Dynamite Analysis

1 Scope

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

2 Introduction

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Other dynamites from foreign manufacturers may also be imported into the United States for sale. Redacted

These procedures will assist in extracting and identifying the various components of this explosive.

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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 electron capture detector (GC/ECD)
- Gas chromatograph with mass spectrometer (GC/MS)
- Ion Chromatograph (IC)
- Liquid chromatograph with mass spectrometer (LC/MS)
- Microscope (optical or digital) with optional digital camera
- Raman spectrometer with macro compartment or microscope attachment
- Scanning electron microscope with energy dispersive X-ray spectrometer (SEM/EDS)
- X-ray diffractometer (XRD)

3.2 Materials

- Autosampler vials and caps
- Disposable plastic syringes
- Kraft paper
- Mortar and pestle
- Spatula
- Syringe filters (0.2 µm nylon)
- Various disposable glassware and plasticware
- XRD sample holders (zero background holder with or without depression)

3.3 Reagents/Solvents/Reference Materials

- Acetone (HPLC grade)
- Air (compressed)
- Anhydrous diethyl ether (reagent grade)
- Deionized water (18.2 MΩ)
- Isopropyl alcohol (70% commercial product)
- Methanol (HPLC grade)
- Nitrogen (high purity)
- Sodium sulfate (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 Standard Operating Procedure (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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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 the individual 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 to conduct a QA/QC check to verify the instrument's reliability and reproducibility from analysis to analysis.

6.1 Examine the material under the microscope and note details of its characteristics such as homogeneity, color, phases, etc. Generally, dynamite samples received by the laboratory appear as an orange or brown gummy/oily conglomerate containing crystalline material and wood-like chips. Redacted

the dynamites appear to be oily and may contain white prills or embedded spheres in them.

6.2 Prepare a supply of diethyl ether drying with sodium sulfate and then filtering into a beaker. Evaporate approximately 6 mL of anhydrous diethyl ether to dryness using heat and/or nitrogen/filtered air (as appropriate) in a test tube. This serves as the negative control. If a residue is present, repeat with fresh anhydrous diethyl ether.

6.3 Extract the organic explosives with anhydrous diethyl ether. Place up to 1 g of material in a test tube and extract with up to 2 mL aliquots of anhydrous diethyl ether (prepared in section 6.2) three times and transfer into a new test tube for evaporation. Evaporate the ether to dryness using heat and/or nitrogen/filtered air as appropriate.

6.4 Extract the residue with up to 5 mL of methanol and place into a new test tube. Retain an equal portion of the methanol as a negative control. Both the negative control and sample may be concentrated using heat and/or nitrogen/filtered air as appropriate. If the sample is too concentrated, further dilution of the extract before analysis may be necessary.

6.5 Analyze the methanol extract and negative control on the GC/ECD, and confirm any organics by GC/MS in electron ionization (EI) or chemical ionization (CI) modes or LC/MS analysis.

6.6 Extract the remaining material in the test tube (from section 6.3) with up to 5 mL of acetone and place into a new test tube. Redacted

6.7 Aged dynamite may exhibit leaching of an oily phase. If an oily phase is seen separating from the bulk material, remove a sample using a spatula and dissolve in methanol for analysis. Follow analysis procedures from section 6.4.

6.8 (Optional) Extract a new portion (up to 1 g) of the original item or a sample of the ether insoluble material with up to 30 mL of deionized water. Retain an equal portion of the water as a negative control. Plasticware containers should be used throughout these procedures to avoid leaching of ions from glassware. Prepare a plastic syringe and a 0.2 µm nylon filter (mounted on a plastic syringe) by flushing with deionized water. Flush portions of the negative control and then the sample through the filter and into their respective autosampler vial for IC analyses to determine Redacted

6.9 Analyze the ether and acetone insoluble material by SEM/EDS to determine the presence of Redacted

6.10 If sufficient material is present, analyze the material (from section 6.9) by Redacted For XRD analysis, grind the residue into a fine powder for analysis. Spread the material onto an XRD sample holder (zero background holder with or without depression) and place in the instrument for analysis.

6.11 (Optional) FTIR and Raman spectroscopy can be utilized for the analysis of other bulk materials which may be present.

7 Calculations

Not applicable.

8 Measurement Uncertainty

Not applicable.

9 Limitations

9.1 The presence of ammonium ions or ammonium nitrate does not constitute confirmation of dynamite as there are many explosive and fertilizer products that contain ammonium nitrate as an ingredient.

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9.4 The identification of uninitiated material may be limited by sample size. Identification of residues from initiated material is possible at detection levels of several micrograms if the unique identifiers as mentioned in section 9.2 are present.

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 (such as 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.

Yinon, J. and Zitrin, S. *Modern Methods and Applications in Analysis of Explosives*, John Wiley & Sons, New York, 1993.

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. Added SAU IOG reference and modified IOSS reference.
4	12/16/2019	Added potassium nitrate to introduction and 6.10. Added language for identifying improvised dynamites in the Limitations section (9.3). Clarified that heat and/or nitrogen/filtered air can be used as appropriate. Removed sample selection from section 5.. Removed SAU Chief and QA from approval lines. Removed unit references to PMPs.

Redacted - Signatures on File

Approval

Explosives Unit Chief Date: 12/13/2019

TL Approval

Explosives Chemistry
Technical Leader Date: 12/13/2019