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Designation: E2998 – 16

An American National Standard

# 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.

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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.

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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

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