# Smokeless Powder Analysis

## 1 Scope

These procedures describe the general process for the analysis of bulk smokeless powders and the identification of their components. These procedures are suitable for bulk samples which are suspected of being a smokeless powder. These procedures apply to caseworking personnel conducting work in explosives chemistry analysis.

## 2 Introduction

Smokeless powders are classified as propellants which are used in ammunition for most firearms. In addition, it can be used as the main charge in pipe bombs and other improvised explosive devices (IEDs).

Powders can be physically distinguished by shape: cylinders, disks, balls (or flattened balls), squares, and irregular. Chemically, they fall into three common categories: single, double and triple-base powders. Single-base powders consist of a nitrated cellulose base known as nitrocellulose (NC), while double-base powders have a certain percentage of nitroglycerin (NG) added to the NC. Triple-base powders are used mainly in military applications Redacted They are similar to double-base powders with nitroguanidine (NQ) added as the third energetic material. These procedures will assist in identifying the various components of smokeless powders.

There are several manufacturers of smokeless powder in the United States used by the various distributors of reloading powders. There are also many foreign companies marketing products to U.S. companies.

Unconsumed smokeless powder grains are easily identified by their unique physical characteristics and chemical composition. If no grains are observed, refer to the Explosive Residue Analysis standard operating procedure (SOP).

## 3 Equipment/Materials/Reagents

Equivalent equipment, materials, and reagents may be substituted as needed.

### 3.1 Equipment

- Fourier transform infrared (FTIR) spectrometer with attenuated total reflectance (ATR) or microscope attachment
- Gas chromatograph with mass spectrometer (GC/MS)

- Liquid chromatograph with mass spectrometer (LC/MS)
- Micrometer
- Microscope (optical or digital) with optional digital camera

### 3.2 Materials

- Autosampler vials and caps
- Kraft paper
- Lighter, torch, or matches
- Various disposable glassware and plasticware

### 3.3 Reagents/Solvents/Reference Materials

- Isopropyl alcohol (70% commercial product)
- Methylene chloride (reagent grade)

## 4 Standards and Controls

All reference materials and reagents will be verified prior to, or in concurrence with, use in casework. Refer to the Verification of Reagents and Solvents SOP, the Verification of Reference Materials SOP, and the Records of Items Used As Known Materials SOP. Refer to the Instrument Parameters and Reagent Preparation SOP for information regarding the components and preparation of all standards and controls referred to in this document.

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### 4.1 Redacted Positive Control

An additional reference standard can be made by extracting a few grains of Redacted with approximately 300 µL methylene chloride. This can serve as a positive control for double-base powders. The standard is made up fresh for each day of use. Validation data for this powder can be retrieved from the appropriate validation file.

## 5 Sampling

Refer to the Sampling Procedures SOP in the Explosives Quality Assurance Manual.

## 6 Procedure

Explosives chemistry personnel will:

Clean work surfaces thoroughly with an isopropyl alcohol solution or other appropriate solvent. Cover the clean work surface with a disposable material such as kraft paper. Refer to the Explosives Contamination Prevention Guidelines for additional details.

Use appropriate personal protective equipment (e.g., safety glasses, laboratory coat, disposable gloves) when examining evidence. This is intended to protect personnel conducting the examination and to prevent contamination of evidence.

Review and understand all safety information contained in Section 10 prior to beginning the following procedures.

For each instrumental technique, refer to the Instrument Parameters and Reagent Preparation SOP for Performance Monitoring Protocol (PMP) information, instrument usage procedures, parameters, and reagent preparation information. Prior to evidence analysis, follow the PMP for the instrument's reliability and reproducibility from analysis to analysis.

**6.1** Debris suspected of being involved in an explosion must be carefully inspected under a microscope for grains. Burned and unburned grains of smokeless powder are often found in pipe threads, on tape material, and in crevices. Suspected grains are removed into a small test tube. Bulk powder samples submitted for analysis should also be inspected under a microscope looking for mixtures or atypical material.

**6.2** Recovered grains or mixtures are then separated into similar groups based upon type, size (diameter and/or length), perforations, and color.

**6.3** Grains are measured using a microscope or micrometer to determine diameter and length or thickness. Also, make note of perforations, color, or other distinguishing physical characteristics of the powder.

**6.4** Extract several grains with approximately 300 µL of methylene chloride, and cap. Allow the solution to stand for at least one hour with occasional vortexing. Lesser quantities of solvent should be used for the analysis of very small quantities or a single grain.

**6.5** Analyze the extract by GC/MS in electron ionization (EI) mode. The Redacted can also be confirmed by LC/MS analysis.

**6.6** A sample set should include a Smokeless Powder Standard, a positive control sample Redacted or nitroglycerin standard, methylene chloride blanks as negative controls, and sample extracts. Several common constituents of smokeless powders include Red acte

**6.7** Analyze the methylene chloride Redacted nitrocellulose.

**6.8** (Optional) If sufficient powder is present, a portion can be subjected to a flame to characterize its burning properties. Smokeless powder will burn rapidly with very little smoke and a brief flash of light.

**6.9** A side-by-side chemical and physical comparison should be done with known reference powders if a brand name determination of an unknown powder is necessary. Smaller quantities of powders should be extracted with the elution time extended to 3-4 hours. This will allow for a more thorough extraction of trace components and more efficient elution and equilibrium of the major components. If available, obtain a microscopic enlargement photo of the unknown powder(s). The FBI Laboratory maintains an extensive database with smokeless powders which can assist in brand name determination. The database includes the physical and chemical properties of smokeless powder.

## 7 Calculations

Not applicable.

## 8 Measurement Uncertainty

Not applicable.

## 9 Limitations

The identification of smokeless powder requires at least one powder grain. This grain must have the morphological characteristics and chemical constituents of smokeless powder. In the absence of visible grains, residues consistent with smokeless powders are identified by the various organic compounds present in propellants.

## 10 Safety

Safety protocols, contained within the FBI Laboratory Safety Manual, will be observed at all times.

Standard precautions will be taken for the handling of all chemicals, reagents, and standards including standard universal precautions for the handling of biological and potentially hazardous materials. Refer to the FBI Laboratory Safety Manual for proper handling and disposal of all chemicals. Personal protective equipment will be used when handling any chemical and when performing any type of analysis.

The handling of some explosive materials is hazardous due to potential ignition by heat, shock, friction, impact, or electrostatic discharge. Personnel should work with small quantities (less than a few grams) and properly store larger quantities in approved containers.

## 11 References

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

FBI Laboratory Safety Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

Explosives Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

Explosives Standard Operating Procedures: Chemistry, Federal Bureau of Investigation, Laboratory Division, latest revisions.

Instrument Operations Manuals for the specific models and accessories used.

*Smokeless Powder Reference Collection*, Federal Bureau of Investigation, Laboratory Division, Explosives Unit.

ASTM Standard E2998, 2016, "Standard Practice for Characterization and Classification of Smokeless Powder," ASTM International, West Conshohocken, PA, 2016, DOI: 10.1520/E2998-16, www.astm.org.

Martz, R. M., Lasswell, L. D. III, Smokeless Powder Identification, Proceedings of the International Symposium On The Analysis and Detection of Explosives, pp. 245-254, 1983.

Wolfe, D., Propellant Profiles, Volume 1, Wolfe Publishing Co., Inc., Prescott, AZ, 1982.

| Rev. # | Issue Date | History |
|---|---|---|
| 3 | 10/04/2018 | Administrative changes for grammar and clarity. Removed testmix components in section 4. Added location-specific PMP references to section 6. Removed autosampler vial for extractions in section 6.4. In section 6.9 changed to longer elution time for smaller quantities. Added SAU IOG reference and modified IOSS reference. |
| 4 | 12/16/2019 | Removed sampling plan from section5. Removed SAU Chief and QA from approval lines. Removed unit references to PMPs. |

**Approval**

Redacted - Signatures on File

Explosives Unit Chief Date: 12/13/2019

**TL Approval**

Explosives Chemistry
Technical Leader Date: 12/13/2019