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Identification of Explosive Residues in Bomb Scene Investigations

Only a few years ago, a bombing or a bomb threat was looked upon as an unusual event, and physical evidence from a bomb scene tended to be a laboratory curiosity by virtue of its rarity. At this writing, the reported bomb incidence rate is about 5.8 bombs per day according to current figures from the National Bomb Data Center, and the examination of physical evidence from these incidents has become a daily occurrence and a major laboratory challenge.

Bomb scene investigations are particularly difficult to conduct. Most of the physical evidence connected with the bomb has either been destroyed, scattered so that it is almost impossible to find, or distorted to such an extent that it is almost unrecognizable. The material from the device that remains is usually extremely limited in quantity and is in a highly contaminated condition since it is mixed with wood, dirt, or other types of debris.

The starting point in any bomb investigation is the identification of the explosive used in the destructive device. While an experienced bomb scene investigator may tentatively identify the explosive used through visual clues at the scene (such as blast effects, odor, or the color of the smoke produced), this method of identifying an explosive is usually not very reliable, especially when improvised mixtures rather than commercial or military explosives are involved. The identification of the explosive is almost always a task for the laboratory examiner.

The procedures described in this paper have been used for the past three years in the Forensic Laboratory of the Bureau of Alcohol, Tobacco and Firearms for examining debris from over 1200 destructive devices. They have been effective in identifying the explosive in about 90 percent of these cases and encompass tests for all of the explosives commonly encountered in criminal bombings. It will be obvious to the reader that these techniques could also be applied to the identification of other explosives such as PETN which, although not currently used to any extent, might be encountered in the future.

Collection and Preservation of Samples

The collection of appropriate samples is a vital step in the identification of explosive residues. Most of the explosive has been consumed upon detonation and usually only a very small fraction of the original material remains. The remaining traces of unconsumed residues are invariably mixed with soil, concrete, or other debris. The residue traces are

rarely, if ever, visible to the naked eye so the collection of the residues depends upon taking samples of debris from those locations at the blast scene most likely to contain them.

The location of the bomb at detonation must be found and the loose soil or other debris from the interior of the blast seat should be scraped out, packaged, and labeled since the debris from the blast seat is most likely to contain traces of the undetonated explosive. Other good sources of explosive residue are the objects located near the device on detonation. Wood, insulation, rubber, or other soft materials which are readily penetrated often collect traces of the explosive. Even metal objects in close proximity to the bomb should be collected since explosive residues have been found on these surfaces in many cases.

A portion of the device itself is an excellent source for recovering traces of the explosive. In pipe bombs, for example, particles of the explosive are frequently found adhering to the pipe cap or to the pipe threads, either by being impacted into the metal by the force of the explosion or by being deposited in the threads during the construction of the bomb.

The samples should be packaged in containers of appropriate size. Soil and other soft, loose material can be stored in plastic bags with suitable closures. Metal or other objects with sharp edges should be wrapped in plastic after placing some protective material such as cardboard around the sharp edges to prevent them from piercing the plastic wrapper. It is important to properly wrap metal or other objects with hard, smooth surfaces since explosive residues clinging to these surfaces are often dislodged in transmitting the package to the laboratory. Loose particles can be recovered from the packaging material if the container has remained tightly sealed.

Examination of the Debris

When the bomb debris arrives at the laboratory, the task of identifying the explosive used in the bomb begins. The first step in this process is a microscopic examination of the debris, a procedure which has been described in some detail by Washington and Midkiff [1]. Small portions of the debris are placed on a 4 by 6 in. white card and viewed with a variable power stereo microscope at a magnification of ×5 to ×15. The debris is slowly sifted with the aid of a dissecting needle or similar probe. The purpose of this microscopic examination is to locate particles of the unconsumed explosive and remove them from the debris for testing.

Metal objects, wood, and other materials taken near the blast seat are similarly examined for the presence of explosive particles. It is advantageous to have pliers and tweezers handy to unfold twisted metal or other brittle objects in order to examine the hidden recesses where these particles are frequently trapped.

While this method of examination is time-consuming, it has two advantages that make it worthwhile: (1) by removing suspected particles of explosives from the debris, the explosive is in effect being concentrated, permitting not only more convenient but more specific chemical testing to be performed on the material; and (2) the visual appearance of the residue gives a clue to its identity and suggests the chemical tests which should be performed.

It should be obvious that the microscopic examination of the debris can only be effective if the examiner has learned to recognize particles of undetonated explosive when he sees them. Black and smokeless powders are relatively easy to detect in debris of most types because of their characteristic shapes and colors. Dynamite, which is an amorphous mixture, is found in a variety of colors and is much harder to find, especially in soil.

Chemical Testing of Isolated Residues

After particles of the suspected explosive have been recovered from the debris, their composition must be chemically confirmed in almost every case. The possible exception to this is smokeless powder which has a unique appearance. In the case of other explosive residues, the general appearance of the material will usually suggest the confirmational chemical test to be run.

Black Powder

Black powder is manufactured in a variety of granulations and can be either glazed or unglazed. Usually it is easily found in debris because it contrasts so markedly with its surroundings.

A particle of the suspected black powder may be readily tested by observing its burning characteristics. Hold a particle of the black powder suspended on the tip of a dissecting needle over a small flame. Black powder will instantly burn with a characteristic flash producing a trace of white smoke.

An alternate method is to extract the particle and test the resulting solution for the presence of nitrates or sulfur by procedures described below. It is felt, however, that the visual appearance of black powder and its burning characteristics are unique and that these are sufficient to identify this substance.

Smokeless Powder

This material is produced in a variety of unique shapes, and the powder can be identified with respect to type and possible manufacturer if suitable reference standards are available. The most commonly encountered smokeless powders are bulk powders used by handloaders. Occasionally, however, ball and other types of powders removed from commercially loaded ammunition are used as the explosive in a bomb. The smokeless powder can be burned if confirmation of its identity is needed.

RDX Compounds

C-4, often referred to as plastic explosive, is white in color and dough-like in consistency. Although it is the most commonly encountered of the RDX-based explosives, it is not frequently used in criminal bombings in this country if the cases examined by the ATF Forensic Laboratory are representative.

A spot test can be performed on a particle of the suspected C-4. Place the particle in a white enamel spot plate with milligram quantities of thymol crystals. Intimately mix these substances with the tip of a glass stirring rod and add 3 drops of sulfuric acid. The formation of a rose color upon the addition of 5 drops of ethyl alcohol indicates the presence of RDX.

That the material is an RDX-based compound can be verified by thin-layer chromatography (TLC) using the procedure described by Jenkins and Yallop [2]. If sufficient material is recovered from the debris, it can also be examined by infrared spectrophotometry (IR) to confirm its identity by comparison with known standards. These procedures are described by Pristera et al [3] who have compiled an impressive list of IR spectra for many explosive compounds.

TNT

Like other military explosives, TNT is rarely used in bombings in this country. Particles of this material can be tentatively identified by their melting point (m.p. 81°C) and confirmed with a chemical spot test.

Place the suspected TNT in a white spot plate and add 2 drops of ethyl alcohol which has been saturated with potassium hydroxide. A deep red color indicates the presence of TNT. Infrared analysis and TLC may also be carried out if a sufficient quantity of the suspected explosive is recovered.

Dynamite

Dynamite is by far the most commonly encountered high explosive used in destructive devices associated with criminal acts in this country. There are a number of types of dynamites including straight dynamite, ammonia dynamite, blasting gelatin, ammonia gelatin, and nitrostarch dynamite. These explosive compounds have wide variations in their colors, ranging from off-white to nearly black.

While there are a variety of dynamite formulations, there are only a few components which need to be identified to confirm the presence of dynamite residues in bomb debris. These components are sulfur, ammonium nitrate, sodium nitrate, and the explosive oils [nitroglycerin (NG) and ethylene glycol dinitrate (EGDN)] absorbed on the binder or "dope" which makes up the bulk of the dynamite compound.

Not all of these components will be present in every type of dynamite and, hence, not in the debris from every dynamite bomb. Even dynamites of the same type have differences in their compositions. For example, straight dynamite up to 30 percent strength contains about 2 percent sulfur, while higher strength dynamites usually do not contain this component.

Most dynamites contain sodium nitrate but only ammonia dynamite contains ammonium nitrate. All commercial dynamites, with the exception of nitrostarch dynamite, will contain explosive oils in their binders. A comprehensive discussion of the specific compositions of the various types of dynamites is a very involved subject and it is not the purpose of this discussion to do more than indicate the variability of their compositions with respect to certain components.

The identification of dynamite in bomb debris is based on the detection of the explosive oils (NG and EGDN) and one or more of the inorganic components. The identification of these individual components is discussed below.

Ammonium Nitrate and Sodium Nitrate—Usually these compounds are easily found in dry debris since they are normally present in the form of prills (spheres about 1 mm in diameter) which contrast markedly with their surroundings. The prill is tested by dissolving it in 2 or 3 drops of water and analyzing the resulting liquid for the presence of sodium, ammonium, and nitrate ions.

The presence of nitrates in the solution may be detected by the diphenylamine, nitron, or modified Greiss tests. Addition of two drops of diphenylamine reagent to the test solution will produce a deep blue color in the presence of nitrates; however, other oxidizing agents will also give the same color reaction. For this reason diphenylamine is most useful as a screening procedure. Addition of nitron reagent to the test solution gives the highly insoluble nitron nitrate as a white precipitate. The Greiss test is performed by adding to the test solution one drop of sulfanilic acid solution followed by the addition of one drop of alphanaphthylamine solution. Addition of zinc dust will reduce nitrate ions to nitrite ions, which in the acid solution will bring about diazotization and coupling to produce a deep red color. The latter two tests are both specific for detecting the presence of inorganic nitrates.

The ammonium ion is detected in the test liquid by the addition of Nessler reagent and the presence of sodium may be detected either by a simple flame test or by atomic absorption.

Sulfur—Sulfur has a characteristic granular yellow appearance in dynamite residues resembling small pieces of cracked whole corn. A very sensitive test for elemental sulfur involves placing a fragment of the suspected sulfur (about one quarter the size of a pin head) in a small test tube and adding 1 ml of pyridine. After gently warming the tube to dissolve the sulfur, add 2 drops of 2 *N* sodium hydroxide solution or a saturated solution of sodium bicarbonate. Depending upon the concentration of sulfur in the test solution, a blue to green color is produced.

Nitroglycerin (NG) and Ethyleneglycol Dinitrate (EGDN)—The detection of NG and EGDN on the particles of dope recovered from the debris is best accomplished by TLC. When sufficient quantity of the dope is present, an IR analysis may also be performed.

The particles to be tested are treated with a few drops of acetone to extract the explosive oils from the dope. The volume of the extract is reduced by air evaporation, the concentrated liquid is spotted on a TLC plate,[2] and the plate is developed in a 4:1 carbon tetrachloride/dichloroethane mixture. As with any TLC analysis, standards are run on the same plate for comparison purposes.

The plate is removed from the tank and dried after the solvent front has moved 10 cm. The plate is lightly sprayed with a diphenylamine/sulfuric acid solution and NG and EGDN appear as well defined and separated spots with R_F values of 0.4 and 0.6, respectively. If additional material from the debris is available, the TLC examination should be repeated using another solvent system such as 1:1 benzene/hexane.

R_F values for dynamite in various solvent systems are shown in Table 1. Although the first three solvent systems are roughly comparable, the best separation and resolution is normally accomplished utilizing the carbon tetrachloride/dichloroethane system.

TABLE 1—*Average TLC R_F values of dynamite.*

Solvent System		EGDN	NG
CCl₄/Dichloroethane	(4:1)	0.6	0.4
Benzene/Hexane	(1:1)	0.6	0.4
Xylene/Hexane	(3:2)	0.6	0.4
Benzene	. . .	0.9	0.8
Hexane/Acetone	(4:1)	0.5	0.5

ANFO

Ammonium nitrate prills mixed with 4 to 8 percent fuel oil is commonly known as ANFO. ANFO is rarely used in bombs because of the ready availability of more easily initiated explosives. To be effective, this material must be packaged in a sturdy container and initiated with a high explosive booster. The resulting bulk is usually undesirable from the bomber's point of view.

The presence of ammonium nitrate can be chemically confirmed by the tests described above. The fuel oil component can be extracted with an organic solvent such as carbon tetrachloride and identified by gas-liquid chromatography.

Improvised Mixtures

Low-order explosive mixtures made from commonly available chemicals have been termed "improvised" mixtures. The majority of the improvised mixtures used in criminal

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[2] Glass plates, precoated with a 200 micron thickness of Silica Gel G, are used for all TLC examinations. These plates were obtained from ANALTECH, Inc., South Chapel Street Extension, Newark, Del. 19711.

bombings in this country fall into two categories: homemade black powder and home-made flash powders. These mixtures are usually confined in some container such as a section of capped pipe, and initiated with an external burning pyrotechnic or homemade fuse.

Homemade black powder is a mixture of potassium nitrate, sulfur, and charcoal. It is usually poorly made and unconsumed particles are abundant in the bomb debris. The components of this mixture can be chemically identified using techniques described previously.

Homemade flash powder mixtures have a variety of formulations. They usually contain finely divided aluminum or magnesium metal and an oxidizing agent such as ammonium nitrate, potassium chlorate, or potassium perchlorate with possible additions of sulfur and/or sawdust.

Some of the components of flash powders can be identified by tests described above or by their microscopic appearance. Magnesium may be identified by its burning characteristics and its solubility in nitric acid. Aluminum metal will dissolve in dilute sodium hydroxide solution with the liberation of hydrogen gas and is insoluble in nitric acid.

The presence of chlorates may be verified with the aniline sulfate test. To a few drops of an aqueous extract of the flash powder, add 2 drops of a 1 percent aniline sulfate solution. Allow 5 drops of concentrated sulfuric acid to slowly run down the side of the test tube so that two layers are formed. After about a minute, the formation of a blue-violet color at the interface of the two liquids is indicative of the presence of chlorates.

Two other techniques, infrared spectroscopy and refractive index measurement, have been found to be useful in the characterization of inorganic salts from flash powders or other explosive mixtures. An extensive collection of the infrared spectra of pure inorganic salts has been reported by Miller and Wilkens [4]. Refractive index measurements may be carried out with extremely small amounts of sample using a polarizing microscope and a set of the appropriate refractive index liquids. The refractive indices for the most commonly encountered inorganic oxidizing agents are shown in Table 2.

Solvent Extraction of Debris

If particles of explosive cannot be found visually in the debris using a low-power microscope, solvent extraction may be tried as a last resort. While solvent extraction is easily performed and requires relatively little time, these benefits are more than offset by two major disadvantages: (1) there are only microscopic quantities of unexploded material in most bomb debris and the large volume of solvent used to extract them serves not only to dilute these quantities, but to introduce interferences; and (2) the physical appearance of the explosive substance suggests the chemical tests to be run. Once the substance to be identified is dissolved, not only are the visual clues to its identity lost, but it is difficult if not impossible to verify the specific substance originally present. For example, a water

TABLE 2—*Refractive indices of selected inorganic salts.*

Compound	Refractive Index (n_D)
Ammonium Nitrate	1.611
Sodium Nitrate	1.587
Potassium Chlorate	1.517
Potassium Nitrate	1.504
Potassium Perchlorate	1.474

extract of soil debris which affords a positive nitrate test is of little value since various nitrate salts are commonly present in soils.

If the presence of a high explosive such as dynamite or C-4 is suspected, the debris may be treated with an organic solvent such as acetone. The acetone recovered from the debris is reduced in volume by air evaporation to concentrate any explosive present, and then the concentrate is examined by TLC.

Examination of Materials Taken from a Suspect

The discussion thus far has been concerned with the identification of the explosive residues present in bomb debris. It should be pointed out that the same analytical methods can also be used to detect explosive residues in a suspect's clothing, on his possession, or even in debris taken from his automobile.

Clothing

During the construction of a bomb, the individual's hands generally contact the explosive. In the case of a dynamite bomb, for instance, the wrapper of the dynamite stick is usually opened to insert the blasting cap and particles of the explosive frequently adhere to the person's clothing or get under his fingernails and are then transferred to his shirt or pants pockets. Debris from a suspect's pockets and even pants cuffs should always be carefully examined microscopically and suspected explosive traces, if found, can be identified by the procedures described above.

Automobile Debris

Vacuum sweepings from the trunk area or automobile floor boards often are excellent sources of residue if the explosive has been transported in the vehicle. The debris should be examined microscopically to isolate any particles of unexploded residues which may be present.

Hand Swabs

The removal of explosive residues from a suspect's hands has proven to be extremely valuable in many investigations. These findings have not only been used for investigative purposes, but have been introduced as court evidence to refute a defendant's statement that he had never handled the explosive.

Techniques have been described for removing residues from a suspect's hands through the use of adhesive tape or with cotton swabs moistened in solvent. A detailed testing and evaluation of the adhesive tape removal procedure for detecting TNT and RDX-based explosives on a suspect's hands has been conducted [5]. Jenkins and Yallop describe the use of cotton-wool swabs soaked in appropriate solvents for the removal of explosive residues. A limited study of the length of time NG and EGDN will remain on an individual's hands was reported by Kempe and Tannert [6] in which this swab removal procedure was used.

The explosive residue removal procedure developed in the ATF Laboratory which has been used by ATF agents for the past three years is similar to that proposed by Jenkins and Yallop. Two or three cotton-tipped swabs saturated with acetone are used to swab the palms, fingers, and underside of the nails of a suspect's hands. These swabs are then preserved in glass vials for subsequent testing.

The swabs are extracted with a few drops of acetone in the ATF Laboratory and the extract, which is concentrated by air evaporation, is spotted on a TLC plate and developed in a suitable solvent system. A visualization spray of diphenylamine/sulfuric acid or di-
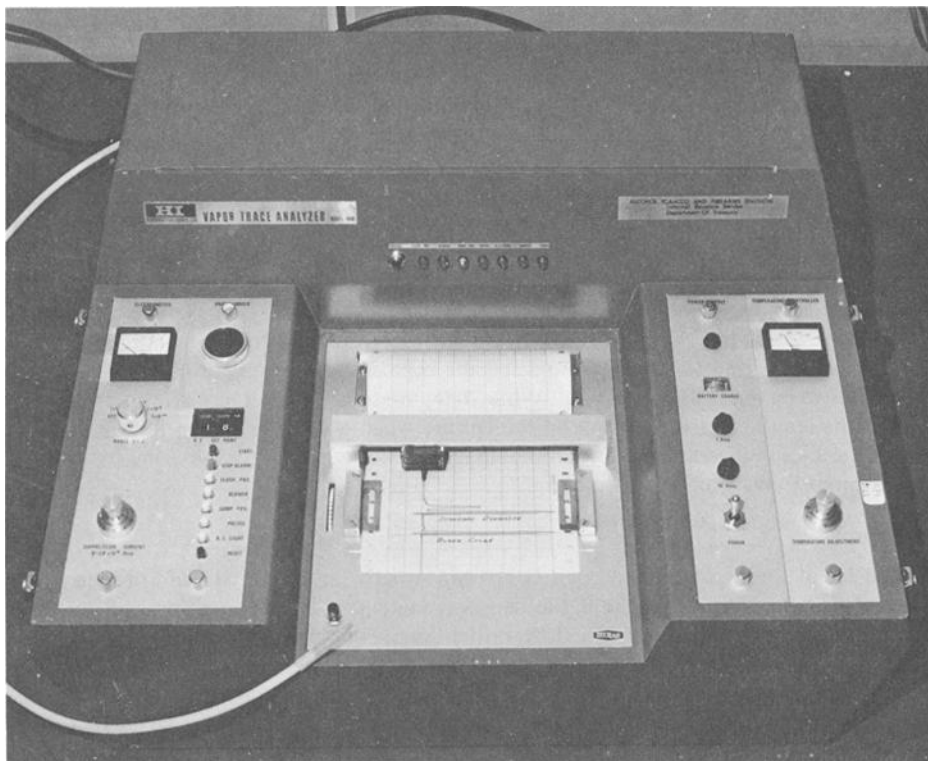
FIG. 1—*The vapor trace analyzer (VTA) is a very sensitive instrument for detecting explosive residues which are present in minute quantities.*

phenylbenzidine/ethanol, followed by exposure to ultraviolet light, is used to detect the presence of explosive compounds on the plate, which are then identified by comparing their R_F values to those of simultaneously run standards.

Vapor Trace Analysis

The microscopic examination of debris for the presence of explosive residues is time-consuming if a large volume of debris is to be searched. The task of examining many bags of debris can be shortened considerably if a single package containing the explosive traces can be isolated from the many bags of debris usually collected in the course of the investigation.

A scanning instrument which has been used with excellent results in our laboratory is a special purpose gas-liquid chromatograph called the Vapor Trace Analyzer (VTA).[3] This instrument, shown in Fig. 1, draws air from the bag containing the bomb debris and traces of explosive vapors in the air are collected in a special sampling valve. The explosive is released from the valve, swept through a short column into an electron capture detector, and identified by its retention time.

[3] Hydronautics, Inc., Pindell School Road, Laurel, Md.

The instrument is extremely sensitive in detecting explosive compounds with relatively high vapor pressures, such as nitroglycerin-based dynamites. Because of this sensitivity, the instrument can also be used to scan the cotton hand swabs used to test a suspect's hands for explosive residues. If a positive VTA response is obtained from a hand swab, the swab is further tested by TLC to identify the explosive present.

There has been some success in testing air samples taken from the scene of the explosion or from a closed compartment which has contained dynamite. In one instance, air was drawn through the keyhole of an automobile trunk lid into an evacuated bottle, and subsequent testing of the air sample with the VTA revealed the presence of dynamite vapors. It was learned that one case of dynamite had been transported in the automobile trunk four days previously.

Bioluminescence may be useful for detecting explosive traces. In this technique, special cultures are sensitized to vapors from a specific explosive and respond to the presence of this explosive by the emission of light. While there are some difficulties with this approach, such as the lengthy activation time of the culture when a new detector cell must be prepared, the bioluminescence units[4] are compact, portable, relatively inexpensive, and do not require extensive operator training.

Summary

Particles of the explosive used in a bomb can usually be recovered from properly collected debris taken at the scene if the debris is meticulously examined microscopically. The explosive can then be identified by rather simple chemical and instrumental procedures. As a last resort, solvent extraction of the debris may be performed, but this is not recommended since it usually lacks the specificity and sensitivity necessary to detect the traces of explosive present in a large volume of debris. In addition, the debris may contain substances that would interfere with subsequent tests of the extract.

Cotton swabs soaked in acetone are effective in removing traces of various explosives from the hands of a subject who has recently handled them. Explosive residues on the swabs can be identified in the laboratory by TLC procedures.

Instruments such as the VTA have value in rapidly scanning debris, cotton hand swabs, clothing, and air samples for the presence of explosives. The explosive present is then confirmed by chemical methods.

The methods described in this paper for examining bomb debris, isolating the explosive particles, and identifying these substances are admittedly tedious. They are also effective, as has been shown by the high degree of success in identifying the explosive used in hundreds of bombings.

APPENDIX

Preparation of Chemical Reagents

The methods of preparing several reagents commonly used in testing explosive residues are listed below. Solvent systems used for TLC and special chemical tests are cited elsewhere in the text.

Alpha-Naphthylamine—Dissolve 0.5 gm of alpha-naphthylamine in 100 ml of methyl alcohol.

Aniline Sulfate—Add 1 part of aniline to 999 parts of distilled water. Add concentrated sulfuric acid by drops to the mixture, with stirring, until the aniline dissolves.

Diphenylamine—Dissolve 1 g of diphenylamine in 100 ml of concentrated sulfuric acid.

Diphenylbenzidine—Dissolve 1 g of diphenylbenzidine in 100 ml of absolute alcohol.

Nessler Reagent—Dissolve 50 g of potassium iodide in 35 ml of distilled water and add enough of a saturated mercuric chloride solution until a precipitate persists (about 200–250 ml).   Add a solution of 200 g of potassium hydroxide in 300 ml of distilled water and dilute the combined solution to 1 l. Filter.

The dry reagent can also be purchased from Scientific Products (Stock No. B-5305).   The reagent is dissolved in distilled water as indicated on the container.

Nitrogen Reagent—Dissolve 1 g of nitron reagent in 20 ml of formic acid.   Nitron reagent can be purchased from the Eastman Company (Stock No. 1077).

Sulfanilic Acid—Dissolve 1 g of sulfanilic acid in 100 ml of 30 percent acetic acid.

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