Supplementary Materials

Bioactive abietane-type diterpenoid glycosides from leaves of *Clerodendrum infortunatum* (Lamiaceae)

Md. Josim Uddin[1,2], Daniela Russo[3,4], Md. Anwarul Haque[5,6], Serhat Sezai Çiçek[1], Frank D. Sönnichsen[7], Luigi Milella[3], and Christian Zidorn[1,*]

1 Pharmazeutisches Institut, Abteilung Pharmazeutische Biologie, Christian-Albrechts-Universität zu Kiel, Gutenbergstrasse 76, 24118 Kiel, Germany; juddin@pharmazie.uni-kiel.de; scicek@pharmazie.uni-kiel.de

2 Department of Pharmacy, International Islamic University Chittagong, Chittagong-4318, Bangladesh

3 Department of Science, University of Basilicata, Viale dell' Ateneo Lucano 10, 85100 Potenza, Italy; daiela.russo@unibas.it; luigi.milella@unibas.it

4 Spinoff BioActiPlant s.r.l., Viale dell' Ateneo Lucano 10, 85100 Potenza, Italy

5 Department of Experimental Pathology, Graduate School of Comprehensive Human Sciences, University of Tsukuba, Ibaraki 305-8575, Japan; a.haque5314@gmail.com

6 Department of Pharmacy, University of Rajshahi, Rajshahi-6205, Bangladesh

7 Otto Diels Institute for Organic Chemistry, University of Kiel, Otto-Hahn-Platz 4, 24118 Kiel, Germany; fsoennichsen@oc.uni-kiel.de

Corresponding Author

*Christian Zidorn: Pharmazeutisches Institut, Abteilung Pharmazeutische Biologie,

Christian-Albrechts-Universität zu Kiel, Gutenbergstraße 76, 24118 Kiel, Germany. Tel.:

+49-431-880-1139. E-mail: czidorn@pharmazie.uni-kiel.de

List of contents

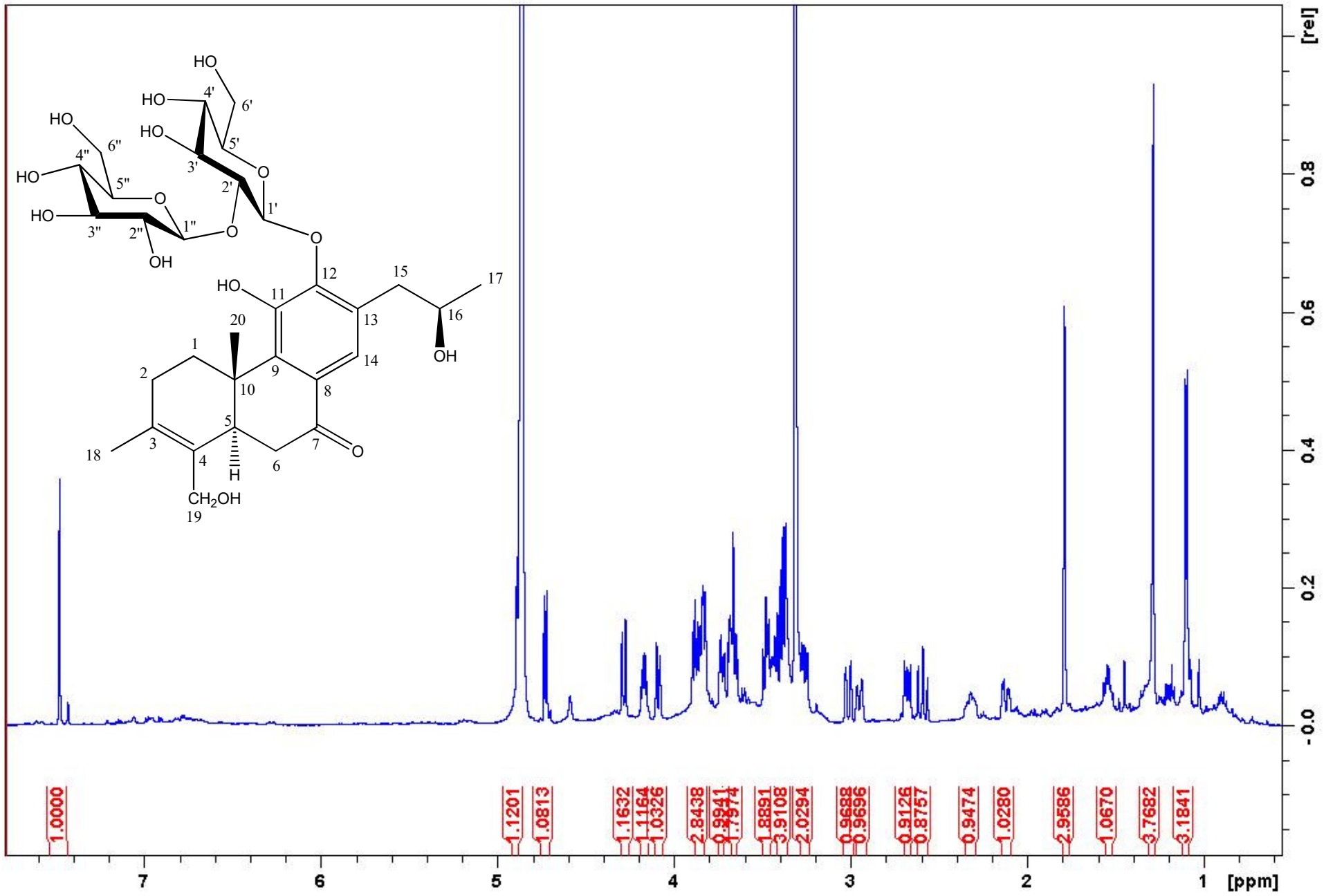


Figure S1-A. ^{1}H NMR spectrum of compound **1** in Methanol-d_4.

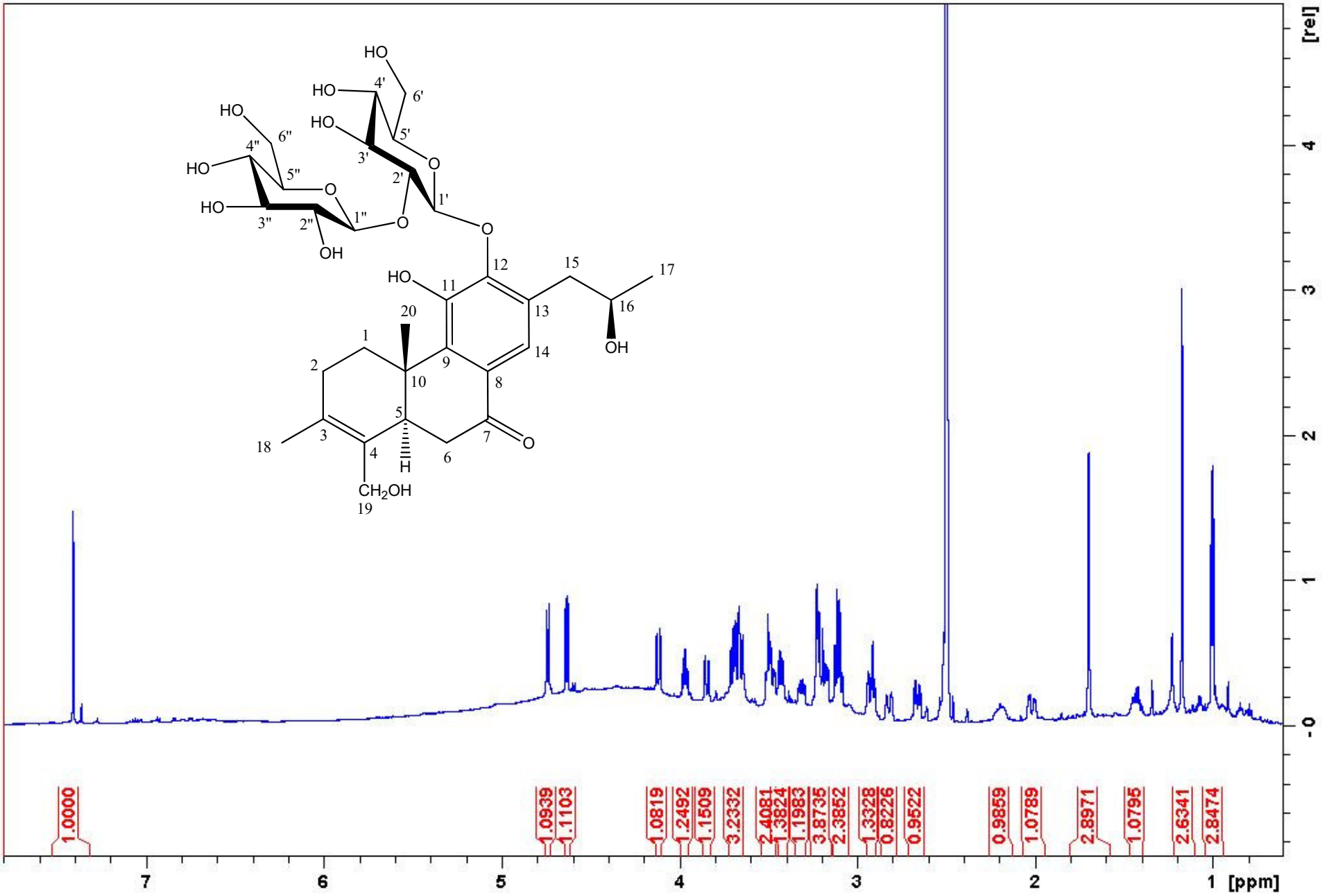


Figure S1-B. ^{1}H NMR spectrum of compound **1** in DMSO-d_6

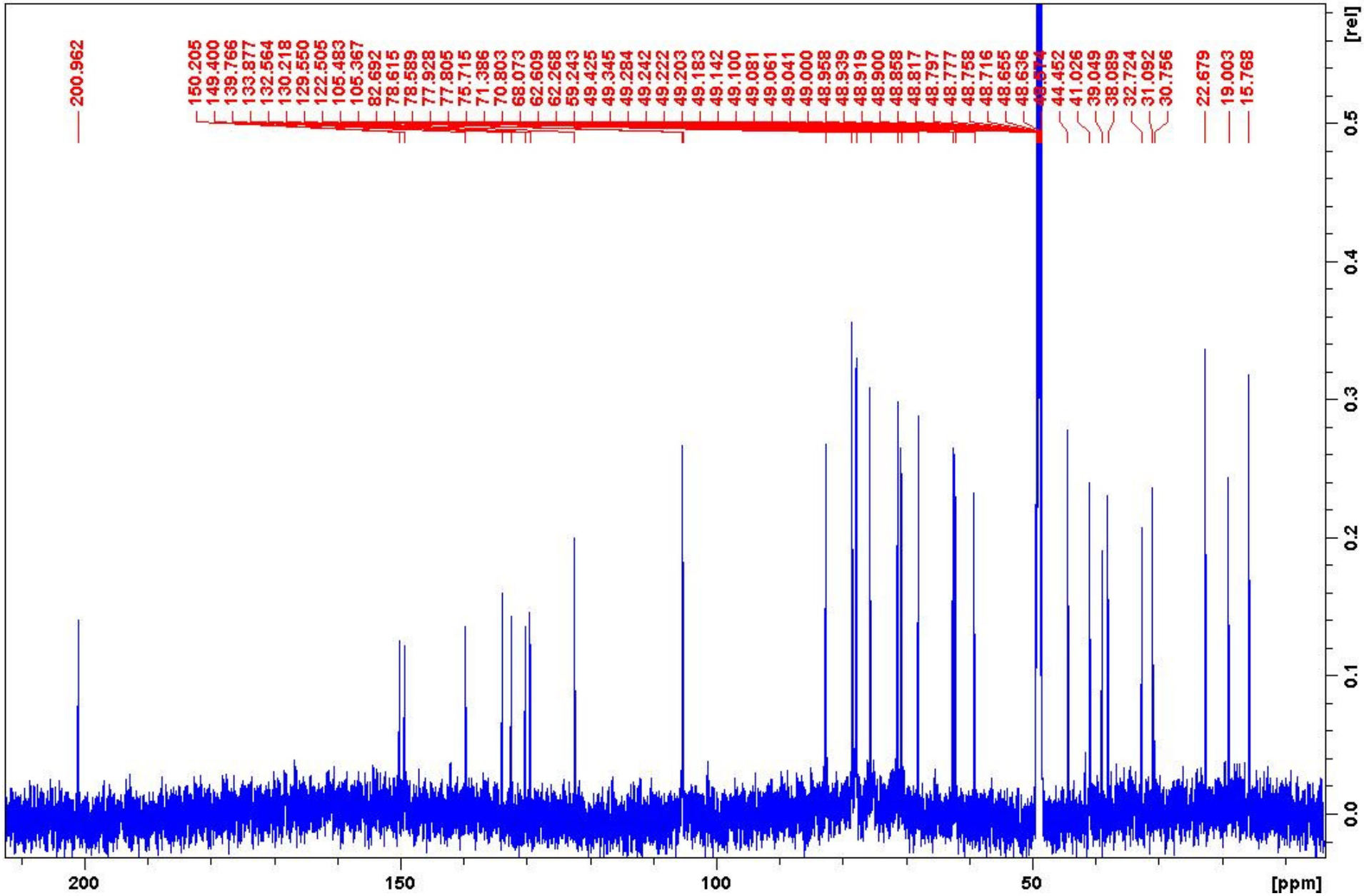


Figure S2-A. ^{13}C NMR spectrum of compound **1** in Methanol-d_4.

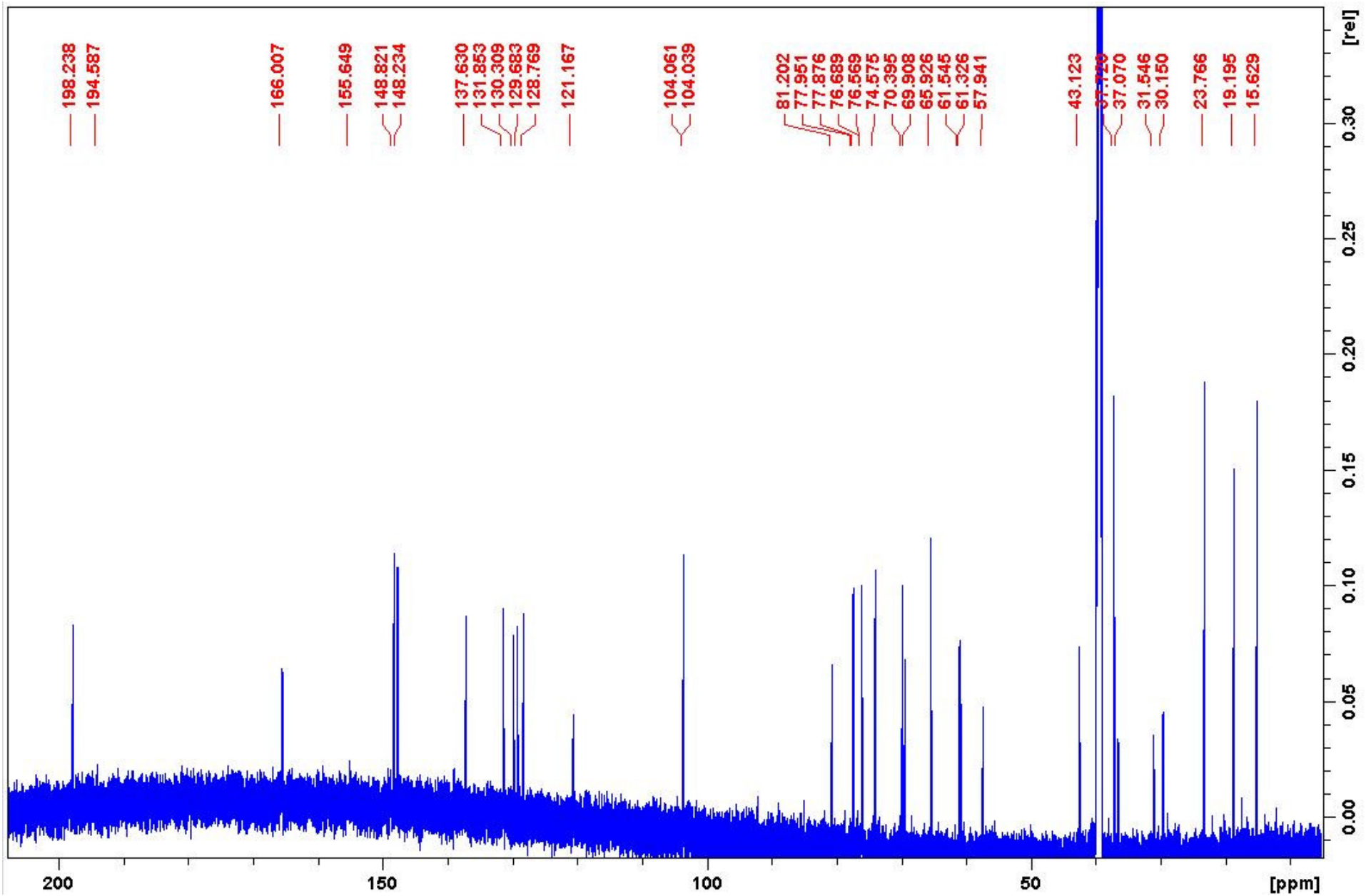


Figure S2-B. ^{13}C NMR spectrum of compound **1** in DMSO-d_6.

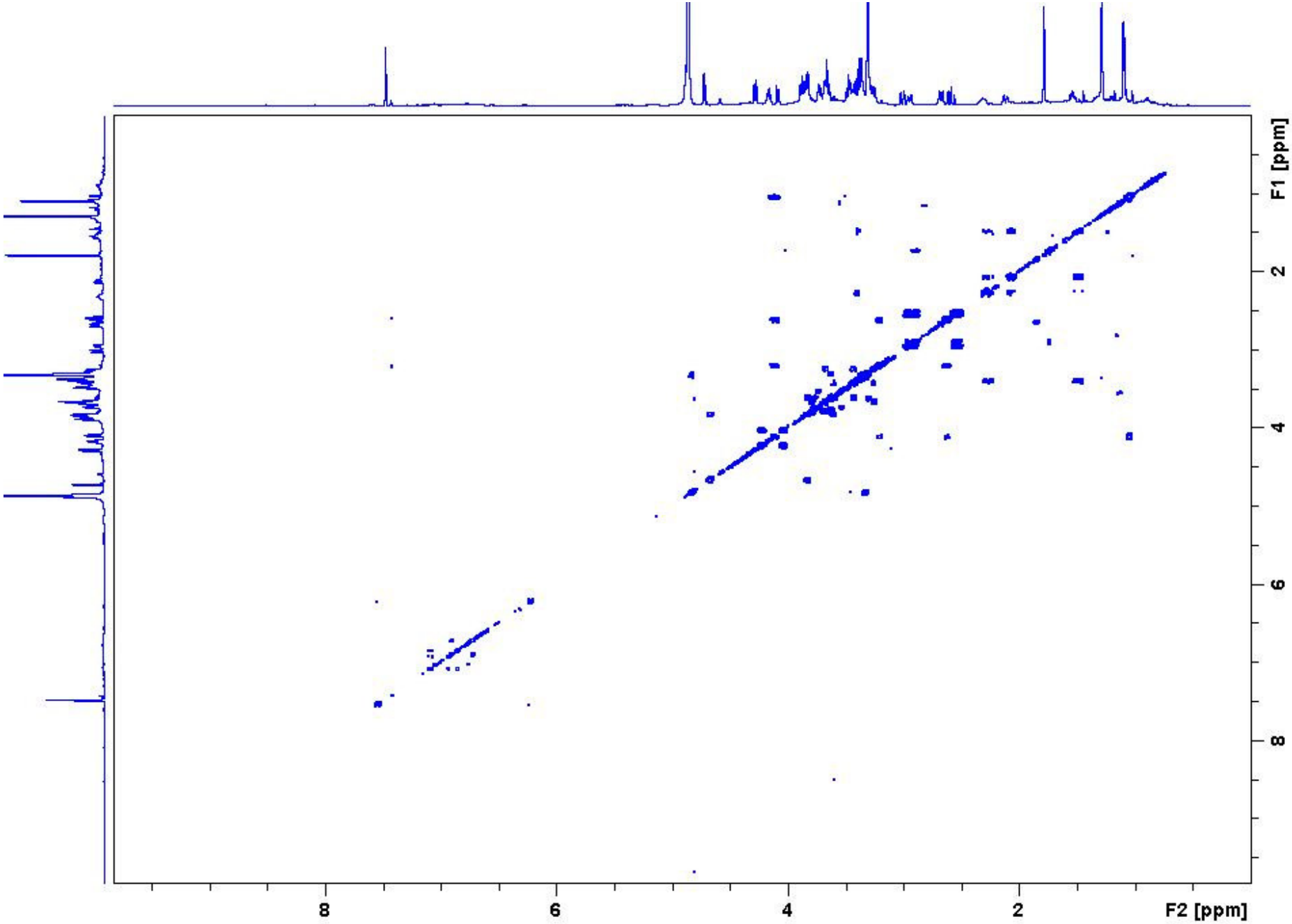


Figure S3-A. COSY spectrum of compound **1** in Methanol-d_4.

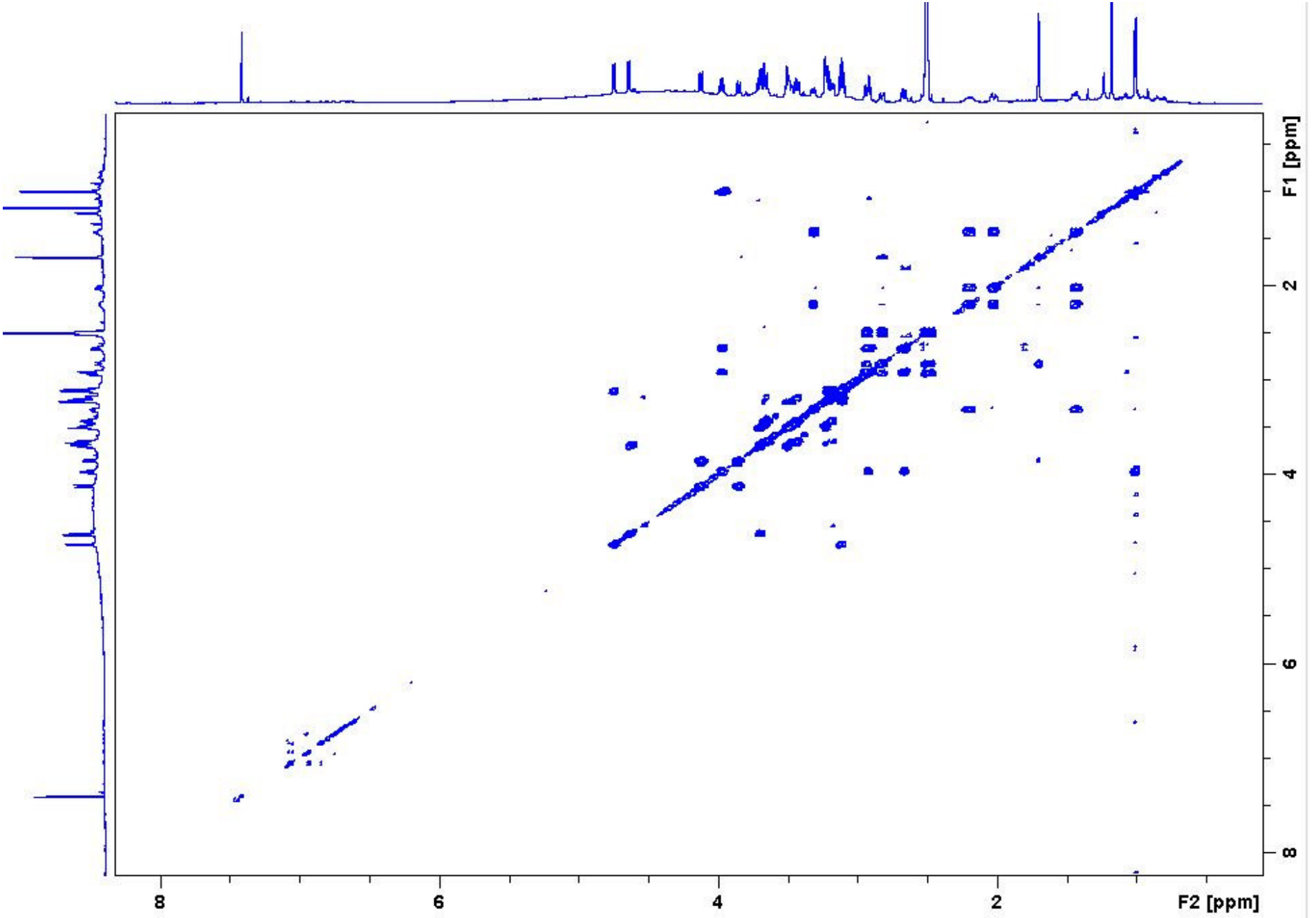


Figure S3-B. COSY spectrum of compound **1** in DMSO-d_6.

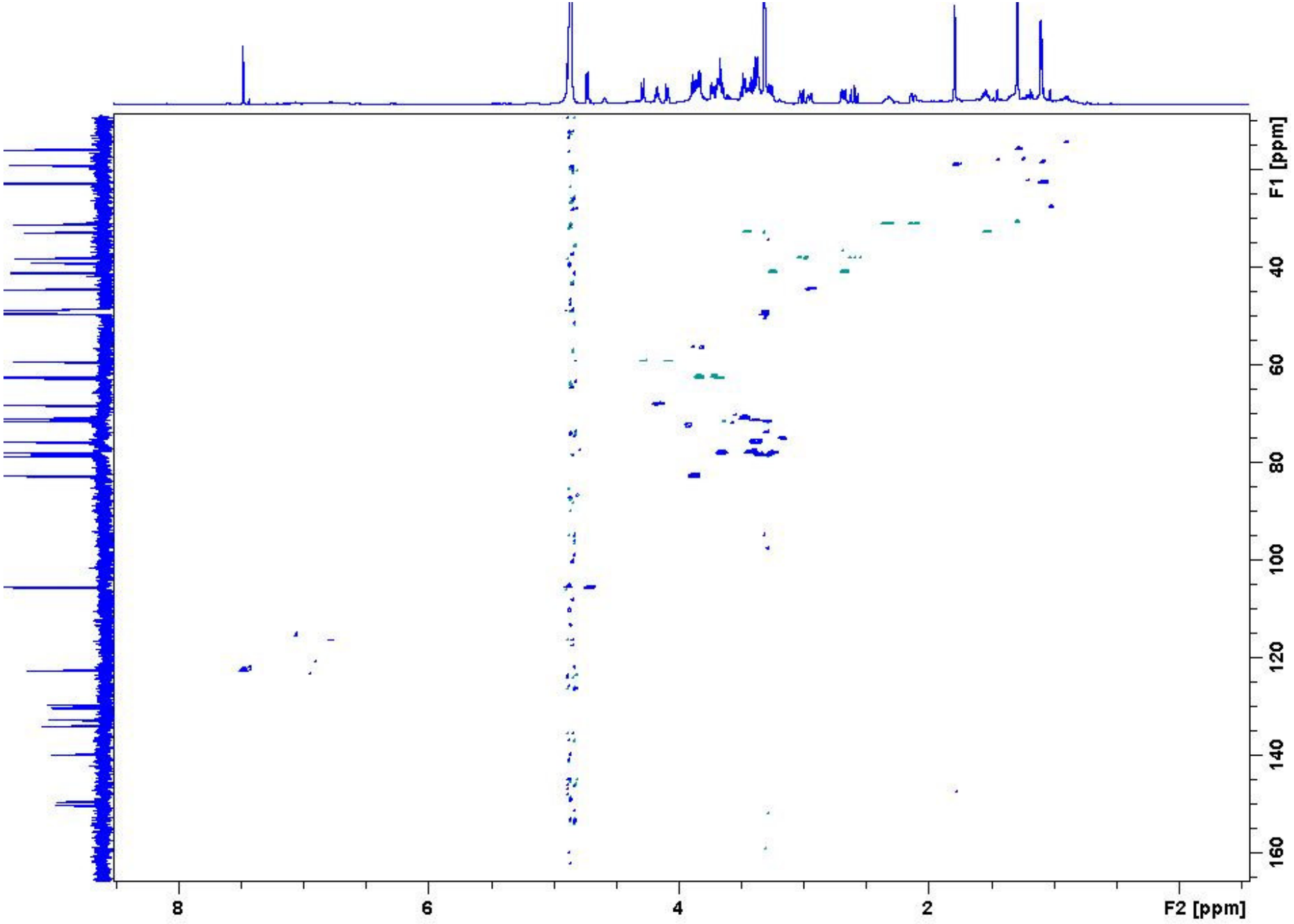


Figure S4-A. HSQC spectrum of compound **1** in Methanol-d_4.

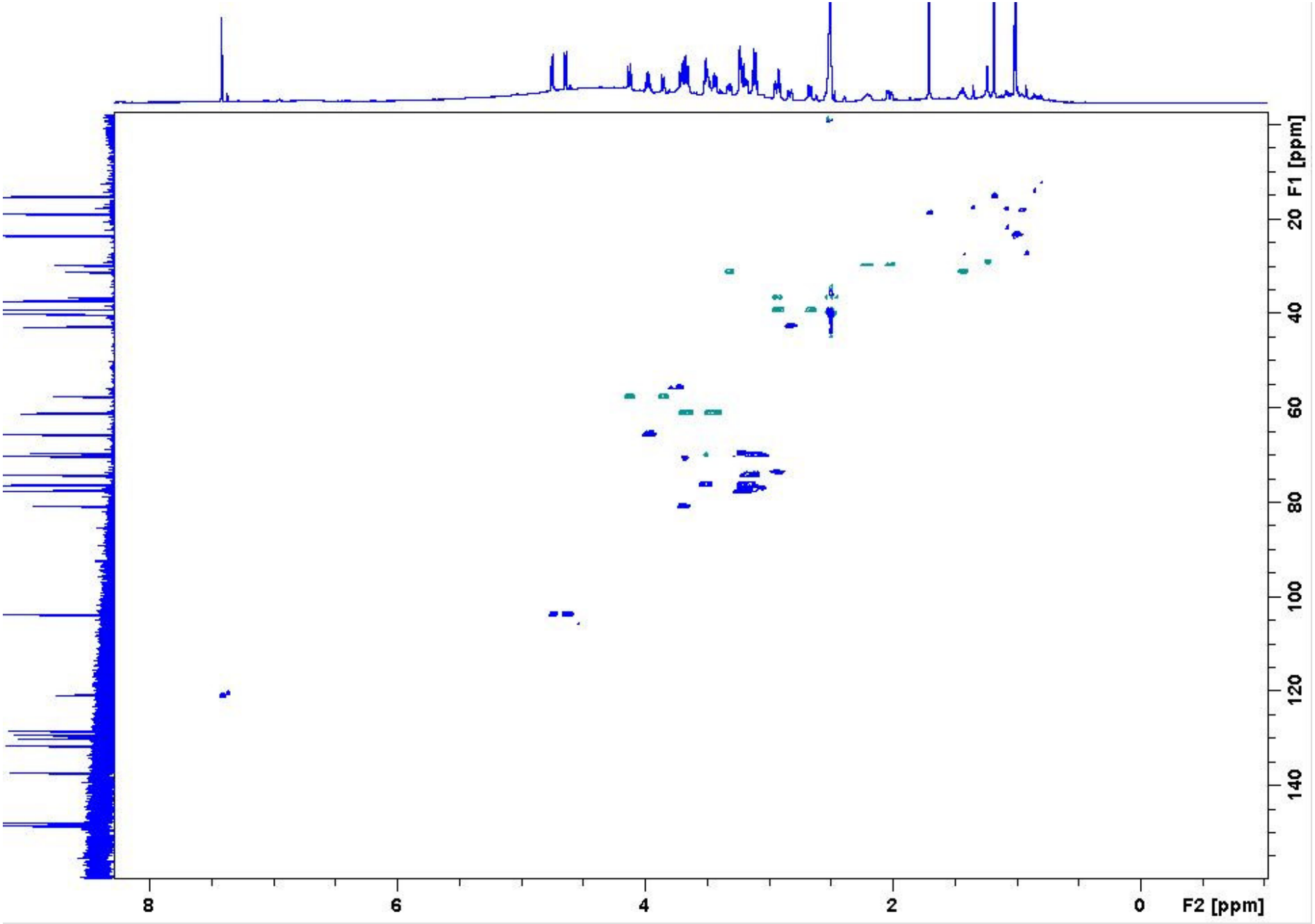


Figure S4-B. HSQC spectrum of compound **1** in DMSO-d_6.

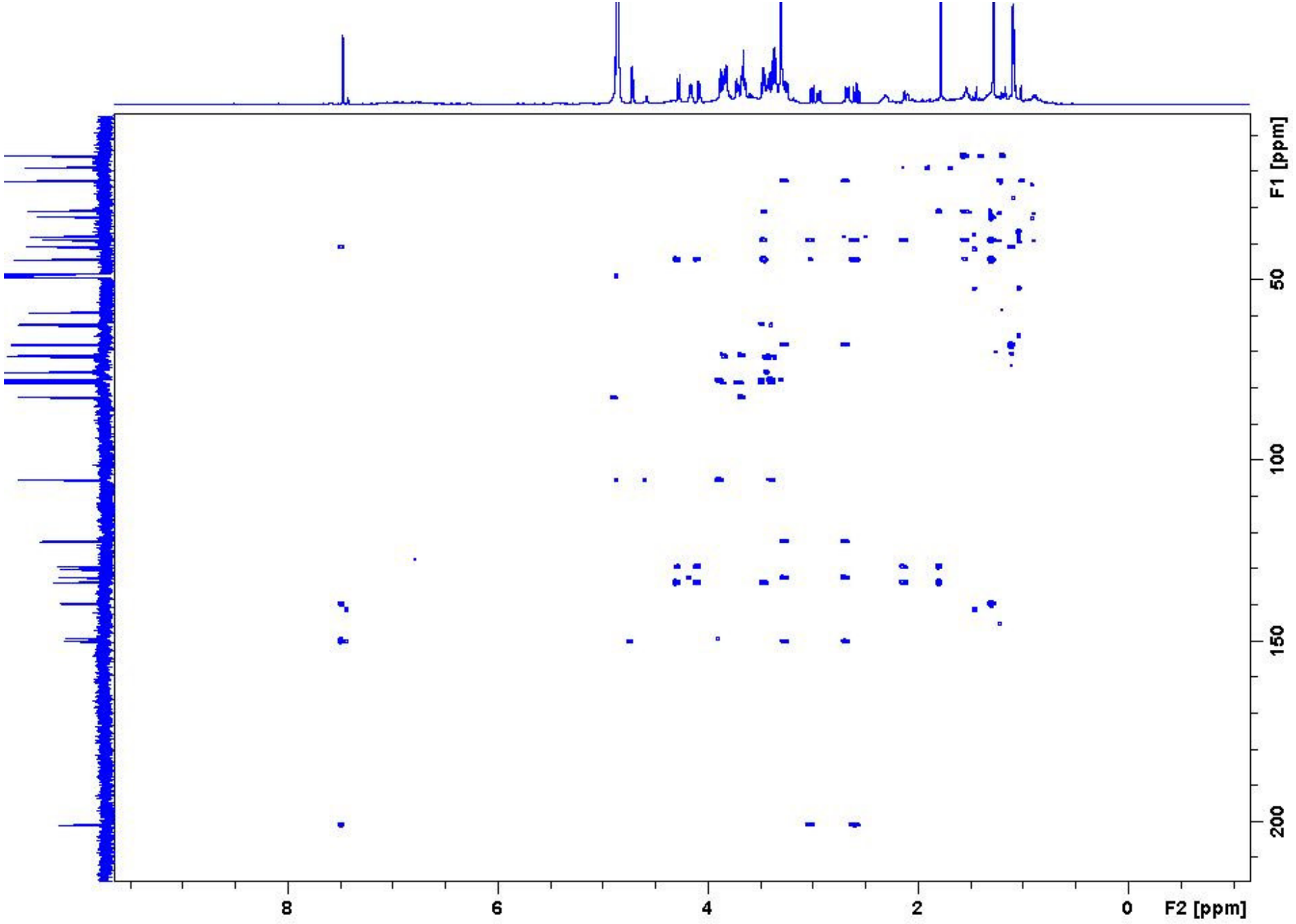


Figure S5-A. HMBC spectrum of compound **1** in Methanol-d_4.

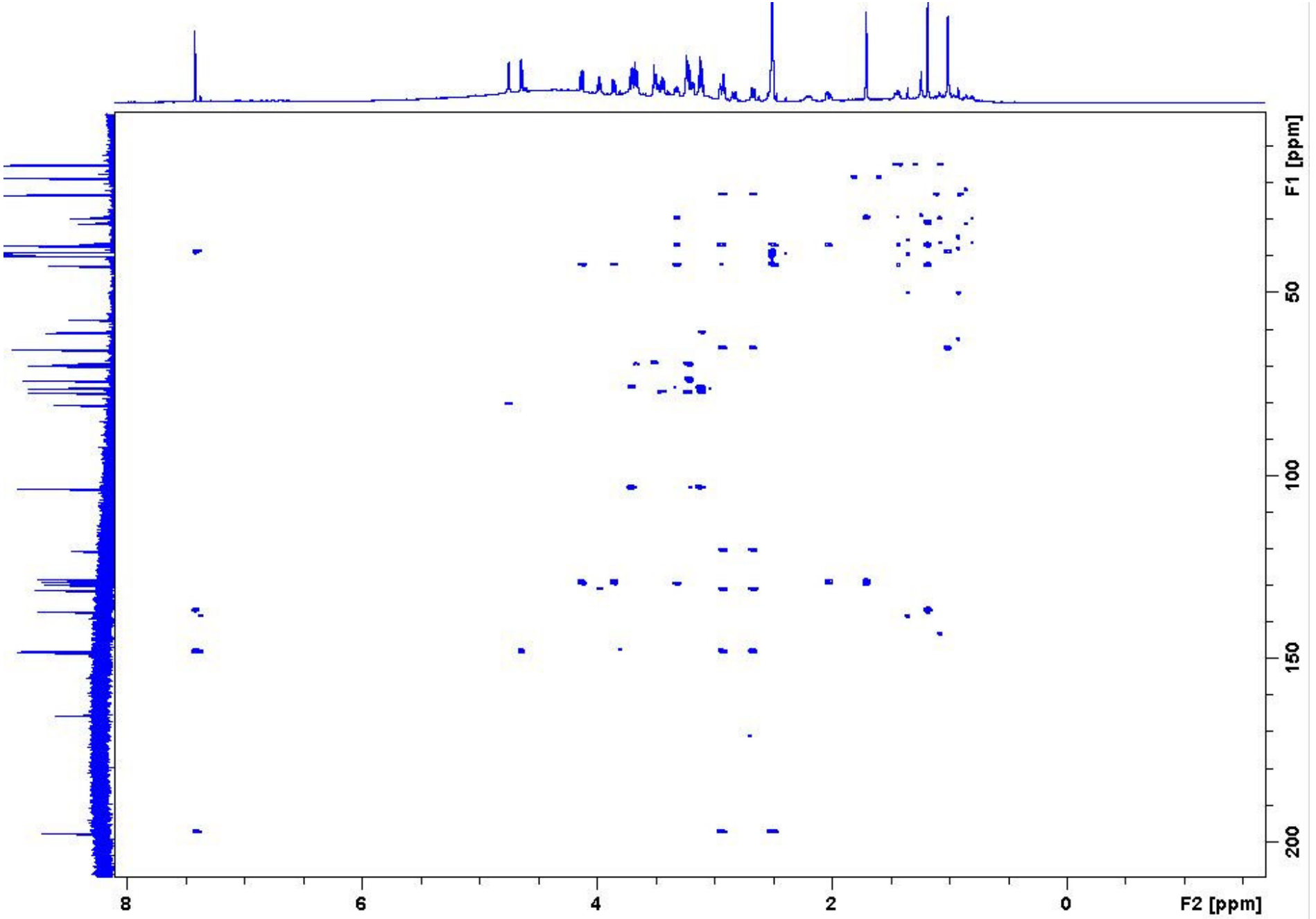
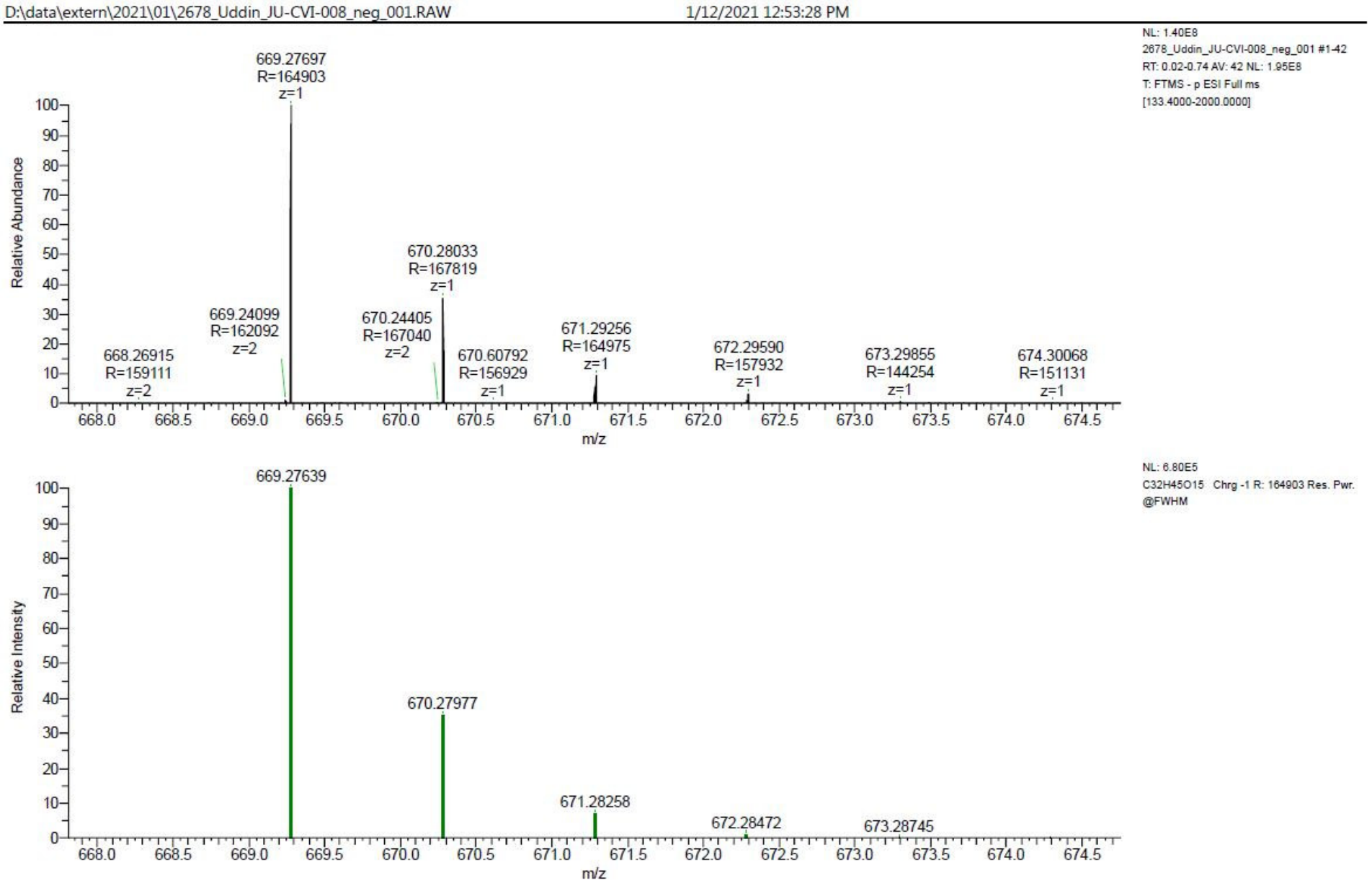


Figure S5-B. HMBC spectrum of compound **1** in DMSO-d_6.



Peak Mass	Display Form...	Combined Fit	RDB	Delta [ppm]	Theo. mass	Rank	Combined Sc...	# Matched Iso.	# Missed Iso.	MS Cov. [%]	Pattern Cov. [...	MSMS Match...
669.27697	$C_{32}H_{45}O_{15}$	94.3841840...	10.50	0.85	669.27639	1	0	5	1	0	0	(Collection)

Figure S6. HR mass spectrum of compound **1** in methanol.

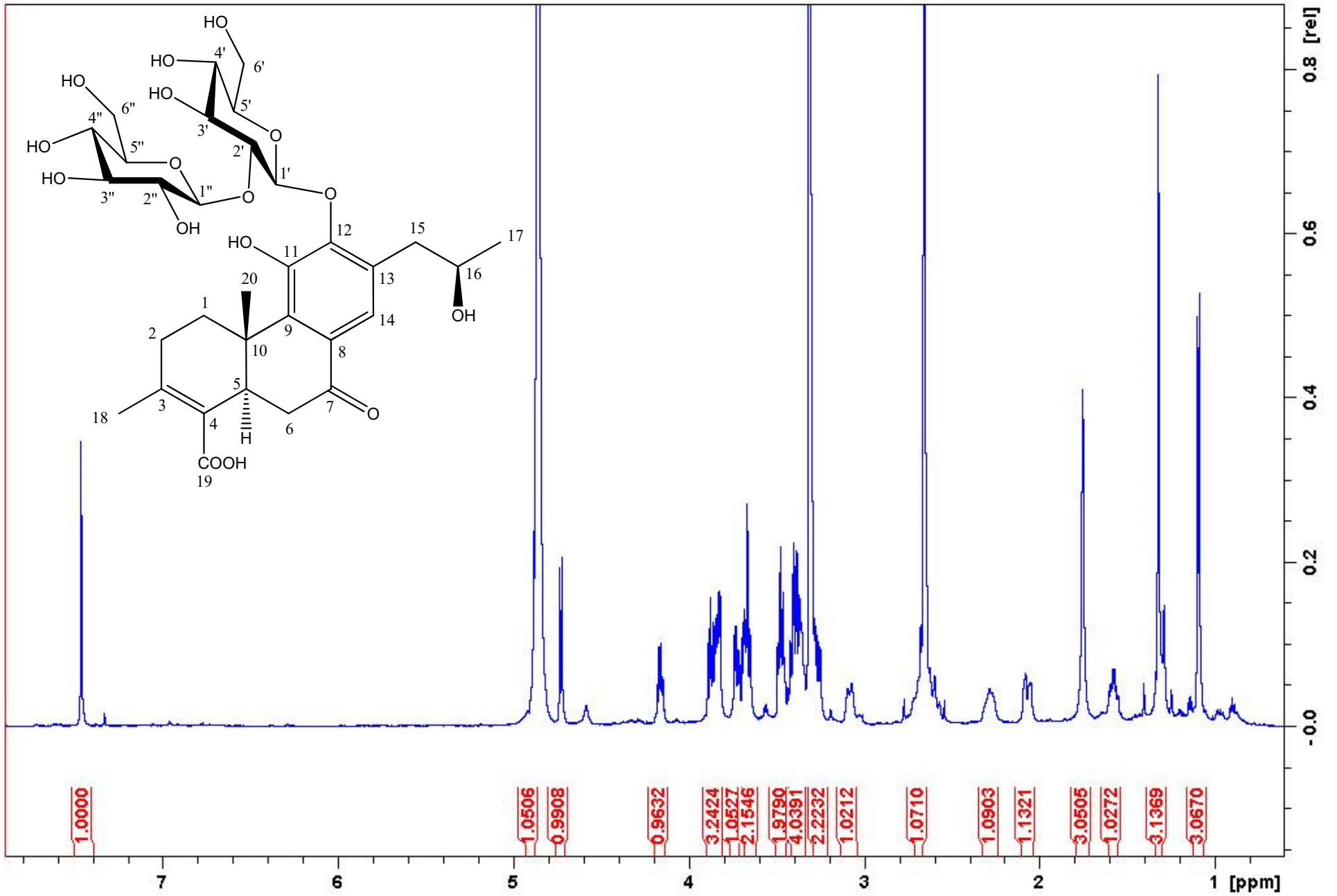


Figure S7-A. ^{1}H NMR spectrum of compound **2** in Methanol-d_4.

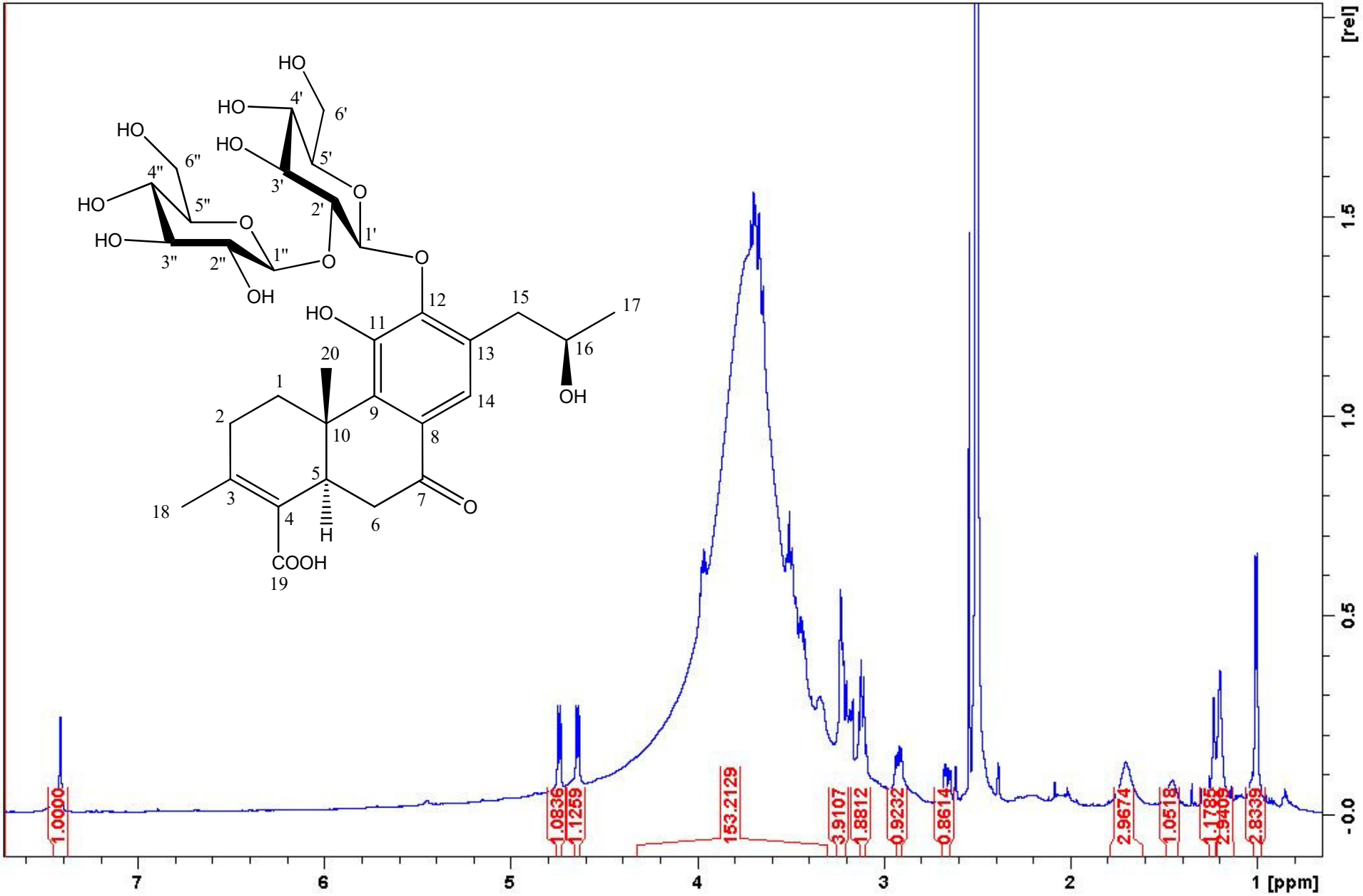


Figure S7-B. ^{1}H NMR spectrum of compound **2** in DMSO-d_6.

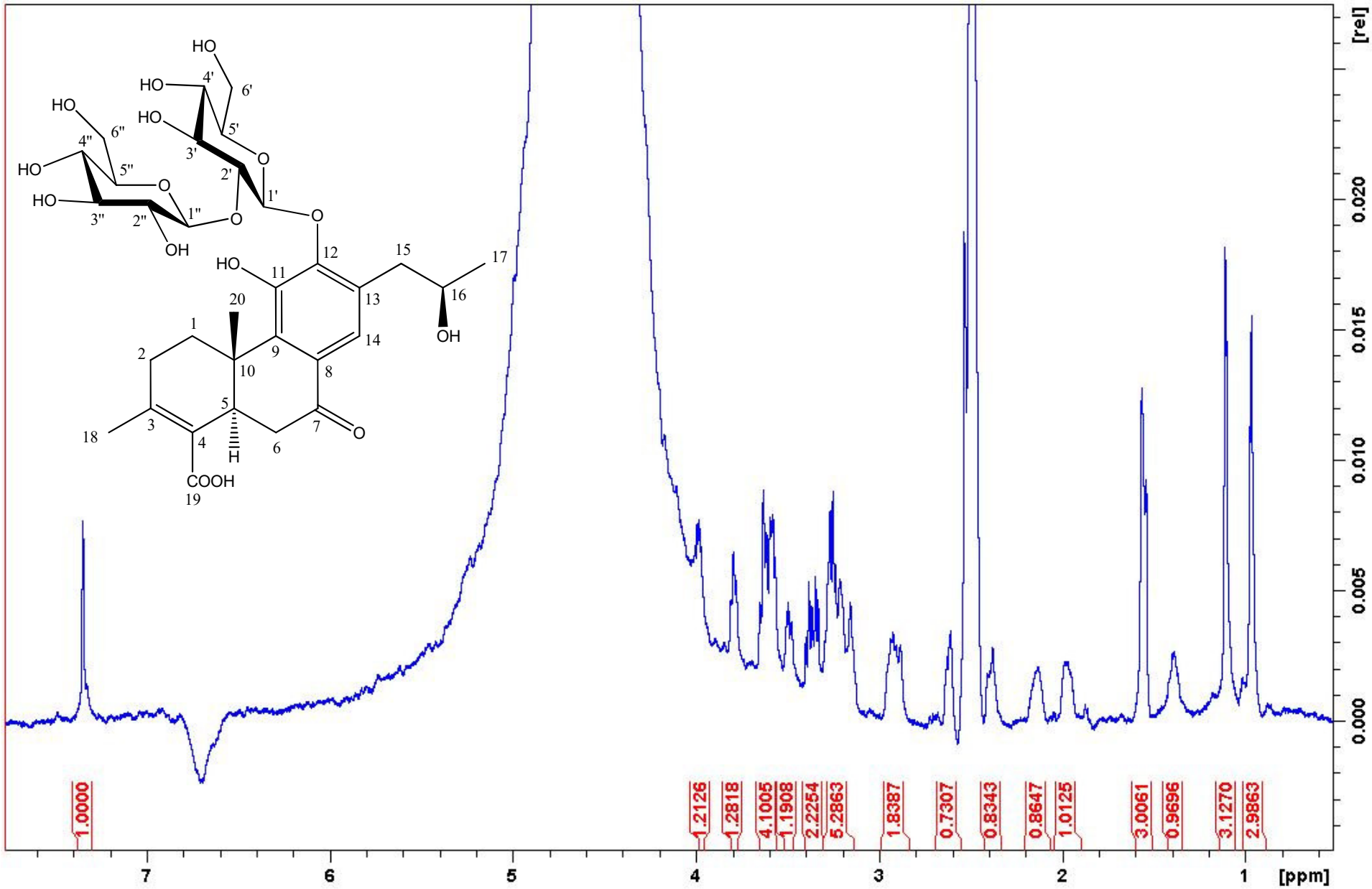


Figure S7-C. ^{1}H NMR spectrum of compound **2** in D_2O.

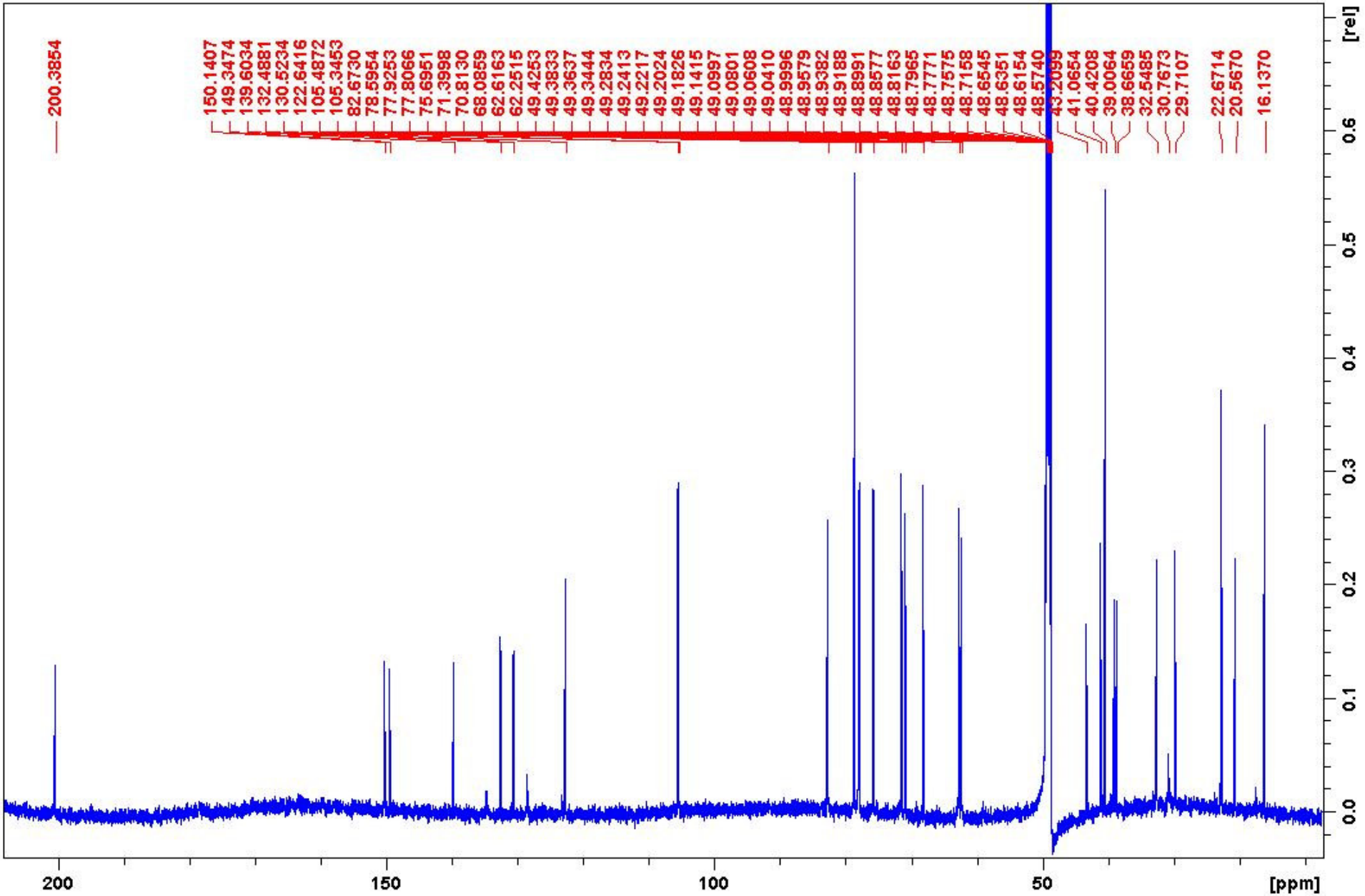


Figure S8-A. ^{13}C NMR spectrum of compound **2** in Methanol-d_4.

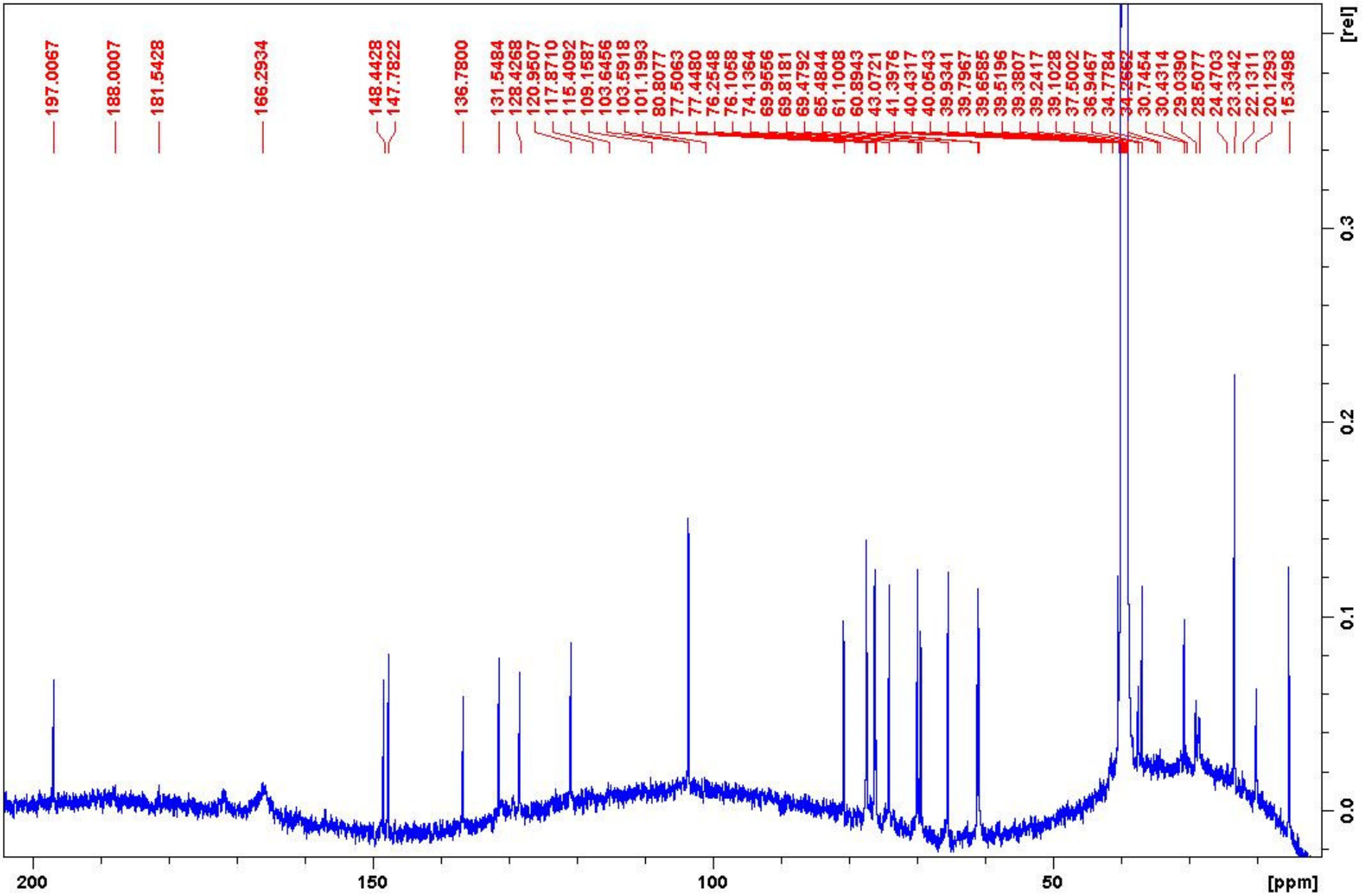


Figure S8-B. ^{13}C NMR spectrum of compound **2** in DMSO-d_6.

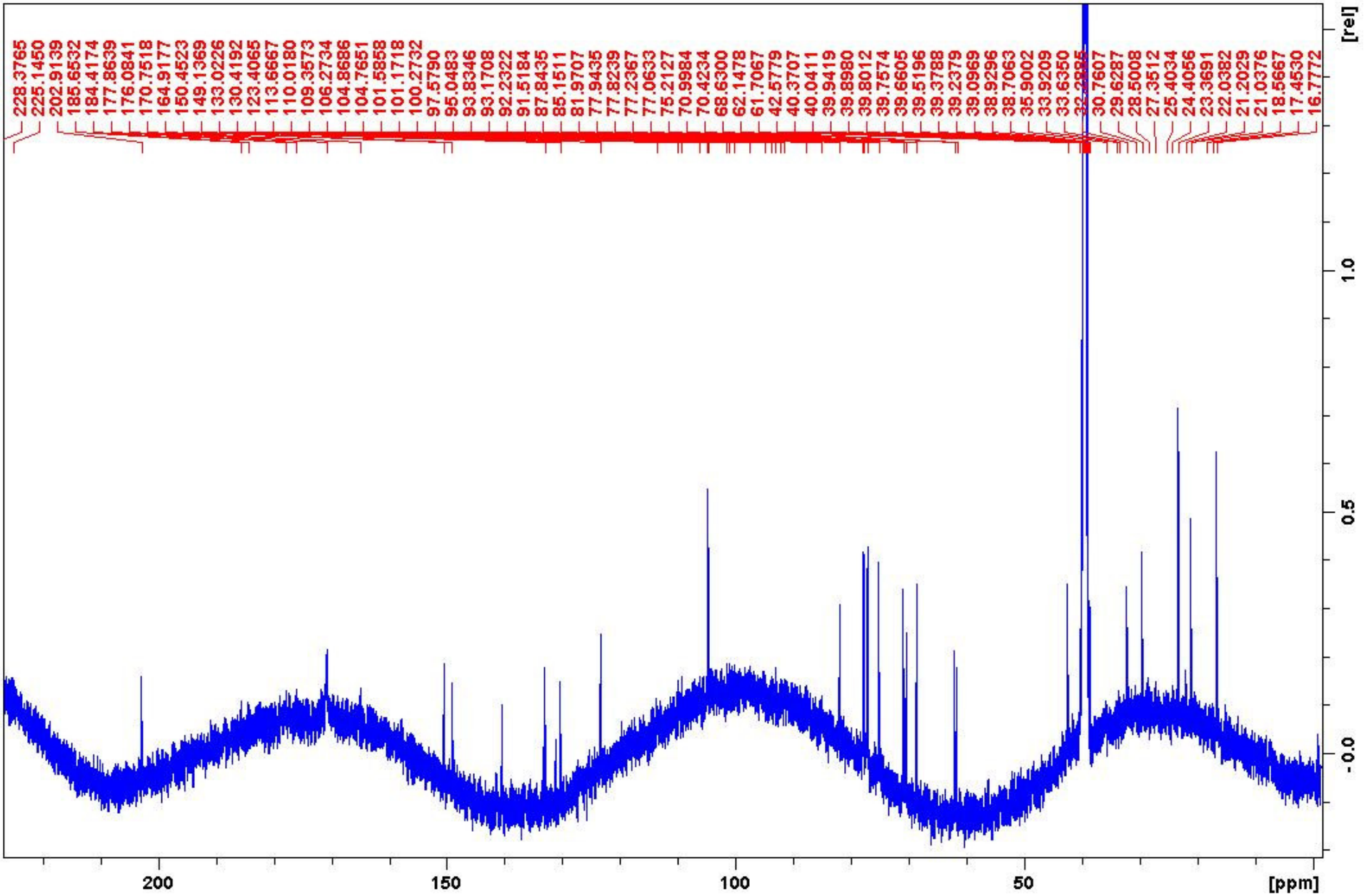


Figure S8-C. ^{13}C NMR spectrum of compound **2** in D_2O.

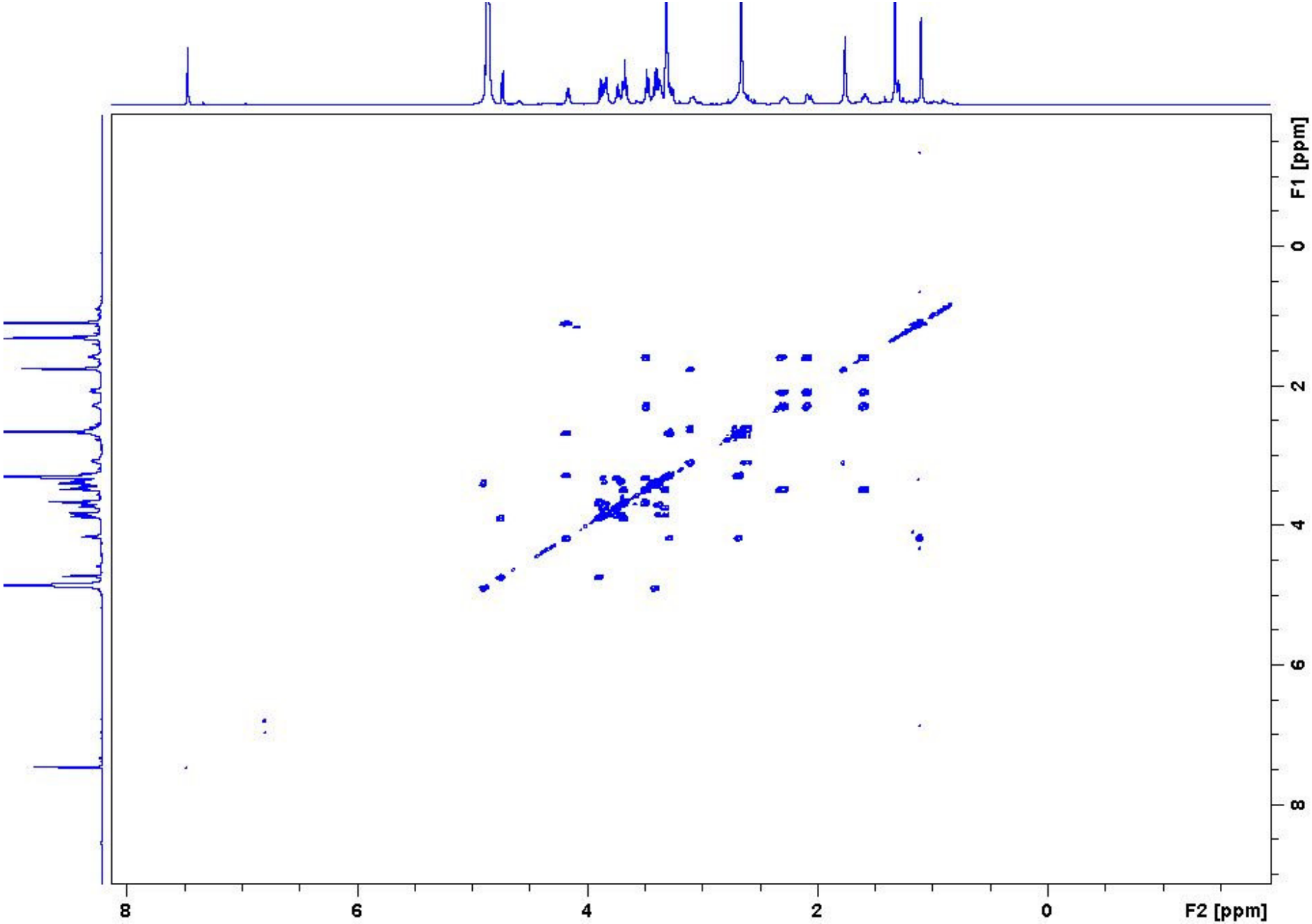


Figure S9-A. COSY spectrum of compound **2** in Methanol-d_4.

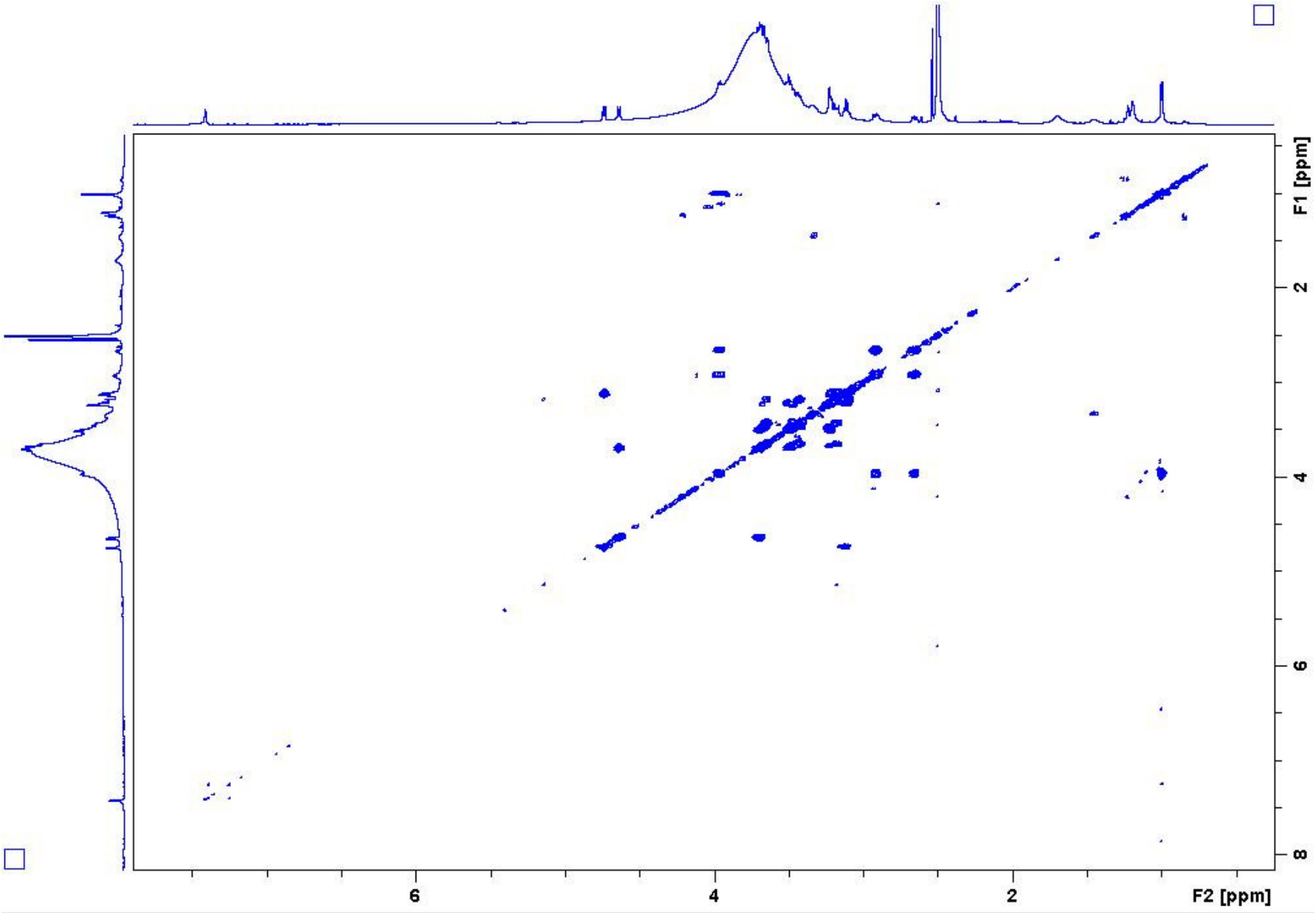


Figure S9-B. COSY spectrum of compound **2** in DMSO-d_6.

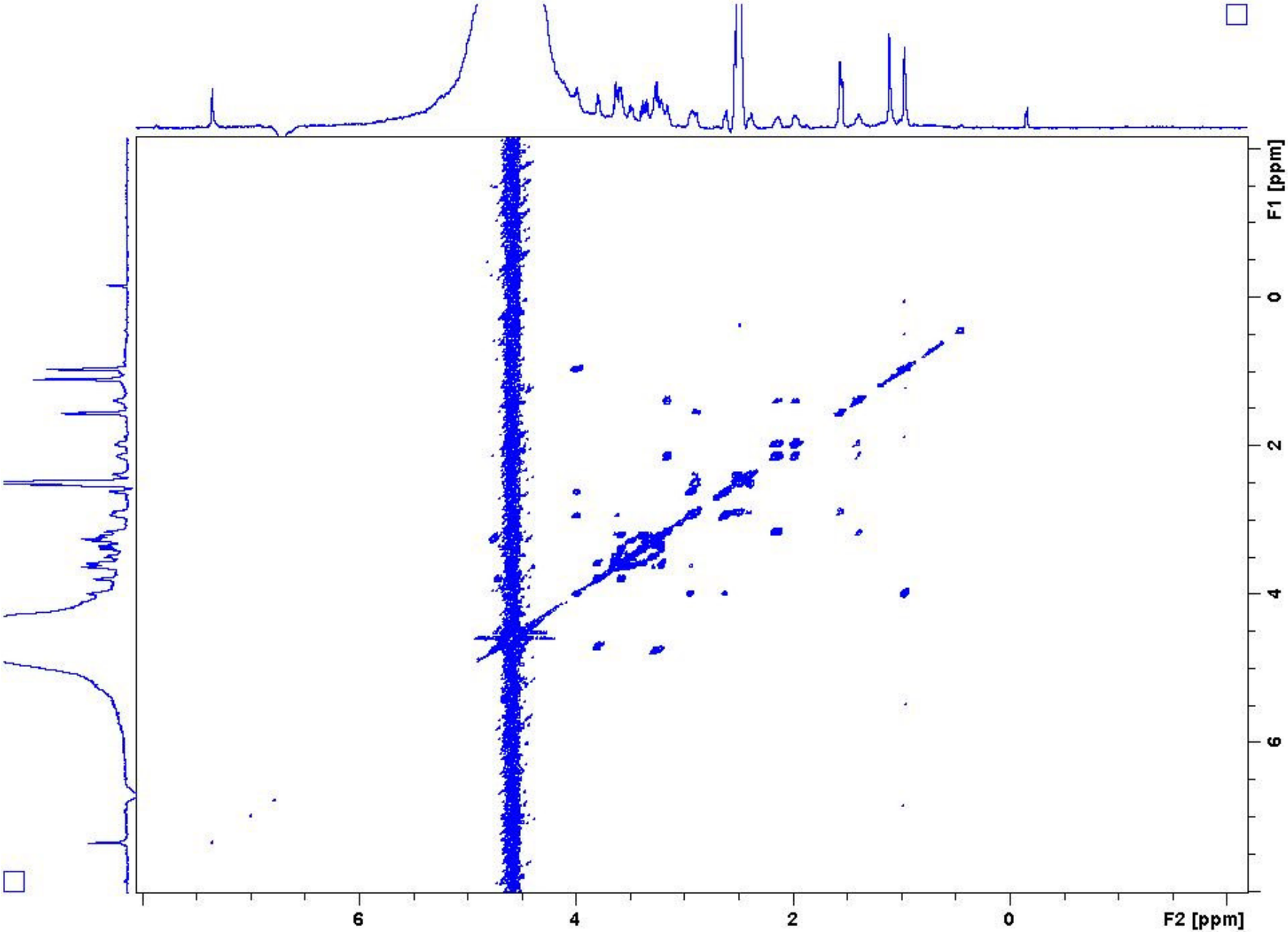


Figure S9-C. COSY spectrum of compound **2** in D_2O.

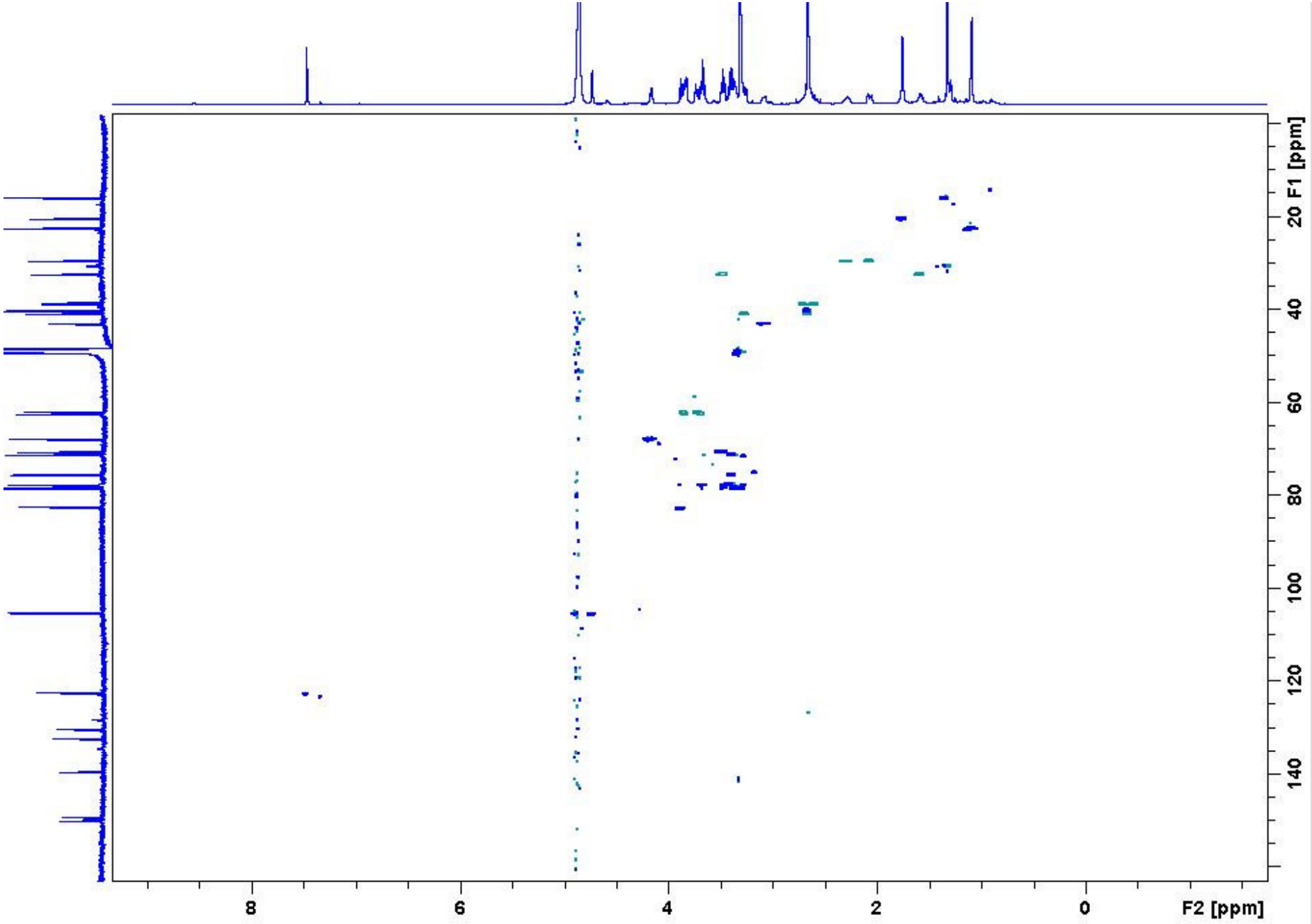


Figure S10-A. HSQC spectrum of compound **2** in Methanol-d_4.

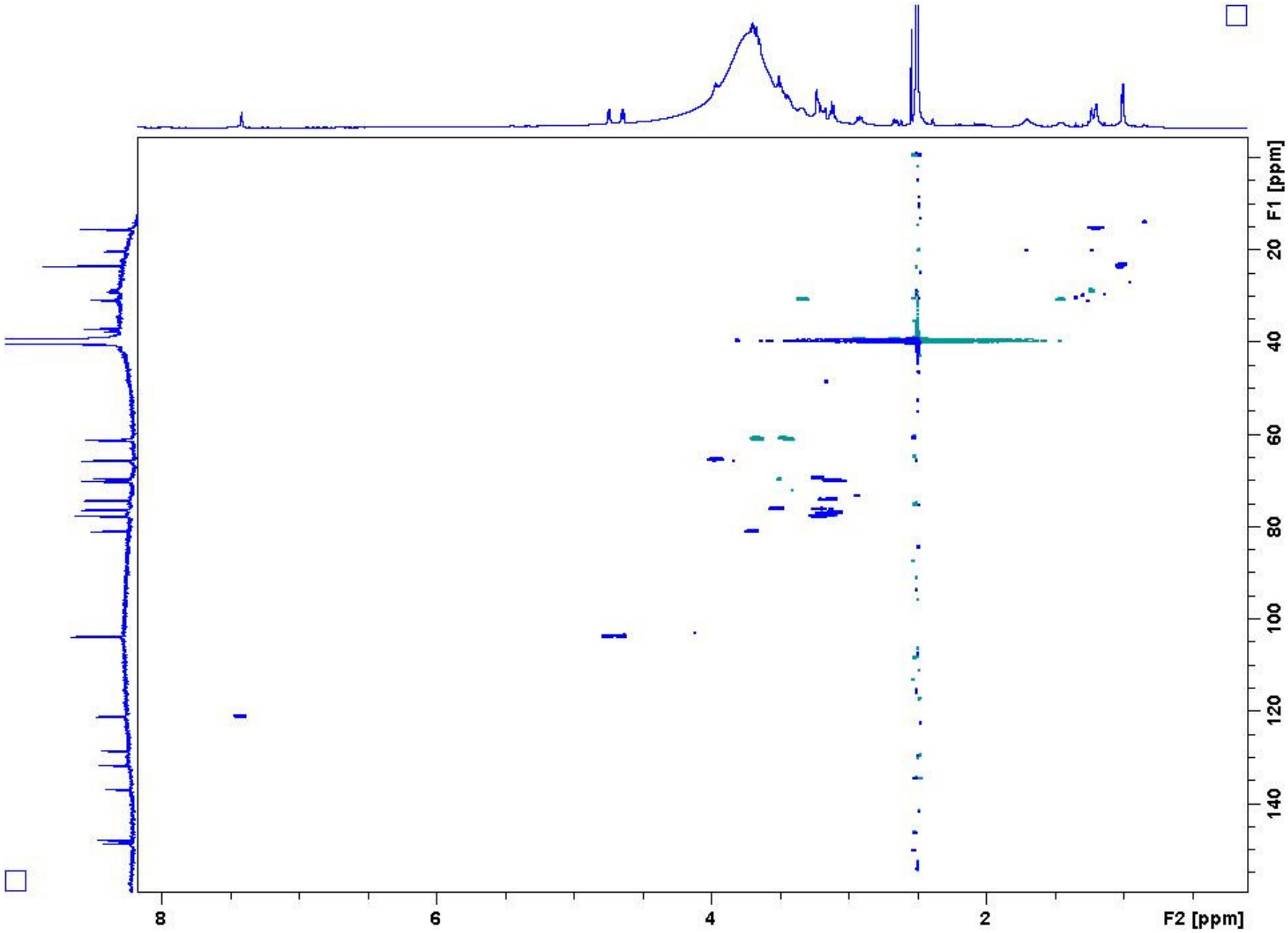


Figure S10-B. HSQC spectrum of compound **2** in DMSO-*d*$_6$.

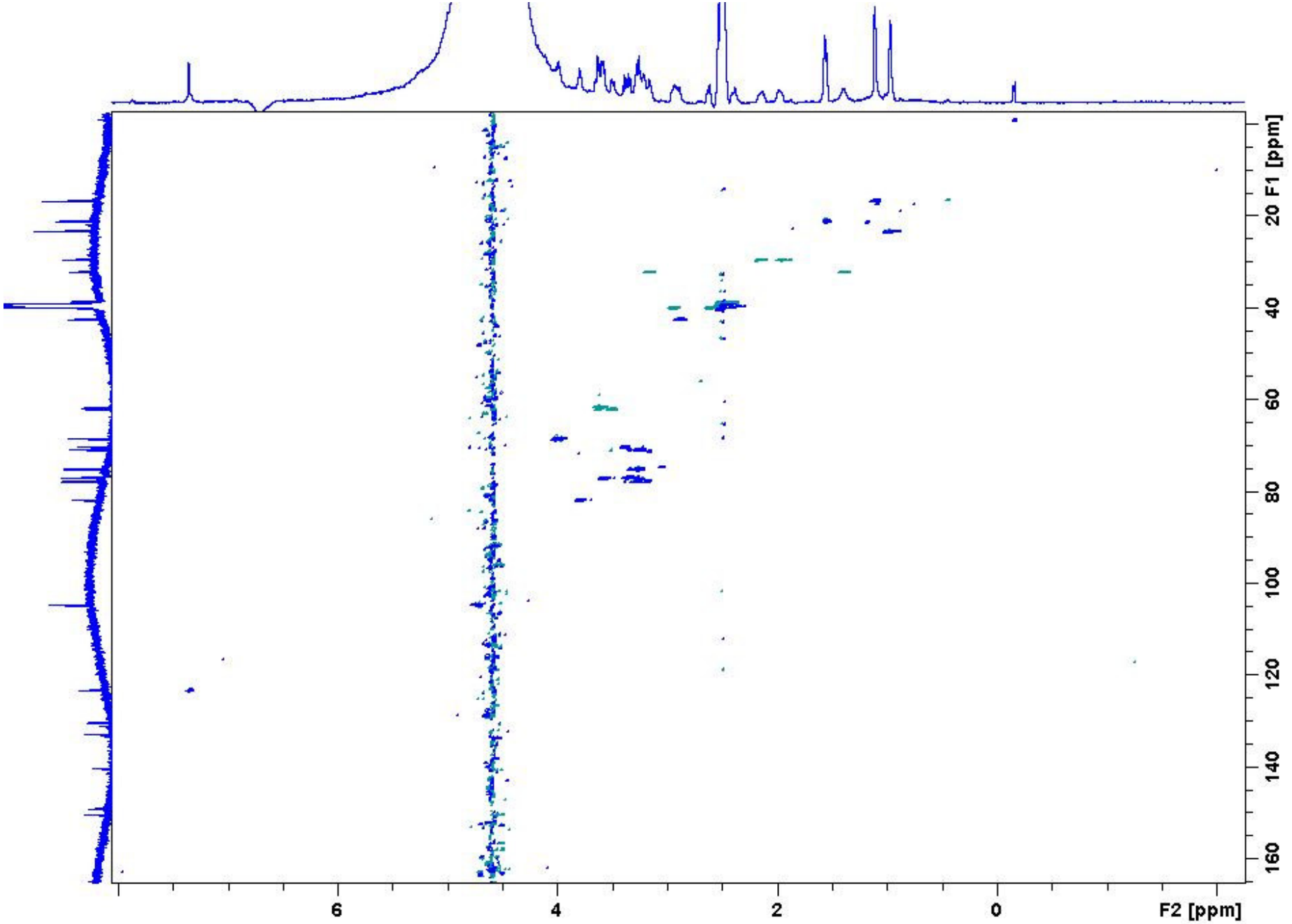


Figure S10-C. HSQC spectrum of compound **2** in D_2O.

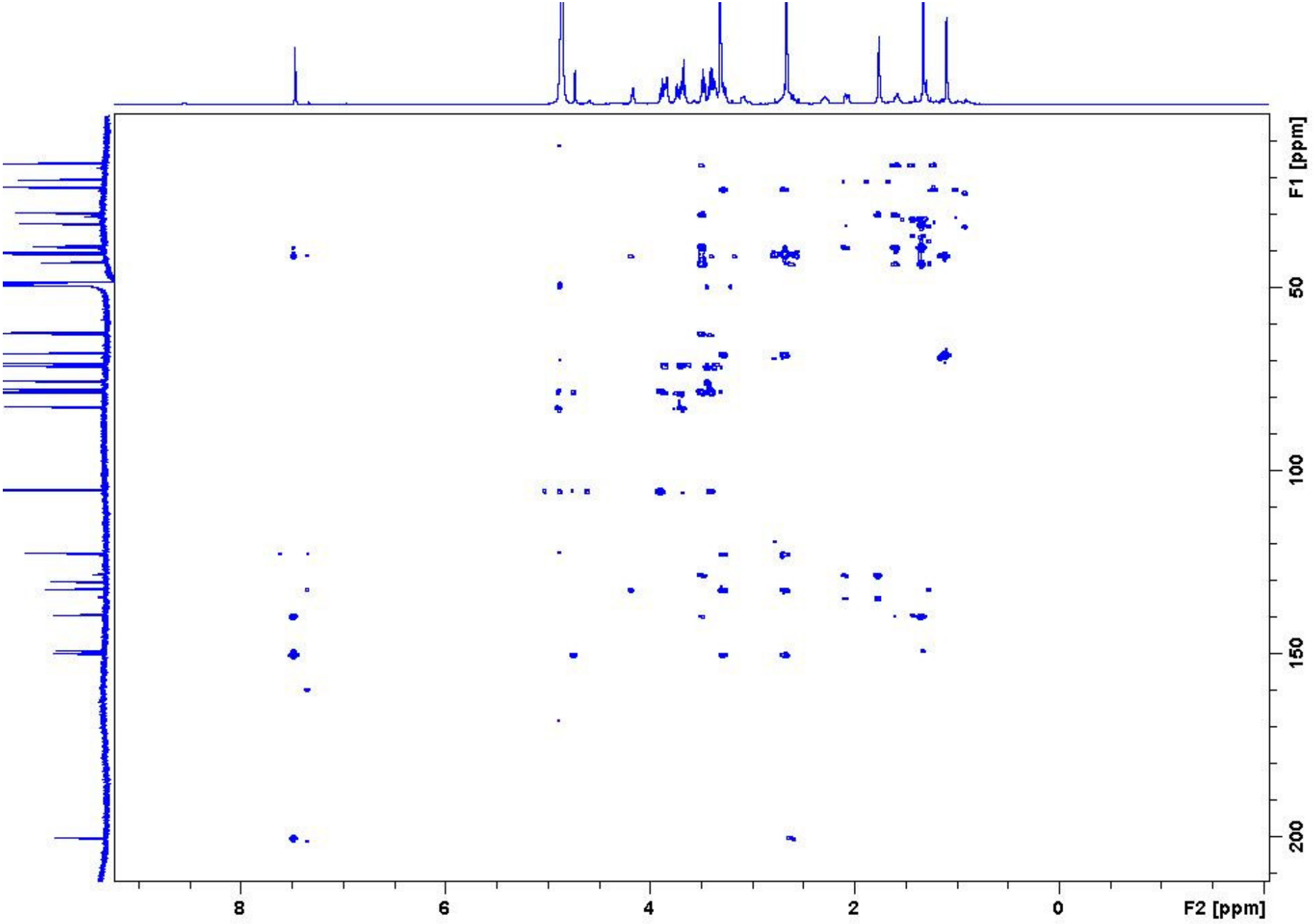


Figure S11-A. HMBC spectrum of compound **2** in Methanol-d_4.

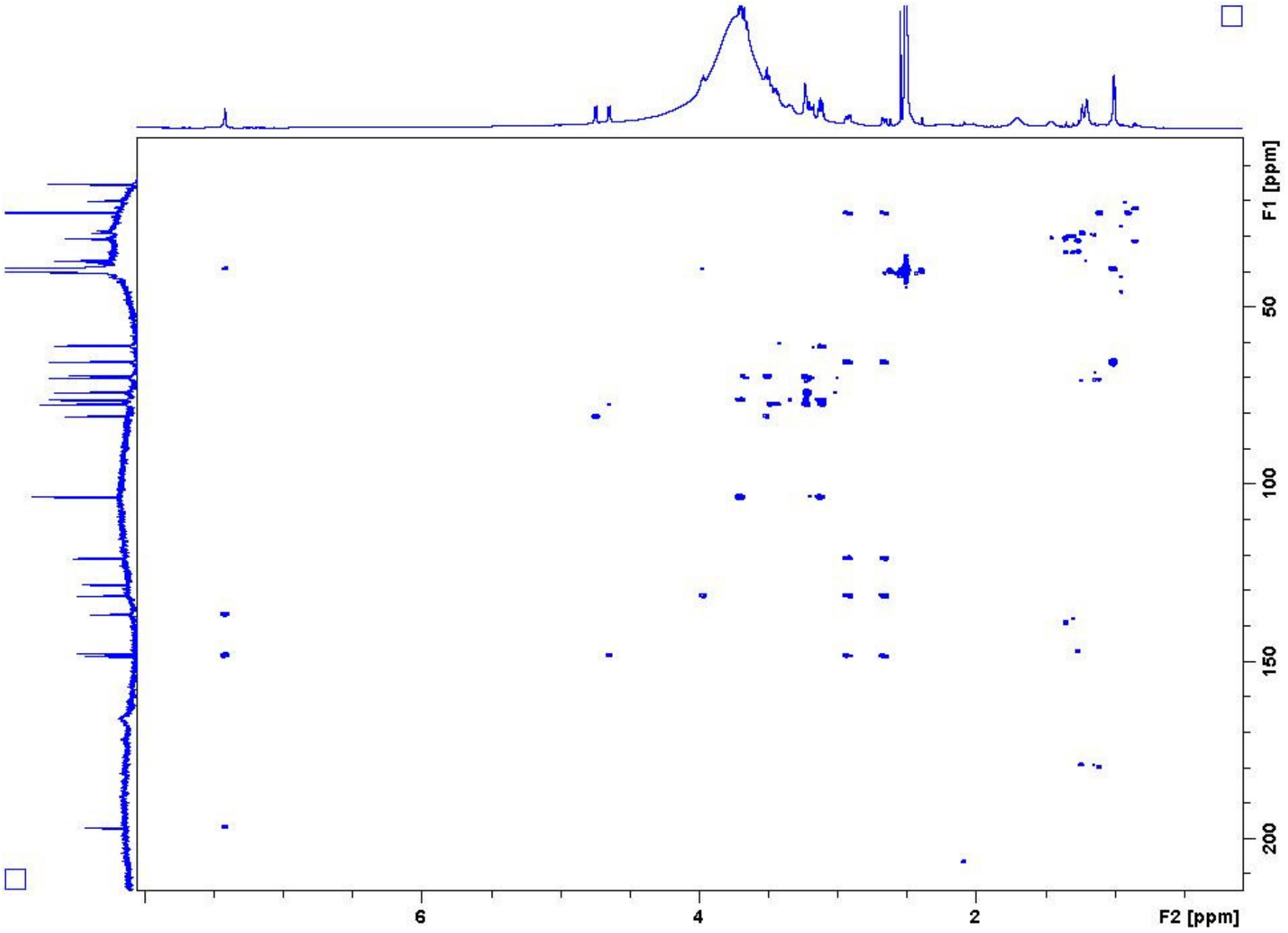


Figure S11-B. HMBC spectrum of compound **2** in DMSO-d_6.

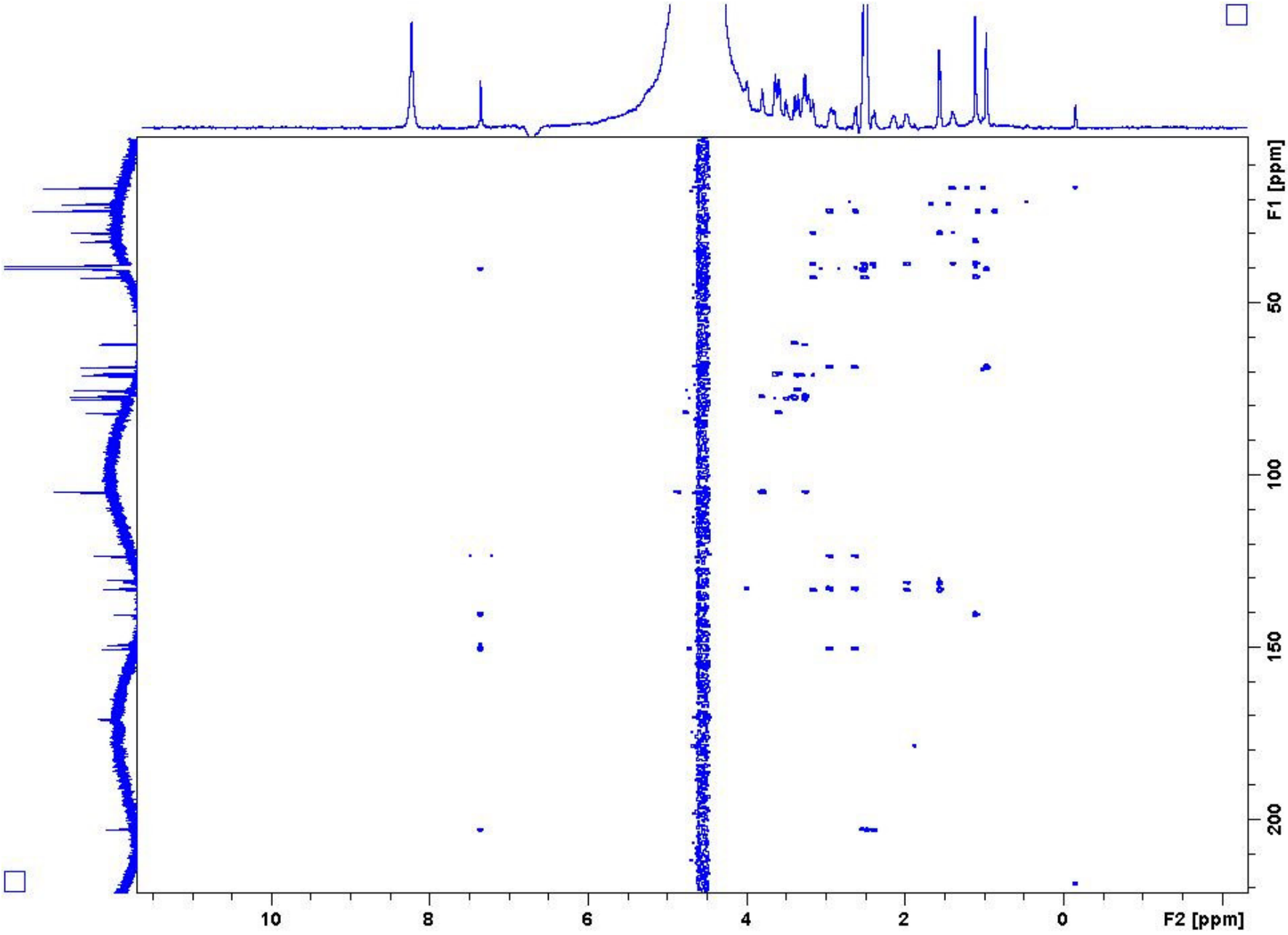
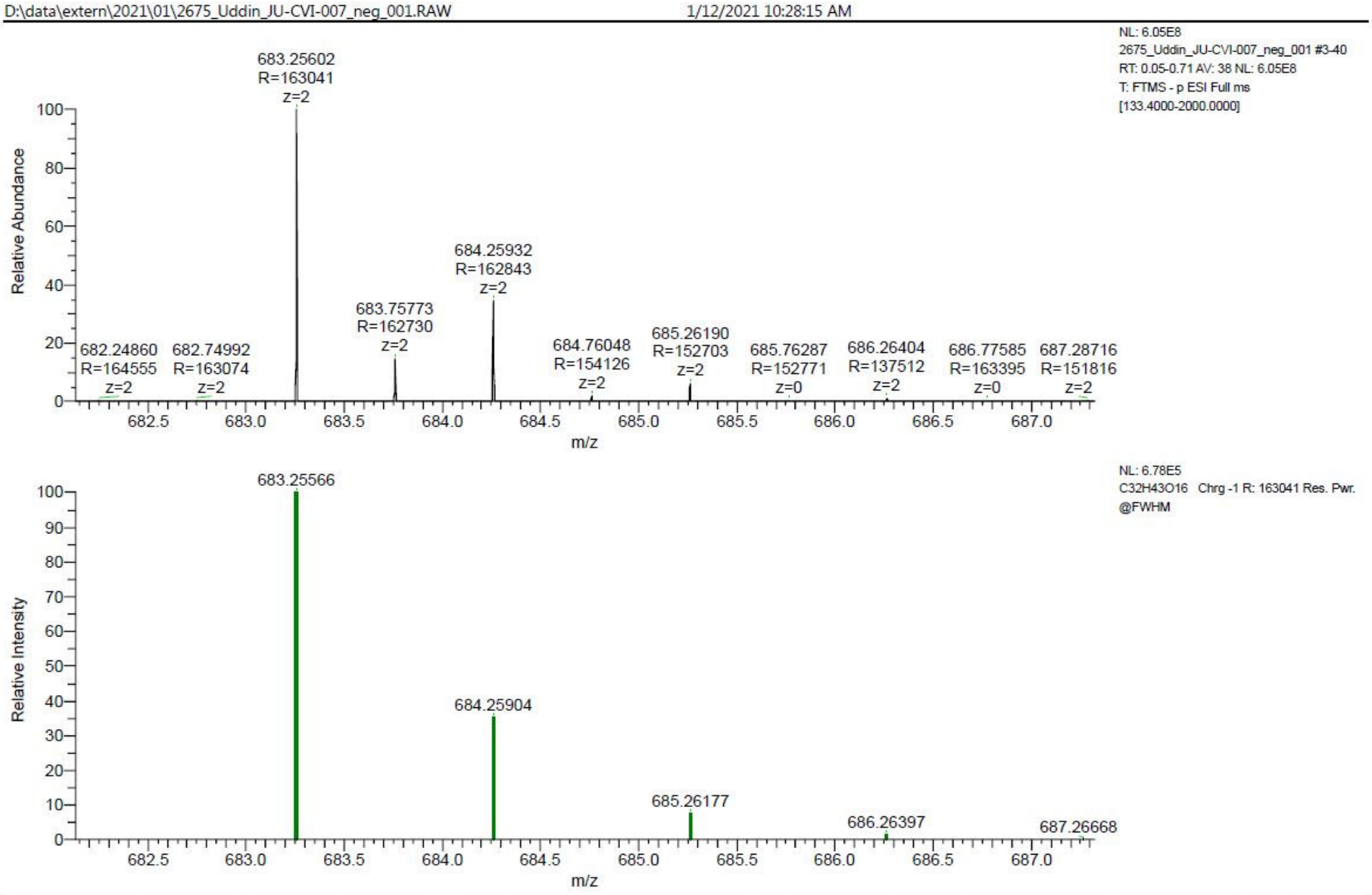
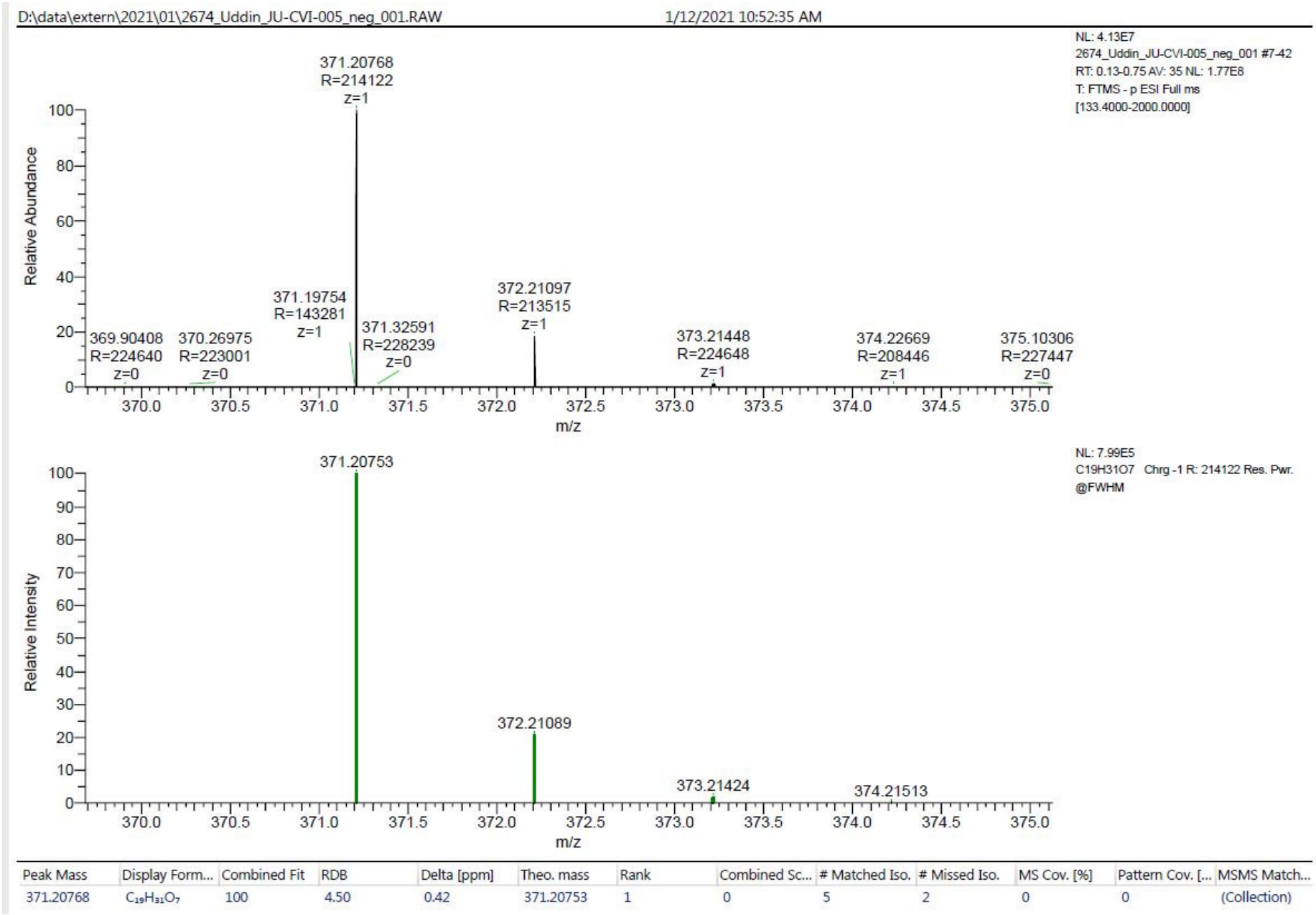


Figure S11-C. HMBC spectrum of compound **2** in D_2O.



Peak Mass	Display Form...	Combined Fit	RDB	Delta [ppm]	Theo. mass	Rank	Combined Sc...	# Matched Iso.	# Missed Iso.	MS Cov. [%]	Pattern Cov. [...	MSMS Match...
683.25602	$C_{32}H_{43}O_{16}$	100	11.50	0.54	683.25566	1	0	6	0	0	0	(Collection)

Figure S12. HR mass spectrum of compound **2** in methanol.



Peak Mass	Display Form...	Combined Fit	RDB	Delta [ppm]	Theo. mass	Rank	Combined Sc...	# Matched Iso.	# Missed Iso.	MS Cov. [%]	Pattern Cov. [...	MSMS Match...
371.20768	$C_{19}H_{31}O_7$	100	4.50	0.42	371.20753	1	0	5	2	0	0	(Collection)

Figure S13. HR mass spectrum of compound **3** in methanol.

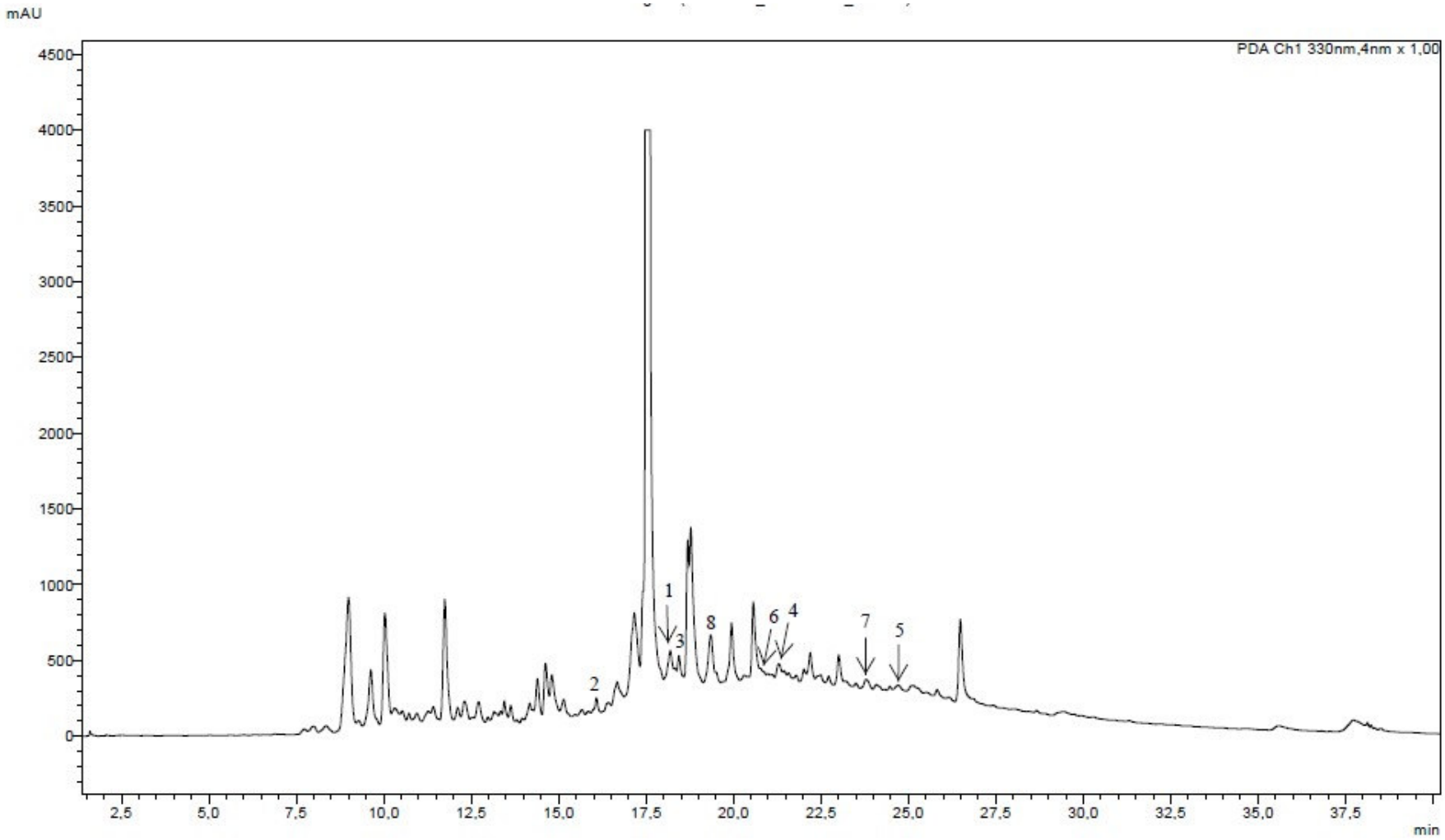


Figure S14. UHPLC (λ = 330 nm) chromatogram of *n*-butanol fraction from leaves of *Clerodendrum infortunatum.*

Table S1. 1D (^{1}H and ^{13}C) NMR spectroscopic data for compound **3**

	3[a]	**3**[b]	**3**[a]	**3**[b]
Position	δ_C, type	δ_C, type	δ_H (*J* in Hz)	δ_H (*J* in Hz)
cyclohexanone moiety				
1	37.3, C	35.9, C		
2	48.1, CH_2	47.0, CH_2	1.97, d (17.5) 2.46, d (17.5)	1.87, d (17.5) 2.34, d (17.5)
3	202.4, C	198.1, C		
4	125.4, CH	124.2, CH	5.80, s	5.72, s
5	170.1, C	166.1, C		
6	52.4, CH	50.1, CH	1.99, m[b]	1.90, t (5.0)
7	26.8, CH_2	25.1, CH_2	1.50, m 1.98, m[b]	1.38, m 1.78, m
8	37.8, CH_2	36.2, CH_2	1.61, m 1.67, m	1.50, m
9	75.5, CH	73.3, CH	3.88, m	3.74, dd (12.0, 6.0)
10	19.9, CH_3	19.5, CH_3	1.18, d (6.0)	1.1, d (6.0)
11	29.1, CH_3	26.8, CH_3	1.01, s	1.00, s
12	27.5, CH_3	28.5, CH_3	1.09, s	0.97, s
13	25.0, CH	24.1, CH	2.05, d (1.0)	1.97, m
glucopyranosyl moiety				
1′	102.1, CH	100.7, CH	4.32, d (8.0)	4.16, d (8.0)
2′	75.2, CH	73.5, CH	3.14, dd (9.0, 8.0)	2.89, t (8.5)
3′	78.2, CH	76.9, CH	3.35, dd (9.0, 9.0)	3.12, t (8.5)
4′	71.8, CH	70.3, CH	3.26, m	3.03, m
5′	77.9, CH	76.8, CH	3.25, m	3.05, m
6′	62.9, CH_2	61.3, CH_2	3.64, dd (12.00, 5.5) 3.85, dd (12.00, 2.0)	3.41, m 3.64, m

[a]Spectra were referenced to solvent residual and solvent signals of CD_3OD at 3.31 ppm (^{1}H NMR, 600 MHz) and 49.0 ppm (^{13}C NMR, 150 MHz), respectively.

[b]Spectra were referenced to solvent residual and solvent signals of $(CD_3)_2SO$ at 2.50 ppm (^{1}H NMR, 600 MHz) and 39.52 ppm (^{13}C NMR, 150 MHz), respectively.