Fast Detection of Triacetone Triperoxide (TATP) from Headspace using Planar Solid Phase Microextraction (PSPME) Coupled to an IMS Detector

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Abstract Triacetone triperoxide (TATP) is a high explosive synthesized from easily available reactants making it accessible for illicit uses. In this study, fast detection of TATP is achieved using a novel planar solid phase microextraction (PSPME) as a preconcentration and sampling device for headspace analysis offering improved sensitivity and reduced sampling time over the conventional fiber-based solid phase microextraction (SPME) when followed by ion mobility spectrometer (IMS) detection. Quantitation and comparison of the retention capabilities of PSPME as compared to the commercially available SPME were determined using TATP standards and analyzed using gas chromatography-mass spectrometry (GC-MS) for SPME analysis and a commercial IMS with no instrumental modification for PSPME. Static and dynamic headspace extractions were used and compared for PSPME extractions, in which low mg quantities of TATP were detected within 30 seconds of static mode sampling and less than 5 seconds in the dynamic mode sampling for PSPME-IMS.

Keywords *Planar solid phase microextraction (PSPME), Solid phase microextraction (SPME), Ion mobility spectrometer (IMS), Triacetone triperoxide (TATP)*

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Introduction

Triacetone triperoxide (TATP) was first discovered and prepared in 1895 by Wolffenstein [1]. Its extreme sensitivity to friction, shock and impact makes it unfavorable for many commercial or military use [1]; however, the ease of synthesis from readily available chemicals, the simple requirements of preparation and the detonation effect attract much interest in criminal and terrorist activities [2,3]. Thus, development of a fast, on-site, contact-free and reliable method for the detection of TATP has received increasing attention within the last decade [4,5].

Since TATP lacks in nitro group and aromatic functionalities, well-established detection methods for the nitro-containing explosives such as 2,4,6-trinitrotolulene (TNT), nitroglycerin (NG) and 1,3,4-trinitro-1,3,5-triazacyclohexane (RDX) are not suitable for this peroxide-based compound [6,7]. Various analytical methods have been developed for the separation and detection of TATP for different purposes. Liquid chromatography (LC) [8] and gas chromatography (GC) [9] are used for the separation of TATP and Infrared [6] and Raman spectroscopy [6,10] techniques have been used to detect TATP in the laboratory. In addition, TATP can also be detected by either mass spectrometry which includes desorption electrospray ionization (DESI) [11] and selected ion flow tube (SIFT) [12] or other sensor-based techniques such as luminescence [13,14], electrochemical [15-17], and biological [18] sensors. However, none of these techniques can fully accomplish two important detection requirements; fast on-site analysis with unambiguous identification and low limits of detection from post-explosion debris with high selectivity. Ion mobility spectrometry (IMS) is another robust tool for the detection of TATP [19,6,20-22] and has been used by law enforcement and homeland security in airports, government buildings, and at border crossings to detect explosives for several decades due to the characteristics of fast detection, high sensitivity, ease of use and on-site analysis, making this detection system attractive over other techniques [23]. However, IMS has not been widely exploited for the detection of TATP and limited research has been published due to the lack of efficient sampling methods that can be coupled to this instrument.

In 2010, a novel preconcentration and sampling technique, planar solid phase microextraction (PSPME) [24], was reported for rapid air sampling in the field. The PSPME devices can be easily and inexpensively made in the laboratory and have similar chemical characteristics as the solid phase microextraction (SPME) fiber [24]. The increased surface area of PSPME significantly shortens the extraction time and increases the extraction efficiency over the fiber SPME and analytes extracted onto the PSPME device can be thermally desorbed into a commercial IMS instrument without modification of the IMS inlet. PSPME extractions can be accomplished in two different modes; static sampling and dynamic sampling. Static sampling is similar to the SPME equilibrium extraction from a confined headspace. Dynamic sampling is assisted with a continuous pumped flow of air through the PSPME device and has been shown shorten sampling time [24] and improve the detection over fiber SPME. Additionally, PSPME does not suffer from potential for SPME fiber breaking and limited adsorption/absorption capacity [25,26].

In this study, we describe a fast method of sampling TATP from headspace followed by detection of IMS within seconds. In addition, a comparative study between SPME and PSPME was conducted to determine the increased retention capability and faster sampling time as a result of

increased surface area and phase volume. Additionally, the PSPME device can be coupled to a portable IMS instrument to allow for on-site analysis with high sensitivity and selectivity. Furthermore, this contact-free air sampling approach reduces the potential for clutter and background commonly found in real-world contact sampling scenarios.

Experimental Section

Instrumentation

Headspace sampling of solid TATP was carried using previously described SPME [27] and PSPME [24] devices and coupled to a GE Ion Track (Wilmington, MA) ITEMIZER 2 IMS instrument. GE N-mode (positive mode) calibration traps containing cocaine were used to calibrate the instrument in the N-mode. Further TATP detection experiments were performed using the Smiths Detection IONSCAN®-LS (Smiths Detection, Warren, NJ) IMS. For this instrument, two different dopants were used, nicotinamide (original dopant in the positive mode this instrument) and isobutylramide purchased from Smiths Detection [28]. The IMS operating conditions for both IMS instruments are shown in Table 1.

Table 1 Conditions for the IMS instruments used as detectors for both SPME and PSPME sampling/preconcentration.

IMS operating conditions	GE Ion Track ITEMIZER	Smiths Detection IONSCAN®-LS
Polarity	Positive (+)	Positive (+)
Desorber temperature (°C)	175	250
Drift tube temperature (°C)	195	235
Sample flow (mL min^{-1})	500	200
Drift flow (mL min^{-1})	350	351
Reagent gas	Ammonia	Nicotinamide/ isobutyramide

Absolute mass quantitation with SPME was analyzed using a Varian (Palo Alto, CA) CP 3800 gas chromatograph coupled to a Saturn 2000 ion trap mass spectrometer and equipped with an 8200 auto sampler (Varian Inc., Walnut Creek, CA). The GC-MS conditions are listed in Table 2. The MS was operated in electron ionization mode (-70 eV) with a scan range of 40-450 m/z and a delay of 3.5 minutes.

Table 2 GC-MS conditions for SPME headspace mass quantitation

Column type	Restek 30 m x 0.25 mm ID x 0.25 um Rxi-5Sil fused silica
Carrier gas	Helium at a flow rate of 1.0 mL min^{-1}
Split ratio	5:1
Injector Temperature	110 °C
Column oven parameters	40 °C, hold for 1 min. 100 °C at 5 °C min^{-1}, hold for 6 mins. 250 °C at 10 °C min^{-1}, hold for 5 mins.
MS Transfer Line temperature	280 °C
MS Ion Trap Temperature	180 °C

Chemicals and methods

TATP explosives were synthesized and prepared in the University of Rhode Island laboratory [29]. Cocaine standards were purchased from Cerilliant (Round Rock, TX) for the positive mode IMS calibration. PSPME sampling devices were prepared by spin-coating a sol-gel PDMS solution on an activated glass fiber filter as previously described [24] and used for both static and dynamic extractions. Approximately 10 mg of solid TATP explosive was placed in a half gallon glass jar and was allowed to equilibrate for 30 minutes. For static extractions, the PSPME devices were suspended over the sample at the opening of the jar. Various sampling times and temperatures (25 °C and 40 °C) were tested in triplicates to determine the minimum amount of time required for the detection of TATP using the PSPME devices. For dynamic extractions, the PSPME device was inserted into a handheld vacuum sampler (Barringer) in order to allow the air sample to flow through the PSPME device at a rate of 0.17 L s^{-1}. All static extractions were equilibrated at the different temperatures; however, all dynamic sampling was performed at room temperature.

Static extractions and TATP calibrations were conducted using certified TATP standards of 0.1 mg mL^{-1} (AccuStandard, New Haven, CT) in acetonitrile (ACN). The TATP stock solution was diluted to concentrations 5, 10, 15, 20, 25 and 30 ng μL^{-1} using methanol or acetonitrile (ACN) of optima grade (Fisher Scientific, Fair Lawn, NJ) for absolute mass quantitation in the SPME-GC-MS and PSPME-IMS sampling/detector configurations.

The optimum equilibrium time was obtained by conducting the static extractions at different times in triplicate. Once the optimized equilibrium time was achieved, 5 µL of standard solutions of known concentration ranging from 5 to 30 ng μL^{-1} were spiked into a quart-sized can containing a suspended PSPME filter and sealed immediately for 5 min static extractions. The signals were recorded and plotted to give a quantitative mass calibration of TATP in the PSPME devices.

Evaluation of the extraction efficiency of SPME and PSPME was achieved by spiking the desired amount of TATP on a quart can and extracting immediately without headspace equilibrium development. Detection of TATP extracted by SPME was performed using the Varian GC-MS, using the conditions described in Table 2. Detection of TATP extracted by PSPME was analyzed with Smiths Detection IONSCAN®-LS IMS without further modification.

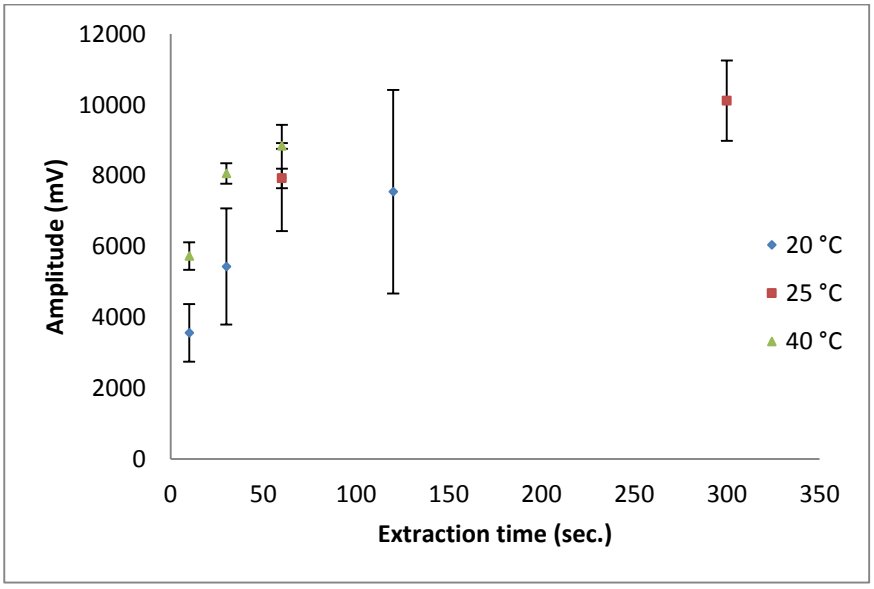
Results and Discussion

Detection of TATP

Various static extraction times were used at room temperature (20 °C), 25 °C and at 40 °C to determine the shortest extraction times for the detection of a 10 mg TATP sample. Detection of TATP was achieved within a 1 minute extraction of the headspace of a half-gallon glass container at room temperature (Fig. 1). In fact, after a one-minute static extraction, the pool of protonated clusters associated as the reactant ion peak (RIP) in the IMS was completely depleted by the TATP on the PSPME device, giving two strong signals at 4.3 ms and 4.7 ms separately corresponding to reduced mobilities (K_0) of 2.13 $cm^2V^{-1}s^{-1}$ and 1.95 $cm^2V^{-1}s^{-1}$ in GE-IMS. The identity of the peak was confirmed by direct spiking 2 µL 1000 ppm TATP in dichloromethane onto a PSPME device forming a peak with the same drift time. Similar results were obtained at elevated temperatures with even shorter extraction times and greater signals. Detection for TATP was achieved within 10 seconds of static extractions at 40 °C.

Dynamic extractions using the PSPME devices produced greater IMS signals with shorter extraction times. A 5 second extraction at room temperature (20 °C) produced a large signal of TATP (4.3 ms peak in the plasmagram) after sampling a total volume of 0.85 L.

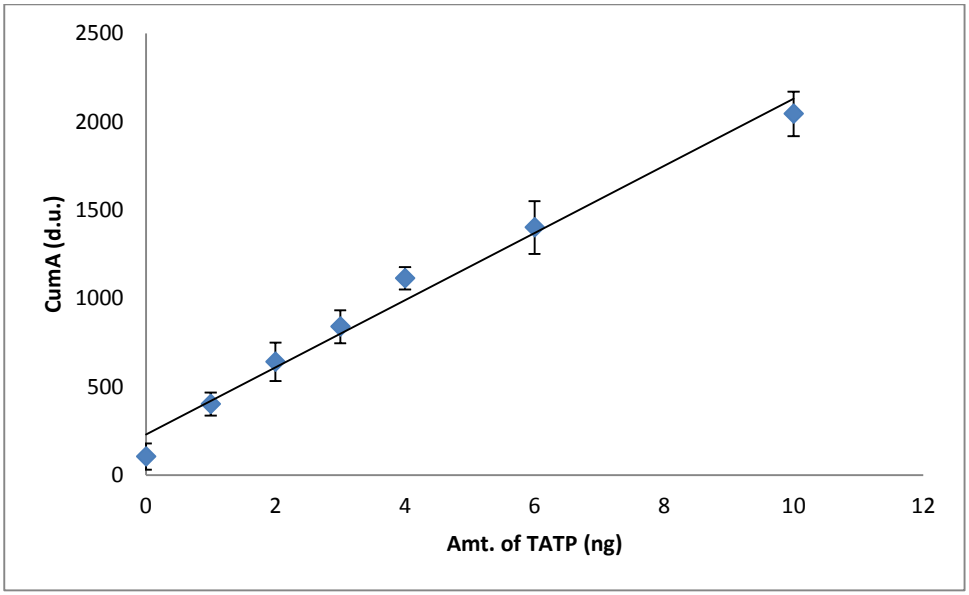
Fig. 1 Signal observed at 4.3 ms at different static extraction times of 10 mg of TATP at different temperature profiles



Alarm in Smiths IMS for TATP was observed within 1 minute of static extraction with 0.5 µg spike of a TATP standard in the quart container, generating a peak with drift time of 6.7 ms and a reduced mobility (K_0) of 2.57 $cm^2V^{-1}s^{-1}$ in close agreement with previously stated reduced mobility of TATP [6]. This reduced mobility is different from reported values[30,31] could be caused by decomposition of TATP at an increased temperature and formation of different adducts with different dopants applied. Additional identification and quantitation of the TATP was

conducted by directly spiking 2 µL of certified standard solutions diluted to concentrations ranging from 0.5-5.0 ng μL^{-1} (Fig. 2).

Fig. 2 TATP calibration by spiking 2 µL TATP of the following concentrations: 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 5.0 ng μL^{-1} onto a PSPME device



Absolute mass calibration of TATP in PSPME filters

A response curve was generated using the observed maximum amplitude (d.u.) from the Smiths IONSCAN resulting in the following linear regression line Eq. 1:

$$(y) = 190.05\ (x) - 229.72,\ r^2 = 0.984 \quad (1)$$

From this method, the minimum detectable amount of TATP was determined to be 1.4 ng.

Since a PSPME extraction is an equilibrium technique similar as SPME, this technique can be used for quantitative analysis. Headspace calibration was achieved by spiking a known amount of TATP into a closed system and headspace sampling using PSPME at the equilibrium time of 5 minutes (Fig. 3). Calibration of TATP was performed using two dopants to evaluate the performance of isobutyramide for peroxide-based explosive detection [28]. Dopant selection is essential for optimal instrument performance in order to form stable and identifiable analyte ions and suppressing ionization of unwanted analytes. Response curves from the Smiths IONSCAN using the nicotinamide and isobutyramide are given in Eq. 2 and Eq.3 respectively:

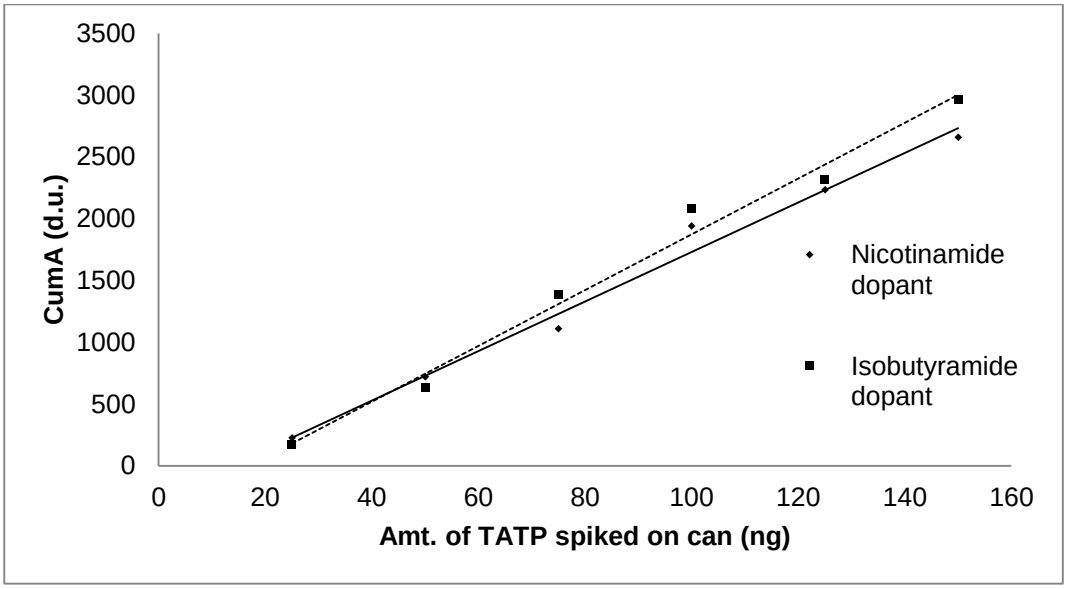
$$(y) = 20.04\ (x) - 272.3,\ r^2 = 0.986 \quad (2)$$

$$(y) = 22.58\ (x) - 384.5,\ r^2 = 0.986 \quad (3)$$

The response signals observed in the IMS were similar using either the nicotinamide or the isobutyramide dopant, thus majority of the experiments were performed using the initially installed nicotinamide dopant. After 5 minutes of static PSPME extractions, the minimum amount of spiked TATP in the can using both dopants was determined to be approximately 19 ng, as shown in Fig. 3. When evaluating the extraction efficiency of the PSPME devices (percentage between the mass

detected and the amount available), the dynamic PSPME devices showed an extraction efficiency of 14%. This significant extraction efficiency shows the preconcentration power of the PSPME devices within minutes of static extraction.

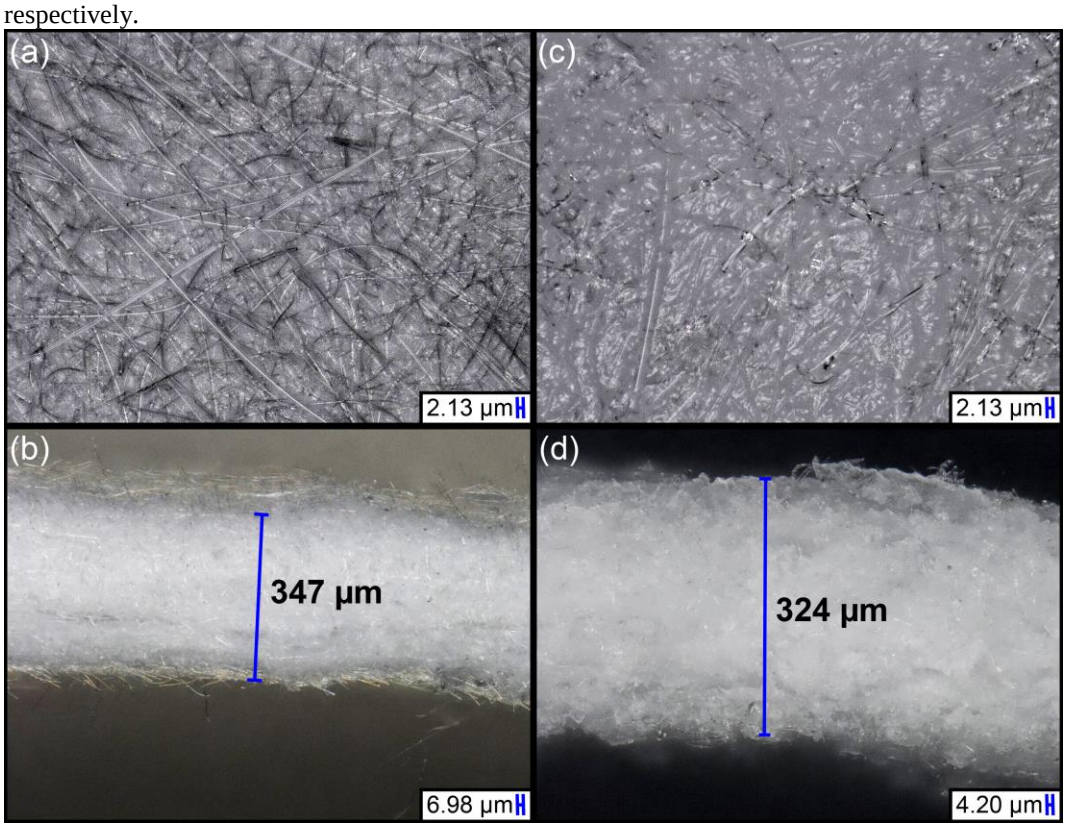
Fig. 3 TATP headspace calibration obtained from 5 minute static PSPME headspace extraction of TATP (spiking 5 µL of solutions of the following concentrations: 5, 10, 15, 20, 25, 30 ng μL^{-1})



Headspace extraction efficiency comparison of PSPME and SPME

The extraction phase volume of a planar SPME disk is calculated to be approximately 35 mm^3, compared to the commercial fiber SPME with a maximum phase volume of 0.6 mm^3 [27], offering greater than 50 times more volume capacity and a surface area of ~ 1000 greater than that of fiber SPME. Digital microscope imaging (Keyence) was performed to characterize the surface of the PSPME in comparison to the uncoated glass filter (Fig. 4). The cross-section thickness of a PSPME device was determined to be ~324 µm (Fig. 4 (b)) while an uncoated glass filters has a cross-section thickness of ~347 µm (Fig. 4 (d)). No increase in cross-sectional thickness indicates the sol-gel based PDMS is well incorporated into the glass-filter surface. Furthermore, surface images (Fig. 4 (a) and (c)) show increased thickness of the glass fibers by ~2 µm in PSPME, thus enhancing the capacity and phase volume.

Fig. 4 Microscope images of the surface and cross-section of an uncoated glass filter, (a) and (b) respectively, and images of the surface and cross-section of a coated PSPME devices, (c) and (d) respectively.



Minimum amount of extraction time for detection of 100 ng of TATP for PSPME was observed to be 0.5 minutes compared to 5 minutes using SPME (Fig. 5). Comparison of the extraction efficiency by varying concentration of TATP was performed by spiking different nanogram-level of TATP standard and extracting for five minutes. The amount of TATP recovered using PSPME was calculated by using an external calibration curve with the regression line in Eq.1. For SPME analysis on the GC-MS, the following linear regression curve Eq.4 was used:

$$(y) = 2302\ (x) - 1661,\ r^2 = 0.964 \qquad (4)$$

Extraction efficiency of TATP on PSPME and SPME was determined to be approximately 15% and 1% respectively as shown in Table 3. Thus, the increased surface area and phase volume of PSPME offers much greater extraction efficiency and faster detection in comparison to the commercially available fiber-based SPME.

Fig. 5 Percent recovery comparison of PSPME and SPME by different static extraction time (0.5 – 30 minutes) of 100 ng TATP

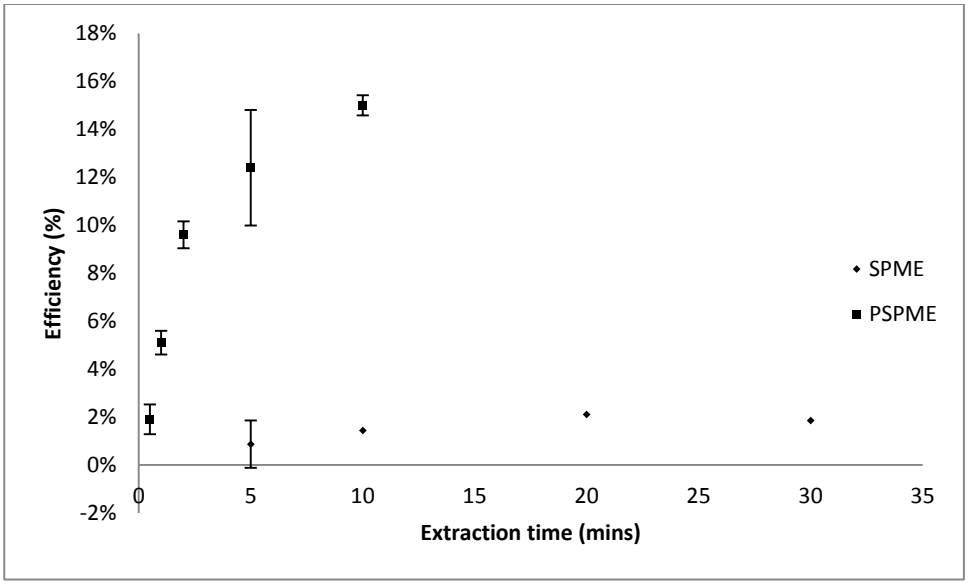


Table 3 Percent recovery comparison of PSPME and SPME by 5 minutes static extraction of different amount of TATP

	PSPME		SPME	
Amt. spiked in can (ng)	Amt. of TATP recovered (ng)	Recovery %	Amt. of TATP recovered (ng)	Recovery %
50	2.58	5.16%		
75	4.63	6.17%		
100	9.00	9.00%	1.22	1.22%
150	21.0	14.0%	1.27	0.85%
200	35.1	17.5%	1.50	0.75%
300	61.8	20.6%	2.36	0.79%
400	79.2	19.8%	3.39	0.85%

Conclusions

As a result of the increased surface area and phase volume in PSPME, TATP was sampled and preconcentrated on a PSPME device in less than 30 seconds by both static and dynamic extractions followed by detection using a COTS IMS that does not require any modification to the sample inlet of the instrument. When using IMS as the detection method, the sampling and detection time of TATP was very short, highlighting the potential use of this method in field analysis with both high selectivity and high sensitivity detection. The total sampling and detection time of TATP was significantly simplified and shortened (~ 35 sec.), in comparison with fiber-based SPME sampling and analysis in GC-MS (~ 22 min). Compared with fiber-based SPME, the extraction efficiency is increased from approximately 1% to approximately 15% when using PSPME. The extraction efficiency for SPME reached a maximum of 2% at 20 min, while PSPME can easily obtain 2% with only a 30 sec. extraction. These results suggest that PSPME devices can be coupled with various

commercial IMS systems to provide high throughput, sensitive detection of different explosives in the field. Future work will include developing receiver operator characteristic (ROC) curves to evaluate the utility of PSPME-IMS in real-world environments.

Acknowledgements

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Tables and Figures

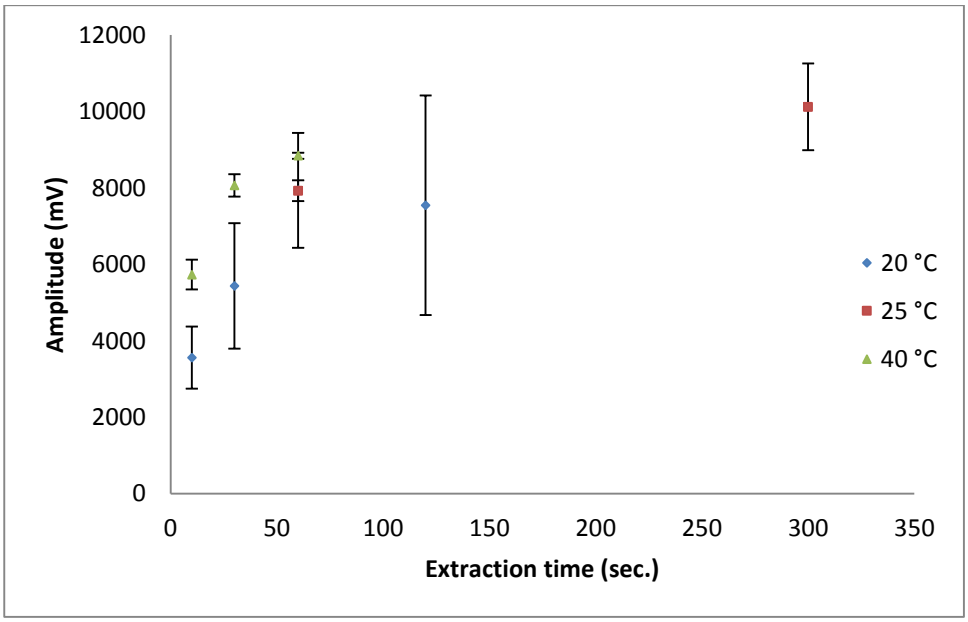
Table 1 Conditions for the IMS instruments used in this experiment

IMS operating conditions	GE Ion Track ITEMIZER	Smiths Detection IONSCAN®-LS
Polarity	Positive (+)	Positive (+)
Desorber temperature (°C)	175	250
Drift tube temperature (°C)	195	235
Sample flow (mL min^{-1})	500	200
Drift flow (mL min^{-1})	350	351
Reagent gas	Ammonia	Nicotinamide/ isobutyramide

Table 2 GC-MS conditions for ACS extraction analysis

Column type	Restek 30 m x 0.25 mm ID x 0.25 um Rxi-5Sil fused silica
Carrier gas	Helium at a flow rate of 1.0 mL min^{-1}
Split ratio	5:1
Injector Temperature	110 °C
Column oven parameters	40 °C, hold for 1 min. 100 °C at 5 °C min^{-1}, hold for 6 mins. 250 °C at 10 °C min^{-1}, hold for 5 mins.
MS Transfer Line temperature	280 °C
MS Ion Trap Temperature	180 °C

Fig. 1 Signal observed at 4.3 ms at different static extraction times of TATP at different temperature profiles



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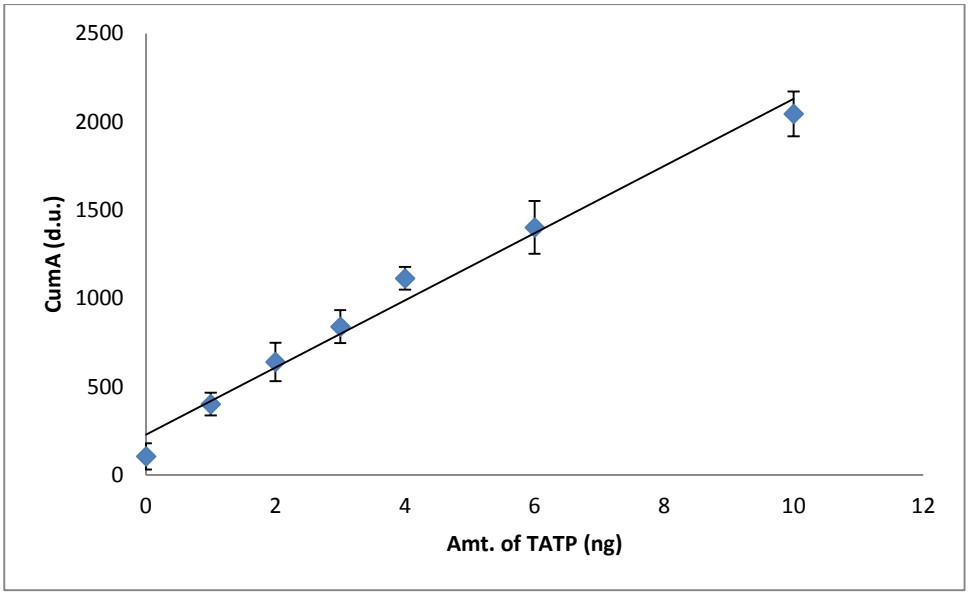

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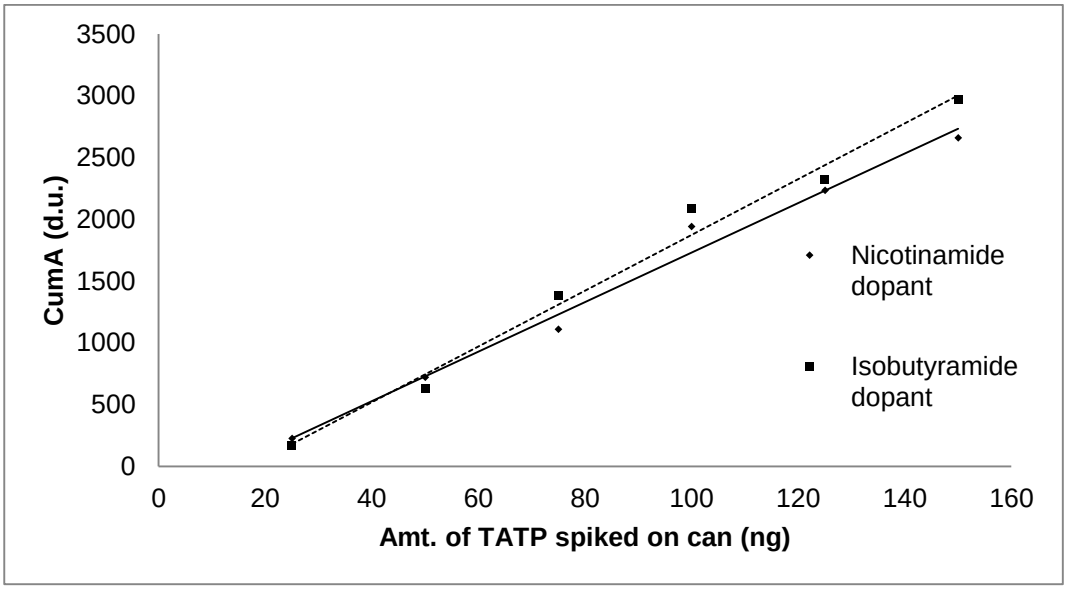

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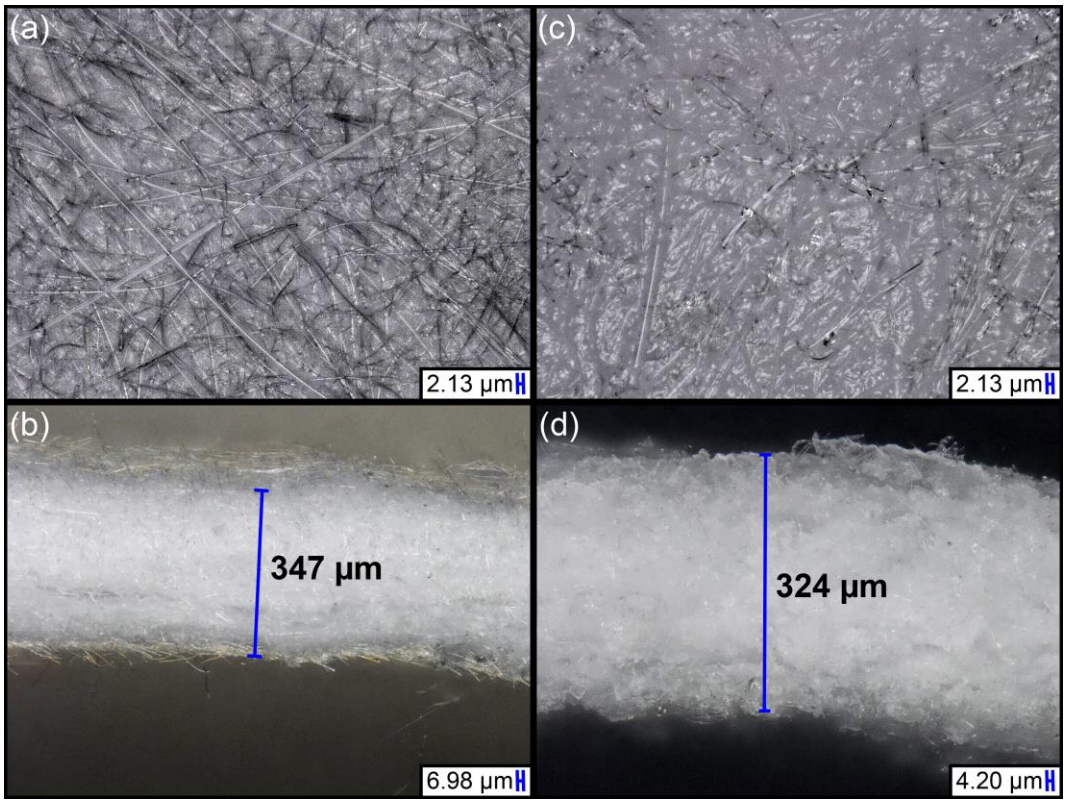

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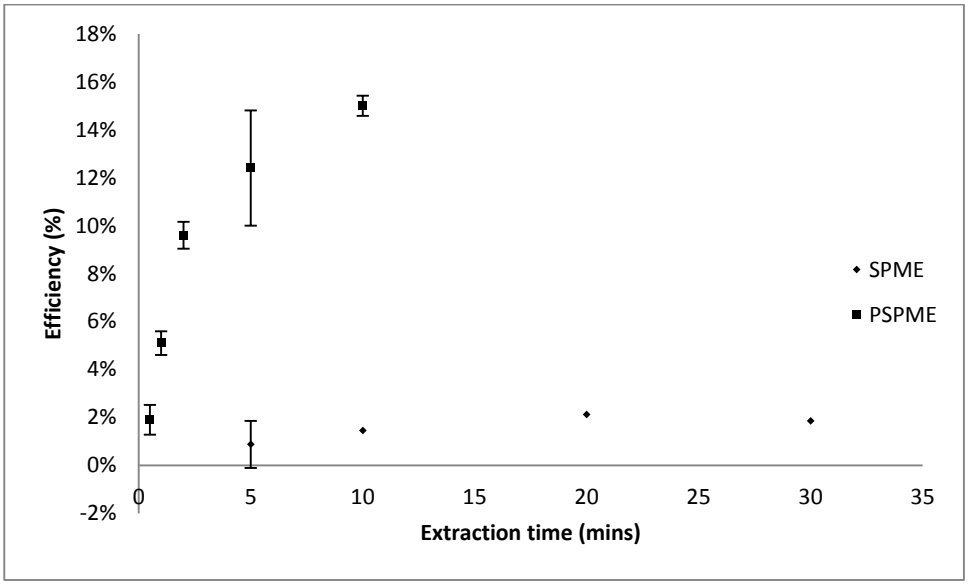

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