

ISOLATION AND STRUCTURE OF A NEW ALKALOID NOR-NERONINE FROM  
THE BULBS OF PANCRATIUM LONGIFLORUM ROXB.\*

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The last decade and half has seen intense activity in the field of chemistry of alkaloids of the plant family Amaryllidaceae. Pancreatium longiflorum Roxb. is one of the rare cases which has escaped attention so far. From the bulbs of this plant growing in Waltair (India) we have isolated a new alkaloid which contains a phenolic hydroxyl group and gives neronine with diazomethane. It is therefore named nor-neronine.

The minced (fresh) bulbs were extracted with alcohol, the combined alcoholic extract partially concentrated, shaken with freshly precipitated lead hydroxide (compare ref.1) and filtered. The reaction of the filtrate was adjusted to pH 6 and the solvent removed under reduced pressure with progressive replacement by water. The predominantly aqueous residue (pH 6) was extracted successively

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\* These results formed part of a talk delivered by the first author at an International Colloquium organised by CNRS, Paris at Noumea in April-May, 1964.

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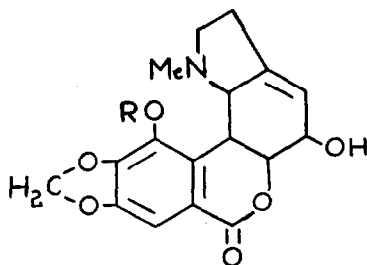
with petroleum ether, ether, chloroform and chloroform-alcohol 2:1. The last two extracts gave colourless needles m.p. 238-40° on crystallisation from chloroform-methanol and then from alcohol (yield, 0.005%).

The main aqueous extract was adjusted to pH 9 by the addition of solid potassium carbonate and extracted with chloroform and chloroform-alcohol 2:1. These two extracts yielded colourless prisms from chloroform-alcohol, m.p. 272-74° (decomp.) (yield, 0.01%); formula  $C_{16}H_{17}NO_4$ ,  $[\alpha]_D^{30} -105.7^\circ$  (absolute alcohol). It gave a positive test for the methylenedioxy group (gallic acid-sulphuric acid test) and formed a hydrochloride m.p. 224-26° (decomp.). With pyridine-acetic anhydride it formed a diacetate  $C_{20}H_{21}NO_6$ , m.p. 216-18°,  $[\alpha]_D^{30} +25.0^\circ$  (chloroform); mixed m.p. with authentic lycorine diacetate from Crinum defixum<sup>2</sup> was undepressed. The properties and composition of the parent compound and its derivatives established its identity as lycorine.

The substance m.p. 238-40° (decomp.) mentioned earlier analysed for  $C_{17}H_{17}NO_6$ ,  $[\alpha]_D^{30} +90^\circ$  (methanol). It gave tests for phenolic (blue violet ferric colour) and methylenedioxy groups (I.R. absorption at  $926\text{ cm}^{-1}$  and positive gallic acid-sulphuric acid test). With sodium acetate and acetic anhydride it gave a diacetate, colourless needles from acetone,  $C_{21}H_{21}NO_8$ , m.p. 248-50° (decomp.),  $[\alpha]_D^{30} +20^\circ$  (chloroform). These properties indicated that it was a new

\* All the compounds whose formulae are given in this communication analysed correctly for C, H and N (and -OCH<sub>3</sub> wherever this applies).

alkaloid. Its I.R. spectrum indicated the presence of a  $\delta$ -lactone ring (strong absorption at  $1710\text{ cm}^{-1}$ ). Treatment of the substance in methanol with ethereal diazomethane gave a product, colourless prisms from ethyl acetate, m.p.  $194-96^\circ$ ,  $[\alpha]_D^{30} +154.6^\circ$  (chloroform), which analysed for  $C_{18}H_{19}NO_6$ , contained one methoxyl group (micro-Zeisel), was not phenolic (negative ferric colour) and contained hydroxyl function (absorption at  $3520-3510\text{ cm}^{-1}$  in the I.R.). This methyl ether was extremely hygroscopic and formed a methiodide,  $C_{18}H_{19}NO_6 \cdot CH_3I$ , colourless prisms from methanol, m.p.  $265-68^\circ$  (decomp.). The properties and composition of the methyl ether and its methiodide correspond to those described in the literature<sup>3</sup> for the amaryllidaceous alkaloid neronine and its methiodide. Hence the new phenolic substance which gives the nonphenolic neronine with diazomethane is called nor-neronine (see also ref. 4 and 5).



R = H, Nor-neronine

R = Me, Neronine

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