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A New Compound from the Bud of *Chrysanthemum indicum* L.

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Abstract: A new bicyclic spiroketone was isolated from the bud of *Chrysanthemum indicum* L. The chemical structure was elucidated as (1R, 9S, 10S)-10-hydroxyl-8 (2′, 4′-diynehexylidene)-9-isovaleryloxy-2, 7-dioxaspiro [5, 4] decane based on the X-ray crystallography.

Keywords: *Chrysanthemum indicum* L., X-ray crystallography, bicyclic spiroketone, (1R, 9S, 10S)-10-hydroxyl-8 (2′, 4′-diynehexylidene)-9-isovaleryloxy-2, 7-dioxaspiro [5, 4] decane.

Chrysanthemum indicum L. (*Compositae*) is a wild herb widely spread in China. The bud of this plant is often used in Chinese traditional medicine for the treatment of inflammation, hypertension and respiratory diseases. Several classes of constituents from *Chrysanthemum indicum* L. have been studied[1-2]. In the present paper, we describe the isolation and the structural elucidation of a new bicyclic spiroketone from the extract of this plant with inhibition on the growth of a human hepatoma cell line ($HepG_2$).

The dried buds of *C. indicum* (8 kg) were macerated with 95 % ethanol at room temperature for 2 weeks. The ethanol extract was evaporated to dryness in vacuum to give a syrupy residue that was partitioned between petroleum ether and water. The petroleum ether fraction was evaporated to give a residue that was chromatographied on silica gel column, eluting with petroleum ether and increasing amount of ethyl acetate (1%-100%). The elution of 10% ethyl acetate in petroleum ether was subjected to flash chromatography over silica gel-H (> 300 mesh) to afford compound **1** (20.0 mg), which was further purified by recrystallization from petroleum ether as white needle crystals, mp 147°C, $[\alpha]_D^{25}$ +13.0 (c 1.1, $CHCl_3$). The molecular formula was determined as $C_{19}H_{24}O_5$ by EIMS. Elemental analysis gave C% 68.61 and H% 7.25 (calcd.: C% 68.60 and H% 7.22). The IR spectrum (KBr) indicated the presence of hydroxyl group (3509 cm^{-1}), ester group (1750 cm^{-1}), triple bonds (2245, 2150 cm^{-1}) and double bond (1645 cm^{-1}). The ^{1}H-NMR (300 MHz, $CDCl_3$) spectrum exhibited proton signals for one oxygenated methylene (δ 3.4, 2H), two oxygenated methines (δ 4.1, 1H and δ 3.9, 1H) and one olefinic proton (δ 5.4, 1H) (**Table 1**). The ^{13}C-NMR spectrum showed nineteen carbon signals for three methyls, four methines, five methylenes and seven

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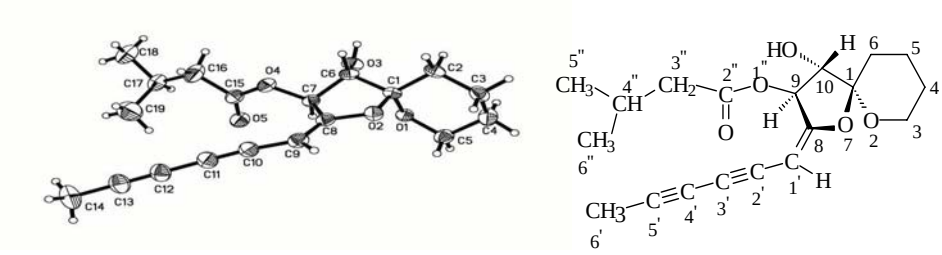
quaternary carbons on the basis of DEPT experiment (**Table 1**). The stereostructure of compound **1** was elucidated by X-ray crystallographic analysis (**Figure 1**). Based on the above results and relevant literatures[2], compound **1** can be regarded as (1R, 9S, 10S)-10-hydroxyl-8 (2′,4′-diynehexylidene)-9-isovaleryloxy-2,7-dioxaspiro [5,4] decane.

Figure 1 X-Ray crystallogram and structure of Compoud **1**



Crystal size = 0.26*0.12*0.10 mm^3. 1.47° < θ < 26.49°. Crystal system: orthorhombic. Space group: $P2_12_12_1$. Unit cell dimensions: a = 0.5914 (2) nm, b = 1.1135 (4) nm, c = 2.7736 (11) nm. Volume = 1.8267 (12) nm^3. Z = 4. Calculated density = 1.209 mg/cm^3, μ (Mo-K_α) = 0.87 cm^{-1}, F (000) = 712 and the final R = 0.0554.

Table l ^1H, ^{13}C-NMR data of compound **1**(δ ppm)

No.	^1H	^{13}C	DEPT	No.	^1H	^{13}C	DEPT
1		98.1	C	3′		107.3	C
3	3.4 (t)	47.2	CH_2	4′		101.2	C
4	2.1 (m)	27.5	CH_2	5′		106.1	C
5	2.1 (m)	23.7	CH_2	6′	1.8 (s)	22.2	CH_3
6	2.7 (m)	27.1	CH_2	2″		170.5	C
8		151.0	C	3″	2.3 (d)	29.8	CH_2
9	4.1 (d)	59.1	CH	4″	1.9 (m)	31.2	CH
10	3.9 (d)	56.8	CH	5″	1.2 (d)	19.2	CH_3
1′	5.4 (s)	119.0	CH	6″	1.1 (d)	18.5	CH_3
2′		102.4	C				

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