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Inexpensive Screening for Smoked Drugs Using an Autosampler/DART/TOFMS

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1 Introduction

Methamphetamine (meth) contamination of real estate is a concern for property purchasers. In 2008 alone, 6783 methamphetamine lab incidents were tallied by the Drug Enforcement Administration. Meth syntheses performed in illicit meth labs produce meth vapor, aerosol and particulates that contaminate floors, walls, ceilings, and objects. Cotton-gauze-pad wipe samples analyzed by conventional mass spectrometric methods have detection limits of 0.05 - 0.1 µg/100 cm² of surface. Based on these detection limits, 15 states have set remediation standards of 0.1-0.5 µg/100 cm².

Meth remediation is expensive, due in part to the cost of meth analyses. NIOSH methods 9106 and 9109 (GC/MS with derivatization) and 9111 (LC/MS) to quantify meth on surfaces require wipe sampling, extraction, clean-up, blow down, and mass spectral analysis using selected ion monitoring. The expense of these methods limits the thoroughness with which meth labs can be characterized. EPA guidelines for meth lab remediation state that "(g)enerally it is more cost-effective to remediate an entire lab than to take pre-remediation samples in an attempt to avoid having to remediate certain areas of a former lab" (1). An inexpensive, high throughput, specific, and sensitive screening technique for meth would encourage collection of more wipe samples and increase the thoroughness of meth lab characterizations before and after remediation.

Over time, the popularity of various illegal drugs changes and new designer drugs could become popular. Hence, a screening technique should be capable of identifying and detecting numerous smoked drugs on surfaces including cocaine and the other illegal drugs listed in Table I. Inexpensive, broadly applicable analysis of wipe samples could be used to routinely screen homes, motel rooms, and other real estate for meth and other drug contamination prior to sales or rentals. A simple sampling technique could enable wipe sample collection by laymen using a kit.

Table I. Optimum Values for Three Variables and Detection Limits for 11 drugs (N=3)

Analyte	Analyte Ion m/z [M+H] ⁺	CID Voltage (% RCPA) ^a	He Temperature (% RCPA) ^a	Pick Up Solvent (% RCPA) ^c	DL (µg/100 cm ²)
Methamphetamine	150.1277	45 V (80,100,86)	300 C (82,86,100)	IPA (59,100,45)	0.05
Ketamine HCl	238.0993	45 V (91,100,75)	200 C (100,91,80)	IPA (82,100,85)	0.025 ^c
Cocaine	304.1543	45 V (93,100,95)	250 C (55,100,80)	IPA (73,100,50)	0.025 ^c
Morphine	286.1438	80 V (73,100,87)	250 C (57,100,94)	IPA (58,100,8)	0.25
Fentanyl	337.2274	50 V (78,100,86)	250 C (74,100,95)	IPA (63,100,48)	0.025 ^c
PCP	244.2060	20 V (92,100,88)	250 C (78,100,62)	Water (77,83,100)	0.025 ^c
Heroin	370.1649	80 V (73,100,89)	250 C (53,100,92)	IPA (49,100,33)	0.025 ^c
Amphetamine	136.1121	50 V (81,100,95)	200 C (100,78,73)	MeOH (100,83,59)	0.025 ^c
THC	315.2318	40 V (72,100,80)	250 C (73,100,83)	IPA (87,100,2)	0.25
Nicotine	163.1230	45 V (67,100,83)	200 C (100,76,86)	IPA (30,100,24)	0.05
Pseudoephedrine HCl	148.1121 ^d	70 V (81,100,94)	200 C (100,91,88)	IPA (97,100,14)	0.025 ^c

^aThe numbers in parentheses are the average (N=3) % relative chromatographic peak areas (% RCPAs) compared to that obtained for the optimum CID voltage (100%). 5 V increments in the CID voltage correspond to the lesser % RCPAs. A broad maximum was apparent for this instrumental parameter.

^bThe helium stream temperature setting was 200, 250, and 300°C for each analyte. The numbers in parentheses are the corresponding % RCPAs.

^cThe testing order for the solvents was MeOH, IPA, and water. The numbers in parentheses are the corresponding % RCPAs.

^dA product ion, [M+H-H₂O]⁺, was the quantitation ion for pseudoephedrine. The [M+H]⁺ ion was used for the other 10 drugs.

^eLowest level tested.

2 Analytical Method

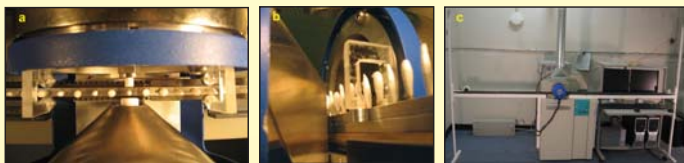


Figure 1. (a) top view, (b) side view, and (c) full view of the autosampler for the DART/TOFMS.

Wipe Sampling. A mirror was partitioned into 10-cm squares with fish line. Different amounts of aqueous solutions of analyte were deposited within the squares with a syringe. After the water evaporated, six-inch-long, glueless, cotton swabs were dipped into 91% isopropanol (IPA) and the swab head was immediately rolled right-to-left and back until the entire 10 cm-square area was sampled. The swab was rotated 90° and the rolling procedure was repeated to ensure a uniform distribution of the analyte along the swab head and around its circumference. Each swab was inserted through a hole drilled through a 3-foot-long, 1/4-inch-square, aluminum support bar. After the swabs dried, the swabs were clipped at the base of the support bar.

Mass Analysis. The support bar with swabs was placed onto two N-scale model railroad flat cars. The bar was pulled by fish line and a variable speed DC motor through a Direct Analysis in Real Time (DART®) ion source interfaced to a Jeol AccuTOF® mass spectrometer as shown in Figure 1 (2). One mg in 1 mL of methanol standards were purchased from Cerilliant to optimize instrument parameters, to select a quantitation ion, and to determine the detection limits of the analytes in Table I.

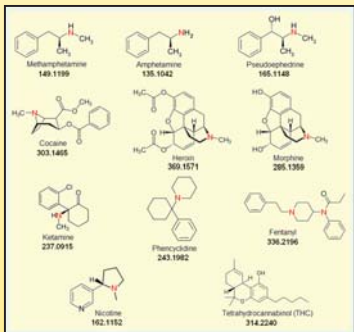


Figure 2. Structures and molecular masses of nine illicitly smoked drugs, nicotine, and pseudoephedrine.

For triplicate wipe samples acquired for 2.5 µg of analyte, the helium stream temperature, CID voltage, and pick up solvent were optimized for the 11 compounds in Figure 2. Table I lists the optimum parameters for each analyte and the detection limit for each using the optimum helium temperature and CID voltage using IPA as the pick up solvent.



Figure 3. Ion chromatogram for the m/z 150 [M+H]⁺ ion from methamphetamine.

3 Discussion

The average %RSD (N=3) for the CID optimum voltage determinations of the eleven compounds was 13.6%. Hence, small differences among the peak areas integrated from ion chromatograms are not significant.

The best pick up solvent was IPA for 9 of the 11 analytes. For PCP and amphetamine, IPA recovered nearly as much as did water and methanol, respectively. IPA was used for all 11 analytes to determine detection limits.

For 8 analytes, broad maxima were observed over a temperature range of 200°C to 300°C. Only for the three largest-mass analytes, heroin, cocaine, and morphine, did the lowest temperature provide significantly less ion abundance, probably due to less complete desorption from the cotton. Hence, 250°C would be suitable for all of the analytes.

For CID voltages, broad maxima were also seen. However, for three illicit drugs, Morphine, PCP, and Heroin, CID voltages of 40-50 V would provide significantly lower ion abundances.

4 Detection Limits

The detection limit (DL) for meth from glass was 0.05 µg/100 cm². As is evident in Figure 3, all three swabs used to collect 0.05 µg from the mirror squares provided visibly larger peaks in the ion chromatogram than for the swabs used to sample clean mirror squares (blanks). The meth DL's for NIOSH methods 9106, 9109, and 9111 are 0.05, 0.05, and 0.1 µg/100 cm², respectively.

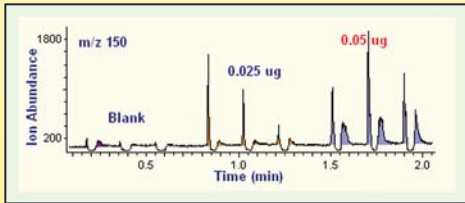


Figure 3. Ion chromatogram for the m/z 150 [M+H]⁺ ion from methamphetamine.

6 Conclusion

The DL for meth using DART/TOFMS is similar to that of the NIOSH methods and could be used as a screening tool for detecting meth and other smoked drugs on surfaces. More numerous wipe samples could be acquired to better characterize meth labs before and after remediation at much less expense than using the NIOSH methods.

The high selectivity of DART/TOFMS could be used to screen for other illicitly smoked drugs that remain on surfaces. The drugs could be identified from their mass spectra and distinguished from the nicotine that might coat surfaces when tobacco is smoked.

Pick up of pseudoephedrine is most successful from impermeable surfaces.

5 Sampling Surfaces

Figure 4 shows an ion chromatogram for the [M+H-H₂O]⁺ ion for cotton-swab wipe samples from various household surfaces onto which 10 µg of pseudoephedrine (PE) in water had been deposited. The PE was obtained by dissolving a cold remedy pill that contained 120 mg of PE. Ample analyte was then available for numerous experiments including determination of the dynamic ranges of PE plotted in Figure 5. A larger DL was observed from the paint surface (Figure 5b), because pseudoephedrine infused into the paint. The ion chromatogram in Figure 4 did not reveal the presence of 10 ug/100 cm² of PE deposited on 6-yr old latex paint. The recovery of PE from paint will probably depend on the age, thickness, and composition of the paint. Additional drugs will be tested for infusion into paint.

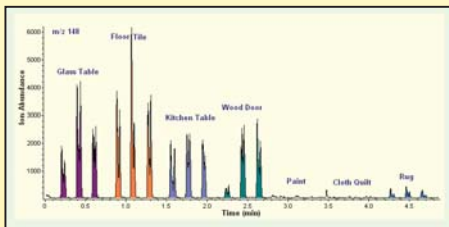


Figure 4. M/z 148 ion chromatogram for cotton-swab wipe samples of 10 µg of pseudoephedrine sprayed as an aqueous solution within 10-cm squares atop household surfaces.

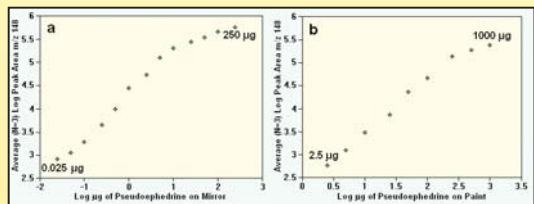


Figure 5. Dynamic range for pseudoephedrine collected from (a) 100 cm² of a mirror surface and (b) 100 cm² of a mirror with two brush-applied coats of acrylic latex paint.

References

1. US EPA Voluntary Guidelines for Methamphetamine Laboratory Cleanup. August, 2009. Available at: http://www.epa.gov/oem/meth_lab_guidelines.pdf
2. Grange, A. H. 2008. An inexpensive autosampler to maximize throughput for an ion source that samples surfaces in open air. *Environmental Forensics* April-Sept.:127-136.