

Slurries, Emulsions, and Water Gels Analysis

Table of Contents

1	INTRODUCTION	2
2	SCOPE	2
3	EQUIPMENT	2
3.1	Equipment.....	2
3.2	Instruments.....	2
3.3	Chemicals/Reagents.....	3
4	STANDARDS AND CONTROLS	3
4.1	Redacted Control	3
5	SAMPLING	3
6	PROCEDURE	3
6.1	Sample Analysis.....	4
6.2	Emulsion Extraction	4
6.3	Slurry/Water Gel Extraction.....	5
7	DECISION CRITERIA	7
7.1	Instrumental Results	7
7.2	Material Identification	7
7.2.1	Emulsion.....	8
7.2.2	Water Gel.....	8
8	MEASUREMENT UNCERTAINTY	8
9	LIMITATIONS	8
10	SAFETY	9
11	REFERENCES	9
12	REVISION HISTORY	9

Slurries, Emulsions, and Water Gels Analysis

1 INTRODUCTION

Explosives used in mining and demolition may involve the use of slurries, emulsions, or water gels. All of these products may contain components such as ammonium nitrate, sodium nitrate, calcium nitrate, sensitizers, aluminum, and glass or phenolic microspheres.

Emulsions are products that contain a substantial amount of a supersaturated oxidizer in water surrounded by an immiscible fuel. These components are blended and do not separate due to the presence of an emulsifying agent. Emulsions are water resistant.

Slurries are products that contain an oxidizer and fuel within an aqueous solution containing a thickener. Slurries are not water resistant.

Water gels are slurries that contain a cross-linking agent and are more water resistant than a slurry.

This procedure will assist in extracting and identifying the various components of these explosives as well as distinguishing between them.

2 SCOPE

This procedure is suitable for uninitiated samples which are suspected of being a slurry, emulsion, or water gel explosive 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)
- SEM stubs or carbon planchets with liquid adhesive (e.g., Duro-tak), carbon adhesive tabs, or aluminum or copper tape
- General laboratory supplies

3.2 Instruments

- Fourier transform infrared (FTIR) spectrometer with attenuated total reflectance (ATR) or microscope attachment
- Gas chromatograph with electron capture detector (GC/ECD)
- Gas chromatograph with flame ionization detector (GC/FID)
- 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
- Scanning electron microscope with energy dispersive X-ray spectrometer (SEM/EDS)
- X-ray diffractometer (XRD)

3.3 Chemicals/Reagents

- Acetone (HPLC grade)
- Air (compressed)
- Anhydrous diethyl ether (reagent grade)
- Deionized (DI) water (18.2 MΩ)
- Hexane (reagent grade)
- 10% Hydrochloric acid (HCl, prepared from reagent grade)
- Isopropyl alcohol (70% commercial product)
- Nitrogen (high purity)
- Pyridine (reagent grade)
- Sodium bicarbonate (reagent grade)
- Sodium sulfate (reagent grade)
- Tri-Sil HTP (Thermo Scientific)

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., monomethylamine, ethanolamine).

Redacted

5 SAMPLING

Refer to the sampling procedures in the [Explosives Quality Assurance and Operations Manual](#).

6 PROCEDURE

Explosive 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.

The identification of these materials is generally limited by sample size. The minimum sample size required to utilize this procedure is approximately 300 mg. If smaller amounts are submitted, only follow steps 6.1.A, 6.1.B, and the steps in section 6.2. See section 7 for sample size effects on material identification.

6.1 Sample Analysis

- A. Examine the material under the microscope and note details of its physical characteristics such as homogeneity, color, phases, etc. The overall appearance of slurries, emulsions, and water gels will vary by purpose and use. In general, these explosives appear as a jelly or paste-like material. Oxidizer prills or crystals are often observed, as well as microspheres.
- B. Analyze the sample by FTIR to determine possible components present in the sample.
- C. Place approximately 100 mg of material in a test tube and add up to approximately 2 mL of hexane. A water gel or slurry will remain essentially unchanged, but an emulsion will disintegrate immediately. If the material disintegrates in hexane, follow the emulsion extraction steps in section 6.2.
- D. If no change is seen in 6.1.C, place 100 mg of material in approximately 2 mL of DI water. Slurries are not durable in water and will start to break up within approximately an hour. Water gels are more durable and may break up or only swell in water. If the material swells or disintegrates in water, follow the slurry/water gel extraction steps in section 6.3.
 1. NOTE - Prior to beginning section 6.3 of this procedure, note that digestion and derivatization of the sample must be completed the same day. Do not begin the digestion of the sample if you do not have the allotted time (about 5 to 6 hours) to complete this portion of the analysis.

6.2 Emulsion Extraction

- A. Label four empty 16 x 100 mm test tubes (e.g., test tube 1 through 4).
- B. Prepare a supply of diethyl ether by drying with sodium sulfate. Prepare a 0.2 μm membrane filter (mounted on a glass syringe) by flushing with diethyl ether, and then filter the dried diethyl ether into a beaker. Evaporate approximately 10 mL of anhydrous diethyl ether to dryness in test tube 4. This serves as a negative control. If visible material is present, repeat with fresh diethyl ether. If no visible material is present, save this test tube for use in section 6.2.I.
- C. Place approximately 100 mg of sample in test tube 1. Extract the sample with approximately 3 mL of anhydrous diethyl ether.
- D. Break up the sample if necessary with a glass stirring rod after it has been placed in the test tube.
- E. Transfer the ether from test tube 1 into test tube 2. If necessary, centrifuge test tube 1 prior to transferring the ether into test tube 2.
- F. Repeat the extraction steps in 6.2.C through 6.2.E three more times.

- G. Save the ether insoluble material that remains in test tube 1 and dry using heat and/or nitrogen/filtered air as appropriate. Submit a small portion of the material to SEM/EDS for analysis. The elemental composition of slurries, emulsions, and water gels may include sodium, calcium, nitrogen, oxygen, aluminum, and silicon.
 1. (Optional) Use SEM to record electron micrographs of the morphology of aluminum and/or microspheres.
 2. Transfer a spatula quantity of the ether insoluble material to a mortar and grind to a fine powder. Prepare the powder for XRD analysis to detect crystalline components such as ammonium nitrate, sodium nitrate, calcium nitrate, and/or aluminum.
- H. Evaporate the ether extract in test tube 2 from section 6.2.F to dryness using heat and/or nitrogen/filtered air as appropriate.
- I. Extract the residue remaining in test tube 2 with up to 5 mL of hexane twice, and transfer the hexane to test tube 3. Retain a portion of the hexane as a blank.
- J. The hexane extract in test tube 3 is analyzed by GC/FID to characterize the hydrocarbon profile (e.g., oil, waxes).
 1. If the results of the FID analysis reveal that the carbon-range distribution falls below ~C₂₅, the extracts may be analyzed via GC/MS in EI mode (FIRE method) for classification of the hydrocarbon present. Refer to the [Fire Debris and Ignitable Liquid Analysis](#) procedure for the FIRE method parameters.
- K. Evaporate the hexane extract in test tube 3 to dryness using heat and/or nitrogen/filtered air (as appropriate) then analyze a portion by FTIR for any emulsifiers.
- L. Dry down the remaining material in test tube 2 from section 6.2.I using heat and/or nitrogen/filtered air as appropriate. Reconstitute the material in several milliliters of acetone to achieve an approximate 10 ppm solution. Retain a portion of the acetone as a blank. Screen the acetone extract by GC/ECD with confirmation of any organic explosives by GC/MS in electron ionization (EI) or chemical ionization (CI) modes and/or LC/MS analysis.
- M. Extract a spatula tip quantity of the bulk specimen or the ether insoluble material in up to 10 mL of DI water. Retain an equal portion of the water as a negative control. Plastic ware containers should be used throughout these procedures to avoid leaching of ions from glassware. Prepare a 0.2 µm membrane filter (mounted on a plastic syringe) by flushing with DI water. Flush portions of the negative control and then the sample through the filter and into their respective autosampler vial for IC analysis to identify ethanolamine or monomethylamine. If the sample extract is too concentrated, further dilution may be necessary. In addition, IC analysis may be helpful in confirming the presence of other chemicals (if not already confirmed).

6.3 Slurry/Water Gel Extraction

- A. Label three empty 16 x 100 mm test tubes (e.g., test tube 1 through 3).
- B. Place approximately 80 mg of sample in test tube 1. Extract the sample with approximately 1 mL of acetone. Prepare a negative control by adding the same volume of acetone to an empty test tube.

- C. Break up/masticate the sample with a glass stirring rod after it has been placed in the test tube and vortex well.
- D. Centrifuge the sample (capped) in test tube 1 and then remove the acetone extract from test tube 1 into test tube 2.
- E. Prepare a 0.2 µm membrane filter (mounted on a plastic syringe) by flushing with acetone. Flush portions of the negative control and then the acetone extract through the filter and into their respective autosampler vials. Screen the acetone extract for any organic explosives by GC/MS in CI mode and LC/MS. If the sample extract is too concentrated, further dilution may be necessary.
- F. Save the acetone insoluble material that remains in test tube 1 and dry using heat and/or nitrogen/filtered air as appropriate.
- G. Once dry, place approximately 20 mg (18-22 mg) of the acetone insoluble material from test tube 1 into test tube 3. Extract the material in test tube 3 with about 3 mL of DI water.
 - 1. NOTE - If more than 22 mg or less than 18 mg of the acetone insoluble will be used, sodium bicarbonate and HCl amounts must be adjusted below.
- H. Break up/masticate the sample with a glass stirring rod and vortex well.
- I. Centrifuge the sample in test tube 3 and then remove as much water extract as possible from the water insoluble.
 - 2. (Optional) This water extract may be saved for IC analysis in step 6.3.R.
- J. Save the water insoluble material that remains in test tube 3 and dry using heat (about 45°C) and/or nitrogen/filtered air as appropriate. The sample will take approximately 1 to 2 hours to dry (less with nitrogen).
- K. Once the water insoluble material in test tube 3 is dry, add 200 µL of 10% hydrochloric acid. Vortex the sample then place it on the heat block (uncapped) for approximately 3 hours at 80-85°C for digestion. Utilize **Redacted** as a positive control (see step 4.1).
- L. While the water insoluble material in test tube 3 is digesting, submit a small portion of the acetone insoluble in test tube 1 to SEM/EDS for analysis. The elemental composition of slurries, emulsions, and water gels may include sodium, calcium, nitrogen, oxygen, aluminum, and silicon.
 - 1. (Optional) Use SEM to record electron micrographs of the morphology of aluminum and/or microspheres.
- M. Transfer a spatula quantity of the acetone insoluble material in test tube 1 directly
Redacted
- N. Once the water insoluble material in test tube 3 has digested for approximately 3 hours, remove it from the heat and add about 48 mg of sodium bicarbonate. Add a few drops of DI water, vortex the sample, and place it on the heat block at about 45°C to dry under a stream of nitrogen/filtered air.
 - 1. NOTE – Sample must be COMPLETELY dry because the Tri Sil HTP reagent to
Redacted

spatula may be used to disturb the material in the test tube, and the test tube can be returned to the heat block under nitrogen/filtered air to continue drying. Dried material should appear as a light yellow to white crystalline substance.

- O. Once the material in test tube 3 is completely dry, add 1 mL of the Tri-Sil HTP reagent to test tube 3. Use a spatula to scrape the material from the bottom of the test tube so that all of the material is exposed to the reagent. Quickly cap the test tube and vortex the sample. Let the sample sit for at least 5 to 10 minutes.
 1. NOTE – It is possible that a white precipitate will form upon adding the Tri-Sil HTP. This is due to the formation of ammonium chloride from the components that make up the derivatizing reagent.
- P. Centrifuge the sample in test tube 3.
- Q. Transfer the derivatized solution into an autosampler vial with insert and prepare a pyridine blank. Analyze the extract by GC/MS in EI mode (SUGAR method). Dilute the derivatized **Redacted** positive control by ~1:5 with pyridine before GC/MS analysis (see step 4.1).
- R. (Optional) Dilute a spatula tip quantity of the bulk specimen or the rest of the acetone insoluble material in test tube 1 in up to 10 mL of DI water. The water extract from step 6.3.I may also be used. 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 0.2 µm membrane filter (mounted on a plastic syringe) by flushing with DI water. Flush portions of the negative control and then the sample through the filter and into their respective autosampler vial for IC analysis. If the sample extract is too concentrated, further dilution may be necessary. In addition, IC analysis may be helpful in confirming the presence of other chemicals (if not already confirmed).

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 slurries, emulsions, and water gel samples.

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.

Utilize extracted ion chromatograms for the following m/z ions to aid in identification of mannose and galactose: 73, 147, 191, 204, 217.

Redacted

Redacted

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.

Redacted

10 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 (less than a few grams) and properly store larger quantities in approved containers.

11 REFERENCES

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

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

12 REVISION HISTORY

Revision	Issued	Changes
05	09/30/2022	Updated to new document template. Updated procedure to include new water gel extraction process. Added decision criteria section. Updated Limitations. Added ASTM references.
06	03/15/2023	Removed GC/ECD from step 6.3.E. Updated step 6.3.M. Added dilution to steps 4.1 and 6.3.Q.