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SHORT COMMUNICATION

ISORHAMNETIN 3-*O*-RUTINOSIDE, THE FLAVONOID PIGMENT IN *BATIS MARITIMA*

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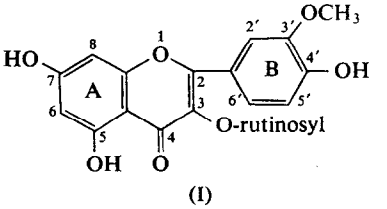
Abstract—Isorhamnetin-3-*O*-rutinoside has been shown to be the only flavonoid in *Batis maritima*.

INTRODUCTION

IN CONNECTION with the taxonomic affinities of the family Batidaceae, we previously discussed[1] the systematic significance of the absence of betalains[2] in *Batis maritima*, one of two species which comprise the family. We have continued our chemotaxonomic investigation of *B. maritima* and now describe its flavonoid content. Several collections were made on Galveston Island, Texas in 1964 and 1965 and no qualitative difference in their flavonoid composition was observed. A single flavonoid spot was detected upon two dimensional paper chromatography of a methanol extract of dried plant material in a 3:1:1 mixture of *t*-butanol, acetic acid and water in the first dimension, and 15% aqueous acetic acid in the second dimension. We wish to report the structure determination of this pigment which has been found to be isorhamnetin 3-*O*-rutinoside (I).



(I)

[1] T. J. MABRY and B. L. TURNER, *Taxon* **13**, 197 (1964).
[2] T. J. MABRY and A. S. DREIDING, in *Recent Advances in Phytochemistry* (edited by T. J. MABRY, R. E. ALSTON and V. C. RUNECKLES), pp. 145–158, Appleton-Century-Crofts, New York (1968).

STRUCTURE DETERMINATION

In order to isolate the flavonoid from the methanol extract, it was necessary to remove a large amount of water soluble components by preliminary column chromatography of the methanol extract over cellulose powder using 15% aqueous acetic acid as solvent. The flavonoid fraction was further purified by preparative paper chromatography using the same solvent and by column chromatography over silica gel. After the pigment was recrystallized from methanol it melted near 250° with decomposition.

With the exception of the melting-decomposition range, the flavonoid from *Batis* was found by standard procedures[3] (u.v., NMR and two dimensional paper chromatography) to be identical with a sample of isorhamnetin 3-*O*-rutinoside which we previously isolated from *Opuntia lindheimeri*.[4]

EXPERIMENTAL

The u.v. spectra were measured with a Coleman-Hitachi 124 recording spectrophotometer. The NMR spectrum was determined in CCl_4 with a Varian A-60 spectrometer using tetramethylsilane as internal standard. The gas chromatographic analysis was performed with a Hewlett-Packard F & M 402 apparatus.

The flavonoid glycoside from *Batis maritima* showed the following properties: u.v. in MeOH: 356, 306sh, 265sh, 254; in NaOMe: 414, 330, 271; in anhydrous $AlCl_3$:[3,5] 401, 368sh, 300sh, 267; in $AlCl_3$/HCl: 400, 360, 302sh, 267; in NaOAc: 398, 320sh, 271; and in NaOAc/H_3BO_3: 360, 305sh, 268sh and 254 nm. NMR (after trimethylsilylation[3,6]): H-6, 6·15 (d, $J=3$); H-8, 6·44 (d, $J=3$); H-2′, 7·50 (d, $J=2$); H-5′, 6·82 (d, $J=8$); H-6′, 7·34 (q, $J=2$ and 8); 3′-methoxy, 3·87; glucose H-1, 5·90; rhamnose H-1, 4·25; rhamnose methyl 0·85 and other signals for 10 sugar protons in the range 3·3–4·0 ppm (δ-scale).

GLC analysis of the sugar fraction (after trimethylsilylation[3,7]) obtained on hydrolyzing the glycoside showed equivalent amounts of glucose and rhamnose. The aglycone was found by standard procedures to be identical with authentic isorhamnetin.

Acknowledgements—We wish to thank the National Science Foundation and the Robert A. Welch Foundation for financial support.

[3] T. J. MABRY, K. R. MARKHAM and M. B. THOMAS, *The Systematic Identification of Flavonoids*, Springer Verlag, New York (1969).

[4] H. RÖSLER, U. RÖSLER, T. J. MABRY and J. KAGAN, *Phytochem.* **5**, 189 (1966). We previously reported a melting point with decomposition of about 177° for the isorhamnetin 3-*O*-rutinoside isolated from *Opuntia lindheimeri*. However, on recrystallizing from methanol the small sample of this material still available we raised the melting point range more than 20°. The melting point difference between the *Batis* and *Opuntia* samples is probably due to their degrees of hydration and to the fact that both samples melt with decomposition.

[5] K. R. MARKHAM and T. J. MABRY, *Phytochem.* **7**, 1197 (1968).

[6] T. J. MABRY, J. KAGAN and H. RÖSLER, *Nuclear Magnetic Resonance Analysis of Flavonoids*, Publ. 6418, University of Texas Press, Austin (1964).

[7] J. KAGAN and T. J. MABRY, *Anal. Chem.* **37**, 288 (1965). We have since found that the analysis is improved by hydrolyzing the glycoside in an initially heterogeneous system without added methanol. The gas chromatograms display only the known sugar peaks, without additional signals.