J Forensic Sci, Jan. 1977, Vol. 22, No. 1

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Identification of Phencyclidine-Related Drugs

The appearance of new drugs within the crime laboratory necessitates the development and improvement of analytical schemes for their detection. One class of particular interest is the phencyclidine-related drugs. The drugs studied are 1-(1-phenylcyclohexyl) piperidine (PCP), commonly known as phencyclidine, "Angel's Dust," or DOA [*1*]; the two homologs 1-(1-phenylcyclohexyl) pyrrolidine (PHP) and 1-(1-phenylcyclopentyl) piperidine (PPP); and an analog 1-(2[thienyl]cyclohexyl) piperidine (TCP).

The analytical procedures evaluated resulted in a positive identification of these drugs. They are microchemical tests, chemical ionization mass spectrometry (CIMS), thin-layer chromatography (TLC), and gas-liquid chromatography.

A comparison of the four drugs by chemical data is presented in Table 1. The ultraviolet (UV) spectrophotometric data, previously compiled [*2,3*], are listed in Table 2. As expected [*4*], only the thiophene analog (TCP) is markedly different. Infrared spectra are also available for each drug but identification by this method can be difficult with a sample that is highly adulturated or in trace quantities.

Experimental Methods

Approximately 3 mg of phencyclidine and the two homologs were placed on a glass slide with 10% aqueous hydrochloric acid or 10% aqueous acetic acid with 2% aqueous potassium permanganate. The crystal formations were observed on a compound microscope at ×400 and are depicted in Figs. 1 and 2.

The chemical ionization mass spectra were taken for all four drugs on a Dupont 21-490 single focusing mass spectrometer. The reagent gas was isobutane (99.9%). All drugs analyzed were admitted through direct probe. The instrument operating conditions were the same as described by Saferstein et al [*5*]. The data are tabulated in Table 3.

Thin-layer chromatography was conducted on 250-μm silica gel plates manufactured by Analtech, Inc., Newark, Del. All drugs were extracted as the free base and spotted with chloroform. After development, the drugs were visualized with 5% aqueous potassium iodoplatinate producing blue-gray spots. All chemicals and solvents were reagent grade and supplied by J. T. Baker, Phillipsburg, N.J. The following solvent systems were used with the resulting Rf × 100 values compiled in Table 4:

(A) cyclohexane:benzene:diethylamine (75:15:15),
(B) ethyl acetate:methanol:ammonium hydroxide (85:10:5),
(C) acetone:dimethylformamide:ammonium hydroxide (85:15:0.5),

Received for publication 7 April 1976; revised manuscript received 1 July 1976; accepted for publication 2 July 1976.

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TABLE 1—*Chemical data.*

Drug	Abbreviation	Empirical Formula (Free Base)	Molecular Weight (Free Base)	Molecular Weight (HCl Salt)	Source
1-(1-phenylcyclohexyl) piperidine (phencyclidine)	PCP	$C_{17}H_{25}N$	243.4	279.9	clandestine
1-(1-phenylcyclohexyl) pyrrolidine	PHP	$C_{16}H_{23}N$	229.4	265.9	Lot P[a]
1-(1-phenylcyclopentyl) piperidine	PPP	$C_{16}H_{23}N$	229.4	265.9	Lot P[a]
1-(1-[2-thienyl]cyclohexyl) piperidine	TCP	$C_{15}H_{23}NS$	249.3	275.8	Lot Q[a]

[a] Parke Davis & Co., Midland, Mich.

TABLE 2—*Ultraviolet spectra.*

Drug	Wavelength Maxima,[a] 0.1*N* HCl
PCP	262, 257, 268, and 252
PHP	262, 257, 268, and 252
PPP	261.5, 256.5, 268, and 252
TCP	232

[a] Strongest maxima are underlined.

(D) methanol:acetic acid (90:10),
(E) chloroform:methanol (90:10),
(F) chloroform:methanol (80:20),
(G) acetone:chloroform (50:50), and
(H) acetone:chloroform (65:35).

A Varian 2700 dual column gas chromatographic with flame ionization detectors at 270°C was used with two 6-ft (1.8-m) by ¼-in. (6.35-mm) outside diameter and 2-mm inside diameter glass columns from Analabs, Inc., North Haven, Conn. These columns were treated with dimethyldichlorosilane and packed with 3% OV-17 and 3% OV-101 on 100/120 mesh Gas Chrom Q. The column temperature was 170°C, and the injection port temperature was 250°C. A flow rate of 30 ml/min was used with medical-grade nitrogen as the carrier gas. The detector gases were H_2(99.999%) and compressed air. All samples were injected as the free base with an 801-μl Hamilton syringe. A graphical presentation was presented on Hewlett Packard Corp. 3380 A integrator. The tabulated data are presented in Table 5 and Fig. 3.

Discussion

The crystal formations produced by PCP and PPP in 10% aqueous HCl and 2% $KMnO_4$ were indistinguishable. They can be described as violet, H-shaped plates, whereas PHP produced crystals also resembling violet, H-shaped plates, only thinner (Fig. 1). In 10% acetic acid and 2% $KMnO_4$ the crystals produced for phencyclidine and its homologs were somewhat dissimilar and can be used for differentiation. Again, PCP formed violet, H-shaped plates, but PHP formed orange, H-shaped plates. The PPP homolog produced definite, violet, X-shaped crystals by this test, as shown in Fig. 2.

The chemical ionization mass spectra for the drugs studied produced the protonated molecular ion (MH^+) in greatest abundance. Phencyclidine (PCP) with an *m/e* of 244 and the thiophene analog (TCP) having an *m/e* of 250 were easily distinguished from the two homologs PPP and PHP having an *m/e* of 230.

The molecular ion M^+ and the M^{-1} ion were also present. The homologs PHP and PPP (molecular weight 229.3) produced the same protonated molecular ions and molecular ions. However, the fragmented ions for PHP and PPP, *m/e* 159 and 165, respectively, allowed differentiation (Table 3).

A fragmented ion product of PCP (*m/e* 159) postulated by Hauber [*6*] as phenylcyclohexene was also present for PHP. Furthermore, the fragmented ion (*m/e* 145) produced by PPP probably corresponds to phenylcyclopentene and the *m/e* of 165, resulting from the fragmentation of the thiophene analog, is most likely thienylcyclohexene.

The alkaline TLC Systems A, B, C and the acidic solvent System D failed to produce adequate resolution. The best results were obtained with the neutral Systems E through H, which successfully resolved PCP, PHP, PPP, and TCP with excellent reproducibility. These systems were composed of various proportions of chloroform with methanol

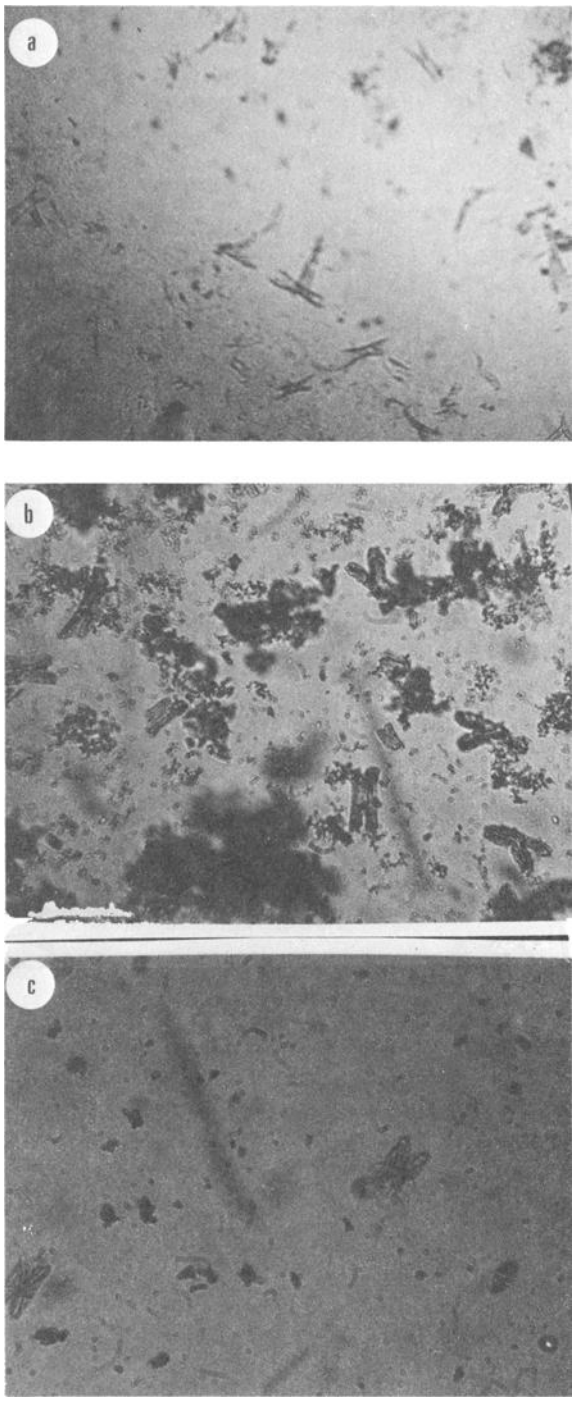

FIG. 1—*Crystal formations observed on a compound microscope with 10% HCl and 10% $KMnO_4$;* (a) *PHP;* (b) *PPP; and* (c) *PCP.*

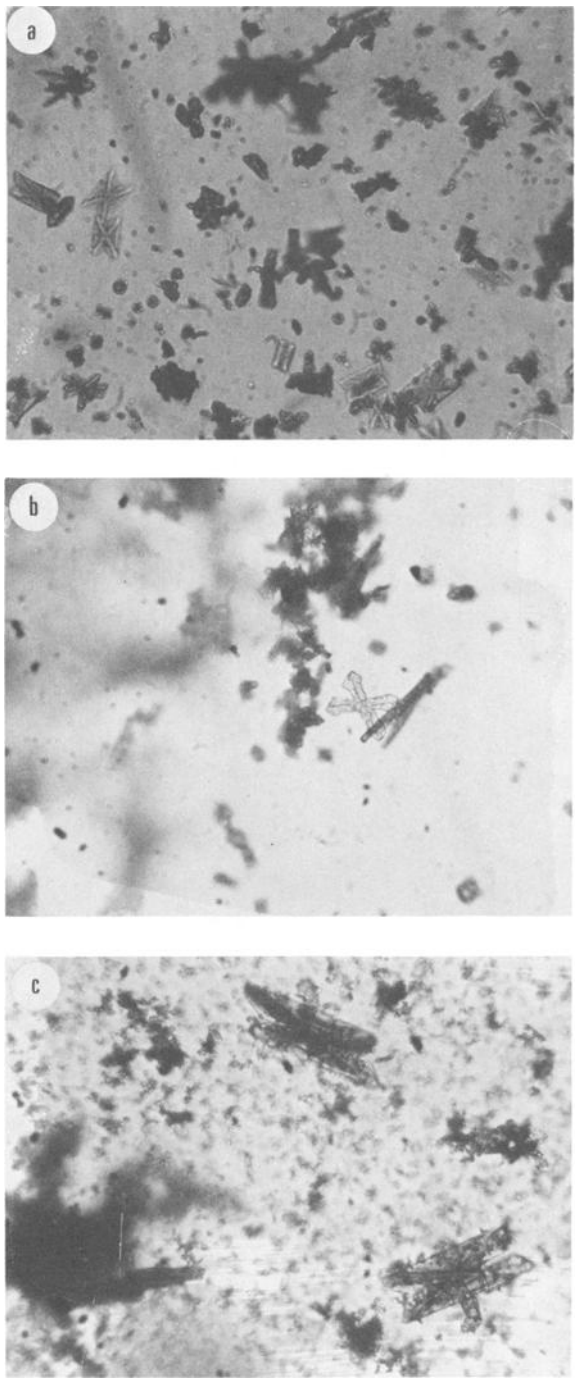

FIG. 2—*Crystal formations observed on a compound microscope with 10% aqueous acetic acid and 2% $KMnO_4$;* (a) *PHP;* (b) *PPP; and* (c) *PCP.*

TABLE 3—*Mass spectral peaks abundance (greater than 20%).*

Drug/Degradation Product	Molecular Weights	*m/e* Peak 1	*m/e* Peak 2	*m/e* Peak 3	*m/e* Peak 4
PCP/phenylcyclohexene	243.4/158.3	244 (100%)	243 (30%)	242 (63%)	159 (32%)
PHP/phenylcyclohexene	229.4/158.3	230 (100%)	229 (65%)	228 (40%)	159 (22%)
PPP/phenylcyclopentene	229.4/144.3	230 (100%)	229 (75%)	228 (40%)	145 (43%)
TCP/thienylcyclohexene	249.3/164.2	250 (100%)	249 (60%)	...	165 (40%)

TABLE 4—*Thin-layer chromatography Rf × 100 values.*

Drug	Solvent Systems							
	A	B	C	D	E	F	G	H
PCP	69	86	98	54	17	29	23	32
PHP	69	75	95	57	10	24	05	13
PPP	69	80	98	54	25	37	29	40
TCP	69	82	98	55	33	51	45	49

TABLE 5—*Gas chromatography data.*

Drug	OV-101 Gas Chrom Q		OV-17 Gas Chrom Q	
	Retention Time, min	Relative Time[a]	Retention Time, min	Relative Time[a]
PPP	4.36	0.65	4.06	0.62
PHP	4.94	0.74	4.83	0.75
PCP	6.70	1.00	6.52	1.00
TCP	6.48	0.97	6.47	0.99

[a] Based on phencyclidine (PCP).

(Systems E and F) and acetone with chloroform (Systems G and H) (Table 4). There was little difficulty in reproducing the chromatographic study of Shulgin [7] for separating PCP and TCP.

Gas chromatography (GC) completely resolved the homologs in the order of PPP, PHP, and PCP using either 3% OV-17 or 3% OV-101 (Table 5). The liquid phases chosen represent the divergent polarities as exhibited by their McReynold's constants [8]. Better resolution of PCP and TCP could be obtained by lowering the column temperature but these drugs can be easily distinguished by their UV spectra as well as by TLC, as previously discussed. A GC/MS interface could probably serve as an efficient tool for rapidly identifying these structurally related compounds.

Some of these phencyclidine-related drugs were studied by Bailey et al [9]; the study resulted in a separation scheme using absorption spectrometry, electron impact MS, protonated magnetic resonance spectra, and several TLC and GC techniques. The drugs of most interest to forensic laboratories, namely PCP, TCP, and PHP, were only partially resolved in the chromatography techniques described. In Bailey's study PPP was not included.

Conclusion and Summary

The analytical techniques presented allow the forensic chemist to readily differentiate phencyclidine (PCP) from the two homologs 1-(1-phenylcyclohexyl) pyrrolidine (PHP) and 1-(1-phenylcyclopentyl) piperidine (PPP) as well as the analog 1-(2-[thienyl] cyclohexyl) piperidine (TCP).

The UV spectra in dilute mineral acid for PCP, PHP, and PPP are indistinguishable, but the TCP spectrum is markedly different because of the thiophene moiety. The $KMnO_4$ crystal test using HCl can only suggest the presence of a phencyclidine-related drug, but acetic acid does offer more distinguishability.

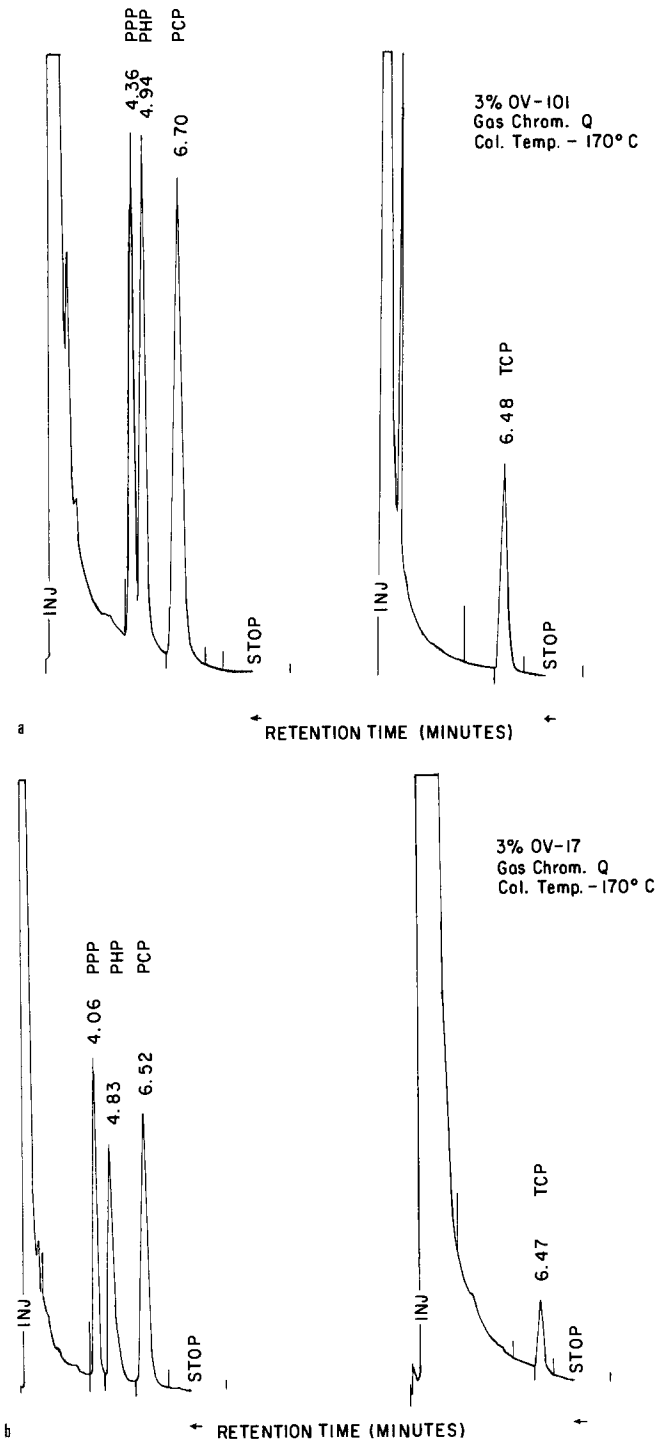

FIG. 3—*Gas chromatographic data.*

Chemical ionization mass spectra of all four drugs studied are different, except that the protonated molecular ion (MH^+) for the homologs PHP and PPP are the same. Both TLC and GC contribute to the separation and confirmation of these drugs after preliminary testing indicated their presence.

Acknowledgments

The authors wish to thank Lt. Joseph Barry and the New Jersey State Police for their cooperation as well as Ms. Michele Senko for her assistance in preparing this paper. The mass spectral data was compiled with the cooperation of the Forensic Science Bureau, New Jersey State Police, West Trenton, N.J. 08625.

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