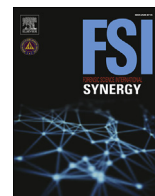


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Forensic Science International: Synergy

journal homepage: <https://www.journals.elsevier.com/forensic-science-international-synergy/>Interpol review of detection and characterization of explosives and explosives residues 2016–2019[☆]Douglas J. Klapec^{*}, Greg Czarnopys, Julie Pannuto

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ABSTRACT

This review paper covers the forensic-relevant literature for the analysis and detection of explosives and explosives residues from 2016–2019 as a part of the 19th Interpol International Forensic Science Managers Symposium. The review papers are also available at the Interpol website at: <https://www.interpol.int/Resources/Documents/Publications>

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1. Introduction and coverage of the literature

This review starts with a recommendation to read the previous three papers covering explosives analysis from 2007 to 2010 presented in 2010 by Richard Strobel and our previous reviews from 2013 to 2016 [13,14,26]. This review is less broad than the previous papers for several reasons including the filtering out of repetitive research. An example would be several papers on a single type of nanotechnology for a single analyte that already is already relatively easy to detect. That said, it is also highly recommended that practitioners in the field of forensic analysis and those on the cutting edge of developing new explosive security measures, peruse the references and determine what may be of use in future real life applications.

As Allied war efforts in regions that have seen many bombings have slowed even more in the past three years than in the last iteration of this topic, we have seen a decrease or flatlining in funding among some governments and even in private companies' research. However, civil wars are still ongoing in countries like Yemen, Syria, Iraq, Libya and Afghanistan, and major bombings are still prevalent, targeting combatants and civilians alike. Manchester, England was the site of a major terrorist bombing in May 2017, which killed 22 people. Large bombings are still prevalent in the Philippines, as one example, and in other nations not involved

in a civil war. Other modes of terrorism also have the attention of forensic practitioners and security experts. These include mass shootings and vehicular attacks, arson and knife attacks. However, the overall threat from explosives, especially in domestic settings, has remained important.

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 a 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 samples. 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.

As stated in our 2016 paper, "The forensic explosive analyst should regularly review literature in the wider scientific community with an emphasis on suitability for employing new techniques in the scheme of analysis. These include both applied and theoretical published research. It helps to get an early start in researching these techniques because of the increasingly stringent accrediting requirements for any new technique" [14]. We are hopeful this review

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will provide additional references and resources to kick start more applied research for the forensic practitioner.

There are increasing scientific and accrediting body requirements to make the transition from research to casework use in the forensic laboratory. More laboratories are requiring vigorous validation before putting any given method into use. As one example, although most forensic explosives chemistry protocols do not require quantitation (the verified detection of an analyte is normally enough to report it) and nor is the amount detected useful in most post-blast analysis, nevertheless, limits of detection may be required for full validation of a technique. There are several reasons for this but include monitoring any given system for performance over time. These issues should not dissuade analysts from attempting to introduce new or improved techniques into their schemes of analysis.

The review of the literature presented here shows that there are many applications in the wider world of explosives that could be of interest to the forensic explosive chemist. The authors, with help of a competent research librarian and hundreds of hours of reviewing abstracts by a team of forensic explosive chemists, have looked for anything to do with explosive manufacturing, theoretical and commercial, explosive detection using any technology, explosive performance and physics, sampling improvements, as well as new or improved analytical techniques for the identification of explosives. The field of explosives detection is still the fastest growing area from which forensics can draw. There are still dozens of references in this area, ranging from theoretical research to applied systems that are already in field use.

There are 1005 references in this review. Each reference has a hyperlink to the abstract or full text article where available. Additionally, the categories in the reference list can be easily accessed using the Navigation page in Microsoft Word. This will aid the navigation of the bibliography section, starting on page 26 of this document. 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 included several review publications. Some are broad schemes of analysis, while many are reviews of a specific class of instrumentation. Still 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. We will be dividing this section into forensic applications versus detection and security applications.

In the area of general overviews, Goodpaster reviewed the current status of explosives analysis from the forensic practitioner standpoint. He reviewed methods including types of spectroscopy, chromatography, and elemental analysis, as well as mass spectrometry [9].

Similarly, Brown et al., in a two part discussion, reviewed the current state of explosives detection. They "... review and critically evaluate the latest (the past five years) important advances in explosives detection, with details of the improvements over previous methods, and suggest possible avenues towards further advances in, e.g., stand-off distance, detection limit, selectivity, and penetration through camouflage or packaging. The review consists of two parts. This part, Part I, reviewed methods based on animals, chemicals (including colorimetry, molecularly imprinted polymers, electrochemistry, and immunochemistry), ions (both ion-mobility spectrometry and mass spectrometry), and mechanical devices" [2].

Peacock, P., et al., comprehensively reviewed the advances in ionization technology from January 2015 to September 2016 [21]. Their work should provide a guide for those working on new techniques to improve mass spectroscopy. Primarily focused towards researchers, the newer ionization techniques here in this paper could be seen commercially or even used directly by enterprising forensic chemists.

Gooch, J., et al. have an interesting review on the use of unique taggants that could be used in countries where taggants are mandated or even by companies interested in tagging their products. With nanotechnologies advancing at an increased pace, "... continuing advances in portable in-field analysis, nanotechnology and material science should have allowed for the development of new and improved forensic marking agents. However, the limited amount of recent research in this area suggests that this is not the case" [8].

Saini, R., has an excellent primer on the latest technologies being investigated for explosives detection [24].

Forbes, T. and Sisco, E. looked at recent advances in ambient mass spectrometry of trace explosives. They write, "These techniques have enabled real-time detection of target analytes in an open environment with no sample preparation and can be coupled to any mass analyzer with an atmospheric pressure interface" [6]. Mostly applicable to security purposes, these are also finding their way into the forensic analysis environment.

Although this paper could be placed in nanotechnology or even novel explosives, Go, B., Qiao, Z., & Yang, G. reviewed the rapidly growing interest in nano-explosives, dividing them into nano-individual explosives, nanocomposites, and nano-cocrystals [7].

Huri, M., Ahmad, U., Ibrahim, R., and Omar, M. presented a nice comprehensive overview of three aspects of explosive residue detection: screening techniques, extraction techniques, and instrumental techniques. Extraction methods include swabbing techniques, solid phase extractions, and solid phase microextractions. Additionally, "Instrumental techniques covered in this review included gas chromatography, high performance liquid chromatography, ion chromatography and capillary electrophoresis" [10].

de Araujo, W.R., et al., presented a review of portable on-site instrumentation and methods to include explosives. They review "A wide range of approaches including electrochemical sensors, microchip electrophoresis, ambient ionization on portable mass spectrometers, handheld Raman and NIR instruments as well as and point-of-need devices, like paper-based platforms" [5].

Zhang, W., et al. reviewed recent developments in spectroscopic techniques for trace explosives detection in the field using terahertz (THz) spectroscopy; laser-induced breakdown spectroscopy (LIBS), Raman spectroscopy; and ion mobility spectrometry (IMS) [32].

3. Explosive standards and references, laboratory quality control, contamination prevention

Lees, H., Zapata, F., Vaheer, M., and Garcia-Ruiz, C. looked at the transfer of nine different explosive residues (ANFO, dynamite, black powder, TNT, HMTD, PETN, NH_4NO_3 , KNO_3 , NaClO_3) to evaluate cross-contamination through fingerprint transfers and other modalities encountered at busy security checkpoints. Some results included, "... that transfer of explosive residues frequently occurred with certain differences among materials. Generally, the amount of explosive particles adhered to the finger decreased in the following order: skin > latex > nitrile, while the transfer of particles from the finger to another surface was the opposite. The adhesion of explosive residues from polycarbonate to the finger was found to be better compared to cotton, while the amount of particles transferred to cotton was higher" [34].

Pawłowski, W., Matyjasek, Ł., Cieślak, K. and Karpińska, M. studied contamination in the laboratory with some common explosives, looking at what stage of an analytical procedure would most likely result in contamination and with what type of explosive. The results are surprising given the static adhesion energy of PETN is well known and that NG, although volatile, can re-absorb on any number of substrates [36].

4. Sampling and concentration of explosive traces

Sampling and concentration of explosives is an important step in explosives analysis and detection. In many post-blast samples, the analytes are present in extremely low quantities or part of difficult matrices, or both. This aspect of explosive analysis is ripe for exploration and research.

A validated solid phase extraction cleanup procedure with Bond Elut NEXUS co-polymeric cartridges was used for soil and swab samples containing pre- and post-blast residues of nitro-organic explosives and reported by Thomas, J., Donnelly, C., Lloyd, E., Mothershead, R., Miller, J., McCollam, D. and Miller, M [69]. They report “The expected explosives were detected in 97% of cases after processing through SPE and analysis by GC/ECD.” And “The results from these matrices were compared to results obtained by syringe filtration. SPE produced equal or better results than syringe filtration in both the ECD screening and MS confirmation tests ...” They report the successful application of the cleanup of organic explosive residues in complex matrices. This was also reported in a separate journal [68].

Chouyyok, W., et al., “... compared the analyte-release performance of standard muslin sampling swipes to that of rationally assembled fiberglass cloth, and used thermal-desorption ion mobility spectroscopy for detection. The fiberglass cloth was chemically modified by covalently bonding phenyl-functional groups to the surface. The rationally assembled sampling materials provide significantly performance improvements over standard muslin sampling materials for detection of TNT, NG, RDX, TATP, and PETN.” For example, phenyl-functionalized fiberglass resulted in over 10 times greater TNT release, compared to muslin cloth, as well as improved response and repeatability after multiple uses of the same swipe [44].

Laster, J. presented novel sampling swabs for the collection of trace explosive residues. Microstructured polypyrrole (PPy) films displayed enhanced particle removal abilities compared to PPy non-structured and current commercial films for IMS detection [54].

Temple, T., Goodwin, C., Ladyman, M., Mai, N., and Coulon, F. reported “Optimised accelerated solvent extraction of Hexahydro-1,3,5-Trinitro-1,3,5 Triazine (RDX) from polymer bonded explosives” [67].

Daaid, N., Holly, A., and Beardah, M. reported that a 2007 European Network of Forensic Science Institutes (ENFSI) Expert Working Group proficiency test with TNT spiked swabs revealed that some laboratories did not detect the analyte. This paper reports on loss of TNT over time and various environmental conditions. “Overall, the cotton swabs stored at room temperature and exposed to daylight showed a very rapid loss of TNT over time, whereas cotton swabs stored in the freezer, and all simulated swab extracts, gave high recoveries over time” [46].

Bors, D., and Goodpaster, J. mapped smokeless powder residues using total vaporisation solid phase microextraction gas chromatography mass spectrometry (TV-SPME/GC/MS) to quantify residues of double-base smokeless powder (nitroglycerin (NG), diphenylamine (DPA), and ethyl centralite (EC)) on post-blast PVC pipe bomb fragments. They report “The analytical method could separate the three constituents in under 5 min with a detection

limit under 1 ppb, which demonstrates high throughput while maintaining high sensitivity. The method was optimised for nitroglycerin, as it is the most indicative of DBSP (double base smokeless powder)” [41].

Abdul-Karim, N., et al., looked at post blast particle morphology in an attempt to aid in collection and recovery. Particles were collected from the detonations of aluminized ammonium nitrate and RDX-based explosive utilizing SEM stubs. They report “Spheroidal particles (10–210 μm) with microsurface features recovered following inorganic charge detonations were dissimilar to the irregularly shaped particles (5–100 μm) recovered following organic charge firings” [38].

Zapata, F., and García-Ruiz, C. used “a wide variety of materials such as glass, steel, plywood, plastic bag, brick, cardboard or cotton subjected to open-air explosions were examined using confocal Raman microscopy, aiming to detect the inorganic oxidizing salts contained in explosives as black powder, chloratite, dynamite, ammonium nitrate fuel oil and ammonal. Post-blast residues were detected through microscopic examination of materials surfaces. In general, the more homogeneous and smoother the surface was, the less difficulties and better results in terms of identification were obtained” [72].

Fisher, D., Zach, R., Matana, Y., Elia, P., Shustack, S., Sharon, Y., et al. examined what types of swabs are best suited for recovery of explosives in the oft-used IMS detection setting. They report, “The adhesion of explosive particles to three typical materials, plastic, metal and glass, were measured using atomic force microscopy (AFM). We found that a strong contribution of capillary forces to adhesion on glass and metal surfaces renders these substrates more promising materials upon which to find and collect explosive residues” [48].

Taudte, R., Roux, C. and Beavis, A. investigated the degradation of compounds from smokeless powders and report that “energetic compounds were generally found to be more stable than smokeless powder additives such as stabilisers including diphenylamine and ethyl centralite, which might be problematic considering that these compounds are common targets for OGSR (*organic gunshot residues*)” [66].

5. Identification of explosives, explosive residues and explosive properties

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

5.1. Commercial explosives

Elbasuney, S., Fahd, A., Mostafa, H., Mostafa, S. and Sadek, R. reported on modified double base propellants with additions of oxidizer-metal fuel (Ammonium perchlorate/Aluminum), and energetic nitramines. The study evaluates the impact of these energetic additives on thermal behavior, chemical stability, and shelf life [83].

Dennis, D., Williams, M., & Sigman, M. utilized “a Bayesian network for inference of the powder manufacturer.” They looked at chemical characteristics of 169 smokeless powders using the most intense ions in their total ion spectra from gas chromatography-electron ionization-mass spectrometry and physical characteristics such as diameter and length, shape, color, luster, bias cut and whether the particles were perforated. The sensitivity and specificity of the fully instantiated network was examined for each manufacturer. They reported, “The PPV ranged from 0.59 to 0.81 for

individual manufacturers when all nodes of the network were instantiated. The NPV for fully instantiated networks ranged from 0.82 to 0.99 for individual manufacturers” [81].

Dennis, D., Williams, M., and Sigman, M. used “Gas chromatography–electron ionization–mass spectrometry (GC–EI–MS) and physical characteristics data for 726 smokeless reloading powders were analyzed by pairwise comparisons of samples comprising the same product and different products.” They looked at 13 organic components/constituents of smokeless powders. Interestingly they reported, “In the discrete and continuous data comparisons, the likelihood ratios for probabilities conditioned on same shape, color, presence/absence of perforation and size were found to provide relatively limited support for either the proposition of same product or different product” [80].

Xu, C., An, C., Li, Q., Xu, S., Wang, S., Guo, H., and Wang, J. have a unique and timely paper on using direct ink writing (DIW) to manufacture pentaerythrite tetranitrate-based composites. The energetic materials were produced using DIW, and “scanning electron microscopy, energy-dispersive x-ray spectroscopy, X-ray diffraction, differential scanning calorimetry, and nanoindentation were used to characterize the printed samples” [111].

While this could be included in the safety section of this paper, Xu, S., Tan, L., Liu, J., Chen, X., Jiang, W., Chen, Y., et al., investigated an accidental event with emulsion explosives and concluded, “The investigation of the accident showed that the reaction between crystalline sodium nitrite and ammonium nitrate (AN) was likely the cause of the spontaneous burning” [112].

5.2. Military explosives

Mao, X., Jiang, L., Zhu, C., and Wang, X. looked at the “Effects of aluminum powder on ignition performance of RDX, HMX, and CL-20 explosives” in *Advances in Materials Science and Engineering*. They showed, interestingly that, “... the energy release of the HMX/Al composite explosive with 10 wt%, 20 wt%, and 30 wt% aluminum powder was only equivalent to 80%, 65%, and 36% of pure HMX, respectively. It was similar to RDX/Al and CL-20/Al composite explosives, except the CL-20/Al mixture with 10% aluminum powder.” Aluminum does not seem to play much of a role except at ignition [131].

5.3. Homemade explosives

The area of Homemade Explosives (HME) is still of tremendous interest to the forensic explosives analyst. 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. Such cases are more likely to include improvised black powder or flash powder than processes such as the nitration of toluene.

The actual usage of HME is constantly changing and it is difficult for forensic laboratories to have adequate protocols for every possibility. Some HMEs or components therein are difficult to detect post-blast unless samples are taken immediately, stored properly, and analyzed quickly. In other instances, a component of the HME might be present in the environment of the explosion (say gasoline used as a fuel in an AN-gasoline mixture used in a car bomb).

In the area of primarily low explosives, many of which can be improvised, Conkling, J.A. & Mocella, C. have published the 3rd edition of *Chemistry of Pyrotechnics: Basic Principles and Theory*. This book is an excellent primer for the forensic analyst wishing to understand behavior of pyrotechnic mixtures and of low explosives, and for understanding the area of Homemade Explosives [149].

DeGreeff, L. and Johnson, K. looked at how vapor detection of Homemade Explosives differs from traditional explosive vapor detection. Specifically, they looked at ammonium nitrate mixtures and organic peroxides [150].

Härtel, M., Klapötke, T., Stiasny, B., and Stierstorfer, J. re-examined the gas phase concentration parameters of the explosives triacetone triperoxide (TATP) and diacetone diperoxide (DADP) [154].

Fraga, C., Mitroshkov, A., Mirjankar, N., Dockendorff, B., and Melville, A. presented a study titled *Elemental source attribution signatures for calcium ammonium nitrate (CAN) fertilizers used in homemade explosives*. They used inductively coupled plasma-mass spectrometry (ICP-MS) to “determine the concentrations of 64 elements in 125 samples from 11 CAN stocks from 6 different CAN factories.” They looked at the elements Na, V, Mn, Cu, Ga, Sr, Ba and U. Partial least squares discriminant analysis was then used to develop a classification model. They report that “for pristine CAN samples, i.e., unadulterated prills, 73% of the test samples were matched to their correct factory group with the remaining 27% undetermined using strict classification.” They then used various fuels in mixtures and still found similar but not the exact results. This is a promising approach to discriminate among CAN samples, especially in those areas where terrorists are frequently using unadulterated CAN as the oxidizer [153].

Newsome, G., Steinkamp, F., and Giordano, B. reported on analyzing bulk ammonium nitrate by using ambient ionization mass spectrometry and a tungsten oxide layer, which absorbs both species and thermally desorbs NH₃ and NO₂. They report that “ammonia was detected successfully, but the pre-concentrator reduced nitric acid to compounds smaller than NO₂, including N₂, that could not be detected apart from background” [159].

Kotrly, M., Turková, I., Beroun, I., and Mares, B. presented Methods for characterization of home-made and non-standard explosives in forensic science which is basically a working scheme of analysis for Homemade Explosives. A later presentation will be explored next but here they present the types of techniques they used and include GC-MSD, GC-ECD, EDS, and imaging by SEM. It is well worth the time to read this [156].

Kotrly, M., Wolker, J., Turkoba, I., and Beroun, I. presented a the first version of an HME database based on a two year running project to “... prepare some of these substances and carry out experimental explosions and tests, and map analyses possibilities using a wide range of available analytical techniques in forensic labs. Samples of primary substances, prepared explosives and post-blast residues are analyzed in a complex way in terms of organic and inorganic components. All data obtained, including visual documentation, are stored in a specialized database for security forces and their expert workplaces.” Again, it is another resource for laboratories attempting to analyze HME [157].

Bannister, W. and Oxley, J. reported on detection issues when dealing with non-nitrogen based explosives. These “include peroxides (used in both monergolic and hypergolic applications); acetylene precursors; and fuel/air bomb systems involving use of olefin oxides, acetylene, other hydrocarbons, and similar high energy agents.” They additionally look at precursors and preparation of these energetic materials. Next they deal with numerous composite explosives in the form of intimate mixtures of fuels and oxidizers such as those that use perchlorate, chlorate or hypochlorite salts as oxidizers. Finally and interestingly they discuss “... self-igniting systems such as boranes, phosphorus and alkali metals” [147].

DeGreeff, L., Cerreta, M., and Katilie, C. looked at degradation products of HMTD for detection and noted variances in detection based upon synthesis method, precursors, storage time, and storage environment. The composition and quantity of these volatiles

were compared across these variables. They did this through headspace analysis of bulk HMTD samples and used solid phase microextraction (SPME) with gas chromatography/mass spectrometry (GC/MS). They also monitored decomposition of HMTD by gravimetric analysis. Two results reported were "... that formic acid is the most abundant decomposition product while formaldehyde is the most commonly detected across all variables" [151].

In a similar report, Steinkamp, F., DeGreeff, L., Collins, G., and Rose-Pehrsson, S. completed a kinetic study of HMTD decomposition in solution (water). They also report a "correlation between degradation rate and the presence of decomposition species identified in the headspace ..." [164].

One interesting paper by Vodochodský, O., Jalový, Z., Matyáš, R. and Novotná, M. reported on using FTIR to do quantitative analysis of triacetone triperoxide (TATP) and hexamethylene triperoxide diamine (HMTD) on different substrates. They tried polymer, plastic, and cellulose matrices. Reporting (in the abstract): "It is based on dissolving, or extraction of, peroxide in the solvent and measurement in cuvettes using the Fourier transform infrared technique. These methods may be useful in analytical techniques of explosive detection and determination" [165].

Lease, N., Kay, L., Chavez, D., Robbins, D. and Manner, V. reported that molten ETN is more sensitive than cast ETN [158].

5.4. Other explosives including novel or new explosives

It was stated in our 2016 paper and still is true now, "Two types of advances in the production of novel explosives are reported here. As in the last review, there are many nanoparticle investigations. Additionally, the need for stability in harsh environments and a push toward environmentally friendlier explosives drive development of new military explosives. Also included are some recently declassified materials" [14].

While this could be seen as an improvement for a well-established technology for commercial explosives, we have included it here under novel explosives. Wang, Y., Ma, H., Shen, Z., Wang, B., Xue, B., & Ren, L. examined the detonation characteristics of emulsion explosives sensitized by hydrogen-storage glass microballoons instead of neutral or air filled microspheres. They reported, "Brisance testing and underwater explosion experiments showed that, compared with traditional emulsion explosives, the shock impulse and specific total energy of hydrogen-storage glass microballoons sensitized emulsion explosives are improved significantly. The brisance (compression of lead block) of hydrogen-storage emulsion explosives is 23.0 mm, 3.2 mm more than that of traditional emulsion explosives." It is unknown if this is a feasible alternative for traditional glass or polymer microballoon for commercial production due to the increased danger in working with hydrogen filled microballoons [244].

Singh, A., Soni, P., Sarkar, C. and Mukherjee, N. discussed reactivity of aluminized polymer-bonded explosives with non-isothermal thermogravimetry and calorimetry. They reported, "Results revealed that the thermal decomposition behavior has been significantly influenced in the presence of Al and HTPB matrix, especially reducing the thermal stability than that of neat HMX" [228].

Abd-Elghany, M., Klapotke, T., and Elbeih, A. studied a new green propellant formulation of a chlorine-free high energy dense oxidizer (HEDO) 2,2,2-trinitroethyl-formate (TNEF) and hydroxyl-terminated polybutadiene (HTPB) as a binder. They characterized the new oxidizer TNEF by nuclear magnetic resonance (NMR) and FTIR and scanning electron microscopy (SEM). They reported, "The results proved that the new oxidizer and its formulation based on HTPB have chlorine-free decomposition products and have higher performance characteristics than the traditional propellants" [169].

In a very interesting article, Gottfried, J., Smith, D., Wu, C. and Pantoya, M. explored coating aluminum particles with aluminum iodate hexahydrate (AIH) to replace the Al_2O_3 layer on Al particles that limits Al oxidation. They stated, "Estimates of the detonation velocity for TNT-AIH composites suggest an enhancement of up to 30% may be achievable over pure TNT detonation velocities. Replacement of Al_2O_3 with AIH allows Al to react on similar time-scales as detonation waves." Again, it is unknown if this could be used on an industrial scale [189].

6. Instrumental analysis of explosives

6.1. LC/HPLC/UPLC

Forensic explosive examiners utilize dozens of instrumental techniques to identify trace amounts of explosives. 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.

Şener, H., Anilnert, B., and Cengiz, S. presented a paper on one of the most popular and newer techniques for ionization with LC systems, that of atmospheric pressure chemical ionization mass spectrometry (LC-APCI-MS/MS). In this presentation they used a fast screening method and examined trace amounts of TNT (trinitrotoluene), RDX (1,3,5-trinitroperhydro-1,3,5-triazine), HMX (cyclotetramethylene-tetranitramine), PETN (pentaerythritol-tetranitrate), tetryl (2,4,6-trinitrophenyl-N-methylnitramine), picric acid (2,4,6 trinitrophenol), 2,6-DNT (2,6-dinitrotoluene), and TMETN (trimethylolethane-trinitrate). They used "a gradient of 2.00 mM ammonium nitrate aqueous solution-methanol mobile system, C18 column, and atmospheric pressure chemical ionization (APCI) (-) ionization mode was used after a single-step solid-liquid extraction procedure from soil matrix." And reported "Limit of detection (LOD) and limit of quantification (LOQ) values obtained from the analysis of the soil samples including explosive mix were between 8.9–161.2 and 13.2–241.5 ngg⁻¹, respectively" [266].

Similarly, using tandem mass spectrometry, Avci, G., Anilnert, B. and Cengiz, S. proposed "A fast and a selective determination method with high recovery was developed for the common explosives 2,4,6-trinitrotoluene (TNT), 3,5-trinitro-1,3,5-triazacyclohexane (RDX), and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) in soil ..." [263].

6.2. Ion chromatography

The technique of ion chromatography (IC) is used in forensic post-blast analysis for the analysis of mostly inorganic but also some organic explosives. The mass spectrometer is the detector of choice even for simple ions, but other detectors are still used as well. Ion chromatography has the advantage over other inorganic characterization methods such as X-ray diffraction and SEM/EDS in that physical particle recovery is not required and, perhaps more importantly, a relative profile of all anions or cations in a sample can be ascertained and judged against known post-blast or post-combustion profiles.

Often, the anionic profile of post-blast residues proves to be most probative. However, in many laboratories, the authors' laboratories included, thiocyanate and perchlorate anions are examined with a separate method from the other typical anions found in post-blast inorganic samples. Here, Gan, Z., Liu, J. and Tang, S. presented a novel method for the simultaneous determination of nine anions (Cl^- , NO_2^- , ClO_3^- , NO_3^- , CO_3^{2-} , SO_4^{2-} , $S_2O_3^{2-}$, SCN^- and

ClO₄⁻) in explosive residues by ion chromatography using a high capacity anion-exchange IonPac AS20 column (250 mm × 4 mm) [267].

6.3. Gas chromatography

As an alternative to, or as an orthogonal technique, for traditional IC detection and identification of anions, Pagliano, E., Campanella, B., D'Ulivo, A. and Mester, Z. reviewed gas chromatography (GC) methods for the determination of inorganic anions after derivatization. The review explores many inorganic anions and their derivatives (already published). They stated, "In this review, most derivatization chemistries employed for anions are discussed with attention to molecular aspects of the conversion, experimental conditions, applications to complex sample matrices, and figure of merits" [275]. It seems useful to have this review available for those who do inorganic ion identifications in post-blast explosives analysis.

Marder, D., Tzanani, N., Prihed, H. and Gura, S. used a splitless programmed temperature vaporizing (PTV)-large volume injection (LVI)-GC-MS-negative chemical ionization (NCI). They improve traditional LVI and the issue of having trouble detecting too many analytes in one sample by having "a unique double-column configuration setup developed for the efficient removal of excess solvent through a flame detector before reaching the MS, with the precise timing of carrier-gas flows and the heating program" [274].

For a method to possibly source plastic explosives, Tsai, C., Milam, S. and Tipple, C. used a comprehensive two dimensional gas chromatography-mass spectrometer (GC × GC-MS) with a statistical approach. The MS was a Time of Flight detector and principal component analysis was done. They report, "This demonstrates accurate classification of PE samples into production lots using these data treatment steps" [277].

Chajstamatious, A. and Bakeas, E. presented unique research into analyzing nitrocellulose (NC) by Gas Chromatography-Electron Ionization-Mass Spectrometry (GC-EI-MS). A rapid method for the identification of NC in bulk explosives using GC-EI-MS was developed. They write, "Results showed that NC was detected, by its trimethylsilyl (TMS) derivatives, in all the explosive mixtures analyzed and no false positives were observed" [270].

In a very important study, Sauzier, G., Bors, D., Ash, J., Goodpaster, J., and Lewis, S. attempt "... a central composite design was used to determine statistically validated optimum recovery parameters for double-base smokeless powder residues on steel, analyzed using total vaporisation (TV) SPME/GC-MS." Importantly, they reported that maximum recovery was by using "... isopropanol-wetted swabs stored under refrigerated conditions, then extracted for 15 min into acetone on the same day as sample collection." It will be interesting to see if this finding translates to other post blast explosives [276].

Katilie, C., Simon, A. and DeGreeff, L. reported an ammonia derivatization in the GC inlet with butyl chloroformate to produce butyl carbamate, a compound that can be used on GC and is compatible with standard GC-MS analysis. The inlet was also cooled. They state, "This method was then used to quantify the ammonia headspace vapor concentration produced from the dissociation of bulk ammonium nitrate as well as from mixtures with aluminum and petroleum jelly, which are fuels commonly used in homemade explosives (HMEs)" [272]. It is unknown how long ammonia stays in the environment in most post-blast scenes but this is interesting work.

Chajstamatiou, A. and Bakeas, E. derivatize thiocyanate and run GC-MS. They correctly assume that SCN is a product of black powder combustion and, while present in most BP post-combustion samples, there are indications this doesn't happen in

all post-combustion scenarios. They report, "In this study, a simple experimental protocol has been developed towards black powder residues identification, using GC-MS. Derivatization of thiocyanates coming from BP deflagration and identification of the relative derivative (PBF-SCN) was achieved by monitoring ions *m/z* 239, 181 and 161." In addition, they observed, "This protocol may be applied directly and without previous preparation to evidence coming from cases of explosions, thus practically contributing in BP residues identification" [269].

6.4. Capillary electrophoresis

Capillary electrophoresis (CE) is a powerful analytical technique for separating analytes. Coupled with mass spectrometry it can identify many species, organic or inorganic, of interest to the forensic explosives chemist.

6.5. 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.

Cruse, C. and Goodpaster, J. proposed coupling of a GC to vacuum ultraviolet (VUV) spectroscopy to possibly increase detection specificity. GC/VUV has already been used for "the analysis of volatile organic compounds, petroleum products, aroma compounds, pharmaceuticals, illegal drugs, and lipids." [297]. Here, they reported on the utility of GC/VUV for explosives analysis, and on thermal degradation within the VUV cell and whether it can be useful. They report, "The general figures of merit and performance of GC/VUV were evaluated with authentic standards of nitrate ester explosives (e.g., nitroglycerine (NG), ethylene glycol dinitrate (EGDN), pentaerythritol tetranitrate (PETN), and erythritol tetranitrate (ETN))" and that "the explosive analytes were thermally degraded in the VUV cell, yielding reproducible, complex and characteristic mixtures of gas phase products (e.g., nitric oxide, carbon monoxide, and formaldehyde)" [297].

Valdes, E. and Hoang, K. looked at the application of X-ray fluorescence spectroscopy (XRF) to analysis of potential explosives via the Primini X-ray fluorescence spectrometer (Rigaku Corporation; Tokyo). They looked at plastic explosives, ammonium nitrate, and calcium ammonium nitrate. XRF is an established technique for elemental analysis in forensic laboratories doing explosives analysis [364].

Pacheco-Londoño, L., Aparicio-Bolaño, J., Galán-Freyte, N., Román-Ospino, A., Ruiz-Caballero, J. and Hernandez, S. used classical Least Squares-Assisted MIR Laser Spectroscopy Detection of High Explosives on Fabrics [342].

6.6. Mass spectrometry

Mass spectrometry continues to be the widest used technique for forensic explosives analysis, especially for post-blast analysis, or for trace detection in security settings. It also is one of the most researched areas in explosives analysis. There are hundreds of applications for mass spectrometry. In many cases, a positive nearly unambiguous identification of an analyte can be achieved. In other cases, orthogonal methods must still be used. Any mass spectrometry technique that does not employ any chromatography or other mode of separation on the front end will almost always trade

some point of identification (i.e. retention time) for speed of use. That said, softer ionization or using chemical adducts can alleviate that potential problem, even for smaller thermally labile explosive compounds.

One of the most researched and promising areas of mass spectrometry for explosives analysis is that of direct analysis in real time mass spectrometry (DART-MS). Williamson, R., Gura, S., Tarifa, A. and Almirall, J. couple capillary microextraction of volatiles with DART for the trace detection and characterization of organic compounds found in smokeless powders and in organic gunshot residues. The analytes are those typically seen in the suite of chemicals in smokeless powders (nitroglycerin (NG), diphenylamine (DPA), ethyl centralite (EC), 2,4-dinitrotoluenes (2,4-DNT), methyl centralite (MC), 2,4,6-trinitrotoluene (2,4,6-TNT) and various derivatives of DPA) [426].

Correa, D., Melendez-Perez, J., Zacca, J., Borges, R., Schmidt, E., Eberlin, M., et al. used DART for looking for TATP on recovered bank notes from an ATM theft. They reported, “Easy ambient sonic-spray ionization mass spectrometry (EASI-MS) is shown to be a simple and selective screening tool to identify peroxide explosives on real banknotes collected from ATM explosion. Analyses were carried out directly on the banknotes surfaces without any sample preparation, identifying triacetone triperoxide (TATP) and diacetone diperoxide (DADP). EASI source coupled to a single quadrupole mass spectrometer provides an intelligent and simple way to identify the explosives TATP, DADP and its domestic synthesis markers” [396].

In another application of DART, Forbes, T., Sisco, E., & Staymates, M. coupled Infrared thermal desorption (IRTD) with (DART-MS) for “the detection of both inorganic and organic explosives from wipe collected samples.” The abstract reported, “IRTD-DART-MS demonstrated the thermal desorption and detection of refractory potassium chlorate and potassium perchlorate oxidizers, compounds difficult to desorb with traditional moderate-temperature resistance-based thermal desorbers. Nanogram to sub-nanogram sensitivities were established for analysis of a range of organic and inorganic oxidizer-based explosive compounds ...” and “The thermal desorption and ionization characteristics of the IRTD-DART technique resulted in optimal sensitivity for the formation of nitrate adducts with both organic and inorganic species” [402].

Forbes, T., Sisco, E., Staymates, M. and Gillen, G. reported on a mass spectrometry (MS) platform coupling resistive Joule heating thermal desorption (JHTD) and direct analysis in real time (DART) for the analysis of inorganic nitrite, nitrate, chlorate, and perchlorate oxidizers. They stated, “JHTD enhanced the utility and capabilities of traditional DART-MS for the trace detection of previously difficult to detect inorganic compounds” [403]. The use of DART-MS for inorganic compounds creates, at a minimum, an orthogonal technique to ion chromatography or capillary electrophoresis.

Also effectively using DART-MS with a unique sample introduction method, Li, F., Tice, J., Musselman, B., and Hall, A. “designed a qualitative analytical approach that utilizes novel sorbent-coated wire mesh and dynamic headspace concentration to permit the generation of information rich chemical attribute signatures (CAS) for trace energetic materials in smokeless powder with DART-MS. Sorbent-coated wire mesh improves the overall efficiency of capturing trace energetic materials in comparison to swabbing or vacuuming.” Constituents of smokeless powders, including nitroglycerin, “... were rapidly and efficiently captured by the Carbopack X wire mesh, followed by detection and identification using DART-MS.” This reduces the analysis time compared to traditional GC-MS approaches as all of the “components that can be detected by GC-MS, were detected by DART-MS in less than a minute” [411].

Bridoux, M., Schwarzenberg, A., Schramm, S., and Cole, R. have a unique approach on the use of Direct Analysis in Real Time (DARTTM) high-resolution OrbitrapTM mass spectrometry (HRMS) in

combination with Raman microscopy. They used this combination on actual explosives including plastic explosives, which have “complex matrix of binders, plasticizers, polymers, and other possible organic additives.” Swabbed particles were “characterized using micro-Raman spectroscopy followed by DART-HRMS providing fingerprint signatures of orthogonal nature.” And “When the polarity was switched to positive mode, DART-HRMS revealed a very complex distribution of polymeric binders (mainly polyethylene glycols and polypropylene glycols), plasticizers (e.g., dioctyl sebacate, tributyl phosphate) ...” [391].

Lising, A. completed a thesis where DART-MS was used on smokeless powder samples in potential matrices that may be encountered in real life samples. DART-MS has been reportedly successful in relatively clean matrices but here smokeless powder was mixed in with motor oil and tested. However, the author reported, “Effective separation was not achieved using the various LLE methods tested. Further testing would be required in order to evaluate the feasibility of implementing the technique as a sample preparation approach prior to analysis by DART-MS.” This is exactly the type of research and reporting that helps forensic laboratories evaluate whether a technique is feasible for (some) real world type samples [412].

Lennert, E. and Bridge, C. have two papers looking at DART-HRMS with smokeless powders [409].

Forbes, T. and Verkouteren, J. reported on the *Forensic Analysis and Differentiation of Black Powder and Black Powder Substitute Chemical Signatures by Infrared Thermal Desorption—DART-MS*. As reported in their abstract, “The trace detection and forensic analysis of black powders and black powder substitutes, directly from wipe-based sample collections, was demonstrated using infrared thermal desorption (IRTD) coupled with direct analysis in real time mass spectrometry (DART-MS)” [401].

Another area for practical scene application is the miniaturization of Mass Spectrometry. Hashimoto, Y. reported on recent developments in this area. He reported, “... on the recent results related to the detection of explosive materials where automated particle sampling using a cyclone concentrator permitted the inspection time to be successfully reduced to 3 s” [408].

One of the mass spectrometry methods and systems that have the most promise for explosives analysis application is the LC coupled with an exact mass detector. Here, Dunn, L., Obaidly, H., and Khalil, S. reported on two semi-quantitative, fast liquid chromatography-mass spectrometry methods. They use an atmospheric pressure chemical ionization source with an accurate mass detector (LC-APCI-QToF-MS) for the analysis of peroxide explosives, namely hexamethylene triperoxide diamine (HMTD) and triacetone triperoxide (TATP). They report, “The limits of detection (LOD) for HMTD and TATP using these methods were determined to be 0.5 ng and 10 ng on column, respectively. The high mass accuracy and narrow mass detection window offer high selectivity with <2 ppm mass difference between measured and calculated values for HMTD” [397].

Ewing, R., Valenzuela, B., Atkinson, D., and Freeburg, E. reported using a commercial mass spectrometer with an atmospheric flow tube (AFT) for inorganic oxidizers in homemade explosives at picogram levels. Specifically, they analyze the thermal desorption of nitrate, chlorate and perchlorate salts [398].

Reese, K., Jones, A. & Smith, R. have a paper titled, *Characterization of smokeless powders using multiplexed collision-induced dissociation mass spectrometry and chemometric procedures*. They compared unburned powders to corresponding fired residues and analyzed them by liquid chromatography-atmospheric pressure chemical ionization-time-of-flight mass spectrometry (LC-APCI-TOFMS). They report “Multivariate statistical procedures were performed to first investigate association and discrimination of the

unburned powders. Principal components analysis (PCA) of the chemical profiles suggested nine distinct groups of powders, according to the dominant organic compounds present. The clusters formed in hierarchical cluster analysis (HCA) were mostly in agreement with PCA groupings although HCA provided a metric to quantify the similarity.” They also caution, “... association of the fired residue (sic) to the corresponding unburned powder was possible although the success was highly dependent on the composition of the unburned powder and the extent of compound depletion as a result of firing” [421].

6.7. Isotope ratio mass spectroscopy, IRMS

Isotope ratio mass spectroscopy is an elusive but still promising technique to source discriminate almost anything. Well established in some drug and agricultural products analysis, it is still in a nascent stage when it comes to practical applications for explosives analysis. Often its utility is in intelligence gathering rather than being reliable for judicial proceedings.

Chesson, L., Howa, J., Lott, M. & Ehleringer, J looked at samples containing RDX, HMX, PETN, TNT, AN, and NC (nitrocellulose) and binders, plasticizers and additives to prepare these different explosive components for stable isotope analysis. They write, “This paper describes the theory and processes used to develop a component-specific approach to prepare explosives samples for isotope ratio analysis, focusing specifically on optimization of solvent extraction methods” [431].

One of the most popular and easily synthesized homemade explosives is TATP. Here, Howa, J., Barnette, J., Chesson, L., Lott, M. and Ehleringer, J. measured the carbon ($^{13}\text{C}/^{12}\text{C}$) and hydrogen ($2\text{H}/1\text{H}$) isotope ratios of the TATP, and one of its precursors, acetone. Acetone is the only source of carbon and hydrogen in TATP. They conducted a survey of acetone from 12 countries to see how much variation there was of $^{13}\text{C}/^{12}\text{C}$ and $2\text{H}/1\text{H}$. They reported, “We observed greater ranges in both C and H isotope ratios of acetone than previously published; we also found that country-of-purchase was a large contributing factor to the observed variation, larger than acetone grade and brand. Following clandestine production methods, we observed that the stable isotope ratios of TATP retained the stable isotope signatures of acetone used in synthesis” [433].

6.8. FTIR

Fourier Transform Infrared Spectroscopy (FTIR) is a workhorse instrument in forensic explosives analysis. Some useful papers are commented upon, below. Many commercial platforms and sampling devices are available [14].

Alvarez, A., Yanez, J., Contreras, D., Saavedra, R., Saez, P., & Amarasiriwardena, D. looked at four propellant brands and characterized them by Fourier-transform infrared photoacoustic spectroscopy (FTIR-PAS). As expected, “Spectra shows characteristic signals of typical compounds in the propellants, such as nitrocellulose, nitroglycerin, guanidine, diphenylamine, etc.” However, they then applied chemometric methods of classification, namely principal component analysis (PCA) and soft independent modeling of class analogy (SIMCA). They state, “Our results show the ability of FTIR-PAS combined with chemometric analysis to identify and differentiate propellant brands in different explosive formulations of IED” [436]. It is unclear if the sample set was vastly increased, whether this technique would work for discrimination but it would be a relatively quick way to do so.

6.9. Raman spectroscopy

Raman spectroscopy has seen increased usage not only on scene, but also in forensic explosive laboratories in the last ten years. It is fast, discriminatory, non-destructive and vetted for legal proceedings. There are still two basic types, stand-off or near stand-off detection, and targeted analysis, sometimes with portable handheld units.

Elbasuney, S., and El-Sherif, A. introduced a study on instant and standoff identification of concealed explosive-related compounds using a customized Raman technique. They reported, “Stokes Raman spectra of common explosive-related compounds were generated and spectrally resolved to create characteristic fingerprint spectra.” As expected they demonstrated “... that the two vibrational spectroscopic techniques were opposite and completing each other” [449].

Almeida, M., Logrado, L., Zacca, J., Correa, D., and Poppi, R. reported using “Raman hyperspectral imaging, in conjunction with independent component analysis” as a “methodology to detect an ammonium nitrate fuel oil (ANFO) explosive in banknotes after an ATM explosion experiment” [441].

Almaviva, S., Palucci, A., Botti, S., Puiu, A., and Ruffoloni, A. reported on using surface-enhanced Raman spectroscopy (SERS) measurements of common trace amounts of military explosives with a micro-Raman system integrated with a Serstech R785 miniaturized device, comprising a spectrometer and detector for near-infrared (NIR) laser excitation (785 nm). They report that “SERS spectra were obtained, exciting samples in picogram quantities on specific substrates ...” [440] *Italics added*.

Zapata, F. and Garcia-Ruiz, C. used vibrational spectroscopy, including both IR and Raman, to study some 72 nitrate, perchlorate and chlorate salts in a non-destructive, non-disassociated (like ion chromatography) manner. They tested whether every salt can be unequivocally identified by IR and Raman. They reported that, “Besides the visual spectra comparison by assigning every band with the corresponding molecular vibrational mode, a statistical analysis based on Pearson correlation was performed to ensure an objective identification, either using Raman, IR or both.” Also, that “Positively, 25 salts (out of 72) were unequivocally identified using Raman, 30 salts when using IR and 44 when combining both techniques” [479]. This is not surprising since many low molecular weight inorganic salts have spectra reflecting the anionic portion of the salt.

6.10. DSC, Thermal analysis, TG

Kohga, M. and Handa, S. analyzed the thermal decomposition behaviors and burning characteristics of propellants with ammonium perchlorate (AP)/ammonium nitrate (AN) particles. It is reported that these greatly depended on the AN content (χ) of the AP/AN sample [485].

7. Nanotechnology

As stated in our previous two reviews, “one of the most exciting aspects in explosives in the last decade has been the development of nanotechnology” [14]. Nanotechnology allows for the miniaturization of instrumentation allowing for very powerful portable analytical use. Another aspect of nanotechnology is the miniaturization of particles in explosives themselves.

Gao, B., Qiao, Z. and Yang, G. presented a review of nano-explosive materials since the 1990's. They write, “Nanotechnology has proved to be a remarkable and indispensable strategy to achieve high-performance nanomaterials for applications. This chapter provides an overview of the main developments of the three types of nanoexplosives (nano-individual explosives, nanocomposites,

and nano-cocrystals) from preparation and characterization of properties, using a comparison of different approaches for preparing nanoexplosives.” This paper is an excellent primer for forensic explosive analysts who will be encountering these types of explosives in criminal or terrorist bombings in the near future (if they have not already) [516].

8. General detection

Mochan, W. and Ramirez-Solis, A. reported that “The GT200 device has been extensively used by the Mexican armed forces to remotely detect and identify substances such as drugs and explosives. A double blind experiment was performed to test its efficacy. In seventeen out of twenty attempts, the GT200 failed in the hands of certified operators to find more than 1600 amphetamine pills and four bullets hidden in a randomly chosen cardboard box out of eight identical boxes distributed within a 90 m × 20 m ballroom. This result is compatible with the 1/8 efficacy expected for a useless device, and is incompatible with even a moderately effective working one” [607]. This is not surprising since the UK Government banned their use in Iraq and Afghanistan in 2010 and the owner of the company, Gary Bolton, was convicted on 26 July 2013 on two charges of fraud relating to the sale and manufacture of the GT200 and sentenced to seven years in prison [564,565, and 584].

Seman, J., Johnson, C. and Giraldo, C. proposed the creation of an identification taggant that survives detonation and can easily be recovered. “This paper shows that traces of two elements, samarium (Sm) and holmium (Ho), can be identified from explosive post-blast residue”. Post-blast residue was analyzed by neutron activation analysis (NAA) and the two elements were detected. The approach is not clear as to whether ratioing or another method would be employed for the thousands of “codes” needed for an identification taggant [622].

A) Canine Explosives Detection

MacCrehan, W., Young, M., and Schantz, M. employed a “novel solid-phase microextraction with externally-sampled internal standard (SPME-ESIS) vapor-time measurements of two volatile compounds associated with canine detection of plastic explosives, 2-ethyl-1-hexanol and cyclohexanone.” They used a polydimethylsiloxane (PDMS)-based material for use as canine training aids [656].

Hall, N. and Wynne, C.D.L. looked at complex odor mixtures with oxidizers and oxidizers alone for canine detection capabilities. They “... evaluated the effect of two training procedures on dogs' ability to identify the presence of a critical oxidizer in complex odor mixtures.” Some dogs “received odor mixtures that varied from trial to trial with and without an oxidizer.” Moreover, some were trained on solely the oxidizer. Their results were that the dogs who were trained on mixtures had “above chance discrimination of the oxidizer from variable backgrounds and dogs were able to readily generalize performance, with no decrement, to mixtures containing novel odorants.” They also reported that dogs trained on oxidizers alone “... led to a precipitous drop in hit rate when the oxidizer was presented in a mixture background containing either familiar and/or novel odorants” [653].

Colizza, K., Gonsalves, M., McLennan, L., Smith, J. and Oxley, J. studied, in depth, the metabolites of triacetone triperoxide (TATP) and compare those to methyl ethyl ketone peroxides (MEKP) in canines to determine possible toxicity of these materials to canines [648].

DeGreeff, L.E., Peranich, K., & Simon, A. looked at “the capability of canines to generalize or discriminate between related target odors including single target odors and binary mixtures” [650].

Ong, T., Mendum, T., Geurtsen, G., Kelley, J., Ostrinskaya, A., and Kunz, R. used a sensitive, real-time vapor analysis mass spectrometer, with a “detection library of nine explosives and explosive-related materials consisting of 2,4-dinitrotoluene (2,4-DNT), 2,6-dinitrotoluene (2,6-DNT), 2,4,6-trinitrotoluene (TNT), nitroglycerin (NG), 1,3,5-trinitroperhydro-1,3,5-triazine (RDX), pentaerythritol tetranitrate (PETN), triacetone triperoxide (TATP), hexamethylene triperoxide diamine (HMTD), and cyclohexanone, with detection limits in the parts-per-trillion to parts-per-quadrillion range by volume.” They found areas of improvement for canine training [657].

B) LIBS Detection

Rezaei, A., Keshavarz, M., Tehrani, M., and Darbani, S. using LIBS, reported how aluminum affected PBX. They reported, “this work introduces a new method on the basis of the laser-induced breakdown spectroscopy (LIBS) technique in air and argon atmospheres to investigate the determination of aluminum content and detonation performance of aluminized PBXs.” They also stated, “By using the LIBS method and the measured intensity ratio of CN/C, an Al content of 15% is found to be the optimum value in terms of velocity of detonation of the RDX/Al/HTPB standard samples” [669].

C) Neutron

Kulcinski, G., Santarius, J., Johnson, K., Megahed, A. and Bonomo, R wrote about using a system to detect landmines or IEDs by the use of small DD or DT neutron sources carried by a drone [679].

D) Terahertz

E) Nuclear Techniques

F) X-Ray

G) Ion Mobility Spectroscopy

Chaffee-Cipich, M., Hoss, D., Sweat, M., and Beaudoin, S. explored the formation of “traps” and malleable surfaces for explosives in IMS sampling in a security setting. Their sampling methods may help in a forensic setting [704].

In a similar fashion Kuzishchin, Y., Kotkovskii, G., Martynov, I., Dovzhenko, D., & Chistyakov, A. reported on a method for detection of ultralow concentration of explosives coupling ion mobility spectrometry (IMS) and laser desorption/ionization on silicon (DIOS). “The DIOS is widely used in mass spectrometry due to the possibility of small molecule detection and high sensitivity” [710].

H) Novel Detection

The references cited in this section are varied. Some are not necessarily completely novel but have a reported significant variation from the standard technology on which they are based.

El-Sharkawy, Y. and Elbasuney, S. used Laser photoacoustic spectroscopy (LPAS). They claimed that theirs is “a novel LPAS technique that offers instant and standoff detection capabilities of trace explosives.” They used this “customized LPAS technique ... for instantaneous trace detection of three main different high explosive materials including TNT, RDX, and HMX” [734].

Adlin, A. and Kumar, K.M. proposed explosive detection by using printed antennas with substrates that can detect explosives based on the E-field excitation value [718].

Zhang, A., Fu, D., Xuan, Y., & Ma, H. introduced a multi-channel system for explosive and drug detection. They reported that they “have developed a new synthetic conjugated polymer with single molecule layer and coated on porous silicon with large surface area to increase quenching signal at least one order, based on this new

film a small handheld explosive detector with sensitivities of 0.1 pg for TNT and 0.1 ng for black gun powder are obtained.” They claimed that “Last year, after face to face competition, our device was selected as the only security detector for the G20 summit held in Hangzhou, China” [808].

Gillanders, R., Samuel, I., & Turnbull, G. “... present a portable photoluminescence-based sensor for nitroaromatic vapours based on the conjugated polymer Super Yellow integrated into an instrument comprising an excitation LED, photodiode, Arduino microprocessor and pumping mechanics for vapor delivery” [739].

A cheap field instrument is reported by Erickson, J., Shriver-Lake, I., Zabetakis, D., Stenger, D. & Trammell, S. using an inexpensive electrochemical assay, with a hand-held “potentiostat for the identification of explosives.” They claimed, “The prototype instrument designed to run the assay is capable of performing time-resolved electrochemical measurements including cyclic square wave voltammetry using an embedded microcontroller with parts costing roughly \$250 USD. We generated an example library of cyclic square wave voltammograms of 12 compounds including 10 nitroaromatics, a nitramine (RDX), and a nitrate ester (nitroglycerine), and designed a simple discrimination algorithm based on this library data for identification” [735].

1) Stand Off

Cole, P., Cal, C.J., Jean, D. R., & Fell N. F. Looked at UV Raman spectroscopy to “determine the effect of additional colors of vehicle paints (*besides white, black and bare metal*) with Clearcoat on the ability of UV Raman to detect explosives on these surfaces.” They reported, “The results clearly show a strong luminescent background in all of the visible Raman spectra and only a weak Raman background signal in the case of UV Raman spectra with 150 backscattering at all 3 UV excitation wavelengths and the onset of luminescence between 1,400 and 1,500 cm^{-1} with 180 backscattering at 257.23-nm excitation” [824].

Kuzovnikova, L., Maksimenko, E., Vorozhtsov, A., Pavlenko, A., Didenko, A. and Titov, S. used an optical-electronic laser complex for the standoff detection of traces explosives. They used Active Spectral Imaging. They reported the results as “Experimental researches in detection of traces of various types of explosives on different substrates were carried out. On average, the probability of detection was 89% and the probability of identification was 91%” [852].

Holthoff, E., Marcus, L., and Pellegrino, P. write on using photoacoustic spectroscopy (PAS), employed in a sensor format. They explained, “PAS is one of the more flexible IR spectroscopy variants, and that flexibility allows for the construction of sensors that are designed for specific tasks. PAS is well suited for trace detection of gaseous and condensed media” [845].

9. Environmental

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.

Ha, Y., Daeid, N.N., Dawson, L.A., DeTate, D., & Lewis, S.W. in an interesting study, looked at explosives that were spiked into soil samples versus actual residues from the detonation of those explosives. They showed how detonations, when examined by scanning electron microscopy, “... reveal that detonations result in newly-fractured planes within the soil aggregates ...”, They also stated that “We demonstrate that detonations cause an increase in

soil porosity, and this correlates to an increased rate of TNT transformation and loss within the detonated soils, compared to spiked pristine soils” [876].

Chatterjee, S., Deb, U., Datta, S., Walther, C., and Gupta, D. demonstrated a review of explosive materials in soils that are contaminated either due to “manufacturing operations, military activities, conflicts of different levels, open burning/open detonation (OB/OD), dumping of munitions etc.”. The review seeks to emphasize the appropriate practices to remediate the contamination [871].

Yu, H., DeTata, D., Lewis, S., and Daeid, N. studied storage effects of explosives in soil. They explain, “in this work, three different soils were spiked with solutions of TNT, RDX and PETN and stored either at room temperature, refrigerated or frozen. Samples were extracted over 6 weeks, with additional samples gamma-irradiated or nitrogen purged prior to storage. Experimental results indicate that TNT underwent very rapid degradation at room temperature, attributed to microbial action, whereas PETN and RDX proved to be more stable” [888].

10. Other (safety, definitions, etc.)

Sisco, E., Najarro, M., Samarov, D. & Lawrence, J. reported on the stability of trace amounts of explosives over time and environmental conditions. Six “explosives were inkjet printed directly onto substrates and exposed to one of seven environmental conditions (Laboratory, $-4\text{ }^{\circ}\text{C}$, $30\text{ }^{\circ}\text{C}$, $47\text{ }^{\circ}\text{C}$, 90% relative humidity, UV light, and ozone) up to 42 days.” At various intervals, samples were extracted and quantified using electrospray ionization mass spectrometry (ESI-MS). The results were, “... compound dependent with minimal sample losses observed for HMX, RDX, and PETN while substantial and rapid losses were observed in all conditions except $-4\text{ }^{\circ}\text{C}$ for ETN and TNT and in all conditions for tetryl.” These are quite interesting results for the authors [957].

Verolme, E., Van der Voort, M., Weerheijm, J., Koh, Y., & Kang, K. tried to extrapolate backwards to see if damage on a post-blast scene can be applied to determine the strength of the original explosion [966].

Of interest for EOD Techs and perhaps other responders, Reid, D., Riches, B., Rowan, A. and Logan, M. proposed a “A new field portable approach using high temperature combustion has been developed and tested to destroy organic peroxides especially TATP. This approach provides a viable alternative to destruction of organic peroxides using explosives, or chemical neutralization. The apparatus is made of commonly available parts, and does not require specialist expertise to safely operate” [944].

Oxley, J., Smith, J., Bernier, E., Sandstrom, F., Weiss, G., Recht, B., and Schatzer, B. mapped pipe fragments for bombs made of steel and PVC. They described pipe fragmentation patterns by fragment weight or surface-area distribution mapping (FWDM) or (FSADM). They make a distinction of presumably steel pipe with cast iron end caps when concluding, “When fillers detonated, detonation velocities of $\sim 4.4\text{ mm}/\mu\text{s}$ were measured. In such cases, side-walls of the pipe were thrown first; the average fragment velocity was $\sim 1000\text{ km/s}^{\dagger 1}$. In deflagrations, the end cap was first thrown; fragment velocities were only $\sim 240\text{ km/s}$ ” [938].

In a macabre paper, Zwirner, J., Bayer, R., Japes, A., Eplinius, F., Dessler, J., & Ondruschka, B. looked at suicide by “the intraoral blast of firecrackers-experimental simulation using a skull simulation.” They stated, “We here report two cases of suicide committed by an intraoral placement of firecrackers, resulting in similar patterns of

¹ Unit of measurement should be m/s and not km/s as reported by the authors in the original paper.

skull injury. As it was first unknown whether black powder firecrackers can potentially cause serious skull injury, we compared the potential of destruction using black powder and flash powder firecrackers in a standardized skull simulant model (Synbone, Malans, Switzerland). This was the first experiment to date simulating the impacts resulting from an intraoral burst in a skull simulant model. The intraoral burst of a “D-Böller” (an example of one of the most powerful black powder firecrackers in Germany) did not lead to any injuries of the osseous skull. In contrast, the “La Bomba” (an example of the weakest known flash powder firecrackers) caused complex fractures of both the viscerocranium and neurocranium. The results obtained from this experimental study indicate that black powder firecrackers are less likely to cause severe injuries as a consequence of intraoral explosions, whereas flash powder-based crackers may lead to massive life-threatening craniofacial destructions and potentially death” [975]. The authors of this paper note that black powder is known to have less velocity upon exploding than typical perchlorate or chlorate-based flash powders.

Final Notes

Papers that were not referenced above can be found in the extensive bibliography. Many of these seem promising as technology advances.

Disclaimer

This is a republication in journal form of a conference proceeding that was produced for the 19th Interpol Forensic Science Managers Symposium in 2019 and was originally published online at the Interpol website: <https://www.interpol.int/content/download/14458/file/InterpolReviewPapers2019.pdf>. The publication process was coordinated for the Symposium by the Interpol Organizing Committee and the proceeding was not individually commissioned or externally reviewed by the journal. The article provides a summation of published literature from the previous 3 years (2016–2019) in the detection of explosives and explosive residues and does not contain any experimental data. Any opinions expressed are solely those of the authors and do not necessarily represent those of their agencies, institutions, governments, Interpol, or the journal.

Declaration of Competing Interests

The authors have no competing interests to declare.

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Other (Safety, Definitions, etc)

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