Washington State Patrol
Crime Laboratory Division

Materials Analysis
EXPLOSIVES TRAINING MANUAL

May 2018

Contents

1 INTRODUCTION

1.1 PURPOSE AND SCOPE

1. This manual contains an outline for training and/or assessing a forensic scientist in the area of explosives analysis. Each scientist will have a unique training program depending on the individual's strengths and weaknesses, previous background, the needs of the laboratory, and available personnel to provide the training. The sequence in which the various sections are presented should not necessarily be considered as a mandatory order of training.
2. This manual endeavors to promote and maintain consistency and quality among forensic scientists performing explosives analyses across the Crime Laboratory Division. Certain inherent aspects of chemical analysis prohibit the establishment of a rigid set of standard procedures to cover every case. Sufficient latitude should be given to allow for independent thought and individual freedom in selecting alternative courses of action. Upon completion of this training program, the trainee will be thoroughly familiar with the options available to perform an examination of most types of explosives evidence that may be received.

1.2 ORGANIZATION OF THE TRAINING MANUAL

The training manual consists of several study segments, each covering different aspects of explosives analysis.

Each study segment is comprised of five parts:

- The *Objectives* summarize the purpose of each training segment.
- The *Overview* designates topics to be included in the training segment.
- The *Safety* section indicates specific safety information relating to the training segment.
- The *Suggested Readings* section lists the reference material that should be read to successfully complete the study segments. The reading assignments are cumulative; comprehension of prior readings may be required to successfully complete study/discussion questions and exercises of subsequent study segments. It may not be necessary or practical to read every reference listed. The trainee will work with the trainer for specifics.
- The *Exercises* are designed to provide the trainee first-hand experience with the main concepts of each study segment. Data or written explanation for each Exercise must be maintained in the training notebooks.

Module 1 covers principles and techniques used in the analysis of commonly encountered explosives:

- Explosives analysis overview
- Color and microcrystalline tests for explosives
- Recrystallization of some common explosives
- Optical properties
- Infrared analysis (IR) of explosives and explosive reaction products
- Scanning electron microscopy/elemental dispersive x-ray (SEM/EDX) of explosives and explosive reaction products
- Micro x-ray fluorescence spectroscopy (µ-XRF) of explosives and explosive reaction products
- Gas chromatography/mass spectrometry (GCMS) of explosives and explosive reaction products
- Capillary electrophoresis (CE) of explosives and explosive reaction products
- Thin layer chromatography (TLC) of explosives and explosive reaction products
- Analytical approach, significance and report writing

Module 2 covers examination of different types of explosives:

- Black powder
- Black powder substitutes
- Pyrotechnics: black powder and effects
- Flash Powder
- Sparklers
- Fusees/flares
- Smokeless powders
- Military explosives

Competency testing will follow Module 2. Supervised casework for Module 2 may begin following successful completion of all competency testing and upon approval from the Quality Manager. An IOC will be written by the trainer upon the successful completion of training recommending the trainee for supervised casework. Once the IOC has been approved through the chain of command, the trainee will be allowed to perform supervised casework for explosives analysis. Following successful supervised casework, an IOC will be routed through the chain of command to the Laboratory Manager recommending independent casework for explosives analysis. A copy of the IOC will be provided to SAS. The instructor is responsible for ensuring that the trainee is prepared to testify as an expert witness.

2 EXPLOSIVES ANALYSIS OVERVIEW

2.1 OBJECTIVES

- To familiarize the trainee with the various types of explosives and their identification.
- To familiarize the trainee with safety concerns surrounding explosives.

2.2 OVERVIEW

An explosive is any chemical compound or mixture (energetic material) whose purpose is to function by explosion (a rapid release of stored energy). Explosive analysis ranges from the examination of simple inorganic materials to highly complex mixtures of inorganic and organic materials. Explosive devices may range from a cardboard tube filled with black powder to a sophisticated device controlled by electronic circuitry and filled with high explosives such as composition-4 (C4), Research Development Explosive (RDX, cyclonite, hexagen) or other materials, and any combination in between.

Explosive analysis entails the identification of the explosives after it has been removed from an explosive device by experienced explosive ordnance experts, as well as the examination of bomb fragments and debris recovered from the scene of an explosion.

Often the explosives analyst will be asked to examine bomb fragments and explosion site debris to not only attempt to identify the explosives used, but to examine the fragments and other debris for the presence of wires, circuitry, batteries and any other material that will allow us to provide information on the nature of the device used. This information may be useful to provide investigative leads.

Other times the explosives analyst may be asked to examine materials to determine if they were incendiary type devices used to initiate a fire or cause damage to a specific object. An example would be the use of thermite to damage a car or gain entry to a metal container.

It should be noted there are many chemical compounds that can be added together to achieve explosive results. Many compounds may not be thought of as an explosive ingredient but they may be used as such. This could be seen as it pertains to the peroxide based explosives that are commonly seen in other parts of the world but are not as common in the United States. This trend could change so the explosives examiner must stay abreast of current journal articles, internet resources and communication with bomb squads.

There is an ever expanding area of do-it-yourself explosives such as dry-ice bombs, pool cleaner devices, over-pressure devices or brake fluid devices which are not discussed here but can still be analyzed using approaches and techniques learned in this training.

2.2.1 Definitions

Binary Explosive – two substances which are not explosive until mixed together.

Blasting Cap – a metal tube containing a primary high explosive capable of initiating most explosives.

Brisance – shattering capacity of a high explosive, primarily determined by detonation pressure.

Booster – a cap-sensitive high explosive used to initiate other, less sensitive high explosives.

Cannon Fuse (Hobby Fuse) – a coated, thread-wrapped cord commonly filled with black powder designed to initiate flame-sensitive explosives.

Combustion – any type of exothermic oxidation reactions (e.g., burning, deflagration, detonation)

Decomposition – the splitting of a material/compound into two or more substances.

Deflagration – a chemical decomposition slower than the speed of sound (subsonic combustion) where the combustion is propagated through heat transfer; burning material heats the next layer of cold material and ignites it.

Detonation – a chemical decomposition greater than the speed of sound (supersonic combustion) where the combustion drives a shock wave propagating directly in front of it.

Detonation Cord (Det Cord) – a plastic/fiber-wrapped cord containing a core of PETN or RDX.

Detonator – a device used for detonating many types of high explosives.

Explosion – a rapid release in energy and increase in volume usually with the generation of high temperatures and the release of gases.

Explosive – a reactive substance that contains a great amount of potential energy that can produce an explosion if released suddenly, often accompanied by light, heat, sound and/or pressure.

Incendiary – a substance capable of producing intense heat.

Fuel – any substance capable of reacting with oxygen or oxidizers.

Munitions (Ordnance) – any and all military explosives.

Oxidizer – a chemical compound that supplies oxygen to a chemical reaction.

Plastic Explosive (Plastique, Putty Explosive) – a soft, moldable solid explosive that is especially suited for demolition.

Propellant – a substance used in the production of energy that is used to create movement and often directed through a nozzle to produce thrust.

Report – A loud sound produced by an explosion.

Shaped Charge – an explosive device which is designed to direct or focus explosive energy into a narrow jet, increasing the heat and energy on the target area.

Shrapnel – objects which are attached to the outside or included inside a device to increase the blast damage and/or injure or kill personnel. The device/container walls themselves can also function in this manner.

Shock Tube – a non-electric explosive initiator that consists of a small-diameter plastic tube containing a tiny dusting of explosive powder (commonly HMX/aluminum). It functions by transporting an initiating signal by shock wave and is safer to handle and store than det cord.

Shock Wave (Shock Front) – a type of propagating/spreading disturbance that carries energy through a medium.

2.2.1.1 Classification of Explosives by Sensitivity:

Primary Explosives – an explosive that is extremely sensitive to impact, friction, heat, static, electricity and/or electromagnetic radiation. They required a small amount of energy to initiate and are often used in detonators for less sensitive explosives.

Secondary Explosives – less sensitive than primary explosives and require more energy to be initiated. More widely used than primary explosives because they are safer to handle and store.

Tertiary Explosives – an explosive that is very shock insensitive and requires the use of an explosive booster for secondary explosive in addition to the primary explosive.

2.2.1.2 Classification of Explosives by Velocity:

High Explosives – compounds where the rate of decomposition is greater than the speed of sounds and generate a supersonic shock wave/front (detonate).

Low Explosives – compounds where the rate of decomposition is less than the speed of sound and generate a subsonic flame front (deflagrate).

2.3 SUGGESTED READINGS

2.3.1 Introduction

Davis TL. **The Chemistry of Powder and Explosives**. San Pedro (CA): GSG & Associates. Reprint of original copyrighted in 1943. Chapters 1, 2, 3, and 6.

Bailey A, Murray SG. **Explosives, Propellants and Pyrotechnics.** London (UK): Brassey's U.K. Ltd. 1989. Chapters 1, 4, and 5.

Brown GI. **The Big Bang a History of Explosives**. Great Britain: Sutton Publishing. 1998. Chapters 1, 2, 8, 10, 11, and appendices I and II.

2.3.2 Scene

U.S. Department of Justice. **A Guide for Explosion and Bombing Scene Investigation**. Washington DC: NIJ. 2000. Scan.

2.3.3 Intact Explosives and Identification

Pickett M. **Explosives Identification Guide**. Albany (NY): Delmar Publishers. 1999. Scan (good for general information).

Cook JR. **The Chemistry and Characteristics of Explosive Material**. New York: Vantage Press. 2001. Chapters 1, 2, and glossary.

Yinin J, Zitrin S. **Modern Methods and Applications in Analysis of Explosives**. New York: John Wiley and Sons. 1993. Chapters 1.1.4, 1.1.6-8, 1.3, 2, 3, and 4.

2.3.4 Chemistry

Cooper PW, Kurowski SR. **Introduction to the Technology of Explosives**. New York: Wiley-VCH. 1996. Chapters 1 and 2.

2.3.5 Pyrotechnics and Chemistry

Conkling JA. **Chemistry of Pyrotechnics Basic Principles and Theory**. New York: Marcel Dekker, Inc. 1985. Chapters 1, 2, 3, 7, and 8.

Russell MS. **The Chemistry of Fireworks**. Cambridge (UK): Royal Society of Chemistry RS•C. 2000. All.

Saxon K. **The Original “Poor Man’s James Bond” (Volumes 1 and 2)**. Eldorado (AR): Desert Publications. 1991, 1992. Scan.

McLean D. **Medicine Chest Explosives, An Investigator’s Guide to Chemicals Used in Home-Cooked Bombs**. Boulder (CO): Paladin Press. 1995. Scan.

Uncle Fester. **Home Workshop Explosives**. Green Bay (WI): Festering Publications. 1990. Scan.

2.3.6 Law

Chapter 296-52 WAC. **Washington Standards for Possession, Handling and Use of Explosives**.

Chapter 70.74 RCW. **Washington State Explosives Act**.

Department of the Treasury, Bureau of Alcohol, Tobacco and Firearms, ATF P 5400.7 (09/12). **Federal Explosives Law and Regulations**. https://www.atf.gov/explosives/docs/publication-federal-explosives-laws-and-regulations-atf-p-54007

2.4 EXERCISES

Find out who your local bomb squad personnel are and establish a rapport.

3 COLOR AND MICROCRYSTALLINE TESTS FOR EXPLOSIVES

3.1 OBJECTIVES

To familiarize the trainee with color tests and microcrystalline tests used for the identification of explosives.

3.2 OVERVIEW

This chapter does not contain all the color and crystal tests that are available for use in explosives analysis. This chapter lists the most common tests used by chemists to identify explosives and explosive reaction products.

Color tests are often instantaneous and can be observed with the naked eye. However, they are non-specific and cannot be used to identify compounds, unless otherwise stated.

Microcrystal tests are simple, highly sensitive and rapid to use. These tests are specific and can conclusively identify inorganic compounds, both anions and cations. All of the tests have to be observed using a polarized light microscope.

When performing these tests always place the reagent on a clean slide, or in the bowl of a clean spot plate, THEN add the unknown/test sample. This ensures there are no contaminants on the slide or the spot plate bowl. If the sample is dissolved in a solvent, the analyst must first perform a procedural blank with the solvent and all reagents involved in the test to ensure the solvent is not contaminated.

3.2.1 Color Test Techniques

1. Solid sample (direct crystal)
    a. Place reagent on a clean slide, or in the bowl of a spot plate, THEN add the unknown/test sample.
2. Liquid sample
    a. First test the solvent with the test reagent(s) to ensure the solvent is not contaminated. If the test requires multiple reagents, the analyst must run them in the same sequence as the actual test to ensure accuracy of the test.
    b. Perform test with the sample by placing reagent on a clean slide, or in the bowl of a spot plate, THEN add the unknown/test sample.
3. Hanging microdrop
   This method is based on the volatility of the sample and is useful as it tends to purify the sample during the process.
    Method one
        1. Place sample on a slide with a well.
        2. Add a drop of base to the sample.
        3. Place a drop of acid on a coverslip, invert over the well, and allow to sit for 10-30 minutes.
        4. Remove the coverslip and place on a slide with the precipitating reagent.
        5. Crystal formation is observed.
    Method two
        1. Place sample on a slide with a well.
        2. Add a drop of base to the sample.
        3. Place a drop of precipitating reagent on a coverslip and invert over the sample in the well.

4. Crystals are observed in the hanging drop.

3.2.2 Color Tests

3.2.2.1 DIPHENYLAMINE TEST for chlorates, nitrates, and nitrated compounds:

Reagent:

- 2.5gm of diphenylamine in 100 mL ethanol

Test:

1. Place 1-2 drops of the diphenylamine solution in a spot plate well.
2. Add a small amount of unknown/test sample.
3. A blue color is a positive indication of nitrates, nitrated compounds and chlorates.

(Jungreis, p 98)

3.2.2.2 ANILINE SULFATE TEST for chlorates:

Reagent:

- 10 gm of aniline sulfate in 100mL deionized (DI) water
- Concentrated sulfuric acid

Test:

1. Place 2 drops of the reagent solution to a test tube.
2. Add a small amount of solid unknown/test sample or 5-10 drops of liquid extract of unknown/test sample to the test tube.
3. Gently tilt the tube to the side and slowly add 2-4 drops of concentrated H_2SO_4, allowing them to run down the side of the tube to make a separate layer.
4. If chlorates are present, a blue ring will form at the interface of the acid and liquids. Nitrates and iodates produce a less distinct reddish-brown ring.

(Merck: Test 3210-Pozzi-Escot Reaction for Chlorate)

3.2.2.3 NAPHTHOL TEST for sugar:

Reagent:

- 15% 1-naphthol in ethanol (1.5 g 1-naphthol in 10mL ethanol)

Test:

1. Place 1 drop of reagent and 2 drops of concentrated H2SO4 to a spot plate well.
2. Add a small amount of solid sample or 1-3 drops of liquid extract of unknown/test sample.
3. If sugar is present, a blue to purple color will appear.

(Jungreis, p 106)

3.2.2.4 PYRIDINE TEST for sulfur:

Reagents:

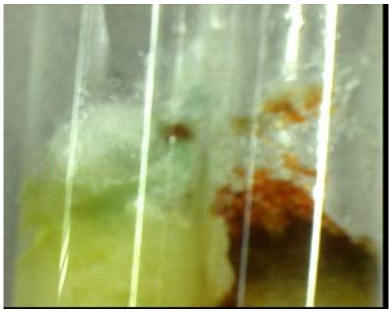
- Pyridine
- 2M Sodium Hydroxide

Test:

1. Place a cotton plug in two small glass pipettes (one for unknown/test sample one for reagent blank).
2. Place two to three drops of 2M sodium hydroxide on each plug.
3. Dissolve a small portion of the unknown/test sample in 2-3 drops of pyridine and wait for two minutes.
4. Add 2-3 drops of pyridine to one of the pipettes with the sodium hydroxide treated cotton plug. Wait one minute and record any color changes.

5. This is the blank. There should be no color changes. If any color develops find a new source of cotton and refill two new pipettes and repeat the above steps.
6. If there was no color reaction in the previous step add two to three drops of the pyridine/unknown solution to the other pipette and record the color changes you see:
7. Green to blue shows a low concentration of sulfur.
8. Red to brown shows a high concentration of sulfur.

(Jungreis, p 107)



3.2.3 Microcrystal Tests

The following are the most commonly used microcrystal tests for inorganic explosives. Unless stated otherwise, results should be observed in one minute or less.
See Appendix A for photomicrographs and expected results.

3.2.3.1 NITRON TEST for Nitrate, Nitrite, Chlorate, Perchlorate, Iodide, Oxalate, Tartrate, and Thiocyanate:

Reagent:

- 1% Nitron in approximately 2N Acetic acid. (0.1 g Nitron in 10mL 2N Acetic Acid)
  -or-
- A drop of 2N Acetic acid is placed on the slide and then a few crystals of Nitron are added to the drop.

Test:

The material in question can be tested directly, in an aqueous solution, or in a solution of 2N Acetic acid. According to the references it is better to use dilute test solutions.

a. METHOD 1: Add sample to a drop of 1% Nitron solution on a glass slide (best for solutions)
   - Remember to test your solvent with the Nitron reagent to ensure there are no contaminants.
b. METHOD 2: Add sample to a drop of 2N Acetic acid on a glass slide after the addition Nitron crystals (best for powders)

(Chamot and Mason, p 326)

3.2.3.2 METHYLENE BLUE TEST for Chlorate and Perchlorate:

Reagents:

- Solution A: 5 g of zinc sulfate and 4 g of potassium nitrate in 20 mL DI water
- Solution B: 0.03 g of Methylene Blue in 100 mL DI water

Test:

1. Place solutions A and B next to each other on a microscope slide.
2. Add a small particle of the unknown/test sample to the solution A drop and allow it to dissolve. Or, if the test material is already in solution, add 1 drop of the test solution to the Solution A drop.
3. Using a glass stir rod or pipette, draw a fine line from the Solution B drop to the Solution A drop.
4. Use the 10X Objective to observe the crystals.
5. If the test solution is too weak, put a drop on the slide and allow it to dry. Then add the Solution A drop to the dried residue and conduct the test in the same manner.

NOTE: The test is more sensitive and faster for perchlorates than chlorates.

(Hopen and Crippin, p 41-45)

3.2.3.3 SILVER NITRATE TEST for Chloride and Sulfate:

Reagents:

- ~5% Silver nitrate in DI water (0.5 g silver nitrate in 10 mL DI water)
- Concentrated ammonium hydroxide

Test:

Large amount of unknown/test sample in aqueous solution:

1. Place a few drops of 5% silver nitrate solution in a test tube.
2. Add the unknown/test sample.
3. Centrifuge and remove the liquid fraction. Retain the liquid fraction for analysis of sulfates (low concentration), nitrates, and acetates.
4. Dissolve the precipitate in concentrated ammonium hydroxide and place on microscope slide. Observe the crystals while the solution is evaporating.

Small amount of unknown/test sample in aqueous solution:

1. Place one drop of 5% silver nitrate solution in a test tube.
2. Add one drop of unknown/ test sample.
3. Use a pipette to remove the clear liquid portion. Reserve the liquid for further testing.
4. Dissolve the precipitate in one drop of concentrated ammonium hydroxide. Observe the crystals while the solution is evaporating.

(Chamot and Mason, p 374, 349)

3.2.3.4 SQUARIC ACID TEST for Potassium, Sodium, Barium, and Strontium:

Reagent:

- Saturated solution of squaric acid in DI water (0.03 g squaric acid in 1 mL DI water)

Test:

1. Place a drop of squaric acid solution on to a clean slide.
2. Add the unknown/test sample (either solid or aqueous solution) to the reagent solution.

NOTE: Can also be used for lithium, calcium, silver, copper, iron and others, however photomicrographs are not found in Appendix A.

(Hollifield, p 81-103)

3.2.3.5 CHLOROPLATINIC ACID TEST for Potassium and Ammonium:

Reagents:

- 5% aqueous solution of chloroplatinic acid (0.5 g chloroplatinic acid in 10 mL DI water)
- 2N sodium hydroxide

Test:

Method 1: Allow a drop of the chloroplatinic acid solution to touch a drop of the unknown/test sample solution.

Method 2: Add a crystal of the unknown/test sample directly to a drop of chloroplatinic acid solution.

NOTE: The potassium and ammonium ions give the same crystals in chloroplatinic acid. If the analyst wants to distinguish between potassium and ammonium the hanging drop method must be used.

1. Place the questioned material in the sample area of a depression or well slide.
2. Add 1 drop of 2N sodium hydroxide to a spot plate well.
3. Place a drop of chloroplatinic acid solution on a microscope slide and invert over the spot plate well.
4. Wait 5 minutes and observe crystal formation.
5. The ammonium ion volatizes and reacts with the chloroplatinic acid in the hanging drop. The potassium ion does not volatize.

(Chamot and Mason, p 65)

3.3 SAFETY

It is important to note that some of the ingredients in microcrystalline reagents pose significant health hazards and before making or using any of these reagents, the appropriate Safety Data Sheet (SDS) should be consulted. All glassware should be disposed of appropriately following analysis.

3.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Microchemical Testing Chapter

McCrone WC, McCrone LB, Delly JG. **Polarized Light Microscopy**. Any edition ok. Chicago (IL): McCrone Research Institute. 1995. p206-207.

Chamot EM, Mason CW. **Handbook of Chemical Microscopy Volume II**. Second Edition Reprint. Chicago (IL): McCrone Research Institute. 1989.

Fiegl F, Anger V. **Spot Tests in Inorganic Analysis**. Sixth Edition (any edition ok). Amsterdam: Elsevier Science B.V. 1972.

Fiegl F. **Spot Tests in Organic Analysis**. Seventh Edition (any edition ok). Amsterdam: Elsevier Science B.V. 1966.

Hollifiend J. **Characterization of Squaric Acid Precipitates**. The Microscope. 2003. Vol 51: p81-103.

Hopen TJ, Crippin JB. **Methylene Blue Microchemical Test for the Detection and Identification of Perchlorates and Chlorates**. The Microscope. 2001. Vol 49(First Quarter).

Jungreis E. **Spot Test Analysis-Clinical, Environmental, Forensic, and Geochemical Applications.** Second Edition. New York: John Wiley & Sons, Inc. 1997.

FBI. **Instrumental Analysis of Explosives and Explosive Residues Training Manual**. Article on Nitron reagent and Article on Test for Potassium.

E. Pozzi-Escot test. Merck's Report for August 1913. Vol 22: p213.

3.5 EXERCISES

The color and crystal test reagents used below may be prepared already, or the analyst may have to prepare them. The analyst may use the photos in this lesson and Appendix A or any other references available to identify these materials.

1. Color Tests

Perform the following color tests and compare your results to the expected ones:

- Diphenylamine: potassium nitrate, potassium chlorate, potassium perchlorate
- Aniline sulfate: potassium nitrate, potassium chlorate, potassium perchlorate
- 1-naphthol: sugar, potassium nitrate, potassium chlorate, potassium perchlorate
- Sulfur: sulfur, potassium nitrate, potassium chlorate, potassium perchlorate, potassium sulfate

2. Crystal Test

Perform all appropriate crystal tests on the following and compare your results to the expected ones:

- Ammonium nitrate
- Ammonium perchlorate
- Potassium nitrate
- Potassium chlorate
- Potassium perchlorate
- Barium nitrate

- Strontium nitrate
- Calcium nitrate
- Sodium chloride
- Ammonium chloride
- Calcium chloride

3. Optional Exercise

If the trainee decides to perform color and/or crystal tests that are not listed in this lesson they must:

a. Cite the reference the test is described in;
b. Provide the formulation(s) for the reagent(s) used in the test;
c. Describe what the test is supposed to detect;
d. the procedure for using the test;
e. Provide drawings of the crystals (photos are better) and a written description.

4 RECRYSTALLIZATION OF SOME COMMON EXPLOSIVES

4.1 OBJECTIVES

- To familiarize the trainee with the crystal structure of some common explosive components after recrystallization from solution.

4.2 OVERVIEW

The explosive analyst is often able to particle pick specific crystals for further analysis by IR, microcrystalline exam, XRF, SEM/EDX, etc. There are times when particle picking isn't sufficient and the analyst will be required to wash the suspected fragments or residues with a solvent (e.g., chloroform, methylene chloride, ethanol, methanol, water). When the solvent dries, the once dissolved materials may form characteristic crystal habits. The crystals can be indicative of a specific substance or used to isolate components for further examination(s). These washes frequently contain mixtures of explosive compounds and/or reaction products.

Recrystallization can be performed on bulk samples with test tubes and watch glasses, or on a small scale using micro-capillary tubes and glass depression slides and observed under PLM. This method can also be used to separate a single crystal into multiple crystals for use in different analyses.

4.2.1 Techniques

1. Saturated solution:
    a. Place 2-3 drops of the solution into the well of a spot plate, or small watch glass, and let it slowly evaporate to dryness.
    b. View crystals under stereoscope and compare to photographs in Appendix B or literature.
2. Powder:
    a. Place 2-3 drops of water into the well of a spot plate, or small watch glass.
    b. Add powder to the water (enough to cover the broad end of a flat toothpick).
    c. Crush with glass stir rod and stir until completely dissolved.
    d. Let solution evaporate to dryness.
    e. View crystals under stereoscope and compare to photographs in Appendix B or literature.

4.3 SAFETY

It is important to note that some of the solvents may pose significant health hazards and before using any of the solvents, the appropriate SDS should be consulted. Appropriate precautions, including use of personal protective equipment and fume hoods, should be utilized.

4.4 SUGGESTED READING

Winchell AN, Winchell H. **The Microscopical Characters of Artificial Inorganic Solid Substances: *Optical Properties of Artificial Minerals***. Third Edition. Chicago (IL): McCrone Research Institute. 1989.

Winchell AN. **The Optical Properties of Organic Compounds**. Second Edition. Chicago (IL) McCrone Research Institute. 1987.

McCrone WC, McCrone LB, Delly JG. **Polarized Light Microscopy**. Any edition ok. Chicago (IL): McCrone Research Institute. 1995. Chapter 8 (All) and p226.

Hopen, Kilbourn. **Characterization and Identification of Water Soluble Explosives**. The Microscope. 1985. Vol 33(1).

Hopen TJ. **Tricks of the Trade: Extraction and Identification of Sulfur**. The Microscope. 2000. Vol 48(1).

4.5 EXERCISES

Using either technique above, or a combination, record your results for the following by writing a brief description of what you see and draw or take pictures of the crystals that form:

- Ammonium nitrate
- Ammonium perchlorate
- Potassium nitrate*
- Potassium chlorate
- Potassium perchlorate*
- Potassium nitrate/potassium perchlorate mixture*
- Sulfur
- Sodium benzoate
- Dicyandiamide
- Dicyandiamide/sodium benzoate mixture
- Sugar/potassium chlorate mixture
- Sugar/potassium perchlorate mixture

*** save for Optical Properties of Explosives Lesson**

5 OPTICAL PROPERTIES OF EXPLOSIVES

5.1 OBJECTIVES

- To familiarize the trainee with using optical properties to analyze inorganic and organic explosives and explosive reaction products.

5.2 OVERVIEW

With experience, determining the optical properties of inorganic compounds is a fast way to confirm the identity of a material. Especially those used in the manufacture of black powder, pyrotechnics and explosives and some of their reaction products.

It is important to be able to recognize if HMX is present in a sample of RDX since it indicates the manufacturing process of the RDX. It is easier to detect HMX in a preparation of RDX if the sample is mounted in an appropriate refractive index medium. A mounting medium refractive index of 1.590 will cause the RDX crystals to almost disappear, where as the HMX crystals will show very strong contrast in nearly all positions. One of the simplest media to use is molten TNT, which has the correct refractive index, is readily available, and, in some cases, is already present in the mixture.

The charts in Appendix C are derived from Winchell's and McCrone and are to be used as a quick reference guide for the materials most likely to be encountered during explosives analysis. Please check other references and do not hesitate to alert the instructor if they appear incorrect.

It is also possible to use polarized light microscopy (PLM) to determine the carbon source in charcoal. This can be especially useful when comparing black powder samples. See Appendix D for photomicrographs of different charcoal types.

5.3 SAFETY

Tools for the manipulation of small particles are sharp and pose cutting and puncture-wound hazards. Care must be exercised in the use of fine tweezers, scalpels, tungsten needles, and other sharp tools. Refractive index immersion liquids may pose health hazards if there is skin/eye contact or ingestion. Strong acids are corrosive on contact with skin.

5.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: Microscopes Chapter

WSP CLD Materials Analysis Secondary Foundation Training Manual: Advanced Microscopy Chapter

Winchell, Winchell. **The Microscopical Characters of Artificial Inorganic Solid Substances: Optical Properties of Artificial Minerals**. Third Edition. MRI. 1989.

McCrone, Andreen, Tsang. **Report On The Microscopic Examination Of High Explosives And Boosters**. Cornell University. August 1944. Section 8.2 Contract Oemsr-193. Declassified 1951.

McCrone WC, McCrone LB, Delly JG. **Polarized Light Microscopy**. Any edition ok. Chicago (IL): McCrone Research Institute. 1995.

5.5 EXERCISES

1. Determine the optical properties of potassium nitrate, potassium perchlorate, and strontium nitrate.

  - Examine the materials straight from the bottle.
  - Examine the materials after being recrystallized, either saved from Recrystallization Lesson or make new by dissolving in a small drop of water and evaporate.

2. Determine the optical properties of potassium nitrate and potassium perchlorate from a recrystallized mixture (use a previously prepared mixture or make a new mixture). Use particle picking to remove the individual crystals for examination.
   Suggestion: This is a good time to use dispersion staining to help rapidly determine the orientation and refractive indices of the crystals.
3. *Examination of charcoal*
   Examine the following types of charcoal using stereomicroscopy and transmitted light/polarized light microscopy (PLM):
   - Burned wood
   - Wood charcoal (two types)
   - Coconut charcoal
   - Animal bone charcoal
   - Charcoal briquette scrapings
   - Carbon black
   - Graphite
   - Oil soot (if available)

6 IR OF EXPLOSIVES AND EXPLOSIVE REACTION PRODUCTS

6.1 OBJECTIVES

- To familiarize the trainee with using IR to analyze inorganic and organic explosives and explosive reaction products.

6.2 OVERVIEW

Infrared Spectroscopy (IR) is a workhorse of identification for explosive materials due to its minimal sample prep of isolated materials, rapid results, and non-destructive nature. Many explosive materials are solid mixtures which need to be separated into individual components before analysis, either by particle picking or by using an extraction scheme. The non-destructive nature of IR makes it especially useful for samples with limited size because a sample can be further analyzed by other techniques such as XRF and/or microcrystalline tests. Because of the prevalence of inorganic materials in both explosives and explosive reaction products, an IR instrument used to examine them should go down to at least 400 cm^{-1}.

Spectral libraries, those made in-house or commercially available, are necessary for identification and comparison of results.

6.3 SAFETY

See IR chapter of WSP CLD Chemical Analysis Technical Procedures.

6.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: IR Chapter

WSP CLD Materials Analysis Primary Foundation Training Manual: FT-IR Chapter

Explosive spectral reference libraries available on instrument

6.5 EXERCISES

1. Perform IR analysis on the samples listed below taking care not to over/under load samples (except as noted). Compare your results to the spectral library of explosives and explosive reaction products. Review results with your instructor.
   Save for printed spectra comparison in Module 2.
   - Pyrodex®
   - Triple se7en®
   - Clean Shot®
   - Clear Shot®
   - Potassium nitrate (weak, medium and heavy loading)
   - Ammonium nitrate
   - Potassium chlorate
   - Nitrocellulose
   - Nitroglycerin
   - Nitroguanidine
   - RDX
   - Prepare a 1:1 solution of potassium nitrate: potassium chlorate in DI water and evaporate down in a watch glass to use as IR samples (analyze fine particles, large crystals near the edge, and crystals from the center)

2. Make sure the compounds listed in Appendix E are searchable with your current IR libraries. Add any that are missing.

7 SEM/EDX OF EXPLOSIVES AND EXPLOSIVE REACTION PRODUCTS

7.1 OBJECTIVES

- To familiarize the trainee with using SEM/EDX to analyze explosives and explosive reaction products.

7.2 OVERVIEW

As a microscope, SEM provides greater resolution, magnification, and depth of field than light microscopy. It is essentially a non-destructive method since the sample can be preserved for later examination, although adhesive and/or coating materials may be added in preparing samples for examination.

As a method of elemental analysis, EDX can quickly identify elements within a sample. A specimen may be analyzed as a bulk sample or as individual particles by spot focusing the electron beam rather than scanning. EDX is unable to detect elements in trace concentrations or below atomic number 12 and some samples need conductive coating before analysis.

7.3 SAFETY

Appropriate safety precautions should be employed when refilling the liquid nitrogen Dewar, if needed. When filling the Dewar, eye level should be above the funnel. Personal protective equipment including safety goggles, face shields, insulating gloves and longs sleeves should be used when handling liquid nitrogen.

Caution should be exhibited when working with vacuum pumps. Waste oil should be treated as hazardous and should be handled and disposed of appropriately.

The SEM/EDX is a high voltage system. After powering off the system, allow any stored energy to discharge prior to performing any maintenance. All maintenance should be undertaken with caution.

7.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: SEM/EDX Chapter

WSP CLD Materials Analysis Primary Foundation Training Manual: SEM/EDX Chapter

Donato F, Paolo M, Scavino G, Ubertalli G, Lenco MG, Pellati G, Piccardo P, Pinasco MR, Stagno E, Costanza G, et al. **Metal Objects Mapping After Small Charge Explosions. A Study on AISI 304Cu Steel with Two Different Grain Sizes**. Journal of Forensic Sciences. Vol 51(3): p520-31.

Kosanke KL, Dujay RC, Kosanke BJ. **Pyrotechnic Reaction Residue Particle Analysis**. Journal of Forensic Sciences. Vol 51(2): p296-302.

7.5 EXERCISES

Properly set up and calibrate the SEM/EDX instrument to perform the exercises which follow. Run all samples at 20 keV unless instructed otherwise.

1. Analyze KNO_3. What elements are seen? Which elements are not?
2. Analyze KCl, $KClO_3$, and $KClO_4$. Can these be differentiated? Why or why not?
3. Analyze a particle of Pyrodex® and a particle of Triple Se7en®. Do these two samples have any elemental similarities? Are there any differences, if so what are they?
4. Prepare and run samples of elemental sulfur, lead carbonate, and sodium molybdate dehydrate.

  a. Do any of these samples appear to have any elemental similarities?
  b. How can you differentiate between sulfur and molybdenum using just the SEM/EDX instrument?
  c. Run elemental analysis of these samples again, but at 30 keV. Are these spectra different? If so, how?

5. Create a mixture of sulfur/sodium molybdate dehydrate mixture and perform elemental analysis.
    a. Does this elemental composition look like anything seem before?
    b. If so, what does this spectrum have similarities to?
    c. If this was an “unknown” how could the presence of sulfur be confirmed or eliminated?
6. Take a small portion of flash powder and place the material on a watch glass. Safely ignite the material with a glowing ember.
    a. Observe the residue using a stereomicroscope. Record observations.
    b. If there are any spheres present, transfer one or more to a stub with carbon tape. Image this particle and perform elemental analysis. What may this be?

8 µ-XRF OF EXPLOSIVES AND EXPLOSIVE REACTION PRODUCTS

8.1 OBJECTIVES

- To familiarize the trainee with using µ-XRF to analyze explosives and explosive reaction products.

8.2 OVERVIEW

X-ray fluorescence (XRF) spectrometry is an analytical method for obtaining information about the elemental composition of a sample. It is predominantly nondestructive in nature allowing further analysis by microcrystal testing on most explosive samples. XRF is especially useful for confirming the identity of monoatomic components or ions in explosive materials and explosive reaction products (e.g., elemental sulfur, potassium ion, chloride ion).

Depending on the specific window material that is used for the XRF detector, the lowest atomic number element that may be detected with this instrument is fluorine (atomic number 9), neon or sodium. The detection limits for the lightest elements depend on whether the analysis occurs in a vacuum or not.

8.3 SAFETY

XRF instruments are surrounded with lead shielding and equipped with safety switches which prevent the X-ray tube from being turned on if one of the access panels is removed.

Certain XRF instruments need liquid nitrogen and appropriate safety precautions should be employed when refilling the liquid nitrogen Dewar, if needed. Personal protective equipment including safety goggles, face shields, insulating gloves and long sleeves should be used when handling liquid nitrogen.

The exit window of the X-ray tube is covered with a thin beryllium window and is highly toxic. If breakage of the window occurs, avoid inhaling the particles and do not allow them to come in contact with skin or clothing.

8.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: µ-XRF Chapter

WSP CLD Materials Analysis Primary Foundation Training Manual: µ-XRF Chapter

8.5 EXERCISES

1. Analyze KNO_3 using the µ-XRF. What elements are seen? Which elements are not?
2. Analyze KCl, $KClO_3$, and $KClO_4$ using the µ-XRF. Can these be differentiated? Why or why not?
3. Compare results from Lead pipe or wire, sodium molybdate and sulfur. Can these be differentiated using µ-XRF?
4. Use the µ-XRF to construct an elemental map of black powder.
5. Analyze a particle of Pyrodex® and a particle of Triple Se7en®. Do these two samples have any elemental similarities? Are there any differences and if so, what are they?
6. Compare samples of $Sr(NO_3)_2$ using diamond anvil cell, mylar film, and adhesive tape sample preparation.

9 GCMS OF EXPLOSIVES AND EXPLOSIVE REACTION PRODUCTS

9.1 OBJECTIVES

- To familiarize the trainee with using GCMS to analyze explosives and explosive reaction products.

9.2 OVERVIEW

GCMS is a well-established technique in analytical organic chemistry and is extremely useful for the analysis of multi-component mixtures, such as identifying the non-base components of smokeless powders. Certain inorganic molecules can also be detected, such as elemental sulfur (S_8).
GCMS analysis of explosives is limited to molecules that can withstand GC conditions. Molecules must be thermally labile but stable enough to not decompose under GC conditions. Highly polar molecules may be difficult to analyze by GCMS because their adsorption to the stationary phase may affect elution. Retention times in the total ion chromatograph (TIC) and mass spectra (MS) from a sample may then be compared to those of reference materials for purposes of identification.

See Appendix F for an instrument method to use as a jumping off point. Individual instrument conditions (e.g., column, carrier gas) will dictate the final method; adjust run parameters as necessary to achieve good separation. It may be necessary to have more than one method for explosives analysis.

The analysis of some explosive compounds (e.g., nitroglycerin, RDX) is sensitive and need a very clean, pristine injector liner to prevent their breakdown in the injection port.

9.3 SAFETY

All systems should be turned off and cooled prior to performing maintenance. Refer to the GC technical procedures for specific safety information regarding the handling of compressed gases.

Scientists should be aware that there are two sources of exhaust on the GC/MSD system: the foreline pump and the GC split vent. The foreline pump outputs gas removed from the vacuum manifold by the high vacuum pumps and the exhaust will contain traces of solvent and sample.

Caution should be exhibited when working with vacuum pumps. Waste oil should be treated as hazardous and should be handled and disposed of appropriately.

9.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: GCMS Chapter

WSP CLD Materials Analysis Primary Foundation Training Manual: GC Chapter and MS Chapter

Yinon J, Zitrin S. **Modern Methods and Applications in Analysis of Explosives**. New York: John Wiley and Sons. 1993.

Beveridge A. **Forensic Investigation of Explosions**. CRC Press, Taylor and Francis Group. 1998.

McLafferty FW, Turecek F. **Interpretation of Mass Spectra**. Fourth Edition. Sausalito(CA): University Science Books. 1993.

9.5 EXERCISES

1. Run the following reference materials using the appropriate explosives method:

Note which substances exhibit good or poor chromatography and the relationship of the mass spectrum to the chemical structure of each analyte.

- Elemental sulfur
- Benzoic acid
- Nitroglycerin
- Organic explosives mixture for smokeless powders (e.g., 2,6-DNT, 2,3-DNT, 2,4-DNT, 3,4-DNT, DEP, DPA, MC, EC, DBP, 2-nDPA, 4-nDPA)
- RDX

2. Run the following reference materials using your standard controlled substance method:
    - Elemental sulfur
    - Benzoic acid
3. Run the following reference materials after silation:
    - Dicyandiamide
    - Benzoate
    - nitrobenzoate

10 CE OF EXPLOSIVES AND EXPLOSIVE REACTION PRODUCTS

10.1 OBJECTIVES

- To familiarize the trainee with using CE to analyze explosives and explosive reaction products.

10.2 OVERVIEW

Capillary electrophoresis (CE) can be used to detect anions, cations or organic molecules, depending on the method, column and reagents used. A significant amount of inorganic anions and cations remain after the deflagration of low explosives. CE can be especially useful for analyzing post blast residues which can be complex mixture of compounds and help determine the nature and source of the explosion.

Aqueous extracts can be used to detect anions or cations; organic extracts can be used in combination with a micelle buffer to detect organic compounds. The low detection limits for inorganic ions (e.g., nitrate, chlorate) make it especially useful for post blast analysis.

See Appendix G for sample run data.

10.3 SAFETY

A high voltage power supply is required to drive the separation in the CE. A safety interlock is in place to shield the user from the high voltage. This safety mechanism should not be tampered with.

Exposure to high powered UV can cause blindness and thermal burns. The interior top cover in the detector chamber should not be opened while the UV lamp is turned on.

The scientist should be aware of any hazards associated with chemicals used as buffers or sample preparation liquids. Appropriate Safety Data Sheets (SDS) should be on file.

10.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: CE Chapter

WSP CLD Materials Analysis Primary Foundation Training Manual: CE Chapter

10.5 EXERCISES

1. Complete the CE portion of primary foundations.

11 TLC OF EXPLOSIVES AND EXPLOSIVE REACTION PRODUCTS

11.1 OBJECTIVES

- To familiarize the trainee with using TLC to analyze explosives and explosive reaction products.

11.2 OVERVIEW

Thin Layer Chromatography (TLC) is a method for separation of chemical mixtures based on differences in component interactions between a stationary phase and a mobile phase. It was once widely used for explosives analysis and is advantageous for being simple, rapid, inexpensive and relatively sensitive. It is important to determine the right separation system (eluting solvent and stationary phase combination) and visualizing agent. TLC of reaction products is often performed on extraction of the debris by acetone, methanol, and/or water, depending on what material is suspected.

11.2.1 TLC Systems for Explosives

11.2.1.1 NITROGLYCERIN SYSTEM

Solvent system

- 3:2 xylene:hexane

Visualization Reagents

- 5% diphenylamine in ethanol
- Concentrated sulfuric acid

Method

1. After plate has developed and dried, spray with 5% diphenylamine solution.
2. Expose plate to short wave UV light for 15 minutes. Record observations.
    - Spots appear olive green
3. Lightly spray with concentrated H_2SO_4. Record observations.
    - Spots turn blue/black with fading

11.2.1.2 C-4 SYSTEM (RDX and HMX)

Solvent System

- 1:1 chloroform:acetone

Visualization Reagents

- 5% diphenylamine in ethanol
- Concentrated sulfuric acid

Method

1. After plate has developed and dried, spray with 5% diphenylamine solution.
2. Expose to long wave UV light for 20-30 minutes. Record observations.
    - RDX appears purple
    - HMX appears grey
3. Lightly spray with concentrated H_2SO_4. Record observations.
    - RDX and HMX spots appear blue

11.3 SAFETY

See TLC chapter of WSP CLD Chemical Analysis Technical Procedures.

11.4 SUGGESTED READING

WSP CLD Chemical Analysis Technical Procedures: TLC Chapter

WSP CLD Materials Analysis Primary Foundation Training Manual: TLC Chapter

Yinon J, Zitrin S. **Modern Methods and Applications in Analysis of Explosives**. New York: John Wiley and Sons. 1993. Chapter 2.1 Thin Layer Chromatography p33-41.

Hiley RW. **Investigations of Thin Layer Chromatography Techniques Used for Forensic Explosives Analysis in the Early 1970s.** Journal of Forensic Science. Vol 38(4): p864-873. 1993.

Taylor, Francis. **Forensic Investigation of Explosions**. Edition by Alexander Beveridge. 1998. p247-249.

11.5 EXERCISES

1. Complete the TLC portion of primary foundations.

NOTE: TLC will be performed on explosive samples during Smokeless Powder and Military Explosives chapters.

12 ANALYTICAL APPROACH, SIGNIFICANCE AND REPORT WRITING

12.1 OBJECTIVES

- To familiarize the trainee with analytical approach, significance of findings and report writing as it pertains to explosives and explosive reaction products such as:
    1. Where do I start with my examination?
    2. What do my results/findings mean?
    3. How do I communicate my results in a report?
    4. Working with an experienced examiner will aid the trainee in finding the answers to these questions. A flow chart which may be useful can be found in Appendix H.

12.2 OVERVIEW

12.2.1 Analytical Approach

In general, good laboratory practices suggest that multiple techniques be employed in any forensic examination and that adequate notes be written along with supporting analytical data which are available for review. The type of analysis may vary and may depend upon if the item is an intact explosive, post blast, or rendered safe item. It is also very important to always remember that other analyses (latent prints, DNA or other trace) may be of forensic interest. Refer to the Trace Evidence Recovery training lesson for specifics.

12.2.2 Intact Material

There are many questions to consider when analyzing intact explosives material. Is the material a commercially available product or homemade? Could it be a low or high explosive? Is it contained in a device such as cardboard or is it a homemade or improvised device? Has this material been seen in the WSP Crime Lab System before? Other important information may be needed if the submitted material had been rendered safe and removed from an improvised device.

Visual and stereomicroscopic examinations are the best way to begin. If there is just bulk powder, record the weight and proceed with examinations as performed in the training plans. If numerous explosive items are submitted, it is up to the analyst to determine the appropriate number of items to examine. Things to consider are how many explosive types and what quantities are involved. If any fusing material is present, it is also important to consider whether it functions as intended.

12.2.3 Post Blast Material

Again, visual and stereomicroscopic examinations are the best way to begin. The residues found on many post blast items include some unconsumed original material, so the goal of the initial examination is to find intact material from the device. Some of the best places to look for intact material are in pipe threads, crevices and on the adhesive side of tape.

For intact or semi-intact devices, record dimensions and sizes of containers, or take pictures. If many pieces are present, try to reconstruct the device to approximate the device size. Note any manufacturer markings on any of the pieces and any alterations, such as a fuse hole.

For analyzing the residue on a device, the analyst has many options such as taking scrapings, performing elemental analysis via SEM/EDX and/or µ-XRF, performing chemical analysis via IR or GCMS, or performing a solvent extraction. Often times, the analyst can perform the analysis on the dried and concentrated residue.

12.2.4 Rendered Safe Items

If an item has been rendered safe, it may be important to find out if it was disrupted and if so, how the item was disrupted. A control sample of the type of disrupter used should be obtained. This may be especially important in cases involving smokeless powder. The weight of the intact material (if available) may also be useful. If it is a mixture of two or more explosives, a sample of each type should be analyzed when practical. There may be an occasion where the analyst will need to separate out each explosive type, weigh them, and proceed with each analysis individually.

12.2.5 Significance

There are no “rules” for the people who create explosives devices to follow. Many submitted items can contain smokeless powder, black powder, pyrotechnic materials and even mixtures of these explosives types. Once the analysis is completed, it is important to interpret the data and form a conclusion. The presence of ammonium nitrate in soil, without a source of fuel, may not be significant. But finding ammonium nitrate (still without a fuel source) in an improvised device with an attached fuse may be significant. The analyst may have to talk to the submitting agency or the bomb squad for more information which may help in drawing conclusions.

12.2.6 Report Writing

The basis of a report is to notify the reader of what was received, what examinations were performed, and the results and conclusions. Information which may be of importance in the report may include the material weight and the device or container size/dimensions.
Your conclusions come from what was received, what analyses were performed, and what was found. A mixture identified as containing potassium nitrate, sulfur and charcoal is referred to as “black powder.” A similar mixture that does not burn, may also be described as a “black powder” formulation but “this material does not function as intended.” Some example reports follow. Guidelines on what analyses are necessary to identify explosives compounds and mixtures can be found in the WSP CLD Materials Analysis Technical Procedures: Explosives Analysis Chapter.

12.2.6.1 Example #1

Overview
The material in Item 6 was examined for the presence of explosives and/or pyrotechnic materials.

Results and Conclusions
The fine gray powder in Item 6 is consistent with a pyrotechnic “flash powder” mixture. If a mixture such as this is confined and ignited, it could act as an explosive. Rice grains and white beans are occasionally used in pyrotechnic mixtures.

Evidence
Item 6: A zip-lock plastic bag containing a mixture of a fine gray powder, rice grains and white beans.

Methods and Observations
Stereomicroscopy, polarized light microscopy, infrared spectroscopy, scanning electron microscopy with energy dispersive X-ray (SEM-EDX) and microcrystalline testing were used to characterize and identify the fine gray powder in Item 6. The rice grains and white beans in the above item were not analyzed.

Analysis of the fine gray powder in Item 6 showed this material to be a mixture of potassium perchlorate and aluminum metal. When a glowing ember is applied to the fine gray powder in Item 6, an ignition with bright sparks is generated.

12.2.6.2 Example #2

Overview

The powder found in Item 1 was analyzed for black powder, or other explosive materials.

Results and Conclusions

The mixture of potassium nitrate, sulfur and charcoal is commonly referred as “black powder” or “gunpowder.” Black powder is used in sporting firearms, commercial and homemade pyrotechnics and explosive devices. If confined and exposed to an ignition source, “black powder” may act as an explosive.

Evidence

Item 1: A sealed plastic bag containing 0.2 gram of black powder.

Methods and Observations

Item 1 was analyzed using stereomicroscopy, polarized light microscopy, infrared spectroscopy, scanning electron microscopy with energy dispersive X-ray (SEM-EDX), gas chromatography-mass spectrometry (GC-MS) and microcrystalline testing.

Analysis of the powder in Item 1 showed the material to be a mixture of potassium nitrate, sulfur and a material consistent with charcoal. When a hot glowing ember is applied to this material, ignition and smoke occurs.

12.2.6.3 Example #3

Overview

The contents of Item 1 were examined for the presence of road flare materials and/or expended road flare materials.

Results and Conclusions

A fusee, or road flare, commonly contains strontium nitrate, potassium nitrate, potassium perchlorate, sulfur, wood and other minor components in various proportions. An expended fusee frequently contains potassium, strontium, sulfate, carbonate and chloride ions, along with the charred wood fragments. The presence of all or some of these ions, in various proportions, is common to expended fusees.

Evidence

Item 1: A zip-lock plastic bag containing a light gray powder with black fragments.

Methods and Observations

Stereomicroscopy, polarized light microscopy, infrared spectroscopy, scanning electron microscopy with energy dispersive X-ray (SEM-EDX), capillary electrophoresis (CE) and microcrystalline testing were used to characterize the light gray powder and black fragments in Item 1.

Analysis of the light gray powder in Item 1 showed that it contained potassium, strontium, sulfate, carbonate and chloride ions. The black fragments are consistent in appearance to burned wood fragments.

Remarks

With current methods and technologies available, a complete analysis of some materials may not be possible. In some samples, there may be components that cannot be detected or identified.

12.3 SAFETY

Standard chemical and laboratory precautions should be considered.

12.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

Minimum of 10 reports by other experienced explosives analysts

12.5 EXERCISES

Listed below are some examples of situations to consider when analyzing evidentiary items. The analyst should look up why these situations may provide issues, record the significance and how the issue(s) can be addressed. These should be discussed your instructor.

1. A device with fuse. Analysis determined the apparent fuse was a fuse “simulator” (does not function as a fuse).
2. A military explosive such as C-4 connected with “hobby” fuse.
3. One particle of smokeless powder among many particles of black powder.
4. A rather small device which was rendered safe that has an equal amount of black powder and smokeless powder particles.
5. A rather large device which was rendered safe that has an equal amount of black powder and smokeless powder particles.
6. A device with no fusing system present.
7. Detonation cord is attached to a device (pipe filled with black powder) via a fuse hole. The detonation cord shows some melting of the fiber jacket.

13 BLACK POWDER

13.1 OBJECTIVES

- To familiarize the trainee with microscopic characteristics of different carbon sources.
- To familiarize the trainee with the analysis of black powder and its reaction products.

13.2 OVERVIEW

Black powder, sometimes known as gunpowder, is the earliest know chemical explosive and contains a mixture of potassium nitrate, sulfur and charcoal. The basic formula has been the same for hundreds of years with most changes and innovations occurring in the manufacturing process. The typical formula for modern black powder is 75% potassium nitrate, 15% charcoal and 10% sulfur. The sulfur and charcoal act as fuels, while the potassium nitrate acts as an oxidizer.

The equation for burning black powder is stated in the book “The Chemistry of Fireworks” by Michael Russell as being:

$$74KNO_3 + 96C + 30S + 16H_2O \longrightarrow \begin{array}{l} 35N_2 + 56CO_2 + 14CO + 3CH_4 + 2H_2S + 4H_2 + 19K_2CO_3 + \\ 7K_2SO_4 + 8K_2S_2O_3 + 2K_2S + 2KSCN + (NH_4)_2CO_3 + C + S \end{array}$$

13.2.1 Analysis of unreacted black powder

1. Perform a burn/ignition test on the material. Record how well the material burned and any characteristic odors, smoke, sputter, color, and/or light. Also note characteristic odors of the burn residue.
2. Observe intact material under the stereomicroscope: record color, size and shape of materials. Record if the material is homogenous. If not, describe the size and shape of the subgroups.
3. Crush a small portion of material and observe under the stereomicroscope.
4. Particle pick the different crystals and perform instrumental and microcrystalline tests, as needed, to identify the components.
5. Wash a portion of the material with chloroform or methylene chloride and analyze for sulfur. Recrystallize if necessary.
6. Wash the residue from the above step with water and let evaporate naturally.
    a. IR crystals
    b. crystal tests, CE and/or elemental analysis (XRF/SEM) for anion and cations
7. Examine the insoluble material (should be mostly charcoal), identify the type of charcoal if possible.

13.2.2 Analysis of burned black powder

NOTE: These may or may not have all of the reaction products that would be expected from the examination of an exploded device. However this will give a close approximation of what would be seen.

1. Burn a sample of black powder on a watch glass
2. Observed under the stereomicroscope
3. Particle pick some of the characteristic spheres that are found. Perform instrumental and microcrystalline testing, as needed, to identify the material.
4. Wash the residue with chloroform/methylene chloride and analyze for sulfur.
5. Wash the residue from the previous step with water, evaporate, and:
    a. Examine with a stereomicroscope for characteristic crystals.
    b. Identify the different crystals using instrumental, microcrystalline tests and PLM.

6. Analyze the insoluble material from the previous step to see if it’s possible to identify the charcoal used in the original material.

13.3 SAFETY

Small quantities of material should be used during a burn/ignition test along with wearing safety glasses or using a blast shield.

13.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

Conkling JA. **Chemistry of Pyrotechnics Basic Principles and Theory**. New York: Marcel Dekker, Inc. 1985.

Cook JR. **The Chemistry and Characteristics of Explosive Material**. New York: Vantage Press. 2001.

Cooper PW, Kurowski SR. **Introduction to the Technology of Explosives**. New York: Wiley-VCH. 1996.

Davis TL. **The Chemistry of Powder and Explosives**. San Pedro (CA): GSG & Associates. Reprint of the original copyrighted in 1943.

McCrone WC, Delly JG. **The Particle Atlas Volumes 2 and 5**. Edition 2. Ann Arbor(MI): Ann Arbor Science Publishers, Inc. 1973. Refer to monographs and photomicrographs for carbon, charcoal, graphite, oil soot. Also available on CD-ROM.

Russell MS. **The Chemistry of Fireworks**. Cambridge (UK): Royal Society of Chemistry RS•C. 2000.

Saxon K. **The Original the Poor Man's James Bond Volume 1**. Eldorado (AR): Desert Publications. 1991.

13.5 EXERCISES

1. Analyze unreacted black powder using all appropriate and available techniques.
    - (save remnants of burn/ignition test)
2. Analyze burned black powder using all appropriate and available techniques.
    - (Or use burn/ignition test from analysis above)

14 BLACK POWDER SUBSTITUTES

14.1 OBJECTIVES

- To familiarize the trainee with the analysis of common black powder substitutes and their reaction products.

14.2 OVERVIEW

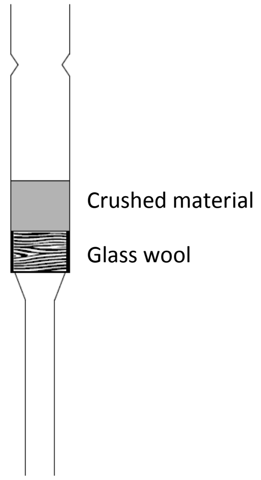
Like black powder, black powder substitutes were developed for hunting or target shooting with black powder firearms but are designed to minimize fouling and corrosion of the weapon.

There are several brands of black powder substitutes available on the market, each with a unique, and widely varying, formulation. Some of the available brands are White Hots ® by IMR, PYRODEX® and Triple Se7en® by Hodgdon Powder Company, Black MZ® and Blue MZ® by Alliant Powder, and Blackhorn 209® by Western Powders Company. CLEAN SHOT® by Clean Shot Technologies, Inc. is now known as American Pioneer® Powder (APP). CLEAR SHOT® and Pinnacle® by GOEX, Black Canyon® by Legend Product Corps., and BLACK MAG® by ARCO haven't been seen on the market for several years.

BLACK POWDER SUBSTITUTE FORMULATIONS	
BRAND	**COMPONENTS**
White Hots ®	Potassium nitrate, potassium perchlorate, sodium benzoate, dextrin
Pyrodex®	Potassium nitrate, potassium perchlorate, sodium benzoate, dicyandiamide, sulfur, carbon
Triple Se7en®	Potassium nitrate, potassium perchlorate, sodium benzoate, 3-nitrobenzoic acid, dicyandiamide, carbon (no sulfur)
Clear Shot®	Potassium nitrate and organic material
Clean Shot®	Potassium nitrate, potassium perchlorate, ascorbic acid, carbon

14.2.1 Analysis of unreacted black powder substitutes

1. Use a stereomicroscope to examine the unreacted material, recording observations. Look for uniform sized composite "spheres/granules" that appear grayish in color with large clear and opaque crystals and smaller crystals that appear bound together.
2. Perform a burn/ignition test of the material. Record how well the material burned and any characteristic odors, smoke, sputter, color, and/or light. Also note characteristic odors of the burn residue.
3. Perform an IR on a portion of the bulk sample, crushed to a uniform consistency.
4. Particle pick as many components as possible and identify them using instrumental, microcrystalline tests, and/or optical properties.
    a. Perform IR, saving crystal on diamond cell for next step.
    b. Perform SEM/EDX or µXRF on the crystal while still on the diamond cell, or other appropriate sample holder.
    c. Scrape the crystal off the diamond cell and perform useful microcrystalline tests. You may also perform PLM to determine optical characteristics.
5. Gently crush a small portion of the material. Place a glass-wool plug in a disposable pipette and put approximately ½-inch layer of material on top of the plug. Perform a series of solvent extractions.



   a. Wash the mixture with chloroform or methylene chloride, retaining the insoluble portion.
      Run extract on GCMS or let the chloroform evaporate. If crystals from the material are likely Pyrodex®, perform IR to determine if any other compounds are present.
         S_8 – diamonds, individual or stacked/layered
   b. Wash the mixture with ethanol or methanol, retain the insoluble portion.
      Run extract on CE or GCMS, or let the liquid evaporate and use particle picking for IR or PLM characterization. This fraction should contain polar organics such as dicyandiaminde, sodium benzoate, and 3-nitrobenzoic acid.
         Dicyandiamide – needles
         Sodium Benzoate – white “halos”
   c. Wash the mixture with water, retaining the insoluble portion.
      Run extract on CE (anions and/or cations), or let evaporate and particle pick for IR, microcrystalline rest, or PLM characterization. This fraction should contain salts such as potassium nitrate and potassium perchlorate.
         $KClO_4$ – large clear, rectangular, glass-like crystals, hexagonal pyramids
         KNO_3 – Blades/long sheaths, grow together, more opaque than KClO4
   d. Examine the remaining insoluble material with a stereoscope and PLM. Try to identify the source of charcoal.

14.2.2 Analysis of reacted black powder substitutes

1. Use a stereomicroscope to examine the material, recording observations. If you see unexploded material, use the method for unreacted black powder substitutes. Look for white spheres, black spheres, white material covered by black film, carbonaceous clumps and a white film.
2. Test as many individual, identifiable components as possible using instrumental, microcrystalline tests, and/or optical properties.
   a. Perform an IR, saving crystal on diamond cell for next step.
   b. Perform SEM/EDX or µXRF on the crystal while still on the diamond cell, or other appropriate sample holder.
   c. Scrape the crystal off the diamond cell and perform useful microcrystalline tests. You may also perform PLM to determine optical characteristics.
3. Wash the sample with water, retaining the insoluble portion.
   Run the extract on CE (anions and/or cations), or let evaporate and analyze by IR, SEM, microcrystalline tests, and/or PLM characterization.

14.3 SAFETY

Small quantities of material should be used during a burn/ignition test along with wearing safety glasses or using a blast shield.

14.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

Conkling JA. **Chemistry of Pyrotechnics Basic Principles and Theory**. New York: Marcel Dekker, Inc. 1985.

Cook JR. **The Chemistry and Characteristics of Explosive Material**. New York: Vantage Press. 2001.

Cooper PW, Kurowski SR. **Introduction to the Technology of Explosives**. New York: Wiley-VCH. 1996.

Davis TL. **The Chemistry of Powder and Explosives**. San Pedro (CA): GSG & Associates. Reprint of the original copyrighted in 1943.

McCrone WC, Delly JG. **The Particle Atlas Volumes 2 and 5**. Edition 2. Ann Arbor(MI): Ann Arbor Science Publishers, Inc. 1973. Refer to monographs and photomicrographs for carbon, charcoal, graphite, oil soot. Also available on CD-ROM.

Russell MS. **The Chemistry of Fireworks**. Cambridge (UK): Royal Society of Chemistry RS•C. 2000.

Saxon K. **The Original the Poor Man's James Bond Volume 1**. Eldorado (AR): Desert Publications. 1991.

14.5 EXERCISES

NOTE: Open air burning of explosive powders may not give all the reaction products that would be expected if the material was detonated in an explosive device. However, there are characteristic reaction products that can be examined and identified which will also be seen in bomb fragments.

1. Analyze unreacted Pyrodex® using all appropriate and available techniques.
   (Save remnants of burn/ignition test)
2. Analyze unreacted Triple Se7en® using all appropriate and available techniques.
   (Save remnants of burn/ignition test)
3. Analyze unreacted Clean Shot® using all appropriate and available techniques.
4. Analyze unreacted Clear Shot® using all appropriate and available techniques.
5. Burn a small portion of Pyrodex® and analyze using all appropriate and available techniques.
   (Or use burn/ignition test from analysis above)
6. Burn a small portion of Triple Se7en® and analyze using all appropriate and available techniques.
   (Or use burn/ignition test from analysis above)
7. Compare your analysis of unreacted and burned Pyrodex®.

15 PYROTECHNICS: BLACK POWDER AND EFFECTS

15.1 OBJECTIVES

- To familiarize the trainee with the analysis of pyrotechnics and their reaction products.

15.2 OVERVIEW

Pyrotechnic mixtures, upon ignition, produce light, color, noise, heat, smoke, and motion. The reactions that occur are highly exothermic oxidation-reduction processes with a number of military and civilian applications, including art and entertainment.

A typical pyrotechnic composition contains an oxidizer, fuel, binder and special effect materials (e.g., color). Considerations for a commercial fireworks mixture are:

- Production of the desired effect (e.g., color, noise)
- Stability in manufacturing and storage
- Low toxicity of ingredients and reaction products
- Reasonable cost

Historically fireworks formulae were a closely guarded secret. With the advent of modern analytical chemistry and instrumentation, it is possible to obtain the component composition. Having the composition, however, does not mean a person can easily reproduce a pyrotechnic effect. The same pyrotechnic formulation shows a great difference in burning behavior depending on particle size and surface area of the components, degree of homogeneity, and loading pressure used to compact the material into a paper tube.

Black powder is used in numerous pyrotechnic devices and is the main-stay of the pyrotechnic industry. It acts as the basic bang and propulsion part of the formulation while all the color effects, smoke, flash and lights are additives to the basic black powder formula.

There are many formulations of effect pyrotechnics available. They are made to sparkle, flash, flash-bang with or without color, burst with sparkles or color, provide colored illuminations and/or smoke. All of the above effects require additives to the basic black powder formulation. Many of the commercial compositions and constructions are protected by major manufacturers.

COMMON PYROTECHNIC INGREDIENTS		
OXIDIZERS		
Potassium nitrate	Potassium chlorate	Potassium perchlorate
Barium nitrate	Barium chlorate	Ammonium perchlorate
Strontium nitrate	Potassium periodate	Sodium periodate
FUELS		
Aluminum	Charcoal	Dextrin
Magnesium	Sulfur	Red gum
Titanium	Antimony sulfide	Polyvinyl chloride
Hexachlorobenzene		
BINDERS		

COMMON PYROTECHNIC INGREDIENTS		
Dextrin	Red gum	Synthetic polymers
FILLERS		
Rice hulls	Rolled grains	

COMMON PYROTECHNIC SPECIAL EFFECT MATERIALS	
Red flame	Strontium nitrate, strontium carbonate
Green flame	Barium nitrate, barium chlorate, boron carbide
Blue flame	Copper carbonate, copper sulfate, copper oxide
Yellow flame	Sodium oxalate, cryolite (Na_3AlF_6)
White flame	Magnesium, aluminum metals
Gold sparks	Iron fillings, charcoal
White sparks	Aluminum, magnesium, aluminum-magnesium alloy, titanium
Whistle effect	Potassium benzoate or sodium salicylate
White smoke	Potassium nitrate/sulfur mixture
Colored smoke	Potassium/sulfur/ organic dye mixture. Sodium bicarbonate is added as an acid neutralizer to decrease accidental ignition

15.3 SAFETY

Standard chemical and laboratory precautions should be considered.

15.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

Conkling JA. **Chemistry of Fireworks-Special Report**. Chemical and Engineering News. 1981. Vol 59(26): p24-32.

Conkling JA. **Chemistry of Pyrotechnics Basic Principles and Theory**. New York: Marcel Dekker, Inc. 1985.

Cook JR. **The Chemistry and Characteristics of Explosive Material**. New York: Vantage Press. 2001.

Cooper PW, Kurowski SR. **Introduction to the Technology of Explosives**. New York: Wiley-VCH. 1996.

Davis T L. **The Chemistry of Powder and Explosives**. San Pedro (CA): GSG & Associates. Reprint of the original copyrighted in 1943.

Hopen TJ. **Identification of Low Explosives**. Presented at: NWAFS meeting Spring 1996.

McCrone WC, Delly JG. **The Particle Atlas Volumes 2 and 5**. Edition 2. Ann Arbor(MI): Ann Arbor Science Publishers, Inc. 1973. Also available on CD-ROM.

Russell MS. **The Chemistry of Fireworks**. Cambridge (UK): Royal Society of Chemistry RS•C. 2000.

Saxon K. **The Original the Poor Man's James Bond Volume 1**. Eldorado (AR): Desert Publications. 1991.

15.5 EXERCISES

1. Pick one of these options: sparklers, smoke bombs, aerial bombs, whistling devices, shooting starts, roman candles, sparkling volcanoes, or cones. Prepare a scheme to analyze the burned

and unreacted materials. Analyze the material you chose, both burned and unreacted, using your prepared analytical scheme.

16 FLASH POWDERS

16.1 OBJECTIVES

- To familiarize the trainee with the analysis of pyrotechnic black powders and flash powders, and their reaction products.

16.2 OVERVIEW

Flash powder is manufactured to produce a flash and a bang. Flash powder is made primarily of powdered aluminum or magnesium mixed with potassium perchlorate or chlorate. Sometimes sulfur is added to increase the sensitivity of potassium chlorate due to the low melting point of the sulfur and its function as "tinder." Clandestinely made flash powders have a wide variety of formulations.

Flash powder is usually found in the M-series of firecrackers, all of which are banned but usually show up around the 4th of July. M-series firecrackers are either commercially manufactured and smuggled in, or are "homemade" in outfits ranging from small scale operations to large scale clandestine factories.

16.2.1 Unreacted Flash Powder Analysis

1. Examine the material with a stereoscope and record observations
2. Perform burn/ignition testing
3. Try to examine some of the components by PLM
4. Preform SEM/EDX or XRF on the sample
5. Perform IR analysis of the sample. An extraction may be necessary to isolate some of the components
6. Perform microcrystalline testing and/or CE to identify the component cations and/or anions.

16.2.2 Burned Flash Powder Analysis

1. Examine the material with a stereoscope and record observations
2. Try to examine some of the components by PLM
3. Preform SEM/EDX or XRF on the sample
4. Perform IR analysis of the sample. Particle pick or an extraction may be necessary to isolate some of the components
5. Perform microcrystalline testing to identify the component cations and/or anions.

16.3 SAFETY

Depending on its formulation, flash powder can be unstable. It made be friction, shock, and heat sensitive and in large quantities may self-initiate. Its detonation velocity can approach high order. It should be handled in small quantities only (less than 1 gram). Great care should be taken when conducting burn testing of suspected flash powder material.

16.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

16.5 EXERCISES

1. Analyze an unreacted flash powder sample using all appropriate and available techniques.
    - (save remnants of burn/ignition test)
2. Analyze a burned flash powder sample using all appropriate and available techniques.
    - (Or use burn/ignition test from analysis above)

3. Analyze cardboard from an expended firecracker. How does it compare to unreacted flash powder?

17 SPARKLERS

17.1 OBJECTIVES

- To familiarize the trainee with the analysis of sparklers and their reaction products.

17.2 OVERVIEW

The production of sparks, and sparkle effects is a principle effect in the special effects industry. There are two main types of fireworks as it pertains to sparklers: wire sparklers and tube sparklers. Wire sparklers consist of a metal wire that had been dipped into a thick pyrotechnic mixture and allowed to dry. Tube sparklers consist of a long paper tube filled with pyrotechnic mixture and attached to a wooden rod using brightly colored tissue paper and/or ribbon. Tube sparklers may also contain layers of different materials resulting in a sparkler that changes color as it burns.

Both types of sparklers consist of a slow-burning pyrotechnic mixture containing the following types of components, one or more from each category may be present:

Metallic fuel, used to make the characteristic sparks

- aluminum (white)
- magnesium(white)
- magnalium (white)
- iron (orange)
- titanium (bright white)
- ferrotitanium (yellow-gold)

Oxidizer, can also double as a colorant

- potassium nitrate
- potassium chlorate
- potassium perchlorate
- barium nitrate
- strontium nitrate

Binder, used to hold the mixture together, can also double as an additional fuel

- sugar
- starch
- shellac
- dextrin
- nitrocellulose

Additional fuel, optional, used to modify burn speed

- charcoal
- sulfur

Pyrotechnic colorant, optional

- chlorides and nitrate of metals

COMMON COLOR EFFECT COMPOSITIONS	
Gold	Charcoal, iron, and barium compounds
Green	Barium compounds
Blue	Barium and Copper compounds

COMMON COLOR EFFECT COMPOSITIONS	
Red	Strontium compounds
White	Aluminum, magnesium, “magnalium,” and titanium compounds

Sparklers can also be used to make an improvised explosives device, or “sparkler bomb,” by bundling several packages of sparklers and wrapping them tightly in tape. The type of tape used and wrapping style varies widely by the creator; aluminum foil may also be used as a part of the wrappings but it’s not necessary for achieving an explosion. One sparkler is often used as a make-shift fuse.

17.2.1 Sparkler Analysis

1. Examine the material with a stereoscope and record observations.
2. Use PLM to identify any isolated components.
3. Perform SEM/EDX or XRF analysis. Be cautious of interpretation since wire sparklers contain metals.
4. Perform IR on the sample. Particle-pick or perform extractions to isolate the components.
5. Perform microcrystalline tests to identify components (cations/anions).

17.3 SAFETY

Standard chemical and laboratory precautions should be considered.

17.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

17.5 EXERCISES

1. Analyze an unreacted sparkler using all appropriate and available techniques.
    - (save remnants of burn/ignition test)
2. Analyze a burned sparkler using all appropriate and available techniques. If using a wire sparkler, examine the wire and differentiate it from the pyrotechnic material.
    - (Or use burn/ignition test from analysis above)

18 FUSEES/FLARES

18.1 OBJECTIVES

- To familiarize the trainee with the analysis of fusees/flares and their reaction products.

18.2 OVERVIEW

Fusees/flares are sometimes found in IEDs as well as at the sites of explosions and fires. They consist of three basic parts, a base, a tube containing fire mix, and a cap containing a striker, or means of igniting the fire mix (such as a matchhead or scratch surface).

The base may incorporate a handle, stand, or anti-roll device.

The fire mix typically contains a mixture of strontium nitrate, sulfur and potassium nitrate and produces a red flame. Organic materials, such as fillers or extenders, may also present. Finely divided sawdust and hydrocarbon oils provide fuel and assist in the manufacture process. Polyvinyl chloride may be added as a binder and color enhancer. Stearic acid is added to accelerate, or retard, the burn rate. The exact composition of the fire mix varies by manufacturer and colors other than red are available.

POSSIBLE FIRE MIX COMPONENTS		
Strontium nitrate	Wood flour	Powdered shellac
Potassium perchlorate	Sawdust and grease	Red gum
Potassium chlorate	Kerosene	Corn starch
Potassium nitrate	Fuel oil	Stearic acid
Strontium oxalate	Paraffin oil	Antimony
Barium nitrate	Petrolatum	Wax
Sawdust	Fine charcoal	Sulfur
Soda ash		

A Matchhead striker has a dark button of material on top of the fuse/flare and the active ingredients are typically strontium nitrate, potassium chlorate, and potassium perchlorate. The addition of quartz or marble dust is often added to make the mixture abrasive. To make the matchhead waterproof, liquid shellac is used as the binder.

POSSIBLE MATCHHEAD COMPONENTS		
Strontium nitrate	Strontium carbonate	Potassium perchlorate
Potassium chlorate	Barium chlorate	Quartz
Marble dust	Charcoal	Burnt umber
Wood flour	Shellac	Red gum
Alcohol	Solvent	

A scratch surface is composed primarily of red phosphorous, although butyl acetate and pumice, as an abrasive, may also be present. The composition is usually mixed with lacquer to make it waterproof and bind it to the cap.

POSSIBLE SCRATCH SURFACE COMPONENTS		
Red phosphorus	Butyl acetate	Potassium chlorate
Antimony sulfide	Manganese oxide	Fine sand
Pumice	Quartz	Glass
Emery	Glue	Lacquer
Thinner	Polyvinyl chloride	Dextrin
Charcoal		

18.2.1 Analysis of unreacted fusee/flare

1. Examine the material with a stereoscope and record observations.
2. Perform a burn/ignition test.
3. Perform SEM/EDX or XRF on the sample
4. Based on the observations, either particle pick or use an appropriate extraction scheme.
5. Perform IR
6. Perform PLM on the components.
7. Perform microchemical testing on the materials to identify the components to include anions and cations.
8. Perform CE on appropriate extracts as necessary

18.2.2 Analysis of burned fusee/flare

1. Examine the material with a stereoscope and record observations.
2. Perform SEM/EDX or XRF on the sample.
3. Based on the observations, analyze the residues. Use instrumentals examinations, extraction procedures, microchemical testing, and PLM to identify the reaction products.
4. Perform CE on appropriate extracts as necessary

18.3 SAFETY

Standard chemical and laboratory precautions should be considered.

18.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

18.5 EXERCISES

1. Analyze an unreacted fusee/flare fire mix and striker using all appropriate and available techniques.
    - (save remnants of burn/ignition test)
2. Analyze a burned fusee/flare using all appropriate and available techniques.
    - (Or use burn/ignition test from analysis above)

19 SMOKELESS POWDERS

19.1 OBJECTIVES

- To familiarize the trainee with the analysis of smokeless powders and their reaction products.

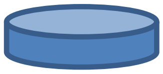
19.2 OVERVIEW

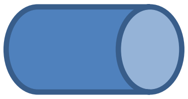
Smokeless powders, sometimes known as ballistite, cordite or propellant, are propellants used in firearms and artillery that produce minimal amounts of smoke when fired. Smokeless powders are divided into three categories depending on base components: single-base (nitrocellulose only), double-base (nitrocellulose and nitroglycerin) and triple-base (nitrocellulose, nitroglycerin, and nitroguanidine).

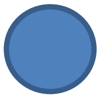
Smokeless powders are formulated to obtain a specific burn rate to ensure a desired performance. Besides the base components, other materials are also added to smokeless powders such as stabilizers, burn modifiers, deterrent plasticizers, flash suppressants, and ignition aids. Synthetic fibers, such as polyester, may also be present to help keep pellets intact. The fibers may be of forensic significance, in which case they should be collected for further analysis by a fiber expert.

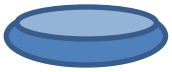
ADDITIONAL SMOKELESS POWDER COMPONENTS		
Deterrent Plasticizers	**Stabilizers**	**Burn Modifier**
Ethyl centralite (EC)	Diphenylamine (DPA)	Dinitrotoluene (DNT)
Methyl centralite (MC)	2-N-diphenylamine(2-nDPA)	Potassium sulfate (K_2SO_4)
Dibutylphthalate (DBP)		
	Ignition Aid	
	Potassium nitrate (KNO_3)	

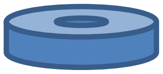
Gun powder manufacturers may also vary the size and shape of the powder to affect its performance.


Disc


Rod


Ball/Sphere


Flattened Ball


Perforated Disc


Tube


Lamel


Agglomerated Ball

Research has been conducted to see if the examination of the physical characteristics and composition of the smokeless powder can point to the manufacturer. Individualization may be possible in the case of extremely unique and special smokeless powders. Usually, however, manufacturers are trying to achieve a specific burn rate and may mix different lots, batches, sizes and shapes in order to achieve the desired performance objective. This makes it difficult to link a specific gunpowder pellet back to a specific manufacturer. However, some manufacturers add marker grains and/or taggants which identify the powder and/or manufacturer.

19.2.1 Methanol Extraction

This is good for getting the nitroglycerin, deterrents, plasticizers, stabilizers and burn modifiers into solution for subsequent analysis by GCMS, CE and/or TLC. The methanol extract can also be dried down to obtain an IR spectrum of nitroglycerin.

1. Sonicate the sample in methanol for 15 to 30 minutes, or leave the sample in methanol overnight.
2. Dry down the insoluble material for an IR analysis. This is the nitrocellulose.

19.2.2 Acetone/Water/Ethanol Extraction

1. Sonicate the sample in acetone for 30 minutes, or leave in acetone overnight.
2. Add four times as much water as the volume of acetone to precipitate the nitrocellulose.
3. Centrifuge and decant the aqueous phase, retaining for later examination.
4. Wash the precipitate twice with methanol, evaporate and perform IR analysis.
5. Add a small portion of diethyl ether of the aqueous phase and extract two times.
6. Analyze a small portion of the ether extract with GCMS, CE and/or TLC. This is the nitroglycerin and some of the other products.
7. Evaporate the remainder and analyze the oily product by IR. This is the nitroglycerin.

19.3 SAFETY

Standard chemical and laboratory precautions should be considered.

19.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

19.5 EXERCISES

1. Take 10 types of smokeless powders (4 to 5 single base, 4 to 5 double base and 1 triple base) and examine each under the stereoscope, recording size, color, and interesting characteristics. (Save and use for exercise 5)
2. Using a double base smokeless powder, extract the following quantities with methanol and perform the listed available analyses:
    a. 20 discs: GCMS, CE(organics)
    b. 5 discs: GCMS, CE(organics), TLC
    c. 1 disc: GCMS, CE(organics)
    d. ½ disc: GCMS, CE(organics)
3. Using the acetone/water/diethyl ether method, perform the following analyses on the listed quantities of a double base smokeless powder:
    a. 20 discs: GCMS, CE(organics)
    b. 5 discs: GCMS, CE(organics), TLC (also do this for single and double base powder)
    c. 1 disc: GCMS, CE(organics)
    d. ½ disc: GCMS, CE(organics)
4. Compare IR spectrum of NG extracted via the acetone/water/diethyl ether method (will be oily) to that of the standard. Note any differences.
5. Determine the components of 10 different smokeless powders using GCMS and CE or TLC.

20 MILITARY EXPLOSIVES

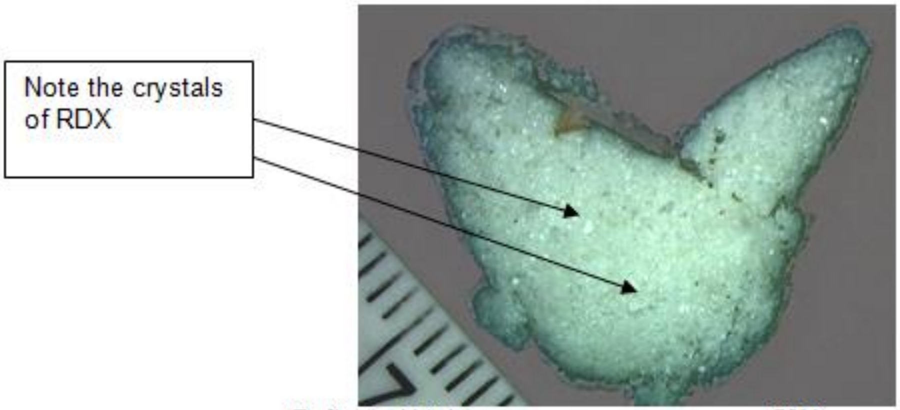
20.1 OBJECTIVES

- To familiarize the trainee with the analysis of military explosives.

20.2 OVERVIEW

Plastic bonded explosive (PBX) is a generic term covering several different formulations. The general composition of these substances includes a high explosive (e.g., RDX or PETN) mixed with a binder, which is typically a polymer mixed with a plasticizer. The two most popular forms are C-4 and SEMTEX.

Military Explosive Compound C-4, sometimes called plastic explosive, is a formulation of RDX, polyisobutylene (binder), either bis-2-(ethylhexyl)adipate or sebacate (plasticizer), and motor oil. C-4 is a white to grey pasty, plastic-like, malleable material. Stereoscopic examination of this material reveals large crystals of RDX surrounded by a plastic-like matrix. C-4 contains varying amounts and combinations of grain sizes and RDX. These characteristics give rise to different classes of C-4.



Reflected Light 50X

Pentaerythritol tetranitrate (PETN) is a very powerful explosive and often used in the explosive core of detonation (det) cord, used to initiate high explosives. It is the least stable of the common military explosives and has been replaced by RDX in many applications, which is more stable and has a longer shelf-life.

PETN mixed with RDX is the main ingredients of Semtex, a general purpose plastic explosive.

20.2.1 Analysis of C-4

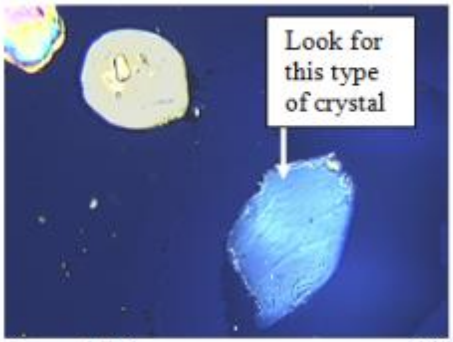
Perform stereoscopic examination.

- Will see large crystals of RDX surrounded by a plastic-like matrix.

Isolation and Identification of RDX

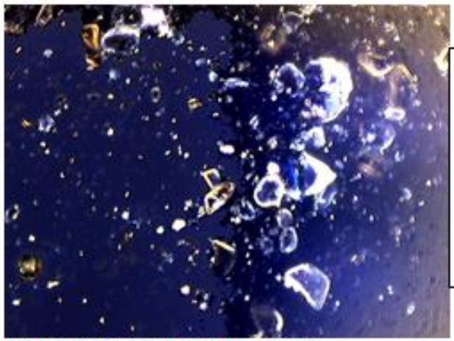
1. Place 50-100 mg of suspected C-4 in a beaker and add 30mL of chloroform.
2. Stir for 30 minutes using a magnetic stir bar.
    - If the material disperses, continue with the instructions, it is likely C-4.
    - If the material does not disperse it is something else, no need to continue.

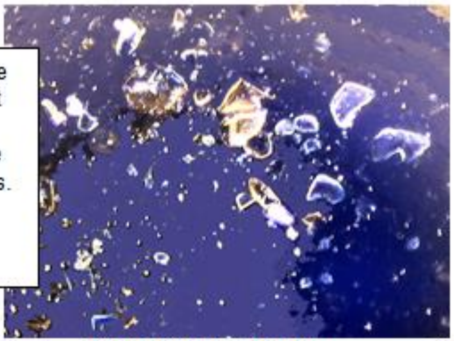
3. Filter the solution using a vacuum funnel with a membrane filter.
    - RDX is not soluble in chloroform and will remain on the filter.
    - The rest of the components are in the filtrate, save for further examination.
4. Examine the chloroform insoluble material under the stereoscope, there will be a mix of large and small grains.
5. Perform GCMS on a small amount of the chloroform insoluble material dissolved in acetonitrile.
6. Perform IR analysis on a small crystal of recrystallized chloroform insoluble material.
    - Dissolve a small amount of suspected RDX in 1mL of acetone and let evaporate on a small watch glass.
7. Perform PLM on the chloroform insoluble material.
    - Nx: 1.5775, Ny: 1.5966, Nz: 1.6015, (-)2V = 55°22’, r>v, Nz-Nx = 0.024


Look for this type of crystal

Crossed Polars 100 X

8. Perform dispersion staining with central stop.
    - Since the RIs of the liquid and crystals are the same in certain orientations, the analyst should see some crystals exhibiting blue to magenta to orange edges in certain orientations.


Note how the crystals start to change colors as the stage rotates.



Plane Polarized Light 100X
Central Stop

Plane Polarized Light 100X
Central Stop, Stage Rotated 90°

9. Perform TLC using the GCMS solution, check the plate in the UV box to make sure enough sample is present on the plate.

Isolation and Identification of the Binder

1. Take the chloroform filtrate saved from step 4 above and add a slightly greater amount of acetone to precipitate the polyisobutylene. Slowly add more acetone to ensure all the binder possible has precipitated.
2. Filter the solution through a membrane filter.
    - The polyisobutylene is not soluble and will remain on the filter.
    - Keep the filtrate for further examination.
3. Perform IR analysis on the sample.

Isolation and Identification of the Plasticizers

1. Take the filtrate from above and evaporate to dryness.
2. Add a small amount of carbon tetrachloride to the residue and mix thoroughly.
3. Filter the solution through a membrane filter.
    - The plasticizers are soluble and will be contained in the filtrate.
    - Discard the membrane filter containing the extraneous material.
4. GCMS on the filtrate.
5. Evaporate some of the filtrate to dryness and perform IR analysis.

20.2.2 Analysis of Det. Cord

The contents of det. cord are often pure enough to particle pick for direct IR.
PETN and RDX are soluble in both acetone and acetonitrile and may be analyzed by GCMS, TLC and/or CE.

20.3 SAFETY

Standard chemical and laboratory precautions should be considered.

20.4 SUGGESTED READING

WSP CLD Materials Analysis Technical Procedures: Explosives Chapter and Appendices

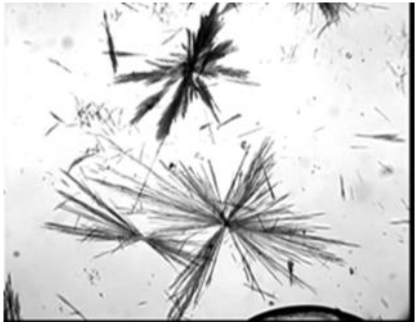
20.5 EXERCISES

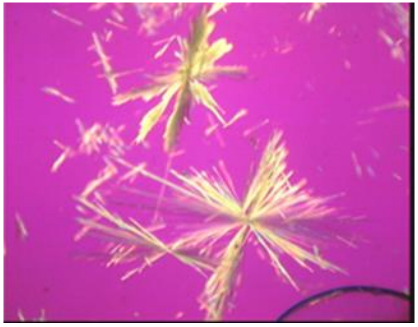
1. Analyze a sample of C-4, isolating the RDX, binder and plasticizer.
   Perform IR analysis on the isolate RDX before and after acetone recrystallization.

21 Appendix A: Microcrystal Tests

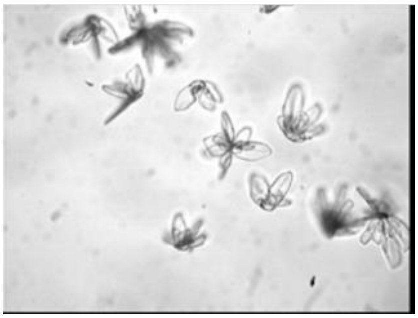
NITRON TESTS

Potassium Nitrate (1000ppm solution)
Immediate precipitation
Needles and hairs forming single tufts and groups of tufts. Sheaves from on standing


Plane Polarized Light, 10x

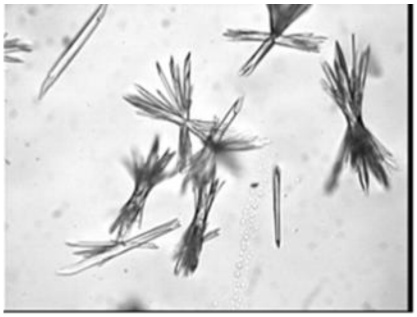

Crossed Polars, 1λ plate, 10x

Potassium Perchlorate (1000ppm solution)
Immediate precipitation
Rods join to form clusters. Rods then develop into blades and clusters of blades


Plane Polarized Light, 40x


Crossed Polars, 1λ plate, 20x

Potassium Chlorate (powder directly added to Nitro reagent)
Immediate precipitation
Rods join to form clusters. Rods then develop into blades and clusters of blades


Plane Polarized Light, 40x

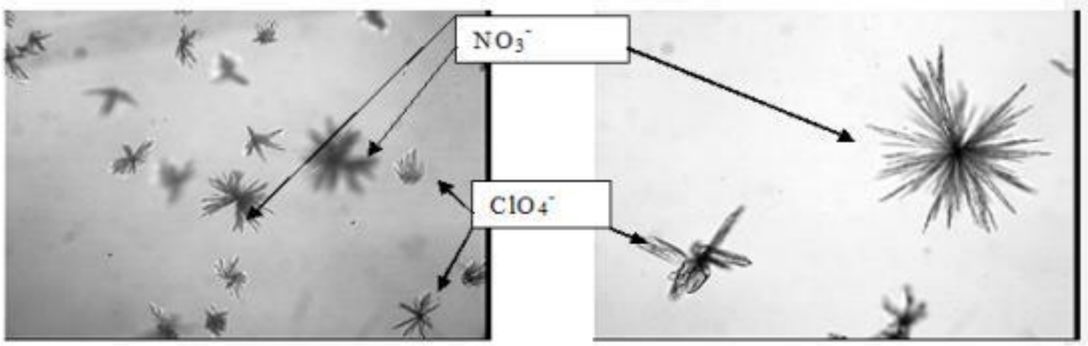

Crossed Polars, 1λ plate, 10x

NITRON TESTS

Potassium Nitrate and Potassium Perchlorate Mixture (1000ppm each)

Immediate precipitation

The nitrate and perchlorate crystals grow individually and are easily discernable from one another

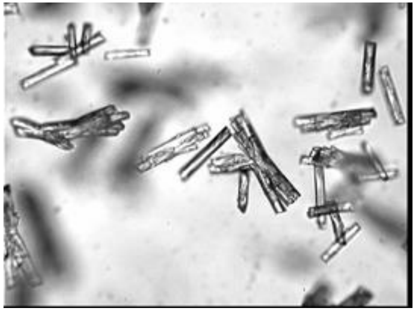


Plane Polarized Light, 10x

Plane Polarized Light, 20x

Potassium Iodide

Very small rods that clump together



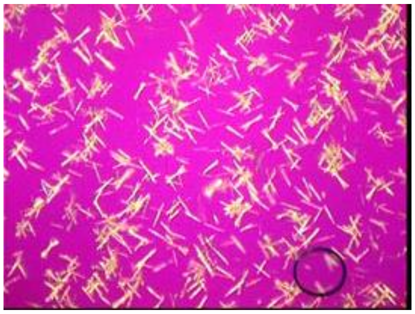
Plane Polarized Light, 10x

Potassium Nitrite

Formation of long, straight needs, singly and in sparse tufts

Closest crystals to nitrates, but they are broader and don’t form sheaves like nitrates do

Plane Polarized Light, 10x

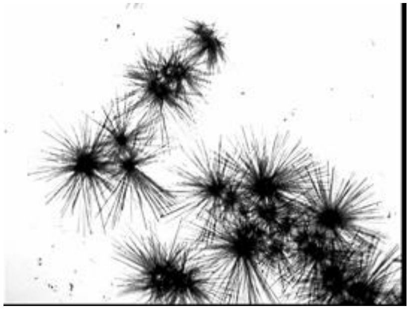


Crossed Polars, 1λ plate, 10x

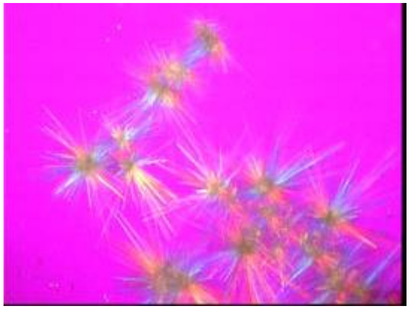
NITRON TESTS

Potassium Oxalate

Rapidly developing burrs (fine needles radiating from a central core)



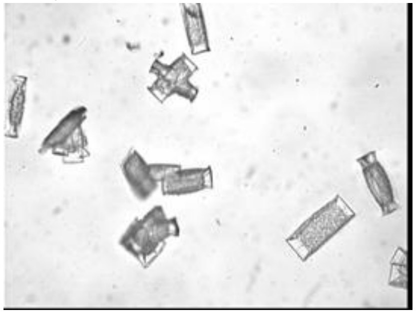
Plane Polarized Light, 10x



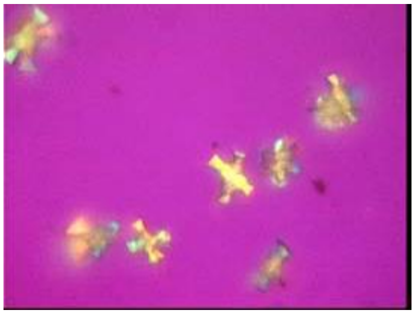
Crossed Polars, 1λ plate, 10x

Potassium Tartrate

Short, stubby barrels with a mottled surface



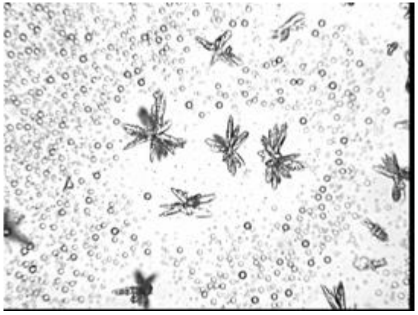
Plane Polarized Light, 40x



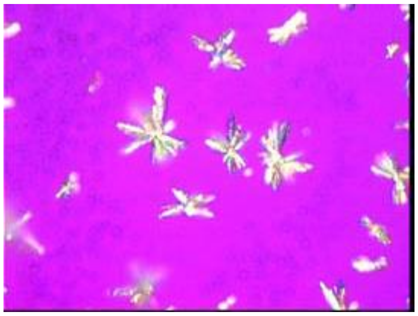
Crossed Polars, 1λ plate, 40x

Potassium Thiocyanate

Small, irregular, coarse blades forming from oily precipitate
Sometimes forming X’s



Plane Polarized Light, 40x



Crossed Polars, 1λ plate, 40x

Potassium Bicarbonate (excess of powder added to Nitron)

Precipitate forms after effervescence stops
Particles start forming oily droplets
No crystals seen after 3 minutes

NITRON TESTS

<u>Potassium Carbonate </u>(excess of powder added to Nitron)
Precipitate forms after effervescence stops
Particles start forming oily droplets
No crystals seen after 3 minutes

<u>Potassium Chloride</u>
Precipitate changing into an amorphous scaly mass

<u>Potassium Chromate</u>
Yellow precipitate. No crystals after 1 minute

<u>Potassium Dichromate</u>
Yellow precipitate. No crystals after 1 minute

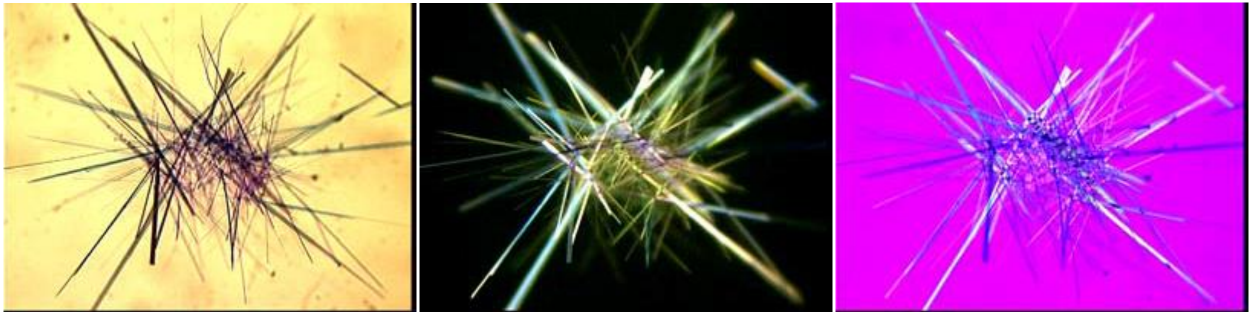
<u>Potassium Ferricyanide</u>
Yellow precipitate. No crystals after 1 minute
White precipitate. No crystals after 1 minute

<u>Potassium Phosphate</u>
No reaction after 1 minute

<u>Potassium Sulfate</u>
No reaction after 1 minute

METHYLENE BLUE TEST

<u>Potassium Chlorate</u>
Mainly blue rosette (sometimes tinged with purple)
Develop slower than perchlorates



Plane Polarized Light, 40x   Crossed Polars, 40x   Crossed Polars, 1λ Plate, 40x

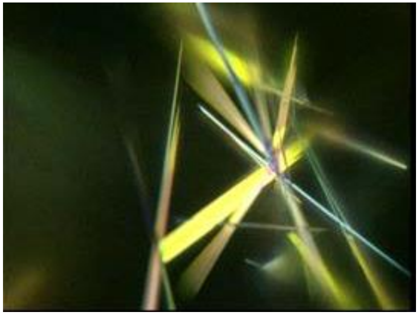
METHYLENE BLUE TEST

Potassium Perchlorate

Fast reaction

Blue needles, sometimes with a purplish tinge, singly and in bundles


Plane Polarized Light, 40x

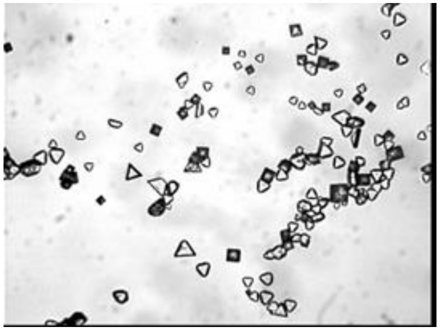

Crossed Polars, 40x


Crossed Polars, 1λ Plate, 40x

SILVER NITRATE TEST

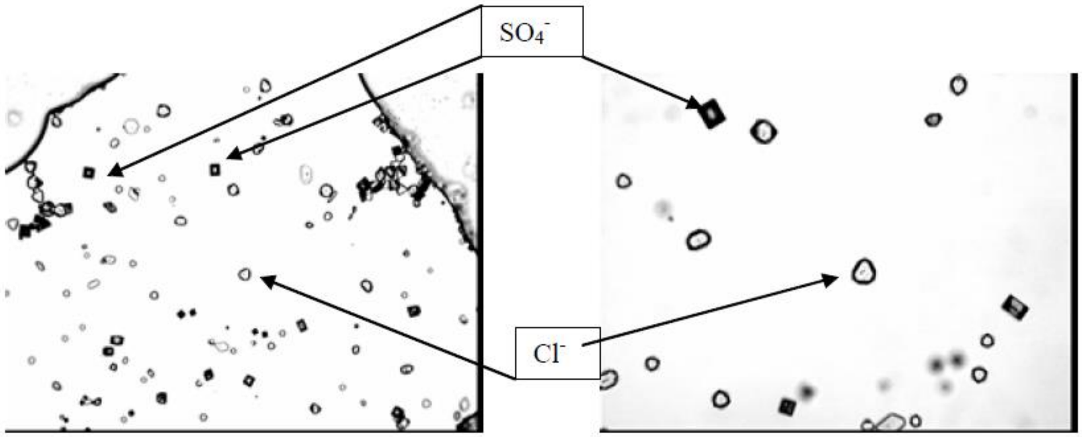
Potassium Chloride

Isotropic octahedral, triangles and hexagonal plates


Plane Polarized Light, 40x

Potassium Chloride and High Concentration of Potassium Sulfate

Sulfate crystals are colorless, highly refractive, rhomb-shaped tablets or short stout prisms with angular ends



Plane Polarized Light, 10 x

Plane Polarized Light, 40x

SQUARIC ACID TEST

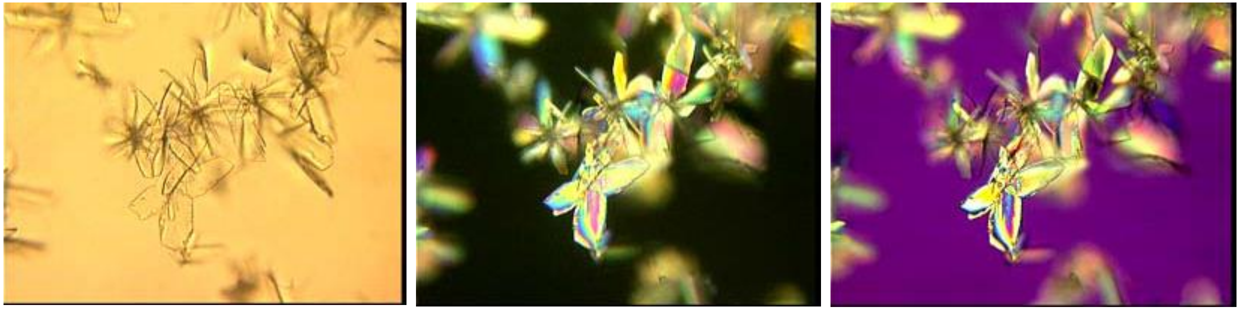
Potassium Chloride

Birefringent, 4-pointed stars, medium retardation colors

Plane Polarized Light, 40x
Crossed Polars, 40x
Crossed Polars, 1λ Plate, 40x

Sodium Chloride

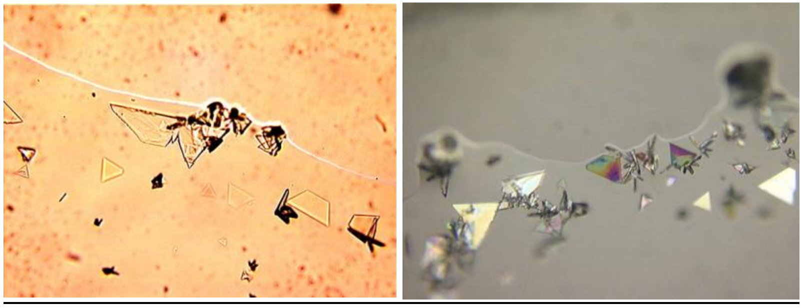
Large birefringent blades, sometimes crossing or radiating from a central point
Medium to high retardation colors



Plane Polarized Light, 40x
Crossed Polars, 40x
Crossed Polars, 1λ Plate, 40x

Barium

Flat blades showing high order retardation colors

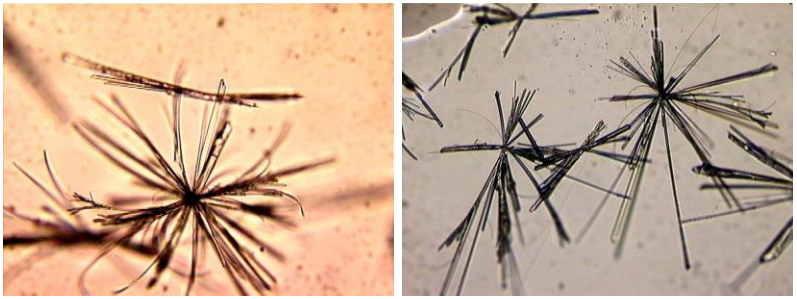


Plane Polarize Light, 100x
Slightly Uncrossed Polars, 200x

SQUARIC ACID TEST

Strontium

Large curved needles showing high order reardation color



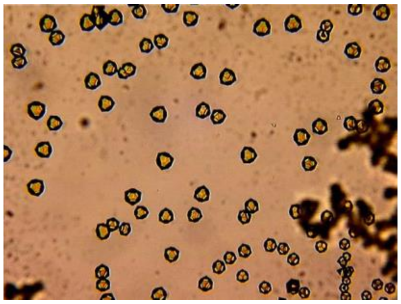
Plane Polarize Light, 200x Slightly Uncrossed Polars, 200x

CHLOROPLATINIC ACID

Potassium and Ammonium

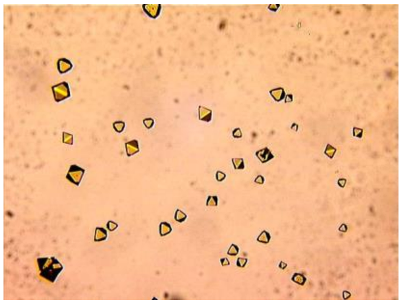
Both ions give the same crystals, hanging drop method must be used distinguish between them.
Ammonium will volatize and react with chloroplatinic acid, potassium will not volatize

Potassium Nitrate



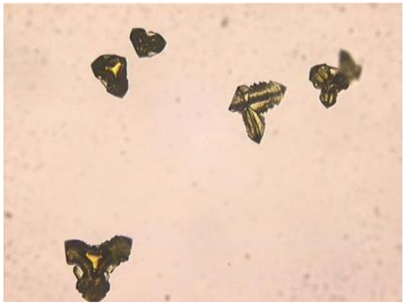
Plane Polarized Light, 200x

Ammonium Nitrate



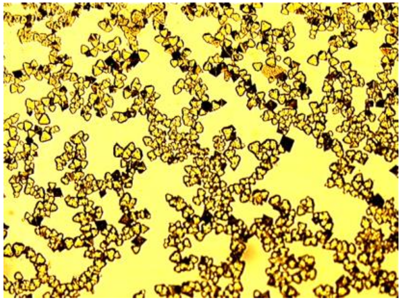
Plane Polarized Light, 200x

Ammonium Nitrate, on standing



Plane Polarized Light, 200x

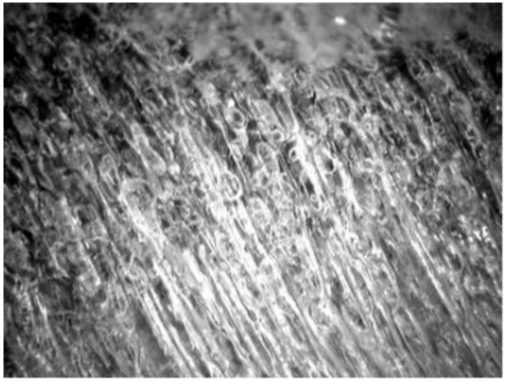
Ammonium Nitrate, hanging drop



Plane Polarized Light, 200x

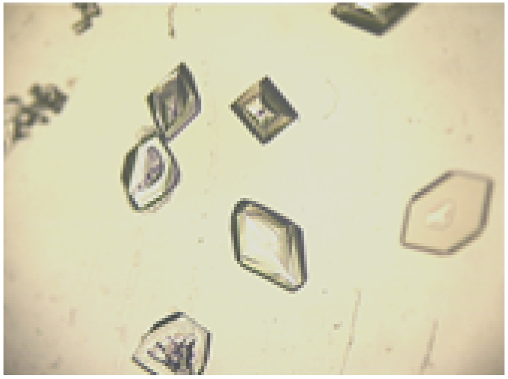
22 Appendix B: Recrystallization Photos

Ammonium Nitrate: recrystallized from water
Crystals tend to elongate and fuse into a large mass
If small crystals form, there is a "halo" of moisture with no distinctive features



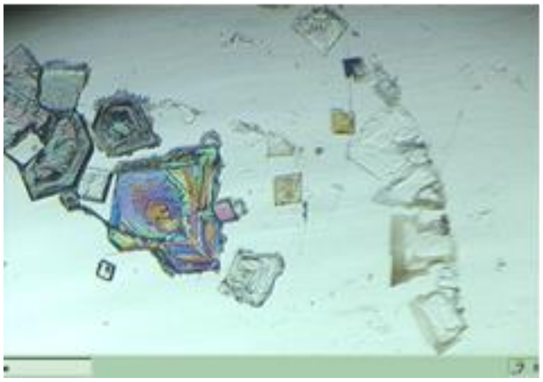
Stereoscope, transmitted light, 100x

Ammonium Perchlorate: recrystallized from water
Nice thick crystals: hexagons, diamonds, plates



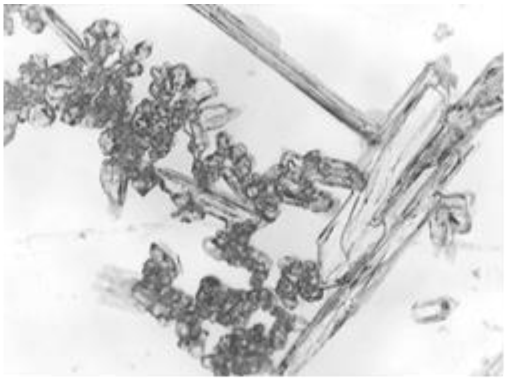
PLM, slightly uncrossed polars, 100x

Potassium Chlorate: recrystallized from water
Forms large plates and tablets that may stick out of solution as it evaporates



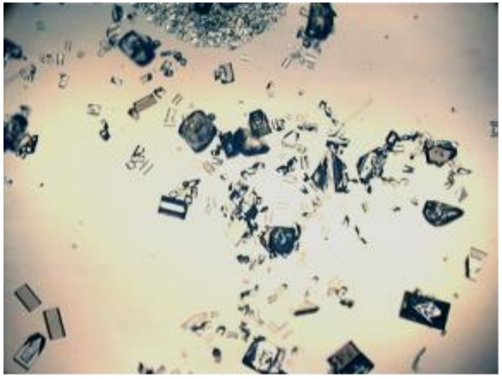
Stereoscope, Slightly uncrossed polars, 100x

Potassium Nitrate: recrystallized from water
Forms branching blades
Clustered around the blades are diamonds, stacked diamonds and plates



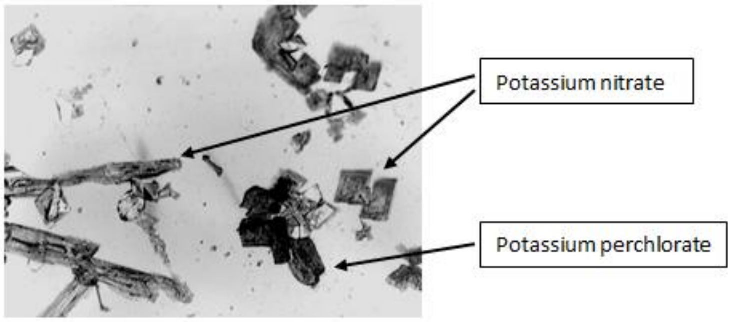
Stereoscope, transmitted light, 100x

Potassium Perchlorate: recrystallized from water
Crystals are thick, elongated and appear to have angular sides



Stereoscope, slightly uncrossed polars, 100x

Potassium Nitrate/Potassium Perchlorate Mixture: recrystallized from water
Potassium nitrate forms blades, plates and tablets
Potassium perchlorate looks like rhinestones throughout the mixture

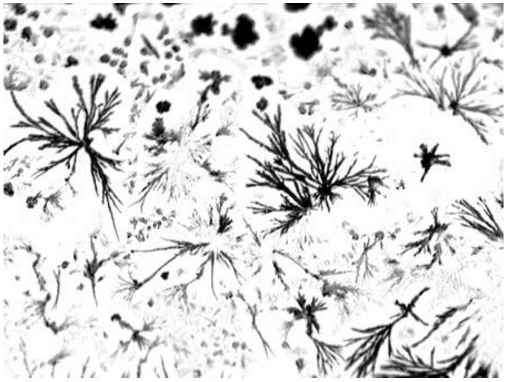


Stereoscope, transmitted light, 100x

Sulfur: recrystallized from chloroform
Pyramidal crystals that appear as individual diamonds or flat chains of overlapping diamonds

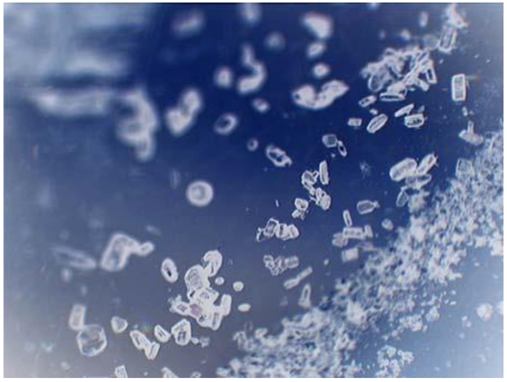
Stereoscope, slightly uncrossed polars, 100x

Sodium Benzoate: recrystallized from ethanol
Crystals are tufts or branching tendrils



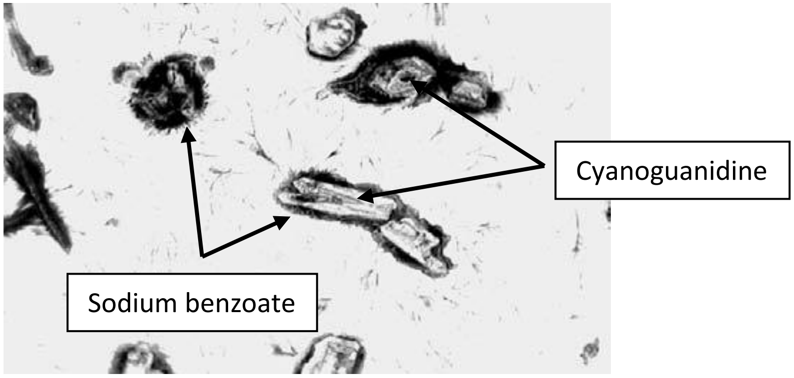
Stereoscope, transmitted light, 100x

Cyanoguanidine (Dicyandiamide): recrystallized from ethanol
Nice plates, prisms and rectangles that grow very slowly



Stereoscope, reflected light, 100x

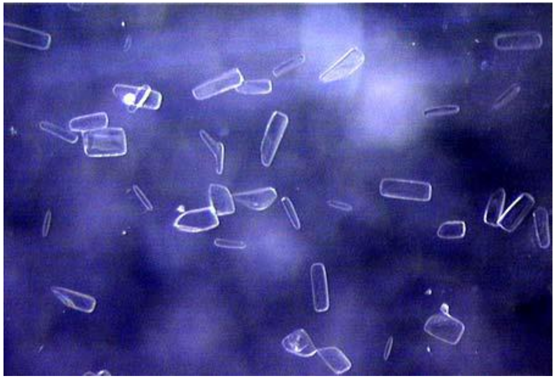
Sodium Benzoate/Cyanoguanidine Mixture: recrystallized from ethanol
Cyanoguanidine grows as clear flat blades
Sodium benzoate grows around the edges of the cyanoguanidine crystals (mold-like)



Stereoscope, transmitted light, 100x

Sucrose: recrystallized from water
No crystals formed when a saturated solution was left or air dry for 5 days

Sugar/Potassium Perchlorate Mixture: recrystallized from water
Potassium perchlorate crystals form in a sugar syrup



Stereoscope, reflected light, 100x

23 Appendix C: Optical Properties

Summary of the Optical Characteristics of Isotropic Explosives		
Name	Formula	n
Ammonium Chloride	HN_4Cl	1.6426
Barium Nitrate	$Ba(NO_3)_2$	1.5711
Copper (I) Chloride	$CuCl$	1.973
Ferrous Oxide (black iron oxide)	Fe_3O_4	2.42
Potassium Chloride	KCl	1.4904
Sodium Chloride	$NaCl$	1.5443
Strontium Nitrate	$Sr(NO_3)_2$	1.5878

Summary of the Optical Characteristics of Uniaxial Explosives					
Name	Formula	ε (n_x)	ω (n_y)	Crystal System	Optic Sign
Calcium Carbonate, Calcite	$CaCO_3$	1.4864	1.6584	Hexagonal	NEG
Ferric Oxide (red iron oxide)	Fe_2O_3	2.78	3.01	Tetragonal	NEG
Lead Oxide (red lead)	Pb_3O_4			Tetragonal	NEG
Magnesium Carbonate	$MgCO_3$	1.509	1.700	Hexagonal	NEG
PETN	Pentaerythritol tetranitrate	1.551	1.556	Tetragonal	NEG
Sodium Nitrate	$NaNO_3$	1.3361	1.5874	Hexagonal	NEG

Summary of the Optical Characteristics of Biaxial Explosives									
Name	Formula/Chemical Name	i			n_x	n_y	n_z	Crystal System	$2V_o$
Ammonium Nitrate	NH_4NO_3				1.463	1.543	1.600	Orthorhombic	-80
Ammonium Perchlorate	NH_4ClO_4				1.4818	1.4833	1.4481	Orthorhombic	69
Barium Carbonate	$BaCO_3$				1.529	1.676	1.677	Orthorhombic	-16
Barium Chlorate	$Ba(ClO_3)_2 \bullet H_2O$				1.562	1.577	1.635	Monoclinic	56
Barium Sulfate	$BaSO_4$				1.6363	1.6373	1.6484	Orthorhombic	37
Calcium Carbonate, aragonite	$CaCO_3$				1.5300	1.6810	1.6854	Orthorhombic	-18
Calcium Sulfate	$CaSO_4$				1.5698	1.5754	1.6136	Orthorhombic	43
Copper(II) Carbonate, Basic	$CuCO_3$				1.73	1.754	1.836	Monoclinic	67

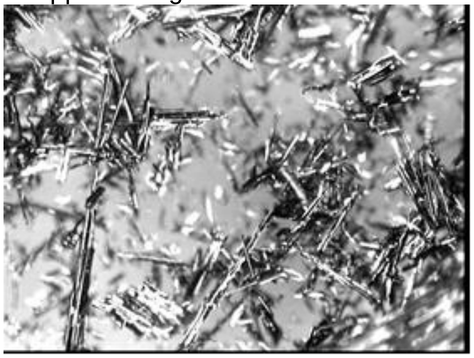
Summary of the Optical Characteristics of Biaxial Explosives

Name	Formula/Chemical Name	i			n_x	n_y	n_z	Crystal System	$2V_o$
Copper (II) Oxide	CuO				2.63	2.84	3.18	Monoclinic	20
Copper (II) Oxychloride	$3CuO \bullet CuCl_2 \bullet 3.5H_2O$				1.831	1.861	1.880	Orthorhombic	-75
HMX, form I	Cyclotetramethylene Tetranitramine				1.589	1.594	1.73	Monoclinic	20
HMX, form II					1.561-1.565 ave: 1.563	1.562-1.566 ave: 1.564	1.72-1.74 ave: 1.73	Orthorhombic	0-30 red, 8-30 blue
HMX, form III					1.537	1.585	1.666	Monoclinic	75
Nitroguanidine	Picrite				1.526	1.694	1.81	Orthorhombic	~-80
Picric Acid	2,4,6-trinitrophenol				1.667	1.699	1.742	Orthorhombic	82
Potassium Chlorate	$KClO_3$				1.4099	1.5174	1.5241	Monoclinic	-27
Potassium Dichromate	$K_2Cr_2O_7$				1.715	1.762	1.892	Monoclinic	64
Potassium Nitrate	KNO_3				1.3346	1.5056	1.5064	Orthorhombic	-7
Potassium Perchlorate	$KClO_4$				1.4731	1.4737	1.4769	Orthorhombic	50
Potassium Sulfate	K_2SO_4				1.4935	1.4947	1.4973	Orthorhombic	67
RDX	Cyclonite, cyclotrimethene trinitramine				1.5575	1.5966	1.6015	Orthorhombic	-53
Sodium Bicarbonate	Na_2SO_4				1.380	1.500	1.586	Monoclinic	-75
Sodium Sulfate	Na_2SO_4				1.471	1.477	1.484	Orthorhombic	84
Strontium Carbonate	$SrCO_3$				1.5199	1.6666	1.6685	Orthorhombic	-7
Strontium Sulfate	$SrSO_4$				1.6215	1.6232	1.6305	Orthorhombic	50

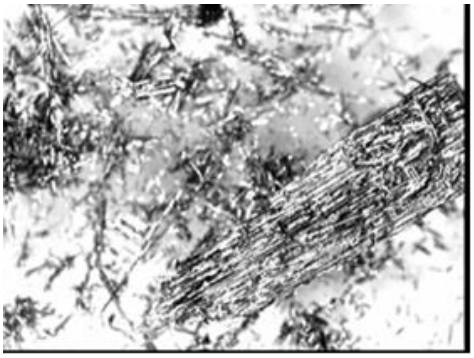
24 Appendix D: Charcoal Photos

BURNED WOOD

Close view shows details of the cellular structure of the wood
carbonization has started but was not complete
material appears to glisten and reflect some of the light back



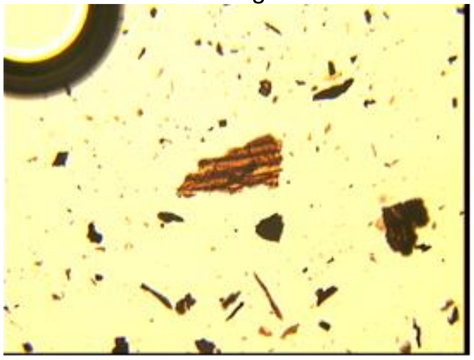
Top Light, Low Mag



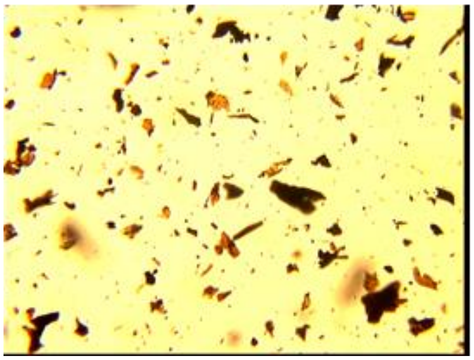
Top Light, Medium Mag

WOOD CHARCOAL

Material reflective in top light. Close observation reveals the cellular structure of the wood.
At 10X most of the material appears opaque, edges appear angular with long fine splinters.
At 40X the smaller particles are transparent, golden brown colored, angular edges with some showing cellular structure. Some fragments exhibit definite woody structure.



Plane Polarized Light, 20X



Plane Polarized Light, 40X

COCONUT CHARCOAL

Smooth appearance in top light, with a yellow/brown color.

Fine granular appearance more like stone than wood.

Difficult to see fine cellular structure, larger pieces show some characteristics.

Top Light, Low Mag

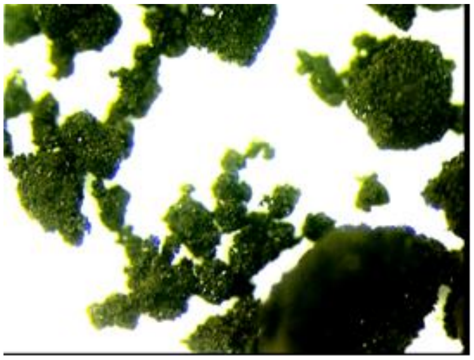
Plane Polarized Light, 40x

ANIMAL CHARCOAL

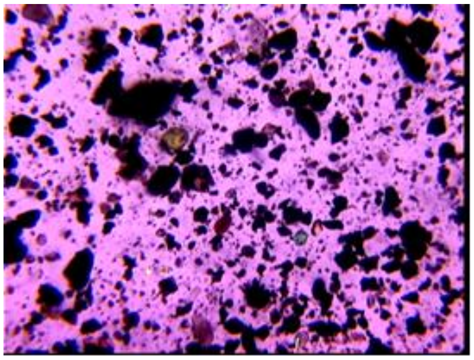
Low mag: light brown color, texture similar to fine clay

High mag: fine reflective crystalline particles can be observed (birefringent with crossed polars and 1λ plate)

No cellular structure evidence



Top Light, High Mag

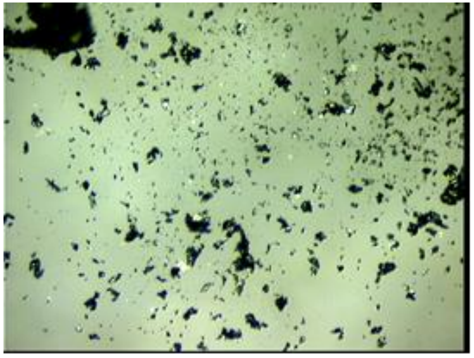


Plane Polarized Light, 1λ Plate, 40x

CHARCOAL BRIQUETTE

Top light: matte finish with clear and opaque reflective particles, few pieces of burned wood.

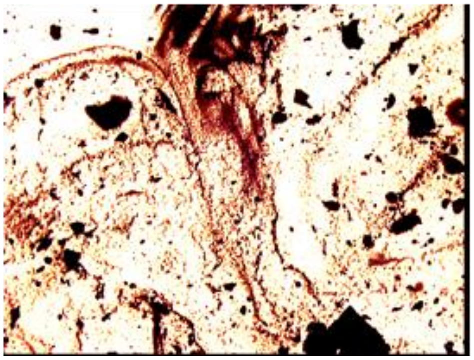
Top Light, Low Mag



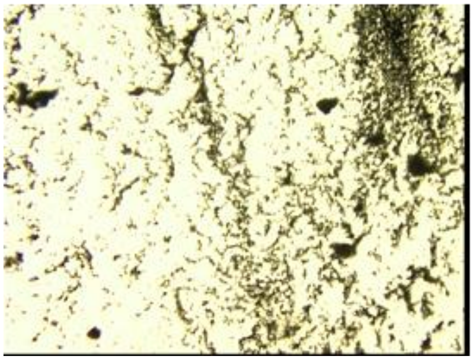
Plane Polarized Light, 40x

CARBON BLACK

Very fine particles ranging from <0.5-10 micron (most are 0.5-4 micron)
No identifiable plant material or birefringent particles.



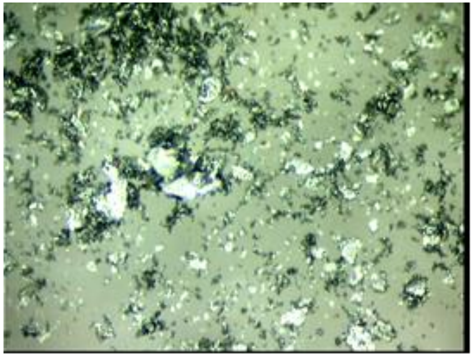
Plane Polarized Light, 10x



Plane Polarized Light, 40x

GRAPHITE

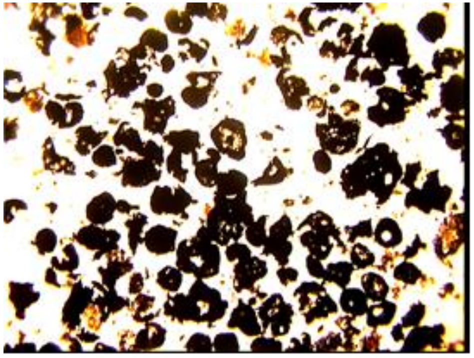
Opaque, sharply angular flakes, smooth surfaces, some layered, angular at times
May contain opaque, magnetic of Fe_3O_4; red, round particles of iron oxide; traces of quartz and calcite



Plane Polarized Light, 40x

OIL SOOT

Mostly brown to black "glassy" spheres, air bubbles trapped in the particles.
Top lighting they are shiny with rough surface
Thinner particles have lacy features at higher magnifications



Plane Polarized Light, 10x

25 Appendix E: IR Explosive Library

IR Library Compounds

Inorganic Explosives

Ammonium nitrate
Ammonium perchlorate
Antimony sulfide
Barium nitrate
Boron
Calcium hypochlorite
Calcium sulfate (CaSO4•½ H2O)
(Plaster of Paris)
Copper (Cupric) chloride
Copper (Cuprous) chloride
Copper (Cupric) oxide
Diatomaceous earth (filler)
Iron (Ferric) oxide
Lead dioxide
Lead nitrate
Potassium chlorate
Potassium dichromate
Potassium nitrate
Potassium perchlorate
Red phosphorus
Sodium bicarbonate
Sodium chlorate
Sodium nitrate
Sodium perchlorate
Strontium nitrate
Sulfur

Explosive Reaction Products & Mixtures

Aluminum oxide
Aluminum sulfate
Antimony sulfide
Barium chloride
Barium oxide
Boron oxide
Calcium carbonate
Copper (Cupric) oxide
Copper (Cupric) sulfate
Iron (Ferric) oxide
Iron (Ferric) sulfate
Lead monoxide
Lithium carbonate
Magnesium oxide
Potassium carbonate
Potassium chloride
Potassium oxide
Potassium sulfate
Pyrodex® powder granule
Silicon dioxide
Sodium bicarbonate
Sodium carbonate
Sodium chloride
Sodium oxide
Sodium sulfate
Strontium carbonate
Strontium chloride
Strontium oxide
Titanium dioxide
Zinc oxide (found on pipe fragments)
Zirconium oxide

Organic Explosives

RDX
HMX
TNT
EGDN
Tetryl
Nitrobenzene
2-nitrotoluene
4-nitrotoluene
2,3-dinitrotoluene
2,4-dinitrotoluene
2,6-dinitrotoluene
3,4-dinitrotoluene
Diethyphthalate
Dibutylphthalate
Diphenylamine

Organics Used In Inorganic Explosives and Smokeless Powders

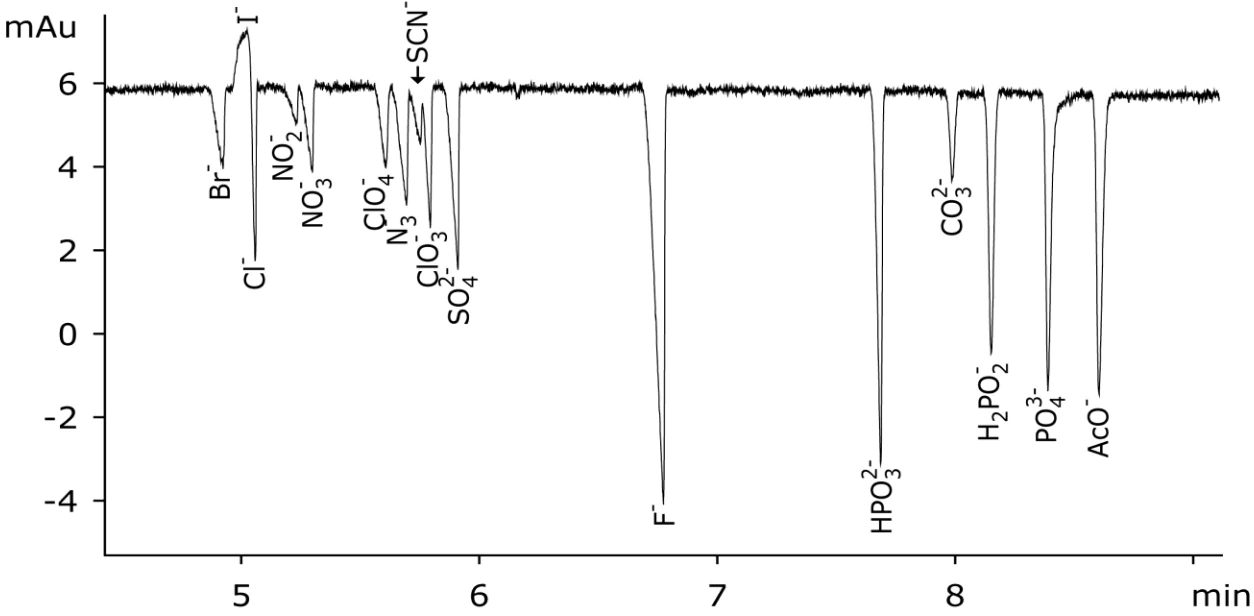
Adipate
Dextrin (a starch)
Dicyandiamide
Diphenylamine
Ethyl centralite
Methyl centralite
Gallic acid
Gum arabic
Nitrocellulose-fibers
Nitrocellulose-from acetone
Nitrocellulose-acetone-precip w/H2O
Nitroguanidine
Nitroglycerine (from smokeless powder)
Sebacate
Sodium benzoate
Starch
Stearic acid
Sugar

26 Appendix F: GCMS Methods

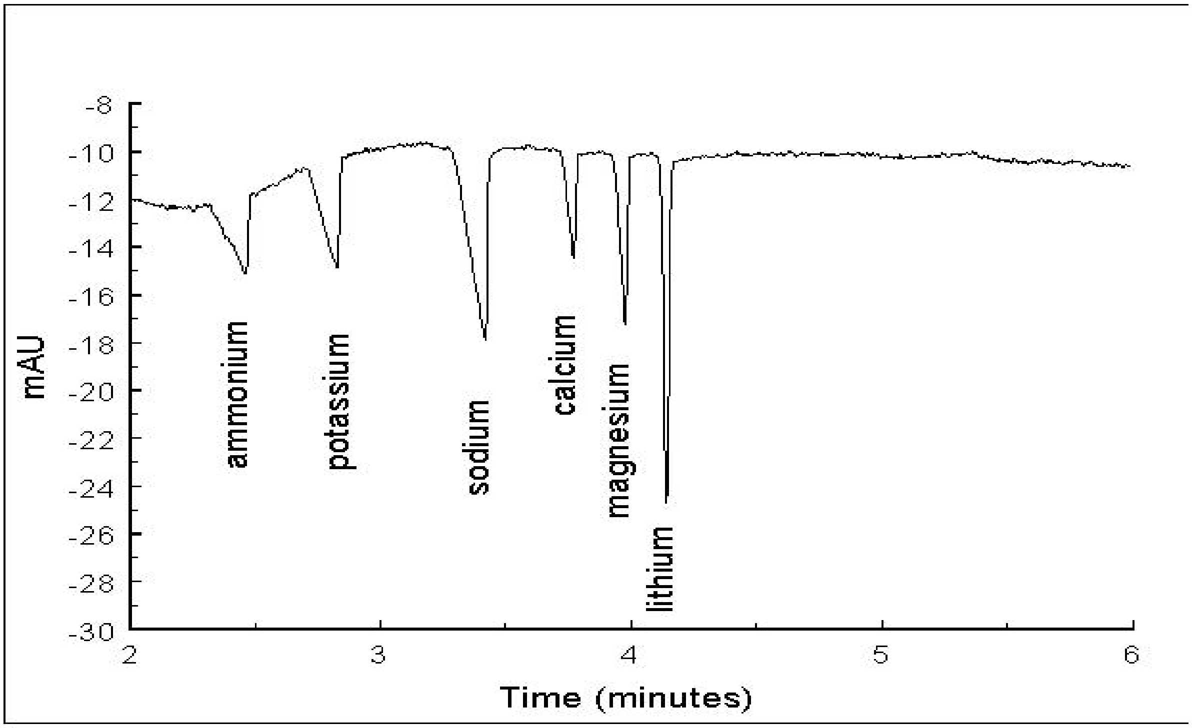
OVEN			
Initial Temperature:	50°C (or 100°C)		
Initial Time:	1.00 min		
Ramp:	Rate	Final Temp	Final Time
	10°C/min	310°C	5 min
Run Time:	32 min		
FRONT INLET			
Mode: Initial Temp: Pressure: Pulse Pressure: Pulse Time: Purge Flow: Purge Time: Total Flow:	Pulsed Splitless 190°C 7.6 psi 20.0 psi 1 min 19.9 mL/min 0.00 min 23.2 mL/min		
INJECTOR			
Injection Volume:	1 µL		

27 Appendix G: CE Run Data

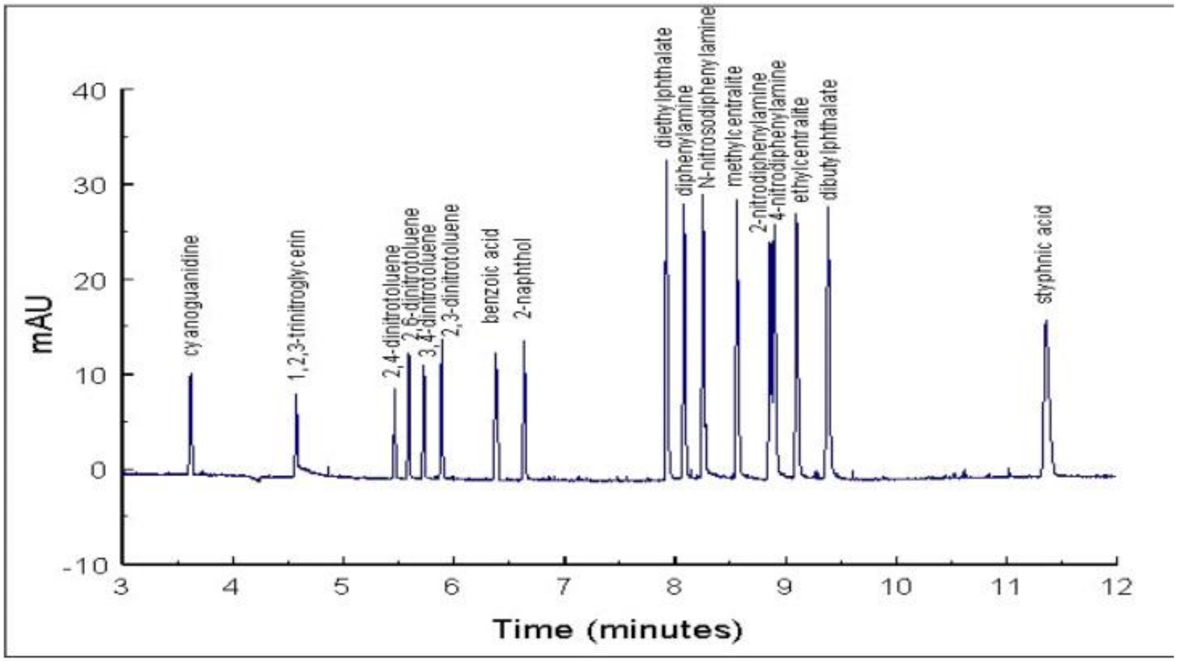
Anion Reference Materials using CElixirOA 8.2 anion method

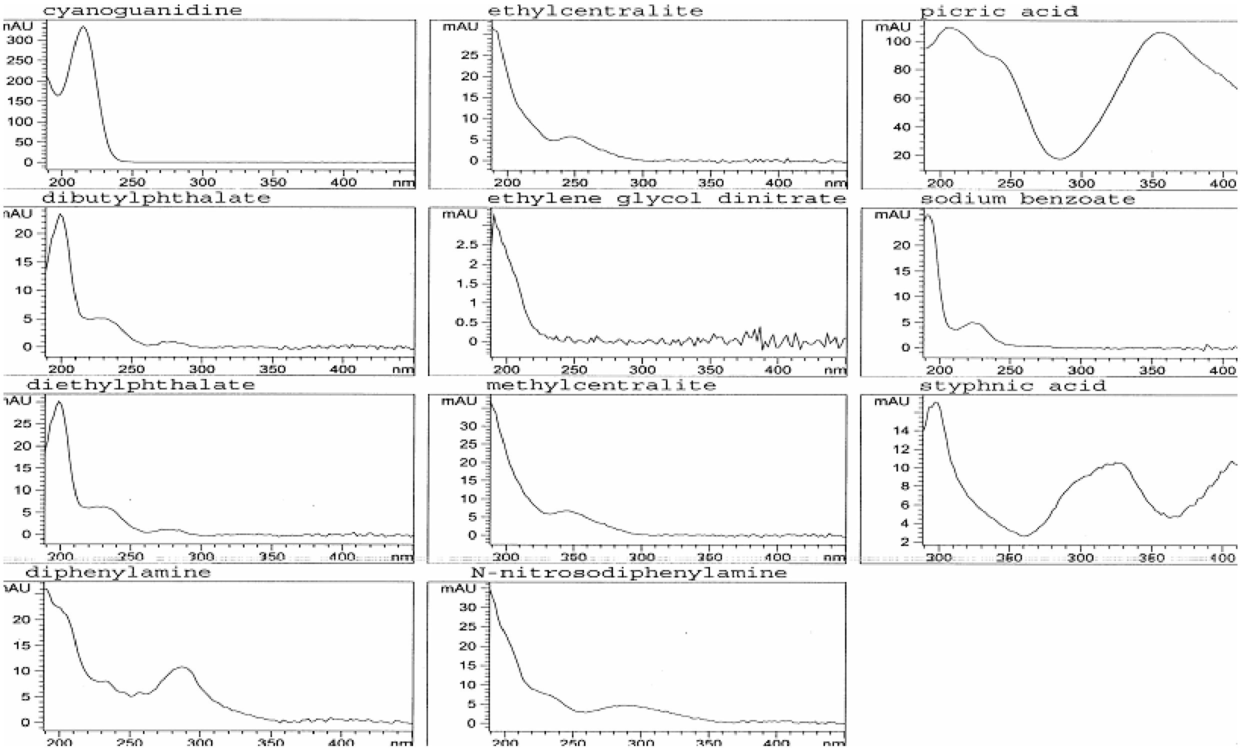


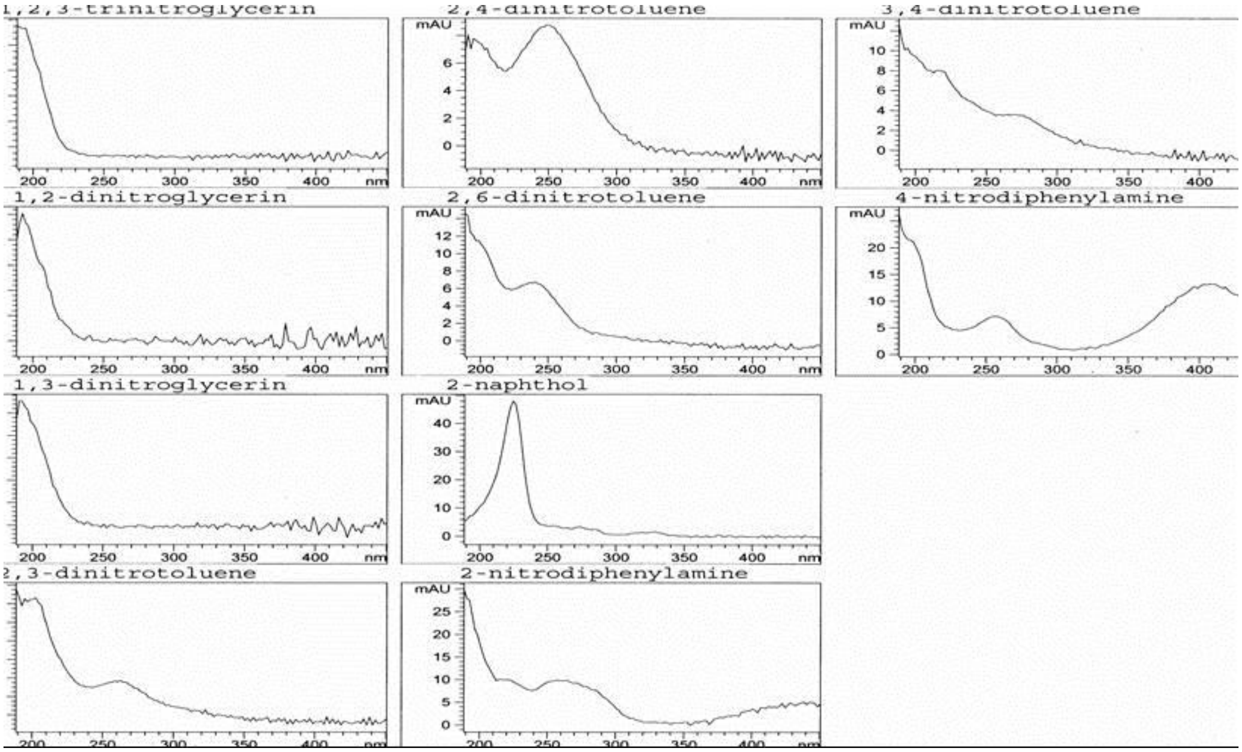
Cation Reference Materials using the CE Cation Method



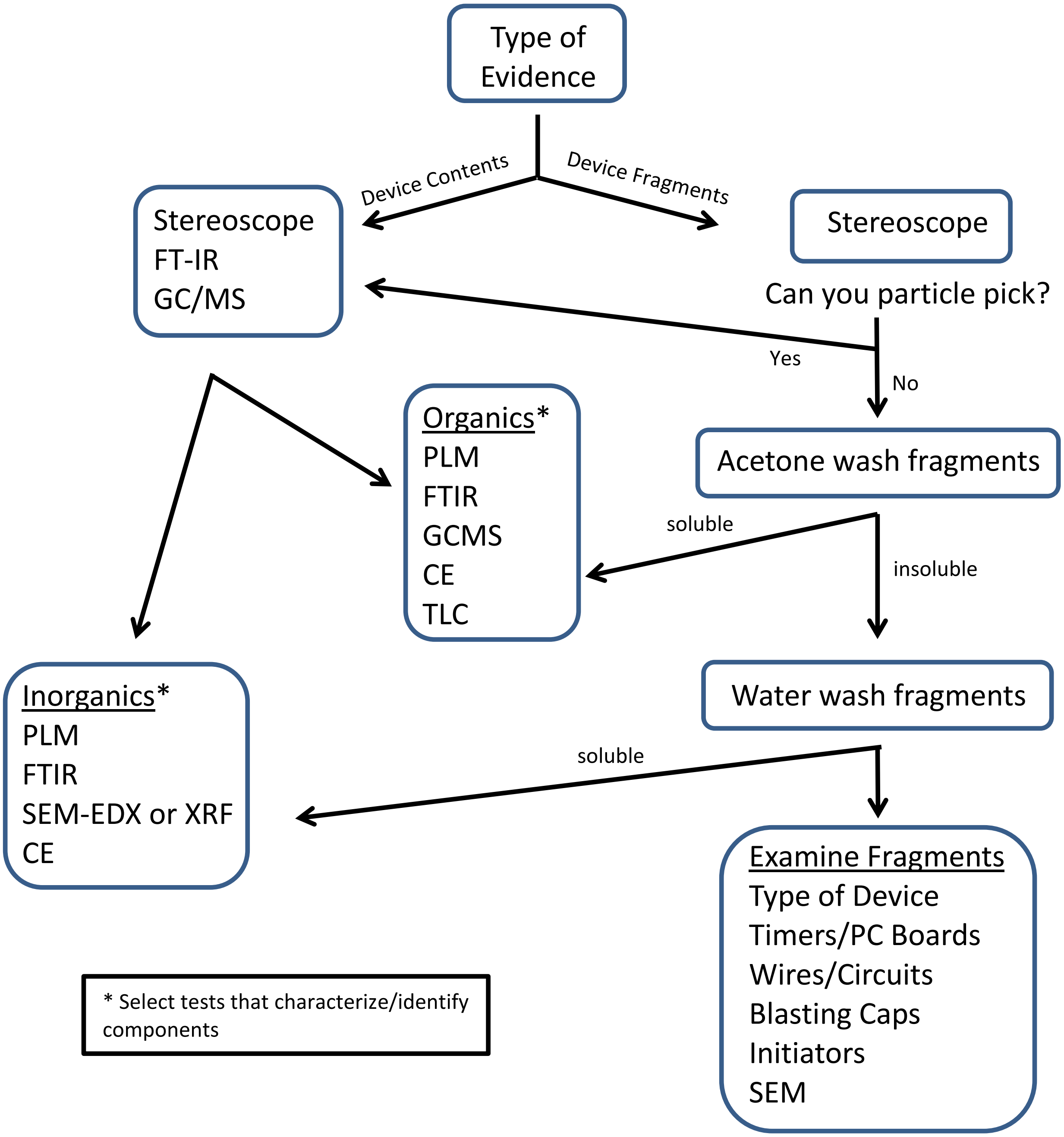
CE of Organic Explosives Reference Materials using SDS Method






1,2,3-trinitroglycerin
2,4-dinitrotoluene
3,4-dinitrotoluene
1,2-dinitroglycerin
2,6-dinitrotoluene
4-nitrodiphenylamine
1,3-dinitroglycerin
2-naphthol
2,3-dinitrotoluene
2-nitrodiphenylamine
mAU
nm
200
250
300
350
400

28 Appendix H: Approach Flow Chart



* Select tests that characterize/identify components

Examine Data and:

- Identify explosive from unreacted material
- Identify explosive reaction products and try to determine the composition of the original mixture
- Try to identify the original device from examination of fragments
- Prepare report

Explosives Training Checklist – Module 1 - Page 1

Trainee:		Trainer:		
		Trainee Initials/Date	Trainer Initials/Date	Time for Completion
Explosives Analysis Overview				
	Reading			
	Exercises			
Color and Microcrystalline Tests for Explosives				
	Reading			
	Exercises			
Recrystallization of Some Common Explosives				
	Reading			
	Exercises			
Optical Properties of Explosives				
	Reading			
	Exercises			
IR of Explosives and Explosive Reaction Products				
	Reading			
	Exercises			
SEM/EDX of Explosives and Explosive Reaction Products				
	Reading			
	Exercises			
µ-XRF of Explosives and Explosive Reaction Products				
	Reading			
	Exercises			
GC/MS of Explosives and Explosive Reaction Products				
	Reading			
	Exercises			
CE of Explosives and Explosive Reaction Products				
	Reading			
	Exercises			
TLC of Explosives and Explosive Reaction Products				
	Reading			
	Exercises			

Explosives Training Checklist – Module 1 - Page 2

Trainee:		Trainer:		
		Trainee Initials/Date	Trainer Initials/Date	Time for Completion
Analytical Approach, Significance, and Report Writing				
	Reading			
	Exercises			

Explosives Training Checklist – Module 2

Trainee:		Trainer:		
		Trainee Initials/Date	Trainer Initials/Date	Time for Completion
Black Powder				
	Reading			
	Exercises			
Black Powder Substitutes				
	Reading			
	Exercises			
Pyrotechnics: Black Powder and Effects				
	Reading			
	Exercises			
Flash Powders				
	Reading			
	Exercises			
Sparklers				
	Reading			
	Exercises			
Fusees/Flares				
	Reading			
	Exercises			
Smokeless Powders				
	Reading			
	Exercises			
Military Explosives				
	Reading			
	Exercises			