Slurries, Emulsions, and Water Gels Analysis

1 Scope

These procedures describe the general process for the analysis of bulk slurries, emulsions, and water gels. These procedures are suitable for bulk samples which are suspected of being a slurry, emulsion, or water gel explosive. These procedures apply to caseworking personnel conducting work in explosives chemistry analysis.

2 Introduction

Explosives used in mining and demolition involve the use of water and oil based gels and slurries. Redacted These procedures will assist in extracting and identifying the various components of these explosives.

3 Equipment/Materials/Reagents

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

3.1 Equipment

- Balance
- 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)

3.2 Materials

- Autosampler vials and caps
- Disposable plastic syringes

- Glass stirring rod
- 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)
- Hexane (reagent grade)
- Isopropyl alcohol (70% commercial product)
- 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.

Redacted

5 Sampling

Refer to the Sampling Procedures SOP in the Explosives Quality Assurance 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 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 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 gels will vary by purpose and use. In general, these explosives appear as a jelly or paste-like material. Redacted

6.2 (Optional) Place up to 5 grams of sample in deionized water. Water gels are not durable in water and will start to break up within approximately an hour. Slurries are durable and will not be affected by the water except to swell. Place another few grams of the bulk material in another test tube, and add up to 2-3 mL of hexane. A water gel or slurry will remain essentially unchanged, but the emulsion will disintegrate immediately.

6.3 Label four 15 mL test tubes 1 through 4.

6.4 Prepare a supply of diethyl ether by drying with sodium sulfate and then filtering into a beaker. Evaporate approximately 10 mL of anhydrous diethyl ether to dryness in test tube 4. This serves as a negative control. If a residue is present, repeat with fresh diethyl ether. If no residue is present, save this test tube for use in section 6.11.

6.5 Place up to 1 g of sample in test tube 1. Dissolve the sample in up to 3 mL of anhydrous diethyl ether.

6.6 Break up the sample if necessary with a glass stirring rod after it has been placed in the test tube.

6.7 Remove the ether from test tube 1 into test tube 2.

6.8 Repeat sections 6.5 through 6.7 three more times.

6.9 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 residue to SEM/EDS for analysis. Redacted

(Optional) Use SEM to record electron micrographs of the morphology of aluminum and/or glass microspheres. Transfer a spatula quantity of the material to a mortar and grind to a fine powder and spread onto an XRD sample holder (zero background holder with or without depression) for analysis by XRD. Redacted

6.10 Evaporate the ether extract in test tube 2 from section 6.7 to dryness using heat and/or nitrogen/filtered air as appropriate.

6.11 Extract the residue remaining in test tube 2 with up to 5 mL of hexane twice transferring the hexane to test tube 3. Retain a portion of the hexane as a blank.

6.12 The hexane extract in test tube 3 is analyzed by Redacted

6.13 Evaporate the hexane extract in test tube 3 to dryness using heat and/or nitrogen/filtered air (as appropriate) then analyze a portion by Redacted

6.14 Dry down the remaining residue in test tube 2 from section 6.11using heat and/or nitrogen/filtered air as appropriate. Reconstitute the residue in several milliliters of acetone. Retain a portion of the acetone as a blank. Redacted

6.15 Dilute a spatula tip quantity of the bulk specimen or the ether insoluble material in up to 10 mL of deionized 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 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 Redacted If the sample extract is too concentrated, further dilution may be necessary.

7 Calculations

Not applicable.

8 Measurement Uncertainty

Not applicable.

9 Limitations

9.1 The presence of the ammonium ion or ammonium nitrate does not constitute confirmation of a slurry or an emulsion explosive as there are many explosive and fertilizer products that contain ammonium nitrate as an ingredient. Combinations of other explosives can also result in similar findings, so personnel should exercise caution in identifying residues having originated from slurries and emulsions.

9.2 The identification of uninitiated material is generally limited by sample size.

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.

Barsotti, D.J., et al, *The Use of Ion Chromatography in the Analysis of Water Gel Explosives*, Proceedings of the International Symposium on the Analysis and Detection of Explosives, 1983.

Bender, E.C. and Crump, J. The Instrumental Analysis of Intact and Post Blast Water Gel and Emulsion Explosives in: *Advances in Analysis and Detection of Explosives: Proceedings of the 4th International Symposium on Analysis and Detection of Explosives, September 7-10, 1992 Jerusalem, Israel* Yinon, J. - Ed. Kluwer Academic Publishers Dordrecht, Holland 1992 pp. 179-88.

Davis, T. L., *The Chemistry of Powder & Explosives*, Angriff Press, 1943.

Kohler, J., Meyers, R., *Explosives*, fourth edition, 1993.

Yinon, J., ed., *Advances in Analysis and Detection of Explosives: Proceedings of the 4th International Symposium on Analysis and Detection of Explosives*, September 7-10, 1992 Jerusalem, Israel, Kluwer Academic Publishers, Dordrecht, Holland, p. 83.

Rev. #	Issue Date	History
3	10/04/2018	Changed title to Slurries, Emulsions, and Water Gel Analysis and made the addition throughout. Administrative changes for grammar and clarity. Removed testmix components in section 4. Added location-specific PMP references to section 6. Changed sections 6.2 and 6.5 to use "up to" a volume of solvent. Added SAU IOG reference and modified IOSS reference.
4	12/16/2019	Clarified that heat and/or nitrogen/filtered air can be used as appropriate. Removed sampling plan from section 5. 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