

Ammonium Nitrate-Based Binaries Analysis

1 Scope

These procedures describe the general process for the analysis of bulk ammonium nitrate-based binaries and the identification of their components. These procedures are suitable for bulk samples which are suspected of being a binary explosive mixture containing ammonium nitrate as the oxidizer and a fuel such as nitromethane or sugar. These procedures apply to caseworking personnel conducting work in explosives chemistry analysis.

2 Introduction

A common class of materials which include binary explosives and simple oxidizer/fuel mixtures based on ammonium nitrate (AN) as the oxidizer are frequently encountered in the FBI Laboratory. Some examples of these materials include AN/fuel oil (ANFO), AN/nitromethane (ANNM), AN/aluminum (ANAL), and AN/icing sugar (ANIS).

All of the individual components are readily available from retail stores, hardware stores, and pharmacies. AN can be found in fertilizers and cold packs, nitromethane is used as a racing fuel and may be purchased in hobby shops for various model engine applications, and fuel oil or diesel fuel is readily available at many gas stations. AN is generally sold in 40 or 50 pound bags of prilled (i.e., pelletized) material. When fertilizer grade AN is mixed with calcium carbonate, it is commonly referred to as calcium ammonium nitrate (CAN) fertilizer. AN is also available in explosives grade form. These procedures will assist in identifying the various components of these explosives.

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 flame ionization detector (GC/FID)
- Gas chromatograph with mass spectrometer (GC/MS)
- Headspace gas chromatograph with mass spectrometer (HS-GC/MS)
- Ion Chromatograph (IC)
- 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

- Carbon disulfide (reagent grade)
- Deionized water (18.2 M Ω)
- Hexane (reagent grade)
- Isopropyl alcohol (70% commercial product)
- Nitrogen (high purity)

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.

- IC Testmixes
- Headspace GC/MS Volatiles Testmix
- Fuel Oil Positive Control
- GC/FID Testmix

4.1 Additional Positive Controls

Additional positive controls may be prepared as necessary in order to identify components of mixed samples (e.g., AN, icing sugar, aluminum). They may be prepared in a manner appropriate for the analytical technique being used.

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 personnel conducting the examination and to prevent contamination of evidence.

Review and understand all safety information contained in Section 11 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 Microscopically observe the material noting any round spheres or prills of AN. AN may also be present in powder form (e.g., when inert materials have been added as a filler to desensitize the product). Describe the liquid phase and analyze separately, if present.

6.2 (Optional) Analyze a portion of the sample on the FTIR spectrometer to assist in identifying AN and any additional sources of fuel or other materials.

6.3 (Optional) Analyze a portion of the sample on the Raman spectrometer to assist in identifying AN and any additional sources of fuel or other materials.

6.4 (Optional) SEM/EDS can be used to analyze any fillers or inorganic coatings associated with the production of AN. These coatings and fillers can be used to compare different samples/sources of AN for likeness. The SEM/EDS results for AN should detect nitrogen and oxygen. Magnesium, calcium, aluminum, silicon, or sulfur may be detected as a result of the manufacturing process of prilled AN.

6.5 (Optional) A small amount of sample can be screened on the Headspace GC/MS for volatile compounds. Small items of evidence may also be screened if the item can fit within a 10

or 20 mL vial. A 0.5 mL sample of the headspace GC/MS volatiles testmix in an autosampler vial serves as a positive control. A sealed blank autosampler vial serves as a negative control. The evidence may be heated up to 90°C, prior to headspace sampling, based on the individual's judgment on how much heating is necessary and for how long. The situation may be that ambient temperature is sufficient, or only gentle heating.

6.6 If indicated by visual appearance, FTIR or Raman analysis, or other preliminary data, the sample can be washed with hexane which will remove any petroleum products, distillates, or similar materials. This could include fuels such as kerosene, diesel fuel, some charcoal lighter fluids, waxes, and cooking oils. Extract with just enough solvent to allow swirling or agitation over the material. The extract may be filtered and/or concentrated using heat and/or nitrogen/filtered air as appropriate. The final volume of the extract will depend on the amount of co-extracted material from the sample as well as the amount of extracted product present. If filtering and/or extract concentration is required, a negative control will also be prepared in the same manner. Transfer the solvent to an autosampler vial for analysis by GC/FID and/or GC/MS in electron ionization (EI) mode.

6.7 A portion of the prills and/or powder will be examined by XRD (before or after the hexane rinse). For XRD, a spatula quantity of prill or powder material is ground to a fine powder then spread onto an XRD sample holder (zero background with or without depression) for analysis. The results are compared to a sample of known AN.

6.8 (Optional) Dilute a spatula tip quantity of the specimen in up to 50 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 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 analysis to determine the presence of any anions or cations. The cations and anions testmixes serve as the positive controls.

7 Decision Criteria

7.1 Instrumental Results

The following criteria will be met in order for a qualitative identification to be made. The identity of a material will be confirmed by comparison to a reference or known material, if available. Reference or known materials may be run concurrently with an unknown sample or may be previously analyzed on the instrument under the same parameters. A reference or known material will be analyzed by at least one spectroscopic technique used for comparison to an unknown. All results should be verified using orthogonal techniques or alternate methods.

When a reference or known material is not available or when only reference data (e.g., from scientific literature, publications, or an instrument library) is used, a material may be reported as “consistent with” a substance.

7.1.1 Chromatography

Peaks should show good chromatographic characteristics, with reasonable peak shape, width, and resolution.

The retention time of the peak of interest should be within $\pm 2\%$ of that for a contemporaneously analyzed reference or known material for gas chromatography and $\pm 5\%$ for liquid chromatography.

The baseline signal-to-noise ratio (SNR) for an analyte should be greater than three to be considered a peak. The signal intensity for an analyte peak should be at least ten times greater than the intensity of any carryover or system peaks which may have been present in analyses just prior to the sample (e.g., blanks or negative controls).

7.1.2 Mass Spectrometry

The mass spectrum of the analyte of interest should compare favorably with that of a contemporaneously analyzed reference or known material.

Characteristic ion plots are reviewed to determine the potential presence of a target analyte. The absence of a primary ion indicates a non-detect.

7.1.3 XRD

The diffraction patterns from the questioned compound should compare favorably to the corresponding reference or known material.

If the unknown material is matched through a library search, a reference or known material may be analyzed for comparison, if available. Tentative identifications may also be confirmed through orthogonal techniques such as FTIR, Raman, SEM/EDS, or GC/MS.

7.1.4 SEM/EDS

Peaks in the EDS spectrum should exhibit a Gaussian peak shape and a minimum SNR of 3:1. The elemental composition of the questioned compound should compare favorably to the corresponding elemental composition of the reference or known material. Elemental assignments made by the software should be verified by the individual conducting the exam.

7.1.5 Other Tests

The results of all tests (e.g., visual inspections, FTIR, Raman, pH) should compare favorably to the corresponding reference or known material.

7.2 Material Identification

An AN binary can be identified based on the visual characteristics and chemical composition (AN in addition to a fuel component). This combination is required to indicate a combination of materials which may be capable of causing an explosion.

The AN and fuel components can be identified when the analytical results from two orthogonal techniques corroborate each other. The AN and fuel components should be compared to reference or known materials.

An example of report wording is as follows: Item 1 was identified as a mixture of ammonium nitrate and a petroleum distillate. Examples of petroleum distillates include fuel oils such as diesel and kerosene. The combination of ammonium nitrate and a fuel oil is commonly known as ammonium nitrate/fuel oil (ANFO), a high explosive.

If an examiner is qualified and authorized in fire debris analysis, then the class of petroleum distillate or product may be reported.

8 Calculations

Not applicable.

9 Measurement Uncertainty

Not applicable.

10 Limitations

10.1 The presence of the ammonium ion or AN does not constitute confirmation of ANFO or other ammonium-based binaries as there are many explosive and fertilizer products that contain AN as an ingredient.

10.2 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; however, a specific combination of compounds is mandatory to establish residues as consistent with an explosive mixture. Refer to the Explosive Residue Analysis SOP.

11 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.

12 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.

Cooper, P. and Kurowski, S. *Introduction to the Technology of Explosives*, VCH, New York, 1996.

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. Removed PLM microscopy from section 6.2. Reworded section 6.6 examples. Removed reference to Fire Debris SOP in previous section 6.9 and ignitable liquids positive controls in section 4.4. Added section 7 Decision Criteria. Added SAU IOG reference and modified IOSS reference.
4	12/16/2019	Changed title to Ammonium Nitrate-Based Binaries Analysis. 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.

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Approval

Explosives Unit Chief

Date: 12/13/2019

TL Approval

Explosives Chemistry
Technical Leader

Date: 12/13/2019

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

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. The use of nitroglycerin is being phased out in favor of less sensitive materials such as gels and slurry-type explosives. These procedures will assist in extracting and identifying the various components of this explosive.

Composition of dynamite (may or may not include all of the following chemicals): 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.

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 M Ω)
- 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.

- IC Testmixes
- GC/ECD Testmix
- LC/MS Testmix
- TSQ Standard Mix

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. Viewed microscopically, wood fibers which are distinguishable by color as well as visible cell structures characteristic of wood fibers may be seen. In addition to wood fibers, 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. Allow the residue to dry, then analyze the residue by FTIR for nitrocellulose. Some dynamites may have material characteristic of wood fibers remaining after the ether extraction. Examine the remaining material in the test tube (from 6.3) under the microscope and note any fibrous material consistent with wood.

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 the presence of ammonium, sodium, calcium, potassium, perchlorate, chloride, carbonate, and nitrate ions. The cations and anions testmixes serve as the positive controls.

6.9 Analyze the ether and acetone insoluble material by SEM/EDS to determine the presence of elements such as nitrogen, sodium, calcium, potassium, carbon, oxygen, and chlorine.

6.10 If sufficient material is present, analyze the material (from section 6.9) by XRD to determine the presence of ammonium nitrate, calcium carbonate, sodium chloride, potassium nitrate, or sodium nitrate. 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.

9.2 If ammonium nitrate and/or sodium nitrate are present with nitroglycerin and/or ethylene glycol dinitrate, then the material/residue is consistent with dynamite. Combinations of other explosives can also result in similar findings. Therefore, caution should be exercised in identifying residues originating from dynamite.

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

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.

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Approval

Explosives Unit Chief

Date: 12/13/2019

TL Approval

Explosives Chemistry
Technical Leader

Date: 12/13/2019

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. These products may contain components such as ammonium nitrate, sodium nitrate, calcium nitrate, sensitizers, emulsifiers (e.g., lecithin), oils, water, aluminum, and glass or phenolic microspheres. 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 M Ω)
- 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.

- IC Testmixes
- Monomethylamine Positive Control
- Ethanolamine Positive Control
- GC/ECD Testmix
- LC/MS Testmix
- GC/FID Testmix

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 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 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. Some examples of materials mixed into this matrix include oxidizers, such as ammonium nitrate (powdered and/or prilled) or sodium nitrate, metals (such as aluminum), glass spheres, rubber, smokeless powder, water, oils, gels, and emulsifiers.

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. The elemental composition of slurries, emulsions, and water gels may include sodium, calcium, nitrogen, oxygen, aluminum, and silicon. (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. Slurries and emulsions may contain ammonium nitrate, sodium nitrate, calcium nitrate, and/or aluminum.
- 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 GC/FID to characterize the hydrocarbon profile (e.g., oil, waxes).
- 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 FTIR for any emulsifiers found in emulsions.
- 6.14** Dry down the remaining residue in test tube 2 from section 6.11 using 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. 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 or LC/MS analysis.
- 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 IC analysis to identify ions of nitrate, ammonium, sodium, calcium, ethanolamine, and monomethylamine. 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

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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.

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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

Fire Debris Analysis

1 Scope

These procedures describe the general process for fire debris analysis. These procedures are suitable for most fire debris samples and can be used to identify single compounds, mixtures, and non-petroleum based ignitable liquids. These procedures apply to caseworking personnel conducting work in fire debris analysis.

2 Introduction

Analysis of fire debris for ignitable liquids used as accelerants plays a large role in arson investigations and may also be of use in investigations of bombing incidents and civil rights violations. Gasoline, fuel oils, charcoal lighter fluids, and paint thinners are the most commonly encountered ignitable liquids in fire debris analysis.

Ignitable liquids are often mixtures containing a large number of hydrocarbon constituents (gasoline, perhaps the most complex, has over 400). The collection and concentration of ignitable liquids takes advantage of the volatility of these constituents. In routine fire debris analysis, a charcoal adsorbent is added to the container then the closed container is heated. Volatile constituents are adsorbed onto the charcoal and then eluted with a solvent for further analysis.

Difficulties in identifying ignitable liquids arise from their complex composition, the interferences caused by the matrices from which ignitable liquids are recovered (i.e., pyrolysis products of styrene from carpet backing), and the loss of volatile constituents from samples through weathering. Consequently, gas chromatography-mass spectrometry (GC/MS) analysis of ignitable liquids in fire debris utilizes ion profile analysis to compare their residues to reference materials and standards.

3 Equipment/Materials/Reagents

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

3.1 Equipment

- Fourier transform infrared spectrometer (FTIR)
- Gas chromatograph-mass spectrometer (GC/MS)
- Oven

3.2 Materials

- Charcoal strips (e.g., Albrayco) cut into approximately 1 cm x 1 cm squares
- Cotton-tipped applicators
- Filter paper
- Nail
- New metal cans with friction fit lids (“paint cans”)
- Nylon bags
- Scissors
- Stainless steel or galvanized wire, small paper clips
- Tape
- Test tubes, beakers, pipettes, and autosampler vials

3.3 Reagents/Solvents/Reference Materials

- Acetone (reagent grade)
- Air (compressed)
- Carbon disulfide (reagent grade)
- Deionized water (18.2 MΩ)
- Ethanol (reagent grade)
- Hexane (reagent grade)
- Ignitable liquids resolution test mixture (e.g., Cerilliant, Restek)
- Isopropyl alcohol (reagent grade)
- Methanol (HPLC grade)
- Methyl ethyl ketone (reagent grade)
- Nitrogen gas (high purity)
- Nitromethane (reagent grade)
- Pentane (reagent grade)
- Reference materials – A collection of commercially available fuels, solvents and specialty products such as gasoline, fuel oil #1, and fuel oil #2
- Toluene (reagent grade)

4 Standards and Controls

All reference materials and reagents will be verified prior to use. 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 preparation of all standards and controls referred to in this document.

4.1 Headspace GC/MS Volatiles Testmix

The volatiles testmix is a 0.01% (0.1 $\mu\text{L}/\text{mL}$) solution of methanol, ethanol, isopropyl alcohol, methyl ethyl ketone, nitromethane, acetone, and toluene in 18.2 M Ω deionized water.

4.2 Standard Accelerant Mix (SAM)

The SAM is a 0.05% (0.5 $\mu\text{L}/\text{mL}$) solution of gasoline, diesel, and kerosene (1:1:1) in carbon disulfide, hexane, or methanol.

4.3 Gasoline Positive Control

The gasoline standard is a 0.1% (1.0 $\mu\text{L}/\text{mL}$) solution of gasoline in either carbon disulfide or methanol.

4.4 Ignitable Liquids Positive Controls

A variety of ignitable liquids are maintained for use as reference materials both in their neat form and in dilutions ranging from 0.05% to 1% in an appropriate solvent (e.g., methanol, hexane, carbon disulfide).

4.5 Charcoal Strips

4.5.1 New lots of charcoal strips are verified for efficacy by extracting a 10 μL gasoline sample in a container using the passive adsorption technique. A blank sample-size charcoal strip from the new lot is extracted with carbon disulfide and analyzed to detect any hydrocarbon contamination of the strips prior to casework. Verification data is retained in the verification files.

4.5.2 For each sample batch, a blank sample-size charcoal strip (from the same lot used for the sample batch) is extracted with carbon disulfide and analyzed for the presence of hydrocarbon contamination.

4.6 Extraction/Dilution Solvents

4.6.1 For each sample batch, an aliquot of the solvent used for final extraction or dilution is analyzed as a solvent blank.

4.6.2 When samples are directly extracted with solvent, as directed in section 6.2.2, an equivalent amount of solvent is filtered, evaporated to a volume approximating final sample sizes, and analyzed as a negative control.

4.7 System Blank

4.7.1 Prepare a system blank by placing a new carbon strip into a clean dry evidence container (e.g., metal can) and processing as described in section 6.1.

4.7.2 When appropriate, prepare a system blank each day that questioned samples are processed.

4.7.3 If the results of the system blank indicate that the system blank was contaminated, review the sample results to determine if and how the question sample results were affected. Evaluate the individual components of the system blank to determine possible contamination origin(s). Process and analyze a new system blank prior to resuming casework processing.

5 Sampling

Fire debris and solid items are routinely examined in their entirety. When a destructive technique such as solvent extraction (section 6.2.2) is to be used, a representative portion of a fire debris sample may be selected for analysis.

A portion of a liquid item is taken for analysis when the item appears to be homogenous. If the item appears to consist of two or more immiscible layers, then a sample is taken of each liquid layer and examined separately.

6 Procedure

Fire debris personnel will:

Cover work surfaces with a disposable material, such as kraft paper before beginning any examinations.

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 to conduct a QA/QC check to verify the instrument's reliability and reproducibility from analysis to analysis.

Evidence items should be received in air-tight and vapor-tight containers: paint cans, nylon or polyester bags, or glass jars. Containers not approved for use with evidence to be examined for trace volatiles or ignitable liquids include paper bags, cardboard boxes or tubes, other paper products/containers, film canisters, coffee cans, or any used containers. Evidence packaged in polyethylene bags (zipper-lock or trash bags), some plastic containers, or other questionable containers will be evaluated by the examiner prior to or after the analysis to determine the extent of any contamination related to the packaging. Improperly packaged fire debris will be noted and returned to the contributor without analysis.

Evidence items will be visually inspected and contents noted just prior to and/or after processing. Obvious odors should be noted to assist with the analysis strategy. However, intentional smelling of the evidence is not recommended as it may pose a health risk.

6.1 Passive Adsorption/Elution Technique

The routine analysis of fire debris samples (burned or unburned) involves extraction of the samples using a passive headspace concentration technique with activated charcoal followed by extract analysis with GC/MS.

6.1.1 For cans and glass jars, punch a hole through the lid using a nail. Remove the lid and thread a wire or paper clip with a charcoal strip attached through the hole. Seal the hole in the lid with tape. The tape also helps keep the wire in place.

6.1.2 Observe the contents of the container before replacing the lid to see that the contents are as described by the contributor. Secure the lid to the container making sure that the charcoal strip, now suspended inside the container, does not touch the contents.

6.1.3 For items in nylon bags, cut open the bag and secure a wire with a charcoal strip inside the bag but without physically contacting the evidence items. Reseal the bag using a heat sealer. If the bag cannot be resealed, then transfer the contents to a new nylon bag or unused metal can along with a charcoal strip.

6.1.4 Heat the container (or nylon bag) in an oven at approximately 80°C for at least three hours. After heating, allow the container to equilibrate to approximately ambient temperature. Heating times and temperatures may be varied depending upon the type of ignitable liquids and the quantity present in each sample.

6.1.5 Open the container in a hood and remove the charcoal strip from the wire. Cut the strip into two pieces and retain one portion in an autosampler vial as secondary evidence. Place the remainder of the strip into a small test tube (~12 x 75 mm) and add approximately 0.25 mL carbon disulfide. Vortex for about 30 seconds then rinse the strip with carbon disulfide repeatedly with the aid of a Pasteur pipette. The extract is then transferred to an autosampler vial and sealed.

6.1.6 Samples are analyzed along with appropriate standards, blanks, gasoline positive controls, and ignitable liquid positive controls by GC/MS in electron ionization (EI) mode.

6.2 Optional Techniques

Samples suspected of containing low-boiling compounds, such as alcohols, may need to be examined using a direct headspace technique. Samples containing heavy petroleum distillates may need to be solvent extracted. Non-aqueous liquid samples may be diluted with solvent and analyzed directly.

6.2.1 Direct Headspace

A small amount of sample can be screened on the Headspace GC/MS for volatile compounds. Small items of evidence may also be screened if the item can fit within a 20 or 40 mL vial. A 0.5 mL sample of the headspace GC/MS volatiles testmix in an autosampler vial serves as a positive control. A sealed blank autosampler vial and/or appropriate system blanks serve as negative control(s). The evidence may be heated up to 90°C, prior to headspace sampling, based on the individual's judgment on how much heating is necessary and for how long.

6.2.2 Solvent Extraction/Rinsing

After performing the passive adsorption/elution or headspace techniques, the sample can be washed with hexane or pentane which will remove any heavy fuels, such as diesel or kerosene. Pour just enough solvent into a clean container (e.g., a beaker) with sample to cover the debris. Agitate or swirl the sample for several minutes. Filter the solvent through filter paper into a clean beaker and concentrate using heat, compressed air, or nitrogen gas as necessary. The final volume of the extract will depend on the amount of co-extracted material from the sample debris as well as the amount of ignitable liquid present. Transfer the solvent to an autosampler vial, seal, and analyze by GC/MS (EI) as described in section 6.1.6.

Non-porous items such as pieces of glass or the inner surfaces of containers that may have held ignitable liquids may be rinsed with a suitable solvent (e.g., pentane, hexane, or carbon disulfide) to remove any ignitable liquid residues. Use the minimum amount of solvent necessary to rinse the appropriate surface. Solvent rinses can be concentrated by reducing the final volume under an air or nitrogen gas stream; however, this can result in the loss of low boiling point compounds.

Upon completion of the analysis, if the sample extract needs to be preserved, a charcoal strip is placed into the autosampler vial to allow the solvent to adsorb onto the strip for long-term storage of the extract.

6.2.3 Liquid Samples

Liquid samples should be tested for hydrophobicity by adding a few drops to an aliquot of deionized water and vortexing to determine if the sample is miscible with water. Liquid samples miscible with water may be analyzed directly by FTIR to determine if the bulk of the sample is consistent with water or an alcohol. Headspace GC/MS can be used to confirm the presence of alcohols and select volatile compounds. For non-aqueous liquid samples, a **Redacted** is prepared for analysis by diluting the equivalent of 5 **Redacted** of carbon disulfide.

Liquid samples may be evaluated for their ability to ignite by testing with an open flame. A few drops of the liquid are applied to a new cotton-tipped applicator and an open flame is introduced to the vapors just above the wetted cotton. If no ignition occurs in the vapor phase, then the flame is brought into direct contact with the cotton. Ease of ignition, flame color, and smoke characteristics should be noted.

6.3 Data Interpretation

Data interpretation of the GC/MS results will consist of comparing chromatographic profiles or extracted ion profiles with known ignitable liquids analyzed on the instrument acquisition data system or against the National Center for Forensic Science online database. Results will be reported using the ASTM E1618 ignitable liquid classification system whenever possible. Appropriate reference materials, if available, will be analyzed for direct comparison.

7 Calculations

Not applicable.

8 Measurement Uncertainty

Not applicable.

9 Limitations

Burned material from which the samples are extracted may contain many of the same components commonly found in ignitable liquids. The examiner must recognize these interferences during data interpretation. Refer to references listed below for a more detailed understanding of analytical limitations and data interpretation.

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.

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.

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

Explosives Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

Instrument Operations Manuals for the specific models and accessories used.

Ignitable Liquids Database, National Center for Forensic Science, <http://ilrc.ucf.edu/>.

ASTM Method E 1386, *Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction*.

ASTM Method E 1388, *Standard Practice for Sampling of Headspace Vapors from Fire Debris Samples*.

ASTM Method E 1412, *Standard Practice for Separation of Ignitable Liquid Residues from Fire Debris Samples by Passive Headspace Concentration With Activated Charcoal*.

ASTM Method E 1618, *Standard Test Method for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry*.

ASTM Method E 2451, *Standard Practice for Preserving Ignitable Liquids and Ignitable Liquid Residue Extracts from Fire Debris Samples*.

Bertsch, W., Holzer, G., and Sellers, C. S., *Chemical Analysis for the Arson Investigator and Attorney*, Huthig Buch Verlag: Heidelberg, 1993.

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Stauffer, E., Dolan, J.A, Newman, R., *Fire Debris Analysis*, Academic Press, Burlington, MA, 2008.

Rev. #	Issue Date	History
5	10/04/2018	Administrative changes for grammar and clarity. Edited use of term “accelerant” in section 2 Introduction. Added FTIR to section 3.1 Equipment. Added cotton-tipped applicators to section 3.2 Materials. Added “Solvents/Reference Materials” to section 3.3 header. Changed concentration units from ppm to % and µL/mL for section 4 testmix and positive control descriptions. Added section 4.7. Expanded section 5 to provide details for sample selections of debris and liquid evidence items. Added location-specific PMP references to section 6. Added paragraph to section 6 introduction discussing visual and olfactory inspection of evidence. Added sentence to section 6.1 summarizing routine fire debris technique used. Section 6.1.7 renumbered as 6.3 Data Interpretation and removed target compound analysis from the interpretation protocol. Edited section 6.2.2 to provide more specifics for solvent extraction/rinsing technique. In section 6.2.3, added FTIR and headspace GC/MS techniques; described flame test for liquid samples. Added CU Quality Assurance and Operations Manual and SAU IOG references. Added Chemistry Unit Chief and Explosives Chemistry Technical Leader to approval signature blocks.
6	12/16/2019	Removed sample selection from section 5. Removed SAU Chief and QA from approval lines. Removed unit references to PMPs.

Approval

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Chemistry Unit Chief Date: 12/13/2019

Explosives Unit Chief Date: 12/13/2019

TL Approval

Explosives Chemistry
 Technical Leader Date: 12/13/2019

Fire Debris Technical
 Leader Date: 12/13/2019

Smokeless Powder Analysis

1 Scope

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

2 Introduction

Smokeless powders are classified as propellants which are used in ammunition for most firearms. In addition, it can be used as the main charge in pipe bombs and other improvised explosive devices (IEDs).

Powders can be physically distinguished by shape: cylinders, disks, 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 a nitrated cellulose base known as 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. These procedures will assist in identifying the various components of smokeless powders.

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.

Unconsumed smokeless powder grains are easily identified by their unique physical characteristics and chemical composition. If no grains are observed, refer to the Explosive Residue Analysis standard operating procedure (SOP).

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 mass spectrometer (GC/MS)

- Liquid chromatograph with mass spectrometer (LC/MS)
- Micrometer
- Microscope (optical or digital) with optional digital camera

3.2 Materials

- Autosampler vials and caps
- Kraft paper
- Lighter, torch, or matches
- Various disposable glassware and plasticware

3.3 Reagents/Solvents/Reference Materials

- Isopropyl alcohol (70% commercial product)
- Methylene chloride (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 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.

- Smokeless Powder Standards
- Nitroglycerin Positive Control

4.1 Hodgdon HS-7 (365) Positive Control

An additional reference standard can be made by extracting a few grains of Hodgdon HS-7 (reference #365) smokeless powder with approximately 300 μ L methylene chloride. This can serve as a positive control for double-base powders. The standard is made up fresh for each day of use. Validation data for this powder can be retrieved from the appropriate validation file.

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 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 to conduct a QA/QC check to verify the instrument's reliability and reproducibility from analysis to analysis.

6.1 Debris suspected of being involved in an explosion must be carefully inspected under a microscope for grains. Burned and unburned grains of smokeless powder are often found in pipe threads, on tape material, and in crevices. Suspected grains are removed into a small test tube. Bulk powder samples submitted for analysis should also be inspected under a microscope looking for mixtures or atypical material.

6.2 Recovered grains or mixtures are then separated into similar groups based upon type, size (diameter and/or length), perforations, and color.

6.3 Grains are measured using a microscope or micrometer to determine diameter and length or thickness. Also, make note of perforations, color, or other distinguishing physical characteristics of the powder.

6.4 Extract several grains with approximately 300 μL of methylene chloride, and cap. Allow the solution to stand for at least one hour with occasional vortexing. Lesser quantities of solvent should be used for the analysis of very small quantities or a single grain.

6.5 Analyze the extract by GC/MS in electron ionization (EI) mode. The nitroglycerin can also be confirmed by LC/MS analysis.

6.6 A sample set should include a Smokeless Powder Standard, a positive control sample (such as Hodgdon HS-7) or nitroglycerin standard, methylene chloride blanks as negative controls, and sample extracts. Several common constituents of smokeless powders include NG, DNT, diphenylamine, ethyl centralite, methyl centralite, and dibutyl phthalate.

6.7 Analyze the methylene chloride insoluble grain by FTIR with an ATR accessory to determine the presence of nitrocellulose.

6.8 (Optional) If sufficient powder is present, a portion can be subjected to a flame to characterize its burning properties. Smokeless powder will burn rapidly with very little smoke and a brief flash of light.

6.9 A side-by-side chemical and physical comparison should be done with known reference powders if a brand name determination of an unknown powder is necessary. 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. If available, obtain a microscopic enlargement photo of the unknown powder(s). The FBI Laboratory maintains an extensive database with smokeless powders which can assist in brand name determination. The database includes the physical and chemical properties of smokeless powder.

7 Calculations

Not applicable.

8 Measurement Uncertainty

Not applicable.

9 Limitations

The identification of smokeless powder requires at least one powder grain. This grain must have the morphological characteristics and chemical constituents of smokeless powder. In the absence of visible grains, residues consistent with smokeless powders are identified by the various organic compounds present in propellants.

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.

Smokeless Powder Reference Collection, Federal Bureau of Investigation, Laboratory Division, Explosives Unit.

ASTM Standard E2998, 2016, "Standard Practice for Characterization and Classification of Smokeless Powder," ASTM International, West Conshohocken, PA, 2016, DOI: 10.1520/E2998-16, www.astm.org.

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Wolfe, D., Propellant Profiles, Volume 1, Wolfe Publishing Co., Inc., Prescott, AZ, 1982.

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3	10/04/2018	Administrative changes for grammar and clarity. Removed testmix components in section 4. Added location-specific PMP references to section 6. Removed autosampler vial for extractions in section 6.4. In section 6.9 changed to longer elution time for smaller quantities. Added SAU IOG reference and modified IOSS reference.
4	12/16/2019	Removed sampling plan from section 5. Removed SAU Chief and QA from approval lines. Removed unit references to PMPs.

Approval

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Explosives Unit Chief

Date: 12/13/2019

TL Approval

Explosives Chemistry
Technical Leader

Date: 12/13/2019

Black Powder, Black Powder Substitutes, and Pyrotechnics Analysis

1 Scope

These procedures describe the general process for the analysis of bulk black powder, black powder substitutes, and pyrotechnics (such as fireworks, flash powder, and flares) and the identification of their components. These procedures are suitable for bulk samples which are suspected of being a black powder, black powder substitute, or pyrotechnic. These procedures apply to caseworking personnel conducting work in explosives chemistry analysis.

2 Introduction

Black powder and black powder substitutes are classified as low explosive propellants. Black powder is commonly used as a propellant in muzzle loading firearms, in pyrotechnics, in blasting, and in fuses.

Commercial black powder is composed of potassium nitrate, charcoal, and sulfur in a ratio of approximately 75:15:10 and may be found in several Fg grades. F denotes “fine” and g denotes “glazed.” Fg powders are larger in grain size than FFFFg powders, which are comprised of extremely fine grains. Improvised or homemade black powders contain similar ingredients; however, they may be composed of different ratios and granular morphologies.

Pyrodex and Triple Seven are commercial black powder substitutes manufactured by Hodgdon Powder Company. Pyrodex is composed of potassium nitrate, potassium perchlorate, sodium benzoate, dicyanodiamide, sulfur, and charcoal and is available in various grain sizes and pellet forms. Triple Seven is composed of potassium nitrate, potassium perchlorate, sodium benzoate, dicyanodiamide, charcoal, and 3-nitrobenzoic acid.

Other black powder substitutes include, but are not limited to, Golden Powder, Black Mag, Black Canyon, Clean Shot, Clear Shot, and American Pioneer. Their chemical components may include, but are not limited to, ascorbic acid, charcoal, dicyanodiamide, fructose/sugar, 3-nitrobenzoic acid, potassium nitrate, potassium perchlorate, sodium benzoate, and sulfur.

A pyrotechnic is a mixture of chemical elements and compounds that is capable of a self-contained and self-sustained exothermic chemical reaction, for the production of heat, light, gas, smoke, or sound. Pyrotechnics may include fireworks, flash powders, smoke grenades, thermites, matches, and flares. Pyrotechnics must contain at least one fuel and oxidizer. Oxidizers include, but are not limited to, barium nitrate, iron oxide, potassium chlorate, potassium nitrate, potassium perchlorate, or strontium nitrate. Fuels include, but are not limited to, aluminum, magnesium, magnalium (aluminum/magnesium alloy), sulfur, antimony trisulfide, sugar, or carbon.

Flash powder is generally gray or metallic in color varying from a visibly homogeneous mixture to a granular heterogeneous mixture. Flash powder is typically composed of a strong oxidizer such as potassium chlorate or potassium perchlorate and a metallic fuel such as aluminum. It will produce a bright flash upon initiation with a flame.

A flare or fusee contains a pyrotechnic material designed to produce a single source of intense light, heat, or radiation for certain durations and can be used for signaling, as a source of ignition, or for other purposes. It is generally composed of a match head, striker, and a pyrotechnic material.

Table 1 contains example compositions of black powder, black powder substitutes, and pyrotechnics.

Table 1: Example compositions of black powders, black powder substitutes, and pyrotechnics

Type	Example Composition
Black Powder	KNO ₃ , S, charcoal
Pyrodex	KNO ₃ , S, charcoal, KClO ₄ , sodium benzoate, dicyanodiamide
Triple Seven	KNO ₃ , charcoal, KClO ₄ , sodium benzoate, dicyanodiamide, 3-nitrobenzoic acid
Black Powder Substitutes (multiple formulations)	KNO ₃ , S, ascorbic acid KNO ₃ , KClO ₄ , ascorbic acid KNO ₃ , charcoal KNO ₃ , ascorbic acid
Flash powder	KClO ₃ , KClO ₄ , Al, Mg
Flare or fusee	Match head: Liquid shellac, Sr(NO ₃) ₂ , SiO ₂ , charcoal, KClO ₄ , KClO ₃ , wood flour, marble dust Scratcher: Lacquer, pumice, red phosphorus Main pyrotechnic: Sr(NO ₃) ₂ , S, KClO ₄ , sawdust, paraffin oil, fuel oil, corn starch, stearic acid
Pyrotechnics	Oxidizers: Ba(NO ₃) ₂ , Fe ₂ O ₃ , KClO ₃ , KClO ₄ , KNO ₃ , Sr(NO ₃) ₂ Fuels: Al, Mg, magnalium, S, Sb ₂ S ₃ , C, sugar

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 flame ionization detector (GC/FID)
- Gas chromatograph with mass spectrometer (GC/MS)
- Ion chromatograph (IC)
- Microscope (optical or digital)
- Raman spectrometer with macro compartment or microscope attachment
- Scanning electron microscope with energy dispersive X-ray spectrometer (SEM/EDS)
- Solids probe mass spectrometer/mass spectrometer (MS/MS)
- X-ray diffractometer (XRD)

3.2 Materials

- Aluminum or copper tape
- Autosampler vials and caps
- Disposable plastic syringes
- Forceps
- Kraft paper
- Lighter, torch, or matches
- Mortar and pestle
- Scalpel
- SEM stubs or carbon planchets with liquid adhesive (e.g., Duro-tak), carbon adhesive tabs, or aluminum or copper tape
- Solids probe cups
- Spatula
- Syringe filters (0.2 μm nylon filter)
- Various disposable glassware and plasticware
- XRD sample holders (zero background holder with or without depression)

3.3 Reagents/Solvents/Reference Materials

- Deionized water (18.2 M Ω)
- Hexane (reagent grade)
- Isopropyl alcohol (70% commercial product)
- Methanol (HPLC 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.

- IC Testmixes

4.1 Black Powder or Black Powder Substitute Positive Control

A positive control of black powder or black powder substitute will be prepared in a manner appropriate for the analytical technique being used.

4.2 Fuel Oil Positive Control

The fuel oil positive control is a 500 ppm solution of fuel oil #2 in an appropriate solvent (e.g., methanol, hexane, or carbon disulfide).

4.3 Fuel and Oxidizer Positive Controls

Various fuels and oxidizers, such as those listed in Table 1, can be used as positive controls. They will be prepared in a manner appropriate for the analytical technique being used.

4.4 Additional Positive Controls

Additional positive controls are prepared as necessary in order to identify components of mixed samples (e.g., silicon dioxide, calcium silicate, collagen, dye, wax as present in a match head). They will be prepared in a manner appropriate for the analytical technique being used.

5 Sampling

Refer to the Sampling Procedures 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, and disposable gloves) when examining evidence. This is intended to protect the individual conducting the exam and to prevent contamination of evidence.

Review and understand all safety information contained in Section 11 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 Macroscopic/Microscopic Examination

Perform a macroscopic examination and note the homogeneity, color, and consistency of the unknown material.

When possible, separate the powder material if it contains grains of different sizes, colors, or shapes. It may be necessary to view particles under a microscope to aid in separation.

Examine the material under a microscope and note physical characteristics (e.g., homogeneity, color, grain size, grain shape, perforations, mixture, atypical material). Measure grain length, thickness, and diameter as necessary. Photographs of the material and relevant positive controls may be recorded.

- Commercial black powder is composed of black, irregularly-shaped grains, often with a glazed coating giving the surface a smooth appearance. The mixing of the potassium nitrate, charcoal, and sulfur is so complete that the individual components are not visible through a stereomicroscope. Improvised black powders may vary in appearance.
- Black powder substitutes vary in appearance based on manufacturing processes and formulation. For example, Pyrodex is a heterogeneous granular material composed of gray and white areas.
- Flash powder is generally gray or metallic in color and can vary from a visibly homogeneous mixture to a granular heterogeneous mixture. It is generally composed of clear to translucent crystals, which are the oxidizer(s), and silvery-metallic particles, which are the fuel(s).
- The pyrotechnic composition within flares generally has an off-white powdery appearance. The bulk of the flare consists of large and small translucent crystals, some being very pale yellow in color, interspersed with very small black or dark gray particles and sawdust. (Optional) After a hexane extraction, fibrous material visually consistent with wood may be seen under the microscope.

6.2 (Optional) Thermal Susceptibility Test

If sample size permits, place a small amount (~50 mg) of material on the tip of a spatula and heat with a lighter, torch, or match. Note the burn properties such as flame, smoke, and residue.

- Black powder and black powder substitutes will both burn rapidly with a flash and smoke and leave a residue.
- Flash powders will burn rapidly with a bright flash.
- A flare should produce a self-sustained reaction.
- Other pyrotechnics will burn with various effects including color, sound, heat, and smoke.

6.3 XRD Analysis

If sample size permits, grind a portion of the sample to a fine powder, as necessary, with a mortar and pestle and analyze by XRD.

- See Table 1 to determine example materials that may be identified by XRD.

Flares and pyrotechnics can contain oils and inorganic materials. A solvent (e.g., water, hexane) extraction may be necessary in order to define the inorganic phases properly via XRD. The material may be extracted with deionized water, the water evaporated from the extract, and the remaining solid residue analyzed by XRD.

6.4 SEM/EDS Analysis

If sample size permits, analyze a portion of the sample (bulk or ground) mounted onto an SEM sample holder to determine its elemental composition.

- See Table 1 to determine example elements that may be identified by SEM/EDS.

Solvent extractions may be necessary in order to identify the composition of individual components.

6.5 (Optional) FTIR Analysis

Analyze a portion of the sample (bulk or ground) on the FTIR spectrometer with an ATR or microscope attachment.

- See Table 1 to determine example materials that may be identified by FTIR spectroscopy.

Dried residues from water or methanol extracts of suspected black powder or black powder substitutes may be analyzed to determine the presence of sodium benzoate, dicyanodiamide, potassium perchlorate, and potassium nitrate.

6.6 (Optional) Solids Probe MS/MS Analysis

For samples of suspected Pyrodex, Triple Seven, or some other black powder substitutes, analysis by solids probe MS/MS will determine the presence of benzoate and dicyanodiamide, which will differentiate these compounds from black powder.

Grind or crush a portion of the sample to a fine powder and extract it in approximately 300 μ L of methanol for about an hour in a test tube. Retain a quantity of the methanol as a blank/negative control. Extract a positive control concurrently with the sample. Fill respective probe cups with the unknown methanol extract, the positive control extract, and the methanol blank and allow all the methanol extracts to evaporate. An oven may be used to speed the evaporation process.

Analyze the extracts by solids probe MS/MS (Electron Impact Ionization).

- Dicyanodiamide will generate the 84 (parent ion), 68, and 43 fragment ions.
- Benzoate will generate the 122 (parent ion) and 105 fragment ion.

6.7 (Optional) IC Analysis

Extract a portion of sample in up to 50 mL of deionized water. Retain an equal portion of water as a negative control. Where possible, plastic containers should be used during these procedures to avoid the leaching of ions from glassware. Flush a 0.2 μ m filter mounted on a plastic syringe with deionized water. Flush portions of the negative control and the sample extracts through the prepared syringe filters into autosampler vials. An autosampler vial of unfiltered deionized water will be used as a blank.

Analyze the extracts by ion chromatography.

- See Table 1 to determine example cations and anions that may be identified by IC.

6.8 (Optional) Raman Spectroscopy Analysis

Analyze a portion of the sample (bulk or ground) on the Raman spectrometer in the macro compartment or using the microscope attachment.

- See Table 1 to determine example materials that may be identified by Raman spectroscopy.

Dried residues from water or methanol extracts of suspected black powder or black powder substitutes may be analyzed to determine the presence of sodium benzoate, dicyanodiamide, potassium perchlorate, and potassium nitrate.

6.9 (Optional) GC/MS Analysis in Electron Ionization (EI) Mode

For samples of suspected flare material, a hexane extract may be used to determine the presence of oils and carboxylic acids such as stearic acid.

Prepare a hexane extract of the suspected flare material, a hexane blank, and appropriate positive controls (e.g., 500 ppm fuel oil #2, stearic acid).

Analyze the extracts by GC/MS (EI).

6.10 (Optional) GC/FID Analysis

For samples suspected of containing waxes and oils, a hexane extract may be used to determine their composition.

Prepare a hexane extract of the material, a hexane blank, and appropriate positive controls.

Analyze the extracts by GC/FID.

7 Decision Criteria

7.1 Instrumental Results

The following criteria will be met in order for a qualitative identification to be made. The identity of a material will be confirmed by comparison to a reference or known material, if available. Reference or known materials may be run concurrently with an unknown sample or may be previously analyzed on the instrument under the same parameters. A reference or known material will be analyzed by at least one spectroscopic technique used for comparison to an unknown material. All results should be verified using orthogonal techniques or alternate methods.

When a reference or known material is not available or when only reference data (e.g., from scientific literature, publications, or an instrument library) is used, a material may be reported as “consistent with” a substance.

7.1.1 Chromatography

Peaks should show good chromatographic characteristics, with reasonable peak shape, width, and resolution.

The retention time of the peak of interest should be within $\pm 2\%$ of that for a contemporaneously analyzed reference or known material for gas chromatography and $\pm 5\%$ for liquid chromatography.

The baseline signal-to-noise ratio (SNR) for an analyte should be greater than three to be considered a peak. The signal intensity for an analyte peak should be at least ten times greater than the intensity of any carryover or system peaks which may have been present in analyses just prior to the sample (e.g., blanks or negative controls).

7.1.2 Mass Spectrometry

The mass spectrum of the analyte of interest should compare favorably with that of a contemporaneously analyzed reference or known material.

Characteristic ion plots are reviewed to determine the potential presence of a target analyte. The absence of a primary ion indicates a non-detect.

7.1.3 XRD

The diffraction patterns from the questioned compound should compare favorably to the corresponding reference or known material.

If the unknown material is matched through a library search, a reference or known material may be analyzed for comparison, if available. Tentative identifications may also be confirmed through orthogonal techniques such as FTIR, Raman, SEM/EDS, or GC/MS.

7.1.4 SEM/EDS

Peaks in the EDS spectrum should exhibit a Gaussian peak shape and a minimum SNR of 3:1. The elemental composition of the questioned compound should compare favorably to the corresponding elemental composition of the reference or known material. Elemental assignments made by the software should be verified by the individual conducting the exam.

7.1.5 Other Tests

The results of all tests (e.g., visual inspections, FTIR, Raman, pH) should compare favorably to the corresponding reference or known material.

7.2 Material Identification

Samples less than several milligrams and grains lacking the physical characteristics of commercial black powder or black powder substitutes can be considered consistent with commercial or improvised (homemade) black powder, black powder substitutes, or pyrotechnics unless otherwise noted below.

7.2.1 Black Powder

A material can be identified as black powder if potassium nitrate, sulfur, and a source of carbon (e.g., charcoal) can be confirmed, and if the material is not adulterated with other chemicals. Physical characteristics of black powder (e.g., glazed coating, uniform size, homogeneity), the source of the material (e.g., fuse, firework), and positive burn characteristics will aid in this determination.

7.2.2 Black Powder Substitutes

A material can be identified as a black powder substitute based on the visual characteristics, chemical composition, and positive burn characteristics in comparison to other known black powder substitutes used as reference or known materials. This identification may be limited in scope to where a specific black powder substitute product cannot be named, but the material is identified as a general low explosive black powder substitute.

A material lacking physical characteristics associated with black powder substitutes can still be identified by the presence of specific target compounds (e.g., dicyanodiamide and sodium benzoate) which are indicative of specific black powder substitutes.

7.2.3 Flash Powder

A material can be identified as a flash powder based on the visual characteristics, chemical composition, and positive burn characteristics producing a bright flash of light. The oxidizers and fuels that compose a flash powder should be compared to reference or known materials.

When a material has the visual characteristics and chemical composition of a flash powder but does not produce positive burn characteristics, it should be identified as a fuel/oxidizer mixture. These fuels and oxidizers should be compared to reference or known materials.

7.2.4 Flare

Flares have three distinct sections: the match head, the scratcher, and the main pyrotechnic material.

The match head of a flare can be identified based on the visual characteristics (typically a solid plug of material) and chemical composition. If enough material is present to break off a small

piece of the match head, then positive burn characteristics will aid in the identification. The oxidizers and fuels composing the match head should be compared to reference or known materials when available.

The scratcher of a flare can be identified based on the visual characteristics and chemical composition. The main chemical component in the scratcher is red phosphorus and should be compared to a reference or known material.

The main pyrotechnic material of a flare can be identified based on the visual characteristics (e.g., sawdust is present), chemical composition, and positive burn characteristics. Oxidizers and fuels present within the main pyrotechnic material should be compared to reference or known materials when available.

7.2.5 Pyrotechnics

A material can be identified as a pyrotechnic composition based on the visual characteristics, chemical composition, and positive burn characteristics producing pyrotechnic effects such as color, sound, heat, and/or smoke. The oxidizers and fuels that compose a pyrotechnic composition should be compared to reference or known materials.

When a material has the visual characteristics and chemical composition of a pyrotechnic composition but does not produce positive burn characteristics, it should be identified as a fuel/oxidizer mixture. These fuels and oxidizers should be compared to reference or known materials.

8 Calculations

Not applicable.

9 Measurement Uncertainty

Not applicable.

10 Limitations

The identification of uninitiated black powder, black powder substitutes, or pyrotechnics may be limited by sample size. Several milligrams of uninitiated material are essential to microscopically observe the manufacturing characteristics.

11 Safety

Safety protocols, contained within the FBI Laboratory Safety Manual, will be observed at all times.

Take standard precautions for the handling of all chemicals, reagents, and standards. Take 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 of material (such as a few grams) and properly store larger quantities in approved containers.

As a safety precaution, it should be noted that dark materials pose a hazard when being analyzed by Raman spectroscopy as they may be initiated by the laser. If this technique will be utilized, then the smallest possible sample amount and reduced laser intensities should be used to minimize the risk and avoid initiation.

12 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, Explosives, latest revision.

Explosives Standard Operating Procedures: Chemistry, Federal Bureau of Investigation, Laboratory Division, latest revision.

Instrument Operations Manuals for the specific models and accessories used.

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Rev. #	Issue Date	History
2	10/04/2018	Administrative changes for grammar and clarity. Added Triple Seven components to section 2 and Table 1. Removed testmix components in section 4. Added location-specific PMP references to section 6. Added SAU IOG reference and modified IOSS reference.
3	12/16/2019	Changed line spacing after section 6.7. 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

Explosives Chemistry and Fire Debris Report Writing Guidelines

1 Scope

These procedures set forth guidelines for writing *FBI Laboratory Reports* for explosives chemistry and fire debris examinations and supplements the requirements of the FBI Laboratory Quality Assurance Manual (QAM) and the FBI Laboratory Operations Manual (LOM). These procedures apply to caseworking personnel conducting work in explosives chemistry and fire debris analysis.

2 Introduction

FBI Laboratory Reports issued by examiners conducting casework in explosives chemistry and fire debris analyses are designed to summarize analytical findings during the routine analysis of evidence. Due to the wide variety of requests and evidence received, these procedures only provide general guidelines for report writing. It is not possible to anticipate every type of report that may be written and this document is designed to provide examples of common occurrences. It is acceptable to use other wording as long as the results of the examinations are accurately communicated, a description of the methodology used to reach the results is included, limitations are addressed, and wording is approved during the technical review process by an authorized technical reviewer in the category of testing.

3 Procedures

All *Laboratory Reports* generated by Explosives Chemistry Examiners and Fire Debris Examiners will follow the requirements set forth in the FBI LOM and will contain the “Results of Examination” and “Remarks” sections.

3.1 Results of Examination

The Results of Examination section contains methods, results, opinions, limitations, interpretations, and/or conclusions of forensic examinations conducted by the examiner. Information about trade names or uses of specific compounds will also be stated, as necessary.

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In addition to analysis of substances submitted as physical evidence, information provided in other formats (e.g., written, video, electronic) can also be assessed to provide opinions and interpretations such as potential use in explosives, opinion of viability, and theoretical calculations. References, if used, will be recorded in the examination records.

Limitations of the results, or limitations of the examinations based on the evidence received will be conveyed. This may include interpretative wording to aid the reader in understanding any

significance of the Results of Examination section.

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Limitations may include the following:

- If examinations were limited based on limited specimen amounts, this will be stated.
- If examinations were limited due to the nature of the packaging of the material, this will be stated. This may result in no examinations being performed.
- If examinations were limited by the method used to collect the samples, this will be stated.

3.2 Remarks

The Remarks section will follow the requirements in the FBI LOM.

4 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.

Rev. #	Issue Date	History
0	03/31/2017	Original Issue. Relevant portions copied or moved from the former Explosives Unit <i>Report Writing Guidelines</i> SOP.
1	10/02/2017	Modified title. Updated for clarity including specificity for fire debris. Administrative changes for grammar, clarity, and conformance to revised QAM and LOM. Removed references to the Explosives Unit to applicability to those conducting explosives chemistry and fire debris examinations. In Appendix A, added techniques used to heavy petroleum distillate example.
2	10/04/2018	Revised technical reviewer requirements. Added second paragraph in section 3.1 regarding analysis of information in other formats.

Approval

Redacted - Signatures on File

Explosives
Unit Chief

Date: 10/03/2018

Scientific Analysis
Unit Chief

Date: 10/03/2018

TL Approval

Explosives Chemistry
Technical Leader

Date: 10/03/2018

Fire Debris
Technical Leader

Date: 10/03/2018

QA Approval

Quality Manager

Date: 10/03/2018

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