

Application of the Ionscan for the Detection of Methamphetamine and Ephedrine in Abandoned Clandestine Laboratories.

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ABSTRACT

Clandestine methamphetamine laboratories are prevalent in southern California. The most common encountered synthesis results in vapor release, and drug residue being left behind. The suspected manufacturing area can be vacuumed and/or methanol wiped and screened immediately at the lab site using the Ionscan. Positive results are confirmed by obtaining vacuum sweep samples with subsequent analysis at the DEA Laboratory. This procedure has been utilized successfully for identifying methamphetamine and ephedrine from clandestine laboratories that have been abandoned and/or remodeled.

INTRODUCTION

Ion Mobility Spectrometry (IMS) is the characterization of chemical substances by measurement of their gas-phase ion mobilities¹. Chemical substances are heated to vaporization, ionized, and allowed to drift within a controlled electric field. Depending on molecular size and structure, the ions move at different speeds². The measured ion mobility, or drift time, is characteristic of the substance from which the ions came. When compared to a known standard, drift time can be used as a presumptive test.

This technology has been used for approximately fifteen years for drug detection. Drugs such as barbiturates³, cocaine, and heroin⁴ dissolved in solution have been studied. Until recently, little work has been done on solid samples. With the introduction of the Barringer Ionscan, solid samples have been studied by Canadian Customs⁵, U.S. Customs⁶, and the FBI⁷. Their work on smuggled drugs has led to increased use of the Ionscan for drug detection throughout law enforcement.

Southern California, especially San Diego, has been known as the methamphetamine capital of the world. The most common synthesis used by clandestine operators in this area is the reduction of ephedrine with hydriodic acid and red phosphorus⁸. The "cooking process" is carried out in round bottom flasks with or without condensers. The extraction and crystallization steps are completed using large tanks, buckets or any other suitable container available. This results in vapor release and liquid spillage. Therefore, in most cases, methamphetamine and ephedrine residues are present at clandestine laboratory sites. Pursuant to clandestine laboratory

investigations, there is often a need to determine the existence and location of suspected lab sites that have been abandoned or remodeled. This had been done previously by conducting vacuum sweeps of the entire suspected manufacturing area with subsequent analysis for drug residue. The process was time consuming and resulted in a large number of samples to analyze. On-site utilization of the Ionscan allows for small areas to be vacuumed and/or wiped and immediately screened for the presence of drug residue.

EXPERIMENTAL SECTION

Instrumentation

Ionscan. The Ionscan used was a Barringer model 250 with a Toshiba laptop computer. Ionscan operating conditions were: drift region length, 6.9cm; drift heater temperature, 225°C; inlet heater temperature, 293°C; desorber heater temperature, 286°C; drift and sample gas, purified ambient air; drift gas flow, 300cc/min.; sample gas flow, 200cc/min.; exhaust gas flow, 502cc/min.; scan period, 20 microseconds; calibrant, niacinamide; desorber time, 4.4 seconds.

Gas Chromatograph. The gas chromatograph (GC) used was a Hewlett-Packard 5890 Series II equipped with a flame ionization detector. It was operated in the split mode (30:1) using a 5M x 0.32mm i.d. capillary column with a 5% phenyl methyl siloxane liquid phase (0.52µm film thickness). The injector temperature was maintained at 280°C. The oven temperature was programmed as follows: initial temperature, 100°C; initial hold, 1min.; temperature program rate, 30°C/min.; final temperature, 270°C; final hold, 5min. Helium was used as the carrier gas at a column flow rate of 1ml/min.

Gas Chromatograph with an Infrared Detector. The vapor phase infrared spectra were obtained on a Hewlett-Packard 5965B Infrared Detector interfaced to a Hewlett-Packard 5890 Series II gas chromatograph (GC-IRD). The gas chromatograph was operated in the splitless mode with a 35 second purge delay then split 40:1 using a 25M x 0.32mm i.d. capillary column with 5% phenyl methyl siloxane liquid phase (0.52µm film thickness). The injector temperature was maintained at 280°C. The oven temperature was programmed as follows: initial temperature, 40°C; initial hold, 0.5min.; temperature program rate, 30°C/min.; final temperature, 270°C; final hold, 5min. Helium was the carrier gas with a flow rate of 5ml/min. The spectra were acquired at 8 wavenumber resolution.

Gas Chromatograph-Mass Spectrometer. Mass spectra were obtained on a Finnigan Incos XL mass spectrometer interfaced to a Hewlett-Packard 5890 Series II gas chromatograph (GC-MS). The gas chromatograph was operated in the split mode (50:1) using a 5M x 0.2mm i.d. capillary column with 5% phenyl methyl siloxane liquid phase (0.25µm film thickness). The injector temperature was maintained at 280°C and the oven was programmed as follows: initial

temperature, 100°C; initial hold, 1 min.; temperature program rate, 30°C/min.; final temperature, 270°C; final hold, 5 min. The ion source temperature was maintained at 180°C under electron impact conditions at 70eV. The mass scanning range was 50-500amu with a rate of 0.5 seconds per scan. Helium was the carrier gas with a column flow rate of 1ml/min.

Materials and chemicals

The cocaine hydrochloride was obtained from Merck, West Point, PA, and the methamphetamine hydrochloride was obtained from Sigma Chemical Co., St. Louis, Mo. The cocaine and methamphetamine were each dissolved in reagent grade methanol to concentrations of 100µg/ml, and 10µg/ml respectively. The teflon filters and holder cards were obtained from Barringer Instruments Inc. A Hamilton ten microliter syringe was utilized for solution delivery.

Procedures

Prior to sample analysis, the Ionscan was calibrated with 100ng cocaine, and 10ng methamphetamine. At various times during sample analysis the instrument was checked, and recalibrated as necessary. Filter cards were run on the Ionscan to ensure they were blank. The cards were then placed into the vacuum holder and a vacuum sample was obtained to ensure the ability to obtain a blank air sample in the sampling area, and the cleanliness of the apparatus. After determination that the filters and vacuum blanks were negative for the presence of drugs, analysis of suspected manufacturing areas was done. A small area was vacuumed, and the filter was run on the Ionscan. If the sample screened positive for the presence of methamphetamine or ephedrine, the area was vacuumed intensely and the filter was placed into a clean clear plastic ziplock bag and further placed into an evidence envelope. The previously obtained blank filter was also placed into a clean clear plastic ziplock bag and an evidence envelope. Both envelopes were sealed and submitted to the DEA laboratory for analysis. After a positive Ionscan run, the vacuum holder was rinsed with water and methanol. Blank samples were obtained to ensure the apparatus was not contaminated with drug residue.

The vacuum samples, and vacuum blanks, and a reagent blank were analyzed at the DEA laboratory using an acid/base extraction procedure. Sufficient acidic water was added to the clear plastic ziplock bag to soak the filter. The solution was poured into a new clean glass vial, made basic, and extracted with diethyl ether. The ether fractions were screened on the gas chromatograph, and methamphetamine and ephedrine were identified utilizing GC-IRD or GC-MS.

RESULTS AND DISCUSSION

Table 1 shows the results of the samples screened using the Ionscan. In 21 out of 25 samples taken, the substance presumptively identified by the Ionscan was confirmed in the laboratory through analysis. Table 2 lists the drift time and K_0 for the substances of interest. The

x-axis of the plasmagram is the drift time in milliseconds (ms) and the y-axis is set in arbitrary display units. Figures 1 and 2 are the plasmagrams for methamphetamine and ephedrine. Figures 3-5 show the vacuum blank and two confirmed samples. The remaining four samples could not be confirmed. For methamphetamine and ephedrine, the criteria for collecting a sample for positive identification is that the sample peak height must exceed the calibrant peak height. The closer the two are in size, the more difficult it is to positively identify the substance. This could be seen in two non-confirmed samples (figures 6 and 7). In both plasmagrams, the sample peak and the calibrant are similar in size. This suggests that the amount of substance present was too small to detect or not enough sample was collected to confirm the presence of the substance. In no case was another substance identified that had interfered or caused a false positive.

Table 2. -Substances of Interest

Peak Number	Substance	K_0	Drift Time
0	Calibrant	1.8400	9.435
1	Cocaine	1.1600	14.966
4	Ephedrine	1.5812	10.979
5	Methamphetamine	1.6441	10.559

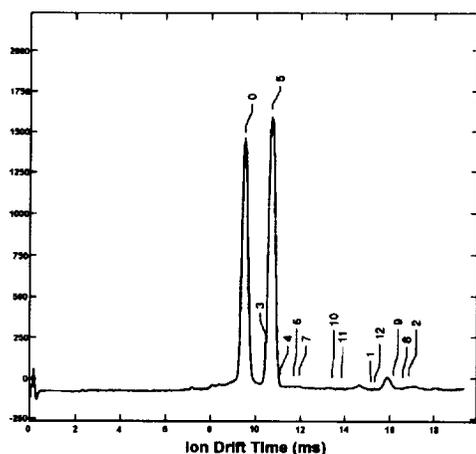


Figure 1.- Plasmagram of methamphetamine

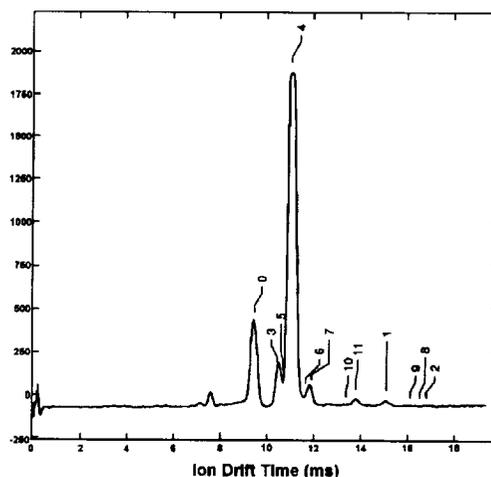


Figure 2.- Plasmagram of ephedrine

Conclusion

The results displayed in this paper show that the Ionscan is a very useful tool for screening samples taken from an abandoned clandestine laboratory. If our criteria for the size of sample peak is met, then confirmation through analysis is not a problem.

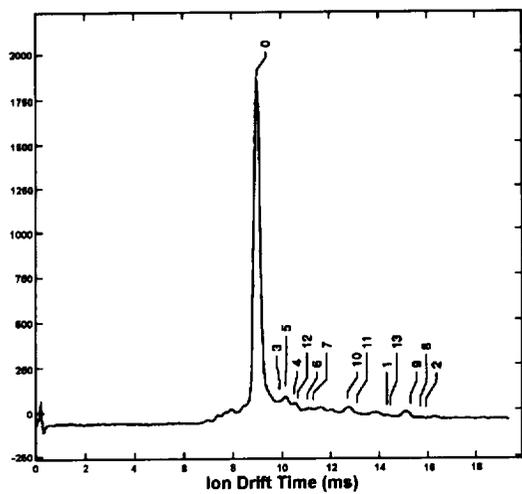


Figure 3- Plasmagram of vacuum blank.

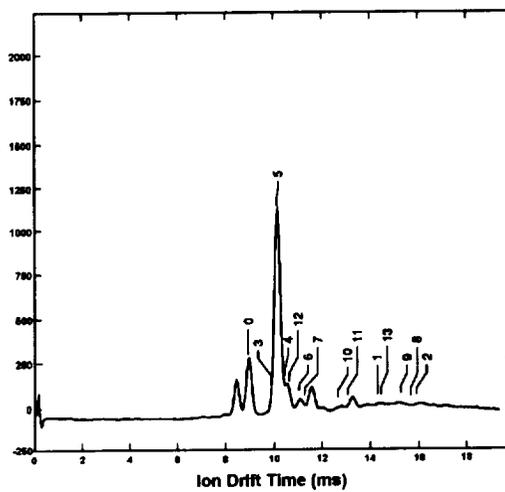


Figure 4- Plasmagram of white circle under rug (Sample 24).

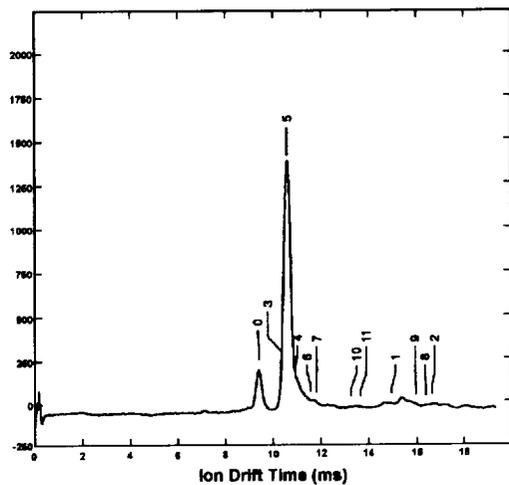


Figure 5- Plasmagram of the shirt (Sample 22).

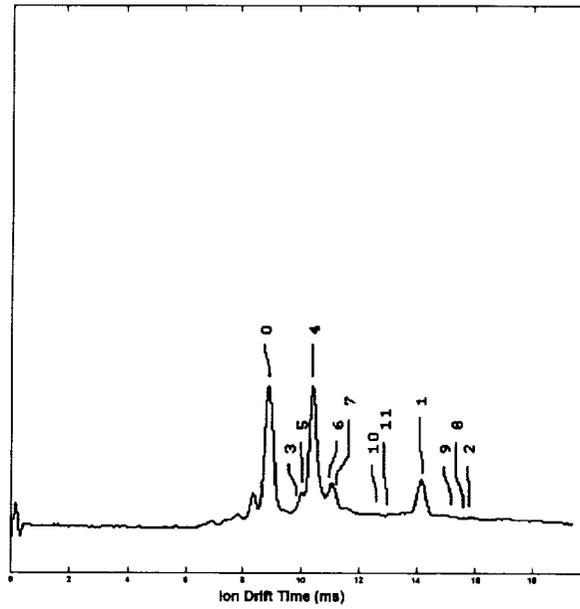


Figure 6-Plasmagram of a vacuum sample from a concrete garage floor (Sample 15).

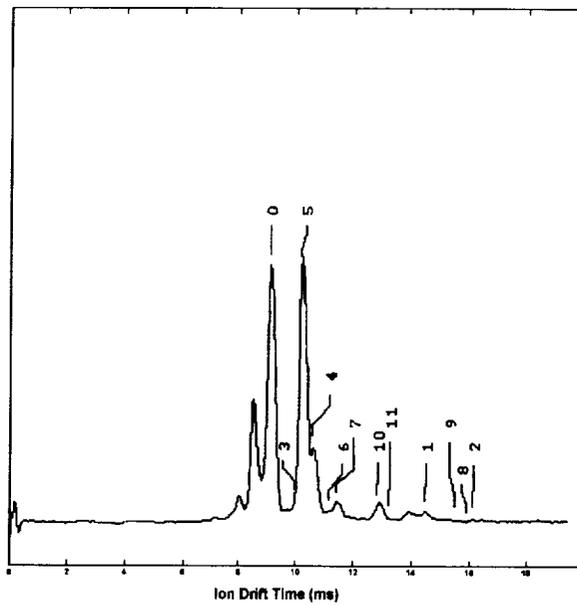


Figure 7-Plasmagram of an alcohol wipe of the register in the downstairs bathroom (Sample 14).

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Reference

1. Eiceman, G.A. and Karpas, Z. *Ion Mobility Spectrometry*. CRC Press, Boca Raton, Fl. 1994 p.2.
2. Ionscan[®] Operators Manual Barringer Instruments Limited, Rexdale, Ontario, Canada, January 1993, p.2-1.
3. Ithakissos, D.S. Plasmagram Spectra of Some Barbiturates. *J. of Chromatographic Sciences*, **18**,88-92 (1980).
4. Karasek, F.W., Hill Jr., H.H., and Kim, S.H. Plasma Chromatography of Heroin and Cocaine with Mass-Identified Mobility Spectra. *J. of Chromatography*, **117**, 327-336 (1976).
5. Harnois, J., Kovar, J., and Pilon,P. Detection of Cocaine and Heroin Using a Custom Built Ion Mobility Spectrometer. *Customs Laboratory Bulletin*, Vol. 4, No. 1, 11-31 (1992).
6. Smith, D., Hoglund, D., Hood, L., Flor, R., Erikson, C., and Trinh, T. A Field Evaluation of Drug Detection Technologies: Laboratory Confirmation of Drug Residues. Presented at Contraband and Cargo Technology International Symposium, Washington, D.C. October 28-30,1992.
7. Fetterolf, D. D., Donnelly, B., and Lasswell, L. D. Portable Analytical Instrumentation : New Weapons in the War on Drugs. Presented at Contraband and Cargo Technology International Symposium, Washington, D.C. October 28-30,1992.
8. Skinner, H.F., Methamphetamine Synthesis Via Hydriodic Acid/Red Phosphorus Reduction of Ephedrine. *Forensic Science International*, **48**, 123-134 (1990).

Table 1. Information from various Clandestine Laboratory Sites.

Sample	Date Cooked	Date Collected	Area Sampled	Type Of Sample	Presumptive Identification	Confirmed
1	11-91	11-92	Ocean Facing Window	Vacuum	Methamphetamine	Yes
2	11-91	11-92	Floor Under Window	Vacuum	Methamphetamine	Yes
3	11-91	11-92	Remaining Floor Area	Vacuum	Methamphetamine	Yes
4	1-93	8-93	Shed Floor	Vacuum	Methamphetamine	Yes
5	8-93	10-93	Wooden Work Bench	Alcohol Wipe	Methamphetamine	Yes
6	8-93	10-93	Concrete Garage Floor	Alcohol Wipe	Methamphetamine	Yes
7	8-93	10-93	Concrete Garage Floor	Alcohol Wipe	Methamphetamine	No
8	8-93	10-93	Trunk Of Car	Vacuum	Ephedrine And Methamphetamine	Yes
9	6-93	10-93	Air Return Register	Alcohol Wipe	Methamphetamine	Yes
10	6-93	10-93	Laundry Room Exhaust Fan	Alcohol Wipe	Methamphetamine	Yes
11	6-93	10-93	Bathroom Air Register	Alcohol Wipe	Methamphetamine	Yes
12	6-93	10-93	Bathroom Exhaust Fan	Alcohol Wipe	Methamphetamine	Yes
13	6-93	10-93	Air Register	Alcohol Wipe	Methamphetamine	Yes
14	6-93	10-93	Register In Downstairs Bathroom	Alcohol Wipe	Ephedrine And Methamphetamine	No
15	6-93	10-93	Concrete Garage Floor	Vacuum	Ephedrine	No
16	6-93	10-93	Counter Top	Vacuum	Ephedrine And Methamphetamine	No
17	6-93	10-93	Separatory Funnel	Vacuum	Methamphetamine	Yes
18	3-92	11-93	Garage Door Opener	Vacuum	Methamphetamine	Yes
19	3-92	11-93	Ceiling Fan	Vacuum	Methamphetamine	Yes
20	3-92	11-93	Ventilation Fan	Vacuum	Methamphetamine	Yes
21	3-92	11-93	Safe Interior	Vacuum	Methamphetamine	Yes
22	3-92	11-93	Shirt	Vacuum	Methamphetamine	Yes
23	3-92	11-93	Pvc Pipe	Vacuum	Methamphetamine	Yes
24	8-92	3-94	White Circle Under Rug	Vacuum	Methamphetamine	Yes
25	8-92	3-94	Yellow Stain On Wooden Floor	Vacuum	Ephedrine And Methamphetamine	No
						Yes