2.3.1 Introduction

The following procedures are designed for cases when unburned low explosive powder(s) is present. The analyst can use their discretion as to when the amount of powder present warrants measuring its weight. If the device has functioned or is fragmented, all pieces should be examined for unburned powder/residue.

The examiner can use their knowledge, training and experience to determine the best or preferred technique for sampling. Techniques to identify the components of the unburned powders include spot tests, microcrystal tests and instrumental techniques. Samples can be taken directly from the unburned particles or from residues from washes with appropriate solvents. In some instances (see below), it may be necessary to use certain wash residues to identify certain components of the unburned powder/particles.

Unburned low explosive powders that have a heterogeneous composition can be picked apart under the stereomicroscope to separate the component.

If multiple types of powders are suspected in one IED, an attempt to identify each type should be undertaken, utilizing the procedures below.

2.3.2 Safety Considerations

See Safety Considerations, section 2.1.2

Some explosives are shock and friction sensitive. Use small samples, avoid sparks with metal objects, process in fume hood with sash lowered if necessary, and use appropriate personal protective equipment.

2.3.3 Equipment and Instrumentation

See Equipment and Instrumentation, section 2.2.3

2.3.4 Minimum Standards & Controls

Blanks should be processed with the same procedure(s) used for the samples.

Note: If no visible residue is obtained in any extraction, no further analysis is required.

2.3.5 Suspected Black Powder

Physical characteristics see section 2.1.3

Total weight of powder present (if warranted)

Flame test (if sample permits)

Perform methylene chloride extraction to isolate Sulfur, see section 2.2.5. #1

Perform water extraction to isolate the oxidizer, see section 2.2.5. #4

Identify the oxidizer (KNO_3 and/or $NaNO_3$) by FTIR and/or IC *and* one of the following: SEM-EDS or XRF

Identify the fuel (Sulfur) by SEM-EDS, GC-MS or XRF (microcrystalline test can also be used if desired, see section 2.2.8.2 Microcrystalline Tests for Cation and Anion Characterization, Sulfur)

2.3.6 Suspected Pyrodex, Triple Seven or Other Black Powder Substitute

Physical Characteristics, see section 2.1.4

Total weight of powder present (if warranted)

Flame test (if sample permits)

Perform methylene chloride (expected residue sulfur), acetone (expected residue cyanoguanidine or ascorbic acid, etc.), methanol (expected residue sodium benzoate and/or sodium 3-nitrobenzoate) and water extractions (oxidizers), see section 2.2.5

Identify the oxidizer by FTIR and/or IC *and* one of the following two: SEM-EDS or XRF

Identify sulfur (if present) by SEM-EDS, GC/MS or XRF (spot test can also be used if desired)

Identify preservatives/antioxidants (methanol and acetone extracts) by FTIR and/or GC/MS.

- Pyrodex contains sulfur whereas Triple Seven does not

Identify ascorbic acid fuel (if present) by FTIR

2.3.7 Flash Powder (Pyrotechnic Powder)

Physical characteristics (note condition of metal flake if present – flakes verses powder), see section 2.1.5

Total weight of powder present (if warranted)

Flame test (if sample permits, results like color, flitter effect, smoke or salute can aid in identification of components)

Perform methylene chloride extraction to isolate Sulfur, see section 2.2.5. #1

Perform water extraction to isolate the oxidizer, see section 2.2.5. #4

Identify the oxidizer(s) by FTIR and/or IC *and* one of the following: SEM-EDS or XRF

Identify metal fuel (i.e., aluminum, magnesium) or other insoluble materials (if present) by SEM-EDS or XRF (microcrystalline can also be used if desired), see section 2.2.5. #5

Examine filler material (if present) by microscopy (if warranted)

2.3.8 Smokeless Powder

Physical characteristics, see section 2.1.6

Total weight of powder present (if warranted)

Flame test (if sample permits)

Perform acetone or methyl alcohol extraction to isolate nitrocellulose and/or nitroglycerine, see section 2.2.5. #1 or #2

Identify nitrocellulose by FTIR

Characterize as single or double base by GC-MS, see 2.1.6 Smokeless Powder Composition

Further characterize if necessary (for comparison to suspect known samples - i.e., identify preservatives/antioxidants by GC-MS)

If a mixture of smokeless powders is present each type should be identified and whether it is single or double base.

2.3.9 Slurries, Emulsions, Water-gels, Blasting Agents

Physical characteristics, see section 2.1.7

Total weight (if warranted)

Identify oxidizer by FTIR and/or IC *and* one of the following: SEM-EDS or XRF

Identify fuel(s):

- The petroleum distillate, nitro-methane and high weight molecular weight oil or wax by GC-MS
- Wax alternately by FTIR
- Aluminum by SEM-EDS or u-XRF

Identify sensitizer by appropriate instrumental technique

- microballoons with microscopy

2.3.10 Other Low Explosives

The examiner can use their knowledge, training and experience to determine which extractions are necessary for homemade, pyrotechnic and other low explosives mixtures encountered which are not covered above.

2.3.11 References

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