Forensic Science Lab Manual



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Table of Contents

Lab: Classification of Fingerprints1
Exercise 1: Classifying Individual Prints
Exercise 2: FBI Cards
Exercise 3: NCIC and Henry Classification Systems
Exercise 4: The FBI Needs Help!
Lab: Dusting and Lifting Fingerprints
Exercise 1: Creating Prints
Exercise 2: Dusting and Lifting Prints
Lab: Latent Detection Methods
Exercise 1: Developing Latent Fingerprints with IKI Fuming
Exercise 2: Developing Latent Fingerprints with Ninhydrin
Exercise 3: Developing Latent Fingerprints with Cyanoacrylate
Lab: Analysis of Glass
Exercise 1: Macroscopic Observations
Exercise 2: Fracture Patterns
Exercise 3: Density of Glass
Exercise 4: Testing Refractive Index
Lab: Arson Investigation
Exercise 1: Arson Investigation
Lab: Explosives and Explosive Residue
Exercise 1: Testing for Explosive Residues

EXPERIMENT

Arson Investigation

Author Hands-On Labs, Inc. Version 42-0171-00-01

Review the safety materials and wear goggles when working with chemicals. Read the entire exercise before you begin. Take time to organize the materials you will need and set aside a safe work space in which to complete the exercise.

Experiment Summary: Students will become familiar with arson investigation, arson chemistry, and the basic concepts of gas chromatography used to analyze the evidence collected in arson cases. Students will solve a simulated arson case by matching hypothetical crime scene gas chromatograms against a database of chromatograms.

Objectives

- To understand the basic chemistry of combustion; including the fire triangle
- To become familiar with the classification of arson accelerants
- To become familiar with basic gas chromatography concepts
- To become familiar with gas chromatograms of commonly used accelerants used in arson cases
- To match hypothetical crime scene chromatograms against a chromatogram data base



Materials

MATERIALS	QTY	ITEM DESCRIPTION	
LabPaq Provides	1	Chromatograms set, commonly used arson accelerants	
		See the file on your CD: <u>46-0025-00-01 SUP, Chromatograms Set.pdf</u>	

Discussion and Review



Figure 1: Total fire engulfment of structure; this is rarely seen in regular house fires.

Figure 2: Arson fire.

Photos courtesy of Richard Tewes

Arson is defined as intentionally setting fire to a house, building, or other property.

The chemical reaction involved in arson cases is a combustion reaction which needs three components to work: fuel, oxygen, and sufficient heat. An example of a combustion reaction is the burning of propane (C_3H_8) : $C_3H_8 + O_2 CO_2 + H_2O$. When fuel (propane) and oxygen (O_2) are present, only a source of heat (such as a match or electricity) is needed for combustion to take place. A fire can be stopped by removing one or more of the components. Water on a fire may remove the heat; aqueous foam, CO_2 , dry chemicals, or a fire blanket may remove the oxygen; mechanically removing unburned combustible material, such as a fire break for a wildfire, removes the fuel.

Fuels can be solids (wood), liquids (gasoline, kerosene), or gases (natural gas, propane). In arson investigations, **accelerants** are solids, liquids, or gases which initiate and enhance flammability. Often, the term *accelerant* is used interchangeably with fuel.

A **flammable liquid** is a liquid that will easily ignite under normal conditions. The vapors given off by flammable liquids will easily ignite when a spark or lighted match is present as an ignition source. **Flash point** is the temperature at which a flammable liquid vaporizes and can ignite if a spark is present. **Ignition temperature** is the temperature necessary for a liquid to give off vapors that can sustain combustion. Examples of ignition temperatures are 536°F for gasoline, 410°F for kerosene, and 494°F for diesel fuel.

In arson, liquid materials are typically used as accelerants because of their ease of ignition and wide availability. Accelerants are nearly always derived from hydrocarbons which are chains of carbon atoms surrounded by hydrogen atoms. Gasoline, methane, propane, and octane are examples of familiar hydrocarbons.

Classification of Accelerants: (from the American Society for Testing)

- 1. Light petroleum distillates (LPD). Examples: lighter fluid, some camping fuels.
- 2. Gasoline.
- **3. Medium petroleum distillates (MPD).** Examples: charcoal starters, paint thinners, mineral spirits, dry-cleaning solvents.
- 4. Kerosene. Examples: aviation fuel, lamp oil.
- 5. Heavy petroleum distillates (HPD). Examples: diesel fuel, heating oil.
- **6. Miscellaneous.** Examples: oxygenated solvents, normal alkanes, isoparaffins, aromatic solvents, and naphthenic/paraffinic solvents.

Arson investigation



Figure 3: Arson crime scene.

Figure 4: Point of origin; in this case, an electric fire.



Figure 5: Pour pattern.

Figure 6: Video evidence.

Photos courtesy of Richard Tewes.

The Arson Crime Scene

When examining a potential arson location, investigators will look for evidence that arson may have been involved. Empty cans or containers located at or near the scene may have trace amounts of liquid in them, and need to be secured as evidence. There may be ignition devices at the crime scene, such as lighters, matches, rags, or candles. Investigators always look for fingerprints, footprints, tool marks, and blood. There may be an odor of petroleum products, paint solvent, or similar flammable liquids at the scene; there may be floor stains, and unusual burn patterns. Often, there is evidence of multiple points of fire origin, where accelerants have been used and ignited at different places within a structure, like the office, and two or three separate locations in a warehouse. Other important components of arson investigation are burn patterns, burn pattern geometry, and accelerant pour patterns. Because arson fires typically involve accelerants, they burn faster and hotter than accidental fires, and structures are often completely engulfed by flames (Figures 1 and 2). In accidental fires, the fire damage is frequently more limited in scope. The color of flames may be an indicator of the fire temperature – arson fires tend to burn hotter. Typically, the lighter the flame color the higher the temperature (Examples: dark red flame: 900–1000°F; dark cherry red: 1100 – 1200°F; bright red: 1400–1500°F; orange: 1700–1800°F; white: 2150–2250°F. Data from David R. Redsicker & John J. O'Connor (1986).

Fires burn upward in a V-shape and frequently such V-shaped patterns can be seen on the walls of burned structures, where the bottom of the V points to the fire source (Figure 4). Generally, the wider the V is, the slower the fire has been burning. Often, accelerant-induced fires have a narrow V. A U-shaped pattern may indicate a "pool" of origin, like a pool of gasoline, rather than a "point" of origin.

Temperatures at floor level are normally below ignition temperature, so accidental fires usually result in very little floor charring, Fires resulting from gasoline (or other accelerants poured on the floor) will usually result in extensive floor charring. Other indicators of the use of flammable liquids may be the presence of V-burns between floor boards. Accelerants may collect between floor boards, and result in small, sharp "V" burn patterns between the floorboard edges. When liquid accelerants are poured, post-burn pour patterns may also be visible on the floor.(Figure 5)

Table 1: Sample tools and methods used to locate and identify accelerants		
ТооІ	Function	
Hydrocarbon Detectors - "Sniffers"	Detect hydrocarbon vapors and help locate possible sources of ignitable liquids.	
Gas Chromatography	Analyze liquid or gaseous samples to determine the proportion of different chemical components present in evidence.	
Mass Spectrometry	Identify and quantify the atoms present in a molecule. Used in conjunction with gas chromatography	
Dogs	Trained to detect the odor of common accelerants. Can detect 0.01 mL of 50% evaporated gasoline 100% of the time. (0.01 mL is roughly the size of a thousandth of a drop.)	

Ignitable liquid residues help forensic scientists determine that a fire was intentionally set, but seldom help to identify the arsonist. Investigators use a variety of tools and methods to locate and identify accelerants used in arson. See Table 1.

Gas chromatography (GC) and mass spectrometry (MS): Gas chromatography is a common technique used to separate gaseous components of a mixture. A small amount of gaseous or liquid sample is injected into the heated injection port of the gas chromatograph. If a liquid

sample is injected, the elevated temperature of the injection port will vaporize it into its gas phase. A *carrier gas* such as helium or argon transports the gaseous material to be analyzed through the GC column, The various components of the injected compound travel at different speeds through the stationary phase and therefore arrive at a Thermal-Conductivity Detector (TCD) or Flame-Ionization Detector (FID) at different times.

From the detector, the signal is sent to a chart printer where it is recorded as a peak. The area under the peak is directly related to the quantity of component molecules passing through the detector. Once a specific compound has passed through the GC, a gas chromatogram is produced which is characteristic for that compound. Ignitable fluids such as gasoline or diesel fuel are combinations of various hydrocarbons and other compounds and produce a very complex pattern of peaks. Figure 7 is a chromatogram of diesel fuel. The peaks represent the various components present; some of them have been identified to demonstrate the concept.



Figure 7: Gas chromatogram of diesel fuel. The numbered peaks represent the following constituents: 1: n-tridecane; 2: n-tetradecane; 3: n-pentadecane; 4: n-heptadecane; 5: n-octadecane (The x-axis shows retention time (RT); y-axis shows intensity of signal, that is, the abundance of respective constituent.)

Gas chromatograms from evidence samples can be compared to those in a chromatogram library to give an indication of the type of accelerant used. However, the gas chromatogram should not be considered an identification technique like fingerprint matching. In fire debris samples, many of the most volatile components of the accelerant are usually lost and will not be present in the chromatogram.

Typically, a gas chromatograph (Figure 8) is coupled with a mass spectrometer (MS), so that individual peaks on the gas chromatogram can be analyzed by the mass spectrometer (Figure 9). As the sample mixture passes directly from the gas chromatograph into the mass spectrometer, it is bombarded by high energy electrons. Each compound is separated into its atomic components in the form of ions and their masses and abundance are determined. The resulting mass spectrum is used to analyze complex samples which cannot be identified by chromatogram pattern recognition alone (Figure 10).



Figure 8: Gow-Mac gas chromatograph. Series 400 TCD Gas Chromatograph photo provided by GOW-MAC Instrument Co., Bethlehem, PA USA



Figure 9: Schematic diagram of gas chromatograph-mass spectrometer (GC-MS).



Figure 10: Mass spectrum of n-octane peak within a chromatogram. Courtesy of U.S. Department of Labor; Occupational Safety & Health Administration.

References

Redsicker, D. R. and O'Connor, J. J. (1986). Practical Fire and Arson Investigation. Elsevier Science Publishing.

Case History

At 3:15 a.m. on April 30, 2010, a fire broke out at the Sheepskin Factory in Glendale, Colorado. The Sheepskin Factory sold a variety of products made of sheepskin, such as seat covers, shoes, hats, rugs, and boots. The fire destroyed the building, causing more than \$500,000 in damages.

In the days following the fire, a message was posted on the Animal Liberation Front's website by someone named "ALF Lone Wolf," claiming responsibility for the fire, saying it had been done "in defense and retaliation for all the innocent animals that have died cruelly at the hands of human oppressors."

The FBI, Denver Fire Department, the Glendale Police Department, and the ATF (Bureau of Alcohol, Tobacco, Firearms, and Explosives) were all assigned to the case and began to review surveillance videos from security cameras in the immediate area. What they found was that standing outside of the building, just minutes before the fire began, was a man carrying a backpack and wearing a dark-hooded, long-sleeve shirt. While the fire was ruled as arson, the ATF were not able to identify the man in the video until July 1 when an informant provided a tip on the suspect. The informant met claimed that a man named Walter Edmund Bond, aka Walter Edmund Zeuhlke, and The Lone Wolf, had claimed responsibility for the fire. Bond was arrested later that day at a Northglenn, Colorado home.



Walter Edmund Bond, aka "The Lone Wolf"- convicted arsonist and "parttime" vegan.

As more information was discovered about the suspect, it was learned that Bond was a parttime vegan, having eaten hamburgers two days before the arson at a friend's house and had been previously jailed for a 1997-house fire in Mason city, Iowa. It was also learned that Bond was under investigation in Utah for two arson attacks: a fire at a leather factory and arson at a restaurant which served foie gras. Bond was officially charged of the crime on July 23, 2010 and indicted on July 27, 2010. While he initially pled not guilty, on November 18, 2010, Bond changed his plea to guilty and stated that he had no remorse for setting the fire. Bond was sentenced to a five-year prison term and responsible for paying nearly \$1.2 million in restitution. The Sheepskin Factory has since reopened at a nearby location and Bond has stated that when he is released from prison he will again set fire to the Sheepskin Factory.

Exercise 1: Arson Investigation

A team of crime scene investigators was called to a burned-out warehouse as arson was suspected. An examination of the premises showed evidence of several points of origin and there was unmistakable odor of petroleum in the air. The investigators used a hydrocarbon-sniffer to locate several areas of higher hydrocarbon concentrations and they collected several charcoal samples for GC analysis.

After investigating the background and circumstances of the fire, as well as reviewing the background of the case, three potential suspects were identified.

- The owner: The owner had bought the warehouse several years ago. He and his company had
 a good reputation, but due to the worsening economy, the business was not doing well. The
 property was somewhat under-insured, and there was no evidence of additional insurance
 purchased recently.
- The warehouse manager: On a background check, it was discovered that the manager had managed a warehouse operation for another company several years ago, and that warehouse also burned down, though arson was not suspected.
- A recently laid-off warehouse worker: Due to the declining economy and resulting reduction in business, the company had to lay off an employee who was very upset about losing his job. He felt that with a little belt-tightening, the company could have survived the financial crisis and that there was no need to let him go.

Using search warrants, the investigators searched the houses of all three suspects. Various cans and containers of potential accelerants were collected from each home.

These were sent to the crime lab for GC analysis.

- 1. Study the gas chromatograms from the samples obtained from the suspects' homes.
- 2. Compare those with the database of reference chromatograms:

Chromatogram Database of Common Flammable Liquids

BP Regular Unleaded gasoline









Phillips 66 Regular Unleaded gasoline

Chevron Aviation Gasolin 100 LL



Klean Strip Klean Kutter Remover



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Klean Strip Lacquer Thinner



EZ Lacquer Thinner



Sunnyside Lacquer Thinner



Klean Strip Turpatine







WD-40





Canadian Northern Charcoal starter









Condea Vista LPA Solvent



STP Gas Treatment





STP Diesel Fuel Treatment



Penske Fuel Injector/Carburetor cleaner









Sunnyside Kerosene







Crime scene chromatograms:

Chromatogram of accelerant found at the warehouse:



Chromatogram of flammable liquid found at the owner's home:







Chromatogram 2 of flammable liquid found at the manager's home:





Chromatogram of flammable liquid found at the worker's home:

Chromatogram Database of Common Flammable Liquids:

See the file on your CD: 46-0025-00-01 SUP, Chromatograms Set.pdf

Arson Investigation

Author Hands-On Labs, Inc. Version 42-0171-00-01

Lab Report Assistant

This document is not meant to be a substitute for a formal laboratory report. The Lab Report Assistant is simply a summary of the experiment's questions, diagrams if needed, and data tables that should be addressed in a formal lab report. The intent is to facilitate students' writing of lab reports by providing this information in an editable file which can be sent to an instructor.

Questions

A. What flammable liquid/accelerant was used at the arson site?

- B. What flammable liquid was found at the owner's house?
- C. What flammable liquid was found at the manager's house?
- D. What flammable liquid was found at the worker's house?

E. Does the presence of charcoal lighter fluid in the worker's home automatically make him the primary suspect?

EXPERIMENT

Explosives and Explosive Residues

HandsOnLabs, Inc.-consulting&content contribution from: Richard Tewes, CSCSA, CFWE, CBPE Version 42-0170-00-02

Review the safety materials and wear goggles when working with chemicals. Read the entire exercise before you begin. Take time to organize the materials you will need and set aside a safe work space in which to complete the exercise.

Experiment Summary:

Students will review the chemistry of explosives and explosive residues and will perform spot tests on "explosive residue" samples for the presence or absence of ions often found in explosives, such as aluminum, nitrate, and nitrite ions.



Objectives

- To review the chemistry of most common explosives
- To perform chemical tests for the presence or absence of ions commonly found in explosive residues

Time Allotment: 2 hours



Materials

MATERIALS	QTY	ITEM DESCRIPTION
Student Provides:	1	Distilled water
LabPaq Provides:	1	0.5 M Potassium nitrite, KNO ₂ – 2mL in pipet
	1	0.1 M Aluminum nitrate - Al $(NO_3)_3$ – 2mL in pipet
	1	Unknown #1– 2mL in pipet
	1	Unknown #2– 2mL in pipet
	1	0.1 M Iron II sulfate – 2mL in pipet
	1	1 M Sulfuric acid – H ₂ SO ₄ -2mL in dropper bottle
	1	Griess Reagent A – 2mL in dropper bottle
	1	Griess Reagent B – 2mL in dropper bottle
	1	Aluminon reagent – 2mL in pipet
	1	3 M Ammonium acetate – 2mL in pipet
	1	3 M Aqueous ammonia – 2mL in dropper bottle
	1	Porcelain spot plate – 6 wells
	1	Empty pipet, 4 mL

Note: The packaging and/or materials in this LabPaq may differ slightly from that which is listed above. For an exact listing of materials, refer to the Contents List form included in the LabPaq.

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Discussion and Review

Explosives are materials that undergo rapid exothermic oxidation reactions that produce large amounts of gas. Because these reactions take place in confined spaces, the gas pressure rapidly increases, causing the explosion. Detonation refers to the ignition of an explosive device.

Most bombings involve an Improvised Explosive Device (IED) (Figure 1). Explosions from these homemade bombs will produce characteristic damage to the surrounding area and will leave debris consisting of bomb components and fragments of surrounding objects and structures. As part of an investigation, forensic scientists analyze the debris and explosive residues through chemical and other means to determine which type of explosive was used in a particular incidence.



Figure 1: Unexploded IED. Courtesy of Richard Tewes.

Pyrotechnics are explosives that produce smoke, light, heat, and/or sound. They are occasionally used in bombs and arson, and include fireworks, flares, and matches. The combustion of *propellants* produces a controllable release of gas which can be used to propel a bullet or a rocket. Examples of propellants include gunpowder, solid and liquid rocket fuels, and gasoline. When confined within a small space such as a metal container, propellants will rupture the container and disperse shrapnel and can therefore be used in simple bombs. There are two main categories of explosives: **Low explosives** have a combustion speed that is lower than the speed of sound. **High explosives** have a speed of combustion faster than the speed of sound causing an almost instantaneous energy release and a wide dispersal of shrapnel. Examples of high explosives include nitroglycerin, TNT, and C4.

Table 1.

Explosive Category	Explosive	Major components
Low explosives	black powder	potassium nitrate, charcoal, sulfur
	smokeless powder:	
	single base –	Nitrocellulose (NC)
	double base –	NC + nitroglycerin (NG)
	triple base –	NC + NG + nitroguanidine
High explosives:	nitroglycerin	1,2,3-trinitroxypropane
Primary	mercury fulminate	Hg(CNO) ₂ - Mercury in nitric acid + ethanol
	silver azide	AgN ₃₋ colorless solid
	ТАТР	triacetone triperoxide
High explosives:	dynamite	NG, ammonium and sodium nitrate, dinitrotoluene, etc.
Secondary	DNT	Dinitrotoluene
	TNT	2.4.6-Trinitrotoluene
	PETN	Pentaerythritol pentanitrate
	RDX	cyclotrimethylene-trinitramene
	НМХ	cyclotetramethylene-
		tetranitramene
	C4	RDX + diethylhexyl (plasticizer) + mo- tor oil
	Tetryl	2,4,6-trinitrophenylmethylnitramine
	Tovex (water gels)	aqueous ammonium nitrate + methyl- ammonium nitrate
	Semtex	PETN + RDX
High explosives:	ANFO	ammonium nitrate + fuel oil
Tertiary	ANNM	ammonium nitrate + nitromethane

Primary explosives are highly reactive to shock, heat, and friction, and thus easily detonated. Nitroglycerin, mercury fulminate, and silver azide are examples. They are often used as primers to detonate a main explosive charge. **Secondary explosives** are less sensitive to heat and shock and can withstand rough handling. They require an *initiator* such as a blasting cap or electronic match to detonate. Dynamite, TNT, RDX, PETN (Pentaerythritol tetranitrate), and C4 are all examples of secondary explosives.

Experiment EXPLOSIVES AND EXPLOSIVE RESIDUES

Dynamite is an explosive frequently used in explosive devices. Originally developed by Alfred Nobel, it consists of three parts nitroglycerine, one part diatomaceous earth, and a small amount of sodium carbonate. (The addition of the diatomaceous earth stabilizes nitroglycerine (which is highly unstable otherwise). Originally, dynamite was then formed into sticks, wrapped in paper, and a fuse was attached. In modern dynamite, referred to as ammonia dynamite, ammonium and/or sodium nitrate and various carbon-based fuels are substituted for part of the nitroglycerin. The fuse has been replaced by electronic detonators.

Finally, **tertiary explosives** require the detonation of a secondary explosive to initiate detonation because they are so insensitive to shock. Tertiary explosives are commonly used in mining, construction, and terrorism. They include ammonium nitrate, ammonium perchlorate, and mononitrotoluene. ANFO (ammonium nitrate and fuel oil) is a common example. Timothy McVeigh and Terry Nichols used ANFO and ANNM (ammonium nitrate and nitromethane, a modified ANFO) in the Oklahoma City bombing of the Murrah Federal Building (Figure 2).



Figure 2: Oklahoma City federal building partially destroyed by an ANFO truck bomb. Courtesy of US Army Corps of Engineers.

Improvised explosive devices use whatever explosive agents that are readily available. For example, the pyrotechnic mixture in road flares or potassium chlorate from fireworks can be sealed in a container to create a pipe bomb. HMTD (hexamethylene triperoxide diamine) and TATP (triacetone triperoxide) are both highly sensitive primary explosives that are employed in terrorist attacks, such as suicide bombings. The highly reactive nature of TATP has earned it the nickname "Mother of Satan" by some terrorist groups. Hypergolic devices take advantage of the violent reaction produced when certain chemicals are combined, for example, sugar and sulfuric acid; potassium permanganate and hydrogen peroxide, or potassium permanganate and glycerin. Aluminum powder is often added to explosives to increase total energy and explosive effect. In current and former war zones, unexploded ordnance (UXO) is obtained from the millions of tons of bombs which have been dropped into the battlefield and failed to detonate. UXO is one of the most commonly-used sources of explosives found in IEDs in war zones.

Explosive device components

In addition to an explosive compound, explosive devices generally consist of the following components: power source, initiator, and trigger or switch. Many explosive devices contain an electric initiator, and therefore, require a power source, typically a battery. Initiators include safety fuses, electric matches, blasting caps, modified flash bulbs, or anything that can produce heat, sparks, flames, or small explosions. Triggers or switches are the means by which explosive devices are set off. When the switch is activated, the circuit is closed, the battery is connected with the initiator, and the detonation takes place. See examples of explosive devices in Figure 3 and 4.



Figure 3: Unexploded pipe bomb. Courtesy of Richard Tewes.



Figure 4: Unexploded IED with circuit board. Courtesy of Rich Tewes.

Laboratory analysis of explosives

An explosion can generate large quantities of evidence: blast crater, bomb components (Figure 5), timer, casing, explosives residue, and vehicle parts (if a vehicle bomb was used). All of these are valuable to the forensic investigator.



Figure 5: Recovered bomb components after explosion. Courtesy of Richard Tewes.

The most common methods of explosive residue analysis include macroscopic and microscopic examination, using optical and scanning electron microscopy (SEM) on bulk samples, and transmission electron microscopy (TEM) on thin sections; wet chemical analyses via chemical spot tests and TLC (thin-layer chromatography), instrumental analyses via X-ray diffraction, and various forms of spectroscopy including gas chromatography/mass spectroscopy (GC/MS).

Experiment EXPLOSIVES AND EXPLOSIVE RESIDUES

A scanning electron microscope (SEM) produces a magnified image by scanning a sample with a high-energy electron beam. The resulting magnification can be as high as 500,000x and allows for a very detailed examination of the sample. Transmission electron microscopy (TEM) is similar to scanning electron microscopy, except that the electron beam is transmitted through a thinsection of the sample.

Thin-layer chromatography (TLC) is a simple chemical technique that can separate a mixture into its various components. An absorbent-coated glass or plastic sheet is spotted near the base with a small sample to be separated. The base of the plate is placed into a solvent-containing development chamber and the upward movement solvent will separate the compound mixture into its components. (This technique is very similar to paper chromatography, where chromatography paper is used in place of the coated plastic or glass plate.)

Gas chromatography (GC) and mass spectrometry (MS): Gas chromatography is a common technique used to separate gaseous components of a mixture. A small amount of gaseous or liquid sample is injected into the heated injection port of the gas chromatograph. The various components of the injected compound travel at different speeds through the stationary phase and therefore arrive at a thermal-conductivity (TCD) or flame-ionization (FID) detector at different times. From the detector, the signal is sent to a chart printer where it is recorded as a peak. Often, a gas chromatograph is coupled with a mass spectrometer (MS), so that individual peaks on the gas chromatogram can be analyzed by the mass spectrometer.

Thermal desorption gas chromatography is a specialized from of gas chromatography, where a vapor sample is passed through a heated thermal desorption tube before entering the gas chromatograph. Figure 6 is an example of a gas chromatogram of an explosives standard.



Figure 6: Thermal desorption GC/MS spectrum of an explosives standard containing the following explosives (using a capillary column): 1. Nitrobenzene; 2. 2-nitrotoluene; 3. 3-nitrotoluene; 4. 4-nitrotoluene; 5. 2,6-dintrotoluene; 6. 1,3-dininitrobenzene; 7.
2,4-dinitrotoluene; 8. TNT; 9. 1,3,5-trinitrotoluene; 10. 4-amino-2,6-dinitrotoluene; 11. 2-amino-4,6-dinitrotoluene. Data from *Shomo et al. 2007*.

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For example, the black powder reaction, $10KNO_3 + 8C + 3S \rightarrow 2K_2CO_3 + 3K_2SO_4 + 6CO_2 + 5N_2 + heat$, creates a residue consisting primarily of potassium sulfate and potassium carbonate. A typical infrared spectrum of black powder residue will show weak nitrate and strong sulfate peaks.

Many homemade bombs contain aluminum and nitrates. Smokeless powders typically produce nitrites, therefore, a nitrate/nitrite spot test may easily verify the presence or absence of these compounds.

Case History

On February 23, 1993, at 12:17 p.m., a terrible explosion occurred in lower Manhattan, New York City. The explosion was the result of a truck bomb which was composed of approximately 1500 pounds of urea nitrate and hydrogen-gas cylinders. The explosion occurred in the parking garage under the World Trade Center, instantly killing six people and injuring thousands more. The explosion left a 100-foot diameter crater and massive rubble surrounding the World Trade Center. The investigators in the crime determined that the bomb was homemade and was built in New Jersey by Islamic radicals, four of whom remain in United States prisons, serving life sentences.



Exercise 1: Testing for explosive residues

This lab exercise will simulate the chemical analysis of explosives residue. Low explosives residue may contain nitrate ions, nitrite ions, and/or aluminum ions, whose presence can easily be identified through a variety of chemical tests. Only a sampling of potential tests will be used, as many common reagents used in the identification of explosives residue are too hazardous to be shipped or used at home.

In the following scenario, a bomb has exploded outside the residence of a corporate executive. The house was partially destroyed, but there was no loss of life and no bodily injuries. From the destruction done to the house, it seems that low explosives were involved. Several pieces of debris were collected by the crime scene investigator and potential explosive residues were isolated and sent to the lab for analysis. It is your job to identify the ions within each sample. Because the preliminary evidence points to low explosives, you might expect to identify ions like aluminum, nitrate, and nitrite.

You will first analyze samples known to contain aluminum, nitrate, and nitrite ions to become familiar with the results of positive tests. Then you will analyze the unknown samples from the crime scene to determine the presence or absence of those ions.

Procedures

Nitrite ion test using iron II sulfate

- 1. Using the porcelain spot plate, add two drops of 0.5 M potassium nitrite (KNO₂) solution to one of the wells.
- 2. Use the empty pipet to add two drops of distilled water to a second well as a control.
- 3. Add one drop of $1M H_2SO_4$ to both wells.
- 4. Add one drop of 0.1 M iron II sulfate to both wells.
- 5. Observe and record the color of the solution in Data Table 1.

Data Table 1			
Test	Color of test solution	Color of water control	
Nitrite ion test using iron II sulfate			
Nitrite test using Griess reagent			
Nitrate test using Griess reagent			
Aluminum ion test using aluminon reagent			

6. Clean the spot plate. Rinse with distilled water (tap water often has contaminants that may result in false positives).

Nitrite and nitrate test using Griess reagent

- 7. Using the porcelain spot plate, add two drops of 0.5 M potassium nitrite (KNO₂) solution to one of the wells.
- 8. Add two drops of distilled water to a second well as a control.
- 9. Add one drop of Griess reagent A to both wells.
- 10. Add one drop of Griess reagent B to both wells.
- 11. Observe and record the color of the solution in Data Table 1.
- 12. Clean the spot plate for the next test. Rinse with distilled water.

Nitrate test using Griess reagent

- 13. Using the porcelain spot plate, add two drops of 0.1 M aluminum nitrate $(Al(NO_3)_3)$ solution to one of the wells.
- 14. Add two drops of distilled water to a second well as a control.
- 15. Add one drop of Griess reagent A to both wells.
- 16. Add one drop of Griess reagent B to both wells.
- 17. Observe and record the color of the solution in Data Table 1.
- 18. Clean the spot plate for the next test. Rinse with distilled water.

Aluminum ion test using aluminon reagent

- 19. Using the porcelain spot plate, add two drops of 0.1 M aluminum nitrate $(Al(NO_3)_3)$ solution to one of the wells.
- 20. Add two drops of distilled water to a second well as a control.
- 21. Add two drops of 3 M ammonium acetate to both wells.
- 22. Add two drops of aluminon reagent to both wells.
- 23. Add one drop of 3 M aqueous ammonia to both wells.
- 24. Observe and record the color of the solution in Data Table 1.
- 25. Clean the spot plate for the next test. Rinse with distilled water.

Analysis of unknowns

- 26. Using the porcelain spot plate, add two drops of Unknown #1 solution to one of the wells, add two drops of Unknown #2 to a second well and add two drops of distilled water to a third well.
- 27. Follow steps 3 6 to test for the presence of nitrites. Record your results in Data Table 2.

Data Table 2			
Test	Test result (positive or negative)		
Unknown #1 – nitrite with iron II sulfate			
Unknown #1 – nitrite with Griess reagent			
Unknown #1 – nitrate with Griess reagent			
Unknown #1 – aluminum			
Unknown #2 – nitrite with iron sulfate			
Unknown #2 – nitrite with Griess reagent			
Unknown #2 – nitrate with Griess reagent			
Unknown #2 – aluminum			

- 28. Repeat step 26, and then follow steps 9 12 to test for the presence of nitrites. Record your results in Data Table 2.
- 29. Repeat step 26, and then follow steps 15 18 to test for the presence of nitrates. Record your results in Data Table 2.
- 30. Repeat step 26, and then follow steps 21 24 to test for the presence of nitrates. Record your results in Data Table 2.
- 31. Clean up: Wash out the well plate and return all supplies to your LabPaq for future use.

References

Ronald E. Shomo, Robert Frey, and John J. Manura, 2007, Detection of explosives on clothing material by direct and air sampling Thermal Desoprtion GC/MS – Presented at American Society for Mass Spectroscopy conference, 2007.

Questions

- A. Give two reasons why nitrite and nitrate tests are included in this exercise?
- B. Why did we test for the presence of aluminum?
- C. Why is nitroglycerin rarely used as the main explosive charge?
- D. Why has ANFO been used in many explosive devices?
- E. How do positive results of Griess nitrite tests differ from positive Griess nitrate tests.
- F. What is the primary difference between "low" and "high" explosives?