2.2.1 Introduction

The purpose of this examination is to observe, document and preserve. The information derived from this examination could be beneficial to the submitting agency to provide direction to an investigation.
The procedures can be entered at any point if certain areas can be skipped because the knowledge/skills/experience of the examiner allow observations of characteristic blast effects and remnants of explosive powder to dictate most likely type of explosive used. These observations and any deviation(s) from the procedures should be documented.

2.2.2 Safety Considerations

See Safety Considerations 2.1.2

2.2.3 Equipment / Instrumentation

Magnifier Lamp
Probes
Forceps
Sample containers (microscope slides, spot test plates, dropper bottles/reagent containers)
Acetone - Reagent Grade
Carbon Tetrachloride - Reagent Grade
Methylene Chloride - Reagent Grade
Methyl Alcohol – Reagent Grade
Water - Distilled/Deionized
Beakers of various sizes
Watch glasses
Disposable glass pipette
Spatula
Ring stand and clamps
Funnels or filter funnels
Sterile cotton, filter paper or other filtering substrate
Hot Plate
Analytical Balance
Stereomicroscope
Polarized Light Microscope

2.2.4 Micro/Macro Procedure for Analysis

If sufficient fragments from the improvised explosive device (IED) are submitted, consider fingerprints and/or Biology/DNA analyses. Use precautions to avoid destroying these types of evidence. Note the type of materials present (ie. cardboard, plastic, paper, pipe, metal, etc). Note any paint or markings on the fragments. This can include letters, numbers, designs, or color coded marks. Any markings on the device or fragments of the device should be photographed and researched in an attempt to determine manufacturer information that could be useful for the investigator.

Physical characteristics of metal pipe fragments can be examined in an attempt to help determine the type of low explosive powder used. Note the size and shape of the fragments, appearance of the broken edges.

- Small fragments can indicate higher explosive force
- Curved or curled shapes
- Fissures in fragments
- Reverse slant fractures
- Spiked teeth edge or thinned edges
- Pitted
- Presence of screw threads

IED fragments and debris should be searched utilizing a stereomicroscope and/or microscope for burned residue and unburned particles which can help determine the type of explosive employed. The threaded areas of pipes and end caps can contain unburned powder even if the device has functioned. Any residue or powder/particles suspected to be from the explosive mixture should be separated if possible; the physical characteristics of the powder/residue should be recorded; and then collected for further analysis.

2.2.5 Solvent Extraction Procedures

Place sample of powder/particles in glass funnel with filter paper or glass pipet with cotton and add the following solvents in the order listed below, collecting and drying each separately in individual watch glass or other glassware:

1. Methylene Chloride
2. Acetone
3. Methanol
4. Deionized/Distilled Water
5. The **insoluble material** remaining on/in the filter may be analyzed for elemental composition.

Note: All solvent extractions are not required for every type of low explosive. See EXP-PM 2.3 for which solvents are commonly used for which low explosive types. The examiner can use their knowledge, training and experience to determine which extractions are necessary for the type of low explosives encountered.

2.2.5.1 Analysis of Extracted Residues

Use appropriate instrumentation and/or techniques to further characterize the residue (see 2.3 and 2.4). These may include:

- SEM-EDS or u-XRF if sample size permits
- GC-MS
- FTIR

- IC
- Microscopy/Microcrystalline tests

2.2.6 Spot Tests

2.2.6.1 Spot Test Preparations

Chemical spot tests are used as a screening test for compounds and/or ions that are associated with explosives or explosive residues. It is recommended spot tests can be performed in a fume hood to minimize exposure. Spot testing can be done on microscope slides, in well plates or other similar type containers. Care should be taken to minimize exposure during microscopic observations.

Recommended Preparations

Recipes can be modified but should maintain original component ratios.

Aniline Sulfate

Option 1: A 5% w/v aqueous solution of aniline sulfate (5g aniline sulfate in 100ml water)

Option 2: 0.1 grams of aniline sulfate in 100 ml concentrated sulfuric acid

Antazoline

Reagent A - 80% H_2SO_4 (80ml H_2SO_4 + 20 ml H_2O)

Reagent B - 2 grams Antazoline Hydrochloride in 100 militers of H_2O

Barium Chloride ($BaCl_2$)
5 g. Barium Chloride in 100 milliliters distilled water

Diphenylamine (DPA)

<u>One</u> of the following three recipes can be used:

0.25 g of DPA in 80% H_2SO_4 (80 ml H_2SO_4+ 20 ml H_2O)

1 g of DPA in 100ml concentrated H_2SO_4

.30 g of DPA + 20ml H_2SO_4 + 10 ml glacial acetic acid

Greiss

Reagent A - Dissolve 1g alpha-naphthylamine in 230 milliliters boiling distilled water. Cool and decant the colorless supernatant liquid and mix with 100 ml glacial acetic acid.

Reagent B - 1g sulfanilic acid in 100 milliliters of 30% acetic acid.
Powdered zinc

KOH/Ethanol

5g of KOH in 100 milliliters Ethanol

LeFitte

Analine:H2O ratio of 1:40
Concentrated HCl

Methylene Blue

Reagent A – Dissolve 5 grams of zinc sulfate and 4 grams of potassium nitrate in 20 ml of water

Reagent B – Dissolve 0.03 grams of methylene blue in 100 ml of water

Methylene Chloride – Reagent Grade

1- Naphthol Solution

3 g of 1-naphthol in 20 milliliters ethanol

Pyridine - Reagent grade

Silver Nitrate ($AgNO_3$)

5 g. Silver Nitrate in 100 milliliters distilled water

2N Sodium Hydroxide

4 g of NaOH in 50 milliliters of distilled water

Triphenylselenium Chloride

0.5 gram triphenylselenium chloride mixed with 5 milliliters deionized water

2.2.6.2 Spot Test Procedure

Spot tests are performed directly on portions or representative samples of the evidence, blanks and controls for color observations.

Note the following presumptive spot tests and recommended parameters. Other tests described in the references and other scientific literature may be used following the same procedures for standards and controls.

Reagents must be tested with a blank and control each day they are utilized on case samples.

The following spot tests are presumptive tests only. These tests are not confirmatory tests.

Aniline Sulfate
Option 1: A drop of the reagent solution is added to the sample, followed by 2-3 drops of concentrated sulfuric acid. The presence of a blue color or blue ring indicates the presence of chlorate.

Option 2: If chlorate is present, a positive reaction is the presence of an orange color with the addition of just the reagent solution to the sample – no additional sulfuric acid necessary.

Antazoline
Add 1-2 drops of Antazoline Hydrochloride solution. Add 1-2 drops of H_2SO_4 (80%). The formation of a deep red color indicates the presence of nitrates.

Barium chloride
Add 2 ml of deionized or distilled water and dissolve material. Add two drops of barium chloride reagent. A heavy white precipitate indicates the presence of a sulfate or carbonate.

DPA
For recipes 1 or 2, cautiously add drops of DPA reagent on the sample.

If using recipe 3 (DPA/ ethanol solution), add 1-2 drops of DPA solution. Let dry and add 1 drop of H_2SO_4.

Dark blue color indicates the presence of an oxidizer.

Griess
Add reagent A and an equal amount of reagent B. A pink/red color indicates nitrites. If no reaction occurs, add a small amount of powdered zinc. A pink/red color indicates nitrates.

KOH/Ethanol
Add 2 drops of KOH reagent. A deep red color indicates TNT.

LeFitte
Place one drop of analine/H_2O solution and one drop of conc. HCl on a slide. Add questioned sample dry. A color reaction going from yellow to green then blue will occur if chlorates are present.

Methylene blue
Add 2 drops of solution A and 1 drop of solution B. A purple color precipitate indicates the presence of perchlorate.

1-Napthol
Add 1 drop of 1-Naphthol solution and 2 drops of concentrated H_2SO_4 to the liquid or solid sample. A blue to purple color indicates the presence of sugar.

Pyridine/NaOH
Add approximately 1 ml of pyridine in a hood and warm pyridine to dissolve the material. Add 2 drops of 2N sodium hydroxide. A range of colors from blue to dark green indicates the presence of sulfur. Color depends upon concentration.

Silver nitrate
Add 2 ml of deionized or distilled water and dissolve material. Add two drops of silver nitrate reagent. A heavy white precipitate indicates the presence of a chloride.

Triphenylselenium Chloride
Place unknown in a black spot plate and add 1-2 drops of reagent. A white opalescent precipitate indicates the presence of perchlorate.

2.2.6.3 Spot Test References

Anger, V., and Feigl, F., Spot Tests in Inorganic Analysis, ed.6., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972

Jungreis, Ervin, Spot Tests Analysis, John Wiley and Sons, Inc.:New York, New York, 1985

Parker, R.G., Stephenson, M.O., McOwen, J.M., and Cherolis, J.A., “Analysis of Explosives and Explosive Residues. Part I: Chemical Tests”, Journal of Forensic Sciences, Vol. 20, No. 1, January 1975, pp. 133-140.

2.2.7 Flame Test (Ignition Test)

2.2.7.1 Introduction

The purpose of the ignition test is to determine how the suspected explosive powder or unconsumed fuse behaves when ignited.

2.2.7.2 Safety Considerations

Perform in a fume hood. Only use a small amount of material and do not perform near other flammable materials.

2.2.7.3 Preparations

Open Flame
Safety glasses

2.2.7.4 Analytical Procedures

For suspected explosive powder, a small portion of the sample may be held over an open flame using a spatula or placed in a glass container and then ignited.

Alternatively, some of the sample may be placed on a Kimwipe then ignited in a glass container.

If trace amounts of sample are present, the examiner may elect to not conduct this test.

For an unconsumed fuse, a small portion of the fuse may be held in a flame with forceps or ignited in a glass container.

If very small lengths of fuse are present, the examiner may elect to not conduct this test.

The reaction of the sample when exposed to an open flame is noted and recorded.

2.2.7.5 Minimum Standards and Controls

It is recommended that the examiner become familiar with flame test results of various explosive and pyrotechnic powders. The examiner should have observed ignition tests of a variety of known explosive powders (black powder, black powder substitutes and smokeless powders) and fuses prior to conducting flame tests on the case sample(s).

2.2.7.6 References

Yallop, H.J., Explosion Investigation, The Forensic Science Society & Scottish Academic Press, Harrogate, England and Edinburgh, Scotland, 1980

Yinon, Jehuda and Zitrin, Shimuel, Modern Methods & Applications In Analysis of Explosives, John Wiley & Sons, Inc.: NY, NY, 1993

2.2.8 Microcrystal Tests Procedure

2.2.8.1 Water Recrystallization

Microcrystal tests are performed directly on portions or representative samples of the evidence, blanks and controls for observations on a microscope slide or other appropriate container. The following is a microscope technique whereby water-soluble inorganic explosive compounds are characterized and can be identified by their crystal shape as they recrystallize from a drop of water on a microscope slide. Once characterized, they can be identified by their optical properties: refractive index, birefringence and extinction angles, and/or by other micro-chemical tests.

To isolate a single particle, physically remove a particle by using fine forceps, needles, or fine tipped glass rods under a stereomicroscope. If the particle of powder is heterogeneous, it may be possible to separate out individual homogenous particles and perform microcrystal tests on them individually.

To dissolve the particle transfer it to a micro droplet of DI water on a clean slide and crush/stir the particle with a glass rod or other micro-tool until it dissolves or disperses. Try not to spread out the micro droplet. Observe the submerged crystals that form (edges have deformed crystals and therefore are not reliable). Any crust that develops at the edge of the drop should be pushed into the center (BE CAREFUL not to spread the drop). Continue as above until large crystals form while the water evaporates.

Note the following microcrystal tests and recommended parameters. Other tests described in the references and other scientific literature may be used following the same procedures for standards and controls.

Reagents must be tested with a blank and control each day case samples are run.

2.2.8.1.1 Perchlorate Compounds

Ammonium Perchlorate (NH_4ClO_4):

Push the crystals that form at the edge, into the center of the droplet.

Two prism views of the crystal soon form.

These are orthorhombic crystals, they normally do not form on a point or develop into perfect crystals.

Potassium Perchlorate ($KClO_4$):

Only slightly soluble in water forming small well-formed crystals shaped like short rods and tablets.

The distinctive habit of KClO4 differentiates it from its isomorphous partner NH4ClO4.

When the crystal is parallel to the c axis both alpha and beta prime have a negative sign of elongation. When the crystal is parallel to the b axis is has a positive sign of elongation.

Sodium Perchlorate ($NaClO_4$•H2O):

Since the hydrate of sodium perchlorate is very soluble it is necessary to have a greater proportion of the solute to the solvent to be assured that the solute will form in large enough crystals before the droplet goes to dryness.

The monoclinic crystal has two views: the most distinctive is the diamond shaped tablet lying on pinacoid face.

The other view will be the same diamond shaped tablet only that it has developed and is lying on its pinacoid face. Both monoclinic crystals have low to moderate retardation colors.

2.2.8.1.2 Nitrate Compounds

Barium Nitrate ($Ba(NO_3)_2$):

This compound is only slightly soluble in water and crystals form at the edge of the droplet almost immediately. Push these crystals into the center of the drop.

Cube and octahedral crystals as well as any combination of these forms plus distortions appear soon.

Octahedral faces have 60° and 120° angles; those of the cube faces have 45° and 90° angles.

These crystals are isotropic and show high relief in water.

Potassium Nitrate (KNO_3):

This compound is very soluble.

The first to form is the high temperature polymorph which is hombohedra; and consists mainly of ill-formed rhombohedra as "chevrons" or "L's" at the very edge of the drop.

As the droplet approaches dryness, crystals of the stable low temperature polymorph appear as well-formed and usually nearly equal combinations of prisms, pinacoid, and bipyramid forms.

These crystals have high-order retardation colors.

Ammonium Nitrate (NH_4NO_3):

This compound is very soluble; a greater ratio of solute to solvent is best here to help in recrystallization.

With care thin rods, blades, tablets and prisms can be obtained.

With ammonium nitrate it is necessary to keep the droplet from spreading and to push the crystals back into the center of the droplet.

As with sodium perchlorate, these crystals appear as the drop nears dryness and develop rapidly making it difficult to measure the profile angles.

These crystals have high to very high-order retardation colors and parallel extinction.

Ammonium nitrate is very deliquescent, slight humidity and slight warming will help develop the crystals. As crystallization continues there is a strong tendency to form long rods. elongated parallel to the b axis.

Sodium Nitrate ($NaNO_3$):

Sodium nitrate is only slightly less soluble than ammonium nitrate. Be sure to add sufficient quantity of the solute to the drop before it becomes too small and flat. Push the edge crust into the center of the drop.

Well-formed rhombohedra will form having very high order retardation colors, and symmetrical extinction. A significant change in relief is noted upon rotation of the stage.

Lead Nitrate ($Pb(NO_3)_2$):

This compound recrystallizes as well-formed octahedra with profile angles of 60° and 120°, but it may also show cube- octahedron combination with 45° and 90° profile angles.

These cubic crystals have very high relief in aqueous solution.

2.2.8.1.3 Chlorate Compounds

Sodium Chlorate ($NaClO_3$):

This compound is very soluble.

Once the cubic crystals start forming the droplet is filled with masses of squares, rectangles, thin rods, well-formed and flattened cubes.

The low refractive index yields a moderate relief.

Potassium Chlorate ($KClO_3$):

An aggregate of diamond-shaped crystals will form as the drop evaporates. Push these back into the center of the droplet.

Tablets lying on their pinacoid face form in the drop.

As crystallization continues other crystals develop, prism faces truncating the acute angles on the pinacoid view.

It shows symmetrical extinction and medium retardation.

2.2.8.2 Microcrystalline Tests for Cation and Anion Characterization

Sodium (Na+) and Lithium (Li+):

Dissolve the unknown in a droplet of water and let it go to dryness.

In a droplet near but not touching the dried residue dissolve a few grains of zinc acetate and uranyl acetate in water that has been acidified with acetic acid.

Draw the reagent drop across the dried unknown; octahedra of the triple acetate appear if sodium and lithium is present.

Potassium (K+):

Dissolve the unknown in a droplet of water.

Place a drop of chloroplatinic acid near the unknown droplet and draw the two droplets together with the drawn-out glass rod tip.

Highly refractive isotropic orange octahedral indicate the presence of potassium.

NOTE: Ammonium ions form isomorphous crystals with chloroplatinic acid and must be resolved for by the method following.

Ammonium (NH_4+):

A small droplet of dilute chloroplatinic acid is placed at the center of a coverslip, the sample to be tested is placed on a microscope slide and encircled with a length of glass tubing (8mm x 12mm I.D. is suggested).

Add a little dilute NaOH to the substance on the slide and cover the glass ring immediately with the inverted coverslip bearing the chloroplatinic acid.

If ammonium is present yellow octahedral appear in the hanging droplet; slight warming of the slide may speed the reaction by liberating the ammonium from the chloroplatinic acid.

NOTE: To be sure that both K+ and NH4+ are not presence, test the residue from the evaporation of NH3 with a droplet of dilute ammonium perchlorate. An immediate precipitate of KClO4 proves K+ is also present.

Lead (Pb++):

Dissolve a little of the unknown crystal in water. Add a very tiny crystal of potassium iodide to a droplet of water near the unknown droplet, and draw them together with the drawn tip of the glass rod.

Bright yellow hexagonal plates will form, often showing thin-film interference colors.

NOTE: Excess KI will convert these hexagons of Pb+2 to white needles of double salt, Pb+2KI•2H20.

Barium (Ba++):

Dissolve a little of the unknown in water slightly acidify with nitric acid; place a saturated drop of aqueous squaric acid on the slide near the unknown.

Allow the two drops to run into each other, barium forms blades and parallelopipeds either singly or in rosettes.

Nitrate (NO_3-):

TEST #1: Dissolve a crystal of nitron in a drop of dilute acetic acid on a slide near a drop of the unknown.

Draw the drops together and at once sheaves of long, very slender needles and imperfect radiates develop if nitrates are present.

TEST #2: Dissolve a crystal of sulfanilic acid and a crystal of an alpha-naphthylamine in a drop of dilute acetic acid on a microscope slide (or a white spot plate).

Add the unknown substance and a minute amount of zinc dust. A deep red color is produced if nitrate is present.

Chlorate/Perchlorate (ClO_3- or ClO_4-):

Reduce the chlorate ion with hydroxylamine sulfate to the Cl^- ion.

Dissolve a small crystal of AgNO3 in a drop of water near the droplet containing the Cl^- ion.

Draw the droplets together and immediately a very fine precipitate of AgCl will form. (These crystals are so fine)

Take the precipitate to dryness and add dilute NH4OH to the dried residue.

AgCl will form an amine complex with the ammonia but AgCl crystal will slowly develop as the ammonia evaporates to yield highly refractive, isotropic cubes and octahedral.

Differentiation of chlorates: Use K^+ to test for $ClO4^-$ and Ag+ to test for Cl^- followed by the above procedure to reduce the $ClO3^+$ to Cl^- and test for it by Ag^+. There are no reported insoluble chlorates.

Sulfur:

Extract the sulfur from the sample with reagent grade methylene chloride (or chloroform). Allow a portion of this extract to evaporate on a microscope slide and observe the crystals present. Crystals that form can range from: dendritic crystals, elongated crystals terminated with points and pointed tablets, well-formed bipyramids and/or circular crystals and droplets. The liquid drops can be touched with a tungsten needle which will cause them to crystallize.

2.2.9 Macro/Micro Examination References

Bender, E. and Klees G. “The Analysis and Investigative Value of Pipe, Pipe Fittings and Explosive Fillers in Improvised Explosive Devices”; Midwestern Association of Forensic Scientists Meeting Workshop Binder/PowerPoint, September 2004, Orlando, Florida

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