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Ammonium Nitrate Recovery from Four Truck Fabrics using Ion Chromatography with Conductivity Detection

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Abstract

The purpose of this work was to determine how various environmental conditions typically encountered when attempting to recover evidence from a detonated ammonium nitrate improvised explosives device affect the ability to extract ammonium and nitrate from four types of truck fabrics. Fabrics were spiked with 100ppm ammonium nitrate and exposed to wind, rain, oil smoke, fabric treatment, humidity, and low and high temperatures. Ammonium and nitrate ions recovered from methanol extraction were measured using ion chromatography with suppressed conductivity detection. We found that fabric treatment, oil smoke, diesel fuel, and environmental conditions of wind and short-term exposure to moderate to high temperatures and moderate humidity do not interfere with the ability to quantify the ammonium and nitrate ions remaining on the fabrics. Other factors, such as rain and prolonged exposure to high temperatures and high humidity resulted in greater loss of ammonium and nitrate from the fabrics but still left enough for their detection.

Keywords: Ammonium; Nitrate; Improvised explosive devices; Ion chromatography

Introduction

Improvised explosive devices (IEDs) have been the weapon of choice for military, insurgent, and extremist groups for decades. IEDs are easily fabricated from widely accessible and inexpensive materials, such as gunpowder [1] and an ammonium nitrate based fertilizer soaked in fuel oil (ANFO) [2]. IEDs cause significant distraction, damage, and loss of life, as demonstrated in the Pan Am 103 flight [3], World Trade Center bombings in 1993 [1], the Oklahoma City Bombing in 1995 [3], the Bali Bombings in 2002 [2] and the Boston City Marathon Bombings in Boston. Therefore, the development of screening methods for chemicals found in IEDs has become of paramount importance for law enforcement and military organizations that are combating the individuals that use these devices [4].

IEDs may be constructed and packaged in a number of ways, one of the most common being an automobile. Vehicle borne improvised explosive devices (VBIEDs), are widely used by terrorist organizations since they are easy to build, conceal, and stage in high traffic areas [4]. Furthermore, these devices can be detonated from a remote location or detonated by a strategically placed detonator that is inadvertently triggered [4]. Due to the nature and environment of a detonation site, analysts are faced with several challenges after a VBIED has exploded, one being evidence collection for post-blast residue analysis. Factors that determine the type of debris collected for analysis are distance from the blast site (usually a radius of 15 to 20 meters maximum will yield trace evidence from the detonated explosive), the analytical instrumentation available to the analyst (an ion chromatograph or capillary electrophoresis column versus a gas chromatograph mass spectrometer), and the types of material available for post-blast residue analysis, i.e. sign posts, sidewalks, soil, etc [5].

As mentioned previously, IEDs are manufactured from widely distributed and inexpensive materials, so there is an issue of environmental interference/contamination during analysis, which has been investigated in several studies. In one study the background ion concentration levels of components that are used to make explosives were measured in the environment of 28 cities. They measured ammonium and nitrate levels in areas such as automobiles, park benches, telephones, mailboxes, etc. and found median levels of ammonium and nitrate recovered to be 26µg and 3µg, respectively. The maximum levels of ammonium and nitrate recovered were 210µg and 110µg, respectively. Out of the 9 automobiles sampled, 0% showed ammonium and 33% showed nitrate ions [5,6]. The potential for contamination from the environment is further complicated by the fact that the ions of an inorganic explosive interact with the surface of the evidence via Van der Waals forces, meaning that the ions could be easily transferred and recovered from the surfaces [7]. It

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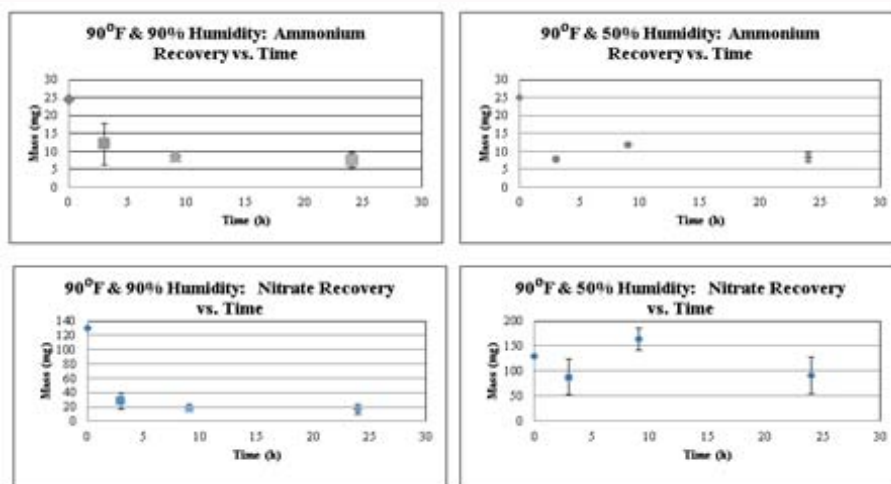


Figure 1: Recovery of ammonium and nitrate at 90°C with 90% and 50% humidity with time. Values reported are averages from all fabrics.

has been found that environmental conditions and the landscape of the blast site affect the results of post-blast residue analysis [3]. For example, a study conducted after TWA Flight 800 found that the type of explosive used and the surface of the material were critical factors in determining the persistence of post-blast residues when exposed to a watery environment [8]. These studies demonstrate the importance of collecting adequate negative controls from the blast site and that the analytical methods used in explosives analysis need to be improved in order to account for environmental factors/contamination.

Current methods employed in pre and post-blast residue collection and analysis are to obtain a sterile cotton swab, moisten the swab with de-ionized water, and then swab the surface [9]; railings, metal sign posts, the exterior of automobiles & buildings, etc. are common surfaces to test at a detonation site [5]. Another method for evidence collection is SPME (solid phase micro-extraction), which involves using a syringe with a coated fiber inside the needle to absorb the post-blast residue vapors; the residue ions are then extracted with a non-interfering solvent or by heat [10]. There have also been studies in the collection and analysis of soil samples with ion chromatography, high-performance liquid chromatography [11], gas chromatography mass spectrometry [12], and liquid-chromatography atmospheric pressure ionization [13] for post-blast residue analysis; however they involve a laborious extraction process compared to traditional methods, and sometimes yield artifact chemicals that were not a component in the detonated IED, but are sometimes found in IEDs, thus leading to an incorrect conclusion regarding the IED [14]. Although methods like gas and liquid chromatography coupled with mass spectrometry offer superb discrimination and identification power for the components involved in the detonated IED, these methods cannot be performed at the blast site, whereas instrumentation such as ion chromatography offers the advantage of being portable by supplying its own power source leaving it operational for 8 hours [15,16]. By developing a screening method that can be performed on site, the investigator would be able to conduct a preliminary analysis of the explosive residues, which would facilitate appropriate evidence collection and more efficient explosive analysis in the laboratory [2].

In this paper, a potential method for preliminary post-blast residue analysis from fabrics using ion chromatography with conductivity detection is presented. This study focuses on ammonium

Table 1: Method extraction efficiency.

Fabric	Ammonium	Nitrate
4119 polyester	69.9%±6.9	30.2%±0.8
341 nylon	35.9%±2.0	17.1%±0.9
448 velour	50.1%±3.0	21.5%±3.6
562 vinyl	67.0%±28.5	30.8%±16.0

and nitrate ion recovery from fabrics since the nitrate ion is a common component in several explosives and the ammonium ion is generally specific to ammonium nitrate containing explosives [2], thus the ability to identify ammonium nitrate during the preliminary screening would greatly increase evidence processing. Ammonium nitrate recovery from fabrics was investigated by spiking various truck fabrics, with a 100g/L ammonium nitrate solution in methanol and then exposing the spiked fabrics to various environmental conditions. The ammonium and nitrate ions were then extracted with methanol and measured using ion chromatography and reported for each fabric. This is the first study to report on the recovery of ammonium and nitrate ions from truck fabrics.

Experimental

Materials

The ion chromatograph buffer was made from sodium carbonate (J.T. Baker now Avantor™, Phillipsburg, NJ) and sodium bicarbonate (Fisher Scientific, Pittsburg, PA.). Methane sulfonic acid and sodium lauryl sulfate were obtained from Sigma Aldrich (Saint Louis, MO). Ammonium nitrate was purchased from Fisher Scientific (Pittsburg, PA) and methanol from VWR (West Chester, PA). Scotch-Gard™ used to treat the fabrics was obtained from 3M (St. Paul, MN).

Standards

A 5 anion standard purchased from Dionex, (Sunnyvale, CA) (fluoride (20mg/L), chloride (30mg/L), nitrate (100mg/L), phosphate (150mg/L), and sulfate (150mg/L) was used to make the external calibration curve for nitrate at concentrations of 0.5mg/L, 5.0mg/L, 10mg/L, 25mg/L, 50mg/L, 75mg/L and 100mg/L. Penylphosphinic acid was used as the internal standard (Sigma Aldrich, St. Louis, Mo) at a concentration of 39.2mg/L. A 6 cation standard purchased from Dionex, (Sunnyvale, Ca.) (lithium (50mg/L), sodium (200mg/L),

Table 2: Effect of rain on ammonium nitrate recovery.

Sample	Mass Recovered (mg)	Percent Recovery	Mass Recovered (mg)	Percent Recovery
Ion	Ammonium	Ammonium	Nitrate	Nitrate
4119	0.45mg± 0.11	1.90%±0.34	0.56mg±0.03	0.71%±0.06
448	0.69mg±0.11	1.91%±0.39	0.63mg±0.05	0.51%±0.06
341	0.98mg±0.06	1.96%±0.15	0.82mg±0.03	0.48%±0.06
562	1.41mg±0.02	6.55%±0.87	1.04mg±0.03	1.40%±0.15

Table 3: Effect of oil smoke on ability to recover ammonium and nitrate ions.

Sample	Mass Recovered (mg)	Percent Recovery	Control Percent Recovery	Mass Recovered (mg)	Percent Recovery	Control Percent Recovery
Ion	Ammonium	Ammonium	Ammonium	Nitrate	Nitrate	Nitrate
4119 polyester	12.2mg	94.3%±37.1	69.9%±6.9	15.0mg	32.6%±10.4	30.2%±0.8
448 velour	12.8mg	51.1%±7.7	50.1%±3.0	16.3mg	18.8%±2.6	21.5%±3.6
341 nylon	11.3mg	22.4%±2.4	35.9%±2.0	13.1mg	7.54%±1.2	17.1%±0.9
562 vinyl	8.13mg	86.6%±17.9	67.0%±28.5	8.05mg	24.9%±8.6	30.8%±16.0

ammonium (400mg/L), potassium (200mg/L), magnesium (200mg/L), and calcium (1000mg/L) was used to make the external calibration curve for ammonium at concentrations of 0.2mg/L, 0.5mg/L, 2mg/L, 20mg/L, 40mg/L and 100mg/L.

Fabrics

Fabrics obtained from Enterprise Upholstery, Inc. (Belair, MD) were representative of the materials commonly used for truck interiors. Fabric 1 is a black vinyl (nonporous) material from GM truck models VIN #562. Fabric 2 is a semiporous woven polyester material from Ford truck models (VIN #4119). Fabric 3 is a blue velour fabric (semi-porous) from Chevy truck models (VIN#448). Fabric 4 is a porous nylon fabric from GM truck models (VIN #341).

Methods

Instrumentation and parameters: The ion chromatograph was an ICS-5000 (Dionex, Sunnyvale, CA.) with suppressed conductivity and Chromeleon 6.80 software to collect and analyze the data. The instrument limits of detection and quantification were determined to be 0.05mg/L and 0.5mg/L, respectively. The established instrument parameters with the gradient column, Ion Pac AS22 4 x 250 mm were a flow rate of 1.2mL/min, column temperature of 22°C, suppressed conductivity recycle mode with an anion self-regenerating suppressor (ASRS 300 4mm), a current of 26mA, a data collection rate of 5.0Hz, and a cell heater of 35°C. The injected volume was fixed at 10µL. Sodium carbonate/sodium bicarbonate eluent was used for the anions and MSA eluent was used for the cations with a 16 minute program for each.

Preparation of samples: Fabrics were cut into 0.5 inch by 2 inch rectangles and treated to remove background ammonium and nitrate (that we determined were present on all fabrics). This procedure resulted in ammonium and nitrate ions below the instrument LOD. The fabrics were placed in a 15 ml conical tube (BD, Franklin Lakes, NJ) with 7.5ml of methanol and sonicated for 10 minutes at 40kHz. The methanol was then decanted into a glass waste beaker and the fabrics were allowed to air-dry completely prior to being weighed and labeled for experiments and analysis. After the fabrics were weighed and labeled, they were sprayed with the 100ppm ammonium nitrate solution, allowed to air-dry, and the treated fabrics were re-weighed prior to exposure to one of the conditions being investigated in this study. After exposure, the ammonium and nitrate ions on the fabrics were extracted with 2.0mL of MeOH in a 15ml conical tube for 10

minutes in the sonicator. The fabrics were removed and allowed to air-dry, while the MeOH solvent was transferred to a glass vial to be evaporated with a 6 mini-port jet air set (Sigma Aldrich, St. Louis, MO). After the MeOH had completely evaporated, 1.0ml of 18.0mΩ filtered water was used to reconstitute the residue ammonium nitrate and then drawn up in a 1.0mL disposable syringe (Franklin Lakes, NJ) and passed through a Fisherbrand 0.45µm nylon filter (Fisher Scientific, Pittsburg, PA) to remove any debris [2]. Phenylphosphinic acid (20µL) internal standard was added to 480µL of the extracted sample in an IC vial (MicroSolv, Eatontown, NJ).

Extraction efficiency and method development: Method development involved determining if spraying versus submerging the fabrics in the ammonium nitrate solution resulted in more ammonium nitrate on the fabrics as well determining if methanol or 18.2mΩ filtered water resulted in a better extraction efficiency of the ammonium and nitrate ions from the fabrics. Spraying the fabrics was a preferable method for spiking the samples, and extracting with methanol yielded significantly greater extraction efficiencies for all fabrics.

Extraction efficiency (Table 1) was calculated by dividing the amount of ammonium and nitrate ions extracted from fabrics without any treatment by the amount of ammonium and nitrate ions deposited onto the fabrics, (measured by the difference between fabric mass after being sprayed with 100g/L ammonium nitrate and the fabric mass after the methanol wash).

Environmental studies (90°F with 90% humidity versus 90°F with 50% humidity): The spiked fabrics were subjected to a controlled temperature chamber (Electro-Tech Systems) which was set to 90°F and either 90% humidity or 50% humidity. The fabric samples were then collected at 3, 9, and 24 h time points to ascertain if increased exposure to atmospheric moisture would affect ammonium and nitrate ion recoveries. The fabric samples were prepared and extracted according to previously described methods.

Fabric samples were subjected to the outdoor weather conditions of Maryland. These samples were pinned to cardboard and set out on a flat surface; the fabrics were collected at 6h, 48h and 72h time points. During the outdoor study, the fabric samples were exposed to an average temperature of 30-40°F, an average humidity of 30% to 40%, as well as little to moderate rain between the 48 hour and 72 hour time points. The fabric samples were prepared and extracted

Table 4: Effect of ANFO on ammonium nitrate recovery.

Fabric	Mass Recovered (mg)	Percent Recovery	Control Percent Recovery	Mass Recovered (mg)	Percent Recovery	Control Percent Recovery
Ion	Ammonium	Ammonium	Ammonium	Nitrate	Nitrate	Nitrate
4119 polyester	6.86mg±1.46	36.1%±0	69.9%±6.9	6.17mg±1.61	26.5%±4.07	30.2%±0.8
448 velour	8.42mg±1.88	55.0%±3.94	50.1%±3.0	6.59mg±0.80	14.4%±1.46	21.5%±3.6
341 nylon	5.51mg±0.057	20.7%±0.33	35.9%±2.0	4.40mg±0.93	6.01%±2.07	17.1%±0.9
562 vinyl	7.32mg±0.734	86.5%±0	67.0%±28.5	8.43mg±3.86	30.9%±4.98	30.8%±16.0

Table 5: Effect of Scotch-Gard™ on ammonium and nitrate recovery.

Sample	Percent Recovery with Scotch-Gard™	Percent Recovery without Scotch-Gard™	Percent Recovery with Scotch-Gard™	Percent Recovery without Scotch-Gard™
Ion	Ammonium	Ammonium	Nitrate	Nitrate
4119 polyester	84.8%±21.3	69.9%±6.9	31.8%±8.14	30.2%±0.8
448 velour	52.4%±7.18	50.1%±3.0	25.9%±8.31	21.5%±3.6
341 nylon	42.6%±5.03	35.9%±2.0	15.7%±2.58	17.1%±0.9
562 vinyl	105%±0	67.0%±28.5	40.8%±9.36	30.8%±16.0

according to previously described methods.

Rain study: Rainwater was collected in a glass pyrex bowl that had been cleaned with 18.2mΩ filtered water. The rainwater samples were run on the IC to determine if there were any trace amounts of ammonium or nitrate ions; it was found that there was ammonium (0.017mg/L) and nitrate (~4.0mg/L) traces present in the rainwater. The fabrics were sprayed with the 100g/L ammonium nitrate solution and allowed to air-dry prior to being exposed to the rainwater. The fabrics were sprayed with the rainwater for 5 minutes in a simulation of a harsh and fast rain shower as an extreme weather condition. The fabric samples were prepared and extracted according to previously described methods.

Wind study: The fabrics were sprayed with 100g/L of ammonium nitrate and pinned to flat cardboard. The cardboard was positioned against a wall and the fan was then placed in front of the fabrics at a 45° angle 2 inches away and 18 inches away. The fan was set at the highest setting, ~5mph, and allowed to blow on the fabrics for a max of 3.5 hours. During the 3.5 hours, the ammonium and nitrate levels were measured in 30 minute intervals to ascertain if there was any observable loss of ammonium nitrate from the fabrics.

Scotch-Gard™ treated fabrics: Some of the the fabrics were treated with Scotch-Gard™ according to the manufacturer's instructions prior to being sprayed with the 100g/L ammonium nitrate solution. One set of the treated fabrics was used to determine the effect of Scotch-Gard™ on the extraction efficiency of ammonium nitrate from the fabrics. The other set of the treated fabrics was exposed to oil smoke to determine if chemical treatment had a protective effect against the oil smoke on the ammonium nitrate treated samples. The fabric samples were extracted according to previously described methods.

Oil smoke study: One set of fabrics were treated with ammonium nitrate solution and then exposed to oil smoke by burning the oil in a flask and setting the fabrics in inverted test tubes on a ring stand to allow the smoke from the oil to settle onto the fabric. The fabrics were subjected to the oil smoke for 10 minutes. Another set of fabrics was treated with Scotch-Gard™, exposed to ammonium nitrate solution and then exposed to oil smoke described above. The fabric samples were extracted according to previously described methods.

Ammonium nitrate fuel oil: ANFO in 6.01% diesel fuel oil was obtained and deposited onto the fabrics. ANFO is a loose powder

that is easily transferred between objects, thus 100 mg of ANFO was pressed and rubbed onto each of the fabrics to ensure adequate transfer. The fabric samples were extracted according to previously described methods.

Mathematical and statistical analysis procedures: In all of the experiments, a sample population of 6 fabrics was used to calculate the mean and standard deviation for the ammonium and nitrate ion percent recoveries. The determined extraction efficiency for each fabric type (previously described) was used as a correction factor for the mass of ammonium and nitrate reported after exposure to one of the variables. A Student's t-test was used to determine if there were significant differences in the results at the 95% confidence level.

Results and Discussion

Wind and rain

Exposure of the fabrics to wind conditions did not result in any significant loss of ammonium nitrate from the fabrics. When the fabrics were exposed to simulated heavy rain, (harsh and fast rain shower by spray for 5 minutes), the ammonium ion recovery was still above the 0.50mg/L LOQ of the instrument for all fabrics with the exception of Fabric 341, which was still well above the 0.50mg/L LOD (Table 2). It is interesting to note that while the ammonium and nitrate measured in the collected rain was 0.017mg/L and 4.0mg/L respectively, the levels we obtained for the nitrate were still well below this level.

Oil smoke

Exposure of the fabrics to oil smoke for 10 minutes did not affect the ability to measure quantitate these ions all of the fabrics. While some of the fabrics showed significantly greater percent recovery over the control, other fabrics showed significantly lower percent recovery when compared to the control (Table 3). When the fabrics that were treated with Scotch-Gard™ were exposed to oil smoke there was no significant difference compared to the fabrics that were not treated with Scotch-Gard™ but exposed to oil smoke.

High temperatures and 50% or 90% humidity

Exposure of the fabrics to fifty percent humidity and 90°C temperatures did not have a significant impact on the ability to recover ammonium and nitrate ions. On the other hand, exposure of the fabrics to 90% humidity and 90°F temperatures resulted in

an average of 37.7% less ammonium recovered from all fabrics in comparison to the 50% humidity setting; for nitrate, and an average of 80.9% less nitrate ions being recovered from all fabrics.

Figure 1, shows that the average percent loss for all fabrics is most significant in the first 3 hours. When the fabrics are exposed to 90°F temperatures and 90% humidity, there was a 58.4% loss of the ammonium and 78.5% loss of nitrate ions, whereas when the fabrics were exposed to temperatures of 90°F and 50% humidity, there was a 53.3% for the ammonium ion and 32.1% for the nitrate ion. Furthermore, the percent loss of ammonium and nitrate ions increases over time at 90°C and 90% humidity by more than 5% by 24 hours, whereas when exposed to 90°C and 50% humidity the percent of ammonium and nitrate ions is not significantly different between 3 and 24 hours. After 24 hours the levels of ammonium and nitrate were still above the LOQ for both set of conditions and thus in these conditions ammonium and nitrate deposited onto such fabrics can still be quantitated.

These results were compared to cooler temperatures and lower humidity in an uncontrolled outdoor study carried out to 72 hours where fabrics were exposed to average temperatures of 37.5°F (30-45°F) and an average humidity of 35% (30-40%) with moderate precipitation between 48 and 72 hours (Figure 2). After 6 hours, an average of 20.4±1.71 mg of ammonium remains; after 72 hours 4.37±2.39 mg is still detected. After 6 hours, an average of 38.0±4.32 mg of nitrate remains; after 72 hours 3.05±2.08 mg is still detected.

Ammonium nitrate fuel oil

In order to determine if our method was able to detect ammonium and nitrate ions in the presence of fuel oil (such as with ammonium nitrate fuel oil (ANFO) explosives), we deposited ammonium nitrate in 6.01% fuel oil onto the fabrics and extracted. We found that fuel oil does decrease the percent of ammonium and nitrate ions recovered from the fabrics when compared to extracting the same amount of ammonium nitrate without the presence of the fuel oil. Table 4 shows that fabric 341 (nylon) was the most affected by the presence of the fuel oil, decreasing the recovery ammonium and nitrate by 18% and 11%, respectively. The fabric 4119 (polyester) was significantly affected by the presence of the fuel oil in regards to the ammonium ion, which decreased by a little over 35%, whereas the recovery of the nitrate ion was not significantly affected by the presence of fuel oil. The fabric 448 (velour) was affected significantly in regards to the nitrate ion, which decreased to 14.4%, but not the ammonium ion. The least affected fabric by the presence of the fuel oil was the GM vinyl fabric (562), which did not exhibit any decrease in the percent recovery of ammonium and nitrate ions.

Scotch-Gard™ treated fabrics

Fabrics are often treated with chemicals to protect and preserve the fabric from stains and wear patterns. We looked at the effect that fabric treatments would have on recovery of ammonium and nitrate ions. In Table 5, the percent recoveries for ammonium and nitrate ions from fabrics treated with both Scotch-Gard™ and ammonium nitrate are reported. In comparing the percent recovery of the Scotch-Gard™ treated fabrics with ammonium nitrate to fabrics only treated with ammonium nitrate, we found that most fabrics showed an increase in percent recovery of ammonium and nitrate. We also looked at the effect that Scotch-Gard™ treated fabrics would have on the ability to recover ammonium and nitrate ions from fabrics that were exposed to oil smoke as described above. These results also showed an increase in percent recovery of ammonium and nitrate

from most fabrics between those treated with Scotch-Gard™ and those that were not treated.

Conclusion

We have shown that ammonium and nitrate ions can be recovered and measured from four fabrics that are typically used in Ford, General Motors, and Chevy trucks. Exposure of these fabrics to a variety of environment conditions including wind, rain, oil smoke, and high temperatures and high humidity after 24 hours still resulted in the ability to detect the ammonium and nitrate ions. It was also found that treatment of the fabrics to SG resulted in an increased recovery for both ions for most fabrics. Conditions of wind, oil smoke and exposure to temperatures of 30-40°F and average humidity of 30% to 40% (after 72 hours) resulted in the lowest loss of ammonium nitrate from the fabrics. Conditions of rain and 90°C temperatures with 90% humidity resulted in the greatest loss of ammonium nitrate from the fabrics, but the levels remaining were still above the method limit of detection. In a typical post blast analysis, recovery of the evidence will occur within 24 hours. Our results show that ion chromatography with conductivity detection could be used as an analytical method for screening post-blast residues deposited on truck fabrics of a detonated IED containing ammonium and nitrate ions exposed to any of the variables noted above. It is further demonstrated that certain fabrics are more suitable for analysis than others; nonporous and semiporous were consistently the best fabrics for analysis due to their highest percent recoveries for ammonium and nitrate ions, and that overall these fabrics were the least affected by the conditions the fabrics were exposed to in this study.

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