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Characterization and validation of ion mobility spectrometry in methamphetamine clandestine laboratory remediation

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ABSTRACT

This project evaluated the efficacy of ion mobility spectrometry (IMS) as a tool for determining remediation success at clandestine methamphetamine laboratory sites. Specifically, limits of detection (LOD), limits of quantitation (LOQ), and matrix effects were investigated as relevant to typical remediation sites and situations. The recoveries of pseudoephedrine and methamphetamine from a range of various surfaces likely to be found in a clandestine laboratory were examined. Portable IMS instruments with thermal desorption were found to be a reliable tool for evaluating the degree of remediation if sufficient procedural and instrumental controls are put into place. In general, detection limits were in the same range as state guidelines as well as laboratory methods using GC/MS and LC/MS. Direct vapor sampling can be used to detect high levels of methamphetamine and potential interferences, but cannot approach the detection limits needed for evaluation of remediation efforts. IMS cannot be used alone to determine the efficacy of remediation efforts; final confirmation using laboratory instrumentation is essential. For the purpose of this study, typical field settings of the IMS were used and the conditions were not optimized.

1. Introduction

The immediate and long-term hazards associated with clandestine methamphetamine laboratories are well known and significant efforts have been directed towards developing reasonable standards for the remediation of clandestine methamphetamine sites [1–15]. Regardless of the synthetic method used to produce methamphetamine, the clandestine laboratory site is typically highly contaminated and requires either demolition or extensive remediation. A recent Federal statue has addressed some of the issues related to remediation and how to gauge if a clean-up has indeed been successful and if a site is safe for re-habitation [13]. In 2005, the United States Drug Enforcement Administration (DEA) published a manual entitled *Guidelines for Law Enforcement for the Clean-up of Clandestine Drug Laboratories* that describes protocols and procedures, but not specific clean-up methodologies [14]. The Environmental Protection Agency (EPA) has drafted guidelines for clean-up, but this document has not yet been released in final form. Various states have adopted different acceptable levels of residual methamphetamine which range from 0.1 to 0.5 μg/100 cm^2 as summarized in the DEA manual [13]. According to the California Study, levels in some states may be as high as 1 μg/100 cm^2 [16]. The quantities refer to the area of a given surface that has been sampled, typically by swiping followed by field or laboratory analysis.

Ion mobility spectrometry (IMS) is frequently used for rapid field evaluation for a variety of compounds [17]. IMS has been deployed as a rugged and reliable field sensing system for chemical warfare agents since the 1980s [18]. It has also found significant use for the detection of explosives, monitoring of environmental compounds, and a drug detection system [19]. More recent applications of IMS include pharmaceutical quality control, verification of the cleaning of pharmaceutical equipment surfaces, pharmaceutical process analysis, and determination of active pharmaceutical ingredients [20–23].

IMS operates at atmospheric pressure and separates ionized analytes as ions and ion/molecule clusters (Fig. 1). With thermal desorption instruments, such as used here, samples are deposited on a Teflon® membrane filter, which are then vaporized by the desorber heater. Ionization occurs from thermal electrons emitted from a ^{63}Ni beta-ray source. The product ions are then gated into a drift region for mobility analysis. Under the influence of an electric field gradient and against the counterflow of a drift gas, the ions move toward the collector plate.

The ion mobility constant, K ($cm^2\ V^{-1}\ s^{-1}$), is used to identify the analyte from the observed ion peaks. Ion mobility constants are calculated according to Eq. (1) [29]:

$$K = d/tE \quad (1)$$

where d is the distance an ion will travel in the measured time (t) under the electric field (E).

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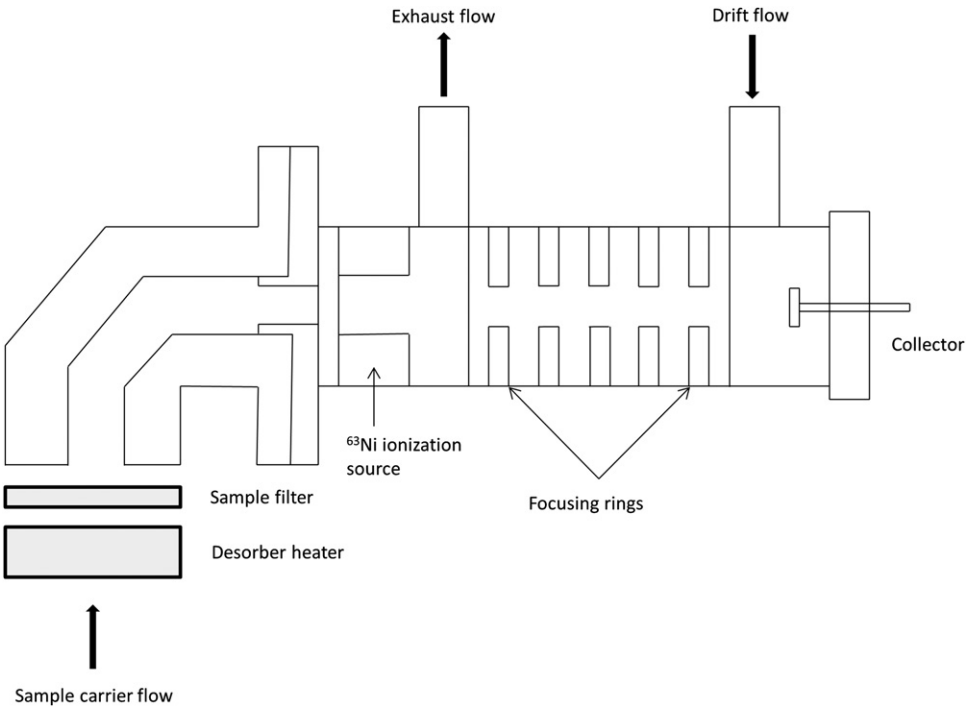


Fig. 1. Schematic representation of IMS, adapted from [24].

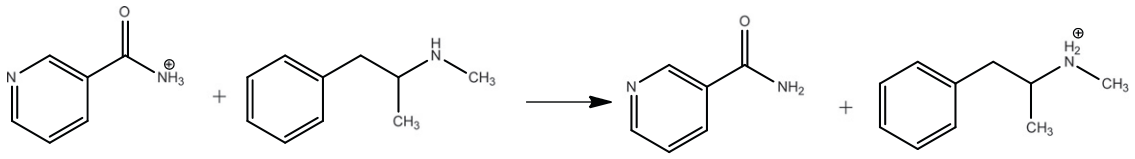


Fig. 2. Formation of protonated methamphetamine from NTA and methamphetamine.

The drift times required by the ions to reach the collector electrode are generally proportional to their masses, but inversely proportional to their reduced ion mobilities K_o ($cm^2\,V^{-1}\,s^{-1}$). The reduced mobility constant compensates and standardizes for pressure and temperature towards standard conditions, as shown in Eq. (2) [29]:

$$K_o = (d/t_d E)\,(273/T)\,(P/760) \quad (2)$$

where d is the length of the drift region (cm), t_d is the time it takes the ion to travel the distance d (s), E is the applied electric field ($C\,cm^{-1}$), T is the temperature of the buffer gas (K), and P is the pressure in the drift region (Torr).

IMS can be operated in the positive or negative mode. For this study, IMS was operated in the positive mode, which is the mode used for drug detection. In this mode, the drift gas contains nicotinamide (NTA) used as both a calibrant and a reactant. In the reaction region, the protonated NTA transfers a proton to the sample molecule, M, as shown in Eq. (3) [29]:

$$[NTA]H^+ + M \rightarrow NTA + [M]H^+ \quad (3)$$

This reaction only proceeds if the proton affinity of the sample molecule is greater than that of the NTA. Methamphetamine responds in a similar fashion, as shown in Fig. 2. The principles and background of IMS has been extensively described elsewhere [17–29].

IMS instrumentation offers many advantages for field use including atmospheric pressure ionization, small instrument size (many commercial hand-held units are available), and low power requirements. Field units can be programmed to respond to the appearance of drift peaks in given drift time windows corresponding to the mobility peaks of target compounds. However, such responses are not unique in that these mobility channels correlate to drift times, cross-sectional areas, and mass-to-charge ratios and not to specific compounds. This can generate false positives, which may result from a number of factors including poor desorption from substrates, low concentration, or competing ion/molecule reactions. A key goal of this study was to identify the strengths and limitations of IMS on specific, but critical field deployment. Lessons learned here can be extended to other field applications.

IMS is frequently used for screening at clandestine laboratory sites and for the detection of methamphetamine [17,25]. Several papers have demonstrated methods of detecting methamphetamine in the presence of nicotine and cigarette smoke which are common interfering compounds seen at clandestine laboratory sites [26]. Accordingly, there is a strong theoretical and practical basis for employing IMS in the context of clandestine laboratory remediation. The goal of this work is to determine the performance limits of detection for residual methamphetamine at remediated clandestine laboratory sites.

2. Materials and methods

2.1. Reagents

For sample preparation (standards of methamphetamine and pseudoephedrine), LC/MS-grade methanol (Fluka/Sigma Aldrich,

St. Louis, MO) was used. Most samplers use methanol and some ethanol. Methamphetamine and pseudoephedrine were obtained from Sigma Aldrich as solids. Stock solutions were prepared from their solid dissolved in methanol and were stored in Miniert™ vials. For swabbing, anhydrous reagent alcohol was used (ACS grade, EMD scientific, Darmstadt, Germany). This alcohol mixture consisted of 89–92% ethanol, 3.5–5.5% methanol, and 4–6% isopropyl alcohol, which is reasonably representative of solvents used in the field.

2.2. Building materials

Several surface samples were obtained locally for methamphetamine analysis. The samples were divided into the following categories: countertop (C1–C4); flooring (F1–F10); glass (G1–G4); miscellaneous (M1–M15); raw (R1–R11); and wall (W1–W3) and are listed in Table 1. The porosity of each material was classified based on appearance and was a qualitative description. To each of these surfaces, 20 μL of a 100 ppm methamphetamine standard (2 μg/application) was directly placed on the sampling surface area. The surfaces were analyzed 30 min, 4 h, 1 day and 3 days after exposure.

2.3. Deposition of samples

Selected surfaces were outlined using 10 cm × 10 cm templates. Methanolic solutions of pseudoephedrine or methamphetamine was deposited directly in the center of the selected surfaces using a clean 100 μL syringe. If the surface area of the material was smaller than what was required, the pseudoephedrine or methamphetamine was applied to the center of the available area.

2.4. Sample wiping protocol

The analysis of each building material nominally requires a surface area of 100 cm^2, although this is not always possible. For example, the electrical outlet (M10), had a much smaller surface area than what is required. A 10 cm × 10 cm template was placed on each individual building material and sampled using a 3 in. × 3 in. 12-ply cotton gauze moistened using methanol. Wiping was performed by one of the following methods: (1) concentric squares wiping; (2) side-to-side wiping (blotting). In both the concentric square and side-to-side wipe methods, the prewetted gauze was folded in half and then in half again. The concentric squares method started in an outer corner of the surface area and wiped with concentric squares until in the center, where the last fold is reversed and the same area is wiped concentrically again. The side-to-side wipe makes use of the folded gauze passing over the surface area in at least five overlapping, side-to-side, horizontal passes followed by a reversed fold and a re-wipe of the same area. The National Institute for Occupational Safety and Health (NIOSH) has published three laboratory methods for swipe analysis targeting methamphetamine and related compounds [30–32]. Methods 9106, 9109, and 9111 recommended by NIOSH were utilized in this study (refer to Table 5 for example photos).

2.5. IMS parameters

IMS analysis was carried out on a Smith's Detection Sabre 4000 (Smiths Detection, Danbury, CT). The operating conditions used are detailed in Table 2.

Typical preventative maintenance was performed followed by a bake-out cycle for 4 h every night at the maximum drift tube temperature (250 °C). For each sampling session, the calibrant

Table 1
Building materials evaluated.

ID	Description	Porosity
C1	Countertop	NP
C2	Back portion of a counter top with particle board	NP
C3	Laminated surface	NP
C4	Plastic strip from the side of a counter top piece	NP
F1a	Wood laminate floor boards (1 of 3)	NP
F1b	Wood laminate floor boards (2 of 3)	NP
F1c	Wood laminate floor boards (3 of 3)	NP
F2	Tan floor tile, adhesive on back	NP
F3	Green floor tile, cracked in corner	NP
F4	Tan floor tile, adhesive on back	NP
F5	Linoleum floor tile	NP
F6a	Ceramic floor tile (1 of 4)	NP
F6b	Ceramic floor tile (2 of 4)	NP
F6c	Ceramic floor tile (3 of 4)	NP
F6d	Ceramic floor tile (4 of 4)	NP
F7	Linoleum floor tile	NP
F8	Shower stall liner	NP
F9	Finished baseboard	MP
F10	Interior wood (old), multiple layers of paint	MP
G1	Plexiglass	NP
G2	Single-paned window	NP
G3	Plexiglass from a lab hood	NP
G4	Plexiglass	NP
M1	Light bulb	NP
M2	Smoke detector	NP
M3	Seat cushion	P
M4	Motor for a heater	NP
M5	Latex cleaning gloves	NP
M6	Windshield wiper	NP
M7	Ceiling tile	P
M8	Window blinds (white)	NP
M9	Bolt lock	NP
M10	Electrical outlet	NP
M11	PVC pipe	NP
M12	PVC pipe	NP
M13	Light switch cover	NP
M14	Outlet cover	NP
M15	PVC pipe	NP
M16	Grout	NP
R1	Sheet rock	P
R2	Untreated wood	P
R3	Wood	P
R4	Interior wood (sanded)	P
R5	Untreated wood	P
R6	Composite wood mimic—material unknown	NP
R7	Painted baseboard	MP
R8	Brick	P
R9	Interior stud	P
R10	Scrap wood	P
R11	T-11, blue painted, old and cracked	MP
W1	Sheet rock	P
W2	Lattice work, painted	MP
W3	Wood molding (finished on one side)	MP

NP=non-porous*.
MP=mildly porous*.
P=porous*.
*Indicates a qualitative and visual evaluation of porosity.

Table 2
IMS operating conditions.

Ion mode	Parameter	Setting
Positive	Drift tube temperature (°C)	150
	Inlet temperature (°C)	145
	Desorber temperature (°C)	125
	Calibrant temperature (°C)	70
	Drift flow (mL/min)	200
	Analysis time (s)	20
	Number of segments per analysis	23

position was updated if needed and backgrounds of a vapor sample and a particulate were obtained. To obtain methamphetamine and pseudoephedrine controls, a clean swab was spiked with 10 μL of a methanolic solution of methamphetamine or pseudoephedrine and was inserted into the desorber. This process was performed a total of five times for each control.

It should be noted that for field use, users rely on an alarm that is set to a certain threshold. However, for this study, the threshold was disabled and peak intensity was used to evaluate methamphetamine detection based on LOD/LOQ considerations as discussed below. In all cases, operation of the instrument was based on how it would be used in the field rather than in a research laboratory context.

3. Results and discussion

3.1. Reproducibility

One of the first studies undertaken was to determine the reproducibility of the IMS over several days with several analysts. The goals were to determine the reproducibility of the reduced mobility of methamphetamine and to determine which peak characteristic to use (amplitude or area). For field identification, peak amplitude is used. The peak intensity is expressed as digital units (dU), which is a voltage representation. Both of these criteria were determined using the software provided by the instrument, as would be the case in the field. Data was collected over ten days by nine analysts using the previously described swab spiking procedure. This study was intended to characterize variability over time and between operators, but did not capture uncertainty contributed by swabbing and building material. The results are summarized in Table 3.

The range (± 1 s) of 1.56–1.63 μs for the reduced mobility (K_o, calculated relative to an internal reference peak) agrees with reported literature values [26,33]. As expected, the reduced mobility was acceptably stable ($< 1\%$ RSD was used as a cut-off) while peak area and peak amplitude showed much greater variation. The variability between peak area and peak amplitude was comparable and as such, either could be used for data evaluation. Indeed both were collected and studied and in all cases, patterns seen in amplitude were mirrored with area. Amplitude was selected in this report based on knowledge of how the instrument is used in the field; the alarm system is based on a peak height exceeding a set threshold and associated variability within a programmed mobility window. However, the variability in the peak height values had to be addressed in the context of determining a realistic and reliable estimate of the LOD and LOQ for the instrument and swab desorption protocol.

In the present application, IMS is not utilized for quantitative analysis, but as a detector that alarms above a certain threshold. Accordingly, it is critical to determine this threshold not in an isolated instrument-only context, but rather in the context of the method as it would be realistically applied in the field. This detection threshold is best labeled as an LOD value, in the sense of associating a voltage with a concentration, but for purposes here, the LOD and LOQ are considered to be the same value. It is important to emphasize that in the context of clandestine laboratory remediation, the instrument is not used quantitatively per se; all that is required is a "positive" response which is defined as a signal above an established threshold. To establish this threshold, hereafter referred to as the LOD, a series of experiments were undertaken that incorporated increasingly more steps of the final field protocol.

It is worth noting that the LOD determinations here are constrained in that they rely on the Sabre/Smith's Detection software. It is not known what algorithms are utilized for integration or related tasks. While the raw data can be exported in an XY format, transferred to spreadsheet software, and processed there to obtain a S/N-based estimation of LOD/LOQ, this would not reflect how the instrument is used in the field for evaluation of methamphetamine remediation. It is also unknown how much the processing algorithm itself contributes to the standard deviations reported in Table 3. However, it is reasonable to assume that peak amplitude values will be less affected than peak area by any software issues. As will be discussed shortly, these affects are likely inconsequential compared to other contributors to amplitude variation.

Table 3
Reproducibility study ($n=65$).

Characteristic	Mean	Standard deviation	%RSD
Reduced mobility (K_o)	1.592 μs	0.0348	0.02
Peak amplitude	392 dU	176	45%
Peak area	147 dU	71	48%

3.2. Variation contributed by analyst, date, and compound

To estimate variations in peak intensity as a function of compound (methamphetamine vs. pseudoephedrine), analyst, and date, a study was conducted with six analysts working over a period of two weeks. Since one analyst could use the instrument at a time given analyst scheduling, it was not possible to isolate variation associated with a different date vs. variation that arises because of a different analyst.

For this study, each analyst prepared and analyzed samples as previously described. The experiment was repeated so that each analyst gathered one set of data on two different dates, if schedules permitted. The results are displayed in Fig. 3. The error bars correspond to one standard deviation unit. Analysts 2 and 4 did not repeat the methamphetamine samples. It was difficult to identify any trends or generalize except to note the wide variability in amplitude over the study period. The methamphetamine peaks are generally, by not universally, larger than pseudoephedrine, a molecule that is structurally similar to methamphetamine save for the addition of a hydroxyl group on the alkyl chain. Thus, a consistent difference in response between the two is expected given that the presence of the hydroxyl group would change the proton affinity of pseudoephedrine relative to methamphetamine. No other generalities can be drawn from this data other than the awareness of inherent variability in response is a dominant factor in the present context.

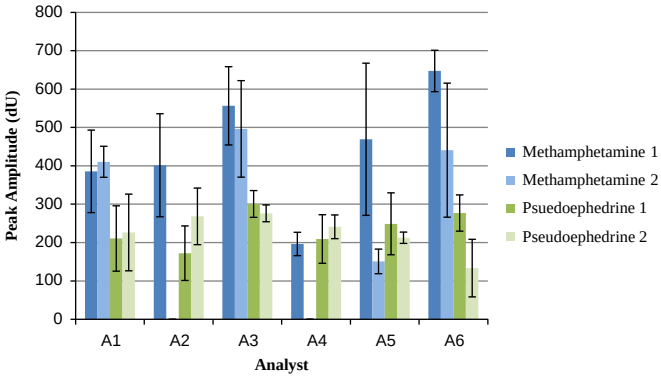


Fig. 3. Variation of peak amplitude by analyst.

Table 4
LOD/LOQ study of peak amplitude as a function of concentration.

Concentration of spiking solution (ppm)	Equivalent μg per swab	Peak amplitude (dU)				
25	0.25	ND				
25		ND				
25		ND				
25		ND				
25		ND				
25		ND				
25		15.8				
25		23.9				
25		36.3	Mean	s	%RSD	95% CI
		Summary	25.3	10.3	41	±11.7
50	0.50	ND				
50		46.3				
50		68.6				
50		47.1				
50		45.3				
50		30.9				
50		46.0				
50		29.0				
50		29.7	Mean	s	%RSD	95% CI
		Summary	42.9	13.2	31	±9.1
100	1.0	101.8				
100		183.0				
100		211.6				
100		113.2				
100		146.6				
100		193.9				
100		60.4				
100		100.9				
100		110.6	Mean	s	%RSD	95% CI
		Summary	135.8	50.8	37	33.2

ND = non-detectable*.
*Non-detectable peaks determined by the integration software.

3.3. LOD/LOQ study

The field LOD of the IMS was estimated by having analysts ($n=9$) analyze a swab spiked with methamphetamine at six different concentrations (1, 5, 10, 25, 50, and 100 ppm) over a two week period. For each experiment, 10 μL of the appropriate methamphetamine solution was spiked onto a swab.

No detectable methamphetamine mobility peak was observed for the 1 ppm (0.01 μg/application), 5 ppm (0.05 μg/application), and 10 ppm (0.1 μg/application) spikes (not shown). At 25 ppm (0.25 μg/application), six out of the nine experiments showed no detectable methamphetamine peak. At 50 ppm (0.50 μg/application), one out of nine showed no detectable peak. At 100 ppm (1 μg/application), a detectable peak was consistently observed for all eight experiments. The data for these peaks is shown in Table 4, which can be compared to the state guideline levels of 0.05–0.5 μg per application.

Several observations can be made based on the data. First, there is no discernible trend in the %RSD as a function of concentration when excluding the "no response" results, which was sufficiently above the LOQ to avoid an increase in %RSD. This is consistent with many current method validation procedures; the LOQ is determined based on acceptability, repeatablility, or reproducibility rather than on 10 S/N. Second, the spread of the data, as measured by the standard deviations, at each calibration level needs to be accounted for in any estimation of the method and instrument LOD and for assigning a cut-off threshold for a positive response. Since the number of replicates at each level is relatively small ($n<10$), the 95% confidence interval is the preferred method for expressing the range to be expected in dU for any given concentration.

The smallest detectable amplitude was 15.8 dU, which suggests that 16 dU is a reasonable estimation for the LOD in dU. Recall here, that detectability is a function of the instrumental software as would be the case in field applications. This was supported by an analysis of all representative spectra ($n=1396$). Over the course of several months of the study, the smallest detectable peak amplitude was identified at 5.5 dU ($n=1$), 18 at less than 10 dU, and 49 at less than 15 dU. Thus, while responses below 16 dU are detectable at some conditions, these responses are not reproducibly detectable. A peak with amplitude of 16 dU should be reliably detectable by the software algorithm and therefore, was adopted as the dU equivalent of the instrument detection limit. As will be discussed shortly, there are instances in which a blank produced higher peak amplitude responses, which were in part due to carryover. This issue must be procedurally addressed using a conservative approach.

This information was used to generate the equivalent LOD in terms of spiking concentration and ng per swabbed area. The data points listed in Table 4 were plotted and fit to a power expression as shown in Fig. 4. The error bars span peak amplitudes of ±34 dU, which corresponds to the largest 95% confidence interval. Using the equation derived, peak amplitudes were calculated from 10 ppm to 70 ppm. The established LOD, a value of 34 dU, was subtracted to define the lower end of the corresponding error bars. This value fell below 16 dU at a concentration of 55 ppm. Therefore, a spike of 10 μL of a 55 ppm solution (0.55 μg/application) onto a swab represents a conservative, but realistic estimate of the instrument LOD as it would be used in the field.

3.4. Foil and glass controls

To establish what peak amplitude at the appropriate K_o value represented a true positive detection of methamphetamine, the background signal was determined. This required that variations associated with the swabbing process be captured and to the extent possible, the background associated with different building materials.

Data was compiled from blanks run on pre-cleaned window glass and foil ($n=27$) with the results shown in Fig. 5a and b. The window and frame assembly were removed during demolition of a house and were not new. In order to increase the incorporation of a matrix, a swab by itself was obtained followed by a swab of the glass and a swab of the foil. It was assumed that a non-porous material, such as the window (G2), would provide the best recovery. In Fig. 5a and b, the *y*-axis is the peak amplitude at the methamphetamine window and the box plot shows the first quartile around the mean ("*t*") blank peak amplitude. Fig. 5b is a scatterplot of the same data. The data series on the left is the

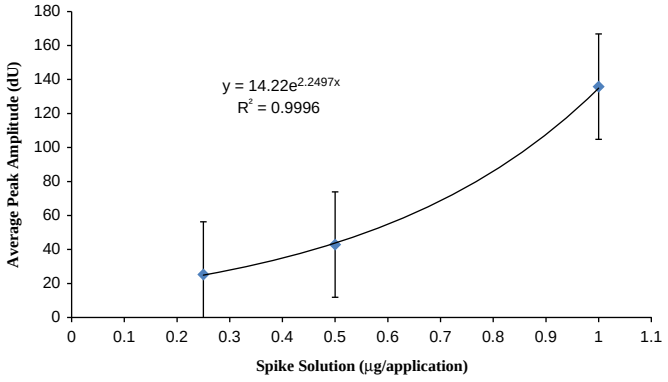


Fig. 4. LOD estimation.

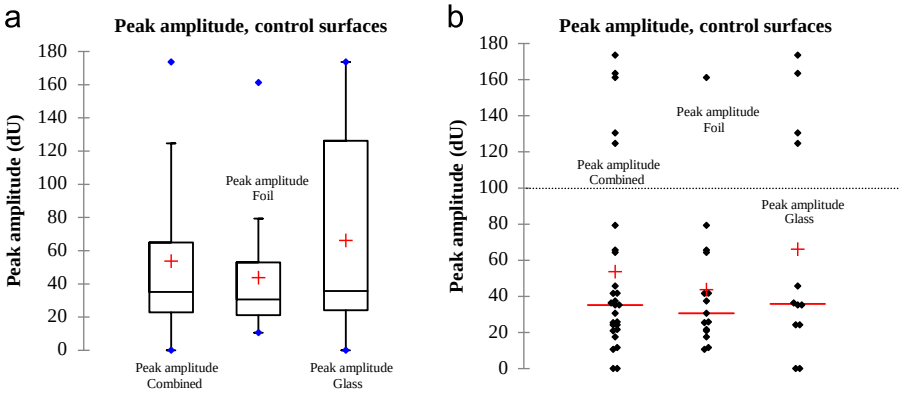


Fig. 5. Glass and foil control blanks: (a) box plot of glass, foil, and combined peak amplitude and (b) scatterplot of the same data.

combined glass and foil, the middle is the foil only, and the right is the glass only.

Trends are immediately obvious. As soon as a realistic building material substrate (i.e. glass) is used, the variation of amplitude increases. However, examination of the combined data suggests that based on the first quartile, the assignment of 60 dU (amplitude) as the minimum detectable signal is reasonable, but not universally applicable. It is worth emphasizing that these spectra were obtained using the previously discussed procedure, which incorporates several cleaning steps. It is assumed that the spread seen here is the minimum that would be expected in field use. The dotted line in Fig. 5b, which represents a minimum peak amplitude of 100 dU, would be reasonable to eliminate most false positives without yielding many false negatives.

3.5. Building material controls

All the building materials listed in Table 1 were included in this study ($n=364$). Given the demonstrated variability using simple matrices, it was difficult to interpret some of the building material results. This was done with caution since factors such as instrument drift, swiping/analyst, and substrate were taken into consideration. Fig. 6 shows the peak amplitudes for all blanks as a function of the date of analysis. A primary concern with field deployable mobility spectrometers is the danger of contamination and subsequent carry-over leading to false positives. Had carry-over been an issue in this study, a continuous upward trend in the blank peak amplitudes would have been noticed. This trend is not evident; however, over the course of a week of work, an upward trend is apparent, but gone a few days later. This suggests that a build-up of methamphetamine over time was not always addressed with typical bake-out. Operators in the field would have to be briefed on this possibility and develop the appropriate procedural controls to address these issues.

In Fig. 7, the same data is displayed as a function of the building material identifier. Of note are the wide spreads associated with the electrical outlet (M10), light switch cover (M13), outlet cover (M14), and PVC pipe (M15). All of the surfaces other than the PVC pipe had large wiping surfaces. The blanks associated with the light bulb (M1), latex gloves (M5), and heater motor housing (M4) were excluded due to small data sets for each item ($n=2$). Note the amount of variance in the building material blanks as opposed to the controls which is further evidence that the controls represent the best case scenario that will likely not be encountered in the field. While the mean peak amplitudes of the blanks are generally below 100 dU, this is not uniformly the case and there is clearly a material dependent variation in the blank signals. While it is impractical for operators to account for all of these factors, it is possible (and strongly recommended) that

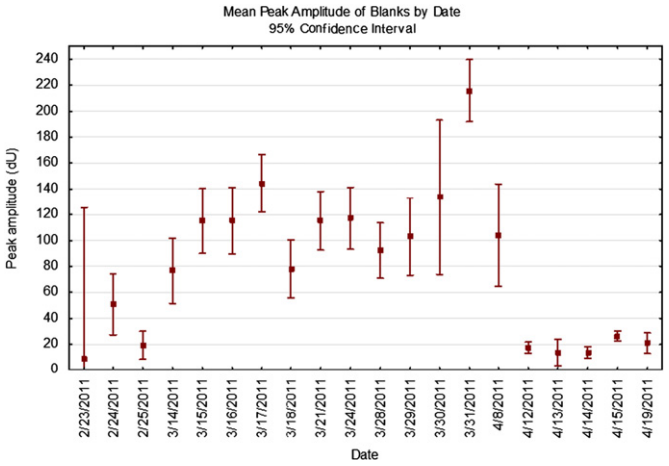


Fig. 6. Variation in methamphetamine peak amplitude as a function of date.

operators establish a control chart that clearly identifies blanks that fall outside of the historical accepted limits. These blanks should include realistic building material matrices.

3.6. Time delay study

Since the building material surfaces were spiked with methamphetamine directly, the protocol for this procedure had to be developed and standardized. The general limits of detection were determined as described above, leaving one key variable for evaluation, the time delay between spiking and swiping for analysis. Methamphetamine as the base is relatively volatile (vapor pressure of 0.163 mmHg) and as such, it was hypothesized that there might be a decrease in recovery as time between spiking and sampling increased, as least on non-porous surfaces such as glass [24–26]. For porous surfaces, the relationship would be more problematic. Several factors, such as the amount of methamphetamine that was absorbed on the surface, the rate that methamphetamine vaporized, the form of the methamphetamine (free base vs. salt), and lastly the reaction of methamphetamine with the atmosphere would have to be taken into consideration. To investigate time effects, several different materials representing a range of apparent porosity were spiked and analyzed after different elapsed times ranging from minutes to days (Fig. 8). The non-porous materials were plastics and glass while the porous materials were wood.

Each building material was thoroughly cleaned with reagent grade alcohol and allowed to dry. The objects were then divided into four 100 cm^2 sections; one served as the blank and the remaining three served as spiking areas. The three areas were

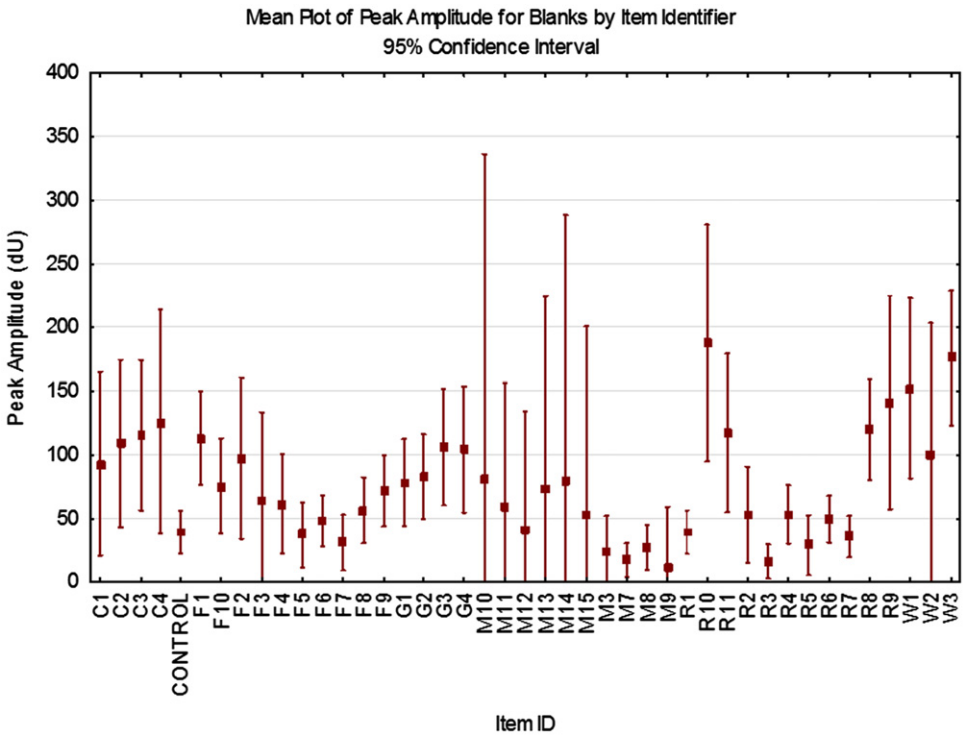


Fig. 7. Variation in methamphetamine peak amplitude on building material surfaces.

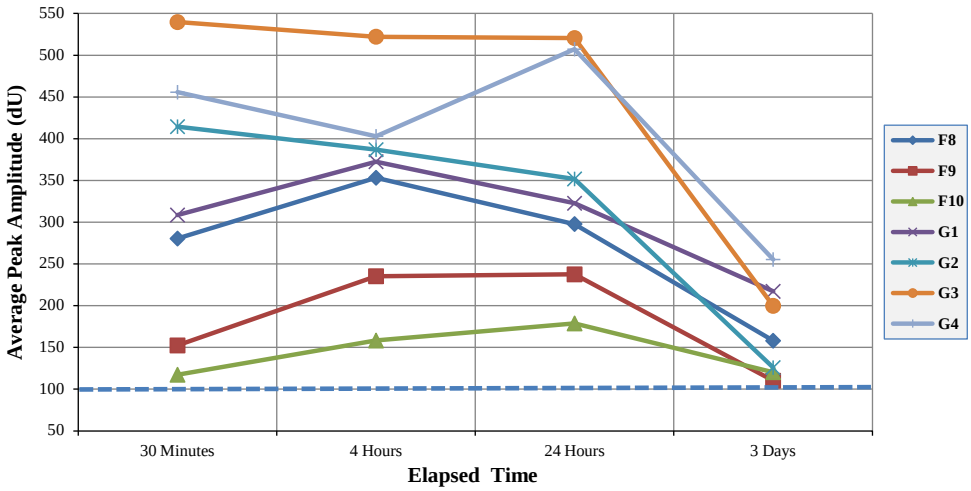


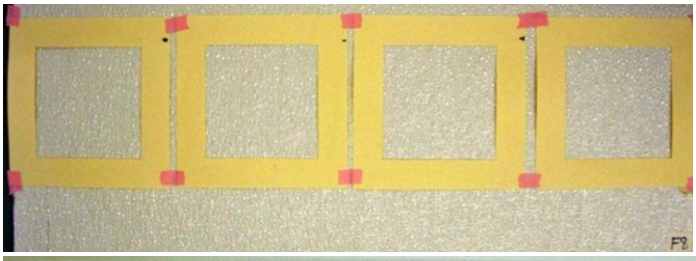
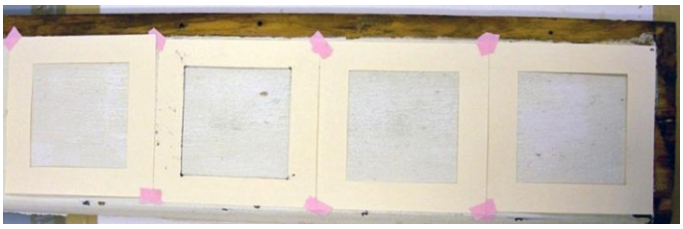
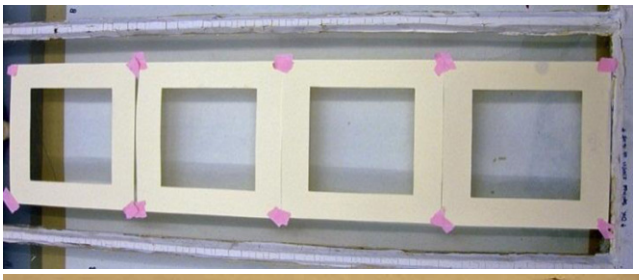
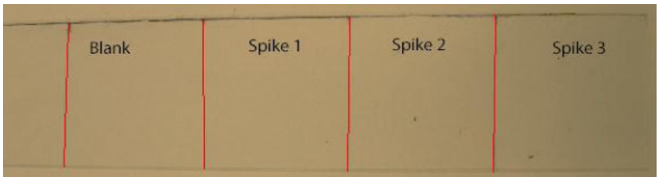
Fig. 8. Elapsed time study for selected building materials.

then spiked with 20 μL of room temperature methamphetamine (100 ppm) using an automatic delivering pipette for an inoculation of 2 μg/application. After the allotted time frame, swabs were moistened with reagent alcohol and applied to the spiking area in circular swiping motions. The swabs were then inserted into the IMS for analysis. This procedure was repeated for every building material and time frame. Two to three clean cycles were executed on the IMS between building materials based on any observed carry-over. Table 5 summarizes the surfaces used and demonstrates how swiping areas were delineated for standard 10 cm × 10 cm squares and for non-standard swipe areas. Note that for the finished baseboard (F9) a 10 cm × 10 cm square area was not feasible.

The recovery from the Plexiglass from a lab hood (G3) with smaller spiked areas remained constant and high until the three day mark. This may be attributed to the small swipe area which allowed for the sample to be swiped essentially completely into the center of the swab. Since the sample was more concentrated in the middle of the swab where the thermal desorption takes place, a higher recovery rate was not surprising.

It appears that with a non-porous surface, the size of the recovery zone plays a role in the total amount of methamphetamine recovered. Moving down to the next two lines in Fig. 8, Plexiglass (G4) and single-paned window (G2) both represent non-porous surfaces and the recovery decreases from 30 min to 4 h elapsed. The reason for the increasing recovery for the single-paned window (G2) from 4 to 24 h is unknown, but could be a reflection of instrument variability. In both cases, the recovery falls off sharply at 3 day. The remaining four materials, Optiplex plastic (G1), shower stall liner (F8), and the interior wood with

Table 5
Time delay surfaces and swiping areas.

Description	Photo showing spiked and swabbed areas
Material: F8 **Relatively non-porous** **Rough surface** Shower wall material with four 10 cm × 10 cm sampling areas. The far left sampling area served as a blank and the other three served as spikes 1, 2, and 3 sampling areas respectively.	
Material: F9 **Relatively porous** **Smooth surface** Painted baseboard molding Sample areas: 7.5 cm × 2 cm	
Material: F10 **Relatively porous** **Slightly roughened surface** Old interior wood, multiple paint layers. Blank cell at left.	
Material: G1 **Relatively non-porous** **Smooth surface** Optiplex plastic	
Material: G2 **Relatively non-porous** **Smooth surface** Window and pane	
Material: G3 **Relatively non-porous** **Smooth surface** Plexiglass strip, sampling size 5 cm × 1 cm	
Material: G4 **Relatively non-porous** **Smooth surface** Plexiglass sheeting	

multiple layers of paint (F10), show similar patterns of increasing recovery from 30 min to 4 h elapsed, followed by declining recoveries thereafter. The dotted line at 100 dU is representative of the estimated limit of detection for the system as previously described.

3.7. Building materials study

This study was designed to capture several sources of potential variation in methamphetamine recovery including analyst, date, substrate (building material), and the time since the spike. On small

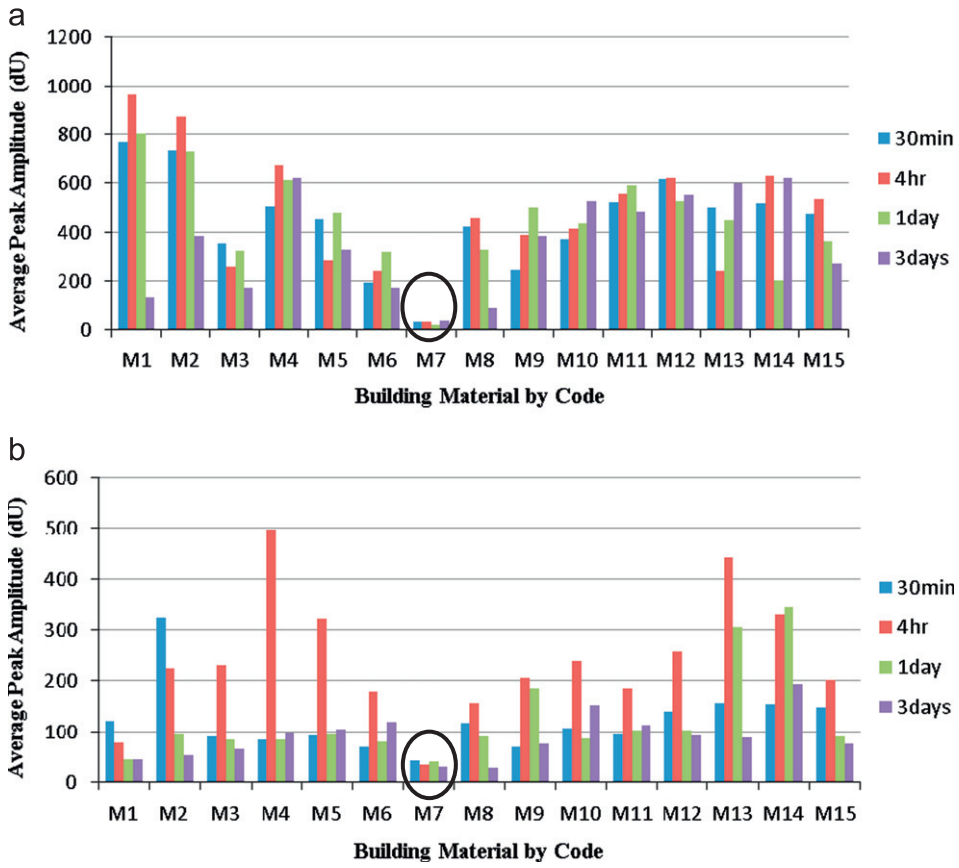


Fig. 9. Variation of certain building materials by analyst and time (a and b).

or oddly shaped surfaces, blocks were marked out and dimensions recorded. One of the blocks served as a negative control while the other three were spiked with 20 μL of the 100 ppm solution which corresponds to a sample load of 2 μg per swabbed zone. After spiking the surface, the analyst returned for wipe analysis after 30 min, 4 h, 1 day and 3 days. Two analysts evaluated each building material on a different series of dates. The materials were divided into the categories previously listed.

Of particular interest were the average amplitudes for the ceiling tile (M7) as shown in Fig. 9a and b. The ceiling tile appeared to absorb a significant amount of methamphetamine as indicated by the recovery. The porous nature of the tile allowed the methamphetamine to be absorbed.

3.8. Methamphetamine recovery based on porosity

The sampling surfaces were grouped visually based upon porosity, which was a qualitative classification, and are listed in Table 6.

The building materials were divided into three categories of porosity: non-porous, mildly porous, and porous. It was believed that the building materials would act in a similar manner when exposed to methamphetamine. Since the four hour time point appeared indicative of all time trials, this was used in the comparison charts.

It was assumed that the non-porous building materials (Fig. 10a) would have higher average amplitudes compared to those of the mildly porous and porous building materials (Figs. 10b and c). The only observable trend is that Trial 1 resulted in higher average amplitudes for all of the miscellaneous bulding materials and that Trial 2 resulted in higher average amplitudes for all of the glass surfaces. The moderately porous building materials exhibited no observable trend. The average amplitudes for both trails were inconsistent. The assumption was made that the porous bulding materials would absorb more methamphetamine than the mildly porous and non-porous materials. This was the case for most of the building materials with the exception of the latex wood (W2) and the wood molding (W3). Trial 1 resulted in higher average amplitudes for 13 of the 15 porous building materials. For the majority of the samples, both trials produced similar results.

Table 6
Surface porosity classification.

Non-porous	Mildly porous	Porous
G1–G4	C1–C4	M3, M7
M1, M4, M6, M9, M11–M12, M15	F1–F10	R1–R11
	M2, M5, M8, M10, M13–M14	W1–W3

4. Conclusion

Portable IMS instruments with thermal desorption capability can be a valuable tool for evaluating clandestine laboratory remediation if proper and reasonable procedures and protocols are employed. The limits of detection of the IMS were in the same range as state guidelines as well as laboratory methods using GC/MS and LC/MS. However, IMS cannot be used as a black box/red-light/green-light device in these applications. Direct vapor sampling (“sniffing”) can be used to detect very high levels of methamphetamine and potential interferents, but vapor sampling cannot approach the detection limits needed for evaluation of remediation efforts. IMS cannot be used alone to determine the

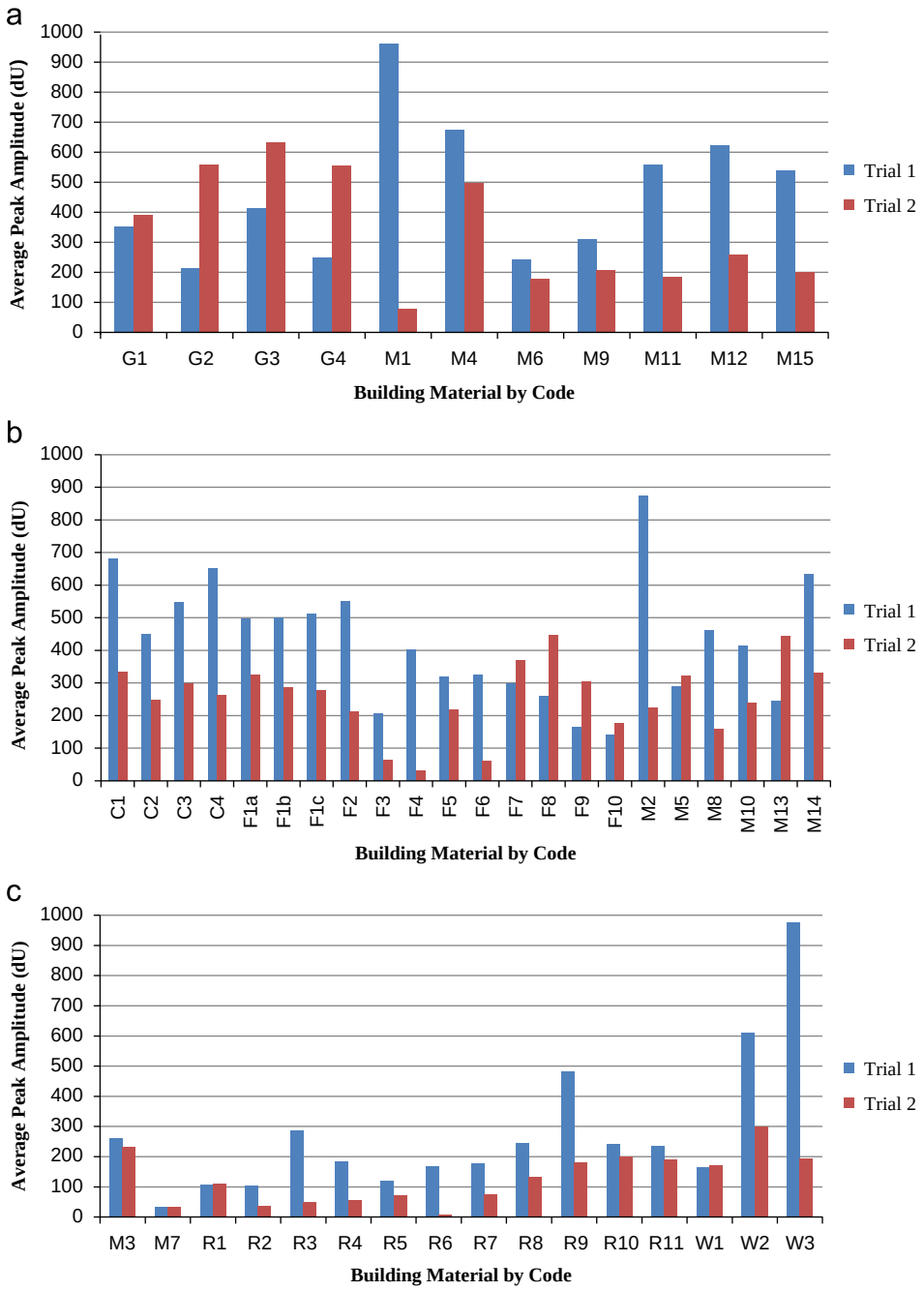


Fig. 10. Building materials based on porosity; non-porous (a), mildly porous (b), and porous (c).

efficacy of remediation efforts; final confirmation using laboratory instrumentation is essential.

Many of the building materials sampled are not amenable to swipe methodology regardless of the instrumentation used. Specific examples identified in this project were sheet rock, untreated wood (the type used in wall studs) and ceiling tiles. It is assumed that methamphetamine penetrates these porous materials resulting in little or no transfer of methamphetamine to the swipes. For remediation purposes, such materials may have to be completely removed and/or new analytical methods developed that can more accurately gauge the level of methamphetamine contamination present.

Under anticipated field conditions, the portable IMS instrument demonstrated detection limits in the same range as state recommended guidelines. The LOD here was approximately 0.09 μg; state levels ranged from 0.05 to 0.5 μg with only Oregon having recommended levels of 0.05 μg. This disparity does not disqualify IMS for use in these situations, but it does require that final levels of residual contamination be confirmed instrumentally.

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