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4th INTERNATIONAL SYMPOSIUM

ON

ANALYSIS AND DETECTION OF EXPLOSIVES

September 7-10, 1992

Jerusalem, Israel

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PROGRAM AND ABSTRACTS

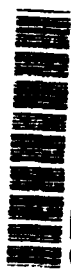
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2006

**FOURTH INTERNATIONAL SYMPOSIUM ON
ANALYSIS AND DETECTION OF EXPLOSIVES**

September 7-10, 1992

Jerusalem, Israel

Sponsored by

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Israel Ministry of Defence

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GENERAL INFORMATION

The Symposium Secretariat at Mitzpeh Rachel will be open daily from 8:30 for the period of the Symposium.

LECTURES

All lectures will be held at the Conference Hall and Lecture Room. Lectures include a 20 minute presentation followed by a 5 minute discussion.

SLIDES

All slides should be handed in to the slide operator prior to each session.

ACCOMMODATIONS

Please note that participants are requested to vacate their rooms on Thursday, September 10, by 12:00.

IDENTIFICATION BADGES

Participants are requested to wear their name tags at all times during the period of the Symposium.

TRAVEL DESK

A representative of ORTRA will be at the Symposium Secretariat to assist in reconfirming return flights or in making post-Symposium travel and site-seeing arrangements.

MEALS

Breakfast	07:00 - 09:00
Lunch	12:00 - 13:30
Dinner	19:00

SYMPOSIUM MIXER

A Symposium mixer will be held on Monday, September 7, 1992, at 20:30 in the Television Room.

SYMPOSIUM DINNER

A Closing Symposium Dinner will be held on Wednesday, September 9, 1992 at 19:30.

SCIENTIFIC PROGRAM

Tuesday, September 8 - Morning

Mitzpeh Rachel, Conference Hall

09:00 OPENING SESSION

CHAIRMAN: J. Yinon, The Weizmann Institute of Science, Rehovot, Israel

GREETINGS

09:30 K1 KEYNOTE LECTURE: The Birmingham Pub Bombings
A. Scaplehorn, Forensic Science Service, Birmingham, U.K.

10:10 COFFEE BREAK

SESSION A: ANALYSIS OF EXPLOSIVE RESIDUES

CHAIRMAN: S. Zitrin, Israel National Police, Jerusalem

10:30 A1 Antibody-based field test kits for explosives
D.D. Fetterolf, FBI Laboratory, Quantico, VA, USA

10:55 A2 A scheme for the analysis of explosives and explosive residues in Japan
T. Kishi, J. Nakamura, Y. Kumo-oka and H. Fukuda, National Institute of Police Science, Tokyo, Japan

11:20 A3 Smokeless powder characterization - an investigative tool in pipe bombings
C.L. Wallace and C.R. Midkiff, Jr., Bureau of ATF, Rockville, MD, USA

- 11:45 **A4** Applications of ion chromatography for determination and identification of chlorate, nitrite and nitrate in explosives and post explosive residues
 S. Abramovich-Bar, Y. Bamberger, M. Ravreby and S. Levy,
 National Police, Jerusalem, Israel
- 12:05 **A5** Sample preparation by supercritical fluid extraction in explosives trace analysis
 P. Kolla, BKA, Wiesbaden, Germany

12:30 LUNCH

PARALLEL SESSION PA: ANALYTICAL METHODS

CHAIRMAN: A.W. Feraday, Defence Research Agency, Fort Halstead, UK

- 10:30 **PA1** Determination of the impurity concentration profile in TNT
 W.F. Ark and T.H. Chen, U.S. Army ARDEC, Picatinny Arsenal,
 NJ, USA
- 10:55 **PA2** Rapid methods for quantitation of stabilizers and some of their reaction products in propellants
 J. Bladek, Military Technical Academy, Warsaw, Poland and M. Miszczak, Military Technical Armament Institute, Zielonka, Poland
- 11:20 **PA3** Prediction of the life-time of nylon 6/6 in contact with JA-2 propellant
 C. Campbell and T.H. Chen, U.S. Army ARDEC, Picatinny Arsenal, NJ, USA
- 11:45 **PA4** Indication of nitroesters concentration in air by gas chromatography
 J. Bil, A. Maranda and J. Nowaczewski, Technical Military Academy, Warsaw, Poland
- 12:05 **PA5** Characterization of coal-mining explosives by classical wet and instrumental analysis
 H. Kohler, Dynamit Nobel AG, Leverkusen, German
- 12:30 LUNCH

SESSION B: ANALYSIS OF SEMTEX AND OTHER EXPLOSIVES

CHAIRMAN: P. Kolla, BKA, Wiesbaden, Germany

- 14:00 **B1** Consideration of some aspects of marking plastic explosive Semtex
 P. Mostak, M. Stancl and V. Preussler, VCHZ Synthesia,
 Pardubice-Semtin, Czechoslovakia
- 14:25 **B2** The Semtex-H story
 A.W. Feraday, Defence Research Agency, Fort Halstead
 Sevenoaks, Kent, U.K.
- 14:50 **B3** Analysis of Semtex explosives
 J.R. Hobbs, U.S. Department of Transportation, Cambridge MA,
 USA
- 15:15 **B4** Slurry and emulsion explosives: new tools for terrorists, new
 challenges for detection and identification
 C.R. Midkiff, Jr. and A.N. Walters, Bureau of ATF, Rockville, MD,
 USA
- 15:40 **B5** The identification of organic peroxides
 J. Chladek, Laboratory of Federal Criminal Police, Prague,
 Czechoslovakia
- 16:05 **COFFEE**
- 17:00 **Reception by the Mayor of Jerusalem and visit of the city.**

Wednesday, September 9, 1992

SESSION C: ANALYTICAL METHODS

CHAIRMAN: C.R. Midkiff, Jr., Bureau of ATF, Rockville, MD, USA

- 8:00 **K2 KEYNOTE LECTURE: Modern methods of LC/MS: Electrospray, thermospray and particle beam**
 R.D. Voyksner, Research Triangle Institute, RTP, NC, USA
- 8:40 **C1 Detection of trace explosives evidence by ion mobility spectrometry**
 D.D. Fetterolf, FBI Laboratory, Quantico, VA, USA
- 9:05 **C2 Fluorometric determination of MMAN in water gel explosives by HPLC using pre-column derivatization with MACF**
 K. Taka-ichi, National Research Institute of Police Science, Tokyo, Japan
- 9:30 **C3 Gas chromatographic retention indices for explosives**
 J.R. Hobbs and E. Conde, U.S. Department of Transportation, Cambridge, MA, USA
- 9:55 **COFFEE BREAK**

SESSION D: ANALYTICAL METHODS

CHAIRMAN: T.H. Chen, U.S. Army ARDEC, Picatinny Arsenal, NJ, USA

- 10:15 **D1 Explosive analysis using capillary electrophoresis**
 B.R. McCord and K.A. Hargadon, FBI Laboratory, Quantico, VA, USA
- 10:40 **D2 The instrumental analysis of intact and post blast water gel and emulsion explosives**
 E. Bender, R. Strobel, and C.R. Midkiff, Jr., Bureau of ATF, Rockville, MD, USA
- 11:05 **D3 The analysis of the explosives of the paper detonator**
 T.R. Hung, C.P. Tang and K. Lin, Chung-Shan Institute of Science and Technology, Lung-Tan, Taiwan, R.O.C.

- 11:30 **D4** Analysis of nitroaromatics and other organic compounds in water samples from ammunition waste in World War II.
 K. Levsen, P. Mussmann and E. Berger-Preiss, Fraunhofer
 Institute for Toxicology and Aerosol Research, Hannover,
 Germany
- 11:55 **D5** Detection modes for the determination of explosives in water and soil
 J. Feltes, UBS, Braunschweig, Germany and J. Koll, IUP,
 Braunschweig, Germany

12:20 LUNCH

**PARALLEL SESSION PC: ANALYSIS OF EXPLOSIVES AND GUNSHOT
RESIDUES**

CHAIRMAN: **C.P. Tang**, Chung-Shan Institute of Science and Technology,
Lung-Tan, Taiwan, R.O.C.

- 8:40 **PC1** Analysis of organic gunshot residues
 C.E. Corless, Metropolitan Police Forensic Science Laboratory,
 London, U.K.
- 9:05 **PC2** The work of the explosives and gunshot residues unit of the
 forensic science service
 R. King, Forensic Science Service, Birmingham, U.K.
- 9:30 **PC3** GSR particles formed by using ammunition with mercury
 fulminate based primers
 A. Zeichner, N. Levin and M. Dvorachek, National Police,
 Jerusalem, Israel

9:55 COFFEE BREAK

PARALLEL SESSION PD: ANALYSIS AND DETECTION OF EXPLOSIVES

CHAIRMAN: **S. Kraus**, Israel National Police, Jerusalem

- 10:15 **PD1** The "Egis" system as an investigative tool
 A. Peer, National Police, Jerusalem, Israel

- 10:40 **PD2** Hidden explosive detection methods
 X.Liu, Explosive Search and Disposal Training Center, Beijing,
 China
- 11:05 **PD3** Analysis of the detonation products of insensitive high explosives
 E.Volk, Fraunhofer Institut fur Chemische Technologie (ICT),
 Pfinztal, Germany

12:20 LUNCH

SESSION E: FRAGMENTATION PROCESSES IN ENERGETIC MATERIALS

CHAIRMAN: **F. Volk**, Fraunhofer Institut fur Chemische Technologie (ICT),
Pfinztal, Germany

- 14:00 **K3** KEYNOTE LECTURE: Synthesis and explosive performance
 characteristics of polynitropolycyclic cage explosives
 A.P. Marchand, University of North Texas, Denton, TX, USA
- 14:40 **E1** Tandem mass spectrometry of two nitramines: HNIW and ADN
 R.J. Doyle, Jr., Naval Research Laboratory, Washington, DC, USA
- 15:05 **E2** Adductions in mass spectrometry of nitramine munitions
 compounds
 E.P. Burrows, U.S. Army Biomedical Research and Development
 Laboratory, Fort Detrick, Frederick, MD, USA
- 15:30 **E3** MS/MS CID fragmentation processes in nitronaphthalenes
 J. Yinon, Weizmann Institute of Science, Rehovot, Israel

15:55 COFFEE BREAK

SESSION F: MASS SPECTROMETRY

CHAIRMAN: **D.D. Fetterolf**, FBI Laboratory, Quantico, VA, USA

- 16:15 **F1** Tandem mass spectrometric identification of explosives adsorbed on
 organic substrates

A.D. Wright, K.R. Jennings and R. Peters, University of Warwick,
Coventry, U.K.

- 16:40 **F2** GC/MS analysis of PETN and NG in post-explosion residues
 T. Tamiri, S. Zitrin, S. Abramovich-Bar, Y. Bamberger, and J.
 Sterling, National Police, Jerusalem, Israel
- 17:05 **F3** Comparative study of RDX and HMX by DEPMS and TSMS
 T.H. Chen, U.S. Army ARDEC, Picatinny Arsenal, NJ, USA
- 17:30 **F4** Diagnostic scheme for polynitrocage compounds
 T.H. Chen and C. Campell, U.S. Army ARDEC, Picatinny
 Arsenal, NJ, USA

19:30 SYMPOSIUM DINNER

CHAIRMAN: **J. Almog**, Israel National Police, Jerusalem.

After-dinner speaker: **Mr. Yigal Carmon**, Advisor to the Prime Minister for
Countering Terrorism.

Thursday, September 10, 1992

SESSION GA: EXPLOSIVE DETECTION

CHAIRMAN: Y. Margalit, Israel Institute for Biological Research, Ness Ziona

8:00 **K4 KEYNOTE LECTURE: Nuclear based explosive detection techniques**
 - 1992 status
 T. Gozani, Science Application International Corporation, Santa
 Clara, CA, USA

8:40 **G1 Explosive detection by pure ^{14}N NQR**
 M.L. Buess, A.N. Garroway, J.B. Miller and Y.P. Yesinowski,
 Naval Research Laboratory, Washington, DC, USA

9:05 **G2 Explosives detection - The case for magnetic resonance**
 J.D. King and A. De Los Santos, Southwest Research Institute,
 San Antonio, TX, USA

9:30 **G3 Test protocol for surface-sampling detectors**
 P. Neudorfl and L. Elias, National Research Council, Ottawa,
 Ontario, Canada

9:55 **COFFEE BREAK**

SESSION GB: EXPLOSIVE DETECTION

CHAIRMAN: D. Fine, Thermedics, Inc., Woburn, MA, USA

10:15 **G4 Explosive and explosive taggant detection with Ionscan**
 L. Danylewych-May, Barringer Research Ltd., Rexdale, Ontario,
 Canada

10:40 **G5 Prediction of the life-time of a taggant in a composition**
 R. Reed, C. Campbell and T.H. Chen, U.S. Army ARDEC,
 Picatinny Arsenal, NJ, USA

11:05 **G6 Analysis of airflows in personnel screening portals**

- J.R. Hobbs and E.P. Conde, U.S. Department of Transportation,
Cambridge, MA, USA
- 11:30 **G7** The ATF canine explosive detection program
 R. Strobel, R. Noll, and C.R. Midkiff, Jr., Bureau of ATF,
 Rockville, MD, USA
- 11:55 **G8** Vapor preconcentration in the detection of explosives by animals in
 an automated laboratory setting.
 G.B. Biederman, University of Toronto, Toronto, Ontario, Canada
- 12:20 LUNCH

SESSION H: VAPOR GENERATORS

CHAIRMAN: **J. Hobbs**, U.S. Department of Transportation, Cambridge,
MA, USA

- 14:00 **H1** Vapor generation for use in explosive portal detection devices
 E.A. Bromberg, D. Dussault, S. MacDonald and W.A.
 Curby, Thermedics, Woburn, MA, USA
- 14:25 **H2** Design concept: Femtogram-level explosives vapor generator
 D.P. Lucero and S.K. Hendrickson, E-Cubed, Inc., Germantown,
 MD, USA
- 14:50 **H3** A model of explosive vapor concentration
 T.A. Griffy, University of Texas, Austin, TX, USA
- 15:15 **H4** Design and calibration of pulsed vapor generators for TNT, RDX and
 PETN
 J.P. Davies, EG&G Idaho, Inc., Idaho Falls, ID, USA
- 16:05 CLOSING REMARKS AND DEPARTURE

LIST OF ABSTRACTS

LIST OF AUTHORS AND PAPER NUMBERS

Abramovich-Bar, S.	A4,F2	Garroway, A.N.	G1
Azk, W.F.	PA1	Gozani, T.	K4
Bamberger, Y.	A4,F2	Griffy, T.A.	H3
Bender, E.	D2	Hargadon, K.A.	D1
Berger-Preiss, E.	D4	Hendrickson, S.K.	H2
Biederman, G.B.	G8	Hobbs, J.R.	B3,C3,G6
Bil, J.	PA4	Hung, T.R.	D3
Bladek, J.	PA2	Jennings, K.R.	F1
Bromberg, E.A.	H1	King, J.D.	G2
Buess, M.L.	G1	King, R.	PC2
Burrows, E.P.	E2	Kishi, T.	A2
Campbell, C.	PA3,F4,G5	Kohler, H.	PA5
Chen, T.H.	PA1,PA3, F3,F4,G5	Koll, J.	D5
Chladek, J.	B5	Kolla, P.	A5
Conde, E.P.	C3,G6	Kumo-oka, Y.	A2
Corless, C.E.	PC1	Levin, N.	PC3
Curby, W.A.	H1	Levsen, K.	D4
Danylewych-May, L.	G4	Levy, S.	A4
Davies, J.P.	H4	Lin, K.	D3
De Los Santos, A.	G2	Liu, X.	PD2
Doyle, Jr. R.J.	E1	Lucero, D.P.	H2
Dussault, D.	H1	MacDonald, S.	H1
Dvorachek, M.	PC3	Maranda, A.	PA4
Elias, L.	G3	Marchand, A.P.	K3
Feltes, J.	D5	McCord, B.R.	D1
Feraday, A.W.	B2	Midkiff, Jr., C.R.	A3,B4, D2,G7
Fetterolf, D.D.	A1,C1	Miller, J.B.	G1
Fukuda, H.	A2	Miszcak, M.	PA2

Mostak, P.	B1	Strobel, R.	D2,G7
Mussmann, P.	D4	Taka-ichi, K.	C2
Nakamura, J.	A2	Tamiri, T.	F2
Neudorfl, P.	G3	Tang, C.P.	D3
Noll, R.	G7	Volk, F.	PD3
Nowaczewski, J.	PA4	Voyksner, R.D.	K2
Peer, A.	PD1	Wallace, C.L.	A3
Peters, R.	F1	Walters, A.N.	B4
Preussler, V.	B1	Wright, A.D.	F1
Ravreby, M.	A4	Yesinowski, Y.P.	G1
Reed, R.	G5	Yinon, J.	E3
Scapelhorn, A.	K1	Zeichner, A.	PC3
Stancl, M.	B1	Zitrin, S.	F2
Sterling, J.	F2		

K1

THE BIRMINGHAM PUB BOMBINGS

**Dr A W Scaplehorn Forensic Science Service, Birmingham
England B5 6QQ**

On the evening of 21 November 1974 explosions occurred in two public houses in central Birmingham that resulted in the death of 21 people and the injury of a further 160. Subsequently 5 men travelling to Belfast from Birmingham were stopped and questioned at the port of Heysham and eventually subjected to handswabbing for traces of explosives using the Griess test. A number of the tests gave positive results and the five were arrested and taken to Birmingham where their number was increased to six by the addition of a friend who had been with them prior to the departure of the train.

The presentation will review the scientific work carried out at the police station and subsequently at the laboratory prior to the trial in 1975 together with that undertaken in advance of the Appeals in 1987 and 1991. This final Appeal was successful and led to the Government establishing a Royal Commission to review Criminal Procedures. The case and other similar ones where forensic science evidence had a significant impact on the trial highlight the need for adequate quality management standards for the benefits of staff and management and the crucial importance of adequate quality assurance procedures. The presentation will consider these issues and the desirability of a code of ethics for forensic science practitioners together with the practicality of the adopting worldwide agreed standards for particular forensic examinations.

A1

ANTIBODY-BASED FIELD TEST KITS FOR EXPLOSIVES

Dean D. Fetterolf, Ph.D., FBI Laboratory,
Forensic Science Research Unit, FBI Academy,
Quantico, VA, 22135, USA

The continued threat of worldwide terrorism has prompted the need for new and innovative methods of explosives detection. For the last several years we have been developing antibody-based field test kits for explosives.

An enzyme-linked immunosorbent assay (ELISA) developed for the detection of Trinitrotoluene (TNT) in munitions wastewater has been adapted to the detection of TNT residue on hands following contact. Using the procedure developed, as little as 50 pg of TNT could be detected. Accounting for sample size and dilution, the 50 pg equates to 15 ng of TNT recovered from the hands. Following contact with TNT, amounts ranging from 53 ng to more than 1500 ng were recovered from hands. The monoclonal anti-TNT antibodies showed no cross-reactivity with several other explosives or common contaminants.

Recently, latex bead agglutination technology has reduced the 3 hour laboratory assay into a 30 second prototype test kit for PETN and NG. The change in optical density as the reaction proceeds is monitored by a hand-held calculator sized spectrophotometer. Preliminary results indicate the detection of as little as 50 ng of PETN. It may be possible to use the kit for the detection of NG from gunshot residue.

These results indicate promise for the commercial development of a simple-to-use, immunoassay-based field test kit for TNT, PETN, and NG based explosives.

A2

A SCHEME FOR THE ANALYSIS OF EXPLOSIVES AND EXPLOSIVE RESIDUES IN JAPAN

T.Kishi, J.Nakamura, Y.Kumo-oka and H.Fukuda,
National Research Institute of Police Science,
Tokyo 102, Japan

During the past 3 years, the small number of bombing incident was occurred in Japan. It may be distinctive feature in Japan that improvised explosive devices are commonly used in criminal case. These improvised explosives are inorganic explosives such as home-made black powder and chlorate explosive. There are few cases of dynamite.

The trace explosive residue was extracted from post-explosion debris with water to identify the inorganic components. Water wash was analyzed by ion chromatography to determine ions. Micro X-ray powder diffractometry is also used for the analysis of water wash to identify chloride and chlorate salts. The debris was also washed with ethyl ether and analyzed by gas chromatography with electron capture detection to identify organic components such as nitroglycerin and ethyleneglycol dinitrate. These techniques allow detection at submicrogram levels.

Capillary electrophoresis is recently used as a new powerful technique for analysis of small ions such as chloride, chlorate, perchlorate, sulfate, and nitrate. These ions can be separated and determined within 5 min.. Cations such as sodium, potassium and ammonium ion also can be determined by this method. These ions are very important ions to identify Japanese improvised explosives. Therefore, capillary electrophoresis is used as a complementary technique to ion chromatography for the analysis of small inorganic ions.

A3

**SMOKELESS POWDER CHARACTERIZATION - AN
INVESTIGATIVE TOOL IN PIPE BOMBINGS**

Cynthia L. Wallace and Charles R. Midkiff, Jr.; Bureau of
Alcohol, Tobacco & Firearms, Forensic Science
Laboratory, 1401 Research Blvd., Rockville, MD, 20850,
U.S.A.

Smokeless powder is the filler in more than 50% of U.S. pipe bombings. The physical and chemical characteristics of smokeless powder may indicate manufacturer, identify the specific powder used, or link powder in a suspect's possession to that in a bomb. Since the manufacturing and blending processes have a significant impact on forensic comparisons, effects of sampling and lot-to-lot variation will be examined. Changes in manufacturing and marketing can date a smokeless powder. Case examples will illustrate the types of information which can be developed and how this information may be used to provide investigative leads or associative evidence in a pipe bombing.

A4

APPLICATIONS OF ION CHROMATOGRAPHY FOR DETERMINATION AND IDENTIFICATION OF CHLORATE, NITRITE AND NITRATE IN EXPLOSIVES AND POST EXPLOSIVE RESIDUES.

Sara Abramovich-Bar, Yair Bamberger, Mark Ravreby and Shlomo Levy. Division of Identification and Forensic Science, National Police Headquarters, Jerusalem, Israel.

Applications of ion chromatography for determination and identification of chlorate, nitrite and nitrate in various combinations and ratios in actual cases of explosives and post explosive residues were investigated. The system consisted of a Dionex Ionpac AS9 column combined with a suppressor column and a conductivity detector. Using a 25 μ l sample size, linear calibration graphs were obtained in the range of 10-100 ppm for nitrite, nitrate and chlorate. Chlorate or nitrate identification in post explosive residues based on spot tests and an infrared spectrum often do not give satisfactory results. In some cases ion chromatography can be used to replace an unsatisfactory infrared spectrum increasing the confidence in the results and increasing the detection limits. Nitrite in post explosive residues is a decomposition product and not a component of the explosive mixture per se. In general the infrared spectrum is of poor quality and the Griess test combined with ion chromatography satisfy the criteria of two independent test results. Post explosive residues containing nitrite and nitrate present special problems. Identification based on spot tests (Griess test with and without zinc reduction) and infrared spectra are frequently problematic partially due to unknown interferences in the nitrate spot test and a high ratio of nitrite to nitrate. Ion chromatography can be very useful to overcome many of these problems and give an indication as to whether the problem is due to an unknown interference or a high nitrite: nitrate ratio.

A5

SAMPLE PREPARATION BY SUPERCRITICAL FLUID EXTRACTION IN EXPLOSIVES TRACE ANALYSIS Dr. P. Kolla, BKA, Wiesbaden, Germany

Extraction with supercritical CO₂ is a versatile method for sample clean-up and trace enrichment. Instead of its novelty, there is yet a wide-spread field of application. It has been used in the analysis of plant constituents for drug analysis, in environmental food and polymer analysis as well as in the residue analysis of plant-protective agents. Supercritical fluid extraction may be superior to classical liquid extraction for speed, completeness and selectivity. In explosives trace analysis especially selectivity is a favoured feature, because of the big concentrations of interfering substances. The combination of SFE with selective analytical methods such as GC-TEA (chemoluminescence) or HPLC-PCD (photolysis-post column derivatization) is a powerful tool in explosive trace analysis in debris, swabs and dust from vacuum cleaner samples. The recovery of most of explosive constituents such as explosive oils, nitro aromatics or high explosives is more than 60 % for traces in the ppb level. Difficulties are only expected if the searched compounds stick to strongly adsorptive surfaces or if the water content of the matrix is very high. But experiences show that the recovery never drops below 30 % and analytical identification is not essentially affected.

The paper will present results and experiences with the application of supercritical fluid extraction in explosives trace analysis.

PA1

**DETERMINATION OF THE IMPURITY CONCENTRATION
PROFILE IN TNT**

W. F. Ark and T. H. Chen

**U. S. Army Armament Research, Development, and
Engineering Center, Picatinny Arsenal, New Jersey
07806-5000, U. S. A.**

Recently, we have conducted a comparative study of the organic impurity concentration profile of 2, 4, 6-trinitrotoluene (2, 4, 6-TNT) samples from two sources by direct capillary gas chromatography employing a bonded silica glass column. This work was conducted to evaluate the suitability and relative merits of both specimens for use in munitions. The objective of this paper is to develop a direct, rapid gas chromatographic method for the intended investigation.

The method developed consists of essentially overloading the column with the sample solution with a relatively high concentration of 2, 4, 6-TNT which enabled direct determinations of impurities down to the level of approximately 10 ppm.

This paper will briefly describe the experimental method developed and discuss the results obtained including the use of impurity concentration profile in the identification of the origin of explosives.

PA2

RAPID METHODS FOR QUANTITATION OF STABILIZERS AND SOME OF THEIR REACTION PRODUCTS IN PROPELLANTS

Jan Bladdek, Military Technical Academy, 01-489 Warsaw, Poland

Maciej Miszczak, Military Technical Armament Institute, 05-220 Zielonka, Poland

Methods are presented for quantitation of stabilizers - diphenylamine (DPA), ethylcentralite (EC) and some of their chemical transformation products in single and double-base propellants - in order to determine their chemical stability.

A considerable number of quantitative analyses of propellants have to be carried out. Because of the high "peak capacity" of TLC methods and the fact that the propellant samples' solutions were of relatively high viscosity, it was decided to use HPTLC coupled with visualization of the chromatograms. The liquid-crystalline and densitometric methods of visualization were used. However, the liquid-crystalline method was useful for quantitative analysis of stabilizers, but was not appropriate for quantitation of many other propellants' components. The densitometric method was found to be a suitable method of quantitation for much more components in propellants than the liquid-crystalline method. Thus, the densitometric method was chosen. During quantitation of DPA, EC and some of their products in propellants, UV absorbance signals were measured at characteristic wave lengths for analysed substances, by dual-wavelength flying spot scanning densitometer. Monotonic (increasing) standard curves were obtained describing changes of values of measured signals vs. concentrations of analysed substances.

PA3

PREDICTION OF THE LIFE-TIME OF NYLON 6/6 IN CONTACT WITH JA-2 PROPELLANT

C. Campbell and T. H. Chen

U. S. Army Armament Research, Development, and Engineering Center, Picatinny Arsenal, New Jersey 07806-5000, U. S. A.

Various plastic materials are used as the integral components of diverse munitions. It is therefore important to assess the possible long-term effects of the propellant fillers on these materials. Currently, this is being done by evaluating the mechanical property changes of tensile-bar specimens following their long-term exposure to the propellant of interest at 60 °C. This long-term test is time-consuming and it could take as long as two years to complete. Further, this method does not provide the predicting capability. The aim of this paper is to develop a general methodology which could rapidly predict the life-time of the plastic materials exposed to various propellants.

We have developed a rapid procedure for predicting the life-time of Nylon 6/6 exposed to JA-2 propellant. The method is based on the correlation of the decrease in the heat of fusion of the crystalline material to the extent of its deterioration.

This paper will describe the experimental method developed and discuss the results obtained including the predicting capability, the mechanism of the deterioration of Nylon 6/6, and assessment of the applicability of the method to other plastic materials.

PA4

INDICATION OF NITROESTERS CONCENTRATION IN AIR BY USE OF GASEOUS CHROMATOGRAPHY

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In many countries such mining explosives as dynamite, which contain nitroesters like glyceryl trinitrate and glycol dinitrate as sensibilizers, are produced in great amounts. These substances are detrimental to human organism causing substantial decrease of arterial-pressure as well as strong headaches, vomiting and even loss of consciousness (faint) by some people. According to actual obligatory norms (PN-89/Z-04212/02 and PN-89/Z-04213/02) highest admissible concentration of glyceryl trinitrate is equal $0,5\text{mg/m}^3$ while for glycol dinitrate it is equal $0,3\text{mg/m}^3$.

In the paper method of air samples collecting from places of employment exposed to exceed the admissible concentration of nitroesters are described. The results of measurements of nitroesters concentration in air carried out by use of gaseous chromatograph with electron capture detector (Pye Unicam GCV type) are presented as well. The results of measurements are compared with standard reference curve obtained for specified concentrations of alcoholic solution nitroglycerin and nitroglycol. The fulfilled measurements were directed to indicate the highly toxic places of employment in the production line of dynamites as well as to specify the health impedance degree to working personnel.

In the paper the measurements of nitroesters concentrations in the atmosphere of mine, which uses dynamites for exploitation firing, are also presented.

PA5

CHARACTERISATION OF COAL-MINING EXPLOSIVES BY
CLASSICAL WET AND INSTRUMENTAL ANALYSIS

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Permitted explosives for coal-mining are mainly mixtures of two or three inorganic salts and nitrate esters e.g. glyceroltrinitrate and glycoldinitrate with minor other components. According to german laws these explosives must always be produced in the same composition within very tight limits. Full analysis of the products is essential for quality control and for fulfilling the legal requirements. At the first thought, exact quantitative analysis of the mixtures of a few inorganic salts seems to be simple, but it isn't. This contribution gives full details of analytic methods, both classical wet and instrumental.

Calculation examples and description of used apparatus will also be presented.

Having methods for full quantitative analysis of these explosives can be helpful for identification of types, origin and manufacturer of these special explosives because of their registered and therefore known composition.

B1

CONSIDERATION OF SOME ASPECTS OF MARKING PLASTIC EXPLOSIVE SEMTEX

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A method for measuring the emission rate of marking agents was developed. The vapours of marking agents evolved from a sample of marked plastic explosive are absorbed under standard conditions in a 8 % methanol-water solution. Then the solution is analyzed by HPLC using a UV detector (215 or 220 nm).

The fluctuations of emission rate of EGDN from marked plastic explosive SEMTEX 1A produced in a plant scale are low, the mean value being 2.52×10^{-5} (g/cm² x h).

The maximum emission rate when marking particles are used is reached in several days, its value being approx. 150 % of that determined for a fresh explosive. The value of the emission rate obtained in case of marking with particles is somewhat lower in comparison to free added EGDN.

The EGDN concentration dependence of the emission rate of EGDN from SEMTEX 1A is represented by a straight line in a range of 0,05 % - 0.2 %, a similar dependence was estimated at marked C-4.

The emission of EGDN vapours from C-4 is only about 50 % of emission obtained from Semtex at the same concentration level of EGDN in the explosive.

The emission rate of marking agents is apparently influenced by the composition and physical-chemical properties of plastic explosive. However, these differences in emission rate are not very important from the practical application point of view.

B2

THE SEMTEX-H STORY

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Details are given of the historical use of SEMTEX-H explosive on the UK mainland by terrorist groups, including the PIRA and various Arab factions, since first encountered in March 1972 until the current date. Comparisons are made which numerically indicate the relatively small proportion of incidents involving SEMTEX-H explosive, leading to the conclusion that the detection of the full range of explosive substances will remain necessary for the foreseeable future.

B3

ANALYSIS OF SEMTEX EXPLOSIVES

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Semtex explosives samples were analyzed for both bulk and trace components. The bulk material was extracted first with hexane which removed the orange dye ingredients leaving the explosives and binders in the solid. The solid was then extracted with acetone which removed the explosives and left the plasticizer and binders. The acetone extract and the trace vapors above Semtex explosives were analyzed by several analytical techniques including gas chromatography-mass spectrometry (GC-MS), gas chromatography with electron capture detection (GC-ECD), and gas chromatography with thermal energy analyzer detection (GC-TEA). The GC-MS headspace data identified the hydrocarbons present in the oils and binders while the GC-MS data of the acetone extract identified the major explosives components. Comparison of the GC-ECD and GC-TEA data indicated which of the electron capturing peaks in the GC-ECD headspace chromatograms were nitro-containing entities. The results of this study indicate that there are numerous impurities and contaminants in Semtex and that these should be considered in anticipated use of explosive detection sniffers.

B4

**SLURRY AND EMULSION EXPLOSIVES: NEW
TOOLS FOR TERRORISTS, NEW CHALLENGES
FOR DETECTION AND IDENTIFICATION**

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With advantages in cost, safety and ease of use, water gel/slurry and emulsion explosives are rapidly replacing traditional explosives. As a result, cap sensitive versions of each are increasingly encountered in criminal activity and a strategy for their identification and characterization is needed. A water gel/slurry explosive is essentially an aqueous solution of an inorganic oxidizer gelled with a carbonaceous gelling agent. Dispersion of fuel or additional oxidizer in the gel produces a slurry, the "water gel explosive" of commerce. Added sensitizers enhance initiation to a detonator with particular compounds protected by patent and specific to a producer. When intact material is available for examination, sensitizer and other component identification characterizes the explosive type. Emulsion explosives are more efficient than gels and differ in that, emulsifying agents suspend droplets of aqueous oxidizer solution in an oil phase. Chemical sensitizers may be used in emulsion explosives but less commonly than in slurry types. Most emulsions are sensitized by microspheres, tiny glass bubbles which both control density and provide "hot spot" initiation. Physical characteristics and composition of typical slurries and emulsions, combined with a systematic analytical approach, are used for the characterization and discrimination of intact gel/slurry and emulsion explosives.

B5

THE IDENTIFICATION OF ORGANIC PEROXIDES

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There are two significant explosives from the group, of organic peroxides, especially triacetone triperoxide (TATP) and hexamethylenetriperoxide-diamine (HMTD). Historic studies showed that TATP and HMTD are unstable for any commercial or military application.

Organic peroxides are interesting for forensic chemists, because of the obvious utility of TATP or HMTD by terrorists.

These peroxides contain neither metallic elements, nor nitro group. Therefore their presence cannot be detected by standard security procedures (transmission RTG, vapour detector etc.).

Preliminary examination is possible by odour, spot tests etc. Identification of these explosives is realized by thin layer chromatography (TLC).

K2

MODERN METHODS OF LC/MS: ELECTROSPRAY, THERMOSPRAY AND PARTICLE BEAM

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Monitoring of many lower molecular weight non-volatile or thermally unstable polar organics (e.g. explosives) relies on the development of sensitive, specific and cost effective LC/MS techniques. Often compromises exist when using soft or hard ionization techniques that limit sensitivity or specificity. The lack of structural information observed when using soft ionization techniques (e.g., electrospray or thermospray) limit those techniques' capabilities for confirmation without more expensive MS/MS resources. On the other hand, hard ionization techniques, such as electron ionization (EI), require sample volatility and may prove ineffective in generating a molecular ion for thermally unstable organics. Perhaps the LC/MS techniques that best juggle the sensitivity/specificity issues have been electrospray, thermospray and particle beam. While these soft and hard ionization LC/MS techniques can be complementary, they are also strongly contrasted. Electrospray has the capability of generating ions from very low quantities of material (pg levels) and can provide structural information by collisional activation decomposition (CAD) in the ion transport region. The CAD spectra resemble MS/MS spectra and can be valuable for confirmation or structural elucidation. The weakness of electrospray lies with its current inability to directly handle high flow rate separations. Thermospray can handle higher flow rate separations but the technique produces spectra that often lack fragmentation for structural elucidation and is less sensitive relative to electrospray. Particle beam provides structurally relevant EI spectra for compounds that can be brought into the gas phase. This rugged technique often lacks sensitivity, permitting only 5-50 ng detection limits.

This presentation will compare and contrast electrospray, thermospray and particle beam LC/MS performance on a variety of compounds. Spectra, detection limits and factors that influence both the appearance of the spectra and sensitivity will be highlighted for each interface for the analysis of explosives and other thermally unstable or nonvolatile compounds.

This work was supported in part by FDA Cooperative Agreement No. FD-U-000589 and EPA Contract No. 68-02-4544.

C1

**DETECTION OF TRACE EXPLOSIVES EVIDENCE BY
ION MOBILITY SPECTROMETRY**

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The continued threat of worldwide terrorism in recent years has prompted the need for new and innovative methods of explosives detection. We have been investigating the application of an ion mobility spectrometer (IMS) known as IONSCAN (Barringer Instruments, Inc). The IONSCAN collects particulate explosives residue on a membrane filter. Sample collection, thermal desorption, and analysis requires less than 1 minute.

Laboratory evaluation has determined the limits of detection for most explosives to be on the order of 50-300 pg. Sampling procedures for examining suspected items or people for hidden explosives have been developed. For example, after touching C-4 ones' hands must be washed numerous times before detectable residue is removed. Explosives transferred from hands to other surfaces can easily be detected. For example, at Superbowl XXV in Tampa in January 1991, RDX residue was detected on the trunk of a car which had C-4 hidden in it for canine training.

Post blast residue can also be detected. Fragments of clothing recovered from a suitcase following an explosion showed detectable RDX residue. Post blast PETN and RDX residue from SEMTEX was detected on a portable radio components. Pipe bomb fragments showed traces of NG from double based smokeless powder. Ammonium nitrate based explosive residue has also been detected following an explosion.

C2

FLUORIMETRIC DETERMINATION OF MONOMETHYLAMINE NITRATE(MMAN) IN WATER GEL EXPLOSIVES(WGE) BY HPLC USING PRE-COLUMN DERIVATIZATION WITH 7- DIMETHYLAMINOCOUMARIN-3-CARBONYL FLUORIDE(MACF)

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A new method for the determination of **MMAN** and its related aliphatic amine nitrates as sensitizing agent in **WGE** is presented.

The reaction of **MACF** with primary or secondary amines proceeds under mild conditions and is complete in 30 seconds even in the presence of water.

The derivatives were highly fluorescent compounds. For aliphatic amine derivatives were separated in 16 minutes.

A linearity range of 20-80 nM/mL was obtained for calibration graph of the **MMAN**.

The detection limit was under 1 picomole range for an injection volume of 10 uL.

C3

GAS CHROMATOGRAPHIC RETENTION INDICES FOR EXPLOSIVES

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Both isothermal (Kovats) and linear temperature programmed retention indices were determined for nitro-alkanes, nitro-aromatics, and explosives on a J&W 0.2 mm diameter, 30 meter DB-5 column with a 0.25 micron film thickness. A Hewlett Packard Model 5890 gas chromatograph equipped with a split-splitless injector at 145 ° C and a Model 5970 mass spectrometer or a flame ionization detector was used to obtain the data. The gas chromatograph was operated isothermal at 140 ° C or linear temperature programmed from 40 ° C to 300 ° C at a rate of 5 ° C per minute. Within a homologous series, the linear temperature programmed retention indices are a linear function of the boiling point. In general the linear temperature programmed retention indices are a linear function of the retention time. These data are being used to form a retention index data base for the identification of explosives compounds by gas chromatography.

D1

EXPLOSIVE ANALYSIS USING CAPILLARY ELECTROPHORESIS

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Capillary electrophoresis is investigated for application as a complementary technique to ion chromatography in the analysis of low explosive residues. Detection limits, interference problems, and matrix effects are examined by comparing the use of ion chromatography and capillary electrophoresis in parallel analyses.

The residue from several different types of explosive devices are examined. The results show capillary electrophoresis to be a useful technique in explosive analysis yielding good sensitivity, high resolution and short analysis times.

D2

**THE INSTRUMENTAL ANALYSIS OF INTACT AND
POST BLAST WATER GEL AND EMULSION
EXPLOSIVES**

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The production of water gel and emulsion explosives is gradually replacing nitroglycerin dynamites. It is therefore important that the forensic explosive examiner have a scheme for the analysis of these materials because of their increased use in criminal activity. Presented here is a procedure for the instrumental analysis of emulsions and water gels both before detonation and post blast.

A pre-blast approach to break these explosives into their basic components for analysis is discussed. Ingredients analyzed include oxidizers, alkyl amine nitrates, oils/waxes, gelling agents, and others.

A scheme for post blast recovery, purification and identification of water gel and emulsion components is described.

D3

The Analysis of the Explosives of the Paper Detonator

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Lung-Tan, Taiwan 32526, Rep. of China

A number of paper made detonators were found in a smuggling case. The explosives were coated on the brown paper and wrapped as cylinder tubes. It can easily pass the X-ray examination during the security check. Two explosive ingredients with different colors were separated for chemical analysis. A locally developed explosive test kit based on spot-test theory as well as IR and GC/MS spectrometric methods were used to identify the explosive compounds.

The orange colored ingredient were determined to be RDX with small amount of wax and orange color pigments. The brown colored sample was identified to be diazodinitrophenol (DDNP) primer. The paper was coated with wax in order to prevent moisture penetration. It has the capability to induce the explosion of TNT as connecting with detonating cord.

D4

ANALYSIS OF NITROAROMATICS AND OTHER ORGANIC COMPOUNDS IN WATER SAMPLES FROM AMMUNITION WASTE FROM WORLD WAR II

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The production of large amounts of ammunition (mainly trinitrotoluene, hexogen and tertryl) during world war II at various sites in Germany was accompanied by the release of waste, which was not properly disposed and is threatening the water supply in many areas in Germany. Analytical methods have been developed for the analysis of the original nitrocompounds (in particular nitrotoluenes, but also hexogen and tertryl) in water samples near these wastes, using liquid/liquid and solid phase extraction and determination by GC (ECD), GC/MS, HPLC (UV) and automated thinlayer chromatography.

In addition, methods have been developed for the analysis of compounds formed by biodegradation of the original nitroaromatics. These are mixed nitroaminoaromatics, aromatic amines and nitrophenols. Quantitative extraction of these compounds from aqueous samples is difficult, but was achieved using either continuous liquid/liquid extraction with a perforator or by solid phase extraction in the presence of 35 % NaCl.

D5

DETECTION MODES FOR THE DETERMINATION OF EXPLOSIVES IN WATER AND SOIL

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Braunschweig, FRG; ²Engineering Bureau IUP, 3300 Braunschweig,
FRG.

Explosives, their degradation- and by- products were determined in surface waters, and soil by GC, GC/MS and HPLC using specific detection modes like a chemiluminescence detector (Thermal Energy Analyzer, TEA) and a mass spectrometer in the negative ion chemical ionization (NICI) and in the selected ion monitoring (SIM) modes in GC, and a photodiode-array detector (PAD) as well as an UV detector in HPLC. The water sample clean-up and enrichment was performed by liquid/solid partitioning using XAD 2/4/8 resins and liquid/liquid partitioning with toluene. Soil samples were extracted with methanol using an ultrasonic bath. In the samples analyzed a wide variety of nitro compounds have been determined like mono-, di- and trinitrotoluenes, nitrotoluidine isomers, dinitroaminotoluenes and hexogen (RDX) in concentrations ranging from 0.03 µg/l to 20 µg/l in water samples and 0.2 mg/kg to 1600 mg/kg in soil samples.

PC1

ANALYSIS OF ORGANIC GUNSHOT RESIDUES.

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Metropolitan Police Forensic Science Laboratory.

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A comparison of the chromatographic methods available for the analysis of organic gunshot residues will be presented.

These were silica capillary gas chromatography with both electron capture and thermal energy analysis detection, and high performance liquid chromatography with dropping mercury electrode detection. The methods were applied to the analysis of nitroglycerine deposited on gun barrels and clothing by various weapons and different types of ammunition.

PC2

THE WORK OF THE EXPLOSIVES AND GUNSHOT RESIDUES UNIT OF THE FORENSIC SCIENCE SERVICE

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ABSTRACT

Explosives and gunshot residues trace analysis has been centralised in a purpose-built facility at the Birmingham Forensic Science Laboratory. A combined swabbing kit has been designed to recover explosives and gunshot residues from skin surfaces and hair. Clothing is examined by vacuum filtration onto membrane filters. Samples are cleaned-up in a micro-column procedure. Most of the major components of explosives and ammunition propellants may be detected sensitively and with substantial specificity by high-performance liquid chromatography with amperometric detection at a mercury drop electrode. For confirmation of their identities the HPLC peaks are trapped out and examined by gas chromatography with a Thermal Energy Analyser. Ammunition primer particles are detected by scanning electron microscopy with X-ray analysis. Examples of the application of the techniques in casework are presented.

PC3

GSR PARTICLES FORMED BY USING AMMUNITION WHICH HAVE MERCURY FULMINATE BASED PRIMERS

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Ammunition having mercury fulminate based primers are commonly manufactured by Eastern Block countries and extensively used in the Middle East. GSR particles formed by firing these types of ammunition were examined. It was observed that much lower percentage of mercury-containing GSR particles were found in samples taken from a shooter as compared to the percentage of such particles in samples from cartridge cases. This fact must therefore be taken into account when interpreting case results. A plausible explanation for the above results is proposed.

PD1

THE "EGIS" SYSTEM AS AN INVESTIGATIVE TOOL

AVI PEER- COMMANDER, ISRAEL POLICE, HEAD OF R & D

The needs of the crime scene investigator to identify/detect the type of explosive right at the scene, has been paramount for many years.

The introduction of the "EGIS" system into the I.N.P. opened a new era in the investigation of explosive incidents.

Today, not only can we identify the type of explosive in a matter of seconds, but we can also conduct additional checks which tie further evidence to the case (such as: safe house used by the perpetrator, escaping car, money handled in explosive transaction, etc).

The lecture will review the capabilities of the "EGIS" system as an investigative tool through the various bomb incidents investigated witht the system.

PD2

HIDDEN EXPLOSIVE DETECTION METHODS

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In recent years, bomb threats have become a big problem. Terrorists have become very sophisticated in their concealment methods, using new explosive materials, which are harder to detect.

This article reviews some explosive detection methods, including very high energy X-ray technology, explosive vapour detection, additional material and detection technology and thermal neutron analysis. The merits and demerits of the various methods are evaluated and the tendency of future developments is discussed.

PD3

**Analysis of the Detonation Products
of Insensitive High Explosives**

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7507 Pfinztal, D**

Abstract

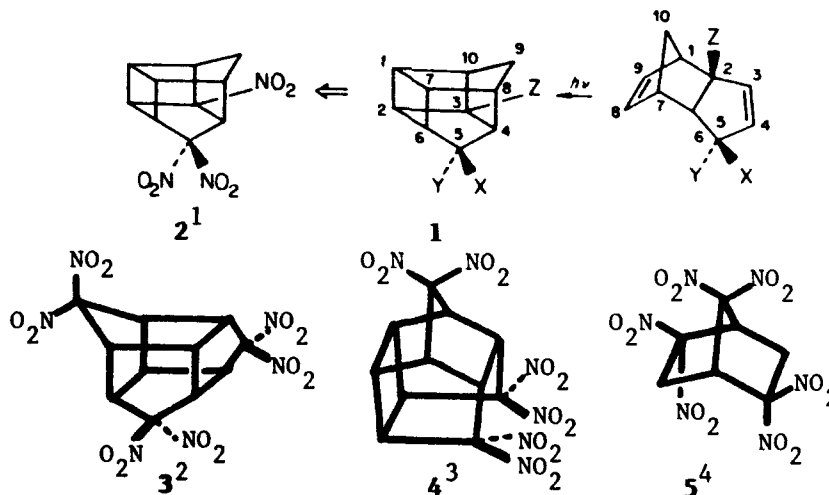
The detonation products of high explosives are dependent on pressure and also on the confinement under which the detonation reaction proceeds. To determine the detonation products of less sensitive high explosives such as TNT/nitroguanidine (NQ) and PBX charges with polybutadiene (PB) binder containing RDX together with or without aluminium (Al) experiments have been performed in a stainless steel chamber of a volume of 1.5 m³. These experiments were done under different ambient argon pressures up to 0.3 MPa. Gaseous reaction products were analysed by mass spectrometry and chemiluminescence analysis. Solid reaction products were analysed for measuring the carbon residue or unreacted Al. It was found that the detonation products were highly dependent on the ambient pressure of argon. The most important changes of the reaction products and therefore also of the energy output were found between vacuum and atmospheric pressure of argon. With increasing pressure, H₂ and CO decrease and CO₂, H₂, C_s, NH₃, HCN and CH₄ increase together with the reaction enthalpy. By analysing the physical structure of the carbon residue, diamonds have been observed between 4 and 7 nm in diameter.

K3

SYNTHESIS AND EXPLOSIVE PERFORMANCE CHARACTERISTICS OF POLY-NITROPOLYCYCLIC CAGE EXPLOSIVES.

Alan P. Marchand, Pendri Annapurna, B. E. Arney, Jr., P. R. Dave, V. R. Gadgil, D. Rajapaksa, G. V. M. Sharma, R. Sharma, and U. R. Zope, Department of Chemistry, University of North Texas, NT Station, Box 5068, Denton, Texas 76203-0068 USA.

Polynitropolycyclic "cage" compounds, by virtue of their highly compact structures, high densities, and relatively high strain energy contents, constitute a promising new class of energetic materials. Preliminary evaluation of their explosive performance characteristics indicates that compounds of this type are relatively powerful, shock-insensitive explosives. Our approach to their synthesis is summarized below. Here, appropriately substituted cage molecules (e.g., **1**, where X,Y = carbonyl oxygen and Z = CO₂H) are constructed, and the substituents (X, Y, & Z) are converted subsequently into C-NO₂ groups. This approach has been used successfully to prepare several polynitropolycyclic cage explosives. Some representative examples in this regard are shown below (compounds **2-5**).



References

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4. Synthesis in progress.

E1

TANDEM MASS SPECTROMETRY OF TWO NEW NITRAMINES: HEXANITROHEXAAZAISOWURTZITANE (HNIW) AND AMMONIUM DINITRAMIDE (ADN)

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Two very different new nitramines have been synthesized recently. The first, hexanitrohexaazaisowurtzitane (HNIW), is a $C_6H_6N_6$ polyazapolycyclic ring system with each of six nitro groups bound to a ring nitrogen. The gas-phase ion chemistry of HNIW has been investigated using high-resolution mass spectrometry and multi-stage mass spectrometry. HNIW demonstrates a much more facile loss of nitro groups than RDX and HMX. Unlike the monocyclic nitramines, HNIW dissociates to yield an unusual number of abundant $C_4H_6N_c^+$ ions that are derived from the $C_6H_6N_6$ ring system and must be formed by the loss of all six nitro groups. It is demonstrated with the use of hybrid MS-MS techniques that $C_4H_6N_c^+$ ions may be formed directly from gas-phase dissociation processes. The aim of this work was (1) to identify unambiguously the most important ions in the HNIW mass spectrum by using high-resolution techniques, (2) to determine the mechanisms by which nitro groups are stripped from HNIW ring system, and (3) to identify dissociation pathways leading from the HNIW molecule to $C_4H_6N_c^+$ ions.

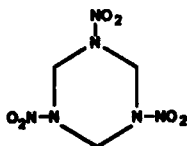
The second new nitramine, ammonium dinitramide (ADN), is an ionic compound containing the ammonium cation (NH_4^+) and the dinitramide anion ($O_2N-N-NO_2^-$). ADN has been investigated using kiloelectronvolt xenon atoms to sputter ionic fragments from the surface of the crystalline solid. Extensive distributions of sputtered ionic aggregates corresponding to the general formulae $([NH_4N(NO_2)_2]_nNH_4)^+$ and $([NH_4N(NO_2)_2]_n[N(NO_2)_2])^-$ were observed with several species exhibiting enhanced abundances. Several ADN aggregates dissociate to yield dinitraminic acid, $HN(NO_2)_2$, the conjugate acid of the dinitramide anion. The objectives of ADN work in progress are to characterize the gas-phase chemistry of ammonium dinitramide, to describe the structures of ADN aggregates, and to determine how ammonium dinitramide ionic aggregates decompose to form dinitraminic acid.

E2

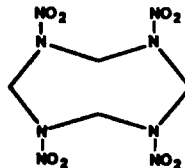
ADDUCT IONS IN MASS SPECTROMETRY OF NITRAMINE MUNITIONS COMPOUNDS. Elizabeth P. Burrows, U.S. Army Biomedical Research and Development Laboratory, Fort Detrick, Frederick, MD 21702-5010, U.S.A.

Fragment-molecule adduct ion formation, first observed for RDX (1) and HMX (2) by Yinon a decade ago, was significant in the EI as well as in the CI (methane) mass spectra of a number of cyclic nitramines and related compounds introduced by direct exposure probe. A recently synthesized polycyclic strained-cage nitramine now under evaluation as a new solid energetic oxidizer also gave rise to fragment-molecule adducts in PCI and NCI modes. In PCI mode, for all cases except 1 and 2, the proton adduct was substantially more abundant than the fragment-molecule adducts; for 1 and 2 one of the latter adducts was at least as abundant as the former. In all cases the reagent gas adducts $[M+C_2H_5]^+$ and $[M+C_3H_5]^+$ were much less abundant or were not observed.

In contrast, an investigation of the ion-molecule reactions of a polar PCI reagent gas, dimethyl ether, with nitramines and related compounds showed that reagent gas adduct ions were by far the predominant species. For 1 and 2 the most abundant ions were $[M+91]^+$ and $[M+61]^+$; in the other cases $[M+45]^+$ was most abundant and usually $[M+47]^+$ was second in abundance. The m/z values of these adducts are uniquely characteristic for a particular nitramine and may be of value, either alone or in addition to data obtained from methane PCI spectra, in the identification of individual compounds in complex mixtures such as post-explosion residues. Collision-induced dissociation of the adducts gives fragments which again are characteristic of the individual nitramine.



1



2

E3

MS/MS CID FRAGMENTATION PROCESSES IN NITRONAPHTHALENES

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Although nitronaphthalenes were widely used in explosive mixtures during World War I, today they are not used in the manufacture of explosives. The main reason is the shortage of naphthalene which is used in the manufacture of phthalic acid and dye intermediates [1].

Some of the nitro derivatives of naphthalene are powerful explosives, some are as powerful as TNT. The advantage of nitronaphthalenes is that they are only slightly toxic, as opposed to the high toxicity of TNT [2]. Because of the environmental toxic effects of TNT, a renewed interest in the nitronaphthalenes has developed.

In order to contribute to the understanding of these compounds we have studied the mass spectral fragmentation processes and pathways of a series of nitro, dinitro and trinitro derivatives of naphthalene. Collision induced dissociation mass spectra were obtained using a tandem BB mass spectrometer.

Major fragmentation pathways included loss of NO_2 from the molecular ion, followed by loss of NO and/or CO . Additional fragmentations included loss of OH from the molecular ion in some of the investigated compounds.

Fragmentation pathways in nitronaphthalenes have been suggested.

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F1

TANDEM MASS SPECTROMETRIC IDENTIFICATION OF EXPLOSIVES ADSORBED ON ORGANIC SUBSTRATES.

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The persistence of explosives on organic materials is of great interest to the forensic scientist. It is well known that the volatile components of propellants and explosives are remain detectable on skin for several days after initial exposure. Mass spectrometry is generally used for the detection of such residues in association with some form of chromatographic separation, i.e. LC/MS. Tandem mass spectrometry has mainly been limited to studies of pure explosives in which the product ion spectra have been recorded of several different explosives.

We present the results of a study of the product ion mass spectra of protonated molecule ions generated by isobutane chemical ionisation of cyclomethylene trinitramine (RDX) and pentaerythrityl tetranitrate (PETN) and product ion spectra of abundant fragment ions formed by electron ionisation of 2,4,6-trinitrotoluene (TNT). Spectra were obtained from both pure explosives and explosives adsorbed on organic substrates such as human hair and man-made fibres.

Experiments were performed using a Kratos Analytical Concept II HH four-sector mass spectrometer. The first mass analyser is used to focus the required precursor ions into an electrically floated collision cell which contains a low pressure of helium. The resulting product ion spectra are recorded by means of a linked scan of the final electrostatic and magnetic fields. The resolution required for the primary separation is generally in excess of 3000.

In order to ascertain whether a particular explosive is present in a sample, the CID spectrum of the sample is recorded. The presence or absence of the peaks characteristic of the explosive is used to indicate the presence or absence of the explosive in question.

The results which we present readily show that tandem mass spectrometry can be applied to this type of problem although the detection limits of these experiments are not known.

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F2

GC/MS ANALYSIS OF PETN AND NG IN POST-EXPLOSION RESIDUES

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GC/MS is used in our laboratory to confirm the identity of explosives in post-explosion debris.

The GC/MS analysis of PETN has often been unsuccessful, mainly due to its thermal decomposition under GC conditions. Changing experimental conditions, using a very short (1.5 m) capillary column, solved the problem.

PETN in post-explosion samples was reported to be occasionally accompanied by its degradation products. We have found a similar behaviour of NG in actual post-explosion samples. The TLC plates of these samples contained several Griess - positive spots which corresponded to mono- and dinitrate esters of glycerine. These esters were independently synthesized and characterized. Their analysis was carried out using silylation, GC/EIMS and GC/CIMS.

F3**COMPARATIVE STUDY OF RDX AND HMX BY DEPMS AND TSMS**

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In the identification of unknown compounds, the molecular weight and the structural information are crucial. In the last several years, we have been developing DEPMS procedures to obtain such information for rapid identification of unknown explosives and propellants, especially RDX and HMX. The latter do not form abundant pseudomolecular ions in PICIMS. The objective of this paper is to compare the DEPMS and TSMS in providing the crucial information needed in the identification of trace quantities of RDX and HMX.

DEPMS was conducted in PICI mode using methane as the reagent gas, while TSMS was performed in EI, PICI, and NICI modes.

This paper will briefly describe the DEPMS procedure developed and compare the capabilities of DEPMS and TSMS in providing the molecular weight and the structural information for RDX and HMX.

F4

DIAGNOSTIC SCHEME FOR POLYNITROCAGE COMPOUNDS

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In the last several years, we have been developing a diagnostic scheme for the identification of non-volatile labile unknowns, in particular, the energetic materials using the DEPMS. This was necessitated by the large numbers of unknowns which needed to be identified. The specimens included the foreign munitions of diverse origins, the environmental samples, the unknown wastes involved in the hazardous waste management programs, and the unknown synthetic products including in many cases, the intermediates.

The unknown identification requires two vital informations regarding the specimen, i. e., the molecular weight and the structural features. The objective of this paper is to describe the diagnostic scheme we have developed recently for the determination of the former for both the strained and the non-strained polynitrocage compounds.

This paper will briefly describe the experimental procedures used and discuss in detail the general diagnostic scheme for molecular weight determination of polynitrocage compounds including the ability of the method in obtaining the structural information in the same experiment.

K4

**NUCLEAR BASED EXPLOSIVE DETECTION TECHNIQUES -
1992 STATUS**

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Over the past several years the attitude towards nuclear based explosive detection systems (EDS) has fluctuated from euphoric to disappointing, and in between. This had to do less with technical merits and more with a host of other issues ranging from changing perceptions of threats and resulting systems' requirements, to the effect of security regulations on the health of commercial airline industry. Despite these tribulations, progress continues. Six thermal neutron analysis (TNA) EDS's have been deployed in several airports in the U.S.A. and abroad. While providing the users with valuable security services, a large amount of data has been collected, allowing further improvements. Indeed the currently deployed TNA systems are an improved version of the first one installed in 1989 at the JFK International Airport's TWA terminal. Significant additional enhancements in performance, which resulted from recent laboratory studies are awaiting decision on implementation in the existing systems.

In the last few years the desire to detect ever smaller amounts of explosives led to the exploitation of fast neutron reactions. Progress in the development of the pulsed fast neutron analysis (PFNA) technique, as the ultimate EDS in terms of sensitivity, low false alarm and high throughput, will be reported. Other nuclear based techniques such as the gamma and neutron resonance attenuation (GRA and NRA, respectively) will be discussed and compared on its own and as part of a total security system. The latter may incorporate several sensors and other attributes of security to provide the most effective system.

G1

EXPLOSIVES DETECTION BY PURE ^{14}N NQR

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Pure ^{14}N nuclear quadrupolar resonance (NQR) is quite promising as a method for detecting explosives such as RDX in quantities which are a threat to aircraft security. We have constructed a proof-of-concept NQR explosives detector which interrogates an active volume of 300 liters in 6 seconds. We first outline the basics of the NQR approach, highlighting strengths and weaknesses, and then present representative results from our RDX detection system.

G2

EXPLOSIVES DETECTION --
THE CASE FOR MAGNETIC RESONANCE

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Distinctive properties of the hydrogen transient magnetic resonance (HTNMR) response from explosives provide a basis for rapid, selective and sensitive detection of these materials concealed in parcels, letters and airline baggage. Most common high energy and commercial nitrogenous explosives as well as many which do not contain nitrogen, are detectable. Full scale inspection systems based on this technology have demonstrated performance comparable to the best reported for any other method.

The theory of HTNMR and related radio frequency resonance methods are briefly reviewed and the development of full scale inspection systems is discussed. MR data on many explosives are presented and compared to common materials found in baggage. The potential to further increase the useful sensitivity and the inspection rate are analyzed as is the possibility for substantially reducing present limitations.

G3

TEST PROTOCOL FOR SURFACE-SAMPLING DETECTORS

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The use of surface sampling and on-site analysis is a recent innovation in "sniffer" technology designed to counter the bomb threat in civil aviation. Augmenting the previous vapour sampling approach, new detector technology is emerging which allows the rapid detection of sub-nanogram quantities of plastic explosive residue which may be present on the exterior of passenger luggage or air cargo as a result of secreted explosives. In assessing the efficacy of a new detector system, the problem arises of providing a realistic and controllable source of plastic explosive residue with which to challenge the system. This paper describes several laboratory and field-simulation techniques which have been researched in our Laboratory, and presents the results of evaluations obtained with the most reliable and useful of these techniques, a "calibrated" thumbprint source.

G4

**EXPLOSIVE AND EXPLOSIVE TAGGANT
DETECTION WITH IONSCAN™**

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A study of a number of commercial explosives and proposed taggants was performed with the commercially available Ion Mobility Spectrometer (IMS), IONSCAN™, manufactured by Barringer for detection of explosives and drugs.

Results on false alarm rate from laboratory and field tests are reported for both discrete and continuous modes of operation of IONSCAN. Different sampling techniques for volatiles (i.e. taggants) and non-volatile explosives or explosives-related substances are discussed. Advantages and/or disadvantages are evaluated for each type of sampling technique.

G5

PREDICTION OF THE LIFE-TIME OF A TAGGANT IN A
COMPOSITION

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As part of our comprehensive tagging program, the life-time of a vapor taggant in a composition is being studied. Life-time is a critical parameter which ranks in importance only next to detectability. The objective of this paper is to apply the semi-finite linear diffusion model to predict the life-time of a vapor taggant in a composition.

A long-term experiment is being conducted to study the loss of the vapor taggant as a function of sample thickness, temperature, and time.

This paper will describe the theory and the experimental design employed and discuss the applicability of the theoretical model to the experimental data obtained to date.

G6

ANALYSIS OF AIRFLOWS IN PERSONNEL SCREENING PORTALS

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A finite element fluid dynamics software model, FIDAP 6.02, was used to model the flow in various designs of personnel screening portals. Two different portal configurations, a walk-in/walk-out portal, such as used in the Thermedics prototype, and a walk-through portal were compared. Laminar and turbulent flow were modeled showing the effects of increasing turbulence on sampling. Parameters measured were streamline plots, pressure plots, speed plots, temperature plots, and mass transfer plots. The effects of heating the subject and blowing high speed jets on the clothing were also included. A parametric analysis was performed for certain scenarios so that various effects could be optimized to provide maximum sample transfer to the detection modules. The results of this study indicate that certain sampling parameters and methods are preferred over others.

G7

THE A.T.F. CANINE EXPLOSIVE DETECTION PROGRAM

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Explosives detection technology has advanced significantly in the past few years. Even with those advances many of the instrumental techniques still are unable to meet current needs. Additionally, evaluation of several canine explosives detection programs found them to be deficient on an operational basis. In response, ATF developed a pilot training program for explosives detection canines in 1991. The training concepts used included: 1. food reward conditioning; 2. scientific oversight of proficiency testing; 3. use of small amounts of a wide range of explosive compounds representative of those in use today. Advantages of the training program included dogs capable of working with multiple handlers and sensitive to a wide range of concealed explosive products at quantities as low as 15 grams. Additionally the training program could be completed in six weeks and at minimal cost.

G8

VAPOR PRECONCENTRATION IN
THE DETECTION OF EXPLOSIVES BY ANIMALS
IN AN AUTOMATED LABORATORY SETTING.

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Gerbils were trained to detect explosives vapor in an automated laboratory setting in order to confirm previous work indicating that detection of explosive composition C4 had taken place. The possibility of contamination with EGDN in the earlier study prompted this reexamination. Samples of C4 uncontaminated with EGDN and separate samples tagged with EGDN were used. In addition, two other plastic explosives: Semtex--currently suspected in recent terrorist incidents, and denta sheet (also known as sheet PETN) were also used in detection training. Each experimental animal was able to detect each of the four explosives in a short-duration training experiment. Control animals failed to learn cues from the vapor presentation system per se, confirming the validity of the present vapor delivery system. Animals were able to detect C4 in its pure and EGDN-tagged forms and were readily able to detect SEMTEX. In a second phase, evidence from an experiment using vapour preconcentration is presented, and the performance of animals is compared using preconcentration and the more usual vapour presentation methods. Improved detection capability could have important implications for anti-terrorist explosives detection.

H1

VAPOR GENERATION FOR USE IN EXPLOSIVE PORTAL
DETECTION DEVICES

by

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In order to properly evaluate portal explosive detection devices both a means of generating a known vapor sample of the different explosives, and a surrogate for bulk explosives are required. What is described is the design and evaluation of a secondary vapor generation source that has proven to be effective in calibrating both the absolute sensitivity and the spacial distribution sensitivity of a portal explosives device. In addition, a bulk explosives surrogate will be described that can safely be used to effectively evaluate a portals ability to actually find hidden explosives.

H2

DESIGN CONCEPT: FEMTOGRAM-LEVEL EXPLOSIVES VAPOR GENERATOR

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The evaluation and response calibration of an explosives vapor detection system for a variety of explosives species can be performed by a vapor generator with unique operating specifications and versatility. This design analysis of a vapor generator concept addresses the evaluation and calibration requirements of an explosives vapor detection system alarming on low explosives vapor mass input ($\sim 1 \times 10^{-15}$ to 5×10^{-15} g) for the low vapor pressure explosives.

This concept uses a special-purpose permeation device (a permeation pillow), with an explosives vapor emission rate that can be determined gravimetrically with commercial microbalances in 1 to 10 days. A special-purpose thermal chamber contains the pillow at a controlled temperature. It uses a porous wall through which carrier air flows and forms a continuously renewed air boundary layer at the porous surface, thus preventing explosives molecule surface adsorption to its walls. Downstream of the chamber and permeation pillow, a heated and temperature-controlled tubular membrane precisely extracts explosives vapor molecules from the chamber carrier air stream at a controlled low flow rate. Thereafter, the explosives vapor molecules are transported out of the generator with either a continuous air stream to simulate the explosives vapor concentration in a detection scenario or with a smaller, pulsed gas flow rate to simulate a localized injection of explosives vapor mass adjacent to the detection system. Regardless of the air flow rate, approximately 1×10^{-15} g of explosives vapor molecules are produced by the generator in a 3-s period.

H3

A MODEL OF EXPLOSIVE VAPOR CONCENTRATION II

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A theoretical model which predicts the expected concentration of explosive vapor which might be available for detection in various scenarios was reported at a previous conference (Mannheim 1989). The results of this model have now been refined and the model extended to apply to other situations. A comparison has been made of the predictions of this model with experimental results obtained in a controlled experiment in a room-sized environment. The experimental results are found to be in good agreement with the theoretical predictions.

H4

Design and Calibration of Pulsed Vapor Generators for TNT, RDX and PETN

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Computer controlled explosive vapor generators for TNT, RDX and PETN were built and calibrated to support an Independent Validation and Verification facility for Explosive Detection Systems (EDS) at the Idaho National Engineering Laboratory. The generators were constructed using pure explosive suspended on quartz beads which were then loaded into a stainless steel tube. The tube was coiled and placed into a temperature controlled chamber. A carrier gas (ultra-pure air) was passed through the coil to carry the explosive molecules. The generators are capable of delivering a pulse of varying explosive concentration through the control of coil temperature, air flow rate, and pulse width. The generators were calibrated in the picogram to nanogram range using an Ion Mobility Spectrometer.