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### The ATF approach to post-blast explosives detection and identification

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THE ATF APPROACH TO POST-BLAST EXPLOSIVES  
DETECTION AND IDENTIFICATION

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ABSTRACT

The majority of all criminal bombings in the United States are investigated by the Bureau of Alcohol, Tobacco and Firearms. Processing the bomb scenes and examining the physical evidence in the laboratory are both performed using teams of highly trained specialists. This paper discusses the team approach to bomb scene processing. The analytical schemes employed by laboratory staff for the post-blast identification of explosives and bomb component is also described.

INTRODUCTION

The Bureau of Alcohol, Tobacco and Firearms (ATF) is a separate agency of the U.S. Department of Treasury. ATF has the dual responsibilities under Federal law of protecting the revenue and enforcing specific criminal laws. Protection of the revenue is concerned with tax

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collection in the firearms, tobacco and alcohol areas. In the law enforcement field, ATF has primary Federal jurisdiction in the firearms, explosives and arson areas. Special Agents investigate violations of the Federal laws enforced by the bureau. Forensic services are provided to the Special Agents by approximately 40 forensic examiners at three facilities. Both in the field for scene investigation and in the laboratory for examination of physical evidence, ATF has adopted a team approach to the investigation of post-blast explosive incidents.

#### BOMB SCENE INVESTIGATION

In 1978, ATF formed four National Response Teams located in Northeast, South east, Midwest and Western regions of the country. Each highly trained team of experts can respond within 24 hours to the scene of a major bombing or arson. Each team is fully equipped, including an investigative response vehicle which, when necessary, is transported to the scene by C-130 Hercules Aircraft operated by the U.S. Cost Guard. A team consists of 13 members including one forensic chemist and one explosive enforcement office. Table 1 outlines the team members and their duties.

TABLE I

Composition of ATF National Response Team

<u>Member</u>	<u>Duties</u>
Team Supervisor	initial scene evaluation coordination with state and local officials
Team Leader	coordinates personnel and search patterns
Photographer	photographs scene, evidence
Evidence Technician	responsible for control of all physical evidence
Schematic Artist	diagrams scene and plots evidence location
Interview Teams	interview witnesses and people associated with target
General and Immediate Area Search Units	establish scene perimeter, collect evidence, identify area of origin
Forensic Chemist	collect and identify evidence for laboratory examination
Explosives Enforcement Officer	evaluate scene, reconstruct events

An initial evaluation of the scene is made on a walk through by the team leader, chemist, and explosives technician. A general approach to be used to process the scene is devised, and an immediate search area established. Each site requires a slightly different approach. Extensive damage to buildings may require using heavy equipment

to remove structural parts. The presence of chemical hazards must be assessed.

The chemist assists the general and immediate area search teams in collecting and evaluating evidence. Actual field tests on evidence are limited to simple chemical tests and component identifications that may provide early investigative leads. The chemist is also responsible for ensuring the proper packaging of the evidence for transmittal to the laboratory. A very challenging task is acting as technical advisor for chemical and laboratory matters at the scene. The forensic chemist is called upon to identify any chemical found at the scene and to be familiar with, or be able to determine, its intended commercial use. The chemist also acts as liaison between technical representatives from businesses and local forensic laboratory personnel.

Other forensic specialists may be called to the scene. The laboratory has portable laser unit which can be taken to the scene by the fingerprint examiners. Laser illumination will cause latent fingerprints to fluoresce. The fingerprint examiners can make an initial evaluation of the presence of latent prints on pieces of evidence. This evidence is then transported to the laboratory for complete latent fingerprint analyses.

The team remains at the scene until all the necessary evidence has been collected and interviews made. Before returning to the laboratory, the chemist will inform the laboratory chief what additional examiners will be needed to analyze the evidence, such as additional chemists, document, fingerprint, and toolmarks examiners. The chemist personally transmits the evidence to the laboratory where analysis begins immediately.

#### LABORATORY ANALYSIS

In the laboratory, the approach taken in examining the evidence is the same as the approach to the scene processing, namely a team approach. On major cases, the team has consisted of several chemists, fingerprint examiners, document examiners, and toolmark examiners. Specialty areas for the chemists include explosives, accelerants and trace evidence including hair, fibers, soil, glass, and tape. Because of the large volume of information that is generated in a short period of time, a case management system was created to ensure that the investigators were informed of all laboratory findings in a timely and organized manner. On major cases, all information developed by the laboratory is routed through an examiner designated to coordinate the analysis to the investigators. The overall concept is to obtain as much information as possible from the bomb evidence using

whatever technical expertise is necessary. Our experience has been that a team of experts working together is the the best situation. This differs from the classical approach of sending the evidence to one examiner, and when he is through, to another.

The detonation of a destructive device will produce a large quantity of fragments and debris, some of which are from the device itself and many more from the surrounding area. To provide as much investigative information as possible, the various components of the device must be reconstructed and identified. Of primary concern on all bombing investigations is the identity of the explosive itself. The type of exhibits submitted to the laboratory for explosive examination are varied and complex. While it would be impractical to apply rigid standard analytical procedures to these non-standard samples, it is possible to suggest an analytical scheme which is applicable in most cases, but can be modified as the circumstances dictate.

#### Analytical Scheme

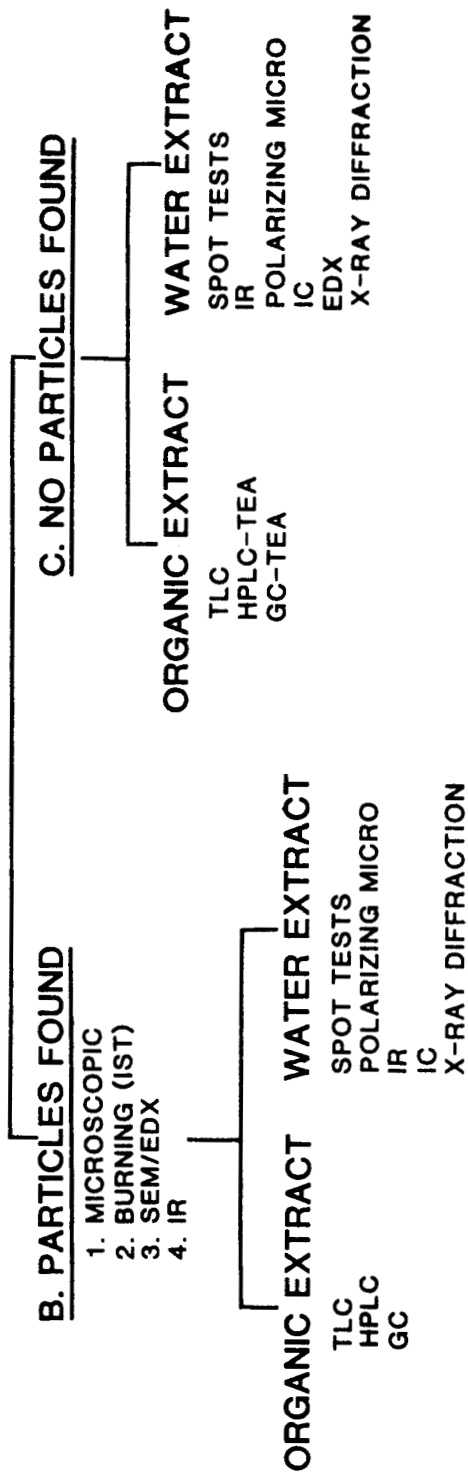
Figure 1 shows a flowchart for the examination of debris from an explosion. This scheme was developed in the ATF Laboratories over a number of years. Other laboratories performing explosive analyses have developed similar schemes<sup>1,2</sup>.

# FLOWCHART FOR THE EXAMINATION OF DEBRIS FROM AN EXPLOSION

FIGURE 1

## A. DEBRIS EXHIBIT

1. VAPOR EXAMINATION
2. VISUAL EXAMINATION
3. MICROSCOPIC EXAMINATION





Volatile organic explosives, such as nitroglycerin, ethylene glycol dinitrate, TNT, RDX and PETN, can be collected from debris by a purge and trap method. Debris is packaged in vapor tight containers to prevent the loss of the volatile explosive in transmittal to the laboratory. When a high explosive is suspected the debris is gently heated in the sample container (to 60°C). The vapor in the container is trapped on a tube of activated charcoal to which a vacuum line is attached. After trapping the vapor, the explosive compounds are eluted from the charcoal with dichloromethane. Appropriate analysis of this liquid sample is conducted to identify the explosive compound.

Visual examination of the debris is a very important step in the analysis scheme. It serves two purposes. First, components of the explosive device, such as containers, wire, batteries, and timers are identified. Second, unconsumed explosive material may be found. This is most likely to occur when a low explosive has been used. A diligent examination of the debris under a low power (5X-30X) magnification is time consuming but most worthwhile. Unconsumed explosive particles found can be readily identified. The morphology will allow a tentative identification and direct the examiner toward the appropriate tests for confirmational analysis.

The manner in which a particle burns is a worthwhile and simple test for many explosive particles. While it is destructive, this "ignition susceptibility test" will clearly differentiate a particle of black powder from a similarly shaped black piece of sand or asphalt. Black powder burns with a "pop" and an orange flame, smokeless powder burns slower with a "poof" leaving no residue, and other explosives such as C-4, flash powders and ammonium nitrate have their own characteristic burning properties.

### Instrumental Analysis

At this point in the analytical scheme, explosive particles have been observed and removed from the surrounding debris, the microscopic appearance and burning characteristics have been noted, and perhaps the solid particle has been examined by an instrumental method such as infrared spectroscopy (IR) or scanning electron microscopy coupled with an energy dispersive X-ray analyzer (SEM-EDX). Depending on the suspected identity of the particle, which is based on the above tests and the known facts surrounding the explosion, the material can be dissolved in an organic solvent or water for further testing. In some cases, presumptive spot tests on a water extract of the particle may have been done along with the microscopic examination prior to any instrumental analysis.

The remainder of the discussion of the analysis scheme will focus on instrumental techniques. These techniques are used to identify unconsumed explosive material, and also to identify the explosive in vapor, organic, and water extracts of the debris.

Most forensic laboratories have an infrared spectrometer. It is perhaps the simplest diagnostic instrument available for identifying relatively pure organic particles removed from debris or for bulk organic explosives. Many inorganic compounds also have unique IR spectra which can be compared with the spectrum from a known material or with a library of IR spectra. It should be noted that while IR is most applicable for the identification of relatively pure substances, it will also be beneficial for mixtures such as RDX/TNT (military dynamite) and sugar/potassium chlorate (improvised explosives).

The SEM-EDX can afford both a high resolution photomicrograph and the elemental chemical composition of an unknown inorganic particle. With explosive analysis, it is best used for identifying the metallic components of flash powders and other explosive formulations utilizing finely divided metals. The SEM has also been used for examining the surface features of smokeless powders and the carbonaceous materials associated with dynamites.

X-ray diffraction spectrometry will positively identify the chemical compounds present in a material. Any crystalline material will give a unique x-ray diffraction pattern. For example, the sulfur and potassium nitrate in black powder can be identified using x-ray diffraction.

In those cases in which no particle can be found in the debris, or the diagnostic effects of the explosion indicate that a high explosive was used and a great deal of loose debris is submitted for examination, identification of the explosive can become a formidable task. The approach involves vapor sampling (discussed previously), extraction of the debris with an organic solvent, and an extraction with water. Organic solvents extract materials other than explosives such as plasticizers, oils, and paint. These extract samples may need pre-analysis clean-up so that the desired explosive residue can be concentrated and the contaminants eliminated<sup>3</sup>. When no intact particles are found, the examiner must be aware of both the original constituents of the explosive and the by-products that result from the combustion of the explosive.

The non-volatile constituents and combustion products are isolated with the water or organic solvent extraction. The usual approach is to either rinse the object with warm, distilled de-ionized water, or cover the debris

with water and warm gently, or cover the debris with water and place in a sonicating bath. Long extraction times are not necessary. The extract is filtered and concentrated by warming it under a stream of air. The reduced volume extracts can be examined by whatever methods necessary to identify the explosive. These procedures would commonly start with presumptive spot tests and proceed to other instrumental techniques such as ion chromatography, and, if sufficient crystalline residue is obtained, x-ray diffraction.

For organic extracts of debris, acetone is the solvent of choice for the organic constituents of explosives. When HPLC-TEA analysis is used, dichloromethane (DCM) is used to extract the debris. The exhibit is rinsed or soaked in the organic solvent, filtered, and concentrated by evaporation. The extract may be examined by thin-layer chromatography (TLC), high pressure liquid chromatography (HPLC), or gas chromatography (GC). In all cases, the instrumental or wet chemical method chosen by the examiner for the identification of the suspected explosive will be dictated by the circumstances of the bombing, the results of the microscopic examination, presumptive tests performed, and the instrumental techniques needed to identify the explosive material. The most important result is obviously the accuracy of

the conclusion reached, and not that a specified number of tests or a certain type of instrument was used.

With training and experience the number of tests carried out can be reduced to a minimum.

### TEA Detector

Following an initial TLC screening of extracts, confirmation of explosives tentatively identified by TLC are obtained using GC and HPLC. The selectivity and sensitivity of the detectors used is of primary importance. In the recent past, there has been an important addition to the detectors available to the analyst, the thermal energy analyzer (TEA®). The TEA is a gas phase, chemiluminescence device which provides excellent selectivity and sensitivity for nitroso- and nitro-compounds<sup>4,5</sup>.

In GC-TEA, it is possible to alter the temperature of the pyrolysis chamber in order to gain selectivity for nitroso-compounds over the response for nitro-compounds. In other words, in the "nitroso mode", the pyrolyzer is held at 550 °C. LaFleur and Mills<sup>4</sup> have shown that nitroaromatics are not readily detected using a pyrolyzer temperature of 500 °C, rather, these compounds require temperature of 800 °C or greater for molar (or near molar) response.

Unfortunately, when LC-TEA is performed, the use of pyrolysis chamber temperatures greater than 550 °C gives rise to substantial baseline noise which severely limits the detector's sensitivity and selectivity. Thus, LC-TEA is always performed in the "nitroso mode". While nitramines (compounds containing N-NO<sub>2</sub> bonds) and nitrate esters (containing the -O-NO<sub>2</sub> moiety) are amenable to LC-TEA under the influence of the lower pyrolysis temperature, nitro-aromatics (containing C-NO<sub>2</sub> bonds) cannot be detected with adequate sensitivity. These differences are understandable, since the C-N bond energy (292 KJ/mol) is significantly higher than the O-N (175 KJ/mol) and N-N (159 KJ/mol) bond energies for nitrate ester and nitramines, respectively.

Several reports in the literature suggest that ultraviolet (UV) irradiation could be used to facilitate TEA detection of the C-nitro compounds. Thus we chose to attempt an on-line, post-column UV irradiation in order to improve the LC-TEA detectability of nitrotoluenes. One important aspect of this LC-Photolytically-Assisted TEA (LC-PAT) approach involved the use of a knitted open tubular (KOT) photochemical reactor design. This KOT, developed by Dr. Ira Krull<sup>6</sup>, significantly reduced the band broadening that occurs whenever long lengths of

tubing, having large dead volumes, are used to construct post-column reaction chambers. Without this inclusion in the experimental design, the sensitivity gained using PAT would be cancelled by the loss in resolution caused by band diffusion. A paper has been prepared which fully describes the experimental metal conditions employed in developing this analytical technique<sup>7</sup>. After optimization of photolytic residence time and cooling bath temperature, PAT allows for an improvement in the detectability of trinitrotoluene and dinitrotoluene by factors of 30 and 16 respectively. Mononitrotoluene, which is not responsive under conventional LC-TEA conditions, may be detected with good sensitivity using LC-PAT. The use of LC-PAT does not interfere with the determination of nitro-based high explosives which exhibit adequate responses under conventional LC-TEA condition.

#### SUMMARY

ATF uses a team concept both in the field for bomb scene investigation and in the laboratory for analysis of the physical evidence. Using the flow scheme described, the laboratory is able to identify the explosive used in 90% of the cases received for examination. The scheme, developed over the last fifteen years, continues to evolve and adapt as new technology becomes available.



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