



Interpol review of the analysis and detection of explosives and explosives residues

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1. Introduction and coverage of the literature

The authors would first recommend that readers review the previous three review papers from 2013, 2016 and 2019 [22–24]. Those may have some articles on technology that laboratory managers might find useful and adaptable to their current approaches to explosives analysis and that may be of use in future real-world applications.

The international situation in 2022 is notably different from that in 2019 when the last iteration of this paper was published. There is considerably less focus on in theater devices and explosives as Allied Forces have mostly withdrawn from the Middle East and Afghanistan. Unfortunately, however, because of this withdrawal and the resulting dearth of human intelligence, people who engage in terrorist activities are less restrained in securing improvised explosives and precursors. Additionally, and dangerously, these terrorists now have access to war material left behind by withdrawing forces, including military grade explosives and munitions. A re-focused effort on traditional military high explosive detection and analysis may be needed for laboratories worldwide if these are deployed in both criminal and terrorist bombings.

Another crisis that may result in concerns for years to come for many countries is the current war in Ukraine. All wars give rise to the long-term problem of criminals and terrorists having access to excess munitions and explosives well after the war has come to a close. This was evident after the wars in Iraq and Afghanistan, but also after the wars during the breakup of former Yugoslavia. Those munitions and explosives bleed over into neighboring countries impacting the security of the region.

Beginning in 2020 the United States experienced a substantial increase in incidents of civil unrest. The criminal misuse of explosives and ignitable liquids was widespread and was recorded from large urban centers to smaller towns. While some explosives were deployed against government entities from police officers to court houses, some opportunistic individuals used the cover of riots and protests to engage in

criminal bombings for monetary gain, especially by trying to breach ATM's. Almost all of the devices deployed were low explosive pyrotechnic devices and some were modified with shrapnel. Almost all of the increases in backlogs at our laboratory were from such civil unrest.

As we wrote in the last (2019) paper, "One of the most important yet difficult areas for the past ten to twenty years for the explosive analyst is the ever-changing type of explosives employed by the criminal bomber and terrorists. Restrictions on widely used commercial and military high explosives are often circumvented by the illicit production of homemade explosives. While there have been attempts to restrict chemical precursors and some oxidizers and fuels, criminal and terrorist bombings are still frequently using homemade explosives. Some of these explosive formulations are difficult to detect in chaotic and contaminated scenes, with matrices that are additionally problematic. The two biggest reasons for failure to identify a post-blast homemade explosive in some of these cases are the failure to collect samples in a timely manner and the failure to properly extract the analytes from difficult matrices. While training of first responders and others may help with the first issue, the second issue falls mostly on the explosives forensic community. There is not a lot of research in this area, but a few referenced papers do address this second issue" [24].

Introducing new techniques into the suite of analytical schemes or standard approaches has become increasingly more laborious as most laboratories are now accredited, many to ISO 17025. Accreditation standards demand more data and study before a new technique can be validated and brought online by a laboratory. This even extends to more simple transitions like swapping out columns with slightly different chemistries. Yet many laboratories do not have dedicated analytical groups to do this kind of validation work. This may add to the hesitancy of laboratory managers to adopt new techniques. If the new techniques fill a crucial need or cover a gap in overall explosives coverage those hesitations should be put aside and efforts made to complete validations.

This review of the literature will hopefully demonstrate that there

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are many applications in the world of explosives analysis from many fields that are of interest to the forensic laboratory manager. The authors and chemists at the ATF National Laboratory Center have spent hundreds of hours of reviewing and classifying abstracts, reading papers, and selecting pertinent papers to highlight in this review. Additionally, hundreds more citations are available in the bibliography. We have drawn from explosives detection, environmental, academic, and case study articles to try to cover the range of probable areas of interest. There are still dozens of references in this area, ranging from theoretical research to applied systems that are already in field use.

There are 1004 references in this review. The bibliography portion of the paper includes numerous references not specifically highlighted in the paper which may be of interest to the reader. It starts on page 23 and follows the same category structure as the paper. Many of these references could fall into two or even three categories. They will not be presented in multiple places, so it would be advantageous for the reader to peruse all of the sections. The organization of this paper follows the same pattern as the previous reviews.

2. Review Articles

This three-year cycle includes several review publications. While some are broad schemes of analysis, others are reviews of a specific class of instrumentation or field of study. Others are self-described as reviews. Review papers are useful to give a broad overview of advances in particular aspects or categories of forensic explosive analysis.

Evans-Nguyen, K. and Hutches, K. have a book on *The Forensic Analysis of Fire Debris and Explosives* and is a very solid accounting of the current state in the analysis of explosives [13].

There is a brief but interesting chapter by Wolfgang Greibel on what end users on the scene of an explosion or bombing have at their disposal instrument wise and additional techniques which may be used in the future [15].

Cagan, A. and Oxley, J. have published a comprehensive Second Edition of *Counterterrorism Detection Techniques of Explosives* which covers “updated research findings that will be used for the next generation of explosive detection technologies.” including the most currently employed open-source detection technologies [10]. While many techniques cannot be directly translated or used in a forensic laboratory because detection has different standards than identification, it is well worth the time reading.

Forbes, T., Krauss, S., and Gillen, G., have published a very thorough and interesting review of the challenges of analyzing for trace homemade fuel-oxidizer mixtures. It is a comprehensive front to back analysis of the current state of analysis and detection for forensic laboratories [14].

Crocombe, R.A., Leary, P.E., and Kammrath, B.W., published Volume One of the technology and instruments used in portable spectroscopy. This covers a general discussion of XRF, UV-Visible, near-infrared, mid-infrared, and Raman. In addition to traditional portable spectroscopy techniques this review also considers LIBS, NMR, and others [11].

In Volume two the above authors show how these instruments are used in various fields. They also discuss the algorithms for detection, library and method development, and the future of portable spectroscopy [11].

Sisco, E., and Forbes, T.P., reviewed the application of DART-MS to a variety of forensic samples including explosives. In addition to a thorough review of the advances for the last five years, the authors identified areas where additional research is needed to continue to advance the technique [39]. As we write below DART-MS is an increasingly useful tool in forensic chemistry.

Herweyer, D., et al. reviewed the advancements made in new “Green” primary energetic materials concentrating on potassium based energetic salts, metal-free ionic energetic salts and metal-free covalent energetic materials. The paper summarizes the “explosive performance, initiability, safety and environmental impact” of several new materials

[18].

Nanoexplosives are another field where new materials continue to be developed. While this paper could be placed in novel explosives, nano-explosive materials have been around long enough to be reviewed by Yetter, R.A. This review covers how to “... understand how to manipulate and build energetic materials at the nanoscale ...” [46].

3. Explosive Standards and References, Laboratory Quality Control, Contamination Prevention

While this category is rather broad, it encompasses many references that could be highly useful in a forensic laboratory setting. As instrumentation becomes more and more sensitive having a framework for things ranging from engineering controls to standard approaches to prevent contamination is imperative.

Gareth Collett has published a fantastic report focused primarily on precursors for Homemade Explosives (HME) including recommendations for preventative actions that could limit access to the materials. It also recommends a global repository for HME based IED’s and additionally links every non war zone HME IED attack with the confirmed or suspected HME used since 1970 [52]. It should be required reading for any analyst in the field.

Schachel, T.D., et al. propose a “pan-European Forensic Substance Database on Explosives” to include not just the base explosives but more importantly for sourcing and or brand identification, the additives in various products. They used a combination of HPLC-HRMS (high resolution mass spectrometry), XRD, and XRF and identified 41 additives with diagnostic potential [55].

Stein, J., did a thesis at Virginia Commonwealth University looking at transfers of explosives used in render safe procedures to device components of simulated IED’s. The author considered specifically the use of a Percussion Actuated Disrupter (PAN) and a water bottle shot with detonating cord. Transfers were detected [56]. This research highlights the necessity of communication between the field and the laboratory.

ASTM International has published *ASTM E2520-21: Standard Practice for Measuring and Scoring Performance of Trace Chemical Detectors* and is a good resource for assessing the performance of trace explosives detectors that use swabs [49].

Also, ASTM International published *ASTM E2677-20: Standard Test Method for Estimating Limits of Detection in Trace Detectors for Explosives and Drugs of Interest* [50]. This standard may be of interest to labs establishing limits of detection for new or existing methods.

4. Sampling and Concentration of Explosive Traces

Despite the volume of research done in this category over the past several years there remain significant gaps in the field of sampling and concentrating explosive samples in the post-blast debris and trace detection realm. Ease of use and robustness of any given technique are necessary as is universality. It is the latter, universality, that is hardest to achieve because there are simply so many classes of explosives and their post-blast reaction products.

Irlam, R.C., et al. researched seven different sorbents (solid phase extraction) for recoveries of 14 types of explosives and matrices that ran the gamut from dirt to cooking oil to wastewater to determine which was the best for analyte recovery. They found that “With the exception of river water, the matrix effects were lowest using dual sorbent SPE, with little/no compromise in recovery.” They report that this approach resulted in an approximate 10-fold limit of detection over the single sorbent approach. Finally, they report that Oasis HLB and Isolute ENV + yielded the best recoveries quantitative wise [68].

Additionally, Irlam, R.C., et al. have applied 3-D printed LEGO® inspired miniature blocks for SPE extractions of explosives in a variety of matrices. It is believed that this is the first reported use of 3-D printed SPE blocks. Efficiencies of the extractions look promising [67].

In the area of ion chromatography, Mauricio F.G.M., et al. evaluated swabs and syringe filters and found that of six syringe filters only two were free of interfering analytes (such as Na, K, Mg, Ca, Cl and SO₄). All “forensic” swabs had significant amounts of these as well. They further analyzed commercially available cotton swabs, cotton balls, and cotton discs and found these to have similar interferents albeit on a, surprisingly, lower level than the forensic ones. Finally, they report that all sampling materials can be cleaned of these ions through three washes [71]. The nature of post-blast analysis of inorganic explosives is such that these types of ions are often all that remains to be detected. Therefore, selection of a swabbing material that has minimal interfering analytes is advantageous and contributes to a stronger conclusion making this particularly relevant research.

While pertinent to an environmental application, Temple T., et al. researched four different types of one step extraction processes (stirring, shaking, sonication and accelerated solvent extraction (ASE)) on five different soil types and report that shaking is the most reproducible, but less efficient than stirring and ASE was the least reproducible. Also, they report that soils high in organic content (>2%) unsurprisingly affected both extraction efficiency and reproducibility [774].

Rodriguez J., and Almirall, J., demonstrated the efficiency in volatile vapor sampling using continuous capillary microextraction and reported recoveries of 3.0–89%. By comparison they reported headspace and simulated open air sampling resulted in less recoveries. The analytes were 3-NT, 2,4-DNT, DPA, EC, DBP and 2-NDPA, all compounds that can be found in smokeless powders [74].

Glackin, J.M.E., et al. published an article on coating swabs with a fluoropolymer which was then used to swab an explosive and thermally desorbed, “... causing the quenching of light emission from a thin film luminescent sensor.” They first tried it with 2,4-DNT and reported an increase of three orders of magnitude in sensitivity over standard colorimetric tests. Then it was used on PETN, RDX, and TNT, and even on post-blast samples [66].

Evans-Nguyen, K.M., et al. reported on employing a solventless, noncontact electrostatic sampling method for drugs and explosives. They used a hand-held Van de Graaf generator and wire mesh held close to the sampling substrate. The particles transferred to the mesh and were analyzed by thermal desorption electrospray ionization and mass spectrometry [62].

Avisar, Y.Y., et al. described using hot water to extract post-blast samples. Then they conducted a liquid-liquid extraction with a very small amount of dichloromethane (DCM) before analyzing by GC-MS. They extracted several different types of substrates (metal, sponge, asphalt, gravel) and reported that this method leads to notably less sensitivity loss in the GC-MS after 40 injections than a traditional solvent extraction. This extraction technique has been used successfully on real life samples [59].

Novoselov, Igor V., et al. completed a study on the recovery of certain high explosives in the swabbing of various surfaces found in security (mass transit) settings. They desorbed the analytes in various ways but found “... nylon had the lowest collection efficiency (CE%) for all cases (appx. 10%) and stainless-steel mesh had the lowest CE% for the evaluated traps” [72].

5. Identification of explosives, explosive residues and explosive properties

There are several articles describing the properties of explosives and theoretical modeling of explosive behavior. Also of interest is the area of novel explosives and proposed improvements to existing commercial and military explosives. Some of these articles also describe analytical techniques.

A. Commercial Explosives

Gao et al. reported on 3-D photopolymerization printing of a CL-20

based propellant with the goal of improve the energy content and combustion performance of propellants made with additive manufacturing techniques [106]. It is unknown if 3-D printing on a large scale will be economically feasible for mass production or more suited for quick synthesis of new combinations of materials.

Ali, F., et al. report on using waste lubricant oil as a partial substitute for fuel oil in ANFO formulations. They found that the formulations exhibited both effective explosive performance and were within the permissible limit for toxic fumes and particulate formation [78].

Lennert, E., and Bridge, C., used statistical analysis to associate smokeless powder residues from burned smokeless powder to the original smokeless powder using Sorensen-Dice similarity coefficients. The samples were run on GC/MS. They also explored the pyrolysis products of the smokeless powder constituents. Their results were mixed, stating, “The complexity of burning a mixture, such as SP, compared to burning single components may contribute to the generally low similarity coefficients observed in the comparison” [118].

Krejcir, K., Adam, J., and Buldra, R. conducted a multi-temperature study on the depletion of three common stabilizers used in smokeless powder over time. The study aims to provide background information for stability testing of smokeless powders [116].

Wang, Y., et al. explored the usage of different gases in the sensitizing microballoons in emulsion explosives and reported that the use of hydrogen gas (as opposed to helium, oxygen and nitrogen) can improve the power and energy of the emulsion explosive. Oxygen and nitrogen provide little improvement and helium is both difficult to incorporate into the explosive system and had a negative influence on performance [147].

B. Homemade Explosives

The area of Homemade Explosives (HME) is of tremendous interest to the forensic explosive’s community. As we stated in our 2019 paper, “Sometimes called Improvised Explosives (as opposed to an Improvised Explosive Device that may or may not use HME), these explosives can, in general terms, be defined as non-factory manufactured explosives. It is uncommon, but not unheard of, however, that makers of HME will attempt to make a “commercial” type of explosive” [24].

We additionally wrote, “The actual usage of HME is constantly changing and it is difficult for forensic laboratories to have adequate protocols for every possibility” [24]. This axiom still holds true now. There is no adequate way for a regular forensic laboratory to have every possibility of HME usage covered in a post-blast situation. Intel and scene examination can help steer analysis but having the protocols in place for every eventuality, especially for a novel HME, is nearly impossible. That said, it can help if analysts keep up with current trends in HME usage worldwide.

Cory Christopher Pye reports a cautionary tale on the long-term storage of a consumer bottle of 2-propanol wherein TATP formation was noted after a dozen years past the expiration date of the product [172]. Anecdotally, the ATF lab has fielded calls where this has been found to have occurred. Fortunately, these calls are infrequent, but it is a valuable safety point for hazmat teams in particular to be aware of as well as for laboratory personnel answering questions from those in the field.

Horvath, T., and Ember, I., discuss the use of Raman and FTIR handheld instruments for on scene identification of HME and highlights the value of using the technologies in parallel [168].

Stromberg, J. and Castillo Rolon, M. conducted a study on TATP headspace using quadrupole LC/MS and found TATP was the only measurable component in the headspace, and it was noted that “the results of this study strongly suggest the proposal that diacetone alcohol and acetone are intractable components of TATP’s ‘vapor signature’ is incorrect” [174].

D’Uva, J., et al. synthesized urea nitrate from a variety of commercially available urea sources and then analyzed the product by FTIR,

SEM/EDS, and most importantly by ICP-MS where trace elements could be determined [166]. Although chemometrics and statistical analysis were not undertaken, this proof-of-concept approach to possibly provide investigative leads etc., is very promising.

C. Other Explosives including Novel or New Explosives:

Throughout the course of history there has been a desire to develop new explosives or improve upon existing ones. The experimentation and development have been towards cheaper, more stable, more energetic, less energetic, more environmentally friendly, less susceptible to accidental initiation, more simple manufacturing processes, and more fit for immediate purpose, to name some of the objectives. Thousands of explosives have been designed and tested for decades if not centuries, but most were rejected as they did not improve upon existing explosives in the areas listed above. In this section we will include both reported improvements to existing military or commercial explosives and some unique explosives.

While not exactly novel, Yang, M., Ma, H., and Shen, Z., proposed and studied the effects of repurposing decommissioned RDX with emulsion explosive formulation. They were able to reduce the critical diameter of the original emulsion [307].

In a similar vein Cui, Q., et al. proposed a novel way of introducing TNT into porous aluminum powders and were able to simplify the method of controlling the amount of TNT through a temperature gradient [191].

Liu, Y., et al. report on a new equation on the theoretical detonation performance of aluminized RDX [251].

In the area of nanotechnology Van Riet, R., et al. report on filling nanoporous carbon with an oxidizer. While oxidizer-carbon mixtures are common, the nanostructure of the carbon is a novel report [282].

Similarly, Ma, X., et al. described improving structural control over nanoenergetic materials in an overview paper [257]. Once costs are brought down and scalability makes these products economically feasible these products will most likely find their way into the marketplace.

Li, M., et al. described the production of “photocurable energetic resin based propellants fabricated by 3D printing [242].

6. Instrumental Analysis of Explosives

A) LC/HPLC/UPLC

As was reported in 2019, “Liquid chromatography (LC), high performance liquid chromatography (HPLC), and ultra-high performance liquid chromatography (UHPLC) are all excellent separation techniques and have the advantage of being less destructive to thermally sensitive high explosives than gas chromatography techniques” [24].

Freye, C.E., et al. researched two types of liquid chromatography for measuring the aging and degradation of the PBX 9501. First low molecular weight components of the explosive were measured with HPLC while the high molecular weight compounds were measured using size exclusion chromatography (SEC) [332]. The parameters for each can be found in their paper. These tools may be useful for forensic applications such as comparisons of PBX explosives.

Steiner, A. and Lurie, I. compiled an overview on using diode array detectors (DAD) coupled with both HPLC and supercritical fluid chromatography (SFC) for a variety of analytes including explosives [332].

Versmortean, C., explored an LCMS method development for eight selected organic high explosive compounds from wastewater. The importance of being able to extract and analyze both the original compounds (TNT, RDX, etc.) and their degradation products and optimize for both was stressed [333].

Freye, C., Nguyen, T., and Tappan, B. used UHPLC-MS/MS to explore the synthesis impurities due to the manufacturing process of ETN. They found 12 such impurities and were able to analyze for them [328].

Kotrlý, M. et al. reported on using derivatization of aliphatic and aromatic amines with HPLC-fluorescent detection and having femtomole-level detection [331].

B) Ion Chromatography

The technique of ion chromatography (IC) is often used in forensic explosives analysis for the analysis of inorganic and some organic explosives. Many detectors are used including mass spectrometry. Ion chromatography has the advantage over other inorganic characterization methods because a relative profile of many anions or cations in a sample can be compared against known post-blast profiles of certain explosive types.

Hutchinson, J.P. et al. report on using two techniques for the forensic analysis of explosives and their post-blast products. Mostly exploring inorganic low explosives, they looked at using capillary electrophoresis and ion chromatography as orthogonal techniques, each instrument with its own pluses and minuses [338].

Gallidabino, M.D. et al. used ion chromatography coupled with high resolution mass spectrometry (IC-HRMS) and an ethanol-based eluent. It was interesting that their method did not detect chloride or nitrite. Their ethanol eluent-based approach was reported to be an improvement over methods using an aqueous eluent [335].

C) Gas Chromatography

Gas chromatography has been a workhorse for the qualitative oriented forensic chemist for many decades now and remains pertinent to the examination of many classes of explosives. It provides for quick separations for a variety of compounds and the methods can be relatively easily modified when required.

Qualley, A., Hughes, G.T., and Rubenstein, M.H. report on a method of improving the data in field-portable GS-MS by using the “pre-incorporation of isotopic analogues (of the target analytes) onto thermal desorption tubes in advance of field distribution ...” [346].

Cruse, C. and Goodpaster, J. reported that they optimized various parameters (inlet temperatures, flow rate, and detector gas pressure) for a GC/VUV (gas chromatography-vacuum ultraviolet) system for the examination of several organic high explosives using statistical analysis called response surface methodology or RSM [343].

M. Li et al. published their work on developing a micro GC with a micro-photoionization detector which reportedly with sub-picogram detection limits on trace vapors for optimizing field GC's [345].

D) Capillary Electrophoresis

Capillary electrophoresis (CE) is a separation technique that utilizes charged fields instead of pressure to examine many types of analytes including explosives. It can be used orthogonally with Ion Chromatography or, with a mass spectrometer, as a solo technique.

Krauss, S.T. et al. demonstrated the utility of a wipe based commercial GreyScan ETD-100 capillary electrophoresis for the detection of several inorganic oxidizers including nitrate, chlorate and perchlorate and post combustion inorganic ions [351].

Rapid capillary electrophoresis screening and chemical classification of fireworks was reported by Bezemer, K. et al. The Dutch police provided samples and it should be noted the Dutch civilian use of fireworks is quite common. The detection was indirect UV-detection and the results showed that chemical classification between primarily homemade and commercial pyrotechnics was determined by Ca²⁺ and Mg²⁺ cations [349].

Krauss, S.T., Forbes, T.P., and Jobs D. reported on using gradient elution moving boundary electrophoresis (GEMBE) as a “robust electrokinetic separation technique” for the separation and detection of inorganic oxidizers frequently seen in explosives. They stated that nitrate, chlorate and perchlorate oxidizers were detected from low

explosives and that many possible inorganic and organic fuels did not interfere, and even detected and separated nitrate in post-blast samples [352].

E) General Spectroscopy: Fluorescence, Luminescence, Spectrophotometric, UV, Chemiluminescence

There are hundreds of research papers and reports in this area. They are varied in their practical application to forensic and/or security work. Some could eventually be used in commercial, military, security and law enforcement applications. Still others will prove to be too costly and are too focused on one class of explosives or even a single explosive. There are a few papers the authors wish to highlight.

F) Mass Spectrometry

Mass spectrometry continues to be the most popular technique for forensic explosives analysis and detection. It could be called the “gold standard” of post-blast explosives analysis. Even so, it remains heavily researched for a variety of reasons including sampling optimization, matrices issues because of the nature of criminal use of explosives, and the similarity of lighter compounds, especially nitrate esters, that are harder to positively identify without an orthogonal approach. Soft ionization is rigorously being examined in many publications. One of the most researched aspects in mass spectrometry in the last three years has been in quick sampling and/or screening techniques such as Direct Analysis in Real Time (DART) or similar sampling methods.

Because canines have shown a propensity to detect potassium chlorate even though the vapor pressure of chlorates at ambient temperatures is negligible and decomposition requires temperatures of 300° Celsius, Cajigas, Perez-Amodovar and De Greeff used on fiber SPME derivatization to detect the resultant chloro-2-propanol proving the outgassing of chlorine [457].

Taudte, R.V., et al. described an automated online Solid Phase Extraction (SPE) for a Triple Quadrupole direct introduction analysis for the determination of five compounds typically encountered in smokeless powders. The analytes were introduced into soil and a simulated organic GSR scenario and extracted by this automated SPE with good results. The report 8 s analysis times [484].

ShuQi An et al. published a paper on DART-MS and varied the discharge gases (helium, nitrogen and argon) and found that for TNT and 2,4-DNT the results were that using nitrogen produced mass spectra “dominated by the oxidation products” suggesting nitrogen provides for a highly oxidative environment. They also tested some substrates with thermal desorption DART-MS and reported better results than traditional DART for 14 explosive compounds [450].

Chelsea Black published a Master’s Thesis on exploring the applicability of DART-MS to identify homemade explosive residues post-blast [453]. And then also published with D’Souza, T., Smith, J., and Hearn, N. a paper on using DART-MS for TATP, HMTD, and MEKP with actual (not spiked) post-blast samples of those. The method worked well with actual IED fragments, dry swabs or wet swabs, with dry swabbing producing the least interferents [454]. The fact that this was done with actual post-blast samples and not simulated or spiked ones is promising.

R. Boyea published a Master’s Thesis on using multivariate statistical analysis using GC-MS to compare fired cartridge cases with unburned smokeless powder [456].

Gaiffe, G. et al. used DART-HRMS to examine 83 plastic explosives and polymer constituents that may be encountered in PBX’s and post-blast samples of some PBX’s. They confirmed a suite of polymeric compounds in Semtex 10. They also were able to see what the changes were post-blast noting “... the best way to describe post-blast polymer samples is that they are less oxygenated and, above all, more unsaturated than the original starting material” [466].

Using ICP-MS and principal component analysis (PCA) Joshua A. D’Uva et al. categorized 48 intact sparkler compositions by looking at 50

elements and could categorize eight distinct groups using the elements V, Co, Ni, Sr, Sn, Sb, and W [461]. It would be interesting to see if in unknown post-blast samples, the patterns could apply as well.

Similar to DART, Fowble, K.L. and Musah, R.A. used laser ablation direct analysis mass spectrometry to hyperfocus on fingerprint ridge details for a variety of illicit substances including the explosive RDX [464].

DART-MS is primarily a fast technique wherein it’s use as a sample screening tool is theoretically very useful. Frazier, J., Benefield, V., and Zhang, M. test five different types of DART-MS sample introduction techniques for a suite of explosives and reported that a Dart gas stream of 200 °C as well as the addition of an acetic acid dopant to wet swabs produced the best results as other methods even including thermal desorption “failed in one aspect or another to be high-throughput, sensitive, and/or robust” [465].

Another sampling technique pre GC-MS/MS introduction is described in a paper by Galmiche, M., et al. Explosives were sampled from water using stir bar sorptive extraction (SBSE). They varied type of stir bars, ionic strength, added organic solvent as well as varied times of extraction and desorption. For those analysts seeking explosives sampling from aqueous solutions, this paper is well worth reading [467].

Gonzalez-Mendez, R., and Mayhew, C.A. published an article on using soft chemical ionization for quantitation of five additives commonly used in smokeless powders through thermal desorption and qToF-MS but all for pre-blast (i.e. intact) smokeless. A follow up study on detection post-blast with this technique might prove useful [469].

Bonnar, C., Popelka-Filcoff, R., and Kirkbride, K. used direct sample analysis ion source integrated with a ToF MS for nitroglycerin and a host of common additives found often in smokeless powders both in intact and post combustion modes with limited results on an attempt at organic gunshot residues on a shooter’s hands. There is a detailed discussion of the ionization and fragmentation patterns well worth perusing [455].

Supajariyawat, P. and Gonzalez-Rodriguez J. validated explosive samples taken using the Ionscan® swabs and run on an Ionscan® system via atmospheric pressure chemical ionization (APCI) in negative ion mode and an LC-qToF mass spectrometer [483].

Kober, S., Hollert, H., and Frohme, M. used a matrix-assisted laser desorption/ionization time of flight mass spectrometry (MALDI-TOF MS) for quick analysis of TNT in soils [475].

McCulloch, R.D. and Amo-Gonzalez, M. reported picogram limits of detection for several explosives using field-free atmospheric pressure photoionization (APPI) combined with a thermal desorption introduction with differential mobility analysis (DMA) tandem mass spectrometry (MS/MS) [478].

Pavlov, J. et al. showed that 1,4-Benzoquinone is an efficient dopant for enhanced ionization and detection of nitramine explosives on a single quadrupole mass spectrometer fitted with a helium-plasma ionization source [481].

Field MS is always a bit tricky for the explosive analyst and mostly requires suitable ambient ionization and often requires operation with no power sources, gas supplies, and flow control or heating devices. Pintabona, L. et al. studied the use of surface acoustic wave nebulization (SAWN) with MS for ambient ionization suitable for field use and report “excellent sensitivity of nitrate-based organic explosives was observed for nitrate-based explosives when operating the MS in the negative mode” [482].

Organic high explosives are frequently either light molecules with high vapor pressures or heavier and nonvolatile. Bi, L et al. developed an “ultrasonic cutter blade coupled barrier discharge ionization” method to detect ultra trace levels of nonvolatile explosives among other nonvolatile compounds [452].

G) Isotope Ratio Mass Spectrometry, IRMS

Isotope ratioing is a technique that can be used to sort or cluster products, in this case explosives, in order to possibly source the origin of

that product. For it to be successful large databases are generally required. There have been successful uses of this technique in both intelligence applications and with agricultural products wherein certain countries are under embargos. It may be useful in forensics especially as an excluder of commonality of two items but, is of limited use in definitive inclusions without the aforementioned database for that product.

Comparison of the homemade explosive erythritol tetranitrate (ETN) with its precursors was explored in a paper published by Bezemer, K et al. Keying in on carbon and nitrogen they report that "... robust linear relationships between precursor and the end product were observed for these isotopes ... that support the hypothesis that a given erythritol or nitrate precursor was used to synthesis a specific ETN batch." [488].

C. Hu et al. propose a recrystallization technique to purify ammonium nitrate (AN) to achieve better results from real world AN comparisons. They found that without purification contributions from other materials mixed in with the AN can skew an isotope ratio analysis [489].

Kim, N.Y., et al. studied the propellants from nine different shotshells from several countries and clustered them by using IRMS. Adding organic component analysis as well, they were able to discriminate all nine [490]. Applying this in a post-blast or post-fired scenario might prove useful for sourcing to provide investigative leads although as always larger databases are needed.

H) FTIR

Fourier Transform Infrared Spectroscopy (FTIR) is a workhorse instrument in forensic explosives analysis. Some useful papers are included in the bibliography.

I) Raman Spectroscopy

Raman spectroscopy continues to be applied for explosives analysis in the lab as well as in the field for hazard assessment. There are numerous advantages to this technique, as we wrote in 2019, "it is fast, discriminatory, non-destructive and vetted for legal proceedings" [24]. Additionally, it does not require sample prep and is capable of sampling materials in situ, through containers, and at significant distances (stand-off).

Gulia, S. et al. reported on using spatially offset Raman spectroscopy (SORS) as a method to overcome the limitations of conventional Raman spectroscopy when applied to testing through colored glass, high density polyethylene (HDPE), Teflon etc. [512]. This will prove useful both in security settings and for examination of materials in searches.

Colob-Gonzalez, F.M., et al. described a method for detecting TNT, PETN, and RDX on a variety of hair types with non-invasive Raman spectroscopy. They found, not surprisingly, that gray hair was the best substrate for detection [505].

In a unique approach to extremely small samples Kotrly, M., and Turkova, I., used a stepped approach with SEM/FIB (focused ion beam) to get 3D reconstruction of the materials in post-blast samples, EDS/WDS for surface elemental mapping, and high-resolution Raman spectroscopy. By using this compliment of techniques, they could easily focus in on materials of interest and carry out complex analysis on microscopic particles [518]. In one regard this can be seen as a sampling technique. This combined instrument set-up could prove valuable for post-blast analysis where minimal residue is available.

J) DSC, Thermal Analysis, TG

This category is applicable to the examination of explosives and mixtures and can further characterize an unknown material. These techniques have been around for decades and while they have limited applications in the forensic lab setting, they are still useful, especially for evaluating new explosives and explosive formulations.

7. Nanotechnology

Nanotechnology can be used for the miniaturization of instrumentation which can be much more easily deployed for field analytical use. It also can significantly enhance detection of very small amounts of analytes. Also in this category is the production and manufacture of nanoexplosives which are more efficient and theoretically at least portend less environmental contamination due to the efficiency of the explosive.

Using Raman spectroscopy coupled with nanomotors Novotny, F., Plutnar, M., and Pumera, M., detected picric acid. Their synthesis of the nanomotors was done using the seeded growth wet chemical method as opposed to planar substrate methods, and they report that this method is highly scalable [551].

Bhatt, P.V., et al. also wrote about using nanotechnology as a means of tagging explosives as well as general advances in nanotechnology and a summation of the current trends [538].

8. General detection

A) Canine Explosives Detection

Canine explosive detection is both very effective, given the right training and reasoned deployment, and simultaneously the most frustrating for analysts who cannot replicate this efficiency and selectivity using man made instrumentation. Research in this area continues to try and shed light on the complexities of how canines process and detect target odors and the optimization of training aids and odor presentation. There are many considerations for training and maintaining an effective explosives detection canine team, some of which are discussed in the papers in this section.

Sacharczuk, M. et al. published a fascinating study on canine performance and selected canine olfactory receptor genes. They identified the ten canines with the best performance and the ten with the worst out of 91 drug detecting canines and 57 explosive detecting dogs. They then examined several genes in order to predict, before training, which canines would be best suited for detection work [566].

One potentially significant problem with canines is their receptiveness to handler cues. Lazarowski, L. et al. reported that responsiveness to human cues decreased with age but the ability to locate the reward increased and "Furthermore, a lack of susceptibility to deceptive social cues was predictive of future success as a detection dog" [564].

DeGreeff, L. and Peranich, K. published an article on using a Mixed Odor Delivery Device (MODD) to increase proficiency in canines for the detection of mixtures that contain a target odor. The paper finds that canines exposed to mixtures in training had more success detecting odors in a mixture than those that had not previously trained on mixtures. The odorants were verified using SPME-GC/MS for quality control [559].

Gazit, I. et al. designed an experiment to test a canine's ability to correctly alert on individual odors when previously exposed to a mixture of those odorants. They found dogs can indeed quickly recognize individual components [563].

Simon, A.G., et al. described using DART-MS to monitor in real time TATP released from a polydimethylsiloxane canine training aid and compared it to other training aids for the same analyte. They report this training aid style had similar odor output to a traditional style container with holes in the lid. The authors also used DART to assess real time odor availability from both styles of training aids in various search configurations [568].

B) LIBS Detection

Romolo, F.S. and Palucci, A. have published a review chapter on the advances with detection techniques for security purposes in the areas of laser induced breakdown spectroscopy (Stand-off) or LIBS, Raman (stand-off), surface enhanced Raman spectroscopy (SERS) and laser

photoacoustic spectroscopy They discuss the detection limits and the drawbacks of these techniques and discuss how they can be used to advance an investigation. One such example was helping to reduce the number of samples sent to the lab [577].

C) Neutron

Seman, J., Giraldo, C.H.C., and Johnson, C.E. reported on a proposed nuclear barcode to be used as a chemical detection agent. Part of the coding employs a unique combination of taggant elements at different concentration levels that could be used to provide specific information about where and when the explosive was manufactured which could provide valuable investigative leads in criminal or terrorist bombings. The paper examines the stability of the ratios over time and found that the technique could work if the concentration levels were separated at least 100 ppb [583].

D) Terahertz

E) Nuclear Techniques

Yu, P., et al. monitored the droplet size distribution in ANFO emulsion explosives at different temperature and storage conditions to assess any impact of the storage condition on the stability of the emulsion. They used Nuclear Magnetic Resonance (NMR) Pulsed Field Gradient (PFG) and found storage at 50° Celsius for 12 weeks lead to a 60% increase in mean droplet size. Adding 5% calcium nitrate to the aqueous phase suppressed this thermal degradation [602].

F) X-Ray

G) Ion Mobility Spectrometry

While Ion Mobility Spectrometry is often used in security screening settings because of its rapid sampling times and relative accuracy, it often has not been used often in forensic laboratories because of its limited ability to separate analytes, its propensity for being overloaded quickly requiring a period of cleaning of the system, and its potential for false positives. There have been advances made to allow for limited separations and quicker recovery times improving the usefulness of this technology.

Hauck B., Harden C., and McHugh V. evaluated five possible calibrants for an ion mobility time of flight instrument. They reported that di(propylene glycol) methyl ether (DPM) was the most useful and recommended against 2,4-pentanedione (PDO) and methyl salicylate (MES) [610].

Thangadurai S. and Gurusamy K. advocated for use of IMS in post-blast debris samples and reported that several explosives were detected in various matrices. They discuss sample preparation and screening and highlight the advantages and limitations of the technique for the detection of explosives [616].

Chilluwal, U. published a dissertation arguing for tandem IMS using chlorine adducts of RDX, PETN and NG for the analysis. By using tandem IMS there was a reported elimination of the false positives often encountered with IMS results due to interferences [608].

In a similar fashion, Mullen, M., and Giordano, B. used the combined technique of secondary electrospray and corona discharge ionization (SECDI) to achieve more sensitivity for TNT and 2,6-DNT [612]. These approaches that eliminate the problems inherent in IMS may prove valuable as a very rapid analytical tool.

The drift tube design for IMS was enhanced in a paper published by Smith, B. et al. They designed a flexible drift tube and report “the DT (drift tube) is constructed from a flexible printed circuit board (PCB) with a bespoke ‘dog-leg’ track design, that can be rolled up for ease of assembly.” This design was reported to have a low cost and low footprint with limits of detection in the low nanograms [615].

Fisher, D., et al. proposed applying machine learning to IMS for a preliminary study using five nitrate explosives, AN, ANFO, Urea Nitrate,

environmental pollution nitrates and blanks. Their supposition is that machine learning can enhance selectivity for actual threats [609].

Bohnhorst, A., et al. wrote about their new approach to enhancing separation by increasing the resolving power of the drift time IMS without employing higher drift voltages. Instead, they employed a moving field IMS (MOF-IMS) to use the available voltage to a smaller segmented drift region [607].

H) Novel Detection

Novel detection is a catchall category for detection that does not fall neatly into another category. It could be completely novel or it could be something based on other techniques but different enough to be included in this section of the paper or bibliography.

Filipi, J., et al. described the use of honeybees as a tool that could be used in humanitarian demining efforts. The bees can be used in passive searches to detect the present of landmines in an area or active searches of pinpointing locations. The system relies on the detection of explosives residue on the body of honey bees that were foraging [638].

Simic, M. et al. described a similar use of honeybees. As a passive system honeybees can “electrostatically collect particles from the air in the flying and foraging areas, which in conjunction with organic-based explosive vapor sensing films, placed at the entrance of a beehive, can be used as a passive explosive sensing mechanism.” Furthermore, honeybees can be trained to react to certain explosives [685].

In a similar vein of using animals to detect explosives, Alsaleh, S., Barron, L., and Sturzenbaum, S. published a paper on the use of the invertebrate nematode *Caenorhabditis elegans* (worm) for the detection of ground soil perchlorate [620].

Liu, C. et al. integrated a smart phone with a urea-functionalized polyionic liquid photonic spheres to construct a colorimetric sensor array that can detect and identify five nitroaromatic explosives [660].

Lefferts, M.J., et al. used “ANFO chemiresistive vapour sensors based on polypyrrole (PPy) percolation networks to enhance” GC-MS results and reported improved sensitivity over thin film and detected “13–180 ppb of ammonia emitted by a variety of different ammonium nitrate ...” mixtures [656].

Blanco, S., et al. proposed wetting TATP as they form TATP-water adducts which could then be detected by microwave spectroscopy [625].

I) Stand Off

9. Environmental

As we wrote in 2019, Environmental scientists and chemists have long sought to test and eventually remediate explosives in environmental samples. Some of these methods can be directly borrowed from this field for use in forensic laboratories. Still other research, such as degradation studies, may assist the analyst in background knowledge of the explosive in certain matrices, especially soils [24].

Nawala, J. et al. used GC-MS/MS, LC-HRMS and NMR to examine explosives excavated from the Baltic Sea. There are tons of unexploded ordnance on the sea bed, so classifying it is important. They found that most samples indicated that it was a TNT, RDX, aluminum mixture known as “torpex” [770].

Rindelaub, J.D., et al. used XRF to measure particulate matter from fireworks usage in New Zealand to postulate source material (e.g. Cl from perchlorate oxidizers) and compared air quality vis-à-vis standard environmental exposure recommendations [776].

10. Other (safety, definitions, etc.)

While this article could be placed in the review category, we have included it here. Richard Crocombe has done an extensive review of portable spectroscopy including a discussion of the future of the

technology. He looked at the deployment numbers and usage of portable IMS, XRF, FTIR and Raman instruments, most of which are deployed in security operations or with fire or police hazmat responders [823].

da Silva, L., et al. published an interesting paper on pipe bomb fragmentation with varying case (wall) thickness. They used aluminized ammonium nitrate detonated with a PETN based detonator and a 5-g booter made of PETN and nitrocellulose and noted that the size and velocity of the fragments correlated to the pipe size [825]. It would be interesting, since the quality control on the pipes and test design was well thought out, to do similar tests with a suite of low explosives and not boosted or detonated.

Amaral, M., et al. studied transfer dynamics of ammonium nitrate showing transfers "... even when contact occurs for a short duration with a relatively low force" [796]. They report a variety of test conditions which may be useful in understanding transfer in the context of crime scenes or swabbing techniques. This reference could also be placed in the sampling category.

Collett, G. et al. tackled the predictive threat assessment process for Homemade Explosives (HME) precursors, specifically the regional distribution of hydrogen peroxide, ammonium nitrate and potassium chlorate [821].

Although there were many articles published on the Beirut Port ammonium nitrate explosion, Yu, G. et al. published perhaps the most comprehensive one. By looking at many post-blast factors, measurements, and reports, they calculated the TNT equivalent of the AN explosion to be approximately 950.3 tons and the fatality radius to be 487 m from the center. They also included an evaluation other safety related considerations relating to the accident and future ammonium nitrate storage considerations [954].

Oluwoye, I., et al. used XRD and IR to examine rust on iron-based containers to show oxygen deficient Fe_2O_3 clusters can serve as an agent to reduce ignition temperatures with ammonium nitrate reactions. They report "The dihydroxylation of hydrated iron (III) oxide, present on the surfaces of rust, exposes the Fe sites that react exothermically with AN, before the material assumes the ordered Fe_2O_3 phase." It may be prudent to monitor AN storage conditions in steel storage containers, especially older ones. AN is a strong oxidizer and does not require a high percentage of fuel to react or even detonate [898].

Rettinger, R., et al. published an article on lab scale (2 g) and field scale (5 kg) testing for combinations of several oxidizers (potassium nitrate, potassium chlorate, potassium permanganate, potassium iodate, ammonium nitrate and ammonium perchlorate) mixed with sucrose and aluminum to compare explosive potentials of each [910].

Tin, D., Margus, C., and Ciottone, G., did a comprehensive review of all terrorist events for the last 50 years, including ones deploying explosives. They report 48.78% of these 168,003 events used explosives since 1970 [934]. This paper is a fitting note to end on, highlighting the continued need for research and development to combat the threat of explosives.

Final notes

We have tried to review as much literature as possible these last few years and have written here about dozens of publications. There are indeed hundreds more citations listed in the bibliography, many of which may have some useful information for your forensic laboratory and explosive analysts.

Acknowledgements

The authors would like to express our deepest gratitude to Ms. Logan Tapscott, Librarian at the Bureau of Alcohol, Tobacco, Firearms and Explosives Laboratory. Additionally, the tireless work of the staff of the Arson and Explosives Sections at the ATF Forensic Science Laboratory, Washington, especially Malinda Durand and Delonn Ng have been invaluable.

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Sampling and Concentration of Explosive Traces

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