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The chemical shift assignments of carbon and proton signals of **Dioscin A** (Compound **1**) in Pyridine-d_5.

Carbon NO.	Dioscin A	
	δ_C (ppm)	δ_C (ppm, *J* in Hz)
1	37.36	1.67 (H_2-1, m)
2	30.11	2.07 (H_2-2, m)
3	78.18	3.87 (H_1-3, m)
4	39.21	2.44 (H_1-4a, t, *J*=12.2 Hz), 2.68 (H_1-4b, d, *J*=13.4 Hz)
5	140.81	
6	121.66	5.30 (H_1-6, d, *J*=4.18 Hz)
7	32.17	1.84 (H_1-7a, m), 1.94 (H_1-7b, m)
8	31.52	1.50 (H_1-8, m)
9	50.18	0.86 (H_1-9, m)
10	39.65	
11	20.96	1.41 (H_2-11, m)
12	36.95	1.69 (H_2-12, m)
13	40.42	
14	56.48	1.35 (H_1-14, m)
15	32.07	1.36 (H_1-15a, m), 1.94 (H_1-15b, m)
16	81.22	4.43 (H_1-16, m)
17	64.11	1.73 (H_1-17, m)
18	19.31	0.89 (H_3-18, s)
19	16.24	0.8 (H_3-19, s)
20	40.7	2.20 (H_1-20, dd, *J*=6.2, 12.7 Hz)
21	16.20	1.18 (H_3-21, d, *J*=6.8 Hz)
22	112.58	
23	30.73	1.73 (H_1-23a, m), 1.99 (H_1-23b, m)
24	28.11	1.32 (H_1-24a, m), 1.79 (H_1-24b, m)
25	34.14	1.88 (H_1-25, m)
26	75.13	3.96 (H_1-26a, m), 3.59 (H_1-26b, m)
27	17.06	0.99 (H_3-27, d, *J*=6.6 Hz)
-OCH_3	47.2	3.25 (3H, s)
3-O-Glc-1'	102.22	4.99 (1H, d, *J*=7.7 Hz)
2'	74.79	4.04 (1H, m)
3'	76.77	3.93 (1H, m)
4'	81.22	4.37 (1H, m)
5'	76.79	4.32 (1H, m)
6'	62.82	4.39 (1H, m), 4.55 (1H, m)
Glc(1→4)1"	104.94	5.22 (1H, d, *J*=7.8 Hz)
2"	71.67	4.21 (1H, m)
3"	75.13	4.04 (1H, m)
4"	74.72	4.12 (1H, m)
5"	78.58	4.23 (1H, m)
6"	62.1	4.48 (1H, m), 4.55 (1H, m)

26-O-Glc-1'''	104.94	4.84 (1H, d, *J*=7.7 Hz)
2'''	74.72	4.12 (1H, m)
3'''	78.18	4.23 (1H, m)
4'''	71.67	4.21 (1H, m)
5'''	78.48	4.01 (1H, m)
6'''	62.38	4.30 (1H, m), 4.55 (1H, m)

The chemical shift assignments of carbon and proton signals of **Dioscin B** (Compound **2**) in Pyridine-d_5.

Carbon NO.	**Dioscin B**	
	δ_C (ppm)	δ_C (ppm, *J* in Hz)
1	37.36	1.67 (H_2-1, m)
2	30.12	2.07 (H_2-2, m)
3	78.42	3.87 (H_1-3, m)
4	39.22	2.44 (H_1-4a, t, *J*=12.2), 2.68 (H_1-4b, d, *J*=13.4 Hz)
5	140.82	
6	121.67	5.30 (H_1-6, d, *J*=4.18 Hz)
7	32.17	1.84 (H_1-7a, m), 1.94 (H_1-7b, m)
8	31.52	1.50 (H_1-8, m)
9	50.19	0.86 (H_1-9, m)
10	39.66	
11	20.97	1.41 (H_2-11, m)
12	36.96	1.72 (H_2-12, m)
13	40.43	
14	56.48	1.35 (H_1-14, m)
15	32.07	1.36 (H_1-15a, m), 1.94 (H_1-15b, m)
16	81.22	4.43 (H_1-16, m)
17	64.11	1.73 (H_1-17, m)
18	16.2	0.8 (H_3-18, s)
19	19.31	0.89 (H_3-19, s)
20	40.71	2.20 (H_1-20, dd, *J*=6.2 Hz, 12.7 Hz)
21	16.25	1.18 (H_3-21, d, *J*=6.8 Hz)
22	112.58	
23	30.74	1.73 (H_1-23a, m), 1.99 (H_1-23b, m)
24	28.11	1.32 (H_1-24a, m), 1.79 (H_1-24b, m)
25	34.15	1.88 (H_1-25, m)
26	76.4	3.94 (H_1-26a, m), 4.04 (H_1-26b, m)
27	17.06	0.99 (H_3-27, d, *J*=6.6 Hz)
-OCH_3	47.2	3.25 (3H, s)
3-O-Glu-1'	102.23	4.99 (1H, d, *J*=7.7 Hz)
2'	78.59	4.37 (1H, m)
3'	76.8	3.93 (1H, m)
4'	75.14	4.04 (1H, m)

5'	78.18	4.32 (1H, m)
6'	62.82	4.39 (1H, m), 4.55 (1H, m)
Glu(1→2)1"	104.95	5.22 (1H, d, *J*=7.8 Hz)
2"	71.68	4.21 (1H, m)
3"	75.14	4.04 (1H, m)
4"	74.81	4.12 (1H, m)
5"	78.59	4.23 (1H, m)
6"	62.1	4.48 (1H, m), 4.55 (1H, m)
26-O-Glu-1"'	104.95	4.84 (1H, d, *J*=7.7 Hz)
2"'	74.73	4.12 (1H, m)
3"'	78.18	4.23 (1H, m)
4"'	71.45	4.21 (1H, m)
5"'	78.49	4.01 (1H, m)
6"'	62.38	4.30 (1H, m), 4.55 (1H, m)

The chemical shift assignments of carbon and proton signals of **Dioscin C** (Compound **3**) in Pyridine-d_5.

Carbon NO.	**Dioscin C**	
	δ_C (ppm)	δ_C (ppm, *J* in Hz)
1	37.37	1.68 (H_2-1, m)
2	30.07	2.09 (H_2-2, m)
3	78.05	4.26 (H_1-3, m)
4	38.85	2.72 (H_2-4, m)
5	140.66	
6	121.76	5.27 (H_1-6, br.s)
7	32.4	1.86 (H_1-7a, m), 2.02 (H_1-7b, m)
8	31.6	1.45 (H_1-8, m)
9	50.25	0.90 (H_1-9, m)
10	39.85	
11	21.02	1.45 (H_2-11, m)
12	37.13	2.02 (H_2-12, m)
13	40.7	
14	57.28	1.11 (H_1-14, m)
15	32.24	1.45 (H_1-15a, m), 2.02 (H_1-15b, m)
16	81.03	4.95 (H_1-16, m)
17	63.77	1.92 (H_1-17, m)
18	17.39	0.89 (H_3-18, s)
19	19.13	1.03 (H_3-19, s)
20	40.6	2.23 (H_1-20, m)
21	16.38	1.31 (H_3-21, d, *J*=6.4 Hz)
22	110.57	
23	37.04	1.75 (H_1-23a, m), 2.02 (H_1-23b, m)
24	28.28	1.67 (H_1-24a, m), 2.02 (H_1-24b, m)

25	34.21	1.92 (H_1-25, m)
26	75.19	3.61 (H_1-26a, m), 3.95 (H_1-26b, m)
27	18.59	0.98 (H_3-27, d, *J*=6.5 Hz)
3-O-Glc-1'	99.91	4.95 (1H, d, *J*=7.2 Hz)
2'	77.66	4.26 (1H, m)
3'	76.12	4.26 (1H, m)
4'	81.97	4.26 (1H, m)
5'	77.18	4.26 (1H, m)
6'	61.99	4.35 (1H, m), 4.56 (1H, m)
Glc(1→4)1"	105.15	6.4 (1H, d, *J*=7.3 Hz)
2"	74.89	4.04 (1H, m)
3"	78.42	4.26 (1H, m)
4"	81.96	4.26 (1H, m)
5"	78.24	3.95 (1H, m)
6"	61.84	4.46 (1H, m), 4.56 (1H, m)
Rha(1→4)1"'	101.74	6.26 (1H, br.s)
2"'	72.39	4.74 (1H, m)
3"'	72.7	4.56 (1H, m)
4"'	74.05	4.35 (1H, m)
5"'	69.4	4.95 (1H, m)
6"'	19.31	1.75 (3H, d, *J*=5.67 Hz)
26-O-Glc-1""	104.87	4.82 (1H, d, *J*=7.6 Hz)
2""	75.12	4.04 (1H, m)
3""	78.53	4.26 (1H, m)
4""	71.6	4.26 (1H, m)
5""	78.21	4.26 (1H, m)
6""	62.73	4.35 (1H, m), 4.56 (1H, m)