Technical Procedure for the Examination of Explosives Evidence

1.0 Purpose – This technical procedure shall be followed for the examination of explosives evidence.

2.0 Scope – This procedure applies to explosives samples and device components including pre- and post-blast low explosives, pre-blast high explosives, and chemical reaction mixtures. In addition, this procedure may be followed for the identification of unknown materials utilizing the included testing techniques.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- Oven
- Gas Chromatograph-Flame Ionization Detector (GC-FID)
- Gas Chromatograph-Mass Spectrometer (GC-MS)
- FT-IR
- SEM/EDX
- XRF
- Stereomicroscope
- Polarized light microscope
- Camera

4.2 Materials

- Pasteur Pipettes
- Screw top glass vials
- Auto sampler vials, 100 µL inserts, and crimp seals
- Vial crimper
- Rubber septa
- 2.5 mL airtight syringe or 3 mL disposable hypodermic syringe
- Tweezers
- Spatula
- Sieves of varying sizes
- pH test strips
- Lighter
- White spot well
- Black spot well
- Syringeless filters
- Glass microscope slides
- Glass rods
- Probe

4.3 Reagents

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- Carbon disulfide - Reagent A.C.S. grade
- Petroleum ether – Optima Grade
- Acetone
- De-ionized water
- Acetic acid
- Ammonium hydroxide
- Cropen reagent
- Methylene blue
- Anthrone spot test
- Silver nitrate spot test
- Barium chloride spot test
- Diphenylamine spot test

5.0 Procedure

5.1 Analytical Approach

5.1.1 Review the request for analysis.

5.1.2 Open the evidence container and describe any liquid, material, or type of debris present, noting any distinctive odors. Determine if the evidence is in a pre-blast or post-blast state.

5.1.3 Locate and identify fragments and/or components of the device (pipe/container, blasting cap, electric matches, leg wires, wrappers, fuses, timing devices, batteries, etc.)

5.1.4 Remove and isolate intact explosives.

5.1.5 Perform a microscopic examination using a stereomicroscope or polarized light microscope.

5.1.5.1 For intact/unconsumed particles, record the following if applicable:

5.1.5.1.1 Morphology (planed lumps, rough spheres, ball, flattened ball, disk, tube, perforated or not perforated, etc.)

5.1.5.1.2 Homogeneity (crystalline inclusions, metal flakes, saw dust, etc.)

5.1.5.1.3 Approximate size of particle(s)

5.1.5.1.4 Presence of any identification markers (color markers in some smokeless powders such as red flakes, blue flakes, green flakes)

5.1.5.2 For consumed particles and residues characterize the type of residue present (ex. White crystalline residue present)

5.1.6 If intact particles are located and are of sufficient quantity to allow a flame test, perform and document the ignition characteristics.

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5.1.7 Characterize or identify the particles and/or residues utilizing the analytical techniques in ***Section 5.2.***

5.2 Analytical Techniques

5.2.1 Solvent Extraction

5.2.1.1 Purpose – An extraction procedure to separate mixtures or isolate particulate matter not readily visible.

5.2.1.2 Procedure

5.2.1.2.1 Select a solvent. For most explosives identifications acetone and water extractions may be performed.

5.2.1.2.2 Wash the debris with the solvent and collect.

5.2.1.2.3 The extract may be filtered with a syringeless filter, if necessary based on training and experience.

5.2.1.2.4 Proceed with additional analytical techniques. Aqueous extracts may be analyzed by spots tests or microcrystalline tests. Organic extracts may be analyzed by GC-MS. Organic and aqueous extracts may be evaporated to dryness and examined by spot tests, flame tests, microcrystalline tests, IR, and SEM/EDS.

5.2.2 Diphenylamine Spot Test

5.2.2.1 Purpose – A color test to indicate the presence of oxidizing ions such as nitrates, nitrites, chlorates, and ferric ions.

5.2.2.2 Procedure

5.2.2.2.1 Place the diphenylamine color test solution in a white spot plate. The solution should be clear and colorless.

5.2.2.2.2 Add a sample of the unknown to the solution.

5.2.2.2.3 Document any color formation.

5.2.2.2.4 The formation of a blue color is a positive indication for the presence of an oxidizing salt.

5.2.2.3 Standards and Controls – The spot test solution shall be tested with a known nitrate with each case in which the test is utilized.

5.2.3 Nessler's Reagent spot test

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5.2.3.1 **Purpose** – A color test to indicate the presence of ammonium ions.

5.2.3.2 **Procedure**

5.2.3.2.1 Place the Nessler's Reagent color test solution in a white spot plate. The solution should be clear with a yellow tint.

5.2.3.2.2 Add a sample of the unknown to the solution.

5.2.3.2.3 Note any color and precipitate formation.

5.2.3.2.4 An orange/brown precipitate formation indicates the presence of ammonium ions.

5.2.3.3 **Standards and Controls** – The spot test solution shall be tested with a known ammonium containing compound with each case in which the test is utilized.

5.2.4 Barium Chloride Spot Test

5.2.4.1 **Purpose** – A precipitation test to indicate the presence of sulfate ions.

5.2.4.2 **Procedure**

5.2.4.2.1 Place the barium chloride spot test solution in a black spot plate. The solution should be clear and colorless.

5.2.4.2.2 Add a sample of the unknown to the solution.

5.2.4.2.3 Note any color and precipitate formation.

5.2.4.2.4 A white precipitate formation indicates the presence of sulfate ions. It should be noted that carbonates will also precipitate out with this spot test. However, the addition of concentrated acetic acid will re-dissolve the carbonate precipitate.

5.2.4.3 **Standards and Control**s – The spot test solution shall be tested with a known sulfate with each case in which the test is utilized.

5.2.5 Silver Nitrate Spot Test

5.2.5.1 **Purpose** – A precipitation test to indicate the presence of chloride ions.

5.2.5.2 **Procedure**

5.2.5.2.1 Place the silver nitrate spot test solution in a black spot plate. The solution should be clear and colorless.

5.2.5.2.2 Add a sample of the unknown to the solution.

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5.2.5.2.3 Note any color and precipitate formation.

5.2.5.2.4 A white precipitate formation indicates the presence of chloride ions. It should be noted that sulfates and other halides may also precipitate out with this spot test. However, the addition of concentrated ammonium hydroxide will re-dissolve the halide precipitates. Halides other than chlorides will form precipitates that vary from off-white to yellow in color.

5.2.5.3 **Standards and Controls** – The spot test solution shall be tested with a known chloride with each case in which the test is utilized.

5.2.6 Anthrone Spot Test

5.2.6.1 **Purpose** – A color test to indicate the presence of carbohydrates including sugars and starches.

5.2.6.2 Procedure

5.2.6.2.1 Place several crystals of anthrone in a white spot plate.

5.2.6.2.2 Add a sample of the unknown and mix.

5.2.6.2.3 Add two to three drops of concentrated sulfuric acid.

5.2.6.2.4 Note any color formation.

5.2.6.2.5 A blue-green color formation indicates the presence of carbohydrates.

5.2.6.3 **Standards and Controls** – The spot test solution shall be tested with a known sugar with each case in which the test is utilized.

5.2.7 Cropen Microcrystalline Test

5.2.7.1 **Purpose** - A microcrystalline test to indicate the presence of chlorates and perchlorates.

5.2.7.2 Procedure

5.2.7.2.1 Place a droplet of the Cropen reagent and a droplet of the methylene blue solution next to each other, but not touching on a microscope slide mounted on a polarizing light microscope (PLM).

5.2.7.2.2 Add a sample of the unknown to the Cropen reagent.

5.2.7.2.3 Use a glass rod or probe to draw the methylene blue solution to the Cropen reagent.

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5.2.7.2.4 Examine the interface area and note any crystal formation.

5.2.7.2.4.1 Perchlorate Crystals - Blue needles, some with a purplish tinge, grow both singly and in bundles, and may develop blunt or split ends.

5.2.7.2.4.2 Chlorate Crystals - Mainly blue rosettes, some with a slight purple tinge. Chlorate crystals are smaller and the needles thinner than the perchlorate form. They develop at a slower rate than perchlorate. Some single crystals may form.

5.2.7.3 **Standards and Controls** – The spot test solution shall be tested with a known chlorate or perchlorate with each case in which the test is utilized.

5.2.8 Samples may be analyzed by the following Trace Evidence Section instrumental procedures:

5.2.8.1 Technical Procedure for Scanning Electron Microscope/Energy Dispersive X-Ray System (SEM/EDX) for non-GSR Casework

5.2.8.2 Technical Procedure for Gas Chromatography

5.2.8.3 Technical Procedure for Gas Chromatography–Mass Spectrometry

5.2.8.4 Technical Procedure for Infrared Spectroscopy

5.2.8.5 Technical Procedure for X-ray Fluorescence Spectrometer

5.2.9 Test for Acids and Bases

5.2.9.1 **Purpose** – A test to determine the pH of a liquid.

5.2.9.2 Procedure

5.2.9.2.1 Place a droplet of the unknown liquid on full range pH test paper.

5.2.9.2.2 Observe the color change while the pH paper is still wet and compare to the scale included with the pH paper.

5.2.9.2.3 Record the reading from the scale comparison.

5.2.9.2.4 This procedure may be repeated using a narrow range pH paper to further classify the acidity or basicity of the liquid.

5.2.9.3 Conclusions

5.2.9.3.1 If the pH is less than 7, the solution is acidic.

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5.2.9.3.2 If the pH is greater than 7, the solution is basic.

5.2.9.3.3 A pH equal to 7 is neutral.

5.2.9.3.4 Strong acids will have a pH of 0.

5.2.9.3.5 Strong bases will have a pH of 13 or greater.

5.3 Criteria for Explosives Identification

5.3.1 The material in question shall be characterized both physically and chemically.

5.3.2 For intact low explosives, physical characteristics such as general appearance, color, morphology, presence of markers, homogeneity, presence of catalysts such as metal, particle size and ignition susceptibility shall be documented.

5.3.3 For intact high explosives, physical characteristics such as general appearance, color, morphology, presence of markers, prills or microballoons, consistency, homogeneity, presence of catalysts such as metal, particle size, and ignition susceptibility shall be documented.

5.3.4 Chemical Characterization shall be performed and documented by two or more of the following methods:

- PLM
- Spot tests
- pH
- FTIR
- SEM/EDS
- GC-FID or GC-MS
- XRF

5.3.4.1 The number of methods for chemical characterization will vary depending on the type of explosive encountered and amount of sample present.

5.3.5 If the physical and/or chemical characteristics do not correspond to the formulations of known explosives, then no identification shall be made. See Appendix 1 for commonly encountered explosive mixtures and post-blast residues.

5.4 Guidelines for Explosives Analysis Result Statements

5.4.1 Explosive Materials prior to deflagration/detonation (Intact)

5.4.1.1 Positive

5.4.1.1.1 In most cases an identification of the explosive material shall be determined in conjunction with a known standard.

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5.4.1.1.2 Example: Examination of Item A revealed the presence of Composition C-4.

5.4.1.2 Negative

5.4.1.2.1 Example: Examination of Item A failed to reveal the presence of explosives.

5.4.1.3 Statements regarding functionality may also be included.

5.4.2 Explosive Residues (Post-Blast)

5.4.2.1 Positive

5.4.2.1.1 Conclusions may include explosive materials, common solid combustion products, and non-explosive components. Statements of functionality may also be included.

5.4.2.1.2 Example: Examination of Item #1 revealed the presence of a damaged cardboard tube, burned safety fuse, and a gray powder residue. The gray powder residue contained aluminum, sulfur, potassium sulfate, and aluminum sulfate, which is consistent with the presence of post-combustion flash powder. These materials are consistent with having come from a post-blast improvised explosive device.

5.4.2.2 Negative

5.4.2.2.1 Example: Examination of Item #1 failed to reveal the presence of explosives.

5.4.3 Hoax Devices

5.4.3.1 Example: Examination of Item #1 revealed the presence of a metal pipe nipple, two end caps, and a union with electrical wires attached. This item is not an explosive device. However, it is consistent in appearance to an explosive device.

5.5 Standards and Controls

5.5.1 If the sample is extracted and analyzed via a liquid injection technique (GC-FID, GC-MS, CE, etc.), then the solvent used for extraction shall be analyzed via the same technique.

5.5.2 The resulting data from the blanks shall have no peaks that interfere with the analysis.

5.6 Calibrations – No additional calibrations or performance checks are required. See associated technical procedures for instrumental performance checks.

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5.7 **Maintenance** – No additional maintenance is required. See associated technical procedures for instrumental maintenance.

5.8 **Sampling and Sample Selection**

5.8.1 No sampling is performed. When sample selection occurs, it shall be based on the Forensic Scientist's training and experience.

5.8.2 Items of evidence shall be analyzed starting with evidence collected from the blast seat and then moving on to evidence that is collected further away. Once identification can be rendered as to the explosive utilized and any device components, analysis may be concluded and a laboratory report generated.

5.9 **Calculation** – N/A

5.10 **Uncertainty of Measurement** – N/A

6.0 Limitations – N/A

7.0 Safety

7.1 Post-blast debris may have sharp edges that may cut skin. Use caution when handling post-blast debris.

7.2 Glass pipettes are sharp and can be dangerous.

7.3 Diphenylamine contains sulfuric acid which may cause severe skin burns. Avoid skin contact with sulfuric acid.

7.4 Care shall be exercised when using the above-listed items and procedures.

8.0 References

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8.2 **Books**

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8.3 Journal Articles

Hoffman, C.M. and E.M. Byall. "Identification of Explosive Residues in Bomb Scene Investigations." *Journal of Forensic Sciences* 19.1 (1974): 54-63.

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9.0 Records – N/A

10.0 Attachments

- Appendix 1 – Commonly Encountered Explosive Mixtures and Post-Blast Residues.

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document
02/01/2013	2	5.1.5.1 was modified to read "For intact/unconsumed particles, record the following if applicable:"
10/18/2013	3	Added issuing authority to header
08/29/2014	4	Updated header to Physical Evidence Section – Trace Unit, issuing authority to Physical Evidence Section Forensic Scientist Manager

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APPENDIX 1

COMMONLY ENOUNTERED LOW EXPLOSIVES	PRE-COMBUSTION / INTACT FORMULA	COMMON SOLID COMBUSTION PRODUCTS & RESIDUES (Traces of original starting material may survive the burn/blast.)
BLACK POWDER	KNO3 Charcoal Sulfur	K2SO4, K2CO3, S, K2S, KHSO4, KSCN, KNO3, KHCO3, KNO2, K2S2O3, C, & associated ions
BLACK POWDER SUBSTITUTES – PYRODEX	KNO3 KClO4 Charcoal Sulfur Cyanoguanidine Sodium Benzoate	K2SO4, KCl, K2CO3, S, K2S, KSCN, KHSO4, KNO3, KHCO3, KNO2, K2S2O3, C & associated ions
BLACK POWDER SUBSTITUTES – 777	KNO3 KClO4 Charcoal Cyanoguanidine Sodium Benzoate	KCl, K2CO3, KNO3, KHCO3, KNO2, C & associated ions
SMOKELESS POWDERS – SINGLE BASED	Nitrocellulose (NC)	NC (K2SO4, KNO2 WEAK)
SMOKELESS POWDERS – DOUBLE BASED	Nitrocellulose (NC) Nitroglycerine (NG)	NG, NC, (K2SO4, KNO2 WEAK)
FLASH POWDERS	KClO3 or KClO4 Aluminum Sulfur	KCl, Al, K2SO4, KClO3 or KClO4, Al2O3, KHSO4, KHSO3, AlCl3, Al2SO4, KAl(SO4)2 & associated ions
CHLORATE / SUGAR MIXTURES	NaClO3 Sucrose	NaCl, NaClO3, Sucrose & Associated ions
ACID / FOIL BOMBS	Strong Acid Aluminum	Al2O3, Al, AlCl3, & presence of residual strong acid

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