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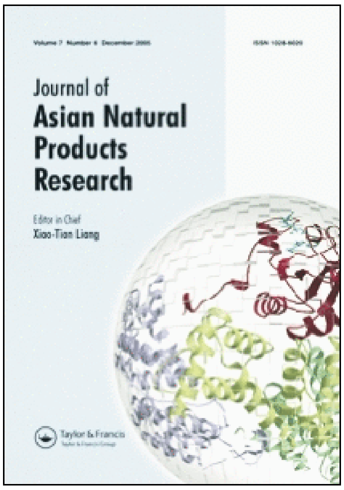

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PLATYPHYLLIN A, A NOVEL COUMARONE FROM THE LEAVES OF *BETULA PLATYPHYLLA* SUK.

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A novel coumarone, platyphyllin A (**1**), was isolated from the leaves of *Betula platyphylla* Suk.. The structure elucidation was accomplished by the analysis of spectroscopic data. Compound **1** was identified as a new coumarone skeleton, which was first isolated from plants.

Keywords: Coumarone; *Betula platyphylla* Suk.

INTRODUCTION

The genus *Betula* contains over 35 species found in temperate and boreal zones of the Northern Hemisphere. *Betula platyphylla* Suk., whose bark and juice were used as anti-inflammatory and cough relieving agent [1], is widespread in China. Triterpenes, flavonoids, lignans and diarylhepatonoids were isolated from birch bark, leaves and spikes [2–7]. This paper describes the isolation and structural determination of platyphyllin A (**1**), which was a new compound with a novel skeleton first found in plants.

RESULTS AND DISCUSSION

Molecular weight determination of **1** by ESI-MS gave a protonated molecular ion at *m/z* 273 $[M+H]^+$ and further confirmed by a negative

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mode ESI-MS showing a strong deprotonated ion at *m/z* 271 $[M-H]^-$. In combination with the ^{1}H- and ^{13}C-NMR spectrum, the formula of **1** was suggested as $C_{15}H_{12}O_5$.

The IR spectrum showed strong absorption bands at 3445 (OH), 1769 (C=O), 1630 and 1601 (phenyl group) cm^{-1}. The sharp band at 907 cm^{-1} indicated the presence of a furan ring, which was supported by analysis of the HMBC data later.

In the ^{1}H-NMR spectrum of **1** (Tab. I) twelve protons were observed. A methylene doublet at δ 4.63 and a hydroxy triplet at δ 5.56 indicated the presence of a hydroxymethyl group. One sharp singlet at δ 3.85 represented OCH_3 group. Three ABX-type protons at δ 6.97, δ 7.17, δ 8.10 reflected a typical trisubstituted benzene ring and one of the substitutions was unsaturated. Two protons at δ 6.70 and δ 7.37 indicated the presence of furan ring in compound **1**.

The ^{13}C-NMR spectrum showed thirteen unsaturated carbon signals and two saturated ones (Tab. I). The signals at δ 56.2 and δ 55.5 were assigned to CH_2OH and OCH_3 group respectively, based on the HMQC spectrum. The signal at δ 169.5 indicated the presence of ester group. In HMBC spectrum, the correlations of δ 3.85 (OCH_3) to δ 155.9 indicated that this methoxyl group was located in the benzene ring. The methylene signal at δ 4.63 (CH_2OH) showed cross peaks with δ 161.5 and δ 111.1 suggesting that the hydroxymethyl group was linked with the furan ring. Further detailed

TABLE I ^{1}H, ^{13}C NMR data and HMBC, NOESY correlations of **1**[†]

Position	*^1H (δ)*	*^{13}C (δ)*	*HMBC*	*NOE*
2		169.5		
3		115		
3a	–	122.3		
4	8.10 *d* (2.6)[‡]	108.5	C-3, C-6, C-5	5-OCH_3, H-6′
5	–	155.9	–	–
5-OCH_3	3.85 *s*	55.5	C-5	H-6′, H-6, H-4
6	6.97 *dd* (8.7, 2.6)	117.1	C-4, C-7a, C-5	H-7, 5-OCH_3
7	7.17 *d* (8.7)	111.1	C-6, C-3a, C-7a	H-6
7a	–	147.4	–	–
1′	7.51 *s*	123.5	C-3, C-3a, C-3′, C-2′, C-2	H-3′
2′		149.6	–	
3′	7.37 *d* (3.4)	124.4	C-4′, C-1′, C-2′, C-5′	H-1′, H-4′
4′	6.70 *d* (3.4)	111.1	C-2′, C-3′	H-3′, H-6′
5′	–	161.5	–	–
6′	4.63 *d* (5.5)	56.2	C-4′, C-5′	H-4, H-4′, 6′-OH, 5-OCH_3
6′-OH	5.56 *d* (5.5)	–	–	H-6′

[†] ^{1}H & ^{13}C NMR recorded on 300 & 75 MHz at 25°C in DMSO with chemical shifts (δ) in ppm from TMS, respectively.

Coupling constants (J) in parentheses (Hz).

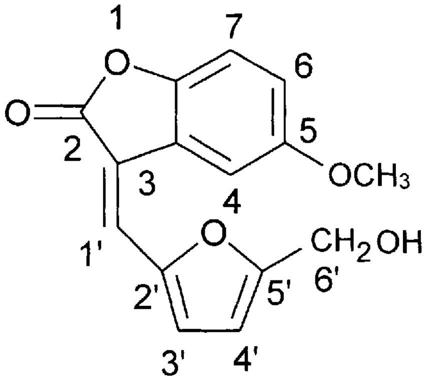

FIGURE 1 The structure of 1.

analysis of HMBC data (Tab. I) led to inferring the structure of **1** as Figure 1. The stereochemistry of **1** was determined by NOESY experiment, which exhibited cross peaks between δ 8.10 and δ 4.63, as well as between δ 3.85 and δ 4.63 suggesting that the double bond at 3(1′) was E isomer and the 1′(2′) single bond was *trans*-configuration. All of the ^{1}H-, ^{13}C-NMR data assignments can be confirmed by HMQC, HMBC and NOESY data.

EXPERIMENTAL SECTION

General Experimental Procedures

Melting point was determined on Yanaco MP-S3 micromelting point apparatus and is uncorrected. IR spectrum was recorded on Bruker IFS-55 spectrophotometer as pressed KBr disk. ^{1}H- and ^{13}C-NMR spectra were measured on Bruker ARX-300 spectrometer in DMSO with TMS as interal standard. Positive and negative ESI-MS were taken on Finnigan LCQ instrument. Optical rotations were measured on Perkin-Elmer 241MC polarimeter using MeOH as solvent.

Plant Material

The leaves of *Betula platyphylla* Suk. were collected at Tong Hua, Jilin of China in July, 1998 and were identified by Prof. Chun-Quan Xu of our University. A sample has been deposited at Department of Natural Products Chemistry, Shenyang Pharmaceutical University.

Extraction and Isolation

Leaves of *Betula platyphylla* Suk. (10 kg) were extracted with water (3 × 100 L) and the extract was precipitated with 70% EtOH. After the EtOH was recovered, the residues was diluted with water to 4 L and partitioned with petroleum ether (3 × 4 L), ether (3 × 4 L), EtOAc (3 × 4 L) and *n*-BuOH (3 × 4 L) successively. EtOAc extract (110 g) was chromatographed on a silica gel column, which was eluted with a $CHCl_3$/MeOH solvent system in increasing ratio of MeOH(0% to 100%). Fr.1 – 15 were then subjected to a vacuum silica gel column chromatography to give compound **1**(10 mg).

Pure **1** was obtained as yellow needles with mp = 165 – 168°C and $[\alpha]_D^{18}$ 0 (c 0.1, CH_3OH). IR (KBr) ν_{max} (cm^{-1}): 3445, 1769, 1630, 1601,1483, 1024, 907, 803. Positive ESI-MS (*m/z*): 273$[M+H]^+$, 255$[M-H-H_2O]^+$, 227$[M+H-H_2O-CO]^+$; negative ESI-MS (*m/z*): 271$[M-H]^-$, 256$[M-CH_3-H]^-$, 228$[M-HCH_3-CO]^-$, 199. The ^1H- and ^{13}C-NMR data see Table 1.

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