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J F S R JOURNAL OF FORENSIC SCIENCE AND RESEARCH
ISSN 2575-0186

Research Article

Impact of Rainy Environments on Nitrate Ion Detection in Post-blast Soil Analysis: A Forensic Study

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Submitted: July 01, 2025
Approved: July 11, 2025
Published: July 14, 2025

How to cite this article: Kumar D, Malik A, Das R, Pawar PS. Impact of Rainy Environments on Nitrate Ion Detection in Post-blast Soil Analysis: A Forensic Study. J Forensic Sci Res. 2025; 9(2): 129-133. Available from: https://dx.doi.org/10.29328/journal.jfsr.1001092

Keywords: Rainy environment; Soil analysis; Nitrate ions; Ion chromatography; Post-blast residues

Abstract

The forensic identification of nitrate ions in post-blast pit soil samples is crucial for investigating nitrate-based explosive incidents owing to environmental factors such as rainfall and monsoon conditions, which can significantly alter the concentration and distribution of nitrate residues at blast sites, as nitrate ions are completely soluble in water. This study investigated the influence of rainfall on the retention and detectability of nitrate ions in pit soil collected from spiked simulated explosive samples, replicating the topographical conditions of hilly regions frequently impacted by insurgent and terrorist activities. Ion Chromatography (IC), a highly sensitive and selective analytical technique, was employed to quantify nitrate ion concentrations within the soil matrix. This study aimed to elucidate the mobility, leaching behavior, and retention of nitrate ions in soils affected by blast under natural drizzling rainfall conditions.

Introduction

Post-blast forensic investigation is critical for identifying inorganic explosive residues in debris and soil samples, which often comprise ions such as nitrates, chlorates, and perchlorates. Among these, nitrate ions are of particular forensic significance because of their widespread use in nitrate-based explosives, such as ammonium nitrate fuel oil (ANFO), urea nitrate, and black powder, which constitute the majority of cases investigated in India and globally because of their accessibility and cost-effectiveness. The detection of these ions provides vital evidence for establishing the use of inorganic explosives at a blast site, contributing significantly to criminal investigations and legal proceedings [1-7].

However, these ions are also commonly present in environmental matrices such as soil, dust, and debris because of the extensive use of nitrate-based fertilizers, further contributing to the background nitrate ion presence in soil samples, posing a challenge in distinguishing explosive-origin nitrates from agriculturally introduced ones.

This challenge becomes even more complicated during the rainy season, when rainwater can significantly influence the distribution and retention of explosive residues in post-blast scenes. Nitrate ions are highly water-soluble, making them particularly susceptible to dilution, leaching, and complete removal from blast sites by rainfall. Such environmental effects increase the risk of false-negative results or inconclusive findings during crime exhibit evaluation, further compromising legal outcomes. Therefore, the analysis of nitrate ions in pit soil during rainy conditions requires highly sensitive and selective analytical techniques capable of detecting trace-level concentrations and differentiating them from the background levels.

This study aimed to investigate the impact of rainfall on the persistence and detectability of nitrate ions in pit soil samples following a simulated blast involving nitrate-based explosives. The focus is on understanding the effects of rainwater on the retention or dispersion of nitrate residues in the blast residue, thereby influencing the reliability of post accounting for the events leading to the explosion. Among the various analytical techniques available, Ion Chromatography (IC) was employed in this study owing to its superior sensitivity, selectivity, and suitability for the trace-level quantitative analysis of inorganic ions [8,9]. The findings of this study are expected to inform the standardization of sampling and analytical protocols for forensic laboratories, particularly in regions experiencing seasonal or heavy rainfall, and to enhance the accuracy of post-blast nitrate analysis in real-world casework.

Objective

This study aimed to systematically examine the effects of rainfall and monsoon conditions on nitrate ion concentrations in pit soil samples using Ion Chromatography (IC) to enhance forensic investigations. The samples were collected after spiking Potassium Nitrate in the simulated pits by mimicking the topographical conditions of hilly regions that are often affected by explosions due to insurgent and terrorist activities.

Materials and methods

Demineralised water (DM) was procured from Labogen Fine Chem Industry, Ludhiana, along with 0.22 µm syringe filters and filter paper obtained from Clairofilt (India), Bhiwandi. For Ion Chromatography calibration, standard solutions of inorganic anions (F^-, Cl^-, NO_2^-, SO_4^{2-}, PO_4^{3-}) were prepared from 10 ppm stock solutions (Sigma Aldrich, Switzerland). The eluent was prepared using AR grade Sodium Bicarbonate and Sodium Carbonate (Merck, Germany), and Acetone (Advent Chembio, Navi Mumbai). Sulphuric acid solution (Sigma Aldrich) was used for suppressor regeneration. Analysis was performed on a Metrohm Advanced Ion Chromatography system with a Metrosep A Supp 19 ion exchange column, packed with a hydrophilic polystyrene/divinylbenzene copolymer containing quaternary ammonium groups [8,9].

Procedure for obtaining spiked soil samples

To simulate the conditions commonly observed in post-blast forensic investigations in hilly and conflict-sensitive regions, especially those affected by Naxalite and terrorist activities, two experimental soil pits were constructed on a sloped surface at the Central Forensic Science Laboratory Pune, each measuring 12 inches in diameter and 6 inches in depth. One pit was designated as the exploded/simulated pit, while the other served as a control to establish the baseline conditions. The exploded pit was spiked with 1 gram of potassium nitrate (KNO_3) of known purity and concentration to simulate the post-blast residue. Subsequently, both pits were filled to 75% of it's volume with water.

Both pits were subjected to natural drizzling rainfall for a week. During this period, a gradual decline in the water level was observed owing to rainwater percolation, leaching, and partial evaporation. Reduced water was replenished with natural rainwater to maintain real environmental conditions. After one week, no further water accumulation or wiping was noted and both pits were left undisturbed to allow for natural drying (Figures 1,2).

Sample collection

After the pits were completely dried, soil samples were collected for subsequent chemical and instrumental analyses. Approximately 100–120 grams of soil was sampled from two distinct layers - surface and subsurface of both the exploded/simulated and control pits. These samples were dried at room temperature and stored in clean, labelled containers.

Sample preparation

The collected soil sample from the top layer of the simulated pit was marked as SPT and the bottom layer of the same was marked as SPB. Similarly the Top and Bottom layers of the controlled pit soils were marked as CPT and CPB respectively. A standard Potassium Nitrate sample of 1g having known concentration was labelled as Standard and a blank DM water sample was labelled as Blank.

To extract nitrate ions from the soil samples, DM Water was added to beakers containing pre-weighed portions of samples – 100ml each for SPT, SPB, CPT, CPB and Standard. The extracts were filtered using filter papers. Each sample was then washed thoroughly with 100ml of DM water, thrice, to ensure complete recovery of nitrate content and the combined filtrates collected from all the samples were evaporated to dryness. It was then reconstituted in 100 ml

Figure 1: Pictorial representation of the simulated pit.

Figure 2: Pictorial representation of the control pit.

of DM Water, followed by thorough mixing for homogeneity. Syringe filtration was conducted to remove fine particulates and 1ml of the filtrate was collected and diluted tenfold with DM water for each sample. The diluted extract was used for IC analysis of the nitrate ions [7-13].

Methodology used in ion chromatography

The ion chromatography (IC) system consisted of a Metrohm 830 IC interface, 819 conductivity detector, 820 column thermostat, 818 IC pump, a separation column, and a Metrosep A SUPP 4/5 guard column. For anionic separation, a Metrosep A Supp 19 column (150 × 4.0 mm, 4.6 µm particle size) was used. The column was packed with a hydrophilic polystyrene/divinylbenzene copolymer, which was functionalized with quaternary ammonium groups. The mobile phase flowed at a rate of 0.7 mL/min, with an eluent composed of 8.0 mM sodium carbonate (Na_2CO_3) and 0.25 mM sodium bicarbonate ($NaHCO_3$) [8-10].

Observation

The data obtained using ion chromatography for the samples are listed in the given table (Table 1) (Figures 3-7).

Table 1: Data obtained for various samples.

Sample type	Area	Height	Concentration
Standard	75.8899	199.207	842.522
Simulated Pit Top Layer (SPT)	3.4739	6.321	38.242
Simulated Pit Bottom Layer (SPB)	2.2790	4.089	24.971
Control Pit Top Layer (CPT)	1.3197	2.473	14.316
Control Pit Bottom Layer (CPB)	0.7852	1.384	8.381

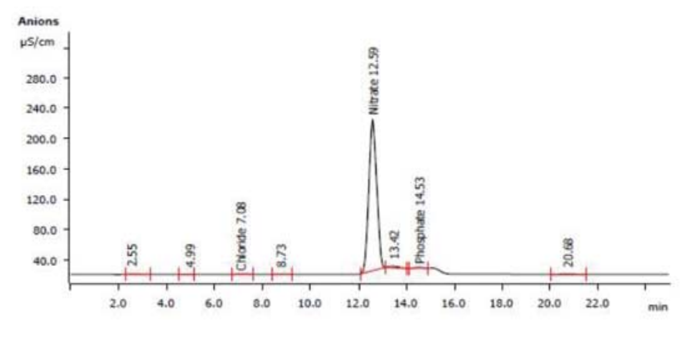

Figure 3: Ion Chromatogram obtained for Standard (Nitrate).

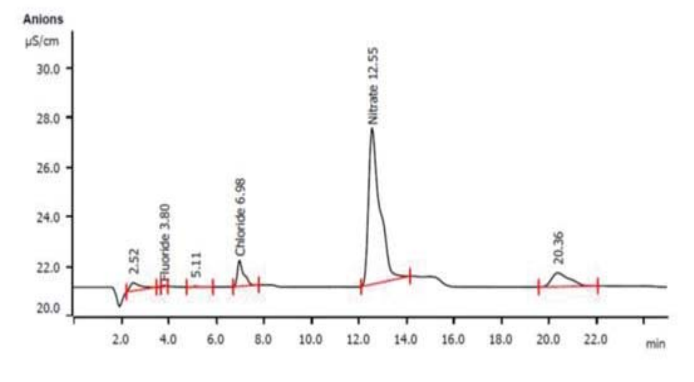

Figure 4: Ion Chromatogram obtained for Simulated Pit Top Layer (SPT).

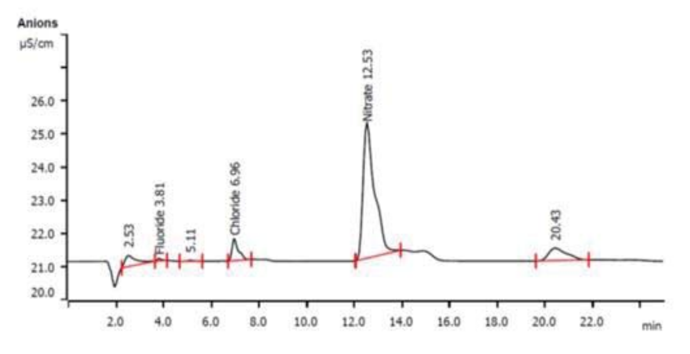

Figure 5: Ion Chromatogram obtained for Simulated Pir Bottom Layer (SPB).

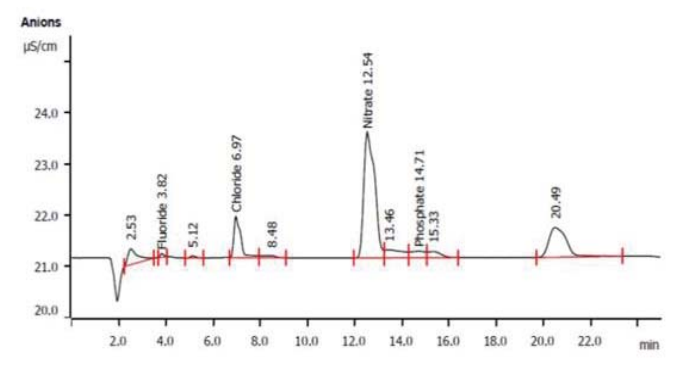

Figure 6: Ion Chromatogram obtained for Control Pit Top Layer (CPT).

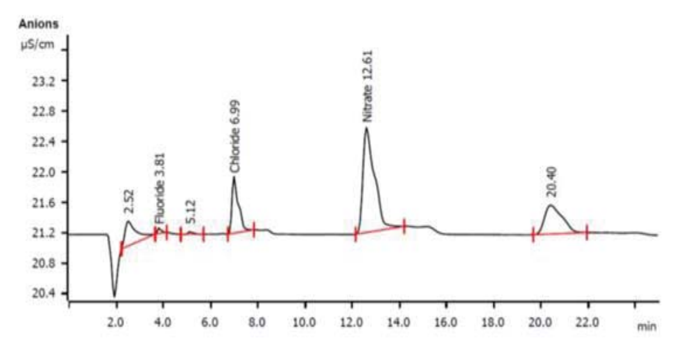

Figure 7: Ion Chromatogram obtained for Control Pit Bottom Layer (CPB).

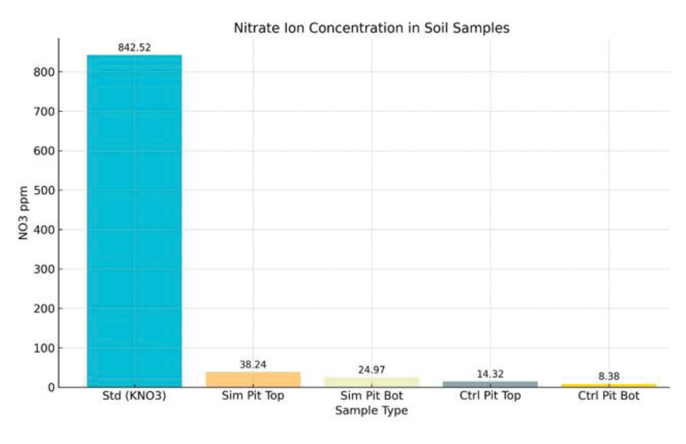

Figure 8: Bar chart showing nitrate ion concentrations in different soil samples.

Results and discussion

Ion chromatography findings

Ion chromatography (IC) was employed to quantify nitrate ion concentrations in soil samples from both simulated post-blast (spiked) and control pits, after exposure to natural drizzling rainfall for one week. The retention times for nitrate ions in all samples (SPT, SPB, CPT and CPB) matched that of the standard potassium nitrate, confirming the presence and correct identification of nitrate ions.

Quantitative results:

- Standard Potassium Nitrate: 842.522 ppm
- Simulated Pit Top Layer (SPT): 38.242 ppm
- Simulated Pit Bottom Layer (SPB): 24.971 ppm
- Control Pit Top Layer (CPT): 14.316 ppm
- Control Pit Bottom Layer (CPB): 8.381 ppm

Graphical representation of results:

Below is a graphical summary of the nitrate ion concentrations measured in the soil samples. The samples include:

- Standard (KNO_3)
- Simulated Pit Top (SPT)
- Simulated Pit Bottom (SPB)
- Control Pit Top (CPT)
- Control Pit Bottom (CPB)

This visual representation allows for a clear comparison of nitrate retention across the different experimental conditions, as shown in Figure 8.

Key observations from the chart:

- The standard potassium nitrate sample showed the highest concentration, serving as a reference for the initial spiking amount.
- Both simulated pit layers (SPT and SPB) had significantly higher nitrate concentrations than the control pit layers, confirming the successful detection of spiked nitrate despite rainfall.
- The top layers (SPT and CPT) had higher concentrations than their respective bottom layers (SPB and CPB), indicating some downward leaching but incomplete percolation.
- The control pit samples showed lower nitrate levels, reflecting background environmental nitrate rather than explosive residues.

This chart visually demonstrates the impact of rainy conditions on nitrate ion retention and the importance of prompt forensic sampling in these environments.

Interpretation of results

Nitrate ion distribution:

Higher in simulated pit: Both the top and bottom layers of the simulated pit showed significantly higher nitrate concentrations than those in the control pit. This confirms that the spiked potassium nitrate was successfully detected after exposure to rain, despite the expected leaching.

Layer differences: The top layer of each pit (SPT, CPT) had higher nitrate concentrations than the corresponding bottom layers (SPB, CPB), suggesting some downward movement (leaching) and possible surface retention due to incomplete percolation or evaporation effects.

Leaching and rainfall effects:

Leaching evident: The presence of nitrate in the bottom layer (SPB) of the simulated pit indicates that rainwater facilitated the downward movement of nitrate ions. However, the concentration in the bottom layer was less than that in the top layer, indicating that not all nitrate was leached away.

Loss due to overflow: The total nitrate recovered from both layers of the simulated pit was much less than the original amount spiked (1g KNO_3 = 842.522 ppm standard), indicating that a significant portion of nitrate was likely lost due to overflow and runoff, especially given the sloped pit design and continuous rainfall.

Background nitrate:

Control pit observations: The control pit, which was not spiked, still contained measurable nitrate levels, attributed to environmental sources such as fertilizers or natural soil content. This highlights the challenge in forensic differentiation between explosive-origin and background nitrate ion in real-world scenarios.

Forensic implications

Rainy environment challenges:

Risk of false negatives: The high solubility and mobility of nitrate ions mean that rainfall can rapidly dilute and remove explosive residues from soil, potentially leading to false negatives or inconclusive results in forensic investigations.

Importance of scene preservation: The findings underscore the need to isolate and preserve blast scenes promptly, especially during rainy conditions, to prevent loss of evidence and it's contamination.

Analytical sensitivity

Role of ion chromatography: The use of IC proved effective for detecting even trace levels of nitrate ions, supporting its suitability for post-blast forensic analysis in challenging environmental conditions.

Recommendations

Crime scene management: Forensic teams should prioritize the rapid collection and protection of soil samples at blast sites, especially during monsoon or rainy seasons.

Protocol development: This study supports the development of standardized protocols for sampling and analysing post-blast residues in environments prone to rainfall, to improve the reliability of forensic evidence.

Conclusion

This study clearly demonstrates that rainy conditions significantly impact the retention and detectability of nitrate ions in post-blast soils. While some nitrate persists and can be detected using sensitive methods such as IC, a substantial portion is lost due to leaching and runoff. This highlights the forensic importance of rapid scene management and the challenges posed by environmental factors in the analysis of explosive residues. The results advocate for enhanced protocols and awareness in forensic practice, especially in regions with frequent rainfall.

Acknowledgement

We would like to thank the Director of the Central Forensic Science Laboratory, Pune, for providing the necessary support and infrastructure for conducting this study.

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