Dynamite Analysis

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1 INTRODUCTION

As of 1997, dynamite is manufactured by only one company in the U.S., Dyno-Nobel of Carthage, MO. Other dynamites from foreign manufacturers may also be imported into the United States for sale. This procedure will assist in extracting and identifying the various components of this explosive.

The various compositions of dynamite include, but are not limited to, the following materials: ammonium nitrate, potassium nitrate, sodium nitrate, trinitrotoluene (TNT), nitrocellulose, wood fiber, carbonaceous material, sodium chloride, newspaper, nitroglycerin (NG), ethylene glycol dinitrate (EGDN), dinitrotoluene (DNT), calcium carbonate, sulfur, and diatomaceous earth.

2 SCOPE

This procedure describes the general process for the analysis of uninitiated dynamites and the identification of their components. This procedure is suitable for uninitiated samples which are suspected of being a dynamite and 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

- XRD sample holders (zero background holder with or without depression)
- General laboratory supplies

3.2 Instruments

- Fourier transform infrared (FTIR) spectrometer with attenuated total reflectance (ATR)
- 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)

3.3 Chemicals/Reagents

- Acetone (HPLC grade)
- Air (compressed)
- Deionized water (18.2 MΩ)
- Isopropyl alcohol (70% commercial product)

- Methanol (HPLC grade)
- Methylene chloride (HPLC grade)
- Nitrogen (high purity)

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 Instrument Parameters and Reagent Preparation procedure for information regarding other positive controls relevant to this procedure (e.g., TSQ Standard Mix).

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 the individual 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. Examine the material under the microscope and note details of its characteristics such as homogeneity, color, phases, etc. Generally, dynamite samples appear as an orange or brown gummy/oily conglomerate containing crystalline material (or prills) and wood-like fibers or chips.
B. (Optional) If an oily phase is seen separating from the bulk material, remove a portion and follow the Identification of General Unknowns technical procedure, confirming any component of interest that is not identified in the remainder of this procedure.
C. Label three empty 16 x 100 mm test tubes (e.g., test tube 1 through 3) and use a balance to record their individual masses. Next, weigh out approximately 100 mg (if appropriate amount of sample is available) of the sample into test tube 1 and record the initial mass of the sample.
D. Extract possible organic explosives in the sample (test tube 1) with ~2 mL methylene chloride. Vortex and/or masticate with a glass stir rod and centrifuge capped as needed. Prepare a 0.2 µm membrane filter (mounted on a glass syringe) by flushing

with methylene chloride, and filter the extract into test tube 2. Repeat this extraction process three more times. Prepare a negative control of the filter in the same manner. Evaporate the remaining methylene chloride in test tube 1 and test tube 2 using heat and/or nitrogen/filtered air as appropriate.

E. Add 5 mL of methanol to test tube 2 and the negative control, then evaporate to dryness using heat and/or nitrogen/filtered air as appropriate. Repeat this step a second time to ensure all methylene chloride is removed. Weigh and record the mass of the test tube 2 dried contents prior to analysis by GC/ECD.
F. Using the mass of the test tube 2 contents, prepare an approximately 10 ppm solution in methanol. Prepare the negative control in the same manner.
G. Analyze the test tube 2 methanol extract and negative control by GC/ECD. Confirm any organic explosives by GC/MS in electron ionization (EI) and/or chemical ionization (CI) modes and/or LC/MS analysis. EGDN and TNT are best identified by GC/MS (CI) while LC/MS should be used to identify NG.
H. Extract the remaining material in test tube 1 (from step 6.1.D) with 5 mL of acetone. Vortex or masticate with a glass stir rod and centrifuge as needed. Transfer the extract into test tube 3. Evaporate test tube 1 and test tube 3 to dryness using heat and/or nitrogen filtered air as appropriate. Weigh and record the masses of the test tube 1 and test tube 3 contents.
I. Analyze any visible material in test tube 3 by FTIR for nitrocellulose. Some nitrate salts may be soluble in acetone. Nitrocellulose, if present, would typically be on the bottom of test tube 3.
J. Some dynamites may have material characteristic of wood fibers remaining after the methylene chloride/acetone extraction in test tube 1. Examine the remaining material in test tube 1 (from section 6.1.H) under the microscope and note any fibrous material visually consistent with wood. Take a photograph if fibrous material is observed.
K. Analyze the methylene chloride/acetone insoluble material in test tube 1 by SEM/EDS to determine the presence of elements such as nitrogen, sodium, calcium, potassium, carbon, oxygen, and chlorine. In addition, some dynamites contain microspheres. If present, photograph and conduct elemental analysis.
L. If sufficient material remains in test tube 1, analyze by XRD to determine the presence of ammonium nitrate, calcium carbonate, sodium chloride, potassium nitrate, or sodium nitrate. For XRD analysis, grind the material into a fine powder. Spread the ground material onto an XRD sample holder and place in the instrument for analysis.
M. (Optional) FTIR and Raman spectroscopy can be utilized for the analysis of other bulk materials which may be present.
N. (Optional) Plasticware containers should be used throughout the following procedures to avoid leaching of ions from glassware. Label a clean plastic test tube, test tube 4. Extract a sample of the methylene chloride/acetone insoluble material from test tube 1 (from section 6.1.H) using deionized water and dilute as necessary to create an approximate 80 ppm solution. Retain an equal portion of the water as a negative control. Prepare a plastic syringe and a 0.2 µm membrane filter (mounted

on a plastic syringe) by flushing with deionized water. Analyze the extract for the presence of anions and cations of interest.

7 CALCULATIONS

A. Based upon the initial approximate mass of the sample from section 6.1.C and approximate masses of the remaining material in test tubes 1 through 3 (steps 6.1.E and 6.1.H), the approximate percentage of each component can be determined. Generally, dynamites can contain various combinations of explosive materials. Noting the ratio of explosive materials may be probative.
B. The mass of the inorganics and other insoluble materials (e.g., nitrate salts, wood chips, microspheres, etc.), is obtained by taking the difference between test tube 1 empty and test tube 1 after the acetone extraction and drying down in step 6.1.H.
C. The mass of the organic explosives material (e.g., NG, EGDN, TNT) in a dynamite is obtained by taking the difference between the mass of test tube 2 empty and test tube 2 after the methylene chloride extraction and drying down of methanol in step 6.1.E.
D. The mass of the nitrocellulose component is obtained by taking the difference of test tube 3 empty and test tube 3 after the acetone extraction and drying down in step 6.1.H.
E. If requested to compare two or more dynamite samples, each sample will be analyzed in triplicate to include estimating the percentages of each component.

8 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 dynamites.

8.1 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.

8.2 Material Identification

The minimum identification requirements for a dynamite are:

- The identification of at least one of the following: NG or EGDN.
- The presence of an absorptive material to include, but not limited to, the following examples:
    - Visual presence of wood fibers, diatomaceous earth, ground seeds, or newspaper
- The confirmation of nitrocellulose by FTIR.
- The identification of at least one nitrate salt, such as ammonium nitrate or sodium nitrate.

Some improvised mixtures may contain ammonium nitrate, potassium nitrate, and/or sodium nitrate with NG and/or EGDN but not have the appearance of a commercially manufactured product. These mixtures may be identified as an improvised dynamite.

9 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.

10 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 material may be limited by sample size. If insufficient material is available for analysis, use the Identification of General Unknowns technical procedure.

11 Safety

The handling of 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.

12 References

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

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

13 Revision History

Revision	Issued	Changes
05	09/30/2022	Updated to new document template. Replaced diethyl ether with methylene chloride and made applicable updates to extraction process. Added calculations and decision criteria sections. Updated limitations. Added ASTM references.