



Standard Test Method for Analysis of Organic Compounds in Smokeless Powder by Gas Chromatography-Mass Spectrometry and Fourier Transform Infrared Spectroscopy¹

This standard is issued under the fixed designation E2999; 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 test method describes the analysis of organic components in smokeless powders by gas chromatography-mass spectrometry (1-6)² and Fourier transform infrared spectroscopy.

1.2 This test method is suited for analyzing samples comprised of visible grains (whole or partial) of smokeless powder.

1.3 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 test method.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.5 This test method 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.

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

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recom-*

mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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

E2998 Practice for Characterization and Classification of Smokeless Powder

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

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

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

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

4. Summary of Test Method

4.1 An extracted specimen of smokeless powder is analyzed by gas chromatography-mass spectrometry (GC-MS) and Fourier transform infrared (FTIR) spectroscopy to identify organic components.

4.2 Post GC-MS data analysis generates extracted ion profiles (mass chromatograms) characteristic of the organic components in smokeless powders. Specific chemical components (target compounds) can be identified by their mass spectra and retention times.

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

Current edition approved Sept. 15, 2017. Published September 2017. DOI: 10.1520/E2999-17.

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.

4.3 FTIR analysis is used in the identification of nitrocellulose and nitroguanidine. An FTIR generates a spectrum consisting of IR absorption bands that can be searched through a reference spectral library for a library match to nitrocellulose or nitroguanidine or compared to known reference materials.

5. Significance and Use

5.1 This test method establishes a procedure for the analysis and identification of organic components of smokeless powder by GC-MS and FTIR.

5.2 FTIR is used in the analysis of nitrocellulose, which is a major component of all smokeless powders, and nitroguanidine in triple-base powders.

5.3 GC-MS is used in the analysis of other organic compounds in smokeless powders, such as energetics, stabilizers, plasticizers, and deterrents.

5.4 This test method is used concurrently with E2998 for the analysis of smokeless powders when whole or partial grains are present.

5.5 GC-MS and FTIR are two techniques used in the analysis of smokeless powders. Additional techniques are available that are acceptable for use in the analysis and identification of organic components of smokeless powders, such as liquid chromatography-mass spectrometry, capillary electrophoresis, and gas chromatography with flame ionization detection.

6. Apparatus

6.1 *Stereo Light Microscope*, with an appropriate light source.

6.2 *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.

6.2.1 *Sample Inlet System*—A sample inlet system that can be operated in either split or splitless mode with capillary columns; the inlet system may use on-column technology.

6.2.2 *Column*—A capillary, bonded phase, methylsilicone or phenylmethylsilicone column or equivalent. Any column length or temperature program conditions may be used provided that each component of the test mixture (see 7.3) is adequately separated.

6.2.3 *GC Oven*—A column oven capable of reproducible temperature program operation in the range from 40 to 300°C.

6.2.4 *Mass Spectrometer*—Capable of acquiring mass spectra from m/z 40 to m/z 400 with unit resolution or better, with continuous data output.

6.2.4.1 *Sensitivity*—The system must be capable of detecting each component of the test mixture (see 7.3) and providing sufficient ion intensity data to identify each component, either by computer library search or by comparison with reference spectra.

6.2.5 *Fourier Transform Infrared Spectrometer*—An FTIR capable of acquiring spectra in the mid-infrared region (wave-number range of 4000 to 650 cm^{-1}).

7. Chemicals, Reagents, and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals or better 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).

7.3 *GC-MS 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.

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

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.

7.6 *Polystyrene Film Standard*.

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

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

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

8. Equipment Calibration and Maintenance

8.1 *GC-MS Calibration and Maintenance*:

8.1.1 Verify the consistent performance of the chromatographic instrument by using blanks and known concentrations of either a prepared test mixture, reference materials, or standards (see 7.3). Optimize gas flows periodically.

8.1.2 Tune and check calibration of the mass spectrometer.

8.1.2.1 Tune the mass spectrometer using perfluorotributylamine (PFTBA), or another appropriate calibration standard,

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.