# Analytical Methods



### **CRITICAL REVIEW**

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# Investigation and analysis of explosive traces in public locations with no military context: a critical review

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This review examines the prevalence of high explosives and gunshot residue (GSR) in public areas with the primary focus on the likelihood of innocent contamination and its implication for forensic investigations. It is found that most studies acknowledge limitations in sample size and geographical scope, emphasizing the need for broader research encompassing diverse locations and environments. Newly applied technologies like Ambient Mass Spectrometry (AMS) and Raman Spectroscopy hold promise for rapid, sensitive, and selective detection of explosives, potentially revolutionising future research and analysis of real-world environments. Multiple studies demonstrate that the detection of high explosive traces like trinitrotoluene (TNT), Research Department Explosive (RDX), and pentaerythritol tetranitrate (PETN) in public areas is statistically rare, indicating a low probability of innocent contamination. In addition, research on GSR prevalence reveals a similarly low risk of transfer from public surfaces, and while some organic GSR (oGSR) components like 2,6-dinitrotoluene (2,6-DNT) are common in non-shooting environments, others like trinitroglycerine (TNG), particularly in conjunction with markers like ethyl centralite (EC), hold stronger evidentiary value. This highlights the importance of analysing oGSR compounds in combination and considering the specific context of the case. More broadly, the dual-use nature of certain chemicals (e.g. ammonium nitrate in both explosives and fertilizers) necessitates cautious interpretation. Similarly, brake pad dust mimicking GSR and low volatility explosives like High Melting Explosive (HMX) present challenges for detection and underscore the need for expert analysis considering environmental factors.

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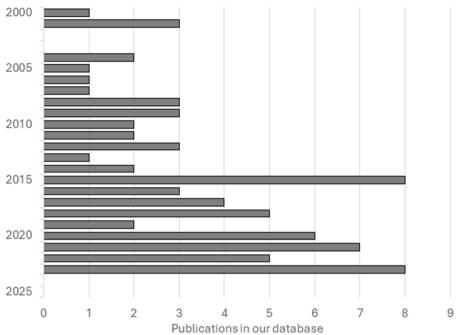
#### Introduction

This review considers the available data on the occurrence and prevalence of explosives in public areas with the primary focus on the likelihood of innocent contamination of hand-contact surfaces and people, and its implication for forensic investigations. In terms of domestic terrorism, the most common materials used are pyrotechnics/fireworks, commercial explosives propellant and improvised/homemade explosives using fuel-oxidiser mixtures.1 However, a recent broad review2 of the forensic detection of explosives (pre- and post-explosion) places a geopolitical component which will broaden the focus. Specifically, it highlights the increased importance of military explosives availability arising from instability in the Middle East and the current conflict in Ukraine. As a means of cataloguing the use of explosive technologies in terrorism, there are two publicly available databases that collate incidents relating to explosions, particularly the United States Bomb Data Centre (US BDC)<sup>1</sup> and The Global Terrorism Database (GTD).<sup>3</sup> These focus on bombing/explosives-related trends (US BDC) and specific

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incidents (GTD), detailing actual explosions or thwarted attempts. The focus of these databases is not however, on the detection of explosive traces.

In parallel there is a thread of published research on environmental contamination by explosives in areas surrounding manufacturing, storage and detonation sites,4 which supports the hypothesis that, away from these areas, the detection of explosives is considered highly discriminating because explosives are rarely found in typical environments, and the level of explosives detectable in public locations varies from nil to minimal.2,5-7 The definitive studies supporting this view were carried out in the 1990s⁵/early 2000s6 in the UK by scientists at the Defence Science and Technology Laboratory (DSTL), with a subsequent study in 2008 in the USA7 with assistance from the Federal Bureau of Intelligence (FBI). However, there remains little data available on the expected occurrence of explosive traces in public locations beyond these. Indeed, this degree of limitation is also observed in the locations of sampling sites where background levels have been determined with almost all being from Europe, the USA and Australia, with none from Africa, for example. This review will collate recent work expanding on early studies that aimed to determine the expected level of background contamination of public spaces with



Source publications on detection of high explosives and gunshot residue from environmental samples (see Appendix).

explosive residues and will restrict itself to high explosives and gun shot residue. This review excludes work on the detection of residues in war zones or military training areas as these would be expected to display significant levels, and there is significant published literature on these locations in the context of decontamination. Fig. 1 shows the distribution of literature sources this review is based on following a database search using search terms based on our sphere of interest plus searches based on the substance names for high explosive related compounds of interest (see Appendix).

This review focuses on the main analytical techniques used to determine explosive traces in the environment. A full range of techniques that have been used, both in research and in the forensic sector, are listed in Table 1.8 As is common for organic compounds in many chemical analysis cases, the use of chromatography - mass spectrometry to not only identify, but quantitate the levels, is an obvious choice. The ability of chromatography to separate the explosive compounds from both one another and other material is invaluable in the accurate isolation and subsequent identification and analysis of trace levels of explosives. Typically, both gas chromatography (sometimes requiring chemical derivatization) and highperformance liquid chromatography have been deployed, coupled with mass spectrometers that include quadrupole, time-of-flight, Orbitrap and magnetic sector. However, even with the use of a mass spectrometer, accurate identification and determination, requires the use of high purity analytical standards to both confirm and identify trace explosives. As the background levels of explosives found in the environment is often at the trace or sub-trace levels definitive analytical

Table 1 Analytical techniques and their figures of merit as deployed in explosive detection (abstracted from ref. 8)

Detection technique	Target analytes	Specificity	Typical LOD
Ion mobility spectrometry (IMS)	Organics	Medium – high	pg–ng
Mass spectrometry (MS)	All (depending on ionisation used)	Medium (unit mass resolution)/ high (high resolution)	pg-ng
Scanning electron microscopy/ Energy dispersive spectroscopy (SEM/EDS)	Elements, $Z > 10$	High (elements)	pg
Raman (including surface enhanced Raman spectroscopy – SERS)	Raman active organics and inorganics	High (pure compounds)/medium (mixtures)	$\mu g/ng$ (SERS)
(μ-) X-ray fluorescence (XRF)	Elements, $Z > 10$	High (elements)	μg
Inductively coupled plasma mass spectrometry or optical emission spectrometry (ICP-MS/OES)	Elements, $Z > 7$ (ICP-MS)	High (elements)	ng
Thermal energy analyser (TEA)	Nitro-containing compounds	High	pg
Electron capture detector (ECD)	Organics	Low	pg

procedures based on assessing the limit of detection and limit of quantitation are required, alongside an assessment of the linearity of the calibration of the determined compounds. A range of other analytical techniques have been deployed including Raman spectroscopy, but again these require the use of analytical standards to identify and confirm the identify of explosives from their characteristic wavelengths based on a structural fingerprint. For inorganic trace explosives the preference has been to use energy dispersive X-ray fluorescence (ED-XRF), often as deployed with scanning electron microscopy (SEM) for quantitative trace analysis. The benefit of this method is it allows direct analysis of trace material with the minimum of sample preparation, and no dissolution of the explosive material. However, ED-XRF is often limited in terms of its sensitivity to detect trace analysis, though with SEM does allow elemental mapping of a surface. Other superior elemental techniques have not yet been fully explored for trace inorganic analysis of explosives. Specifically, the extreme lower limits of detection capability of inductively coupled plasma mass spectrometry (ICP-MS), that could be deployed into this field of analysis considering its maturity (the first commercial instruments being available in the 1980s) as an analytical technique.

### Detection methodology and databases

Comprehensive texts are available detailing explosives, detection methods and the various applications thereof,9 but are now somewhat dated (pre-2000), so miss some of the recent advances in detection techniques, particularly regarding the analysis of precursors and degradation products that can give insights into the explosive's origin.10 The single most important analytical platform technology in this field is chromatography (to separate the compounds of interest from the background and from each other for quantitation). The most common form of detector used in series with chromatography is mass spectrometry. 11,12 Recent reviews 13 highlight that there has been a rapid evolution of technology used to detect and analyse explosives with a growing emphasis on detecting minuscule amounts (nanograms) of explosives residues. This is crucial for pre-blast investigations, linking suspects to explosive materials, and uncovering clandestine activities. Another recent review<sup>2</sup> analysed 477 publications on physicochemical traces of ignitable liquids, cosmetics, explosives, narcotics, and paints within an online Transfer Traces at Activity level Database (TTADB). Of compounds identified as being on this database, 116 relate to explosive technologies. Sadly, this resource is no longer online.

The European Network of Forensic Science Institutes Best Practice Manual for the Forensic Recovery, Identification and Analysis of Explosives Traces,<sup>8</sup> aimed at forensic practitioners, outlines best practices for handling trace explosives evidence, from scene investigation to laboratory analysis and presentation of findings. It focuses on the recovery, analysis and identification of microscopic explosives traces, and covers crime scene and laboratory procedures, but excludes: first response and general management of explosives-related crime scenes; investigation of bulk explosive materials; trace impurity profiling/individualisation of explosives; and identification of

precursor materials for illicit explosives manufacturing. The manual stresses the importance of trained and competent personnel at all stages of the investigation with key competencies for the different roles of crime scene attendant, forensic analyst, and forensic investigator. In addition to measurement methodologies, the manual provides detailed contamination recommendations for both scene and laboratory work that includes personnel decontamination, use of disposable equipment, and designated laboratory spaces for trace and bulk explosives analysis. It also recommends prioritising non-destructive analytical techniques before destructive methods and the use of regular internal proficiency testing and participation in external proficiency testing schemes where available for quality assurance. The manual acknowledges that determining the original explosive composition solely from trace analysis is often impossible. However, advances in equipment and techniques have recently been demonstrated on several classes of explosive10 to give better insight into sources of materials via trace impurity and precursor identification.

# Methodologies for recovery of trace explosives

ASTM International<sup>14</sup> has a recently developed method (ASTM E1588-20) for scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDX) analysis of the inorganic part of gunshot residue (GSR),<sup>15</sup> but there are few other recent standards available in the scientific literature. However, at the time of writing there is an active set of ASTM working committees looking to develop new standards in the areas of terminology,<sup>16</sup> collection of gunshot residue (GSR),<sup>17,18</sup> analysis by liquid chromatography-mass spectrometry (LC-MS)<sup>19</sup> and gas chromatography-mass spectrometry (GC-MS).<sup>20</sup> The use of benchmarking laboratories,<sup>21,22</sup> to enable comparison of results from different sites, sets a minimum acceptable performance level and allows inter-operability.

To prepare explosive trace samples for analysis, specific techniques are used depending on the surface type (Table 2).8

It is important to follow anti-contamination procedures throughout the entire process of collecting, preparing, and analysing explosive trace samples. This includes using clean and dedicated equipment, appropriate personal protective equipment, and control samples. Crowson<sup>24</sup> highlights the critical need to prevent contamination in trace explosives analysis as even minute traces of explosives can compromise results and lead to misinterpretations in forensic investigations. The Forensics Explosives Laboratory (FEL) proposes a rigorous multilayered contamination control system<sup>24</sup> that includes:

#### Personnel protocols

Showering, changing clothes, restricted access, and training on contamination prevention.

#### Controlled environment

Positive air pressure, HEPA filtration, particle monitoring, designated lobby area with specific entry procedures, and routine cleaning.

Table 2 Recommended sampling techniques

Non-porous surfaces	Porous surfaces	Possible additional processing steps
Swabbing the surface <sup>23</sup> with a swab that has been moistened with a solvent. Ethanol/water mixtures are commonly used solvents Solvent wash/extraction of the explosive residues from the surface. Suitable solvents include methanol, methanol/water mixtures, and ethanol/water mixtures	Solvent extraction to extract the explosive residues from the porous material	Ultrasonication to enhance the extraction of explosive residues from the sample
	Vacuuming to collect weakly adhered residues from the surface Scraping to physically remove residues from the surface Headspace sampling for volatile explosives Solid-phase microextraction uses a fibre coated with an adsorbent material to extract analytes from the sample	particles from the liquid extract

#### **Material control**

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Approved suppliers, staged unwrapping of materials, and use of dedicated equipment and reagents. Several suppliers can provide small quantities of selected explosives for use as reference standards for the purposes of equipment calibration (for example, Accustandard<sup>25</sup>).

#### Ion mobility spectrometry (IMS) screening

Helps identify and prevent the entry of contaminated individuals into the laboratory.

#### Monitoring regime

The core of the quality assurance system is a robust monitoring regime, encompassing:

**Weekly Sampling.** Benches, floors, and other surfaces in the main trace laboratory are swabbed and analysed using GC/TEA.

**Monthly Sampling.** Less frequently accessed areas, such as telephones and windowsills, are swabbed and analysed.

Control samples. Processed alongside monitoring swabs, these ensure the integrity of the sampling materials and analytical process.

**Spiked samples.** Help assess the efficiency of extraction and clean-up procedures.

#### **Action criteria**

A graduated response protocol is in place depending on the detected amount of explosives, ranging from no action to thorough inquiries and procedural changes.

#### **Continuous improvement**

FEL operates on a continuous improvement model. The QA regime is constantly reviewed and updated based on:

**Monitoring data analysis.** Identifying contamination trends and potential sources.

**Root cause analysis.** Investigating contamination incidents to identify weaknesses and implement corrective actions.

The intention of this protocol is that contamination of samples could only take place if multiple breaches of the procedure occur.<sup>24</sup> It should be noted however, that in forensic science, contamination is defined as the sum of the background *i.e.* the presence on a crime scene of traces that are not relevant to the crime investigated, and pollution *i.e.* the unintentional insertion of target analytes in a sample during its analysis.

Detection and identification of explosives is a rapidly developing area. However, there is a tension between adoption of new techniques that provide additional insights versus use of established methods with a proven track record. Several reviews13,26 examine emerging technologies with potential in explosives detection, including terahertz spectroscopy, nuclear techniques, and advanced imaging methods. Klapec13 provides an extensive list of recent patents demonstrating ongoing research and development in explosive detection technologies, highlighting areas like enhanced sensitivity, new material development, and improved detection devices. In addition to technology development, there is also a push to implement some of the existing laboratory-based techniques in public transportation hubs.27 GC-MS and LC-MS remain the mainstay of studies (see for example<sup>28-30</sup>) but significant advances have been made in the use of Ambient Mass Spectrometry (AMS).31 Ambient Mass Spectrometry has emerged as a powerful tool for trace explosives detection, offering advantages such as minimal to no sample preparation, rapid analysis times (within seconds), direct analysis of various surfaces and substrates, the use of soft ionization minimizing fragmentation (particularly crucial for labile peroxide-based explosives) and adaptability for field-deployable instruments. Hopper<sup>32</sup> has developed a method for simultaneous analysis of anions and cations in low explosive residue. Using this technique, a wide variety of ions pertinent to low explosives analysis, including chloride, nitrite, nitrate, sulfate, perchlorate, thiocyanate, chlorate, and cyanate as well as ammonium, potassium, sodium, calcium, and magnesium can be identified and quantitated in a single run.

Colón-González *et al.*<sup>33</sup> have demonstrated the feasibility of detecting explosives *i.e.* Trinitrotoluene (TNT), Pentaerythritol

Tetranitrate (PETN), and Research Department Explosive (RDX) on hair using non-invasive Raman spectroscopy. While Kresge<sup>34</sup> has applied method development of Raman to trace analysis. This novel approach identifies inorganic ions commonly found in homemade explosives (HMEs), potentially offering a rapid, cost-effective, and accurate way to analyse trace evidence. Further research by Jander<sup>35</sup> has developed ultraviolet (UV) resonance Raman spectroscopy for the detection of trace explosives. For real-world applications, it is necessary to develop algorithms that can pick out the spectral features of explosives against the typical background in a sample spectrum, and this work demonstrates a methodology that can tolerate high levels of fluorescence background whilst enabling the detection of traces of Ammonium Nitrate Fuel Oil (ANFO) and TNT explosives at surface coverage levels of 55 µg cm<sup>-2</sup> in a blind test experiment. In a different approach Yu et al.36 utilized NMR pulsed field gradient techniques to monitor ANFO emulsion stability under different storage conditions. Their findings provided valuable insights into the impact of temperature and the stabilizing effect of calcium nitrate.

Mullen and Giordano<sup>37</sup> have reported increased sensitivity in TNT and 2,6-dinitrotoluene (2,6-DNT) detection by employing a novel ionization technique combining secondary electrospray and corona discharge with IMS. This highlights the potential of combined approaches in enhancing existing technologies. However, while IMS is often used for security screening due to its rapid sampling times and relative accuracy, it is not often used in a forensic context. This is because of its limited ability to separate analytes (based on their ion mobility), its tendency to become overloaded quickly (requiring cleaning before continuation), and perhaps most importantly its potential for false positives.

# Background environmental levels of GSR

Significant investigation into GSR has been reported in the literature with 149 publications between 2006 and 2020 being reported in 2021 by Crispino,<sup>2</sup> with almost half of those being published since 2015. Studies have looked at the detectable components of GSR from an elemental, molecular and microstructure perspective as well as investigating best practice in detection and quantitation.

In 2010, Dalby<sup>38</sup> reviewed the literature surrounding the contamination, distribution, and transfer of GSR. In addition, the review lists 49 organic and 52 inorganic compounds that can potentially be found in GSR. The most common elemental components of the inorganic fraction of GSR were lead (Pb), barium (Ba) and antimony (Sb).<sup>39,40</sup> As such, this composition is considered characteristic of inorganic GSR and can be said to originate from primers such as Sinoxid primer, which is composed of lead styphnate, barium nitrate, and antimony sulfide. In forensic terms, trace analyses containing a combination of any two of these elements are considered consistent with GSR.<sup>41</sup> While single element particles containing only lead, antimony, or barium are also produced from firearm discharge,

and they may be classified as consistent with or commonly associated with GSR, little evidential weight is attached to their presence. The presence of other elements, such as iron, in particles of retrieved materials is not conclusive in distinguishing GSR from other sources.  $^{42}$  The shape of the particles is also a factor considered when typing them as GSR or not  $^{42}$  though, it is considered secondary in evidentiary value to the elemental composition. GSR particles are often spheroidal, non-crystalline particles between 0.5 mm and 5.0 mm in diameter. However, some are irregular in shape, vary from 1 to  $^{100+}$  mm in size, or both.  $^{41}$ 

In 2023, Blakey *et al.*<sup>43</sup> investigated the potential for inadvertent transfer of gunshot residue to individuals through contact with public environments in the UK using Scanning Electron Microscopy with Energy Dispersive X-ray Analysis (SEM-EDX) for elemental analysis of microparticles. In total 262 samples from various public locations across England, including trains, buses, taxis, and train stations were analysed. No characteristic GSR particles (containing lead, barium, and antimony) were detected on any of the samples collected. Four indicative/consistent particles *i.e.*  $2 \times Ba/Al$  and  $2 \times Pb/Sb$ , were found on a single train seat back. However, as these particles can originate from sources unrelated to firearms activity, the study concluded that the potential for GSR transfer through contact with publicly accessible locations appears to be insignificant.

Brożek-Mucha<sup>44</sup> quantified the drop-off in detectable inorganic GSR among "frequent shooters" grouped into daily, weekly, monthly and quarterly shooters. This demonstrated detectability at the level of monthly use. Subsequently, measurement of background levels of GSR was carried out across a population of 100 non-shooters of varying occupations to demonstrate only one had detectable GSR residue i.e. Pb/Sb/ Ba. This is contrasted with two firearm-related crime cases, demonstrating the use of GSR analysis to identify the shooter. In 2015, Hannigan41 had reported on the incidence of GSR on clothing and hands. The probability of finding "one or two" characteristic 3-element particle i.e. Pb/Sb/Ba, by chance was put at less than 0.02, and hence characterised as a relatively uncommon event. Subsequently, Lucas et al.45 performed a similar study in Australia yielding comparable results to those of Brożek-Mucha,44 with a frequency of 0.3% in a random sample of 289 persons. These studies41-45 support the hypothesis that locations with no connection to firearms or firearm materials are very unlikely to contain materials that are identifiable as GSR. They strongly support the position that the presence of GSR on an individual is more likely due to direct or indirect contact with firearms or a firearms discharge, rather than from unwitting, casual contact with public surfaces which are contaminated by others.

Fruneaux<sup>46</sup> studied the frequency of occurrence of organic gunshot residue (oGSR) in non-shooting environments in the USA. They found that 2,6-DNT was detected in 100 out of 119 samples (83%). However, the ubiquity of 2,6-DNT in various industries confirms its limited value as a standalone oGSR marker, consistent with previous findings.<sup>39</sup> Notably, trinitroglycerine (TNG) was absent in all samples, supporting its

potential as a stronger indicator of oGSR when found in conjunction with other markers, as proposed by Tobin.<sup>47</sup> The study emphasizes the importance of evaluating oGSR compounds in pairs, like TNG and ethyl centralite (EC), for reliable forensic interpretation. The absence of this pair in the nonshooting cohort strengthens their association with actual firearm discharge. The qualitative nature of the Fruneaux study,<sup>46</sup> which focused on presence rather than quantification, limits the interpretation of oGSR prevalence. Additionally, differences in firearms regulations are also expected to influence background levels of GSR in public places.<sup>43</sup> This is potentially seen in the difference between the Fruneaux<sup>46</sup> study (USA) with ubiquitous 2,6-DNT, and Blakey<sup>43</sup> (UK) where GSR was essentially absent on subjects without connection to firearms.

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Several similar studies have been carried out in Italy,<sup>42</sup> Poland,<sup>44</sup> Australia,<sup>45</sup> Ireland<sup>41</sup> and a broad study inside/outside Europe.<sup>39</sup> There is general agreement that GSR presence correlates strongly with exposure to firearms, and that presence of GSR in the general population is extremely low. In Switzerland, Manganellia *et al.*<sup>48</sup> studied eight oGSR compounds on 122 civilians and 115 individuals working in police services. Results indicated a non-negligible prevalence in the civilian sample, with 18% of the 122 civilian participants having relevant compounds on their hands and 11.5% on their wrists or sleeves. For the police population, the prevalence was higher than for civilians, with 36.5% of the hand samples and 33% of the wrist samples being positive in a similar manner. These higher values were due to the possession of service weapons and their regular use in shooting exercises.

# Background environmental levels of explosives

Lahoda's 2008 study in the USA7 aimed to establish background levels of explosives and related compounds in various public areas (e.g. public buildings, ATMs, mailboxes, transportation hubs) across 28 cities in the United States and Puerto Rico. Over three hundred samples were tested. For inorganic ion analysis, swabs were extracted with deionized water and analysed using capillary electrophoresis with a dual opposite injection method to detect both anions and cations simultaneously. The inorganic compounds were separated into ubiquitous ions (chloride, nitrate, and sulfate), highlighting the need for careful interpretation in forensic contexts, and rare ions (ammonium, nitrite, perchlorate, thiocyanate, chlorate, and cyanate), suggesting their potential value as specific markers for inorganic explosive residues. With the exception of nitrate, most low explosive oxidizers were found to be rare in the environment and may be considered indicative of the presence of inorganic explosive residue.7 For organic analysis, swabs were extracted with acetone and analysed using gas chromatography with a pulsed-discharge electron capture detector (PDECD). In comparison, no organic explosives (e.g. TNT, RDX) were definitively identified in any of the samples suggesting that the chances of finding high explosive residues in a public area in the USA are minimal.7

In the UK in 2004, Cullum *et al.*<sup>6</sup> investigated the prevalence of high explosive traces in various public locations across four major UK cities, aiming to assess the likelihood of innocent contamination. The study found a very low prevalence of high explosive traces in public areas. Only four out of 501 samples (excluding controls) tested positive for explosives, representing <0.5% of the total. Only three types of explosives were detected in trace amounts:

RDX (7.5 ng found on train seats).

NG (3.6 ng found on a taxi floor and 11.7 ng in a hotel wardrobe).

2,4-Dinitrotoluene (2,4-DNT) (15.2 ng found on an airport X-ray machine).

Notably absent were traces of TNT, pentaerythritol tetranitrate (PETN), ethylene glycol dinitrate (EGDN), nitrobenzene (NB), and other nitrotoluenes, despite the analytical techniques being capable of detecting them at appropriately low levels. Cullum et al.6 suggests that the detected traces could originate from legitimate sources like firearms (NG), previous presence of armed police (NG), or industrial applications (2,4-DNT). The study concluded that it was unlikely that visitors to the investigated public areas could become significantly contaminated with explosives. However, Cullum et al.6 recommended expanding the research to include a wider variety of public locations and different types of sites, plus analysis for less volatile explosive substances like High Melting Explosive (HMX). The Cullum et al.6 study had built on previous work5 in 1996 which had investigated the prevalence of explosives traces in various public places and police environments to assess the likelihood of innocent contamination. These earlier findings agreed with its conclusion5 that traces of common high explosives (NG, TNT, PETN, and RDX) are rare in public areas like transport hubs and airports. The Crowson et al. 5 study had only detected four low-level traces of RDX across all public areas, indicating this is not a common environmental contaminant. While, NG, which is often associated with firearms, was detected in several police stations and related vehicles. This emphasizes the importance of considering occupational exposure when interpreting NG traces on suspects. In addition, RDX and PETN were primarily found in controlled police environments, such as, a secure unit office and a vehicle that had (probably) been used for explosives training.

Resendre<sup>49</sup> investigated contamination issues related to the transfer and persistence of explosive residues, specifically TNT, PETN, RDX, HMX, triacetone triperoxide (TATP), and ammonium nitrate (AN), on surfaces and individuals handling these materials. The study examined the presence and quantity of explosive residues on surfaces (benchtops, aluminium foil) and individuals' hands before, during, and after handling various explosives. The amount of residue transfer varied significantly between individuals, highlighting the influence of personal handling techniques and hygiene practices. It was reported that the average sampled PETN was 4  $\mu$ g, but with a standard deviation of 6  $\mu$ g, after handling detonating sheets.<sup>49</sup>

An environmental survey on emulsion/gel explosives, reported in 2001,<sup>4</sup> assessed their constituent inorganic ions and sugars *via* 71 samples at four urban sites and two rural sites across the UK.

Summer and winter sampling was carried out to account for environmental variance due to winter road gritting. The results indicated that the anions, chloride and sulfate were commonly detected, nitrate and phosphate less so, fluoride, nitrite (except for one sample), bromide and chlorate were only detected at very low levels while thiocyanate and perchlorate were not detected. Sodium was found to be extremely common, calcium was recovered in 71% of samples, potassium and magnesium were recovered in approximately half of the samples, while ammonium was recovered in fewer samples (but was present in significant levels in wall samples). Relevant sugars were present in most of the house, hotel and car vacuum samples.

Alternate scientific approaches have also explored more conventional approaches, often deployed in drug analyses, that instead of looking for the parent molecule, investigate its degradation product with a view to provide a more accurate identification of the explosive used. This approach has been deployed on contaminated sites, where explosives have been extensively used, as part of remediation strategies. For example, it is known that TNT readily photo decomposes, by exposure to UV radiation, to 1,3,5-trinitrobenzene, 4,6-dinitroanthranil, 2,4,6-trinitrobenzaldehyde and 2,4,6-trinitrobenzonitrile.<sup>9,43</sup> While 2,4,6-trinitrophenyl-methylnitramine (tetryl) is hydrolysed to N-methyl picramide43 and then undergoes photolysis to the picrate ion, N-methyl picramide, methyl nitramine, nitrate and nitrite.38 And finally, RDX has been shown to undergo biodegradation under anaerobic conditions,8 though this is less relevant for the identification of trace explosives in the predominantly aerobic conditions of the public environment.

## Limitations of currently published research

From a forensic perspective while Letendre et al.2 highlighted significant knowledge gaps in understanding the transfer and persistence of physicochemical traces, and Cullum et al.6 emphasised that while trace amounts were found on some surfaces, transfer to individuals is likely to be <100% efficient, affecting the possibility of contamination occurring, all suffer from the same aspect. Ultimately, the number of sites and environments investigated5-7,43 is relatively small and further research is needed to increase data reliability. It was noted in the preparation of this review, no work carrying out further large-scale systematic testing has been identified since Lahoda<sup>7</sup> in 2008. While there is a common approach following ENSFI recommendations,8 there is variability in sample collection details, which could influence the efficacy of extraction, such as the choice of extraction solvent. While methodology has evolved over time, it varies between different research groups, and depending on which target explosive is being investigated (e.g. methyl-tert-butyl ether,5 ethanol: water,6 and more recently 40:60 acetone: IPA46). Previously, Dalby et al.38 had reviewed the studies assessing solvent efficacy with regards to NG extraction and found ethanol to be the most effective. In summary there is a small body of studies that provide valuable baseline data for interpreting the presence and levels of explosive-related compounds in forensic

samples, but further work is needed to generalize the results and create a definitive methodology for linking the presence or absence of a compound to a forensic recommendation.

More research is also required on individualisation i.e. linking the recovery and detection of a specific explosive from a crime scene to the explosive material or its raw materials that are associated with a suspect. Brust has explored individualisation of trace explosives, 10 linking the materials to their source materials, via identification of impurities and/or variations in isotopic composition. Focussing on casework in the Netherlands, the research highlights10 data from the Netherlands Forensic Institute (NFI), indicating the predominance of PETN, TNT, RDX, and HMX in both pre- and post-blast cases. This data emphasizes the need for effective molecular profiling techniques applicable to these commonly encountered explosives and offers some methodologies for this.10 Trace impurities introduced during manufacturing or degradation are a powerful tool for source determination. Analysis of TNT was done using vacuum-outlet GC-MS, emphasizing the potential for profiling thermolabile explosives more generally.10 Alternatively, variations in isotopic ratios, which can be measured using isotope ratio mass spectroscopy (IRMS), can differentiate between batches of explosives based on their origin and production processes e.g. analysis of hexamethylene triperoxide diamine (HMTD).10 For the most chemically simple explosive components e.g. ammonium nitrate, Brust advocated10 combining isotopic analysis with techniques like inductively-coupled plasma mass spectrometry (ICP-MS) for elemental profiling. A further reason for profiling an explosive and its degradation products is to use the molecular profile for "activity level" evidence crucial for reconstructing events and supporting or refuting suspect claims, and this was performed for PETN.10

The analysis of environmental samples could provide information on source apportionment of explosives. Wastewater analysis offers a new avenue for intelligence gathering in explosivesrelated investigations.50 Using the presence of trace levels of explosives and their by-products to pinpoint potential areas of illicit activity and the types of explosives being involved has been studied.51 A methodology was developed and validated using LC-MS for detecting trace quantities of explosives like HMTD and PETN in wastewater. Like the successful application of wastewater analysis in monitoring illicit drug use, Gamble51 proposes that the detection of specific explosives and their precursors in wastewater can provide valuable information about activities occurring within a particular sewage catchment area. Another underexploited area of research is bank note analysis. While research has been published on the results of banknote analysis for post-explosion residues,52 typically used to connect the bank note with attacks against Automated Teller Machines (ATMs). No research looking at pre-explosion transfer residues has been carried out.

The interpretation of forensic analytical data can sometimes lead to wrongful interpretation *i.e.* so-called misleading evidence. Some studies have attributed detection of analytes flagged as indicating explosives to non-malign sources instead. For example, Fruneaux<sup>46</sup> cited that EC was detected in 25 samples (21% of the total number of samples investigated), primarily in individuals within industrial occupations (*e.g.* drivers handling

Critical Review chemicals), suggesting potential environmental sources which can confuse the analysis. Some of this confusion may be due to home-made explosives (HME), something that complicates

the dual-use nature of many chemicals, including those used in regulation.<sup>53</sup> The legitimate use of these materials means they might reasonably expect to be detected in the public environment. Examples of such compounds include ammonium nitrate which despite its widespread use in fertilizers, is used in HME necessitating stricter global standards, and indeed, past efforts to mitigate the explosive potential of AN with diluents have been circumvented by non-state actors.53 Oxidizers, such as hydrogen peroxide and potassium chlorate, also fall into this category, particularly the use of hydrogen peroxide in HME, and specifically in organic peroxide explosives (OPEs) and potassium chlorate as a favoured HME component after restrictions on AN, demonstrating the cause and effect dynamic of precursor regulation.<sup>53</sup> Other common materials carry the potential for confusion despite little functional similarity to explosives. Tucker et al. 54 investigated whether modern automotive brake pads have the potential to produce particles that could be mistaken for gunshot residue (GSR), a concern raised by previous research in the early 2000s. The study found that initiatives to eliminate lead from automotive components have been successful. This has significantly reduced, though not eliminated, the possibility of brake pads producing particles containing lead, a key component of traditional GSR. In this case the use of morphology and composition are key differentiators. While some brake pads produce particles containing barium and antimony (also found in the inorganic part of GSR), their morphology and composition differ significantly from GSR. Brake pad particles are generally angular and flaky, unlike the spheroidal shape of GSR particles formed under high temperature and pressure during firearm discharge. Brake pad particles often contain a heterogeneous mix of elements, including high levels of iron and sulfur, not typically found in the inorganic part of GSR. Another potential metal particle contaminant in the environment is from welding. Brożek-Mucha<sup>55</sup> reported how SEM-EDX can be used to distinguish welding fumes from the inorganic part of GSR. Deployment of airbags can lead to materials that could be confused with GSR39,56,57 but it has been found that the associated high levels of aluminium metal can be used to distinguish them from GSR.56 The common nitro functional group can also lead to potential for false positive identification at the molecular level. Examples include angina medication (glyceryl trinitrate)46,58 and nitrocellulose and 2,6-DNT which are innocently found in paints, surface coatings and dyestuffs though often in niche applications. 46,59 Finally, volatile components in fruits, such as, grapefruit, oranges, and pears, have been shown to produce peaks at the same retention time as EC in some forms of chromatography. 46,60 However, adaptations to tandem ion mobility spectrometry (IMS) methodology has been cited, using chlorine adducts, for improved selectivity in explosives detection.61

#### Conclusion

Multiple studies5-7 concur that detecting high explosive traces (e.g. TNT, RDX, PETN) in public areas is statistically rare,

indicating that there is a low probability of innocent contamination. Research on GSR reveals a similarly low risk of transfer from public surfaces.43 The absence of characteristic GSR particles in diverse public settings strengthens the evidentiary value of GSR found on individuals, suggesting direct or indirect links to firearms. While some oGSR components like 2,6-DNT are common in non-shooting environments,46 others like TNG demonstrate stronger links to firearm discharge when found in conjunction with specific markers (e.g. EC). This highlights the importance of contextual interpretation and analysing oGSR compounds in combination. The dual-use nature of numerous chemicals necessitates careful interpretation of findings. For example, ammonium nitrate, while potentially used in explosives, have legitimate applications in fertilizers.53 Brake pad dust can mimic GSR, underscoring the need for expert analysis (rather than an automated or generalized process) and considering environmental factors.54 Certain explosives, like HMX, pose challenges for routine detection due to low volatility, potentially leading to false negatives.6

These conclusions have been developed from studies with limited sample size and rather specific geographical scope or environments, emphasizing the need for further research which broadens sampling across diverse locations and environments. There are several emerging technologies such as Ambient Mass Spectrometry (AMS) and Raman Spectroscopy which are showing promise for rapid, sensitive, and selective detection of explosives, potentially improving future research analysis.31,33

An area of research that is currently underdeveloped is the role that standardisation of sampling methodologies (i.e. beyond sharing of best practice but measuring similarity) and the application of quality assurance and quality control procedures to the analysis of trace explosives for what are often individual samples. The role of a scientific government body in coordinating and delivering proficiency testing methods is required to ensure data integrity in terms of sampling protocols and the generated interpretation of the analytical data.

### Explosives chemical names

#### **Abbreviations**

AKII Akardite II (1-methyl-3,3-diphenylurea)

ANFO Ammonium nitrate fuel oil

DMDNB 2,3-Dimethyl-2,3-dinitrobutane

3,5-DNA 3,5-Dinitroaniline 1,3-DNB 1,3-Dinitrobenzene 2,4-DNT 2,4-Dinitrotoluene

2,6-DNT 2,6-Dinitrotoluene 3,4-DNT 3,4-Dinitrotoluene DPA Diphenylamine 2-nDPA 2-nitrodiphenylamine 4-nDPA 4-Nitrodiphenylamine

N-nDPA *N*-nitrosodiphenylamine Ethyl centralite (1,3-diethyl-1,3-diphenylurea) EC

Ethylene glycol dinitrate **EGDN** 

**ETN** Erythritol tetranitrate **Analytical Methods** Critical Review

**HMTD** Hexamethylene triperoxide diamine

HVMX High melting explosive (octahydro-1,3,5,7-tetranitro-

1,3,5,7-tetrazocine)

MCMethyl centralite (1,3-dimethyl-1,3-diphenylurea)

N,N-DPF N,N-Diphenyl formamide

NB Nitrobenzene NG Nitroglycerine 2-NT 2-Nitrotoluene 3-NT 3-Nitrotoluene 4-Nitrotoluene 4-NT Picric acid

**PETN** Pentaerythritol tetranitrate

RDX Research department explosive (1,3,5-

trinitroperhydro-1,3,5-triazine or cyclotrimethylene

trinitramine)

R-salt Hexahydro-1,3,5-trinitroso-1,3,5-triazine TATP Triacetone triperoxide Tetryl 2,4,6trinitrophenylmethylnitramine

1,3,5-1,3,5-Trinitrobenzene

TNB

TNG Trinitrogylcerine 2,4,6-2,4,6-Trinitrotoluene

TNT

### **Appendix**

Search terms used to accumulate the papers automatically were:

- Forensic.
- Explosives.
- Firearms.
- Gun shot residue (GSR).
- Improvised explosive device (IED).
- Incendiary.
- Smokeless powders (SLPs).
- Surfaces.
- High performance liquid chromatography mass spectrometry (HPLC-MS).
  - Liquid chromatography mass spectrometry (LC-MS).
  - Gas chromatography mass spectrometry (GC-MS).

Subsequent specific substance searches were carried out individually against the following materials: 1,3,5-TNB (1,3,5-Trinitrobenzene), 1,3-DNB (1,3-Dinitrobenzene), 2,4-DNT (2,4-Dinitrotoluene), 2,6-DNT (2,6-Dinitrotoluene), 2-nDPA (2-nitrodiphenylamine), 3,4-DNT (3,4-Dinitrotoluene), 3,5-DNA (3,5-Dinitroaniline), 4-nDPA (4-nitrodiphenylamine), 2-NT (2-nitrotoluene), 3-NT (3-nitrotoluene), 4-NT (4-Nitrotoluene), 2,4,6-TNT (see also TNT below) (2,4,6-trinitrotoluene), AKII (Akardite II (1-Methyl-3,3-diphenylurea)), ANFO (Ammonium nitrate fuel oil), DMDNB (2,3-Dimethyl-2,3-dinitrobutane), DPA (Diphenylamine), EC (Ethyl centralite (1,3-Diethyl-1,3-diphenylurea)), EGDN (Ethylene glycol dinitrate (ethylene glycol dinitrate)), ETN (Erythritol tetranitrate), HMTD (Hexamethylene triperoxide diamine), HMX (High melting explosive (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)), MC (Methyl centralite (1,3-Dimethyl-1,3-diphenylurea)), N,N-DPF (N,N-Diphenyl formamide), NB (Nitrobenzene), NG (Nitroglycerine), N-nDPA

(N-nitrosodiphenylamine), PA (Picric acid), PETN (Pentaerythritol tetranitrate), RDX (Research department explosive (1,3,5trinitroperhydro-1,3,5-triazine or cyclotrimethylene trinitramine)), R-salt (Hexahydro-1,3,5-trinitroso-1,3,5-triazine), TATP (Triacetone triperoxide), TNT (Trinitrotoluene), Tetryl (2,4,6trinitrophenylmethylnitramine), and TNG (Trinitrogylcerine). Papers were collected and reviewed by the authors. The focus of the work was to identify studies relevant to the detection of explosive traces in civilian areas without recent history of terrorism or warfare. Therefore, papers were eliminated from the study that focussed on any of the following scenarios:

- (a) Detection of explosives used based on analysis of residues post-explosion.
- (b) Detection of explosives used in military training areas (that were mainly focussed on soil remediation), or military operations.

The remaining papers were taken forward into this review.

### Data availability

No primary research results, software or code have been included and no new data were generated or analysed as part of this review.

#### Conflicts of interest

There are no conflicts to declare.

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