University of Queensland

PAPERS

DEPARTMENT OF CHEMISTRY

Volume I 1947 Number 30

Alkaloids of Queensland Flora

BY

I. R. C. BICK, B.A., M.Sc., A.A.C.I., and T. G. WHALLEY, B.Sc.

Price : One Shilling.

PUBLISHED AS AN ORIGINAL PAPER BY THE UNIVERSITY OF QUEENSLAND

DATE OF PUBLICATION :
6th JUNE, 1947.

DEPARTMENT OF CHEMISTRY.

VOLUME I. 1947. NUMBER 30.

ALKALOIDS OF QUEENSLAND FLORA.

PART II.—ALKALOIDS OF DAPHNANDRA DIELSII.

By

I. R. C. BICK, B.A., M.Sc., A.A.C.I.; and T. G. WHALLEY, B.Sc.

Chemistry Department, University of Queensland.

[Published as an original paper by the University of Queensland.]

DATE OF PUBLICATION:
6th JUNE, 1947.

A. H. TUCKER, Government Printer, Brisbane.

ALKALOIDS OF QUEENSLAND FLORA.

Part II.—Alkaloids of Daphnandra Dielsii.

By I. R. C. Bick, B.A., M.Sc., A.A.C.I.; and T. G. Whalley, B.Sc.

In the first paper of this series (1), dealing with the alkaloids of *Daphnandra repandula*, the authors described the isolation and characterisation of two new alkaloids, repanduline and repandine. In view of the close botanical relationship between *D. repandula* and *D. Dielsii*, the bark of the latter has been investigated, in order to discover whether the same alkaloids occur in both species. The present paper describes the isolation of a yellow crystalline alkaloid from the bark of *D. Dielsii* and its identification as repanduline. Contrary to expectation no indication of the presence of repandine was found.

D. Dielsii, Perkins, of the family Monimiaceae, is closely allied to *D. repandula* but differs in being hairy on the underside of the leaves. This species is locally termed "yellow-wood." The bark which is about three eighths of an inch thick, is bright yellow in colour when cut, and is practically odourless. It has a bitter taste and gives strong alkaloid tests.

The total alkaloid content was determined by the method previously described (1), the figure amounting to about 5½% of the air-dried bark. This value is comparable with that found by the authors in *D. repandula* and by Pyman (2) in *D. micrantha*.

The bark used in this investigation was collected on the Atherton Tableland, North Queensland, by Mr. L. J. Webb and was air dried for twenty-four hours at a temperature not more than 66°C. A sample has been deposited in the C.S.I.R. Herbarium, Canberra. (No. 1061).

EXPERIMENTAL.

Isolation of the Alkaloid.

500 grams of the finely-ground air-dried bark was exhaustively extracted with ethyl alcohol in a glass extractor. The extraction was carried out under reduced pressure at about 47°C in order to minimise resinification of repanduline which occurs very readily at higher temperatures.

The alcoholic extract was evaporated down under reduced pressure to a small bulk. A quantity of water was added, the solution again distilled to remove the remaining alcohol, and then acidified with ½% hydrochloric acid. After allowing the solution to stand, the insoluble resinous material was filtered off and washed free of alkaloid with more dilute acid, the acid washings being added to the main extract.

The acid solution was basified with aqueous ammonia which produced a thick yellow precipitate of crude amorphous alkaloid. The mixture was extracted with chloroform by shaking in a separating funnel until the chloroform extracts gave fairly weak alkaloid tests. The extracted ammoniacal solution still gave good alkaloid tests. On carefully neutralising this solution with dilute hydrochloric acid, no precipitate resulted, indicating the possible presence of a water-soluble alkaloid; however, none has so far been isolated in a pure state. The chloroform solution was extracted with sodium hydroxide (8%) which removed some colouring matter. The extract on neutralising gave no precipitate but gave alkaloid tests, and thus if a phenolic alkaloid is present, it must be water-soluble.

The extracted chloroform was then dried over anhydrous sodium sulphate and evaporated to a syrup under reduced pressure (250 mms.). On addition of a little ethyl alcohol and ethyl acetate the solution yielded a heavy precipitate of yellow needles. The yield of crystalline alkaloid obtained on cooling and filtering the mixture was about 4·25 grams. The filtrate, on evaporation and further addition of alcohol and ethyl acetate, yielded another ·5 gram of crystals. This crude material consisted almost entirely of repanduline.

In order to separate any repandine which might be present, the crude material was eluted several times with small quantities of cold benzene, which dissolves repanduline readily, but not repandine. No indication of any repandine was obtained by this method nor during the subsequent recrystallisations.

The crude alkaloid was recrystallised from ethyl acetate, and then from dry ether. The pure product gave the colour tests for repanduline (1).

As repanduline decomposes without melting, the specific rotation of the purified base was determined in benzene solution:

$$\alpha_D^{23} = + 5{\cdot}22^\circ ; \; C = {\cdot}5012 ; \; l = 2 \text{ dcm} ; \; [\alpha]_D^{23} = + 521^\circ$$

The specific rotation of authentic repanduline determined in benzene solution at 19°C was found to be + 525°.

Thus it may be concluded that the bark of *D. Dielsii* is a rich source of repanduline, the yield of crude alkaloid being about 1% of the air-dried bark, and that no repandine is present.

ACKNOWLEDGEMENTS.

The authors wish to thank the Council for Scientific and Industrial Research and Mr. L. J. Webb for making available the bark of *Daphnandra Dielsii*.

The investigation was carried out with the aid of a research grant from the Council for Scientific and Industrial Research.

The authors also wish to express their thanks to Professor T. G. H. Jones for his encouragement in this work.

REFERENCES.

(1) BICK, I. R. C. and WHALLEY, T. G., Univ. of Q'ld. Papers, Chem. Dept. Vol. 1, No. 28. (1946).

(2) PYMAN, F L., Trans. Chem. Society 105, 1679 (1914).

A. H. TUCKER, Government Printer, Brisbane.