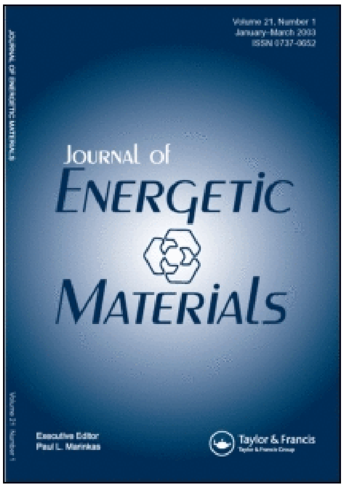

Journal of Energetic Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713770432

Explosives residue analysis in the mid-1980's-an expanding and challenging role for the forensic scientist

Alexander Beveridge[a]
[a] Royal Canadian Mounted Police Forensic Laboratory, Vancouver, B.C., Canada

To cite this Article Beveridge, Alexander(1986) 'Explosives residue analysis in the mid-1980's-an expanding and challenging role for the forensic scientist', Journal of Energetic Materials, 4: 1, 29 — 75

To link to this Article: DOI: 10.1080/07370658608011333

URL: http://dx.doi.org/10.1080/07370658608011333

EXPLOSIVES RESIDUE ANALYSIS IN THE MID-1980's - AN EXPANDING AND CHALLENGING ROLE FOR THE FORENSIC SCIENTIST

Alexander Beveridge
Royal Canadian Mounted Police
Forensic Laboratory
Vancouver, B.C. Canada

ABSTRACT

The Forensic Scientist who undertakes explosives residue analysis in the 1980's must not only be a skilled analyst, but also a skilled student, educator and consultant. The subject is multi-disciplinary, the scope is international, and the keywords are "experimentation" and "communication". This paper expands on each area, drawing illustrations from recent Canadian experience which includes visits to major Forensic Laboratories on three continents. Specific emphasis is given to reviewing current literature on explosives analysis and considering the criteria which must be met by a new method in order for the Forensic Scientist to add it to his routine scheme for systematic analysis of explosives residues.

Journal of Energetic Materials vol. 4, 29-75 (1986)

Published in 1986 by Dowden, Brodman & Devine, Inc.

Explosives residue analysis is a much more complex challenge for the Forensic Chemist in the mid-eighties than it was two decades ago. Then, literature on methods of analysis dealt with infrared spectroscopy[1], spot tests[2] and thin-layer chromatography[3] [4]. Background information on most explosives was available through texts such as Urbanski's excellent series[5]. Now, however, there are new classes of commercial explosives such as cap-sensitive water gels/ slurries, new components with names like "Metriol trinitrate"[6] and an upcoming new generation of military explosives in the form of nitrated cubanes as described by Dr. Alster[7] in the preceding paper.

As the list of new formulations grows, so too does the list of new analytical methods and instruments offering enhanced sensitivity and specificity - and cost and complexity.

As the scientist attempts to maintain industrial and scientific currency, however, the investigators relying on his expertise continue to ask the same two questions:

i) did an explosion occur?

ii) what caused the explosion?

The first question typically arises in disasters such as an aircraft crash. The second is universal. Both are always accompanied by pressure for results - for intelligence, for investigation - and increasingly are being asked in an international context as bombs cross borders.

The thesis which I am advancing in this paper is that to answer these questions most efficiently and effectively, today's forensic scientist must be not only a skilled analyst, but also a skilled educator, a practical consultant and a dedicated student. The key words, I believe, are "experimentation" and "communication".

As Commissioner Kraus so accurately stated in his opening remarks to this conference, terrorism using explosives is an international problem and any country could be next. The counter must be international cooperation. There is no place for rivalry - interlab or interscientist - nor for "tunnel vision" - concentrating on one way of doing things to the exclusion of more practical alternatives.

Canada has certainly not been immune to terrorist acts in the last few years and this has caused the R.C.M.P. Forensic Laboratories to reassess explosives casework. The first step has been to concentrate experience by undertaking such work in only two of the eight Laboratories. Thus, the Vancouver Laboratory essentially covers explosives incidents in Western Canada while the Central Forensic Laboratory in Ottawa covers incidents falling under federal jurisdiction in Central and Eastern Canada. The second step has been to review our methodology by assessing current literature methods and by visiting major forensic laboratories which specialize in explosives analysis. These visits have spanned three continents and have permitted comparison of methods in the literature to those in actual use, as well as assessment of why some methods have been adopted and others not. This will be discussed in the second half of this paper.

Firstly, however, I would like to consider the scientist's role as educator, consultant and student.

Scientist as Educator

The context is the scene of an explosion or disaster. The usefulness to an investigator of the results of explosives residue analysis using the most sophisticated instrumentation, the most experienced scientists and the most comprehensive data bases is wholly dependent on the quality and pertinence of the exhibits submitted for examination. The scientist can help the investigation greatly by prior education of the first people to arrive at a scene - most likely the police and fire departments. The purpose is to aid their recognition of physical evidence and train them how to safeguard it prior to expert-supervised collection. This involves close liaison with the "bomb squad" and lecturing to detachment policemen, scenes of crime officers, emergency response teams, dive teams and fire departments. A fire department made aware of the water-soluble nature of many residues, and the contamination complications induced by dry chemical extinguishers, can do much by judicious action at a scene, to minimize further environmental contamination. The R.C.M.P. runs courses with an extensive practical component for each of those groups.

Training interested investigators leads, of course, to two-way communication and recognition of new problems.

For example, two recent questions from a dive team course were:

i) do you want a metallic exhibit to be submitted in water from which it was recovered, in fresh water, dried, or sprayed with oil to prevent corrosion?

ii) which residues will survive submersion?

These closely related questions are not directly addressed in the literature. A paper on aircraft sabotage investigation by Higgs 'et al'[8] deals with both chemical and physical aspects of post-explosion debris including material recovered from the sea, and papers by Beveridge 'et al'[9] [10], and Hayes[11], deal in part with insoluble residues. However, while this enabled some answer to be given, it was clear that we would have little practical background if faced with a situation like the sinking of the 'Rainbow Warrior'. Hence, a test program of underwater explosions is being initiated. One immediate benefit was the discovery that virtually all fragments were recoverable in a very small area, and made excellent "damage". standards. A more detailed answer to the questions should be available by the next course!

Scientist as Consultant

It is often advantageous to have the perspective of a forensic chemist at a post-blast scene to ensure the pertinence of all material seized as exhibits for residue analysis. This normally requires the scientist to advise and answer questions about what to collect, how to package, etc. The scientist must, therefore, know what to look for in terms of likely residue-bearing evidence. This can be learned in part by reading - e.g. Bomb Data Centre, military and underground publications - and by accompanying more experienced colleagues to scenes. But the best preparation, surely, is by experimentation with a wide range of explosives in an equally wide range of environments like buildings, cars, boats and aircraft. That is, the scientist who goes to a scene as consultant via an explosives range is likely to achieve more than one who goes directly from the bench. I add that the forensic scientist at the scene is one of those in coveralls on his knees and not one of those standing around in a suit or overcoat.

The R.C.M.P. is presently formalizing formation of an Explosives Incident Response Team, to consist of an explosives disposal and bomb technology group member, a forensic chemist, and a scenes of crime specialist.

In international incidents and investigations where a device or explosives originate in one country and explode in another, the scientist has another key consultative role - to inform and advise his foreign colleagues about domestic explosives and their residues, and about special skills and experience in his country which could aid the investigation.

Closely allied to this is simply knowing whom else to call - domestically or foreign - when local resources are exhausted. This is best achieved by the personal contact which arises from attending conferences like this, from professional visits and from knowledge of the literature.

Scientist as Student

Having ensured, as educator and consultant, that optimal exhibits have been received, the scientist is faced with two tasks:

a) analysis.
b) interpretation and presentation of the significance of the results.

The scientist prepares for these tasks in the role as student, in three primary areas:

i) reading, asking, listening, visiting - e.g. by reading scientific, government and other literature, contact with explosives manufacturers, regulatory agencies and users, attending conferences, visiting other laboratories.

ii) experimenting on an explosives range - e.g. by obtaining experience and practice by blowing up devices using as comprehensive a range of explosives as possible in conjunction with the most commonly encountered substrates - e.g. soil, cars, buildings, boats, aircraft - and collecting the residues and maintaining a physical data base.

iii) experimenting in the laboratory - e.g. by applying the range of available methods - routine and novel - to live explosives, to substrates, to substrates plus live explosives, and finally to post- explosion debris. This permits assessment of methodology and raises many questions leading to practical research into new methods - scientist as researcher. The on-going process also permits maintenance of an analytical data base[12].

Dealing first with areas (ii) and (iii), the knowledge and experience gained by this type of practical training - the explosions carried out always with the 'bomb squad' - enables the scientist to function effectively at a crime scene and in the lab by knowing what to look for, when seeking evidence of an explosion or residue from an explosive. It also permits ready recognition of device remains or components which merit further forensic examination by the chemist or by colleagues in other disciplines. Experience of the analytical problems caused by substrate contamination also greatly assists in method selection and result interpretation.

Communication of analytical results is also vitally important in an international context. Forensic scientists lack an equivalent to publications like those of National Bomb Data Centres which do an excellent job of communicating information on devices. It would be very useful to have a similar medium for lab results - bearing in mind the requirements of the intelligence community. The Proceedings of this conference and its predecessor are positive steps in this direction.

Area (i), in the context of the literature and visits to other labs will take up the remainder of the paper.

Explosives Analysis Literature - A Review

In the period up to 1970, there were five papers of note. These were a compendium of IR Spectra of explosives by Pristera 'et al'[1], systematic analysis of commercial dynamites (using spot tests) by Amas and Yallop[2], the application of thin layer chromatography to analysis of nitroglycerine by Lloyd[3], and to a wider range of explosives by Jenkins and Yallop[4], and a study of explosive damage to metal by Tardif and Sterling[13]. These papers are as useful today as when they were written.

The paper by Jenkins and Yallop[4], set an excellent example for successive authors in the explosives field by testing their method on explosives residues. While this may seem self-evident, there has been some tendency in recent years to report new methods for explosives analysis based on analysis of pure unreacted explosives and application to 'real world' samples from cases. A method is much more likely to gain acceptance by the forensic scientist if its ability to enhance specificity and sensitivity is demonstrated by successful identification of explosives components from test explosions on 'real world' substrates. A good example of the Jenkins and Yallop approach is the field testing of the Thermal Energy Analyser (TEA) detector[14].

The paper by Tardif and Sterling[13] underlined the interdisciplinary nature of post-blast forensic examinations by applying metallurgical techniques to identify evidence of explosive damage to metal fragments produced in an explosion. This work has proved to be very valuable in answering the question 'did an explosion occur?', and is particularly applicable to aircraft disasters. Investigation of the recent tragic crashes of Air India 182 off Ireland in 1985, and of the U.S. Space Shuttle 'Challenger' off Florida this year, has shown that technology now exists to locate and recover debris from the furthest depths of the ocean. Even in absence of residue, the metallurgist can determine, given the pertinent material, and following the test results illustrated by Tardif and Sterling, whether the signatures of an explosion are present. This has been well demonstrated by the U.K. Royal Armament Research and Development Establishment (RARDE) who have shown themselves, through extensive experience, to be world leaders in forensic examination of debris from aircraft. Their approach is well explained in a paper by Higgs, Markham 'et al'[8], which is required reading for anyone faced with this type of investigation. This paper should be read in conjunction with a paper by Higgs and Hayes[15], dealing with post-blast recovery and persistence of nitroglycerine on polymeric material including suitcases.

Returning to general residue analysis, in the 1970's the literature was dominated by the application of routine techniques to common explosives. This represented excellent communication, in which a leading role was played by the U.S. Treasury, Bureau of Alcohol Tobacco and Firearms (ATF). The major authors were Hoffman and Byall[16], Washington and Midkiff [17 18 19 20 21], and Parker 'et al'[22 23 24]. Also in the same period, a systematic analytical scheme developed in Canada was published by Beveridge 'et al'[9], which was based on analysis of residues from a series of test explosions of commercial, military and 'homemade explosives'. Specific emphasis was paid to identifying not only unreacted explosive components, but also inorganic reaction products and insoluble residues such as barium sulphate and manganese dioxide. The Israeli National Police Forensic Science Service published two papers in this period, by Kaplan and

Zitrin[25], and by Kraus 'et al'[26]. These papers concentrated on identification of military and homemade explosives. Twibell[27] 'et al', published a paper on differentiation of sodium chlorate samples. Yander and Hanley[28], described the collection of explosives vapors at the scene of a blast, and subsequent analysis using electron capture detection on a gas chromatograph. Camp [29], showed the way to the future by describing a procedure using thin layer chromatography (TLC) for clean-up, followed by high pressure liquid chromatography (HPLC) separation, and identification by chemical ionization mass spectrometry following on the lead of Vouros 'et al'[32], and Yinon[31]. The papers in references 12, 26, 27, 28 and 29 originated from the 'New Concepts Symposium and Workshop on the Detection and Identification of Explosives' sponsored by U.S. Departments of Treasury, Energy, Justice and Transportation in 1978. The primary theme was tagging of explosives, but this concept did not survive long enough to become of practical use. Some useful reviews of explosives analysis in the 1970's were given by Yinon[31] (general), Moler[32] (general), Krull (HPLC)[33], and Yinon[34] (mass spectrometry). The most detailed and comprehensive review is given in the excellent text 'Analysis of Explosives' (1981) co-authored by Yinon and Zitrin[35].

The eighties brought major changes to residue analysis, as new methods first introduced at the New Concepts Symposium in 1978 were refined and developed, and further methods came on-stream. The literature branch dealing with systematic analysis continued, as exemplified by Hayes[11], who updated the original Amas and Yallop[2], scheme, and Beveridge 'et al'[10], who studied the residues produced from confined and unconfined chemical mixtures, and from water gels. These authors continued to used more established methods. But in 1983, a systematic scheme published by Rudolph and Bender[36], of the F.B.I. Laboratory was a dramatic contrast to that presented (but not published) by the F.B.I. only five years previously at the New Concepts Symposium. The 'old' scheme was similar to that published by Beveridge 'et al'[9] in 1975; the new scheme added ion chromatography (IC), and HPLC with UV and TEA detectors, along with gas chromatography/ mass spectrometry (GC/MS). These, and other developments, led to the F.B.I. to organize and convene the first 'International Symposium on the Analysis and Detection of Explosives' at the F.B.I. Academy in 1983. The Proceedings of that meeting stand as a tribute to the organizers and participants and as a first rate compendium of advances in explosives residue analysis up to 1983. It is a prime example of 'communication' of 'experimentation' (the key words cited earlier).

In reviewing the literature of the eighties, a close parallel is apparent between developments in forensic drug analysis and forensic explosives analysis. The drug analyst/ toxicologist uses HPLC routinely, but drug identification is based more on spectroscopic methods (IR, MS, NMR) than on chromatography. This has almost been dictated by the courts.

Explosives analysis is moving in this direction in some laboratories, whereas others are satisfied with a new generation of nitro/nitroso-specific detectors for GC and HPLC, or by using dual chromatographic analysis with different columns. It is my distinct impression that we are now in a period of consolidation following the innovations presented in 1983, as seen by the continuity in topic from that conference to this.

The remainder of this review looks at developments in the forensic/scientific literature up to the present from the early eighties. The review is restricted to literature readily available in our forensic laboratory library system, and excludes medical/pharmacological publications.

The introduction of new explosives and formulations has resulted in several papers dealing with identification of specific components. Thus, water gels or cap-sensitive slurries marketed as dynamite substitutes have been analyzed by Parker[24] (IR), Peterson 'et al'[37] (TLC), Prime and Krebs (HPLC)[38], Beveridge 'et al'[10] (IR; post-blast residues), Reutter 'et al'[39 40] (ion chromatography) and Barsotti and Hoffman[41] (ion chromatography).

The introduction of a 'no headache' dynamite 'Hercodyne' by the Hercules Company produced the opposite effect on the scientists who first encountered it. Two components 'metrioltrinitrate (MTN)'[6], and diethyleneglycoldinitrate (DEGDN) were successfully identified by HPLC and GC/MS[42]. Two unusual peroxide explosives[43], hexamethylenetriperoxidediamine (HMTD)[42 44] and triacetonetriperoxide (TATP)[44 45] received as 'unknowns' were identified by detailed systematic analysis. Interpretation of the analytical results was very well presented[42 44 45]. Two unusual pipe bomb fillers originating from pyrotechnics have been described by Fung[46]. The compound trioxane was identified as an adulterant in an IED; identification was by IR/NMR[47]. These papers are very useful to all chemists who may encounter the same compounds as future 'unknowns'..

High Pressure Liquid Chromatography (HPLC)

HPLC has dominated recent literature on explosives residue analysis, having proved more sensitive and specific than thin layer chromatography, and less likely to produce thermal decomposition than gas chromatography.

The applicability of HPLC to explosives analysis and the literature to the early eighties are covered in a detailed and useful review by Krull[33]. The technique is well established in forensic laboratories, usually having been used in drug analysis before being applied to explosives. Recent developments have centred on detectors.

The UV detector has been widely used for explosives analysis. In 1982, Neumann[48], described its application to analysis of RDX, TNT and PETN and to NG in smokeless powder. In 1983, Lyter[49] described its application to analysis of seven common commercial dynamite and military explosives, and in 1984, Prime and Krebs[50] extended their earlier work on commercial dynamite[38], to cap-sensitive slurries[50]. The latter paper described the test-explosion debris work-up procedure used to test the method. Most recent applications have involved determination of traces of nitro-organics in munitions plant waste water [51 52].

In the early eighties, Kissinger and co-workers began to apply electrochemical detection to HPLC analysis of explosives (LC-EC). In 1981, Bratin, Kissinger 'et al'[53] described application of reductive electrochemical detection to a wide range of explosives, and at the International Symposium at Quantico in 1983, the technique was reviewed by Kissinger[54]. Its application to analysis of explosives and gunshot residue was described by Briner, Bratin and Longwell,[55]. The method has subsequently been applied by Maskarinec 'et al'[56], to detection of munitions components in water.

Krull 'et al'[57] have modified the method to enhance specificity by incorporating photolysis between the HPLC effluent outlet and the electrochemical detector, which was operated in the oxidative mode. This system (LC - photolysis - EC), has been applied to analysis of both drugs and explosives, including post-blast residue samples. At this conference, Dr. Selavka will describe improvements in sensitivity, and extend the application to water-gel formulations.

Another form of electrochemical detector is that developed by Lloyd - the pendant mercury drop electrode (PMDE). In a series of papers in 1983/4, he described the practical aspects of the technique itself[58] [59] and its application to analysis of organic explosives components [60], explosives on hand swabs[61] [62], and detection and differentiation of nitrocellulose traces of forensic interest[63]. It is interesting to contrast Lloyd's 1967 TLC paper[3], wherein it was reported that nitroglycerine could not be detected at a level of 0.1 μg, to the PMDE [60] with a reported detection limit for nitroglycerine and other organic explosives of 7-49 pg for 20 μL injected. Dr. Lloyd's presentation to this conference, will include the application of LC-PMDE to organic components of gunshot residue.

The third type of detector to be used with HPLC of explosives is the 'thermal energy analyzer' or 'TEA' detector. This is a nitro/nitroso specific detector which can be used with or even interchanged between a liquid or gas chromatograph. Its potential application to explosives residue analysis was introduced at the New Concepts Symposium in 1978, by Lafleur, Morrison and Fine[64], and following further development and testing in conjunction with a forensic science laboratory[14] was presented in greater detail by Goff, Yu and Fine[65] [66], at the International Symposium in 1983. The detector, being selective, has the advantage of minimizing the clean-up procedures for extracts of post-explosion debris which are a pre-requisite for most other forms of instrumental analysis (see below). Additionally, sensitivity for common explosives is in the range of 4 to 5 picograms injected on-column [14]. Its application has been described for the detection of commercial and military explosives both pure and in post-blast debris[66] [14], for handswabs[14], and for smokeless powders[67]. This detector is in use for forensic explosives analysis in the U.S., Canada and the U.K.

A fourth detector for the HPLC is the mass spectrometer (MS). The potentially potent combination of the separation ability of HPLC with the identification ability of the MS has been reviewed by Yinon[68], and applied to explosives analysis by Parker, Yinon 'et al'[69] and Yinon 'et al' [70] [71] [72] [73] [74], the latter work dealing in large measure with determination of explosives metabolites in body fluids. This technique was not yet in use in any of the forensic laboratories visited, but due to these publications, we know that we need look no further than the Weizmann Institute for the instrumentation and expertise (scientist as consultant)!

Two other HPLC detectors reported in the eighties were electron capture by Krull, Kraus 'et al'[75], and the fourier transform infrared spectrometer (FTIR) by Riddell and Mills [76]. Papers by Rudolph and Bender[36], and Albanbauer 'et al'[77], described the systematic application of HPLC to post-blast residue analysis as part of a routine analytical scheme.

Ion Chromatography

Ion chromatography is a sub group of LC applied to the separation of ions. Good commercial instruments are now available, and the method has been described in detail by Reutter and Buechele[39]. Papers on its application to analysis of explosives have been given by Reutter 'et al' [40] (general application to explosives and their post-blast residues), Rudolph[78] (low explosives), and Barsotti 'et al'[41] (water gel explosives). The application of ion chromatography within a systematic analysis scheme has been presented by Rudolph and Bender[36], and this method was found in use in most labs visited.

Ion chromatography offers an advantage over traditional techniques for ion identification such as X-ray powder diffraction, infrared spectroscopy and spot tests, which are usually applied to the residue of evaporated solvent extracts. This is that solvent extracts may be analyzed for ions without the evaporation or heating which have been shown to cause loss of evidence[39 40]. Fortunately, ions such as ammonium and 'monomethylamine' (MMA) which can be lost on aqueous solution evaporation can be readily isolated from acetone extracts, and qualitatively identified by established methods[10]. However, the sensitivity and ability to quantitate with ion chromatography are further good features of the technique. IC does not, of course, assist in identifying ions in water-insoluble compounds and does not replace traditional methods - it complements them.

Gas Chromatography (GC)

Gas chromatographic analysis of explosives using an electron capture detector (ECD) has been used in Japan since the mid-1970's[79 80], but did not achieve wide-spread use until the introduction of fused silica capillary columns in the late seventies. The development and application of GC analysis explosives to in the seventies may be reviewed in the text by Yinon and Zitrin[35]. ECD was combined with fused silica capillary columns by Douse[81] who achieved detection levels in the low picogram range for components of pure samples of common commercial and military explosives. Douse[82] then successfully applied GC/ECD to analysis of explosives in hand swab extracts. The method incorporated a clean-up procedure based on selective adsorption using an "XAD-7" resin. Douse[83] later applied the same method to GC/ECD analysis of benzodiazepine drugs, thereby underlining the close parallel between drug and explosives analysis. Douse's[84] next step in explosives analysis was to replace the ECD detector with a TEA detector (GC/TEA). This resulted in selective detection of several common explosives in the low picogram range. His ultimate step[85] was to use both the EC and TEA detector to identify low nanogram levels of explosives in hand swabs, using a clean-up procedure to protect the column. He concluded that the TEA detector approached the sensitivity of the ECD, but was more selective.

In an independent study of hand swabs in the same period, Twibell 'et al'[86] performed a comparative study of GC/ECD, GC/MS and TLC for analysis of recovered traces of nitroglycerine. They selected GC/ECD as the most accurate and sensitive method. In a comparative study for recovery of common organic explosives from hand swabs for analysis by GC/ECD, Twibell 'et al'[87] found centrifugal microfilter extraction to be most efficient. Twibell 'et al'[88] also studied the persistence of military explosives on hands using GC/ECD for detection.

GC/ECD was used by Yip[89] for analysis of nitroglycerine (NG), ethylene glycol dinitrate (EGDN), and ethylene glycol mononitrate (EGMN: a water gel sensitizer). In a useful contribution by industry, Penton [90] described detection of NG at subpicogram levels using cold on-column injection onto a fused silica column using an ECD. GC/ECD was also used by Balkin 'et al'[91] for analysis of explosives traces in water.

GC/Mass Spectroscopy

The application of mass spectroscopy to analysis of explosives was reviewed by Yinon [34] in 1982. The technique, being a specific spectroscopic method of identification and being very widely used in the forensic drug/toxicology field, seems well suited to explosives analysis. Its use as a detector for liquid chromatography of explosives has been cited above[70]. Cumming and Park[92] have discussed the analysis of trace levels by explosives by gas chromatography/ mass spectroscopy (GC/MS), and concluded that the combination of electron impact, negative chemical ionization MS and capillary G.C. is a very powerful analytical system. GC/MS has been included in recent systematic analysis schemes[10 29 36]. Analysis of explosives by tandem mass spectroscopy (MS/MS) is a new "high tech" development reported by Yinon[93], and McLucky 'et al'[94]. Indeed, Dr. Yinon will be presenting a paper to this conference on analysis by MS/MS of some polynitrated cubanes - the new class of explosives just introduced to us by Dr. Alster[7]. This is an outstanding example of keeping-up with new technology - both technical and industrial. This work follows from studies of the collision-induced dissociation of RDX and HMX using electron impact and chemical ionization[95]. The fragmentation pattern of RDX using chemical ionization was also studied by Zitrin [96]. Papers at this conference by Dr. Zitrin and Ms. Tamiri will give further practical dimensions in the application to explosives of this potent technique.

Clean-Up and Recovery

A major topic in recent literature has been clean-up and recovery procedures to concentrate explosives traces and minimize environmental contamination.

Two methods offer minimal need for clean-up procedures. One example is a heated portable device which absorbs explosives vapors onto Tenax® resin. This was reported by Wardleworth and Ancient[97] at the International Symposium in 1983. The paper includes discussion of the advantages over solvent extraction. The second method used the nitro/nitroso specific TEA detector discussed above[14 65 66 84 85].

The interdisciplinary nature of recovery was illustrated by Laposata[98] who discussed optimal collection of trace evidence from bombing victims at autopsy.

Most papers, however, dealt with preconcentration of extracts for trace analysis of explosives. Douse[82, 83 85] has described clean-up procedures for GC/ECD analysis. This was based on adsorption using "Amberlite XAD-7" porous polymer beads. Strobel and Tontarski[99] studied a series of bonded resins and derived a scheme for clean-up of samples for HPLC analysis. Higgs and Hayes[15] described the persistence of nitroglycerine on polymeric materials and recovered NG both by solvent extraction and thermal desorption. Twibell 'et al'[100] assessed solvents for recovery of nitroglycerine from hands using cotton swabs, and concluded that ethanol was best. The most recent papers were by Lloyd[101] [102] [103] who has described a centrifugal filter clean-up system for trace analysis[101] [87] and microcolumn clean-up system for traces of explosives and propellants from explosives debris and gunshot residue[102]. He has also undertaken a very thorough comparative survey of the adsorption characteristics of organic explosives on typically-used adsorbants[103].

Other Methods

Other methods reported since 1982 are raman spectroscopy [104] and polarizing microscopy [105].

Literature Summary

The most cited source in this review was the Proceedings of the International Symposium in 1983. Some of the Proceedings' papers have been published in journals. In these instances, only the journal has been cited. The two most frequently cited journals from 1982 to the present were the Journal of Chromatography and the Journal of Forensic Sciences. Over 70% of the papers were authored by forensic scientists working in government laboratories, and about 16% were from research institutes - primarily from Dr. Yinon's group at the Weizmann Institute. The only university input to forensic explosives analysis, was from Dr. Krull's group at Northeastern University in the U.S. The cited literature originated from only five countries. The major contributors were the from U.K. and U.S. followed by Israel. Contributions also were made by Canada and Germany.

There were some encouraging examples of cooperation, specifically international co-authorship in mass spectroscopy [69], international forensic laboratory/university co-authorship on HPLC/ECD[75], forensic laboratory/industry co-authorship on the TEA detector[14], government/university co-authorship in HPLC analysis of explosives in water[51] [52], and university/industry co-authorship in HPLC - photolysis - EC[57]. The task of advancing the field, however, continues to fall primarily on the forensic scientist researcher, who, unless working from a research position, has also to carry his share of casework. Progress in research contributions from the operational chemist requires skilled management of time and resources, and recognition that time spent on R & D has as much benefit to the investigator by enhancing analysis and interpretation of the results as does actual casework itself.

Scientist as Analyst

The range of possible explosives residues as unreacted components and products, and the range of interfering environmental contaminants, makes explosives residue analysis one of the most challenging applications of analytical chemistry.

Experience gained from test explosions and casework enables the scientist to know what to look for and where to look for it. The literature review illustrates the methodology available. Laboratories dealing regularly with explosives casework follow a systematic scheme of analysis, honed over years by experience, and by familiarity with the methods. If the scheme is to be changed by adding new instruments, accessories and methods, then there will be costs in capital expenditure, training and testing time. In return, there must be benefits in simplicity of use, sensitivity and/or selectivity. Thus, a new method to separate and identify common explosives like NG or RDX is unlikely to excite much interest in an operational laboratory unless it offers significant advantages over established procedures - and it must fall within budget restrictions. Further, a new method must be reliable and ready-to-go when required. A method which yields the above benefits to a skilled researcher may require extensive modification for routine use in a busy operational laboratory. Cost factors alone may dictate centralization of 'high tech' methods. In this regard, Prof. Kind[106] offered some words of caution in his presidential address to the International Association of Forensic Sciences in Oxford - namely that a danger in consolidating 'high tech' methods in a central facility, remote from the crime scene, is concentration on the method rather than on the problem. While not specifically referring to explosives analysis, the caution is still apt for the forensic scientist - but not for the academic or institutional researcher who is pursuing knowledge without application restrictions.

Our visits to leading government laboratories specializing in explosives residue analysis show that new methods will be adopted if they offer sufficient benefits. Two prime examples are ion chromatography which permits identification of ions otherwise lost on evaporation of aqueous extracts, and the TEA detector for GC and/or LC which permits selective detection of nitro/nitroso-containing compounds. Ion chromatography was observed in most forensic laboratories visited, and the TEA detector was observed in North America and the U.K. Both methods, significantly, were thoroughly tested by a forensic laboratory on samples from test explosions, and the results published - 'experimentation' and 'communication'.

The visits showed that HPLC is now the method of choice for separation, with the older TLC technique being used more for screening or preparatively for clean-up. HPLC detectors observed were UV, TEA, and in Britain, the pendant mercury drop electrode electrochemical detector. Gas chromatography was also widely used, with the most favoured configuration being direct on-column injection onto fused silica capillary columns using EC and/or TEA detectors. Mass spectrometry was used less than anticipated. LC/MS and MS/MS were not observed in any forensic labs, and while GC/MS was used in some for explosives analysis, the use was much more directed to drugs in comprehensive-service laboratories with one mass spectrometer. As noted above, ion chromatography was widely used, but, so far, little study seems to have been made on IC of typical explosives residue environments like soil or clothing.

We found that infrared spectroscopy using a diamond cell on an FTIR instrument, which is a prime non-destructive technique for organic and inorganic explosives residues in R.C.M.P. laboratories, was little used elsewhere. But this did lead to useful discussions. Several, though not all, labs used X-ray powder diffraction. The same applies to SEM/EDX where applications ranged from physical examination for explosives signatures through elemental analysis to elemental mapping. The mapping technique, using overlaid photomicrographs, showed which elements were associated in unextracted residue[107]. Stereobinocular microscopy was almost universally used to examine debris prior to extraction, and most labs used spot tests in one form or another. Within the U.K., two areas of particular R&D interest and practical application were hand swabs, and analysis of gunshot residue for organic components (see literature review above). Those methods cited in the literature, but not in use in the major forensic labs visited, remain valuable sources of information, putting their authors in the position of scientist as consultant to the scientist as student.

Without publication of ideas and data, the essential communication process fails.

Most chemists can analyze. The forensic chemist is distinguished from his peers by possessing the background knowledge required to interpret the significance of analytical results and to present the interpretation to the investigator and the courts. There is no more complex field than explosives residue analysis. The incidents often involve death, terrible injury and destruction - and publicity, and pressure, and cross-examination of every step taken and every opinion reached. The 'analysis' is but part of explosives residue analysis. The forensic scientist who concentrates his preparation for casework on experimentation with explosives residues and communication of his results will be best able to assist investigation of explosives incidents amid the information overload of the eighties. That is, the forensic scientist best prepared to undertake explosives residue analysis must be an educator, consultant and student as well as an analyst.

While our next specific meeting on the analysis and detection of explosives is three years away, I hope that some of you will make your way next year to my home city of Vancouver, and contribute to the 1987 Meeting of the International Association of Forensic Sciences.

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