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Application of Spectral Libraries for Characterization of Oxidizers in Post-Blast Residues by Electrospray Mass Spectrometry

ABSTRACT: The goal of this paper is to demonstrate that electrospray mass spectrometry when used with spectral libraries becomes a useful method for rapid identification of inorganic oxidizers commonly present in commercial and improvised explosives.

Electrospray ionization mass spectra (ESI-MS) of oxidizers reveal a series of characteristic cluster ions. Such a set of cluster ions can be regarded as a "fingerprint" of a particular oxidizer. With the aid of a spectral library, tentative identification of inorganic oxidizer becomes automated and an easy-to-implement process. The oxidizer identity may be further confirmed by using tandem mass spectrometry (MS/MS). Because it is not necessary to separate the components before analysis, results become available in real time. Little sample consumption and the ease of sample preparation should also be noted. The methodology can be readily employed on various ESI-MS systems that are already in use in analytical laboratories. This is another important benefit as the ESI-MS instrumentation is becoming increasingly common in forensic laboratories.

KEYWORDS: forensic science, post-blast, inorganic oxidizers, liquid chromatography-mass spectrometry, spectral libraries

Many explosives used in mining, construction, and military applications contain inorganic oxidizers and fuel as the main components. An oxidizer can be defined as a material such as chlorate, permanganate, or nitrate salt, that will give off oxygen readily, thus stimulating the combustion of organic materials and generating a vast amount of hot gaseous combustion products.

The need to characterize accurately these oxidizers is evident as these materials are available to the general public and have been used as explosive ingredients in improvised explosive devices (IED). A number of methods have been employed for forensic analysis of inorganic oxidizers in explosive formulations and post-blast residues. They can be arranged in three groups as follows:

Methods Based on Crystal Properties

Microscopical characterization is probably the fastest way to establish the identity of oxidizer crystals when done by a trained person. The Particle Atlas by McCrone and Delly (1) provides a thorough description of principles and techniques of microscopical examination of various particles. X-ray diffraction is a technique for unambiguous characterization of inorganic compounds based on the measurement of crystal lattice parameters that are unique to every salt (2). These methods allow characterization of a substance as an entity. The very nature of these techniques demands that solid material is available for the analyses. Given the high solubility of salts in water it is obvious that the crystals may not be always readily available. Also, there is a need for a relatively large amount of material, which is not always available in post-blast residues.

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Chemical Spot Tests

A few chemical spot tests have been used for presumptive identification of explosive material ingredients. They include Nessler reagent for the presence of ammonium cations, Griess test for the presence of nitrate ions, and copper tetrapyrindine for the presence of perchlorate ions (3). Since these tests use chemistry pertinent to a particular anion or cation, it may be required to use several different tests before the oxidizer is identified correctly. Also, these tests are not always specific enough and need more material than sometimes is available.

Ion Separation Techniques

Another group of methods including ion chromatography (4) and capillary electrophoresis (5) makes use of different acid-base properties of ionic species and differences in their behavior in an applied electric field to afford sensitive identification of inorganic oxidizers. For each oxidizer, two measurements have to be made, as separate columns are needed for anion and cation analysis. The result of the measurements will determine the presence of an anion and/or cation, but will not confirm the presence of an entire oxidizer molecule. Each one of the resulting ion confirmations could be attributed to an ion present in the environmental background, and not necessarily to the expected oxidizer.

Forensic Science Need

It is difficult to utilize existing methods in those situations where several inorganic oxidizers are present, which is often the case in improvised explosive device investigations. On the other hand, because the amount of material necessary for the analyses is often higher than the amount of post-blast residue available, there are limited possibilities for confirmatory analysis. Complex matrices may also limit the usefulness of some of these methods. It is

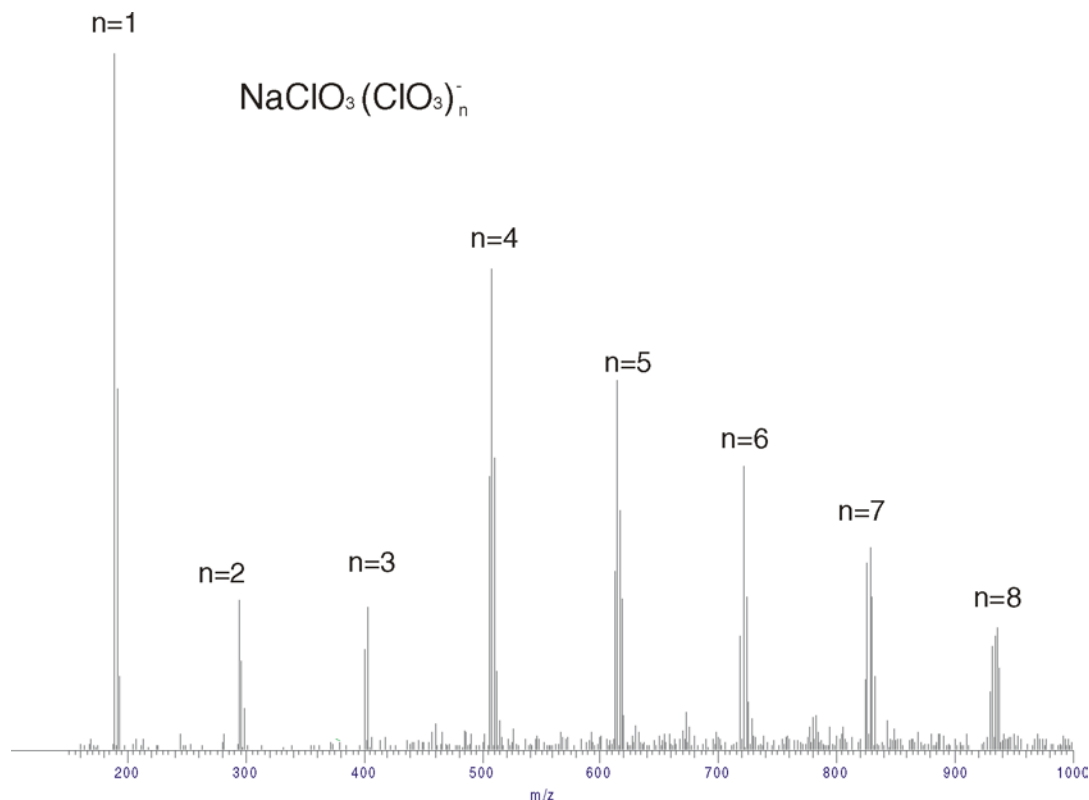


FIG. 1—ESI mass spectrum of 1 mM NaClO_3 solution in water.

evident that there is still a need for a rapid, efficient and reliable method to identify inorganic oxidizers (salts) commonly present in ammonium nitrate and fuel oil (ANFO), emulsion, gel and improvised explosives.

Previous studies in our laboratory (6,7) showed that electrospray ionization mass spectrometry (ESI-MS) is a suitable technique for the analysis of inorganic oxidizers. Our group has been successful in the characterization of nitrates, chlorates, and other inorganic salts in explosive formulations. ESI mass spectra of studied oxidizers reveal a series of cluster ions containing the entire oxidizer molecule. Such a series can be regarded as a “fingerprint” of a particular oxidizer as illustrated in Fig. 1. Oxidizers of similar composition produce similar spectra in terms of cluster ion composition and relative abundances, but due to different mass the pattern will be different.

Whereas we already have demonstrated that ESI-MS is a versatile and adequately sensitive method for the analysis of small amounts of post-blast residues, there is still a need for a methodology that can be used by the practicing forensic scientist to rapidly provide initial assessment on the presence and identity of inorganic oxidizers.

A feasible way to have a real-time analysis capability is to use spectral libraries. This approach has been successfully implemented in gas chromatography/mass spectrometry (GC/MS) analysis of controlled substances (8).

Experimental

Chemicals and Equipment

The inorganic salt standards were purchased from Aldrich-Sigma Co. (Milwaukee, WI) as solid material in purest grade available. High-performance liquid chromatography (HPLC)-grade water was

obtained from Burdick & Jackson Co. (Muskegon, MI). Sugar powder was obtained in a local grocery store. Solid-phase extraction (SPE) cartridges containing 500 mg C18 phase were obtained from Fisher Scientific Co. (Pittsburgh, PA).

Electrospray Mass Spectrometry

MS analyses were carried out using a Thermo-Finnigan LCQ_{DUO} ion trap mass spectrometer (San Jose, CA). Helium was used as damping and collision gas, while nitrogen was used as sheath and auxiliary gas. Heated capillary temperature was held at 200°C for all salts except ammonium nitrate for which it was held at 150°C. The spray voltage was set at 4 kV. Salts were dissolved in water to achieve 1 mM concentration. The solution was introduced into the mass spectrometer at 0.025 mL/min using a syringe pump, unless noted otherwise.

Forensic Samples

Ammonium nitrate-based explosives were received from the Florida Department of Law Enforcement Tallahassee Regional Crime Laboratory. Prior to analyses ten milligrams of material were dissolved in 10 mL of deionized water. Undissolved residue was removed by filtration.

In order to obtain post-blast residue samples with maximum resemblance to those found in bombing scenes, a series of miniature pipe bombs filled with 1–3 g of oxidizer (NaMnO_4 , NH_4ClO_4 , KClO_3 , and NaBrO_3)-sugar mixtures were exploded with the assistance of the local sheriff's department. For safety reasons cardboard pipes were used. The pipe fragments along with surrounding soil were immediately collected into sample jars. The post-blast residues along with a soil sample collected prior to detonation were extracted

with 10 mL of deionized water for every gram of debris, followed by filtration.

Samples for solid-phase extraction were obtained by spiking soil (1 g) with 0.1 mL of 1% KNO_3 water solution followed by water extraction. A SPE cartridge was rinsed with HPLC-grade water (1 mL) with the aid of vacuum suction. The water extract followed by water (2 mL) was flushed through the cartridge and retained for further analyses.

Results

ESI-MS of Inorganic Oxidizers

Positive ion ESI mass spectra usually consist of a series of abundant cluster ions that can be described as one or more salt molecules (typically between one and eleven) centered around the positively charged metal or ammonium cation. In the negative ion mode the cluster center is the negatively charged anion. Table 1 shows major ions found in ESI mass spectra of oxidizer standard solutions.

MS/MS

As can be seen in Fig. 1 and Table 1, ESI-MS produces a highly characteristic pattern for an individual oxidizer, which can be used for tentative identification of post-blast residues.

Unless the m/z value is accurately (within 10 ppm) determined, it alone cannot be used to assign elemental composition to a particular ion because there may be other ions with the same nominal m/z ratio. Therefore it is desirable to have the capability to exclude all other ions with the same m/z ratio as the ion of interest.

Tandem mass spectrometry (MS/MS) is an indispensable tool for identifying compounds in complex matrices and for determining structures of unknown substances. Zhao and Yinon (6) used product ion scanning, the most frequently used MS/MS mode, to confirm the cluster ion composition. Loss of one or more salt molecules (i.e., incremental decrease of n , Table 1) is a general fragmentation pathway and can be used as a confirmative test, thus allowing unambiguous identification of an inorganic oxidizer. Figure 2 demonstrates how the ion identity can be established by selecting the ions of interest followed by collision-induced dissociation (CID). In this example, the ion $[(\text{NaClO}_3)_2\text{ClO}_3]^-$ at m/z 295 has been chosen as the parent ion (Fig. 2a). The MS/MS daughter-ion spectrum (Fig. 2b) shows a loss of 112 mass units (NaClO_3), which serves as evidence of the ion identity.

Spectral Libraries

We have chosen to use the NIST/EPA/NIH (National Institute of Science and Technology/Environmental Protection Agency/National Institutes of Health) mass spectral library software (9) as a platform to create a library containing the ESI mass spectra of common oxidizers because this software is regarded as an industry standard. Every major MS instrumentation manufacturer offers library creation, management, and search capabilities that support the NIST format, thus assuring compatibility between different systems and future expansion.

The spectral library "oxidizers" was created as follows: ESI spectra of oxidizers listed in Table 1 were acquired in positive and negative ion modes at conditions specified in the experimental section using standard 1 mM salt solutions. Then each spectrum was transferred into the library browser, annotated and appended to the library.

The library was first validated using standard salt solutions to ensure there are no false positives (cross reactivity). Because the very nature of a forensic sample precludes one from knowing the analyte concentration, it is important that the analytical method has sufficient dynamic range. We are reporting that the spectral library allows one to identify inorganic oxidizers in a concentration range from 0.1 mM (10 ppm for $M=100$) to 10 mM (0.1%). At salt concentration below 0.1 mM the abundance of characteristic cluster ions becomes too low for reliable pattern matching whereas a concentration higher than 0.1% causes rapid salt buildup and clogging of the ion source.

Figure 3 shows a library search result view for NH_4ClO_4 -sugar post-blast residue sample. It consists of the following windows:

1. Hits list. This window contains library entries that match the data arranged by correlation success.
2. Structure window. It displays identifiers (such as chemical structure, formula, molecular weight, and library index number) associated with selected in the previous window compound.
3. Comparison of the sample spectrum with the library entry.
4. Difference spectrum (raw data minus library entry). Peaks above the horizontal axis are relatively more intense in the sample spectrum than in the library entry. Peaks below the X-axis are relatively more intense in the library than in the sample spectrum. This feature provides the user with an opportunity to judge the match quality for him(her)self.

It can be seen that ammonium perchlorate indeed constitutes the main component of the extract. The confidence level can be easily increased by running the same sample in the negative ion mode. Figure 4 shows a library search result on a spectrum of the same sample but acquired in the positive ion mode. Again, ammonium perchlorate is the best match.

Post-Blast Residues Analysis

A reaction between fuel and oxidizer (combustion) rarely proceeds to its fullest extent unless special precautions are taken. Consequently, one will expect to find a mixture of combustion products, unreacted species and intermediates in most post-blast residues. With the exception of NaMnO_4 based mixture, the analysis of all post-blast samples revealed the presence of oxidizers themselves along with reduction products (i.e., Cl^- and Br^- containing clusters), which is in agreement with the literature (10). The extract of sodium permanganate post-blast residue did not contain any other than permanganate ions since the main product MnO_2 is not soluble in water. Mass spectrum of the blank (extract of soil which was taken prior to detonation) did not reveal any abundant ions or ions that can be associated with oxidizers.

Figure 5 shows a library search result window of a sample of a common ammonium nitrate-based explosive, Powermite, in the positive ion mode. Search results for the same sample in negative ion mode are displayed in Fig. 6. While ammonium nitrate is the major component, contribution from sodium nitrate is also evident, when comparing to library entry. Analysis of other ammonium nitrate-based explosives (Iremite and KinPak) confirmed NH_4NO_3 as the major ingredient.

In order to minimize errors, analysis should be conducted in both positive and negative-ion modes. An MS/MS experiment may be warranted if further confirmation is desired.

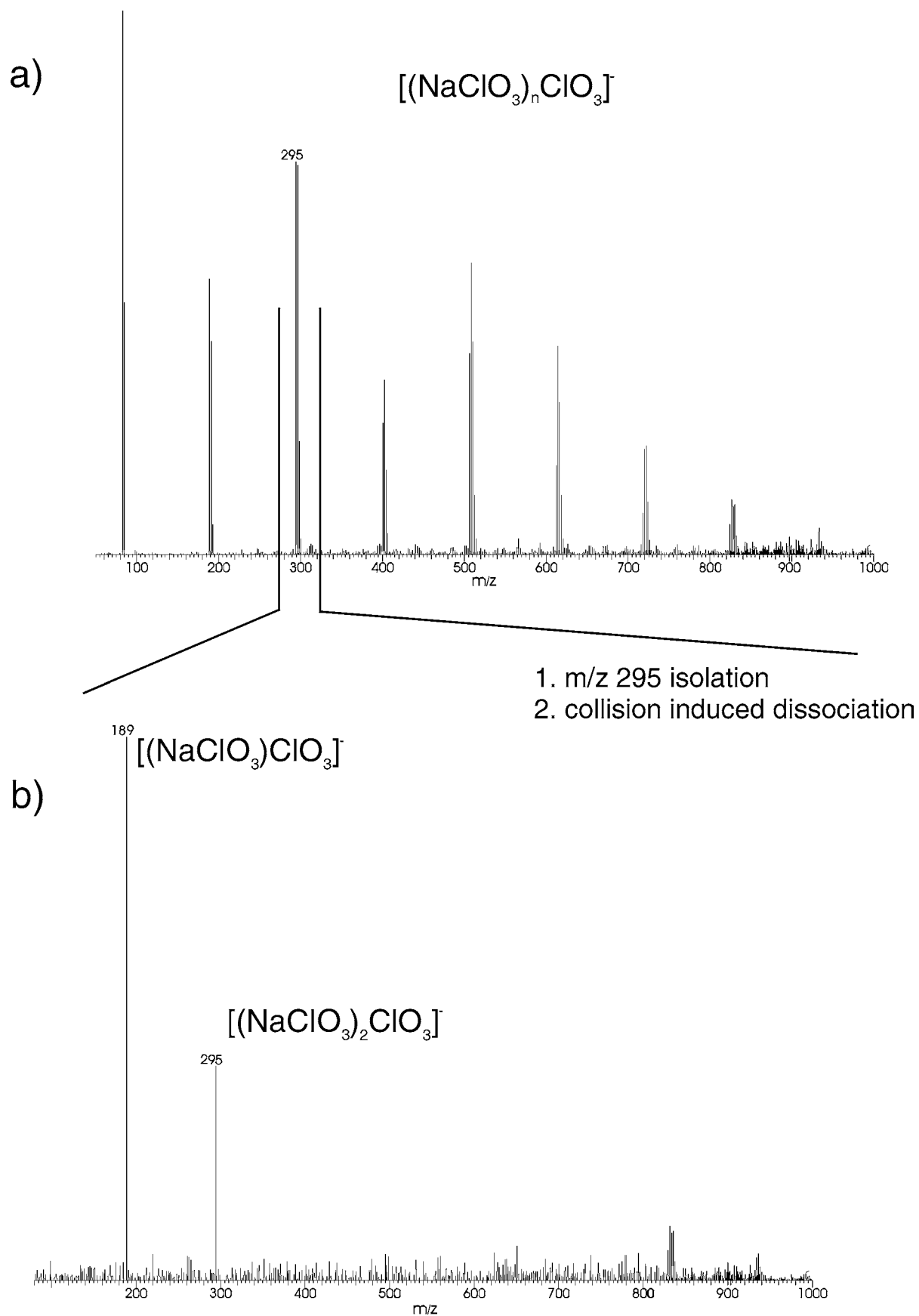
TABLE 1—ESI-MS of inorganic oxidizers. The most abundant ion in a series is shown in bold; its m/z value is in parentheses.

Salt	Formula	Ion Composition, the Most Abundant Ion and Its m/z	
		Positive Ions	Negative Ions
Sodium nitrate	NaNO ₃	[(Na(NO ₃)) _n Na] ⁺ , $n = 1-11$ [(Na(NO₃))₆Na]⁺ (533)	[(Na(NO ₃)) _n NO ₃] ⁻ , $n = 1-11$ [(Na(NO₃))₂NO₃]⁻ (147)
Potassium nitrate	KNO ₃	[(K(NO ₃)) _n K] ⁺ , $n = 1-11$ [(K(NO₃))₃K]⁺ (342)	[(K(NO ₃)) _n NO ₃] ⁻ , $n = 1-11$ [(K(NO₃))₄NO₃]⁻ (466)
Barium nitrate	Ba(NO ₃) ₂	[Ba(H ₂ O) _n] ²⁺ , $n = 0-6$ [Ba(NO ₃)(H ₂ O) _n] ⁺ , $n = 0-4$ [Ba(H₂O)₅]²⁺ (113.5)	[(Ba(NO ₃) ₂) _n NO ₃] ⁻ , $n = 1-3$ [(Ba(NO₃)₂)₂NO₃]⁻ (586)
Ammonium nitrate	NH ₄ NO ₃	[((NH ₄)NO ₃) _n NH ₄] ⁺ , $n = 1-5$ [((NH₄)NO₃)₂NH₄]⁺ (178)	[((NH ₄)NO ₃) _n NO ₃] ⁻ , $n = 0-2$ [NO₃]⁻ (62)
Potassium permanganate	KMnO ₄	[(KMnO ₄) _n K] ⁺ , $n = 1-12$ [(KMnO₄)₄K]⁺ (670)	[(KMnO ₄) _n MnO ₄] ⁻ , $n = 1-8$ [(KMnO₄)₄MnO₄]⁻ (277)
Sodium permanganate	NaMnO ₄	[(NaMnO ₄) _n Na] ⁺ , $n = 1-12$ [(NaMnO ₄)(H ₂ O) _n Na] ⁺ , $n = 1, 2$ [(NaMnO₄)₄Na]⁺ (590)	[(NaMnO ₄) _n MnO ₄] ⁻ , $n = 1-4$ [(NaMnO₄)₄MnO₄]⁻ (261)
Sodium sulfate	Na ₂ SO ₄	[(Na ₂ (SO ₄)) _n Na] ⁺ , $n = 1-6$ [Na ₂ (SO ₄)(H ₂ O)Na] ⁺ [(Na₂(SO₄))₄Na]⁺ (591)	[HSO ₄] ⁻ [HSO ₄ (H ₂ O)] ⁻ [NaSO ₄ (H ₂ O) _n] ⁻ , $n = 1, 2$ [HSO₄]⁻ (97)
Potassium sulfate	K ₂ SO ₄	[(K ₂ (SO ₄)) _n K] ⁺ , $n = 1-6$ [K ₂ (SO ₄)(H ₂ O)K] ⁺ [K₂(SO₄))K]⁺ (213)	[HSO ₄] ⁻ [KSO ₄ (K ₂ SO ₄) _n] ⁻ , $n = 0, 2$ [KSO₄]⁻ (135)
Ammonium sulfate	(NH ₄) ₂ SO ₄	[(NH ₄ H(SO ₄)) _n NH ₄] ⁺ , $n = 1-6$ [(NH₄H(SO₄))₂NH₄]⁺ (248)	[HSO₄]⁻ (97)
Sodium chlorate	NaClO ₃	[(NaClO ₃) _n Na] ⁺ , $n = 1-9$ [(NaClO₃)₅Na]⁺ (555)	[(NaClO ₃) _n ClO ₃] ⁻ , $n = 1-8$ [(NaClO₃)ClO₃]⁻ (189)
Potassium chlorate	KClO ₃	[(KClO ₃) _n K] ⁺ , $n = 1-9$ [(KClO₃)₂K]⁺ (283)	[(KClO ₃) _n ClO ₃] ⁻ , $n = 0-6$ [(KClO₃)₄ClO₃]⁻ (573)
Ammonium perchlorate	NH ₄ ClO ₄	[(NH ₄ ClO ₄) _n NH ₄] ⁺ , $n = 1-8$ [(NH₄ClO₄)₆NH₄]⁺ (722)	[(NH ₄ ClO ₄) _n ClO ₄] ⁻ , $n = 0-8$ [ClO₄]⁻ (99)
Sodium perchlorate	NaClO ₄	[(NaClO ₄) _n Na] ⁺ , $n = 1-8$ [(NaClO ₄) ₆ Na] ⁺ (756)	[(NaClO ₄) _n ClO ₄] ⁻ , $n = 0-7$ [ClO₄]⁻ (99)
Sodium bromate	NaBrO ₃	[(NaBrO ₃) _n Na] ⁺ , $n = 1-6$ [(NaBrO ₃)(H ₂ O)Na] ⁺ [(NaBrO₃)₂Na]⁺ (325)	[(NaBrO ₃) _n BrO ₃] ⁻ , $n = 0-3$ [BrO₃]⁻ (128)
Potassium bromate	KBrO ₃	[(KBrO ₃) _n K] ⁺ , $n = 1-6$ [(KBrO₃)₂K]⁺ (373)	[(KBrO ₃) _n BrO ₃] ⁻ , $n = 0-3$ [(H ₂ O)BrO ₃] ⁻ [BrO₃]⁻ (128)
Potassium cyanate	KOCN	[K(KOCN) _n] ⁺ , $n = 1-6$ [K(KOCN)₄]⁺ (363)	No abundant characteristic ions
Potassium carbonate	K ₂ CO ₃	[K(KOH)] ⁺ [HKCO ₃] ⁺ [K(K ₂ CO ₃) _n (KHCO ₃) _m] ⁺ , $n = 1, 2, m = 0-2$ [K(K₂CO₃)]⁺ (177)	[HCO ₃] ⁻ [HCO ₃ (H ₂ O)] ⁻ [KCO ₃ (H ₂ O) ₂] ⁻ [HCO₃(H₂O)]⁻ (79)
Sodium carbonate	Na ₂ CO ₃	[Na(Na ₂ (CO ₃)) _n (NaHCO ₃) _m] ⁺ , $n = 1-3, m = 0-3$ [Na(Na₂(CO₃))₂(NaHCO₃)]⁺ (319)	[HCO ₃] ⁻ [HCO ₃ (H ₂ O)] ⁻ [NaCO ₃ (H ₂ O) ₂] ⁻ [HCO₃(H₂O)]⁻ (79)
Ammonium dichromate	(NH ₄) ₂ Cr ₂ O ₇	No abundant characteristic ions	[HCrO ₄ (CrO ₃) _n] ⁻ , $n = 0-4$ [HCrO₄]⁻ (117)
Ammonium chromate	(NH ₄) ₂ CrO ₄	No abundant characteristic ions	[HCrO ₄ (CrO ₃) _n] ⁻ , $n = 0-4$ [HCrO₄]⁻ (117)

Organic Matter Removal

While organic matter present on the test site did not cause an interference problem, it is quite possible that some samples collected from a heavily contaminated surface (pavement, machinery parts, etc.) may contain organic matter in the amount that will give rise to abundant ions capable of hindering those from oxidizer.

When this is the case, solid-phase extraction (SPE), a common procedure for isolating organic substances from aqueous medium (11), may be used to remove excessive organic contamination. Figure 7 shows improvement in the potassium nitrate cluster ions signal after SPE. Two soil samples were used. The first sample, which did not have visible contamination, was taken from a surface



Hit	SI	RSI	Name	Library Name
1	318	414	Ammonium Perchlorat	
2	159	252	Sodium Perchlorate	
3	38	151	Potassium Chlorate	
4	73	83	Sodium Chlorate	
5	24	57	Sodium Permanganate	

Ammonium Perchlorate
Formula ClH₄NO₄, MW 117, CAS# NA, Entry# 14

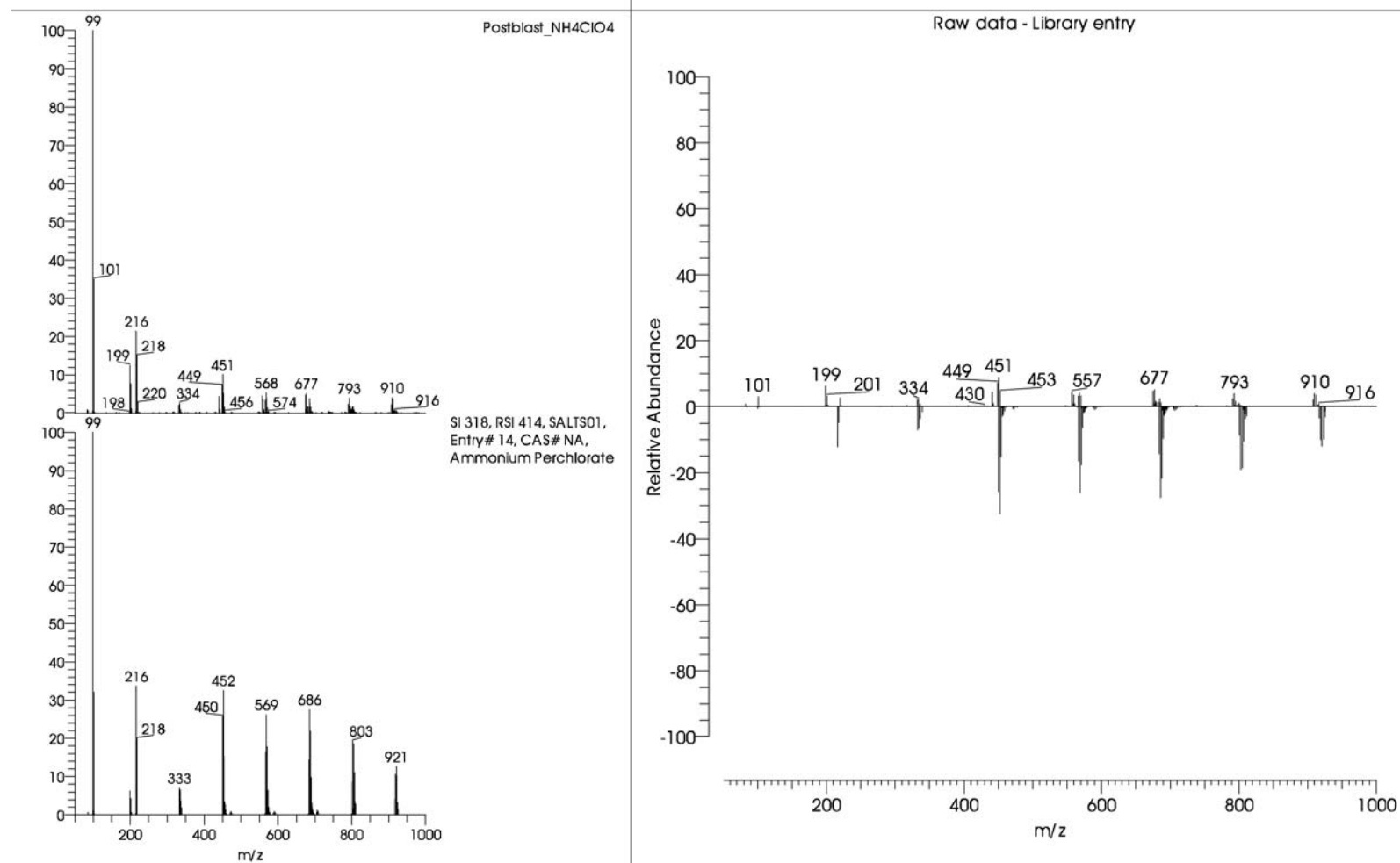


FIG. 3—Spectral library search view for post-blast residue of ammonium perchlorate—sugar mixture. Spectrum acquired in the negative ion mode.

Hit	SI	RSI	Name	Library Name
1	422	491	Ammonium Perchlorat	
2	205	363	Potassium Permangan	
3	158	345	Sodium Chlorate	
4	130	311	Sodium Perchlorate	
5	251	304	Sodium Chlorate	
6	94	235	Sodium Bromate	
7	73	226	Potassium Permangan	

Ammonium Perchlorate
Formula ClH4NO4, MW 117, CAS# NA, Entry# 15

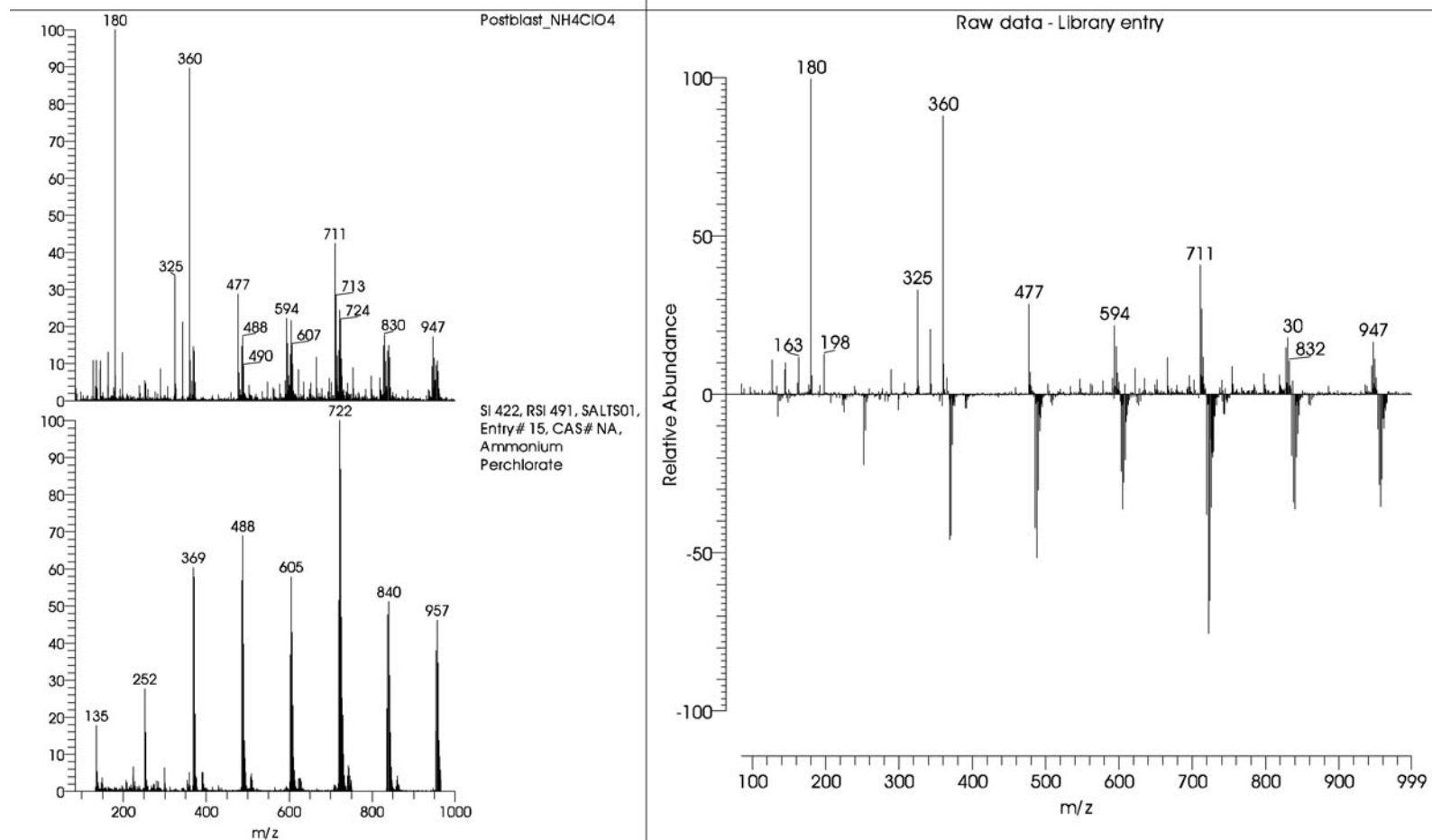


FIG. 4—Spectral library search view for post-blast residue of ammonium perchlorate—sugar mixture. Spectrum acquired in the positive ion mode.

Hit	SI	RSI	Name	Library Name
1	543	544	Ammonium Nitrate	
2	264	282	Sodium Nitrate	
3	61	143	Potassium Bromate	

Ammonium Nitrate
Formula H₄N₂O₃, MW 80, CAS# NA, Entry# 13

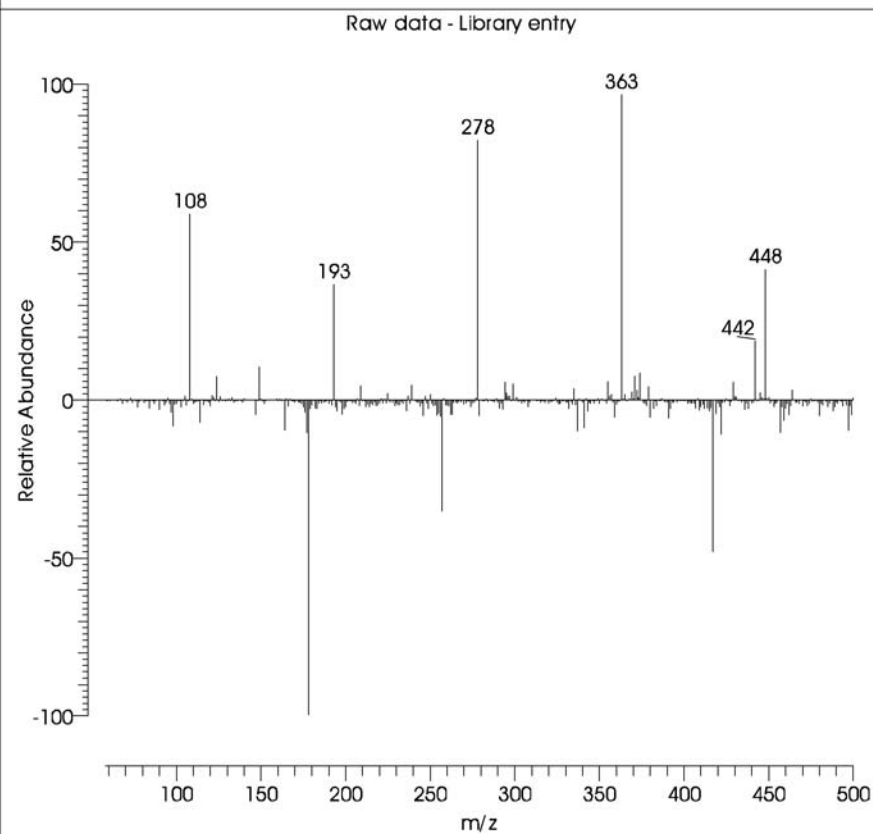
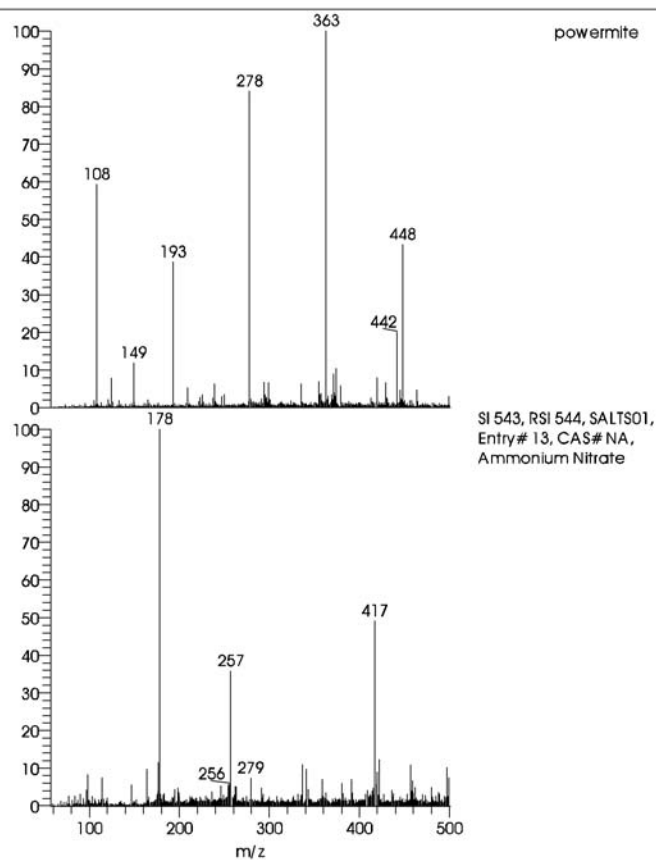


FIG. 5—Spectral library search view for Powermite spectrum acquired in the positive ion mode.

Hit	SI	RSI	Name	Library Name
1	516	525	Ammonium Nitrate	
2	282	297	Sodium Nitrate	
3	222	250	Sodium Permanganate	

Ammonium Nitrate
Formula H₄N₂O₃, MW 80, CAS# NA, Entry# 12

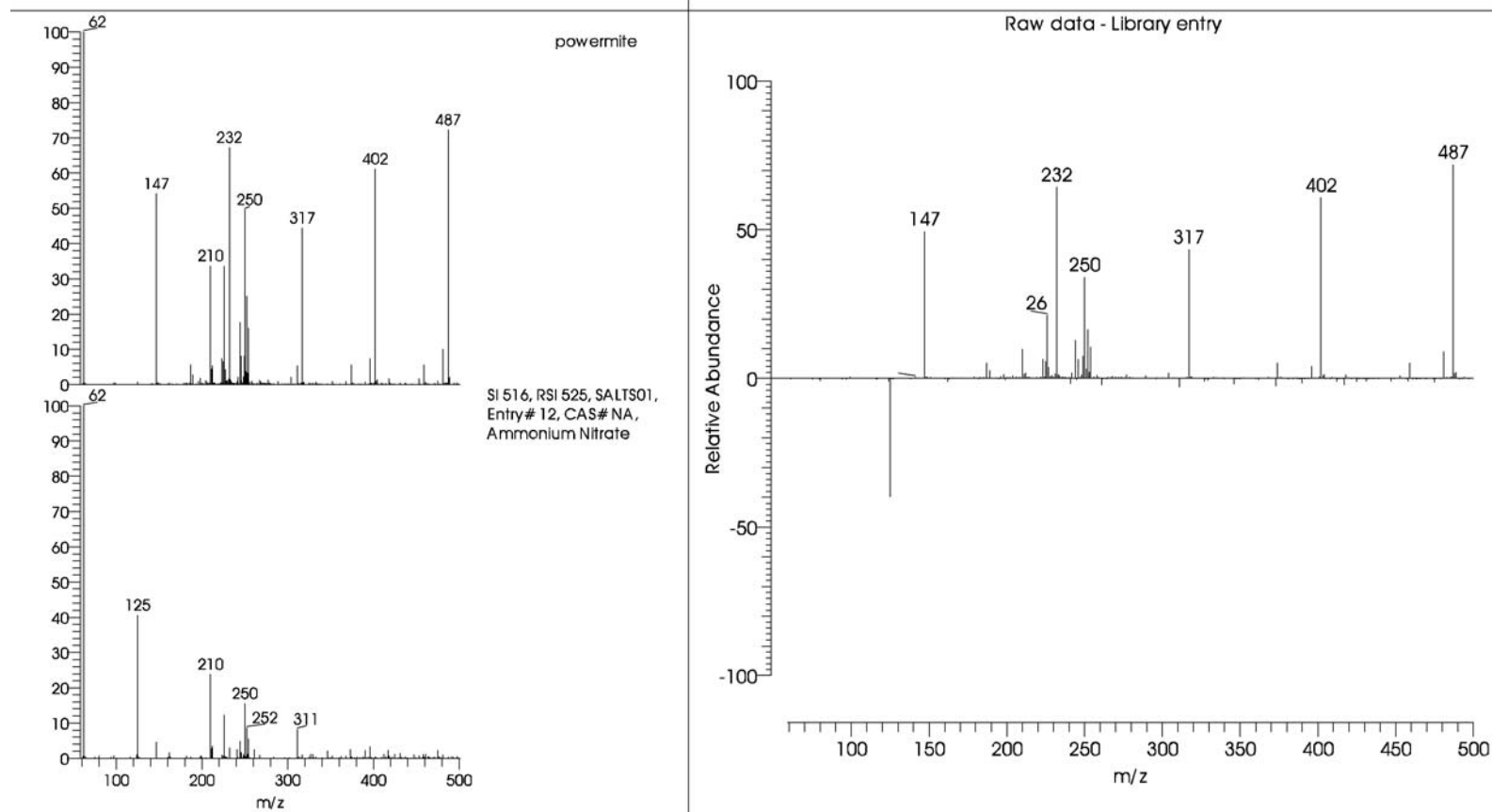


FIG. 6—Spectral library search view for Powermite spectrum acquired in the negative ion mode.

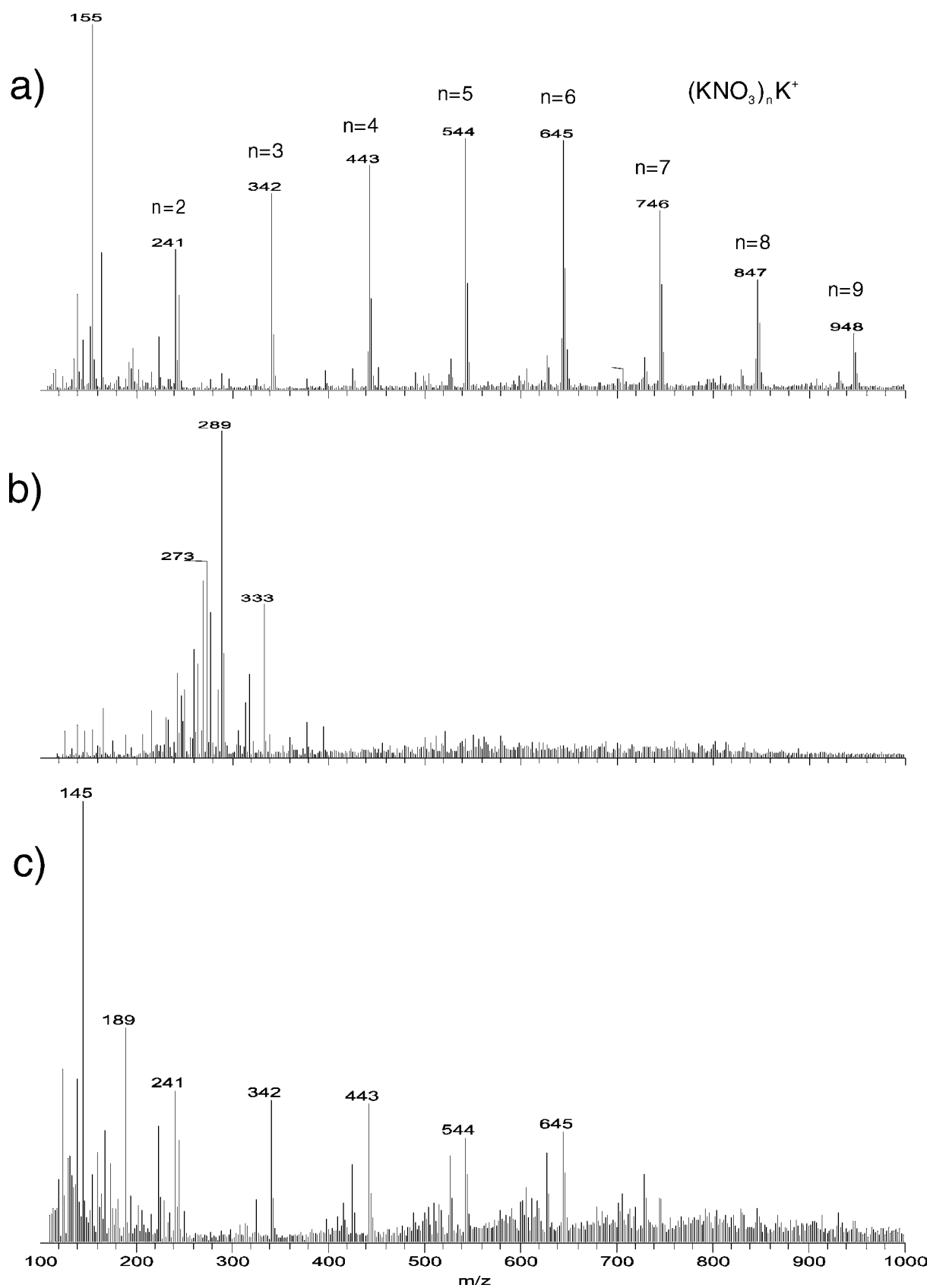


FIG. 7—Mass spectra of water extracts of KNO_3 spiked sand (a), engine oil contaminated parking lot soil (b), and same as (b) after organic matter removal by SPE (c).

of a parking lot whereas the second sample was taken from a spot (20 cm away from the first one) heavily contaminated with engine oil.

Conclusions

In this and previous communications we have demonstrated that ESI-MS has adequate sensitivity, while requiring no or very simple sample preparation. With the aid of spectral libraries this method can be used for real-time screening for and tentative identification of inorganic oxidizers. MS/MS provides the capability to unambiguously confirm the presence of a particular oxidizer. LC/MS-ESI seems to be an appropriate technique for characterization of inorganic oxidizers in unexploded devices and post-blast residues.

Acknowledgments

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