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CACTACEAE

ISOLATION OF CANDICINE FROM *TRICHOCEREUS CHILENSIS*

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Trichocereus chilensis (Colla) Br. & R. is the only Chilean member of its genus figuring in the literature as having been examined for alkaloids, although previous reports are negative. Djerassi and coworkers[1] found no alkaloids in this cactus. Agurell,[2] who specifies that no attempt was made to detect quaternary bases and discusses the sensitivity of his methods for non-quaternary amines, also reports no alkaloids in detectable amounts in *T. chilensis*. In the present communication we wish to report on the presence of both quaternary and non-quaternary bases in this species.

Due to extremely low yields, only candicine was isolated and identified as its iodide, following Castrillón's method.[3] TLC of the chloroform-soluble material, containing large amounts of non-alkaloidal substances, showed five Dragendorff-positive spots; the separation and structural assignment of these alkaloids is now being attempted. The candicine iodide was analyzed for C, H and N, and both its analysis and its m.p. are in full accordance with Reti's data.[4] Mixed m.p. with an authentic sample and comparison of the IR spectra confirmed our identification.

EXPERIMENTAL

M.ps were taken on a Kofler block and are uncorrected. For TLC silica gel G (Merck) plates (0·25 mm) were used, developing with $CHCl_3$–*n*-BuOH–NH_4OH (50:50:2·5) (System 1) or $CHCl_3$–EtOH (1:1) (System 2). Whole cacti (aerial part) were collected in September 1970 in Mallarauco Pass, near Santiago, sliced, air-dried and milled.

Non-quaternary bases. T. chilensis powder was extracted (Soxhlet) with ethanol. The ethanol solution was concentrated *in vacuo* and the sticky, dark-coloured syrup was taken up in 5% HCl and extracted continously with ethyl ether until no colour was noticeable in the extract. The aqueous layer was filtered, basified with NH_4OH, and extracted with $CHCl_3$. The combined $CHCl_3$ extracts were dried over Na_2SO_4 and evaporated under reduced pressure yielding a dark-brown residue (3 g) which was examined by TLC (System 1) showing five Dragendorff-positive spots.

Candicine iodide. The $CHCl_3$-extracted aqueous phase was acidified with HCl and filtered, and excess Mayer's reagent was added. After standing overnight at room temp., the precipitate was filtered off, dissolved in hot water, and decomposed with H_2S. The hot solution was filtered and concentrated to a final volume of approximately 10 ml. After standing 20 hr a pale yellow precipitate (100 mg), m.p. 227–231°, was collected. This salt was recrystallized from hot water and decolourized with charcoal, furnishing 60 mg colourless crystals, m.p. 232–233°. (Found: C, 42·94; H, 5·88; N, 4·62%. (Calc. for $C_{11}H_{18}INO$: C, 42·85; H, 5·86;

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[1] C. DJERASSI, A. BOWERS, S. BURSTEIN, H. ESTRADA, J. GROSSMAN, J. HERRÁN, A. J. LEMIN, A. MANJARREZ and C. PAKRASHI, *J. Am. Chem. Soc.* **78**, 2312 (1956).

[2] S. AGURELL, *Lloydia* **32**, 206 (1969).

[3] J. CASTRILLÓN, *Dissertation*, University of Buenos Aires (1950).

[4] L. RETI, in *The Alkaloids* (edited by R. H. F. MANSKE and H. L. HOLMES), Vol. III, p. 321, Academic Press, New York (1953).

N, 4·56%.) No m.p. depression was observed upon admixture with authentic candicine iodide. R_fs (System 2) and IR spectra (KBr discs) were identical, with strong absorption at 3300–3200 (phenolic O–H), 1220 (amine C–N), 1200 (C–OH), and 835 cm^{-1} (1,4-disubstituted benzene ring).

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Key Word Index—*Trichocereus chilensis;* Cactaceae; alkaloids; candicine.

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COMPOSITAE

TERPENOIDS OF *CENTAUREA AMERICANA**

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Plant. Centaurea americana. Uses. In folk medicine. *Previous work.* On sister species.[1–6] *Aerial parts.* From 700 g of the aerial part of *C. americana*: 21 g of resinous material were extracted with $CHCl_3$, this solution was chromatographed on an inverted column. The elution of each fraction was with successive portions of $CHCl_3$, acetone and ethanol. The NMR of the $CHCl_3$ extract exhibited the typical signals of a germanicrol type of sesquiterpenelactone. The following compounds were obtained.

β-Sitosterol. Identified by mixed m.p. IR, UV, NMR and co-TLC, of the sterol and its acetate. Acetate of a triterpene of the β-amyrin type; $C_{32}H_{52}O_2$ (M^+ 468 by mass spectroscopy). Found, C, 80·18; H, 10·84; O, 8·18; Calcd. C, 81·99; H, 11·18, O, 6·83, m.p. 183°, $[\alpha]_{589}$ + 84·5°; $[\alpha]_{546}$ + 102·4°; $[\alpha]_{436}$ + 176·4°; (Chl) L-B negative, TNM; positive, IR, 2940, 2880, 1740, 1630, 1460, 1440, 1375–1365, $(CH_3)_2C$), 1250, 1020, cm^{-1}. On saponification, alcohol, m.p. 138–140°. $[\alpha]_{589}$ +28·8; $[\alpha]_{578}$ + 34·8, $[\alpha]_{546}$ + 37·8 (Chl) IR, 2920, 2840, 1630, 1450; 1380, 1360, 1040 cm^{-1}. On oxidation of the alcohol, a ketone was obtained, m.p. 222–226°. IR. 2920, 2850. 1690 (C=O) 1460, 1385 and 1375 cm^{-1}.

New compound: $C_{28}H_{38}O_{10}$. Found, C, 62·65; H, 6·65; H, 6·92; O, 30·14; Calcd. C, 62·91; H, 7·17; O, 29·93. m.p. 355–360°, IR, 3450, 2930, 2850, 1755, 1720, 1640, 1450, 1430, 1390, 1260, 1145, 1050–1010, 950, 900, 800, 750 cm^{-1}. NMR, 6·6 (m) S.

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[1] R. HEGNAUER, *Chemotaxonomie der Pflanzen*, Vol. 3, p. 502, Birkhauser Verlag, Basel (1964).
[2] F. BOHLMANN and C. ZDERO, *Tetrahedron Letters* 3239 (1967).
[3] J. L. BRETON, A. G. GONZÁLEZ and S. O. RUIZ, *An Quím.* (*Spain*) **63**, 703 (1967).
[4] Z. F. AHMED, F. M. HAMMANDA, A. M. RUZK and S. I. ISMAIL, *Planta Med.* **18**, 227 (1970).
[5] J. L. BRETON, A. G. GONZÁLEZ and M. R. RINCONES, *An. Quím.* (*Spain*) **65**, 297 (1969).
[6] J. L. BRETON, D. G. MARRERO and A. G. GONZÁLEZ, *An. Quím.* (*Spain*) **64**, 187 (1968).