

# Extraction and Post-Explosion Trace Analysis of Explosions

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**Abstract.** Recently, the Beirut port catastrophic explosion left behind a tremendous number of deaths and injuries. This accident highlighted the critical impact of the accidental explosion. Post-explosion investigations revealed the key role of the probable swap sampling and extraction of residues, either organic or inorganic, collected from the crime scene in achieving a fast, accurate and reliable forensic analysis. On the other hand, unqualified strategies which focused only on the analysis of organic explosives could misguide the whole forensic investigations. Therefore, more detailed forensic investigations for the detection of materials, which is still a serious puzzle for technical specialists in the field of explosives analysis, become a must. This work develops a simple on-site scheme for the precious swap sampling, separation, and identification of both organic and inorganic residues. Application of Ion Trap Mobility Spectrometry (ITMS) was essential to ensure the existence of explosive traces in post-explosion collected residues. Then, separating organic and inorganic traces was an essential step before utilizing the analysis techniques. Fine-tuned High-performance Liquid Chromatography (HPLC) was able to detect organic explosive traces with a minimum detection limit of 0.5ppm. On the other hand, Ion Chromatography (IC) was capable to effectively discriminate the levels of inorganic anions and cations of ammonium nitrate, ammonium perchlorate, potassium chlorate, and black powder explosive with a minimum detection limit of 0.1 ppm.

**Keywords:** Inorganic explosives, Trace analysis, Forensic Investigations, Explosion residues, and Post-explosion analysis.

## 1. Introduction

Improvised explosive devices (IEDs) originated in the 1970s from the British Army and the Irish Republican Army [1]. They consist of an explosive charge, detonator, and initiation system. The Belarussian Rail War and the IRA used IEDs during World War II and the 1969-1997 campaign [2]. The IRA developed sophisticated anti-handling devices, such as mercury tilt switches, and used remote-controlled roadside bombs to detonate bombs accurately [3]. The number of explosive devices (IEDs) used in Afghanistan has increased by 400% since 2007 and has been the main cause of death among NATO troops [4]. In the Iraq War, 63% of U.S. deaths occurred due to IEDs. IEDs are mounted on animal carcasses, cans, and boxes, and are used against coalition forces. In 2010, Taliban fighters developed a new generation of IEDs that are almost undetectable due to their lack of metal or electronic parts [5].

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NC, NQ, and composition B, and utilizing a simple, accurate, and feasible on-site post-explosion scheme to extract explosive residues for both real and artificial explosions.

## 2. Experimental Work

### 2.1 Chemicals

Acetone, Ethanol, Methanol, Acetonitrile, Potassium bromide, Carbon tetrachloride, and Hexane were purchased from Sigma-Aldrich (Steinheim, Germany). All Solvents were HPLC-grade and were utilized without any further purification. Military explosives used in this study; TNT, RDX, ANFO, NC, NG, PETN, plastic explosive mixture (PETN + (plasticizer, nylon, rubber), Sheet explosive (PETN + Binders), Black powder, Ammonium nitrates, and Potassium nitrates, were acquired from local military factories.

### 2.2 Analysis of explosives residues in soil (artificial explosion)

Post-explosion analysis is challenging due to limited and contaminated explosive residues. A simple, accurate, and feasible on-site analysis scheme involves creating an artificial post-explosion sample, extracting, filtration, and concentration, and subjecting the organic extract to analytical chromatographic methods like ITMS and HPLC, with the minimum detection limit determined gradually [7],[8]. The extraction process involved acetone as the optimum solvent for explosives, followed by coldwater flow and heating. Filtration was done using a bucket funnel and vacuum pump. The sample concentration was achieved through fractional distillation, heating, and filtering. Ion trap mobility spectrometry confirmed trace explosives' presence.

### 2.3 Real post-explosion analysis

Real explosions were carried out using a 1 Kg explosives mixture of TNT and RDX (2:1); classified as military explosives, and 10 Kg of ANFO, and burning 0.5 Kg of Black Powder classified as homemade explosives. Soil and debris were collected from each explosion scene separately. The explosion scene was examined using magnification and light to determine debris and unexploded explosive charge. Post-explosion samples were collected and analyzed using aqueous and organic extractions. Post-explosion analysis is crucial due to limited original explosive material. Sample collection is essential, and acetone extractions are used for organic explosives and related compounds. Soil sample filtration is essential for removing fine particles from organic extracts. A vacuum pump is used for efficient filtration, followed by a disposable filter cartridge for analysis. Solid-phase extraction technique (SPE) is employed to remove explosive traces, and the cartridge is used for standard mixtures and post-explosion samples [9][10][11].



Figure 1. Solid-phase extraction technique SPE column.

### 2.4 Ion trap mobility spectrometry (ITMS) in the detection of explosives

ITMS instrument is a trace detection system of explosives; it employs a new technology to identify explosives with high efficiency and sensitivity. The patented ion trap surpasses conventional IMS in ionization efficiency and sensitivity, enhancing detection peaks for positive and negative ion modes. The detection capability of ITMS is significantly expanded since most explosive ions are detected in the negative mode while some of them are detected in the positive mode. The ITMS system model –GE Ion Track Company was adjusted at its factory for trace detection and identification system of the following explosives substances such as TNT, EGDN, NG, RDX, PETN, HMX, AN, and smokeless powder. ITMS instrument is used in milli, micro, and nanograms concentration detection of explosives. It is applying on dry samples after the clean-up process for fast detection of explosives

within few seconds. Swabs are taken by the trap and inserted in the slot located in the front of the instrument as shown in Figure 3 [12][13]



**Figure 2.** Collection of Samples with Trap



**Figure 3.** Inserting sample trap into disrober.

### 2.5 HPLC for identification of explosive residues

The Agilent 1100 series HPLC apparatus was used for an investigation, consisting of an autosampler, quaternary pump, and UV-Visible detector [14][15]. The system was operated under specific conditions, including a regulated temperature reversed-phase column and a UV-Visible detector. The explosives examined included TNT, RDX, HMX, PETN, NQ, composition B, and NC.

### 2.6 Ion Chromatography (IC) in Identification of Explosives

Ion chromatography (IC) is a useful analytical method for analyzing inorganic homemade explosives, with the FBI developing identification methods based on IC for slurry explosives and bombing debris. The IC-3000 system was used to analyze ANFO and Black powder, using standard solutions of inorganic cations and anions [16]. Dionex ICS-1100 ion chromatography was used for simultaneous separation of anions and cations, with a 25  $\mu$ l sample loop for measurements. The first ICS-1100 Dionex system was used for anions separation and suppressed conductivity detection, with operating conditions such as column size, flow rate, injection volume, and system backpressure. The second ICS-1100 Dionex system was used for cations separation and suppressed conductivity detection [16][17][18].

## 3. Results and Discussions

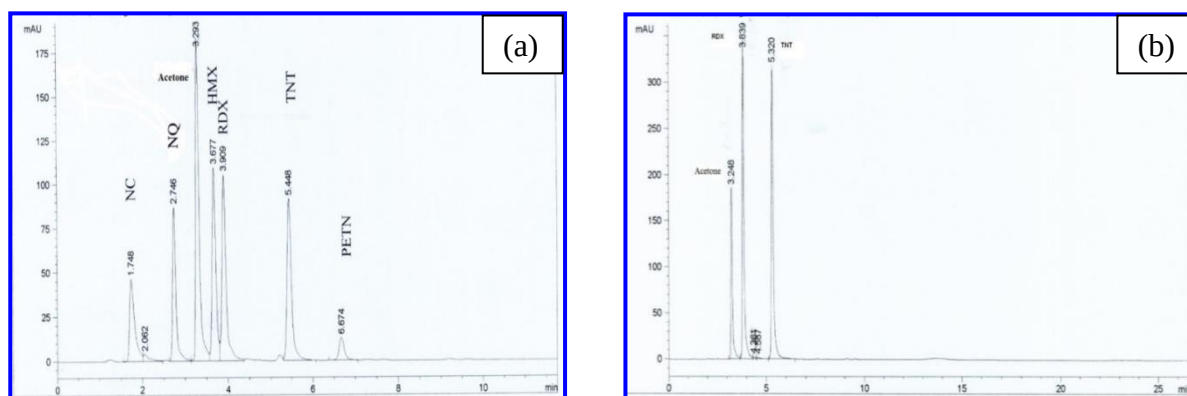
### 3.1 ITMS in the detection of explosives (artificial)

ITMS was successfully utilized for fast detection of explosives in time not exceed few seconds (4 -10) and for the detection of explosives traces in hand swabs. ITMS plasmagram of the detected explosives shows the strength–time relation for each explosive sample. Results showed that ITMS was able to detect explosive mixtures in milligrams concentrations without interfering results to confirm the presence of traces in the examined sample before the extraction step. ITEMS failed to distinguish the difference between NG and NC where the determination of either of them sometimes was refereed to nitro peak at retention time 3.830 in the negative ion mode of the plasmagram. Other times NC was detected as a smokeless powder at retention time 7:4 in the positive ion mode. So that ITMS used for qualitative analysis of traces explosives.

### 3.2 HPLC in the identification of explosives

This work makes use of an Agilent 1100 series HPLC apparatus. This apparatus consists mostly on a UV-Visible detector, quaternary pump capable of running with four eluents at regulated ratio and flow rate, and autosampler. Stationary phase was All system parameters may be automatically regulated by HP-3DLC chemostation software using a controlled temperature reversed-phase column [14][15]. The operating conditions followed: stationary phase: 150mm by 10mm - inverted-phase column Zorbax SB C–18 mobile Phase: 60% ACN, 40% Water, flow rate: 0.4 ml/min; pressure: 80 bar; linear velocity: 0.137 cm/sec; injection volume: 1  $\mu$ l, Detector: 220 nm UV

Detector Post-run: thirty minutes, column temperature: thirty-five °C Investigated explosives were TNT, RDX, HMX, PETN, NQ, composition B, and NC.



**Figure 4.** HPLC Chromatogram for; (a) NC, NG, HMX, RDX, TNT, and PETN, (b) composition B (TNT+RDX)

**Table 1.** The minimum detection limit (MDL) of all investigative explosive samples

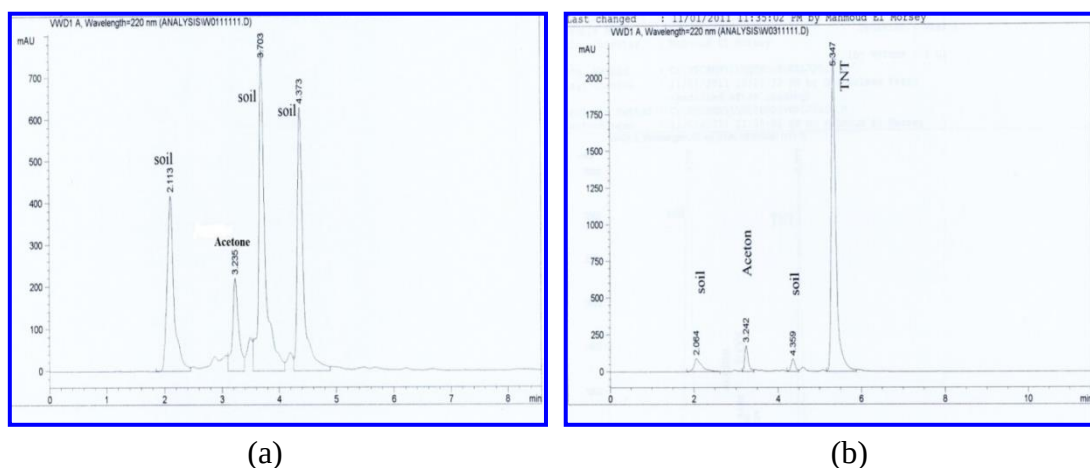
| NO. | Explosive     | Minimum detection limit MDL( ppm) |
|-----|---------------|-----------------------------------|
| 1   | TNT           | 0.5                               |
| 2   | RDX           | 0.5                               |
| 3   | HMX           | 0.5                               |
| 4   | PETN          | 5                                 |
| 5   | NQ            | 1                                 |
| 6   | NC            | 50                                |
| 7   | Composition B | 5                                 |

### 3.3 IC in the identification of explosives

Results showed that Ion chromatography (IC) is an ideal technique for the analysis of homemade inorganic residues after the explosion. Analysis of explosives residue using IC technique overcomes difficulty to detect homemade explosives based on inorganic materials. This technique showed the ability to identify the original inorganic explosive through the determination of the levels of inorganic anions and cations present. [43, 44] Figures 8 and 9 show the high potential for separation, discrimination, and effectiveness of this technique in post-explosion trace analysis of ammonium nitrate, ammonium perchlorate, and black powder explosives anions and cations respectively.

### 3.4 Application of HPLC for analysis of explosive residues in soil

To simulate a post-explosion sample, 10 g of TNT was mixed with 100 g of soil sample. This sample was subjected to 6 hours of extraction, repeat 4 times, and then filtration and concentration were performed. An organic extract of a blank soil sample was concentrated and subjected to investigation by the HPLC technique. The obtained HPLC chromatogram of the blank sample is shown in Figure 5 (a). The sample which consists of a mixture of soil and TNT was cleaned up by solid-phase extraction technique (SPE) to remove any interfering substances, especially in a heavily contaminated matrix. The obtained HPLC chromatogram of TNT residues in the soil is shown in Figure 5 (b).

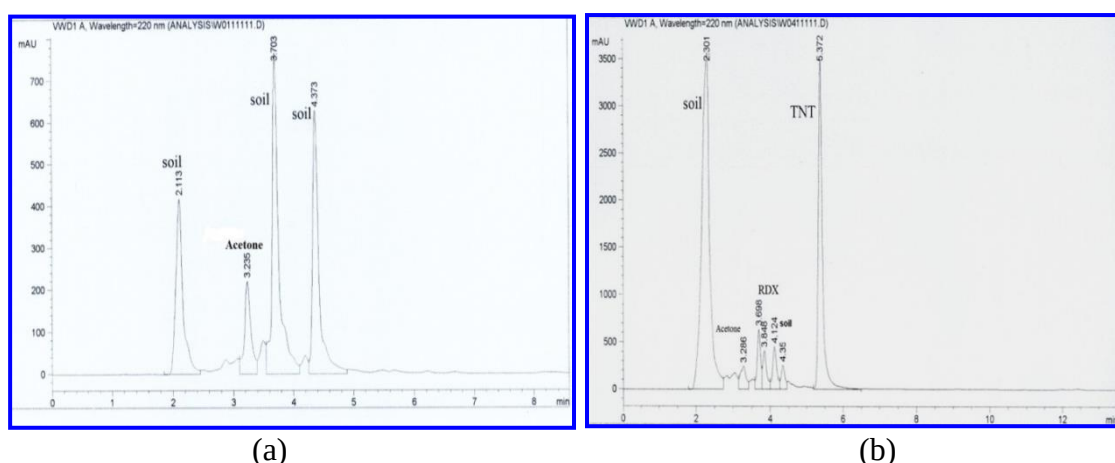


**Figure 5.** HPLC chromatograms of (a) the blank soil sample, (b) TNT residues in soil

### 3.5 Real post-explosion analysis

#### 3.5.1 Application of HPLC for real post-explosion sample analysis

Real explosions are carried out by using a 1Kg explosives mixture of TNT and RDX (2:1) classified as military explosives and 10Kg of ANFO based on ammonium classified as homemade explosives, also burning of 0.5 Kg of Black Powder. The post-explosion analysis scheme was implemented through proper sample collection debris, organic and aqueous extractions, filtration and concentration then concentrated post-explosion organic extracts were investigated by chromatographic analytical techniques such as HPLC for trace analysis of organic explosives residues and IC for trace analysis of inorganic homemade explosives residues. HPLC chromatograms of pure soil samples and soil samples taken from the center of a real explosion of 1Kg explosives mixture of TNT and RDX (2:1) are shown in Figure 6.



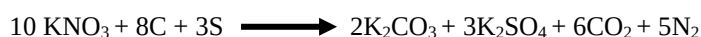
**Figure 6.** HPLC chromatograms for (a) blank soil sample, and (b) TNT and RDX explosives mixture collected after Real Post Explosion.

#### 3.5.2 Application of IC for real post-explosion sample analysis

ANFO sample extraction with carbon tetrachloride (CCl<sub>4</sub>) to remove any residue of fuel oil and filtration through a millipore membrane filter (pore size 0.45  $\mu$ m, 47 mm diameter) then the sample was diluted using de-ionized water to be analyzed using the ion chromatography. Standard solutions of inorganic cations and anions were purchased as 1000 ppm individual ion chromatography standards from Merck and diluted as required. Distilled Water treated with a Water pro - Labconco deionization system to obtain water of 18.2 megaohms resistivity was used in preparing standard solutions and diluting the samples. Generally, the minimum detection limit in ion chromatography for all cations and anions is 0.01 ppm while the maximum limit may vary from 100 to 200 ppm

according to the desired ion. For analyzing higher concentrations a dilution process is required or reduces the volume of injected sample. By analyzing post-blast residue samples of the real explosion of ANFO, it was found that the investigated sample contained salts of sodium, ammonium, potassium, magnesium, and calcium as cations and chloride, nitrate, and sulfate as anions but the major constituents were ammonia and nitrate. The obtained experimental results for the real explosion of ANFO using the IC instrument.

As shown in Table 2 the detection of the nitrate ion from the real explosion of ANFO is very clear at which the dilution of 1-1000 is suitable in this case, increasing the dilution the detection still available but some sort of negative error is obtained which is increased by increasing the dilution factor. Detection of the nitrate ion is easy even if the dilution was up to 1-1000000 with an error of -16 % of the true value which is an excellent result compared with huge dilution factor; that means the analysis of post-blast residues samples of real explosion enough to know and estimate the type of the inorganic anions and cations present in explosion field and will give an idea about the components of the explosive. Black powder is a mixture of 75% potassium nitrate (KNO<sub>3</sub>) which supplies oxygen for the reaction, 15% charcoal (C), and 10% sulfur (S) which serves as a fuel. A simple equation for the combustion process is:



For the pre-blast analysis about 0.5g of black powder was dissolved in 1 liter of deionized water and filtered through a millipore membrane filter (pore size 0.45µm, 47mm diameter) then the sample was diluted (1-100) using deionized water to be analyzed using the ion chromatography. By using IC the obtained experimental results pre-blast analysis of black powder cations and anions. For the post-blast analysis, a sample of about 100 g of black powder was burned in a crucible which was weighted and then washed with 100 ml deionized water to remove the entire burning residue and reweighed to the determined weight of the burning residue. This dissolved residue was filtered through a millipore membrane filter (pore size 0.45µm, 47mm diameter) then the sample was diluted (1-50) using deionized water to be analyzed using ion chromatography.

**Table 2.** Effect of dilution on the detection of nitrate ion

| Experiment No. | Sample dilution | Nitrate ion conc. (ppm) |
|----------------|-----------------|-------------------------|
| 1              | 1-1000          | 69712                   |
| 2              | 1-2000          | 69634                   |
| 3              | 1-4000          | 67047                   |
| 4              | 1-8000          | 65177                   |
| 5              | 1-16000         | 64620                   |
| 6              | 1-32000         | 64476                   |
| 7              | 1-64000         | 63041                   |
| 8              | 1-128000        | 63422                   |
| 9              | 1-256000        | 63708                   |
| 10             | 1-512000        | 59654                   |
| 11             | 1-1024000       | 58552                   |

## Conclusions

Post-explosion trace analysis is a complex technique influenced by various materials used in military, commercial, and homemade explosives, potential contamination, and modern analytical techniques. This study aimed to identify post-explosion trace analysis from bombing scenes using real and artificial explosions. The study found that applying both real and artificial explosions was crucial for comparing results and ensuring the accuracy of analytical methods. Ion trap mobility spectrometry (ITMS) was used to ensure the minimum concentrations of trace explosives in post-explosion residues. A post-explosion scheme was used for separation and concentration processes in both real and artificial explosions, suitable for analysis using HPLC and IC. The use of Porapak RDX resins for analyzing sub-part per billion levels was successful. HPLC technique was effective for identifying organic explosives, while IC was the most sensitive method for inorganic explosives. The application of an efficient post-explosion scheme and devices like HPLC, IC, and ITMS provided an excellent system for detecting and analyzing all types of explosives used in Improvised Explosive Device (IED) manufacturing.

## References

- [1] Ronald L. Woodfin, Trace Chemical Sensing of Explosives, John Wiley & Sons, Inc., 2006.
- [2] B. A. Forensic Investigation of Explosions, CRC press, 2011.
- [3] 'Welcome to Belarus, Belarus during the Great Patriotic War - History.', 2008. .
- [4] 'Mine Warfare in Afghanistan', 2009. [Online]. Available: [http://www.history.navy.mil/wars/ in Afghanistan](http://www.history.navy.mil/wars/in%20Afghanistan)
- [5] "American Experience" Transcript, PBS', 2009. [Online]. Available: <https://www.pbs.org/wgbh/americanexperience/>.
- [6] Frontline world, "Suicide Terrorism: A Global Threat", 2009. .
- [7] Walsh M. E., Jenkins T. F., and Thorne P. G., 'Laboratory and analytical methods for explosives residues in soil', J. Energ. Mater., 1995, 13(3-4), 357-383.
- [8] Bjella KL, 'Pre-screening for explosives residues in soil prior to HPLC analysis utilizing Expray', Eng. Res. Dev. Cent. HANOVER NH COLD Reg. Res. Eng. LAB, 2005.
- [9] Frontanals F. B. N., Marce R.M., 'New Hydrophilic materials for solid-phase extraction.pdf', Trends Anal. Chem., 2005, 24(5), 394-406.
- [10] J. W. Matthew Smith, Greg E Collins, 'Microscale solid-phase extraction system for explosives', J. Chromatogr. A, 2003, 991(2), 159-167.
- [11] L. M. E. J. Fernández de Córdova M.L., Protocols for Extraction of Pesticide Residues., Sustainabl. 2021.
- [12] G E Ion Track Corporation, 'Total ion trap mobility spectrometry Itemizer 3 data sheet', Washington DC, 2009.
- [13] C. M. Caitlyn Norman, Brian McKirdy, Gillian Walker, Pat Dugard, Niamh Nic Daéid, 'Large-scale evaluation of ion mobility spectrometry for the rapid detection of synthetic cannabinoid receptor agonists in infused papers in prisons', Drug Test. Anal., 2021, 93(5), 2933-2941.
- [14] Agilent corporation, 'Total ion trap mobility spectrometry Itemizer 3 data sheet', Washington DC, 2009.
- [15] Gaurav P. K., D., Malik A. K., and Rai, 'High-Performance Liquid Chromatographic Methods for the Analysis of Explosives.', Crit. Rev. Anal. Chem., 2007, 37(4), 227-268.
- [16] H. P. Johns C, Shellie RA, Potter OG, O'Reilly JW, Hutchinson JP, Guijt RM, Breadmore MC, Hilder EF, Dicinoski GW, 'Identification of homemade inorganic explosives by ion chromatographic analysis of post-blast residues.', J. Chromatogr. A, 2008, 1182(2), 205-14.
- [17] Dicinoski P. R., Shellie G. W. and Haddad R. A., 'Forensic Identification of Inorganic Explosives by Ion Chromatography.', Anal. Lett., 2006, 39(4), 639-657.
- [18] Kolla P., 'Gas chromatography, liquid chromatography and ion chromatography adapted to the trace analysis of explosives.', J. Chromatogr. A, 1994, 674(1-2), 309-18.