

Supporting Information

Production of Terretonin N and Butyrolactone I by Thermophilic *Aspergillus terreus* TM8 promoted apoptosis and cell death in human prostate and ovarian cancer cells

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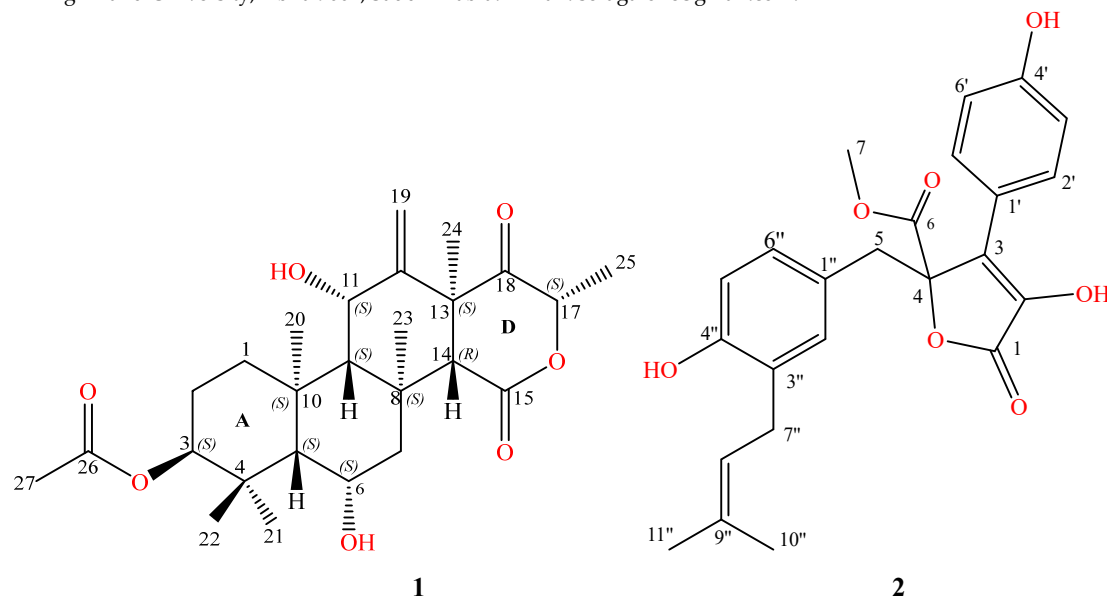


Table of Contents:	Page
Physico-properties of terretonin N (2) and butyrolactone I (2)	S3
Figure S1-S10: MS and NMR spectral data of terretonin N (1)	S4-S13
Figure S11: COSY (—) and key HMBC (↷) correlations of terretonin N (1)	S14
Table S1: ¹³ C (125 MHz) and ¹ H (500 MHz) NMR data of terretonin N (1) in CDCl ₃	S14
Figure S12-S17: MS and NMR spectral data of butyrolactone I (2)	S15-S20
Figure S18: H,H COSY (—) and selected HMBC (↷) correlations of butyrolactone I (2)	S21
Table S2: ¹³ C (125 MHz) and ¹ H (500 MHz) NMR data of butyrolactone I (2) in Acetone- <i>d</i> ₆ :	S21

Physico-properties of terretonin N (2) and butyrolactone I (2)

Terretonin N (1)

C₂₆H₃₈O₇ (462), colorless solid, *R_f* = 0.54 (CH₂Cl₂/ 5% MeOH), UV non absorbing-absorbing (254 nm) or fluorescence, pink and later as violet staining with anisaldehyde/sulfuric acid. $[\alpha]_{\text{D}}^{20}$ 114 (*c*= 0.1, MeOH). ¹H NMR (500 MHz, CDCl₃, see Table S1), ¹³C NMR (125 MHz, CDCl₃, see Table S1). (+)-ESI MS: *m/z* (%) = 485 ([M+Na]⁺, 100), 947 ([2M+Na]⁺, 0.58). (-)-ESI MS: *m/z* (%) = 461 ([M-H]⁻, 62), 891 ([2M-H]⁻, 4), (-)-ESI-HRMS: *m/z* = 461.2547 [M-H]⁺ (Calc. 461.2545 for C₂₆H₃₇O₇).

Butyrolactone I (2)

C₂₄H₂₄O₇ (424), colorless solid, *R_f* = 0.43 (CH₂Cl₂/ 7% MeOH), UV absorbing (254 nm), intense violet staining with anisaldehyde/sulfuric acid. ¹H NMR (500 MHz, Acetone-d₆), ¹³C NMR (125 MHz, Acetone-d₆), see Table (3). (+)-ESI MS: *m/z* (%) = 447 ([M+Na]⁺, 81), 871 ([2M+Na]⁺, 100). (-)-ESI MS: *m/z* (%) = 423 ([M-H]⁻, 13), 847 ([2M-H]⁻, 4), (+)-ESI-HRMS: *m/z* = 447.1414 [M+Na]⁺ (Calc. 447.1414 for C₂₄H₂₄NaO₇), (-)-ESI-HRMS: *m/z* = 423.1435 [M-H]⁻ (calc. 423.1449 for C₂₄H₂₃O₇).

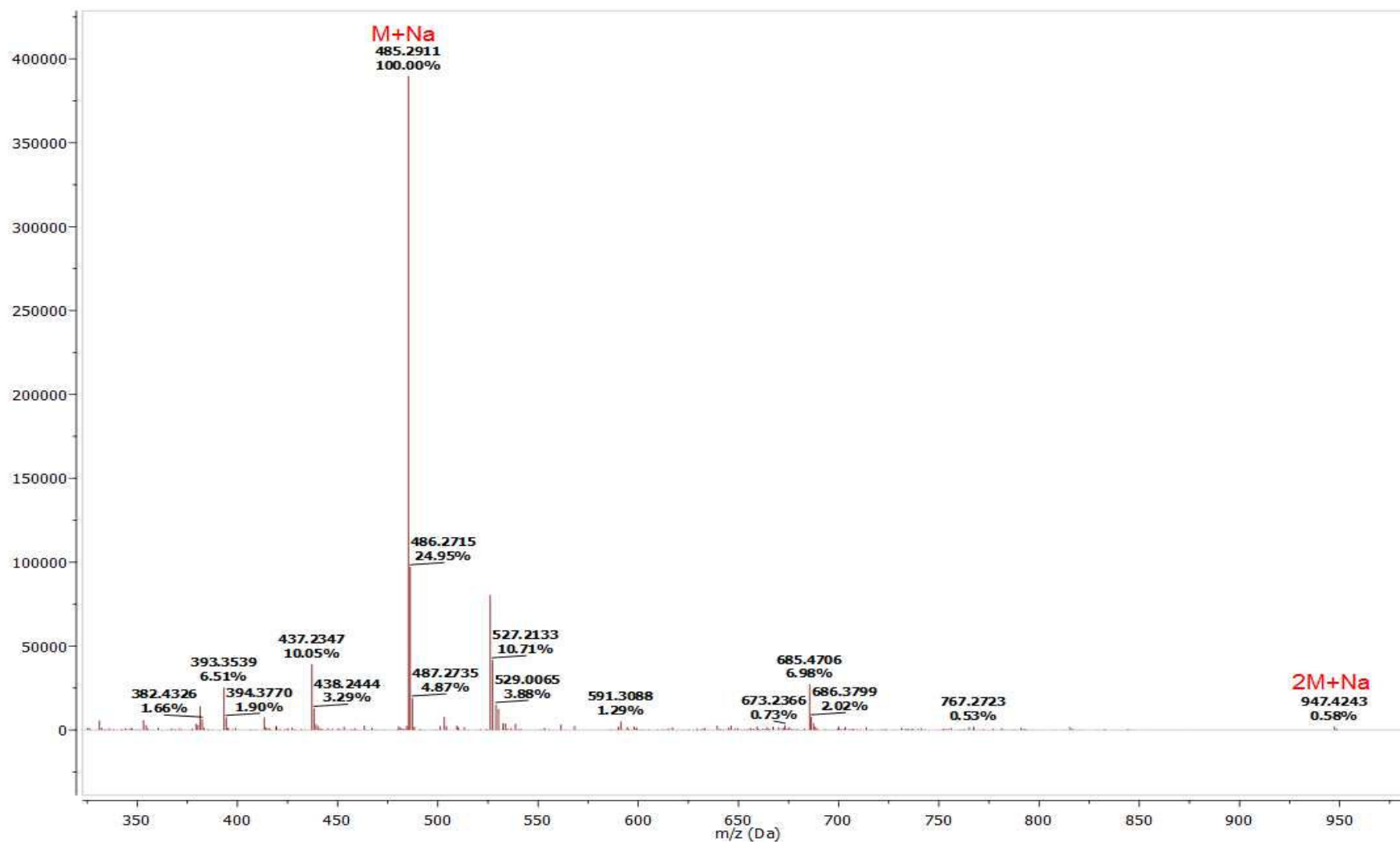


Figure S1: (+)-ESIMS of terretonin N (**1**)

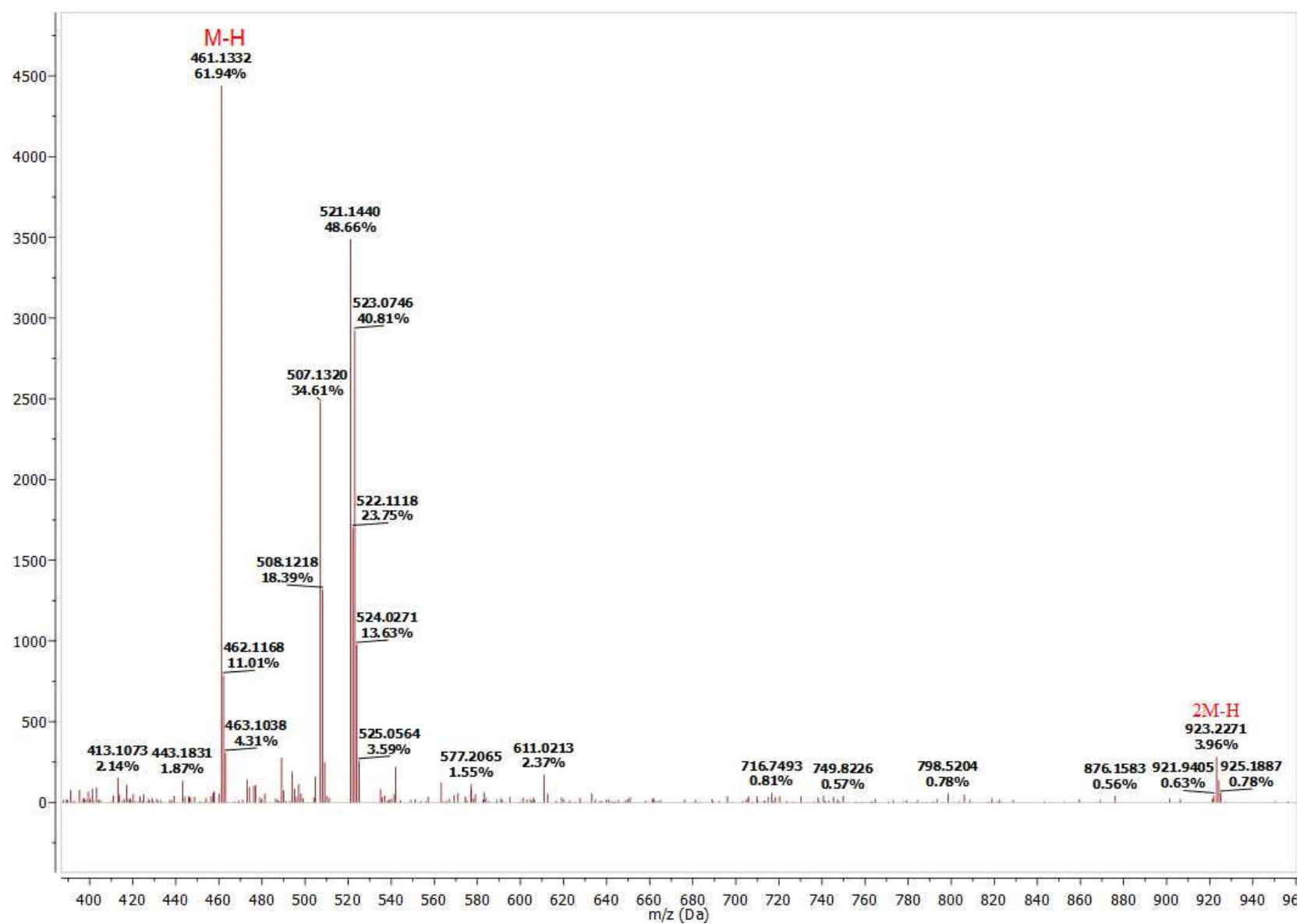


Figure S2: (-)-ESIMS of terretinin N (1)

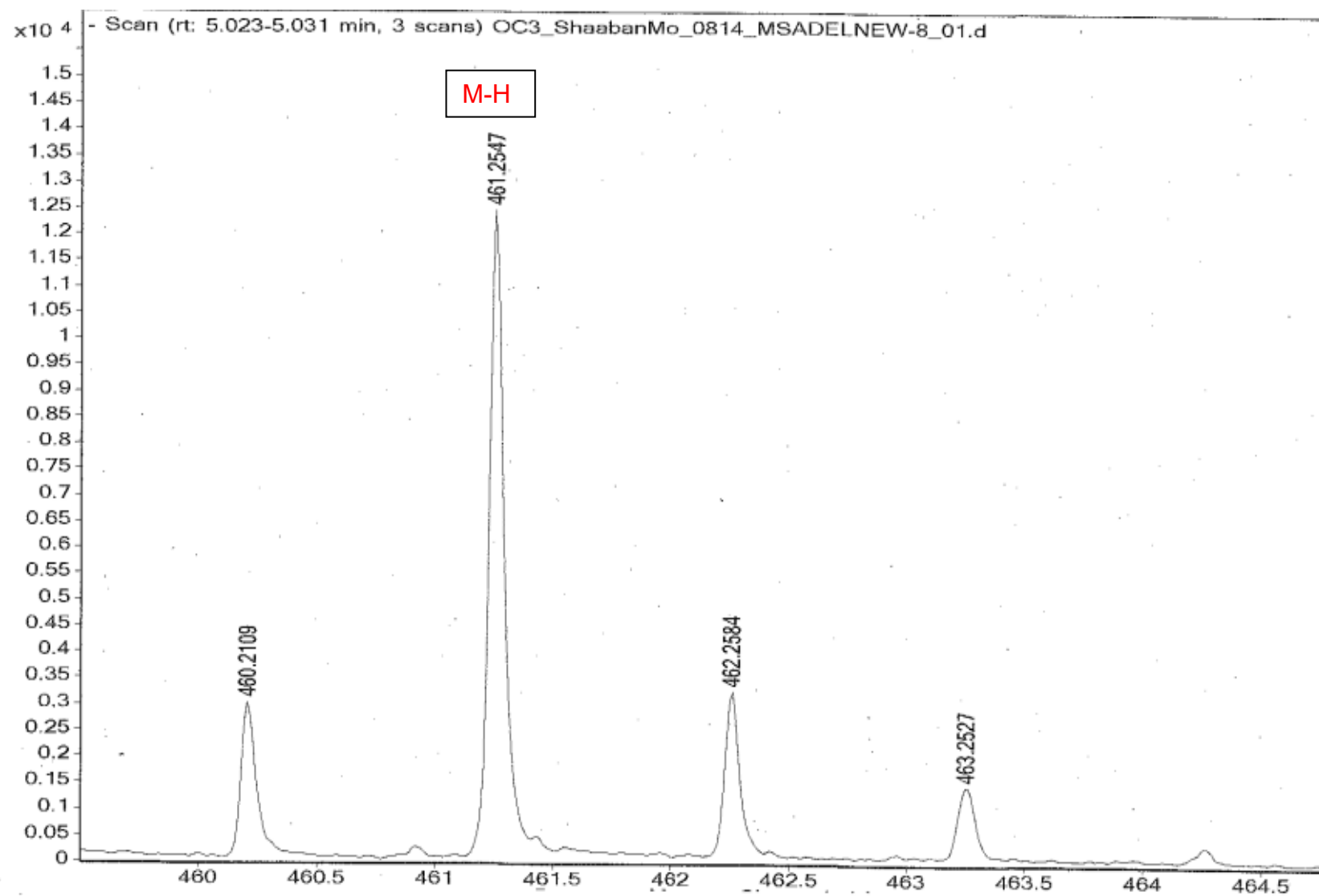


Figure S3: HR-ESI MS of terretonin N (1)

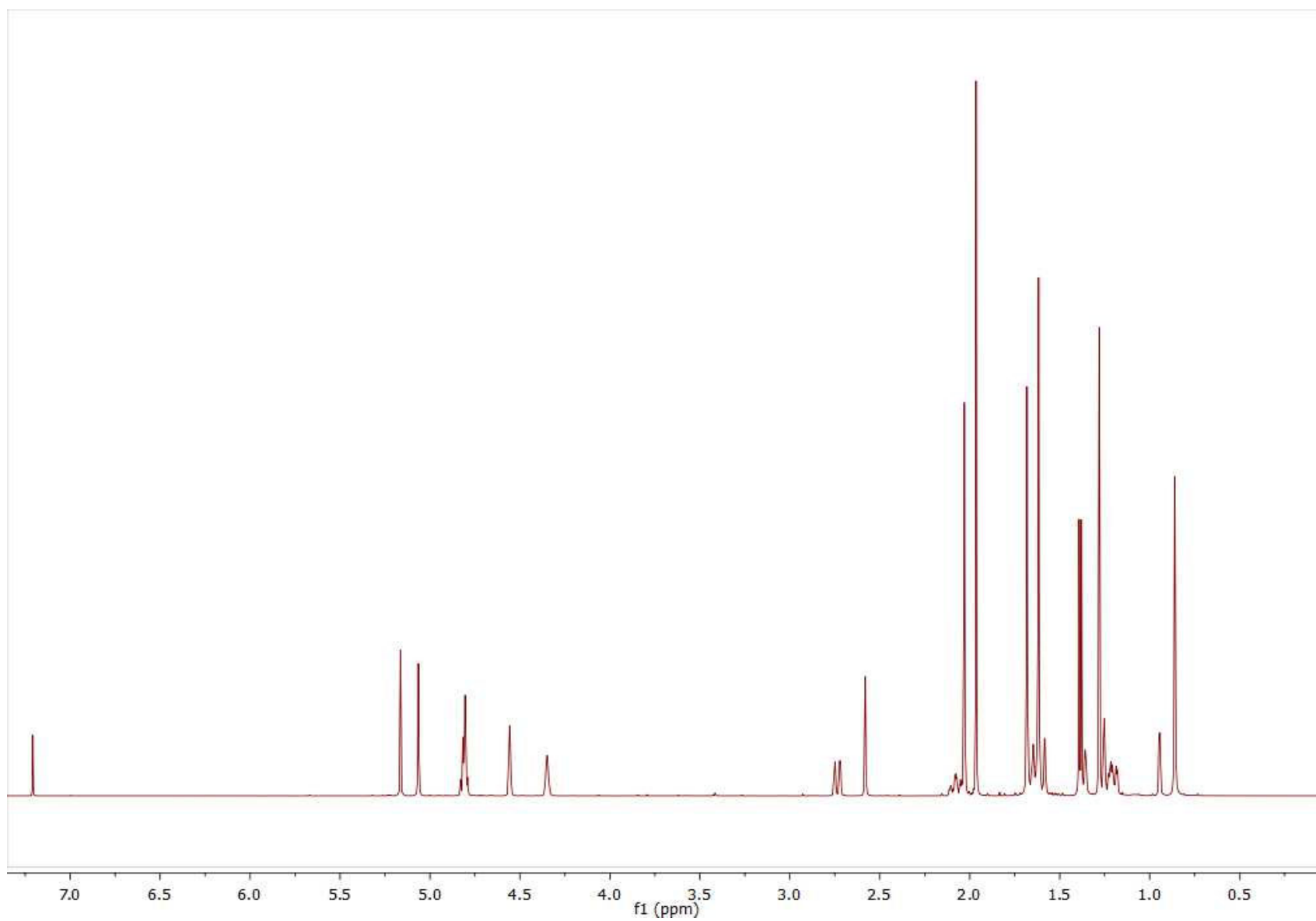


Figure S4: ^1H NMR spectrum (CDCl_3 , 500 MHz) of terretinin N (**1**)

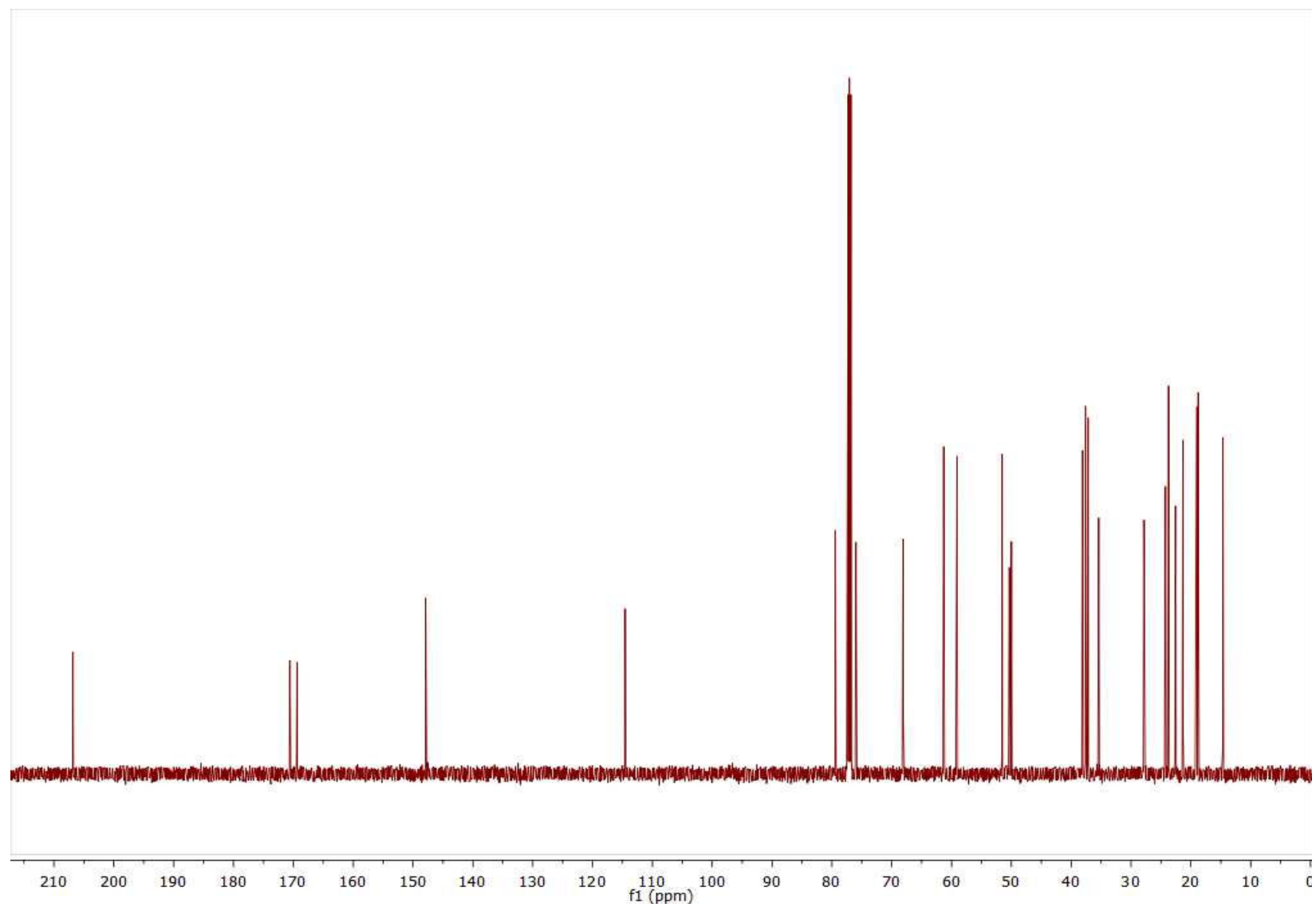


Figure S5: ^{13}C NMR spectrum (CDCl_3 , 125MHz) of terretonin N (**1**)

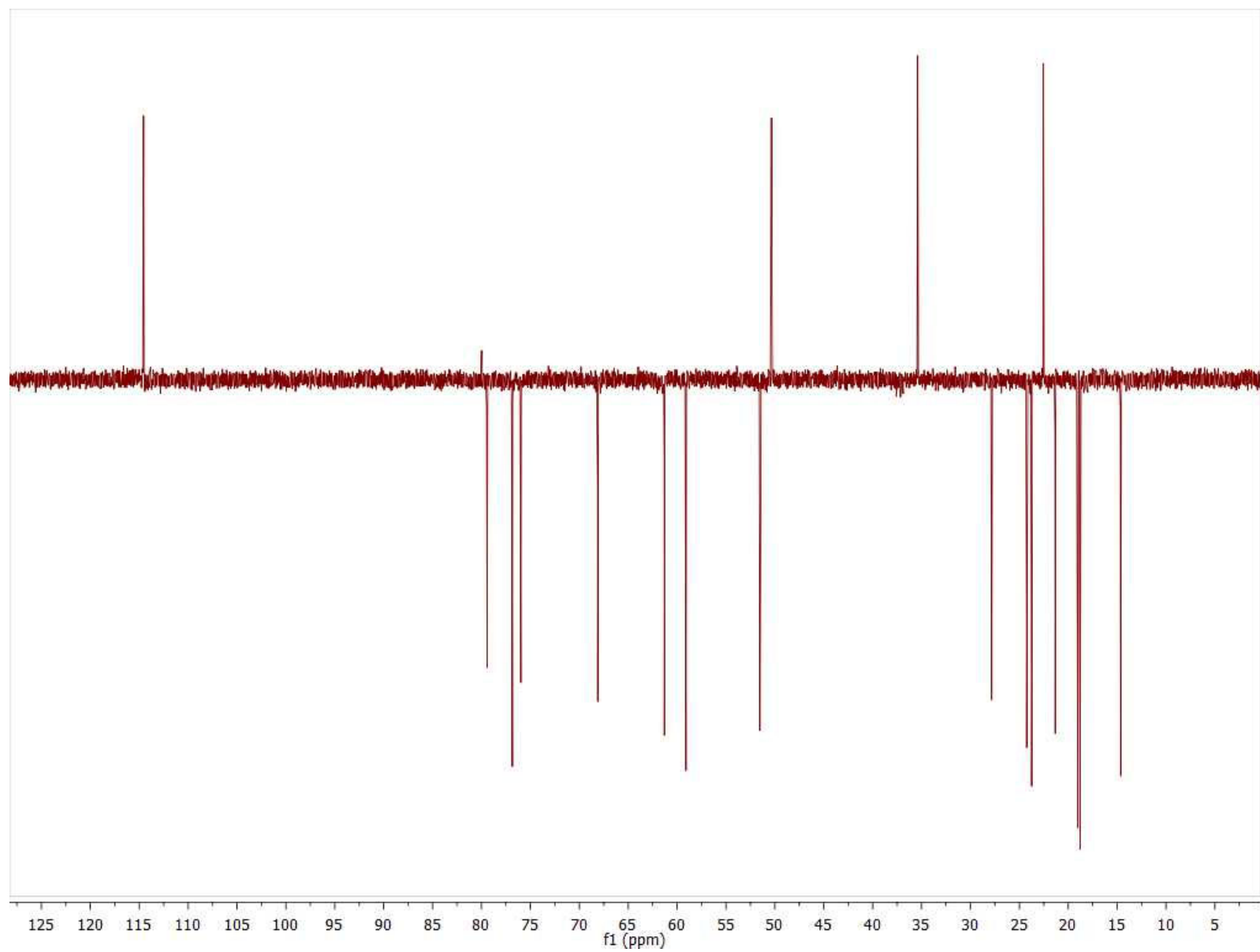


Figure S6: DEPT spectrum (CDCl₃, 125 MHz) of terretonin N (**1**)

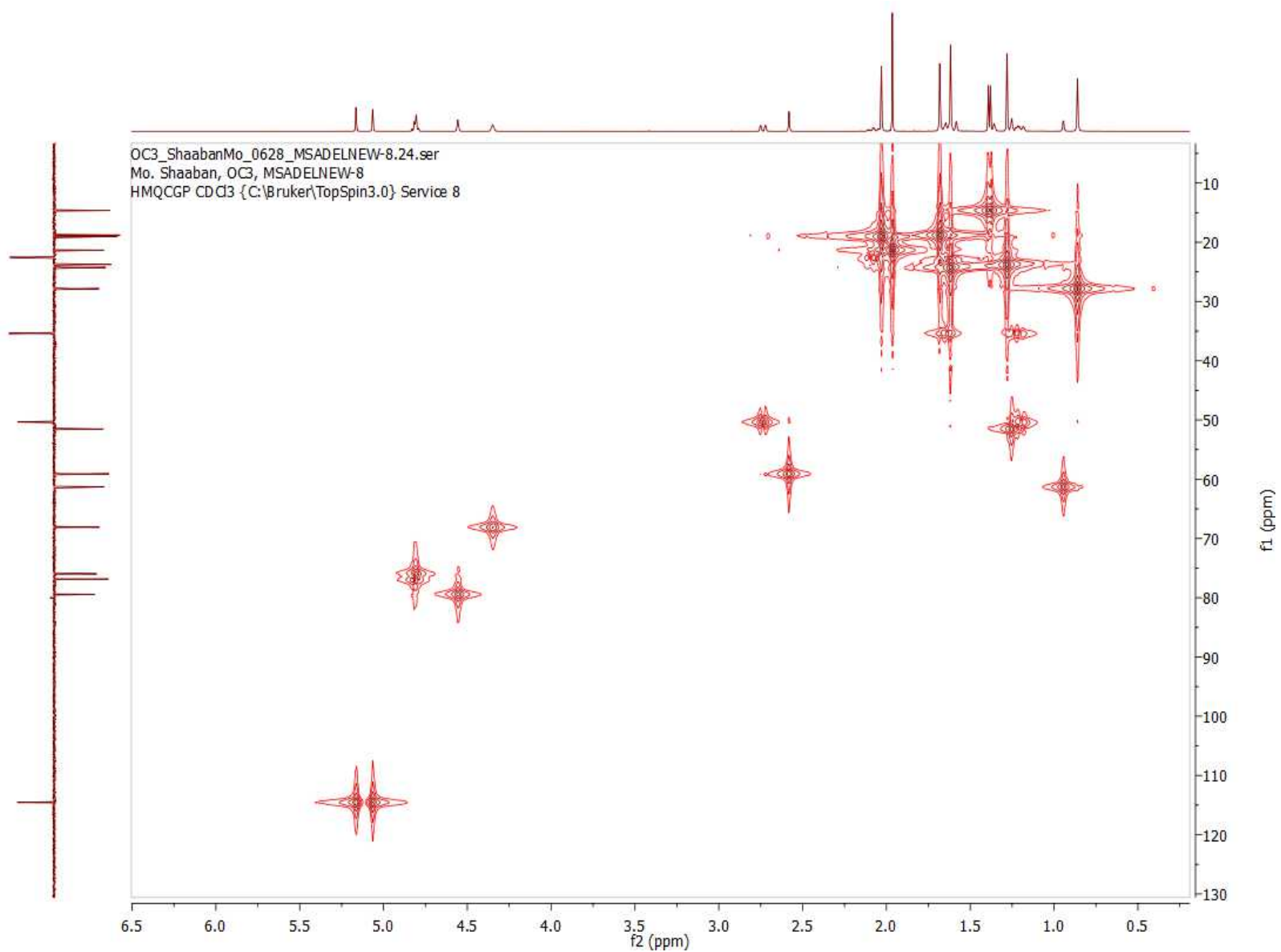


Figure S8: HMQC spectrum (CDCl₃, 500 MHz) of terretonin N (**1**)

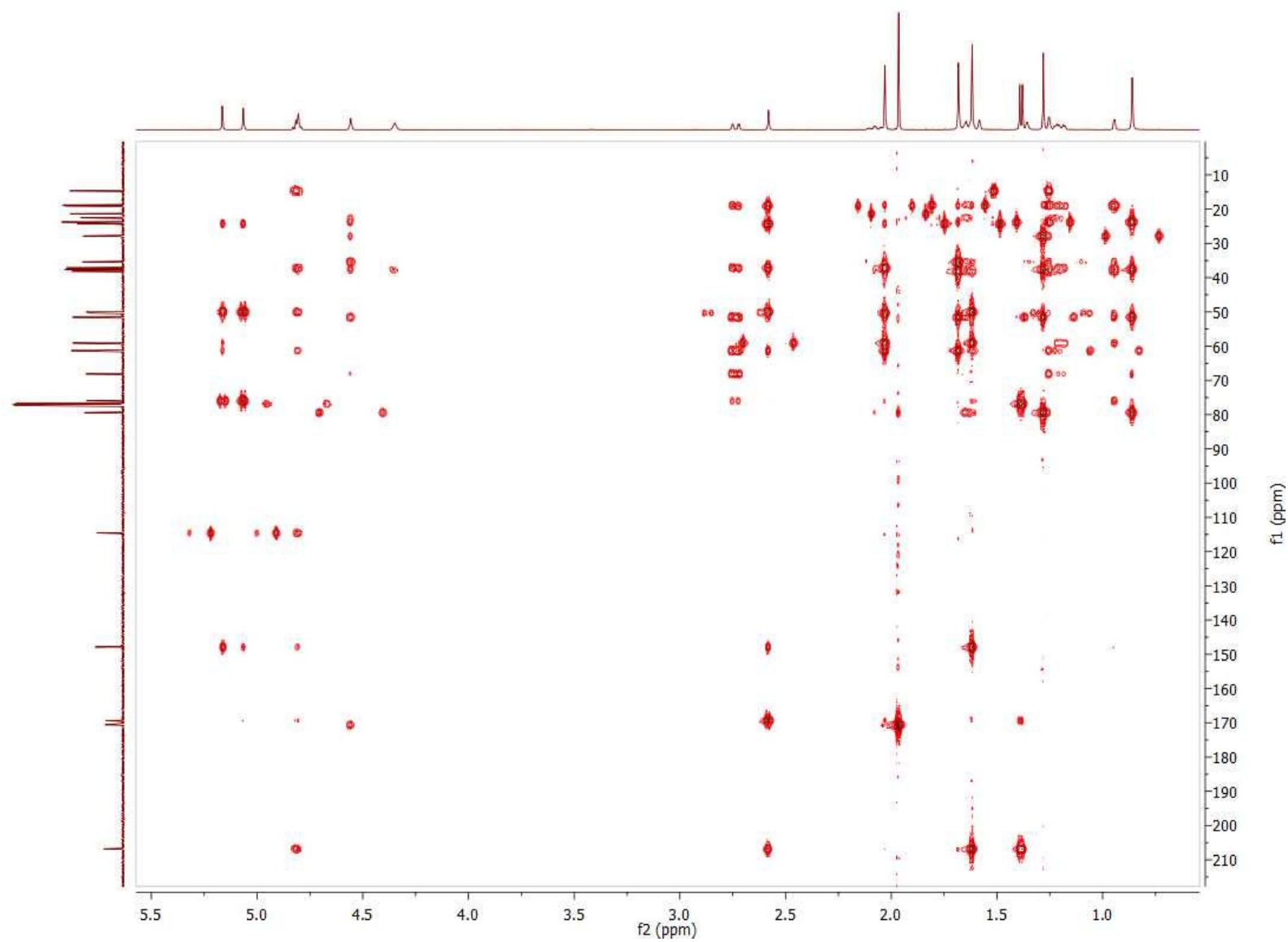


Figure S9: HMBC spectrum (CDCl₃, 500 MHz) of terretinin N (**1**)

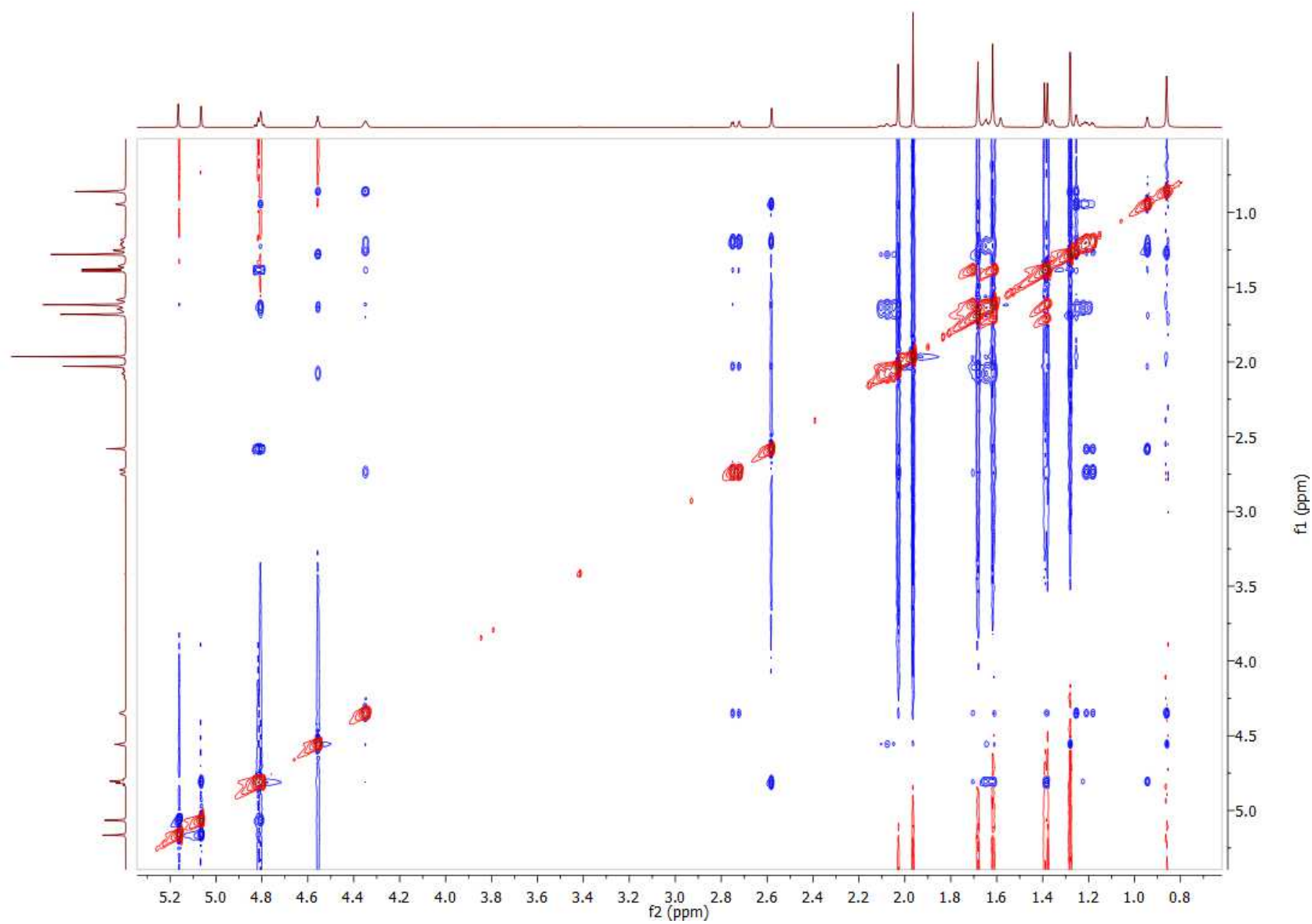


Figure S10: NOESY spectrum (CDCl_3 , 500 MHz) of terretinin N (**1**)

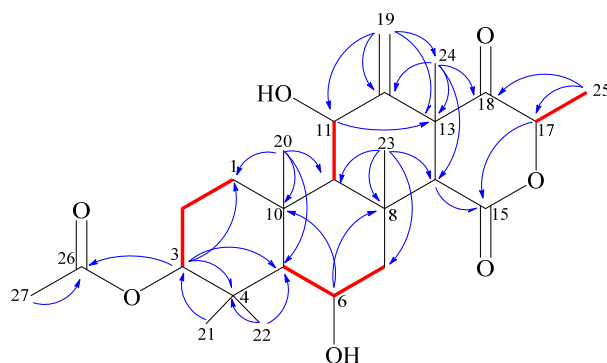


Figure S11. COSY (—) and key HMBC (↷) correlations of terretinin N (**1**)

Table S1: ^{13}C (125 MHz) and ^1H (500 MHz) NMR data of terretinin N (**1**) in CDCl_3

Position	δ_{C} [ppm]	δ_{H} [ppm] (m, J [Hz])	Position	δ_{C} [ppm]	δ_{H} [ppm] (m, J [Hz])
1	35.4	1.65 (m), 1.21 (m)	14	59.1	2.58 (s)
2	22.5	1.95 (m), 1.65 (m)	15	169.4	-
3	79.4	4.55 (t, 3.0)	17	76.8	4.81 (m)
4	37.6	-	18	206.8	-
5	51.5	1.25 (m)	19	114.5	5.16 (s), 5.06 (s)
6	68.1	4.35 (t, 3.0)	20	18.8	1.68 (s)
7	50.3	2.74 (dd, 14.5, 2.7), 1.19 (m)	21	27.8	0.86 (s)
8	37.1	-	22	23.7	1.28 (s)
9	61.3	0.95 (d, 2.4)	23	19.0	2.03 (s)
10	38.1	-	24	24.2	1.62 (s)
11	76.0	4.81 (m)	25	14.6	1.38 (d, 6.4)
12	147.9	-	26	170.6	-
13	50.0	-	27	21.3	1.96 (s)

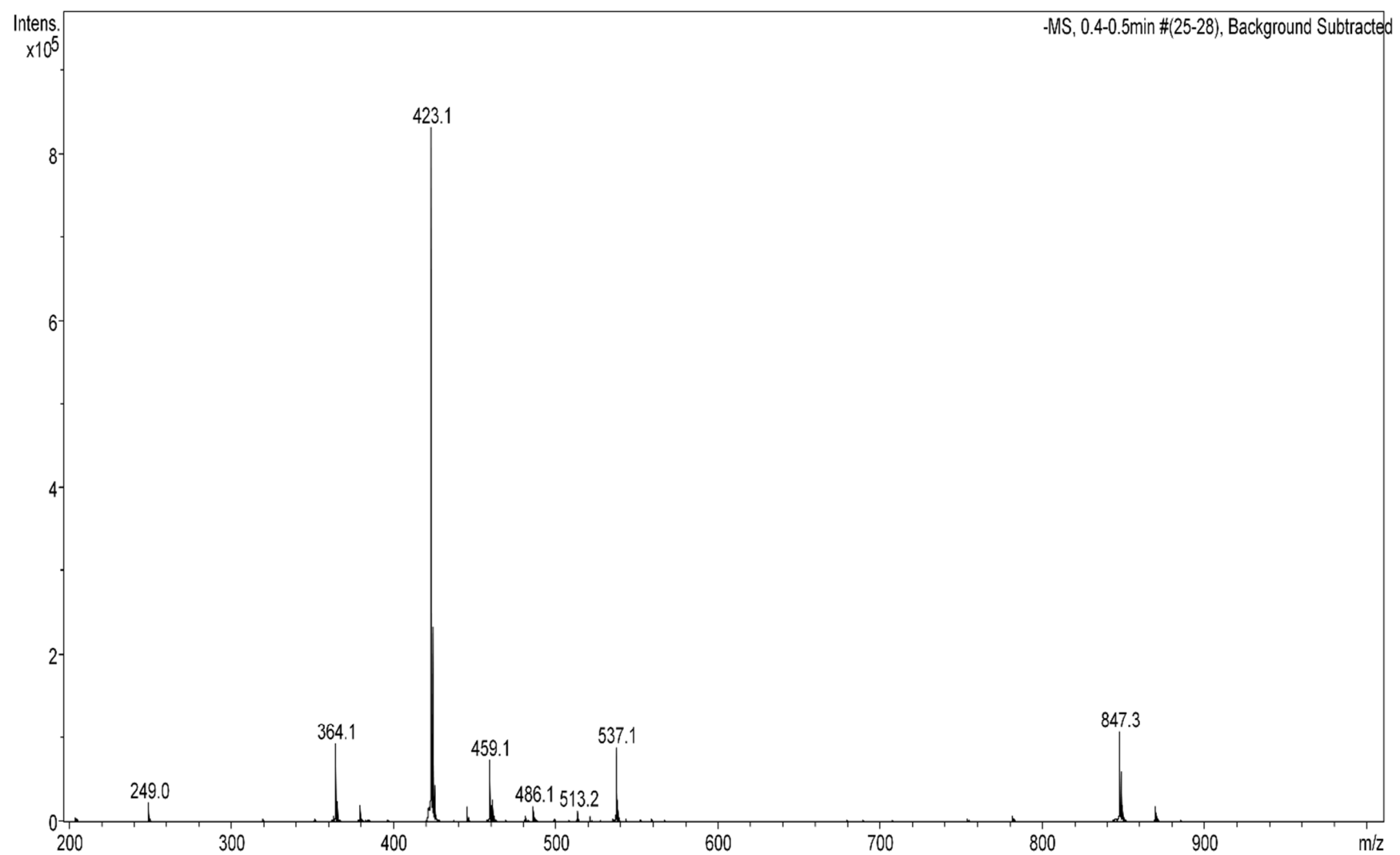


Figure S12: (-)-ESIMS of butyrolactone I (**2**)

OC3_ShaabanMo_0917_MSDA-3a.10.1.1r
Mo. Shaaban, OC3, MSDA-3a
PROTON Acetone {C:\Bruker\TopSpin3.0} Service 15

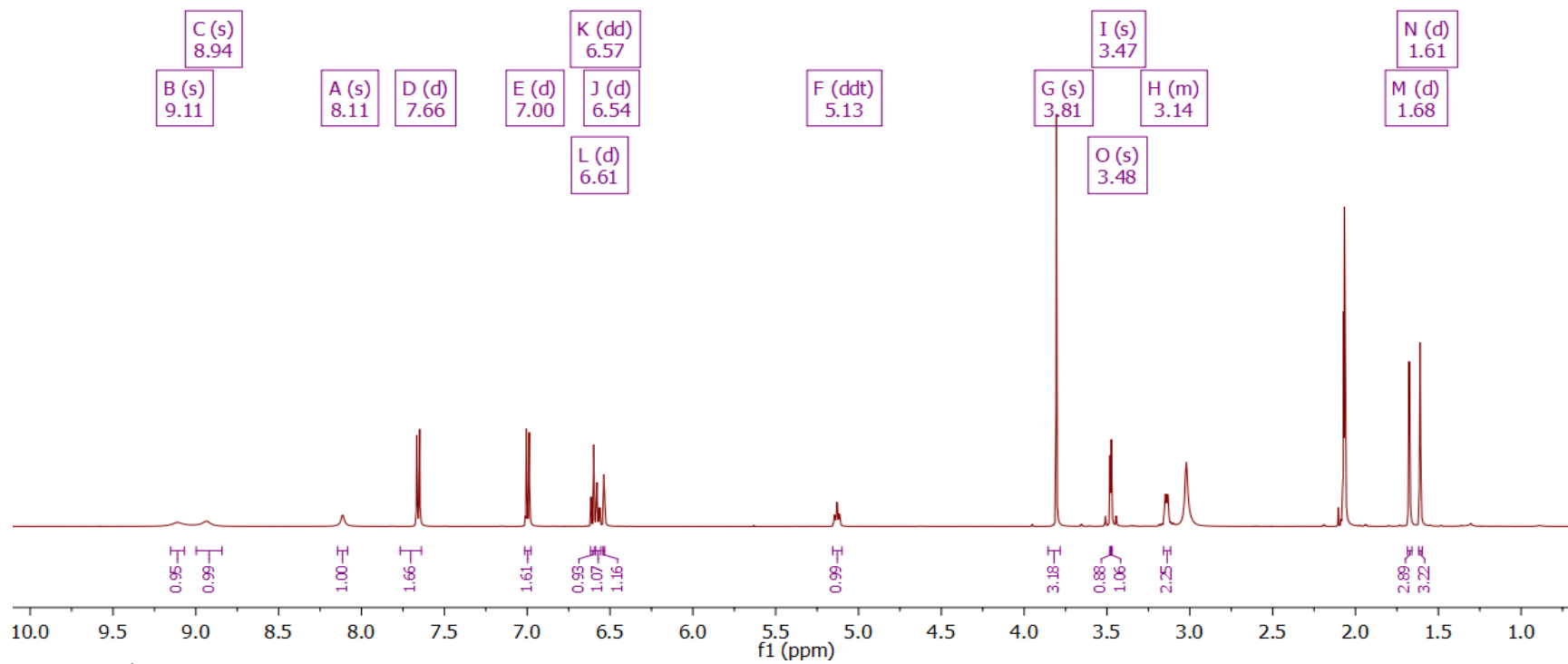


Figure S13: ¹H NMR (500 MHz, Acetone-d₆) of butyrolactone I (2)

OC3_ShaabanMo_0917_MSDA-3a.14.1.1r
Mo. Shaaban, OC3, MSDA-3a
C13CPD Acetone {C:\Bruker\TopSpin3.0} Service 15

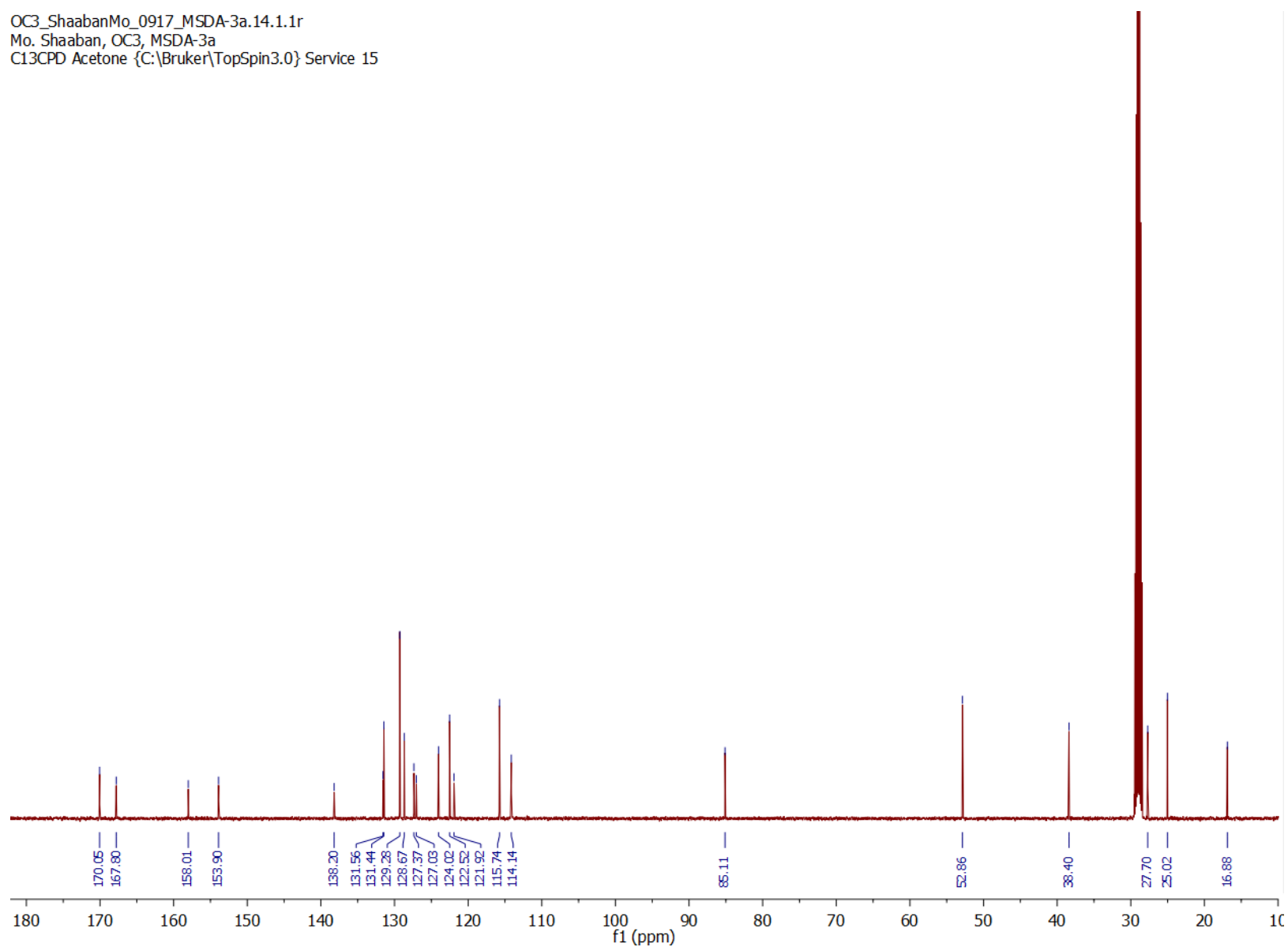


Figure S14: ¹³C NMR (125 MHz, Acetone-d₆) of butyrolactone I (2)

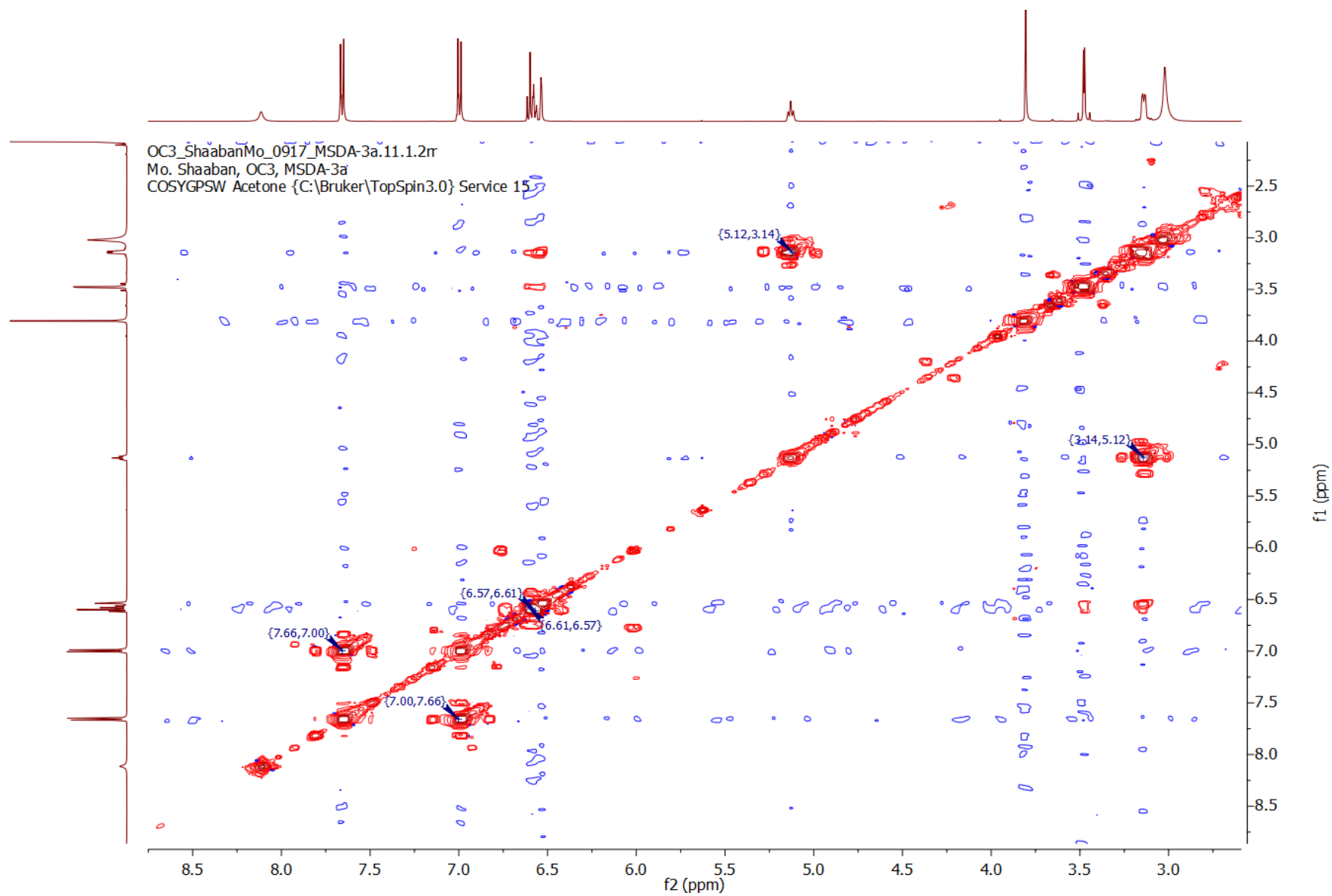


Figure S15: H-H-COSY spectrum of butyrolactone I (2)

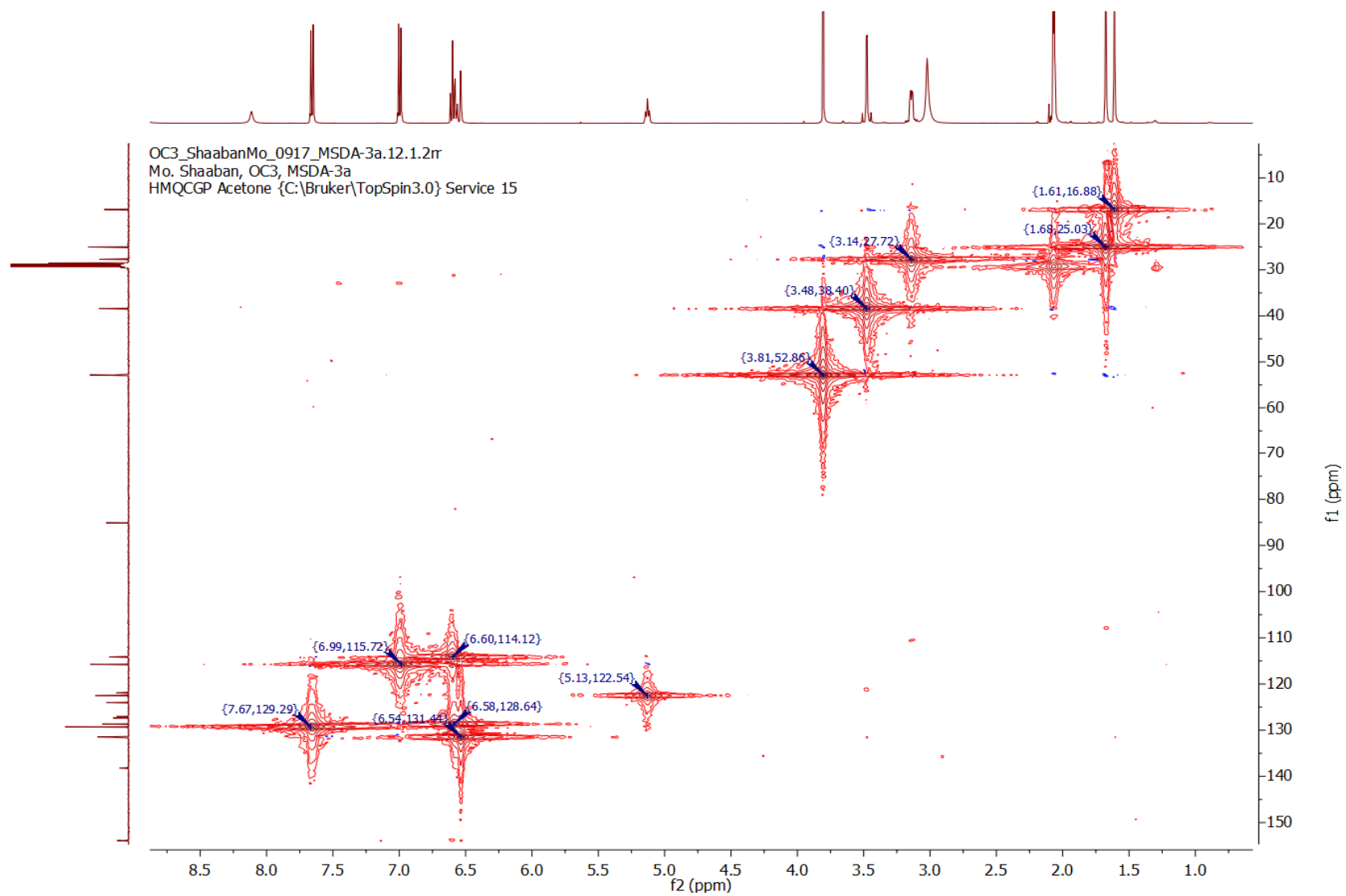


Figure S16: HMQC spectrum of butyrolactone I (2)

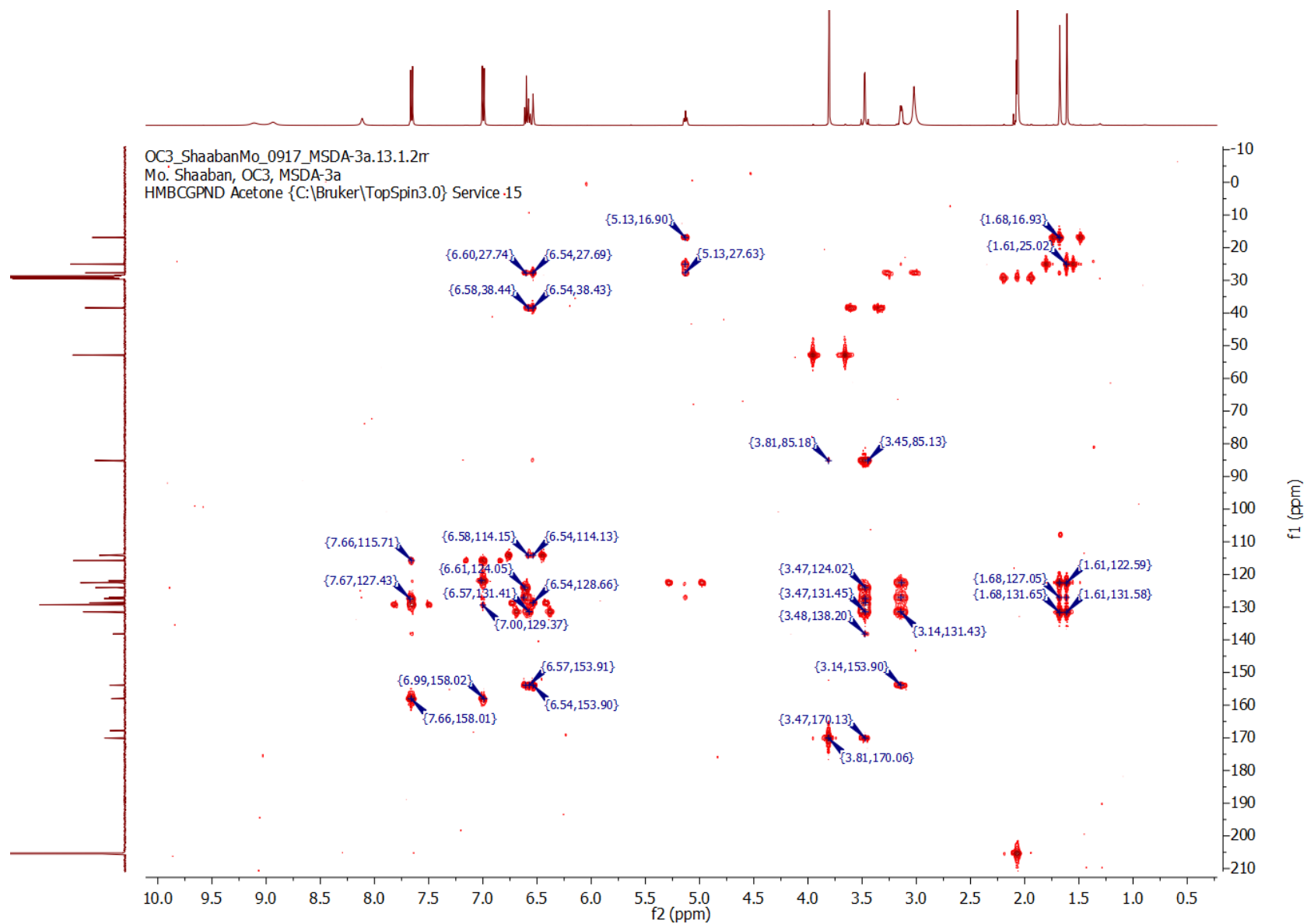


Figure S17: HMBC spectrum of butyrolactone I (2)

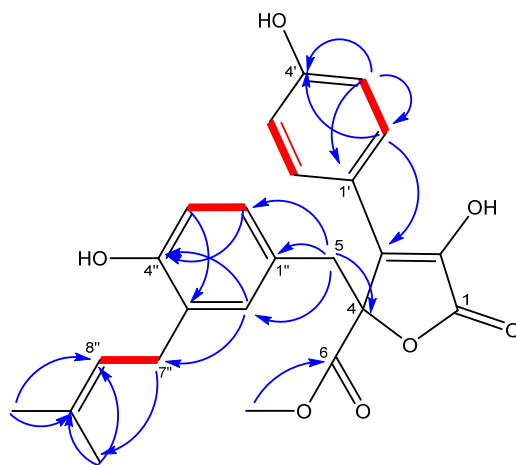


Figure S18: H,H COSY (—) and selected HMBC (→) correlations of butyrolactone I (**2**)

Table S2: ^{13}C (125 MHz) and ^1H (500 MHz) NMR data of butyrolactone I (**2**) in Acetone- d_6 :

Position	δ_{C} (ppm)	δ_{H} (J in Hz)	position	δ_{C} (ppm)	δ_{H} (J in Hz)
1	167.8	-	1''	124.0	-
2	138.2	-	2''	131.4	6.54 (d,2.2)
3	127.4	-	3''	127	-
4	85.1	-	4''	153.9	-
5	38.4	3.48 (s), 3.47 (s)	5''	114.1	6.61(d,8.1)
6	170.1	-	6''	128.7	6.57(dd,8.2,2.2)
7	52.9	3.81 (s)	7''	27.7	3.14(m)
1'	121.9	-	8''	122.5	5.13(ddt,8.8,5.9,1.5)
2',6'	129.3	7.66(d,8.9)	9''	131.6	-
3',5'	115.7	7.0(d,8.8)	10''	25.0	1.68(d,1.4)
4'	158.0	-	11''	16.9	1.61(d,1.2)