

## Detection and Analysis of Explosive Residues Using Advanced Instrumental Techniques

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### Abstract:

Explosive is a substance that undergoes rapid chemical reaction either oxidation or redox action to produce heat, light, gas and a violent expansion. The remnants or debris left behind after an explosion takes place is called explosive residue. Detection and analysis of post explosion residue is technically difficult and more challenging because of the presence of huge amount of complexity. This is very effective in criminal investigation and in terrorism act. Proper sample collection, extraction, filtration, and clean up procedure must be followed for post explosion analysis. There are various techniques which can be used for trace evidence examination in explosion such as X-ray diffraction, gas chromatography-mass spectrometry, Fourier transform infrared spectroscopy, and ion mobility spectrometry while analytical chromatographic methods mainly HPLC, TLC, and spot test used for analysing the amount of explosive residues in soil. With the use of these techniques, forensic experts can precisely detect and categorise the explosive substance which aid in the determination of the origin of explosives. The application of non-factory manufactured explosives like Homemade explosive (HME) is constantly evolving which makes it more challenging for forensic labs to establish detail protocols for every potential scenario.

**Keywords:** Explosion analysis, Post explosion residue, Homemade explosive, PETN, Nuclear magnetic resonance, Analytical techniques

### 1. INTRODUCTION

While the modern world is constantly changing on a daily basis, criminal activity is also increasing in tandem with these improvements. These days, explosives are used in guerilla warfare, terrorist strikes, and bank robberies<sup>1</sup>. The majority of explosives are used by the military after being taken by criminals during transit. Both civilians and defense security are at risk from the bombs<sup>2</sup>. Additionally, miners and commercial industries that demolish buildings employ explosives<sup>3</sup>. The explosives leave behind residues, leftovers, debris, and other things in their immediate surroundings when they are used<sup>4</sup>. They can be discovered in the earth, the air, or on the user's clothing, fingerprints, and surroundings. Trace evidence analysis, which identifies explosive remains, is a crucial field of forensic research<sup>5</sup>.

According to the Indian Explosive Act (1884), "explosive" means gunpowder, nitroglycerine,

nitroglycol, gun-cotton, di-nitro-toluene, tri-nitro-toluene, picric acid, di-nitro-phenol, tri-nitro-resorcinol (styphnic acid), cyclo-trimethylene-trinitramine, penta-erythritol-tetranitrate, tetryl, nitro-guanidine, lead azide, lead styphnate, fulminate of mercury or any other metal, diazo-di-nitro-phenol, coloured fires or any other substance whether a single chemical compound or a mixture of substances, whether solid or liquid or gaseous used or manufactured with a view to produce a practical effect by explosion or pyrotechnic effect; and includes fog-signals, fireworks, fuses, rockets, percussion-caps, detonators, cartridges, ammunition of all description and every adaptation or preparation of an explosive as defined in this clause<sup>6</sup>.

After an explosion the remnants left are called the explosive residues. Usually, the explosives found in the residues are TNT (Tri-Nitro-Toluene), HMX (High Melting eXplosive), PETN

(pentaerythritol tetranitrate), ANFO (Ammonium Nitrate Fuel Oil), Dynamite, gun powder, organic explosives such as nitro-compounds, chlorates and perchlorates<sup>7</sup>. Many analytical methods, including gas chromatography, mass spectrometry, ion chromatography, TLC, Direct-Analysis in real time (DART), x-ray diffraction, and others, are typically used to detect and analyse these explosives<sup>8</sup>. This overview explains the different analytical techniques, detection, sample extraction, and concentration.

## 2. HISTORY OF EXPLOSIVES

Explosives are the most chaotic and noisiest inventions ever made in human history. In the 9th century of the Tang Dynasty, Chinese alchemists mixed sulfur, charcoal, and potassium nitrate (saltpeters) and named as gun powder<sup>9</sup>. These chemical ingredients were easily available in ancient times. They used gunpowder as they packed gunpowder into bamboo tubes to make grenades and highly flammable arrows to kill the soldiers in warfare. Europeans got gunpowder through trading routes and they created cannon guns which made it more disastrous<sup>10</sup>.

In 1846, Ascanio Sobrero did a reaction by mixing concentrated nitric acid with glycerol and an oily product formed as glyceryl trinitrate also called nitroglycerin. Alfred Nobel invented the blasting cap in 1863, a device that revolutionized the technology of explosives. It is inserted in the closed end of mercury fulminate which consists of the small copper capsule<sup>11</sup>. Nobel wanted to make it safer so that it would not explode during its transportation because his brother died in an explosion in a factory in the 1860s, then he mixed the nitroglycerin with kieselguhr, a naturally occurring soft, siliceous powder, and the mixture was named as dynamite which was far safer to handle than nitroglycerine. He made this patent in 1867<sup>12</sup>. During 1870s ammonium nitrate has been widely used as a supplemental oxidant in dynamites<sup>13</sup>.

Julius Wilbrand, a German chemist synthesized 2,4,6-trinitrotoluene by nitration of toluene in 1863 which is also known as TNT and used as a high explosive Carl Häussermann light on its explosive properties in 1891 like its stability, strength, and perfect for shells and bombs. TNT and chlorine gas were used as brutal chemical weapons in World War I. TNT is used in combination with high explosives and acts as an energetic binder<sup>14</sup>.

2,4,6-trinitrophenol (TNP), more commonly known as picric acid, was produced by nitration of horns of animals, and natural resins. If picric

acid reacts with the metal shells to form metal picrates, known as primary explosives. Ammonium picrate, also known as Dunnite, is an explosive made by the US Army during World War I<sup>15</sup>. By World War II, things became more advanced and scientists developed RDX. However ultimate crossover was the atomic bomb which was dropped in 1945 on Hiroshima and Nagasaki. Nowadays experimental and laser-guided weapons have more potential to make an explosion even more precise and terrifying.

## 3. TYPES OF EXPLOSIVES

An explosive is simply defined as a substance that undergoes rapid chemical reaction to produce heat, light, gas, energy and violent expansion<sup>16</sup>. Explosives are classified based on their velocity of detonation which is used in the military and classification based on their source/application is used in field of forensic science<sup>17</sup>.

### 3.1. Based on Velocity of Detonation

The explosive's deflagration/detonation power is influenced by the detonation velocity, meaning that detonations traveling at supersonic speeds will be more destructive. When the detonation velocity in the unreacted material surpasses the sound speed, detonation takes place. Remember that sound travels at around 340 m/s in an air medium at ambient temperature. However, because atoms and molecules are closer together in liquid or solid states, sound travels faster through these media (for example, 1500 m/s in water, 3000 m/s in wood, or 6400 m/s in aluminium)<sup>18</sup>. The speed of sound in most explosives, which are often made in powdered form, is predicted to be between 1000 and 3000 m/s; this is significantly slower than the speed of sound in aluminum metal that is tightly packed. Hence, the velocity of detonation is established to differentiate between high explosives and low explosives<sup>19</sup>.

**3.2. High energy explosives:** When high-energy explosives go off, a supersonic shock wave is produced that typically travels at speeds of over 4,000 meters per second<sup>20</sup>. They are subdivided according to their sensitivity. The classification according to sensitivity is given in Table 1.

**3.3. Low energy explosives:** When low energy explosives detonate, a subsonic wave front is produced that is slower than sound. The overpressure created inside a container by the build-up of gas products from deflagration is what causes the explosion that low explosives may cause<sup>21</sup>. Low explosives are mostly used as propellants in pyrotechnics, space rockets, or

handgun ammunition due to the progressive creation of these gas products during their deflagration<sup>22</sup>. Typical low explosives include

flash powder, black powder, and smokeless powder. The classification based on application is given in Table 2.

**Table 1.** Classification of explosives according to their sensitivity<sup>23</sup>.

Primary explosives	Secondary explosives	Tertiary explosives
Primary explosives are highly sensitive to several minor stimuli that cause detonation, such as heat, sparks, or friction. They typically don't have much power.	Secondary explosives typically need a powerful shock to detonate because they are essentially insensitive to weak stimuli.	Tertiary explosives are an additional category that is occasionally introduced to the classification in order to include and distinguish those insensitive explosives that are frequently employed for mining and demolition tasks
Due of these characteristics, they are typically employed sparingly as supplementary high explosive detonators.	With explosion velocities exceeding 6000 m/s, the majority of secondary explosives are incredibly potent.	They have lower detonation velocities than military secondary explosives.
Lead azide and Mercury Fulminate are examples of primary explosives.	RDX, PETN, HMX, TNT are few examples.	Typical tertiary explosives include dynamite, ANFO, and ANAI ingredients.

**Table 2.** Classification based on their application or use<sup>24</sup>.

Military explosives	Commercial explosives	Home-made explosives
High explosives that adhere to stringent specifications for functionality, performance, and safe handling, storage, and transportation are known as military explosives. They need detonators to detonate because they must be strong and insensitive to weak or moderately strong stimuli	Commercial explosives are those that are permitted for non-military uses, such as those used in demolition, mining, and as propellants for weapons, rockets, and pyrotechnics.	All "do-it-yourself" explosives are considered homemade explosives.
Typically, military explosives are organic compounds made up solely of nitrogen, oxygen, hydrogen, and carbon atoms.	Compared to military explosives, these high explosives have comparatively low detonation velocities.	Since the advent of the Internet in the previous century, producing or synthesizing explosives no longer requires a scientist.
In particular, nitro-groups (-NO <sub>2</sub> ) are usually present in these compounds as oxygen and nitrogen atoms. Military explosives are sometimes referred to as nitro-explosives.	This allows more exact control over the power needed to move and shatter rocks and structures.	Online resources offer freely available recipes that walk users through the process of obtaining, creating, and synthesizing various explosives.
Common military explosives include TNT, HMX, PETN, and RDX.	Nowadays, the most often used commercial explosives for mining and demolition are dynamite and ANFO formulations	Usually, oxidizer-fuel explosive combinations like ANFO are included in these compositions.

#### 4. DETECTION AND ANALYTICAL TECHNIQUES

There is increasing interest in using a wide range of techniques to detect trace explosive residues because trace detection of explosives has been a persistent problem for decades and has emerged as one of several crucial issues in defence science, public safety, and international counterterrorism.

##### 4.1. Gas Chromatography and Mass Spectrometry (GC-MS)

In gas chromatography, the explosives-containing gaseous or vaporized sample is introduced into the GC apparatus<sup>25</sup>. The GC

column, a long, coiled tube coated with a stationary phase, is where the sample is placed. Depending on their volatility and affinity for the stationary phase, the sample's constituent parts are separated. The sample is moved along the column by a heated carrier gas, usually nitrogen or helium, and different compounds separate as they elute at different times <sup>26</sup>.

Mass spectrometry, As the separated compounds exit the GC column and enter the mass spectrometer, ionization takes place. Two common ionization techniques are chemical ionization and electron impact. Fragmentation: Ionization causes molecules to break apart into

smaller pieces<sup>27</sup>. The specific material being studied has a distinct fragmentation pattern. The ions produced during ionization are identified by the mass spectrometer using its mass to-charge

ratio ( $m/z$ ) detector<sup>28</sup>. The detector creates a mass spectrum, which shows ion abundance against  $m/z$ .



**Figure 1.** Gas Chromatography-Mass spectrometry (GC-MS)

### 4.2. High Performance-Liquid Chromatography (HPLC)

Mikhail S. Tswett initially provided it during his research on plant pigment separation. When the stationary phase is solid and the moving phase is liquid, HPLC is just the opposite of GC<sup>29</sup>. According to A Chromatographic and

Electrophoretic Methods/A High-Performance Liquid Chromatography, the liquid is passed through a column that contains solid particles. As the sample passes through the column, the liquid delays the various components at different points in time. It is sufficient to do the experiment in HPLC at room temperature<sup>30</sup>.



**Figure 2.** High Performance Liquid Chromatography

### 4.3. Capillary Electrophoresis

While electrophoresis does not employ a moving liquid phase, it does employ the similar criteria of migrating on a stationary phase. This method involves applying an electric voltage across the

stationary phase or medium<sup>31</sup>. When using the capillary electrophoresis procedure, an electric field caused the analyst to move within the electrolyte solution. Typically, the system includes a detector, electrodes, source, and vials<sup>32</sup>.





**Figure 3.** *Capillary electrophoresis*

#### 4.4. Raman Spectroscopy

Adolf Smekal made the initial prediction in 1923, and in 1928, the Indian scientist C.V. Raman gave it its name. The optical system must be composed of quartz or glass, and the substance being tested must be colourless and pure. It is made up of a light source, detector, wavelength selector, and sample lighting system<sup>33</sup>. Forensic laboratories have made extensive use of Raman spectroscopy to identify explosive substances. Nevertheless, this approach can be time-

consuming and could eventually lead to pattern infection. Raman spectroscopy helps scientists identify substances like PETN, TNT, HMX, and RDX that have the potential to cause immense destruction<sup>34</sup>. These spectrometers can detect even minute amounts of substances without destroying the evidence because they are incredibly sensitive and non-destructive. Additionally, they help to improve protection by minimizing scientists' direct contact with explosive substances<sup>35</sup>.



**Figure 4.** *Raman Spectroscopy*

#### 4.5. X-Ray Diffraction (XRD)

XRD is a non-destructive analytical technique used to determine the crystalline structure of materials, including explosives. The concept that X-rays interact with the atoms in a crystalline sample when

they are focussed on it and scatter at specific angles forms the basis of X-ray diffractometric (XRD)<sup>36</sup>. The resulting diffraction pattern allows material identification by displaying the crystal structure in exquisite detail<sup>37</sup>.



**Figure 5.** *X-ray Diffraction*

#### 4.6. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a powerful analytical method that can be used to identify explosives and analyse their chemical composition.

Each chemical composition has its own unique absorption bands, which are based on the theory that molecules absorb infrared light at specific

wavelengths<sup>38</sup>. This is the basic idea behind FTIR. When the sample is subjected to an infrared beam, specific infrared light frequencies are absorbed as a result of interactions between the sample's molecular structure and the beam. Use of Infrared Light When infrared radiation passes through the sample and absorbs specific infrared light frequencies, the chemical bonds in the explosive molecules vibrate<sup>39</sup>.



**Figure 6.** *Fourier Transform Infrared Spectroscopy (FTIR)*

**4.7. Thin Layer Chromatography (Tlc)** - To separate the components of a mixture, the TLC technique uses the stationary phase as well as moving phases that are solid and liquid. Here, a small coating of aluminium oxide or silica gel is made to act as the stationary phase, and the sample to be examined can be put straight onto

the plate<sup>40</sup>. Due to capillary action, the sample begins to ascend. In this manner, the sample's constituents are dispersed among the liquid flowing phase and the immobile solid phase. Greater affinity for the moving phase causes components to move more quickly than those in the stationary phase. Next, the retention value is determined<sup>41</sup>.



**Figure 7.** Thin Layer Chromatography (TLC)

#### 4.8. Direct Analysis in Real Time (DART-MS)

When used in conjunction with mass spectrometry (MS), ambient ionization sources like direct analysis in real time (DART) can evaluate materials immediately on surfaces and provide quick, sensitive analysis with high chemical specificity with little to no sample preparation<sup>42</sup>. A Thermo LTQ XL mass spectrometer with an Ion Sense DART-SVP ionization source make up the DART-MS. All

DARTMS studies employed helium as the ionization gas, and the temperature of the gas heater was set at 200 °C<sup>43</sup>. With the exception of HMTD, which was examined in positive mode, the mass spectra for every experiment were gathered in the negative-ion mode for all explosives within the  $m/z$  range of 50–400. Four DART-MS techniques—direct-insert, transmission, thermal desorption, and QuickStrip—were assessed<sup>44</sup>.



**Figure 8.** Direct Analysis in Real Time-Mass spectrometry (DART-MS)

## 5. DISCUSSION

Advanced tools like high-performance liquid chromatography (HPLC), gas chromatography (GC), and gas chromatography-mass spectrometry (GC-MS), Raman spectroscopy are very effective in determining what's left behind after an explosion. There are superiorities and limitations to these techniques and a lot of space for further improvement.

HPLC is a good exceptional for tackling polar and non-polar substances like- nitro aromatic

compounds and inorganic ions which are scattered around after an explosion. HPLC is able to handle liquid samples and complex molecules according to their interactions with the stationary phase. It was reported in 2019 that liquid chromatography (LC), high-performance liquid chromatography (HPLC), and ultra-high performance liquid chromatography (UHPLC) are perfect techniques for separation and are considered non-destructive and less harmful to highly explosive, thermally sensitive material than gas chromatography methods.

There are 2 types of liquid chromatography for measuring the degradation and aging of PBX9501. Compounds with high molecular weight were measured by size exclusion chromatography (SEC) while compounds with low molecular weight were measured by using HPLC. These tools can be useful for the application in forensic field like in comparison of PBX explosives. Combining HPLC-fluorescent detection with femtomole-level detection helps to derivatize aliphatic and aromatic amines.

In gas chromatography, vaporized or gaseous sample material with having explosive substance in it is sent into the GC system then the sample is inserted into the long, coiled tube column which is covered with stationary phase and also known as the GC column. On the basis of the stationary phase and the volatility of the sample, components are separated out. Carrier gas of helium or nitrogen is heated and it transports the sample along the column and different chemicals are removed at different periods, resulting in their separation. When the separated chemicals enter the mass spectrometer (MS) after leaving the GC column, the process of ionization occurs. Ionization molecules split into tiny pieces called fragmentation and this pattern of fragmentation is unique to specific substances.

For determining the crystalline structure of explosive compounds X-ray diffraction is considered which is a non-destructive analytical method. When X-rays are passed from the sample, X-rays interact with the crystal lattice of the sample consisting of atoms which are scattered at particular angles, it helps to give more detail through different diffraction patterns and helps to determine the identification.

Fourier Transform Infrared Spectroscopy (FTIR) is an analytical technique that can identify explosives and their chemical composition. Different chemical compounds absorb infrared light at particular wavelengths and thus show distinctive absorption bands.

The sample is crushed and spread into a thin film then the sample is exposed to an infrared beam and the molecular structure of the sample absorbs the frequencies of infrared light. Transmitted or reflected infrared light from a sample is detected by a detector and is transformed into an interferogram, a process called Fourier Transform. By comparing absorption bands, exact functional groups present in explosives can be easily determined.

Advanced laboratory methods, such as X-ray diffraction, nuclear magnetic resonance (NMR) spectroscopy, Fourier transform infrared spectroscopy, and gas chromatography-mass spectrometry (GC-MS), are used to collect evidence and preserve the integrity of evidence should be followed and ensure that the proper chain of custody is maintained, these all are helpful during the reconstruction of an explosive event.

Raman spectroscopy technique is widely used in laboratories of forensic for the detection and analysis of explosive compounds, it allows experts to gather more detail of samples and helps in distinguish between different explosives such as ammonium nitrate (AN), pentaerythritol tetranitrate (PETN), trinitrotoluene (TNT), high melting explosive (HMX) also known as octagon or cyclotetramethylene and royal demolition explosive (RDX). Spectrometers are highly sensitive. Raman spectroscopy isn't used as a confirmatory test in a court of law.

HPLC, GC, Raman spectroscopy, GC-MS, X-ray diffraction, and FTIR have revolutionized how we analyze post-explosion residues, turning messy chaos into crucial forensic evidence. Even though they're not perfect—each has its blind spots—their combined power is unquestionable. The challenge now is to rectify them, making them faster, more rigid, and smarter against the messy reality of an explosion site.

## 6. CONCLUSION

In conclusion, forensic investigations and security operations heavily rely on the analysis of explosive trace evidence. Identifying and interpreting minute traces of explosives can help determine the origin of explosives used in criminal operations or acts of terrorism, connect suspects to explosive devices, and understand the nature of explosive occurrences. In order to detect and analyse residues of explosives, various analytical methods are used. Determine the best method to detect and analyse explosives depends on factors such as type of explosive, the matrix the residue is on, age and condition of the residue, the sensitivity required and whether the analysis needs to be done in the field or in laboratory.

However, considering the strengths and limitations of each technique, the GC-MS technique is most reliable and most versatile in detecting and analysing explosive residues. This technique has high sensitivity and helps in detecting the trace elements on the matrix. They are excellent for complex mixtures and provides both qualitative and quantitative information. In



order to enhance security procedures, protect public safety, and guarantee the prompt and effective resolution of explosives-related incidents, explosive trace evidence analysis is crucial given the ongoing threats posed by explosives. By continuing research, developing new technologies, and collaborating with experts and government representatives, we can address the issue of terrorism and criminality related to explosives and make the world a safer place.

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