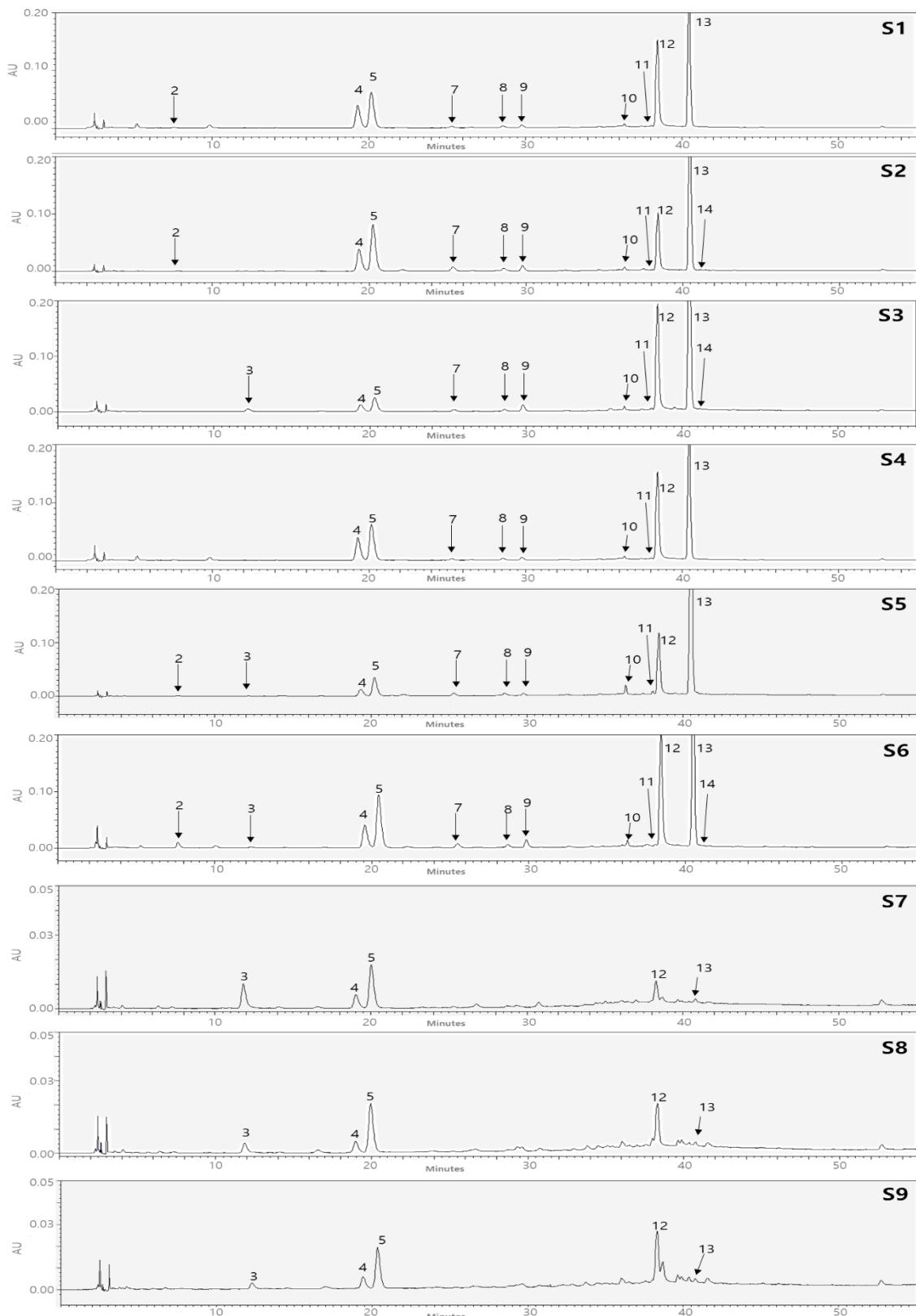
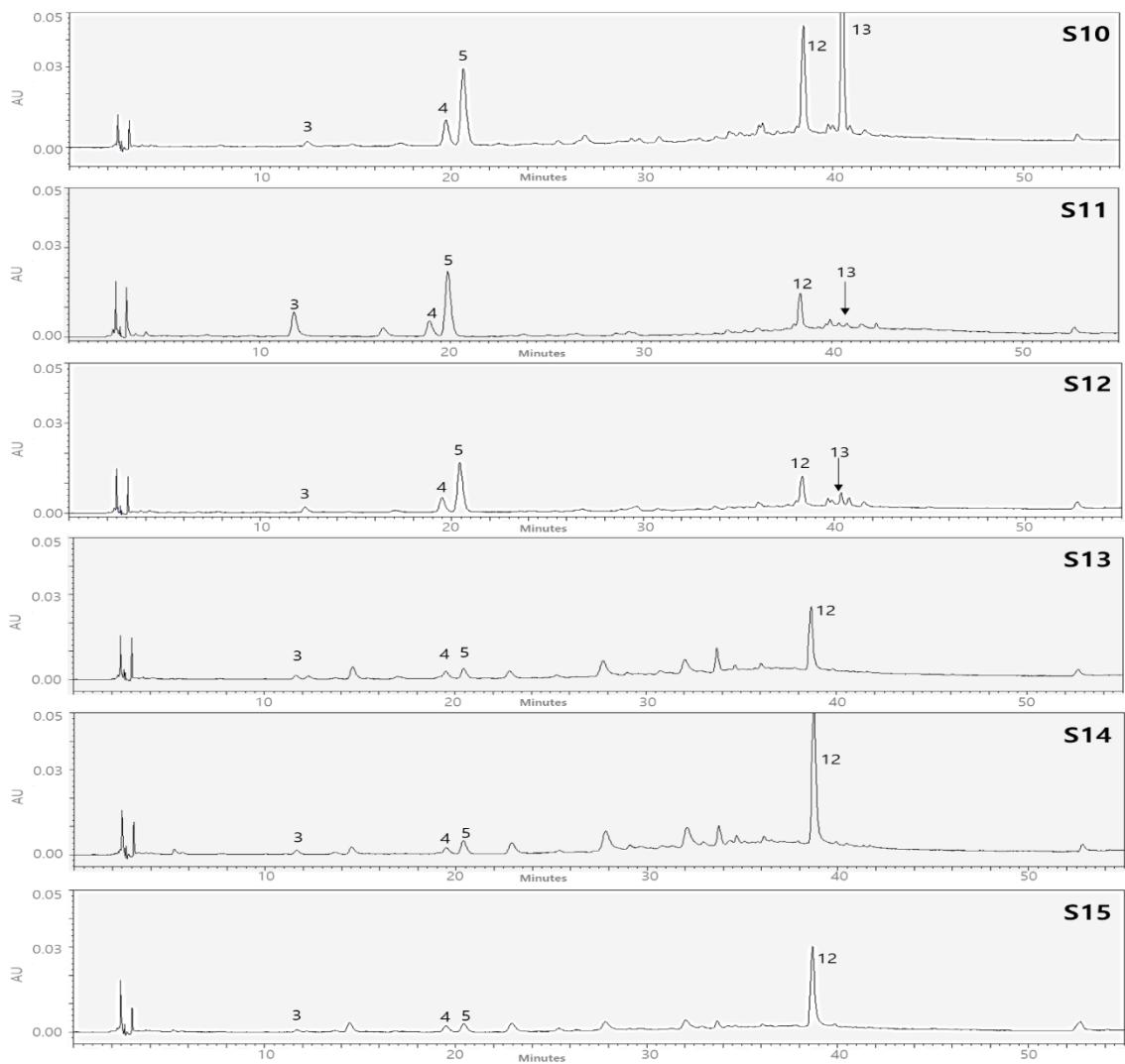


## Supplementary Materials





**Figure S1.** HPLC chromatograms of *D. fortunei*, *D. mollis*, and *D. mariesii* samples.

2: compound 2, 3: compound 3, 4: compound 4, 5: compound 5, 7: compound 7, 8: compound 8, 9: compound 9, 10: compound 10, 11: compound 11, 12: compound 12, 13: compound 13, 14: compound 14, S1: Sample 1, S2: Sample 2, S3: Sample 3, S4: Sample 4, S5: Sample 5, S6: Sample 6, S7: Sample 7, S8: Sample 8, S9: Sample 9, S10: Sample 10, S11: Sample 11, S12: Sample 12, S13: Sample 13, S14: Sample 14, S15: Sample 15

**Table S1.** NMR and mass spectrometry data of 14 compounds isolated from *D. fortunei*.

Analyte	NMR and mass spectrometry data
Compound 1	C <sub>7</sub> H <sub>6</sub> O <sub>5</sub> ; ESI/LTQ-Orbitrap-HRMS m/z: 169.0125 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.05(2H,s,Ar-H); <sup>13</sup> C-NMR (150 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 170.38(C=O), 146.36(C-3,5), 139.57(C-4), 121.95(C-1), 110.31 (C-2,6).
Compound 2	C <sub>6</sub> H <sub>6</sub> O <sub>3</sub> ; ESI/LTQ-Orbitrap-HRMS m/z: 127.0397 [M + H] <sup>+</sup> , <sup>1</sup> H-NMR (600 MHz, CDCl <sub>3</sub> -d) δ: 9.50(1H,s,H-1), 7.14(1H,d,J=3.6Hz,H-4), 6.44(1H,d,J=4.2Hz,H-3), 4.63 (2H,s,H-6), 3.34(1H,brs,OH); <sup>13</sup> C-NMR (150 MHz, CDCl <sub>3</sub> -d) δ: 178.63(C-1), 161.58(C-2), 153.35(C-5), 110.94(C-4), 58.60(C-6).
Compound 3	C <sub>7</sub> H <sub>6</sub> O <sub>4</sub> ; ESI/LTQ-Orbitrap-HRMS: 153.0178 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.43-7.41(2H,m,Ar-H), 6.79(1H,d,J=8.3Hz,Ar-H) ; <sup>13</sup> C-NMR (150 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 170.50(C-7), 151.39(C-4), 146.02(C-3), 123.85(C-1), 123.52(C-6), 117.75(C-2), 115.74(C-5).
Compound 4	C <sub>15</sub> H <sub>18</sub> O <sub>8</sub> ; UPLC/QTOP-MS: 371.0959 [M + HCOOH - H] <sup>-</sup> , 325.0909 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS: 371.0963 [M + HCOOH - H] <sup>-</sup> , 325.0911 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz , CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.46(2H,d,J=9Hz,H-2,H-6), 7.44(1H,d,J=16.2Hz,H-7), 7.07(2H,d,J=9Hz,H-3,H-5), 6.38(1H,d,J=16.2Hz,H-8), 4.94(1H,d,J=7.8Hz,H-1'),3.89(1H,dd,J=12,2.4Hz,H-6'a), 3.37(1H,dd,J=12,5.4Hz,H-6'b), 3.51-3.36(4H,m,H-2',3',4',5'); <sup>13</sup> C-NMR (150 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 176.59(C-9), 159.66(C-4), 141.37(C-7), 130.89(C-1) 129.69(C-2,6),117.68(C-8) 117.60(C-3,5), 101.67(C-1'), 77.87(C-3'), 77.63(C-5'), 74.57(C-2'), 71.04(C-4'), 62.18(C-6').
Compound 5	C <sub>15</sub> H <sub>18</sub> O <sub>9</sub> ; UPLC/QTOF-MS m/z : 341.0847 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS: 341.0857 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHZ, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.48(1H,d,J=16.2Hz,H-7), 7.18(1H,d,J=8.4Hz,H-5), 7.08(1H,d,J=1.2Hz,H-2), 7.01(1H,dd,J=8.4,1.5Hz,H-6), 6.32(1H,d,J=15Hz,H-8), 4.91(1H,d,J=4.8Hz,H-1'), 3.90(1H,dd,J=12,1.8, H-6a), 3.71(1H,dd,J=12.6, 5.4Hz,H-6b), 3.39-3.52(4H,m,H-2',3',4',5') <sup>13</sup> C-NMR (150MHZ, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 148.51(C-4), 148.47(C-3), 144.64(C-7), 131.65(C-1), 121.84(C-6),119.92(C-5), 118.23(C-8), 115.736(C-2), 103.62(C-1'),78.38(C-3'), 77.55(C-5'), 74.9(C-2'), 71.29(C-4'), 62.40(C-6')
Compound 6	C <sub>7</sub> H <sub>6</sub> O <sub>3</sub> ; ESI/LTQ-Orbitrap-HRMS: 137.0223 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.87 (2H, m, J=6.6, 4.4 Hz, H-3, 5); 6.81 (2H, m, J=7.2, 2.4 Hz, H-2, 6), <sup>13</sup> C-NMR (150 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 170.21 (C-7), 163.50(C-4), 133.14 (C-3, 5), 122.83 (C-1), 116.17 (C-2, 6).
Compound 7	C <sub>16</sub> H <sub>20</sub> O <sub>9</sub> ; UPLC/QTOF-MS m/z : 355.1024 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS: 401.1064 [M + HCOOH - H] <sup>-</sup> , 355.1014 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHZ, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.33 (1H,d,J=16.0Hz,H-7), 7.17(1H,d,J=1.2Hz,H-2), 7.13(1H,d,J=8.4Hz,H-5), 7.06(1H,dd,J=8.4,1.8Hz,H-6), 6.40(1H,d,J=15.6Hz,H-8), 4.93(1H,d,J=7.8Hz,H-10), 3.87(1H,dd,J=11.4,1.2Hz,H-6b), 3.69(1H,dd,J=12,4.8Hz,H-6a),3.51-3.38(4H,m,H-2',3',4',5'), <sup>13</sup> C-NMR (150MHZ, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 171.06(COOH), 148.65(C-3), 148.49(C-4), 145.62(C-7), 131.31(C-1), 122.02(C-6), 118.48(C-8), 118.17(C-5), 115.88(C-2), 103.51(C-1'), 78.35(C-5'), 77.51(C-3'), 74.78(C-2'), 71.27(C-4'), 62.38(C-6').
Compound 8	C <sub>17</sub> H <sub>22</sub> O <sub>10</sub> ; UPLC/QTOF-MS m/z : 385.1131 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS: 431.1170 [M + HCOOH - H] <sup>-</sup> , 385.1102 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHZ, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 7.43(1H,d,J=15.6Hz,H-7), 6.87(2H,s,H-2,6), 6.43(1H,d,J=15.6Hz,H-8), 4.94(1H,dd=7.2,H-1'a), 3.88(6H,s,Me), 3.77(1H,m,H-6'a), 3.69(1H,m,H-6'b), 3.50-3.38(4H,m,H-2',3',4',5'); <sup>13</sup> C-NMR (150 MHz, CD <sub>3</sub> OD-d <sub>4</sub> ) δ: 170.66(C-9), 154.45(C-

	3.5), 143.34(C-7), 137.47(C-4), 133.13(C-1), 106.82(C-2,6), 105.00(C-1'), 78.35(C-3'), 77.78(C-5'), 75.66(C-2'), 71.29(C-4'), 62.42(C-6'), 57.12(C-OCH <sub>3</sub> ).
Compound 9	C <sub>21</sub> H <sub>26</sub> O <sub>13</sub> ; UPLC/QTOF-MS m/z : 531.1338 [M + HCOOH - H] <sup>-</sup> , 485.1292 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS m/z: 531.1332 [M + HCOOH - H] <sup>-</sup> , 485.1280 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, CD3OD-d4) δ: 7.90(1H,d,J=6.0Hz,H-2), 6.23(1H,d,J=2.4Hz,H-8), 6.22(1H,d,J=1.8Hz,H-6), 6.15(1H,d,J=6.0Hz, H-2), 5.25(1H,d,J=1.8Hz,H-1''), 5.09(1H,d,J=7.8Hz,H-1''), 3.93(1H,dd,J=1.8Hz,3.0Hz,H-2''), 3.81-3.86(1H,m,H-5''), 3.70(1H,dd,J=4.8Hz,12Hz,H-2'), 3.66-3.62(1H,m,H-6'), 3.60(1H,t,J=9.6Hz,H-3''), 3.52-3.55(1H,m,H-3''), 3.42-3.41(1H,m,H-5''), 3.40-3.37(1H,m,H-4''), 1.29(1H, d,J=5.6Hz,H-6'').
Compound 10	C <sub>27</sub> H <sub>30</sub> O <sub>15</sub> ; UPLC/QTOF-MS m/z : 593.1519 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS m/z: 593.1479 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, DMSO-d6) δ: 7.80(2H,d,J=8.4Hz,H-2'6'), 6.93(2H,d,J=9.0Hz,H-3'5'), 6.75(1H,d,J=2.0Hz, H-8), 6.49(1H,d,J=1.8Hz,H-6), 5.40(1H,s,H-1''), 5.05(1H,d,J=7.2Hz,H-1''), 4.22(1H,dd,J=2.4Hz,3.6Hz,H-4''), 3.92(1H,dd,J=1.8Hz,12Hz,H-5''), 3.72-3.69(2H,m,H-6'',H-5''), 3.54-3.47(2H,m,H-3'',2''), 3.40-3.35(2H,m,H-3'',2''), 3.34-3.32(1H,m,H-4''), 0.92(3H,d,J=6Hz,rhamnosylCH3); <sup>13</sup> C-NMR (150 MHz, DMSO-d6) δ: 177.78(C-4), 162.80(C-7), 160.76(C-5), 160.10(C-4''), 157.66(C-2), 155.96(C-9), 134.91(C-3), 130.53(C-2'6'), 120.12(C-1), 115.29(C-3'5'), 105.619(C-10), 101.62(C-1''), 99.70(C-1''), 99.19(C-6), 94.49(C-8), 77.04(C-5''), 76.25(C-3''), 72.96(C-2''), 70.94(C-3''), 70.53(C-2''), 70.16(C-4''), 69.91(C-5''), 69.39(C-4''), 60.44(C-6''), 17.330(C-6'').
Compound 11	C <sub>26</sub> H <sub>28</sub> O <sub>15</sub> ; UPLC/QTOF-MS m/z : 579.1352 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS m/z: 579.1431 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, DMSO-d6) δ: 8.07(2H,d,J=9.0Hz,H-2', H-6'), 7.31(2H,d,J=7.2Hz,H-3'H-5'), 6.85(1H,d,J=7.8Hz,H-8), 6.81(1H,d,J=8.4Hz,H-6), 5.63(1H,s,H-1''), 5.28(1H,d,J=7.2Hz,H-1''), 4.07-4.05(1H,Brs,H-2''), 4.01-3.99(1H,Brs,H-3''), 3.92-3.84(1H,m,H-4''), 3.79/3.77(1H,m,H-5''), 3.45(1H,brs,H-2''), 3.44(1H,brs,H-3''), 3.39(2H,brs,H-4'',H-5''), 3.69-3.65(1H,m,H-6'').
Compound 12	C <sub>27</sub> H <sub>32</sub> O <sub>15</sub> ; UPLC/QTOF-MS m/z : 595.1682 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS m/z: 595.1637 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, DMSO-d6) δ: 12.04(1H,s,5-OH), 9.08(2H,br.s,H-3'4'-OH), 6.86(1H,d,8.4Hz,H-6'), 6.74(2H,d,J=4.8Hz,H-2'5'), 6.10-6.07(2H,m,H-6,8), 5.46-5.39(1H,m,H-2), 5.14-5.10(1H,m,H-1''), 5.10-5.08(1H,m,H-1''), 3.22-3.71(10H,m,H-6'',5'',5'',3'',2'',2'',4'',4''), 3.20-3.09(1H,m,H-3a), 2.73-2.66(1H,m, H-3b), 1.14(3H,dd,J=2.4Hz, 6Hz, H-6''); <sup>13</sup> C-NMR (150 MHz, DMSO-d6) δ: 197.69(C-4), 165.37(C-7), 163.45(C-5), 163.22(C-9), 146.37(C-4''), 145.75(C-3''), 129.65(C-1'), 118.6(C-6'), 115.89(C-2'), 114.95(C-5'), 103.85(C-10), 100.95(C-1''), 97.81(C-1''), 96.75(C-6), 95.64(C-8), 79.18(C-2), 77.64(C-3''), 77.41(C-5''), 76.69(C-2''), 72.34(C-4''), 70.99(C-2''), 70.9(C-3''), 70.11(C-4''), 68.8(C-5''), 60.95(C-6''), 42.71(C-3), 18.7(C-6'').
Compound 13	C <sub>27</sub> H <sub>32</sub> O <sub>14</sub> ; UPLC/QTOF-MS m/z : 579.1733 [M - H] <sup>-</sup> , ESI/LTQ-Orbitrap-HRMS m/z: 579.1685 [M - H] <sup>-</sup> , <sup>1</sup> H-NMR (600 MHz, DMSO-d6) δ: 7.31(2H,d,J=8.4Hz,H-2', H-6'), 6.82(2H,d,J=7.8Hz,H-3'H-5'), 6.17(1H,d,J=1.8Hz,H-6), 6.15(1H,d,J=2.4Hz,H-8), 5.37-5.35(1H,m,H-2), 5.25-5.24(1H,m,H-1''), 5.10-5.07(1H,m,H-1''), 3.93-3.30(10H,m,H-6'',5'',5'',3'',2'',2'',4'',4''), 3.15-3.14(1H,m,H-3a), 2.74(1H,d,J=15.6Hz,H-3b), 1.28(1H,d,J=6.6Hz,H-6''); <sup>13</sup> C-NMR (150 MHz, DMSO-d6) δ: 198.51(C-4), 166.47(C-7), 164.86(C-5), 164.58(C-9), 159.03(C-4''), 103.64(C-1'), 129.16(C-2'6'), 116.38(C-3'5'), 104.89(C-10), 102.44(C-1''), 99.34(C-6), 97.84(C-8), 96.73(C-1''), 80.60(C-2,4''), 78.88(C-3''), 78.85(C-3''), 78.06(C-2''), 73.84(C-5''), 72.10(C-2''), 71.18(C-5''), 69.92(C-4''), 62.19(C-6''), 44.03(C-3a), 44.79(C-3b), 18.10(C-6'').

Compound 14	$C_{28}H_{34}O_{15}$ ; ESI/LTQ-Orbitrap-HRMS m/z: 609.1791 [M - H] <sup>-</sup> ; <sup>1</sup> H-NMR (600 MHz, DMSO-d6) δ: 12.01(1H,s,5-OH), 6.93(1H,d, <i>J</i> =1.8Hz,H-2'), 6.93(1H,d, <i>J</i> =8.4Hz,H-5'), 6.89(1H,dd, <i>J</i> =2.8Hz,8.4Hz,H-6'), 6.13(1H,d, <i>J</i> =2.4Hz,H-8), 6.11(1H,d, <i>J</i> =3.0Hz,H-6), 5.49 (1H,dd, <i>J</i> =12.0Hz,3.0Hz,H-2), 4.96(1H,d, <i>J</i> =7.2Hz,H-1"), 4.51(1H,brs,H-1"), 3.76(3H,s,4-OCH <sub>3</sub> ), 3.64-3.10(10H,m,H-2",3",4",5",6",2'",3'",4'",5'",3a), 2.76(1H,dd, <i>J</i> =17.4Hz,3.0Hz,H-3b), 1.07(3H,d, <i>J</i> =6.6Hz,H-6); <sup>13</sup> C-NMR (150 MHz, DMSO-d6) δ: 197.02(C-4), 165.12(C-7), 163.02(C-5), 162.49(C-9), 147.95(C-4'), 146.44(C-3'), 130.89(C-1'), 117.94(C-6'), 114.14(C-2'), 112.02(C-5'), 103.31(C-10), 100.60(C-1"), 99.42(C-1"), 96.37(C-6), 95.53(C-8), 78.36(C-2), 76.25(C-5"), 75.50(C-3"), 72.97(C-5"), 72.05(C-2"), 70.69(C-4"), 70.25(C-2"), 69.57(C-3"), 68.30(C-4"), 66.02(C-6"), 55.67(4'-OCH <sub>3</sub> ), 42.04(C-3), 17.82(C-6").
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NMR: Nuclear Magnetic Resonance

**Table S2.** Intra-day and Inter-day precision and accuracy of 12 compounds.

Analyte	Conc. ( $\mu$ g/mL)	Precision (C.V.%)		Accuracy (%)	
		Intra-day	Inter-day	Intra-day	Inter-day
Compound 2	60	1.56	0.76	98.87	97.19
	30	1.06	0.82	101.49	104.90
	15	0.70	0.82	102.78	101.51
Compound 3	80	1.14	2.14	98.68	96.54
	40	0.18	1.39	97.13	104.75
	20	1.57	0.92	99.12	100.80
Compound 4	80	2.07	1.64	100.10	96.54
	40	0.74	1.22	99.23	102.49
	20	0.64	0.81	101.69	103.20
Compound 5	80	2.10	1.43	100.07	96.79
	40	0.56	1.04	99.39	98.86
	20	0.30	1.02	101.41	102.12
Compound 7	80	2.85	1.47	100.06	95.75
	40	0.75	0.88	99.48	103.35
	20	1.36	1.18	100.86	102.55
Compound 8	80	2.51	2.13	99.69	95.95
	40	0.79	0.58	104.26	105.75
	20	0.82	0.89	95.03	102.76
Compound 9	80	1.06	1.41	99.32	101.27
	40	0.78	2.37	102.15	100.33
	20	0.06	1.77	104.43	102.72
Compound 10	80	0.56	0.73	99.95	99.03
	40	0.86	0.67	100.40	100.10
	20	1.41	2.83	98.83	100.22
Compound 11	80	1.83	0.52	100.17	98.56
	40	1.67	2.35	98.83	101.58
	20	1.72	0.37	101.99	100.76
Compound 12	80	0.72	1.07	100.76	98.72
	40	1.04	1.65	96.56	99.27
	20	1.38	1.84	98.91	101.16
Compound 13	80	1.61	1.68	99.85	96.61
	40	0.81	1.09	99.20	99.26
	20	1.78	1.33	101.15	102.84

	80	0.50	0.69	96.58	96.47
Compound 14	40	0.90	0.99	104.75	104.12
	20	0.90	0.24	100.94	102.65

Each value was calculated with mean of triplication

**Table S3.** Repeatability of retention time and peak area of 12 compounds.

Analyte	Retention time		Peak area	
	Mean (min)	RSD (%)	Mean ( $\mu\text{V}^2\cdot\text{sec}$ )	RSD (%)
Compound 2	7.56	0.30	2407028	1.22
Compound 3	12.13	0.26	399648.7	2.23
Compound 4	19.32	0.33	586888.8	1.53
Compound 5	20.19	0.29	542672.8	2.09
Compound 7	25.28	0.12	709714	1.44
Compound 8	28.54	0.08	182826.2	2.05
Compound 9	29.75	0.11	69236.5	1.37
Compound 10	36.29	0.05	253620.8	1.82
Compound 11	38.00	0.07	83329.33	2.20
Compound 12	38.45	0.06	421485.3	0.68
Compound 13	40.47	0.06	586656.8	1.17
Compound 14	41.00	0.06	444231.3	1.08

Each value was calculated with mean of triplication. RSD : Relative standard deviation

**Table S4.** Recovery of 12 compounds for three different spiked concentrations.

Analyte	Spiked concentration ( $\mu\text{g/mL}$ )	Recovery rate (%)
Compound 2	5	103.27
	25	99.71
	50	103.47
Compound 3	5	97.94
	25	99.54
	50	100.81
Compound 4	5	103.05
	25	103.63
	50	103.71
Compound 5	5	103.24
	25	102.20
	50	103.41
Compound 7	5	101.32
	25	100.14
	50	103.96
Compound 8	5	100.67
	25	103.89
	50	103.48
Compound 9	5	100.46
	25	103.65
	50	103.05

	5	103.92
Compound 10	25	103.03
	50	100.53
	5	102.66
Compound 11	25	103.09
	50	103.44
	5	103.74
Compound 12	25	102.74
	50	100.44
	5	101.33
Compound 13	25	103.72
	50	99.66
	5	101.12
Compound 14	25	102.69
	50	102.78

Each value was calculated as mean of triplication