

Supporting Information to:

Four New Lignans from the Stems of *Kadsura heteroclita*

Wei Wang^{1,2}

Jinzhi Liu³

Rongxia Liu²

Zhengren Xu¹

Min Yang^{1,2}

Weixing Wang¹

Peng Liu¹

Gular Sabia¹

Xiaoming Wang²

Dean Guo^{1,2}

Affiliation: ¹ The State Key Laboratory of Natural and Biomimetic Drugs, School of Pharmaceutical Sciences, Peking University, Beijing, P. R. China

² Shanghai Research Center for Modernization of Traditional Chinese Medicine, Shanghai Institute of Materia Medica, Shanghai Institutes for Biological Sciences, Chinese Academy of Sciences, Shanghai, P. R. China

³ Peking University Shenzhen Hospital, Shenzhen City, Guangdong Province, P. R. China

Correspondence: Prof. Dr. Dean Guo

School of Pharmaceutical Sciences

Peking University

Xueyuan Road 38

Beijing 100083

People's Republic of China

Phone: +86-10-8280-2024

Fax: +86-10-8280-2700

E-mail: gda@bjmu.edu.cn

Known compounds isolated

Kadsurarin (5): obtained as a colorless powder from MeOH; m.p. 255 – 256 °C; $[\alpha]_D^{20}$: −63.5° (*c* 0.12, MeOH); UV (MeOH): λ_{\max} (log ε) = 210 (4.86), 300 (sh) nm; IR (KBr): ν_{\max} = 3535, 2941, 1725, 1624, 1595, 1461, 1377, 1231, 1106, 1046 cm^{−1}; EI-MS (70 eV): *m/z* = 572 (M⁺), 512, 440, 412, 369, 359, 344, 329, 328, 83 (100), 55; ¹H-NMR (CDCl₃, 400 MHz) and ¹³C-NMR (CDCl₃, 100 MHz) data, see Tables S1 and S2.

Kadsulignan E (6): obtained as a colorless powder from MeOH; m.p. 190 – 192 °C; $[\alpha]_D^{20}$: −66° (*c* 0.11, MeOH); UV (MeOH): λ_{\max} (log ε) = 221 (4.82) nm; IR (KBr): ν_{\max} = 3427, 2920, 1741, 1713, 1649, 1633, 1597, 1256, 1068, 1034, 719 cm^{−1}; EI-MS (70 eV): *m/z* = 578 (M⁺), 442, 414, 353, 339, 311, 283, 218, 178, 122, 105 (100), 77; ¹H-NMR (CDCl₃, 400 MHz) and ¹³C-NMR (CDCl₃, 100 MHz) data, see Tables S1 and S2; CD (MeOH): Δε (nm) = −28.31 (220), −11.48 (232), −39.34 (248), +0.56 (326).

D-Epigalbacin: obtained as yellow crystals from EtOAc; m.p. 116 – 117 °C; $[\alpha]_D^{20}$: +44° (*c* 0.14, MeOH); UV (MeOH): λ_{\max} (log ε) = 206 (4.52), 237 (3.36), 287 (3.28) nm; IR (KBr): ν_{\max} = 2907, 1495, 1444, 1250, 1038, 996, 933, 801 cm^{−1}; EI-MS (70 eV): *m/z* = 340 (M⁺), 190 (100), 175, 162, 145, 135, 117, 91, 77; ¹H-NMR (CDCl₃, 400 MHz): δ = 6.8 (6H, m), 5.94 (4H, s, -O-CH₂-O-), 5.39 (1H, d, *J* = 4.2 Hz), 4.59 (1H, s, *J* = 9.3 Hz), 2.38 (2H, m), 0.98 (3H, d, *J* = 6.6 Hz), 0.60 (3H, d, *J* = 6.9 Hz); ¹³C-NMR (CDCl₃, 100 MHz): δ = 147.77, 147.38, 146.88, 146.21, 136.98, 134.49, 119.52, 118.98, 107.91, 107.84, 106.71, 106.41, 100.89, 100.77, 85.63, 84.68, 47.50, 43.34, 11.72, 9.39.

meso-Dihydroguaiaretic acid: obtained as a colorless powder from EtOAc; m.p. 79 – 80 °C; $[\alpha]_D^{20}$: 0° (*c* 0.11, MeOH); UV (MeOH): λ_{\max} (log ε) = 206 (4.54), 228 (4.21), 281 (3.78) nm; IR (KBr): ν_{\max} = 3492, 3404, 2965, 2922, 1608, 1514, 1441, 1372, 1269, 1154, 1029, 797 cm^{−1}; EI-MS (70 eV): *m/z* = 330 (M⁺), 192, 165, 151, 137 (100), 122, 94; ¹H-NMR (CDCl₃, 400 MHz): δ = 6.61 (3H × 2, m), 3.85 (3H × 2, s, -CH₃O × 2), 2.70 (1H × 2, dd, *J* = 5.1, 13.5 Hz), 2.24 (1H × 2, dd, *J* = 9, 13.2 Hz), 1.76 (1H × 2, m),

0.83 (3H × 2, d, J = 6.6 Hz, Me × 2); ^{13}C -NMR (CDCl_3 , 100 MHz): δ = 146.27, 143.51, 133.75, 121.66, 113.91, 111.38, 55.81, 39.14, 38.84, 16.17.

Table S1 ^1H -NMR spectral data of compounds **5** and **6** (CDCl_3 , 400 MHz)^{a,b}

<i>Proton</i>	5	6	<i>Proton</i>	5	6
4	6.79 s	6.56 s	3-OCH ₃	3.93 s	
6	5.67 s	5.82 s	14-OCH ₃	3.73 s	
8	2.11 m	2.11 m	7-OH	2.13 brs	2.72 s
9	5.67 s	5.68 s	Acetoxy Moiety		
11	6.43 s	6.45 s	Me-	1.59 s	1.80 s
17	1.34 s	1.30 s	Angeloxy Moiety		
18	1.25 d (7.2)	1.33 d (7.2)	2'	5.96 br q (7.5)	
19		4.32 d (8.8)	3'	1.84 dd (1.5, 7.2)	
		4.44 d (9.2)	4'	1.39 s	
-OCH ₂ O-	5.90 d (1.5)	5.98 d (1.6)	Benzoxy Moiety		
	5.91 d (1.5)	6.03 d (1.2)	2'', 6''		7.73 dd (1.2, 8.4)
1-OCH ₃	3.62 s	3.69 s	3'', 5''		7.41 t (8.0)
2-OCH ₃	3.88 s	2.83 s	4'		7.56 t (7.6)

^a Chemical shifts in ppm relative to TMS; coupling constants (J) in Hz.

^b Assignments were made by ^1H - $^1\text{HCOSY}$, HSQC and HMBC data.

Table S2 ^{13}C -NMR spectral data of compounds **5** and **6** (CDCl_3 , 100 MHz)^{a,b}

<i>Carbon</i>	5	6	<i>Carbon</i>	5	6
1	151.16	165.50	2-OCH ₃	60.35	58.74
2	141.01	133.74	3-OCH ₃	55.91	
3	151.87	182.50	14-OCH ₃	59.03	
4	110.08	131.29	Acetoxy Moiety		
5	130.03	148.37	-CO-	168.85	169.54
6	84.73	81.11	Me-	20.47	20.26

7	73.92	75.44	Angeloxy Moiety	
8	42.99	44.45	-CO-	165.69
9	83.49	84.51	1'	126.95
10	132.62	129.91	2'	139.88
11	102.01	100.36	3'	15.65
12	148.71	150.32	4'	19.78
13	135.58	129.91	Benzoxo Moiety	
14	140.82	143.95	-CO-	165.95
15	120.36	119.99	1''	128.52
16	121.76	55.79	2''	129.50
17	28.80	28.62	3''	129.21
18	17.05	17.72	4''	133.95
19		84.51	5''	129.21
OCH ₂ O	100.94	102.09	6''	129.50
1-OCH ₃	60.49	61.02		

^a Chemical shifts in ppm relative to TMS.

^b Assignments were confirmed by HSQC and HMBC data.

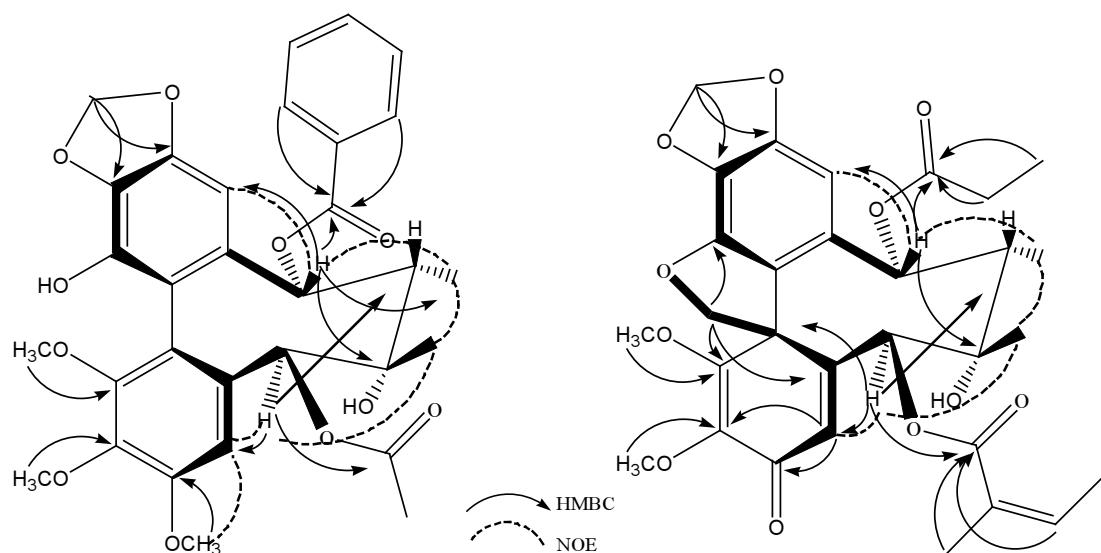


Fig. S1 Key HMBC and NOE correlations of compounds 1 and 4