Supplementary Materials

Potent xanthine oxidase inhibitory activity of constituents of *Agastache rugosa* (Fisch. & C.A.Mey.) Kuntze

Heung Joo Yuk [1], HyungWon Ryu [2],* and Dong-Seon Kim [1],*

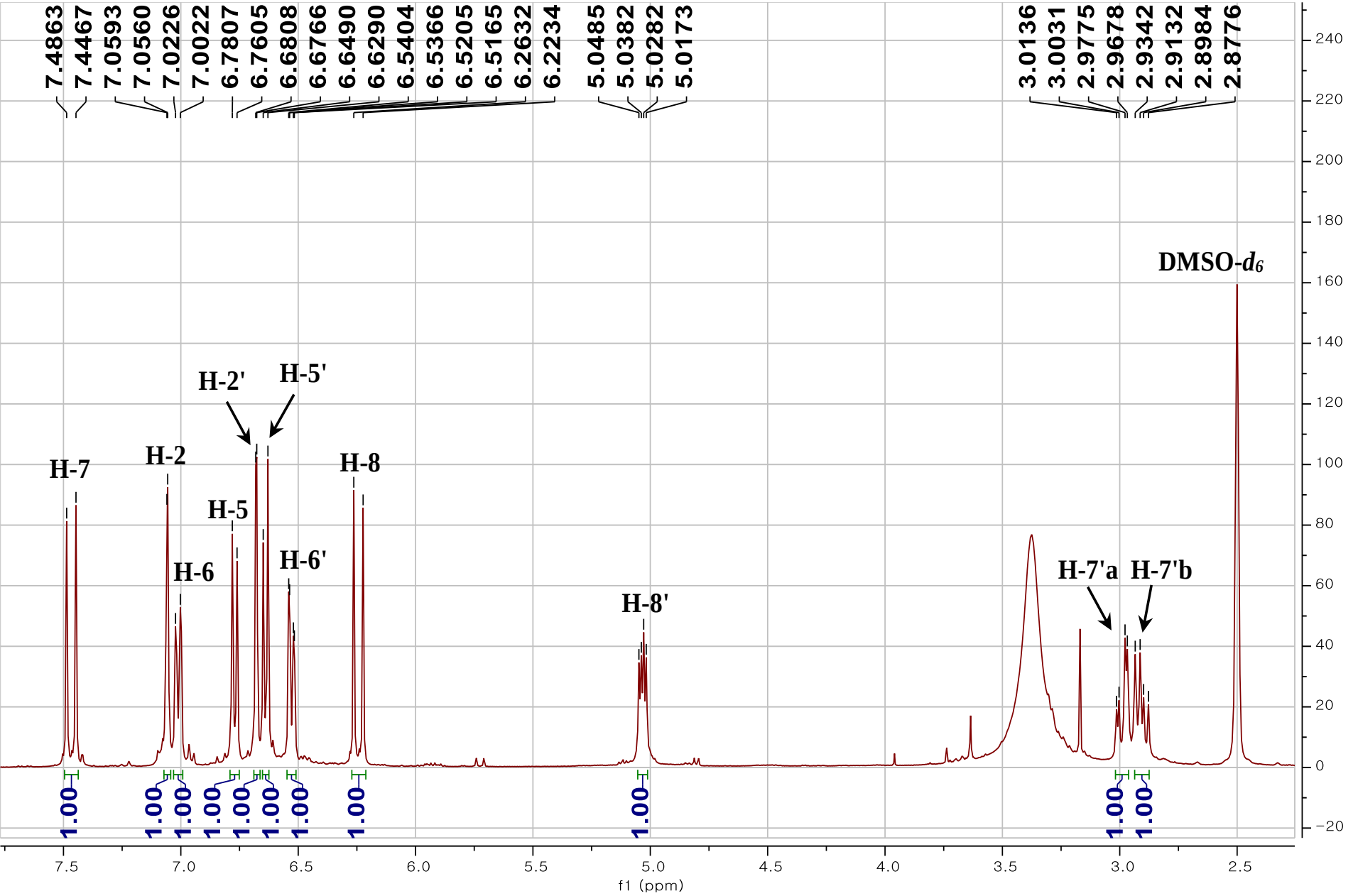

Figure S1. The ^{1}H NMR spectrum of compound **1** (400 MHz, DMSO-d_6).

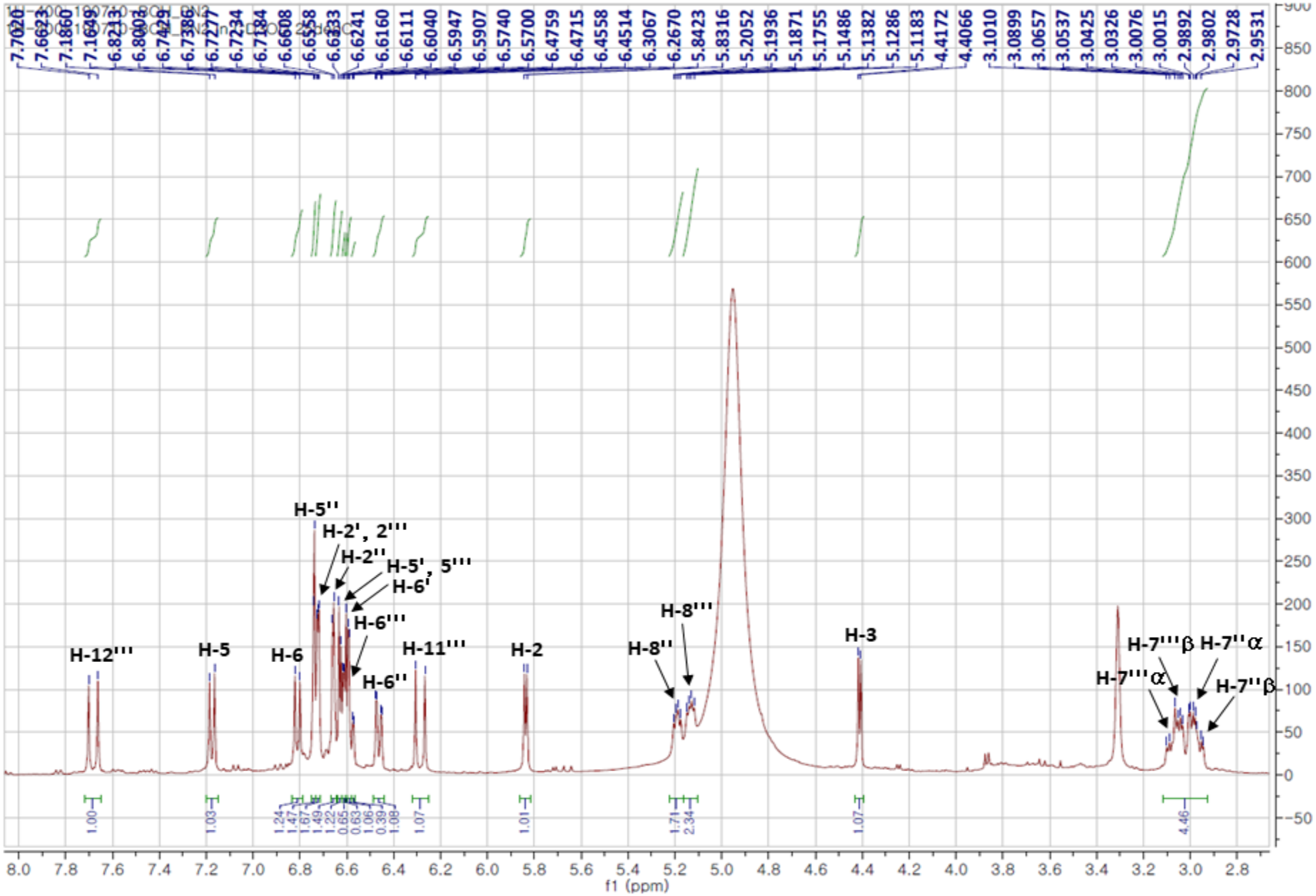

Figure S2. The ^{1}H NMR spectrum of compound **2** (500 MHz, CD_3OD).

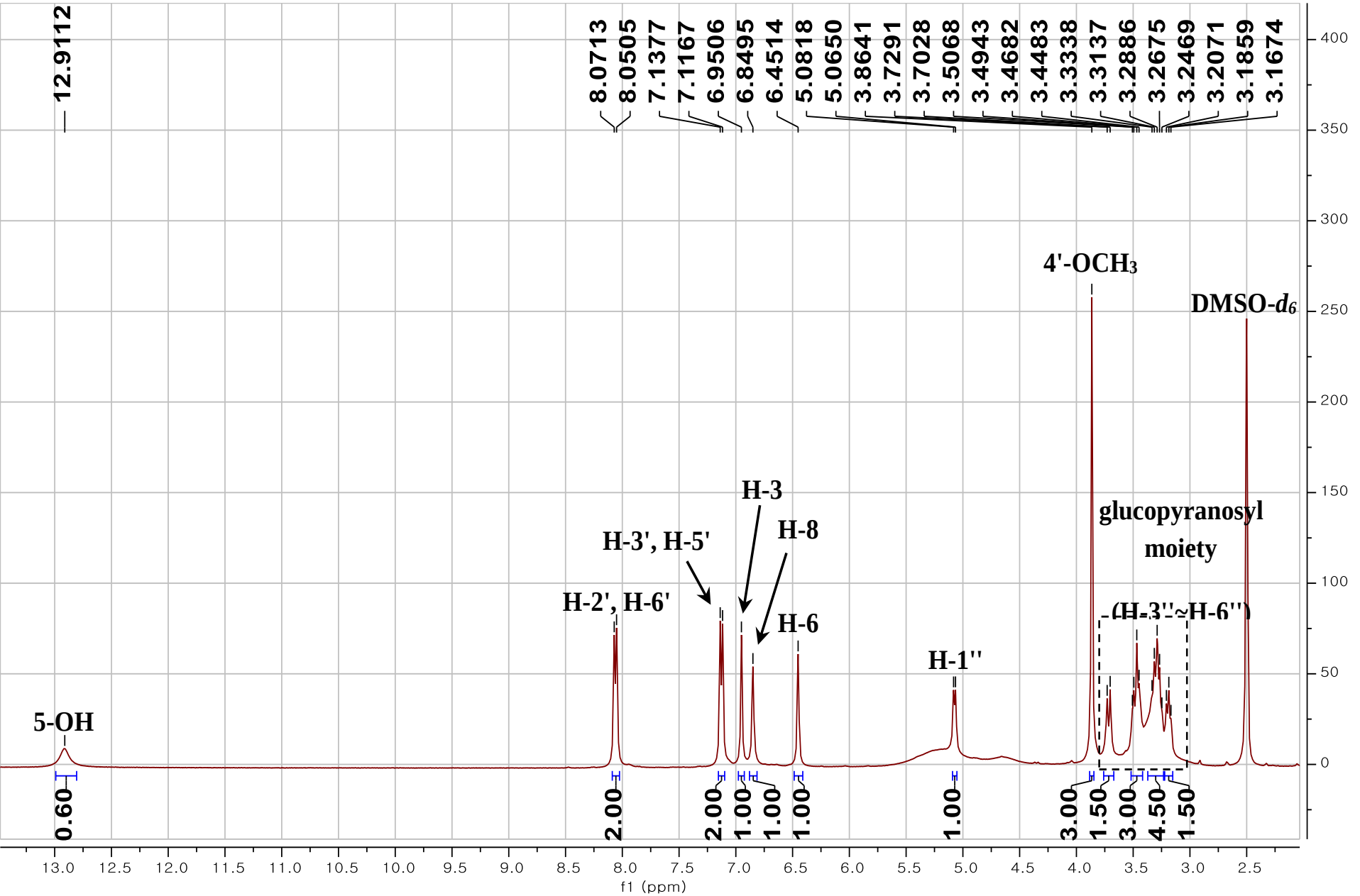

Figure S3. The ^{1}H NMR spectrum of compound **3** (400 MHz, DMSO-d_6).

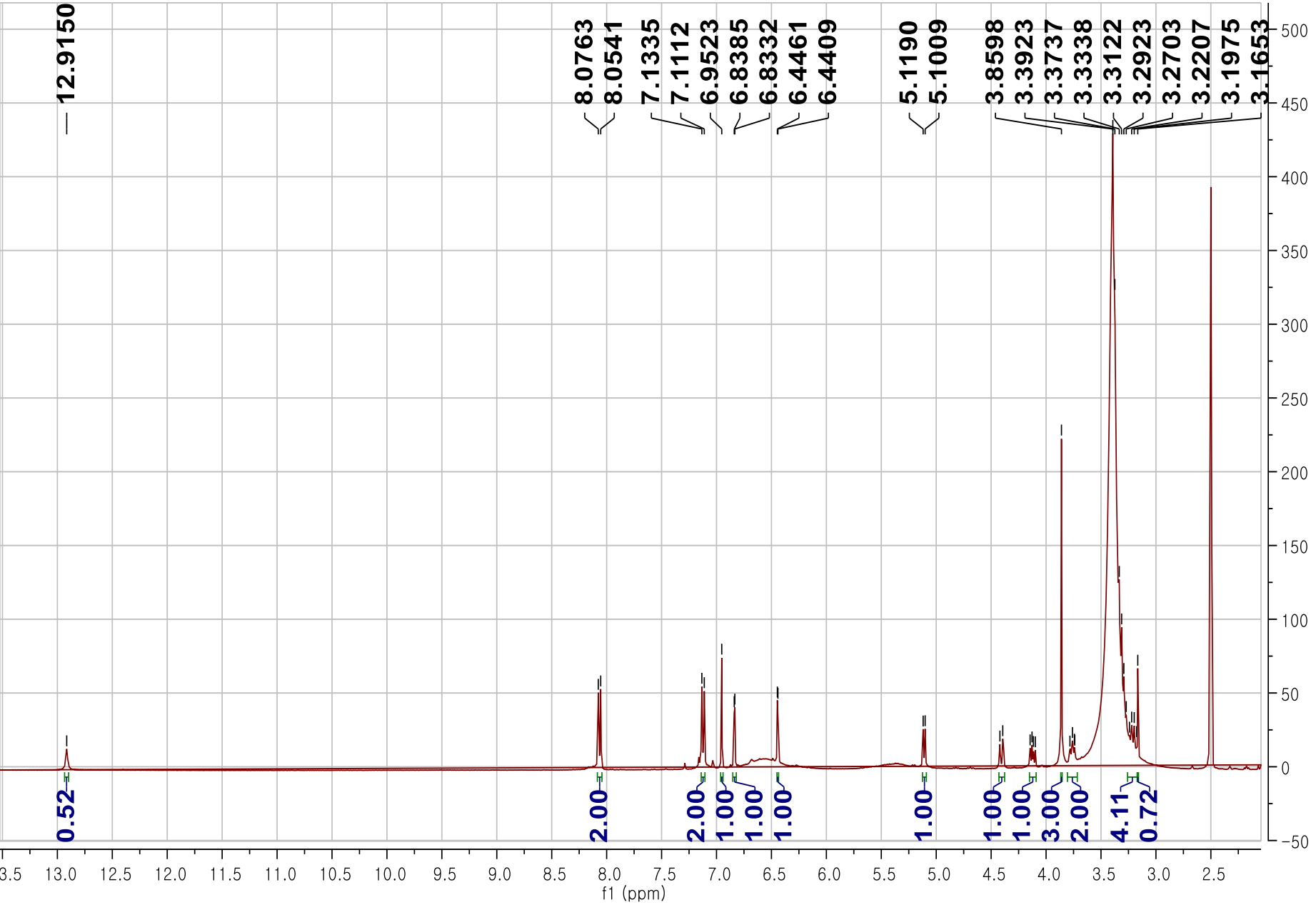

Figure S4. The ^{1}H NMR spectrum of compound **4** (400 MHz, DMSO-d_6).

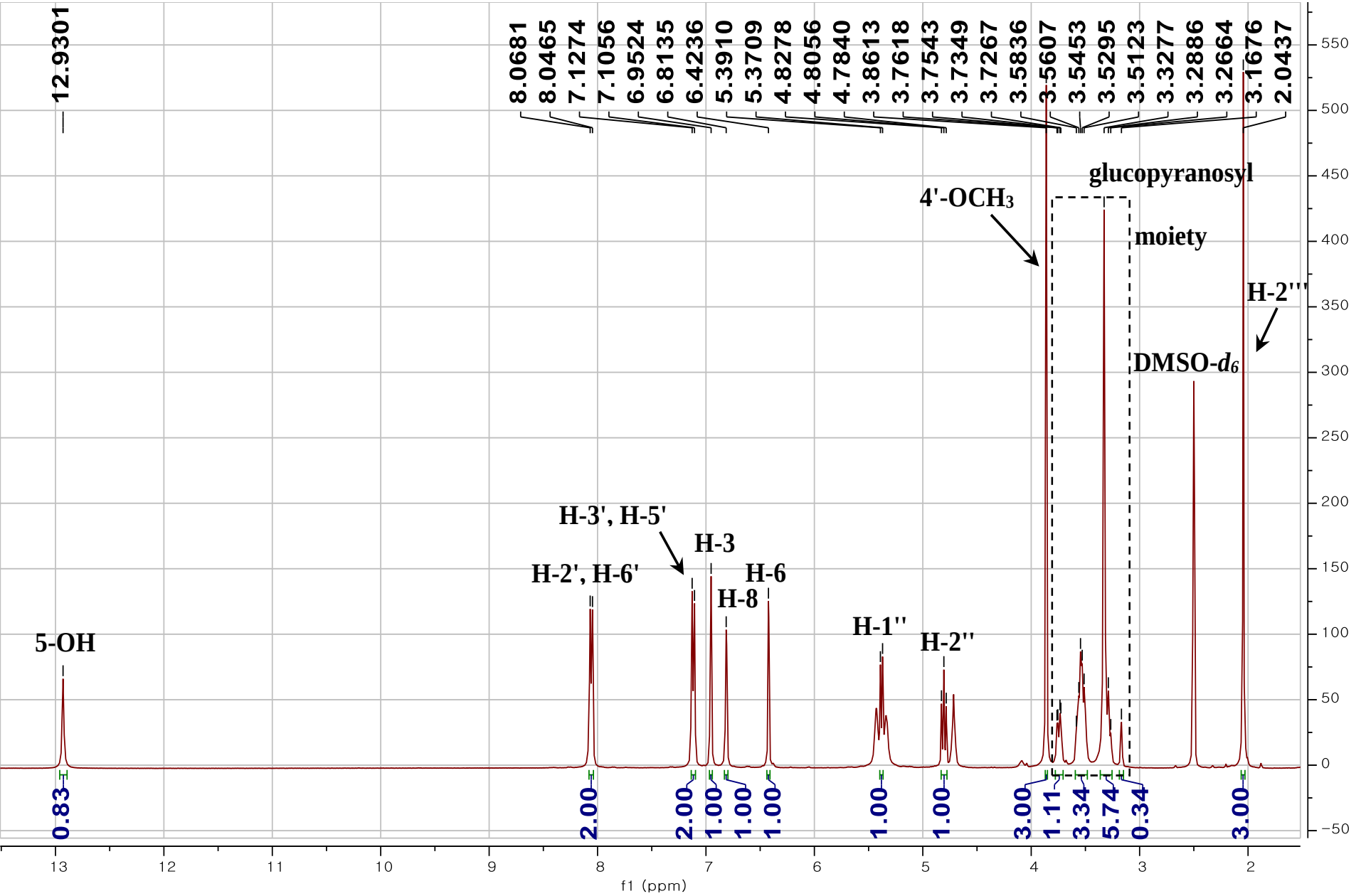

Figure S5. The ^{1}H NMR spectrum of compound **5** (400 MHz, DMSO-d_6).

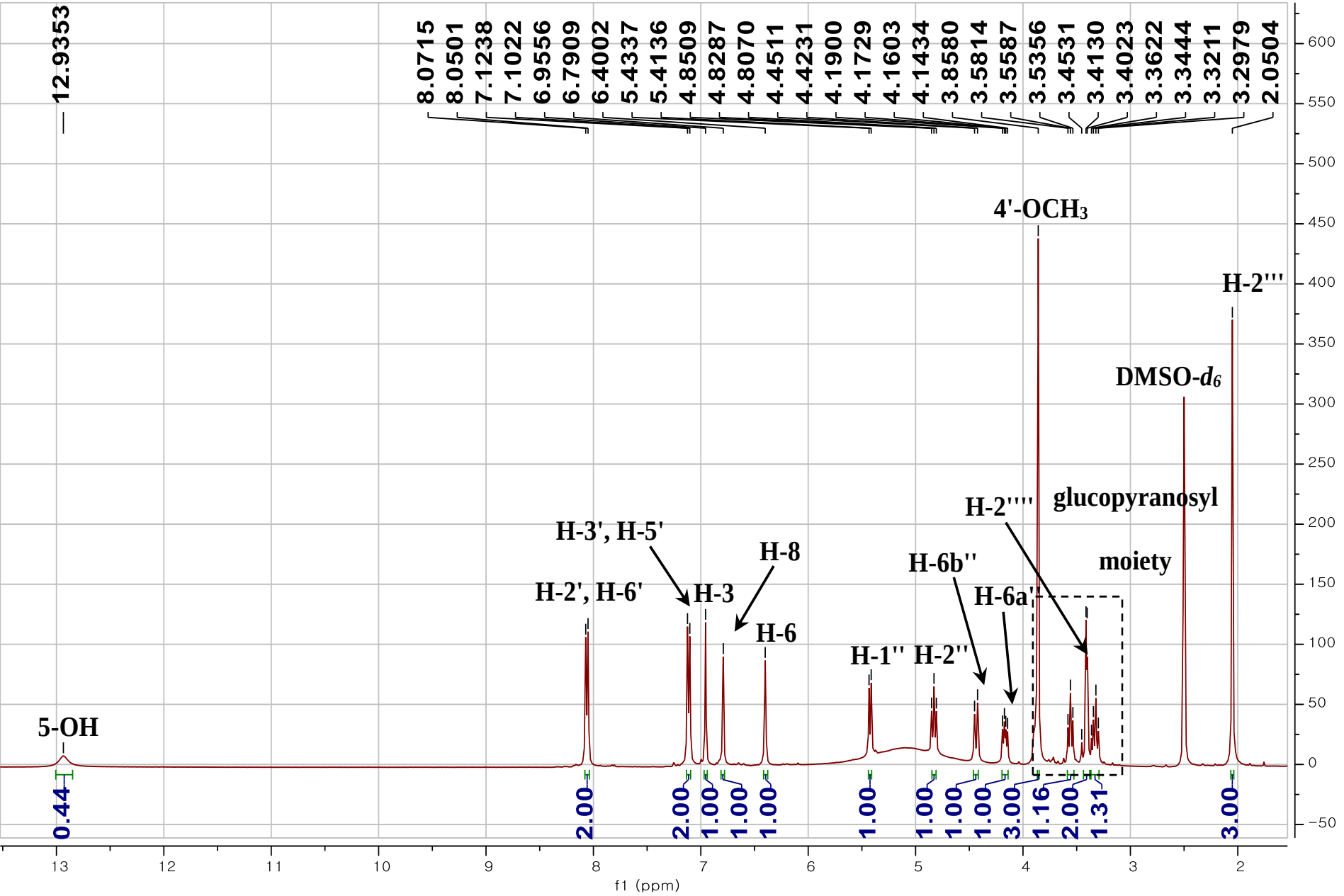

Figure S6. The ^{1}H NMR spectrum of compound **6** (400 MHz, DMSO-d_6).

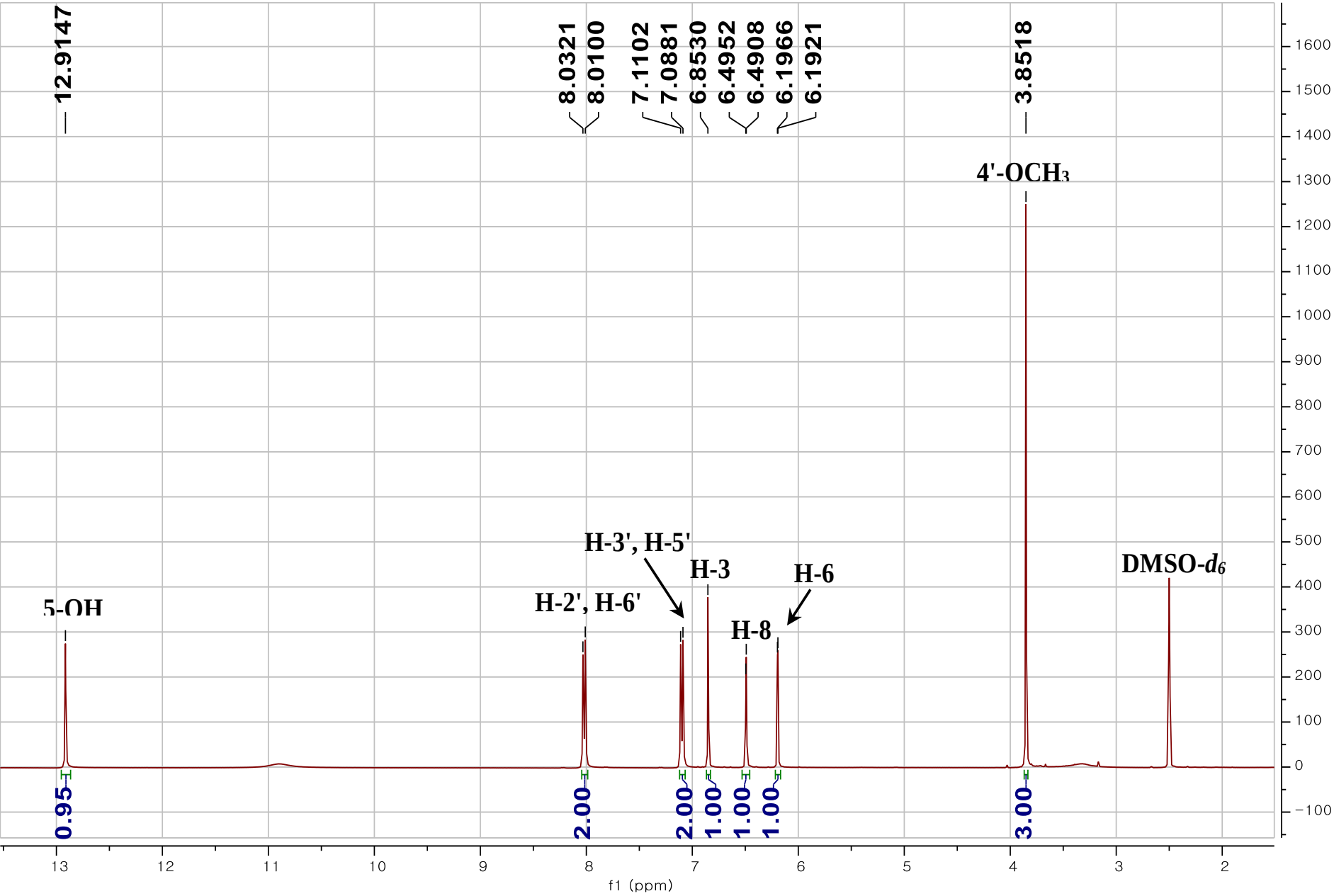

Figure S7. The ^{1}H NMR spectrum of compound **7** (400 MHz, DMSO-d_6).

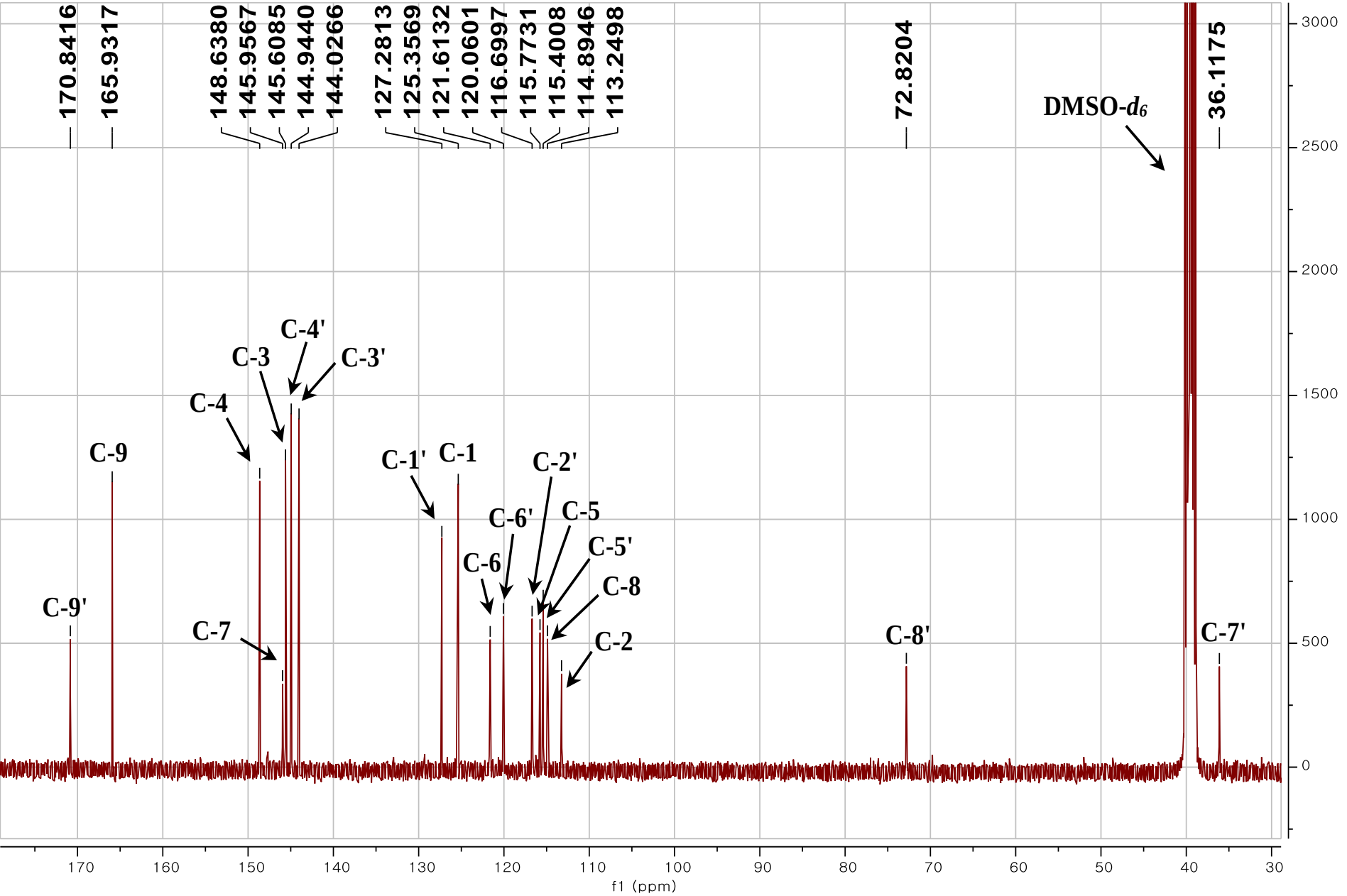

Figure S8. The ^{13}C NMR spectrum of compound **1** (100 MHz, DMSO-d_6).

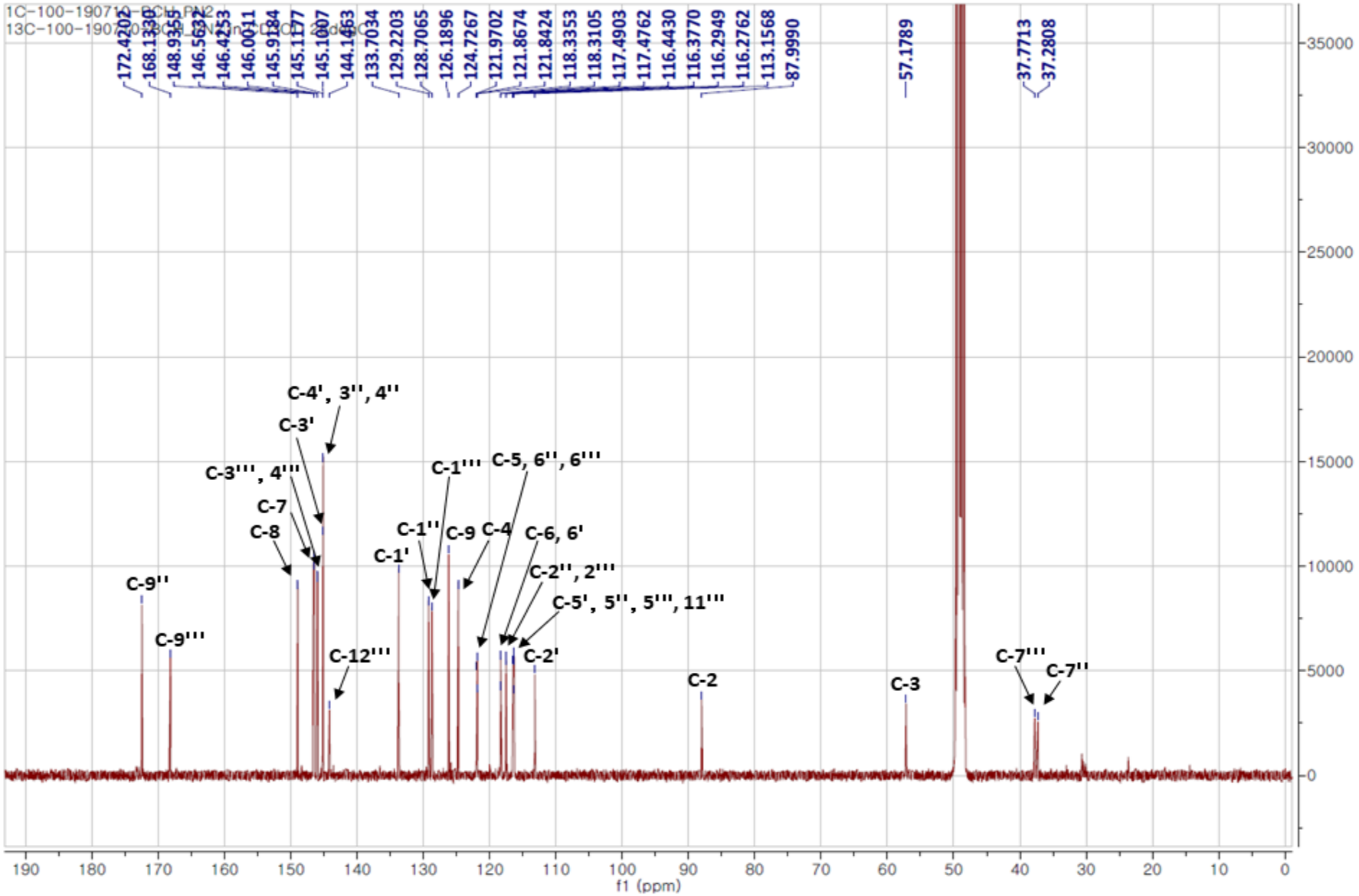

Figure S9. The ^{13}C NMR spectrum of compound **2** (125 MHz, CD_3OD).

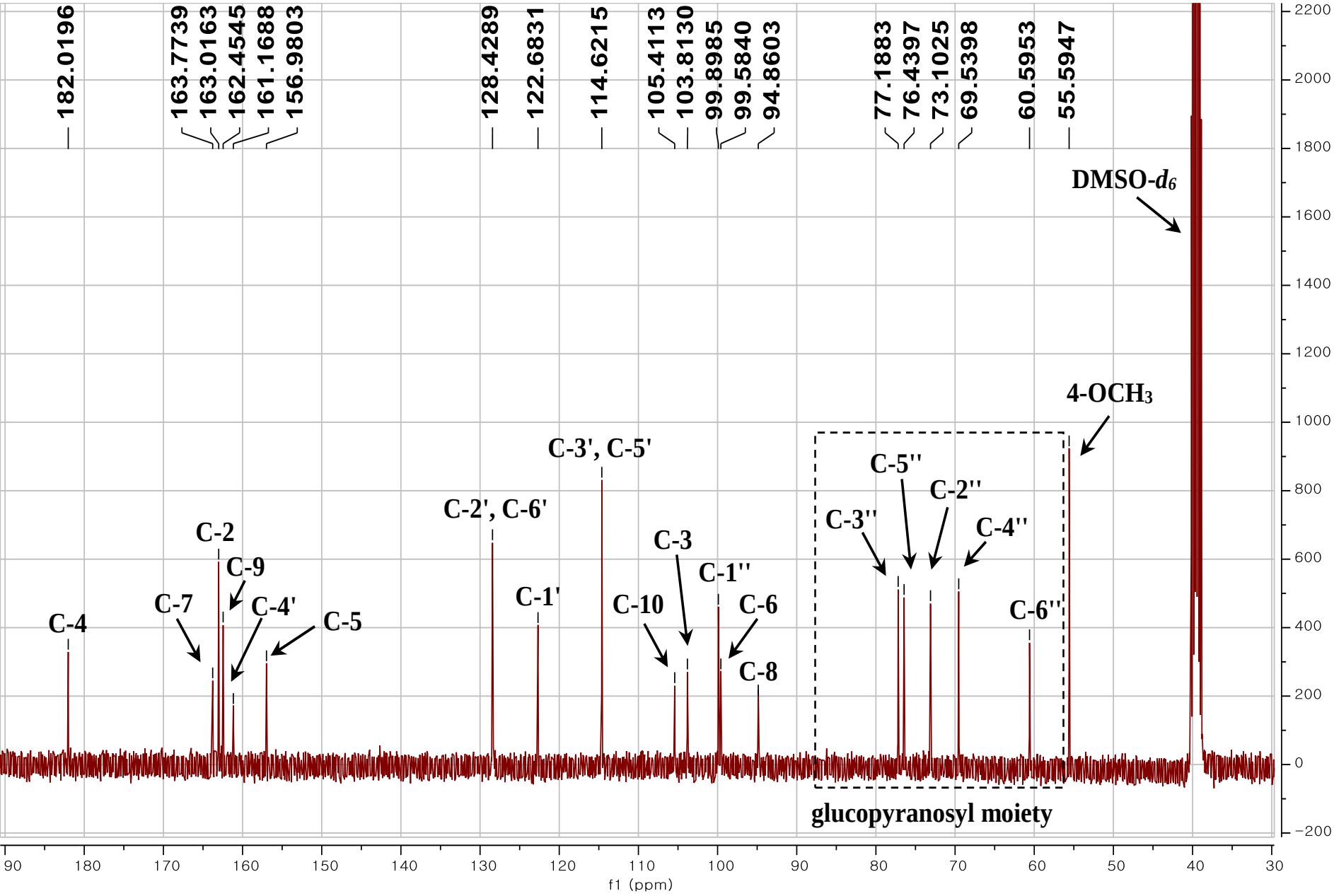

Figure S10. The ^{13}C NMR spectrum of compound **3** (100 MHz, DMSO-d_6).

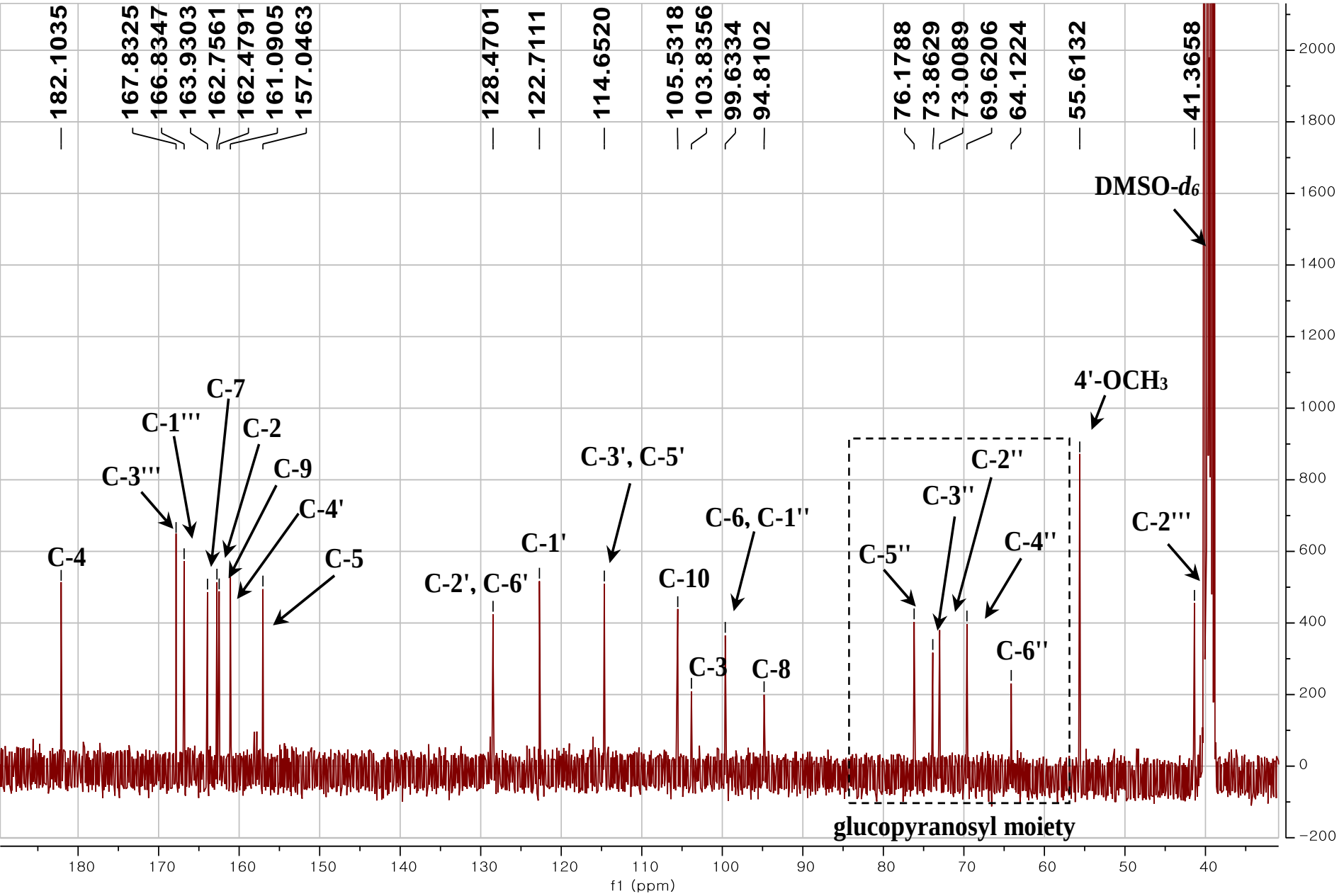

Figure S11. The ^{13}C NMR spectrum of compound **4** (100 MHz, DMSO-d_6).

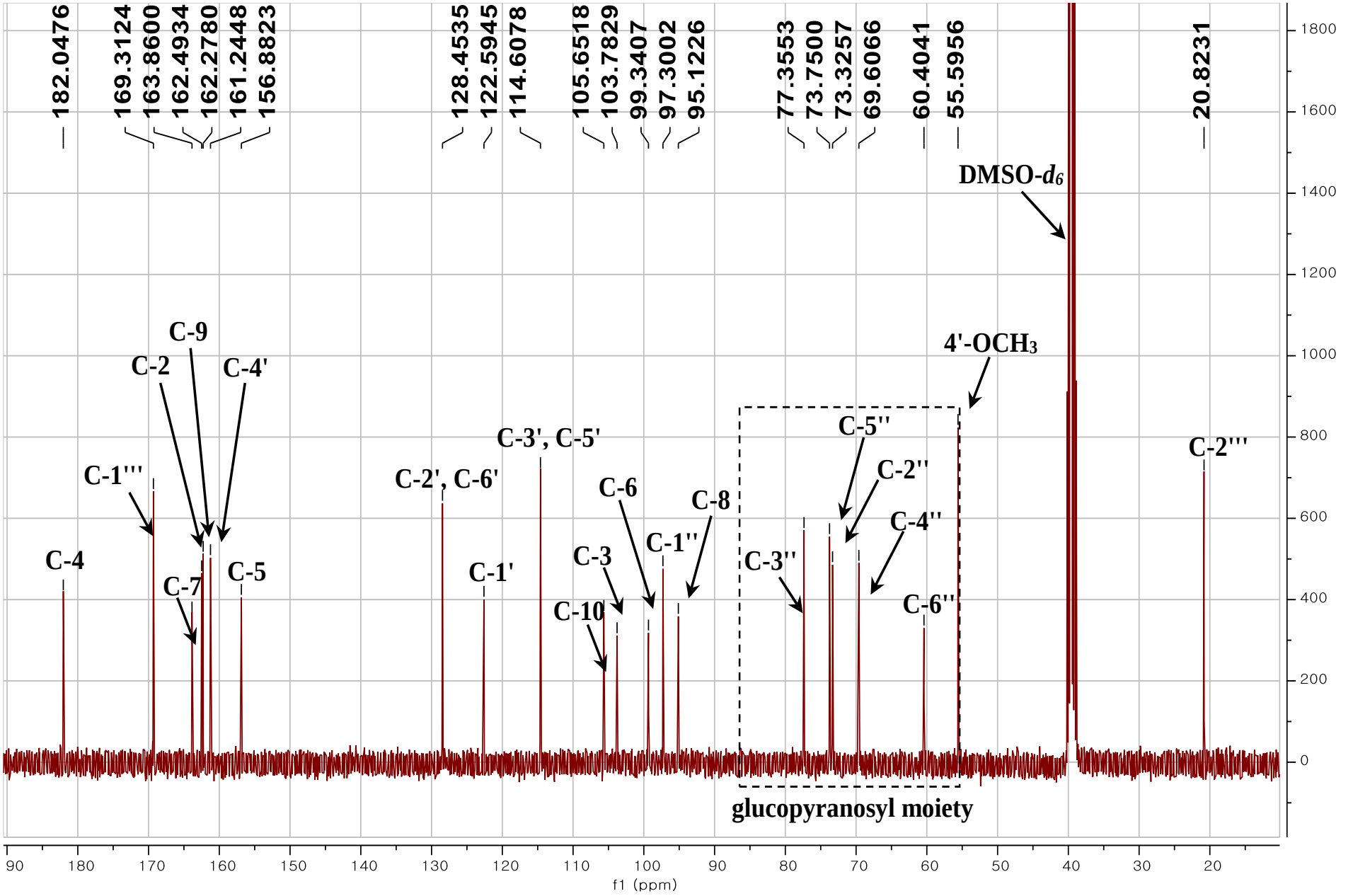

Figure S12. The ^{13}C NMR spectrum of compound **5** (100 MHz, DMSO-d_6).

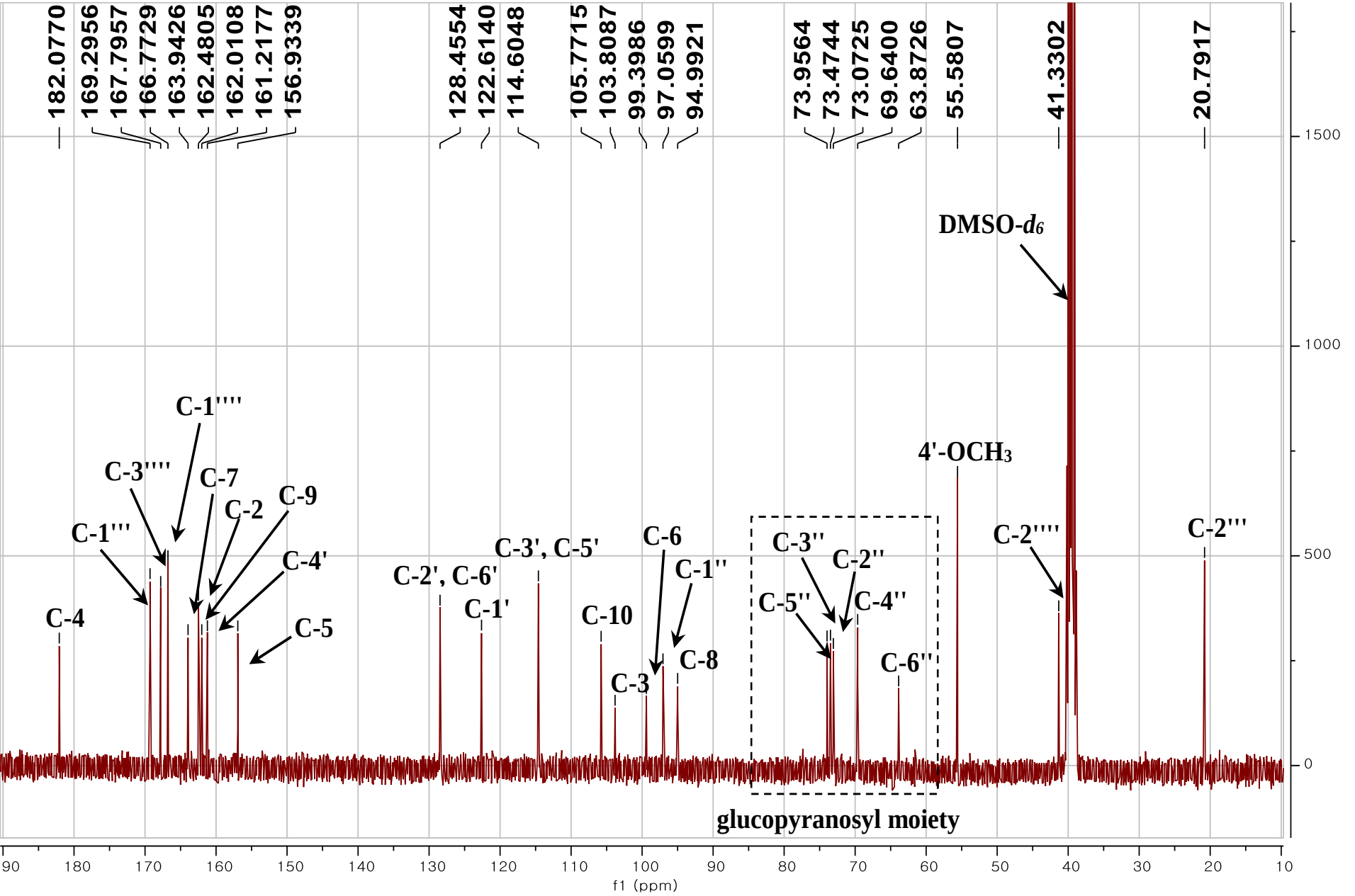

Figure S13. The ^{13}C NMR spectrum of compound **6** (100 MHz, DMSO-d_6).

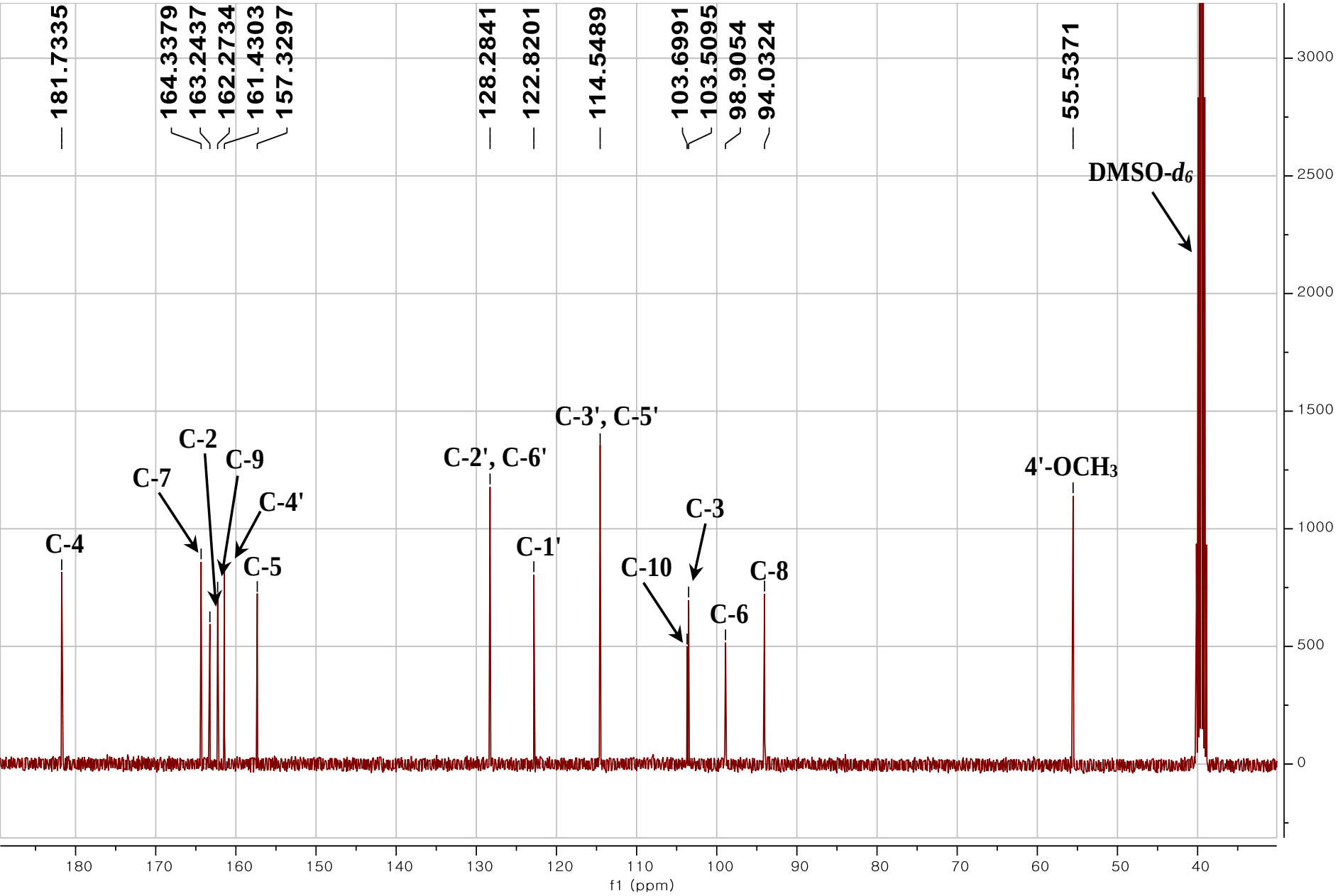

Figure S14. The ^{13}C NMR spectrum of compound **7** (100 MHz, DMSO-d_6).

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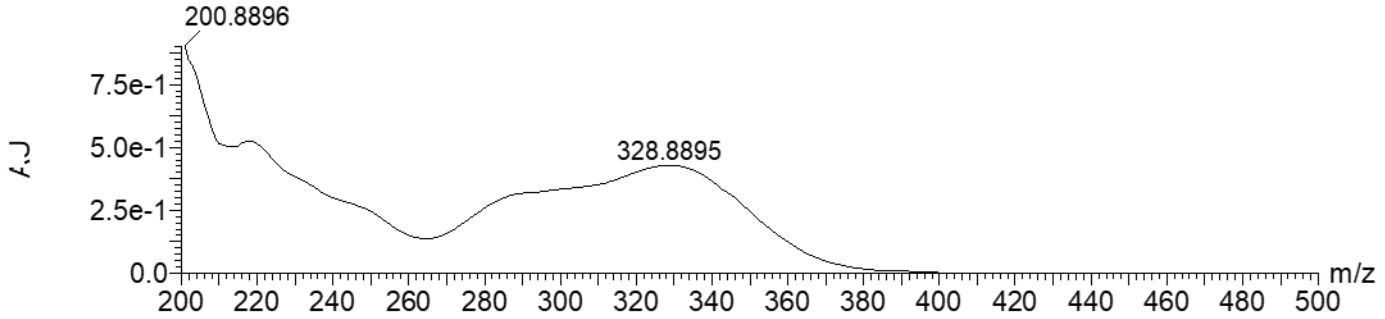
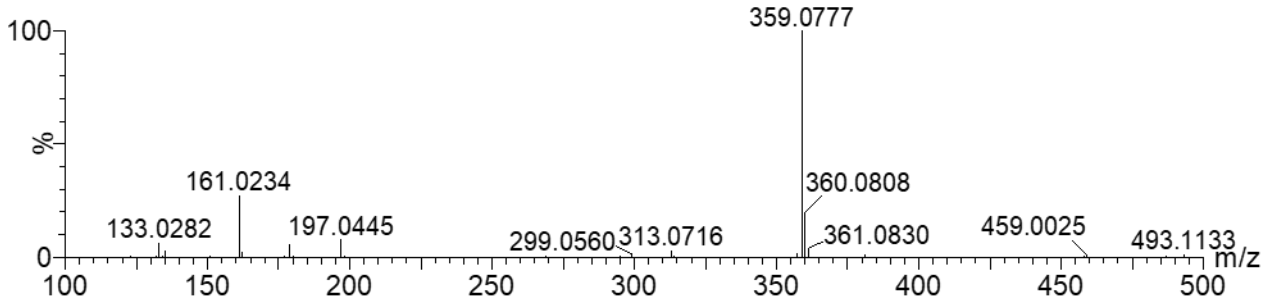
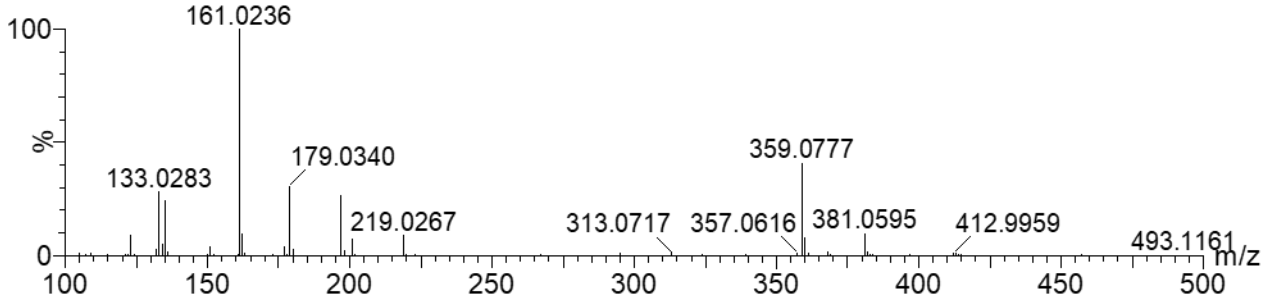
[UV spectrum]



[MS Chromatogram] negative



[MS/MS Chromatogram] negative



Elemental Composition: [HR-ESI/MS] negative

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

76 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

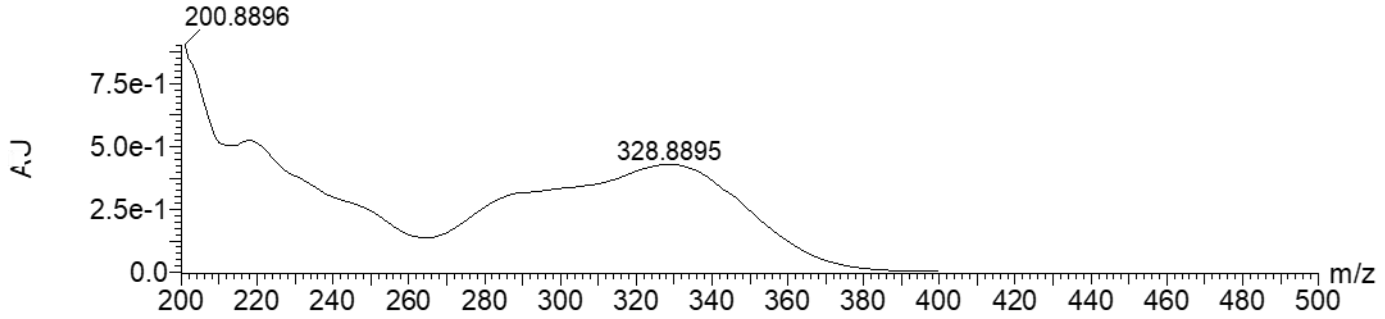
Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
359.0777	359.0767	1.0	2.8	11.5	C18 H15 O8	844.2	n/a	n/a	18	15	8

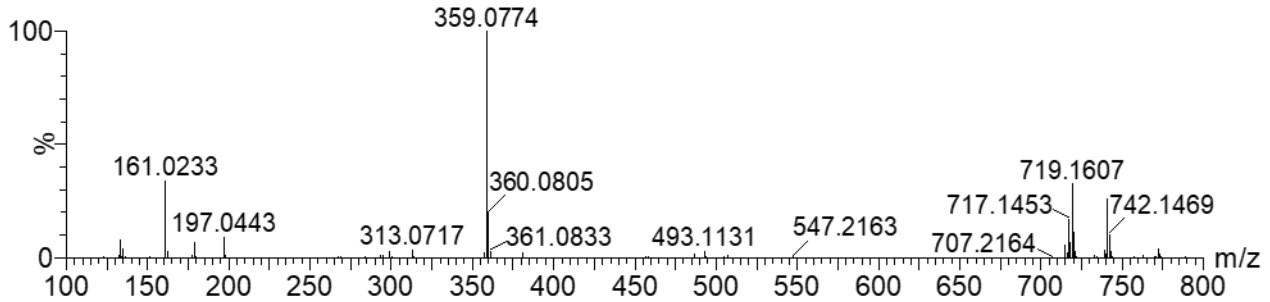
Figure S15. Identification of compound **1** by UPLC-QTof MS.

.

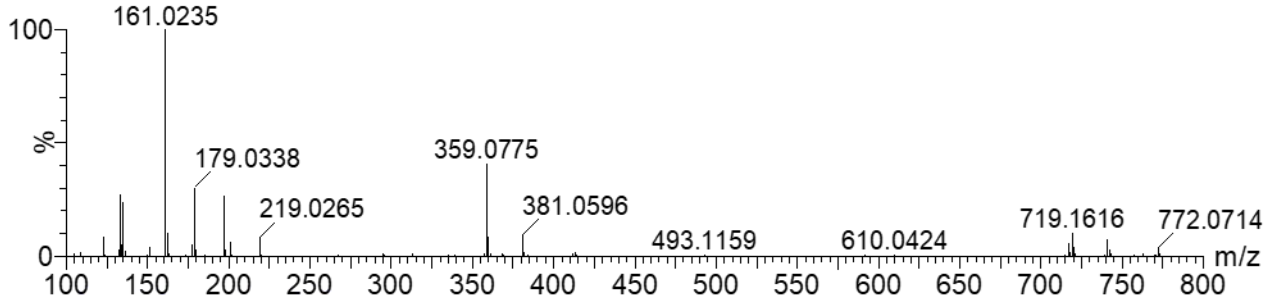
[UV spectrum]



[MS Chromatogram] negative



[MS/MS Chromatogram] negative



Elemental Composition: [HR-ESI/MS] negative

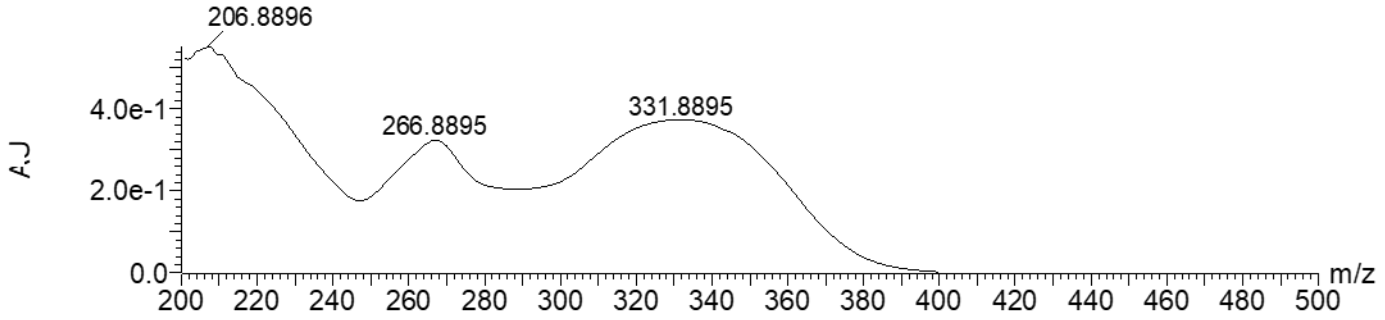
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
237 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
717.1453	717.1456	-0.3	-0.4	22.5	C36 H29 O16	324.0	0.350	70.47	36	29	16
	717.1421	3.2	4.5	0.5	C18 H37 O29	324.9	1.220	29.53	18	37	29

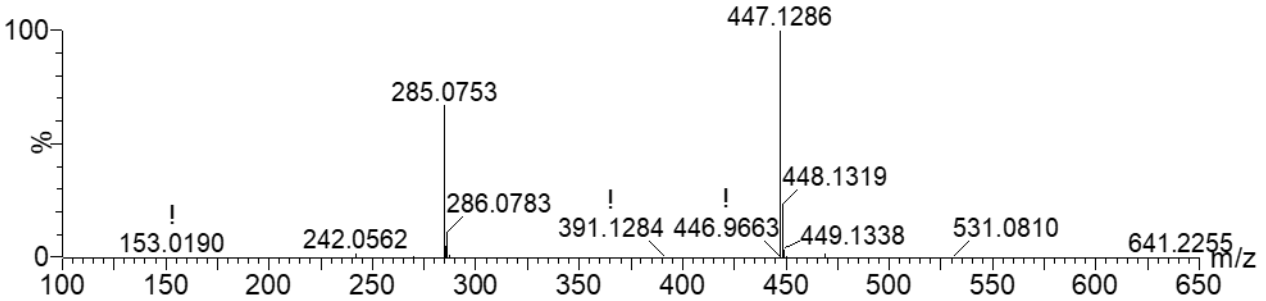
Figure S16. Identification of compound **2** by UPLC-QTof MS.

.

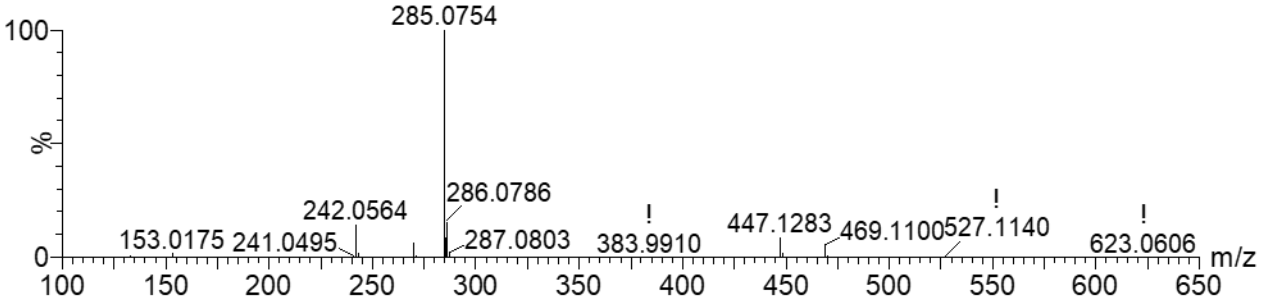
[UV spectrum]



[MS Chromatogram] positive



[MS/MS Chromatogram] positive



Elemental Composition: [HR-ESI/MS] positive

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

108 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

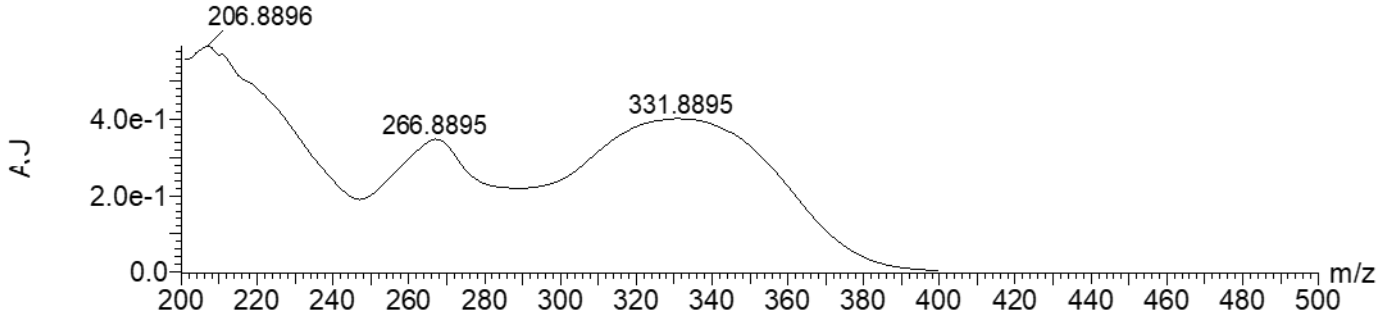
Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
447.1286	447.1291	-0.5	-1.1	11.5	C22 H23 O10	554.5	n/a	n/a	22	23	10

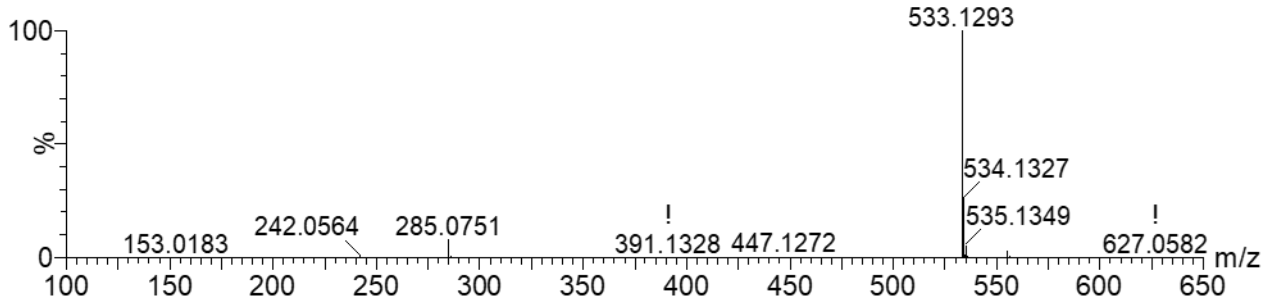
Figure S17. Identification of compound **3** by UPLC-QTof MS.

.

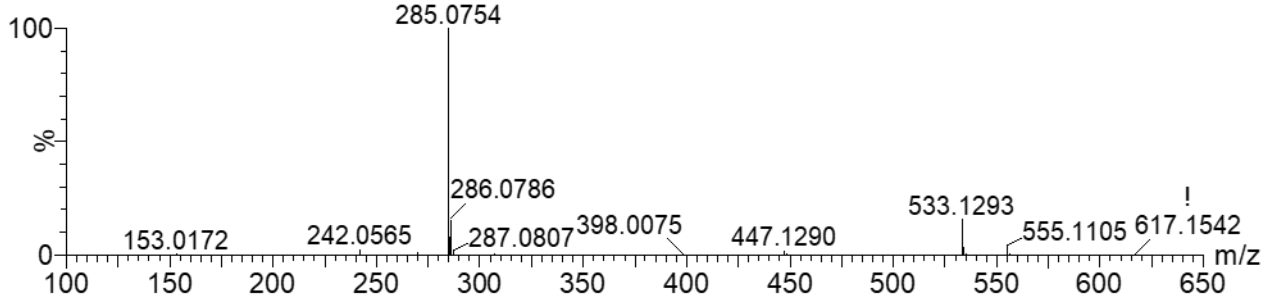
[UV spectrum]



[MS Chromatogram] positive



[MS/MS Chromatogram] positive



Elemental Composition: [HR-ESI/MS] positive

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

141 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

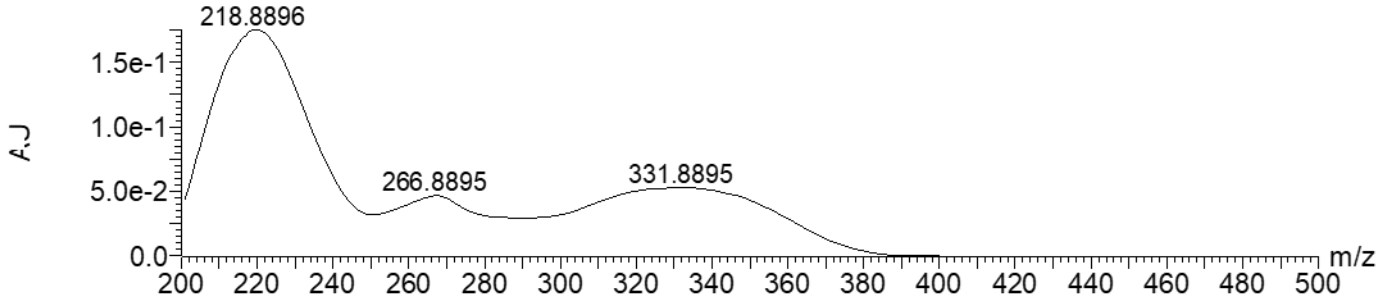
Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
533.1293	533.1295	-0.2	-0.4	13.5	C25 H25 O13	669.7	n/a	n/a	25	25	13

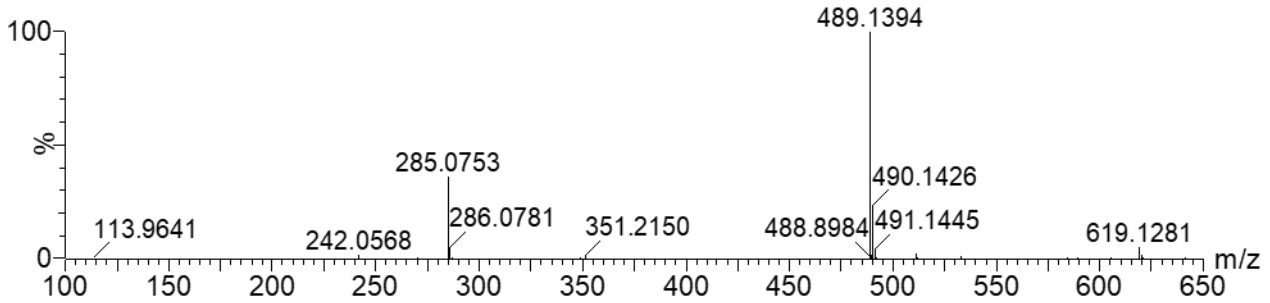
Figure S18. Identification of compound **4** by UPLC-QTof MS.

.

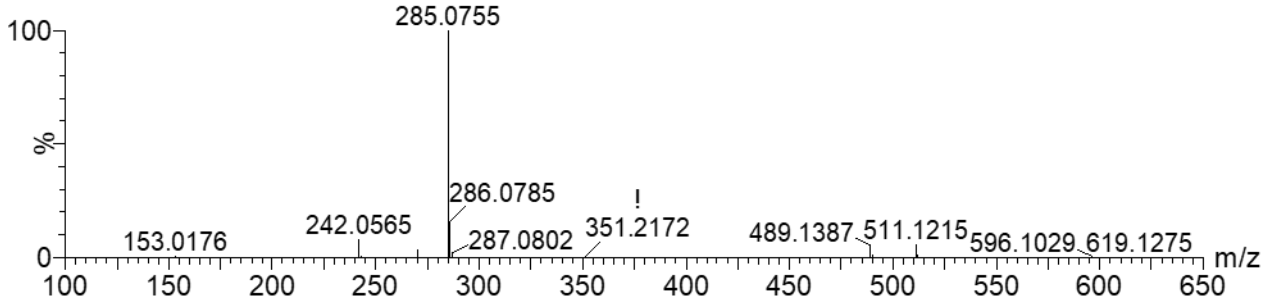
[UV spectrum]



[MS Chromatogram] positive



[MS/MS Chromatogram] positive



Elemental Composition: [HR-ESI/MS] positive

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

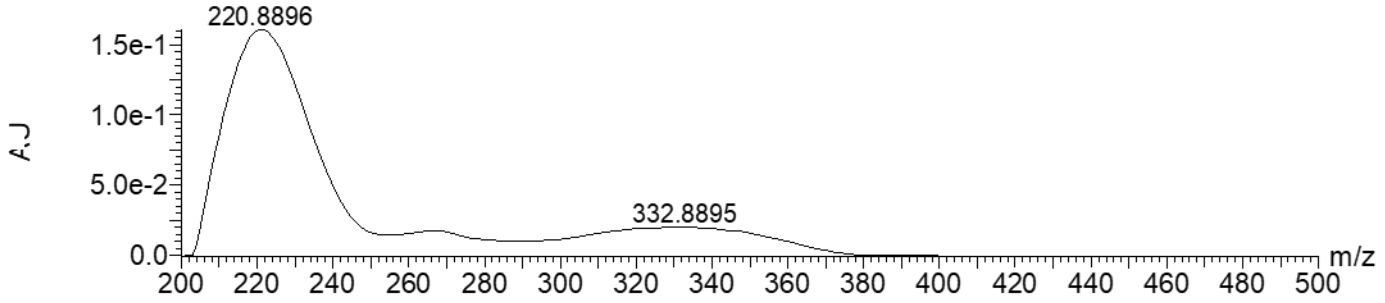
121 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

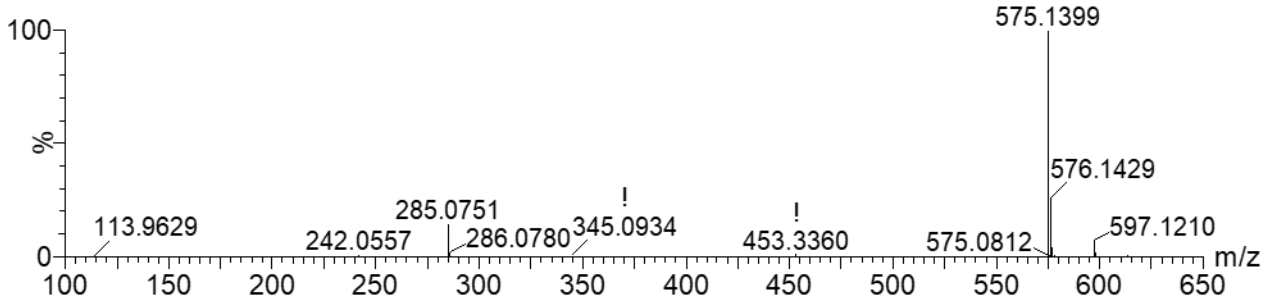
Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
489.1394	489.1397	-0.3	-0.6	12.5	C24 H25 O11	326.7	n/a	n/a	24	25	11

Figure S19. Identification of compound **5** by UPLC-QTof MS.

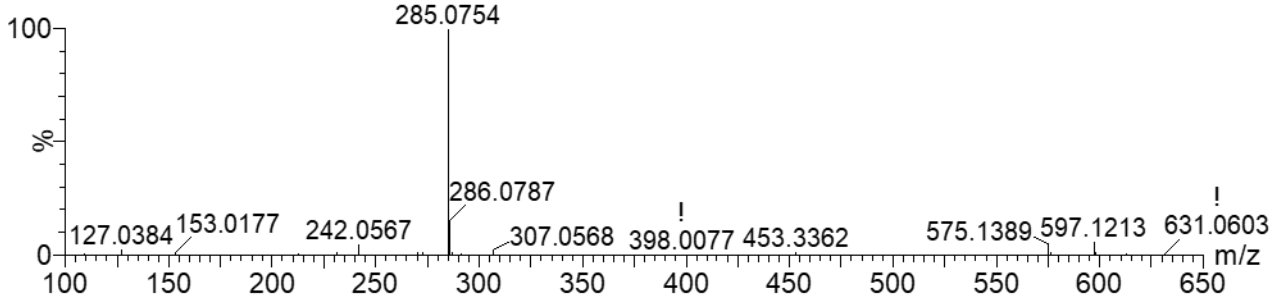
[UV spectrum]



[MS Chromatogram] positive



[MS/MS Chromatogram] positive



Elemental Composition: [HR-ESI/MS] positive

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

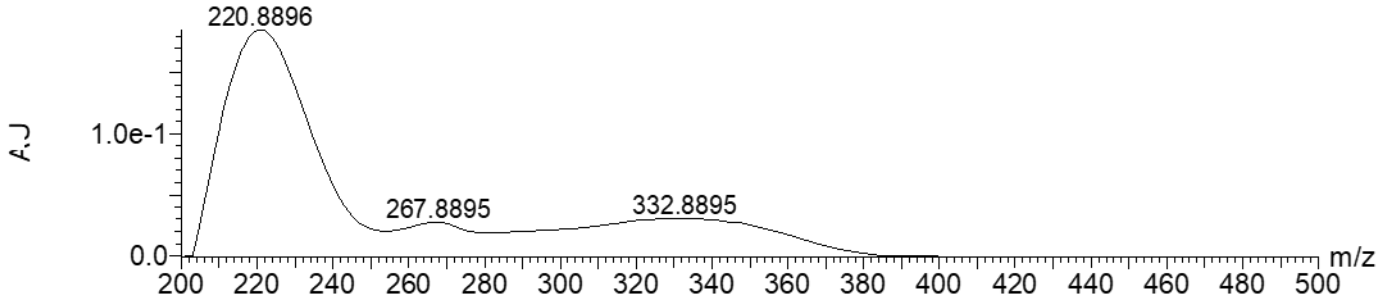
166 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

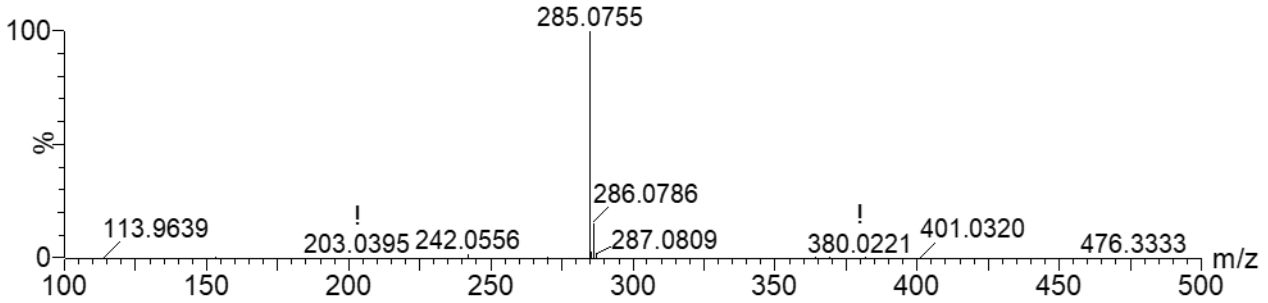
Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
575.1399	575.1401	-0.2	-0.3	14.5	C27 H27 O14	353.2	n/a	n/a	27	27	14

Figure S20. Identification of compound **6** by UPLC-QTof MS.

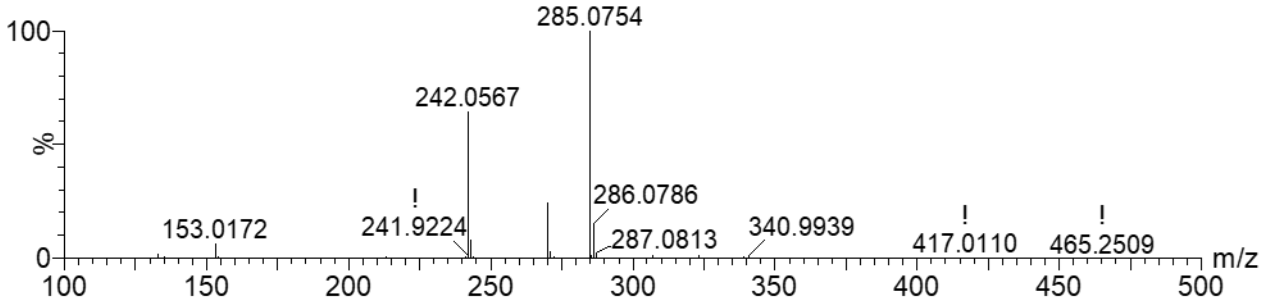
[UV spectrum]



[MS Chromatogram] positive



[MS/MS Chromatogram] positive



Elemental Composition: [HR-ESI/MS] positive

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

48 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	O
285.0755	285.0763	-0.8	-2.8	10.5	C16 H13 O5	473.3	n/a	n/a	16	13	5

Figure S21. Identification of compound **7** by UPLC-QTof MS.

Table S1. ^{1}H- and ^{13}C-NMR assign of compounds (**1**−**7**).

Comp.	assign
1	^{1}H NMR (400 MHz, DMSO-d_6) δ_H 2.88 (1H, dd, J = 8.2 Hz, 8.2 Hz, H-7'b), 2.99 (1H, dd, J = 14.2 Hz, 4.2 Hz, H-7'a), 5.03 (1H, dd, J = 8.2 Hz, 4.2 Hz, H-8'), 6.24 (1H, d, J = 16.0 Hz, H-8), 6.53 (1H, dd, J = 8.0 Hz, 1.6 Hz, H-6'), 6.64 (1H, d, J = 8.0 Hz, H-5'), 6.68 (1H, d, J = 1.6 Hz, H-2'), 6.77 (1H, d, J = 8.0 Hz, H-5), 7.01 (1H, dd, J = 8.0 Hz, 1.2 Hz, H-6), 7.06 (1H, d, J = 1.2 Hz, H-2), 7.47 (1H, d, J = 15.8 Hz, H-7). ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C 36.12 (C-7'), 72.82 (C-8'), 113.25 (C-2), 114.89 (C-8), 115.40 (C-5'), 115.77 (C-5), 116.70 (C-2'), 120.06 (C-6'), 121.61 (C-6), 125.36 (C-1), 127.28 (C-1'), 144.03 (C-3'), 144.94 (C-4'), 145.61 (C-3), 145.96 (C-7), 148.64 (C-4), 165.93 (C-9), 170.84 (C-9').
2	^{1}H NMR (500 MHz, CD_3OD) δ_H 3.10-2.95 (4H, m, H-7"α, 7"β, 7'"α, 7'"β), 4.40 (1H, d, J = 4.4 Hz, H-3), 5.11-5.20 (2H, m, H-8", 8'"), 5.83 (1H, d, J = 4.4 Hz, H-2), 6.29 (1H, d, J = 16.0 Hz, H-11'"), 6.46 (1H, dd, J = 2.0, 8.0 Hz, H-6"), 6.57-6.65 (8H, m, H-6'", 6', 5', 5'", 2", 2', 2'", 5"), 6.76 (1H, d, J = 8.4 Hz, H-6), 7.17 (1H, d, J = 16.0 Hz, H-5), 7.68 (1H, d, J = 16.0 Hz, H-12'"). ^{13}C NMR (125 MHz, CD_3OD) δ_C 172.4 (C-9"), 170.1 (C-10"), 168.1 (C-9'"), 166.9 (C-10'"), 148.9 (C-8), 146.5 (C-7), 146.4 (C-3'", 4'"), 145.9 (C-3', 4'), 145.1 (C-3",4"), 133.7 (C-1'), 129.2 (C-1"), 128.7 (C-1'"), 126.2 (C-9), 124.7 (C-4), 144.1 (C-12'"), 121.9 (C-5, 6", 6'"), 118.3 (C-6, 6'), 117.5 (C-2", 2'"), 116.3 (C-5', 5", 5'", 11'"), 113.2 (C-2'), 88.8 (C-2), 74.4 (C-8"), 74.4 (C-8"), 57.2 (C-3), 37.8 (C-7'"), 37.2 (C-7").
3	^{1}H NMR (400 MHz, DMSO-d_6) δ_H 3.16-3.72 (6H, m, Glc-H), 3.86 (3H, s, 4'-OCH_3), 5.07 (1H, d, J = 7.6 Hz, H-1"), 6.45 (1H, d, J = 2.0 Hz, H-6), 6.85 (1H, d, J = 2.0 Hz, H-8), 6.95(1H, s, H-3), 7.13 (2H, d, J = 8.0 Hz, H-3', H-5'), 8.06 (2H, d, J = 8.0 Hz, H-2', H-6'), 12.91 (1H, br s, 5-OH). ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C 55.59 (4'-OCH_3), 60.60 (C-6"), 69.54 (C-4"), 73.10 (C-2"), 76.44 (C-5"), 77.19 (C-3"), 94.86 (C-8), 99.58 (C-6), 99.90 (C-1"), 103.81(C-3), 105.41 (C-10), 114.62 (C-3', C-5'), 122.68 (C-1'), 128.43 (C-2', C-6'), 156.98 (C-5), 161.17 (C-4'), 162.45 (C-9), 163.02 (C-2), 163.77 (C-7), 182.02 (C-4).
4	^{1}H NMR (400 MHz, DMSO-d_6) δ_H 3.15-3.80 (4H, m, Glc-H), 3.39 (2H, s, H-2'"), 3.85 (3H, s, 4'-OCH_3), 4.12 (1H, dd, J = 11.8 Hz, 6.8 Hz, H-6"a), 4.41 (1H, dd, J = 11.8 Hz, 1.6 Hz, H-6"b), 5.11 (1H, d, J = 7.2 Hz, H-1"), 6.44 (1H, d, J = 2.0 Hz, H-6), 6.84 (1H, d, J = 2.0 Hz, H-8), 6.95 (1H, s, H-3), 7.12 (2H, d, J = 8.8 Hz, H-3', H-5'), 8.07 (2H, d, J = 8.8 Hz, H-2', H-6'), 12.92 (1H, br s, 5-OH). ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C 41.37 (C-2'"), 55.61 (4'-OCH_3), 64.11 (C-6"), 69.61 (C-4"), 73.00 (C-2"), 73.85 (C-3"), 76.17 (C-5"), 94.80 (C-8), 99.62 (C-6, C-1"), 103.40 (C-3), 105.53 (C-10), 112.70 (C-1'), 114.64 (C-3', C-5'), 128.46 (C-2', C-6'), 157.04 (C-5), 161.08 (C-4'), 162.47 (C-9), 162.75 (C-2), 163.92 (C-7), 166.82 (C-1'"), 167.82 (C-3'"), 182.09 (C-4).
5	^{1}H NMR (400 MHz, DMSO-d_6) δ_H 2.04 (3H, s, H-2'"), 3.15-3.80 (5H, m, Glc-H), 3.86 (3H, s, 4'-OCH_3), 4.81 (1H, dd, J = 9.4 Hz, 8.2 Hz, H-2"), 5.39 (1H, d, J = 7.6 Hz, H-1"), 6.42 (1H, d, J = 2.0 Hz, H-6), 6.81 (1H, d, J = 2.0 Hz, H-8), 6.95 (1H, s, H-3), 7.12 (2H, d, J = 8.8 Hz, H-3', H-5'), 8.06 (2H, d, J = 8.8 Hz, H-2', H-6'), 12.93 (1H, br s, 5-OH). ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C 20.82 (C-2'"), 55.60 (4'-OCH_3), 60.40 (C-6"), 69.60 (C-4"), 73.33 (C-2"), 73.75 (C-5"), 77.36 (C-3"), 95.12 (C-8), 97.30 (C-1"), 99.34 (C-6), 103.78 (C-3), 105.65 (C-10), 114.61 (C-3', C-5'), 122.59 (C-1'), 128.45 (C-2', C-6'), 156.88 (C-5), 161.24 (C-4'), 162.28 (C-9), 162.49 (C-2), 163.86 (C-7), 169.31 (C-1'"), 182.04 (C-4).
6	^{1}H NMR (400 MHz, DMSO-d_6) δ_H 2.05 (3H, s, H-5'"), 3.30-3.95 (3H, m, Glc-H), 3.41 (2H, s, H-2'""), 3.86 (3H, s, 4'-OCH_3), 4.17 (1H, dd, J = 11.0 Hz, 6.8 Hz, H-6"a), 4.44 (1H, d, J = 11.0 Hz, H-6"b), 4.83 (1H, m, H-2"), 5.42 (1H, d, J = 8.0 Hz, H-1"), 6.40 (1H, d, J = 2.0 Hz, H-6), 6.79 (1H, d, J = 2.0 Hz, H-8), 6.96 (1H, s, H-3), 7.12 (2H, d, J = 8.8 Hz, H-3', H-5'), 8.06 (2H, d, J = 8.8 Hz, H-2', H-6'), 12.94 (1H, br s, 5-OH). ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C 20.79 (C-2'"), 41.33 (C-2'""), 55.58 (4'-OCH_3), 63.87 (C-6"), 69.64 (C-4"), 73.07 (C-2"), 73.47 (C-3"), 73.95 (C-5"), 94.99 (C-8), 97.06 (C-1"), 99.40 (C-6), 103.81 (C-3), 105.77 (C-10), 114.60 (C-3', C-5'), 122.61 (C-1'), 128.46 (C-2', C-6'), 156.93 (C-5), 161.22 (C-4'), 162.01 (C-9), 162.48 (C-2), 163.94 (C-7), 166.77 (C-1'""), 167.79 (C-3'""), 169.30 (C-1'"), 182.09 (C-4).
7	^{1}H NMR (400 MHz, DMSO-d_6,) δ_H 3.85 (3H, s, 4'-OCH_3), 6.19 (1H, d, J = 1.8 Hz, H-6), 6.49 (1H, d, J = 1.8 Hz, H-8), 6.85 (1H, s, H-3), 7.10 (2H, d, J = 8.8 Hz, H-3', H-5'), 8.02 (2H, d, J = 8.8 Hz, H-2', H-6'), 12.92 (1H, br s, 5-OH). ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C 55.54 (4'-OCH_3), 94.03 (C-8), 98.91 (C-6), 103.51 (C-3), 103.70 (C-10), 114.55 (C-3', C-5'), 122.82 (C-1'), 128.28 (C-2', C-6'), 157.33 (C-5), 161.43 (C-4'), 162.27 (C-9), 163.24 (C-2), 164.34 (C-7), 181.73 (C-4).