

192. Studies on Argentine Plants. Part II. Aspidospermine from *Vallesia glabra* and *Vallesia dichotoma*.

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The alkaloid occurring in *V. glabra* and *V. dichotoma* has been identified as aspidospermine.

MAININI (Thesis, Buenos Aires, 1904) isolated from *V. glabra* ("ancoche"), an alkaloid, vallesine, which was first obtained in a crystalline form, melting at 184—185°, by Stuckert (Inv. Lab. Quím. Biol., Córdoba, 1938). Hartmann and Schlittler (*Helv. Chim. Acta*, 1939, **22**, 547) identified it with aspidospermine, also present in the bark of the "quebracho blanco" tree. We had already arrived at the same conclusion in an independent way (Deulofeu, Labriola, and De Langhe, *Rev. Inst. Bact.*, 1939, **9**, 224) and have now shown that the alkaloid found in *V. dichotoma* by Cárcamo (*Bol. Soc. Quím. Perú*, 1936, **2**, 25) is also identical with aspidospermine.

The identification in both cases was effected by transformation of the alkaloid into deacetylaspidospermine and aspidosine by Ewins' method (J., 1914, **105**, 2738). The alkaloid was isolated from the leaves and branches. Extracts from the root of *V. glabra*, though giving a strong alkaloidal reaction, could not be induced to crystallise.

EXPERIMENTAL.

Preparation of Aspidospermine.—The preparation from the leaves of *V. glabra* is described as an example. The same method applies to the branches. 3 Kg. of leaves were extracted with 20 l. of water containing 5% of tartaric acid (when hydrochloric acid was used, no crystals were obtained). After 8 days the solution was filtered and the extraction was repeated with 3% tartaric acid solution (20 l.) and again with a 2% solution (20 l.). The united extracts were evaporated to 6 l. and shaken with ether, non-basic substances thus being partially eliminated. The aqueous solution was then made alkaline with sodium carbonate and extracted with ether. After being washed with water, the ether was evaporated, and the gummy residue dried in a desiccator and dissolved in alcohol; the aspidospermine crystallised readily, m. p. 204° after recrystallisation from alcohol. The alkaloid was freed from a minute quantity of non-basic substances by solution in water acidified with tartaric acid and shaking with ether; after recovery from the aqueous solution, made alkaline with sodium carbonate, by ether extraction it had m. p. 207—208°, $[\alpha]_D^{20}$ —91.1° (c 1.48) in chloroform (Found: C, 74.85; H, 8.5; N, 7.8;

OMe, 8.5. Calc. for $C_{22}H_{30}O_2N_2$: C, 74.6; H, 8.65; N, 7.9; OMe, 8.75%). The literature gives m. p. 208° and $[\alpha]_D -93^\circ$.

The yield of aspidospermine per kg. was 3.6—3.9 g. from the leaves and 2 g. from the branches. From *V. dichotoma* a yield of about 4 g. per kg. was obtained.

Deacetylaspidospermine was prepared according to Ewins (*loc. cit.*) by the action of hydrochloric acid on aspidospermine; it formed fine needles, m. p. $108-109^\circ$, from aqueous acetone (Ewins gives $110-111^\circ$). Aspidosine, prepared from aspidospermine by the action of hydriodic acid, separated from alcohol in prisms, m. p. 245° (Ewins gives $244-245^\circ$).

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