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Law Enforcement and Corrections Standards and Testing Program

Trace Detection of Narcotics Using a Preconcentrator/Ion Mobility Spectrometer System

NIJ Report 602–00

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Prepared for:
National Institute of Justice
Office of Science and Technology
Washington, DC 20531

April 2001

NCJ 187111

National Institute of Justice

The technical effort to develop this report was conducted
under Interagency Agreement 94–IJ–R–004,
Project No. 99–003CTT.

This report was prepared by the Office of Law Enforcement
Standards (OLES) of the National Institute of Standards
and Technology (NIST) under the direction of
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Kathleen M. Higgins, Director of OLES.
The work resulting from this report was sponsored by the
National Institute of Justice (NIJ), Dr. David G. Boyd, Director,
Office of Science and Technology.

FOREWORD

The Office of Law Enforcement Standards (OLES) of the National Institute of Standards and Technology (NIST) furnishes technical support to the National Institute of Justice (NIJ) program to strengthen law enforcement and criminal justice in the United States. OLES's function is to conduct research that will assist law enforcement and criminal justice agencies in the selection and procurement of quality equipment.

OLES is: (1) Subjecting existing equipment to laboratory testing and evaluation, and (2) conducting research leading to the development of several series of documents, including national standards, user guides, and technical reports.

This document covers research conducted by OLES under the sponsorship of the National Institute of Justice. Additional reports as well as other documents are being issued under the OLES program in the areas of protective clothing and equipment, communications systems, emergency equipment, investigative aids, security systems, vehicles, weapons, and analytical techniques and standard reference materials used by the forensic community.

Technical comments and suggestions concerning this report are invited from all interested parties. They may be addressed to the Office of Law Enforcement Standards, National Institute of Standards and Technology, 100 Bureau Drive, Stop 8102, Gaithersburg, MD 20899–8102.

Dr. David G. Boyd, Director
Office of Science and Technology
National Institute of Justice

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COMMONLY USED SYMBOLS AND ABBREVIATIONS

A	ampere	H	henry	nm	nanometer
ac	alternating current	h	hour	No.	number
AM	amplitude modulation	hf	high frequency	o.d.	outside diameter
cd	candela	Hz	hertz	Ω	ohm
cm	centimeter	i.d.	inside diameter	p.	page
CP	chemically pure	in	inch	Pa	pascal
c/s	cycle per second	IR	infrared	pe	probable error
d	day	J	joule	pp.	pages
dB	decibel	L	lambert	ppm	parts per million
dc	direct current	L	liter	qt	quart
°C	degree Celsius	lb	pound	rad	radian
°F	degree Fahrenheit	lbf	pound-force	rf	radio frequency
dia	diameter	lbf·in	pound-force inch	rh	relative humidity
emf	electromotive force	lm	lumen	s	second
eq	equation	ln	logarithm (base e)	SD	standard deviation
F	farad	log	logarithm (base 10)	sec.	section
fc	footcandle	M	molar	SWR	standing wave ratio
fig.	figure	m	meter	uhf	ultrahigh frequency
FM	frequency modulation	min	minute	UV	ultraviolet
ft	foot	mm	millimeter	V	volt
ft/s	foot per second	mph	miles per hour	vhf	very high frequency
g	acceleration	m/s	meter per second	W	watt
g	gram	N	newton	λ	wavelength
gr	grain	N·m	newton meter	wt	weight

area=unit^2 (e.g., ft^2, in^2, etc.); volume=unit^3 (e.g., ft^3, m^3, etc.)

PREFIXES

d	deci (10^{-1})	da	deka (10)
c	centi (10^{-2})	h	hecto (10^2)
m	milli (10^{-3})	k	kilo (10^3)
μ	micro (10^{-6})	M	mega (10^6)
n	nano (10^{-9})	G	giga (10^9)
p	pico (10^{-12})	T	tera (^{1012})

COMMON CONVERSIONS
(See ASTM E380)

0.30480 m = 1 ft	4.448222 N = 1 lbf
2.54 cm = 1 in	1.355818 J = 1 ft·lbf
0.4535924 kg = 1 lb	0.1129848 N m = 1 lbf·in
0.06479891g = 1gr	14.59390 N/m = 1 lbf/ft
0.9463529 L = 1 qt	6894.757 Pa = 1 lbf/in^2
3600000 J = 1 kW·hr	1.609344 km/h = 1 mph

Temperature: $T_{(°C)} = (T_{(°F)} - 32) \times 5/9$
Temperature: $T_{(°F)} = (T_{(°C)} \times 9/5) + 32$

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TRACE DETECTION OF NARCOTICS USING A PRECONCENTRATOR/ION MOBILITY SPECTROMETER SYSTEM

J.E. Parmeter,[1] Gary A. Eiceman,[2] Jaime E. Rodriguez[2]

This report discusses work performed in the area of trace drug detection, primarily at New Mexico State University (NMSU), during fiscal year 1999. These experiments combined a chemical preconcentrator and associated control hardware developed at Sandia National Laboratories (SNL) with an ion mobility spectrometer (IMS) constructed at NMSU. The preconcentrator is of the same patented design used in the SNL trace detection portal that screens personnel for explosives, and the IMS is of a cross-flow design where analyte molecules are ionized downstream from the ion source region. The overall goal of these studies was to investigate the efficacy of the preconcentrator in the general field of drug detection. In addition, it was hoped to make an initial determination concerning the feasibility of a trace drug detection portal for personnel screening that would operate on the same principles as the explosives detection portal. Based on our current results, it appears that such a drug detection portal could be developed, but more research and development is needed to work toward this goal. The next logical step in the development of such a portal would be to extend the present studies to include detection of both trace and bulk drug samples in a mock-up portal. The principal results discussed in this report include the use of the IMS to detect drugs with and without nicotinamide dopant and studies of the preconcentrator efficiency. Limits of detection were not investigated in detail, but the IMS could easily detect one microgram of all the drugs studied. The primary drugs studied are methamphetamine, cocaine, heroin, and tetrahydrocannabinol (THC).

We are indebted to Dr. Alim Fatah of the NIST Office of Law Enforcement Standards for programmatic support and for numerous useful discussions about the technical content of this report. At Sandia National Laboratories, Charles Rhykerd Jr., Frank Bouchier, and Lester Arakaki were responsible for the development and construction of the preconcentrator and associated system controls. We thank Dr. William MacCrehan of NIST for a thorough review of this report.

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1. INTRODUCTION

Due to the major societal problems associated with narcotics abuse, the detection of illicit drugs is currently an area of major research interest. Several broad categories of detection techniques are important, including imaging methods such as x-ray based technologies, the use of trained canines, and trace chemical detection utilizing various "sniffer" technologies. The last category involves indirect detection of a drug by collecting and analyzing minute quantities of vapor or particle contamination. Several technologies have been developed for this type of application, of which ion mobility spectrometry (IMS) is perhaps the most widely utilized [1].[3] This technology has a number of advantageous features, including the potential ability to detect almost all drugs of interest, moderate cost, near instantaneous response time, and sensitivity in the sub-parts per billion range in some cases.

Despite the advantages associated with the use of IMS to detect illicit drugs, there are also disadvantages that can be of considerable importance in many applications, especially when large volumes of personnel need to be screened rapidly. Foremost among these is the problem of collecting and delivering to the detector a sample of the drug that is large enough to result in a positive detection. In the past, collection of particle samples has usually relied on surface swiping, i.e., taking a sampling pad and wiping it across a surface to try to collect particle contamination. This can be very effective, but it is sufficiently slow that it limits throughput to about one sample every 30 s. This is too slow in some cases, such as when there is a desire to screen every person entering an airport terminal, or at a busy border crossing. Furthermore, swiping of clothing or skin may be considered excessively invasive when personnel screening is involved. The alternative to particle collection is to collect vapor or airborne particulate material using some form of vacuuming. This is less invasive and potentially faster, but the extremely low vapor pressures of some drugs make it difficult or impossible to collect an adequate amount of sample in a sufficiently concentrated form when using this collection method. For these reasons, there is at present a need to improve sampling techniques in order to apply the trace detection of illicit drugs to the problem of personnel screening.

Similar problems exist in the area of trace detection of explosives, and an explosive detection portal recently developed at SNL with primary funding from the Federal Aviation Administration employs technology that is likely also to be applicable in the field of illicit drug detection. A schematic representation of this portal is shown in figure 1. The portal uses specially designed airflows over the body of a test subject to entrain explosives vapor and/or particle contamination, and then directs the airflows into a patented preconcentrator. The preconcentrator collects high molecular weight organic compounds[4] such as explosives (and potentially drugs) via adsorption, while allowing the air to flow through to an exhaust line. After this adsorption cycle, the adsorbing surface in the preconcentrator is heated to return the explosive material back into the gas phase in a much more concentrated form, and a much

[3] Numbers in brackets refer to references in section 5.

[4] By "high molecular weight" is meant molecules with molecular weights on the order of 100 to 200 atomic mass units (amu) or more, i.e., high in comparison to the main constituents of air such as nitrogen, oxygen, argon, etc.

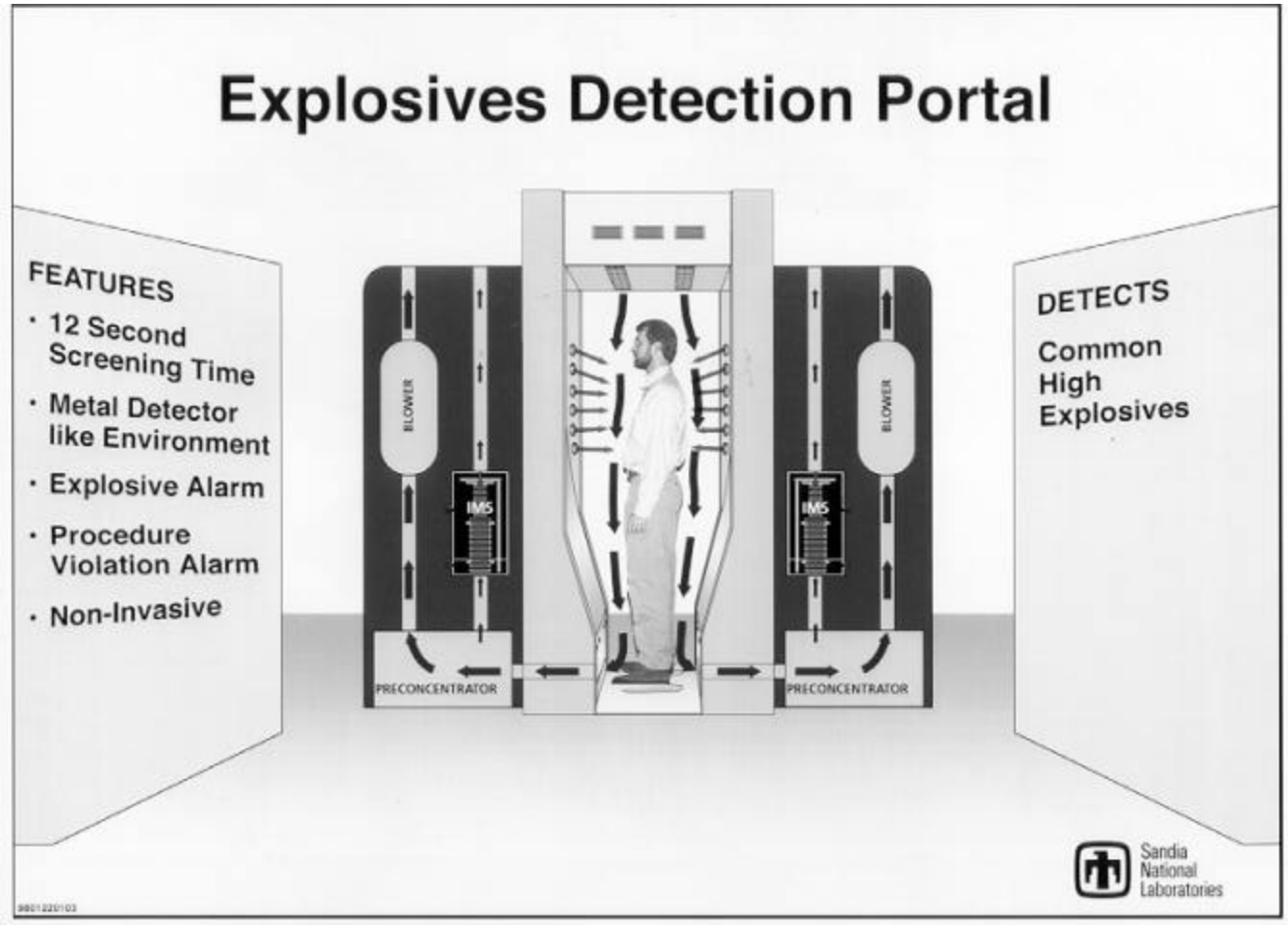


Figure 1. Schematic representation of the explosives detection portal developed at Sandia National Laboratories

smaller airflow delivers the explosive material into an IMS for detection. This portal can currently process people at the rate of one every 12 s, and thus helps address both the speed and sample collection problems associated with trace chemical detection of organic analytes.

The above discussion makes clear that it would be very useful to extend the trace detection portal technology from explosives detection to the area of illicit drug detection. With this goal in mind, the National Institute of Justice (NIJ) through the Office of Law Enforcement Standards (OLES) at the National Institute of Standards and Technology (NIST) provided funding in fiscal year 1999 to investigate the use of a preconcentrator/IMS system in a laboratory setting for the application of drug detection. A substantial part of the funding was utilized in system development and construction, so the scope of the experimental work was limited. Nevertheless, the present work is aimed at laying the groundwork for the development of a trace drug detection portal in the near future, provided that suitable funding can be obtained.

2. EXPERIMENTAL METHODS[5]

2.1 IMS System

These studies utilized a high temperature, side-flow IMS that was designed and built in the Department of Chemistry and Biochemistry at NMSU. A schematic of this instrument is shown in figure 2, and a photo is shown in figure 3. The IMS consists of a high-voltage region with a ^{63}Ni ionization source; a medium-voltage region that accepts the cross flow of air containing the incoming sample, and in which charge transfer occurs; and a low-voltage drift region where ion separation occurs. The system was operated with a voltage gradient of 250 V/cm. The high-voltage region consists of a repeller plate at the highest voltage (ca. 4050 V), followed by the ion source and three drift rings that are separated and independently insulated. The medium-voltage region contains two focusing rings that allow charge transfer to occur between the ions entering from the high-voltage region and the neutral analyte molecules coming out of the preconcentrator and entering the IMS in the cross flow at the sample inlet. These focusing rings produce an electric field to focus the generated positive ions into the drift region, while neutral molecules are exhausted through the opposite side of the instrument. The low-voltage or drift region contains an ion shutter at its entrance, four drift rings, an aperture grid, and the detector plate that collects the ions. All of these are uniformly separated and independently insulated. The IMS high temperature is applied by an Omegalux[5] flexible heating tape and controlled by a series 6100 temperature controller, both from Omega Engineering (Stamford, CT). The IMS high-voltage is generated by a Gamma High-Voltage Research (Mt. Vernon, NY) power supply. The other electronic devices needed to operate the IMS, such as the shutter control unit and current amplifier, were designed and built in-house.

In some experiments, the IMS alone was used to detect drugs, while in others it was coupled to a chemical preconcentrator. The preconcentrator and associated controls were designed and constructed in Department 5848 at SNL. A photo of the complete IMS/preconcentrator system is shown in figure 4, and a schematic of how the preconcentrator works is shown in figure 5. In essence, the preconcentrator is a molecular filter, in which a large airflow containing analyte molecules is pulled through a high-density mesh of stainless steel fibers known as metal felt. The metal felt adsorbs a large fraction of any high-molecular weight organic molecules such as drugs (whether in vapor or particulate form), while allowing air to pass through and continue to an exhaust line. Once the heavy organic molecules are collected on the metal felt, the felt can be heated resistively to desorb these molecules back into the gas phase. At this point, an airflow that is much smaller than the original flow into the preconcentrator, and in an orthogonal direction, carries the desorbed molecules into the IMS for detection. The preconcentrator allows analyte vapors to be concentrated by a factor of 100 to 1 000 prior to their entry into the detector and also reduces the airflow containing the sample to one that can be accommodated by an IMS.

[5]Certain commercial equipment, instruments, or materials are identified in this paper to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by neither the National Institute of Standards and Technology (NIST), nor the U.S. Department of Justice, nor does it imply that the equipment, instruments, or materials are necessarily the best available for the purpose.

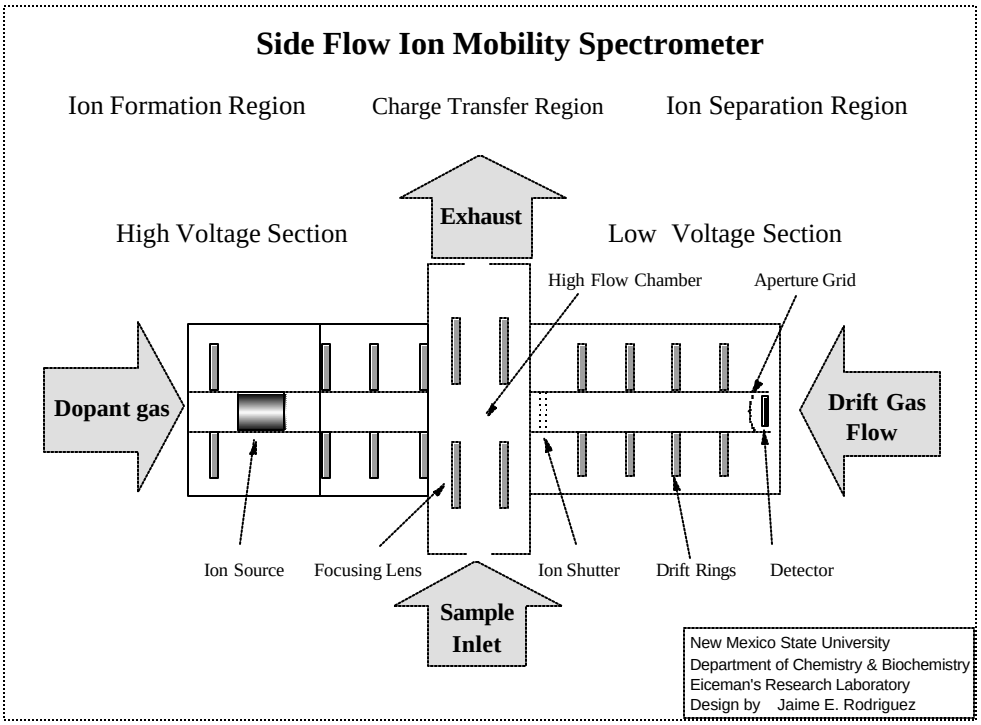
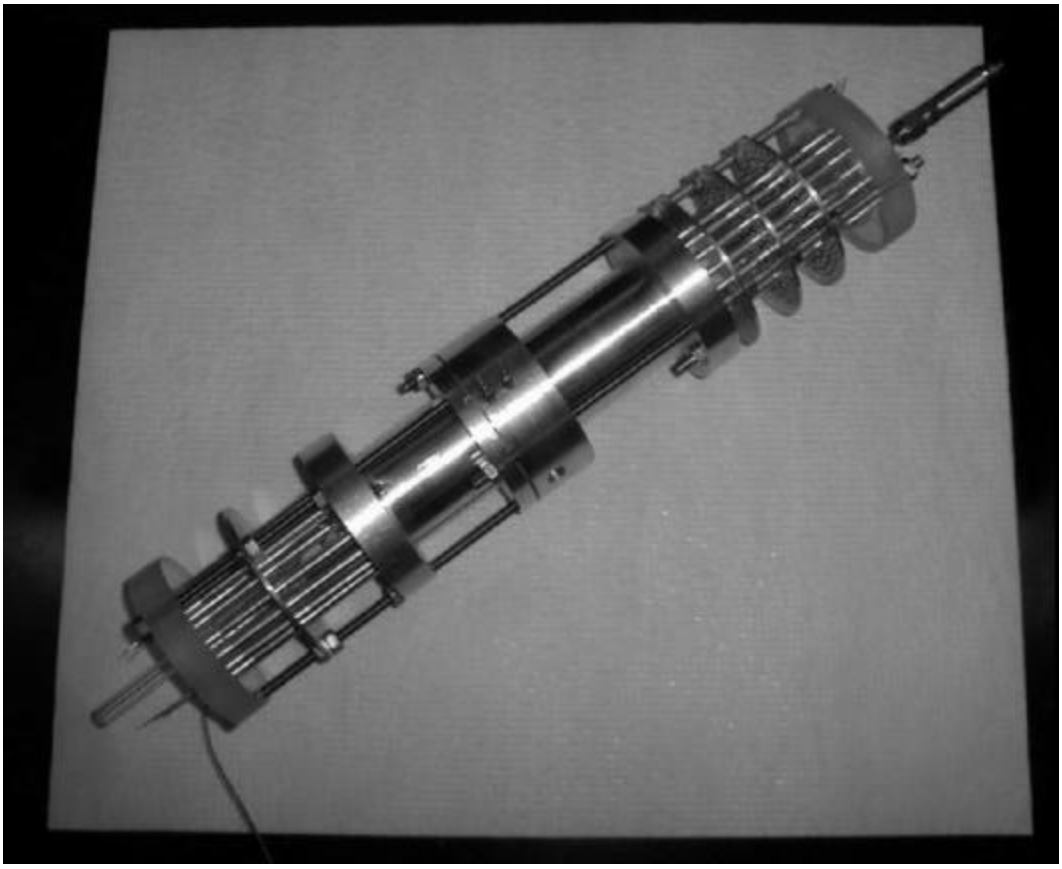


Figure 2. Schematic representation of the IMS used in these studies, which was designed and built at New Mexico State University



Fiaure 3. Photo of the IMS. The ion formation reaion is at the lower left

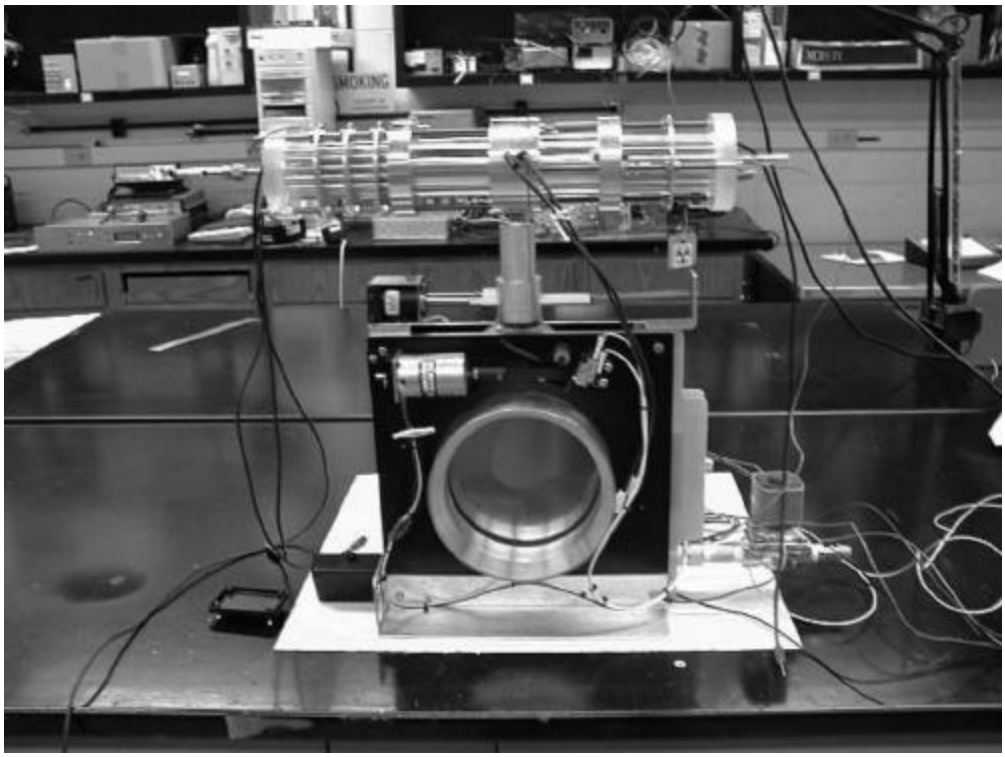


Figure 4a. The IMS mounted on top of the Sandia preconcentrator

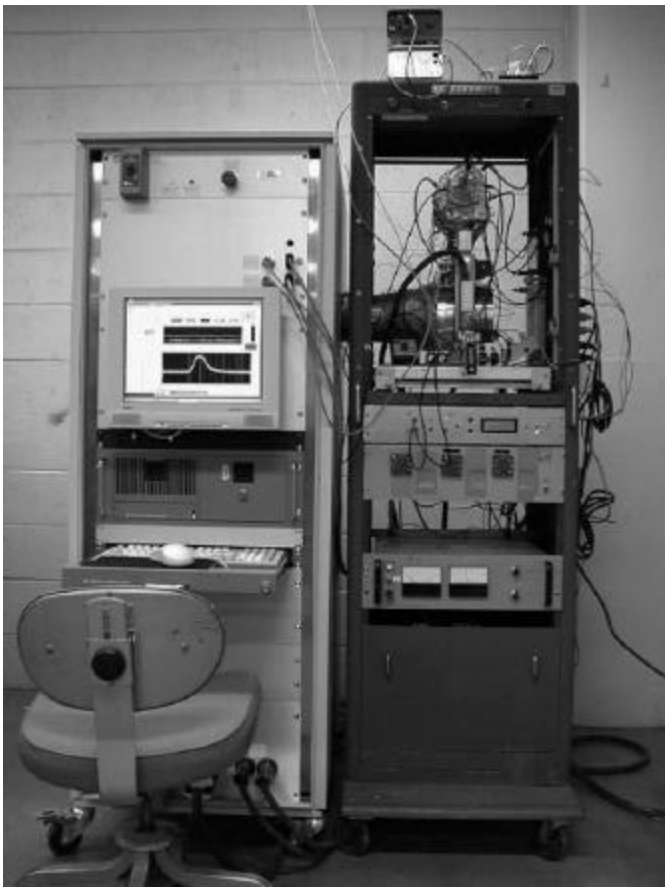


Figure 4b. Power racks containing the IMS, preconcentrator, and system controls

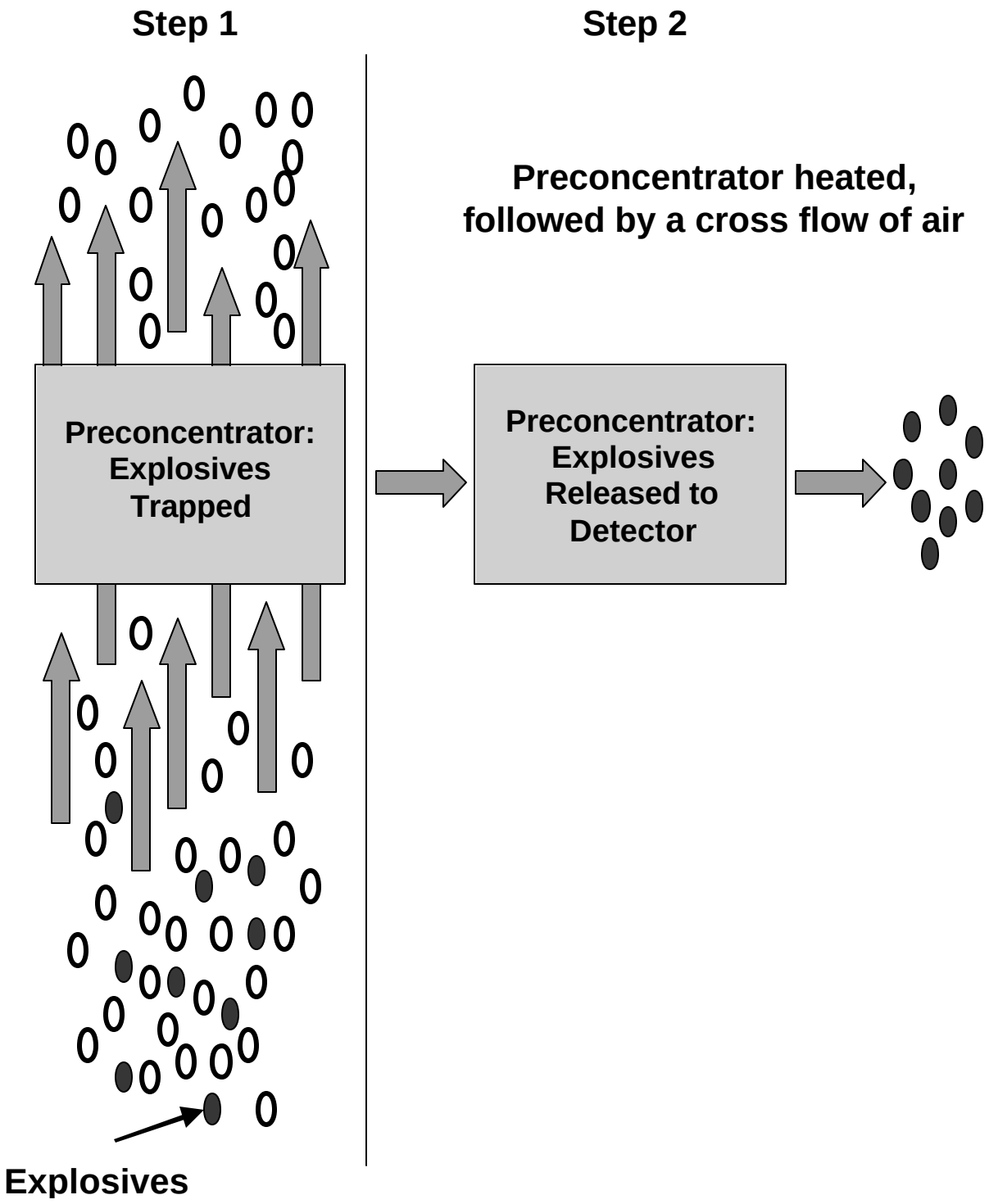


Figure 5. Schematic representation of the trapping of analyte molecules in the preconcentrator, with subsequent delivery to the detector

Experimental parameters in these studies included the following: IMS operation temperature, 250 °C; drift gas flow rate, 350 ml/min; and maximum voltage, 4050 V. The gate pulse width for the ion shutter was 400 μs, and 100 drift measurements were signal averaged to produce a single spectrum. Ten such spectra were acquired for each sample injection. In the studies used to estimate collection efficiencies, 10 separate injections were performed for each chemical, in order to produce the most valid possible average values. Nicotinamide dopant was used in most of these studies to create a reference ion peak and aid in the ionization of the drugs under investigation. The nicotinamide was packed in a glass tube and wrapped with a flexible heating tape. An airflow of 150 ml/min and a temperature of 150 °C were applied, and the nicotinamide was introduced directly into the high voltage region containing the ion source.

2.2 Preconcentrator

In experiments utilizing the preconcentrator, samples of the various drugs could be introduced into the preconcentrator either by placing a drop of solution containing the drug directly onto the preconcentrator's metal felt using a syringe ("direct injection"), or by desorbing drug vapor into the inlet airflow of the preconcentrator, resulting in adsorption of some of the vapor onto the metal felt ("flash desorption" or "vapor inhale"). In the direct injection experiments, the metal felt was heated to approximately 240 °C in 1.55 s. One microliter (1 μL) of drug solution was deposited onto the metal felt. The drug solutions in methanol solvent were obtained from Alltech. The specific solutions and concentrations used were as follows: 9-THC, catalogue # 013873, 100 μg/mL; cocaine, catalogue # 018003, 1.0 mg/mL; d-methamphetamine, catalogue # 010013, 1.0 mg/mL; and heroin, catalogue # 013653, 1.0 mg/mL. The gas flow rate ("desorb flow") from the preconcentrator to the IMS was approximately 4 L/min.

Flash desorption experiments utilized identical metal felt heating temperatures, desorb flow rate, and sample solutions. Generation of vapor into the inlet airflow of the preconcentrator was accomplished by placing 1 μL of sample solution onto a stainless steel scoop located at the tip of a thermal desorption probe. The probe was then resistively heated to approximately 350 °C for 10 s, while it was held in the inlet airflow of the preconcentrator approximately 2.54 cm (1 in) from the metal felt. The inlet airflow was run for a total of 15 s, with an approximate rate of 100 L/s.

3. RESULTS

3.1 IMS Detection of Key Drugs

Typical IMS spectra of several key drugs are shown in figures 6 and 7. In these studies, drugs were introduced directly into the IMS without use of the preconcentrator. This was accomplished by inserting a syringe into the charge transfer (medium-voltage) region through the exhaust port (see fig. 2), and then depressing the syringe to emit 0.5 μL of drug solution. At the IMS temperature of 250 °C, such a drop vaporizes very rapidly. In each figure, the bottom spectrum is a typical spectrum with no added drug, corresponding to the spectrum of air in figure 6, and of nicotinamide in figure 7. The remaining spectra, from the bottom to the top of the figure, are for d-methamphetamine, cocaine, heroin, and tetrahydrocannabinol (9-THC). Note that the spectra obtained with nicotinamide dopant tend to show fewer peaks than that obtained in pure air. This indicates that the presence of nicotinamide tends to stabilize large drug ions, while in the absence of nicotinamide there is an increased tendency for the drugs to break up into smaller fragments. In addition to the nicotinamide peak with a drift time of approximately 7.5 ms, the spectra of drugs with nicotinamide dopant show the following peaks: d-methamphetamine, 9 ms; cocaine, 13 ms; heroin, 15 ms (major) and 13.5 ms (minor); and THC, 15 ms (major) and 11 ms (minor). The relative drift times for d-methamphetamine (molecular weight = 149 atomic mass unit (amu), cocaine (303 amu), and heroin (369 amu) are in accord with the relative molecular weights of these species, while that of THC (251 amu) is higher than would be expected based solely on molecular weight. This last result is not necessarily surprising, since drift time is a complex function not only of molecular weight and ion charge, but also of molecular shape. In general, these results indicate that all four of these drugs exhibit distinctive IMS spectra with well-defined peaks when using nicotinamide dopant, and hence IMS should be a good method for the detection of these species. However, the primary peaks for THC and heroin in figure 7 are at nearly identical drift times, suggesting that these two drugs may be difficult to distinguish from one another. More work is needed to determine whether this result can be altered by changing the experimental conditions, or if it might be an artifact due to cross contamination of the samples used.

3.2 Combined Preconcentrator/IMS System

A preconcentrator used by itself is difficult to study quantitatively, since it has no inherent detection capability. However, it gains this capability when coupled with an IMS, so studying the two as an integrated unit allows the important performance characteristics of the preconcentrator to be determined. Perhaps the single most important parameter is the preconcentrator efficiency.

Preconcentrator efficiency, hereafter referred to simply as efficiency for the sake of brevity, is a measure of how effective the preconcentrator is at collecting the substance to be detected from the incoming airflow. The value of the efficiency must be between zero and one: it will be zero if the preconcentrator does not collect any of the incoming drug vapor/particle contamination, and one if the preconcentrator collects all of this residual material. Clearly it is desirable to have the efficiency be as close to one as possible, and knowing the value of this parameter even approximately permits conclusions to be drawn about how much it might be possible to improve

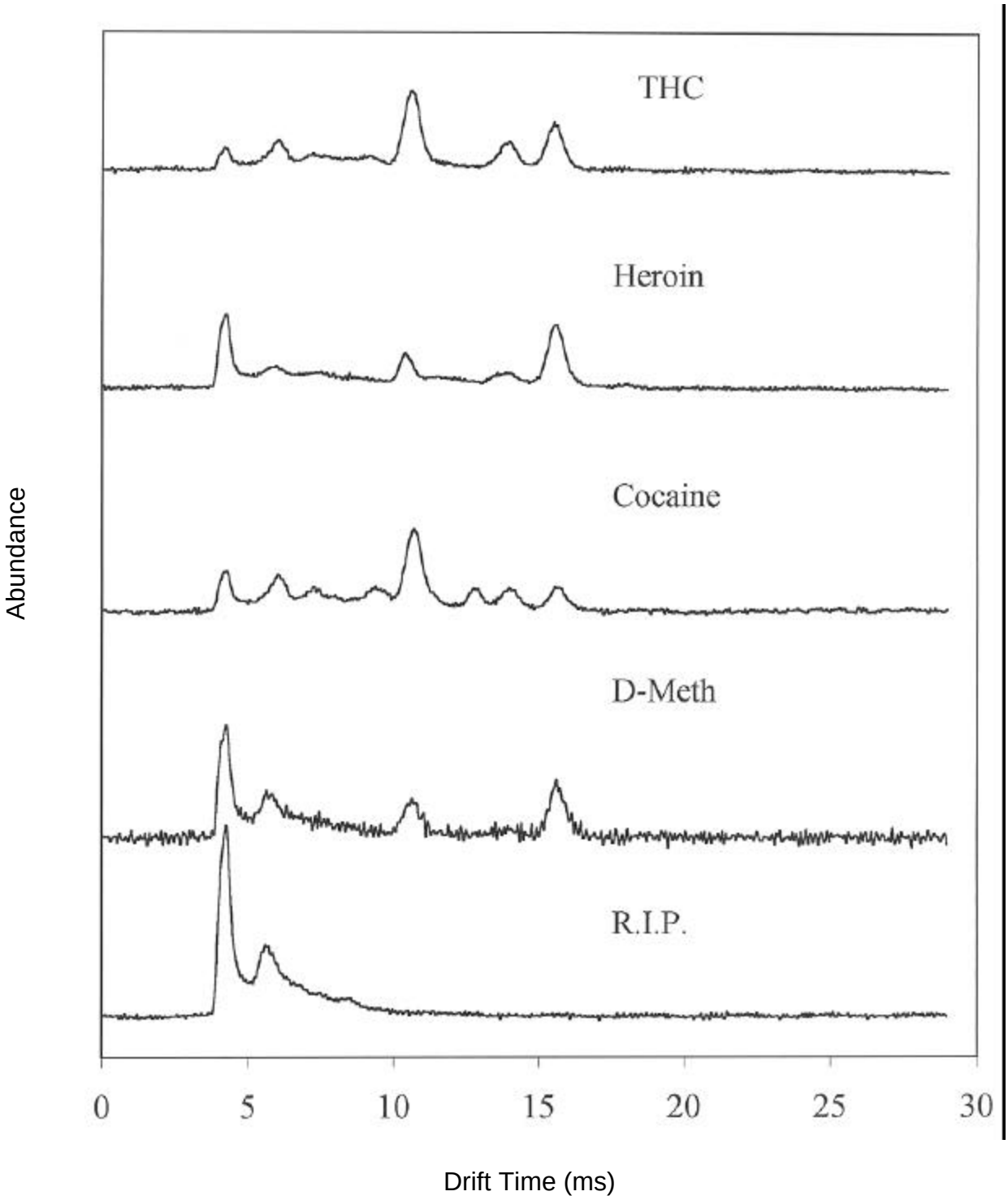


Figure 6. IMS spectra for different drugs without the use of nicotinamide dopant. The quantity of all drugs injected into the IMS was 500 ng in all cases, except for THC where only 50 ng was injected

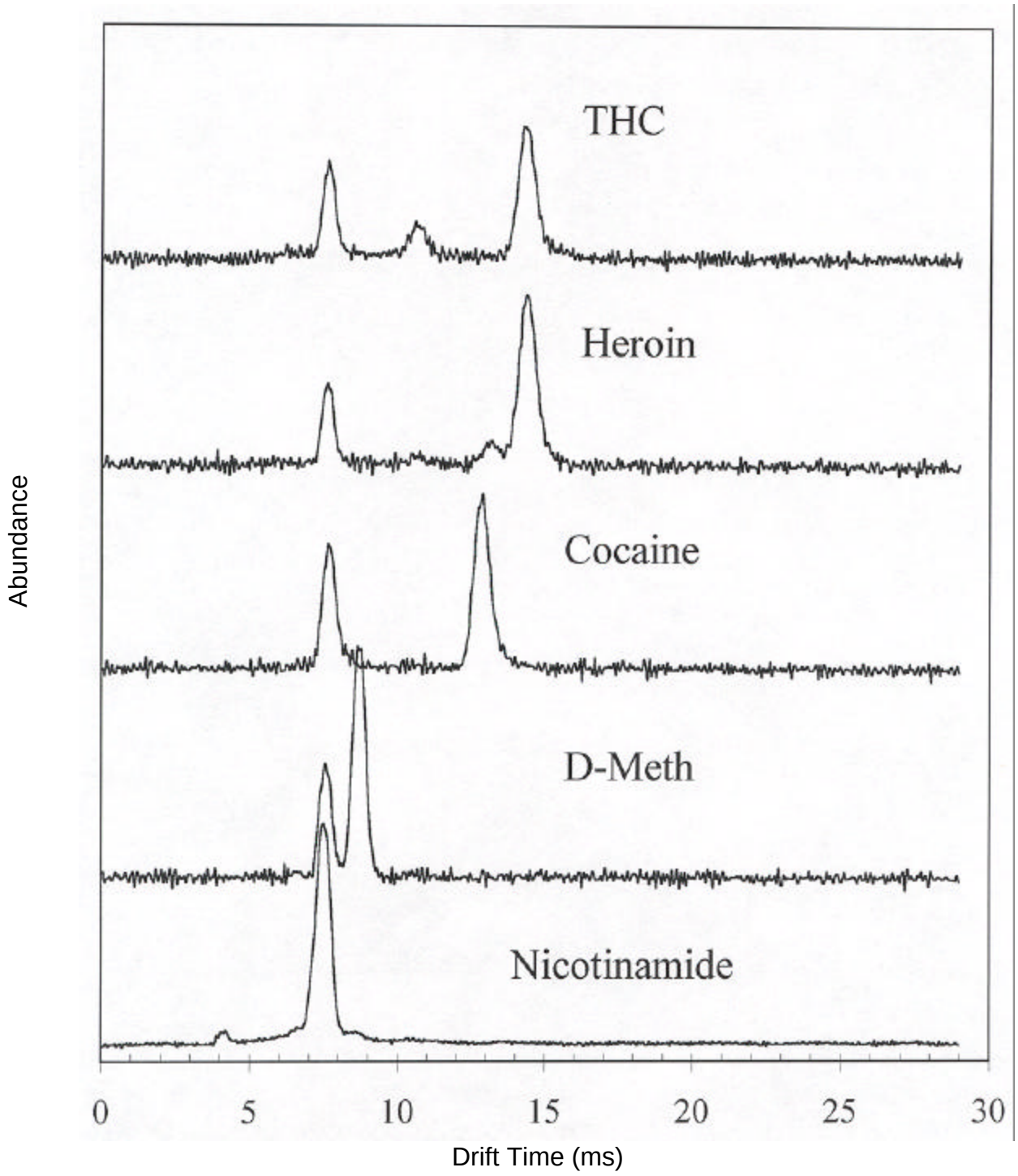


Figure 7. IMS spectra for different drugs with the use of nicotinamide dopant. The quantity of drug injected is 1 mg in all cases except for THC where 100 ng was used

the preconcentrator. With the present preconcentrator design, a good approximation of the efficiency can be obtained by performing two measurements. In the first, a given mass of a particular drug from a standard solution is deposited onto the tip of a flash desorber and then desorbed into the inlet airflow of the preconcentrator. After this occurs, the metal felt in the preconcentrator is heated to desorb the collected drug molecules back into the gas phase, and the material is then delivered to the IMS for detection. The magnitude of the resulting signal is the result of this first measurement. The second measurement is largely the same, except that a syringe is used to directly deposit the drop of solution containing the drug onto the metal felt in the preconcentrator. This assures that 100 % of the trace drug material is collected on the felt, and this material can then be delivered to the IMS as in the first measurement. This will yield a second, larger quantitative signal. Taking the ratio of the signal obtained in the first measurement to that obtained in the second measurement provides an approximate efficiency value. This estimate does not take into account the fact that some of the drug may decompose on the flash desorber tip when it is heated; a phenomenon that is important with some drugs and which would tend to reduce the estimated efficiency value.

Figure 8 shows a plot of a series of such measurements made with methamphetamine. To ensure good statistics, 10 measurements of both the first and second types described above were obtained, alternating between flash desorptions and "drop-on-the-screen" (or direct deposition) tests. The x-axis plots the trial (measurement) number, and the y-axis shows the associated IMS signal intensity. Experimental conditions were as follows: probe temperature approximately 350 °C during flash desorption; preconcentrator inlet airflow approximately 100 L/s; metal felt temperature approximately 200 °C during heating to desorb the drug; 1 μg of methamphetamine in 1 μL of standard solution was used in both types of tests; and the IMS was run with a drift cell temperature of 250 °C. As expected, direct deposition of the drug onto the screen always resulted in a larger signal than a flash desorption into the inlet airflow, though the value of this ratio varied considerably over the course of the 10 tests. On average, the signal obtained for a flash desorption test was 57 % of the signal obtained for a direct deposition, indicating an efficiency value of nearly 60 %. This indicates that even a theoretically perfect preconcentrator would be less than a factor of two better than the present preconcentrator, at least for this drug under this particular set of circumstances. It is noteworthy that the signal for the flash desorptions tended to decrease during the course of the 10 measurements, while that for the direct depositions tended to increase slightly.

Figure 9 shows a similar set of measurements for cocaine, with identical experimental conditions. For this drug the average efficiency has the nearly identical value of 56 %, though there is considerably more scatter in the individual data points. This may be related to the much lower vapor pressure of cocaine compared to methamphetamine, that may lead to greater variations in the amounts that decompose if there are minor variations in the heating temperatures of both the flash desorber and the preconcentrator metal felt. Figure 10 shows a similar figure for experiments performed with heroin. In this case, there is again a large amount of scatter in the data (especially for the direct depositions), and the average efficiency is estimated to be only 17 %. This is probably indicative of decomposition of the heroin on the tip of the flash desorber, rather than an inability of the preconcentrator to collect the heroin, although additional work will be needed to clarify this. The vapor pressure of heroin is very low even in comparison to that of cocaine, being in the low parts per trillion range. Therefore, it

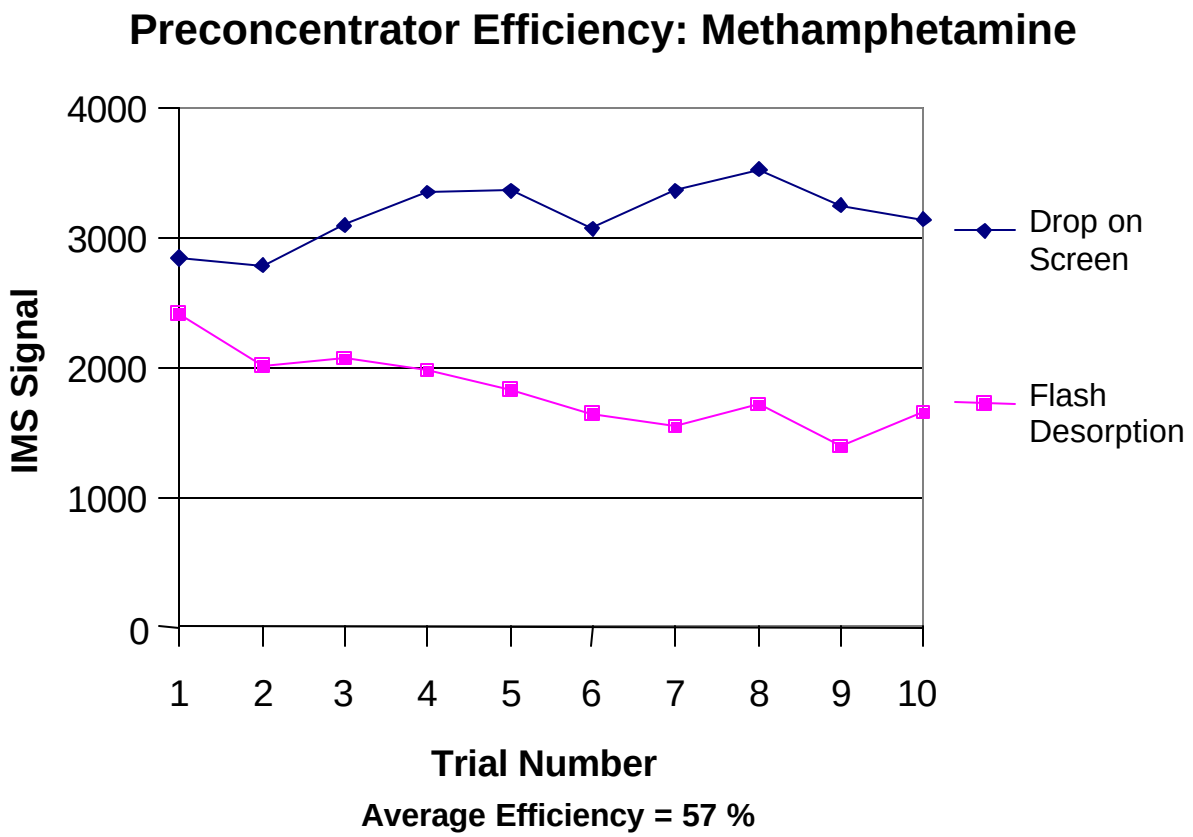


Figure 8. Set of 10 measurements used to estimate preconcentrator collection efficiency for methamphetamine

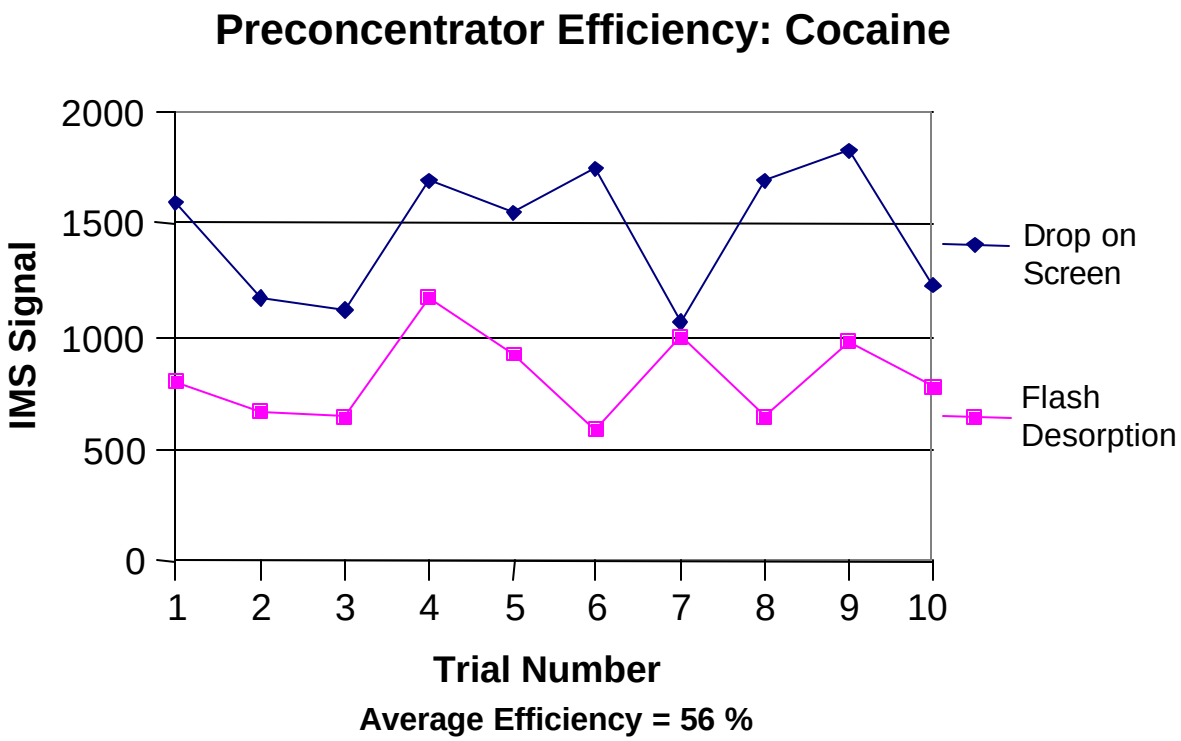


Figure 9. Set of 10 measurements used to estimate preconcentrator collection efficiency for cocaine

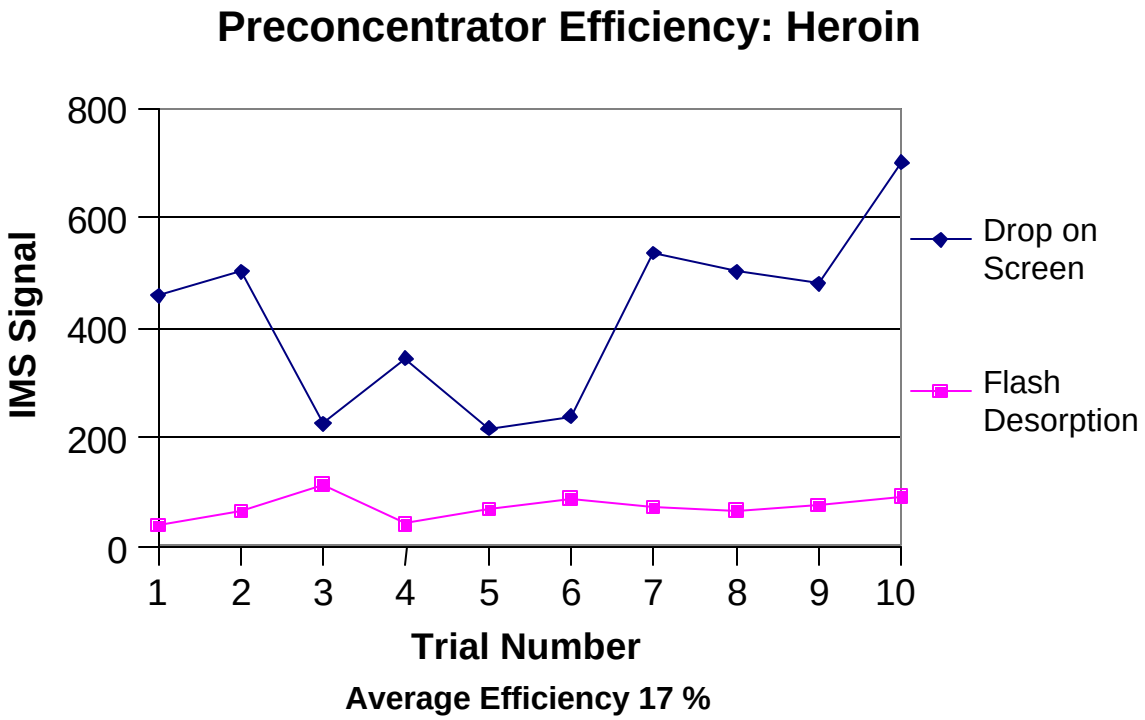


Figure 10. Set of 10 measurements used to estimate preconcentrator collection efficiency for heroin

seems likely that in the course of heating the desorber tip to 350 °C, much of this drug decomposes before being liberated into the gas phase. This suggests that further studies are needed, either utilizing lower flash desorber temperatures, or completely different means of heroin volatilization.

A combination of time and funding limits, as well as difficulties with the equipment, prevented the performance of detailed detection limit studies for the preconcentrator/IMS unit. However, the tests represented by figures 8 and 9 showed that 1 μg of drug vapor desorbed into the preconcentrator could be detected easily, at least in the case of methamphetamine and cocaine. Since little effort was invested in trying to optimize the experimental parameters, the 1 μg figure can be considered an upper bound on the limit of detection.

4. CONCLUSIONS AND FUTURE WORK

The results of this study are generally encouraging regarding the future application of this technology to trace detection of illicit drugs, and in particular to the development of a personnel portal for that use. The major drugs of interest are all readily detected using IMS, particularly when nicotinamide dopant is utilized. More work is needed to investigate issues such as the distinction of heroin and THC, and the detection of the drugs in a real-world environment, when various interferents may be present. The preconcentration efficiency for both methamphetamine and cocaine is greater than 50 %, indicating that the preconcentrator is effective in collecting these molecules from an incoming airflow. Detection limits of the preconcentrator/IMS system are at worst on the order of 1 μg.

The next logical extension of this work would be to conduct trace drug detection experiments in a mock-up personnel portal already existing at SNL. The studies to be performed would include experiments both with true trace samples, such as clothing contaminated with fingerprints containing drug particles, and with bulk drug samples concealed under clothing. In addition, some further laboratory studies in trace detection at NMSU are desirable; especially to obtain some refined data on detection limits and preconcentrator efficiency for more types of drugs. Given that all of the necessary equipment has been purchased and developed, and also that much experience has been gained in learning how to use the equipment for trace drug detection, a relatively small funding investment in the future could produce significant research returns in terms of knowledge gained.

5. REFERENCE

[1] Eiceman, G. A. and Z. Karpas, *Ion Mobility Spectrometry*, CRC Press, Boca Raton, 1994 (presents a summary of the use of IMS in the area of drug detection).

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