Latinoam. Quim. 7, 83
8. Voirin, B. (1983) *Phytochemistry* **22**, 2107.
9. Jay, M., Gonnet, J. E., Wollenweber, E. and Voirin, B. (1975) *Phytochemistry* **14**, 1605.
10. Schmid, R. D. (1972) *Tetrahedron* **28**, 3259.
11 Schmid, R. D, Varenne, P. and Pàris, R. (1972) *Tetrahedron* **28**, 5037
12 Wagner, H. and Seligmann, O (1973) *Tetrahedron* **29**, 3029
13 Seshadri, T. R. (1962) *The Chemistry of Flavonoid Compounds* (Geissman, T A., ed) p 180 Pergamon Press, Oxford
14 Beirne, J J., Carroll, N. M, O'Sullivan, W. I and Woods, J (1975) *Tetrahedron* **31**, 265

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2,3-DIMETHYLNONACOSANE AND TROPANE ALKALOIDS FROM *HYOSCYAMUS ALBUS*

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Key Word Index—*Hyoscyamus albus*; Solanaceae; hyoscyamine, hyoscine; 2,3-dimethylnonacosane.

Abstract—In addition to hyoscine and hyoscyamine, a new compound isolated from the leaves and stems of *Hyoscyamus albus* has been characterized as 2,3-dimethylnonacosane by spectral studies.

INTRODUCTION

Hyoscyamus albus is known to contain tropane alkaloids [1, 2] which have mydriatic, anti-spasmodic and anticholinergic properties. This plant has also been reported to possess chlorogenic acid [3]. A re-investigation of this plant has shown it to contain a new branched hydrocarbon and a total crude alkaloid content of *ca* 0.24%, of which *ca* 40% consists of hyoscyamine, *ca* 30% of hyoscine and *ca* 15% of two unidentified bases.

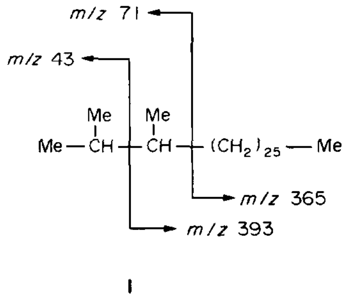
RESULTS AND DISCUSSION

Silica gel column chromatography of the *n*-hexane extract of the plant yielded a crystalline isolate. The isolate, mp 62°, had IR absorption bands at 2960, 2920, 2850, 1465, 720 (long chain) and 1375, 1360, 1150 cm^{-1} (isopropyl group) [4]. An $[M]^+$ ion at m/z 436 in the mass spectrum of the compound suggested the molecular formula $C_{31}H_{64}$. The formation of an ion at m/z 421 $[M-Me]^+$ indicated the compound to be of branched chain nature carrying a methyl group as substituent. The intensity of the C_nH_{2n+1} peaks after a maximum at $n = 4$, steadily declined up to $n = 25$, followed by a strong peak at $n = 26$ (m/z 365). From the intense peak at $n = 26$ $[M-71]^+$, it followed that one methyl group is attached at C-3 [5]. The other significant ions at m/z 393 and 43 indicated the presence of a second methyl group at C-2 which is also a part of the isopropyl group. These fragmentations are depicted in structure **1**. The ^{1}H NMR spectrum of the compound integrated for 64 protons. One of the terminal methyl groups was seen as a triplet ($J = 4$ Hz) at δ0.84 and the two methyls of the isopropyl function appeared as a doublet ($J = 6$ Hz) at δ0.80. The methyl group at C-3 was also present as a doublet ($J = 5$ Hz) at δ0.76. These data led us to establish the structure of this compound as 2,3-dimethylnonacosane (**1**).

The structure of compound **1** is in full agreement with the data now available. It has not been found previously in nature. It is interesting to note that the Solanaceae is a good source of branched hydrocarbons. Earlier they were reported from *Solanum torvum* [6] and *Duboisia myoporoides* [7]. Fractions obtained after **1** contained large amounts of oil, which was not worked up further.

EXPERIMENTAL

Mp is uncorr. The IR spectrum was recorded in KBr and the 90 MHz ^{1}H NMR spectrum in $CDCl_3$ with TMS as internal ref. TLC was carried out on silica gel G and the spots were visualized by exposure to I_2 vapour or Dragendorff spray. Plant material



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was cultivated at the experimental farm of this Institute and a voucher specimen has been deposited at the Botany Department.

Extraction and isolation. Dried and powdered leaves and stems (1.88 kg) of *H. albus* were extracted with *n*-hexane (6 × 4 l.). The hexane extract was concd to dryness (39.30 g) and was chromatographed over silica gel (1.2 kg, 60–120 mesh, BDH). Elution was carried out in hexane, hexane–$CHCl_3$ (9:1, 4:1, 7:3, 1:1, 1:3) and $CHCl_3$. Fractions of 250 ml each were collected and monitored by TLC.

2,3-*Dimethylnonacosane* (**1**). Removal of solvent from hexane fractions (1–8) provided a residue (2.10 g), mp 62° (hexane). IR ν_{max} cm^{-1}: 2960, 2920, 2850, 1465, 1375, 1360, 1150, 720. ^1H NMR: δ0.84 (3H, *t*, $J = 4$ Hz, H_3-29), 0.80 (6H, *d*, $J = 6$ Hz, isopropyl group), 0.76 (3H, *d*, $J = 5$ Hz, H_3-3), 1.86 (1H, *m*, H-3), 1.62 (1H, *m*, H-2), 1.22 (50H, *br s*, $(CH_2)_{25}$). MS *m/z* (rel. int.): 436 $[M]^+$ ($C_{31}H_{64}$) (1.9), 421 $[M - Me]^+$ (1.6), 393 (9), 365 (8), 351 (1), 337 (2), 323 (2), 309 (3), 295 (3), 281 (3), 267 (5), 253 (5), 239 (5), 225 (5), 211 (6), 197 (7), 183 (8), 169 (9), 155 (11), 141 (12), 127 (18), 113 (20), 99 (28), 85 (55), 71 (78), 57 (100), 55 (20), 43 (58).

Isolation of alkaloids. The material left after the above hexane extraction was extracted with EtOH (5 × 4 l.) and the EtOH extract was concd to dryness. The residue was treated with HCl (2%, 6 × 100 ml) and filtered. The filtrate was extracted with hexane (2 × 300 ml). Hexane-insoluble material was basified with NH_4OH and extracted with $CHCl_3$ (6 × 300 ml, 4.40 g, 0.24%). The alkaloid mixture was again converted into hydrochlorides and the pH of this soln was adjusted to 6 by the gradual addition of a saturated soln of $NaHCO_3$. At this pH, two unidentified bases (0.66 g) were extracted in $CHCl_3$ (6 × 50 ml); at pH 6.5, hyoscine (1.32 g) was extracted in $CHCl_3$ (6 × 50 ml); and towards higher pH 8–11, hyoscyamine (1.76 g) was extracted in $CHCl_3$ (6 × 50 ml). Hyoscine and hyoscyamine were identified by direct comparison with authentic specimens.

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REFERENCES

1. Cromwell, B. T. (1955) in *Modern Methods of Plant Analysis* (Paech, K. and Tracey, M. V., eds.), Vol. IV, p. 496. Springer, Berlin.
2. Ghani, A., Evans, W. C. and Wooley, V. A. (1972) *Bangladesh Pharm. J.* **1**, 12.
3. Politis, J. (1948) *C. R. Acad. Sci.* **226**, 692.
4. West, W. (1967) *Technique of Organic Chemistry*, Vol. 9, p. 351. Interscience, New York.
5. Bentley, K. W. (1963) *Elucidation of Structure by Physical and Chemical Methods*, Part I, p. 291. Interscience, New York.
6. Mahmood, U., Shukla, Y. N. and Thakur, R. S. (1983) *Phytochemistry* **22**, 167.
7. Shukla, Y. N. and Thakur, R. S. (1984) *Phytochemistry* **23**, 799.