Nitrocellulose-Based Propellant Analysis

Table of Contents

Nitrocellulose-Based Propellant Analysis

1 INTRODUCTION

Nitrocellulose-based propellants are classified as low explosives. These include smokeless powders used in most firearm ammunition and nitrocellulose (NC) sheets/rods/products used in some munitions and other firearm products.

Smokeless powders can be physically distinguished by shape to include: cylinders, discs, 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 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 and are rarely encountered in forensic work. They are similar to double-base powders with nitroguanidine (NQ) added as the third energetic material.

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.

Other NC-based propellants include Hodgdon Triple 8 and Blackhorn 209. These products include guanidine nitrate as part of their chemical composition.

2 SCOPE

This procedure describes the general process and is suitable for the analysis of suspected uninitiated NC-based propellants and the identification of their components. This procedure applies to caseworking personnel conducting work in explosives chemistry analysis.

3 EQUIPMENT

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

3.1 Equipment

- General laboratory supplies

3.2 Instruments

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

3.3 Chemicals/Reagents

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

4 STANDARDS AND CONTROLS

Refer to the Explosives Quality Assurance and Operations Manual for details regarding verification of reference materials. Testmix components and preparation instructions are recorded in the applicable instrument performance document(s). Refer to the Instrument Parameters and Reagent Preparation procedure for information regarding other positive controls relevant to this procedure (e.g., Smokeless Powder Standards).

4.1 Hodgdon HS-7 (or Similar) Positive Control

Prepare a NG-based reference standard by extracting a few grains of Hodgdon HS-7 (or another known double-base smokeless powder) with approximately 300 µL of methylene chloride. This serves as a positive control for NG and the extraction process. The standard is made up fresh for each day of use. Reference data for the powder chosen is recorded in the reference material file.

5 SAMPLING

Refer to the sampling procedures in the Explosives Quality Assurance and Operations 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 Quality Assurance and Operations Manual for additional details regarding explosives contamination prevention.
- 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.
- For each instrumental technique, refer to the Instrument Parameters and Reagent Preparation procedure for instrument usage procedures, parameters, and reagent preparation information. Prior to evidence analysis, follow the applicable instrument performance document(s) to conduct a performance check.

6.1 Sample Analysis

A. Uninitiated powder samples submitted for analysis should be inspected under a microscope to look for mixtures or atypical material (such as partial grains).
B. Separate grains, sheets, and/or rods based upon type, size (diameter and/or length), perforations, and color.
C. Measure the approximate diameter, length, and/or thickness (as applicable) using a microscope. Also, make note of perforations, color, or other distinguishing physical characteristics of the powder.
D. Extract several grains (or a single sheet or rod) with approximately 300 µL of methylene chloride in a glass vial or test tube. Cap and allow the solution to stand for at least one hour with occasional vortexing.

1. Prepare a negative control in the same way as the sample extracts (to include extraction time). This negative control may also serve as a blank if prepared directly in an auto-sampler vial.

E. Analyze the extract using electron ionization (EI) GC/MS (SP method).
   1. A sample set should include a Smokeless Powder Standard, a positive control sample (such as Hodgdon HS-7 or other known double-base smokeless powder), methylene chloride as the blank, negative controls (if applicable), and sample extracts.

F. Analyze the methylene chloride insoluble grain by FTIR with an ATR accessory to determine the presence of NC, nitroguanidine, and/or guanidine nitrate.

G. (Optional) If sufficient powder is present, subject a portion of the original material to a flame to characterize its burning properties. Smokeless powder will burn rapidly with very little smoke and an orange flame.

H. If requested to compare two or more smokeless powder samples (such as between evidentiary items or for brand determination), a side-by-side chemical and physical comparison should be done in triplicate. 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. When comparing samples, utilize similar sample mass, solvent volume, and extraction time when possible. The FBI Laboratory maintains an extensive database with smokeless powders which may assist in brand determination. The database includes the physical and chemical properties of smokeless powder.

7 DECISION CRITERIA

Refer to the Explosives Chemistry Report Writing Guidelines and the Report Wording Examples for Explosives Chemistry Analysis document (level 4) for additional details regarding reporting of smokeless powders.

7.1 Instrumental Results

Refer to the Instrument Decision Criteria for Explosives Chemistry Analysis procedure for details regarding the acceptance of data generated using the instruments and methods described above.

7.2 Material Identification

The minimum identification requirements for a NC-based propellant are:

- The presence of NC.
- The absence of other energetic components except NG, NQ, guanidine nitrate, trinitrotoluene (TNT), and dinitrotoluenes (DNTs).
- Visual characteristics of the material.

If the material has a distinct grain morphology as stated in the introduction, the material may be further identified as a smokeless powder, unless guanidine nitrate is present. If guanidine nitrate is present, the material will be identified as a NC-based propellant.

If grain morphology is present, and the material is identified as a smokeless powder, the following additional requirements should be considered:

- NC is present as an energetic substance, but without NG and NQ – single-base smokeless powder
- NC and NG are present as energetic substances, but without NQ – double-base smokeless powder
- NC, NG, and NQ are present as energetic substances – triple-base smokeless powder

8 Measurement Uncertainty

Although infrequent, the mass of a crude material may be requested by the contributor. When requested, refer to the Explosives Quality Assurance and Operations Manual for information regarding measurement uncertainty of these results.

9 Limitations

This procedure does not address the analysis and limitations of explosive residue examinations.

When an item is tested, a representative sample is tested (if not the whole item). However, the results of the analysis only pertain to the portion of the item tested.

The identification of uninitiated smokeless powder requires at least one intact or partial grain.

NG may physically transfer between mixed powders; therefore, the examiner should exercise caution when reporting mixed smokeless powders.

10 Safety

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

ASTM E2998-16, Standard Practice for Characterization and Classification of Smokeless Powder, ASTM International, West Conshohocken, PA, 2016 (or latest version).

ASTM E2999-17, Standard Test Method for Analysis of Organic Compounds in Smokeless Powder by Gas Chromatography-Mass Spectrometry and Fourier Transform Infrared Spectroscopy, ASTM International, West Conshohocken, PA, 2017 (or latest version).

ASTM E3196-21, Standard Terminology Relating to the Examination of Explosives, ASTM International, West Conshohocken, PA, 2021 (or latest version).

ASTM E3253-21, Standard Practice for Establishing an Examination Scheme for Intact Explosives, ASTM International, West Conshohocken, PA, 2021 (or latest version).

12 REVISION HISTORY

Revision	Issued	Changes
05	09/30/2022	Updated to new document template. Changed title from Smokeless Powder to NC-Based Propellant. Broadened introduction and procedure to include other NC-based materials. Updated HS-7 positive control to include similar, known DB powders. Added decision criteria section. Updated limitations. Added ASTM references.