Phytochemistry, 1981, Vol. 20, p. 177. © Pergamon Press Ltd. Printed in England. 0031-9422/81/0101-0177 $02.00/0

A TERPENE–COUMARIN DERIVATIVE FROM *ETHULIA CONYZOIDES*

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(*Received* 5 *May* 1980)

Key Word Index—*Ethulia conyzoides*; Compositae; new terpene–coumarin derivative.

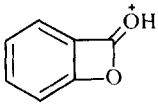
We have very recently characterized four isomeric terpene–coumarin derivatives with the formula $C_{20}H_{22}O_5$ from the aerial parts of *Ethulia conyzoides* L. [1]. This report deals with the isolation and structural elucidation of another major compound, $C_{20}H_{24}O_6$, representing a hydrated derivative of the above isomers. Its ^1H NMR (see Table 1) and MS data clearly establish structure **1**. The stereochemistry at C-5′ and C-6′, however, has not been determined. The proposed structure was also confirmed through treatment with periodate to afford the lactone **2** previously obtained from two of the isomers after similar treatment [1, 2].

Table 1. ^1H NMR data for **1** (270 MHz, $CDCl_3$, TMS as internal standard)

Protons	δ	J (Hz)
6-H	7.17 *d* (*br*)	6, 7 = 8
7-H	7.37 *dd*	
8-H	7.04 *d* (*br*)	7, 8 = 8
9-H	2.76 *s*	
1′*t*-H	5.25 *d*	1′*t*, 2′ = 17.5
1′*c*-H	5.30 *d*	1′*c*, 2′ = 10.5
2′-H	6.23 *dd*	
$4'_1$-H	2.57 *d*	$4'_1, 4'_2$ = 15
$4'_2$-H	2.26 *d*	
6′-H	3.42 *s*	
8′-H	1.44 *s*	
9′-H	1.42 *s*	
10′-H	1.62 *s*	

EXPERIMENTAL

The reported extraction and purification procedure [1] was repeated on another batch of plant material, 250 g. The ether extract was subjected to column chromatographic analysis using SiO_2 and ether–petrol (= E–P) in increasing order to successively elute the isomeric compounds [1] and lastly with E–P, 1:1, to get **1**. The latter was further purified by preparative TLC on SiO_2 to yield 56 mg, colourless crystals, mp. 128–129°, IR ν_{max}^{KBr} cm^{-1}: 3450 (OH), 1695, 1600 (coumarin); MS (70 eV, direct inlet): M^+ *m/e* 360.157 (2%) ($C_{20}H_{24}O_6$); 342 (3) ($M-H_2O$); 327 (2) (342–•Me); 302 (4) ($M-Me_2CO$); 271 (25) ($M-HO\dot{C}HC(OH)Me_2$); 229 (65) ($271-C_2H_2O$);



135 (100).

To 5 mg **1** in 0.5 ml MeOH 10 mg $NaIO_4$ in 0.1 ml H_2O was added. After 30 min the reaction product was extracted with ether and the residue was crystallized from E–P as colourless crystals, mp. 148° (3 mg), identical with an authentic sample (mp, ^1H NMR).

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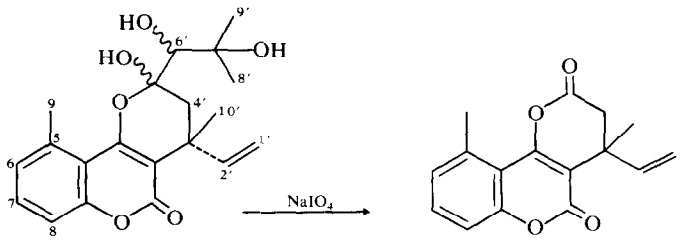

1 **2**