Supporting Information

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Three New Phenolic Sulfates from *Acrostichum aureum* Collected from Coastal Area of Thai Binh Province, Vietnam and Their Cytotoxic Activity

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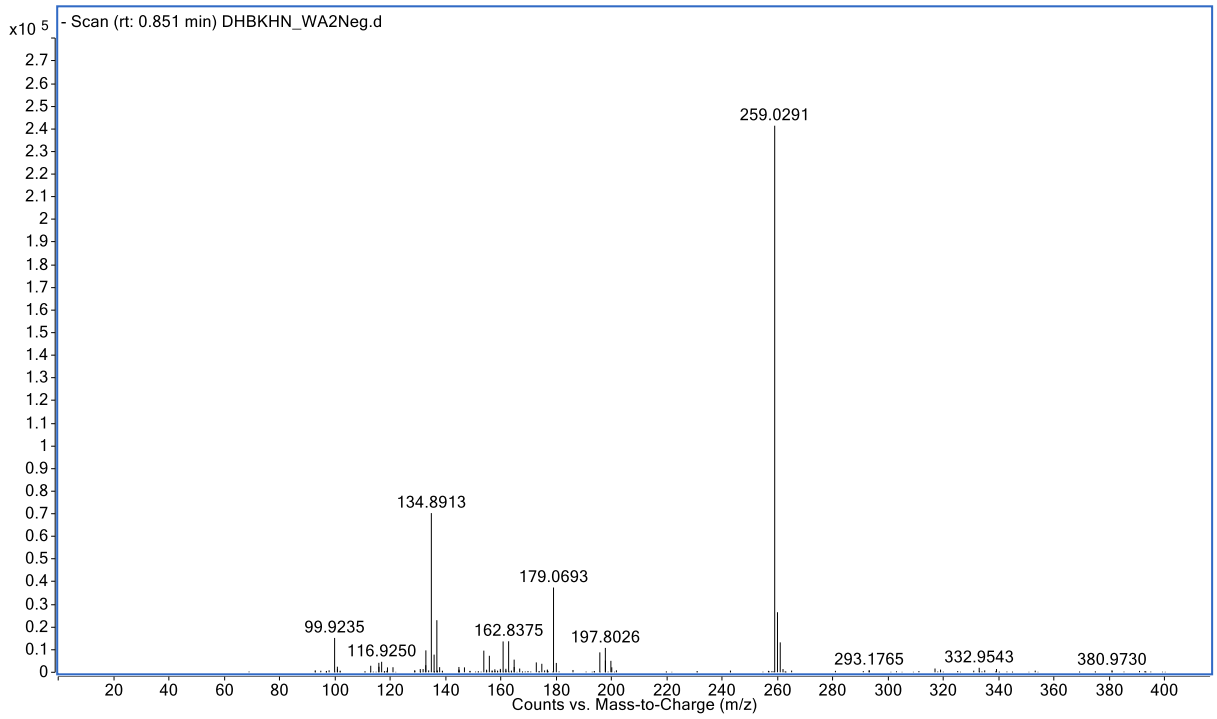


Figure S1: HR-ESI Mass Spectrum of **1** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2-butanone)

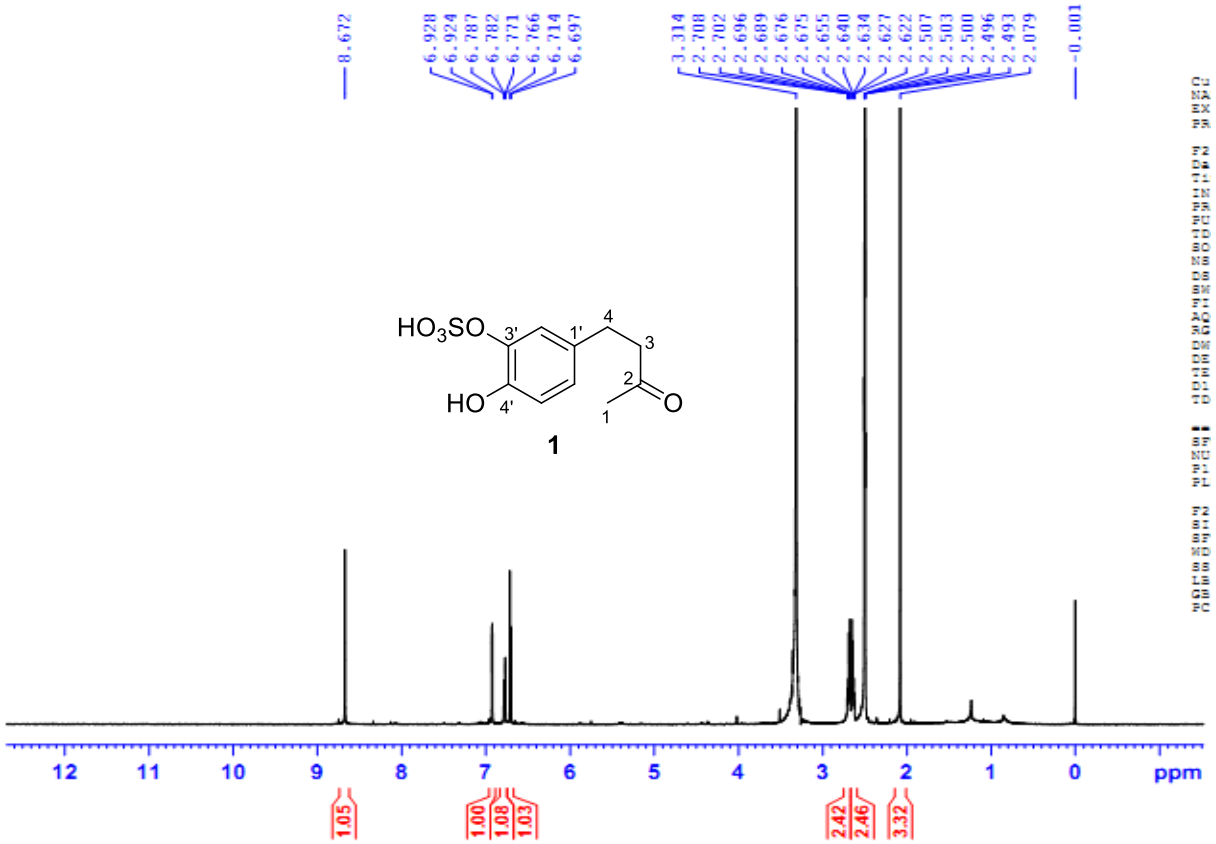


Figure S2: ^{1}H-NMR (500 MHz, DMSO) Spectrum of **1**

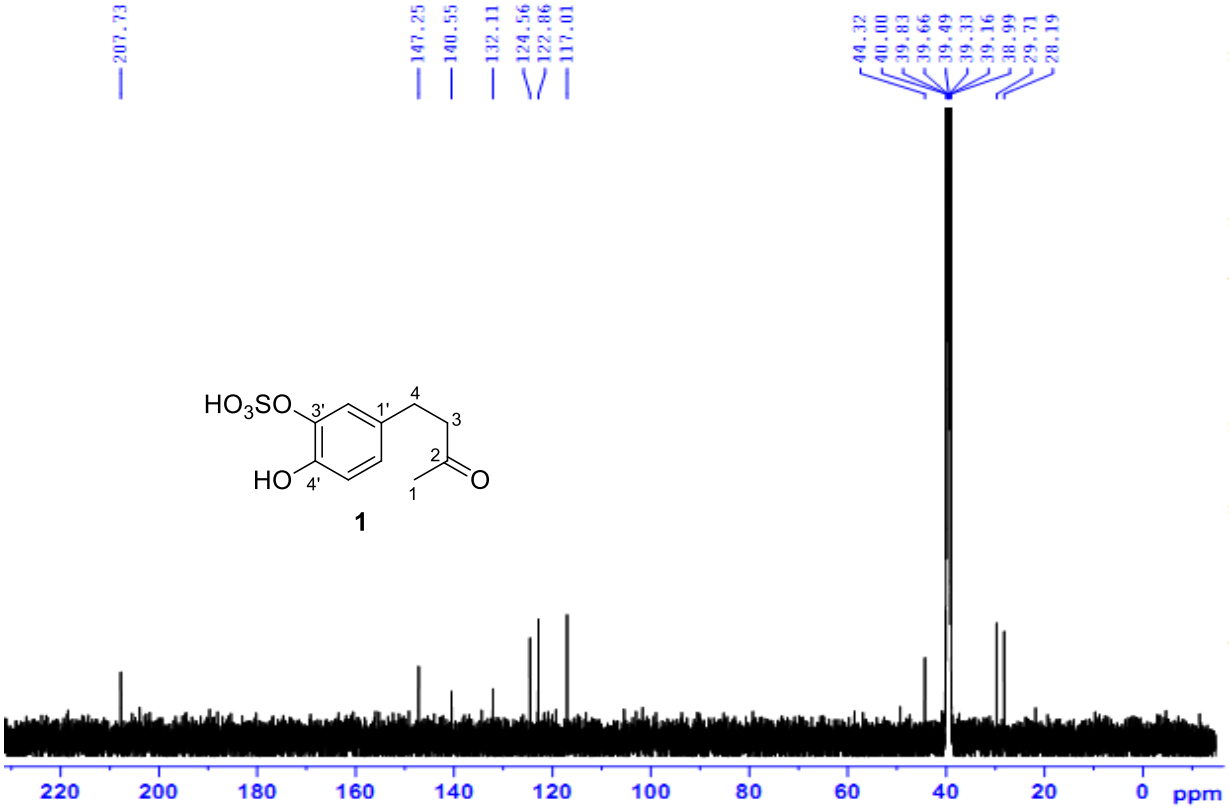


Figure S3: ^{13}C-NMR (125 MHz, DMSO) Spectrum of **1**

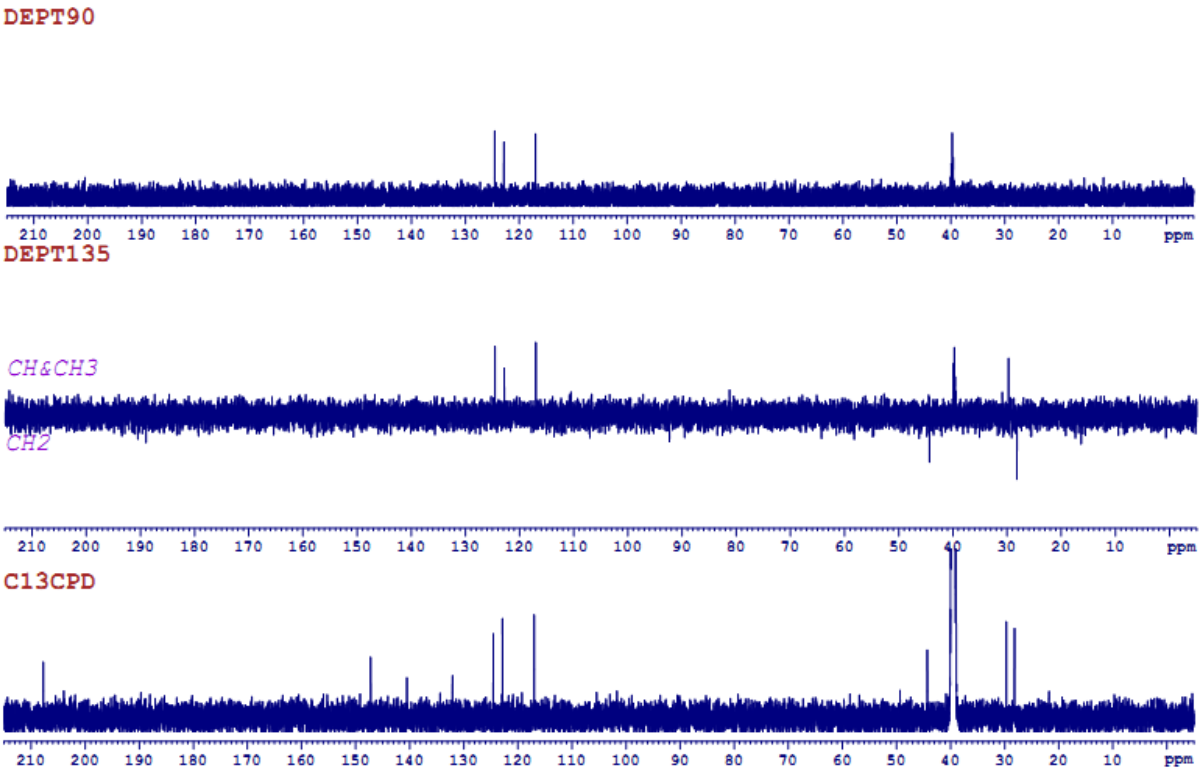


Figure S4: DEPT90 and 135 (125 MHz, DMSO) Spectrum of **1**

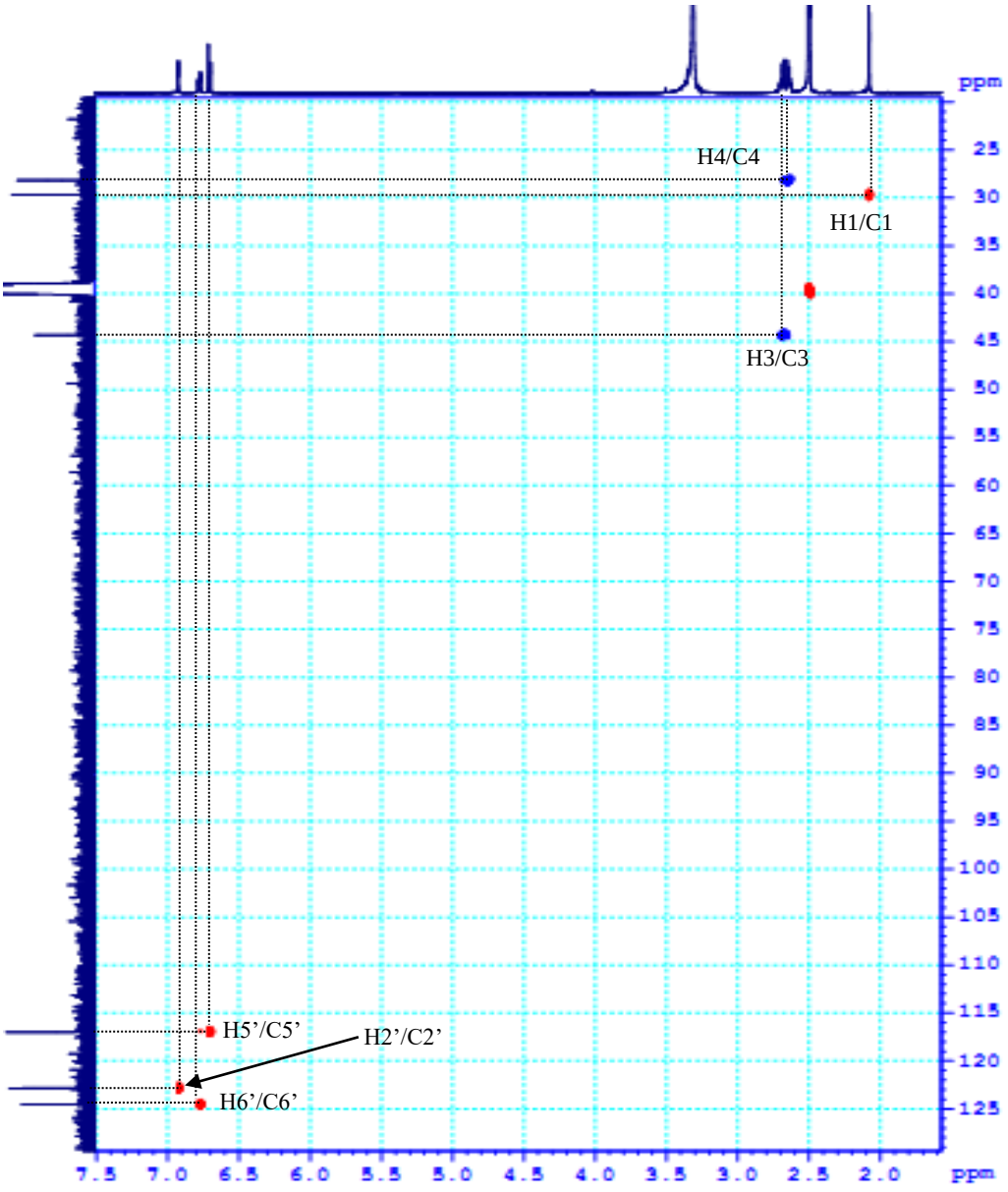


Figure S5: HSQC Spectrum of **1** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2-butanone)

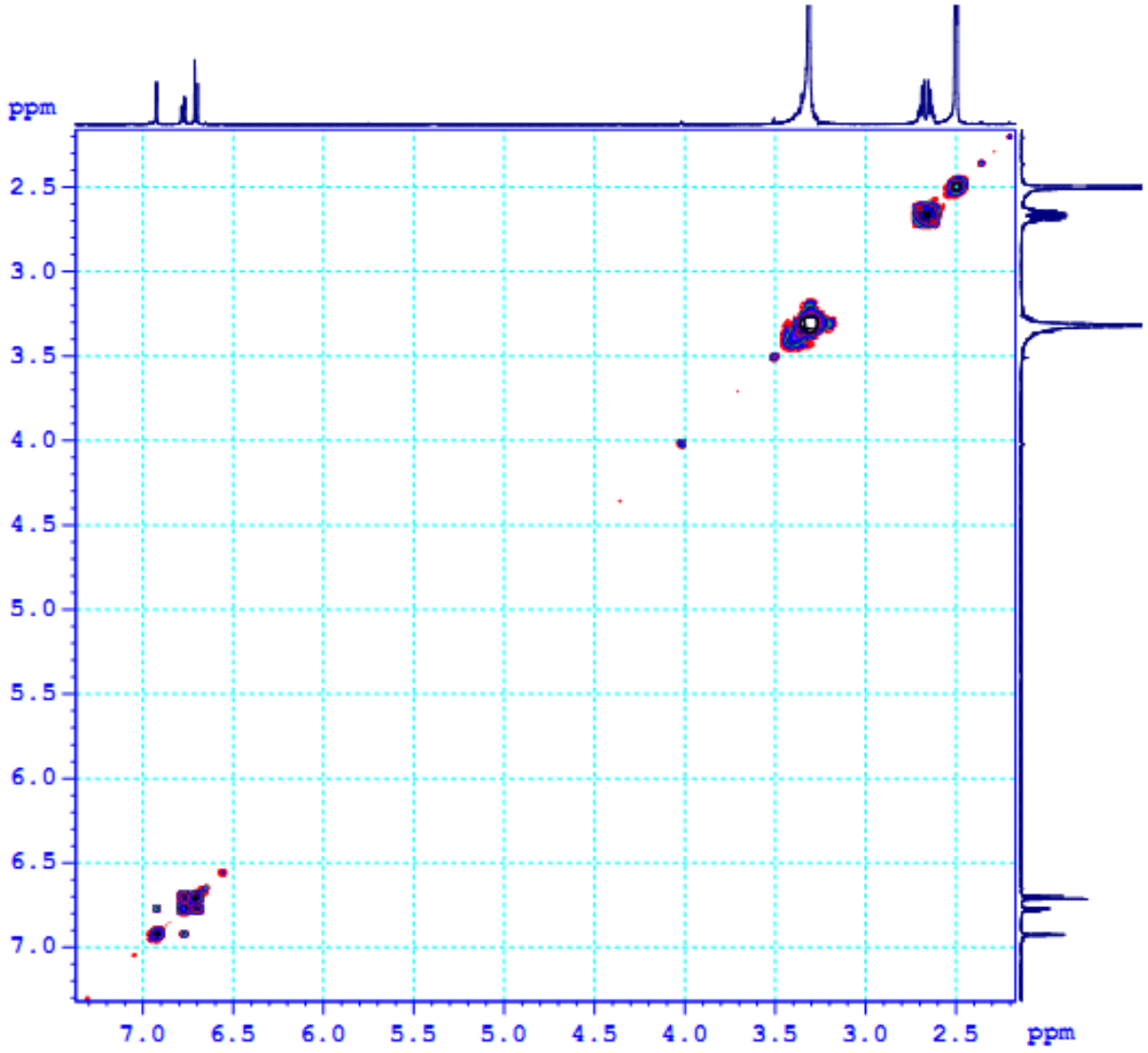


Figure S6: ^{1}H-^{1}H COSY Spectrum of **1** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2-butanone)

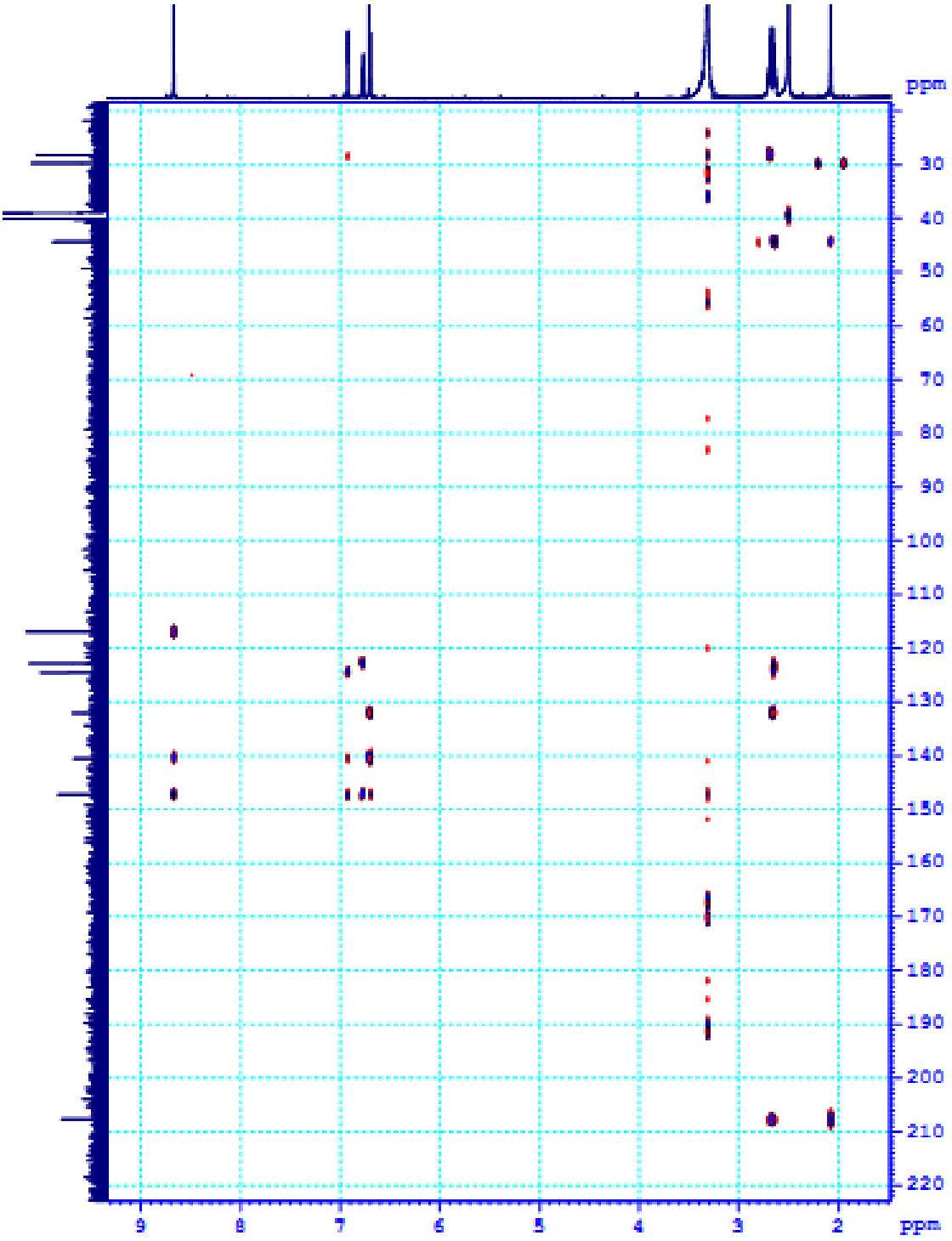


Figure S7: HMBC Spectrum of HMBC of **1** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2-butanone)

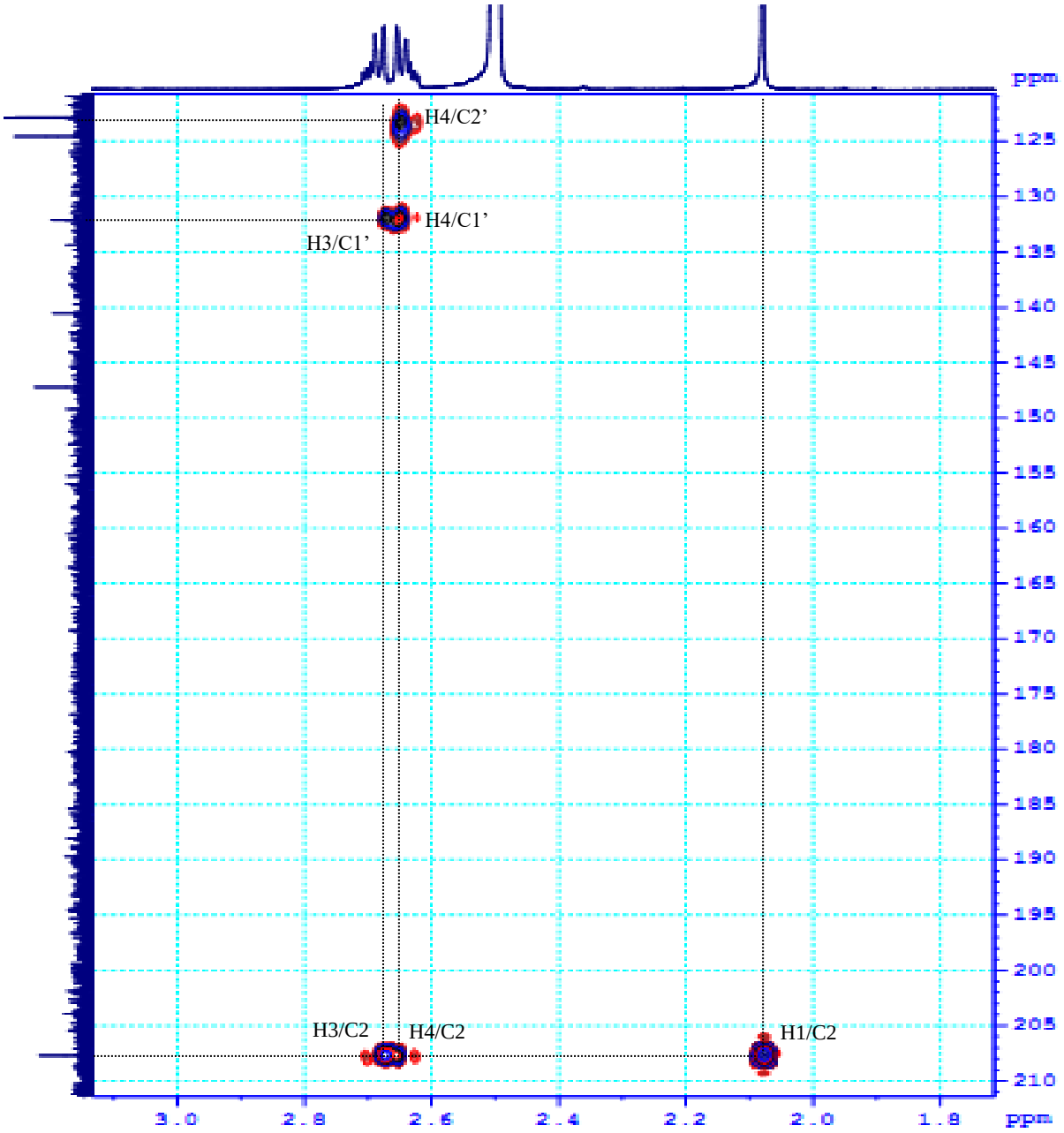


Figure S8: HMBC Spectrum of **1 (**From δ_C 120 ppm to δ_C 210 ppm)

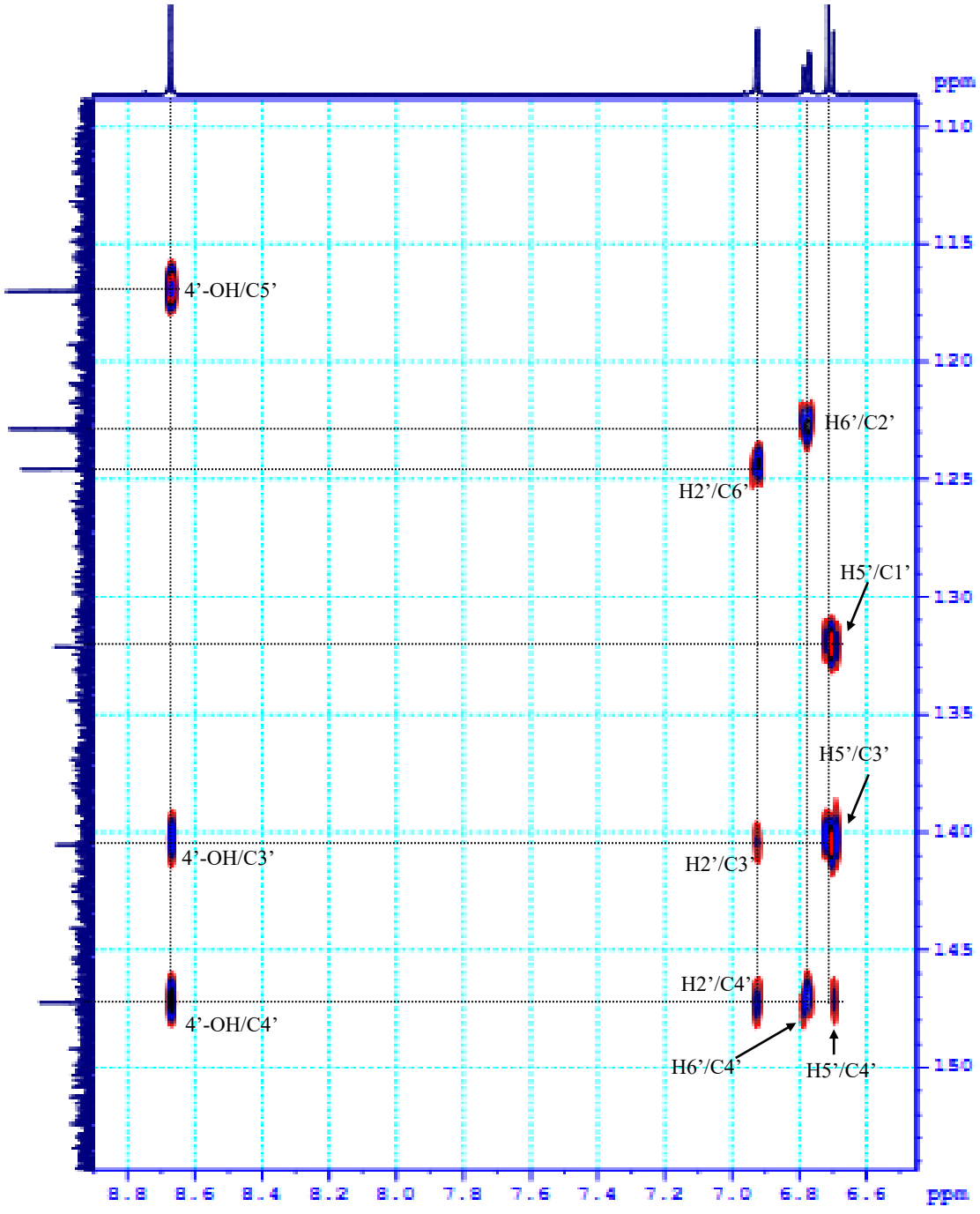


Figure S9: HMBC Spectrum of **1** (From δ_C 110 ppm to δ_C 150 ppm)

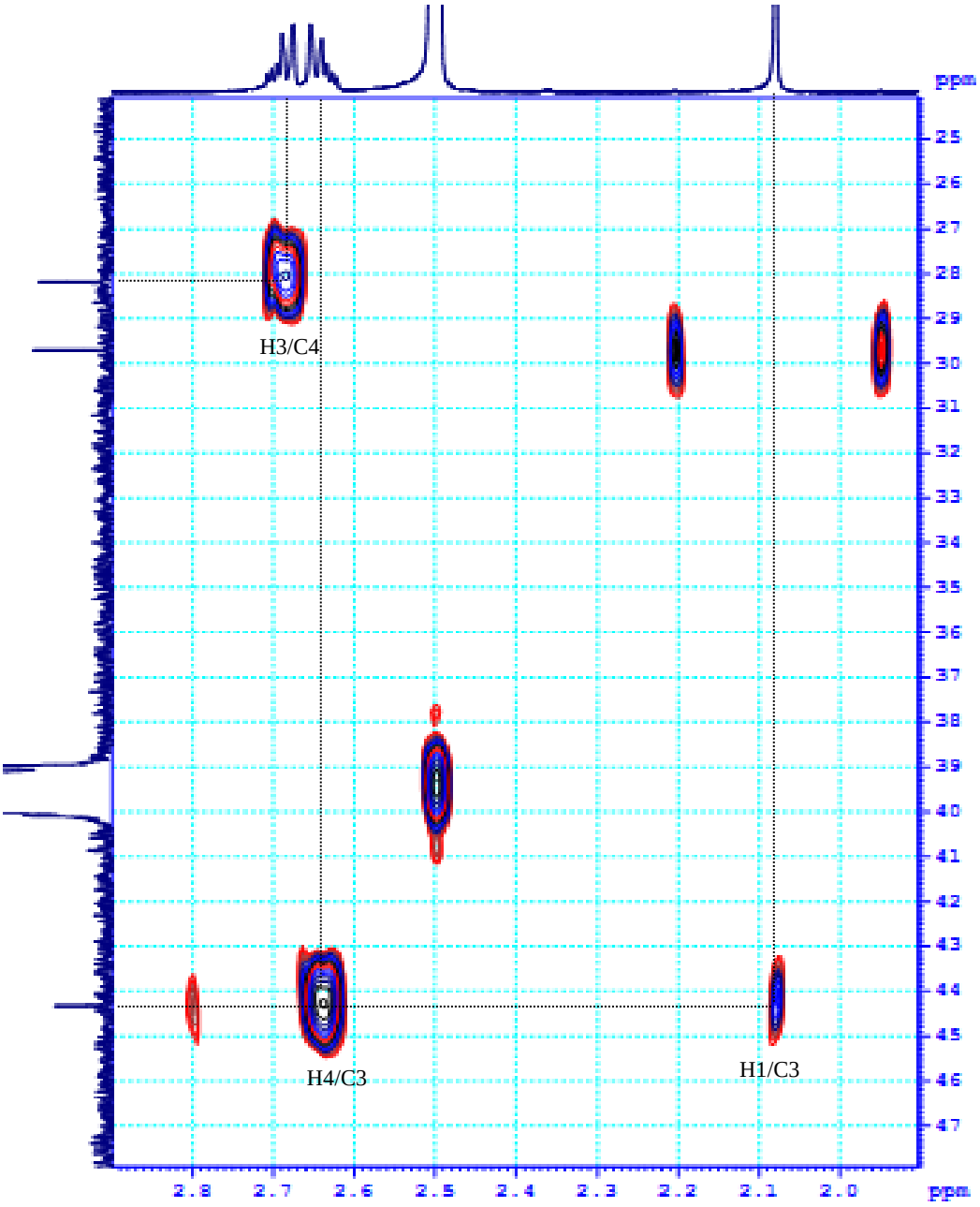


Figure S10: HMBC Spectrum of **1 (**From δ_C 25 ppm to δ_C 45 ppm)

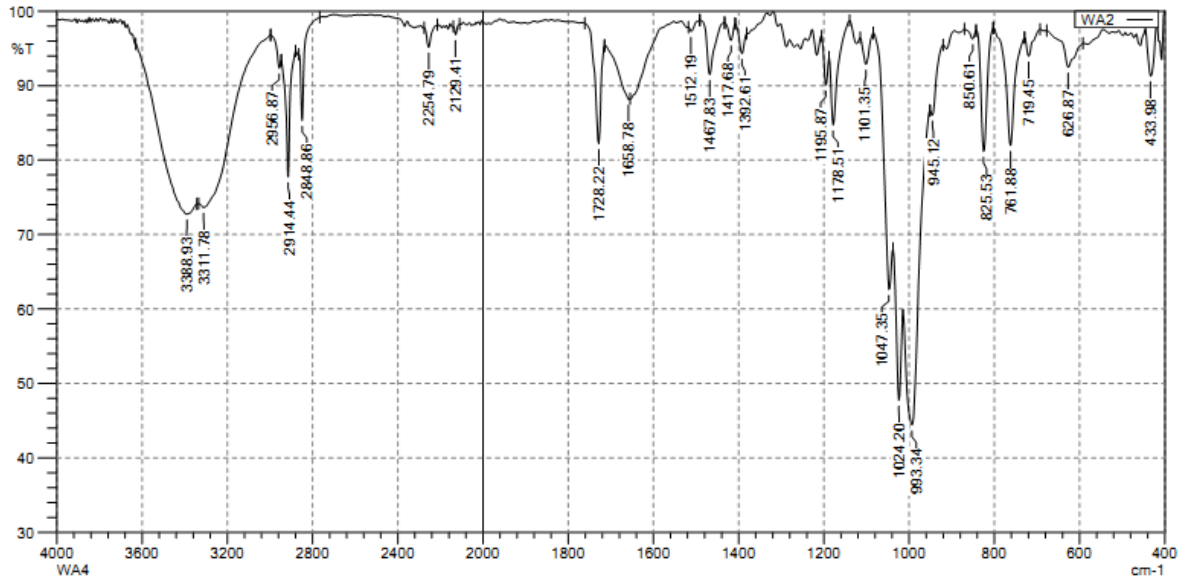


Figure S11: FT-IR Spectrum of **1** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2-butanone)

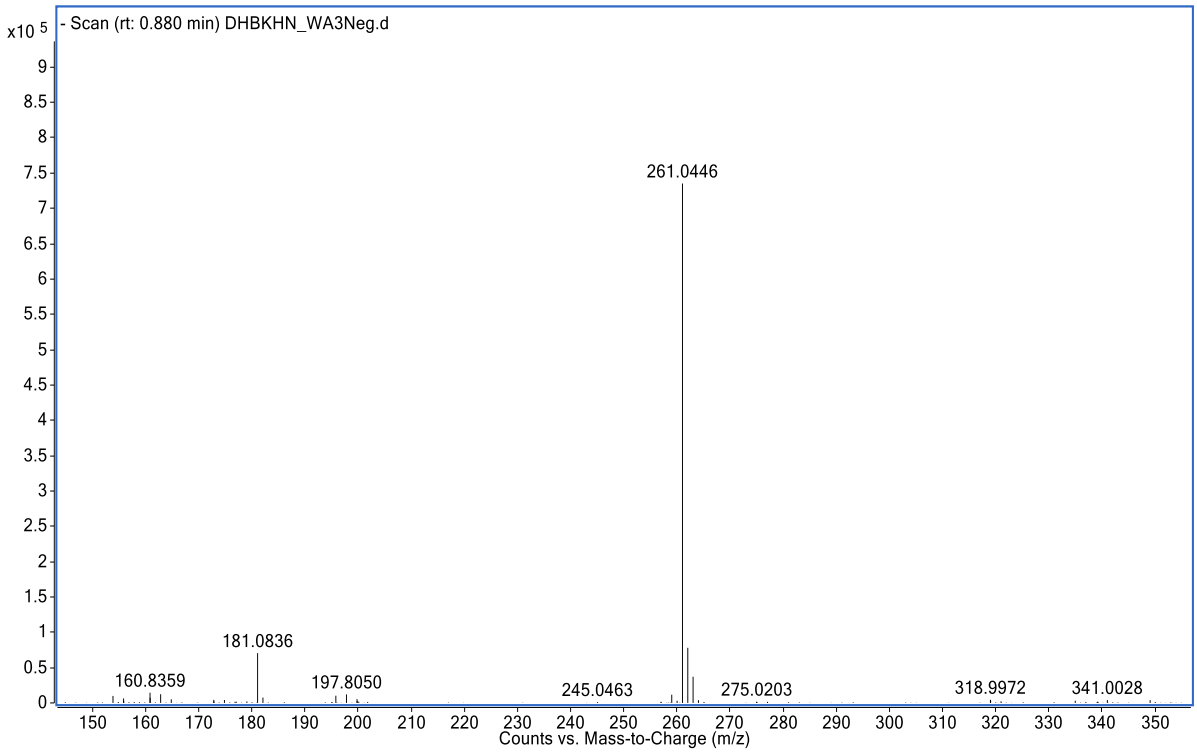


Figure S12: HR-ESI Mass Spectrum of **2** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2(*R*)-butanol)

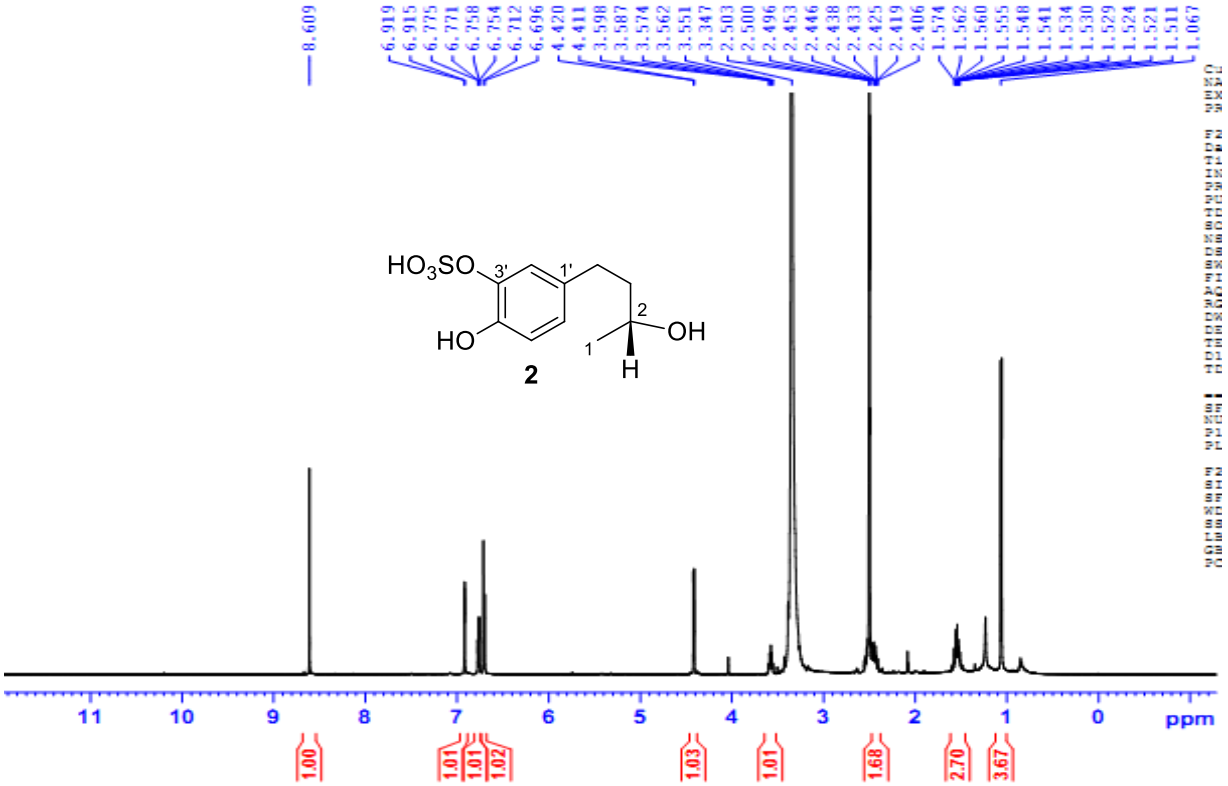


Figure S13: ^{1}H-NMR (500 MHz, DMSO) Spectrum of **2**

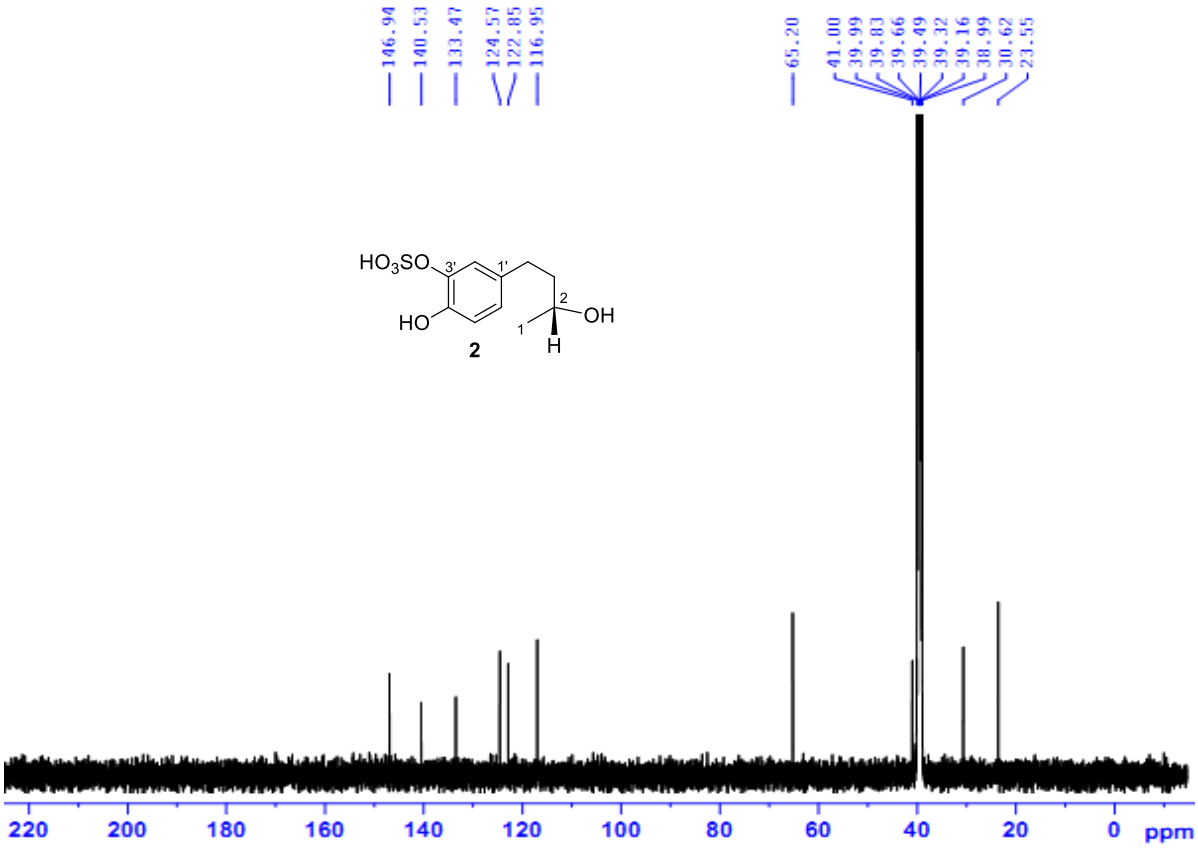


Figure S14: ^{13}C-NMR (125 MHz, DMSO) Spectrum of **2**

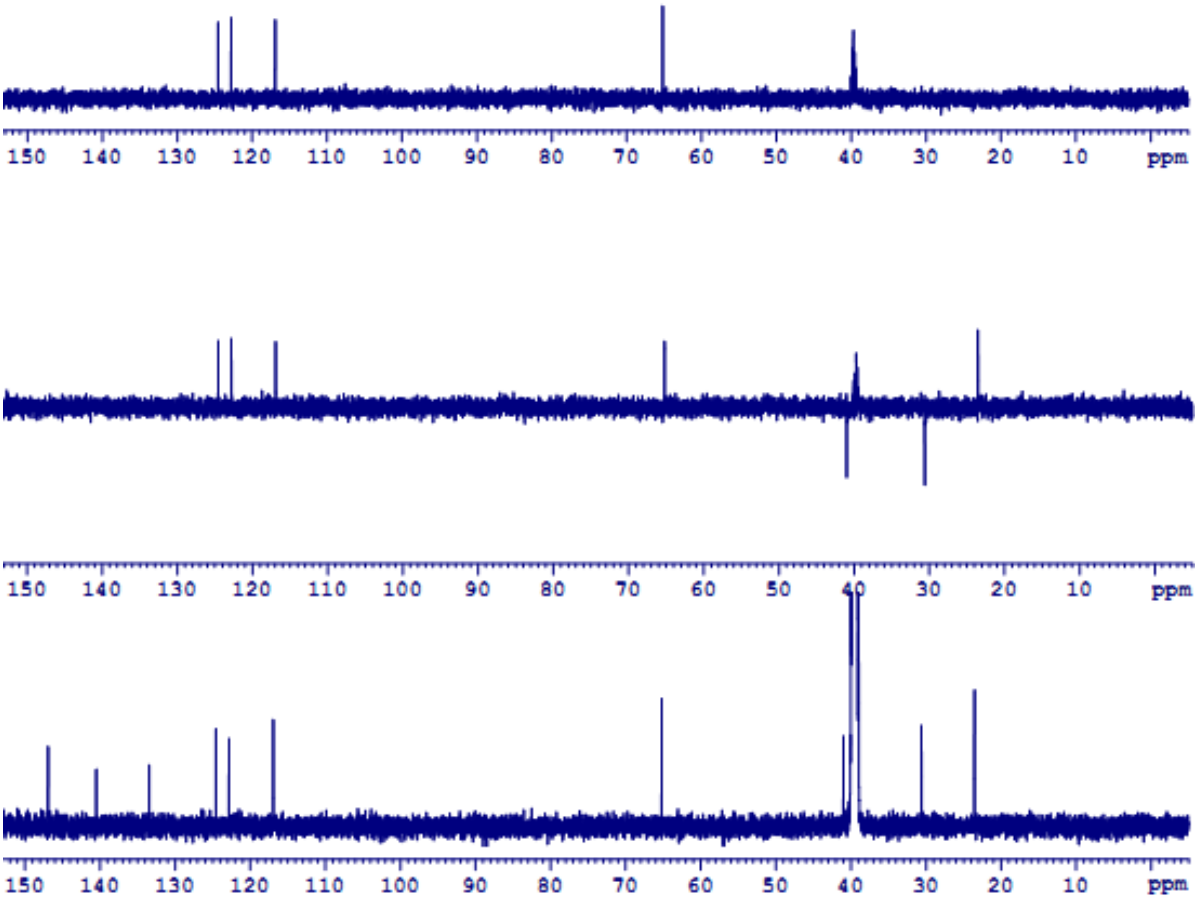


Figure S15: DEPT90 and 135 (125 MHz, DMSO) Spectrum of **2**

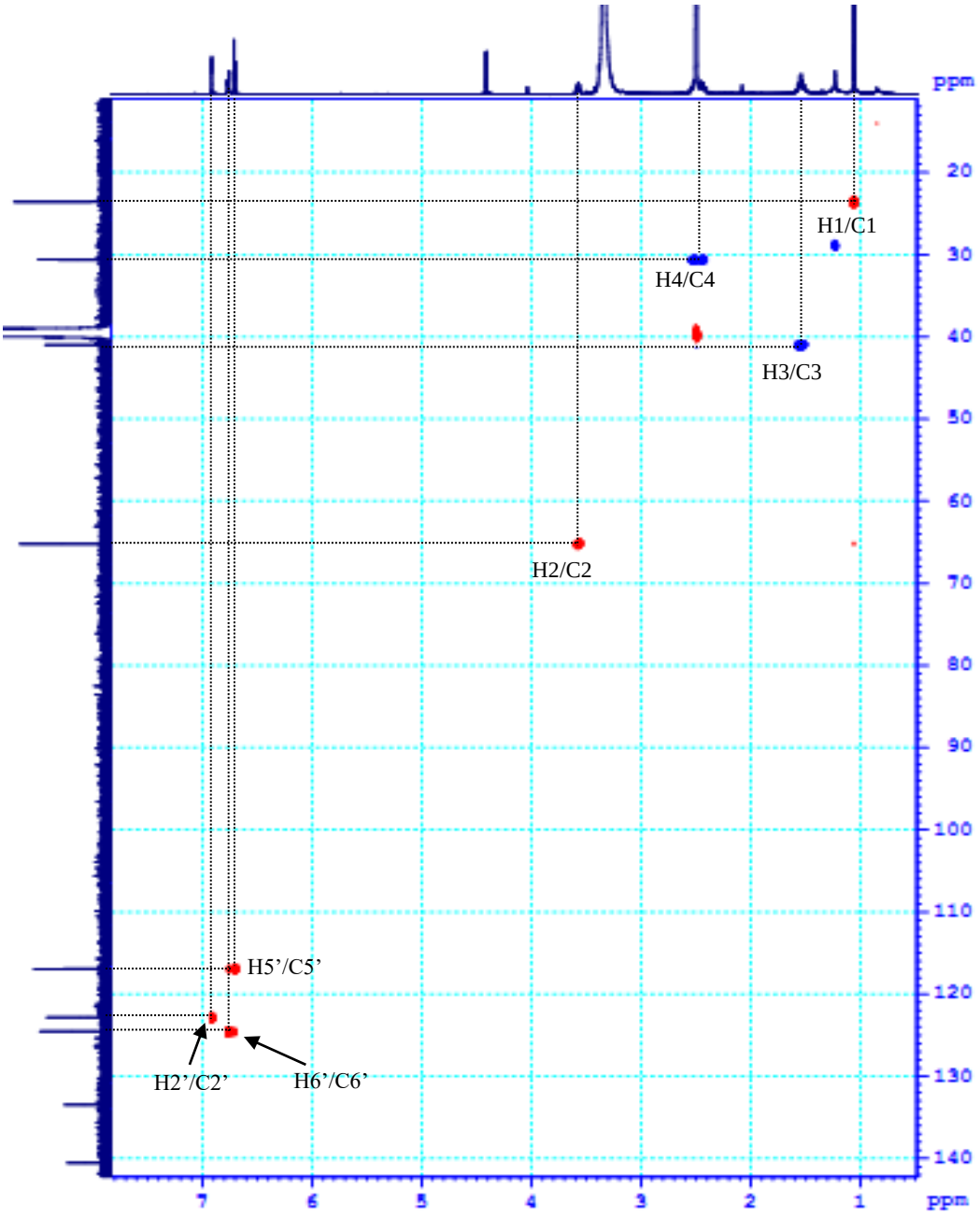


Figure S16: HSQC Spectrum of **2** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2(*R*)-butanol)

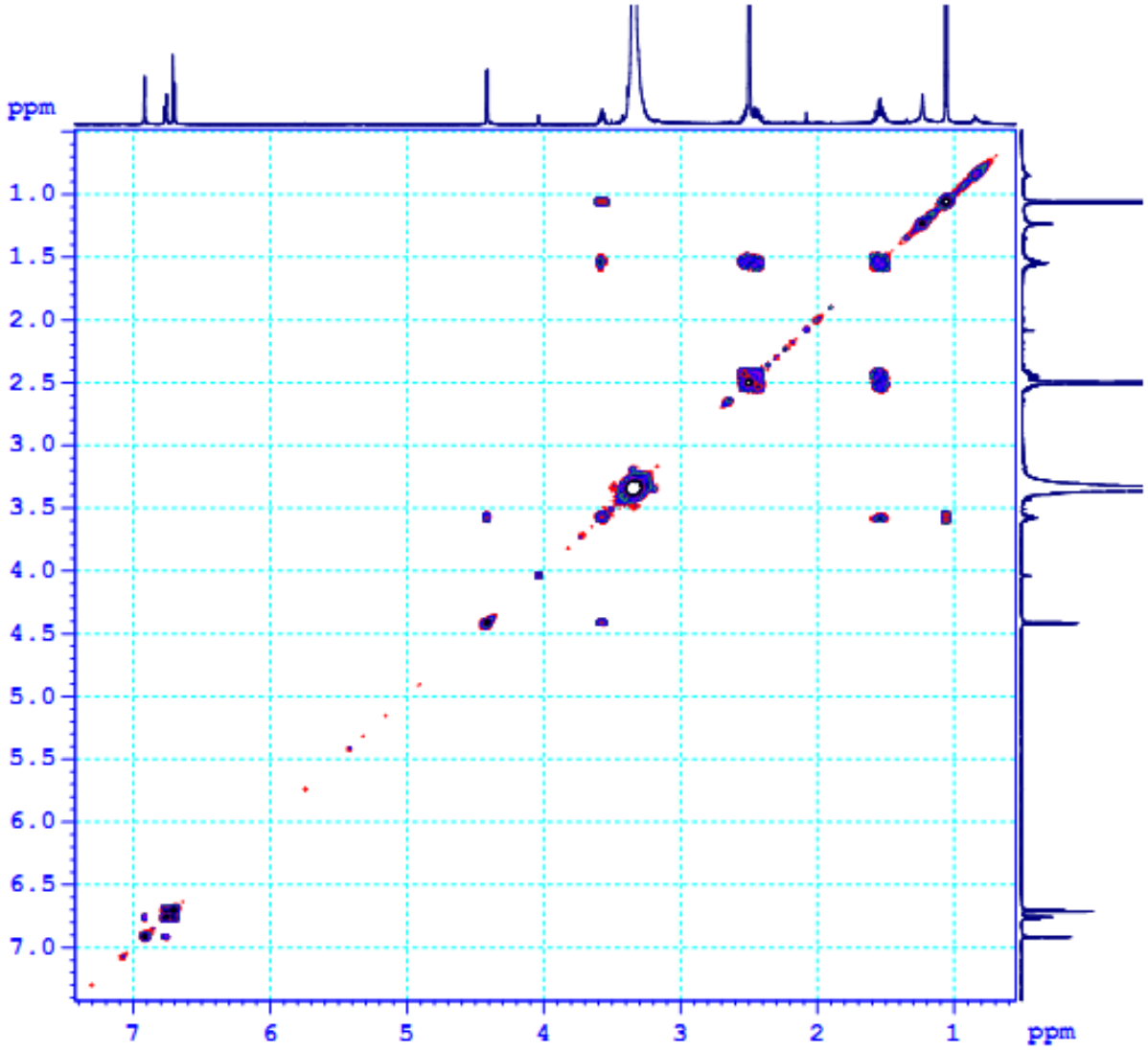


Figure S17: ^{1}H-^{1}H COSY Spectrum of **2** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2(*R*)-butanol)

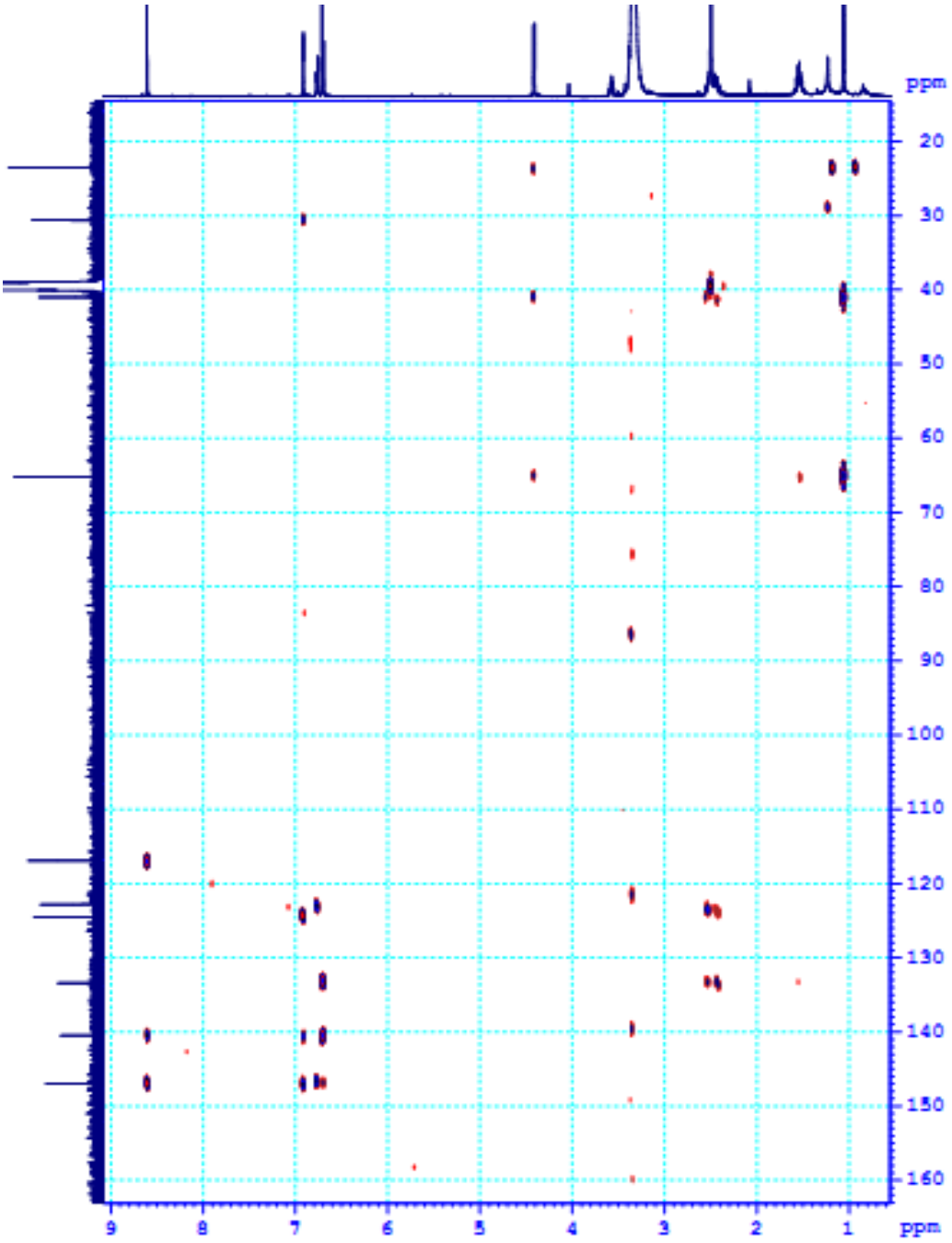


Figure S18: HMBC Spectrum of **2** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2(*R*)-butanol)

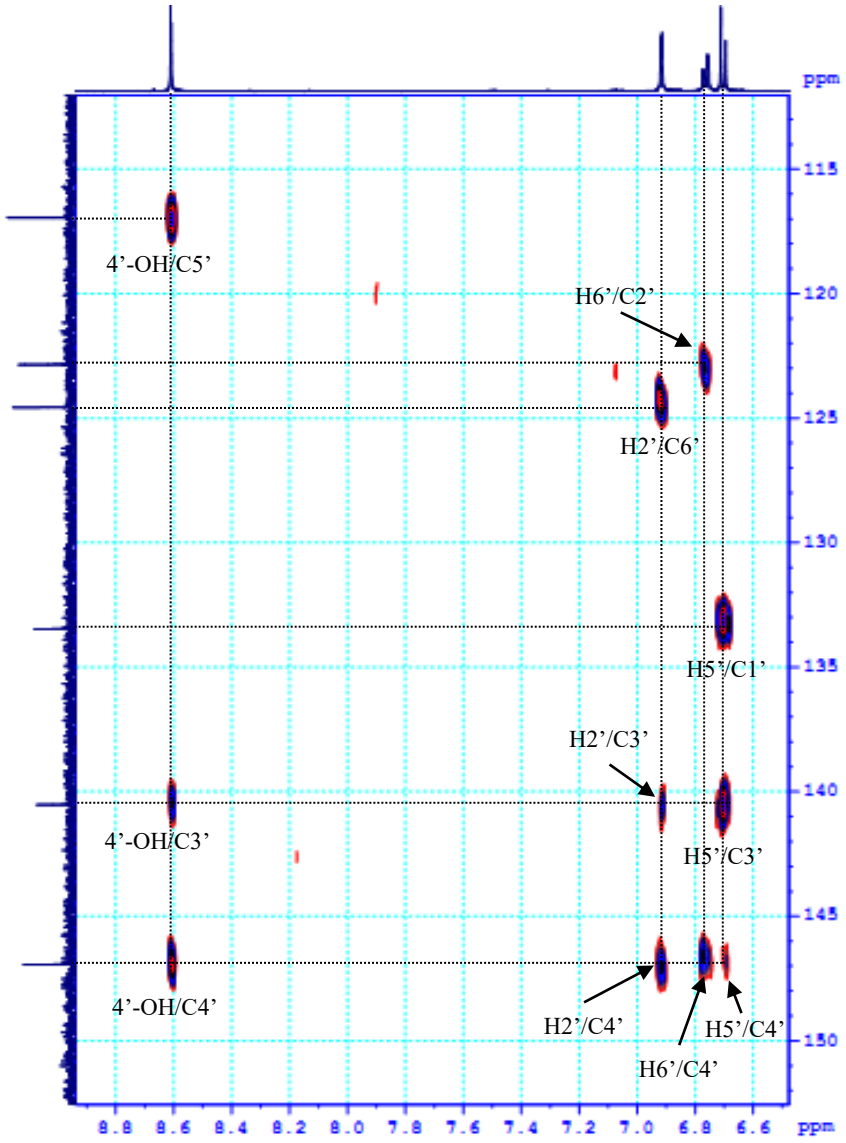


Figure S19: HMBC Spectrum of **2** (From δ_C 115 ppm to δ_C 150 ppm)

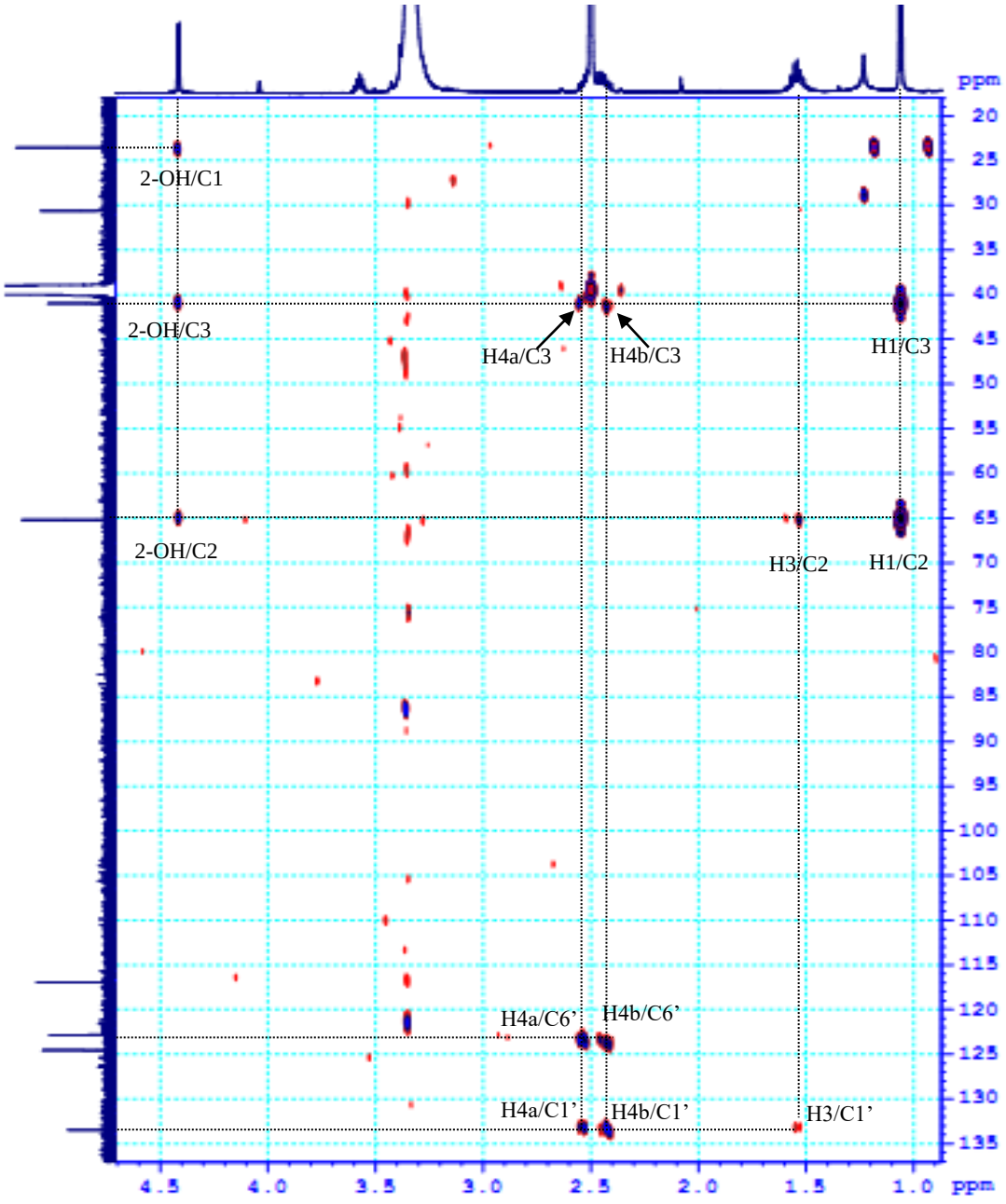


Figure S20: HMBC Spectrum of **2** (From δ_C 20 ppm to δ_C 135 ppm)

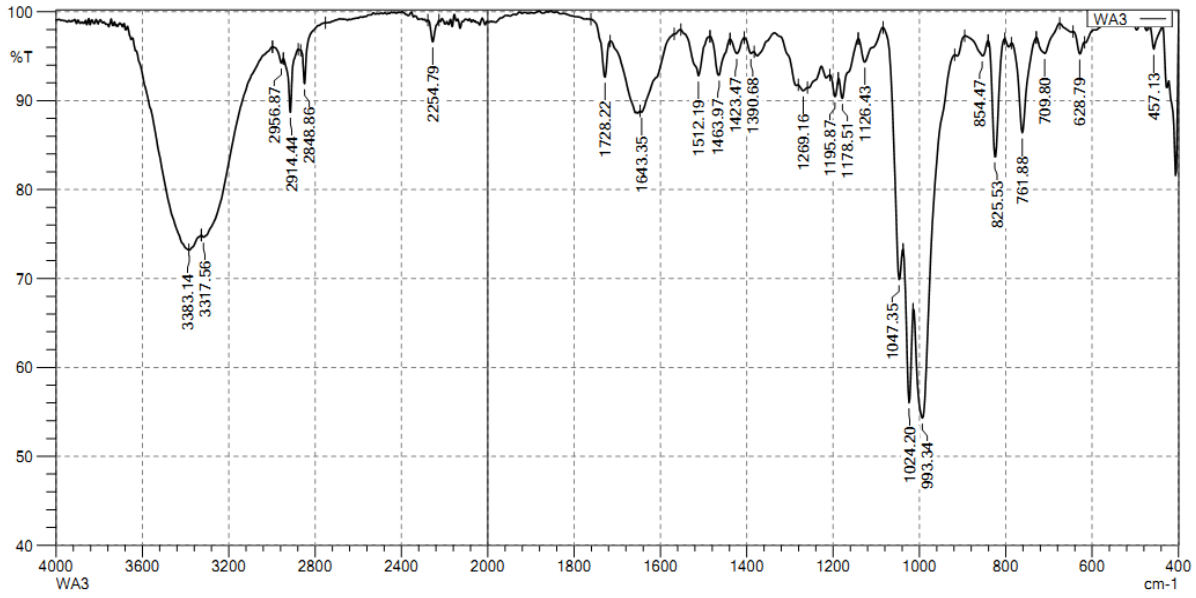


Figure S21: FT-IR Spectrum of **2** (4-(3′-*O*-sulfate-4′-hydroxyphenyl)-2(*R*)-butanol)

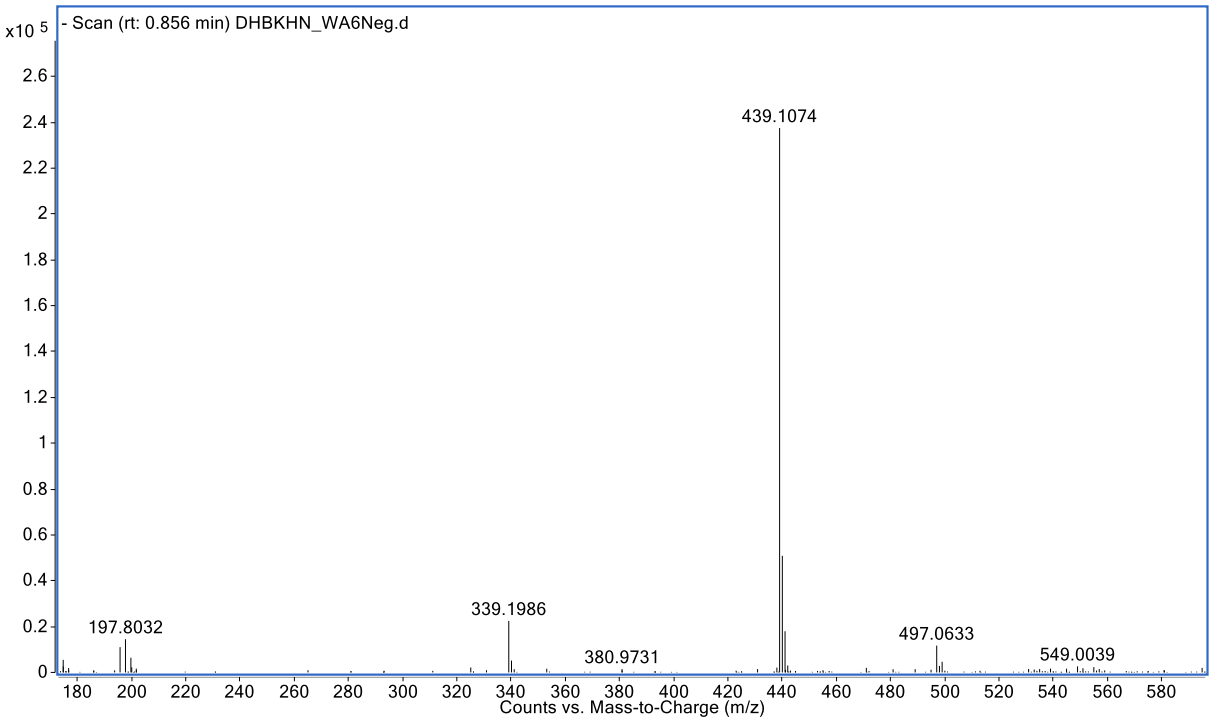


Figure S22: HR-ESI Mass Spectrum of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

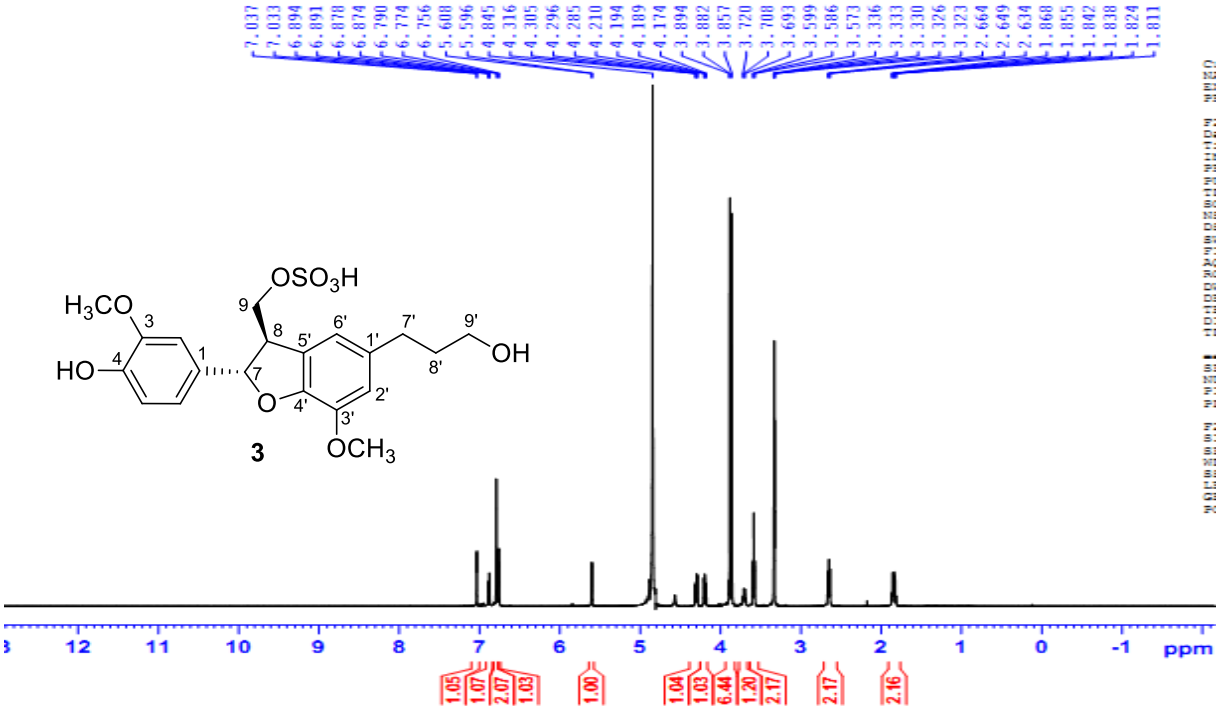


Figure S23: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of **3**

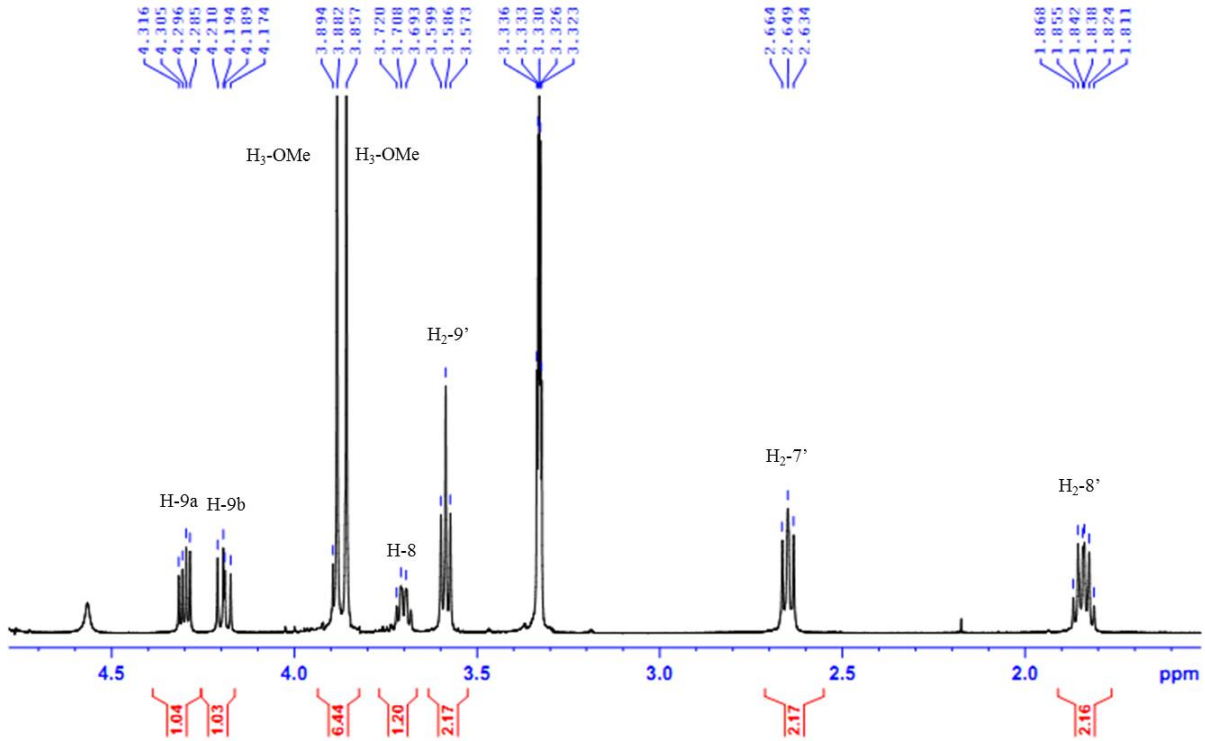


Figure S24: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of **3** (From δ_C 1.5 ppm to δ_C 4.5 ppm)

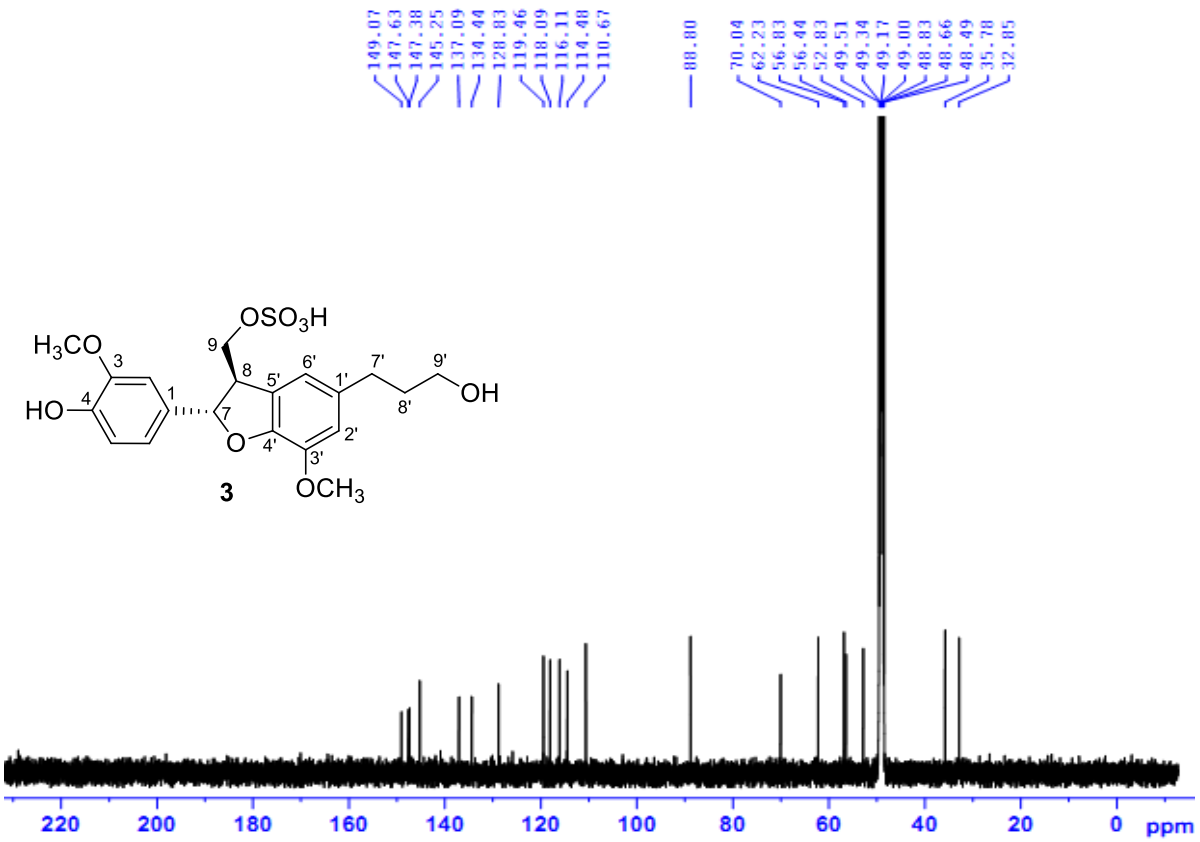


Figure S25: ^{13}C-NMR (125 MHz, CD_3OD) Spectrum of **3**

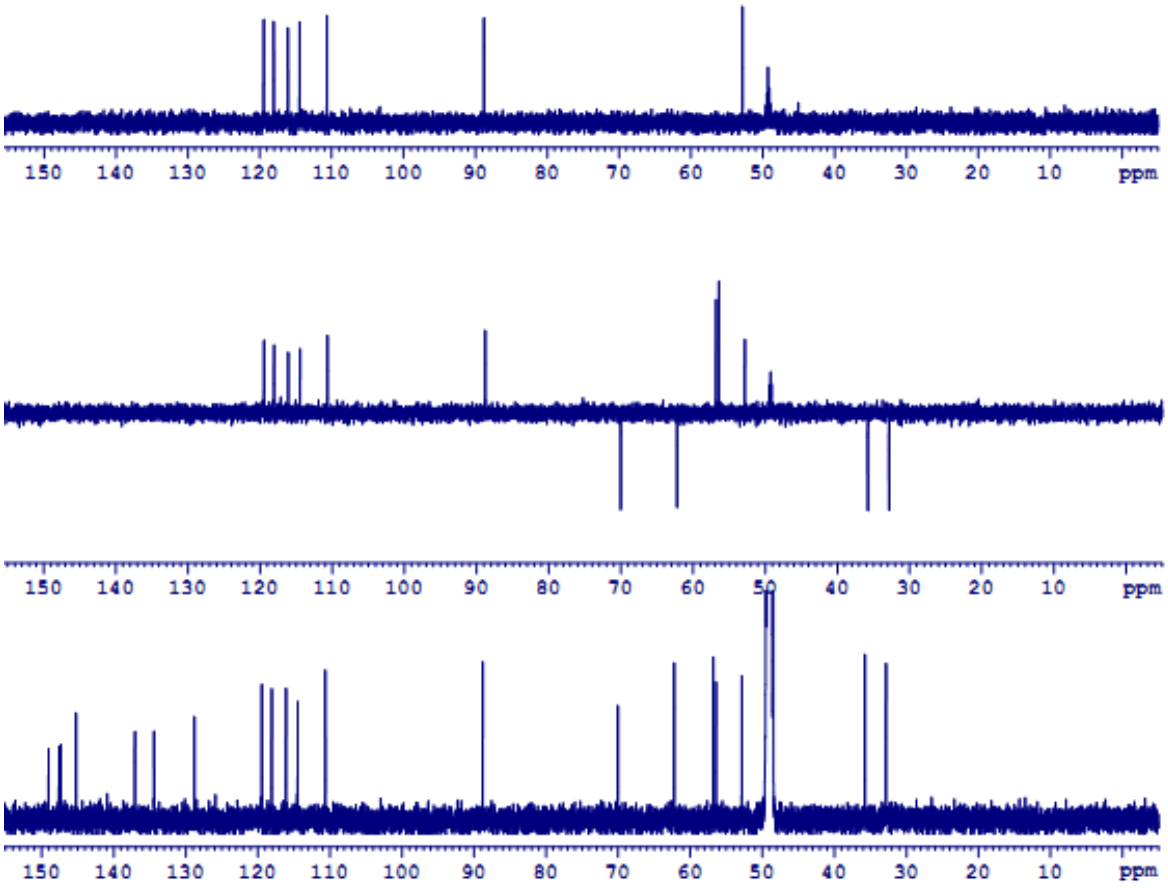


Figure S26: DEPT90 and 135 (125 MHz, CD_3OD) Spectrum of **3**

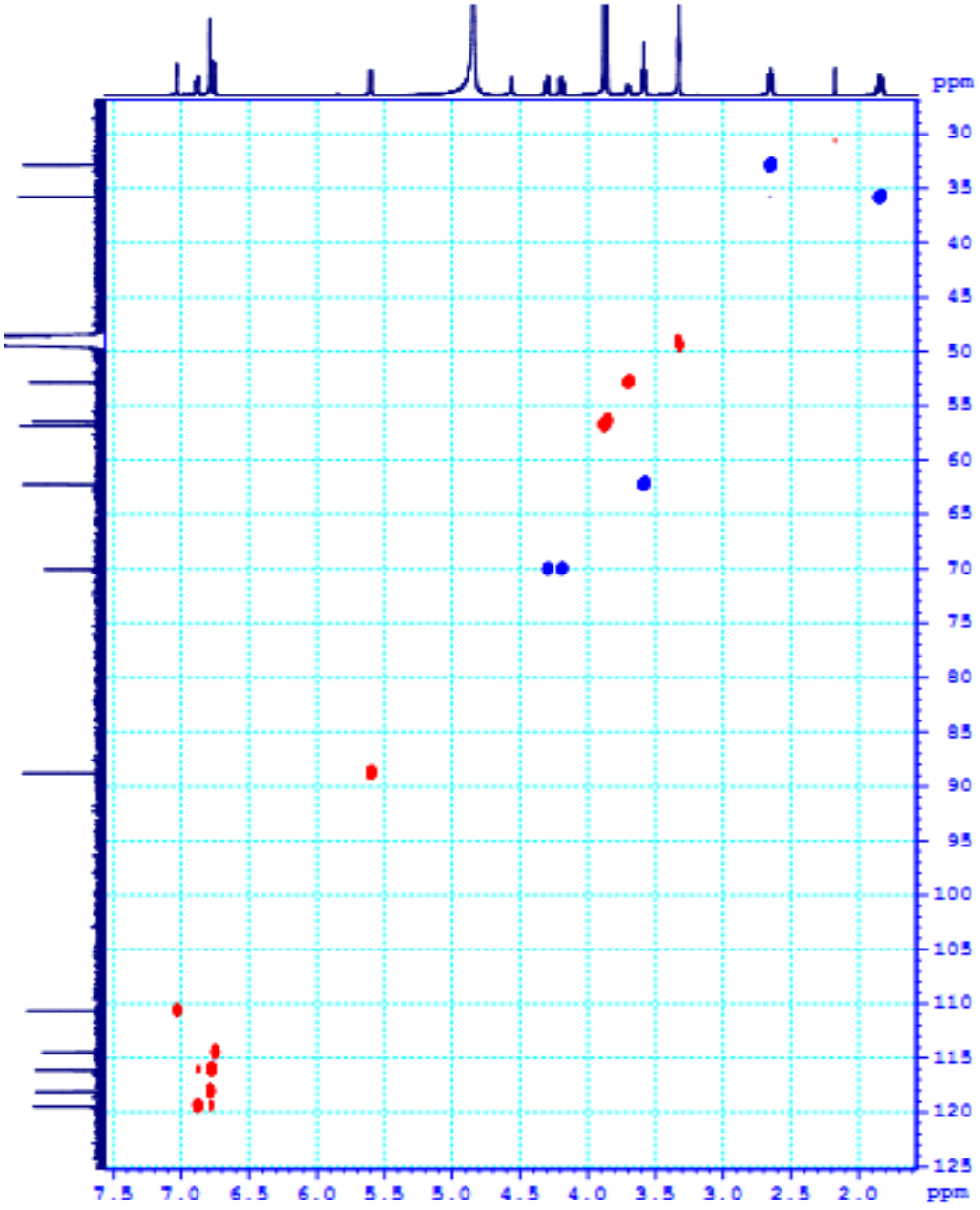


Figure S27: HSQC Spectrum of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

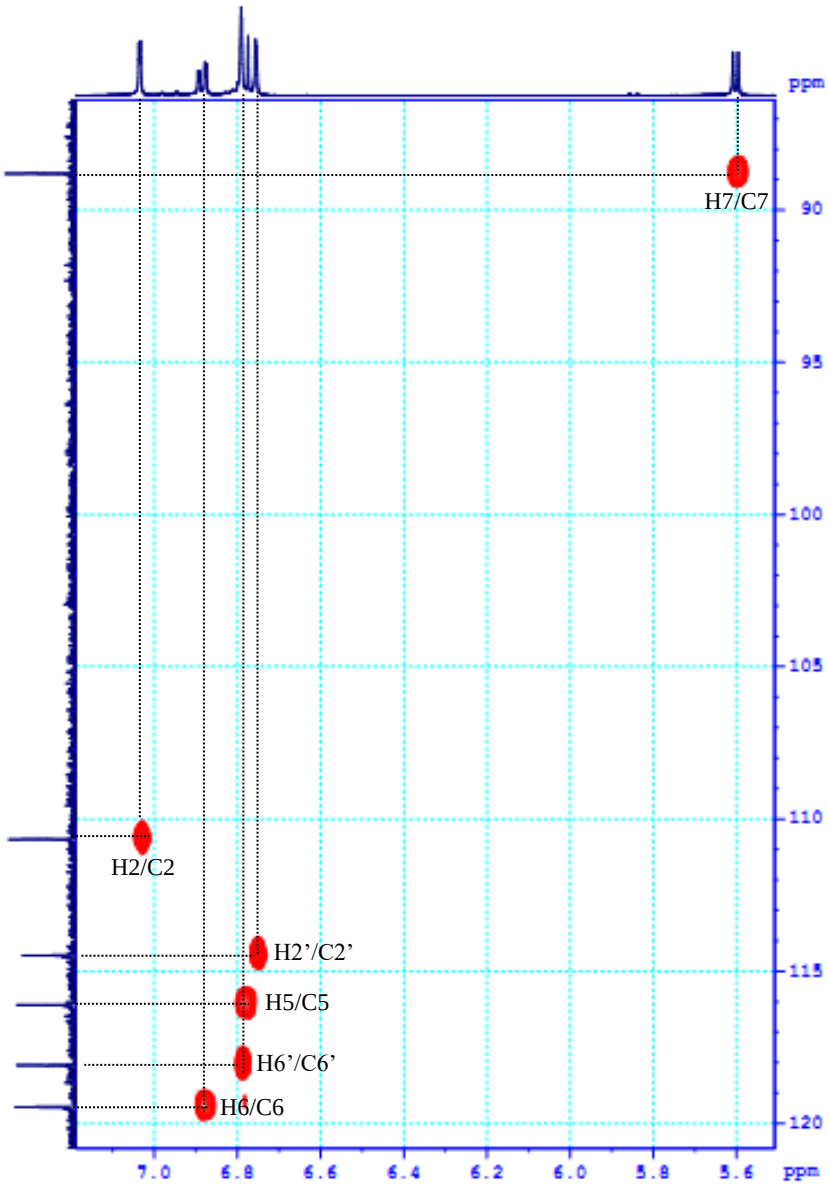


Figure S28: HSQC Spectrum of **3** (From δ_C 85 ppm to δ_C 120 ppm)

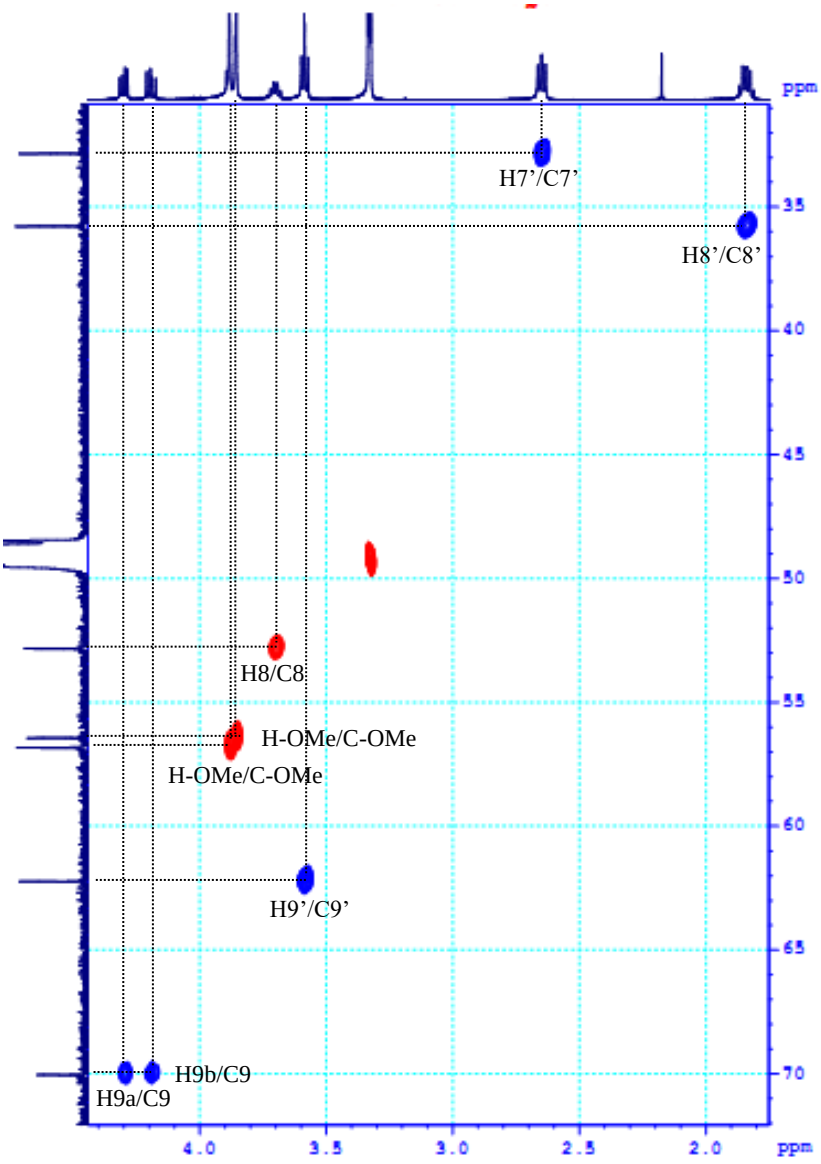


Figure S29: HSQC Spectrum of **3** (From δ_C 30 ppm to δ_C 70 ppm)

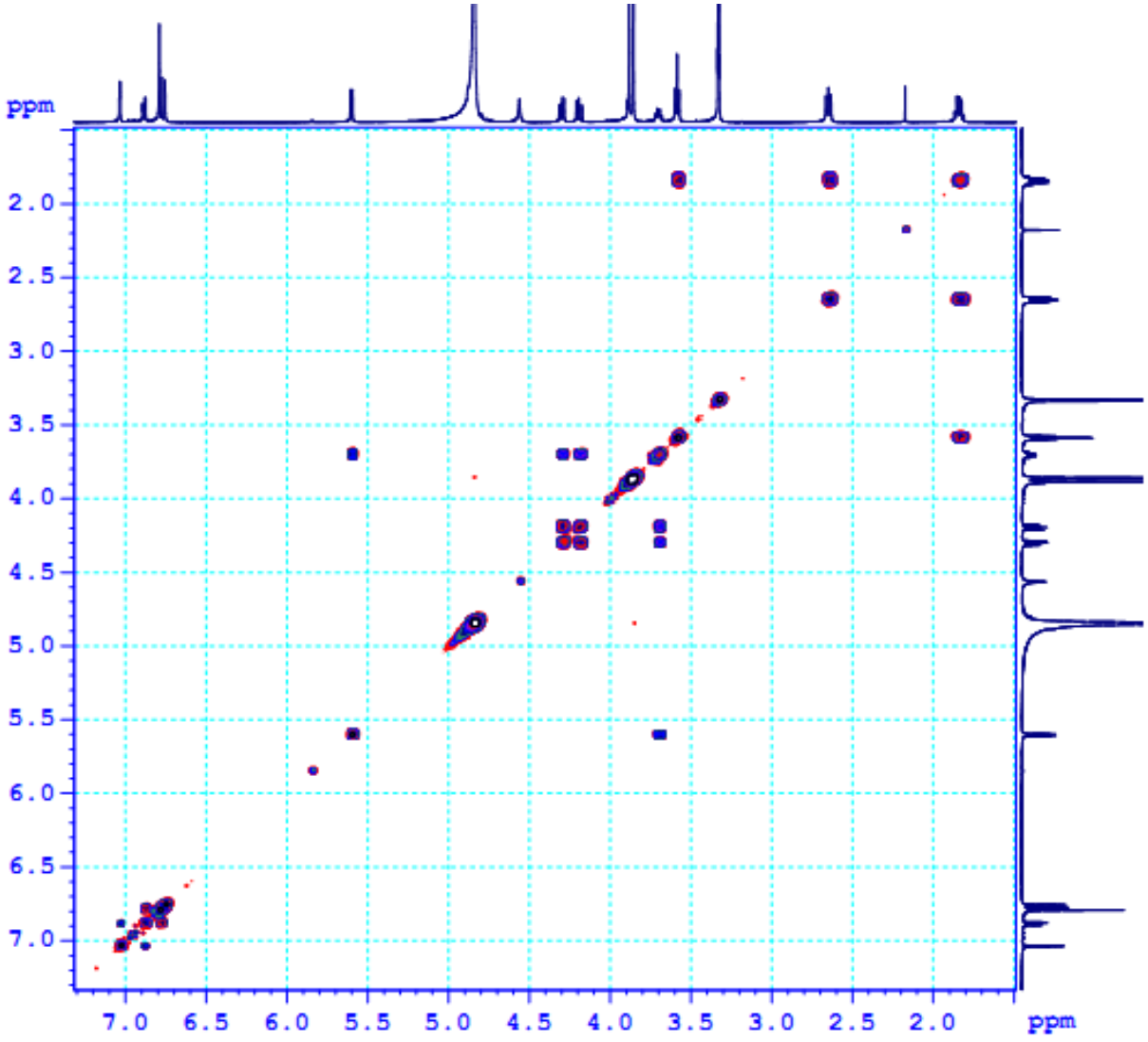


Figure S30: ^{1}H-^{1}H COSY Spectrum of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

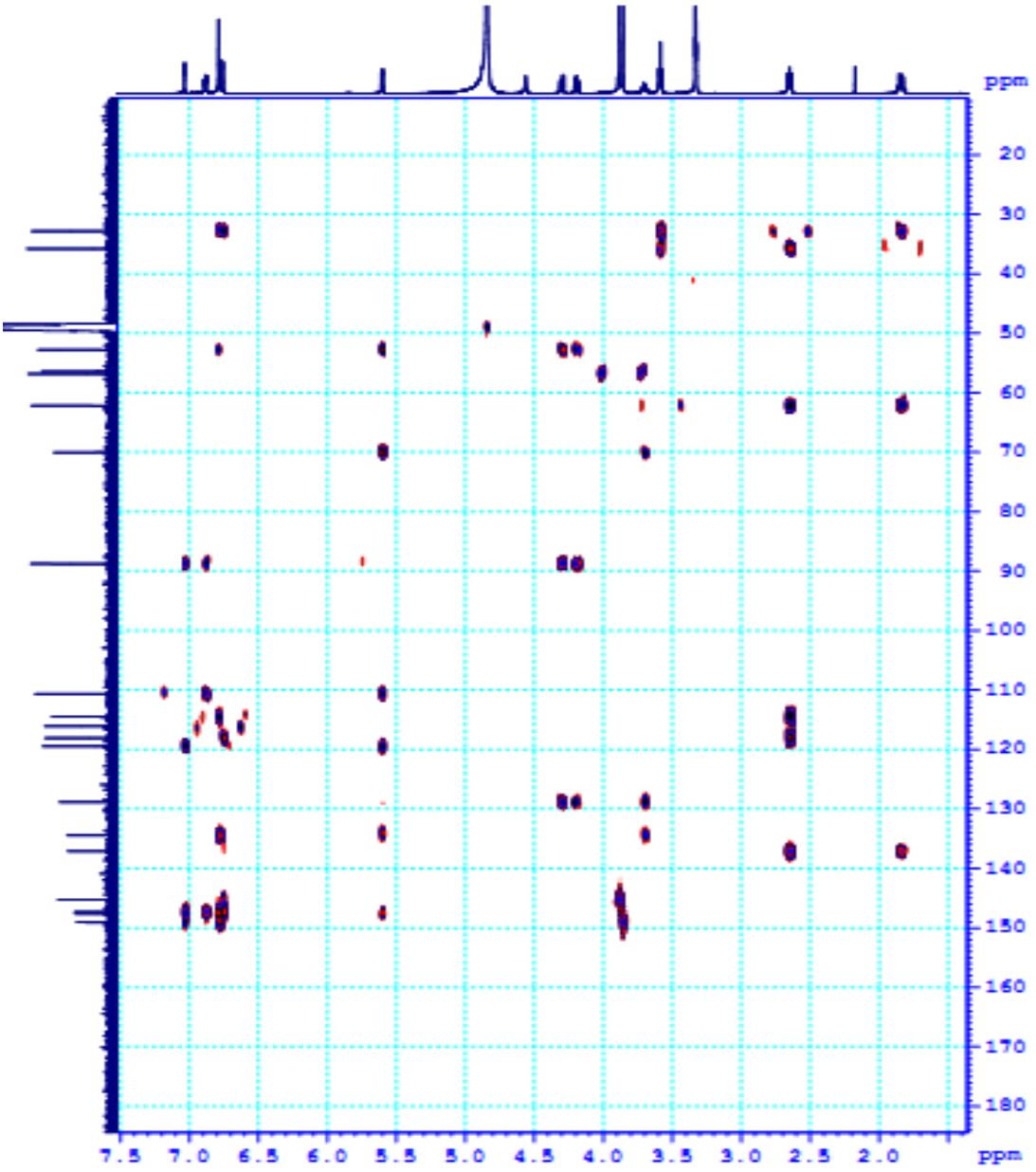


Figure S31: HMBC Spectrum of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

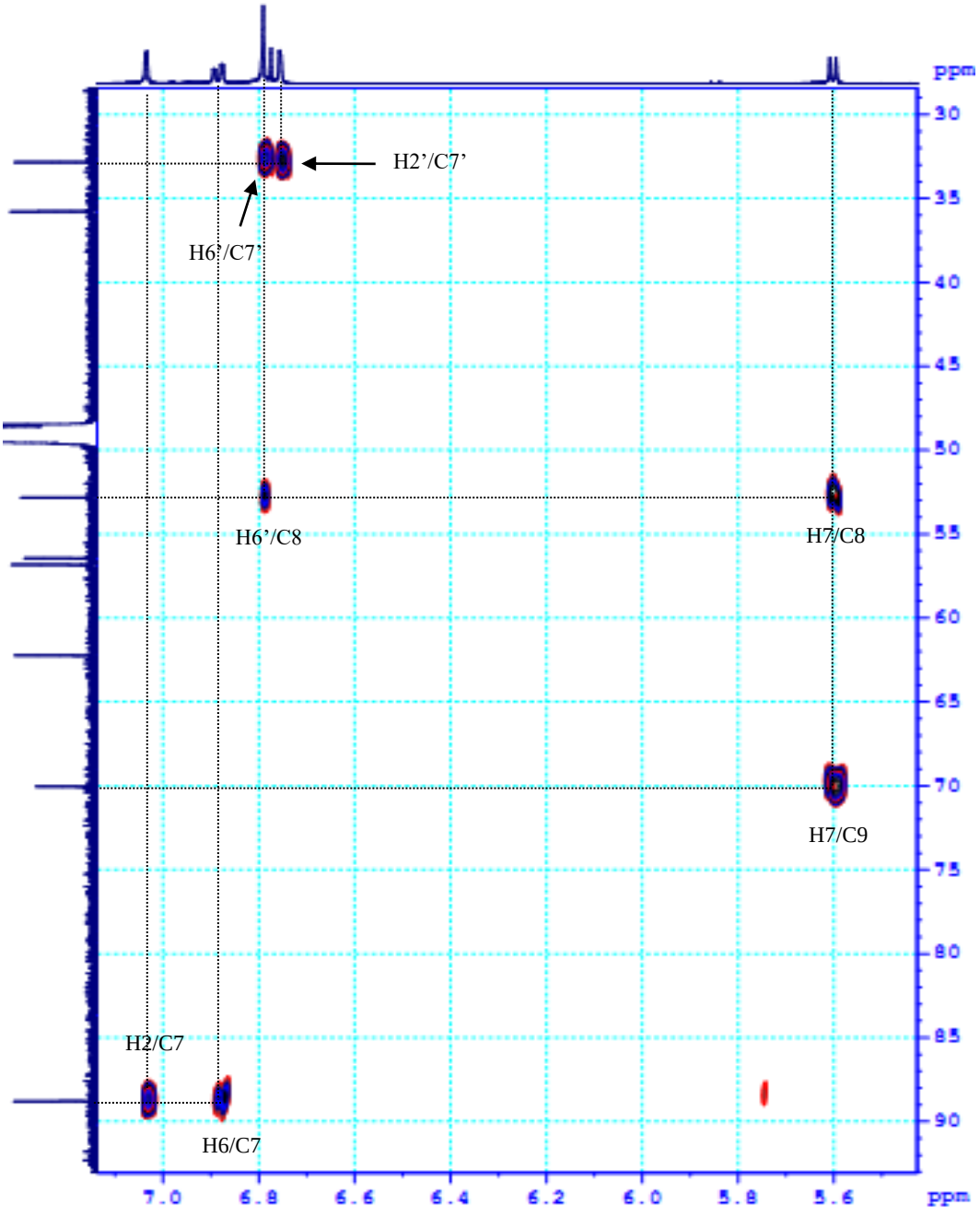


Figure S32: HMBC Spectrum of **3** (From δ_C 30 ppm to δ_C 90 ppm)

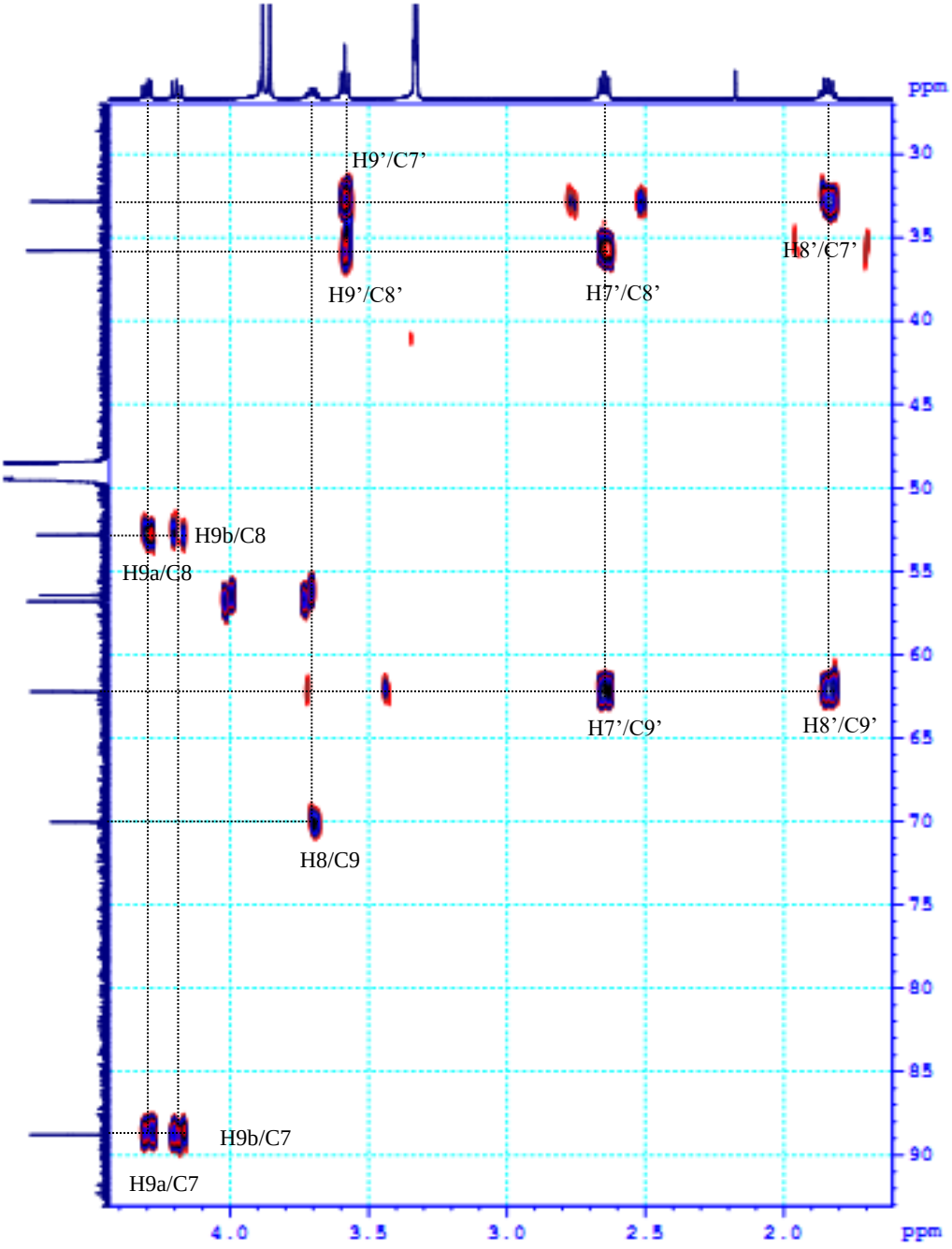


Figure S33: HMBC Spectrum of **3** (From δ_C 30 ppm to δ_C 90 ppm)

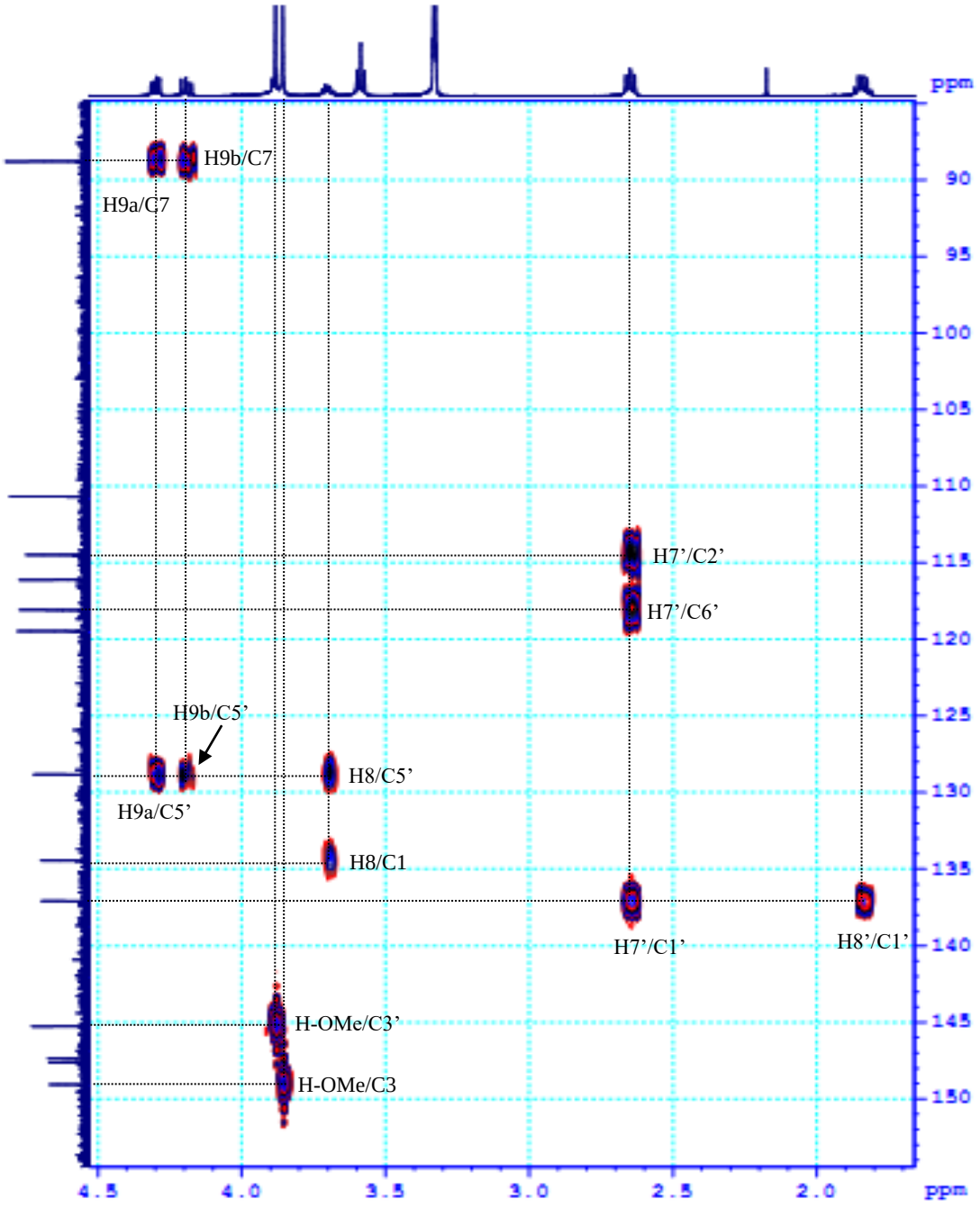


Figure S34: HMBC Spectrum of **3** (From δ_C 90 ppm to δ_C 150 ppm)

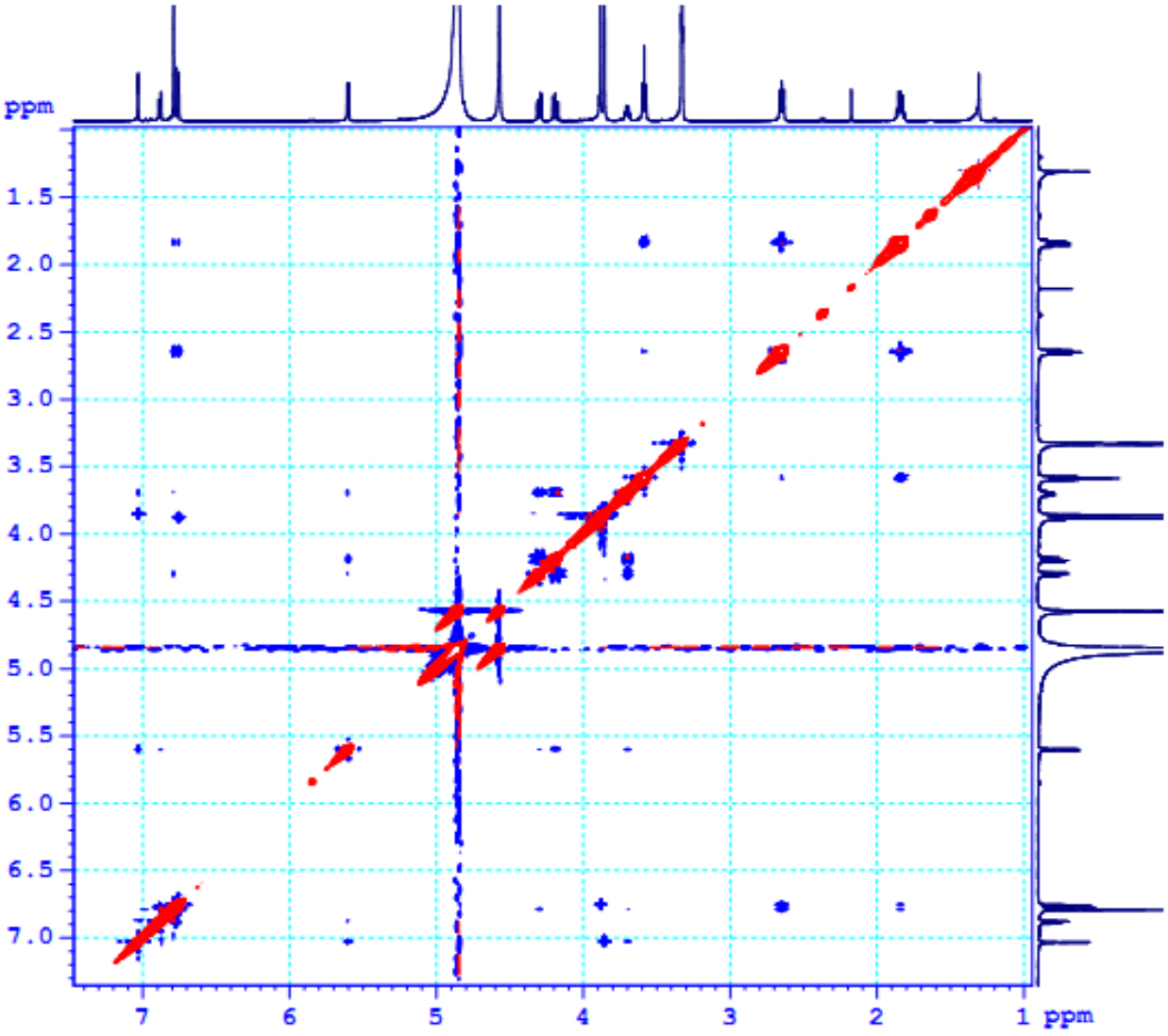


Figure S35: NOESY Spectrum of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

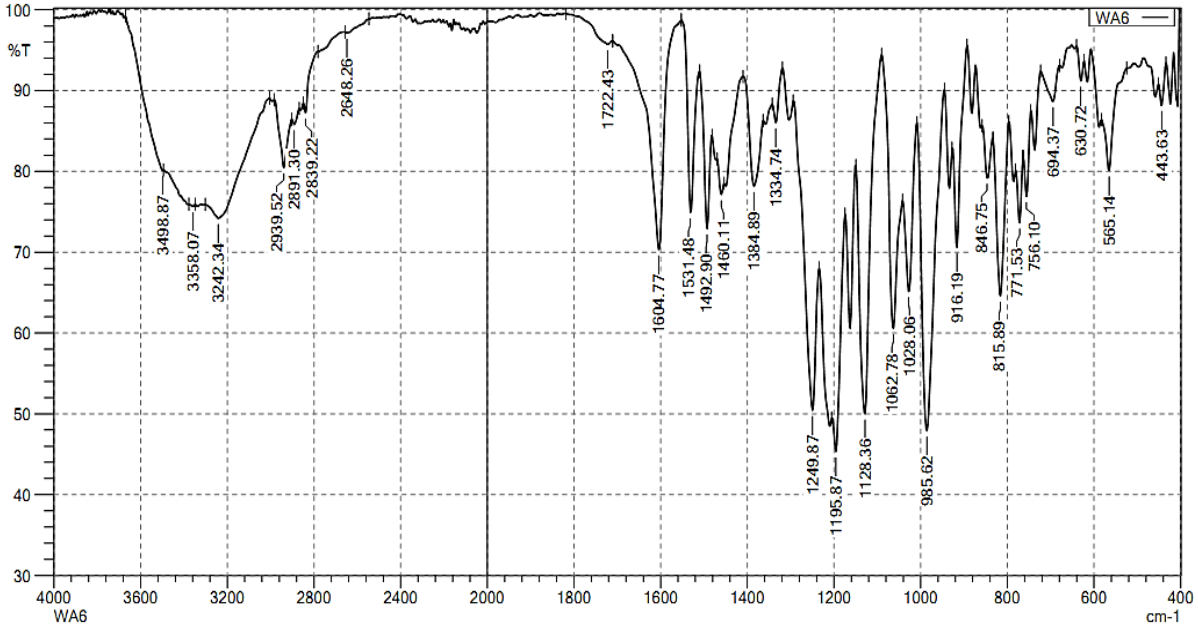


Figure S36: FT-IR Spectrum of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

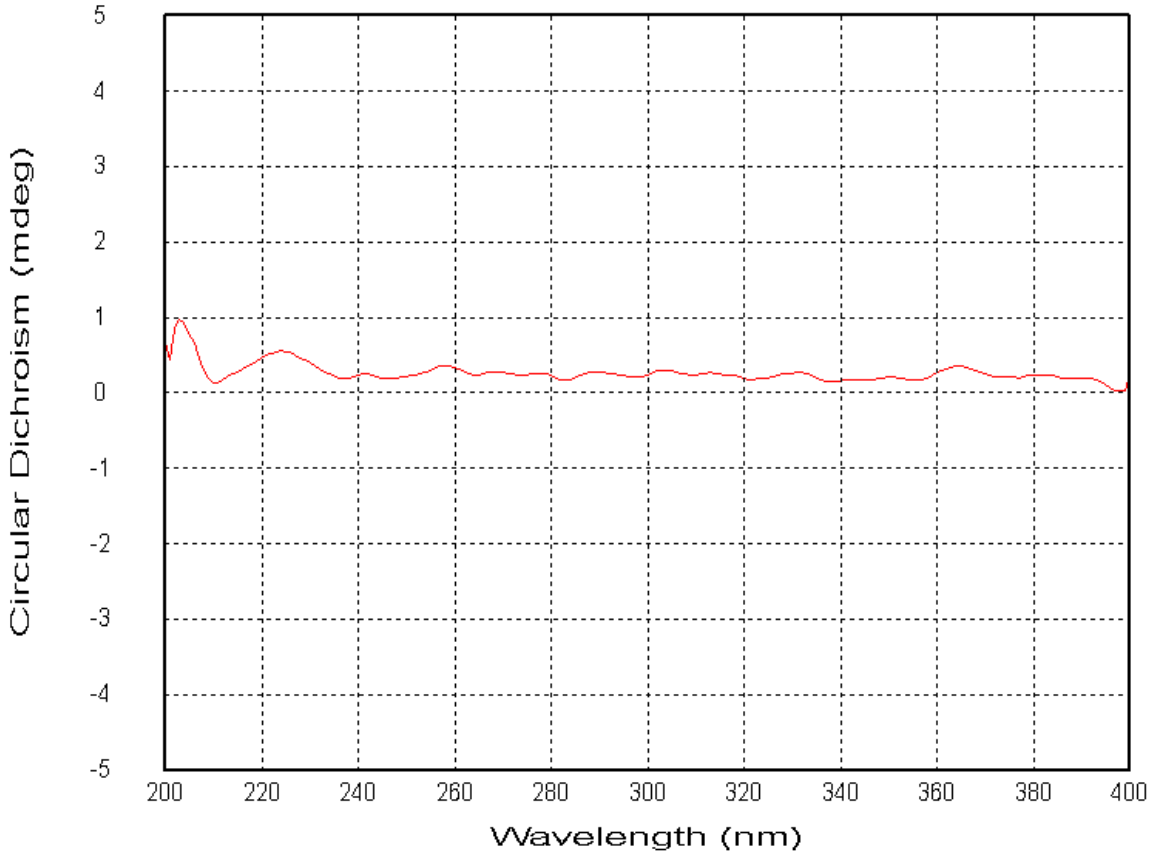


Figure S37: CD Spectrum (CH_3OH) of **3** (dihydrodehydrodiconiferyl alcohol-9-*O*-sulfate)

(+)-pinoresinol-4-O-sulfate (4): ^{1}H NMR (500 MHz, DMSO-d_6) δ_H 8.87 (1H, *s*, 4-OH), 7.04 (1H, *d*, *J* = 8.0 Hz, H-5), 6.92 (1H, *d*, *J* = 1.5 Hz, H-2), 6.89 (1H, *d*, *J* = 1.5 Hz, H-2′), 6.82 (1H, *dd*, *J* = 1.5, 8.0 Hz, H-6), 6.76 (1H, *dd*, *J* = 1.5, 8.0 Hz, H-6′), 6.72 (1H, *d*, *J* = 8.0 Hz, H-5′), 4.67 (1H, *d*, *J* = 4.5 Hz, H-7), 4.60 (1H, *d*, *J* = 4.5 Hz, H-7′), 4.14 (2H, *m*, H-9a, H-9′a), 3.76 (3H, *s*, 3′-OCH_3), 3.75 (2H, *m*, H-9b, H-9′b), 3.74 (3H, *s*, 3-OCH_3), 3.05 (2H, m, H-8, H-8′). ^{13}C NMR (125 Hz, DMSO-d_6) data (Table S2).

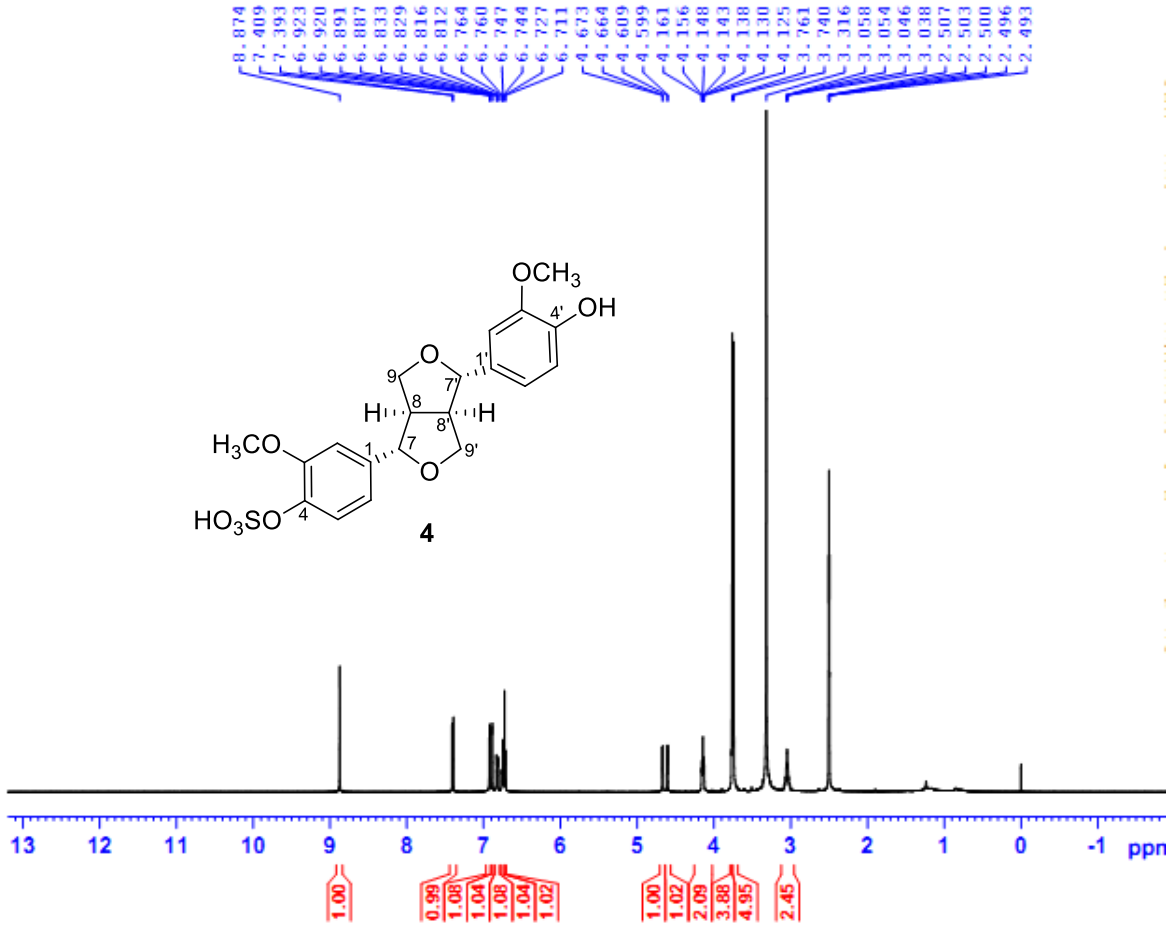


Figure S38: ^{1}H-NMR (500 MHz, DMSO) Spectrum of (+)-pinoresinol-4-*O*-sulfate (**4**)

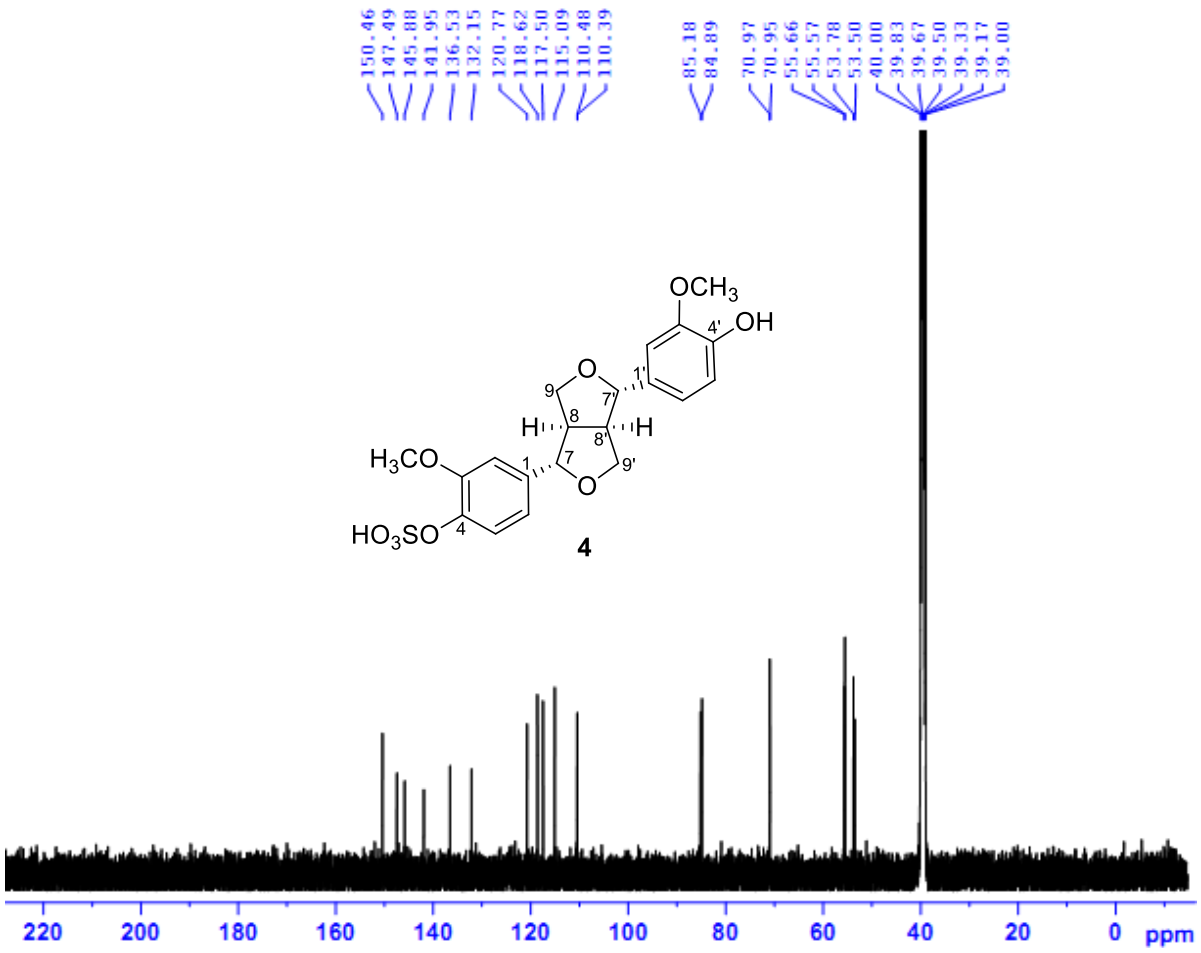


Figure S39: ^{13}C-NMR (125 MHz, DMSO) Spectrum of (+)-pinoresinol-4-*O*-sulfate (**4**)

(+)-pinoresinol-4-*O*-glucoside (5): ^{1}H NMR (500 MHz, CD_3OD) δ_H 7.17 (1H, *d*, *J* = 8.0 Hz, H-5), 7.05 (1H, *d*, *J* = 2.0 Hz, H-2), 6.97 (1H, *d*, *J* = 2.0 Hz, H-2′), 6.95 (1H, *dd*, *J* = 2.0, 8.0 Hz, H-6), 6.84 (1H, *dd*, *J* = 2.0, 8.0 Hz, H-6′), 6.79 (1H, *d*, *J* = 8.0 Hz, H-5′), 4.90 (1H, *d*, *J* = 7.0 Hz, H-1″), 4.79 (1H, *d*, *J* = 4.5 Hz, H-7′), 4.74 (1H, *d*, *J* = 4.5 Hz, H-7), 4.27 (2H, *m*, H-9a, H-9′a), 3.89 (3H, *s*, 3-OCH_3), 3.88 (3H, *s*, 3′-OCH_3), 3.88 (2H, *m*, H-9b, H-9′b), 3.16 (2H, *m*, H-8, H-8′), 3.88 (1H, *m*, H-6″a), 3.71 (1H, *m*, H-6″b), 3.40-3.55 (4H, *m*). ^{13}C NMR (125 Hz, CD_3OD) data (Table S2).

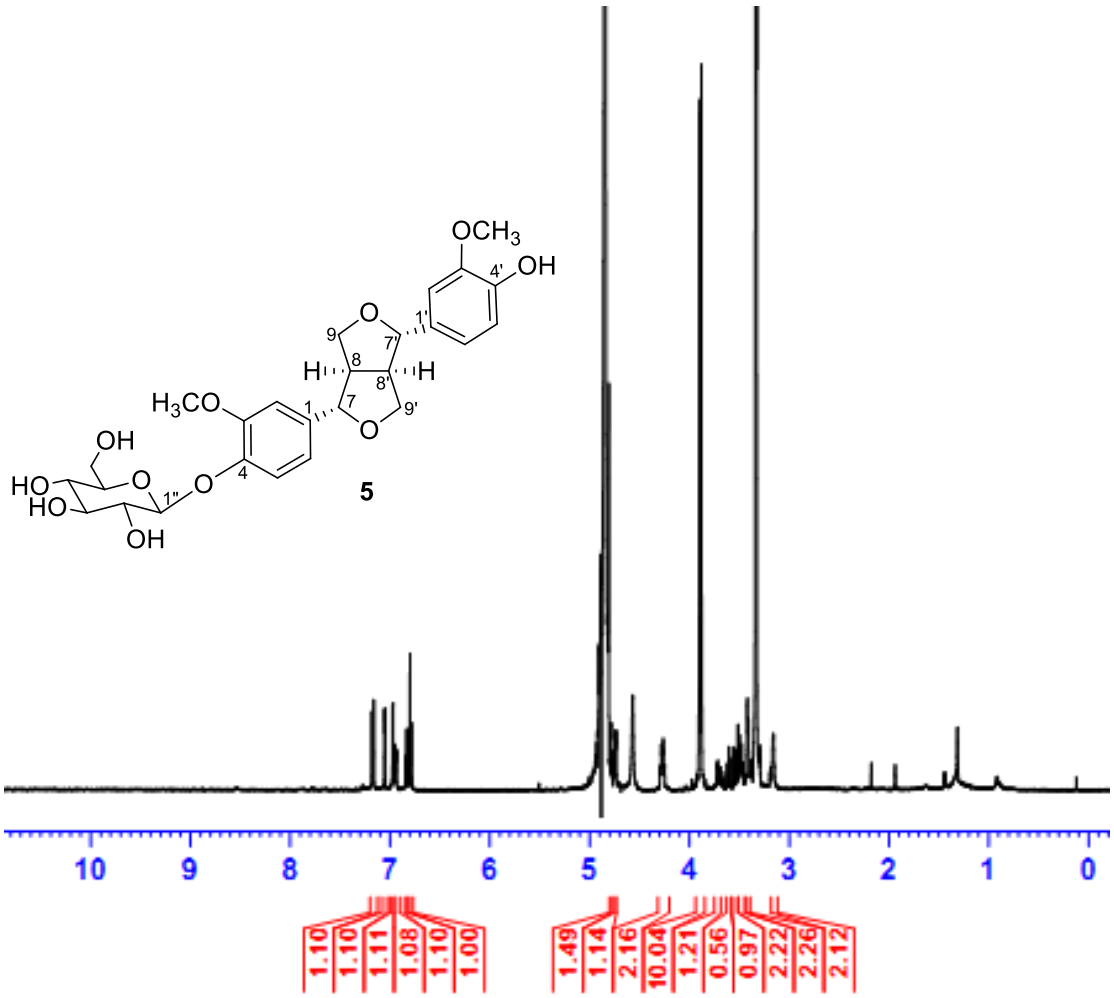


Figure S40: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of (+)-pinoresinol-4-*O*-glucoside (**5**)

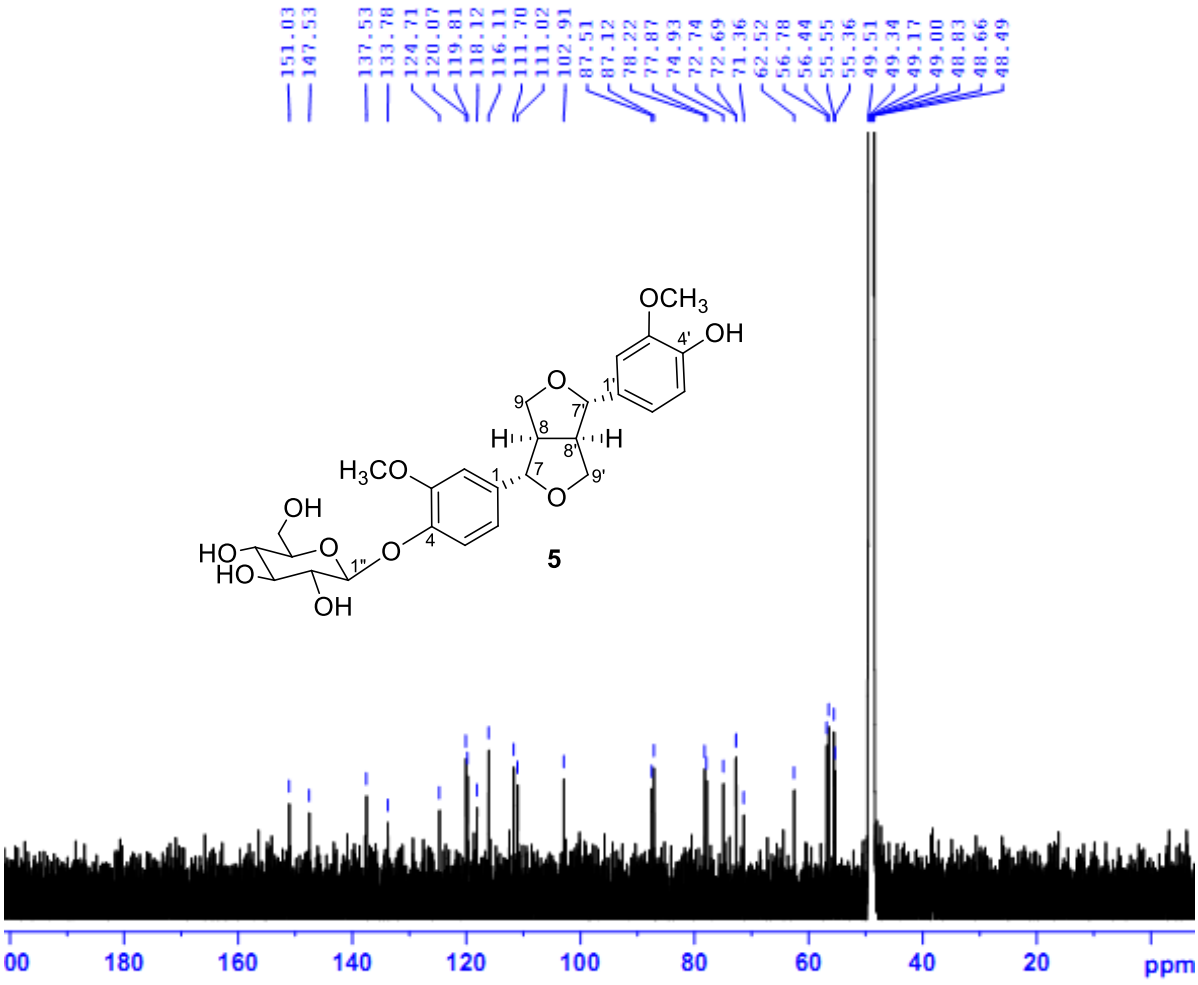


Figure S41: ^{13}C-NMR (125 MHz, CD_3OD) Spectrum of (+)-pinoresinol-4-*O*-glucoside (**5**)

Dihydrodehydrodiconiferyl alcohol-4-*O*-glucoside (6): ^{1}H NMR (500 MHz, CD_3OD) δ_H 7.17 (1H, *d*, *J* = 8.0 Hz, H-5), 7.05 (1H, *d*, *J* = 2.0 Hz, H-2), 6.96 (1H, *dd*, *J* = 2.0, 8.0 Hz, H-6), 6.75 (1H, *s*, H-2′), 6.73 (1H, *s*, H-6′), 5.58 (1H, *d*, *J* = 6.0 Hz, H-7), 4.85 (1H, *d*, *J* = 7.5 Hz, H-1″), 3.88 (3H, *s*, 3-OCH_3), 3.86 (2H, *m*, H-6″a, H-9a), 3.85 (3H, *s*, 3′-OCH_3), 3.76 (1H, *m*, H-9b), 3.69 (1H, *m*, H-6″b), 3.60 (2H, *t*, *J* = 6.5 Hz, H_2-9′), 3.50 (1H, *m*, H-8), 3.41-3.47 (4H, *m*), 2.64 (2H, *t*, *J* = 7.5 Hz, H_2-7′), 1.83 (2H, *m*, H_2-8′). ^{13}C NMR (125 Hz, CD_3OD) data (Table S2).

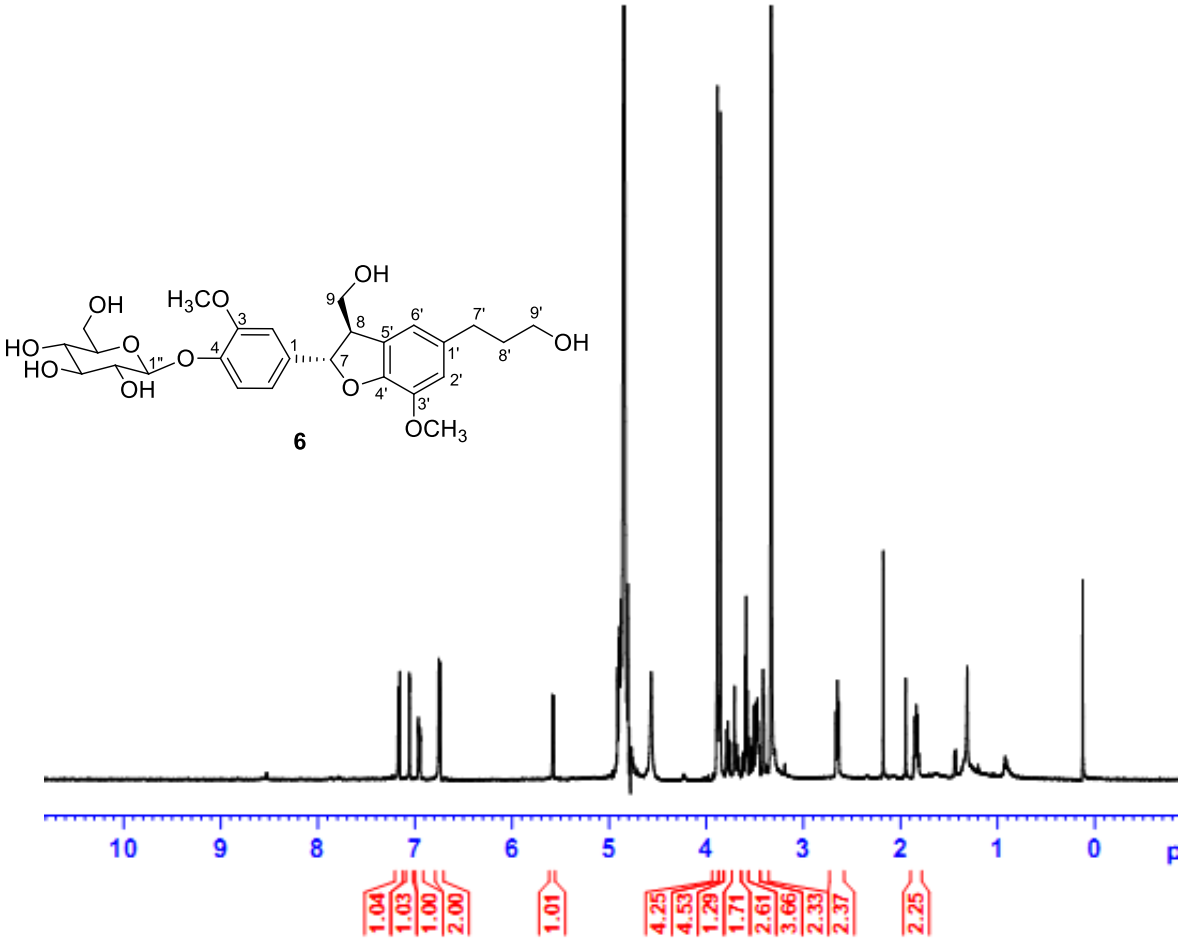


Figure S42: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of dihydrodehydrodiconiferyl alcohol-4-*O*-glucoside (**6**)

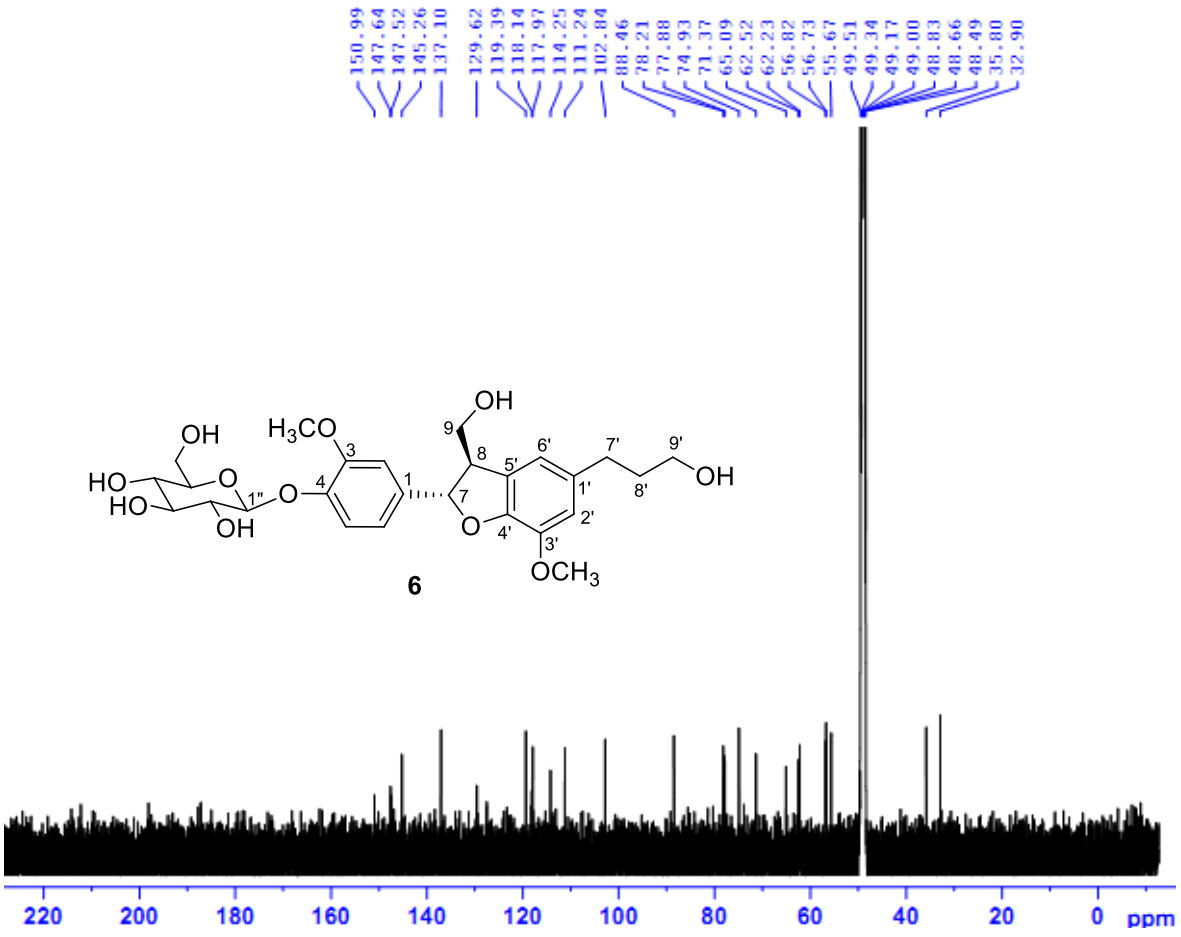


Figure S43: ^{13}C-NMR (125 MHz, CD_3OD) Spectrum of dihydrodehydrodiconiferyl alcohol-4-*O*-glucoside (**6**)

(+)-isolarisiresinol-9-*O*-sulfate (7): ^{1}H NMR (500 MHz, CD_3OD) δ_H 6.78 (1H, *d*, *J* = 1.5 Hz, H-2), 6.75 (1H, *d*, *J* = 8.0 Hz, H-5), 6.68 (1H, *s*, H-2′), 6.62 (1H, *dd*, *J* = 1.5, 8.0 Hz, H-6), 6.22 (1H, *s*, H-5′), 4.13 (1H, *dd*, *J* = 3.0, 10.0 Hz, H-9a), 3.91 (1H, *dd*, *J* = 3.0, 10.0 Hz, H-9b), 3.96 (1H, *d*, *J* = 10.5, H-7), 3.83 (3H, *s*, 3′-OCH_3), 3.82 (3H, *s*, 3-OCH_3), 3.79 (1H, *dd*, *J* = 4.0, 11.0 Hz, H-9′a), 3.73 (1H, *dd*, *J* = 6.0, 11.0 Hz, H-9′b), 2.85 (1H, *m*, H-7′), 2.07 (1H, *m*, H-8′), 1.94 (1H, *tt*, *J* = 3.0, 10.0 Hz, H-8). ^{13}C NMR (125 Hz, CD_3OD) data (Table S2).

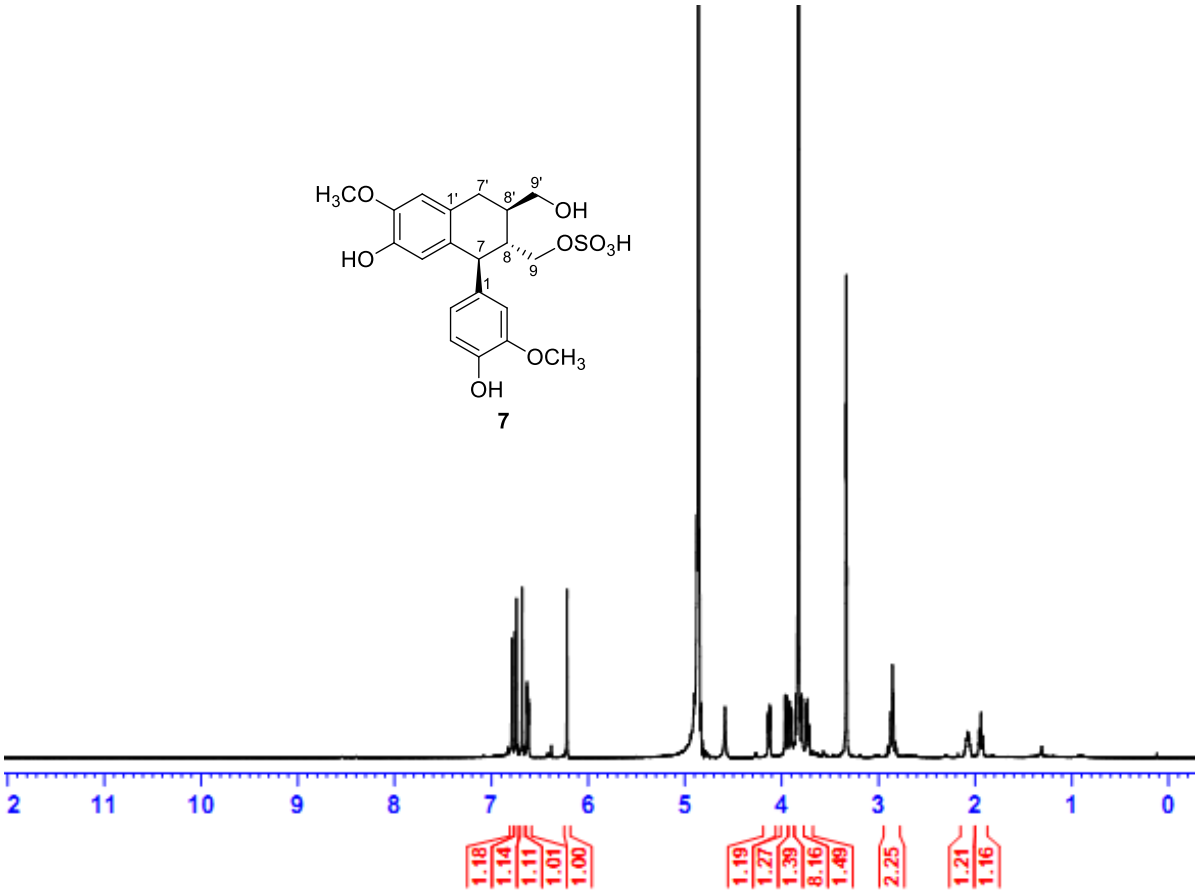


Figure S44: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of (+)-isolarisiresinol-9-*O*-sulfate (**7**)

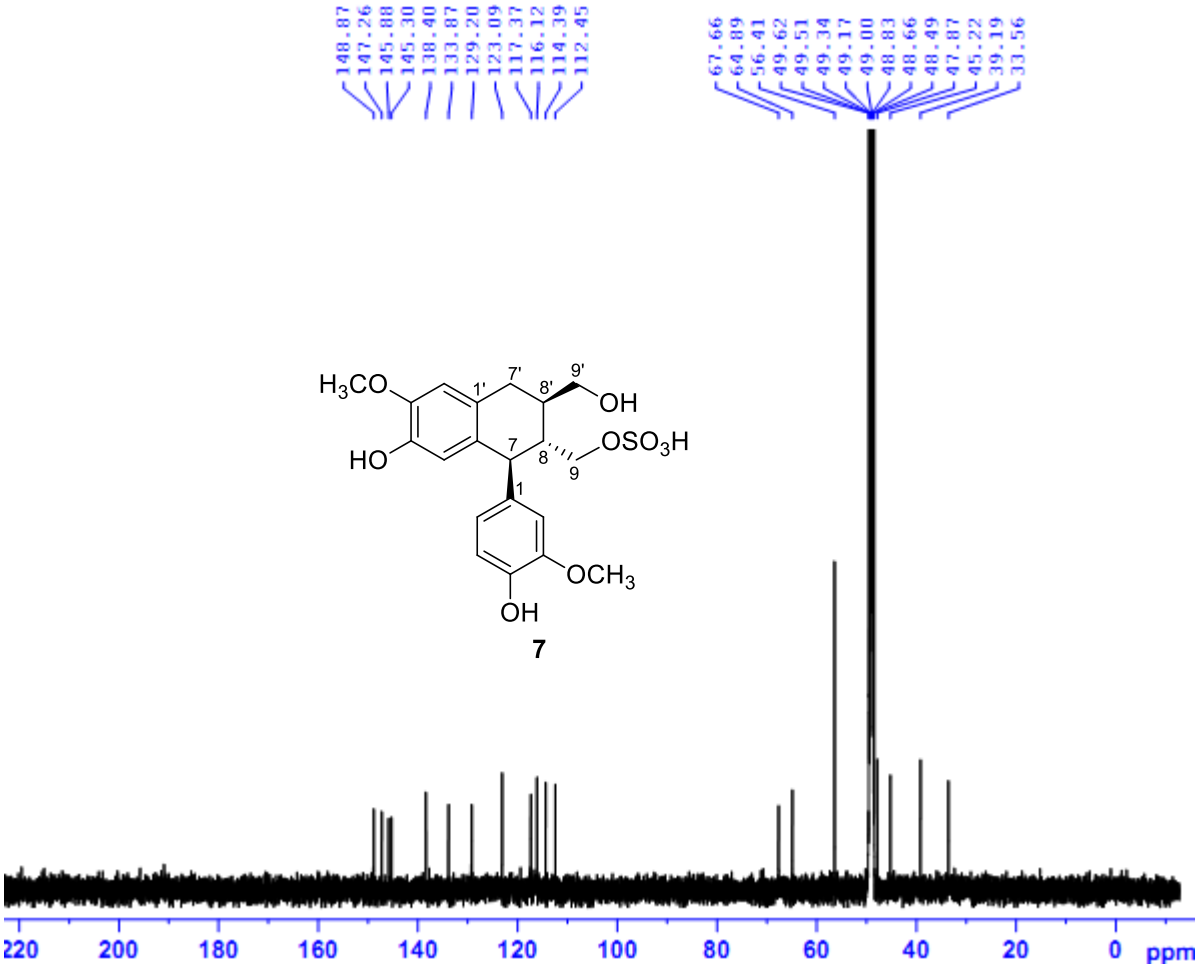


Figure S45: ^{13}C-NMR (125 MHz, CD_3OD) Spectrum of (+)-isolarisiresinol-9-*O*-sulfate (**7**)

Isotachioside (8): ^{1}H NMR (500 MHz, CD_3OD) δ_H 7.03 (1H, *d*, *J* = 8.5 Hz, H-6), 6.49 (1H, *d*, *J* = 2.5 Hz, H-3), 6.32 (1H, *dd*, *J* = 2.5, 8.5 Hz, H-5), 4.71 (1H, *d*, *J* = 8.5 Hz, H-1′), 3.88 (1H, *dd*, *J* = 2.5, 12.0 Hz, H-6′a), 3.83 (3H, *s*, 2-OCH_3), 3.71 (1H, *dd*, *J* = 5.5, 12.0 Hz, H-6′b), 3.35-3.45 (4H, *m*, H-2′, H-3′, H-4′, H-5′). ^{13}C NMR (125 MHz, CD_3OD) δ_c 154.9 (C-4), 152.0 (C-2), 141.0 (C-1), 120.5 (C-6), 107.6 (C-5), 104.3 (C-1′), 101.8 (C-3), 78.1 (C-3′), 77.8 (C-5′), 75.0 (C-2′), 71.4 (C-4′), 62.5 (C-6′), 56.5 (2-OCH_3).

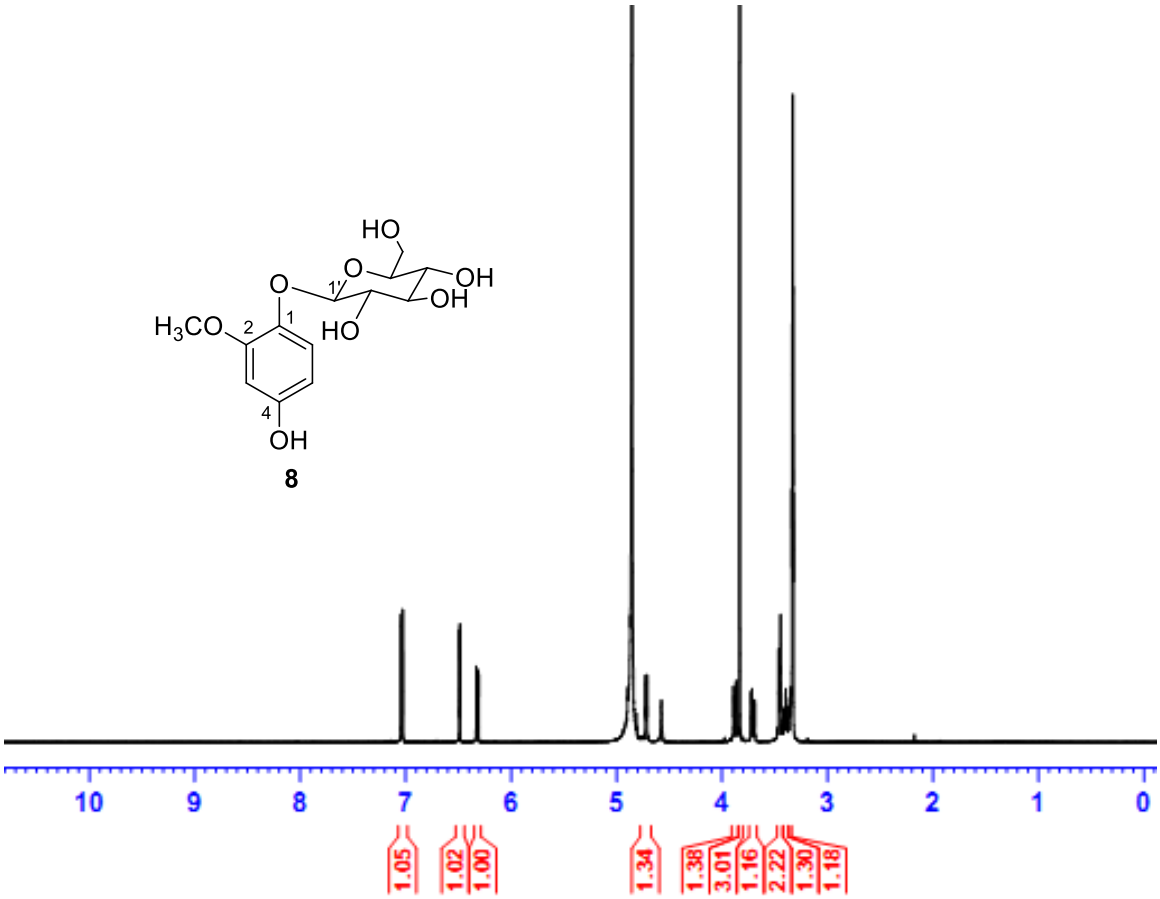


Figure S46: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of isotachioside (**8**)

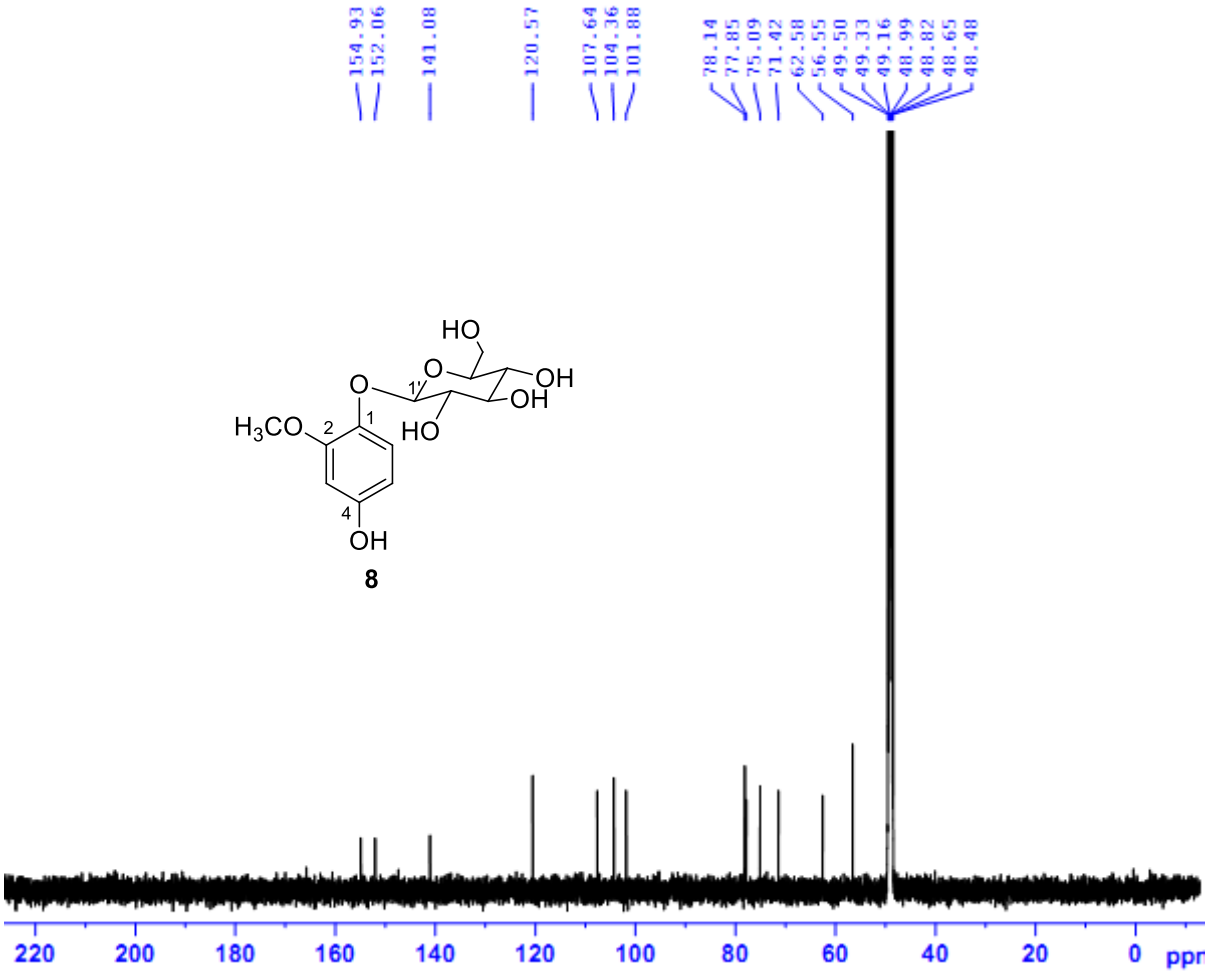


Figure S47: ^{13}C-NMR (125 MHz, CD_3OD) Spectrum of isotachioside (**8**)

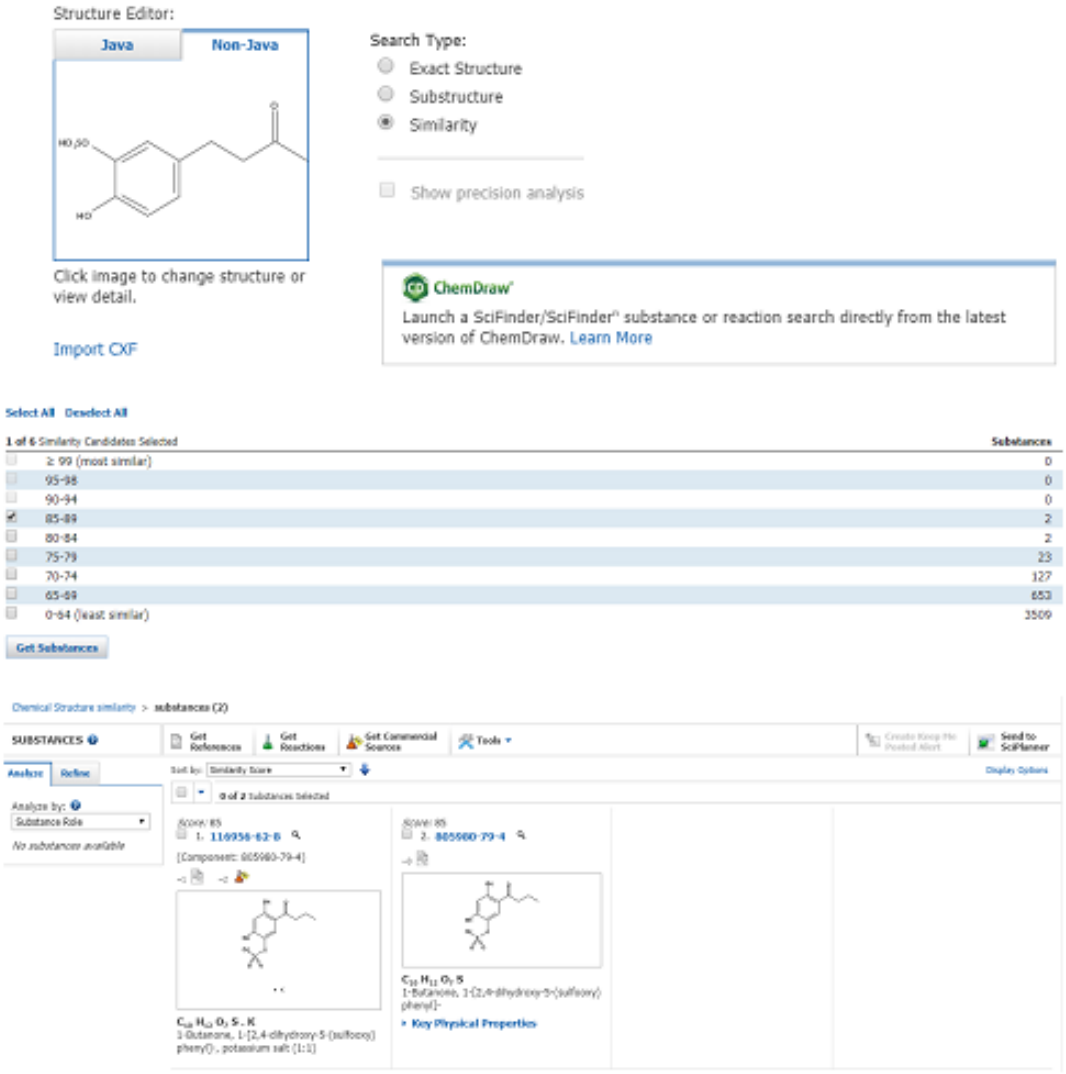


Figure S48: SciFinder Search Results of **1**

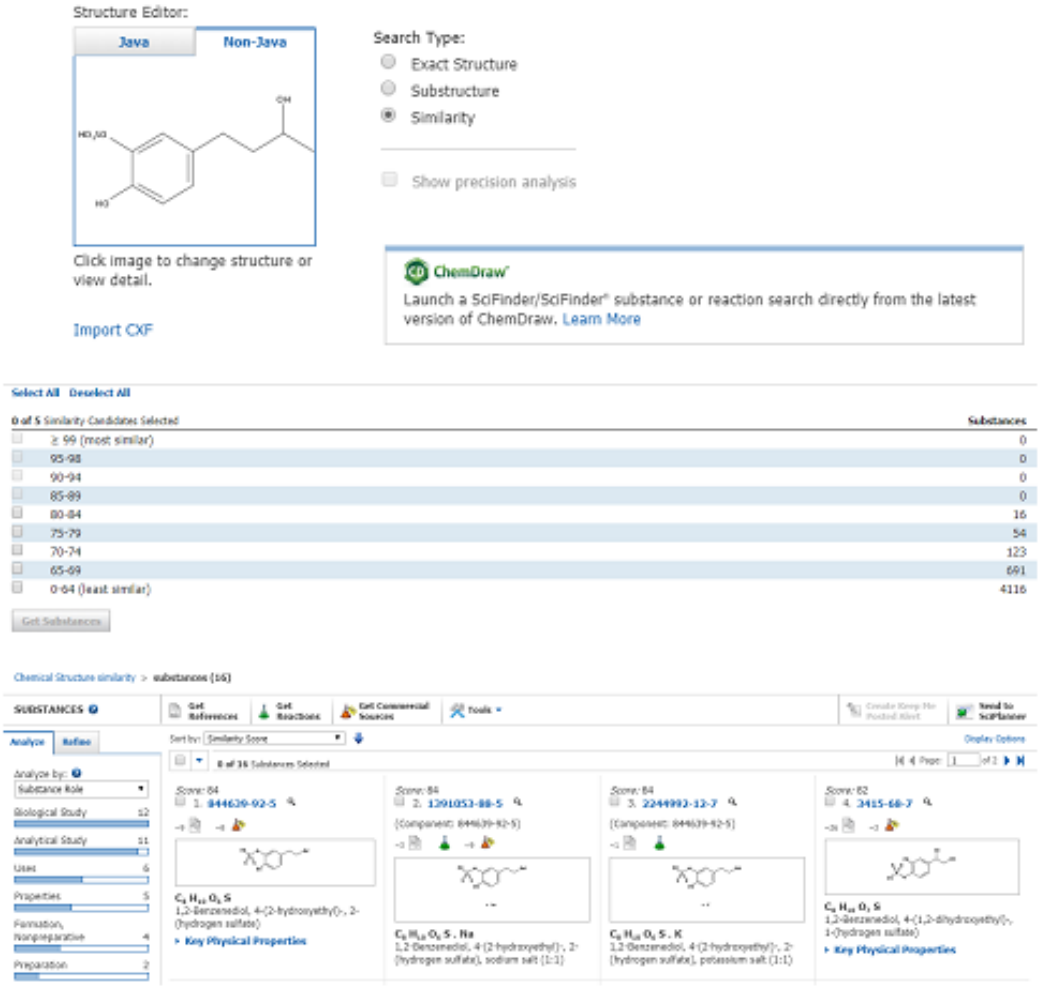


Figure S49: SciFinder Search Results of **2**

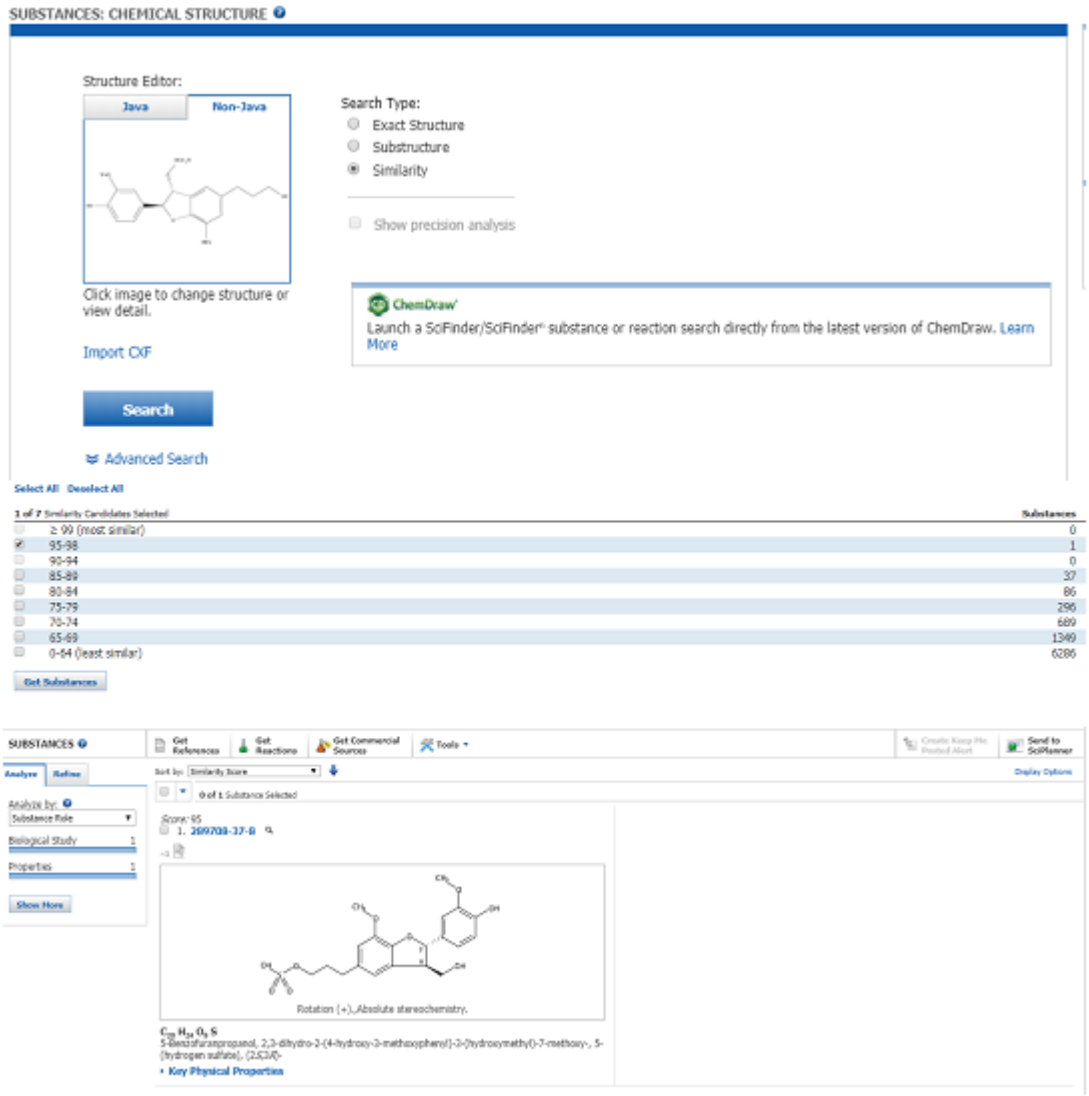


Figure S50: SciFinder Search Results of **3**

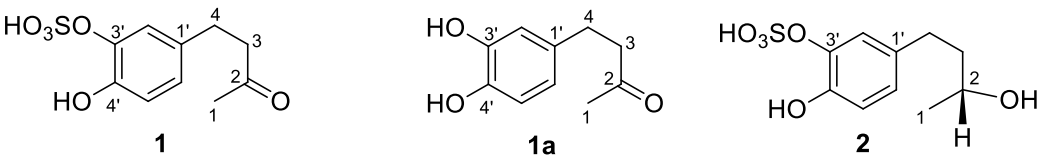


Table S1 : ^{13}C NMR (125 MHz) data for compounds **1**, **1a** and **2** in DMSO-d_6

Position	**1**	**1a**	**2**
1	29.1	30.3	23.5
2	207.7	208.4	65.2
3	44.3	45.1	41.0
4	28.1	28.9	30.6
1′	132.1	132.3	133.4
2′	122.8	115.9	122.8
3′	140.5	145.4	140.5
4′	147.2	143.7	146.9
5′	117.0	116.1	116.9
6′	124.5	119.1	124.5

1a: 4-(3′,4′-dihydroxyphenyl)-2-butanone [T. T. Le, J. Yin, M. W. Lee (2017). Anti-inflammatory and anti-Oxidative activities of phenolic compounds from *Alnus sibirica* stems fermented by *Lactobacillus plantarum* subsp. *argentoratensis*, *Molecules* **22**, 1566-1574.]

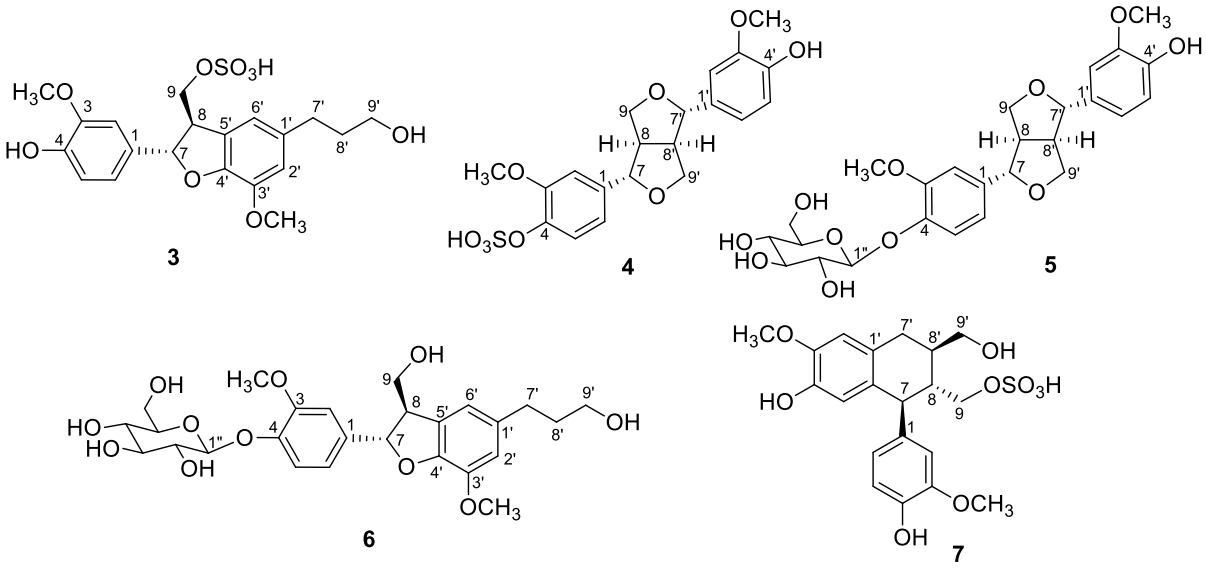
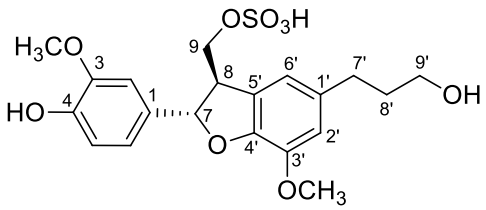


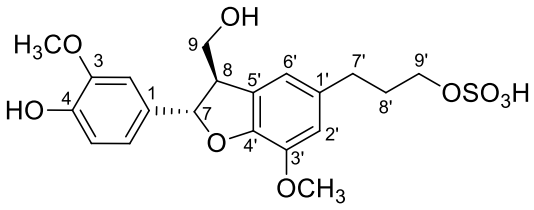
Table S2 : ^{13}C NMR (125 MHz) data for compounds **3-7**

Position	**3**[a]	**6**[a]	**4**[b]	**5**[a]	**7**[a]
1	134.4	138.5	136.5	137.5	138.4
2	110.6	111.2	110.5	117.5	114.4
3	149.0	151.0	150.4	151.1	148.8
4	147.3	147.6	141.9	147.5	145.2
5	116.1	118.1	120.7	118.1	116.1
6	119.4	119.4	117.5	119.8	123.1
7	88.8	88.4	84.9	87.5	47.8
8	52.8	55.6	53.7	55.5	45.2
9	70.0	65.1	71.0	72.7	67.6
1′	137.1	137.1	132.1	133.7	129.2
2′	114.4	114.2	110.4	111.0	112.4
3′	145.2	145.2	147.5	148.0	147.2
4′	147.6	147.5	145.8	147.5	145.9
5′	128.8	129.6	115.1	116.1	117.4
6′	118.1	117.9	118.6	120.0	133.8
7′	32.8	32.9	85.2	87.1	33.5
8′	35.7	35.8	53.5	55.3	39.2
9′	62.2	62.2	70.9	72.6	64.9
OCH_3	56.8	56.8	55.6	56.7	56.4
OCH_3	56.4	56.7	55.5	56.4	56.4
1″		102.8		102.9	
2″		74.9		74.9	
3″		78.2		78.2	
4″		71.3		71.3	
5″		77.8		77.8	
6″		62.5		62.5	

[a] Recorded in CD_3OD, [b] in DMSO-d_6



3



3a

Table S3. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) data for compounds **3** and **3a** in CD_3OD

Position	**3**		**3a**	
	δ_C	δ_H (*J* in Hz)	δ_C	δ_H (*J* in Hz)
1	134.4 (C)	-	135.0	-
2	110.6 (CH)	7.03 (1H, *d*, *J* = 2.0)	110.7	6.95 (1H, *d*, *J* = 2.0)
3	149.0 (C)	-	149.2	-
4	147.3 (C)	-	147.8	-
5	116.1 (CH)	6.77 (1H, *d*, *J* = 8.0)	116.2	6.77 (1H, *d*, *J* = 8.0)
6	119.4 (CH)	6.89 (1H, *dd*, *J* = 2.0, 8.0)	119.8	6.83 (1H, dd, *J* = 2.0, 8.0)
7	88.8 (CH)	5.60 (1H, *d*, *J* = 6.0)	89.1	5.50 (1H, *d*, *J* = 6.0)
8	52.8 (CH)	3.70 (1H, *m*)	55.5	3.84 (1H, m)
9	70.0 (CH_2)	4.30 (1H, *dd*, *J* = 6.0, 10.5)	65.0	3.47 (1H, *dd*, *J* = 6.0, 11.0)
		4.19 (1H, *dd*, *J* = 8.0, 10.5)		3.76 (1H, *dd*, *J* = 7.0, 11.0)
1′	137.1 (C)	-	136.5	-
2′	114.4 (CH)	6.75 (1H, *s*)	114.9	6.68 (1H, *s*)
3′	145.2 (C)	-	145.3	-
4′	147.6 (C)	-	147.5	-
5′	128.8 (C)	-	129.9	-
6′	118.1 (CH)	6.79 (1H, *s*)	118.2	6.68 (1H, *s*)
7′	32.8 (CH_2)	2.64 (2H, *t*, *J* = 7.5)	32.7	2.68 (2H, *t*, *J* = 7.0)
8′	35.7 (CH_2)	1.84 (2H, *m*)	32.8	1.94 (2H, m)
9′	62.2 (CH_2)	3.58 (2H, *t*, *J* = 6.5)	68.2	4.01 (2H, *t*, *J* = 6.0)
3-OCH_3	56.4 (CH_3)	3.85 (3H, *s*)	56.5	3.83 (3H, *s*)
3′-OCH_3	56.8 (CH_3)	3.88 (3H, *s*)	36.9	3.87 (3H, *s*)

3a: Dihydrodehydrodiconiferyl alcohol 9′-*O*-sulfate [H. Otsuka, E. Hirata, T. Shinato, Y. Takeda (2000). Isolation of lignan glucosides and neolignan sulfate from the leaves of *Glochidion zeylanicum* (Gaertn) A. Juss, *Chem. Pharm. Bull.* **48**, 1084-1086].