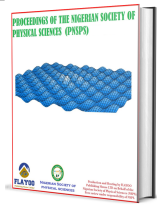
Published by Nigerian Society of Physical Sciences. Hosted by FLAYOO Publishing House LTD

Proceedings of the Nigerian Society of Physical Sciences

Journal Homepage: https://flayoophl.com/journals/index.php/pnspsc



Forensic investigation of flagged import goods

T. Muhammad[a,*], D. J. Adeyemo[b], J. A. Yusuf[b], D. B. Hasan[c], A. Said[c]

[a]*Nuclear Science and Technology Section, Centre for Energy Research and Training, Ahmadu Bello University, Zaria, 810261, Kaduna State, Nigeria*
[b]*Health Physics and Radiation Biophysics Section, Centre for Energy Research and Training, Ahmadu Bello University, Zaria, 810261, Kaduna State, Nigeria*
[c]*Material Science and Development Section, Centre for Energy Research and Training, Ahmadu Bello University, Zaria, 810261, Kaduna State, Nigeria*

ABSTRACT

Trade is central to social and economic activity, but imported goods must be screened carefully to protect public safety and national security. In this study, a consignment declared as household products was flagged by national security agencies for forensic investigation. Initial field tests at the port of entry used portable X-ray fluorescence (XRF), spectroscopic screening, and radiation surveys. The radiation survey showed no significant deviation from background levels, and the initial XRF measurements did not reveal suspicious elements. Spectroscopic screening, however, indicated high nitrate/nitrite levels in a material labelled as bath salt, where such levels were not expected. The samples were therefore subjected to laboratory analysis by high-accuracy energy-dispersive X-ray fluorescence (EDXRF), instrumental neutron activation analysis (INAA), ultraviolet–visible (UV–Vis) spectroscopy, gas chromatography–mass spectrometry (GC–MS), and Fourier-transform infrared (FTIR) spectroscopy. INAA and XRF showed that the product labelled as bath salt (magnesium sulphate) contained magnesium at only about 0.23% and sulphur at 6.27%, which is inconsistent with the declared composition. UV–Vis spectroscopy confirmed nitrate/nitrite at 15.7% (m/v), while FTIR revealed nitrate absorptions together with hydrocarbon and amine features. GC–MS supported these findings by identifying complex hydrocarbon chains and related fragmentation patterns. Taken together, the results showed that the flagged goods were not household products but hazardous materials with potential ballistic applications. The investigation provided critical support to national security infrastructure by preventing suspicious materials from bypassing regulatory control.

Keywords: Forensic analysis, XRF, INAA, FTIR, GC–MS, Ballistic material, Safeguards.

DOI:10.61298/pnspsc.2026.3.268

1. INTRODUCTION

The movement of goods across state borders requires rigorous inspection to safeguard national security and public safety. Discrepancies between declared shipping manifests and the actual contents of consignments can lead to cargo being flagged for further forensic analysis to determine the true nature of the materials involved.

In this study, a 20-foot shipping container docked at a national point of entry was flagged by Nigerian security authorities. The shipping manifest listed household cleaning products and cosmetics, along with materials for recreational smoking (shisha). The aim of the study was to apply forensic science techniques

*Corresponding author Tel. No.: +234-806-2394-919.
e-mail: tmuhammad@abu.edu.ng (T. Muhammad)

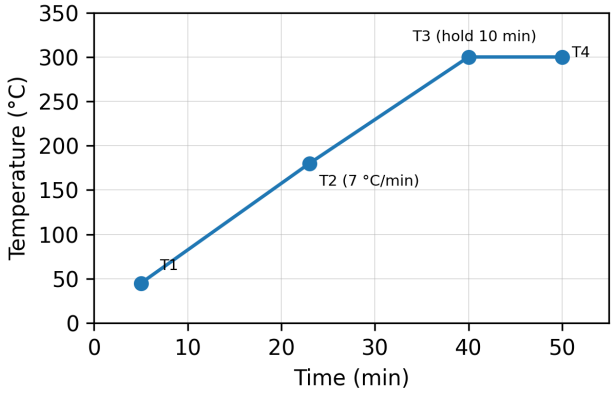


Figure 1. GC oven temperature profile [7].

Table 1. Results of spectrophotometric analysis of nitrate/nitrite.

Sample	Sample name	Result	Stdev	Mda	Unit
1	Cosmetic A	29.9	1.87	29.0	mg kg^{-1}
2	Cosmetic B	16.8	1.03	17.0	mg kg^{-1}
3	Cosmetic C	9.17	0.47	9.50	mg kg^{-1}
4	Cosmetic D	13.6	0.61	13.2	mg kg^{-1}
5	Cleaner A	8.67	0.47	9.00	mg kg^{-1}
6	Cleaner B	7.85	0.41	7.56	mg kg^{-1}
7	Bath salt	15.7	0.08	15.7	% (m/v)
8	Aluminium foil	32.5	3.89	34.9	mg kg^{-1}
9	Charcoal	5.00	0.00	5.00	mg kg^{-1}
10	Fuel tablet	48.7	0.62	48.5	mg kg^{-1}

to investigate whether the consignment contained materials that posed safety or security concerns. Radiological surveys, spectroscopic screening, and chemical tests were first conducted on-site to rule out immediate hazards.

Preliminary field tests revealed inconsistencies in nitrate/nitrite concentrations and elemental composition that did not match the manifest. These findings led to comprehensive laboratory investigations using standardized analytical methods.

The integration of energy-dispersive X-ray fluorescence spectroscopy (EDXRF), instrumental neutron activation analysis (INAA), ultraviolet–visible spectroscopy (UV–Vis), Fourier-transform infrared spectroscopy (FTIR), and gas chromatography–mass spectrometry (GC–MS) demonstrates how conventional analytical science can assist frontline security personnel in identifying disguised materials and strengthening national safeguards against illicit and dangerous imports.

2. MATERIALS AND METHODS

Materials selected for further investigation were chosen on the basis of the field-screening results and observable physical variation. All sample collection and preparation steps were performed in triplicate to ensure precision [1–3]. Particular emphasis was placed on nitrate/nitrite species because of their relevance as precursors in improvised explosive materials. Nitrate/nitrite species were measured using a CHEMetrics V-2000 photometer with Vacu-vials ampoules. Nitrate ions were reduced to nitrite in the presence of cadmium, forming chromophore complexes detected at specific wavelengths.

Elemental analysis was performed by XRF and INAA. Both

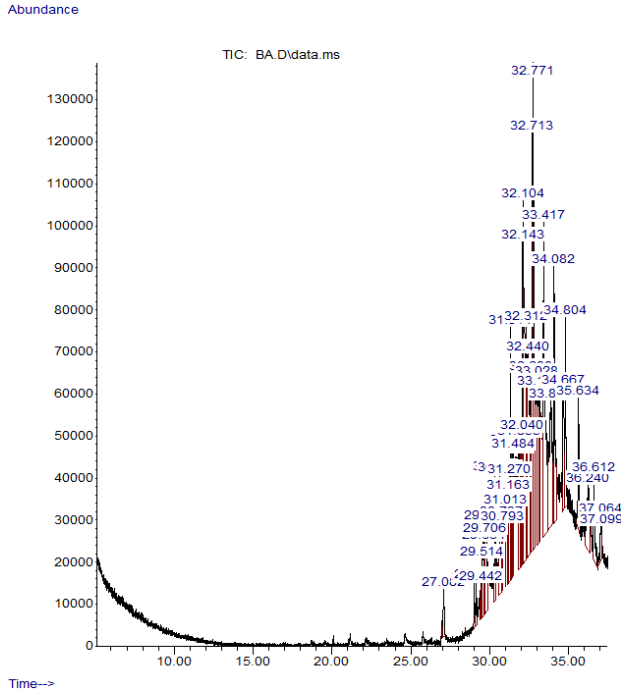


Figure 2. Bath salt sample chromatogram.

techniques require limited sample preparation and are non-destructive, thereby supporting bulk analysis and minimizing blank-related errors. Solid and liquid samples were prepared using standard pelletizing and containment methods. The results were validated against certified reference materials (CRMs), namely NIST 1633C and WEPAL ISE-3 [4].

Further investigations were carried out using mid-infrared scans from 4000 to 400 cm^{-1} to identify molecular vibrations associated with nitro groups, hydrocarbons, amines, and related functional species. Solid samples were analysed using potassium bromide (KBr) pellets [5]. Evidence of organic materials from FTIR analysis led to additional investigations by GC–MS. Components were separated on a non-polar capillary column with helium as the carrier gas, followed by electron-impact ionization. Identifications were based on retention times, mass-to-charge ratios, and National Institute of Standards and Technology (NIST) spectral-library comparisons [6, 7].

In total, 26 samples were processed with minimal contamination risk. Solutions were prepared at 1000 ppm where applicable, using dissolution protocols tailored to solids, liquids, and metals. Each solution was prepared in triplicate to ensure reproducibility, following relevant standard methods [6–8].

3. RESULTS AND DISCUSSION

Spectrophotometric analysis showed that most samples, including cosmetics, cleaning agents, aluminium foil, charcoal, and fuel tablets, contained nitrate/nitrite concentrations within normal background ranges. In contrast, the sample labelled as bath salt showed a nitrate/nitrite content of 15.7% (m/v), far exceeding the levels attributable to natural variation or manufacturing impurities.

Table 1 presents the spectrophotometric results for nitrate/nitrite species in the samples. Standard deviation (Stdev)

Table 2. CRM validation by Z-score for WEPAL ISE 2017 and NIST 1633C.

WEPAL ISE 2017					NIST 1633C				
Element	This work	Reported value	Stdev	Z-score	Element	This work	Reported value	Stdev	Z-score
Al	89340	91800	4950	0	Al	13210	13280	610	0
As	29	27.7	1.29	1	As	189	182.6	3	2
Br	6.4	7.66	1.40	-1	Ba	1615	1126	330	1
Ca	5938	6790	481	-2	Ca	13050	13650	400	2
Fe	56082	52800	2190	1	Cr	267	258	6	-2
K	21590	20491	886	1	Cs	8.89	9.39	0.22	-1
Mn	452.3	455	26.2	0	Dy	18.7	18.7	0.3	0
Rb	124.3	129	5.23	-1	Fe	104600	104900	3900	0
Th	13.5	13.9	0.735	-1	K	17890	17730	66	2
V	127.7	147	7.41	-3	La	86.5	87	2.6	0
					Mg	6846	5980	520	2
					Mn	234	240.2	34	0
					Na	1657	1707	59	-1
					P	1872	1920	100	0
					Pb	102	95.2	2.5	3
					Rb	117	117.42	0.53	-1
					S	1054	1100	19	-1
					Sc	37.1	37.6	0.6	-1
					Si	226500	213000	5700	2
					Ta	1.35	1.58	0.03	-1
					Tb	2.99	3.12	0.06	-2
					Th	23.5	23	0.4	1
					Ti	738.2	724	30	0
					V	3060	2860	79	3

Table 3. Elemental analysis of the study samples.

Element	Cosmetic A	Cosmetic B	Cosmetic C	Cosmetic D	Cleaner A	Cleaner B	Bath salt	Foil	Charcoal
Fe	1.66	3.18	0.02	BDL	3.66	20.4	89.1	34.0	9400
Cu	0.55	125	BDL	0.42	145	1.01	5.30	0.01	78.7
Zn	1.11	1.05	0.01	BDL	0.65	1.25	15.5	0.16	127
Al	308	231	2.50	BDL	326	363	BDL	975000	6500
Mg	1181	938	11.2	995	1200	1300	2300	124	7100
S	439	370	60.8	4200	710	567	62700	2.31	9100
P	BDL	BDL	BDL	5.30	BDL	34.8	81.0	BDL	1800
Ca	7.02	12.2	1.95	47.3	35.7	93.4	166	0.39	27700
K	3.20	77.0	0.20	12.9	8.63	16.9	BDL	BDL	18900
Mn	BDL	BDL	BDL	BDL	BDL	1.27	BDL	8.27	646
Br	BDL	1.14	0.28	7.00	BDL	BDL	2.12	0.31	13.6
Cl	31.9	38.5	43.3	2400	82.0	18.5	668	BDL	9100
V	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.87	11.7
Na	250	116	82.4	3700	118	173	BDL	BDL	BDL
Ti	BDL	BDL	BDL	BDL	BDL	BDL	800	1.04	301

BDL = below detection limit.

Table 4. Absorption intensities from FTIR analysis.

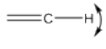
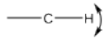
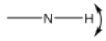
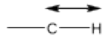
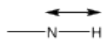
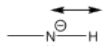
S/N	Peak	Intensity (% transmittance)	Functional group	Representative structure
1	501 cm^{-1} 663 cm^{-1} 794 cm^{-1} 1003 cm^{-1}	49.3 39.7 42.0 9.77	Fingerprint region for sp^2 alkene bending	
2	1234 cm^{-1} 1442 cm^{-1}	32.2 76.6	Nitro group	
3	1365 cm^{-1}	57.0	sp^3 alkane bending	
4	1620 cm^{-1}	71.0	Amine bending	
5	2874 cm^{-1} 2924 cm^{-1}	28.5 25.7	sp^3 alkane stretching	
6	2144 cm^{-1}	67.0	Amine stretching	
7	3958 cm^{-1}	43.1	Free anionic amine	

Table 5. GC chromatogram intensities.

PK	RT	Area (%)	Library/ID	CAS
26	32.103	3.3563	Heneicosane	000629-94-7
27	32.146	8.2511	trans-13-Octadecenoic acid	000693-71-0
28	32.310	4.2148	Methyl stearate	000112-61-8
29	32.442	5.8921	Ergost-22-en-3-ol, [3α,5β,22E]; Heneicosenol	055527-92-9
31	32.712	6.4660	Hexacosane; Heneicosenate	000630-01-3
32	32.771	7.0013	Docosane	000629-97-0
35	33.417	7.8493	Eicosane	000112-95-8
36	33.888	5.7615	Hexahydropyridine, 1-methyl-4-[4,5-dihydroxyphenyl]; Eicosane	094427-47-1
37	34.082	4.0064	Eicosane	000112-95-8
38	34.666	3.0405	1-Bromoeicosane; Eicosane	004276-49-7

and median (Mda) are reported as dispersion and location statistics, respectively, at a 95% confidence interval.

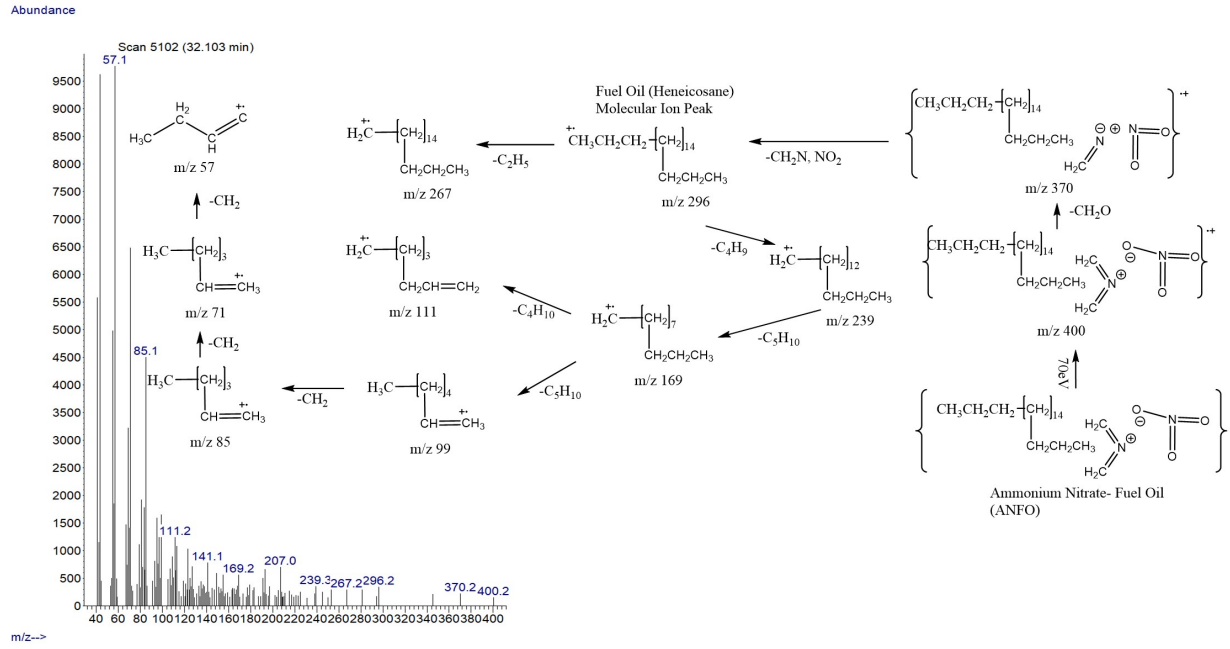


Figure 3. Fragmentation pattern of the compound at RT 32.103 min.

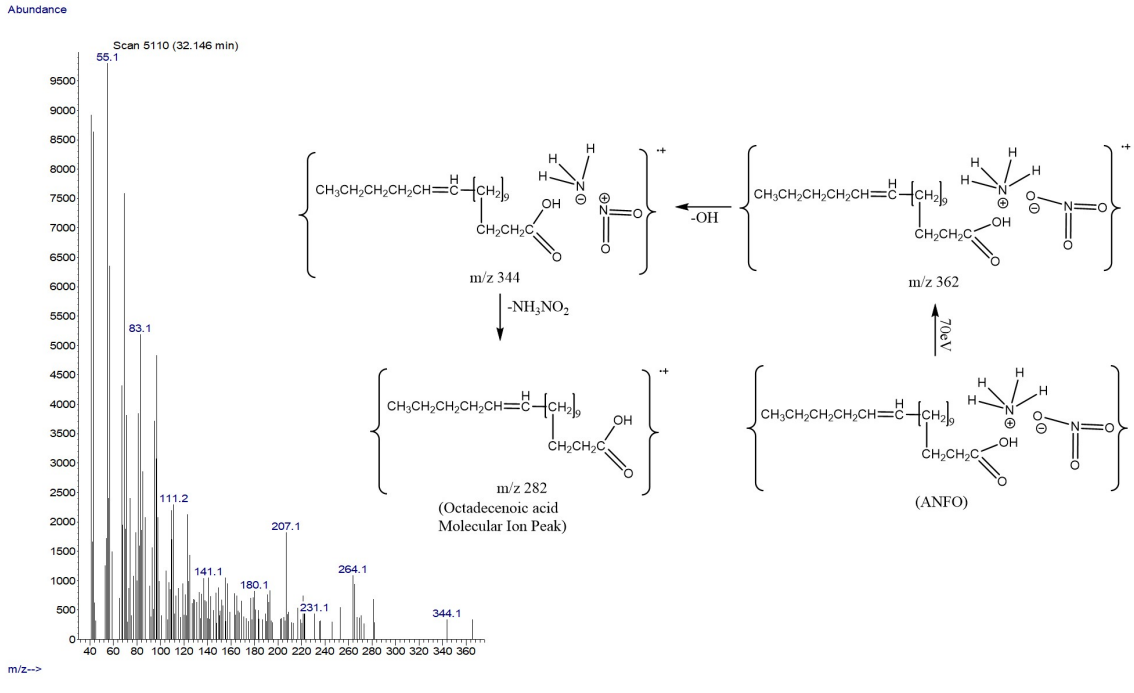


Figure 4. Fragmentation pattern of the compound at RT 32.146 min.

Table 1 shows that most samples contained nitrate/nitrite concentrations in the parts-per-million range, consistent with expected environmental background levels and trace impurities introduced during manufacturing. These values are relatively low and did not indicate immediate concern.

The bath salt sample, however, was an outlier, with a nitrate/nitrite concentration of 15.7% (m/v), approximately 10,000 times higher than the nominal background represented by the other samples. This anomaly indicated the deliberate presence of nitrate/nitrite-containing compounds. Because bath salt labelled as magnesium sulphate ($MgSO_4$) should not contain substantial nitrate/nitrite, and because this component was not declared in the shipping manifest, the product was treated as suspicious and potentially mislabelled.

Tables 2 summarizes CRM validation by Z-score. ‘‘This work’’ gives the measured value, ‘‘Reported value’’ gives the certified or reported mean, and ‘‘Stdev’’ gives the standard deviation of the certified mean at a 95% confidence interval. The

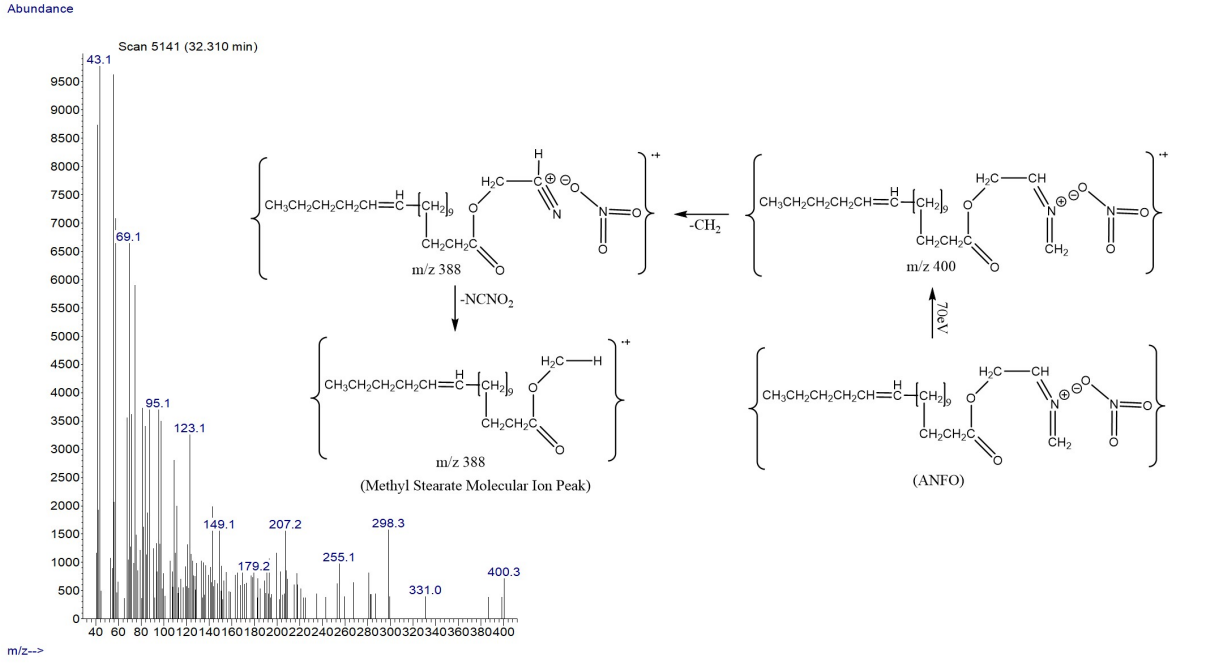


Figure 5. Fragmentation pattern of the compound at RT 32.310 min.

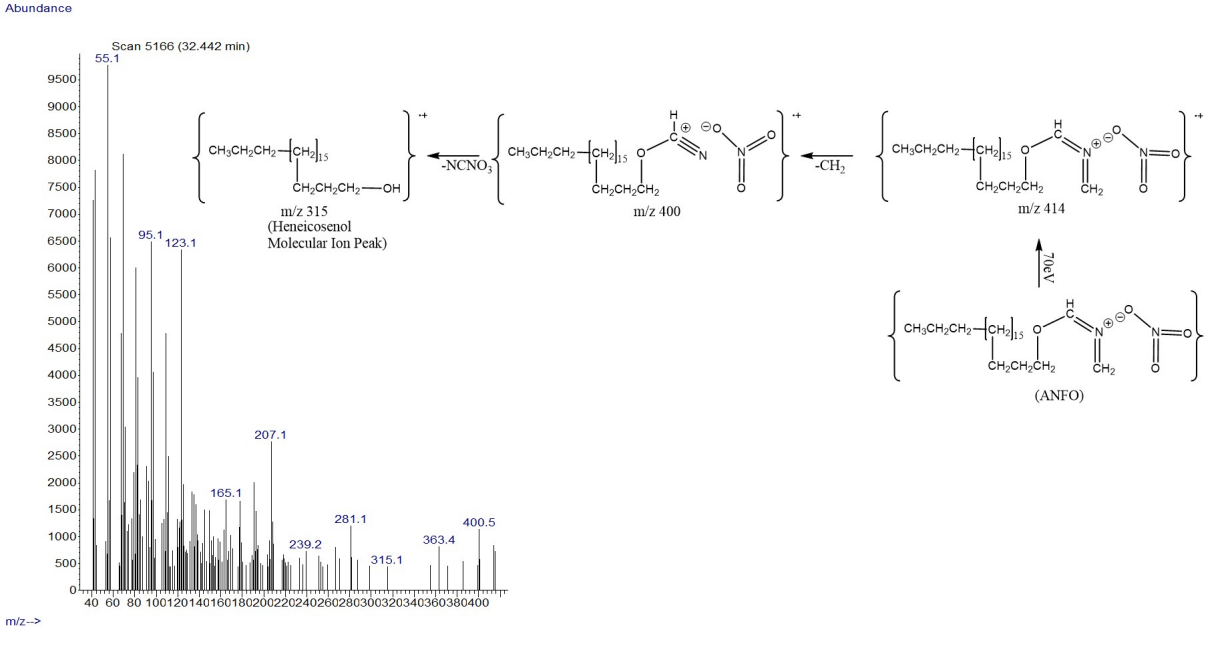


Figure 6. Fragmentation pattern of the compound at RT 32.442 min.

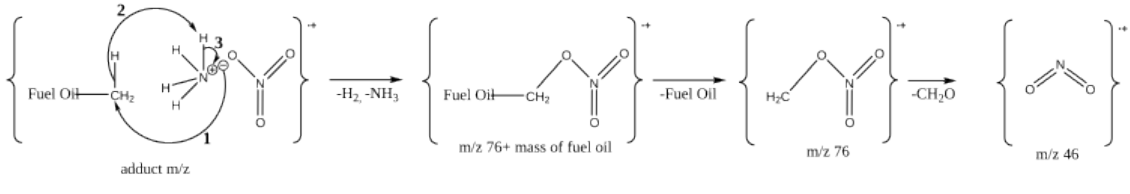


Figure 7. Characteristic fragments associated with long-chain fuel-oil components.

elemental analysis of the study samples is presented in Table 3.

Statistical validation using Z-scores produced values mostly between −3 and +3, confirming the accuracy of the analytical methods. Al, Mn, and Dy agreed closely with reference values,

while Ca and V in WEPAL ISE showed minor deviations that may be attributable to weak gamma signals, low isotopic abundance, and instrumental sensitivity limits.

For the bath salt-related elements, Mg and S in the NIST 1633C dataset gave Z-scores of +2 and −1, respectively. Both values are within acceptable limits and support the reliability of the methods used to evaluate the sample set.

The elemental composition of the sample set (Table 3) shows that the cosmetic products contained very low concentrations of Fe, Cu, Zn, Al, Mg, and S. The cleaning products showed moderate Mg, Ca, and Cl levels, consistent with detergent formulations. Aluminium foil showed 97.5% aluminium, confirming its authenticity, while charcoal contained Fe, Ca, and K at 0.94%, 2.77%, and 1.89%, respectively, values consistent with reported literature [9]. These samples therefore showed no evidence of adulteration.

The bath salt sample again stood out. Although it contained sulphur at 6.27%, earlier spectroscopic analysis indicated that the sulphur was not present as sulphate. In addition, Mg was present at only about 0.23%, whereas a genuine magnesium sulphate product would be expected to contain substantial proportions of magnesium, sulphur, and oxygen.

Further studies of the flagged sample by FTIR revealed nitro groups, alkane stretches, and amine vibrations, consistent with organic nitrogen-containing compounds. GC–MS analysis showed an amorphous mixture containing long-chain hydrocarbons and unsaturated fatty acids. Fragmentation-pattern studies supported the presence of heteroatom-containing compounds typical of fuel oils. Comparison with the NIST database indicated that the material was consistent with an ammonium nitrate–fuel oil (ANFO)-type material [10–13].

FTIR functional-group results are presented in Table 4. Duplicate analyses yielded a relative standard deviation of 2.83. The sample chromatogram is shown in Fig. 2, and the most abundant GC–MS peaks are listed in Table 5. Fragmentation-pattern reconstruction is shown in Figs. 3–6.

The infrared spectroscopy results (Table 4) showed N–H stretching near 3400 cm^{-1}, C–H stretching near 2900 cm^{-1}, and N=O absorption near 1000 cm^{-1}, indicating organic nitrate species. The C–H doublet near 2900 cm^{-1} also suggests an asymmetrical molecular environment. The chromatograms (Fig. 2) confirm that the sample was not homogeneous but was instead an admixture, with separations occurring between 27 and 37 min. Fragmentation studies established that the sample largely contained long-chain (C_{11}–C_{28}) species with branching, unsaturation, and heteroatoms. This interpretation is further supported by principal fragments at m/z 46 and 76, which are consistent with markers for long-chain hydrocarbons, as illustrated in Fig. 7.

The combined data for the bath salt sample indicate that it was not a cosmetic or cleaning product, as implied by its label, but a controlled or hazardous material concealed among household cosmetics and cleaning products. ANFO is a secondary explosive used primarily in military and industrial applications. Mislabelling of such materials has been documented as a means of evading regulatory control and enabling malicious applications [17].

4. CONCLUSION

A shipment declared as household cleaning and cosmetic products raised suspicion when a mislabelled material was discovered among the goods. To determine its true identity, this study employed a suite of analytical techniques. UV–Vis spectrophotometry revealed unusually high nitrate/nitrite levels, while elemental analysis contradicted the declared composition by showing low magnesium and an unexpected sulphur profile for a product labelled as magnesium sulphate. These inconsistencies prompted deeper inquiry.

FTIR and GC–MS provided decisive evidence that the material was not a benign household product but an organic nitrate-containing material with signatures consistent with an ANFO-type explosive mixture. This study demonstrates that the combined use of forensic science and advanced chemical analysis is essential for identifying hazardous materials disguised as consumer products. Beyond its immediate forensic value, the study underscores the importance of integrated analytical capability in safeguarding national security and disrupting the illicit movement of hazardous materials.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

FUNDING

The authors declare that no funding was received during the preparation of this manuscript.

References

[1] APHA, *Standard methods for the examination of water and wastewater*, 21st ed., Method 5400-NO_3-E, 2005. https://www.nemi.gov/methods/method_summary/4708/

[2] ASTM International, *Standard test methods for Nitrite-Nitrogen and Nitrate-Nitrogen in water*, ASTM D3867-04, 2004. https://doi.org/10.1520/D3867-04

[3] CHEMetrics Inc., "Self-filling ampoules" (Vacu-Vials™), U.S. Patent No. 3,634,038, 1972. https://www.chemetrics.com/product/nitrate-hr-chemets-kit/

[4] J. Coates, "Interpretation of infrared spectra, a practical approach," Encyclopedia of Analytical Chemistry **12** (2000) 10815. https://doi.org/10.1002/9780470027318.a5606

[5] "Thermo Fisher Scientific, OMNIC FTIR software training," 2021. https://www.thermofisher.com/us/en/home/industrial/spectroscopy-elemental-isotope-analysis/molecular-spectroscopy/fourier-transform-infrared-spectroscopy/software/omnic-software-training.html

[6] Y. Kaplan and R. Garnica, "Improved sensitivity and dynamic range using the Clarus SQ 8 GC/MS system for EPA Method 8270D semi-volatile organic compound analysis," Gas Chromatography/Mass Spectrometry Application Note, PerkinElmer Lifesciences, Boston, MA, 2011. https://www.perkinelmer.com/CMSResources/Images/44-132032APP

[7] U.S. Environmental Protection Agency, "Semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS)," Method 8270D, 2014. https://19january2017snapshot.epa.gov/sites/production/files/2015-12/documents/8270d.pdf

[8] W. L. F. Armarego, *Purification of laboratory chemicals: 9th ed Part 2: Inorganic Chemicals, Catalysts, Biochemicals, Physiologically Active Chemicals, Nanomaterials*, Butterworth-Heinemann, 2022. https://www.inspectioncopy.elsevier.com/book/details/9780323909686

[9] C. O. Ijagbemi, S. O. Adepo & K. S. Ademol, ''Evaluation of combustion characteristic of charcoal from different tropical wood species,'' IOSR Journal of Engineering **4** (2014) 50. http://196.220.128.81:8080/xmlui/handle/123456789/3599

[10] K. Nakagawa, A. Hiroki, B. Ronny, T. Yuji & H. Takahiro, ''Use of sterols to monitor surface water quality change and nitrate pollution source,'' Ecological Indicators **107** (2019) 105534. https://doi.org/10.1016/j.ecolind.2019.105534

[11] M. Fernández-Martínez, J. Corbera, X. Domene, F. Sayol, F. Sabater & C. Preece, ''Nitrate pollution reduces bryophyte diversity in Mediterranean springs,'' Science of the Total Environment **705** (2020) 135823. https://doi.org/10.1016/j.scitotenv.2019.135823

[12] E. Rosenthal, M. Magaritz, D. Ronen & R. Roded, ''Origin of nitrates in the Negev Desert, Israel,'' Applied Geochemistry **2** (1987) 347. https://doi.org/10.1016/0883-2927(87)90050-3

[13] G. E. Ericksen, ''The Chilean nitrate deposits'', American Scientist **71** (1983) 366. https://www.jstor.org/stable/27852136

[14] B. Laffon, ''Fuel oils,'' in: P. Wexler, *Encyclopedia of toxicology*, 3rd ed., Academic Press, Cambridge, United States, 2014, pp. 667–670. https://doi.org/10.1016/B978-0-12-386454-3.00024-5

[15] Z. X. Zhang, ''Explosives and detonators,'' in: *Rock Fracture and Blasting*, Butterworth-Heinemann, Oxford, United Kingdom, 2016, pp. 179–195. https://doi.org/10.1016/B978-0-12-802688-5.00008-7

[16] M. A. Cook, *The science of high explosives*, American Chemical Society Monograph Series, No. 139, 1958. https://archive.org/details/the-science-of-explosives

[17] National ''Academies of Sciences, Engineering, and Medicine,'' *Reducing the threat of improvised explosive device attacks by restricting access to explosive precursor chemicals*, National Academies Press, Washington, DC, 2018. https://doi.org/10.17226/24862