

SHORT COMMUNICATION

2-HYDROXY-2-METHYLPROPYL GLUCOSINOLATE IN *RESEDA ALBA*

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Abstract—Seeds of *Reseda alba* L. (Resedaceae) contain, on the basis of chromatographic analysis, one glucosinolate, identified as 2-hydroxy-2-methylpropylglucosinolate (II), new to the family, but previously encountered in a few species of the family Cruciferae.

INTRODUCTION

GLUCOSINOLATES (I) seem to be present in virtually all species of the families Cruciferae and Capparidaceae.¹ The family Resedaceae, generally accepted as part of the same phyletic unit, has also served as the source of glucosinolates; however, the number of species studied is still small. Thus far, 2-phenylethyl- (Ia) and 2(*S*)-hydroxy-2-phenylethylglucosinolate (Ib) represent the only glucosinolates firmly established as constituents of Resedaceae.¹ A few years ago, however, Schraudolf² presented evidence for the presence of 3-indolylmethylglucosinolate (Ic) in etiolated seedlings of several species of the genus *Reseda*. We now present the first finding of a glucosinolate with a purely aliphatic side-chain in a *Reseda* species.

RESULTS AND DISCUSSION

On paper chromatography, a methanolic seed extract of *Reseda alba* L. was shown to contain a single glucosinolate. This was extracted, purified, and subjected to enzymatic hydrolysis with myrosinase. One of the hydrolysis products was extracted with chloroform and isolated in pure, crystalline state. Melting point, composition, and spectroscopic properties disclosed its identity as 5,5-dimethyl-2-oxazolidinethione (III), undoubtedly deriving from 2-hydroxy-2-methylpropylglucosinolate (II), as previously discussed.³ This thioglucoside, which can be crystallized as the potassium salt,⁴ was first discovered in seeds of the crucifer *Conringia orientalis* (L.) Andr.,^{3, 5} but shown to be present also in three species of *Cochlearia* (Cruciferae).³ Its presence in *R. alba* L. suggests the operation in this species of a biogenetic pathway leading from leucine, or a close relative, to (II), similar to the one prevailing in the conversion of phenylalanine into the corresponding 2(*S*)-hydroxy-2-phenylethylglucosinolate (Ib) in *R. luteola* L.⁶

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¹ A. KJÆR, *Fortschr. Chem. Org. Naturstoffe* **18**, 122 (1960).

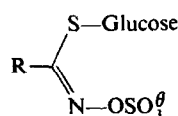
² H. SCHRAUDOLF, *Experientia* **21**, 520 (1965).

³ A. KJÆR, R. GMELIN and R. BOE JENSEN, *Acta Chem. Scand.* **10**, 432 (1956).

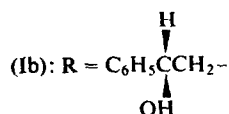
⁴ R. GMELIN and A. I. VIRTANEN, *Acta Chem. Scand.* **13**, 1718 (1959).

⁵ O. -E. SCHULTZ and W. WAGNER, *Arch. Pharm.* **289/61**, 597 (1956).

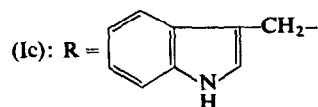
⁶ E. W. UNDERHILL, *Can. J. Biochem.* **43**, 184 (1965).



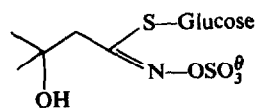
(Ia): $R = C_6H_5CH_2CH_2-$



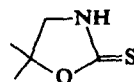
(Ib): $R = C_6H_5CH_2CH_2-$



(Ic): $R =$



(II)



(III)

Delaveau⁷ reported, on the basis of paper chromatography, the presence of 2-phenylethyl isothiocyanate, probably deriving from the glucosinolate (Ia), in roots of *R. alba* L. We have not had opportunity to confirm this finding.

EXPERIMENTAL

Seeds (100 g) of *Reseda alba* L. were ground and defatted with petroleum ether. The seed powder was extracted with two 800-ml portions of 70% MeOH. The combined extracts were concentrated to a small volume, and lead acetate (20% solution) was added to precipitate undesired contaminants. The filtrate was treated with a 20% solution of Na_2HPO_4 , and precipitated lead phosphate was filtered off. The filtrate was diluted with a citrate buffer (pH 6.4), and a myrosinase solution (2 ml) and a trace of ascorbic acid were added. After a few hours at room temperature, the mixture was extracted three times with $CHCl_3$. On evaporation of the dried solution, a crystalline fraction (500 mg) remained. Several recrystallizations from ethyl acetate: petroleum ether gave a pure specimen (180 mg) of a product, C_5H_9NOS , m.p. 105° , alone or in admixture with an authentic specimen of 5,5-dimethyl-2-oxazolidinethione (III).³ The isolate displayed spectroscopic properties identical with those of the authentic sample.

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⁷ P. DELAVEAU, *Bull. Soc. Bot. France* **105**, 224 (1958).