

Hemislin B Glucoside, a New Lignan from *Hemistepta lyrata*

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Abstract: From the ethanolic extract of the whole plant of *Hemistepta lyrata* Bunge., a new lignan, named hemislin B glucoside, has been isolated and identified as α -(*trans*-3-methoxy-4-O- β -D-glucopyranosylbenzylidene)- β -(3,4-dimethoxybenzyl)- γ -butyrolactone. Its structure has been established on the basis of UV, IR, FABMS, $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, HMQC and HMBC spectral analysis.

Keywords: *Hemistepta lyrata*, hemislinoside.

Hemistepta lyrata Bunge is a plant of few of one-plant genus in the nature. It is widely distributed in China, but its chemical constituents are little reported. Its extract has been used in Chinese traditional medicine for treatment of anal fistula, sore and furuncle, mastitis, parotitis. The rich resource and multiple pharmacological properties encouraged us to initiate detailed chemical investigation of this plant. After systematic chemical study, we isolated about 20 compounds from this plant. They were identified as sterol, triterpene, flavonoid, dihydroflavonoid, phenolic acid, chrone, ceramide and lignan. Among them dihydroflavonoid and lignan were the main components in this plant. In this article, we report the isolation and structural identification of a new unsaturated dibenzylbutyrolactone-lignan, hemislin B glucoside **1**, which is very rare in the nature.

Hemislin B glucoside **1**, mp 112-113°C (from methanol); $[\alpha]_D^{20}$ -9.0 (c, 0.183, MeOH). It showed positive result in Molish test. The glucose could be examined after hydrolysis. According to FABMS m/z ($M^+ + H$: 533.1), $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra, its molecular formula was determined to be $\text{C}_{27}\text{H}_{32}\text{O}_{11}$. In its UV spectrum, there were three bands of absorption at λ_{max} (MeOH, lge) 224 (4.24), 290 (4.10) and 316 (4.16) nm, which are in good agreement with those of the known unsaturated lignans containing exocyclic double bonds in α -position of γ -lactone ring^{1,2}. The absorption bands at 1745 cm^{-1} and 1640 cm^{-1} in its IR spectrum, exhibited the presence of an α , β -unsaturated- γ -lactone and a conjugated double bond. The seven proton signals at the low field in $^1\text{H-NMR}$

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spectrum (δ 6.75-7.48), belonged to two benzene rings with 1, 3, 4- trisubstitution (**Table 1**). The signals at δ 3.84 in $^1\text{H-NMR}$ spectrum indicates the presence of three methoxy groups in the benzene rings.

Additionally, in the $^1\text{H-NMR}$ spectra, the signal at δ 2.96, 2.65, 4.32, 4.20 and 4.11 were attributed to $2\times\text{H-7}'$, $2\times\text{H-9}'$, and $2\times\text{H-8}'$, respectively. Another one signal at 4.95 ppm (d, 1H, $J=7.5$ Hz) was assigned to the anomeric proton of β -D-glucose. The location of substituents in the γ -lactone ring and the location of glucose are confirmed by HMBC (see **Figure 1**).

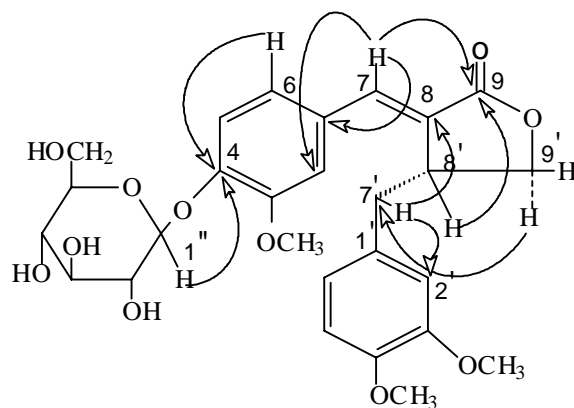
Table 1 The $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectral data of compound **1** (δ ppm)

No.	δ_{H} (J Hz)	δ_{C}	No.	δ_{H} (ppm J Hz)	δ_{C}
1		127.5	6'	6.87(d,1H, $J=8.0$)	120.9
2	7.38(s,1H)	115.1	7'	2.96(dd,1H, $J=14.0,3.5$) 2.65(dd,1H, $J=14.0,10.0$)	36.0
3		147.5	8'	4.11(m,1H)	38.2
4		148.2	9'	4.32(t,1H, $J=8.0$) 4.20(d,1H, $J=9.0$)	69.3
5	7.23(d,1H, $J=8.5$)	113.8	1''	4.95(d,1H, $J=7.5$)	99.7
6	7.34(d,1H, $J=8.5$)	123.9	2''	3.49(m,1H)	73.1
7	7.48(s,1H)	136.0	3''	3.47(m,1H)	76.8
8		130.2	4''	3.48(m,1H)	69.5
9		171.9	5''	3.47(m,1H)	77.1
1'		126.7	6''	3.49(m,1H)	60.6
2'	6.80(s,1H)	112.8	OCH ₃	3.84(s,3H)	55.6
3'		148.6	OCH ₃	3.84(s,3H)	55.7
4'		148.9	OCH ₃	3.84(s,3H)	55.5
5'	6.77(d,1H, $J=8.0$)	111.9			

According to the reference², alternative IR measurement reveals that the frequency of carbonyl and conjugated double bond absorption for hibalactone (1735 and 1640 cm^{-1}) is higher than that of isohibalactone (1713, 1585 cm^{-1}). The absorption bands of compound **1** at 1745 and 1640 cm^{-1} are in good agreement with hibalactone, on the other hand, its $^1\text{H-NMR}$ spectral data of H-7', H-9', and H-8' are very similar to hibalactone. So the configuration of C-8' was identified as R.

According to all above data, the structure of the compound **1** was elucidated as α - (*trans*-3-methoxy-4-O- β -D-glucopyranosylbenzylidene- β - (3,4-dimethoxy benzyl)- γ -butyrolactone (**Scheme 1**). The $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectral data of compound **1** were summed up in **Table 1**. The HMBC correlation of compound **1** is shown in **Scheme 1**.

Figure 2 The HMBC of compound 1



References

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