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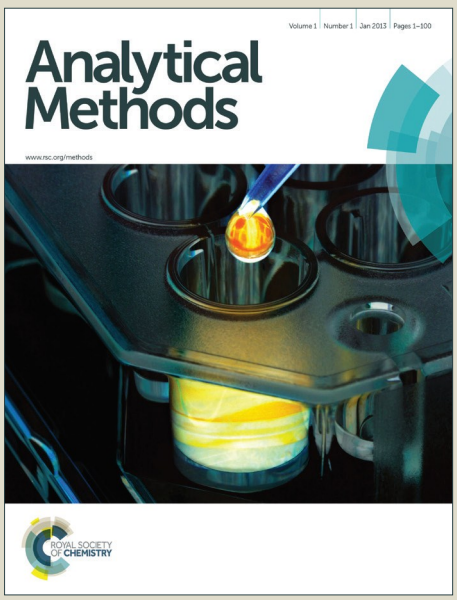

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Mapping Explosive Residues on Galvanized Pipe Bomb Fragments Using Total Vaporization Solid Phase Microextraction (TV-SPME)

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Solid phase microextraction (SPME) is a popular sampling technique whereby analytes are sorbed to a coated fiber and subsequently desorbed into an analytical instrument. In headspace SPME, analytes partition between the sample, the headspace above the sample, and the SPME fiber coating. In total vaporization SPME (TV-SPME), sample extracts are heated until both the solvent and analytes completely vaporize, whereupon the analytes partition between the vapor phase and the SPME fiber. In this study, TV-SPME using a polyethylene glycol fiber was coupled with fast gas chromatography/mass spectrometry to identify components of double-base smokeless powder (DBSP). Nitroglycerin (NG), diphenylamine (DPA) and ethyl centralite (EC) were separated in under 5 min. For NG, the optimal sample volume (70 µL), extraction temperature (60 °C) and extraction time (20 min) resulted in a method that was over twelve fold more sensitive than traditional liquid injection and with a detection limit below 1 ppb. This method was then used to quantify DBSP residue on post-blast debris from five galvanized steel pipe bombs. The mean concentration of NG on the fragments was 0.25 ppm (w/w). An average of 1.01 mg of NG was recovered from the devices. Finally, the distribution of NG could be "mapped" by tracking the original locations of each fragment within the device. These maps showed that the distribution of NG was far from uniform. In fact, the concentration of the NG on fragments originating from the end caps was several fold higher than in other locations. This finding can help guide the selection of bomb fragments for chemical analyses in real-world scenarios.

Introduction

The identification of explosive residues at bombing scenes and on post-blast debris plays an important role in explosives investigations. This can determine what explosive was originally present, which may link the device to a particular suspect. In the absence of intact explosive particles, the standard methodology involves extracting one or more pieces of debris with an organic solvent (i.e., dichloromethane and/or acetone) and then analyzing the extract(s) via infrared spectroscopy and/or liquid injection GC/MS[1]. Specific guidelines on the analysis of post-blast debris have been established by the Technical Working Group on Fires and Explosions (TWGFEX)[2, 3].

Solid-Phase Microextraction (SPME) is a popular and wide-spread pre-concentration sampling technique where analytes are sorbed onto a coated fiber and then desorbed into the inlet of an analytical instrument[4-7]. The use of SPME in forensic science has been well-established for many years[8]. Typically, SPME is carried out in either headspace or immersion mode. In headspace SPME, the fiber extracts analytes from the headspace above a sample. In immersion SPME, the fiber extracts analytes directly from a liquid sample.

Headspace SPME sampling has been used extensively in the analysis of intact explosives. For example, triacetone triperoxide (TATP) was detected from headspace using planar SPME with an ion mobility spectrometer[9]. Nitroaromatic explosives in air and soil have been analyzed using headspace cavitand-based SPME gas chromatography-mass spectrometry (GC-MS)[10]. Finally, studies using headspace SPME-GC/MS have been used to determine the volatiles that are associated with explosives such as smokeless powder, PETN-based sheet explosive, Composition C-4 and TNT[11-16].

Immersion SPME sampling has been used in environmental applications to extract organic explosives in water and/or aqueous soil extracts followed by GC-MS and GC-electron capture detection[17, 18]. Some specific examples of explosives that have been identified in this way include 2,6-dinitrotoluene, TNT, PETN, NG (dynamite) and RDX (Composition C-4)[19-21]. Neither headspace nor immersion SPME is routinely applied to the analysis of explosive residues on post-blast debris, despite several previous reports describing its unique utility, such as the analysis of single particles of smokeless powder[22] or extraction of explosive residues from soil samples gathered from the blast seat following an explosion[21, 23].

Total vaporization (TV) is a technique that has been used previously in conjunction with dynamic headspace sampling. When applied to solid samples, residual solvents are released from the matrix. When used with liquid samples, the entire sample is vaporized prior to headspace sampling with a gas

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syringe. This technique has been applied to various samples such as residual solvents in solids[24], odor compounds in aqueous solutions[25] and ethanol in fermentation liquor[26].

The approach described in this paper couples TV and SPME (TV-SPME), which was recently demonstrated as offering greater sensitivity and lower detection limits for nicotine and cotinine in the hair of tobacco users[27]. TV-SPME is a technique where a sample extract is heated until it vaporizes and a SPME fiber is used to pre-concentrate analytes from the resultant vapor[27]. At equilibrium, the volume of sample that can be analyzed by TV-SPME is directly related to the properties of the solvent and the extraction temperature, as expressed by Equation (1):

$$V_s = \left(\frac{\left(10^{A-\frac{B}{T+C}}\right)V}{RT}\right)\left(\frac{M}{\rho}\right) \quad (1)$$

Where V_s is the volume of sample (mL), A, B, and C are the Antoine constants describing the solvent vapor pressure at temperature T (K), V is the total volume of the vial (L), R is the ideal gas constant (8.3145×10^{-2} L bar/K mol), *M* is the molar mass of the solvent (g/mol), and ρ is the density of the extract solvent at room temperature (g/mL).

Note that equation (1) describes equilibrium conditions, however quantitative extractions are able to be achieved regardless of whether the system reaches equilibrium. In a non-equilibrium situation, the amount of analyte extracted is dependent on the extraction time, which is represented by a simple exponential term. When the extraction time is constant, the extracted amount is proportional to the initial concentration[5].

Combining total vaporization with SPME is conceptually similar to large volume injection (LVI) techniques in that large sample volumes (e.g., ~300 µL) result in increased sensitivity. However, in TV-SPME there is no need for modifying the GC instrument such as adding retention gaps or exits for solvent vapor. In addition, filtration of sample extracts is not necessary as any insoluble or non-volatile components remain on the surface of the vial.

A critical feature of TV-SPME is that despite the evaporation of the liquid sample into a much larger volume, the ability of the SPME fiber to pre-concentrate the analyte from the vapor more than compensates for this dilution. In addition, proper choice of SPME fiber chemistry can add selectivity to the analysis.

Post-blast Analysis of Pipe Bomb Fragments

The samples of interest to this paper originate from pipe bombs, which consist of a rigid container (the pipe with end caps), a low explosive filler and a chemical fuse. Given their simplicity and ease of construction, pipe bombs are a common form of improvised explosive device (IED) in the United States. For example, materials such as pipes and endcaps are found in most hardware stores, and low explosive propellants are widely available at sporting goods stores. In particular, double-base smokeless powder (DBSP) is a popular propellant that is based on nitrocellulose and nitroglycerin (NG). DBSP also contains stabilizers and burn-rate modifiers such as diphenylamine (DPA) and ethyl centralite (EC).

Residue from the explosive filler in a pipe bomb can be identified on post-blast container fragments using a variety of spectroscopic, chromatographic or mass spectrometry methods[28]. In particular, smokeless powder constituents can be identified using ultra performance liquid chromatography/tandem mass spectrometry (UPLC/MS/MS)[29], gas chromatography coupled to either a thermal energy analyzer (GC/TEA) or a mass spectrometer (GC/MS)[30], and capillary electrochromatography[31].

It is important to note that, in practice, the amount of residue is not quantified. This is true because explosives investigators wish to know what explosive is present, not necessary how much. This paper does not seek to contradict that view. Instead, we present a quantitative approach to understand, in a general sense, the distribution of explosive residue on pipe bomb fragments. In turn, this "residue mapping" may indicate what portion of the device is most likely to yield higher levels of residue. In addition, the actual concentration of residue on device fragments dictates the sensitivity and detection limit of any analytical scheme that is applied. Lastly, mapping of the residue may shed light on the specific process by which a pipe bomb container fails and then fragments. Thus far, this has only been studied using high-speed filmography[32, 33].

Overall, this paper reports several novel findings: the use of SPME (TV-SPME in particular) to analyze trace residues of low explosives on actual post-blast debris, the quantitation of these residues on device fragments, and the determination of how these residues are distributed within the device itself.

Materials and methods

Materials

Nitroglycerin (1 mg/mL) was purchased from Restek. Diphenylamine (ACS grade) was purchased from Acros Organics. Methylene chloride (HPLC grade), ethyl centralite (99%) and all SPME fibers were purchased from Sigma Aldrich. Galvanized steel (8" x 1"diameter) and cast iron endcaps (1" diameter) were purchased at Home Depot, and the Alliant Red Dot double-base smokeless powder was obtained from Gander Mountain. SPME vials and caps were acquired from Gerstel.

Instrumental Analysis

A Thermo Trace Ultra GC with a DSQ II MS and a TriPlus Autosampler was used for all analyses. Samples were incubated for 5 minutes at the desired extraction temperature. Various extraction temperatures and times were used and are discussed below. After extraction, the SPME fibers were desorbed in the GC inlet for 1 minute. A PTV inlet ramp was used with the initial temperature at 200 °C for 0.21 minutes, ramped 10 °C/s to 250 °C and held for 0.21 minutes. The fiber was then conditioned offline at 240 °C for 3 minutes. The column used was a Zebron ZB5-MS with dimensions of 10 m x

0.18 mm x 0.18 µm. Helium was used as the carrier gas with a flow rate of 1.5 mL/min. The oven program began at 40°C for 1 min, then it was ramped at 45 °C/min to 250 °C, immediately set to 300 °C, and then held for 1 min. The transfer line to the MS and the ion source were both held at 250 °C. Pulsed positive ion negative ion chemical ionization (PPINICI) was used with a methane reagent gas flow of 1.3 mL/min. Selected ion monitoring (SIM) was used to detect nitroglycerin (m/z 62 in negative mode), diphenylamine (m/z 170 in positive mode) and ethyl centralite (m/z 269 in positive mode). The total scan time was 0.1 s and the dwell times were 5 ms.

Effect of Fiber Chemistry

Preliminary experiments were conducted to compare several SPME fiber chemistries. A set of calibrants consisting of 5 ppb-5 ppm nitroglycerin in dichloromethane were prepared. Four fibers were evaluated: polydimethylsiloxane (PDMS), polydimethylsiloxane-divinyl benzene (PDMS-DVB), polyethylene glycol (PEG) and polyacrylate (PA). In each case, 50 µL of each calibrant was extracted at 50 °C for 30 min. The fibers were desorbed at 200 °C in the inlet for 1 min. The fiber was conditioned offline at 240 °C for 2 min. The column used in this study was a Zebron ZB5-MS with dimensions of 60 m x 0.25 mm x 0.25 µm. Hydrogen was used as the carrier gas with a flow rate of 2.0 mL/min. The oven program began at 40 °C and was ramped 20 °C/min to 320 °C and held 1 min. The transfer line was 220 °C and the ion source was 200 °C. Electron impact ionization was used in SIM mode with m/z values of 46 and 76 (NG).

Effect of Sample Volume

A study of the effect of sample volume with either a constant concentration (0.5 ppm) or a constant mass of NG, DPA and EC (50 ng) was completed. Sample volumes of 50 µL, 60 µL, 70 µL, 80 µL, 90 µL and 100 µL were analyzed in 20 mL SPME vials. The extraction time was 20 min and the extraction temperature was 60 °C.

Optimization

Many parameters are incorporated into a TV-SPME method, including SPME fiber type, extraction temperature, extraction time, desorption temperature, desorption time, and sample volume. The effect of some of these variables in headspace and immersion SPME of explosives has been explored[34]. For this study, response surface methodology (RSM) and central composite design (CCD) were utilized to optimize the system[35-37]. RSM uses statistical techniques to analyze responses that are dependent on numerous variables. The ultimate goal is to optimize the response. A second order RSM model was used in this paper, as shown in Equation (2):

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum \sum_{i<j} \beta_{ij} x_i x_j + \in \quad (2)$$

where y is the response, β_0 is a constant, β_i is the coefficient of the linear term, x_i is the linear variable, β_{ii} is the coefficient of the square term, x_i^2 is the square variable, β_{ij} is the coefficient of the interaction terms, $x_i x_j$ is the interaction variable term, $\in$ is the error in the response, and k is the number of variables.[38] The variable terms are coded to show values on a scale from -1 to +1.

In order to get the most effective results, a proper experimental design must also be used. CCD is the most popular design used to fit response surfaces. In CCD, two parameters are chosen which will determine the design for fitting the model: α, which is the distance of the axial points from the center value and n_c, which is the number of center points. The parameter n_c is selected to provide adequate experimental data to properly model the response (e.g., >3)[9].

In this case, a face-centered CCD with α=1 and n_c=6 was used to optimize NG, DPA, and EC. The three parameters and ranges studied were incubation temperature (40-120 °C), extraction time (5-30 min) and sample volume (10-50 µL). A constant mass of NG was used in all studies (50 ng). This required 20 experimental runs. In all cases, a polyethylene glycol (PEG) SPME fiber was used.

Sensitivity Comparison

Comparison to liquid injection involved preparing a series of nitroglycerin calibrants in DCM ranging from 1 pg/mL - 1 µg/mL. These were analyzed using the optimized TV-SPME method with an extraction time of 20 min at 60 °C. The same solutions were also analyzed via liquid injection, with 1 µL of each calibrant injected with a total splitless time of 1 min.

Pipe Bomb Study

Assembling and functioning of the pipe bombs was completed by the Indiana State Police Bomb Squad.

Prior to assembly, the exterior of the pipe and endcaps were color coded with paint so that the assembled devices had five distinct sections: left end cap (1.8 in x 1.2 in), left pipe body (1.2 in x 2.67 in), center pipe body (1.2 in x 2.67 in), right pipe body (1.2 in x 2.67 in), and right end cap (1.8 in x 1.2 in). In the device, the overlap of the endcap over the threaded portion of the pipe was 0.35 in at each end.

Blast cages constructed of a welded steel frame and two layers of metal grating were used to trap as many fragments as possible. Approximately 50 g of Alliant Red Dot DBSP was used in each device. A time fuse inserted through a hole in the right endcap was used to initiate each device. After the explosions, fragments from within the cages were collected by gloved personnel and placed in paint cans specific to each device. The pipe bomb fragments were then transported to the laboratory and stored at room temperature until needed. Prior to extraction, the fragments were sorted by pipe location/color and photographed as a whole. Each fragment was assigned an identification number according to the convention, device

number – location – number. Fragments were also photographed individually, weighed, and placed in plastic bags.

Extraction of Fragments

Each pipe bomb fragment was placed in a small, medium or large screw-top glass jar depending upon the fragment's size. Volumes of 10 mL, 20 mL, or 50 mL of dichloromethane were added to the jars using volumetric pipets. The jars were closed, sealed with wax film and then placed on a shaker table for 15 min. 70 µL of the extract was transferred (without filtering) to a SPME vial for analysis using the optimized TV-SPME/GC/MS method.

Results and discussion

In the discussion that follows, NG, DPA and EC were analyzed under various conditions. However, there will be an inherent emphasis on the determination of nitroglycerin based upon the focus of forensic science laboratories. Under most forensic protocols, identifying NG on post-blast debris is required in order to report that residues of double-base smokeless powder were present. In contrast, the stabilizers and other compounds in smokeless powder can help identify the brand of the powder, but they are not unique to the explosive.

Prior to systematically gathering data, several internal standards were considered for use in the quantitation of nitroglycerine. The candidates included nitropropane (b.p. 131-132 ^{o}C), nitrobenzene (b.p. 210-212 ^{o}C) and triacetin (b.p. 257-259 ^{o}C). The relative response of nitropropane was very low whereas the response of nitrobenzene and triacetin were not sufficiently reproducible between runs.

The use of external standardization was further justified by determining the extraction efficiency of the method. Extracting three steel post-blast fragments twice in succession proved that the first extraction was exhaustive and the mean recovery was 99.9% of the NG present. Lastly, the accuracy of external standardization was confirned by using a 0.1 ppm test mix to challenge the calibration curve ranging from 3 ppb to 1 ppm. The mix was calculated experimentally to be 0.102 ppm, representing a 2% error.

Effect of Fiber Chemistry

The results of a SPME fiber comparison are summarized in Table 1. By far, the more polar fibers (PA and PEG) exhibited the greatest sensitivity, exceeding that of the PDMS and PDMS-DVB fibers by almost two orders of magnitude. The PEG fiber was ultimately selected as it also exhibited the widest linear range, spanning three orders of magnitude.

Table 1: Effect of fiber chemistry on the linear range, sensitivity, and linearity of TV-SPME for nitroglycerin.

Fiber	Linear Range	Slope	R^2
PDMS	50 ppb – 5 ppm	2.47×10^6	0.987
PDMS-DVB	10 ppb – 5 ppm	1.84×10^6	1.000
PA	50 ppb – 5 ppm	1.18×10^8	0.998
PEG	5 ppb – 5 ppm	1.26×10^8	0.997

Effect of Sample Volume

Various volumes of NG, DPA and EC standards in methylene chloride were analyzed using a PEG fiber at 60 ^{o}C. In this case, one set of calibrants had the same concentration for all analytes (0.5 ng/µL) whereas the other set of calibrants had differing concentrations so that the total amount of each analyte in the vial was fixed at 50 ng. The calculated maximum volume of methylene chloride that can be vaporized at 60 °C in a SPME vial was 95 µL. This is based upon Equation (1) and a calibration of the volume of the SPME vials using water (20.9 ± 0.1 mL).

As shown in Figure 1, when sample volume increases and the mass of NG is constant, the response is initially flat (as expected) followed by a rapid decrease at volumes larger than 70 µL. On the one hand, it would be expected that the response in TV-SPME would drop precipitously once the sample volume exceeds the calculated maximum. Under these conditions, some portion of the liquid sample would remain and significantly perturb the distribution of analyte. The fact that this decline actually begins at much lower sample volumes may be due to the concentration of DCM vapor in the vial, which exceeds 33 ppm (v/v) with sample volumes greater than 70 µL. Given that the fiber coating does not swell (as verified by immersing the fiber in DCM), a decrease in the distribution coefficient would result in a decrease in sensitivity.

When sample volume increases and the mass of NG is also increasing, the response reaches a maximum at 70 µL followed by a less dramatic decline. This is consistent with the competing effects of decreased partition coefficient (as discussed above) and increasing mass of analyte. Based upon these results, the experimentally determined maximum of 70 µL for NG was used for the remainder of the study.

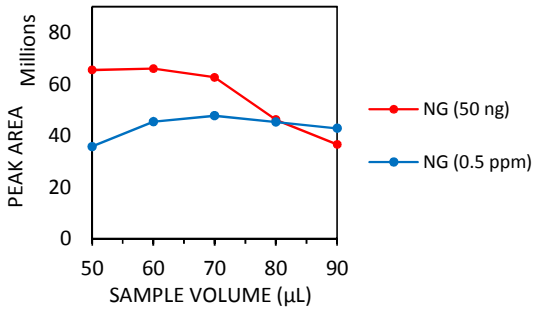

Figure 1: Response to nitroglycerin (m/z 62) as a function of sample volume at 60°C.

Optimization

In the RSM optimization, the amount of each solute was held constant in all vials by adjusting the concentration of the solutions. It became clear that the recovery of NG was much more sensitive to temperature than DPA and EC. Figure 2 shows three of the twenty optimization runs that utilized an extraction time of 17.5 min but with differing extraction temperatures. The peak intensities have been normalized to the response at 40 °C.

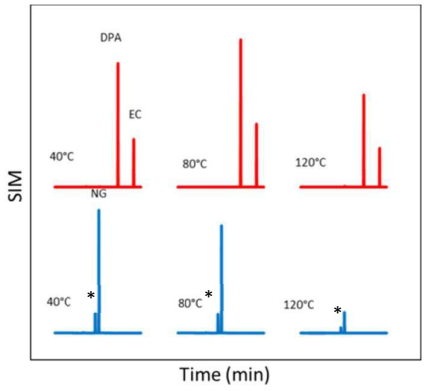

Figure 2: TV-SPME chromatograms of smokeless powder components using an extraction time of 17.5 min and three different extraction temperatures (Top: positive m/z 170 + 269; bottom: negative m/z 62). The peak marked with star (*) has been identified as dinitroglycerin (see text).

As can be seen in Figure 2, NG is also exhibiting some amount of chemical degradation in the GC inlet, resulting in two chromatographic peaks. The thermally instability of NG has been reported elsewhere[39]. The degradation product results from the hydrolysis of one of the nitro functional groups on trinitroglycerin to form dinitroglycerin. Additional experiments varying the inlet temperature program (data not shown) have indicated that this peak can be significantly reduced by using a lower inlet temperature.

The optimized parameters for NG, DPA and EC are shown in Table 2 along with the overall optimum. Desirability ranges from 0 to 1 and is an indicator of how well the calculated parameters result in the optimum response. The desirability of the global optimum is noticeably lower than the optima that were found for each single component. This is primarily due to the large difference in optimal extraction temperature for NG, DPA and EC. Given the focus of this study, the optima determined for NG were used for all subsequent experiments. The ideal sample volume was determined to be 50 µL, the maximum volume investigated. Due to this, a separate volume study was done (see previous) with an expanded range to determine the optimal value.

Table 2: Results of the CCD optimization of TV-SPME parameters for DBSP components (R^2=0.81).

Analyte	Optimal Extraction Temperature (°C)	Optimal Extraction Time (min)	Desirability (0 – 1)
NG	60	20	0.990
DPA	80	20	0.974
EC	108	22	0.903
All	80	20	0.756

Figure 3 shows the results of a separate extraction time study spanning the same range as the optimization, 5-30 minutes. For extraction times up to 20 minutes, the signal for all three components increases. However, by 30 minutes, the signal for NG has significantly decreased, whereas the signal for DPA and EC have leveled off. Based on these results as well as those obtained during the optimization, a 20 minute extraction time was used for the remainder of the study.

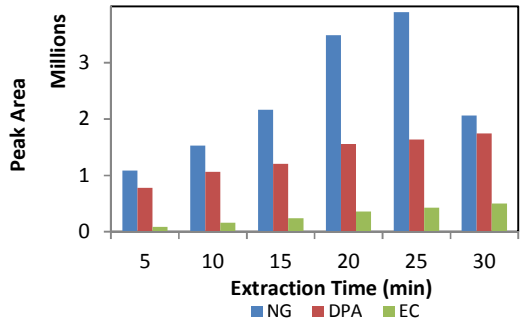

Figure 3: Comparison of peak area relative to extraction time for three double base smokeless powder components.

Sensitivity

The current "gold standard" for determining smokeless powder residues on bomb fragments is liquid injection GC/MS. Calibration curves were generated for nitroglycerin over a range of 10 ng/mL to 1 µg/mL using both liquid injection and TV-SPME injection. The slope was calculated for both plots, and the sensitivity for TV-SPME was more than an order of magnitude larger than liquid injection. Furthermore, the signal to noise for the 10 ng/mL calibrant was over an order of magnitude higher using TV-SPME (Table 3). The estimated limit of detection for NG using the TV-SPME method is 100 pg/mL (S/N = 5).

Table 3: Sensitivity and linearity for nitroglycerin by liquid and TV-SPME injection.

Method	Slope	R^2	S/N (10ng/mL)
Splitless (1 µL)	2.05×10^6	1.00	37
TV-SPME (70 µl)	2.52×10^7	0.98	399

Analysis of Pipe Bomb Fragments

The optimized TV-SPME method was then applied to real post-blast pipe bomb fragments. A summary of the masses of the container, propellant and residues is shown in Table 4.

Table 4: Summary of results for the steel devices.

	Device	1	2	3	4	5
Pipe	Initial Mass (g)	724.93	740.71	737.96	738.44	744.83
	Mass Recovered (g)	617.46	489.59	437.62	729.21	505.69
	% recovery	85	66	59	99	68
	# fragments	37	54	50	36	47
Smokeless Powder	Total (g)	52.08	52.10	52.03	52.02	52.03
	NG (g)	9.37	9.38	9.37	9.36	9.37
Post-Blast Residues	NG (mg)	1.14	0.61	0.47	2.20	0.61
	DPA (µg)	-	22.42	11.99	12.90	2.00
	EC (µg)	-	3.61	3.89	-	-

Figure 4 shows a sample chromatogram for a galvanized steel pipe bomb fragment. Nitroglycerin and diphenylamine were able to be quantified. Ethyl centralite was present in a few extracts, but in others it was below the limit of quantitation.

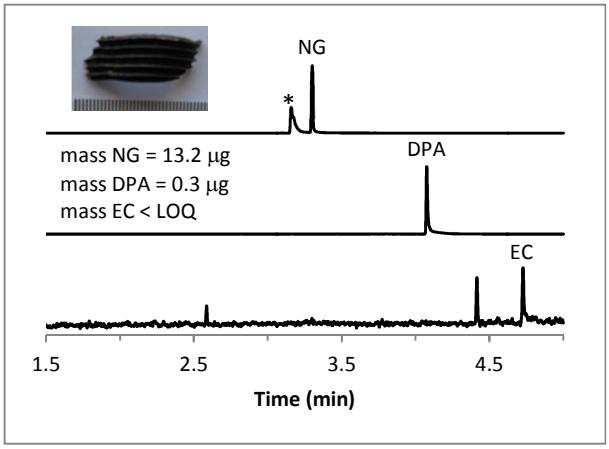

Figure 4: Photo (scale in mm) and chromatogram for a post-blast steel fragment (Top: negative m/z 62; middle: positive m/z 170; bottom: positive m/z 269). The peak marked with a star (*) has been identified as dinitroglycerin (see text).

The mass of NG recovered from different locations on the devices is shown in Figure 5 as a color-coded "heat map". In all five devices, the highest mass of NG was located on the endcap. The star represents where intact DBSP particles were found, leading to a higher recovery of NG in that location.

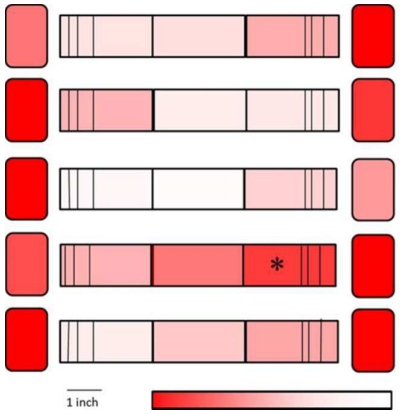

Figure 5: Heat maps of the five devices showing the NG distribution. The color scale is normalized to the highest amount of NG within each device (* indicates the location of the intact DBSP particles).

In similar fashion to NG, the highest concentrations of DPA were located on or near the endcaps (data not shown). The total amount of DPA recovered was much lower than NG, averaging 12.3 µg. The devices yielded a total of 7.5 µg of EC.

The extent to which explosives residues accumulate preferentially on the end caps of a pipe bomb has not been reported previously. High speed video footage of pipe bomb explosions has shown that steel devices rupture first at one of their end caps[32, 33]. Therefore, the end cap regions of a pipe bomb may inherently capture and/or shield the explosives residue from the heat of the blast regardless of how the device container initially fails. This trend will need to be further confirmed in additional devices.

Conclusion

A TV-SPME method has been designed, characterized and optimized for the analysis of explosive residues on pipe bomb fragments. In this work, sample volume, incubation temperature, and extraction time of the TV-SPME method were optimized. Optimized parameters for nitroglycerin were a 60 °C incubation temperature, a 20 minute extraction time, and a 70 µL sample volume. Additionally, sensitivity was compared to liquid injection, and TV-SPME was more than 12-fold more sensitive with lower detection limits (i.e., less than 1 ng/mL).

When applied to actual pipe bombs, this method determined that the mean concentration of nitroglycerin on the steel fragments was 0.25 ppm (w/w) and the mean mass of NG recovered was 1.0 mg. Fragments from the end caps yielded the highest amount of NG and DPA. These results add to the understanding of how small IEDs function as well as inform analysts regarding the sensitivity that is required for post-blast analysis of smokeless powder. In the future, other types of smokeless powder (single and triple based) could be investigated. Additionally, this technique could be applied to other container types, such as PVC.

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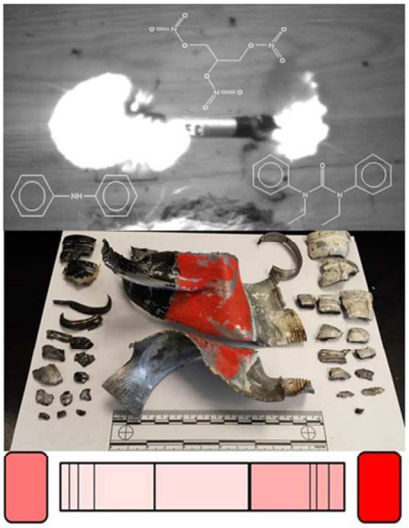

Graphical Abstract
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