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Drug Identification by the Application of Gas Chromatography/ Time-of-Flight Mass Spectrometer Technique

The identification of illicit drugs presents many problems to the forensic laboratory because there is a broad range of possibilities which must be considered in order for a chemist to arrive at a definite identification. Obviously, this is a task that requires a constant search for new analytical techniques to meet such demands. Furthermore, these new techniques must not only facilitate the testing procedure but must provide the necessary confidence in their results as required by our court system.

In recent years, analytical methods which utilize both gas and thin-layer chromatographic techniques have found widespread use in many forensic laboratories, although there still remains an apparent lack of specificity in these methods [*1–4*]. To overcome this problem and to improve the efficiency of drug screening, a procedure was developed using a gas chromatograph/mass spectrometer (GC/MS) system. This system is rapid, accurate, and, because of the small sample required, is virtually nondestructive.

In this approach a CVC Products MA-2/015/2500 GC/MS system was used. In order to eliminate the need for a restrictive interface system and to ensure 100 percent efficiency, a direct effluent coupling between the GC/MS was achieved by the use of a differential vacuum system which allows the ion source to accept the entire effluent from the gas chromatograph. In this manner all the sample vapor flows directly into the ionization region, resulting in optimum sensitivity.

Although other papers [*5–7*] dealing with related methods of drug analysis have been published since the inception of the GC/MS in 1957, the purpose of this paper is to report the results of a study undertaken in this laboratory in which a time-of-flight mass spectrometer was used to identify very small samples of various illicit drugs available on the street level.

Apparatus

All measurements were made with a CVC Products Mass Spectrometer (Model MA-2) equipped with a (Model MA-015) differential pumping unit. A CVC Products (Model-2500) four-column, temperature programmed gas chromatograph was used for the chromatography and was directly coupled to the mass spectrometer, to allow the total

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TABLE 1—*Gas chromatograph temperatures and major mass spectra peaks used to identify common drugs.*

Drug	Column Temperature, °C	Molecular Weight (M.W.)	amu Peaks 1	2	3	4	5	6	Molecular Ion (M^+) Seen	Common Name
Alkaloids										
Cocaine	230	303	82	182	83	77	94	105	Yes	
Procaine	230	236	86	99	120	58	42	65	Yes	
Amethocaine	230	264	58	72	73	42	106	177	Yes	Tetracaine
Hallucinogens										
Methyl -3,4-methylene-dioxyphenethylamine	200	179	44	136	135	51	77	179	Yes	MDA
Mescaline	230	211	30	181	180	167	211	182	Yes	
Phencyclidine	225	243	200	91	243	84	242	186	Yes	PCP
Cannabinol	250	310	297	310	238	119	239	249	Yes	Marijuana[a]
Cannabidiol	250	314	231	246	121	313	193	173	Yes	Marijuana[a]
Tetrahydrocannabinol	250	314	314	299	231	271	243	258	Yes	Marijuana[a]
2,5-Dimethoxy-4-methylamphetamine	175	209	43	153	44	38	69	42	No	STP
Narcotics										
Mepiridine	200	247	71	70	57	247	91	103	Yes	Demerol
Methadone	230	309	72	73	42	57	56	44	Yes	
Diacetylmorphine	275	369	43	42	327	369	81	310	Yes	Heroin
Sedatives										
Barbital	200	184	156	55	141	69	98	83	No	
Glutethimide	250	217	117	132	91	189	115	51	Yes	Doriden
Butabarbital	200	212	156	41	141	57	69	157	No	
Amobarbital	200	226	156	41	141	42	55	157	Yes	Amytal
Pentobarbital	200	226	156	141	157	43	41	55	Yes	Nembutal
Phenobarbital	230	232	204	117	118	51	232	77	Yes	
Secobarbital	200	238	168	167	43	41	97	124	Yes	Seconal
Methaqualone	230	250	235	91	250	76	65	233	Yes	Sopor
Stimulants										
Amphetamine	150	135	44	91	65	45	42	39	Yes	
Methylamphetamine	150	149	58	42	91	59	56	65	Yes	
Caffeine	180	194	67	55	109	194	82	42	Yes	
Tranquilizers										
Diazepam	230	284	283	256	281	257	284	285	Yes	Valium
Chlordiazepoxide	230	299	282	281	283	274	77	220	No	Librium
Chlorpromazine	230	318	58	318	86	231	319	272	Yes	Thorazine
Flurazepam	250	386	86	87	58	99	386	183	Yes	Dalmane
Meprobamate	250	218	56	84	41	55	43	62	No	Miltown
Miscellaneous										
Phentermine	150	149	58	42	91	41	59	65	Yes	
Phenmetrazine	175	177	71	42	43	56	77	51	No	
Nicotinamide	200	122	51	78	122	106	50	52	Yes	Vitamin B_3
Methyprylon	200	183	55	83	98	140	155	69	Yes	Noludar
Promazine	230	283	58	86	283	199	198	237	Yes	
Pentazocine	230	285	217	285	70	69	110	202	Yes	Talwin
Imipramine	250	280	58	85	235	234	280	193	Yes	Tofranil

[a] Method—The marijuana vegetable matter is extracted with petroleum ether and taken to dryness, redissolved in chloroform, and chromatographed on an aluminum oxide column. Two bands appear; the first to elute is a yellow band, and is discarded; the second is a green band that contains the compounds of interest. This second mixture is collected, dried, redissolved in methanol, and injected into the GC.

GC effluent to be put into the ionizing region of that unit. Spectra were displayed on a high-frequency oscilloscope (Hewlett-Packard Model 180-A with 1801-A dual channel amplifier) and recorded on a Dixson (Model 100) direct recording oscillograph.

A 6 ft, ¼ in. OD glass column packed with 3 percent OV-17 on Chromasorb 80/100 mesh was used for the analyses. Chromatography was performed under the following conditions using the total output monitor of the mass spectrometer as the detector: column and injection port temperatures depend upon the volatility of each substance and are listed separately under each compound; carrier gas, helium with 20 ml/min average linear flow rate, 1.0–2.0 μl of samples were used.

The mass spectral data tabulated in this study were obtained using the following spectrometer operating parameters: electron ionization energy 70 eV; accelerating voltage, 2.7 kV; filament current, 2.5 A; total output monitor range 10^{-7} d-c A; total multiplier range 10^{-9} d-c A; and predynode gating set at 25 amu. The ¼-in. OD glass transfer tube connecting both units was maintained from 250–300°C. Compounds eluted from the GC were also monitored on a Perkin-Elmer recorder (Model 165) connected to the output monitor of the MS, and were used to determine the most effective time to record the mass spectral data as they passed into the detector region.

Procedure

The drugs tabulated in this paper were obtained in two forms, either as pure salts or as tablets and capsules available from commercial sources. Where necessary the pure drugs were isolated by simple extraction procedures and then dissolved in methyl alcohol to prepare standard solutions in concentrations of approximately 10 μg/μl. Because illicit samples of heroin are often diluted with lactose, trimethylsilyl derivatives of these powders were prepared in pyridine solution by reaction with hexamethyldilazane, similar to the method proposed by Grooms [8].

Results and Discussion

Table 1 presents the mass spectral data obtained for the various chemical families of commonly abused drugs. This table, in keeping with the listing of the *ASTM Index of*

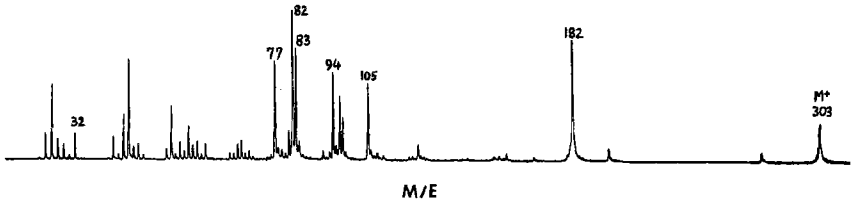

FIG. 1—*Mass spectrum of cocaine.*

FIG. 2—*Mass spectrum of methadone.*

FIG. 3—*Mass spectrum of tetrahydrocannabinol.*

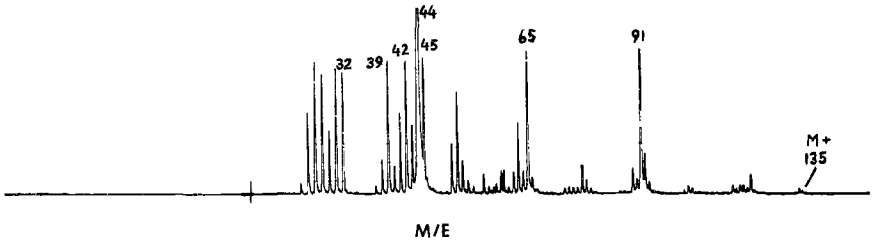

FIG. 4—*Mass spectrum of dl-amphetamine.*

Mass Spectral Data [*9*], gives both the molecular ion m/e peak values and six of the most intense fragment ion m/e peak values for each of the compounds examined.

Results of some of the GC/MS analyses are also illustrated in Figs. 1–4, which show typical scans of recorded spectral data. In all traces the m/e values increased from left to right and were run on the Dixson Recording Oscillograph at a scan rate of 1.8 in./s. Identification of unknown m/e peaks are made by overlaying a calibration chart using perfluorokerosene as a reference spectrum [*10*].

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