

Narcissine : an alkaloid from the bulb of the common daffodil (Narcissus pseudonarcissus) / by Arthur James Ewins.

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NARCISSINE:
AN ALKALOID FROM THE BULB OF
THE COMMON DAFFODIL
(NARCISSUS PSEUDONARCISSUS)

BY

ARTHUR JAMES EWINS, B.SC.

(From the "Transactions of the Chemical Society," Vol. xcvi, 1910)



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CCXLV.—*Narcissine: an Alkaloid from the Bulb of the Common Daffodil (Narcissus pseudonarcissus).*

By ARTHUR JAMES EWINS.

IN 1878 a paper was published by Ringer and Morshead (*J. Physiol.* 1, 437), entitled "On the physiological action of narcissia, an alkaloid obtained from the bulb of the common daffodil (*Narcissus pseudonarcissus*).” This work contained a detailed account of experiments carried out on men and frogs with an alkaloid or with extracts containing the alkaloid, which had been obtained by Gerrard from the bulb of the common daffodil. From experiments carried out with extracts obtained from bulbs in the resting stage and from bulbs of the flowering plants, the authors concluded that the action of the alkaloid present in the extracts from the resting bulbs was similar to that of pilocarpine, whereas that present in the extracts from the bulbs of the flowering plants closely resembled atropine in action. The alkaloid obtained by Gerrard from both extracts nevertheless appeared to be the same in general chemical properties.

In view of these statements and of the fact that no reference to "narcissia" or to any alkaloid obtained from the bulbs of the daffodil can be found in chemical literature, it appeared to be of interest to obtain the alkaloid in a pure state, in order that a more detailed investigation of its chemical and physiological properties might be made, more especially as the alkaloids which have been obtained from monocotyledons are comparatively few in number.

The bulbs of a cultivated variety of the daffodil (*Narcissus princeps*), being more readily obtainable than the variety mentioned above, were first employed in the investigation. From these bulbs, however, the extracts obtained from the resting or flowering bulbs gave only traces of alkaloidal reactions, and the isolation of the alkaloid was obviously hopeless. With the bulbs of the wild daffodil (*Narcissus pseudonarcissus*), much more satisfactory results were obtained. From these a crystalline alkaloid was readily obtained. From the resting bulbs a yield of approximately 0.2 per cent. of the dried material, and from the flowering bulbs only about 0.1 per cent., was obtained. The alkaloid, which, in accordance with modern usage, it is suggested be called "narcissine," rather than "narcissia," was the same in both cases.

Narcissine has been found by analyses and molecular-weight determination to possess the formula $C_{16}H_{17}O_4N$. The alkaloid is characterised by very great stability, and on that account and owing to the small amount of material available (about 3 grams only), no light has been thrown on its probable constitution. The nitrogen present is tertiary, since nitrous acid is without action on the alkaloid, and treatment with methyl iodide produced an amorphous product which was probably the methiodide of the base, but which could not be crystallised for analysis. The action of hydriodic acid (Zeisel) showed the absence of methoxy-groups, but on very strongly heating, a very small amount of methyl iodide was evolved, and the residue on suitable treatment yielded a solution which gave a violet coloration with ferric chloride, the phenolic substance being extracted from its acid solution by ether. The amount of substance so obtained was, however, extremely small, and further attempts to hydrolyse by means of acids yielded no crystalline product.

Fusion with potassium hydroxide yielded no better results. At temperatures up to 220° the alkaloid was only very slowly attacked, traces of alkaline vapour being evolved. Heating with the naked flame for one and a-quarter hours was required in order to decompose completely about 0.7 gram of the alkaloid. The reaction product dissolved in water gave on addition of ferric chloride a violet coloration, quickly passing to a dirty brown, with separation of a

brown, flocculent precipitate. This polyphenolic substance, again, could be extracted by ether from its acid solution, but on evaporating off the solvent only a very small quantity of a brown, amorphous product was obtained, and all attempts to obtain a crystalline product were fruitless.

On account of the relatively large number of oxygen atoms present in the molecule, it was thought possible that a carboxyl group might be present, but attempted esterification showed the absence of such grouping. For this reason and from the absence of methoxy-groups as shown by the Zeisel reaction, the formation of a polyphenolic derivative such as has been described is very possibly due to the presence in the molecule of a methylenedioxy-grouping and a phenolic bridge oxygen. The stability of the alkaloid would seem to support such a view, although there is, of course, no direct evidence of such structure.

Experiments with regard to the physiological action of the alkaloid were carried out in these laboratories by Dr. P. P. Laidlaw, to whom I am indebted for the following account of its action. As tested on frogs and cats, the alkaloid showed no action in any way similar to that of pilocarpine or of atropine. 0.125 Gram, given by mouth to a cat, caused nausea, vomiting, salivation, and purgation. The salivation was not, however, similar to that produced by pilocarpine, since it could not be produced on the anæsthetised animal.

EXPERIMENTAL.

Preparation of the Alkaloid.

Two thousand five hundred bulbs of the common daffodil (*Narcissus pseudonarcissus*), weighing approximately 4 kilos., were dried at a temperature of about 40°. The weight of the dried product was 1400 grams. The substance was finely ground and extracted for about six hours with hot alcohol (97 per cent.), and the alcoholic extract evaporated to about 200 c.c. To the dark syrupy, acid liquid was added an equal volume of water, and a very dark resinous precipitate which formed was collected. This precipitate was re-suspended in a little very dilute acid, and again collected after thoroughly shaking. The filtrate and washings were combined, and the acid liquid extracted twice with about one-third of its volume of ether. The aqueous solution was then rendered alkaline by addition of sodium carbonate, when, after some time, a precipitate formed, which consisted mainly of bunched prisms. The crystals were collected, and after recrystallisation from 90 per cent. alcohol were obtained as colourless, short, stout prisms, melting at 266—267° (bath at 250° at commencement of heating). The substance was dried first in air, then in a vacuum over sulphuric acid,

and finally at 110° , no alteration in weight taking place under these varying conditions:

0.1448 gave 0.3552 CO_2 and 0.0754 H_2O . $\text{C}=66.9$; $\text{H}=5.8$.

0.1283 „ 0.3159 CO_2 „ 0.0684 H_2O . $\text{C}=67.1$; $\text{H}=5.9$.

0.1051 „ 5.0 c.c. N_2 (moist) at 13.5° and 732 mm. $\text{N}=5.4$.

$\text{C}_{16}\text{H}_{17}\text{O}_4\text{N}$ requires $\text{C}=66.9$; $\text{H}=5.9$; $\text{N}=5.0$ per cent.

A determination of the molecular weight was made by Barger's microscopic method (Trans., 1904, **86**, 286) in glacial acetic acid. 0.060 Gram, in 1.197 grams of solvent, was intermediate between 0.19 mol. and 0.20 mol., whence $\text{M.W.}=257$. $\text{C}_{16}\text{H}_{17}\text{O}_4\text{N}$ requires $\text{M.W.}=287$.

Properties of Narcissine.—The alkaloid, as before stated, is obtained by recrystallisation from alcohol in colourless prisms, which melt at 266 — 267° with some decomposition and formation of a red liquid. The crystals are insoluble in water or dilute sodium hydroxide, but readily soluble in dilute acid. The acid solution gives all the usual alkaloidal reactions, for example, with Meyer's reagent, with a solution of iodine in potassium iodide, and with phosphotungstic acid. The crystalline product is only very sparingly soluble in methyl alcohol, ethyl alcohol, ethyl acetate, or acetone, moderately so in pyridine, nitrobenzene, or glacial acetic acid, and insoluble in ether or chloroform. A determination of its solubility in absolute ethyl alcohol showed that one part by weight of the alkaloid was soluble in 284 parts of boiling alcohol and in 340 parts of alcohol at 15° .

The alkaloid is laevorotatory. 0.166 Gram, made up to 100 c.c. with absolute alcohol, gave, in a 2.2-dcm. tube at 10° , $\alpha_D -0.35^{\circ}$, whence $[\alpha]_D^{10} -95.8^{\circ}$.

The alkaloid dissolves in concentrated sulphuric acid, producing a deep red solution, which slowly becomes reddish-brown on keeping.

Narcissine Hydrochloride, $\text{C}_{16}\text{H}_{17}\text{O}_4\text{N} \cdot \text{HCl}$.—0.3 Gram of crude narcissine was dissolved in dilute hydrochloric acid, and the solution evaporated to dryness over potassium hydroxide in a desiccator. The resulting crystalline product was dissolved in hot 90 per cent. alcohol, and after treatment with a little blood-charcoal, the solution, on cooling, deposited the hydrochloride in long, thin prisms, melting at 198 — 199° . Yield, 0.27 gram:

0.2737 gave 0.1176 AgCl . $\text{Cl}=10.6$.

$\text{C}_{16}\text{H}_{17}\text{O}_4\text{N} \cdot \text{HCl}$ requires $\text{Cl}=10.9$ per cent.

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