal techniques, such as GC-ECD, GC-FID, GC-TEA, CE, and LC analysis, are acceptable to identify nitroglycerin and other organic compounds **(11, 12)**.

9.4.1.6 **Warning—**Methylene chloride may not be an approved or optimal solvent for some instrumental techniques. If indicated, follow the steps in 9.4.3 using an appropriate solvent for analysis.

9.4.1.7 The nitroglycerin in double- and triple-base powders should be removed through solvent extraction prior to analysis of the nitrocellulose by FTIR. The alternate solvent extraction scheme (see 9.4.3) may be used for this purpose.

9.4.2 *Extraction and Analysis Scheme Using Methylene Chloride:*

9.4.2.1 Put approximately 10 mg (5–50 grains depending upon the grain size) of powder in a test tube and extract with approximately 500 µL methylene chloride for up to 30 minutes with occasional agitation or vortex mixing. Typical extraction times will vary from 5 to 30 minutes depending upon grain morphology, size, and surface coatings. Extending extraction times beyond 30 minutes is acceptable and has no undesirable affects on a powder sample.

NOTE 3—Any suitable item of glassware, such as an extraction vial, watch glass, or small beaker may be used in lieu of a test tube throughout this practice. Plastic labware is not recommended as it may contain phthalates similar to plasticizers used in smokeless powders that may dissolve into the extraction solvent.

9.4.2.2 Smaller specimen sizes are permitted and solvent volumes should be reduced accordingly.

9.4.2.3 After extracting the specimen, transfer an aliquot of the extract into an appropriate vial for analysis. An analysis sequence should include solvent blanks, reference materials, controls, and standards as necessary.

NOTE 4—Specimens may be diluted as necessary depending on the sensitivity of the instrumental technique(s) used for analysis.

9.4.2.4 For specimens containing no nitroglycerin, analyze the extracted grains by FTIR to detect nitrocellulose.

9.4.2.5 For samples containing nitroglycerin, the alternate extraction and analysis scheme (see 9.4.3) should be used to remove nitroglycerin from the powder prior to analysis for nitrocellulose as spectral similarities are noted between the two components.

9.4.3 *Alternate Solvent Extraction and Analysis Scheme for Organic Compounds:*

9.4.3.1 Acetone is used in this extraction and analysis scheme for the purpose of dissolving powder grains to increase the efficiency of extraction of organic compounds from the nitrocellulose. This extraction scheme may also be used to extract nitroglycerin from double- and triple-base powders to enable FTIR analysis of the nitrocellulose. If the powder specimen was previously extracted using another solvent, remove any remaining solvent prior to beginning this extraction scheme.

9.4.3.2 Add approximately 500 µL of acetone to a test tube containing up to 10 mg of powder. Allow the acetone to completely dissolve the grains which will take approximately 60 minutes with occasional agitation or vortex mixing. Once dissolved, allow the acetone to evaporate to dryness. A stream of purified air or an inert gas can be used to accelerate evaporation without heat.

NOTE 5—Some of the organic components have low boiling points and could be lost if heat is applied during evaporation.

9.4.3.3 Extract the dried residue with up to 500 µL of methylene chloride for up to 15 minutes with occasional agitation. Transfer the methylene chloride extract to a second test tube. Repeat these steps two additional times, transferring the methylene chloride to the second test tube after each extraction. After the last extraction, allow any residual methylene chloride in the first test tube to evaporate to dryness.

9.4.3.4 Aliquots of the methylene chloride from the second test tube can be analyzed by the desired techniques. Dilute or concentrate aliquots as necessary.

NOTE 6—This step is not necessary if the sample was previously extracted and analyzed following 9.4.2.

9.4.3.5 For an instrumental method incompatible with methylene chloride, transfer an aliquot (about 300 µL) of the methylene chloride extract into another test tube and evaporate to dryness. A stream of purified air or an inert gas can be used to accelerate evaporation (without heat). Reconstitute using an appropriate solvent and analyze by the desired technique.

9.4.3.6 Analyze the specimen extracts by the appropriate combination of techniques along with solvent blanks, reference materials, controls, and standards as necessary.

9.4.3.7 Remove a portion of the dried residue remaining in the first test tube (from 9.4.3.2) for FTIR or Micro-FTIR

analysis for nitrocellulose. Acetone can be added to dissolve a portion of the nitrocellulose to transfer to the FTIR stage or a salt plate.

NOTE 7—The nitroglycerin and most other organic compounds are removed from the nitrocellulose in double- and triple-base powders by the methylene chloride rinses in the previous steps.

9.5 *Analysis of Nitroguanidine in Triple-Base Powder:*

9.5.1 Nitroguanidine is a colorless crystalline organic compound present in triple-base smokeless powders.

9.5.1.1 Nitroguanidine is a major component in some triple-base powders and can be analyzed by FTIR typically with little sample preparation.

9.5.1.2 Sample preparation will be necessary for some triple-base powders that contain smaller amounts of nitroguanidine.

9.5.2 Take a single grain of a suspected triple-base powder and slice open lengthwise. Observation under the microscope may reveal a crystalline material embedded lengthwise on the interior of the grain.

NOTE 8—Triple-base smokeless powder grains may be difficult to slice requiring the use of a microtome or other cutting device.

9.5.2.1 Place the flat portion of the grain face down on the FTIR stage to analyze in attenuated total reflectance (ATR) mode, or remove a portion of the crystalline material from the grain and prepare as necessary for FTIR analysis.

9.5.3 If crystalline material is not obviously present, extraction of the nitroguanidine from sample grains may be necessary, such as in some smaller grain triple-base powders.

9.5.3.1 Dissolve several grains (or up to 200 mg of powder) in a test tube with up to 1 mL of acetone with occasional agitation or vortex mixing. This may take as long as 2 hours or more. Once dissolved, allow the acetone to evaporate to dryness. A stream of purified air or an inert gas can be used to accelerate evaporation.

9.5.3.2 Extract the dried residue with up to 2 mL of deionized water for up to 60 minutes with occasional agitation or vortex mixing. The application of heat (not to exceed 100°C) will increase the solubility of nitroguanidine in the deionized water. **(16)**

9.5.3.3 Remove the water to a watch glass (or other suitable glass container) and evaporate to dryness. Heat may be applied but do not exceed 100°C.

9.5.3.4 Take a portion of the dried residue and prepare as necessary to analyze by FTIR (ATR or transmission mode).

9.5.4 If sample size is limited, the residue remaining in the test tube from the extraction in 9.4.3.2 may be used in lieu of performing the extraction in 9.5.3. Note that the initial specimen size used in 9.4.3.2 is much less (10 mg).

9.5.5 Other techniques or combination of techniques, such as LC and MS, are also acceptable and may be used to analyze the extracted residue for nitroguanidine.

9.5.5.1 LC, MS, and other sensitive techniques may be more suitable than FTIR for analysis of nitroguanidine for smaller specimen sizes or specimens that require extraction.

9.6 *Burn Test:*

9.6.1 If sufficient sample is available, a portion of the powder may be burned noting its burning characteristics.

9.6.2 Smokeless powders ignite easily with a flame and burn with a minimal amount of smoke.

10. Smokeless Powder Classification Scheme

10.1 *Criteria for Class Identification of Smokeless Powder:*

10.1.1 The minimum criteria for class identification of a smokeless powder as a propellant or low explosive are identification of nitrocellulose and physical characteristics consistent with smokeless powders.

10.1.2 Physical characteristics to consider would include size and shape of the powder being consistent with known smokeless powders and a positive burn test.

10.1.3 Chemical analysis is used in the identification of nitrocellulose.

10.2 *Criteria for Classifying a Smokeless Powder as Single-Base:*

10.2.1 The minimum criteria to classify a smokeless powder as single-base are identification of nitrocellulose, physical characteristics consistent with single-base smokeless powders, and chemical analysis to detect other organic compounds associated with smokeless powder (see Table 1).

10.2.2 Physical characteristics to consider would include size and shape of the powder being consistent with known single-base powders and a positive burn test.

10.2.3 Chemical analysis (for example, GC-MS or LC-MS) must indicate that nitroglycerin is not present as a component in the smokeless powder. Chemical analysis is used in the identification of nitrocellulose and may be used if necessary to identify other organic compounds related to smokeless powder (see Table 1).

10.2.3.1 Diphenylamine and dinitrotoluene compounds are common components of single-base powders.

10.3 *Criteria for Classifying a Smokeless Powder as Double-Base:*

10.3.1 The minimum criteria to classify a smokeless powder as double-base are identification of nitrocellulose and nitroglycerin, and physical characteristics consistent with double-base smokeless powders.

10.3.2 Physical characteristics to consider would include size and shape of the powder being consistent with known double-base powders and a positive burn test.

TABLE 1 Compounds Commonly Used in Manufacturing Smokeless Powder

Component	Use
Nitroglycerin	Energetic (in double- and triple-base only)
Nitrocellulose	Energetic
Nitroguanidine	Energetic and flash reducer (in triple-base only)
Nitrotoluenes	Energetic
Methyl Centralite	Deterrent
Ethyl Centralite	Deterrent, Stabilizer
Phthalates	Plasticizer
Diphenylamine	Stabilizer
Akardite II	Stabilizer
Nitro- and Nitrosodiphenylamines	Stabilizer
Calcium carbonate	Stabilizer and flash reducer
Potassium salts	Flash reducer
Graphite	Lubricant and static reducer
Camphor	Other
Adipates	Other

10.3.3 Chemical analysis is used in the identification of nitrocellulose and nitroglycerin and may be used if necessary to identify other organic compounds related to smokeless powder (see Table 1).

10.3.3.1 Diphenylamine, ethyl centralite, and dibutylphthalate are other common components of double-base powders.

10.4 *Criteria for Classifying a Smokeless Powder as Triple-Base:*

10.4.1 The minimum criteria to classify a smokeless powder as triple-base are identification of nitrocellulose, nitroglycerin, and nitroguanidine, and physical characteristics consistent with triple-base smokeless powders.

10.4.2 Physical characteristics to consider would include size and shape of the powder being consistent with known triple-base powders and a positive burn test.

10.4.2.1 Triple-base powders are generally manufactured for military use for large caliber weapons with grain sizes much larger than most commercial single- and double-base powders.

10.4.3 Chemical analysis is used in the identification of nitroglycerin, nitrocellulose, and nitroguanidine and may be used if necessary to identify other organic compounds related to smokeless powder (see Table 1).

10.5 *General Comments:*

10.5.1 Partial grains of smokeless powder in the absence of whole grains may not exhibit enough physical characteristics to accurately determine the shape (morphology) of the powder. This does not preclude a class identification of the sample but may require more supportive data (additional chemical analyses) than a sample containing whole grains.

10.5.1.1 Class identification is possible on a partial grain in the absence of a morphological characterization if significant target compounds are identified. For example, the identification of nitrocellulose as well as nitroglycerin, dinitrotoluenes, diphenylamine, or centralites are indicative of a smokeless powder whereas phthalates alone are not.

10.5.1.2 The identification of nitrocellulose is based upon a combination of FTIR analysis, and microscopy of the physical properties of the grain or a positive burn test.

10.5.2 The chemical composition of partially burned smokeless powder grains may differ from the unburned powder.

10.6 *Limitations:*

10.6.1 The analyst should use caution when classifying individual powders as single- or double-base in mixtures of smokeless powders containing both types because of cross contamination of granular particles or volatile components.

10.6.1.1 Powder samples that contain mixtures of single- and double-base smokeless powders would require the analyst to evaluate the chemical composition and physical properties of a powder to properly classify a powder specimen as single- or double-base if desired.

10.6.2 Some compounds used in smokeless powders may be difficult to identify by the analysis schemes suggested in this practice. The techniques may not effectively extract or isolate a compound for analysis or the instrumental analyses used herein are not capable of detecting or identifying some compounds.

10.6.3 Some components in smokeless powders are proprietary materials. The unavailability of a reference material, standard or equivalent material for comparison may hinder the analyst's ability to identify such components.

10.6.4 Isomers of some organic compounds used in smokeless powders, such as phthalates and dinitrotoluenes, have similar spectra or retention times and may be difficult to discriminate on some analytical systems.

10.6.5 The analyst should limit identification of powder components to the set of compounds within test mixtures or available reference standards when recording conclusions in a forensic report.

10.6.6 Class identification of smokeless powders does not require the analyst to identify every component in a questioned powder sample nor identify every peak in the chromatographic profile of an instrumental analysis of a specimen extract.

10.6.7 The analyst must perform appropriate chemical analyses to satisfy the minimum criteria to further classify a smokeless powder sample as single-, double-, or triple-base if required to reach such a conclusion in a forensic report.

10.6.7.1 An analysis plan which involves techniques that do not identify nitroglycerin would not be sufficient for an analyst to reach a conclusion that further classifies a smokeless powder sample as single-, double-, or triple-base.

10.6.7.2 An analysis plan which involves techniques that do not identify both nitroglycerin and nitroguanidine would not be sufficient for an analyst to reach a conclusion that further classifies a smokeless powder sample as triple-base.

10.6.7.3 Nitroguanidine is used in triple-base military propellants which are significantly larger than commercially available single- and double-base smokeless powders. If the grain size of a powder is not indicative of a military propellant, chemical analysis for nitroguanidine is not required.

10.6.8 The criteria for intercomparison of smokeless powders or the association of a powder sample with a manufacturer or by brand name (commercial product) would involve a detailed examination which includes the identification of the chemical components and physical characteristics of powders. The complexity of such an analysis is beyond the scope of this practice.

11. Report Writing

11.1 Refer to Practice E620 for general information on report writing.

11.2 *Reporting Conclusions for Class Identification of Smokeless Powder:*

11.2.1 The following are suggested phrases to be used to report a conclusion that a material was identified as a smokeless powder:

11.2.1.1 "Item A was identified as smokeless powder."

11.2.1.2 "The material removed from Item A was identified as smokeless powder."

11.2.2 Similar wording is used to classify a sample as a single-, double-, or triple-base powder:

11.2.2.1 "Exhibit A was identified as a single-base smokeless powder."

11.2.2.2 “Grains of disc-shaped double-base smokeless powder were identified in the threads of the pipe nipple on Item #7.”

11.2.2.3 “The powder grain was identified as a triple-base smokeless powder.”

11.2.3 Qualifying statements may be added to report findings that further identify the material and its components:

11.2.3.1 “The material removed from Item #9 was identified as a disc-type double-base smokeless powder containing nitroglycerin, diphenylamine, and ethyl centralite.”

11.2.4 A statement summarizing the techniques used in the identification of a smokeless powder should also be included in a report:

11.2.4.1 “The following techniques were utilized in the analysis of Item #1: Gas chromatography-mass spectrometry, Fourier transform infrared spectroscopy, optical microscopy, and a burn test.

11.2.5 Phrases such as “consistent with” or “similar to” shall not be used when reporting conclusions for class identification of smokeless powder.

11.2.5.1 Smokeless powders are not similar to any other class of energetic material by the nature of their unique morphology and chemical composition.

12. Keywords

12.1 double-base; nitrocellulose; nitroglycerin; nitroguanidine; single-base; smokeless powder; triple-base

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