Supporting Information

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Cyclocarioside Z15, a new 3,4-*seco* dammarane triterpenoid glycoside from the leaves of *Cyclocarya paliurus* with *α*-glucosidase inhibitory activity

Qian Yao[1], Xi-Fan Wei[1], Lu Wang[1], Rui Gui[1], Yi-Heng Liu[3], Dan-Dan Li[2], Xiao-Ai He[3], Xia Yu[1], Gui-Ming Deng[2,*] and Kang-Ping Xu[1,*]

[1]*Xiangya School of Pharmaceutical Sciences, Central South University, Changsha 410013, China*

[2]*The First Hospital of Hunan University of Chinese Medicine, Changsha, 410208, China*

[3]*Haikou People's Hospital and Central South University Xiangya School of Medicine Affiliated Haikou Hospital, Haikou, 570208, China*

[*]Corresponding Author: Kang-Ping Xu. Email: xukp395@csu.edu.cn; Gui-Ming Deng. Email: dengguiming@hnucm.edu.cn.

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Experimental

General experimental procedures

Optical rotation was measured on SGW-3 automatic polarimeter (Shanghai INESA Physico-Optical Instrument Co., Ltd, Shanghai, China) at 25℃. HR-ESI-MS spectra were recorded on UHPLC-Vanquish H/Q Exactive system (Thermo Fisher, MA, USA) in negative ion mode. 1D and 2D NMR spectra were performed on Bruker AV-600 MHz spectrometer (Bruker, Karlsruhe, Germany) with TMS as an internal standard. Silica gel (80-100, 200-300 or 300-400 mesh, Qingdao Peremanent Sea Silica Ltd., Qingdao, China), HP20 macroporous adsorption resin (Mitsubishi Chemical Corporation, Tokyo, Japan), polyamide (80-100 mesh, Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) were used for column chromatograph; Silica gel TLC plates (GF254, Yantai Jiangyou Silicone Development Co., Ltd.) were used for qualitative analysis. Analytical HPLC experiments were implemented with a H&E SP ODS-A column (5μm, 4.6 mm × 250 mm) in Agilent 1260 (Agilent Technologies, Ltd.) equipped with a diode array detector (DAD) under reversed-phase. Both semipreparative and preparative HPLC separations were conducted by using Shimadzu system with a H&E SP ODS-A column (5 μm, 250 × 10 mm; 5 μm, 250 × 20 mm). GC-MS spectra were conducted by Thermo Scienlific Q Exactive GC system (Thermo Fisher, MA, USA) with a TG-5MS (0.32 mm × 30.0 m, 0.25 μm). Unless otherwise specified, all chemical reagents involved in this study are analytical grade and purchased from Chengdu Kelong Chemical Co.,Ltd.

Biological Activity Assay

The α-Gucosidase Inhibitor Activity Assay Kit was purchased from Beijing Solarbio Science & Technology Co.,Ltd. (BC6385). All compounds were evaluated for their α-glucosidase inhibitory activity according to the manufacturer's kit instructions.

Acid hydrolysis

Compound **1** (1.0 mg) was weighed in a 25 mL round bottom flask, and 1 M HCl (2 mL) was added to stir and reflux in a water bath (80°C, 2 h). Na_2CO_3 was used for neutralization reaction. Then the reaction mixture was extracted with ethyl acetate (EtOAc) for three times, and the aqueous layer was concentrated and dried to yield the sugar moiety. The mixture was dissolved in pyridine (1 mL). Subsequently, L-cysteine methyl ester hydrochloride (2.0 mg) was added, and the solution was stirred and refluxed in an oil bath (60°C, 1 h). After the reaction, trimethylsilylimidazole (1 mL) was added, and the mixture was stirred in a water bath (60°C, 30 min). The sample was filtered through a microporous membrane and analyzed by GC-MS (front inlet 300℃, column 60℃-300℃ at 15℃/min). L-arabinopyranose was subjected to the same derivatization procedure.

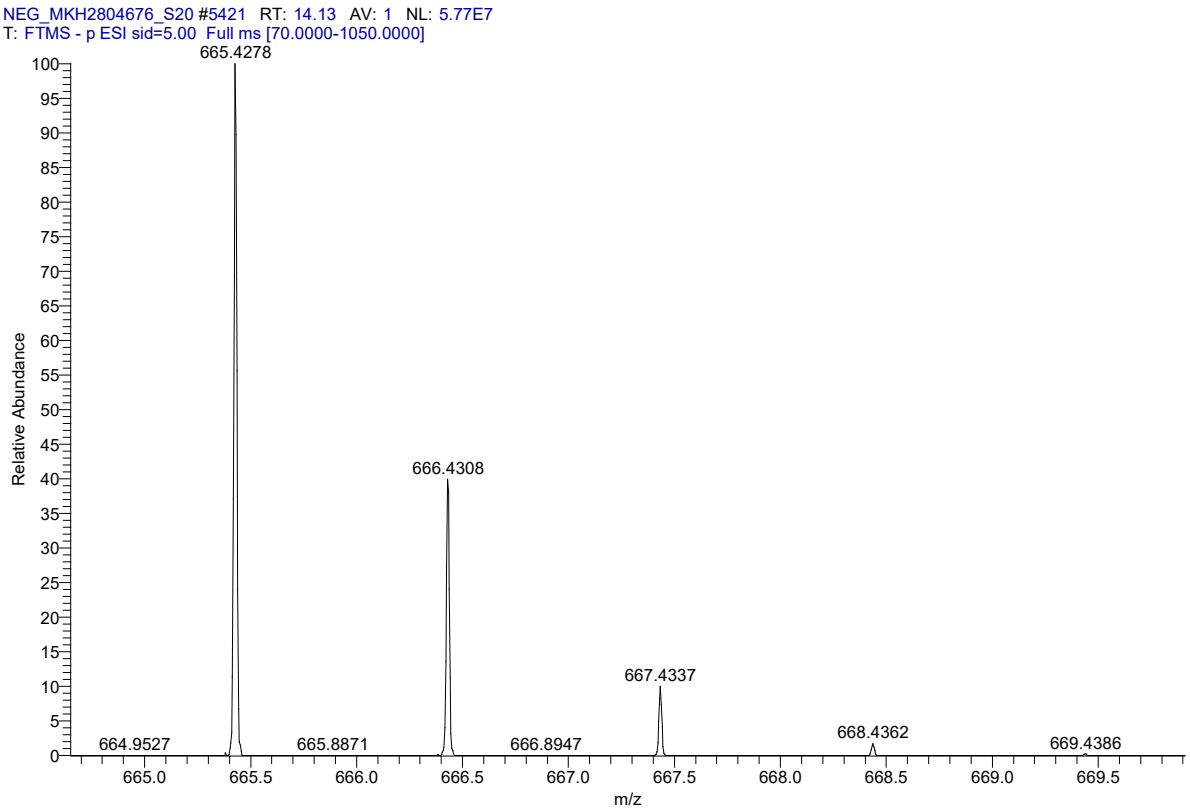


Figure S1: The HR-ESI-MS spectrum of compound **1**

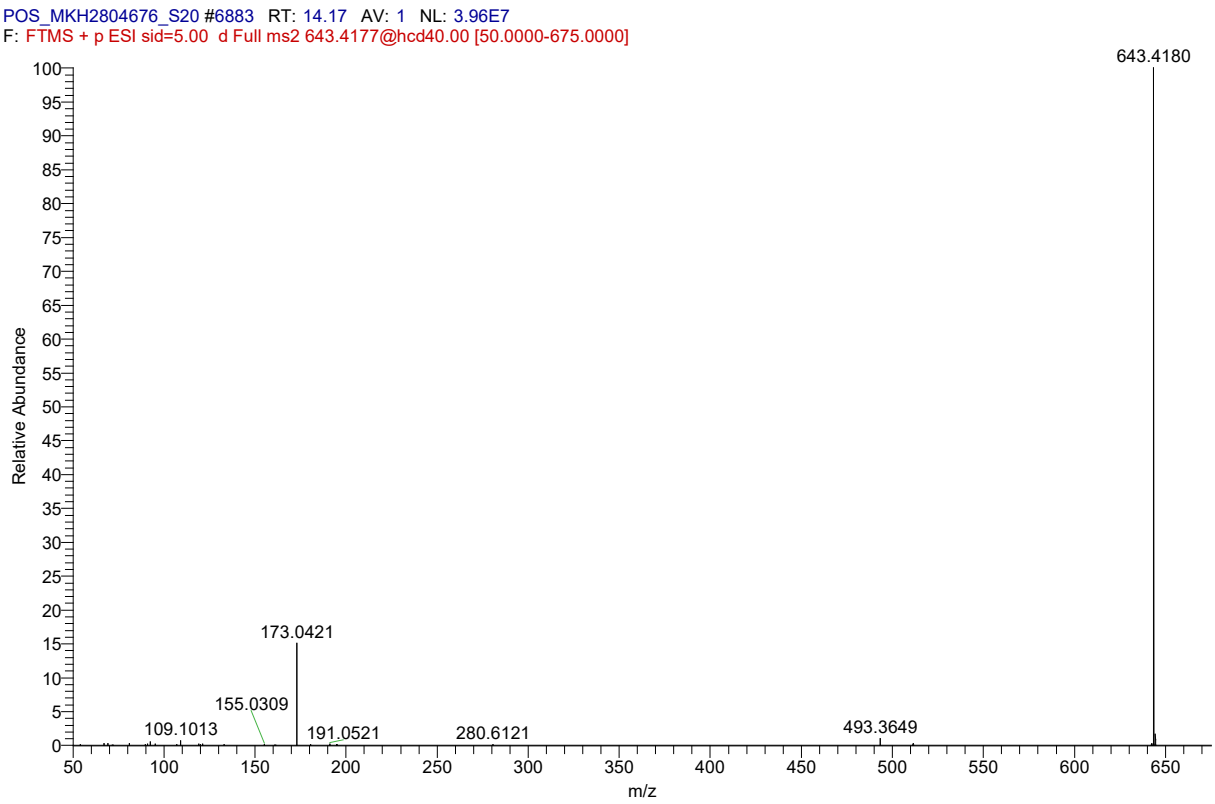


Figure S2: MS/MS spectrum of compound **1** (positive-ion mode)

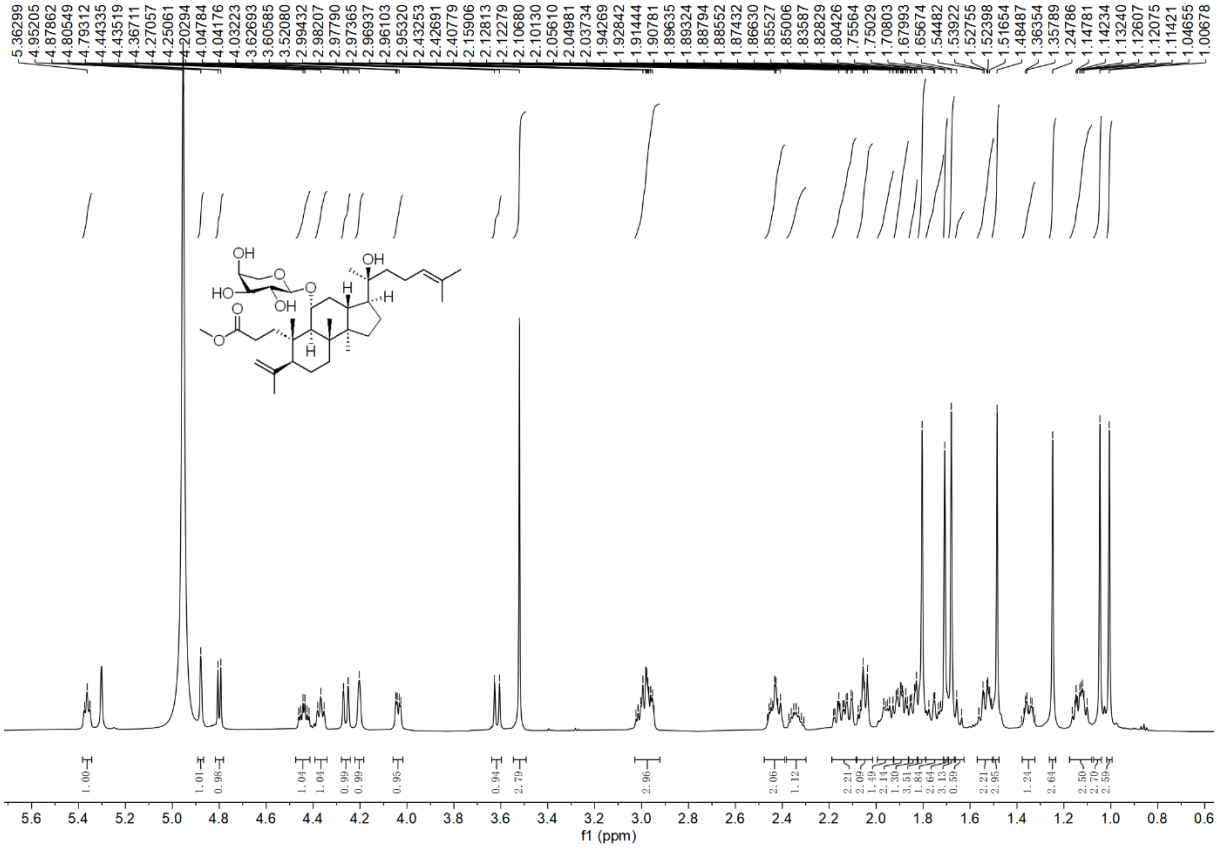


Figure S3: The ^{1}H NMR spectrum of compound **1** (Pyridine-d_5, 600 MHz)

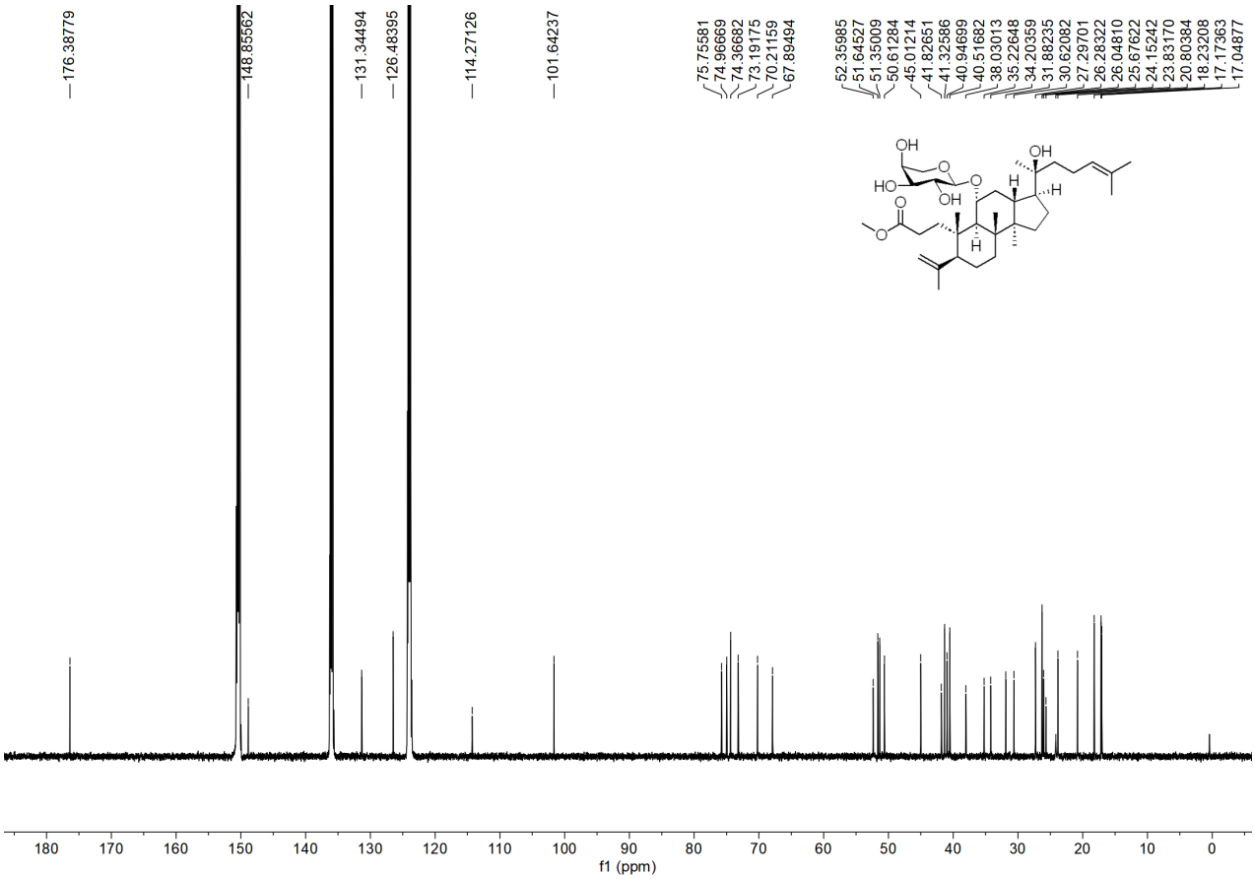


Figure S4: The ^{13}C NMR spectrum of compound **1** (Pyridine-d_5, 150 MHz)

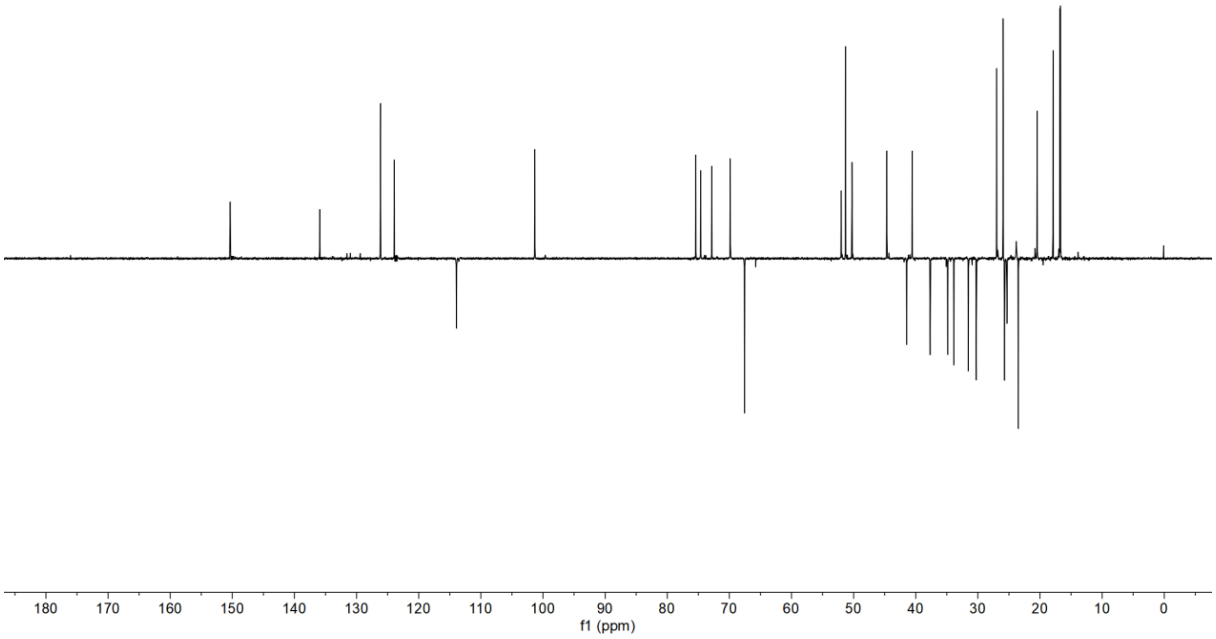


Figure S5: The DEPT 135 spectrum of compound **1** (Pyridine-d_5, 150 MHz)

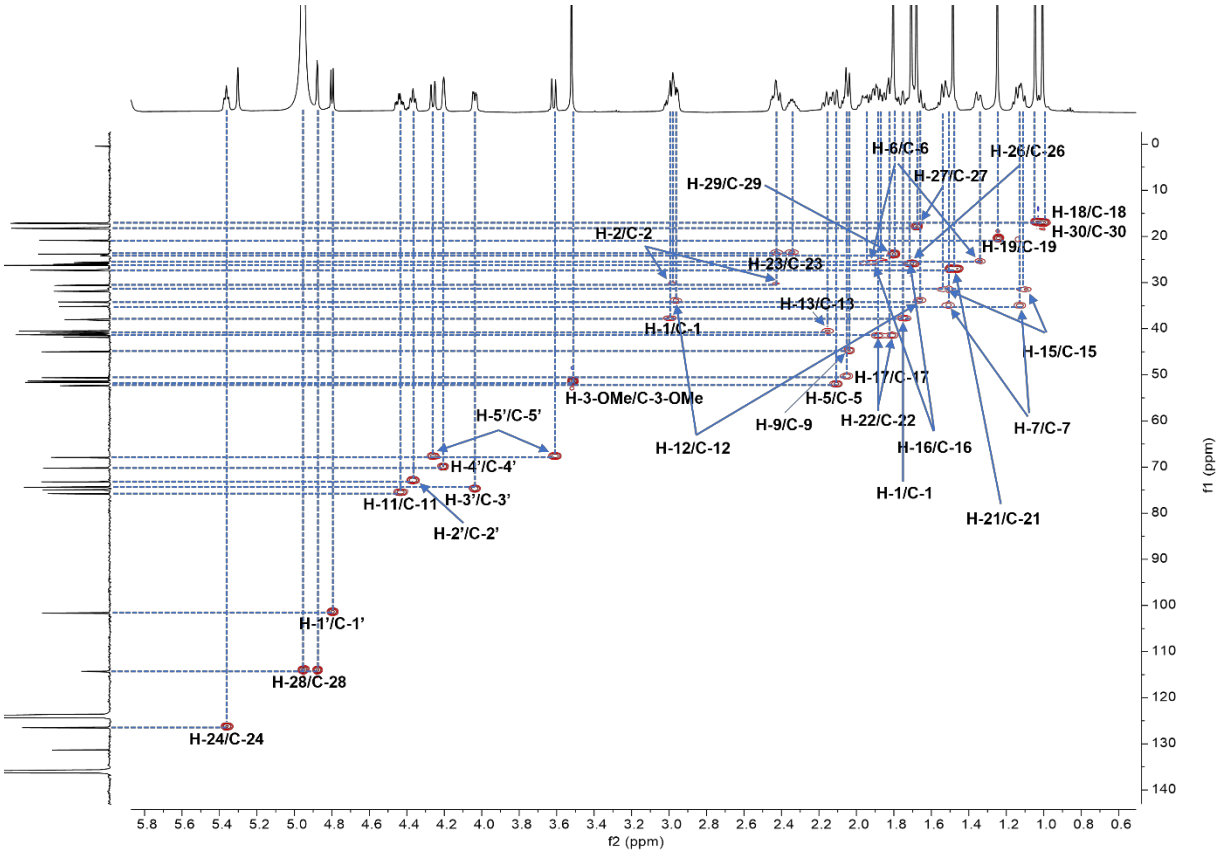


Figure S6: The HSQC spectrum of compound **1**

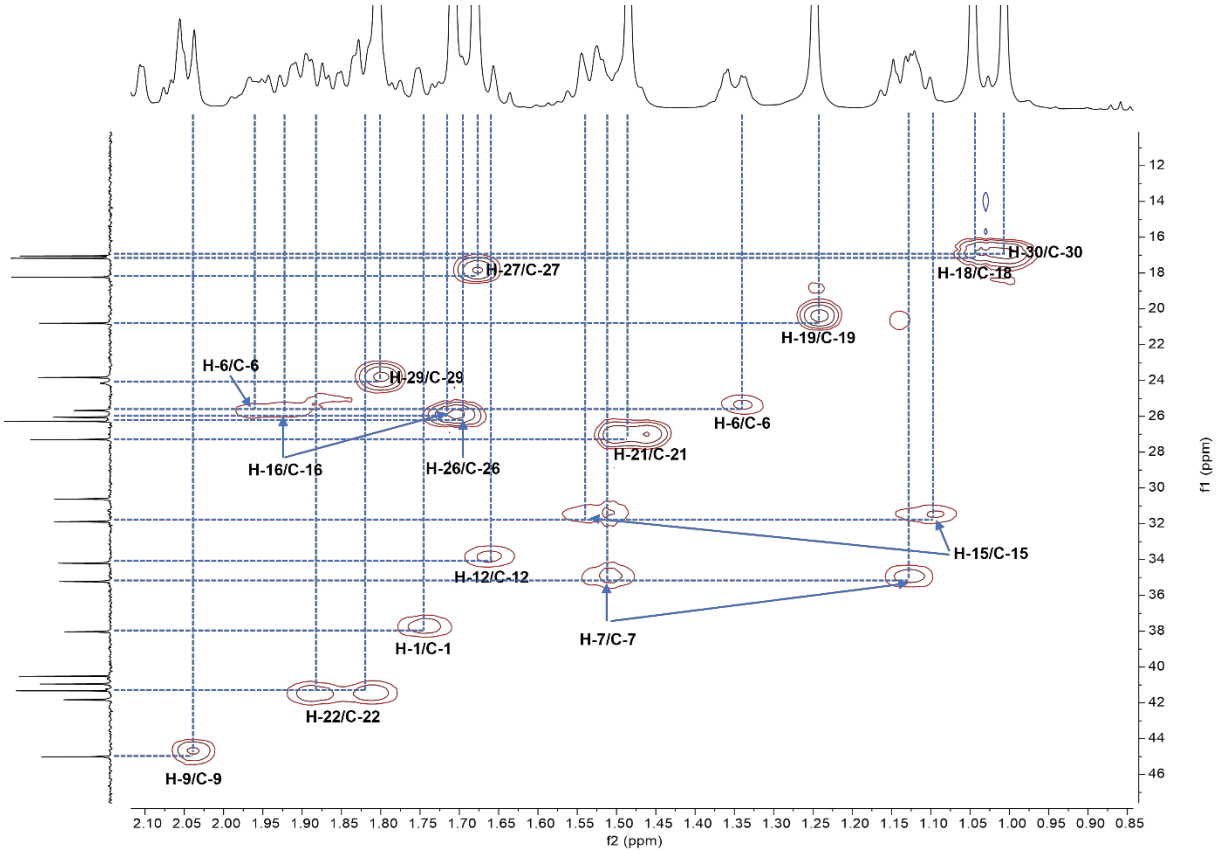


Figure S7: Enlarged HSQC spectrum of compound **1**

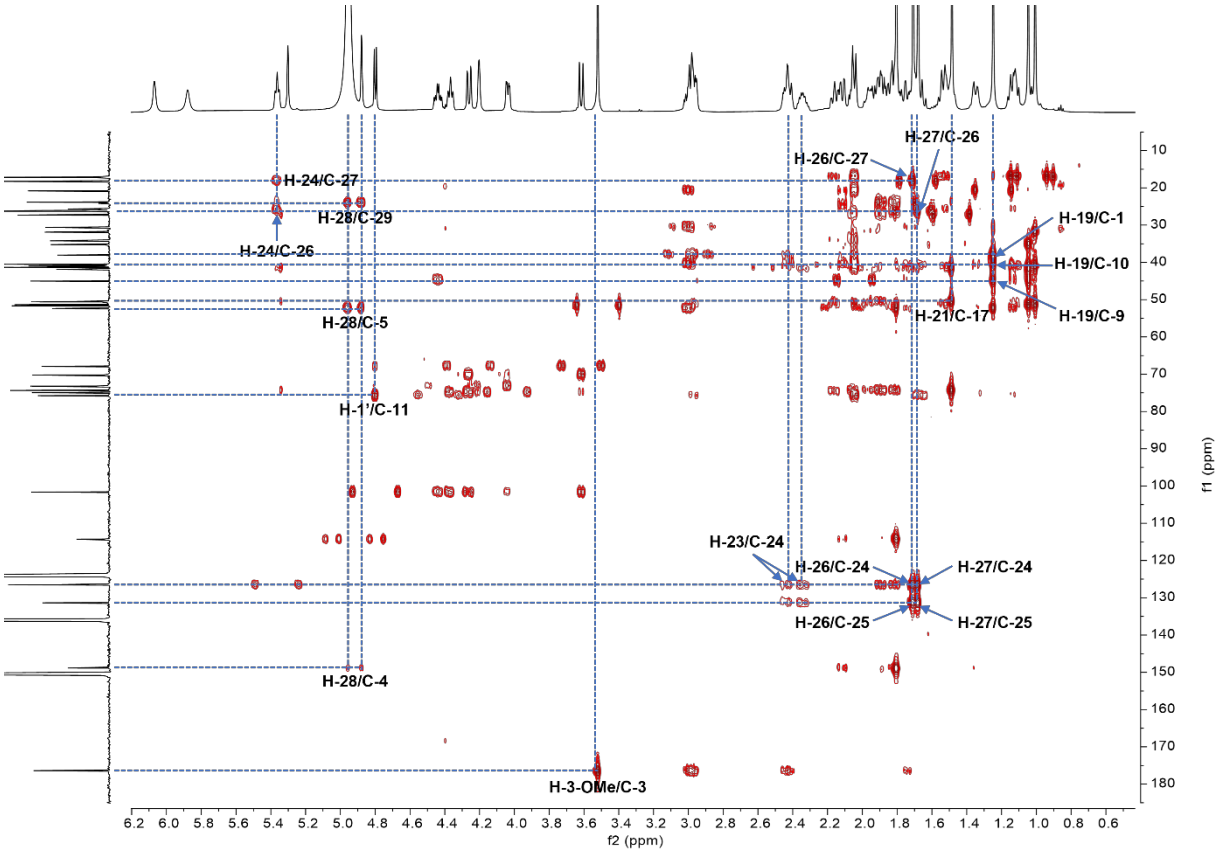


Figure S8: The HMBC spectrum of compound **1**

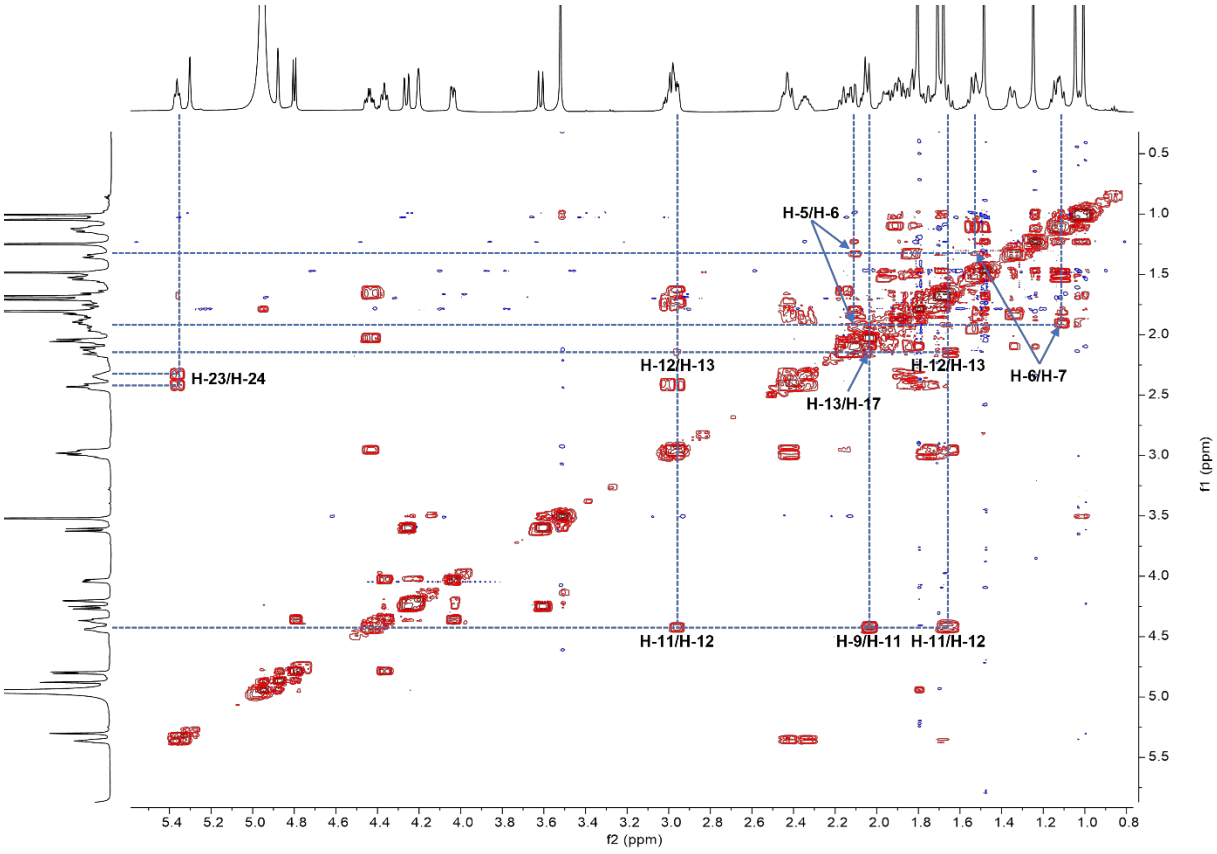


Figure S9: The ^{1}H-^{1}H COSY spectrum of compound **1**

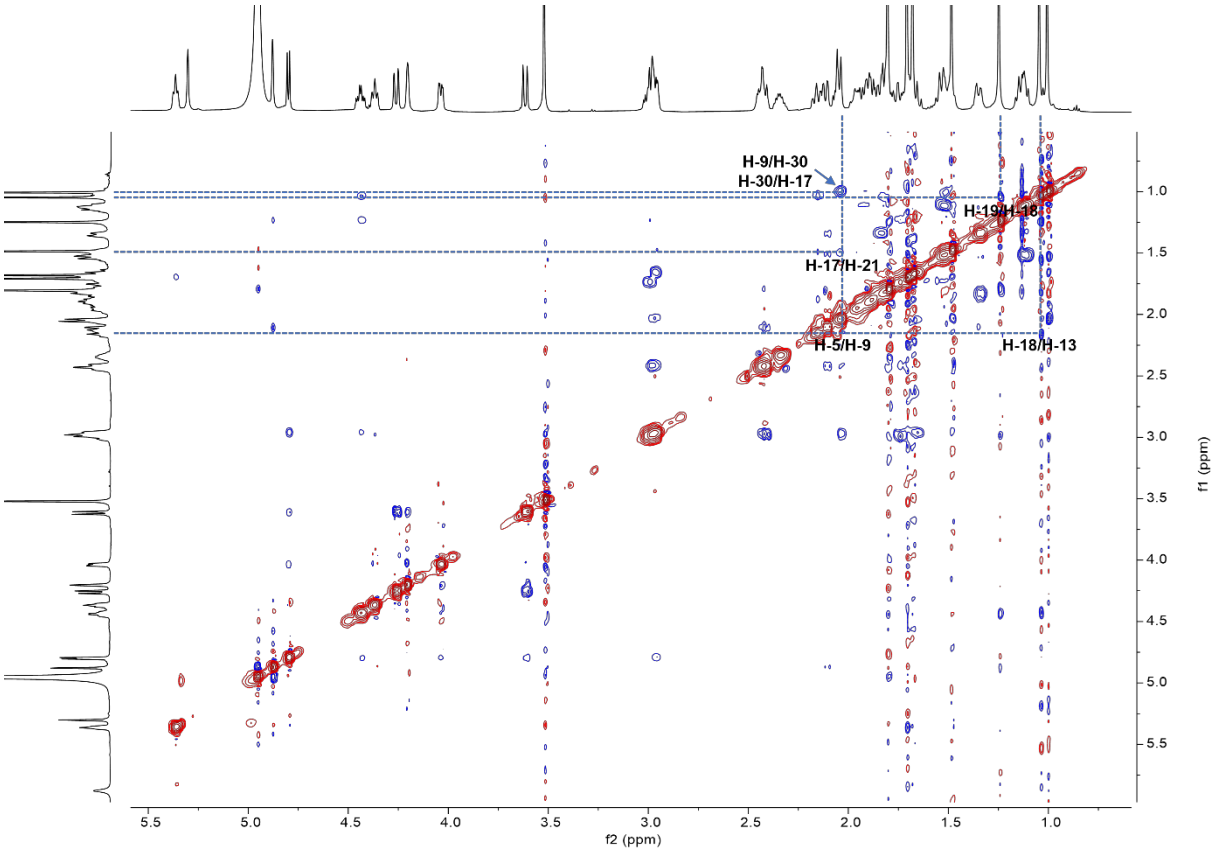


Figure S10: The ROESY spectrum of compound **1**

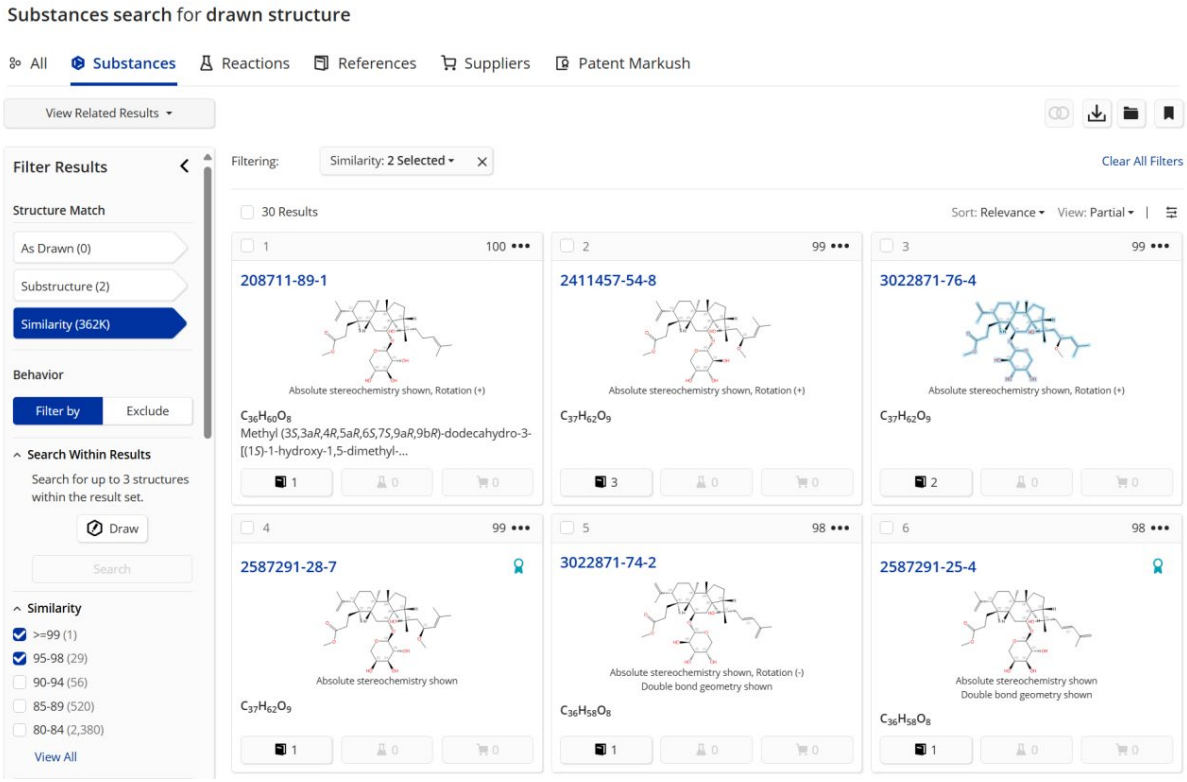


Figure S11: Scifinder similarity report for compound **1**

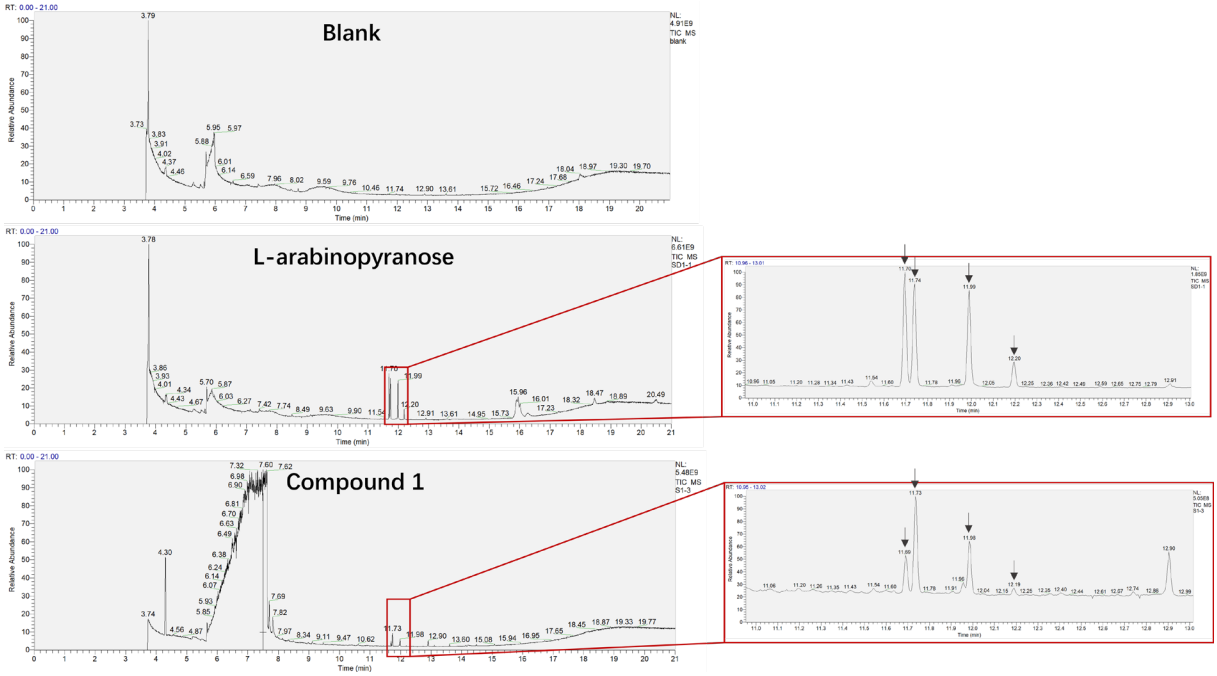
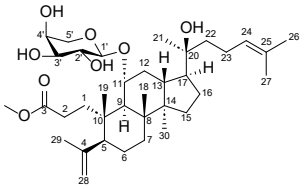
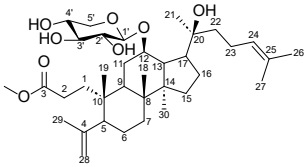
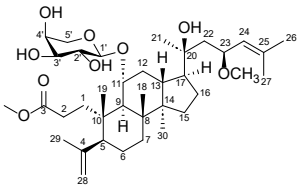


Figure S12: GC-MS spectra of compound **1** and L-arabinopyranose

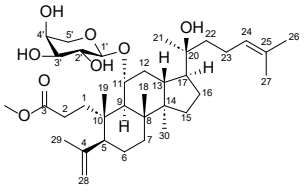
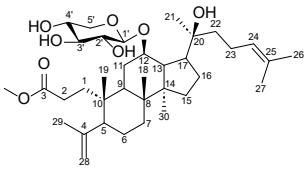
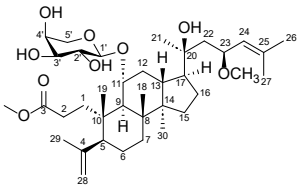
Table S1: ^{1}H NMR data of compound **1** (cyclocarioside Z15), betula-schmidtoside A and cypaliuruside D

Name	cyclocarioside Z15	betula-schmidtoside A[a]	cypaliuruside D
CAS Registry Number	-	208711-89-1	3022871-76-4
Structure			
position	δ_H (Pyridine-d_5, 600 MHz)	δ_H (Pyridine-d_5)	δ_H (Pyridine-d_5, 500 MHz)
1	1.75, 2.98, m	-	1.71, 2.96, m
2	2.43, 2.98, m	-	2.40, 2.96, m
3	-	-	-
4	-	-	-
5	2.14, m	-	2.08, m
6	1.35, 1.95, m	-	1.32, 1.81, m
7	1.13, 1.53, m	-	1.09, 1.49, m
8	-	-	-
9	2.05, m	-	2.01, d (10.9)
10	-	-	-
11	4.44, td (10.7, 4.8)	-	4.45, m
12	1.65, 2.98, m	-	1.65, 3.00, m
13	2.14, m	-	2.10, m
14	-	-	-
15	1.13, 1.53, m	-	1.06, 1.47, m
16	1.75, 1.89, m	-	1.81, 1.84, m
17	2.05, m	-	1.95, m
18	1.05, s	0.87, s	1.02, s
19	1.25, s	0.94, s	1.23, s
20	-	-	-
21	1.48, s	1.33, s	1.47, s
22	1.84, 1.89, m	-	1.73, 2.16, m
23	2.34, 2.43, m	-	4.42, m
24	5.36, t (7.0)	5.33, t (7.0)	5.21, d (8.7)
25	-	-	-
26	1.71, s	1.64, s	1.71, m
27	1.68, s	1.64, s	1.71, m

28	4.88, 4.95, br s	4.76, 4.92, s	4.85, 4.93, s
29	1.80, s	1.71, s	1.78, s
30	1.01, s	0.74, s	0.96, s
1’	4.80, d (7.4)	5.18, d (7.6)	4.80, d (7.5)
2’	4.37, t (8.1)	-	4.34, m
3’	4.04, dd (9.3, 3.6)	4.30, dd (11.0, 4.9)	4.01, dd (9.3, 3.6)
4’	4.20, br s	-	4.17, m
5’	3.62, d (12.6); 4.26, d (12.0)	-	3.59, d (9.3) 4.23, d (12.4, 1.9)
3-OMe	3.52, s	-	3.50, s
23-OMe	-	-	3.22, s

[a] “-” means the signal is not attributed.

Table S2: ^{13}C NMR data of compound **1** (cyclocarioside Z15), betula-schmidtoside A and cypaliuruside D

Name	cyclocarioside Z15	betula-schmidtoside A	cypaliuruside D
CAS Registry Number	-	208711-89-1	3022871-76-4
Structure			
position	δ_C (Pyridine-d_5, 150 MHz)	δ_C (Pyridine-d_5)	δ_C (Pyridine-d_5, 150 MHz)
1	38.0	24.7	37.5
2	30.6	28.6	30.1
3	176.4	174.3	175.9
4	148.9	147.5	148.4
5	52.4	40.5	51.9
6	25.7	28.7	25.2
7	35.2	33.5	34.8
8	51.4	39.7	50.7
9	45.0	50.3	44.5
10	40.5	39.7	40.0
11	75.8	28.2	75.3
12	34.2	76.5	33.7
13	41.0	46.8	40.4
14	41.8	52.8	40.9
15	31.9	31.5	31.3
16	26.1	27.2	25.6
17	50.6	53.9	51.2
18	17.2	15.5	16.6
19	20.8	20.2	20.3
20	75.0	72.7	74.2
21	27.3	27.0	27.5
22	41.3	34.4	44.7
23	23.8	23.0	75.7
24	126.5	126.6	127.0
25	131.3	130.5	135.4
26	26.3	17.7	25.7
27	18.2	25.8	18.2

28	114.3	114.0	16.6
29	24.2	23.3	23.7
30	17.1	17.3	113.8
1’	101.6	100.6	101.2
2’	73.2	75.0	72.7
3’	74.4	78.8	74.4
4’	70.2	70.7	69.7
5’	67.9	67.4	67.4
3-OMe	51.7	51.5	51.2
23-OMe	-	-	55.1