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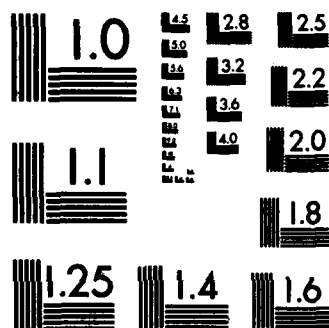
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which is sealed. This phase (II) covers nonvapor detection. This work is supported by the Naval Sea Systems Command (NAVSEA 06H3). This study covered the period of June 15 through November 21, 1983.

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Department of Energetic Systems  
November 23, 1983

Commander, Department of the Navy  
Naval Sea Systems Command NAVSEA-06H3  
Attention: Mr. Ed Daugherty  
Washington, DC 20362

Subject: SwRI Project 15-5607-825  
Final Report on Phase II  
"Feasibility of Detection of Explosives in  
Cased Munitions: Phase II Nonvapor Detection  
Methods"

Dear Mr. Daugherty:


In accordance with the MIPR sent through the Defense Electronics Systems Command (DESC) three copies of the Final Report on SwRI Proposal 06-0604A, Phase II funded under contract modification number P00031 dated 15 June 1983, line item 0001AV to NTIAC Contract DLA 900-79-C-1266 and titled: "Feasibility of Detection of Explosives in Cased Munitions: Phase II Nonvapor Detection Methods," by Dr. Harold J. Gryting are forwarded.

For Phase I \$10,475 is now needed in accordance with the proposal. On completion of Phase I, the two sections will be bound together into one report (some additions will be made to Phase II as not all requested information has been received). The report will also contain a brief list and discussion of advantages and disadvantages of vapor vs nonvapor detection.

Recommendations for near term research into the best of the nonvapor detectors has been made in the attached report.

We are happy to have the opportunity to serve and hope to continue to assist in this important endeavor.

Sincerely yours,



Alex B. Wenzel, Director  
Department of Energetic Systems

ABW:lr  
Enclosure

cc: H. N. Abramson  
H. J. Gryting  
R. Priegel  
C. du Menil  
W. R. Herrera



SAN ANTONIO, TEXAS  
WITH OFFICES IN HOUSTON, TEXAS, AND WASHINGTON, D.C.

ASSESSMENT OF THE FEASIBILITY OF PERFORMING  
INFIELD NONDESTRUCTIVE EVALUATION TO DETERMINE THE  
PRESENCE OF EXPLOSIVES MATERIALS WITHIN CASSED MUNITIONS

Phase II: Nonvapor Detection

By

Harold J. Gryting

SwRI Final Report (Phase II)  
Project 15-5607-825

Prepared For

The Naval Sea Systems Command  
NAVSEA 06H3

November 1983



APPROVED

*Alex B. Wenzel*  
Alex B. Wenzel, Director  
Department of Energetic Systems

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## FOREWARD

The Navy has the ongoing problem of ordnance being loaded with simulants for various requirements including practice items being difficult to differentiate from explosive-filled ordnance. In addition some practice bombs contain energetic marker materials. Ideally, one portable instrument for all differentiation problems would be chosen if available. The current survey is to determine the status of explosives detection instruments and possibly viable concepts for such detection for Navy ordnance, much of which is sealed. This phase (II) covers nonvapor detection.

This work is supported by the Naval Sea Systems Command (NAVSEA 06H3). This study covered the period of June 15 through November 21, 1983.

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## DETECTION OF EXPLOSIVES PHASE IN NONVAPOR DETECTION

### INTRODUCTION

A proposal to review concepts and new and improved methods for explosives detection was accepted by NAVSEA06H in June 1983. Phase II Nonvapor Detection was funded first. To conserve time, the abstracts for both phases were obtained at the beginning of Phase II. Full reports (microfiche) were ordered for 46 articles related to Phase II. Approximately 150 reports were noted as related to Phase I.

Data bases searched by computer from 1980 through July 1, 1983 included (1) INSPEC Data Base, which is the largest data base in the English language in the fields of physics, electrotechnology, computers, and control. (2) CA Search Data Base. This covers bibliographic data from all documents covered by the Chemical Abstract Service. (3) NTIS Data Base. This covers Government-sponsored research, development, and engineering, plus analyses prepared by Federal agencies, their contractors and grantees, and (4) Compendex Data Base which is from the Engineering Index with a worldwide coverage of 3500 journals, publications of engineering societies and organizations including papers from the proceedings of conferences, and selected Government reports and books. In addition to the above data base computer searches, personal contact was made with a number of people known to be involved with detection to obtain updates in their particular specialties that have occurred since 1979. This included many people whose papers are referenced in the SwRI report of December 1981. In addition, a summary from the FAA-sponsored Cambridge, Mass., (April 19, 1983) Conference on Detection was obtained, as were abstracts of papers from the FBI-sponsored detection meeting at Quantico, W. Va. (March 29-31, 1983). These two conferences were directed toward finding explosives that are brought in or planted by terrorist or other saboteurs.

The field of quantitative nondestructive evaluation has expanded and progressed markedly since 1980. A meeting was attended at the University of California, Santa Cruz, sponsored by the Center for Advanced Nondestructive Evaluation, Ames Laboratory, Iowa State University, in cooperation with the Office of Basic Energy Sciences of the US Department of Energy, the Defense Advanced Research Projects Agency, the Naval Sea Systems Command

and the Air Force Wright Aeronautical Laboratories/Materials Laboratory. Although the NDE papers given did not include results of use of any instrument specifically developed for detection of explosives, some of the techniques, given additional exploration dedicated to differentiation between simulants and explosives, may become useful in the future. Some of these will be considered here.

Although for many of the types of detection and detectors no major breakthrough type improvements have been made that allow field detection in sealed bombs instantly with portable devices, there is still the potential for some of these methods to be developed to do what is required to differentiate the true explosive from the inert simulants. Among these can be included nuclear gauging, dual energy tomography and nuclear magnetic resonance. The latter cannot be used for sealed weapons, however, so its use would require development of equipment which could drill holes suitable for a small transducer. NMR can be used with a large or small specimen (down to a few grams size).

The theory and mode of operation of nonvapor detectors is discussed in some detail in our previous report<sup>(1)</sup> and the evaluation by Henegar<sup>(2)</sup> of various metal detectors has apparently not been superseded by a newer detailed comparison of instruments.

#### COMPUTERIZED TOMOGRAPHY (CT)

As discussed previously,<sup>(1,3)</sup> CT is the reconstruction numerically of a cross-sectional image from data obtained at different aspect angles. These are determined using x-rays or gamma-rays together with radiation detectors. An update of Roder's previous report<sup>(3)</sup> has just been received for review; however, the author indicated that whereas the previous report indicated that tomography was not good enough to differentiate between explosives and their simulants, he now considers that using Dual Energy Computerized Tomography, (DECT), depending upon the system, may work with warhead cases up to one-half-inch thick, and above that it would not work. If, on the other hand, the organic simulant simulates the density of the explosive exactly and atomic numbers do not differ by more than about 10%, DECT probably would not be

satisfactory for the differentiation required<sup>(4)</sup>. Both Aerospace-Georgetown University sponsored by BATF and Varian Associates sponsored by the FAA have performed DECT studies.<sup>(5)</sup> These showed that explosives could be seen as high density materials with low average atomic numbers and could be reasonably distinguished from innocuous objects. One explosive, it was noted, was not distinguished from a block of cheese.

A breadboard type system suitable for realistic measurements was designed by Aerospace-ARACOR-USCF under BATF sponsorship. This system includes a precision conveyer belt, a panoramic X-Ray tube, eighty separate scintillators ( $\text{CdWO}_4$ ) coupled to photodiodes, and a data acquisition and control system. This system is to be evaluated at the FAA Tech Center.

To summarize recent CT technology advances Dr. Frederick Roder is quoted:

"In the four years that have passed since development of the breadboard DECT system was initiated, the state-of-the-art in CT has advanced on several fronts. A prototype system configured along the lines of the breadboard system would require  $\sim 10^4$  detector elements. In 1979 that seemed unthinkable and  $10^4$  is still more detectors than has ever been incorporated in a single scanner. However, one commercial system has employed  $2.4 \times 10^3$  detector elements. The breadboard system requires that the X-ray voltage be changed between complete scans. However, an X-ray source designed for dual energy work and capable of switching between two fixed voltages at 60 Hz is currently being used at the Stanford Medical Center. But most significantly, the last four years has seen the development of a very high speed CT scanner designed primarily for cardiac imaging. This unit, termed a cinetomographic scanner by the developing group at the University of California, San Francisco Medical School, has already been employed to obtain cross sectional images of human hearts at 36 slices/sec. This system utilizes no moving parts during a scan. Instead a high current ( $\sim 600$  mA) electron beam is electromagnetically swept over a  $210^\circ$  tungsten target, producing the same effect as an X-ray tube rotated through the same angle. The slice thickness is 7.5 mm, with a 47 cm diameter reconstruction circle. The present system is designed for single energy operation. The next system, currently being fabricated, will have DECT capability. Figure 7<sup>(6)</sup> is a photograph of the cinetomography system before the detector rings were installed. Figure 8<sup>(6)</sup> illustrates the scanning position for a patient."

"The cinetomographic scanner could obtain a full three-dimensional DECT image of a 100-cm long suitcase in .3.7 seconds. However, the resolution of this system (2.2 mm FWHM) is far in excess of the requirements for explosives detection and the 47 cm diameter reconstruction circle will only accommodate relatively small luggage items (~35-cm high by 30-cm wide). Reconstruction time is also a limitation: at present about 7s are required per 256 x 256 slice. Consequently, although it is believed that the cinetomography system would be an excellent tool for demonstrating the feasibility and efficacy of DECT explosives detection, further engineering will be required to embody this technology in a system configured for explosives detection."

"Finally there is the matter of cost. As a medical system, the estimated \$1.5 million price tag of the cinetomography system is quite acceptable. However, for security applications such a price tag would be unacceptable under other than the most dire circumstances. Consequently, cost reduction engineering must be an essential aspect in developing a DECT explosives detector."

#### NUCLEAR GAUGING

Work in this area by Weber, Lukens, and others has been summarized in a paper from IRT Corporation<sup>(1)</sup>. The early work reported a nuclear gauging system consisting of three transmission type gauges with signal acquisition electronics. They determined density using a dual energy gamma gauge with <sup>241</sup>Am (59.5 KeV energy) and <sup>109</sup>Cd (22.1 and 25.0 KeV) and hydrogen content with a thermal neutron gauge with <sup>252</sup>Cf as the source and a tritium-filled gas proportional counter as the detector. Letter bombs were detected at near 100% accuracy.

More recently nuclear gauging was used to determine feasibility of detection of phosphorus in practice bombs.<sup>(8)</sup>

#### Nuclear Gauging by Compton Scattering - Applied to Practice Bombs

Nuclear gauging has been examined by IRT Corporation using a Compton Scattering Inspection System.

A Compton scattering detection system was developed by IRT Corporation which they indicate has been shown in a feasibility study conducted at

NWC, China Lake, to be capable of meeting requirements of range clearing operations for the NR-76/BDU-33 and NK-106 practice bombs. Compton scattering, or collimated photon scattering, a technique developed by IRT for nondestructive examination of ordnance, uses a collimated beam of photons from a "pencil beam" intersecting a practice bomb at a particular inspection location. A series of collimators is focused on this location and the attached sodium iodide detectors and associated electronics provide accurate counting rates for photons which are scattered away from the "location" (or interior inspection volume). The scattered photons can yield information concerning whether the energetic load has been expended or remained in whole or part.

A three-step inspection process was developed:

1. Physical inspection using calibrated, hand-held measuring rods.
2. Inspection of remaining bombs using the Compton Scattering System.
3. Inspection of those gauged to be live on doubtful by the Compton System by dismantling, sawing near spotting charge location and visually verifying whether live or not.

A validation laboratory study (IRT-399-902) confirmed the initial feasibility study which was then followed by a pilot study which included design and fabrication of a prototype Compton Scattering System and transporting to NWC Baker Test Range where results led to establishing the parameters for an optimized system for full-scale range cleanup of MK-76/BDU-33 and MK-106 practice bombs using this method.

The live round false accept probabilities were: for MK106 =  $1.9 \times 10^{-8}$ , for MK 76/BDU-33 =  $1 \times 10^{-5}$ , whereas empty round false reject probability was: for MK106 =  $4.4 \times 10^{-5}$  and for MK-76/BDU-33 =  $3.9 \times 10^{-5}$ .

Nuclear gauging research is underway for FAA also at Westinghouse; recent reports from there have not been received. Discussion with Hurwitz has indicated that should this technique be used, spent nuclear materials would be exchanged for new specimens from the furnishing plant so the Government should not be required to dispose of used materials as hazardous materials.

G. Entine et al. of Radiation Monitoring Devices (private communication to H. J. Gryting) have developed a small detector with some capability for

detecting explosives as shown in Figure 1 and 2. In its present state, complex differentiations as indicated earlier would have the limitations noted for this mode. It could detect certain explosives in bicycle frames, for example, but is not a universal tool for all ordnance.

#### NUCLEAR MAGNETIC RESONANCE

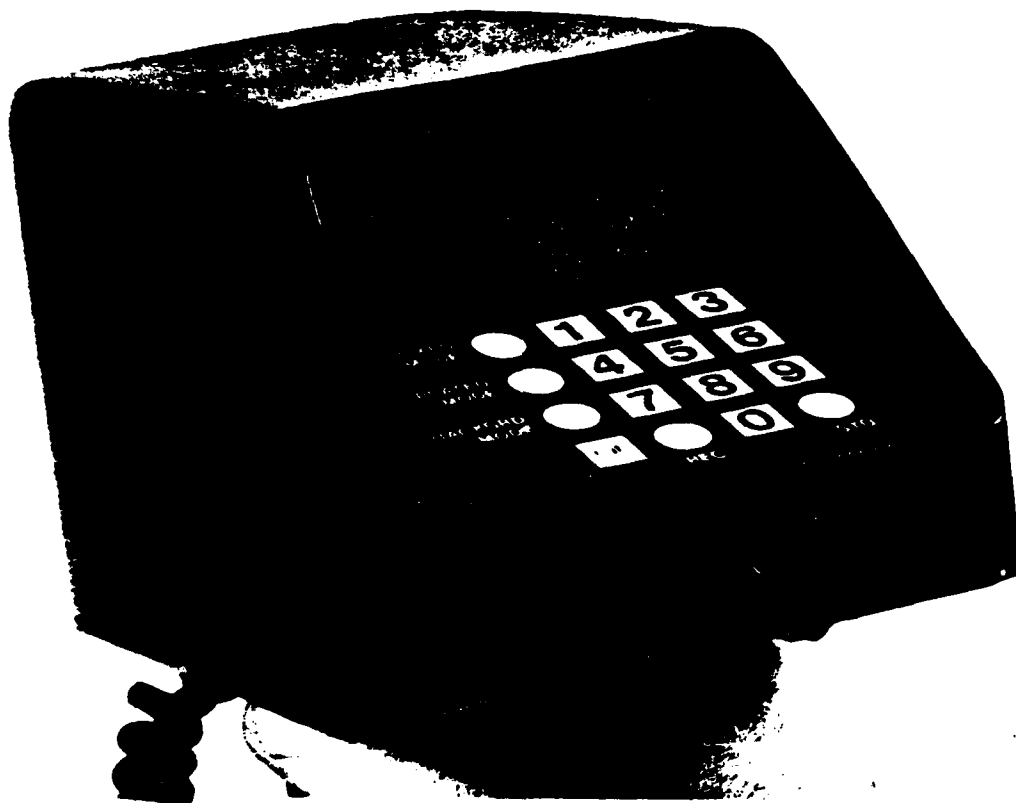
Since Ref. 1 was issued, there have been continuous efforts related to nuclear magnetic resonance and nuclear quadruple resonance mostly to improve the techniques and to extend the quantitative measurement capability to additional compositions. In work conducted during the last year, advances have been achieved that allow explosives to be detected more reliably and with fewer false alarms in imperfect magnetic fields. These advances resulted from efforts to improve the NMR system for inspecting baggage to detect concealed explosives but these should also be useful to detect explosives sealed in ordnance by means of making a small hole in the steel containment wall. In normal NMR, the magnetic effects of the steel casing on the signal obtainable with a small sensor probe inserted through a thick wall could seriously degrade the results. However, use of these more recent techniques along with adequate probe design could be instrumental in improving the probability of success with such probe measurements.<sup>(9)</sup> No effort has been conducted to the author's knowledge to determine explosives through small holes. A number of companies producing lasers have been asked whether their equipment could gently open a hole in a steel case without igniting an explosive or other energetic material. Not a great deal of optimism was expressed for such an approach.

Nuclear magnetic resonance for use in detection of explosives could be a viable concept providing the ordnance is not sealed totally. Metallic cases do not allow the electromagnetic waves to pass into the explosive unless there is a hole large enough for a small probe (size to be determined by the smallest transducer that is determined experimentally to be effective) to be inserted in such a way that the electromagnetic waves are not all adversely reflected or absorbed. Significant progress has continued in the field of nuclear magnetic wave use for quantitative determination of such explosives as the cyclotols and Composition B.

# **MODEL 8200**

## **Portable Contraband Detector**

### **Detects Hidden Contraband Without Disassembly**



The RMD Model 8200 Portable Contraband Detector is a compact, lightweight instrument for the detection of narcotics and currency hidden in the body panels, tires and seats of vehicles.

Developed under contract for the U.S. Customs Service, the Model 8200 uses a modern microprocessor-based technique to detect contraband beneath metal surfaces. The scanner may be used on all types of vehicles, aircraft, vessels and shipping containers.

The detection technique utilizes a nuclear flux, emitted from a small source directed into the volume of interrogation. The backscatter flux is measured using a highly sensitive solid-state detector and analyzed by digital electronics. The operator is alerted to the presence of contraband by an audible alarm as well as a numerical display.

Figure 1. RMD Explosives Detector

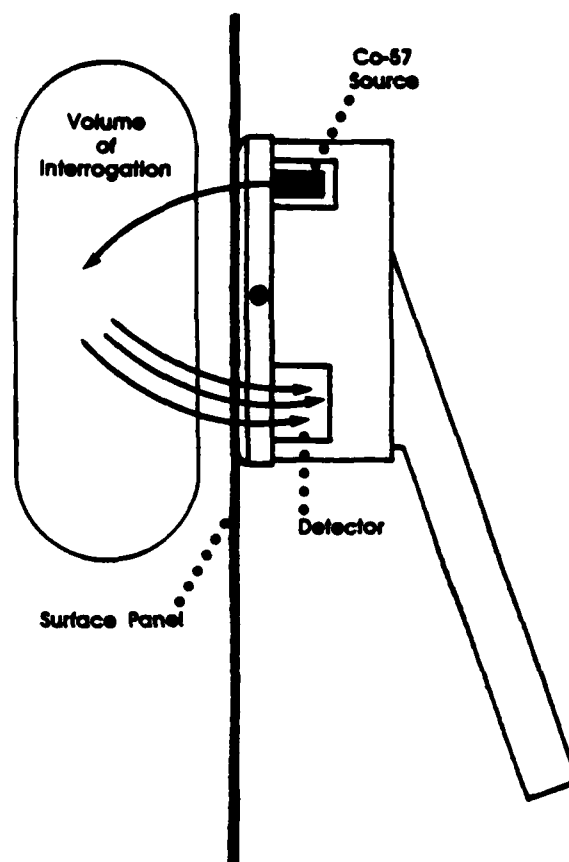
**RMD**

Radiation  
Monitoring  
Devices,  
Inc.

Advanced  
Instruments  
For  
Industry

## Features

- Fast:** A typical vehicle can be inspected inside and out in less than five minutes, including hollow body panels, tires, trunk and seats.
- Accurate:** State-of-the-art sensor and microprocessor signal enhancement allow positive identification of contraband targets.
- Highly Sensitive:** Narcotics packages of 4 oz. (0.1 kg.) and currency packages over 100 bills can be readily detected.
- Flexible:** The scanner can be used to locate a wide range of drugs and currencies.
- Easy-To-Use:** Requires less than two hours of operator training. Small Co-57 source needs no NRC license.



## Specifications

- Size:** 10" high by 9" long by 4" wide (25 x 23 x 10 cm)
- Weight:** 3.25 lbs. (1.5 kg.)
- Power:** Rechargeable NiCd batteries provide a full work week of operation before recharging.
- Display:** Alphanumeric with backlighting for night operation.
- Response Time:** 0.25 seconds.
- Scanning Rate:** 1 foot (0.3m)/second.

Figure 2. Characteristics of RMD Explosives Detector

**RMD**

Radiation  
Monitoring  
Devices,  
Inc.

44 Hunt St., Watertown, MA 02172  
(617) 928-1167  
Telex 951661 RMD WTN

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## X-RAY TECHNIQUES

Greater sophistication in X-ray techniques obtained in the last few years makes the measurements more meaningful including the successful identification of certain specific objects; however, the full differentiation between explosives loads and their inert simulants has not been achieved by these techniques.

Excerpts from the Cambridge FAA meeting summary indicate status of progress. X-Ray Fluorescence Spectrometry used in semi-quantitative analyses of known explosives compositions and the detection of metallic elements in small quantities is indicative of some of the branching techniques which are being developed.

### MAX-E System

A digital radiographic system, is a direct successor to <sup>133</sup>Ba gamma-ray transmission system. The MAX-E consists of a 1 KW constant potential X-ray source which is continuously variable over 40-140 KV, a luggage conveyor operated at 24 cm/sec, a linear array of 512 photodiodes coupled to CdWO<sub>4</sub> scintillators, a data acquisition system, a general purpose computer, and an array processor. This currently is an assembly/test stage. It is designed for two mm resolution over a 512 x 512 pixel image, 256 statistically significant grey levels and a target dynamic range of 1000:1. Explosive detection software is incomplete, and subject to FAA procurement.

### Pattern Recognition Software for MAX-E System

Eight proposals are under review by FAA.

Pattern recognition can apply to recognition of shapes (rectangles, cylinders, etc.) or it may apply to the recognition of density distributions. Pipe bombs and packaged high explosives may be recognized by shapes, whereas military explosives may be recognized as compact areas of low atomic number. Ability to determine average atomic number results from MAX-E capability to obtain dual energy data. False alarms may be obtained from organic solids such as cheese and liquids in plastic bottles. Explosives contained within glass or thin metallic containers could escape detections.

At the FAA seminar they felt the MAX-E approach should provide a meaningful although far from comprehensive explosives detection capability.

### X-Ray Fluorescence Spectrometry in Quantitative Analysis<sup>(10)</sup>

For low atomic numbers  $Z = 9$  (F) to  $Z = 14$  (Si) conditions used are: a Cr-X-ray tube is used with maximum current and voltage settings (50 KV, 40 ma). A large sample holder (3.18 cm diameter sample, pressed) is employed, and a TAP analyzing crystal with vacuum of  $<150 \mu$ . A flow counter (p-10 gas) with an ultrathin window ( $1 - \mu$  thick) (the TATB is pressed to  $\sim 1.5 \text{ g/cm}^3$ ).

For a TATB/KelF composition a minimum diameter particle for this PBX is 1000 - 2000  $\mu$  and penetration is about 1000  $\mu$  whereas the fluorine X-ray travels only 2 to 3  $\mu$  from where it was produced. Therefore the signal from fluorine is manifest at the surface. The signal will depend upon surface conditions and also upon local inhomogeneities (local variations of HE to binder ratios, particle orientation and flow during pressing operation).

Accuracy is restricted to within 10 or 20% for this PBX. Signal/noise ratio is poor and the lower limit of qualitative scan is  $\sim 4100$  ppm (minimum for detection).

For sodium the fluorescence yield is low and the lower detection limit is 80 ppm.

Mg lower detection limit is 250-300 ppm.

Al lower detection limit is 250-300 ppm.

Si lower detection limit is 250-300 ppm.

Si with change of crystal from TAP to EDdt  $\rightarrow$  50 ppm.

Phosphorous and sulfur  $\sim 10-15$  ppm.

Cl 270 ppm.

K 10 ppm.

Derivation of the relationship between X-ray intensity and the concentration of the element in the sample is given in the Appendix to Worley's article.

### METAL DETECTION TECHNIQUES

Morita<sup>(11)</sup> reviews and documents technical effort and results for identification and screening of promising remote sensing systems for detecting mines with emphasis on surface laid minefields with short detection reaction time. The minefields considered were in the European Theater.

Techniques and their highlights include:

- Aerial Photography - recommended for continued effort.
- Spot light Radar - may give quantitative data. (The 10.6μ active scanner was suggested for greater future emphasis.)
- Image intensifiers and TV devices - indicated as only having limited potential.
- Explosive Detection - Methods were not useful here as it is necessary to get close to the item to detect the explosive.
- Sight Systems - little or no potential.
- MTI and Pulse Doppler Radars - are capable of giving inferential
- Acoustic and Seismic Sensors - information - (recommendations on these techniques are not covered in this report).

#### THERMAL NEUTRON CAPTURE (WESTINGHOUSE) <sup>(5)</sup>

The Thermal Neutron Capture system utilizes a  $^{252}\text{Cf}$  source and 96 plastic scintillators, each 2-in. wide by 15-in. long to produce a mapping (~4 in. resolution) of the nitrogen distribution within a suitcase using the  $^{14}\text{N}(n,\gamma)^{15}\text{N}$  reaction. The emitted gamma-ray has an energy of 10.8 MeV and is higher in energy than most other neutrons emitted by common elements, although gamma-rays produced by iron, chlorine, and chromium do produce a significant background. This background is presently being compensated for by obtaining a better-defined spectrum with a NaI(Tl) detector. There is also a count problem from the 2.2 MeV hydrogen capture gamma rays. Consequently, increasing the source size would not improve the data acquisition time, which is currently minutes, unless changes are made in the neutron polyethylene moderator.

Nitrogen is contained in almost all explosives, as well as in wool, leather, nylon, orlon, cheese, and lean meat. However, the nitrogen concentration in explosives is considerably higher than the concentration in these innocuous materials. To date, field data obtained with this system has been quite limited, so background nitrogen concentrations and distributions in the checked luggage population are largely unknown. Preliminary results with this system showed approximately a 95% detection rate and an approximate 3% false alarm rate for one sample.

The use of the thermal neutron capture approach for air cargo warrants consideration, since cargo tends to contain large amounts of metal; and thus, is not readily inspected via x-ray techniques.

#### NONDESTRUCTIVE EVALUATION (SANTA CRUZ MEETING AUG 1983)

The Santa Cruz NDE symposium did cover certain detection aspects of tomography, including computer tomography NDE of solid rocket motors.<sup>(12)</sup> This pertained to the development of CT inspection capability of a range from small to 2.5 meter diameter solid rocket motors and components. One system, the AF/ACT-I, was operational early in 1983 and has been used to scan rocket motors and other aerospace hardware components for flaws. Scudder<sup>(13)</sup> describes a system that can make images of objects up to two inches in diameter and has a spatial resolution of 0.010 inch.<sup>(13)</sup> It has been used primarily for aircraft engine turbine blades and can also create good x-ray images of most industrial materials. Good contrast images can be made from materials ranging from light organics such as plastics to heavy metals as steel and copper.

Acoustics was one of the major subjects of the Santa Cruz NDE meeting.<sup>(14)</sup> Several people were contacted with respect to potential usefulness of such techniques. From what has been gleaned from the meeting and from discussions with a number of people no direct application to explosives detection or differentiation from inert simulants in sealed systems has yet been found for acoustics. Acoustic transmission and reflection spectra from inert and explosives would have to be determined and compared before viability could be either predicted or indicated as impossible. Discussion with Bruce Maxfield and Dick Bossi of Sigma Research where acoustic holography and ultrasonics studies are in progress for other purposes also indicated no current explosive detection capability by these means.

#### INTERNATIONAL SYMPOSIUM ON ANALYSIS AND DETECTION OF EXPLOSIVES

The FBI sponsored an explosives detection meeting at Quantico, Virginia in March (1983). Abstracts were sent by Terry Rudolph, however, the final

papers are to be compiled soon. Significant information relating to detection as concerns this project will be added during the time for accomplishment of Phase I. Attachment of these abstracts as Appendix I will indicate where much of the detection effort aimed at thwarting terrorists is going on.

When the papers arrive they will provide more information for comparing the potential utility of nonvapor vs vapor detectors for sealed explosives.

#### COMPARISON OF CANDIDATE NONVAPOR-EXPLOSIVES-DETECTION TECHNIQUES

Table I is an updated comparison for nonvapor detection techniques. The table was modified from an Aerospace Corporation table used previously in Ref. (1) by permission from Bob Moler.

#### CONCLUSIONS AND RECOMMENDATIONS

Within the nonvapor detector group, there appears to be most promise, at least in the near term, for those detectors based upon nuclear gauging techniques and on nuclear magnetic resonance together with its associated proton and electron magnetic resonance.

Some of the other possibly viable techniques will require considerably more basic research and for several the advances needed cannot be predicted. The need for excellent overall detectors becomes greater with the advent of many new types of explosives and with new situations. In addition to making our ranges and range clean up safer, the terrorist threat to our troops can also be lessened if we can detect, track, and halt illegal and/or enemy movement of explosives which perils our peacekeeping troops and friendly nations.

It is recommended that in addition to reviewing the vapor detectors (Phase I to be funded) that a detailed comparison of most promising nonvapor detecting methods, based upon nuclear gauging methods and nuclear magnetic resonance be made for the ten (or more) ordnance items considered jointly by SwRI and NAVSEA to be the greatest problem.

It is recommended that methods of safely making holes in ordnance cases without endangering the explosive toward ignition be initiated.

TABLE I. COMPARISON OF CANDIDATE NONVAPOR-EXPLOSIVES-DETECTION TECHNIQUES

METHOD	SCENARIO	TYPE OF BOMB	DETECTION CRITERIA	SPECIFICITY	RESPONSE TIME (sec)	COMPLEXITY	BOMBING RANGE SCENARIO: POTENTIAL SUITABILITY FOR EXPLOSIVES IN BOMBS VS INERT SIMULANTS	
Conventional X-Ray	Controlled Access	All	Density: Operator Interpretation	Low	2-5	Moderate		Poor
Automated Gamma/X-Ray Radiography	Controlled Access	All	Density: Automatic Shape Discrimination	Low	2-5	Moderate		Poor
Dual-Energy Gamma/X-Ray Transmission	Controlled Access	Pipe	Density: Atomic Number	High	2-5	Moderate		Potentially possible for some comparisons
Gamma/X/Neutron Transmission	Letters/ Flats	Plastic	Density: Atomic Number: Hydrogen Content	High	0.01	Moderate		Possibly for some
Gamma/X-Ray Scattering	Controlled Access	All	Density	Moderate	10-30	High		Feasibility demonstrated photon scattering 1981
Dual-Energy Tomography	Controlled Access	All	Density: Atomic Number	High	10-30	High		Some potential perhaps mostly for fuzes
Thermal Neutron Capture	Controlled Access	All	Nitrogen Content	Moderate	10-30	High		Information limited
Dielectric	Letters/ Flats	Plastic	High Dielectric Constant	High	0.01	Low		Poor
Nuclear Resonance	Controlled Access	All	Hydrogen Resonance Decay Times	Moderate	10-30	High		Steady progress access ports required
Radiometric/Imaging	Concealed Contraband Explosives on Body	All	Microwave (Neal) Millimeter Wave Reflection & Reradiation	Poor for explosives, good for metals	2-5	Moderate		Poor

This would allow greater ease of detection also by the vapor detectors as well as by NMR and possibly by nuclear gauging. In addition microscopic identification and use of chemical detection kits could be made practical for previously sealed weapons.

An analysis of all situations wherein terrorists or potential enemies can intrude with explosives into peacekeeping missions needs to be made to determine what methodology and instrumentation must be developed to solve this urgent problem.

## ACKNOWLEDGMENTS

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APPENDIX I

Abstracts from FBI International  
Symposium on Analysis and Detection of  
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#### THE TRACE ANALYSIS FOR EXPLOSIVES AND RELATED COMPOUNDS VIA LIQUID CHROMATOGRAPHY-ELECTROCHEMISTRY (LCEC)

The trace analysis for organic nitro compounds and explosives via HPLC and various detection methods has become quite popular within the past decade. However, it is only within the past few years that LCEC has been applied in the reductive mode for any explosives. This work has now been extended to cover a wide variety of explosive materials, and to real world samples of gunshot residues or post-blast explosion debris. At the same time, reductive LCEC has been applied to a number of other nitro derivatives, and derivatization for non-EC active substrates has been extended to form certain nitroaromatic derivatives off-line.

On the other hand, there has been no attempt to utilize oxidative LCEC for any nitro derivatives or explosives, since it is readily apparent that such materials will not undergo EC oxidation. In order to use this LCEC approach, it is first necessary to convert the initial explosive to a derivative product that would be EC active in the oxidative mode. However, normal derivatization approaches would use off-line techniques, followed by oxidative LCEC, a more difficult approach than on-line derivatization-LCEC. We have developed an approach that releases nitrite from organic nitro compounds after their HPLC separation and just before the EC detector. This release of  $\text{NO}_2^-$  as nitrite from the compound is then followed by oxidative LCEC - detection of the  $\text{NO}_2^-$ . There are several advantages of this approach to explosives detection, essentially in the on-line, real-time generation of an EC active derivative from an analyte that is not EC active at the start. These methods have now been applied to a variety of organic nitro compounds and explosives, and we have obtained calibration plots and detection limits. These methods could be readily applied to real world post-blast residues containing various combinations of explosives. At the same time, the overall methods could be readily applied to other organic nitro compounds including environmental pollutants, such as nitro-PAMs, veterinary drugs, pharmaceuticals, agricultural chemicals, industrial raw materials and final products, consumer products, and other sample matrices. The instrumentation for oxidative LCEC analysis of organic nitro compounds should be inexpensive and readily available. (This work was supported, in part, by an NIH Biomedical Research Support Grant, No. NS07143, to Northeastern University, DNS.)

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#### SCREENING FOR ORGANIC EXPLOSIVE COMPOUNDS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY WITH DETECTION AT A REDUCTIVE MEMBRANE ELECTRODE

The electrochemical detection of explosive compounds, separated by high performance liquid chromatography (HPLC), at a mercury film (thin layer) electrode (MFE) can be considerably improved both in terms of use and sensitivity of a reagent (hanging) mercury drop electrode (HMDE). The electrode characteristics are highly reproducible, the electrode may be renewed during or at the start of a chromatogram, and it is not subject to the contamination problems of the HMDE. With 5  $\mu\text{m}$  particle HPLC columns the detection limits for a wide range of nitrate and nitro compounds are in the range 7-49 ng per 20  $\mu\text{l}$  injected sample. These limits are approximately a tenfold improvement on those reported for the HMDE technique, and are comparable with those of electron capture detection (ECD) in gas chromatography, compared with which technique the MFE is superior in specificity.

A facile clean-up procedure has been developed to enable the MFE-HPLC technique to be used for screening handloads for traces of 75 organic explosive compounds. Examples of the application of the technique will be presented.

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#### DETECTION AND ANALYSIS OF POLYNITROPHENOLS IN WATER BY REVERSED-PHASE ION-PAIR LIQUID CHROMATOGRAPHY

The separation and quantitative analyses of mixtures of up to nine different polynitrophenols in water including both picric and etyphnic acids by ion-pair liquid chromatography are described. Using Pic-A reagent (t-butyl ammonium phosphate) to produce the counter cation in methanol-water systems, quantitative results were obtained at phenol concentrations as low as 0.1 ng/liter (0.1 ppm). Details of a preconcentration step for the analyses of polynitrophenols at the parts per billion (ppb) level are given.

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#### LIQUID CHROMATOGRAPHY/ELECTROCHEMISTRY DETECTION OF EXPLOSIVES. P.T. Kissinger, Department of Chemistry, Purdue University and Analytical Systems, Inc., West Lafayette, IN 47906.

Many commonly used explosive substances are electrochemically reducible to exhibit negative potentials (below -1.0 volt vs. Ag/AgCl). As a result, electrochemistry provides excellent selectivity for these substances because very few naturally occurring materials contain nitro groups. The combination of reversed phase chromatography with electrochemical detection therefore provides a unique opportunity to determine very small amounts (typically 1 ng) of various explosive substances. In this presentation, the principles and experimental practice of LCEC will be reviewed with specific reference to optimization of the quantitation and identification of individual explosive materials.

A series dual-electrode scheme was applied to the detection of explosive compounds in standard, gunshot residue and environmental samples. The series dual-electrode thin-layer transducer can extend the specificity and detection limits (for compounds reduced at higher energies) of the amperometric detector and can also provide better assurance of peak identity. In the case of polynitro aromatic explosives, the detection limits obtained with the dual-electrode transducer (signal measured at the downstream electrode) were higher by a factor of 3-4 than with a single electrode transducer because the decrease in the baseline noise did not fully compensate for the decrease in the electrolysis current at the downstream electrode. Operating the reductive LCEC system with a series dual-electrode transducer allows a direct injection of the sample solution without the need to remove dissolved oxygen prior to the injection. The described methodology permits detection of explosive compounds at detection limits below 10 ppb, depending on the particular compound.

LCEC appears to have significant advantages vs. gas phase techniques for the determination of nitro-based explosives for environmental and forensic purposes. The primary future direction is to improve reliability for the occasional user of the technique. When large numbers of samples need to be processed on a regular basis, the method is well established and few problems are encountered with dedicated instrumentation.

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#### CHARACTERIZATION OF PLASTICS, POLYMERS, AND EXPLOSIVES BY DIRECT SIZE EXCLUSION CHROMATOGRAPHY

This study was directed at the various plastics and polymers that can be characterized by gel permeation chromatography (GPC)/size exclusion chromatography (SEC). Many commercial products are fabricated exclusively, or contain, polymers that give the product its desired physical properties. Products such as automobile tires, tailights, and wire insulation are produced commercially by many companies. When analyzed, each product reveals a characteristic fingerprint or chromatogram that can be traced to the producer or origin of manufacture.

The same method would also apply to smokeless powders used in commercial ammunition, explosives, and their residual byproducts. Representative samples of these powders, explosives, plastic wedding, primer material, and bullet lubricants were characterized by this technique of GPC. The residue remaining after detonation was washed from the empty cartridge and barrel of the weapon and then analyzed. Comparisons were then determined. The same procedure was also applied for residue obtained from spent explosive material.

GPC is a predictable mode of analysis since it is a molecular sorting process based upon the size of molecules. In many instances direct comparison of the chromatograms can reveal significant differences between two supposedly identical materials. This is because the complete distribution of each molecular weight species, in order of molecular size, and in amount, is presented in the chromatogram. A size separation can be used on molecules with a molecular weight as low as 1000 up to the way up to large high polymers, in excess of 10 million MW.

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#### Identification of Reaction Products in Explosive Residues

Reaction products and unreacted components in explosive residues have been identified in test explosions of:

- i) "home-made" explosive mixtures of oxidizers and fuels,
- ii) high explosives of the "water-gel" type.

The "home-made" explosives were two-component mixtures of oxidizers (chlorates, perchlorates, nitrates) with fuels (sugar, sulphur, aluminum) and were ignited both confined and unconfined.

The high explosives were sticks of "water-gel" explosive produced by two different manufacturers. The composition of residues from these explosives is compared to residues from dynamite.

Residues were systematically analysed by routine solvent extraction methods and analytical procedures.

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#### IDENTIFICATION AND TRACING OF NON-EXPLOSIVE COMPONENTS IN EXPLOSIVES Harold Mueller, Metropolitan Police Department, 1200 Clark Avenue St. Louis, Mo. 63103 Telephone 1-314-444-5837

Non explosive components retrieved from bomb scenes frequently can be of value in determining the type of explosive device used. Additionally information may be gained to characterize these components for tracing possible origins.

Methods of analysis for these components are those frequently utilized in forensic laboratories. Examples given will include macroscopic examinations, x-ray fluorescence, infrared and pyrolysis G.C.

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#### EXPLOSIVE ANALYSIS KIT

An explosive analysis kit has been developed to allow the timely field separation and identification of 26 different explosives and energetic materials. The kit uses solubility and thin layer chromatography (TLC) to separate mixed explosives and identify each component.

The kit, with sufficient materials to perform ten (10) analyses, is packaged in a briefcase. The solvents for sample dissolution and TLC separation are packaged in single-use lead tubes to ensure composition and lack of contamination. The kit is assembled from commercial components, with custom packaging required for filling and sealing the lead solvent tubes. A battery-powered UV light visualizes the TLC plates, eliminating the need for corrosive reagents and ambiguous color-forming reagents. A worksheet provided with the instructions guides the operator through the analysis and is used to record data and help make the identification. The kit identifies the components of booster and main charge explosives, as well as selected oxidizers, propellants, and other energetic materials.

After limited training, 62 U.S. Marine Corps ESD technicians tested the kit. Their performance was good, and their reaction to the kit was very favorable.

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**ORGANIC SOLVENT SEPARATION OF EXPLOSIVE RESIDUES: CLEAN-UP TECHNIQUE.** Richard A. Strobel, Richard E. Tantaraki, Kenneth Chabata, Forensic Science Branch, Bureau of Alcohol, Tobacco and Firearms, 1401 Research Blvd., Rockville, MD 20850

A number of sensitive techniques exist today for the detection of explosives. However all their sensitivity and specificity may be lost when "dirty" or "real world" samples are subjected to analysis. Improved chromatographic techniques and sensitive detectors have aided the situation. Unfortunately, many of the "real world" samples encountered contain a complex mixture of interfering substances. The chromatography system can not adequately separate the explosives of interest from these contaminants. These "dirty" samples require a pre-treatment prior to being subjected to chromatography and detection. The use of porous polymers and bonded phase adsorbents as a clean-up procedure is explored in this work. Retention properties of both explosives and contaminants are studied, and an analytical scheme is presented which optimizes the separation of the explosive from the contaminants.

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#### A Scheme for the Analysis of Explosives and Explosive Residues

The FBI Laboratory has developed a new scheme for the analysis of explosive residues. This scheme is based on a water and/or organic solvent wash of bombing debris. Inasmuch as several high explosives such as dynamite and water gel/slurries contain both organic and inorganic species, it is often necessary to fully identify the explosive, to perform both washes on the debris. This scheme highlights the use of X-ray powder diffraction and ion chromatography (IC) for the analysis of the water wash. Such hard to identify inorganic species as ammonium nitrate can easily be identified with the IC methods reported. The organic solvent wash highlights the use of HPLC and GC/MS methods. HPLC methods utilize both normal and reverse phase chromatography and variable UV wavelength and TMA detectors.

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#### INITIAL DETONATION BEHAVIOR OF SUSPENDED EXPLOSIVES AS OBSERVED FROM UNREACTED RESIDUES

##### ABSTRACT

An investigation was conducted by the Bureau of Mines to recover, collect, and identify the solid explosive products of commercial explosives. These condensed products can include unreacted, partially reacted, and completely reacted ingredients.

Various unconfined commercial explosives were fired suspended in a sphere in air. The solid residues were collected and studied. Unexpectedly large amounts of residues were found. In general, the granular explosives produced more residue than the semisolid and water

gels. Many of the residues were found to be thermally reactive, not unlike the original explosives, when they were evaluated by thermal analysis tests. The residues from the water gel explosives were the least reactive thermally. Preferential consumption of the ingredients was indicated by wet chemical analysis of a few of the residues.

Residues were also collected from two semiconfined charges and one confined charge fired in cannon tests. As expected, the amounts of these residues were much smaller.

A good inverse correlation was found between the amount of unreacted residue and the square of the ratio of unconfined and confined detonation velocities, (D unconfined/D confined), in agreement with the hydrodynamic theory of detonation.

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**Instrumental Techniques Utilized in the Identification of Smokeless Powders.** Proton Magnetic Resonance (PMR) and Gas Chromatography (GC). RICHARD E. MEYERS, M.S., and JOHN A. MEYERS, B.S., Forensic Science Branch, National Laboratory Center, Bureau of Alcohol, Tobacco and Firearms, and the Drug Enforcement Administration North Central Field Laboratory, respectively.

An approach to identifying the manufacturer of domestic commercial smokeless powders has been evaluated at the Bureau of Alcohol, Tobacco and Firearms (ATF) Headquarters Laboratory and the Drug Enforcement Administration (DEA) North Central Field Laboratory. The procedure utilizes a combination of proton magnetic resonance (PMR), and Gas Chromatography (GC).

The use of PMR permits discrimination between similar products of the major U.S. manufacturers, namely, Dupont, Hercules, or Winchester-Wadsworth. Using PMR alone, some discrimination can be made within a particular manufacturer's products. The GC profile permits the observation of minor components. When PMR is combined with GC one is able to also identify each type within the manufacturer. These results were obtained using undetonated samples.

This presentation will discuss the differences in both the PMR spectra and GC Chromatograph, to include variations seen in products from different manufacturers and those observed in products from a particular manufacturer. Examples of the results showing both similarities and differences will be given. Application of the techniques to typical forensic problems will be described.

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#### THE USE OF MULTIPLE DETECTION IN THE GAS CHROMATOGRAPHIC ANALYSIS OF ORGANIC NITRO COMPOUNDS AND EXPLOSIVES (GC-ECD/PID)

The trace organic analysis for nitro derivatives and explosives has traditionally been hampered by a lack of suitably selective and sensitive detectors. Except for the mass spectrometer, most other GC detectors are not suitably selective for nitro compounds to provide for unambiguous identification of trace amounts. In recent years, various workers have applied a combination of detectors, in series or parallel, for improved compound identification. We have utilized a parallel arrangement of electron capture detection (ECD) and photoionization detection (PID), together with certain Permapond GC packing materials, for the improved resolution and specific identification (speciation) of numerous organic nitro compounds and explosives. The combination of GC-ECD/PID for nitro derivatives provides relative response factors (RRFs) vs a common internal standard, as well as ratios of ECD/PID RRFs that are often unique for individual nitro compounds. We have applied these analytical methods to a wide variety of nitro derivatives, including: mono-nitro toluenes, dinitrotoluenes, dinitrobenzenes, nitro aliphatics, nitro-PAMs, PAMs, and various explosive compounds. Separations of mixtures of the aromatic nitro compounds, PAMs, nitro-PAMs, or explosives were obtained using packed glass columns of Permapond Methyl Silicone Permapond PEG 20M, and/or Permapond PAM packings. The combination of ECD and PID provides for vastly different RRFs in comparing the PAMs and nitro-PAMs, so that the normalized RRFs for the ratio of ECD/PID responses become vastly different on an absolute scale. Thus, in certain cases, PAMs and their nitro-PAM derivatives can exhibit 3-7 orders of magnitude differences in their RRFs for ECD/PID ratios.

The use of combined ECD/PID ratios and RRFs in GC analyses for organic nitro compounds and explosives provides a unique method of utilizing readily available and inexpensive detectors for improved analyte identification and speciation at little added overall cost. These methods are directly applicable to environmental samples having trace amounts of nitro derivatives and/or explosives. GC-multiple detection is therefore a very viable and useful method for the trace analysis and speciation of these classes/types of compounds. (This work was supported, in part, by an NIH Biomedical Research Support Grant, No. RR07143, to Northeastern University, DMS.)

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Section 1 - explosive analysis  
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#### Determination of Nitro Explosives by Gas Chromatography Utilizing an On-Column Capillary Injector

Nitroglycerin and other nitrated esters are important compounds because of their use as explosives and as drugs in the treatment of cardiac disorders.

The compounds have been determined by gas chromatography using conventional packed columns but the analysis can be quite difficult. Temperatures must be carefully controlled and the column must be prepared and conditioned very carefully so that active sites on which the nitrated esters can decompose are eliminated.

Capillary columns made of fused silica (pure synthetic SiO<sub>2</sub> without metal contaminants) have been found to be of value when analyzing reactive compounds. When combined with a capillary on-column injector,

Some compounds that could not be analyzed by gas chromatography or could only be determined with great difficulty can now be measured.

In this study, an electron capture detector was used and nitroglycerin was determined down to sub-picomogram levels. The sample was injected directly into the fused silica column under non-vaporizing conditions. There was no sign of tailing or decomposition. Linearity of response was obtained over a concentration range of  $10^{-3}$  and the correlation coefficient to a straight line was 0.997. Use of the non-vaporizing on-column injector was critical - nitroglycerin could not be detected when a splitless injector was used.

In the discussion, the on-column injection technique will be described and contrasted with the older capillary injection techniques (split and splitless). Problems that one might encounter will be mentioned and results with other explosives such as nitramine and pentaerythritol tetranitrate will be given.

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#### THEORETICAL ANALYSIS OF PYROTECHNICS AND EXPLOSIVES

Differential thermal analysis (DTA) has proven to be a valuable tool for the identification and qualitative analysis of pyrotechnic and explosive mixtures as well as for the investigation of the chemical mechanisms of pyrotechnic reactions.

In DTA, processes that absorb heat from the surroundings, such as melting, boiling, and crystalline phase transitions, produce downward peaks, termed endotherms, in a plot of  $T$  versus  $DT$  (temperature difference between sample and thermally-inert reference material). Processes that release heat, such as exothermic reactions, produce upward peaks (exotherms) in the plot. The resulting diagram, upon heating from some temperature to 500, 800, 1200° or higher, is termed a **thermogram**.

The pattern for a particular pure material yields a thermal "signature" that can be used for qualitative purposes and for purity determinations. The thermogram also yields information regarding the thermal stability of new materials and mixtures.

The thermogram of a pyrotechnic or explosive mixture is a combination of the thermograms of the individual components, up to the ignition temperature of the material. At that point, a strong exothermic peak is observed corresponding to the occurrence of a self-propagating reaction.

Typical thermograms of pyrotechnic and explosive materials will be presented, and some of the chemical implications regarding ignition behavior will be discussed.

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#### Identification of Two Rare Explosives

Two unusual and rare explosives were identified in two separate cases of terrorist activity. The two explosives were identified as hexamethylenediamine peroxide and triacetone triperoxide. The identification was based upon interpretation of spectral characteristics of the compounds. These included mass spectrometry under electron impact (EIMS) and chemical ionization (CIMS) conditions, infrared (IR) spectroscopy and nuclear magnetic resonance (NMR) spectroscopy. The explosives were first identified when no known spectra of them were available in our laboratory library. Their identity was later confirmed by comparison with data from literature; the comparisons included melting points and IR spectra.

The two explosives, which are organic peroxides, were described extensively in older literature, but their current use as military explosives has not been reported. Although the explosive properties of the two compounds correspond to those of primary explosives, one of them, triacetone triperoxide, was employed by terrorists as a main charge.

Special significance should be given by law enforcement agencies to the simplicity of preparation of the two explosives, as well as to the ready availability of the starting materials needed for their synthesis. In the case of triacetone triperoxide, the preparation was described in the testimony of an apprehended terrorist.

An interesting point which could be relevant to the detection of these explosives by x-rays is that contrary to common primary explosives (e.g., lead azide or mercury fulminate), these peroxide explosives contain no metallic elements. Therefore, their presence cannot be detected by standard airport security procedures.

#### IDENTIFICATION, STRUCTURAL DETERMINATION, AND QUANTIFICATION OF AN UNKNOWN EXPLOSIVE

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The methodologies employed in the identification, structural determination, and quantitation of the constituents of an unknown explosive mixture will be described. The techniques used include chemical tests, elementary analysis, thin-layer chromatography (TLC), infrared absorption spectroscopy (IR), ultraviolet-visible absorption spectroscopy (UV-VIS), nuclear magnetic resonance (NMR), gas chromatography/mass spectrometry (GC/MS), positive and negative ion chemical ionization mass spectrometry (PFI/MS), and high resolution electron impact mass spectrometry (HR/MS).

The structural determinations were based primarily on the MS/MS data along with chemical stability considerations, the molecular weight and the structural data derived from PFI/MS, IR, and UV. The positive identification of the unknown was made by synthesizing the compound and comparing its characteristics with those of the unknown in question by various instrumental techniques. Both IR and NMR were used in the composition analysis.

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#### Analysis of an Unusual Explosive: Methods Used and Conclusions Drawn from Two Cases

Samples of explosive material are often submitted to the FBI Laboratory for identification. In most cases, the sample is quickly identified by Gas Chromatography/Mass Spectrometry analysis and, if a sufficient quantity of pure substance is available, by Infrared Spectroscopy. The resulting spectra are compared with library spectra or spectra obtained from known samples in the laboratory. When no matching spectra can be found among these resources, the examiner must deduce the molecular structure of the material by using fundamental chemical knowledge and by employing whatever appropriate spectroscopic techniques are necessary. The choice of those techniques will depend both on the quantity of material available and on the complexity of the structure.

Two recent cases will be discussed with emphasis on the methodologies used by the examiners.

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#### DESCRIPTION OF A NITRO/NITROSO SPECIFIC DETECTOR FOR THE TRACE ANALYSIS OF EXPLOSIVES

A nitro/nitroso specific detector for both capillary column gas chromatography (GC-TSA) and high-performance liquid chromatography (HPLC-TSA) is described.

Sensitivity is typically better than 10 picograms for such compounds as nitroglycerine, 2,4-DNT, TNT, and RDX. At 0.1 ng level the coefficient of variations are  $\pm 4.1\%$ ,  $\pm 3.5\%$ ,  $\pm 3.6\%$ ,  $\pm 3.4\%$  for NG, 2,4-DNT, TNT, and RDX, respectively.

Confirmation as to the identity of the compound under suspicion is achieved by the use of parallel GC-TSA and HPLC-TSA analysis.

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#### APPLICATIONS OF THE NITRO/NITROSO SPECIFIC DETECTOR TO EXPLOSIVE RESIDUE ANALYSIS

The specificity of the TSA<sup>®</sup> Analyzer interfaced to a gas chromatograph (GC-TSA) and/or a liquid chromatograph (HPLC-TSA) renders the technique a useful tool for the analysis of explosive residues in a wide variety of forensic and environmental applications. Specific applications to the analysis of explosive residues will be described, including post explosion debris and washings from persons who have handled explosives. Examples of environmental data will also be included.

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#### X-RAY PHOTOELECTRON SPECTROSCOPIC (XPS) DETECTION AND IDENTIFICATION OF EXPLOSIVE RESIDUES

X-ray Photoelectron Spectroscopy (XPS) is a sensitive and powerful analytical technique with which residues in the nanogram range over an area of a square centimeter can be detected and identified. The spectrum of nitrogen is particularly useful because the chemical shift of the line by the oxidation state helps distinguish between the nitrate ester, nitro, nitroso and amine groups. Their relative ratios in the debris can be determined. Also, the modes of molecular fragmentation of different explosives provide fingerprint spectra. Combined with thin layer chromatography, XPS was proved to be a highly successful technique for forensic and malfunction investigations. Examples of specific applications are given.

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#### Characterization & Identification of Water Soluble Explosives by Light Microscopy

This paper describes a simple technique whereby water soluble inorganic explosive compounds are characterized by their crystal shape, size, and interfacial angles as they recrystallize from a drop of water on a microscope slide. Once the crystals are characterized, the inorganic explosive compound(s) can be confirmed by other optical properties such as refractive indices, extinction angles, and birefringence, and/or by conducting microchemical tests. The advantage of this procedure is that it is fast, inexpensive, only a small amount of sample is needed, and does not require a broad knowledge of optical crystallography. A discussion of the characteristic crystals and the identification of the common inorganic explosive compounds will be presented. The application of the technique to cases received in the forensic laboratory will be demonstrated.

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#### Identification of Water-Soluble Explosives and Their Post-Blast Residues by Ion Chromatography

Explosives which contain primarily water soluble ingredients are frequently encountered by the forensic scientist. Determining the identity of the many slurry explosives now being used in a growing number of bombing cases is one area where ion chromatography (IC) has been most useful. There are numerous commercial dynamites and blasting agents sold by approximately a dozen major manufacturers which are all comprised primarily of  $\text{H}_2\text{N}_2\text{O}_3$ ,  $\text{KNO}_3$ , and  $\text{NaNO}_3$ , with varying amounts of water and both ionic and non-ionic additives. If the wrapper has been removed from these products before they are incorporated into an improvised explosive device (IED), their identification can be a formidable task.

With the voluntary cooperation of explosive manufacturers, the FBI Laboratory is building a collection of commercial products most likely to be used in IED's. In order to analyze these explosives, the Laboratory has developed new and novel procedures for sample preparation and has developed IC procedures for some ions which were not previously determined by IC.

The extremely high sensitivity and selectivity of IC makes the technique extremely valuable for the analysis of post-blast residues. Following extraction of the debris with water or a water-methanol solution, the extract is simply filtered and run on the IC under conditions identical to those used for the analysis of the undetonated explosive. We have shown that this form of analysis offers several advantages over other techniques when the ionic residues are volatile or electrochemically active.

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THE USE OF ION CHROMATOGRAPHY IN THE ANALYSIS OF WATER SOLUBLE EXPLOSIVES. D. J. Barnett, R. H. Hoffman, R. P. Wenger. This method involves the use of 10%  $\text{HNO}_3$  to break down the crosslinked network of the water gel. The resulting solution is filtered to remove the insoluble components, i.e. glass, perlite, hydrolyzed wax and organic fuel. The filtrate is diluted to decrease the individual ion concentrations to approximately 100 ppm. Two sets of chromatographic conditions are necessary for a complete analysis, one set for the monovalent cations  $\text{Na}^+$ ,  $\text{CH}_3\text{NH}_3^+$ ,  $\text{NH}_4^+$ , etc. and the multivalent ones such as calcium, etc. Using dual column, dual conductivity detector ion chromatography the concentration is determined using a previously determined response factor for each ion of interest. The described method will reduce the analysis time from several hours to thirty minutes. The precision (2%) is 50 relative.

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#### The Characterization of Some Low Explosive Residues by Ion Chromatography (IC)

A large percentage of improvised explosive devices encountered in bombing matters received by the FBI Laboratory involved the use of low explosives such as black powder, potassium chlorate/potassium nitrate and the commercial black powder substitute, Pyrodex. The combustion residues of most of these low explosives are inorganic in nature and water soluble. Because of this water solubility they can be analyzed both qualitatively and quantitatively by IC. The characterization of several different low explosives by IC is reported. This analysis provides a simple and rapid identification for these residues.

#### Identification of Explosives Containing Alkylammonium Nitrates by Thin Layer Chromatography

The sensitizers, dimethylamine nitrate (DMAN) and mono-ethylamine nitrate (EMAN), contained in duPont and Hercules ester gel explosives respectively, can be uniquely identified in evidentiary samples from bombings by utilizing the three thin layer chromatography (TLC) systems discussed in this paper. These TLC methods also identify the presence of other explosive ingredients and contaminants commonly found in debris from bombings.

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#### ANALYSIS OF EXPLOSIVES BY LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY

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In many applications of forensic analysis, an analytical method is required which combines good separation characteristics with highly specific and sensitive detection. The Liquid Chromatography/Mass Spectrometry (LC/MS) system has such specifications and has an advantage over GC/MS in that it is suitable for thermally sensitive and involatile compounds.

We have interfaced an HPLC with a magnetic sector mass spectrometer. The mass spectrometer is a home built 90° 4-inch radius magnetic sector instrument with a high-speed differential pumping system. The HPLC consists of an Eldex High Pressure Pump, an Eldex Solvent Programmer, a Rheodyne Model 7125 Sample Injector and a Waters 441 UV Detector. The column used was a RP-8 reversed-phase column. Mobile phases were methanol:water and acetonitrile:water at various relative concentrations.

The LC/MS interface is a commercial Hewlett-Packard Direct Liquid Insertion Probe LC/MS interface which is a variable split-type interface.

A series of standard explosive mixtures including TNT, RDX, IMX and NG, as well as commercial explosives have been analyzed by this LC/MS system. LC/MS spectra of these explosives will be shown in order to demonstrate the usefulness of this technique in forensic analysis.

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#### The Analysis of Post Detonation Carbon Residues by Mass Spectrometry

In the forensic analysis of specimens from scenes of explosions it is usual to find some traces of the undetonated explosive. However, it has proved impossible to detect and identify 2,4,6-Trinitrotoluene (TNT) in residues by the normal methods of rubbing followed by Gas Chromatography of the extract.

As an oxygen deficient explosive TNT deposits carbon in the form of particles on surfaces in the vicinity of the site of the explosion, and it was considered that this carbon could provide a means of identifying the explosive, since decomposition products from the detonation could well be trapped within it. Samples of carbon were examined by Mass Spectrometry using a pyrolysis probe. The detailed method of sample preparation will be described together with an account of the problems encountered. By flash heating the carbon sample in the pyrolysis chamber, which was directly connected to the Mass Spectrometer source, it was possible to examine the materials released. Our initial studies indicated that unreacted TNT was present and could be identified.

These preliminary studies produced a success rate of 20% and further work has improved the technique considerably. This has included modifications to the probe design to permit its use in the Chemical Ionization and Negative Ion modes. The modifications and their effects will be described and the implications for the use of the technique with other carbon depositing explosives such as RDX and PETN will be discussed.

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ON-LINE COMPUTER SEARCH SYSTEM APPLIED TO EXPLOSIVES  
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The analysis of explosive residues by electron impact gas chromatography/mass spectrometry and on line computer searching of spectra will be presented. A microprocessor based GC/MS system and related software was used to approach the problem. Real time computer searching of G.C. peaks utilized a pre-selected library of explosive spectra. Methods of sample collection and preparation will be reviewed.

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### Identification of Smokeless Powders and Their Residues by Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS)

Until recently, forensic comparisons of smokeless powders were made based upon physical properties (e.g., size, shape, color) and positive matches would be tentative, especially in the case of burned residues. For the last several years, comparison and identification of both burned and unburned smokeless powder residues have been done in the Federal Bureau of Investigation Laboratory by a combination of physical comparison and chemical analysis using high performance liquid chromatography. Examining both the chemical and physical properties of the smokeless powders allows a more definitive comparison or identification. This presentation will describe a new technique for comparing and identifying smokeless powders based upon the analysis of the trace organic constituents by capillary column GC/MS.

The chemical examinations consist of extracting the powder or powder residue with chloroform, and separating and identifying the soluble constituents with a fused silica capillary column GC/MS system equipped with a cold on-column injector. Although this procedure can resolve smokeless powder extracts into as many as 30 major and minor components, only the major components are used for the chemical comparisons. On our GC/MS data system, we have established a smokeless powder "library" (representing about 100 powders) with each entry being a composite spectrum generated by merging the spectra of the major peaks found in the powder extract. Identification of a smokeless powder is effected by computer searching the composite spectrum of the questioned powder against the library. Confirmation of the computer identification is made by comparing the relative amounts of the various components as found by the GC/MS analysis, and by comparison of the physical properties of the known and questioned smokeless powders.

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### FINGERPRINTS OF DETONATION PRODUCTS FROM NAVY EXPLOSIVES

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Detonation products of explosives in air and nitrogen are being studied at the Naval Weapons Center mainly to learn what toxic compounds are formed in underwater detonations. We have seen a large variety of molecules and particulates found in the detonations. These compounds differ between TNT and PETN explosives and it seems very possible that they can be used to fingerprint the explosive used.

Detonations in air and nitrogen atmospheres have been carried out in a steel-lined room where we are able to collect the product gases, vapors, and particulates. The nitrogen atmosphere explosions produce much more understudied products and should simulate underwater conditions and products. There is a sufficient amount of the products that it would be collectible in the water from the vicinity of an underwater detonation for a reasonable time after the explosion. This water could then be analyzed by any GC/MS. The GC peaks would give a fingerprint which could be compared to those found in our studies.

Our presentation will show the fingerprints given by TNT and some polymer bound explosives when detonated in air, collected, and analyzed using a gas chromatograph/mass spectrometer.

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### The Analysis of Trace Levels of Explosive by Gas Chromatography/Mass Spectrometry

The identification of trace levels of explosive is a problem faced by forensic analysts. In principle the Mass Spectrometer is a very powerful tool for use in these analyses, since considerable structural information can be obtained from the mass spectrum. However, when dealing with explosives in a conventional Mass Spectrometer major problems are experienced due to the extensive fragmentation, particularly with the non-aromatic nitrate esters and nitrocompounds such as nitroglycerine and RDX. Much use can, however, be made of this limited information by the use of a high efficiency capillary Gas Chromatograph coupled to the Mass Spectrometer. Some of the problems will be described and the detection limits possible with Single Ion Monitoring of the most abundant ion (typically in the low m/z range) will be discussed.

Other forms of ionization are possible, however, and the application of the negative ion mode will be described. This technique is particularly appropriate to the analysis of explosives, since electron capture is the principal mechanism of ionization, and common explosives are strongly electron capturing, a property which can be used in their Gas Chromatographic analysis. Not only does the negative ion mode produce improved detection limits, but also introduces a degree of discrimination not available in conventional Mass Spectrometry. The implications of these newer developments in Mass Spectrometry for the analysis of explosive traces will be discussed.

### Analysis of Explosives and Explosive Residues with Ion Mobility Spectrometry (IMS)

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Techniques for the analysis of explosives with Ion Mobility Spectrometry will be described. These include vapor sampling, solvent extraction, adsorption/desorption, and sample heating. Ion Mobility Spectrometer instrumentation required to support these techniques will be discussed.

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The analysis for ethyleneglycolmononitrate and monomethylamine nitrate in commercial blasting agents and post blast samples.

The commercial blasting agents, POWEREX (C.I.L.) and TOVEX (DuPont) have been encountered in a number of seized, disrupted or hoax explosive devices and have been suspected in a number of bombing incidents. Work has been done to develop a procedure to detect the sensitive ethyleneglycolmononitrate (EGMN) from Powermax and monomethylamine nitrate (MMAN) from Tovex using high performance liquid chromatography.

Analysis of samples of debris recovered from test blasts has been successful after using an appropriate pre-concentration technique.

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High pressure liquid chromatography (HPLC) is gradually supplanting thin layer chromatography (TLC) as a tool in the identification of explosive residues. Debris from explosion scenes is often extracted using bulk solvents or by headspace concentration and analyzed by HPLC. Identifications are made by comparison of retention time data with known standards. It would be desirable, however, to develop a more specific method for identification of these elements using Fourier Transform Infrared Spectroscopy (FTIR).

The tremendous separatory power of HPLC allows the analyst to "prep" each component of the explosive residue in suitable purity and quantities to generate useful infrared spectra. Normal phase HPLC with 5 micron silica columns is particularly useful because eluents are then composed of organic solvents which are readily evaporated to yield a film on KBr plates or a pressed KBr pellet of the sample.

Since FTIR has proven to be considerably more sensitive than dispersive IR, the FTIR systems can be used as a real-time chromatographic detector. Using ultra-micro HPLC flowcells (0.2 ul volume) as detector cells, the FTIR becomes an on-the-fly detector. Special software such as the Nicolet chemigram programs and liquid nitrogen cooled Mercury Cadmium Telluride detectors serve to provide a very selective detector sensitive only to changes in absorptions in a narrow infrared wavelength region. Alternatively, the full spectra can be taken of the eluent on-the-fly. Subtraction routines then can be used on this data to remove the contribution the eluent solvents to the eluate spectrum. The resultant spectrum then can be confirmed by comparison with reference spectra via a computer library search of various standard explosive infrared spectra.

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### The Analysis of Smokeless Powders Using High Performance Liquid Chromatography

The analysis of smokeless powders or propellants have been of long interest to the forensic examiner. Smokeless powders contain not only explosives such as nitroglycerine and nitrocellulose but also stabilizers, gelatinizers and their various decomposition products which are thermally labile. High Performance Liquid Chromatography allows their accurate characterization and quantitation. By using tandem UV/TEA detectors these compounds can be analyzed in the low nanogram range which is a requirement for some forensic applications. Diphenylamine, 2-nitrodiphenylamine, 4-nitrodiphenylamine, nitroglycerine, 2,6-dinitrotoluene and 2,4-dinitrotoluene have been separated, identified and their relative quantities used to characterize the gun powder.



# DETECTION OF EXPLOSIVE RESIDUES BY MICROBORE HPLC

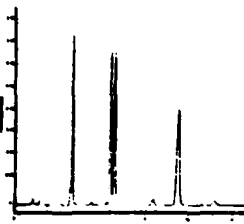
Microbore HPLC is, in most cases, a less sensitive technique than conventional bore HPLC due to limitations of injection size and detector cell volume needed to maintain resolution using these narrow bore columns. However, in cases where the absolute amount of sample is limited, microbore HPLC can provide up to 20 times greater mass sensitivity from a given sample because the lower flow rates result in less dilution of a fixed sample size. Examples illustrating this point, including advantages and limitations, will be presented.

Sample resulting from the total extraction of explosion wreckage are extremely complex and filthy. Column lifetime analyzing such samples can be as short as one run. The cost per analysis is then quite high. Microbore columns contain less than 5% of sorbent and if trashed can be replaced easily in less than half an hour. A description of a packing system and detailed procedures for the packing of microbore columns will be presented.

Figure 1  
Explosives by Microbore

Column: 90 cm x 1 mm  
Lichrosorb RP-18,  
10 micron  
Solvent: 50/50 MeCN/H<sub>2</sub>O  
Flow Rate: 80 µl/min  
Pressure: 1000 PSI  
Detection: UV @ 210 nm

- (1) RDX 120 ng
- (2) TNT 120 ng
- (3) DNT 150 ng
- (4) PETN 200 ng



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# DIFFERENTIATION OF NITRATE ESTERS, NITRAMINES, NITROAROMATICS, AND THE ANALYSIS OF BIOLOGICAL FLUIDS BY LIQUID CHROMATOGRAPHY WITH A NITRO/NITRONE SPECIFIC DETECTOR

As a consequence of work with cardiovascular drugs, the capability now exists to evaluate the potential occupational hazard associated with human exposure to explosives via skin contact and/or vapor inhalation in biological fluids. A technique using the TNA Analyzer interfaced to a high-performance liquid chromatograph (HPLC) is described for the trace level determination of nitroglycerin, pentaerythritol tetranitrate, and their metabolites in blood and urine. The method developed is capable of detecting 0.1 nanogram of each of the nitrate esters, corresponding to a detection limit of one part per billion (ppb). The precision of the HPLC-TNA method at 1 ppb level was established to be 7.4% and 5.7% relative standard deviation (RSD) for nitroglycerin and pentaerythritol tetranitrate, respectively. Analytical methodology developed for the detection of ethylene glycol dinitrate (EGDN), triallatolone (TWT) and RDX in biological matrices and wastewater effluents are also discussed.

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# Explosive Residue Detection by Liquid Chromatography Using An Electrochemical Detector

Reductive and oxidative electrochemical detection with liquid chromatography is applied to the determination of nitro aromatics, nitrate esters, nitramines, and diphenylamines in military explosives and double base smokeless propellants. A sensitive and highly selective method is presented for the detection of organic "gunshot residue" on the hand of individuals who have discharged a weapon. The detection limits as 5/10-3 are of the order of 0.5, 1, 2, and 0.3 picogram for nitro aromatic, nitramine and nitrate ester explosive compounds, and diphenylamines, respectively.

In the last five years, oxidative made electrochemical detection in liquid chromatography has become widely accepted for solving many problems of clinical, pharmaceutical, and environmental interest. Several reviews have been published on the advantages resulting from the combination of liquid chromatography with electrochemical detection (L.C.-e.c.). Progress in reductive made (L.C.-e.c.) has been slow because of problems associated with dissolved oxygen, metal impurities, and the lack of reliable electrodes. Recent technological advances in detector design and the availability of more suitable electrode materials has generated a renewed interest in this technique.

This paper describes the application of reductive made (L.C.-e.c.) using glassy carbon and amalgamated gold electrodes to quantify explosive compounds in military explosives and smokeless propellants, and the development of a highly sensitive and selective method for the detection of nitroglycerin, 2,4-dinitrotoluene (2,4-DNT), and diphenylamine (DPA) in gunshot residue.

Most explosives can be classified into one of several groups represented by nitro compounds, nitro acid esters, nitramines, salts of picramic, and chloric acids, azides and other miscellaneous compounds capable of producing an explosion, and mixtures of explosives from the above groups. Representatives of common commercial and military explosive compounds suitable for trace determinations using reductive made L.C.-e.c. will be discussed.

THE EVALUATION OF PETN AS A DETECTOR FOR THE HPLC ANALYSIS OF  
EXPLOSIVE RESIDUES. Richard A. Strobel, Richard E. Tontarski,  
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HPLC has been used for some time, with a variety of detectors, for the identification of explosives. Unfortunately, the detector systems used with HPLC have lacked the necessary specificity for identification purposes. The examiner must use alternate techniques to confirm his findings. In analyses involving actual cases the examiner is often confronted with chromatograms that provide him with multiple peaks having retention times close to those of known explosives. Being able to distinguish contaminants from explosives using only these retention times is inadequate for identification.

If a sensitive and specific detector system could be applied to each component as it is eluted, the need for additional confirmatory techniques could be eliminated. PETN is such a detector. The use of HPLC coupled with PETN as a detector is explored in this work, and its applicability to actual cases is evaluated.

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# Proton NMR Characterisation of Explosives

Qualitative and quantitative determination of significant by-products and trace impurities is useful as additional forensic information to differentiate between materially equal explosive compounds originating from different sources. For this purpose chromatographic techniques (especially HPLC) and nuclear magnetic resonance are used.

An example of the application of NMR-spectrometry for the rapid and simple characterisation of organic explosives is demonstrated. The detection of low levels of a specific impurity (trinitroanisole) in trinitroanisole samples allows comparative analysis and also the establishment of relevant connections. Furthermore, the NMR-analytical identification of this characteristic explosive impurity furnishes indications concerning the manufacturing process.

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# RADIOFREQUENCY RESONANCE ABSORPTION SPECTROSCOPIC (RRAS) METHODS FOR THE DETECTION AND ANALYSIS OF EXPLOSIVES

The field of radiofrequency resonance absorption spectroscopy (RRAS) comprises the techniques of nuclear magnetic resonance (NMR), nuclear quadrupole resonance (NQR), and electron magnetic resonance (EMR), all of which are useful in the detection and qualitative and quantitative analysis of explosives. Each of these techniques can be used on some but not all explosives. Hydrogen NMR signals can be obtained with good signal-to-noise ratio from all explosives except black powder. Nitrogen NMR signals are also available from many explosives. Nitrogen NMR spectra can be obtained from a few explosives. Free electron EMR responses are available from those explosives containing pyrolyzed materials. Each of these techniques will be briefly described, their expectations compared, and some results, from many years of measurement experience, displayed.

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# PROGRAM ON EXPLOSIVES VAPOR DETECTION

A survey of four areas of work relating to the enhanced detection of explosive vapors will be discussed as follows: the basic research on materials used in metallic preconcentrators, a TWT calibrator for determining instrument sensitivity, the development of a "soft" ionization source for mass spectrometer and the test results from our evaluation of a Gas Chromatograph-Thermo Electron Analyzer (GC-TEA).

\*This work was supported by the U. S. Department of Energy Contract DE-AC04-76DP00789.

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#### TEMPERATURE DEPENDENCE OF ADSORPTION EFFECTS OF EXPLOSIVE MOLECULES

Adsorption effects must be considered when selecting structural materials used for explosives vapor handling. Quartz, pyrex, teflon, stainless steel, and nickel absorb explosive vapors at room temperature. The reduction in the adsorptive capabilities of these materials at elevated temperatures is discussed. Data were obtained by passing TNT, DNT, or PETN vapor through heat cleaned tubing. A gas chromatograph equipped with an electron capture detector was used for analysis. The temperature needed to assure passage of explosive vapors range from 105°C for glass and pyrex to 170°C for nickel.

This work was supported by the U. S. Department of Energy Contract DE-AC04-76DP00789.

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#### The detection of explosives on human hair

In the absence of bulk explosives, demonstration of illegal explosives involvement relies heavily on trace explosives concentrations. In their most significant form traces will be closely associated with a suspect, for example on the hands. Such traces usually result from contact contamination, but experiments have shown that some common explosive constituents, for example ethylene glycol dinitrate (EGDN) have a sufficiently high vapor pressure to contaminate nearby objects via the vapour-phase. This opens up a wide area of study, some aspects of which are considered in a separate paper. Such contamination could be expected to occur on clothing, exposed skin and head hair, and it is the latter which will be discussed.

Preliminary studies indicated that thermal desorption (i.e. heating contaminated hair in a purge gas) gave variable recoveries of explosive vapour, and that adequate results were obtainable by solvent extraction despite some difficulties due to lipid. In subsequent experiments this recovery technique was used to study the influence of humidity, hair type and hair cleanliness on vapour uptake and desorption. The potential of hair examination was assessed by exposing a volunteer to vapours from explosives and subsequently removing small hair samples for analysis. Identifiable traces of EGDN were recoverable for several hours after exposure. Aspects of this approach which would benefit from further work are interpretation of a clean-up, a population contamination survey and an informed review of the legal implications.

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#### A small method for the recovery of volatile explosive traces.

Although trace explosive contamination can sometimes be detected with an explosive vapour detector, further positive identification is usually necessary. For reasons of practicality this will generally involve recovery of the explosive traces from the substrate, ideally without contaminating other materials which could interfere with analysis. Recovering volatile explosive such contamination can be achieved by employing high-temperature vapour-trapping, in which a purge gas desorbs vapours to a suitable trapping arrangement. In rapid recovery of several items such as clothing is required, or where the sample is large, such as a vehicle, thermal recovery using conventional laboratory apparatus is inappropriate, and to overcome this problem portable primary equipment has been devised. This consists of a heated plenum which is placed in contact with the surface, an air pump and a trap containing Tenax (TR), a polymeric adsorbent. In validation experiments this device recovered ethylene glycol dinitrate (a constituent of ammoniated gelatin) and other explosive vapours from a variety of substrates. In most instances it was much more efficient than solvent-washing, the main alternative. With a sampling rate of five minutes the limits of detection was 5 µg per square centimetre. At this level of sensitivity the forensic scientist can recover vapours resulting from vapour-phase contamination, in addition to the contamination resulting from contact or from application, and the technique has been successfully used in a number of forensic cases.

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#### THE IMPROVED DETECTION OF EXPLOSIVES BY THERMAL MASS SPECTROMETRY

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The THERM GDS Thermal Mass Spectrometer (MS/MS) coupled with an atmospheric pressure chemical ionization (APCI) source provides a non-invasive method of detecting vapors from explosives in a variety of samples. The APCI source, fitted with appropriate sampling lines, allows instantaneous detection of these vapors in the sub-ppm (parts-per-billion) concentration ranges. The tandem mass spectrometer portion of the instrument provides a highly specific analysis of the vapors which are detected. The first mass analyzer of the MS/MS system is used as a separator which separates the ions formed from the various components of the sampled air according to their molecular weight. These ions are fragmented in a collision region, and the second mass analyzer is used to determine which fragment ions are formed. The fragment ion spectrum of a compound is a "fingerprint" of

that compound which can be used for the identification of the compound from a spectral library.

Applications of the APCI/MS/MS technique to the detection of several explosives will be presented. Nitroglycerine, in the form of dynamite and double-base propellants, has been detected on aircraft, on the human body, in sealed cardboard boxes, in a matched-up cargo container and on a cleaned revolver. Involatile explosives such as TE can be detected from the vapors given off by solvents (cyclohexanone) or impurities.

Airborne sampling for relatively involatile explosives or for particles from explosives is accomplished using a hand-held probe concentrator which traps the vapors or particles on an organic coating. The sampling probe may be sealed and returned to the THERM MS/MS system for analysis.

Customized computer software allows for near "black-box" operation of the system for non-technical operators.

Descriptions of the instrument, sampling techniques, software and applications related to explosive detection will be presented.

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#### A MINI-PORTABLE GCMS FOR EXPLOSIVE DETECTION

A highly integrated design, light-weight and low power consumption gas chromatograph-mass spectrometer has been developed and successfully operated as part of the NASA Viking mission to the surface of Mars. The rigorous criteria established by NASA for the Viking Mars Lander resulted in an instrument that was physically compact, highly shock resistant and capable of remote operation through a command and control linkage with data return to earth. The design of this instrument evolved over an eight year period of extensive analytical testing and refinement of system elements to meet the specifications established for remote planetary operations.

The principal elements of the Viking GCMS are being re-packaged into a configuration suitable for terrestrial analytical applications ranging from environmental monitoring to a variety of forensic and security uses, including explosives detection. The result is a unique analytical tool that combines the power and sensitivity of a laboratory GCMS instrument in a small valve-sized, non-portable device that will allow field measurements and identification of unknown volatiles with maximum sensitivities of the order of 0.1 parts per billion (in air by volume).

The essential design and operational characteristics of the Viking GCMS system will be highlighted in connection with explosives detection applications.

(1) Bushneck, Diaz, et al, "Viking Gas Chromatograph-Mass Spectrometer", Rev. Sci. Instrum. 49(6), June 1978.

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#### TRACE VAPOR DETECTION OF HIDDEN EXPLOSIVES

Laboratory and field trials are outlined in the development of a prototype GC explosives Analyzer. Field trials involved aircraft, building and vehicle search scenarios.

The results of a study on the identification of a common volatile constituent of organonitrate explosives are also presented.

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#### SAMPLING OF EXPLOSIVES WITH MULTIPLE, PORTABLE PRECONCENTRATING CARTRIDGES

A portable personal sampler has been developed to be used in searches for explosives. A cartridge is inserted into a personal sampler which draws air through the cartridge. Explosive vapors are preferentially adsorbed in the cartridge. This preferential adsorption results in a pre-concentration of vapors. The cartridge is then removed and inserted into IonTech's Model GC-710 Explosive Detector where the cartridge filament is heated and the flushed off explosive vapors are detected with an electron capture detector. Commercial TNT, C-4, dynamite, and data sheets have been detected. Retention time and sampling time curves for each explosive show that detection is possible up to 30 minutes after sampling. The sampler is small (1.5 pounds) and relatively inexpensive. Several cartridges may be used with one sampler and several samplers may be used with one GC-710, thereby reducing the investment for search equipment.

#### REMOTE DETECTION OF EXPLOSIVES USING TRAINED CANINES

Through investigative research, Allied-General Nuclear Services, operating under contract to the U. S. Department of Energy, has developed a facility and technique for the remote detection of explosives. This work currently involves the remote searching of personnel entering sensitive areas of a facility. The work was generic in nature and the results can be applied to any situation where the carrying of explosives on personnel constitutes a threat. In that this system utilizes a remote detection concept, it does not violate individual civil liberties.

The developed system involves placing the search subject in a booth and circulating a volume of filtered and air-conditioned air across and around the subject. The booth air is also recycled to assure proper mixing and minimum dilution. The booth air is exhausted and a sample of the air stream extracted by way of an isokinetic sampler. This sample of air is investigated by a trained canine in an area divorced from the search subject. Trained canine response indicates the presence or absence of explosive odors.

While a number of explosives have been investigated using this remote detection concept, Commercial Dynamite and C-4 were used in full testing. Results showed nearly 100% detection and an error rate of less than 2%. Testing was conducted using small concealable samples of two to four ounces. Processing time during testing was twenty to thirty seconds per four-person group.

Through auxiliary tests, certain limitations of canine use in explosive detection were revealed. These involved odor concentration and discrimination problems. Methods of minimizing and overcoming these problems have been addressed and will be discussed.

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#### THE SCIENTIFIC DEVELOPMENT OF AN EFFICIENT DETECTOR DOG THROUGH OLFACTORY AND BEHAVIORAL MODIFICATION

A progressive learning sequence departing from the traditional methods of training dogs results in an olfactory sensitive dog compatible with a learned behavior to communicate recognition of a primary vapor. In essence, the developed dog with a skilled handler becomes a portable, mobile biological vapor detector.

The innovative procedure researched and practiced at Southwest Research Institute, San Antonio, Texas, emphasizes timely, positive reinforcement of desired behavior in qualified dogs. Initial methods sensitizes the dogs olfactory system to a practically pure primary odor mixed with a nitrogen gas carrier in an olfactometer. The dog then learns an associative behavior which communicates recognition or discrimination of the primary vapor (explosives, narcotics, etc.).

A scientifically developed dog, properly managed by an educated handler, can augment any law enforcement team and in a practical sense equal most known mechanical or electronic detection devices.

The discussion will feature a research study on the dog's recognition and olfactory sensitiveness to ethylene glycol distearate, an insignificant component with respect to quantity in five dynamite samples.

Some research studies sponsored by the Department of Defense, Drug Enforcement Agency, Department of the Interior, Department of Agricultural and Industrial Companies will be discussed. Moreover, a casual overview of how much further expanding the usefulness of detector dogs in service to man will be presented.

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#### INDICATOR TUBES FOR THE DETECTION OF TRINITROTOLUENE

A field detector kit for trinitrotoluene (TNT) in water has been developed at the Naval Weapons Center. In addition, a simple extraction technique has been developed which permits the use of this kit to detect TNT in soil. This kit has been developed in order to assist maritime commanders and military organizations in their pollution abatement efforts. Portable applications are also envisioned when it is necessary to determine if ordnance used to commit a crime contained TNT.

The operation of this detector kit involves passing an aqueous solution of TNT through a bisecting indicator tube. The basic oxide pretreatment section of the tube converts the TNT to its bisecting anion. The indicator section of the tube contains an alky quaternary ammonium chloride anion exchange resin which traps the colored anion, forming a stable blue length in proportion to the flow rate, volume, and concentration of the TNT solution. Indicator tubes have been developed for use in two different concentration ranges. The first tube is useful in the 0.1-10.0 ppm range while a low concentration tube is useful in the 10-200 ppb range.

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#### THE TAGGING OF EXPLOSIVES; THE NEW SWISS LAW ON EXPLOSIVES; DEVELOPMENT, ACHIEVEMENTS AND FIRST EXPERIENCES

In Switzerland the marking of explosives, safety fuses, detonating cords and fuselages is embodied in law. This act and its administrative rules are herewith presented, completed by the description of the present day situation in Switzerland, the applied investigation procedures for bombings, the efforts taken so far (details on the two systems "MICROTAGGANT" and "EXPLOTRACER") and future developments.

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#### INTERNAL STANDARD CHEMICAL LABELING OF INTACT EXPLOSIVES & THE SUBSEQUENT ON LINE THIN LAYER-FLAME IONIZATION IDENTIFICATION OF NANOGRAM QUANTITIES OF THESE STANDARDS IN SPENT EXPLOSIVE RESIDUES.

It is now possible to analyze quickly and with no laborious sample preparation; non-volatile, high molecular weight complex organic molecules at levels low enough to make chemical labeling of explosives and their subsequent detection feasible to incriminate would be users of explosives in a terrorist manner.

First of all, an explosive manufacturer could ID his own product line with a unique, isolated, non-volatile organic chemical of choice to, without doubt, identify his product.

Then when utilized for a suspicious purpose, residue samples may be taken on-site and spotted on a Thin Layer Chromatograph with a Flame Ionization Detector. This unit is a turn key automatic system complete with a preprogrammed data system set up for internal standard integrator chromatography analysis showing a CRT and hard copy "hit ratio" percentage of internal standard possibilities in the Basic language software executive.

With this technique, the low minimum detectable levels required for a meaningful analysis can be attained. These nanogram or parts per billion levels usually attainable here-to-fore only on volatile compounds that can be gas chromatographed; can now be obtained on non-volatile compounds that will not totally disappear upon explosion. They will be left afterwards to allow tracing of the explosive to the buyer to seller to manufacturer as evidence to prosecute the appropriate guilty party.

Preferential Category:  
1. Explosive Residue Analysis  
2. Explosive Analysis  
3. Remote Detection of Explosives

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#### The Analysis of Organic Components in Gunshot Residue

Procedures for the detection of organic gunshot residues on the hands and clothing of persons suspected of firing a weapon are described. The strongest evidence in this context is produced by using a scanning electron microscope (SEM) to detect metallic primer residues, which have characteristic shape and elemental composition. Unfortunately the SEM procedure is slow and thus limits the number of cases that can be examined. Also for some primer compositions the SEM results are not conclusive evidence that the residue arises from a firearm discharge.

In an attempt to find an alternative approach that could be used to rapidly screen cases before submission for SEM analysis a study has been made of methods for detecting propellant residues, eg., nitroglycerine, diphenylamine, nitrocellulose and inorganic nitrites. Capillary GC, TLC and HPLC with electrochemical detection have been used and the conclusions drawn are that nitroglycerine and diphenylamine residues can be determined at levels which may prove of value, whereas nitrocellulose and nitrites are less promising.

The initial levels of residue deposited vary considerably with the types of weapon and ammunition used. Some combinations produced as little contamination that residues could only be detected, using current methods, if the fire was sampled immediately, whereas others produced a high discharge and nitroglycerine, on hands and clothes, and diphenylamine, on clothes, could be detected up to 4 hours after firing.

Organic gunshot residue analysis has a future in forensic science but we must improve the sensitivity and selectivity of our techniques before it is routinely applicable.

As in other countries, attacks by improved explosive devices (IED) in the name of politically motivated terrorism play an important role in Iraq. The first assessment of their effects in Iraq was undertaken both in order to avoid self-deception and to provide a basis for future studies. Assessments are made to answer this question by comparative blessings but mostly the appropriate methods are not available to obtain really evident and comparable measuring results. Therefore, at Bundeskriminalamt (BKA) studies of IED effects have been carried out using statistical analysis in a scientific way. Objective measurement data on the effects of IED ...

measurement methods and apparatus for the experimental detection of the blast and fragmentation effects of TCs are described since they are the most hazardous for human beings. The problems of providing conditions for carrying out suitable studies on explosion effects by means of comparative blasting are discussed. Exemplary measurement results and their conclusions are presented.

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### THE SEVEN DIMENSIONS OF CLASSIFYING EMPLOYEE UNIONS

A film of a number of clandestine explosive devices in action, both at normal film speeds and high speed will illustrate the various phenomena found in these devices. Such observations can be useful in understanding some of the effects observed in past container debris.

A series slides taken at very high speed will illustrate the  
 various detonation of a pipe bomb with different explosive charges.

