A STUDY TO DETERMINE THE COMMON ORIGIN OF ILLICIT HEROIN SAMPLES IN MICHIGAN USING CHEMICAL FINGERPRINTING TECHNIQUES

By

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ABSTRACT

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Presently there is no chemical data routinely supplied to police personnel which can allow them to effectively allocate personnel and resources to conduct investigations involving particular persons and areas known to be the originating source of narcotics or drugs.

An experiment was conducted using gas chromatographic techniques to establish the common origin of illicit heroin samples. Morphine and monoacetylmorphine are impurities in the manufacture of heroin and their quantitative ratio relative to that of heroin (HMM ratio) should be characteristic of each individual batch even after dilution and adulteration. This ratio was determined in 100 illicit heroin samples.

The adulterants were identified in each sample and this data was used along with the HMM

ratios to establish the common origin of the samples.

A proficiency of 90 percent was attained by this investigator during a blind trial study based soley on HMM ratio data.

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Chapter 1

INTRODUCTION

Undercover narcotic agents at the state and county level of government in Michigan are presently assigned to conduct investigations concerning violations of the Public Health Code of 1978 in various areas around the state based upon information and requests made by local or county law enforcement officials. The requests are usually generated by a sudden rise in the number of narcotic and drug seizures made by patrol personnel or by pressures applied by the local citizenry due to the sudden availability of narcotics and drugs in schools, at places of employment, or openly in public places.

For purposes of this paper, narcotics and drugs will be comprised of those chemical substances specifically listed under the Public Health Code of 1978 as amended.[1] Although numerous substances are listed under this act, a relatively small number of them are actually encountered by law enforcement personnel in

[1]Narcotics and drugs are specifically defined in Section 7105, (6) and Section 7107, (a), (b) of the State of Michigan Public Health Code Act 368 of 1978. Certain drugs were previously listed as narcotics which are presently listed as non-narcotic drugs under the new act as amended.

the field, with only a fraction of those encountered with any frequency. The ones most frequently encountered include heroin, cocaine, phencyclidine (PCP), and lysergic acid diethylamide (LSD).

Assignment of undercover narcotic agents based soley on this type of information may result in an increase in the number of arrests made for controlled substances law violations but the majority of the arrests will be for mere possession of controlled narcotics and drugs rather than for the delivery of the substances. In 1974 and 1975 respectively there were 1,395 and 1,250 arrests for sale of narcotics or drugs as compared to 11,268 and 9,378 arrests respectively for possession of narcotics or drugs (1). It is very rare for an investigation to lead to the arrest of persons who are actually the initial suppliers or manufacturers of the narcotics or drugs. In addition, this results in the confiscation of only small quantities of the substances while the supplies maintained by the initial suppliers remains unaffected. This can readily be observed in a criminalistics laboratory where the majority of evidence submitted in narcotic and drug cases consists of only one or two small packets of powdered substance or one or two tablets or capsules containing drugs. Seldom does a raid result in the confiscation of a large number of packets or a

large amount of powdered substance or tablets intended for future delivery. Therefore, it appears that the goal of the narcotic and drug investigation units, that of halting or reducing the increase in the distribution of narcotics and drugs, may be severely hampered by this means of personnel assignment. It is very possible that more effective deployment systems might be developed especially if intelligence information included distribution route patterns of the narcotics and drugs under investigation.

This study was conducted to determine the feasibility of chemically "fingerprinting" suspected heroin samples purchased or confiscated by police personnel from various areas in the state of Michigan in an effort to establish the common origin of some of the samples and possibly the trade route followed by certain batches of heroin. Heroin was chosen from among the more frequently encountered controlled substances since it is generally found in an impure form with several diluents and adulterants present which would allow the samples to be chemically individualized or "fingerprinted". Heroin is also a narcotic of great concern to everyone. This concern can be partially realized by the increased penalties provided in the Public Health Code of 1978 as amended which provides stiff penalties for the delivery or

possession of heroin.[2] The data compiled during the chemical fingerprinting of the heroin samples and identification of routes the sample followed on its way to the user will supply necessary intelligence information which may possibly be used to more effectively assign narcotic agents to those areas where the supplies of heroin are originating.

The chemical fingerprinting of narcotic or drug exhibits has not been done by the Federal Drug Enforcement Administration on a routine basis but only upon special request in certain cases which usually involve the alleged shipment of narcotics or drugs across state borders. At the present there is no program of this nature being conducted in the state of Michigan.

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[2]Under Section 7402,(2),(a),(i) and Section 7403, (2),(a),(i) of the Public Health Code of 1978 a person convicted of delivering or possessing 650 grams or more of any mixture containing any amount of heroin shall be imprisoned for life. Additional stiff penalties are provided for delivering or possessing lesser specified amounts of the mixture.

Chapter 2

REVIEW OF THE LITERATURE

The clandestine synthesis of heroin originates with raw opium which is the dried latex from the unripe seed capsules of the opium poppy, Papaver somniferum. There has been considerable work done on determining the amounts of the various natural alkaloids present in opium with 18 different ones being isolated (2). Only three of these alkaloids have been found to consistently comprise one percent or more of the alkaloid content by weight of the opium. They are morphine (3-20%), narcotine (1-12%), and codeine (0.7-2.5%). The concentrated aqueous opium extract is treated with strong calcium chloride solution, and calcium lactate, sulfate and meconate are precipitated and removed. From the mother liquor a mixture of morphine and codeine hydrochlorides crystallize. This mixture, which is predominantly morphine, is reacted with acetic acid or acetic anhydride to produce the desired diacetylmorphine (heroin) with various amounts of O^6- and O^3-monoacetylmorphine, acetylcodeine, and unreacted morphine and codeine as impurities of manufacture. A flow chart of the synthesis of heroin

from raw opium is presented in Figure 1. Depending, therefore, on the relative amounts of morphine and codeine which crystallized out of the mother liquor and on the efficiency of the acetylation process each separate batch of heroin manufactured will have a certain characteristic relative content ratio of the heroin and the impurities of manufacture.

Illicit heroin samples encountered at the street level are also frequently adulterated with certain chemicals or drugs which can be easily and legally obtained. They are added in some cases to mask the presence of a small amount of heroin thus increasing the profit. Others are added to enhance the physiological affects of the heroin. The more common adulterants include caffeine, procaine (novocaine), methapyrilene, and quinine. Sugars such as lactose and mannitol as well as starch are commonly added as diluents. The presence or absence as well as the relative amounts of these adulterants and diluents in illicit heroin samples can also be used to describe the chemical fingerprint of a particular sample.

The establishment of illicit narcotic or drug sources has been primarily the responsibility of the Drug Enforcement Administration (DEA) established within the Federal Justice Department. Chemists in the Special Testing and Research Laboratory have

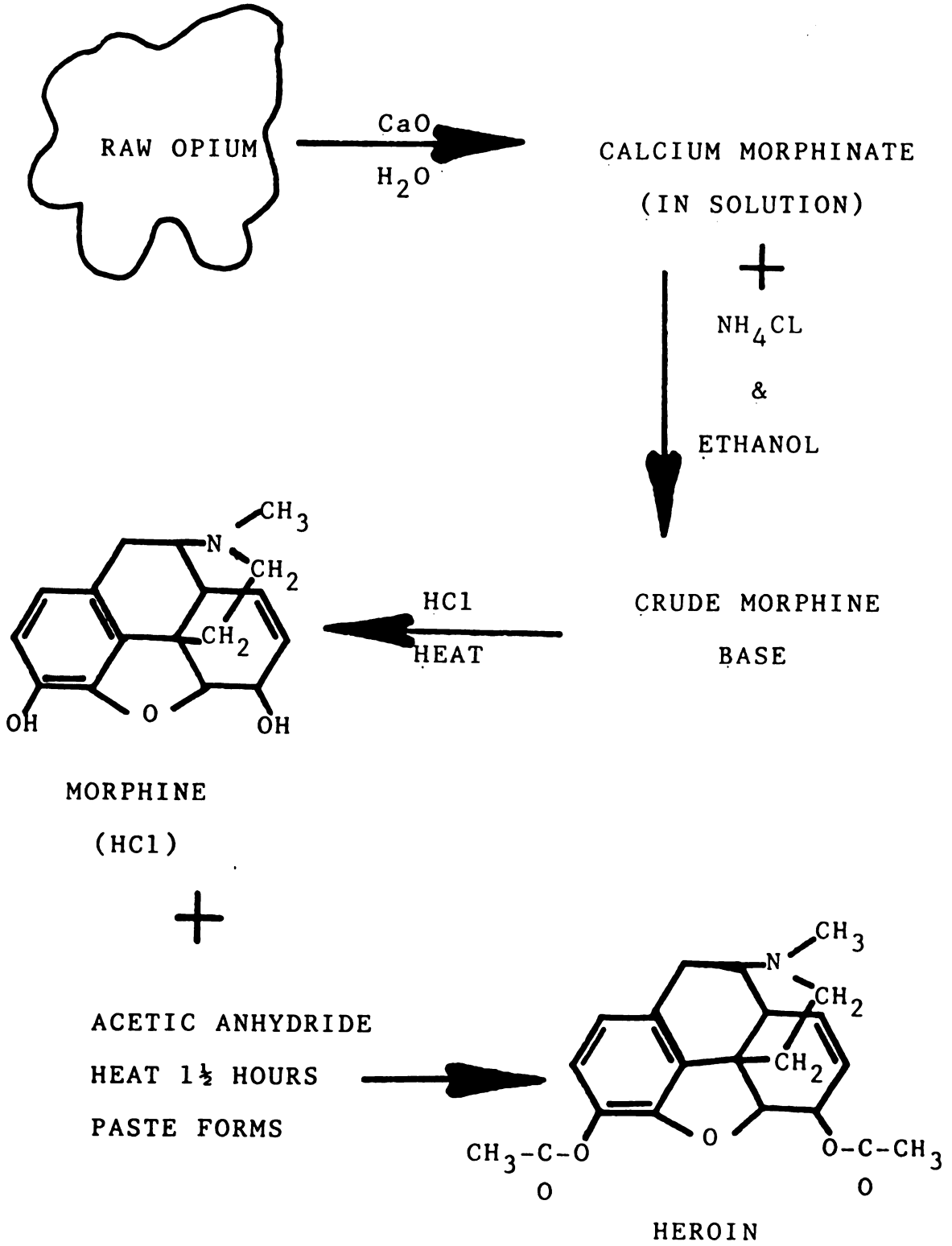


Figure 1.--Flow diagram for the synthesis of brown heroin from raw opium.

investigated direct and derivative gas chromatographic methods for chemically fingerprinting drug exhibits (3). The qualitative and quantitative identification of as many different chemical constituents as possible in each sample is the goal in the fingerprinting of narcotic or drug samples (4).

Additional information about each individual sample can be obtained by a microscopic examination. Each crystalline adulterant or diluent has its own set of specific optical properties which serve to identify the compound. These properties include refractive indicies, extinction angle, axial angle, sign of elongation, and optic sign (5). Eisenberg and Tillson (6) utilized this procedure to identify crystalline substances present in counterfeit barbiturate and amphetamine tablets and capsules by mounting small portions of the substances present in a suitable series of calibrated refractive index oils and examining them under a polarizing microscope. Tables and files of optical data were then consulted to identify the crystalline substances.

Table 1 shows the quantitative results obtained by chemists in the DEA laboratory when they examined six heroin cases from Texas (T-prefix on laboratory number) and two from Michigan (M-prefix on laboratory number), (7). It can be seen that samples numbered

T-1775, T-1776, and M-1832 are very similar with regard to the percent of heroin present and also the absence of lactose which is one of the more common diluents.

Table 2 shows the quantitative data obtained by the DEA chemists in terms of the ratios of the adulterants of the same eight exhibits as given in Table 1, (7). Even though many of the exhibits had been adulterated, a constant relationship of the by-products was found with the exception of the last sample, M-1833.

X-ray fluorescence analysis was also utilized in the above case to provide information on the elemental composition of each sample. The elements that were detected and measured were potassium, calcium, and iron. Table 3 shows the results obtained on the eight samples after the x-ray fluorescence examination (7). It can be seen from Table 3 that each of the samples which had no lactose present had similar elemental compositions.

The microscopic appearances of the three heroin samples which did not contain lactose were identical. The other five samples revealed the presence of poorly crystallized lactose monohydrate. Based upon the results of the four examinations described above, the DEA chemists concluded that the heroin in one of the

Table 1

Heroin Composition and Presence or Absence of Lactose in Eight Illicit Heroin Exhibits from Texas and Michigan

Lab Number	Percent Heroin HCl	Lactose
T1771[a]	30.2	+
T1772	32.6	+
T1774	31.4	+
T1775	94.9	-
T1776	93.0	-
T1777	36.0	+
M1832[b]	95.1	-
M1833	19.3	+

[a]The "T" prefix denotes an exhibit from Texas.

[b]The "M" prefix denotes an exhibit from Michigan.

Table 2

Ratio of By-products in Eight Illicit Heroin Exhibits from Texas and Michigan.

Lab No.	Morphine	Monoacetyl morphine	Codeine	Acetyl codeine	Heroin
T1771	-	1.70	0.13	2.69	95.5
T1772	-	1.81	0.09	2.85	95.2
T1774	-	1.89	-	2.88	94.7
T1775	0.03	1.90	0.06	2.97	95.0
T1776	-	1.95	0.12	3.25	94.7
T1777	-	1.80	-	3.01	95.2
M1832	0.03	1.92	0.10	3.13	94.8
M1833	0.39	2.40	0.30	3.35	93.2

Table 3

Elemental Composition of Eight Illicit Heroin Exhibits from Texas and Michigan.

Lab Number	Potassium	Calcium	Iron
T1771	+	5700[a]	+
T1772	+	6000	+
T1774	+	5300	+
T1775	-	9950	+
T1776	-	10050	+
T1777	+	6200	+
M1832	-	9600	+
M1833	+	4000	+

[a]Counts per minute.

exhibits (M-1832) in the Michigan case did correspond with those found in Texas. Further, it was concluded that all exhibits in the Texas case had a common origin and that two sources of heroin existed in the Michigan case (7).

In a similar study, Van der Slooten and Van der Helm (8) of the University of Amsterdam, determined the quantitative composition of thirty-two illicit heroin samples and found that is was possible to draw conclusions about the common origin or trade route of the different samples. These researchers expected that the relative amounts of impurities of manufacture of the heroin which included monoacetylmorphine and morphine would not change as the heroin was diluted and adulterated by successive dealers. They felt that a quantitative determination of these impurities of manufacture together with a determination of adulterants, may provide information about illegal drug traffic.

The ratio, heroin:monoacetylmorphine:morphine (HMM) of thirty-two illicit heroin samples, together with a number of common adulterants, was determined by gas chromatography for the purpose of tracing: (a) heroin samples with the same HMM ratio, in order to get information about a possible common source, and (b) heroin samples with the same composition as far

as adulterants were concerned, in order to find out whether they had passed through the same chain of dealers.

Three different gas chromatography columns were used along with four different internal standards for quantitation purposes. The results of the analysis are summarized in Table 4, (8).

The data in Table 4 was used to represent the composition of the samples as shown in part by Figure 2. In Figure 2 some groups can clearly be distinguished: samples 509, 522, and 648 (group I), show a very similar composition, similarly 476 and 590 (group II), 591 and 683 (group III), 856, 857, and 860 (group IV), and 757 and 795 (group V). By representing the composition of the samples as in Figure 2, addition of other substances has no influence on the "heroin pattern". So it can be seen that samples 591 and 683 have the same batch of heroin as a basis, but to 591 an amount of caffeine has been added (see Table 4).

The data in Table 4 was also used to compare samples such as number 1731 and number 1732 as in Figure 3. From Figure 3 it can be seen that the samples are derived from different batches of heroin. Yet, they are likely to have passed to the same person or persons, because of the striking similarity in amphetamine, caffeine and cocaine content. The route,

Table 4

Composition of 32 Illicit Heroin Samples, Percentage by Weight, Sample = 100.

SAMPLE NUMBER	HER	MONO MOR	MOR	CAFF	AMPH	EPHE	STRY
458	4.8	55.0	4.0	19.0			
476	9.7	59.0	3.7	26.0			
509	37.0	22.0	1.7	27.0			
522	44.0	25.0	1.7	26.0			
526	2.0	1.6	-----	1.0			
542	43.0	26.0	2.2	22.0			
557	55.0	24.0	2.5	13.0			
590	10.0	56.0	3.0	24.0			
591	27.0	15.0	0.4	21.0			
648	42.0	22.0	1.7	24.0			
674	24.0	13.0	2.3	----			
683	59.0	32.0	0.8	----			
757	32.0	24.0	1.9	30.0			
795	39.0	28.0	2.2	36.0			
800	20.0	52.0	3.7	12.0			
820	19.0	50.0	4.6	25.0			
821	18.0	51.0	4.1	25.0			
856	14.0	40.0	4.4	32.0			
857	16.0	47.0	5.0	30.0			
858	63.0	21.0	2.9	----			
859	72.0	23.0	3.1	----			
860	13.0	38.0	4.3	27.0			
868	6.4	59.0	4.8	24.0			
941	0.05	1.1	2.8	----	----	0.67	----
950	17.0	36.0	0.68	14.0	0.73	----	----
953	0.08	9.0	2.3	86.0	----	----	----
954	-----	----	22.0	5.8	0.27	----	27.0
1731	36.0	12.0	0.57	7.7	0.25	----	7.1
1732	32.0	32.0	2.1	9.0	0.24	----	----
2731	86.0	9.0	0.78	----	----	----	----
4041	54.0	37.0	1.3	6.3	----	----	----
4042	44.0	20.0	0.72	12.0	----	----	1.6

HER = Heroin, MONOMOR = Monoacetylmorphine, MOR = Morphine, CAFF = Caffeine, AMPH = Amphetamine, EPHE = Ephedrine, and STRY = strychnine.

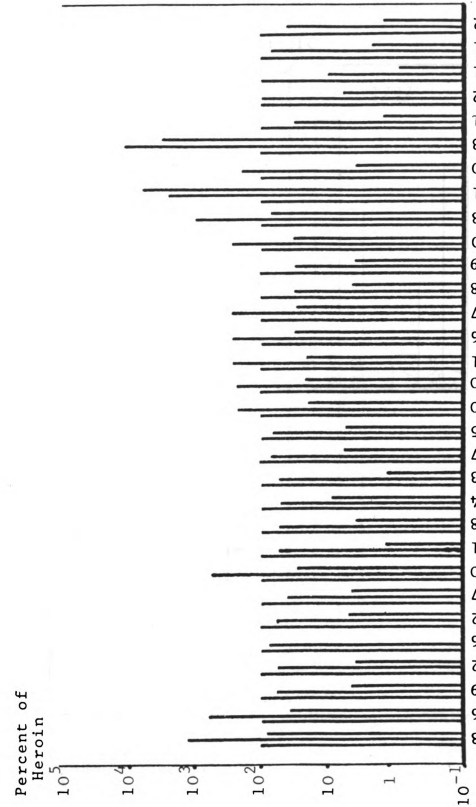


Figure 2.--Composition of 31 illicit heroin samples.

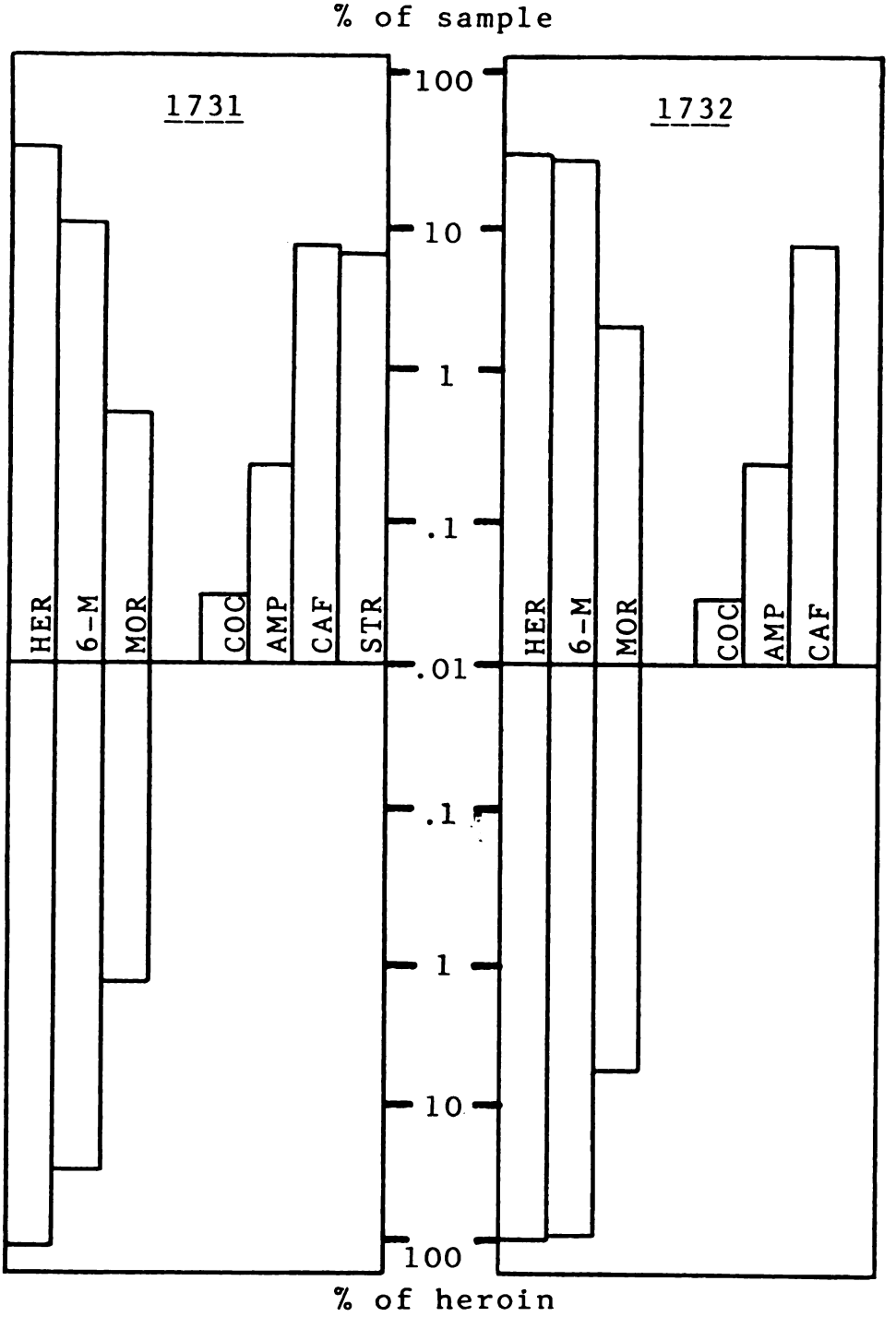


Figure 3.--Comparison of the composition of two heroin samples, represented as the percentage of the heroin content (downwards) and as the percentage of the sample (upwards). HER = heroin, MOR = morphine, COC = cocaine, CAF = caffeine, STR = strychnine, 6-M = monoacetylmorphine, and AMP = amphetamine.

however, was not exactly the same; somewhere sample 1731 acquired an ample amount of strychnine.

Van der Slooten and Van der Helm (8) summarized that one may conclude that by means of rather uncomplicated methods of analysis, sufficient information can be gathered to indicate the common origin of different heroin samples. Also, in certain cases it seems to be possible to draw conclusions about the route the sample followed on its way to the user.

Chapter 3

RESEARCH DESIGN AND PROCEDURES

The background study and subsequent research was conducted at the Michigan State Police Bridgeport Regional Crime Laboratory. All equipment and materials were the property of this laboratory. It was conducted under the authority granted by the Controlled Substance Registration Permit number 01217 issued by the State of Michigan Department of Licensing and Registration, the Controlled Substances Registration Certificate number PB0129963 issued by the Drug Enforcement Administration of the United States Department of Justice and by Section 7303,(2) of the Public Health Code of 1978 which deals with conducting research with controlled substances.

For clarity, this chapter is separated into five divisions under the headings of (1) materials, (2) sample selection, (3) experiment 1: total sample assays, (4) experiment 2: heroin quantitation, and (5) experiment 3: heroin:monoacetylmorphine:morphine ratio determination.

MATERIALS

Solvents and reagents. Triacontane (Lot 3538) and tetracosane (Lot 1261) internal standards were obtained from Applied Science Laboratories, State College, Penna. N,O-Bis-(trimethylsilyl)-trifluoro-acetamide (BSTFA) silylating reagent with 1% trimethylchlorosilane (TMCS) was obtained from Pierce Chemical Company, Rockford, Illinois. The chloroform, 1,1,1-trichloroethane, acetic acid, hydrochloric acid, and potassium phosphate, dibasic used in this experiment were all ACS reagent grade chemicals.

Glassware used in this experiment consisted of disposable pipets, test tubes, and microscopic slides. Twelve, one milliliter size reaction vials were used to make the silyl ether derivatives and were obtained from Pierce Chemical Company. A new disposable 12 millimeter diameter teflon coated disc was used in the vial caps during each derivatization procedure.

Standard alkaloids and drugs. All of the drugs as well as the morphine and codeine alkaloids were the property of the Michigan State Police Bridgeport Regional Crime Laboratory and are normally used as standards during the normal course of business. Their purity was verified by infrared spectrophotometry and by gas chromatographic analysis. The acetylcodeine and monoacetylmorphine standards were prepared by

acetylation of portions of the codeine and morphine standards with acetic anhydride. The purity of these two standards was also verified by infrared spectrophotometry and gas chromatography.

Instrumentation. All gas chromatographic analyses were conducted on a Varian Aerograph Model 2100 gas chromatograph utilizing a flame ionization detector and a dual differential electrometer. Recording was done on a Varian Aerograph Model A-25 strip chart recorder. A Varian Aerograph Model 485 electronic digital integrator was used to integrate the area under the gas chromatographic peaks.

A Sartorius Model 2492 analytical balance with a ±0.05 milligram precision was used for all weighings.

A Perkin-Elmer Model 457 infrared spectrophotometer was used to analyze the drug and alkaloid standards using the potassium bromide pellet technique.

Miscellaneous equipment. Hamilton ten microliter syringes were used to introduce the samples into the gas chromatograph. A Vortex Genie mixer Model 58223 from Scientific Products, Bohemia, N.Y., was used to agitate the liquid solutions in test tubes. A Clay-Adams Safety-Head centrifuge was used to centrifuge all samples. A Wig-L-Bug dental amalgamator was used to homogenize all heroin samples.

GC Column. A ¼" O. D. U-shaped glass column four feet in length was silanized using the procedure described by Applied Science Laboratories, Inc. (9) and then packed with 3% OV-101 on 120-140 Mesh Gas Chrom Q. The packing was obtained pre-coated and tested from Applied Science Laboratories (Lot SP-1895).

SAMPLE SELECTION

The samples analyzed consisted of 100 individual illicit heroin samples which at various times were submitted to the Michigan State Police Bridgeport Regional Crime Laboratory for analysis involving actual case investigations. The submissions were subsequent to purchases made by undercover narcotics agents and/or confiscations made incident to arrests or execution of search warrants. The cases from which the samples were taken are now closed so that the samples are no longer of any evidentiary value.

Approximately 150 mg aliquotes were taken from exhibits involving cases where they were purchased or seized between the dates of September 21, 1974 and June 8, 1978. They were all submitted by various police agencies within the geographical area served by the Bridgeport Regional Crime Laboratory. Each individual sample was assigned a random five digit number for reference purposes. The samples cannot

be associated with their previous laboratory number from the information provided in this study.

Since it would be impossible to ascertain that two or more of the illicit heroin samples studied actually came from the same original source, the sampling method was selected to insure that two main categories would be present. The first category would consist of those samples which were submitted as individual purchases or confiscations where there was apparently no connection between them. The second category consisted of samples where two or more exhibits were submitted as purchases or confiscations which came about as the result of the investigation of one particular case or suspect. In this second category one may reasonably expect some of the samples to have come from the same original source since some of them were the result of multiple purchases or confiscations made at the same time from the same person or place. In addition, there were some investigations where possibly a double purchase was made on a certain date and a single purchase made several weeks later. This would involve the same case but one might reasonably expect this second purchase to have originated from a different batch of heroin as did the first two. Table 5 lists the illicit heroin samples by their assigned random number, groups

Table 5

One Hundred Illicit Heroin Samples Analyzed.

Case No.	Random No.	Date Obtained	Location
1	22368	9-21-74	Bay City
2	86591	8-21-75	Saginaw
3	42167	11-04-75	Bay City
4	96301	11-05-75	Bay City
5	03237	11-10-75	Saginaw
6	28918	12-23-75	Bay City
7	07119	1-27-76	Bay City
8	51085	2-05-76	Saginaw
9	07056	4-06-76	Buena Vista
10	48663	6-08-76	Saginaw
11	91921	12-20-76	Saginaw
12	00582	12-27-76	Saginaw
13	69011	1-13-77	Saginaw
14	25976	1-18-77	Saginaw
15	09763	1-18-77	Saginaw
16	91567	2-16-77	Saginaw
17	14577	5-19-77	Freeland
18	98427	6-28-77	Saginaw
19	92608	7-25-77	Saginaw
20	30405	10-25-77	Lapeer
21	38935	11-02-77	Saginaw
22	16631	11-07-77	Flint

Table 5 - Continued

Case No.	Random No.	Date Obtained	Location
23	44657	3-22-78	Saginaw
24	96773	3-31-78	Saginaw
25	50001	4-03-78	Saginaw
26	27504	4-12-78	Saginaw
27	37169	4-14-78	Saginaw
28	11508	4-14-78	Saginaw
29	37449	4-14-78	Bay City
30	60336	4-20-78	Saginaw
31	03299	4-26-78	Saginaw
32	18039	5-11-78	Saginaw
33	79556	5-16-78	Saginaw
34	38534	6-08-78	Bridgeport
35	10480	6-22-78	Saginaw
36	57491	6-26-78	Saginaw
37	59037 42488 46764	9-05-75	Saginaw
38	37570 99562	11-04-75	Saginaw
39	77921 01011	11-04-75 2-25-76	Bay City

Table 5 - Continued

Case No.	Random No.	Date Obtained	Location
40	85475	12-22-75	Mt. Morris
	63553	1-06-76	
	10365	1-15-76	
41	09429	1-06-76	Bay City
	00725	12-30-76	
42	02368	2-13-76	Saginaw
	52162	2-25-76	
	08362	2-25-76	
43	23982	6-29-76	Buena Vista
	09915		
44	54164	10-06-76	Saginaw
	32639		
45	29334	10-08-76	Saginaw
	02488		
46	00742	12-15-76	Saginaw
	05366		
47	17955	3-15-77	Saginaw
	46503		

Table 5 - Continued

Case No.	Random No.	Date Obtained	Location
48	34914 70060	7-14-77	Saginaw
49	53976 76072	8-11-77	Saginaw
50	90725 08962 73115	8-15-77	Saginaw
51	64364 95012 15664 16408 18629	8-15-77	Saginaw
52	31624 78919	2-18-78	Saginaw
53	03931 74426 09066 42238 16153	3-14-78	Saginaw

Table 5 - Continued

Case No.	Random No.	Date Obtained	Location
53	21457 21581 55612	3-14-78	Saginaw
54	91340 91227 92157	3-28-78 3-28-78 4-12-78	Saginaw
55	65390 24130 21885 89579	4-12-78 4-12-78 4-18-78 4-18-78	Saginaw
56	82486 81525 97656 29676	4-18-78 4-18-78 4-25-78 4-25-78	Saginaw
57	46515 30986 63798 43937	4-21-78	Saginaw
58	79626 85636	4-27-78	Saginaw

them as to case investigations, and indicates the date purchased or confiscated and geographical area where the sample came from. As can be seen from Table 5, 36 samples are of the type described in the first category and 55 are of the type described in the second category with the remaining 9 samples being individual purchases or confiscations after a time period has passed after the original multiple purchase or confiscation. The 64 samples in the second category are divided into 22 separate case investigations.

TOTAL SAMPLE ASSAY

Experiment 1

Initially the presence or absence of lactose was determined microscopically for each sample using a polarizing microscope with the sample mixed with glacial acetic acid on a microscope slide.

Some samples were in a powdered form and others were hard crystalline masses. Some also appeared as a mixture of white crystalline powder and dark brown colored crystalline material. To ensure the homogeneity of the samples for chemical testing each one was pulverized on the dental amalgamator in disposable plastic containers with disposable plastic agitator balls for a period of one minute. Blank runs were made using spectral grade potassium bromide and the

plastic balls for three minute agitation periods. No substances were detected due to the agitation in the plastic vials.

A portion of each sample was weighed into individual test tubes to which 1.0 ml of 1.0 mg/ml triacontane in chloroform solution was added. Each sample was agitated on the vortex mixer for one minute after which each sample was injected into the gas chromatograph. The analysis was conducted in the temperature programmed mode from 150°C to 260°C at the rate of 10°C/min on the 3% OV-101 column, 30 ml/min nitrogen carrier gas flow rate, 32 x 10^{-11} amps/mv sensitivity attenuation, with a 1 cm/min strip chart speed.

Standard solutions of caffeine, procaine, methapyrilene, cocaine, methadon, papaverine, phenacetin, strychnine, and quinine were also run under the same gas chromatographic parameters as the heroin samples. The adulterants were identified and quantitated.

HEROIN QUANTITATION

Experiment 2

Additional aliquots of each sample were weighed out individually into test tubes. Two milliliters of 0.1 N hydrochloric acid was added to each sample to dissolve it. Two milliliters of 1,1,1-trichloroethane were added to each solution followed by agitation on

the vortex mixer for one minute and then centrifuged. The 1,1,1-trichloroethane was used to remove the adulterants which could interfere in the derivatization procedure described in experiment 3. The acid layer was drawn off each sample and transferred to separate test tubes. One milliliter of 1.0 N potassium phosphate solution was added to each sample to make it basic and then 1.0 ml of a standard solution containing 1.0 mg and 5.0 mg of tetracosane and triacontane respectively per milliliter of chloroform was pipetted into each sample. Each sample was agitated for one minute on the vortex mixer and then centrifuged. Approximately 0.6 ml of the organic layer was drawn out of each sample tube and transferred to separate reaction vials for derivatization. The remaining organic extract was analyzed on the gas chromatograph isothermally at 230°C, with 30 ml/min nitrogen carrier gas flow rate, 32×10^{-11} amps/mv sensitivity attenuation, on the 3% OV-101 column, also at a strip chart speed of 1 cm/min.

A standard solution of heroin alkaloid was prepared and extracted as above and analyzed on the gas chromatograph under the same parameters as the heroin samples. The standard sample was injected 20 times to allow for a statistical analysis of the heroin quantitation.

The heroin was quantitated in each of the 100 samples.

HEROIN:MONOACETYLMORPHINE:MORPHINE RATIO DETERMINATION

Experiment 3

0.2 milliliter of O-Bis-(trimethylsilyl)trifluoroacetamide (BSTFA) was added to each organic aliquote in the reaction vials. The resulting mixtures were heated at 75°C for one hour. The derivatized samples were then analyzed on the gas chromatograph isothermally at 230°C on the 3% OV-101 column, with 30 ml/min nitrogen carrier gas flow rate, 16 x 10^{-11} amps/mv sensitivity attenuation, with a strip chart speed of 1 cm/min. The heroin:monoacetylmorphine:morphine quantitation ratio was determined for each of the 100 samples.

Reproducability of the results was calculated by examining two separate samples from each of which ten aliquotes were weighed out into test tubes and the same procedure was performed on each of the 20 samples as described above. Identical gas chromatographic parameters were used to examine the 20 trial samples as was used to analyze the 100 heroin samples.

Chapter 4

ANALYSIS OF THE DATA

CALCULATIONS

All calculations in these experiments were performed by hand using a Hewlett-Packard HP-35 portable calculator. The quantitation calculations for the heroin as well as for the adulterants were made using the following general formula:

$$\frac{\frac{\text{Area Standard Peak}}{\text{Area Internal Std. Peak}}}{\text{Number of mg/ml Std.}} = \frac{\frac{\text{Area Component Peak}}{\text{Area Internal Std. Peak}}}{\text{X mg/ml Component}}$$

Division by the total sample weight analyzed multiplied by 100 gives the percent by weight of the component in the sample.

The heroin:monoacetylmorphine:morphine (HMM) ratios were calculated by adding the peak areas of those three components in each sample as determined by the integrator and then dividing the peak area of each individual peak by that sum. The three results provide the HMM ratios.

The values calculated for the bar graphs were

made by calculating the percentage of the heroin peak the monoacetylmorphine and the morphine peak areas represent for each sample analyzed.

RELIABILITY CRITERIA OF THE ASSAYS

Linearity Study

The quantitative analysis of heroin samples in the Michigan State Police Crime Laboratory is conducted routinely as well as the quantitation of the adulterants encountered in toxicological cases. Linearity studies have been conducted previously on these substances and the quantitative results have been found to be linear in the concentrations encountered during this research on the instrument and column type used.

Recovery and Precision

By utilizing a single solution containing internal standards for all dilutions in these experiments any difference in the extraction efficiencies will be corrected for since all calculations were made relative to the area of the internal standard peak areas.

The data generated by repetitive analysis of a single sample and the repetitive analysis of two samples weighed out and extracted ten times each are presented in Table 6. The coefficient of deviation for the 20 repetitive analyses of the same sample for the heroin quantitation was 0.44% and averaged 1.44% for the

Table 6

Repetitive Analyses

Twenty Repetitive Analyses of a Single Heroin Sample.

Component	Range	Standard Deviation	Coefficient[a] of Deviation
Heroin	0.76	0.21	0.44
Monoacetyl-morphine	0.47	0.11	0.94
Morphine	0.92	0.29	4.41

Ten Separate Repetitive Analyses of Heroin Sample A.

Component	Range	Standard Deviation	Coefficient[a] of Deviation
Heroin	4.84	1.67	2.45
Monoacetyl-morphine	1.19	0.64	4.16
Morphine	3.94	1.24	7.52

Ten Separate Repetitive Analyses of Heroin Sample B.

Component	Range	Standard Deviation	Coefficient[a] of Deviation
Heroin	0.52	0.20	0.42
Monoacetyl-morphine	1.17	0.46	0.94
Morphine	1.10	0.41	10.43

[a]Coefficient of deviation in percent.

repetitive analysis of the 20 samples produced from weighed aliquots of two original samples.

EXPERIMENT 1--TOTAL SAMPLE ASSAY

The microscopic examination performed to determine the presence of lactose or starch showed that none of the samples contained starch as a diluent. Further, it was found that the presence of lactose could be described in four ways: (1) well crystallized (WC), (2) poorly crystallized (PC), (3) not present but sample soluble in glacial acetic acid (SOL), and (4) not present and sample not soluble in glacial acetic acid (NSOL).

Only three different adulterants were found in the samples analyzed. Procaine was found in 43 of the samples, caffeine in 2 of the samples, and methapyrilene in 1 of the samples. Codeine and acetylcodeine were also found as impurities of manufacture in 33 of the samples. In all cases the quantity of codeine or acetylcodeine was less than 2½%. The results of experiment 1 are presented in Table 7.

EXPERIMENT 2--HEROIN QUANTITATION

The percentage of heroin was determined for each of the 100 samples. The percentages ranged from 1.37% to 32.96% with the mean value of 5.54%. The heroin

Table 7

Total Sample Assay and Heroin Quantitation Data - Percent by Weight.

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
1	22368	PC[a]	3.13	27.90				
2	86591	PC	1.71					
3	42167	PC	10.76					
4	96301	PC	11.52					
5	03237	PC	32.96		0.30	0.34		
6	28918	PC	6.10	53.75				
7	07119	WC[b]	4.60					
8	51085	WC	17.04		0.34			
9	07056	PC	5.86					
10	48663	SOL[c]	11.92	84.01	0.77			
11	91921	PC	7.23					
12	00582	PC	11.29					
13	69011	PC	6.28	10.22				

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
14	25976	WC	6.15		0.42			
15	09763	WC	7.20		0.75			
16	91567	PC	10.49					
17	14577	PC	9.89					
18	98427	PC	6.70	79.43				
19	92608	SOL	6.57	88.53		2.01		
20	30405	PC	3.25	46.23				
21	38935	PC	13.43					
22	16631	WC	2.21	3.43				
23	44657	NSOL[d]	6.47	45.08	0.46			
24	96773	PC	9.43					
25	50001	SOL	7.19	89.56	0.90			
26	27504	PC	4.03					
27	37169	NSOL	4.91	69.70	0.54			

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
28	11508	PC	3.40					
29	37449	PC	4.26					
30	60336	PC	3.76					
31	03299	PC	1.37	47.67	0.69		22.23	10.5
32	18039	PC	7.69	76.81	1.49			
33	79556	PC	7.63	76.85	0.59			
34	38534	PC	7.54	75.91	0.76			
35	10480	WC	5.40					
36	57491	PC	8.93					
	59037	SOL	6.97	83.85	0.58			
37	42488	SOL	15.81	77.58				
	46764	SOL	7.09	83.86	1.06			

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
38	37570	SOL	11.55	83.98				
	99562	SOL	13.06	82.05				
39	77921	PC	9.27					
	01011	PC	3.48					
40	85475	PC	14.61					
	63553	PC	8.08					
	10365	PC	10.56					
41	09429	WC	3.36	40.60	1.13			
	00725	WC	8.21	31.35				
42	08362	PC	2.90				7.70	
	52162	PC	2.55					

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
42	02368	PC	7.91					
43	23982	NSOL	26.70			2.11		
	09915	NSOL	25.21			2.31		
44	54164	PC	5.64					
	32639	PC	3.87					
45	29334	PC	6.07					
	02488	PC	5.68					
46	00742	PC	5.60					
	05366	PC	5.61					
47	17955	PC	4.91					
	46503	PC	4.03		1.07			

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
48	34914	PC	3.15					
	70060	PC	2.34					
49	53976	PC	4.21	87.85				
	76072	PC	4.70	87.80				
50	90725	SOL	7.07	87.03	2.20			
	08962	SOL	5.55	88.90	1.85			
	73115	SOL	6.36	89.38	1.67			
51	64364	PC	4.75	85.04				
	95012	PC	5.15	88.07				
	15664	PC	5.11	89.01				
	16408	PC	4.45	88.53				
	18629	PC	5.32	88.41				

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
52	31624	WC	9.18					
	78919	WC	4.62					
53	03931	PC	6.45	84.30				
	74426	PC	5.62	85.11	0.24			
	09066	PC	5.62	84.95				
	42238	PC	6.23	85.10	0.69			
	16153	PC	2.26		0.58			
	21457	SOL	6.87	89.85	0.88			
	21581	PC	5.55	84.08				
	55612	PC	6.09	83.91	0.56			
54	91340	PC	4.06					
	91227	PC	11.55		0.57			
	92157	PC	4.45					

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
55	65390	SOL	6.92	89.51	1.00			
	24130	SOL	6.11	90.02	0.49			
	21885	SOL	7.95	88.91	0.61			
	89579	SOL	5.35	80.36	0.52			
56	82486	PC	5.73					
	81525	PC	6.47					
	97656	PC	7.14					
	29676	PC	6.62					
57	46515	PC	2.82					
	30986	PC	3.15					
	63798	PC	3.60					
	43937	PC	5.81					

Table 7 - Continued

Case Number	Sample Number	Lactose	Heroin	Procaine	Acetyl-codeine	Codeine	Caffeine	Metha-pyrilene
58	79626	PC	6.65					
	85636	PC	6.56					

[a]PC Poorly crystallized.

[b]WC Well crystallized.

[c]SOL Soluble in glacial acetic acid.

[d]NSOL Not soluble in glacial acetic acid and no lactose present.

quantitation results are also listed in Table 7.

EXPERIMENT 3--HEROIN:MONOACETYLMORPHINE:MORPHINE RATIO

The HMM ratio was calculated for each of the 100 samples. The results were tabulated according to increasing heroin ratio for the 36 supposedly unrelated cases and then for each series in the 22 cases studied. It was very difficult to make any type of comparison manually from this listing of the ratios. Therefore, the method used by Van Der Slooten and Van Der Helm (8) was employed and the HMM ratio was calculated as the percentage of the heroin peak area represented by the peak areas of the monoacetylmorphine and morphine. These results, presented in Table 8, were used to construct bar graphs on semi-logarithmic graph paper for each of the 100 samples depicting the HMM ratios. A visual comparison was then possible to compare the HMM ratios of the samples with each other.

The 36 supposedly unrelated cases were examined first. Eighteen of the samples were found to have HMM ratios that did not compare with any other samples. The remaining eighteen samples were found to have the same HMM ratio within experimental limits which could be grouped into eight different HMM ratio patterns indicating the involvement of eight different original batches of heroin. Figure 4 presents the bar graphs

Table 8

Heroin:Monoacetylmorphine:Morphine Ratios
and Percent of Heroin Peak Area

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
1	22368	56.65	36.52	6.82	64.47	12.04
2	86591	56.62	21.90	21.48	38.68	37.94
3	42167	91.54	2.07	6.39	2.26	6.98
4	96301	87.91	4.80	7.21	5.46	8.19
5	03237	90.92	4.81	4.27	5.29	4.70
6	28918	71.76	20.34	7.90	28.35	11.01
7	07119	80.27	13.32	6.41	16.59	7.99
8	51085	90.40	4.51	5.09	4.99	5.63
9	07056	73.20	16.68	10.12	22.79	13.83
10	48663	82.63	10.27	7.10	12.43	8.59
11	91921	77.78	8.77	13.45	11.28	17.29
12	00582	76.53	15.13	8.34	19.77	10.90
13	69011	80.58	7.56	11.86	9.38	14.72

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
14	25976	60.36	34.19	5.45	56.64	9.03
15	09763	55.94	37.41	6.66	66.88	11.91
16	91567	74.24	16.04	9.71	21.61	13.08
17	14577	83.35	7.63	9.02	9.15	10.82
18	98427	73.31	13.88	12.81	18.93	17.47
19	92608	70.17	19.04	10.79	27.13	15.38
20	30405	61.20	22.94	15.86	37.48	25.92
21	38935	88.48	5.17	6.35	5.84	7.18
22	16631	53.98	21.33	24.69	39.52	45.74
23	44657	85.74	7.26	7.01	8.47	7.01
24	96773	75.84	13.39	10.77	17.66	14.20
25	50001	81.56	9.79	8.65	12.00	10.61
26	27504	79.78	14.24	5.98	17.85	7.50
27	37169	81.28	9.79	8.93	12.05	10.99

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
28	11508	75.53	9.97	14.49	13.20	19.18
29	37449	66.57	18.18	15.25	27.31	22.91
30	60336	81.10	12.91	6.00	15.92	7.40
31	03299	81.66	16.49	1.85	20.19	2.27
32	18039	76.63	13.00	10.38	16.97	13.55
33	79556	76.43	15.40	8.17	20.15	10.69
34	38534	70.99	21.61	7.40	30.44	10.42
35	10480	81.98	10.63	7.39	12.97	9.01
36	57491	88.64	4.93	6.42	5.56	7.24
	59037	46.37	49.29	4.34	106.30	9.36
37	42488	73.43	21.38	5.18	29.38	7.05
	46764	47.45	47.54	5.01	100.19	10.56

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
38	37570	74.98	20.56	4.46	27.42	5.95
	99562	73.88	21.21	4.92	28.71	6.66
39	77921	86.63	8.63	4.74	9.96	5.47
	01011	83.24	9.02	7.74	10.84	9.30
40	85475	73.40	20.46	5.96	27.88	8.12
	63553	66.40	23.49	10.11	35.38	15.23
	10365	77.79	16.08	6.13	20.67	7.88
41	09429	73.10	17.67	9.23	24.17	12.63
	00725	66.52	7.63	5.85	11.47	8.79
42	08326	58.05	33.63	8.32	57.47	14.33
	52162	66.08	23.94	9.97	36.23	15.09

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
42	02368	76.32	12.21	11.47	16.00	15.03
43	23982	73.73	19.48	6.79	26.42	9.21
	09915	74.26	18.87	6.88	25.41	9.27
44	54164	52.92	38.66	8.43	73.05	15.93
	32639	48.08	42.81	9.12	89.04	18.97
45	29334	63.02	30.32	6.67	48.11	10.58
	02488	62.66	26.63	10.72	42.50	17.11
46	00742	64.21	22.79	13.01	35.49	20.26
	05366	67.78	22.86	9.37	33.73	13.82
47	17955	72.93	15.18	11.89	20.82	16.30
	46503	74.99	15.14	9.86	20.19	13.15

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
48	34914	80.02	10.35	9.63	12.93	12.04
	70060	84.14	11.05	4.81	13.13	5.72
49	53976	77.49	14.67	7.84	18.93	10.12
	76072	78.48	12.92	8.60	16.46	10.96
50	90725	70.26	18.53	11.21	26.37	15.96
	08962	65.84	22.49	11.66	34.16	17.71
	73115	72.38	17.39	10.24	24.03	14.15
51	64364	66.55	21.00	12.45	31.56	18.71
	95012	73.28	18.90	7.82	25.79	10.67
	15664	75.90	18.21	5.89	23.99	7.76
	16408	67.37	17.83	14.81	26.47	21.98
	18629	76.37	18.38	5.25	24.07	6.87

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
52	31624	79.30	9.31	11.40	11.74	14.38
	78919	72.22	11.50	16.28	15.92	22.54
53	03931	77.12	10.55	12.33	13.68	15.99
	74426	88.59	5.82	5.59	6.57	6.31
	09066	74.32	11.20	14.49	15.07	19.50
	42238	82.82	8.33	8.85	10.12	10.69
	16153	79.25	16.45	4.30	20.76	5.43
	21457	81.86	8.66	9.48	10.58	11.58
	21581	79.89	9.26	10.85	11.59	13.58
	55612	85.17	7.52	7.31	8.83	8.58
54	91340	72.54	15.23	12.23	21.00	16.86
	91227	80.78	11.88	7.34	14.71	9.09
	92157	77.11	14.79	8.11	19.18	10.52

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
55	65390	80.39	10.90	8.71	13.56	10.84
	24130	82.96	10.22	6.82	12.32	8.22
	21885	84.94	8.37	6.69	9.85	7.88
	89579	82.48	10.37	7.16	12.57	8.68
56	82486	75.73	14.98	9.29	19.78	12.27
	81525	75.34	16.27	8.39	21.60	11.14
	97656	82.55	9.77	7.68	11.84	9.30
	29676	74.33	13.30	12.36	17.89	16.63
57	46515	80.50	11.40	8.10	14.16	10.06
	30986	70.25	14.38	15.36	20.47	21.87
	63798	83.64	10.70	5.66	12.79	6.77
	43937	83.18	11.02	5.80	13.25	6.97

Table 8 - Continued

Case Number	Sample Number	HMM Ratio Heroin	Monoacetyl.	Morphine	Percent of Heroin Peak Area Monoacetyl.	Morphine
58	79626	85.12	9.27	5.61	10.89	6.59
	85636	73.22	13.19	13.60	18.01	18.57

for these samples.

In addition, all of the 36 supposedly unrelated samples were compared with the HMM ratios obtained for the remaining 64 samples from the 22 cases studied. Correlations could be made with 4 of the 18 samples with no previous HMM matches with 4 of the 22 cases studied. Comparisons could be made with 7 out of the 8 groups of similar samples from the 36 supposedly unrelated samples with 7 of the 22 cases studied. Figure 5 presents the bar graphs for these comparisons.

The bar graphs for the 64 samples involving 22 cases were arranged in the order of the date of their purchase or confiscation. As a result it could be seen that as time progressed a certain batch of heroin would disappear and one with a different HMM ratio would appear on the scene. Eventually the new one disappears and another shows up with a different HMM ratio. The older samples characterized by a particular HMM ratio do not seem to reappear after a period of several months presumably because they have been consumed.

When the bar graphs are arranged in groups according to which of the 22 cases they are involved with several correlations can be made from examination of the HMM patterns. Nine of the cases examined involve exhibits produced from nine original batches

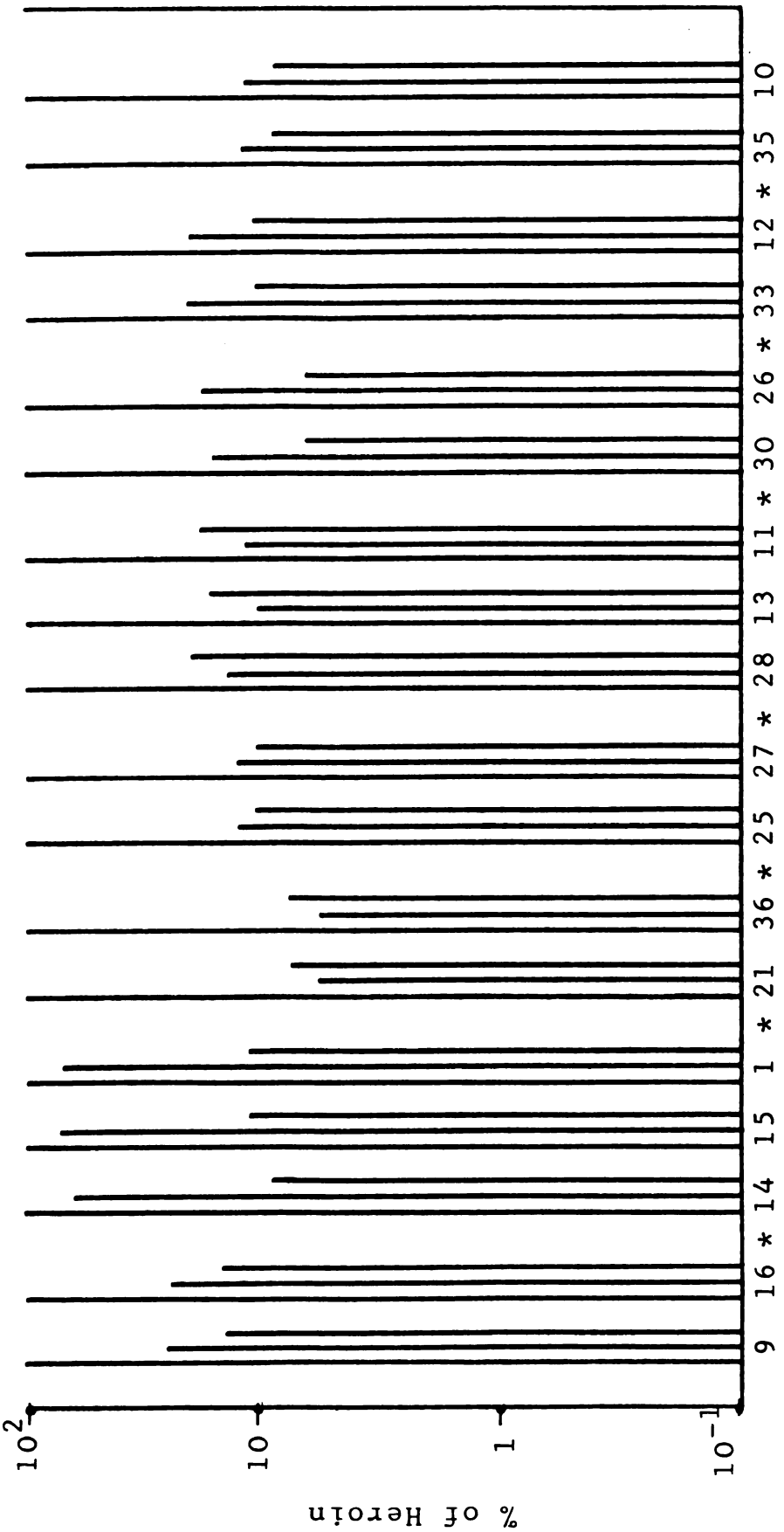


Figure 4.--HMM ratios of 18 unrelated samples forming 8 groups of similar patterns.

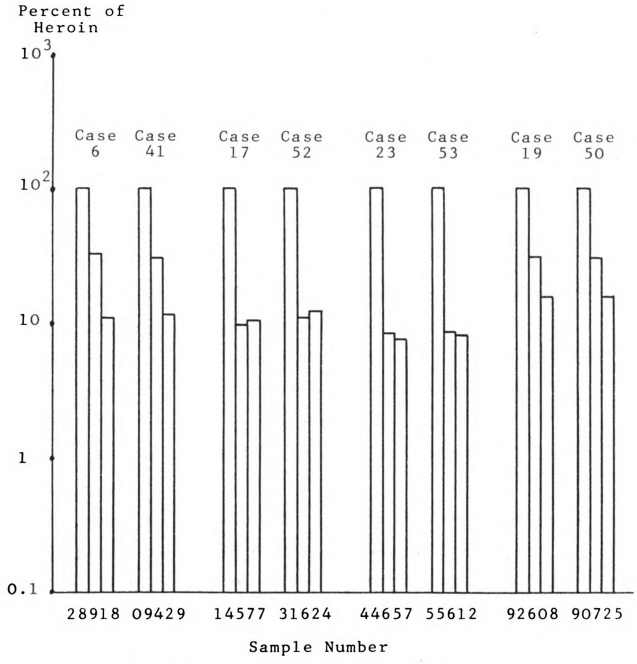


Figure 5.--HMM ratio comparisons of 4 of the 18 unrelated samples having no previous HMM matches with 4 of the 64 samples involving 22 cases.

of heroin. Each case contains exhibits produced from the same original batch so that the HMM ratios of the exhibits in each case are different from those in each of the other eight cases. Figure 6 shows the bar graphs produced from the HMM ratios for these nine cases.

Three cases each involve three exhibits. It can be shown that these three cases are composed of two exhibits produced from the same original batch of heroin while the third exhibit was produced from a different batch. In addition, the two similar exhibits in one of the three cases have the same HMM ratio as the third non-matching exhibit in one of the other cases. Figure 7 presents the bar graphs for these comparisons.

Four cases can each be shown to be composed of two exhibits produced from two separate original batches of heroin. The HMM ratios of all eight exhibits involved in these four cases are different with one exception. The HMM ratio of two of the exhibits each from a different case are the same. The dates of confiscation of the two exhibits are approximately two months apart and both were from Bay City. Figure 8 presents the bar graphs for these cases.

The remaining six groups of bar graphs were prepared from HMM data derived from a total of 28

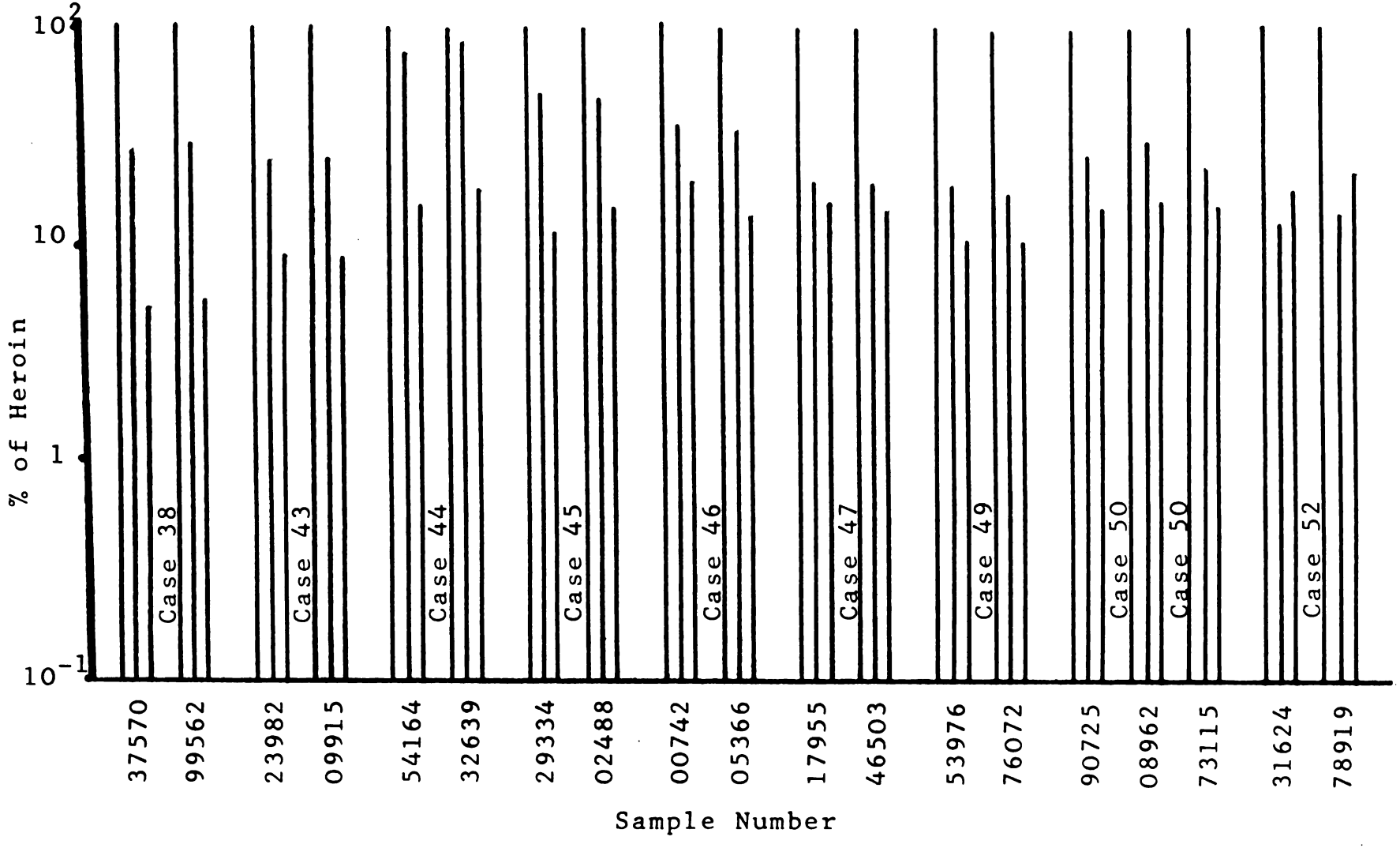


Figure 6.--HMM ratios of samples from nine separate cases indicating the existence of nine separate batches of heroin.

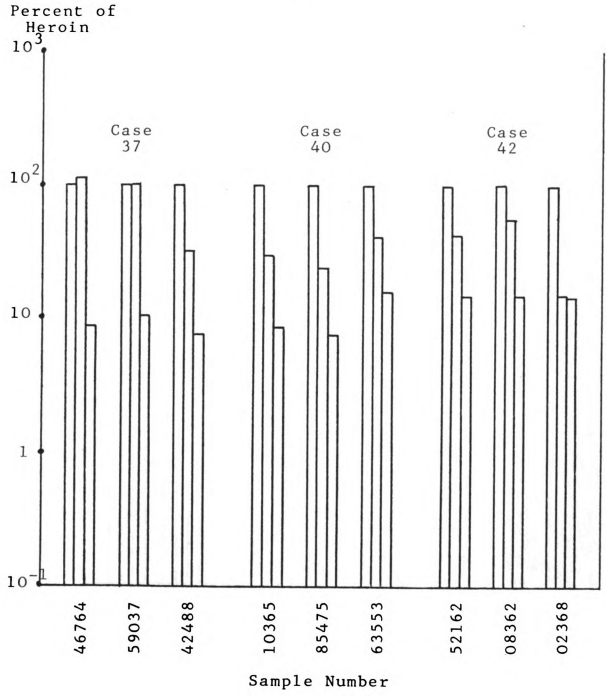


Figure 7.--HMM ratios of 3 samples in each of 3 cases; 2 of the 3 samples in each case have similar HMM ratios while the third is different indicating the existence of two original batches of heroin for each case.

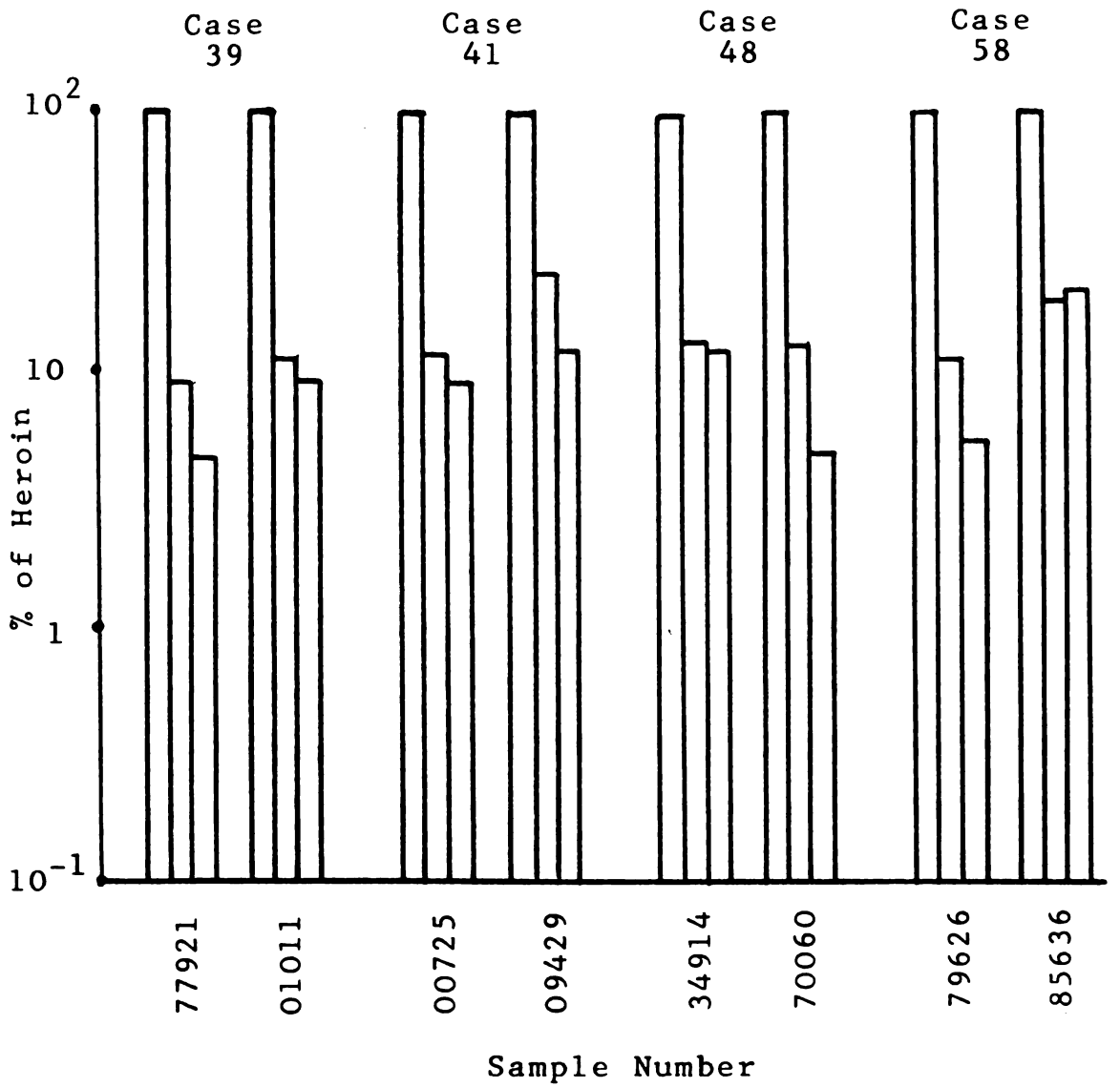
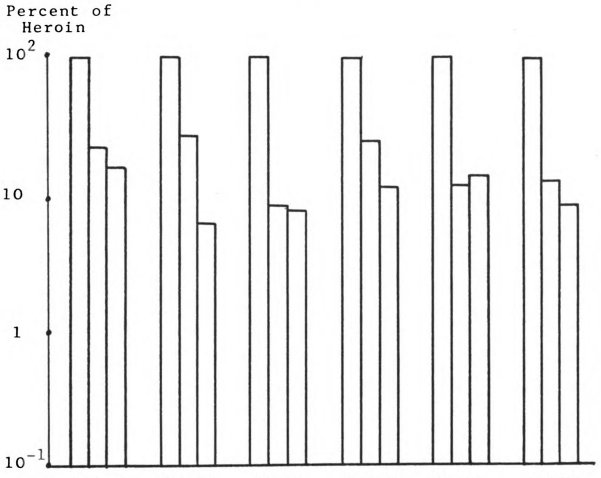


Figure 8.--HMM ratios of 2 samples in each of 4 cases, indicating the presence of four original batches of heroin.

different exhibits. There appeared to be considerable overlap between cases, i.e., exhibits with similar HMM ratios appeared in more than one case. Therefore, these exhibits from the six cases were examined as a single group. From this examination six different HMM ratio patterns could be determined. Figure 9 presents the six bar graphs of the HMM ratios characteristic to the samples in the six cases and indicates which exhibits have each particular HMM ratio pattern. It may also be noted that all 28 exhibits came from the Saginaw area between 8-15-77 and 4-25-78, an eight month period.

Additional correlations can be made between the HMM ratios of the exhibits in particular cases. As previously stated, one of the exhibits present in case number 38 has the same HMM ratio as two exhibits in case number 37. It is also similar to the HMM ratio of one exhibit in case number 40 and to both exhibits in case number 43. Similarly, one of the exhibits involved in case number 39 has a similar HMM ratio as one of the exhibits involved in case number 41. Case number 40 involves an exhibit with an HMM ratio which matches that of exhibits in cases numbered 42 and 46. Case number 51 involves exhibits with HMM ratios that match those of exhibits involved with cases numbered 50 and 53. Exhibits involved with



16408 91340 29674	18629 15664 16153	74426 55612 21885	92157 82486 81525 95012 64364	42238 21457 03931 09066 21581 30986	63798 43937 91227 24130 89579 65390 46515 97656
Samples With Similar HMM Patterns					
51 54 56	51 53	53 55	51 54 56	53 57	54 55 56 57
Case Numbers Involved					

Figure 9.--Six HMM ratio patterns characteristic to 28 samples involving 6 cases.

case number 52 have nearly the same HMM ratios as exhibits involved with case number 53. Correlations between the remaining cases have been previously dealt with when individual case exhibits were being considered above.

It should be noted that all of these comparisons have been made entirely based upon a visual examination of the HMM ratios of the various exhibits as depicted in bar graphs. The results from experiment 1 and 2 will be considered in the discussion section along with these observations to attempt to confirm the conclusions drawn from the graphs alone.

Blind Trial Study

Five separate heroin samples were prepared by weighing out 5.0 mg of heroin, 5.0 mg of monoacetylmorphine, and 0.8 mg of morphine into five separate test tubes. Twenty milligrams of procaine and twenty milligrams of lactose were added to each of the test tubes. Two of these samples were set aside and 8.0 mg of codeine was added to each of the three remaining test tubes and to one of these was added 8.0 mg of acetylcodeine. This resulted in five samples which should all have very close HMM ratios which would differ only due to the error of weighing out such small quantities. This procedure will also demonstrate the difficulty in trying to reproduce the same

HMM ratio in different batches of heroin even when one is trying to do it intentionally using an analytical balance.

Three aliquots were taken from each of the five samples produced above. To one of the aliquots from each of the five samples was added 20 mg of lactose, to another was added 10 mg of procaine, and to the last group was added 10 mg of caffeine thus producing an additional 15 samples. Each of the 20 samples thus produced was assigned a random 5 digit number for reference and each was extracted, derivatized, and analyzed on the gas chromatograph under the same parameters as the 100 samples analyzed in experiment 3. The data obtained was processed to provide the necessary information to construct bar graphs indicating the HMM ratio of each sample. Visual examination of the bar graphs led this investigator to judge 18 out of the 20 samples correctly, a 90% proficiency. It must be noted, however, that this judgement was based solely on the HMM ratio comparisons without any knowledge of the adulterants present.

Chapter 5

DISCUSSION

The purpose of this study was to devise a method by which one could routinely examine illicit heroin samples submitted to the crime laboratory in such a fashion that the data obtained would allow one to determine a common source of origin for various samples. The aim was to provide a simple procedure that would make samples originating from a common source readily apparent to the analyst even though previous dilution or adulteration made the exhibits appear entirely different to the naked eye or to produce gas chromatographic charts which are obviously different. The rather simple extraction procedure followed by derivatization and gas chromatographic analysis as done in this study to determine the HMM ratios of various exhibits satisfies the first part of this aim. The comparison of HMM ratios would satisfy the second part of this aim. However, additional information may still be needed to confirm the conclusions reached in examining only the HMM ratios as some of the following examples illustrate.

When the 36 supposedly unrelated cases were

considered on the basis of their HMM ratios only it was concluded that eight groups of similar exhibits existed. Further, it was concluded that four out of the 18 exhibits with no previous HMM matches could be matched with four of the 22 cases studied and that seven of the eight groups could also be matched with seven of the 22 cases studied as depicted in Figures 3 and 5. Now, consider some of these correlations in light of the additional data supplied by Experiments 1 and 2.

Exhibit 88231 was found to match exhibit 52636. From Tables 5 and 7 it can be seen that both came from Bay City approximately two weeks apart and that both contained procaine. Exhibit 88231 contained 6.10% heroin with poorly crystallized lactose while exhibit 52636 contained 3.36% heroin with well crystallized lactose. Based upon all of this information it could be concluded that even though the quantity of heroin present in one of the exhibits is roughly twice that of the other sample the two samples could very well have originated from the same batch of heroin. A 1:1 dilution of exhibit 88231 with lactose commercially available which should be well crystallized would yield a product similar to exhibit 52636 while not changing the HMM ratio.

Exhibits 25976, 09763, and 22368 were also

determined to have similar HMM ratios. Additional data has shown exhibits 25976 and 09763 to both contain well crystallized lactose, no procaine and nearly equal amounts of heroin and acetylcodeine. Exhibit 22368 however, contains poorly crystallized lactose, it contains procaine and only about half as much heroin as the other two exhibits with no acetylcodeine. All were from the Saginaw area. It is relatively easy to show that these three exhibits still originated from the same source. The addition of procaine to exhibit 25976 or 09763 in a 1:1 ratio would produce the results listed for exhibit 22368. The lactose would appear poorly crystallized as the procaine dissolved in the acetic acid. The adulteration with procaine would account for the lower percentage of heroin present and would reduce the acetylcodeine to to an undetectable level. The only problem in concluding that these three exhibits originated from the same batch of heroin is the dates of the confiscations. Exhibits 25976 and 09763 were both confiscated on 1-18-77 while exhibit 22368 which is the exhibit that would have to have been adulterated with procaine was confiscated on 9-21-74, approximately two years prior to the confiscation of the other two exhibits. Based upon all of the information then it would be more reasonable to conclude that exhibits 25976 and 09763

originated from the same batch of heroin while exhibit 22368 originated from a different batch of heroin with a similar HMM ratio to that of the first batch.

Exhibits 27504 and 60336 were among the 36 supposedly unrelated samples that were found to have similar HMM ratios. Further, they were found to have HMM ratios similar to exhibits 82486, 81525, 63798, and 43937 which are four exhibits involving two separate cases from among the 22 cases studied as listed in Table 5. Additional data further showed that all six of the exhibits came from the Saginaw area between 4-12-78 and 4-21-78. All contained poorly crystallized lactose, none contained procaine and all contained heroin quantitatively determined to be within a range of 3%. Considering all of this data it would be reasonable to conclude that all of the exhibits came from one original batch of heroin. This also illustrates a series of correlations that were correctly made using only the HMM ratios.

One more example will be taken from the comparisons made between the 22 cases studied for discussion purposes. Case number 37 involves exhibits 59037, 42488, and 46764. Exhibits 46764 and 59037 have a similar HMM ratio while exhibit 42488 is different. Case number 38 involves exhibits 37570 and 99562. Both of these exhibits have similar HMM ratios which

are similar to that of exhibit 42488 from case number 37. Additional data indicates that all five of the exhibits came from Saginaw between 9-5-75 and 11-4-75. Exhibits 37570 and 99562 from case number 37 and exhibit 42488 from case number 38 all contain procaine, none contain lactose since all of the samples were soluble in glacial acetic acid and all contain heroin within roughly a 4% range. Exhibits 59037 and 46764 which are both from case number 37 are similar except that they each contain roughly 50% less heroin than the other three exhibits. Adulteration of the other three exhibits with procaine could produce this observed decrease in the amount of heroin present. However, the two exhibits with the lower amount of heroin present also contain detectable amounts of acetylcodeine with a mean value of 0.8% which should of course be greater before adulteration. The other three exhibits did not contain any detectable amount of acetylcodeine. Therefore, it is reasonable to conclude that two original batches of heroin are involved and that exhibits 42488, 37570, and 99562 involved with both cases originated from a single original batch of heroin.

The blind trial study indicated that this researcher was able to differentiate with 90% proficiency between exhibits with very similar HMM ratios

even after subsequent dilution and adulteration of the samples. Illicit heroin samples are not carefully mixed or ground up to ensure homogeneity of the heroin batches. Therefore, one should expect rather significant variations in the HMM ratios of heroin samples from the same granular type of heroin source than from finely ground batches. It is likely that some of the illicit heroin batches being distributed at the street level will also end up being mixed with remaining portions of other batches thus creating supplies with HMM ratios different than the original batches.

Chapter 6

APPLICATION

The procedure presently employed by analysts in the Michigan State Police Scientific Laboratories system for analyzing heroin exhibits would not require a radical change to provide the data necessary to compare exhibits and determine a common source of origin. Each heroin exhibit submitted to the laboratory is qualitatively analyzed using infrared spectrophotometry or mass spectroscopy to establish the presence of heroin. The results of these analyses along with the quantitation data for the heroin obtained by a gas chromatographic analysis are recorded on computer cards by blackening out the appropriate number codes. The cards also record the laboratory number assigned to each individual exhibit, the date the exhibit was received at the laboratory and the police department which confiscated the exhibit.

The major change in this routine procedure would involve the chemical derivatization procedure prior to the quantitative analysis so that the amount of monoacetylmorphine and morphine could be determined

as well as the amounts of the adulterants or other products of manufacture such as codeine or acetylcodeine. Each individual unit within the laboratory system has a computer card and program designed to handle the data generated by that particular unit. All involve the recording and storing of statistical data such as the number of examinations versus the number of identifications in a particular case as well as the results of quantitative examinations such as the percent by weight of heroin in a particular sample or the weight percent of alcohol per 100 milliliters of blood. Therefore, it would be relatively simple to design a computer card and program to record and store the quantitative data provided by the gas chromatographic analysis of the derivatized sample. In effect, the computer card would contain the data necessary to determine the HMM ratio, date of confiscation, location of confiscation as derived from the submitting police department code number, presence or absence of diluents and the amounts of adulterants or other products of manufacture. The computer program could be written so that it would match those samples with HMM ratios found to be the same within the limits determined by experimental error and eliminate those matches which involve cases found to have been submitted to the laboratory on dates more than a

predetermined period of time apart such as one year. The printout could include the additional information such as amounts of adulterants or presence of diluents for each exhibit to further establish a common source of origin.

This data submission procedure would not have any city, county or state boundaries. Any forensic laboratory which should be equipped with a gas chromatograph would provide the means for a chemist to perform the derivatization procedure on a portion of each heroin exhibit and obtain the data necessary to make comparisons to establish a common source of origin. The data could be submitted to one location where it would be transferred to the computer cards and stored in the computer memory. This would involve only the transfer of data obtained during the analysis of a particular exhibit identified only by a laboratory number. There would be no transfer of a portion of the physical evidence itself eliminating any legal ramifications such as the chain of custody of the evidence.

Computer data indicating the common origin of two or more exhibits could be presented in the form of an intelligence report stating the probability that certain samples have a common source of origin. The report would be submitted to the intelligence units

of the departments originally submitting the similar exhibits. It would then be the responsibility of the various departments to utilize the data in a cooperative effort to assign their narcotics agents to those areas where the heroin appears to be originating.

The additional time required to perform the derivatization procedure and quantitative calculations per sample is approximately 1½ hours and the only additional cost is for the derivatizing reagent. At the present time this amounts to approximately 50 cents per sample over the cost of performing the routine analysis.

Chapter 7

SUMMARY

Gas chromatography was initially employed to identify and quantitate the adulterants present in 100 illicit heroin samples which had been previously submitted to the Michigan State Police Bridgeport Regional Crime Laboratory as exhibits in narcotic cases. The presence of lactose or starch was determined microscopically.

The amount of heroin was determined in each case also using the gas chromatograph employing an internal standard and an electronic digital integrator.

A derivatization technique was employed to examine the 100 samples by gas chromatography to determine the heroin:monoacetylmorphine:morphine (HMM) ratio in each sample. The ratios were then calculated in terms of the percentage amount the monoacetylmorphine peak and morphine peak represented relative to the heroin peak. Bar graphs on a semi-logarithmic scale were constructed for each of the 100 samples representing the HMM ratios. Visual comparisons were made between the bar graphs and correlations

were made among the samples as to common sources of origin. Several of these correlations were discussed at length while taking into account the additional data provided by the total sample assay and heroin quantitation experiments.

A blind trial study was conducted on twenty samples with similar HMM ratios following dilution and adulteration. This researcher was able to correctly match the samples based soley on the HMM data in 18 out of the 20 trials for a 90% proficiency rating.

Practical applications of this type of analysis was discussed. Computer application would allow data storage and rapid retrieval for intelligence reports that could aid participating police departments in assigning their personnel to areas where narcotics or drugs are originating.

The major conclusion of this study is that the comparison of HMM ratios to determine a common source of origin for illicit heroin samples is a reliable method for investigative purposes. Additional data would be necessary for any conclusions to be made for court purposes.

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