Identification of a Street Drug as *N*-Ethyl-1-phenylcyclohexylamine, a Phencyclidine Analog

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Abstract □ The identification of *N*-ethyl-1-phenylcyclohexylamine in a street drug sample is described. Spectroscopic data suitable for the identification of the substance are presented.

Keyphrases □ *N*-Ethyl-1-phenylcyclohexylamine—identified in street sample by NMR, IR, and mass spectral data □ Abuse drugs—*N*-ethyl-1-phenylcyclohexylamine identified in street sample by NMR, IR, and mass spectral data □ Phencyclidine analogs—*N*-ethyl-1-phenylcyclohexylamine identified in street sample by NMR, IR, and mass spectral data

Phencyclidine (I) is a commonly encountered illicit synthetic drug on the street (1–3). Details of the synthesis of I and its analogs and homologs have been published (4–6). Because I is controlled under Schedule III of the Controlled Substances Act in the United States and under the Narcotic Control Act in Canada, it is to be expected that analogs having a similar pharmacological profile will be prepared and distributed on the illicit market (7, 8). The thiophene analog (known as thio-PCP) (II) and the pyrrolidine analog (III) have been positively identified in street samples (8), and the former is controlled under Schedule I of the Controlled Substances Act.

Methods for the unequivocal identification of I–III and three closely related tertiary amines have been published (9). The ethyl homolog, *N*-ethyl-1-phenylcyclohexylamine (IV), also known as cyclohexamine, PCE, CL-45, and CI-400, is said to have been available in California since about 1969 (8), but information and descriptions suitable for identification have not been published.

Psychic disturbances were seen with I and IV when their potential as anesthetics was explored (10), and investigations in humans were rapidly abandoned. This paper reports the identification of IV in a street sample. Spectroscopic and chromatographic properties of authentic material useful for its identification are described.

EXPERIMENTAL

Apparatus—A gas chromatograph equipped with a flame-ionization detector[1] and fitted with a 6-mm × 1.8-m glass column packed with 5% OV-7 on 80–100-mesh Chromosorb W (HP)[2] was used. The operating temperatures were 150, 250, and 275° for the oven, injector, and detector, respectively.

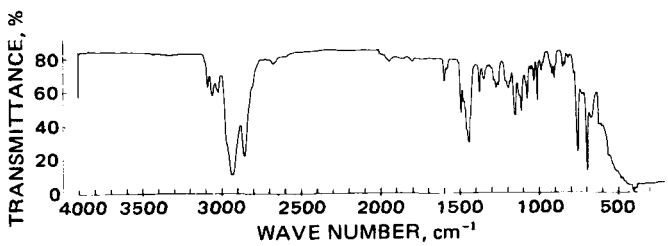


Figure 1—*IR spectrum of IV base as film on sodium chloride plates.*

TLC was carried out with 0.25-mm silica gel[3] precoated on glass[4]. The compounds were detected with iodoplatinate spray reagent, with which they produced magenta spots.

NMR spectra were recorded[5] at 40°, using tetramethylsilane as the reference standard. IR spectra were recorded[6] using natural films between sodium chloride plates for the free bases and potassium bromide disks for the salts. UV spectra were recorded[7] in ethanol solutions. Mass spectra were obtained by direct probe insertion on a spectrometer[8] operated with the probe at 160° and an ionization energy of 70 ev.

Spot Tests—The original sample was examined with Marquis, Mecke, and Froehde reagents (11); in each case, there was effervescence without color development. A flakey blue precipitate, insoluble in stannous chloride, was obtained with the cobalt thiocyanate test (12).

Isolation of IV (13)—The sample (about 25 mg of colorless crystals) was dissolved in 1% tartaric acid solution (0.5 ml) and triturated with a mixture of chloroform (1 ml), sodium bicarbonate (1 g), and acid-washed diatomaceous earth[9] (1 g). The mixture was transferred to a short glass

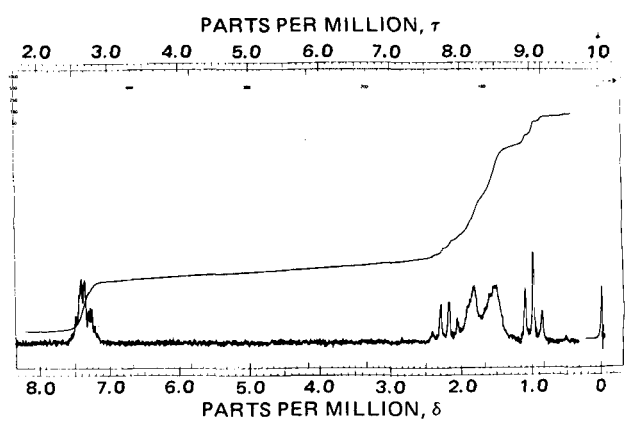
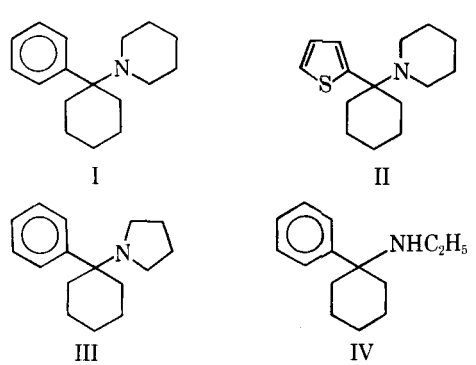


Figure 2—*NMR spectrum of IV base in deuterochloroform with deuterium oxide added.*



I II III IV

[1] Series 3000, Hydroflow Co., Toronto, Ontario, Canada.
[2] Chromatographic Specialties, Brockville, Ontario, Canada.
[3] Merck.
[4] EM Laboratories, Elmsford, N.Y.
[5] Model A-60A spectrometer, Varian Instruments, Palo Alto, Calif.
[6] Model 625 spectrophotometer, Perkin-Elmer Corp., Norwalk, Conn.
[7] Model DBGT spectrophotometer, Beckman Instruments, Fullerton, Calif.
[8] Hitachi Perkin-Elmer RMU-6L, Perkin-Elmer Corp., Norwalk, Conn.
[9] Celite 545, Chromatographic Specialties.

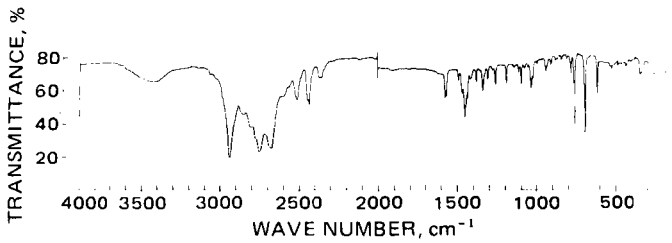


Figure 3—*IR spectrum of IV hydrochloride in potassium bromide disk.*

column, and the liberated components were eluted with chloroform (15 ml). The base was then extracted into 0.1 *N* H_2SO_4 (2 × 1 ml), which, in turn, was made alkaline with concentrated ammonia and finally extracted with chloroform (0.5 ml). Evaporation of the solvent gave IV as a colorless oil.

This procedure successfully isolated IV from excipients present in samples in the form of "decks" and "microdots" submitted for analysis on subsequent occasions.

Synthesis of IV—Authentic IV was synthesized in two ways: (*a*) by acetylation of 1-phenylcyclohexylamine (14) with acetic anhydride in acetic acid, followed by reduction of the resulting acetamide with lithium aluminum hydride (4); and (*b*) by the reaction of phenyllithium with *N*-ethylcyclohexanecarbonitrile (5). The product was converted into the hydrochloride salt, which was purified by recrystallization from a mixture of 2-propanol with ether (~3:1), mp 232–234° (with sublimation) [lit. (5) mp 240–242° and 236–238°].

Anal.—Calc. for $C_{14}H_{21}N$·HCl: C, 70.12; H, 9.25; N, 5.84. Found: C, 70.08; H, 9.27; N, 5.80.

The base was regenerated from the salt by addition of potassium carbonate solution and extracted into ether. IR, NMR, and mass spectra of the substance isolated from the sample and of authentic IV were compared. (The spectral data presented in this paper were obtained with authentic IV.)

RESULTS AND DISCUSSION

The results of the spot tests resembled those observed in these laboratories for samples containing cocaine or phencyclidine (I). The IR spectrum of the isolated base (Fig. 1) resembled spectra of I and its phenyl analogs (9) but notably lacked absorption bands at 2800 and 960 cm^{-1}. The bands at about 760 and 700 cm^{-1} suggested that a monosubstituted benzene ring was present, and a phencyclidine-like structure was inferred. The integrated proton NMR spectrum (Fig. 2) indicated that the compound had both cyclohexyl (δ 1.2–2.0 ppm) and ethylamino (δ 0.98-ppm triplet and 2.20-ppm quartet) moieties present with a mono-substituted benzene ring (δ 7.15–7.55 ppm), and it was surmised that the isolated material was IV.

Authentic IV was compared by IR, proton NMR, and mass spectral data with the isolated unknown, and the samples were found to be identical. The salt form of the original sample was not known, but a similar sample received at a later date was shown (mass spectrometry) to be the hydrochloride. IR, proton NMR, and mass spectral data from IV and its hydrochloride, suitable for identification purposes, are presented in Figs. 1–5. The IR spectrum of IV hydrochloride (Fig. 3) was quite different from those of tertiary amine analogs (9), especially in

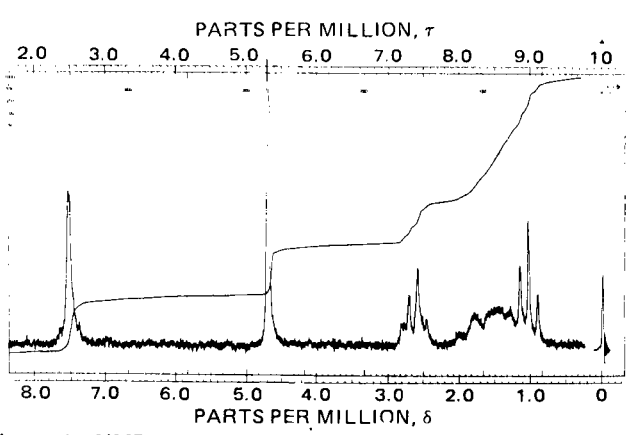


Figure 4—*NMR spectrum of IV hydrochloride in deuterium oxide.*

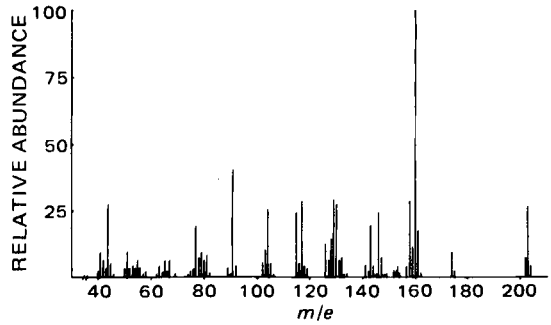


Figure 5—*Normalized mass spectrum of IV.*

showing strong absorption from 2660 to 2800 cm^{-1}, associated with the NH stretching of a secondary ammonium ion.

The mass spectrum of IV (Fig. 5) resembled spectra of the phenyl analogs reported (9) in having a moderately intense molecular ion M^+ (at 203), the base peak at M − 43 (160), and intense ions at 158, 143, 130, 129, 117, 115, 104, and 91 amu, all of which are of very minor intensity in the thienyl series. The UV spectrum of IV hydrochloride in ethanol was exactly like spectra of I and III hydrochlorides, having λ_{max} 252, 257, 261, and 268 nm. Compound II hydrochloride is distinguished by having λ_{max} 232 nm (9).

Phencyclidine (I) and analogs II and III were easily distinguished from IV by TLC on silica gel, using acetone–12 *N* ammonia (99:1), because they had R_f values of 0.66, 0.68, 0.55, and 0.49, respectively. It was difficult to distinguish III and IV with ethanol–5 *N* ammonia (9:1) (9), however, which gave R_f values of 0.70, 0.72, 0.63, and 0.65 for I–IV, respectively.

GLC of I–IV on 5% OV-7 at 150° gave peaks at 24.7, 24.6 (decomposition product at 3.8), 17.8, and 5.1 min, respectively, and the emergent material from IV was identified by mass spectrometry as the unchanged amine. Raising the column temperature resulted in their increasing decomposition to materials of shorter retention times, which have not been investigated.

It is possible that a cursory examination of IV by spot tests might lead to its reported (8) confusion with the commonly found phencyclidine. The IR spectra of the two bases are also quite similar in the fingerprint region, 1500–400 cm^{-1}. Therefore, the complete IR spectrum should be examined, and particular attention should be paid to the behavior at 2800 and 960 cm^{-1} where phencyclidine, but not IV, exhibits absorption bands. Proton NMR and mass spectrometry appear to be suitable for the unequivocal identification of these compounds (9).

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