

Suggested Guide for Explosive Analysis Training

This is a **suggested** guideline for the training of an examiner in the field of explosive analysis by an appropriately qualified explosives examiner/analyst. If an appropriate instructor is unavailable, the corresponding sections of the training should be sought externally. This training guide may be modified to fit an agency's training requirements and goals. All training must be conducted following proper safety procedures as prescribed by the appropriate agency/organization guidelines, as well as all applicable laws/regulations. When practical and available, coordination with local bomb squads is highly recommended.

All training must include proper documentation upon completion of each section/module. The trainee may not have to complete all sections. The needs and available instrumentation of the agency will dictate the individual training program selected from the following guidelines. However, the trainee needs to be aware of other analytical methods that are utilized elsewhere.

Methods of instruction may include instructions by trainer, self study and/or online guides and/or external instruction.

Method of evaluation may include oral, written and/or practical exercise(s).

Suggested readings may be found for each section in the accompanying bibliography.

I. Introduction

Introduction to Explosives

Objectives:

Upon completion of this unit the student will be able to:

1. Describe the historical development of explosives. (*i.e.* black powder, TNT, smokeless powder, nitroglycerin, pyrotechnics, etc.)
2. Describe the different types of explosives and how they can be classified. Examples of the different classification systems should include: low vs. high, deflagrating vs. detonating, DOT shipping classifications, chemical classifications, etc.
3. Explain legal uses of explosives. (*i.e.* blasting, propellants, military, pyrotechnic, etc.)
4. Explain illegal uses of explosives. (*i.e.* terrorism, Improvised Explosive Devices etc.)

Practical Exercises:

1. None recommended.

II. Chemistry of Explosives

Chemistry of commercially produced Black Powder/Black Powder Substitutes

Objectives:

Upon completion of this unit the student will be able to:

1. Recognize Black Powder and Black Powder substitutes, including particle sizes and morphologies.
2. Describe Black Powder and Black Powder substitute formulations.
3. Explain the Black Powder and Black Powder substitute combustion products.
4. Explain the various mechanisms that can be used to initiate Black Powder and Black Powder substitutes and describe any resulting differences, if any.

Practical Exercises:

1. Examine at least ten samples of Black Powder and/or Black Powder substitutes, classify them by physical characteristics. These characteristics should include but not be limited to: color, texture, luster, particle size, pellets, etc.
2. Make a list of the formulations of Black Powder (*ie.* Goex) and Black Powder substitutes (*ie.* Black Canyon, Pyrodex, Triple 7, Clear Shot, Clean Shot, etc).
3. Make a list of the combustion products of Black Powder and Black Powder substitutes.
4. Burn samples of at least five Black Powder and/or Black Powder substitutes, noting smoke production, gas production, flame color and other characteristics of the burning material, utilizing proper safety procedures. Examine partially burned/consumed particles.
5. Visit and summarize commercial manufacturer websites associated with black powder and black powder substitutes.
6. Visually examine at least five unknowns and classify them as Black Powder and/or Black Powder substitutes.

Chemistry of smokeless powder

Objectives:

Upon completion of this unit the student will be able to:

1. Recognize smokeless powder.

2. Describe the different morphologies and how they relate to speed of burn, for example, disc vs. perforated disc, rod vs. tube, size, etc.
3. Describe the difference between single, double, and triple base smokeless powder.
 - a. Single - nitrocellulose
 - b. Double - nitrocellulose & nitroglycerine
 - c. Triple - nitrocellulose, nitroglycerine & nitroguanidine
4. Describe the different additives that may be present in various types/brands of smokeless powders, as well as their intended purpose.
5. Explain the various mechanisms that can be used to initiate different types of Smokeless Powder and describe any resulting differences, if any.

Practical Exercises:

1. Examine at least five samples of Smokeless Powder and classify them by physical characteristics. This should include at least one sample of each type of morphology.
2. Make a list of common ingredients used in Smokeless Powder and their purpose.
3. Describe the combustion products of Smokeless Powder.
4. Burn samples of at least five different types of Smokeless Powder, noting smoke production, gas production, flame color and other characteristics of the burning material, utilizing proper safety procedures. Examine partially burned/consumed particles.
5. Visit and summarize commercial manufacturer websites associated with smokeless powders.
6. Examine at least five unknowns and classify them as the type of Smokeless Powder.

Chemistry of Pyrotechnics

Objectives:

Upon completion of this unit the student will be able to:

1. Recognize the difference between a pyrotechnic mixture and black powder/black powder substitutes or smokeless powder.
2. Discuss the different oxidizers, fuels and burn modifiers used in pyrotechnic formulations.
3. Discuss combustion products of pyrotechnic mixtures.

4. Describe possible end uses of pyrotechnic mixtures based on their formulation. (for example, strontium nitrate, sulfur, and sawdust are consistent with road fusee or similar device)
5. Be able to describe methods of preparation of various types of pyrotechnic devices including but not limited to: sparklers, M-80's, firecrackers, Roman candles, whistles, stars, fountains, etc.
6. Explain the various mechanisms that can be used to initiate pyrotechnic powders and mixtures and describe any resulting differences, if any.

Practical Exercises:

1. By direct visual examination and a stereomicroscope, examine at least five powders from pyrotechnic devices.
2. Make a list of formulations from the literature of at least five pyrotechnic devices or powders including, but not limited to, flash powder, firecrackers, railroad fusee, road flare, cones/fountains, piccolo/whistling Pete, sparklers, Roman candle, torpedo/snap pops, smokes, and animal control devices (e.g. gopher gassers, etc.)
3. Based on the formulation of pre-blast products of at least five pyrotechnic powders, list the possible post blast products.
4. Burn samples of ten powders separately, noting smoke production, gas production, flame color and other characteristics of the burning material, utilizing proper safety procedures. Examine partially burned/consumed particles.
5. Visit and summarize commercial manufacturer websites associated with pyrotechnics.

Chemistry of Primary High Explosives

Objectives:

Upon completion of this unit the student will be able to:

1. Describe different types and formulations of primary high explosives (i.e. lead styphnate, mercury fulminate, Armstrong's mixture, TATP, HMTD, lead azide).
2. Explain the combustion products of primary high explosives.
3. Describe possible uses of primary high explosives.
4. Be able to describe methods of manufacture/preparation of primary high explosives.

5. Explain the various mechanisms that can be used to initiate primary high explosives.

Practical Exercises: (Due to safety concerns, actual hands-on work should be limited and proper safety precautions must be taken)

1. Make a list of formulations from the literature of at least five primary high explosive materials including, but not limited to lead styphnate, mercury fulminate, Armstrong's mixture, TATP, HMTD, lead azide, etc.
2. Describe safe handling procedures associated with primary high explosives.
3. Provide summaries of websites or other documentation that describes manufacturing procedures for primary high explosives.

Chemistry of Secondary High Explosives

Objectives:

Upon completion of this unit the student will be able to:

1. Discuss the different types and formulations of secondary high explosives to include but not be limited to dynamites, TNT, ANFO, binary, slurries/emulsions, plastic bonded explosives, detonating cord, military explosives, etc.
2. Discuss the combustion products of secondary high explosives.
3. Describe the uses of secondary high explosives.
4. Be able to describe methods of manufacture/preparation of secondary high explosives, including chemical marking agents &/or taggants.
5. Explain what is meant by an explosive train and give examples (both electrical and non-electrical) that could be used to initiate secondary explosives.
6. Discuss issues related to the method of initiation. This discussion should include such topics as low vs. high order detonations, critical diameter, etc.

Practical Exercises:

1. By direct physical examination and using a stereoscope, examine at least five secondary high explosives.
2. Burn samples of five secondary high explosives separately, noting smoke production, gas production, flame color and other characteristics of the burning material, utilizing proper safety procedures. Examine partially burned/consumed particles.

3. Visit and summarize commercial manufacturer websites associated with secondary high explosives.

Improvised Explosive Mixtures

(If possible enlist the assistance of the local bomb squad for supplement demonstrations, lectures, etc).

Objectives:

Upon completion of this unit the student will be able to:

1. Describe and identify chemical components used in improvised explosive mixtures, such as chemical reaction bombs, “home-made” explosive mixtures, etc.
2. Describe how the different types of improvised explosive mixtures react and cause an explosion.
3. Describe the possible reaction products of different improvised explosive mixtures.

Practical Exercises:

1. Appropriate practical exercises may be conducted at the discretion of the trainer.

III. Presumptive Chemical Testing Procedures

Color Spot Tests for Explosives

Objectives:

Upon completion of this unit the student will be able to:

1. Correctly perform a color spot test for various cations and anions.
2. Correctly perform a color spot test for various explosives (e.g., TNT, RDX, etc.) and explosive components (e.g., sulfur, aluminum).
3. Understand the interfering substances and limitations of each color spot test used.
4. Where possible, describe the chemical reaction of the color spot test.

Practical Exercises:

1. Prepare the appropriate reagents and conduct a positive control to ensure reagents are performing correctly.
2. Using various oxidizers, fuels, low explosives and high explosives perform relevant color spot tests.
3. Using various standards or reference materials of post blast products and combustion products, perform relevant color spot tests.

Thin Layer Chromatography

Objectives:

Upon completion of this unit the student will be able to:

1. Be familiar with practical thin layer chromatography.
2. Demonstrate the different retention factors (R_f) for different explosives.
3. Demonstrate the use of visualization methods for explosives.

Practical Exercises:

1. Utilizing single and double based smokeless gunpowders, run two different TLC systems and develop each using appropriate visualization methods.
2. Determine the components present in each and compare to literature data.

3. Utilizing different types of high explosives (example TNT, RDX and PETN), run two different TLC systems and develop each using appropriate visualizing methods.

IV. Microscopy of Explosives

Basic Microscope Knowledge and Particle Manipulation

Objectives:

Upon completion of this unit the trainee will be able to:

1. Name the important parts of a stereomicroscope and polarizing light microscope.
2. Explain the basic theory of optics, resolution, and how magnification is achieved.
3. Describe basic theory of polarized light microscopy and optical crystallography.
4. Setup and align any of the light microscopes used in the examination and analysis of explosives.
5. Handle small particles using the naked eye, stereomicroscope and PLM.

Practical Exercises:

1. Properly setup a stereomicroscope.
2. Properly setup and align a transmitted light microscope (e.g. Kohler illumination).
3. Practice small particle manipulation using the naked eye, stereomicroscope and PLM.

Water Recrystallization Method of Inorganic Oxidizer Identification

Objective:

Upon completion of this unit the trainee will be able to:

1. Recognize common inorganic oxidizers through water recrystallization.

Practical Exercises:

1. The trainee shall recrystallize known standards or reference materials of common inorganic oxidizers. This should include at least the following: NH_4NO_3 , NaNO_3 , KNO_3 , KClO_3 , KClO_4 , $\text{Ba}(\text{NO}_3)_2$ and NH_4ClO_4 .
2. During recrystallization the trainee should note the optical crystallographic properties such as morphology, refractive index (indices), interfacial angles, crystal system, birefringence, optic angle, and optic sign for each of the common inorganic oxidizers.

Fusion Method of Identification of Inorganic Oxidizers

Objective:

Upon completion of this unit the trainee will be able to:

1. Recognize common inorganic oxidizers by using the fusion method.

Practical Exercises:

1. Recrystallize melts of standards or reference materials of common inorganic oxidizers, to include at least the following: NH_4NO_3 , NaNO_3 , KNO_3 , KClO_3 , KClO_4 , $\text{Ba}(\text{NO}_3)_2$ and NH_4ClO_4 .
2. The trainee should note the optical crystallographic characteristics of the fusion preparation during and after recrystallization.

Microchemical Crystal Tests

Objective: Upon completion of this section the trainee will be able to:

1. Perform microchemical crystal tests to identify cations and anions found in common oxidizers.

Practical Exercises:

1. Perform microchemical crystal tests on oxidizers to determine the cations, using the corresponding reagents. These cations should include: K^+ (Chloroplatinic acid), NH_4^+ (Chloroplatinic acid - hanging drop test), Na^+ (zinc acetate/uranyl acetate), & various cations (Squaric acid).
2. Perform microchemical crystal tests on oxidizers to determine the anions, using the corresponding reagents. These anions should include: NO_3^- (Nitron), ClO_4^- (Methylene Blue, Nitron, or Strychnine Sulfate), & ClO_3^- (Nitron or Methylene Blue).
3. The trainee will conduct water extractions on burned and unburned pyrotechnic materials (to include Black Powder, Pyrodex, and flash powder) and perform the microchemical crystal tests on the extracts.

Recrystallization of Sulfur using Suitable Organic Solvents

Objective: Upon completion of this section the trainee will be able to:

1. Recognize sulfur through organic solvent recrystallization.

Practical Exercises:

1. The trainee shall recrystallize sulfur from known standards or reference materials.
2. During recrystallization the trainee should note the optical crystallographic properties such as morphology, refractive indices, interfacial angles, crystal system, and birefringence for sulfur.

Identification of Organic High Explosives

Objective:

Upon completion of this unit the trainee will be able to:

1. Recognize common organic high explosive by PLM techniques.

Practical Exercises:

1. Examine the following preparations found under both the Identification of Organic High Explosives section and in the Recrystallization of Organic High Explosives section and note the characteristic optical crystallographic properties such as morphology, refractive index (indices), interfacial angles, crystal system, birefringence, optic angle, and optic sign for each standard or reference material used in both practical exercises.
2. Prepare temporary and/or permanent microscopical mounts for organic high explosives standards or reference materials such as, but not limited to: TNT, PETN, HMX, RDX, etc.

Recrystallization of Organic High Explosives

Practical Exercises:

1. Organic high explosives standards or reference materials from appropriate solvents such as, but not limited to:
 - A. HMX from acetone
 - B. RDX from nitromethane
 - C. Picric acid from ethanol/water (1:1)
2. Fusion Method of Identifications of Organic High Explosives.
 - A. Prepare fusion melts for organic high explosives such as, but not limited to: TNT, PETN, Tetryl, and HMX.

- B. Prepare a mixed fusion melt for organic high explosives such as, but not limited to: TNT/ Ammonium Nitrate (e.g.: Amatol), Picric Acid/ Thymol, and TNT/RDX (e.g.: Composition B or Military Dynamite).
- C. Recrystallize organic high explosives such as, but not limited to, HMX and RDX, by sublimation.

V. Instrumental Methods

Infrared Spectroscopy (IR/FTIR)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of IR/FTIR and be able to explain the function of the major components of the instrument.
2. Compare and contrast different sampling methods and available IR/FTIR accessories.
3. Explain and be able to perform appropriate performance evaluation procedures and/or quality checks as well as routine instrument maintenance.
4. Prepare samples for analysis choosing the technique most appropriate to the sample. Interpret the results obtained using library searches or comparison to known standards or reference materials and/or spectral subtraction when appropriate.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory and explain the function of each component. These should include, but not necessarily be limited to, the energy source, the optics and the detector.
2. Perform appropriate performance evaluation and/or quality checks before using the instrument.
3. Analyze at least one substance using all IR/FTIR accessories available, and compare the results obtained, ease of analysis, and benefits of each technique.
4. Analyze at least five samples of explosive to include both low and high explosives.

Raman Spectroscopy (RS)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of Raman and be able to explain the function of the major components of the instrument.
2. Discuss the applications of available Raman accessories.
3. Explain and be able to perform appropriate performance evaluation procedures and/or quality checks as well as routine instrument maintenance.

4. Prepare samples for analysis choosing the technique most appropriate to the sample. Interpret the results obtained using library searches or comparison to known standards or reference materials and/or spectral subtraction when appropriate.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory and explain the function of each component. These should include, but not necessarily be limited to, the energy source, the optics and the detector.
2. Perform appropriate performance evaluation and/or quality checks before using the instrument.
3. Analyze at least one substance using all IR/FTIR accessories available, and compare the results obtained, ease of analysis, and benefits of each technique.
4. Analyze at least five samples of explosive to include both low and high explosives.

Gas Chromatography (GC)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of GC and be able to explain the function of the major components of the instrument.
2. Compare and contrast column types used in GC.
3. Discuss the different sample introduction techniques and detectors available for GC.
4. Explain and be able to perform appropriate performance evaluation procedures and/or quality checks as well as routine instrument maintenance.
5. Prepare samples for analysis choosing the technique most appropriate to the sample. Interpret the results obtained using library searches or comparison to known standards or reference materials.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory and explain the function of each component.
2. Perform all appropriate performance evaluation and/or quality checks before using the instrument.

3. Analyze known explosive components such as NG, nitromethane, fuel oil, wax and other suitable materials.
4. Analyze at least five unknown samples by GC, interpret the results, and discuss the limitations of the interpretation.

Gas Chromatography /Mass Spectrometry (GC/MS)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of GC/MS and be able to explain the function of the major components of the instrument.
2. Discuss the available sample introduction and ionization techniques.
3. Explain and be able to perform appropriate performance evaluation procedures and/or quality checks and routine instrument maintenance.
4. Prepare samples for analysis choosing the technique most appropriate to the sample.
5. Interpret the results obtained using library searches or comparison to known standards or reference materials when appropriate.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory and explain the function of each component.
2. Perform all appropriate performance evaluation and/or quality checks before using the instrument.
3. Analyze samples from each type of appropriate explosive available including smokeless powder, high explosives, and other appropriate materials.
4. Analyze at least five unknown samples by GC/MS, run spectral library searches or ion profiling as appropriate and discuss the limitations of the interpretation.

High Pressure Liquid Chromatography (HPLC)/Ion Chromatography (IC)/Capillary Electrophoresis (CE)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of HPLC/IC/CE and be able to explain the function of the major components of the instruments.
2. Explain and be able to perform appropriate quality checks and routine instrument maintenance.
3. Be able to explain the strengths and limitations of the technique and of the different detectors.
4. Prepare samples for analysis, choosing the technique most appropriate to the sample.
5. Interpret the results obtained in comparison to known standards or reference materials.

Practical exercises:

1. Diagram the components of the instrument available in your laboratory, and explain the function of each component.
2. Perform all appropriate calibration and/or quality checks before using the instrument.
3. Analyze appropriate extracts from a variety of known explosive standards or reference materials and explosive residues.
4. Analyze at least five unknown samples by HPLC/IC/CE, compare to standards or reference materials and discuss the limitations of the interpretation.

X-Ray Fluorescence (XRF)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory and instrumentation used in XRF.
2. Explain and be able to perform appropriate performance evaluation procedures and/or quality checks and routine instrument maintenance.
3. Discuss the strengths and limitations of the technique.
4. Prepare samples for analysis, choosing the technique most appropriate to the sample.
5. Interpret the results obtained.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory, and explain the

function of each component. This should include, but not necessarily be limited to, the x-ray source, optics and the detector.

2. Perform all appropriate performance evaluation and/or quality checks before using the instrument.
3. Analyze common explosive materials, combustion products and explosive device components available.
4. Analyze at least five unknown samples by XRF and discuss the limitations of the interpretation.

Scanning Electron Microscope / Energy Dispersive X-ray Spectrometry (SEM/EDS)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of SEM/EDS and be able to explain the function of the major components of the instrument.
2. Explain and be able to perform appropriate quality checks and routine instrument maintenance.
3. Discuss the strengths and limitations of the technique including factors which may effect the resulting spectrum such as escape peaks, sum peaks, peak overlaps, and peak ratio shifts in a spectrum.
4. Prepare samples for analysis choosing the technique most appropriate to the sample.
5. Interpret the results obtained.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory, and explain the function of each component. This should include, but not necessarily be limited to, the x-ray source, optics and the detector.
2. Perform all appropriate performance evaluation and/or quality checks before using the instrument.
3. Analyze common explosive materials, combustion products and explosive device components available.
4. Analyze at least five unknown samples by SEM/EDS, and discuss the limitations of the interpretation.

X-Ray Diffraction (XRD)

Objectives:

Upon completion of this unit the student will be able to:

1. Explain the basic theory of XRD and be able to explain the function of the major components of the instrument.
2. Explain and be able to perform appropriate quality checks and routine instrument maintenance.
3. Be able to explain how sample displacement, preferred orientation, amorphous substances, sample flatness and crystal size can influence the resulting diffraction pattern.
4. Know how sample preparation can compensate for many of these, and be able to use several different sample preparation techniques.
5. Explain the strengths and limitations of the technique.
6. Prepare samples for analysis using a variety of methods.
7. Interpret the results obtained using library searches and/or comparison to known standards or reference materials.

Practical Exercises:

1. Diagram the components of the instrument available in your laboratory and explain the function of each component.
2. Perform all appropriate quality checks before using the instrument.
3. Analyze common explosive materials and combustion products using different sampling techniques available. Compare the results obtained, ease of preparation, and benefits of each technique.
4. Analyze samples from many types of explosive available including black powder and post-combustion black powder, black powder substitutes, and other explosives commonly encountered.
5. Analyze at least five unknown samples by XRD, run library searches, and discuss the limitations of the interpretation.

VI. Device Reconstruction

It is recognized that the different laboratory systems will authorize different levels of device reconstruction determination. It is possible to determine device components without reconstructing an actual device.

Device Reconstruction

In an ideal situation it is highly recommended that the bomb squad and laboratory personnel establish a mutually beneficial working relationship.

Objectives:

Upon completion of this unit the student will be able to:

1. Recognize device components to include, but not be limited to:
 - a. firing train components.
 - i. electrical
 1. timers, switches, cellphones, remote control devices, etc.
 - ii. non-electrical/mechanical
 1. timers, hobby fuse, safety fuse, shock tube, etc.
 - b. containers
 - i. pipe nipples, end caps, fittings, etc.
 - ii. cardboard tubes, glass objects, plastic bottles, CO2 cartridges, or any other type of unconventional packaging (fire extinguishers, duct tape, flashlights, etc.)
 - c. misc. parts
 - i. Solder, tape, glue, etc.
2. Describe the different components used in explosive devices and possible device configurations.

Practical Exercises:

1. Under the supervision of qualified EOD personnel, observe the construction of devices and recover device debris after initiation. These devices should include different types of initiating systems including cannon fuse, safety fuse and improvised initiators to include both electric and nonelectric types.
2. Observe and document all surviving device components.
3. Document all unaccounted device components.
4. If possible examine a minimum of five different devices of known construction in post-blast condition. The devices must be constructed utilizing different types of containers, construction and explosive filler.

5. Describe the characteristics of the remaining device components. These characteristics can include visual observations, odors, measurements, etc.

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