

CHALCONE DERIVATIVES FROM *POLYGONUM LAPATHIFOLIUM*

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Key Word Index—*Polygonum lapathifolium* L.; Polygonaceae; chalcone derivatives.

Abstract—The investigation of *Polygonum lapathifolium* L. afforded, in addition to 2'-hydroxy-4',6'-dimethoxychalcone, three new derivatives. The structures were elucidated by high field ^1H NMR techniques.

INTRODUCTION

The plant is widely grown in Bangladesh. Previous investigation of leaves and seeds extract of this plant afforded flavonoids [1] and 5,7-dihydroxychromone [2]. 2'-Hydroxy-4',6'-dimethoxychalcone was first reported by Mahesh *et al.* [3] from the heart wood of *Pinis griffithi*. Our present investigation gave only chalcones but no chromone compounds could be detected. The results are discussed in this paper.

RESULTS AND DISCUSSION

The aerial parts gave the chalcones **1**, **2**, **3** and **4**, the last two compounds not being separated. The structures of the compounds **2**, **3** and **4** followed from the ^1H NMR data (Table 1). The spectra showed more or less the same type of signals for all three compounds differing only in signals for ester residues. The compounds are all chalcones and it is evident from two multiplets at δ 7.58 and 7.39 indicating aromatic protons of the monosubstituted ring A. The *trans*-olefinic protons of **7** and **8** positions are represented by two doublets at δ 7.88 and 7.79 respectively with a coupling constant of 15 Hz. The second aromatic ring is highly substituted and only one singlet can be seen at δ 6.07. The other four substituents are 2-hydroxyl groups, one methoxy group and an ester. The relative position of one of the hydroxyl groups followed from the chemical shift at around δ 14.4 due to hydrogen bonding and those of the methoxy group from observed NOE with H-8 and aromatic signal. As the latter showed an identical chemical shift in all three compounds and the hydrogen bonded protons influenced by the ester group differ slightly, the proposed arrangement of the two residual groups is more likely. It is further supported by an up-field shift of the aromatic signal.

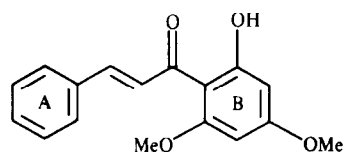
EXPERIMENTAL

The plant material was collected in Dhaka, Bangladesh in 1986. The plant was identified by National Herbarium of Bangladesh. The sun-dried whole plant was ground and 500 g of the powder was extracted with petrol and ether (2:1). The extract gave four fractions by CC (silica gel). The solvent system was petrol and ether with increasing polarity. The compounds were later purified by repeated TLC (silica gel, GF 254). Fraction I of

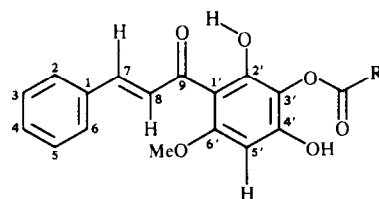
CC gave by TLC 5 mg **1** and fraction III afforded three compounds: 4 mg **2** and 5 mg **3** and **4**. Compounds **3** and **4** could not be separated. The known compound **1** was identified by comparing the 400 MHz ^1H NMR spectra with that of authentic material.

6'-Methoxy-2',4'-dihydroxy-3'-angeloyloxychalcone 2. Colourless gum; IR $\nu_{\text{max}}^{\text{CCl}_4}$ cm^{-1} : 3640, 3500 (OH), 1720 ($\text{C}=\text{CCO}_2\text{R}$), 1630, 1560 ($\text{PhCOC}=\text{C}$); MS m/z (rel. int.): 368 $[\text{M}]^+$ (2) (calc. for $\text{C}_{21}\text{H}_{20}\text{O}_6$: 368.126), 286 $[\text{M}-\text{C}_5\text{H}_6\text{O}]^+$ (100), 271 $[\text{286}-\text{Me}]^+$ (2), 269 $[\text{M}-\text{OAng}]^+$ (2), 209 $[\text{286}-\text{C}_6\text{H}_5]^+$ (6), 182 $[\text{286}-\text{C}_6\text{H}_5\text{CH}=\text{CH}_2]^+$ (86), 83 $[\text{C}_4\text{H}_7\text{CO}]^+$ (100), 57 $[\text{85}-\text{CO}]^+$ (86).

6'-Methoxy-2',4'-dihydroxy-3'-(2-methylbutyryloxy)-chalcone 3 and 6'-methoxy-2',4'-dihydroxy-3'-isovaleryloxychalcone 4. Colourless gum; IR $\nu_{\text{max}}^{\text{CCl}_4}$ cm^{-1} : 3660, 3520, 3300 (OH), 1750 (COOR), 1730, 1560 ($\text{PhCOC}=\text{C}$); MS m/z (rel. int.): 370 $[\text{M}]^+$ (5) (calc. for $\text{C}_{21}\text{H}_{22}\text{O}_6$: 370.141), 286 $[\text{M}-\text{ketene}, (\text{Me})_2\text{CH}-\text{CH}=\text{C}=\text{O}]^+$ (90), 269 $[\text{M}-\text{RCOO}]^+$ (2), 209 $[\text{286}-\text{C}_6\text{H}_5]^+$ (5), 182 $[\text{286}-\text{C}_6\text{H}_5\text{CH}=\text{CH}_2]^+$ (100), 85 (12), m 57 (64).



1



- 2** R = Ang
3 R = MeBu
4 R = iVal

Table 1. ¹H NMR spectral data of **2**, **3** and **4** (400 MHz, CDCl₃, δ-values)

H	2	3	4
2,6	7.58 <i>m</i>		7.58 <i>m</i>
3,4,5	7.39 <i>m</i>		7.38 <i>m</i>
7	7.88 <i>d</i>		7.88 <i>d</i>
8	7.79 <i>d</i>		7.79 <i>d</i>
5'	6.07 <i>s</i>		6.07 <i>s</i>
OH	14.44 <i>s</i>	14.36 <i>s</i>	14.41 <i>s</i>
OMe	3.91 <i>s</i>		3.90 <i>s</i>
OCOR	6.36 <i>qq</i>	2.75 <i>tq</i>	2.53 <i>d</i>
	2.11 <i>dq</i>	1.35 <i>d</i>	2.30 <i>tqq</i>
	2.13 <i>dq</i>	1.68 <i>ddq</i>	1.09 <i>d</i>
		1.90 <i>ddq</i>	
		1.06 <i>t</i>	

J [Hz]: 7,8 = 15; OAng: 3,4 = 7; 3,5 = 4,5 = 1.3; OMeBu: 2,3 = 2,5 = 3,4 = 7; 3', 3'' = 14; OVal: 2,3 = 3,4 = 3,5 = 7.

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NEOCORYMBOSIDE, A DI-C-GLYCOSYLFLAVONE FROM *ATRACTYLIS GUMMIFERA*

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Key Word Index—*Atractylis gummifera*; Compositae; di-C-glycosylflavone, neocorymboside; 6-C-β-L-arabinopyranosyl-8-C-β-D-galactopyranosylapigenin.

Abstract—Neocorymboside, a new glycosylflavone from *Atractylis gummifera* roots was shown to be 6-C-β-L-arabinopyranosyl-8-C-β-D-galactopyranosylapigenin

INTRODUCTION

Atractylis gummifera L. grows on Mediterranean shores except on the French littoral. Its roots contain atractyloside which is very toxic to hepatic cells. Orientin, homoorientin and isoschaftoside were found in *Atractylis gummifera* leaves [1]. In this paper, we discuss the identification of a new di-C-glycosylflavone in the roots. Corymboside, its α-L-arabinopyranosyl isomer was found in *Carlina corymbosa* roots [2] and it is known that *Carlina* and *Atractylis* are closely related systematically.

RESULTS AND DISCUSSION

The compound was isolated from the butanol-soluble fraction of a methanolic extract of dry powdered roots collected in Algeria. The UV spectrum and diagnostic shifts suggested apigenin with free 4',7 and 5-hydroxyl groups [3]. Its chromatographic and hydrolytic properties were those of C-glycosides: no sugar was obtained on acid hydrolysis. Negative-ion FAB mass spectrum of the free compound and electron impact mass spectrometry of its PM ether showed the product to be a 6-C-