ISOLATION OF *NN*-DIMETHYL 4-METHOXYPHENYLETHYLAMINE FROM *TECLEA SIMPLICIFOLIA**

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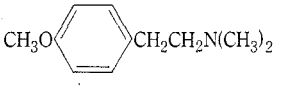
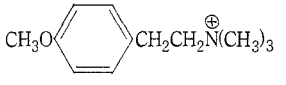
Teclea simplicifolia is found in tropical Africa, and grows to a height of about 60 ft. It is a member of the family Rutaceae.

Extraction of the finely ground bark with alcohol gave the triterpene lupeol, and a mixture of bases which was separated into a chloroform-soluble fraction and a water-soluble fraction. The chloroform-soluble fraction gave a crude alkaloid having an absorption spectrum similar to those of the furoquinoline alkaloids;[1] but the pure alkaloid could not be obtained.

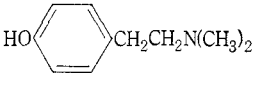
Purification of the water-soluble fraction gave a pure alkaloid which has been identified as *NN*-dimethyl 4-methoxyphenylethylamine (I). Its structure was established by physical methods, and by conversion into the methiodide (II). This was shown to be identical with a specimen prepared by methylation of *NN*-dimethyl 4-hydroxyphenylethylamine (hordenine, III). It is of some interest that although many phenylethylamine alkaloids have been reported in the literature,[2] *NN*-dimethyl 4-methoxyphenylethylamine has not previously been obtained.



(I)



(II)



(III)

Experimental

Extraction.—The finely ground bark (5 kg) was extracted at room temperature with methylated spirit (12 l.) for 3 days. The solution was concentrated to 500 ml and the resulting solid collected and recrystallized from aqueous ethanol to give lupeol, m.p. 214°C (lit. 215°C); its acetate had m.p. 222°C (lit. 218°C) and benzoate, m.p. 274°C (lit. 273–274°C).

The filtrate was exaporated to dryness and the residue extracted with dilute HCl (10×200 ml). The resulting acid solution was extracted with chloroform (3×100 ml) and the aqueous layer then brought to pH 8 by addition of Na_2CO_3. A small amount of tar was removed. Excess ammonium reineckate was added and the precipitate (80 g) collected and dissolved in a mixture of 50% aqueous acetone (2 l.) and 10% HCl (50 ml). The solution was extracted with ether (1 l., then 10×200 ml) until the ether layer was no longer pink, and the aqueous layer then evaporated to dryness. The residue was then extracted with absolute ethanol (300 ml), the extract filtered, and then evaporated to give the salt (8 g). Recrystallization from absolute ethanol gave NN-*dimethyl 4-methoxyphenylethylamine hydrochloride* as colourless blades, m.p. 279–280°C (decomp.) (Found: C, 61·1; H, 8·3; N, 6·2; Cl, 16·9, 16·4; active H, 0·23 (30°C), 0·29% (95°C). $C_{11}H_{18}ClNO$ requires C, 61·2; H, 8·4; N, 6·5; Cl, 16·5; 1 active H, 0·46%). The

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[1] Lahey, F. N., Lamberton, J. A., and Price, J. R. (1950).—*Aust. J. Sci. Res.* A **3**: 155.

[2] Reti, L. (1953).—In "The Alkaloids." Vol. 3. (Eds. R. H. F. Manske & H. L. Holmes.) (Academic Press: New York.)

picrate crystallized from ethanol in yellow prisms, m.p. 164–165°C (Found: C, 50·3; H, 4·9; O, 31·1%. $C_{17}H_{20}O_8N_4$ requires C, 50·0; H, 4·9; O, 31·3%). The *reineckate* crystallized from aqueous acetone and had m.p. 173°C (Found: C, 36·2; H, 5·1%. $C_{15}H_{24}CrN_7OS_4$ requires C, 36·1; H, 4·85%). The n.m.r. spectrum of the hydrochloride in D_2O showed a complex set of bands at $\tau = 6{\cdot}8$ (which integrated for 14 protons) assigned to the CH_2, OCH_3, and NCH_3 protons, and two doublets at $\tau = 2{\cdot}7$, 2·85, 3·03, and 3·2 (which integrated for 4 protons) characteristic of a *p*-substituted benzene derivative. Its infrared spectrum was also consistent with this structure.

The chloroform extract of the HCl solution was evaporated to dryness to give crude alkaloid (2·0 g). Attempts to purify this base have not been successful.

NN-*Dimethyl 4-Methoxyphenylethylamine Methiodide.*—(i) A mixture of *NN*-dimethyl 4-hydroxyphenylethylamine sulphate (≡ hordenine sulphate, 0·3 g), K_2CO_3 (0·5 g), acetone (10 ml), water (2 ml), and methyl iodide (2·5 g) was refluxed for 8 hr. The cooled mixture was filtered, the filtrate evaporated, and the residue recrystallized from ethanol. NN-*Dimethyl 4-methoxyphenylethylamine methiodide* formed colourless prisms, m.p. 204–206°C (Found: C, 44·9; H, 6·3; N, 4·2%. $C_{12}H_{20}ION$ requires C, 44·9; H, 6·3; N, 4·4%). The same methiodide was also obtained following methylation with methyl sulphate and conversion to the iodide on an ion-exchange column.

(ii) A suspension of $AgCO_3$ (0·1 g) in warm water (15 ml) was added to a solution of *NN*-dimethyl 4-methoxyphenylethylamine hydrochloride (0·1 g) in ethanol (20 ml), and the mixture shaken for a few minutes and filtered. Methyl iodide (3 ml) was added to the filtrate which was then refluxed for 1 hr. Ether was added to precipitate the product which was recrystallized from ethanol to give prisms, m.p. 204–206°C, alone or admixed with an authentic specimen prepared as above.

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