

Supplementary Materials: Preparative Separation of Main Ustilaginoidins from Rice False Smut Balls by High-Speed Counter-Current Chromatography

Weibo Sun, Xuejiao Dong, Dan Xu, Jiajia Meng, Xiaoxiang Fu, Xiaohan Wang, Daowan Lai, Ligang Zhou and Yang Liu

Table S1. The ¹H-nuclear magnetic resonance (¹H-NMR, 600 MHz) data of compounds 1–7.

Position	1	2	3	4	5	6	7
2	-	4.42 ddq (11.9, 2.4, 5.9)	-	4.44 s	4.34 d (9.5)	-	4.33 d (11.7)
3	6.11 s	2.80 dd (17.4, 11.9)	6.16 s	2.80 dd (17.2, 11.9)	2.92 dd (17.3, 12.5)	6.17 s	2.90 dd (17.4, 12.3)
3'	6.11 s	2.72 dd (17.2, 2.4)	6.16 s	2.68 d (15.6)	2.61 d (17.2)	6.17 s	2.59 dd (17.2, 2.4)
7	6.62 s	5.90 s	6.16 s	5.64 s	5.70 s	6.17 s	5.68 s
7'	6.62 s	6.50 s	6.59 s	6.46 s	6.47 s	6.61 s	6.45 s
10	6.62 s	6.60 s	6.60 s	6.59 s	6.57 s	6.61 s	6.57 s
10'	6.36 s	6.11 s	6.16 s	6.19 s	6.17 s	6.20 s	6.19 s
10''	6.36 s	6.38 s	6.20 s	6.21 s	6.20 s	6.20 s	6.19 s
2-CH ₃	2.30 s	1.34 d (5.9)	2.24 s	1.27 d (6.2)	-	-	-
2'-CH ₃	2.30 s	2.31 s	-	-	2.27 s	-	-
2-CH ₂ OH	-	-	-	-	3.53 dd (18.4, 2.8)	4.27 d (4.4)	3.52 dd (18.7, 3.3)
2'-CH ₂ OH	-	-	4.27 s	4.30 s	-	4.27 d (4.4)	4.29 d (2.5)
2-CH ₂ OH _H	-	-	-	-	4.98 br. s	nd.	nd.
2'-CH ₂ OH _H	-	-	5.76 s	5.79 s	-	nd.	nd.
6-OH	-	-	10.03 s	9.98 s	9.97 s	9.97 s	9.93 s
6'-OH	-	-	10.03 s	9.98 s	9.97 s	9.97 s	9.93 s
8-OH	-	-	9.82 s	9.82 s	9.78 s	9.82 s	9.78 s
8'-OH	-	-	9.82 s	9.82 s	9.79 s	9.82 s	9.78 s

Note: Compounds 1 and 2 were measured in acetone-*d*₆, and 3–7 were measured in DMSO-*d*₆. Chemical shifts were given on the δ (ppm) scale with TMS as the internal standard and coupling constants (*J*) were given in Hz. The letters s, d, dd, ddq and br.s mean singlet, doublet, doublet of doublets, doublet of doublet of quartets, and broad-singlet, respectively, in NMR spectrum. nd.: not detected.

Table S2. The ^{13}C -nuclear magnetic resonance (^{13}C -NMR, 150 MHz) data of compounds **1–7**.

Position	1	2	3	4	5	6	7
2	171.0	74.1	169.7	72.8	77.3	172.4	77.3
2'	171.0	171.0	172.4	172.4	169.8	172.4	172.4
3	106.3	43.6	103.4	42.8	37.7	103.3	37.7
3'	106.3	105.3	105.9	103.4	105.8	103.3	103.3
4	184.7	184.7	183.3	183.5	183.3	183.4	183.5
4'	184.7	199.4	183.4	198.1	198.0	183.4	198.0
4a	102.8	102.7	101.7	101.7	101.7	102.1	101.8
4a'	102.8	102.7	102.2	102.2	101.8	102.1	102.1
5	160.7	161.7	162.7	162.6	162.6	162.6	162.5
5'	160.7	163.2	162.7	164.9	164.7	162.6	164.6
5a	106.6	102.8	105.9	104.4	104.3	105.9	104.3
5a'	106.6	105.3	106.4	105.9	105.8	105.9	105.8
6	154.6	156.6	158.1	157.9	157.9	158.0	157.9
6'	154.6	159.6	158.1	158.9	158.8	158.0	158.8
7	99.6	99.6	98.0	98.2	98.0	98.2	98.2
7'	99.6	100.1	98.2	98.4	98.5	98.2	98.5
8	159.7	160.6	159.1	159.1	159.0	159.2	159.1
8'	159.7	161.6	159.2	160.1	160.1	159.2	160.0
9	107.0	106.6	106.4	106.6	106.6	106.4	106.6
9'	107.0	106.9	106.4	106.9	106.9	106.4	106.9
9a	141.2	141.1	139.8	139.9	139.7	139.9	139.8
9a'	141.2	143.1	140.0	141.6	141.6	139.9	141.6
10	101.5	100.7	100.8	100.0	100.0	100.9	100.0
10'	101.5	101.4	100.8	100.8	100.7	100.9	100.8
10a	153.4	153.4	151.7	151.6	151.9	151.6	151.6
10a'	153.4	154.6	152.0	155.0	154.8	151.6	154.8
2-CH ₃	20.6	20.6	20.2	20.4	-	-	-
2'-CH ₃	20.6	20.9	-	-	20.2	-	-
2-CH ₂ OH	-	-	-	-	62.7	59.8	59.8
2'-CH ₂ OH	-	-	59.8	59.8	-	59.8	62.7

Note: Compounds **1** and **2** were measured in acetone- d_6 and **3–7** were measured in DMSO- d_6 . Chemical shifts were given on the δ (ppm) scale with TMS as the internal standard. a and a' refer to the positions in the chemical structures shown in Figure 2.