



Supporting Information

for

Semisynthesis, characterisation, and antibacterial evaluation of a novel lecanoric acid-derived amide library

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Beilstein J. Org. Chem. **2026**, 22, 1023–1032. [doi:10.3762/bjoc.22.81](https://doi.org/10.3762/bjoc.22.81)

NMR data tables and 1D/2D NMR spectra for natural products 1–5 and semisynthetic amide analogues 6–13, additionally, HRESIMS data for all semisynthetics

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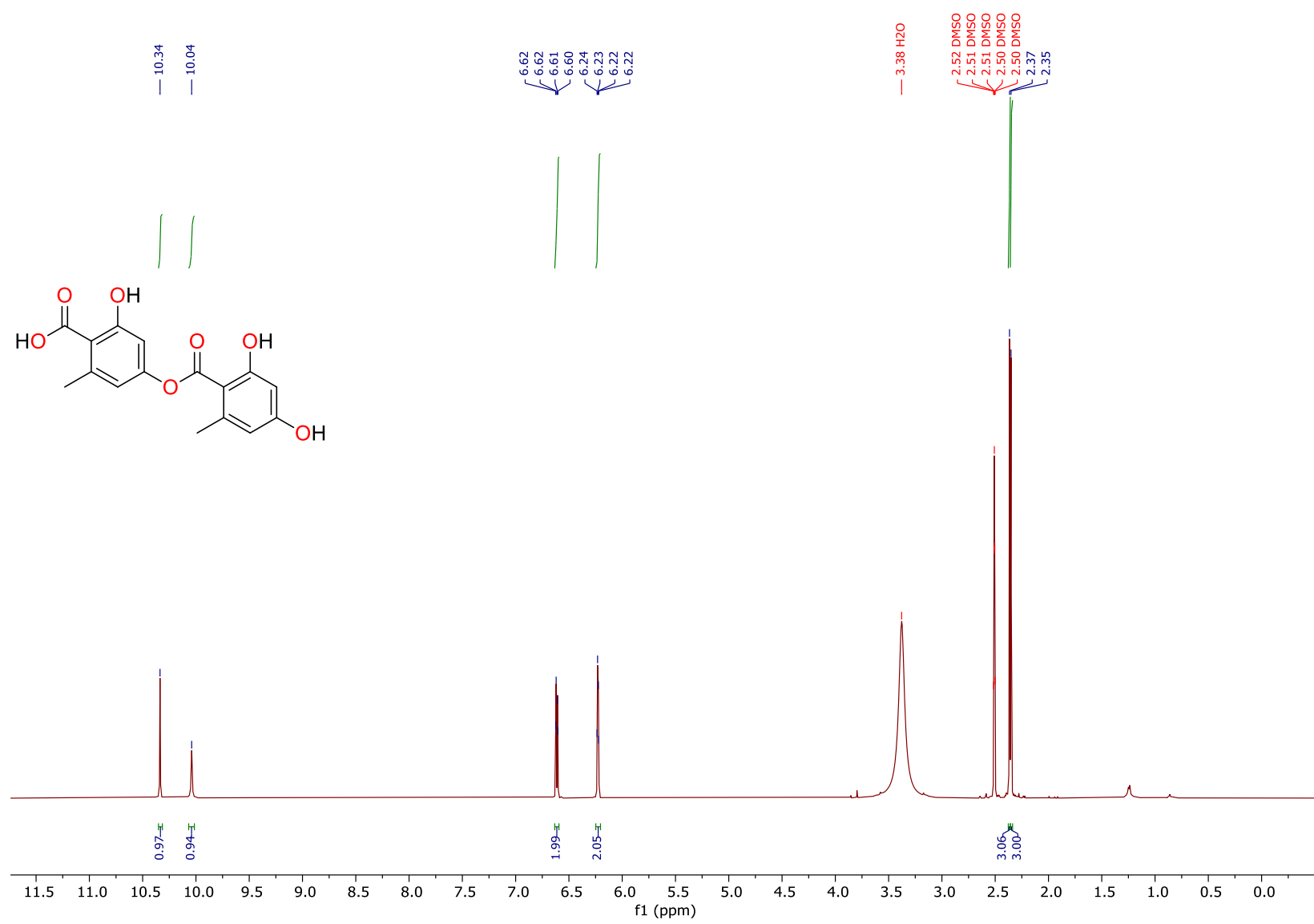
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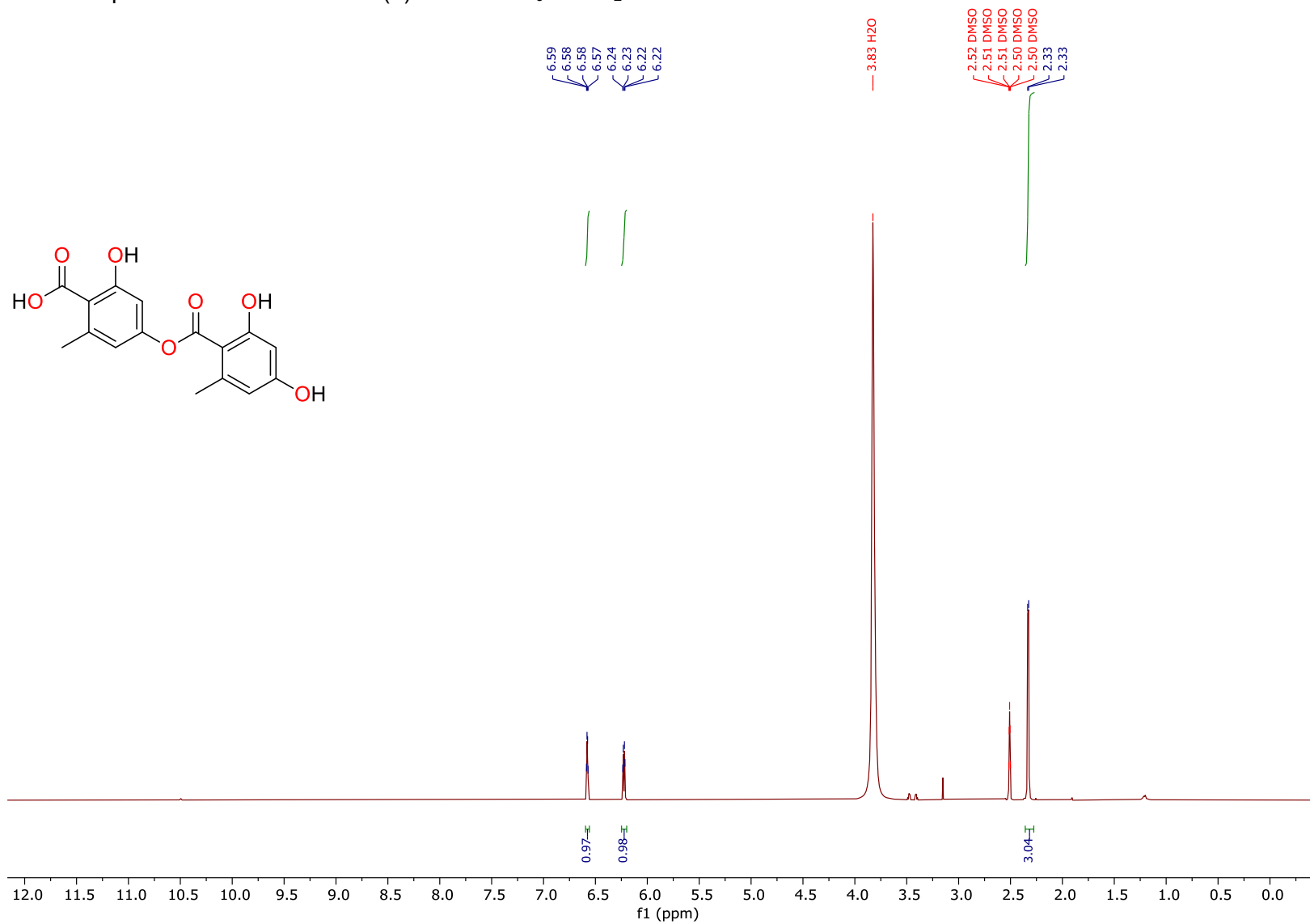
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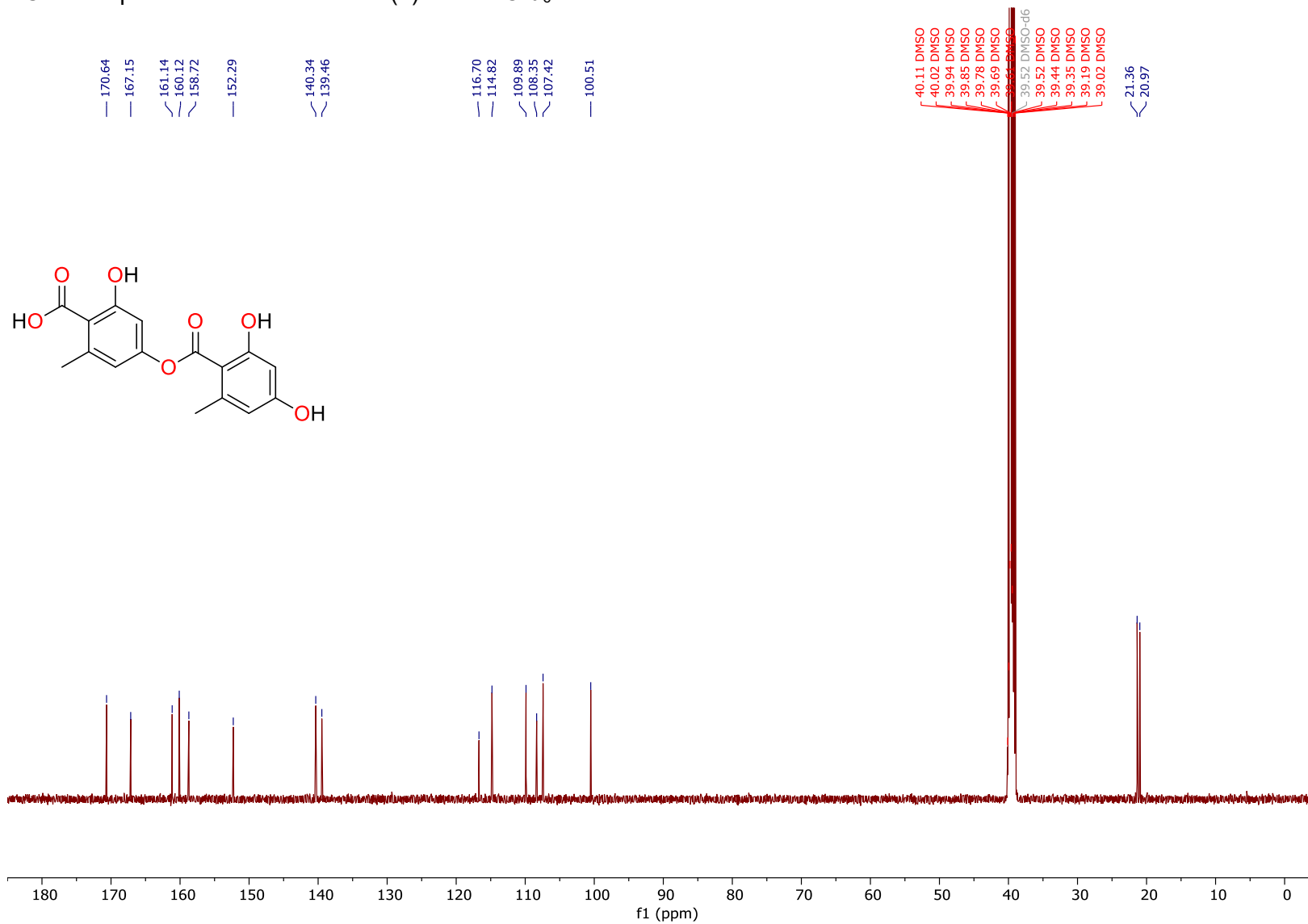
¹H NMR spectrum of lecanoric acid (1) in DMSO-d₆



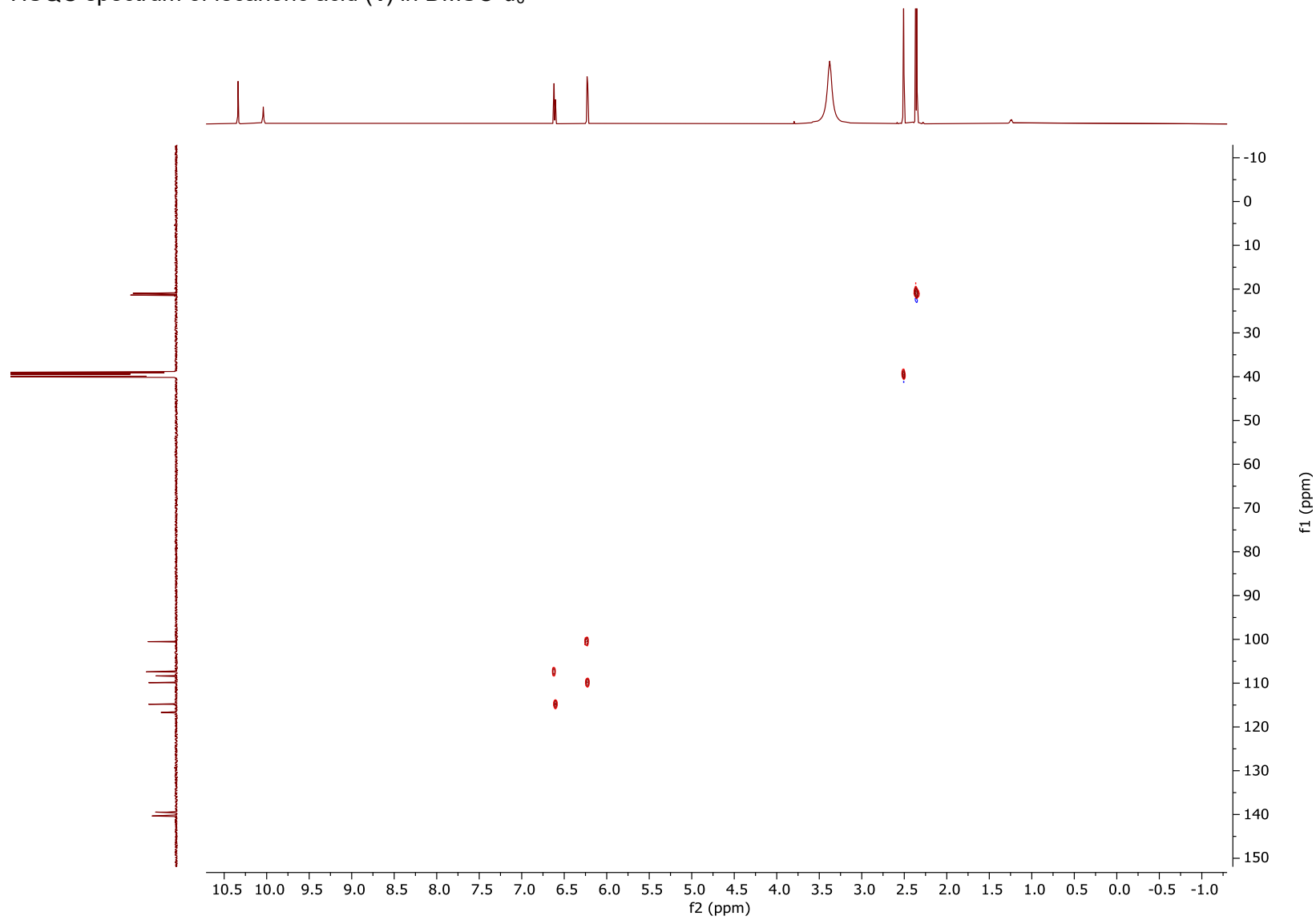
^1H NMR spectrum of lecanoric acid (**1**) in $\text{DMSO-}d_6$ and D_2O



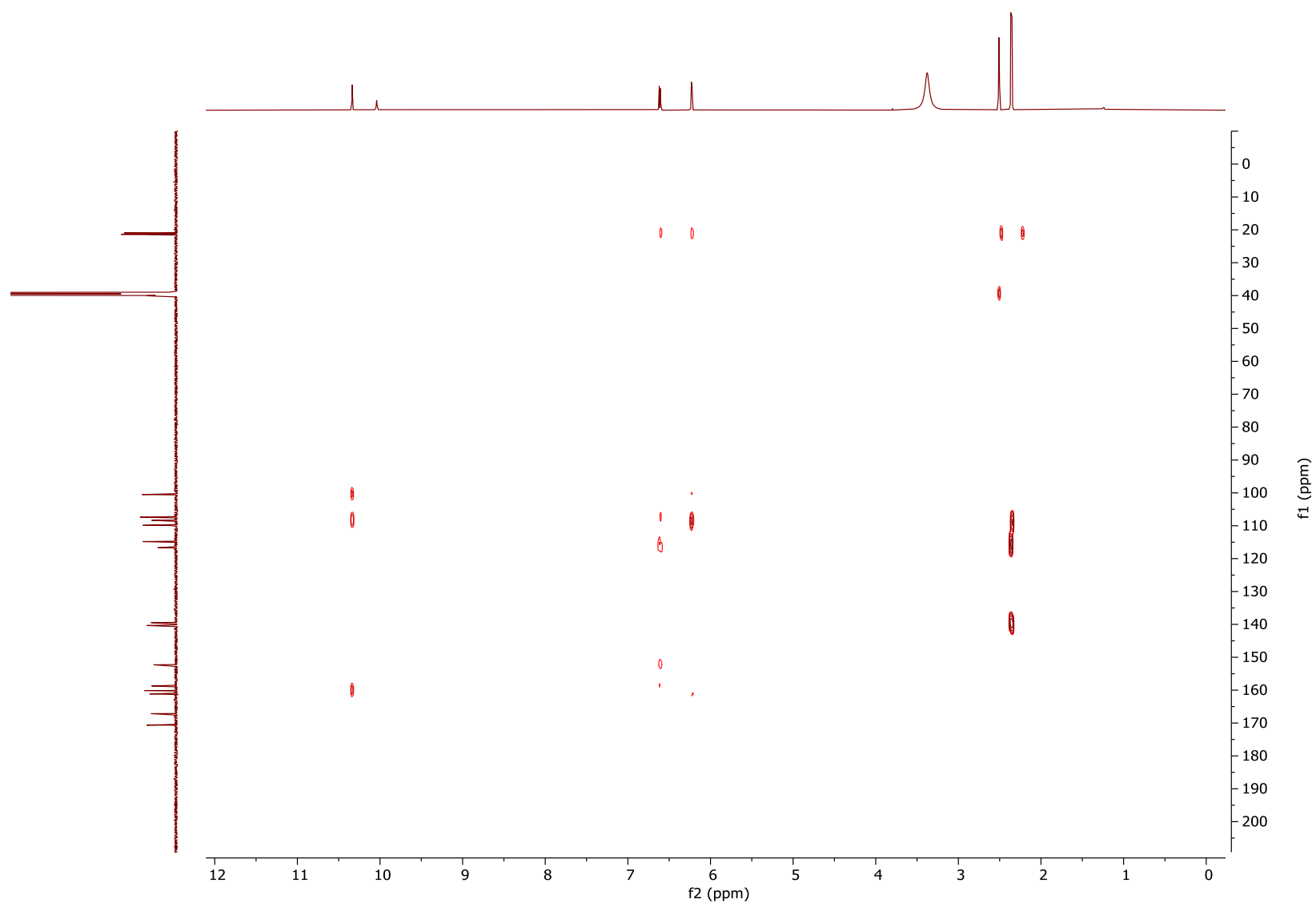
¹³C NMR spectrum of lecanoric acid (**1**) in DMSO-*d*₆



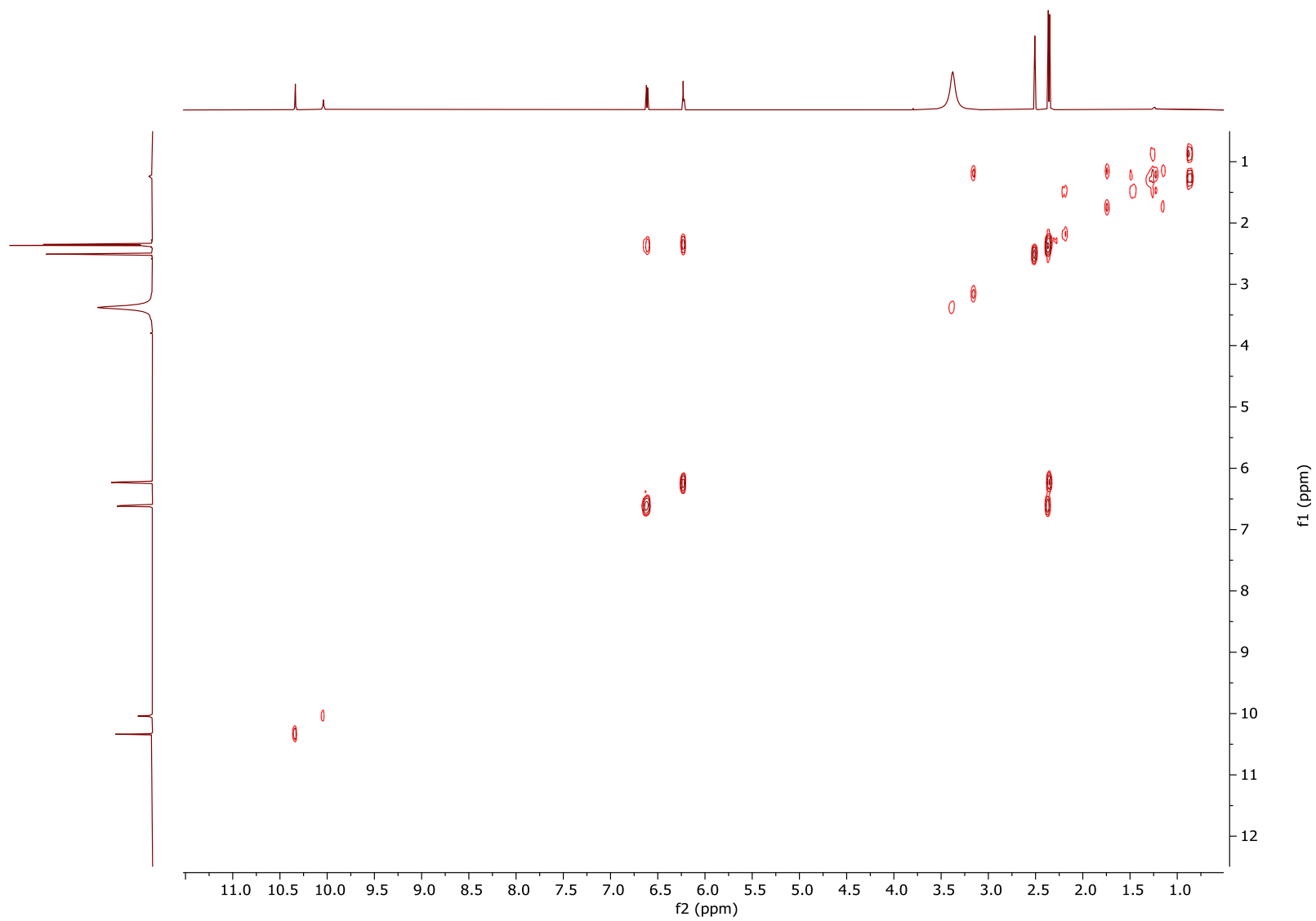
HSQC spectrum of lecanoric acid (**1**) in DMSO- d_6



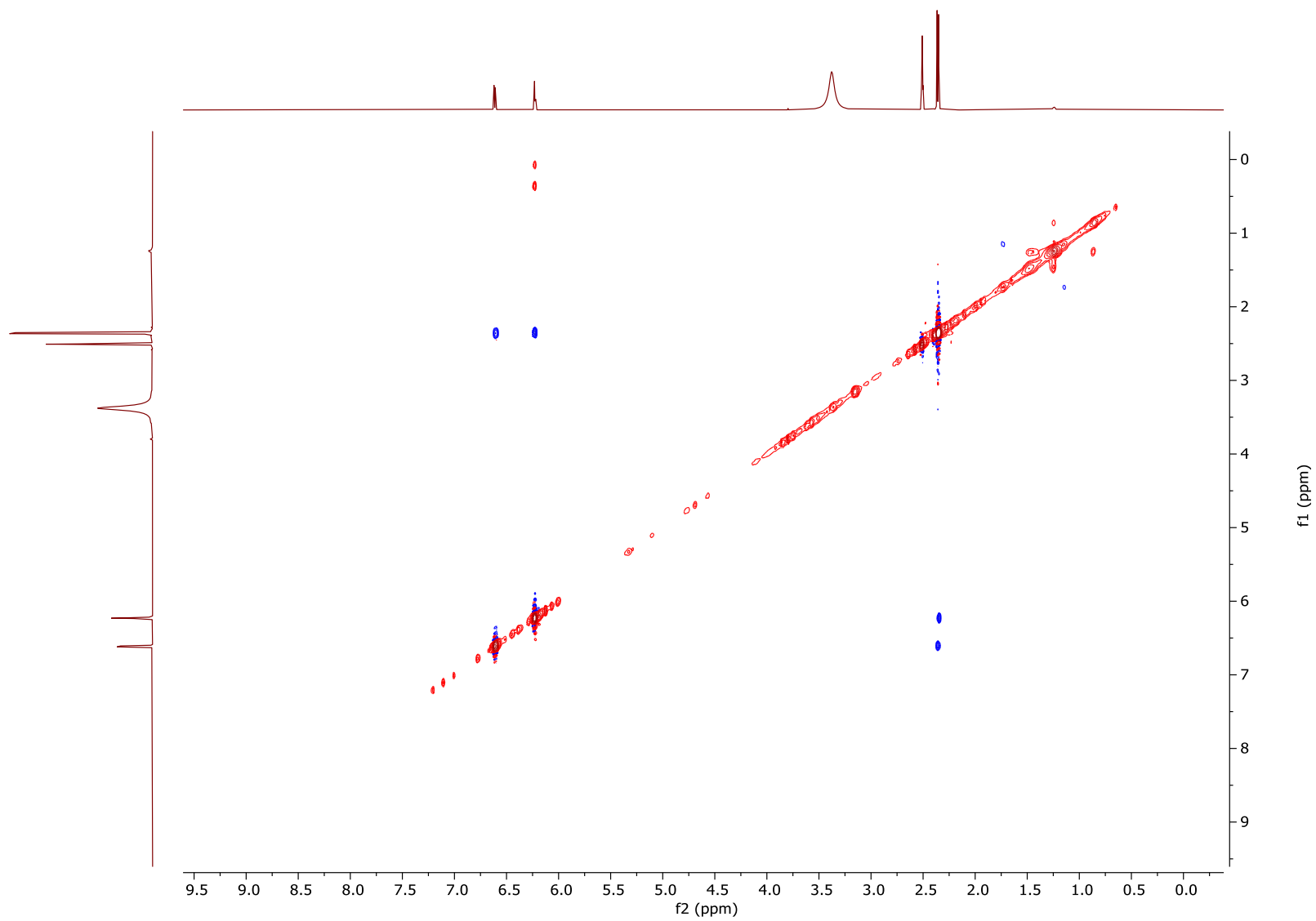
HMBC spectrum of lecanoric acid (**1**) in DMSO- d_6



COSY spectrum of lecanoric acid (1) in DMSO- d_6



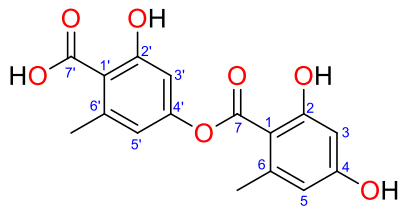
ROESY spectrum of lecanoric acid (**1**) in DMSO- d_6



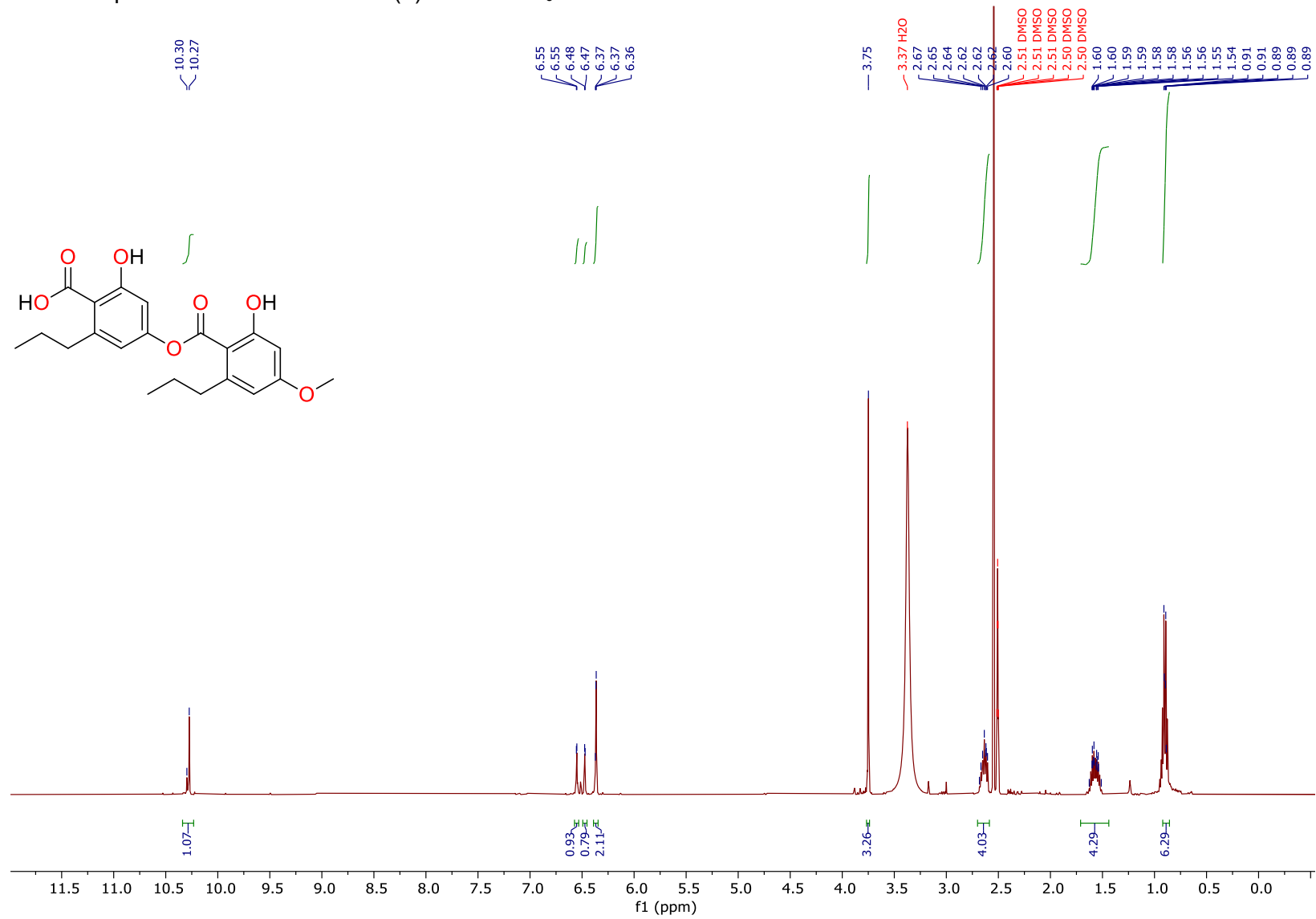
NMR data table for lecanoric acid (**1**) in DMSO- d_6^a

Position	δ_H (mult., J in Hz)	δ_C , type	COSY	HMBC	ROESY
1		108.4, C			
2		160.1, C			
2-OH	10.34 (s)			1,2,3,4,5	
3	6.23 (d, 2.3)	100.5, CH	5,6-Me ^w	1,2,4,5,6-Me	
4		161.1, C			
4-OH	10.04 (brs)				
5	6.22 (dq, 2.3, 0.7)	109.9, CH	3,6-Me	1,2,3,4,6-Me	6-Me
6		140.3, C			
6-Me	2.35 (s)	21.4, CH ₃	3 ^w ,5	1,2 ^w ,5,6,7 ^w	5
7		167.2, C			
1'		116.7, C			
2'		158.7, C			
2'-OH	b				
3'	6.62 (d, 2.3)	107.4, CH	5',6'-Me ^w	1',2',4',5'	
4'		152.3, C			
5'	6.60 (dq, 2.3, 0.7)	114.8, CH	3',6'-Me	1',3',4',6'-Me	6'-Me
6'		139.5, C			
6'-Me	2.37 (s)	21.0, CH ₃	3' ^w ,5'	1',2' ^w ,5',6',7' ^w	5'
7'		170.6, C			
7'-OH	b				

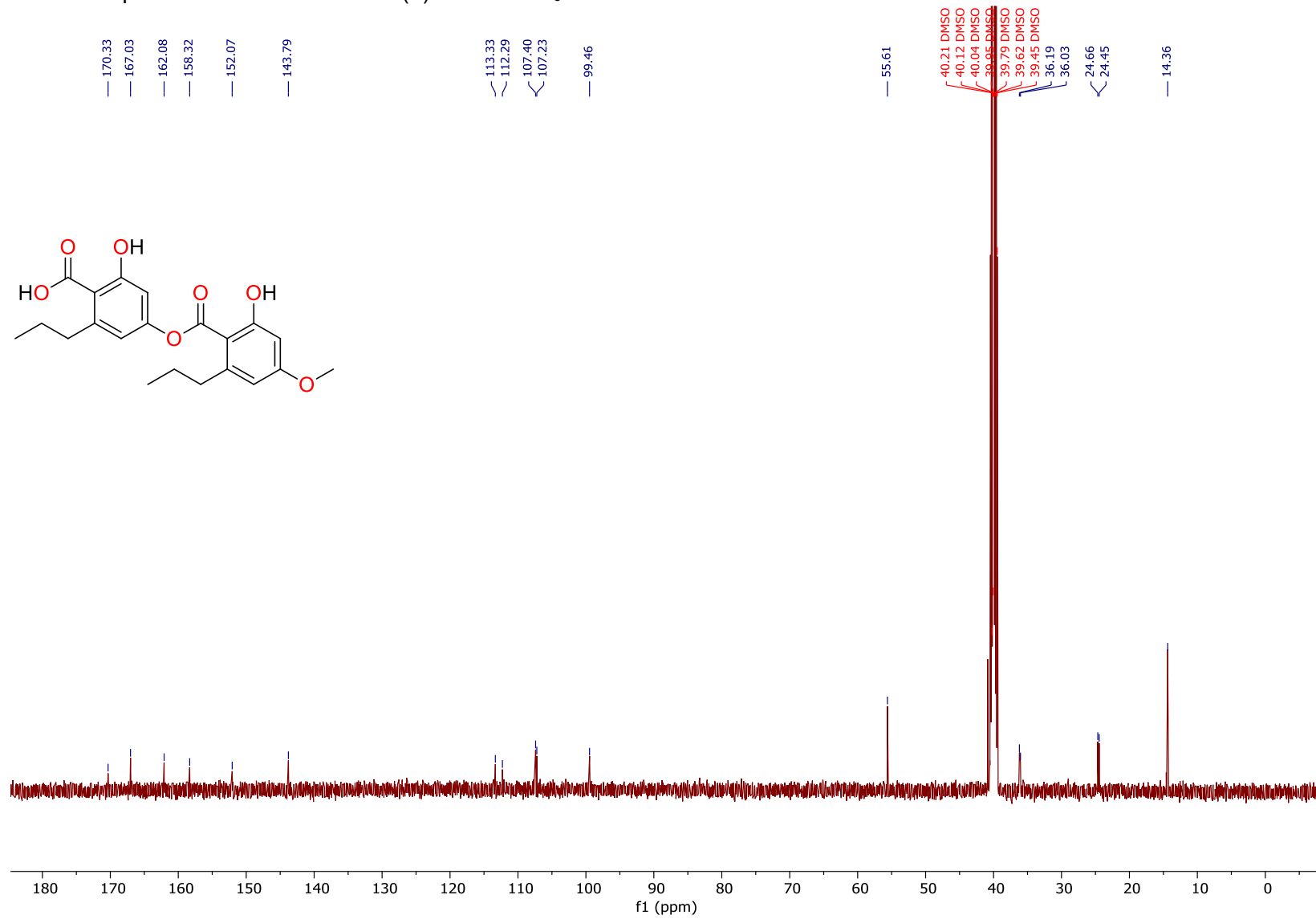
^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Not observed; ^w Weak correlation.



¹H NMR spectrum of divaricatic acid (**2**) in DMSO-d₆



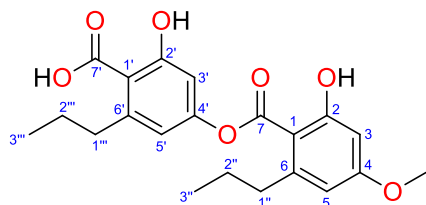
¹³C NMR spectrum of divaricatic acid (2) in DMSO-d₆



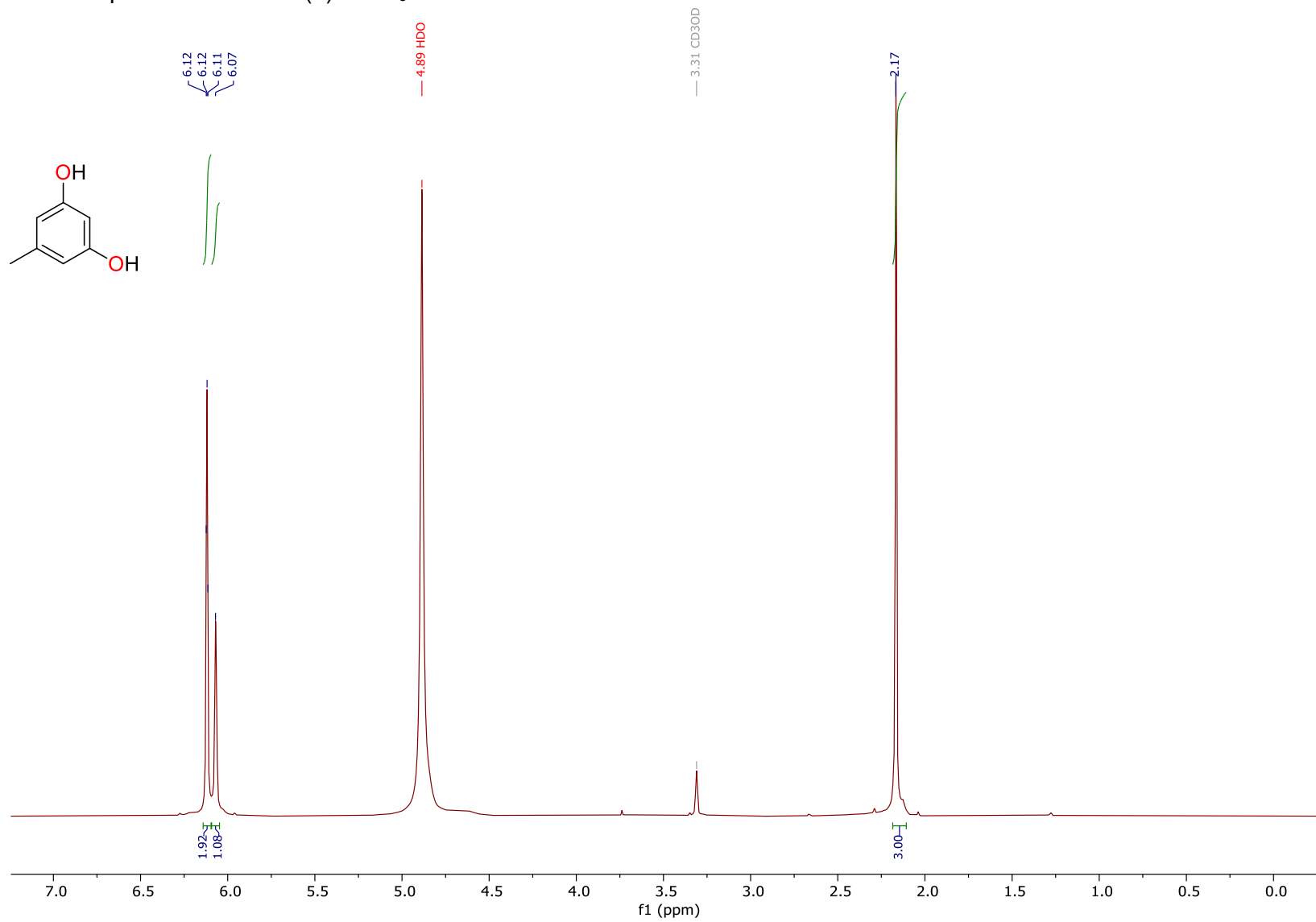
NMR data table for divaricatic acid (**2**) in DMSO- d_6^a

Position	δ_H (mult., J in Hz)	δ_C , type
1		107.2, C
2		^b
2-OH	10.30 (s)	
3	6.47 (d, 2.2)	99.5, CH
4		152.1, C
4-OMe	3.75 (s)	55.6, CH ₃
5	6.55 (d, 2.2)	113.3, CH
6		^b
7		167.0, C
1'		112.3, C
2'		158.3, C
2'-OH	10.27 (s)	
3'	6.36 (d, 2.4)	107.4, CH
4'		162.1, C
5'	6.37 (d, 2.4)	^b
6'		143.8, C
7'		170.3, C
1''	2.60 (t, 7.8)	36.0, CH ₂
2''	1.51–1.63 (m)	24.5, CH ₂
3''	0.89 (t, 7.3)	14.4, CH ₃
1'''	2.64 (t, 7.8)	36.2, CH ₂
2'''	1.51–1.63 (m)	24.7, CH ₂
3'''	0.91 (t, 7.3)	14.4, CH ₃

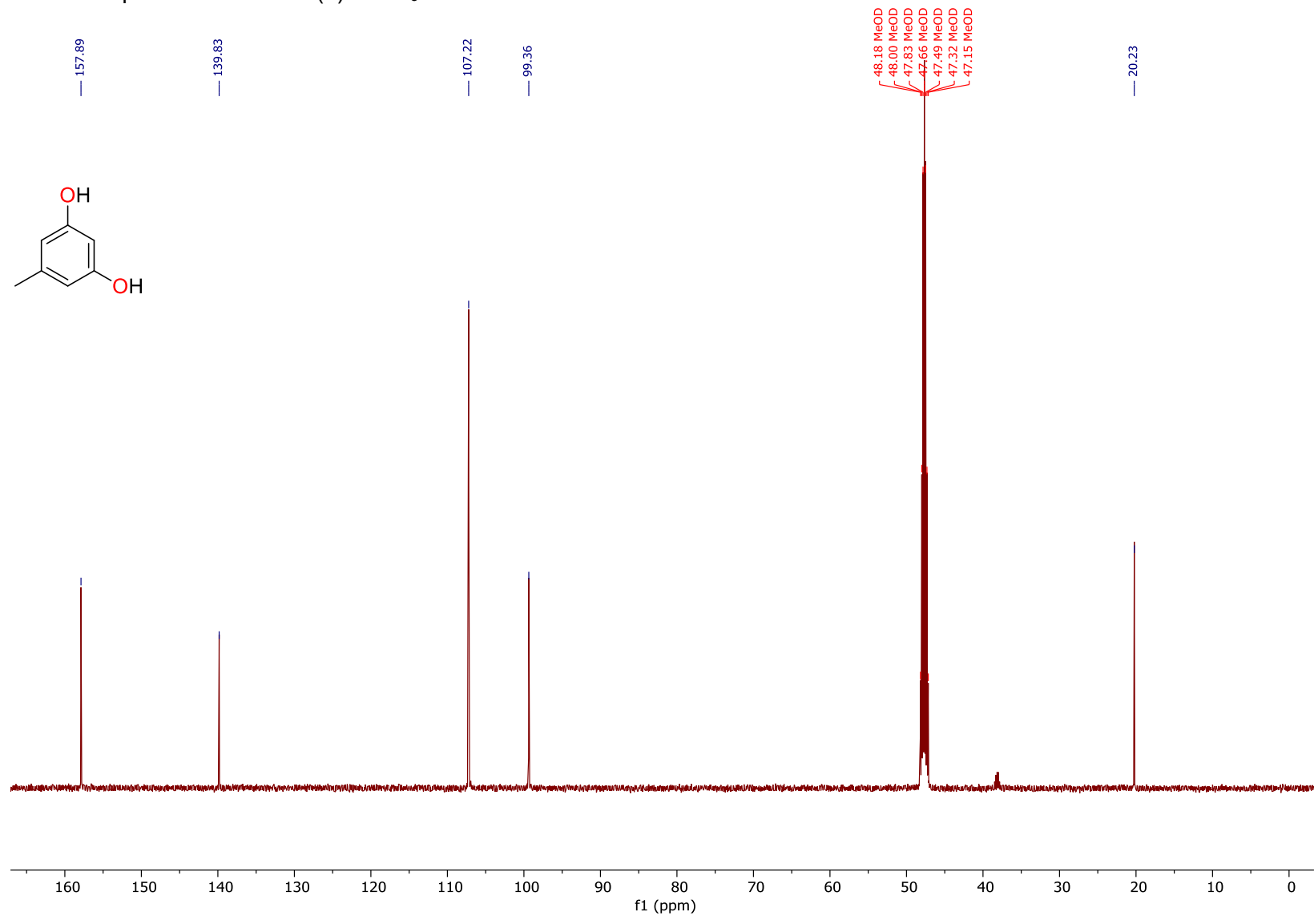
^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Not observed.



^1H NMR spectrum of orcinol (**3**) in CD_3OD



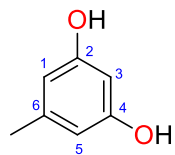
¹³C NMR spectrum of orcinol (**3**) in CD₃OD



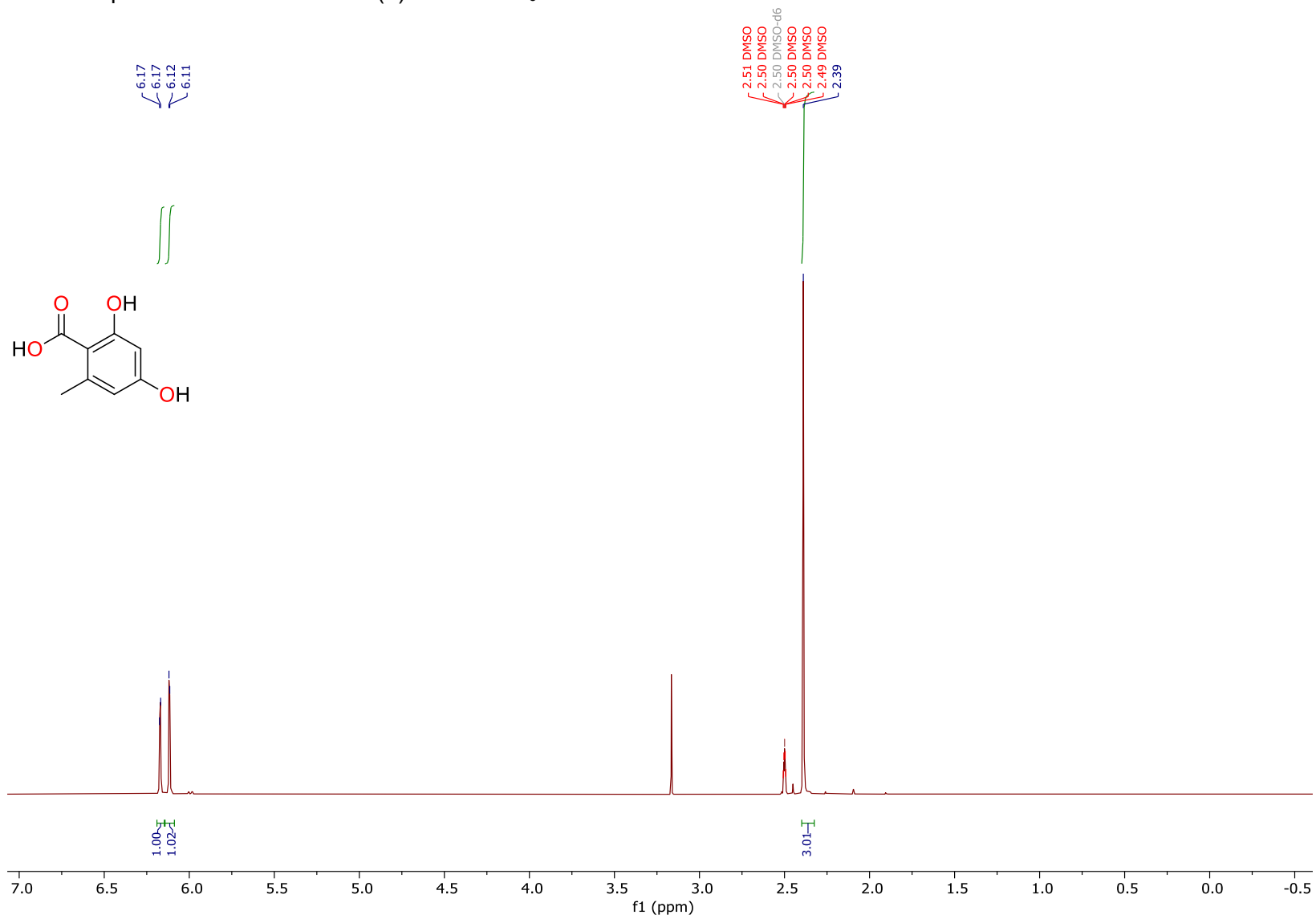
NMR data table for orcinol (**3**) in CD₃OD^a

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type
1	6.12, d (2.4)	107.2, CH
2		157.9, C
2-OH		
3	6.07, t (2.3)	99.4, CH
4		157.9, C
4-OH		
5	6.12, d (2.4)	107.2, CH
6		139.8, C
6-Me	2.17 (s)	20.2, CH ₃

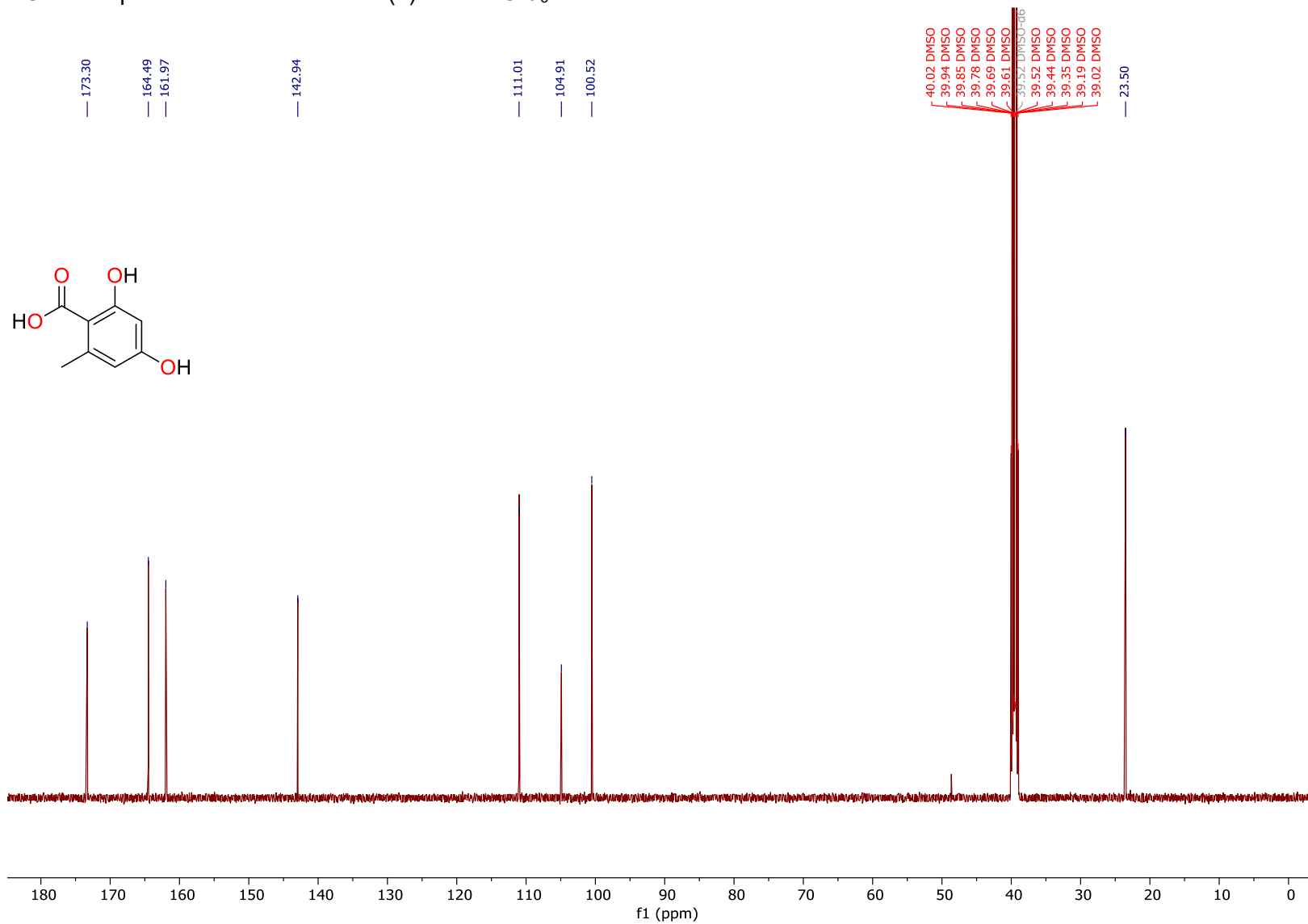
^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR).



¹H NMR spectrum of orsellinic acid (**4**) in DMSO-*d*₆



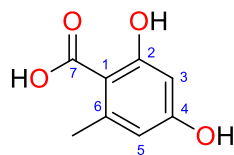
¹³C NMR spectrum of orsellinic acid (**4**) in DMSO-d₆



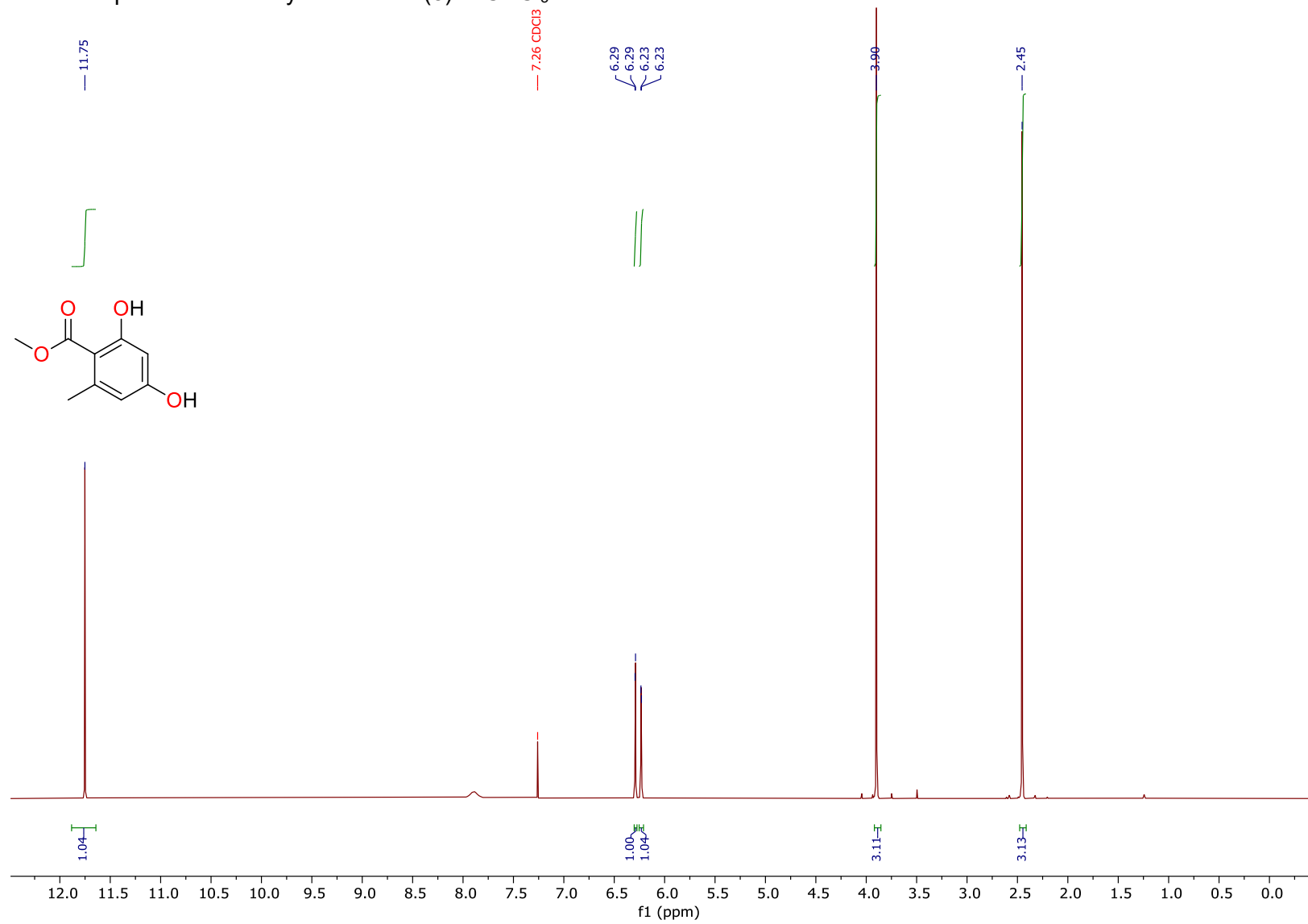
NMR data table for orsellinic acid (**4**) in DMSO- d_6^a

Position	δ_H (mult., J in Hz)	δ_C , type
1		104.9, C
2		162.0, C
2-OH	b	
3	6.17 (d, 2.4)	100.5, CH
4		164.5, C
4-OH	b	
5	6.12 (d, 2.4)	111.0, CH
6		142.9, C
6-Me	2.39 (s)	23.5, CH ₃
7		173.3, C
7-OH	b	

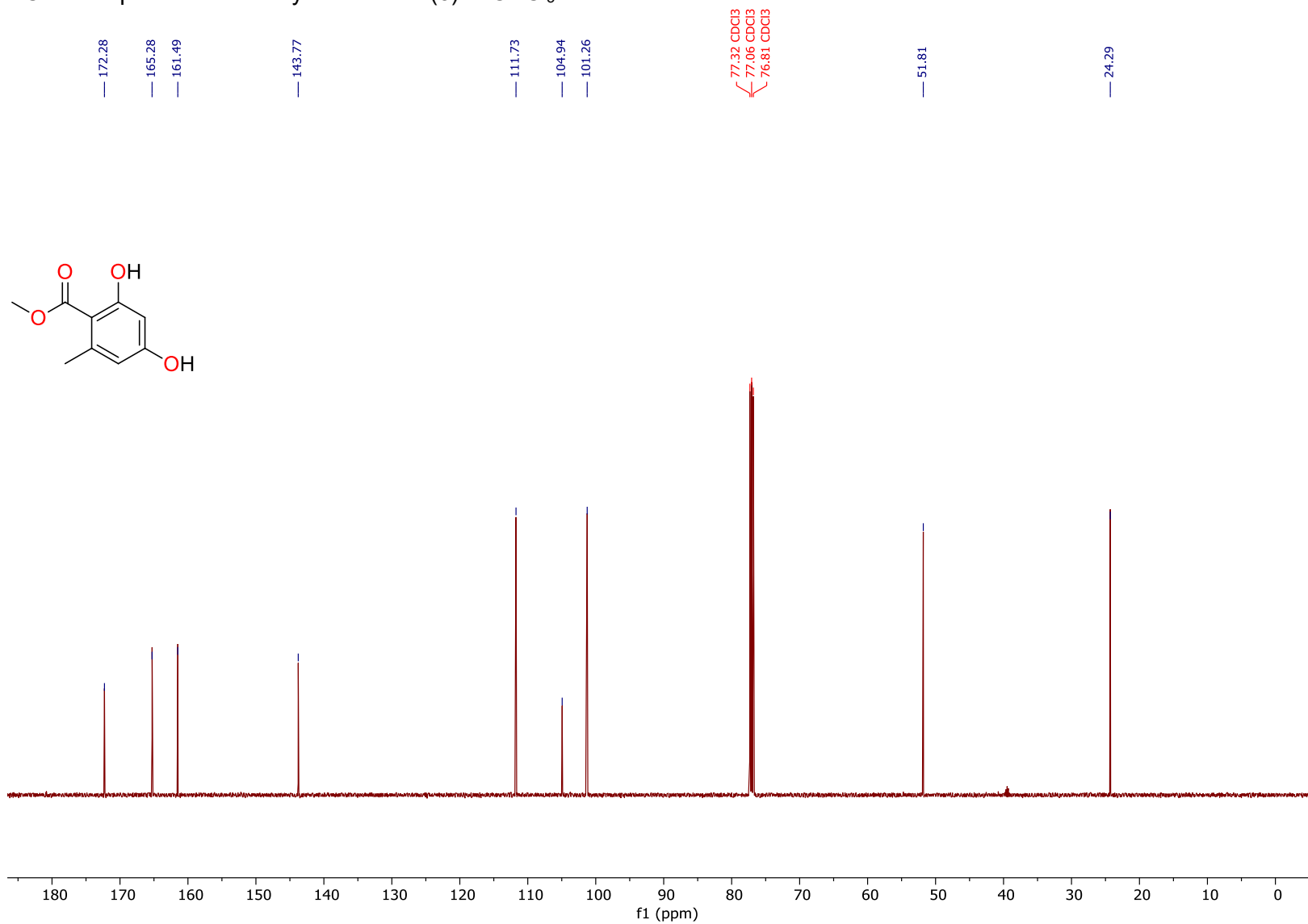
^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Not observed.



^1H NMR spectrum of methyl orsellinate (**5**) in CDCl_3



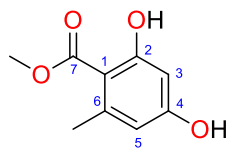
¹³C NMR spectrum of methyl orsellinate (**5**) in CDCl₃



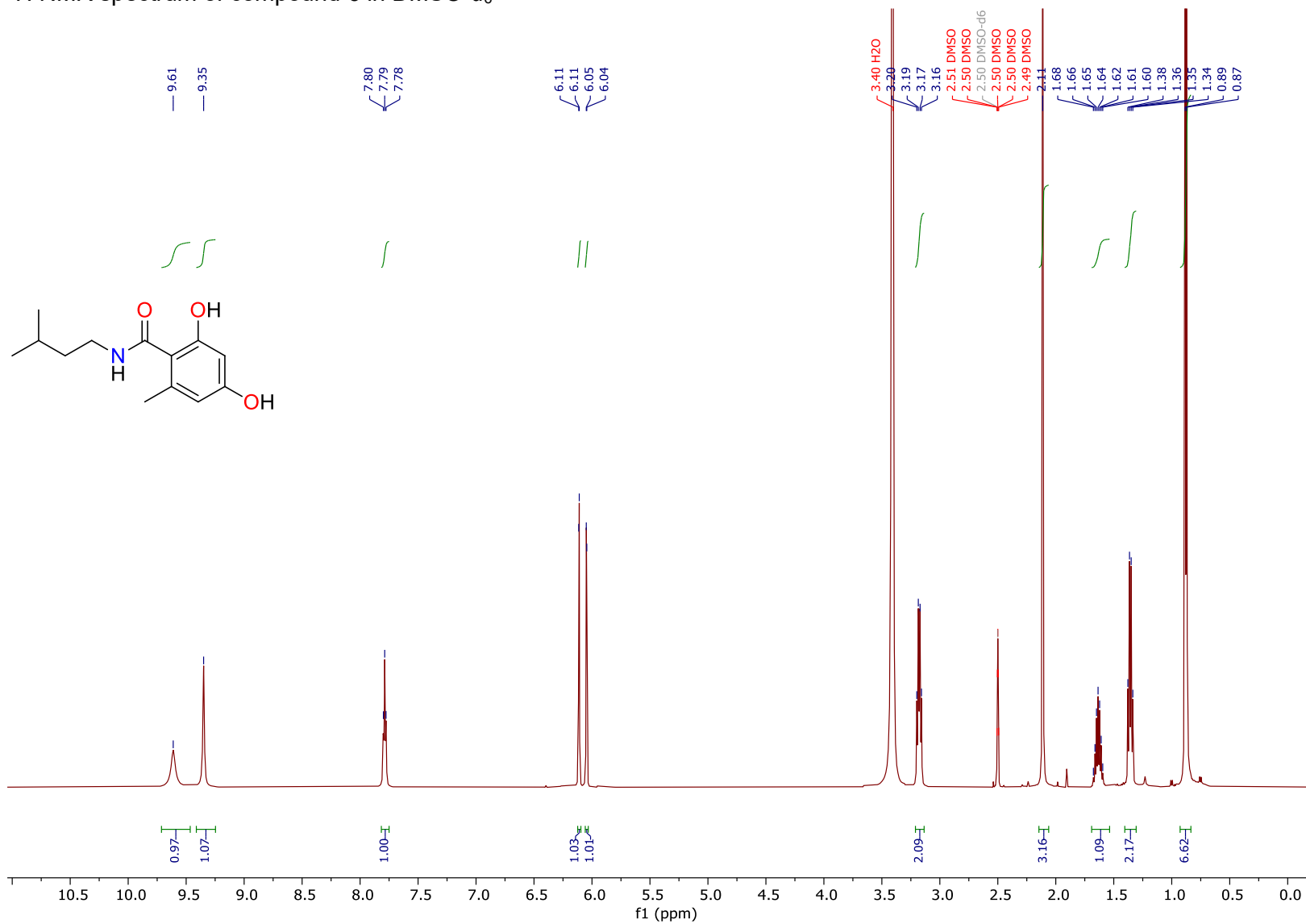
NMR data table for methyl orsellinate (**5**) in CDCl₃^a

Position	δ_H (mult., <i>J</i> in Hz)	δ_C , type
1		101.3, C
2		161.5, C
2-OH	11.75 (s)	
3	6.29 (d, 2.5)	104.9, CH
4		165.3, C
4-OH	^b	
5	6.23 (d, 2.5)	111.7, CH
6		143.8, C
6-Me	2.45 (s)	24.3, CH ₃
7		172.3, C
7-OMe	3.90 (s)	51.8, CH ₃

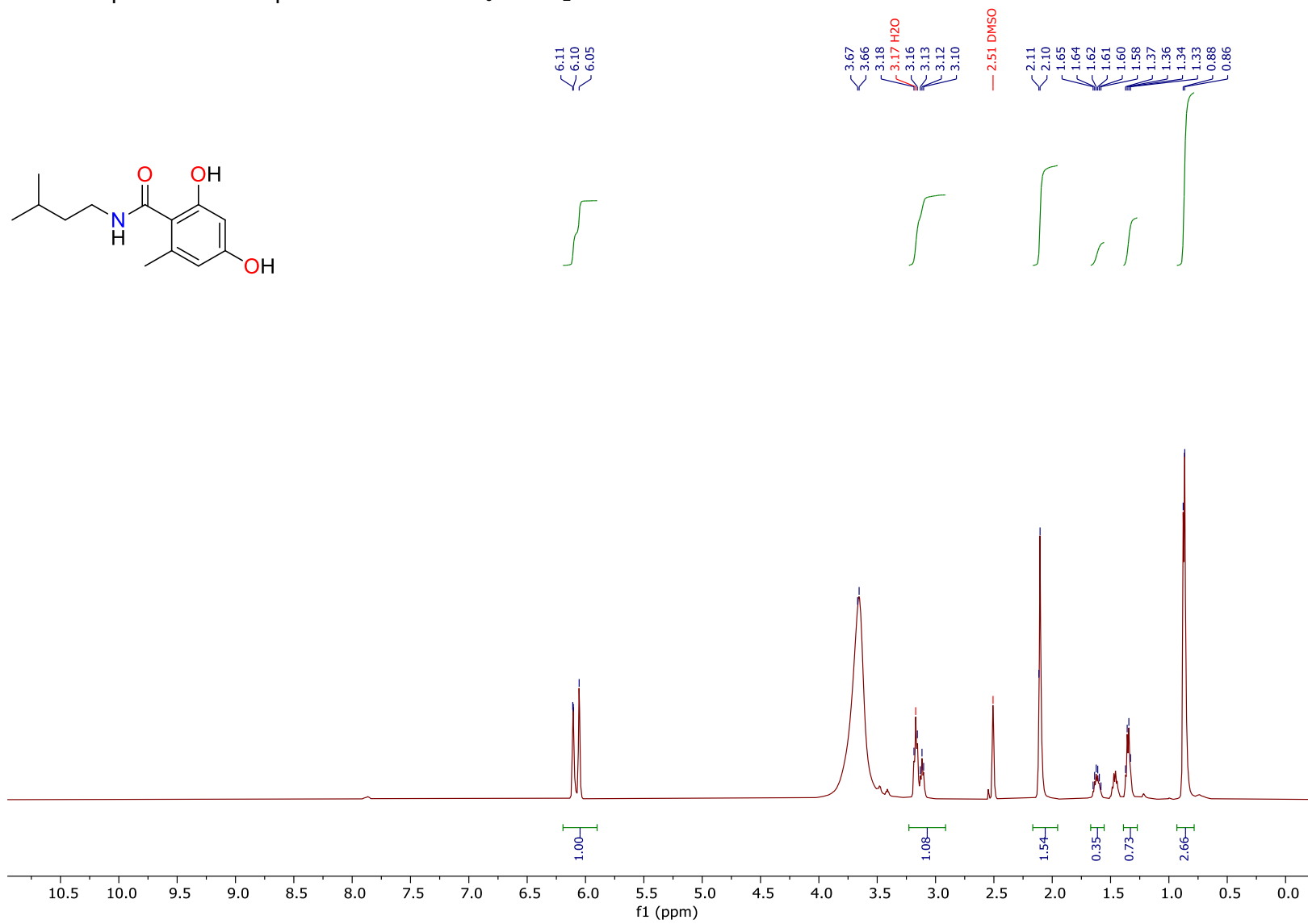
^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Not observed.



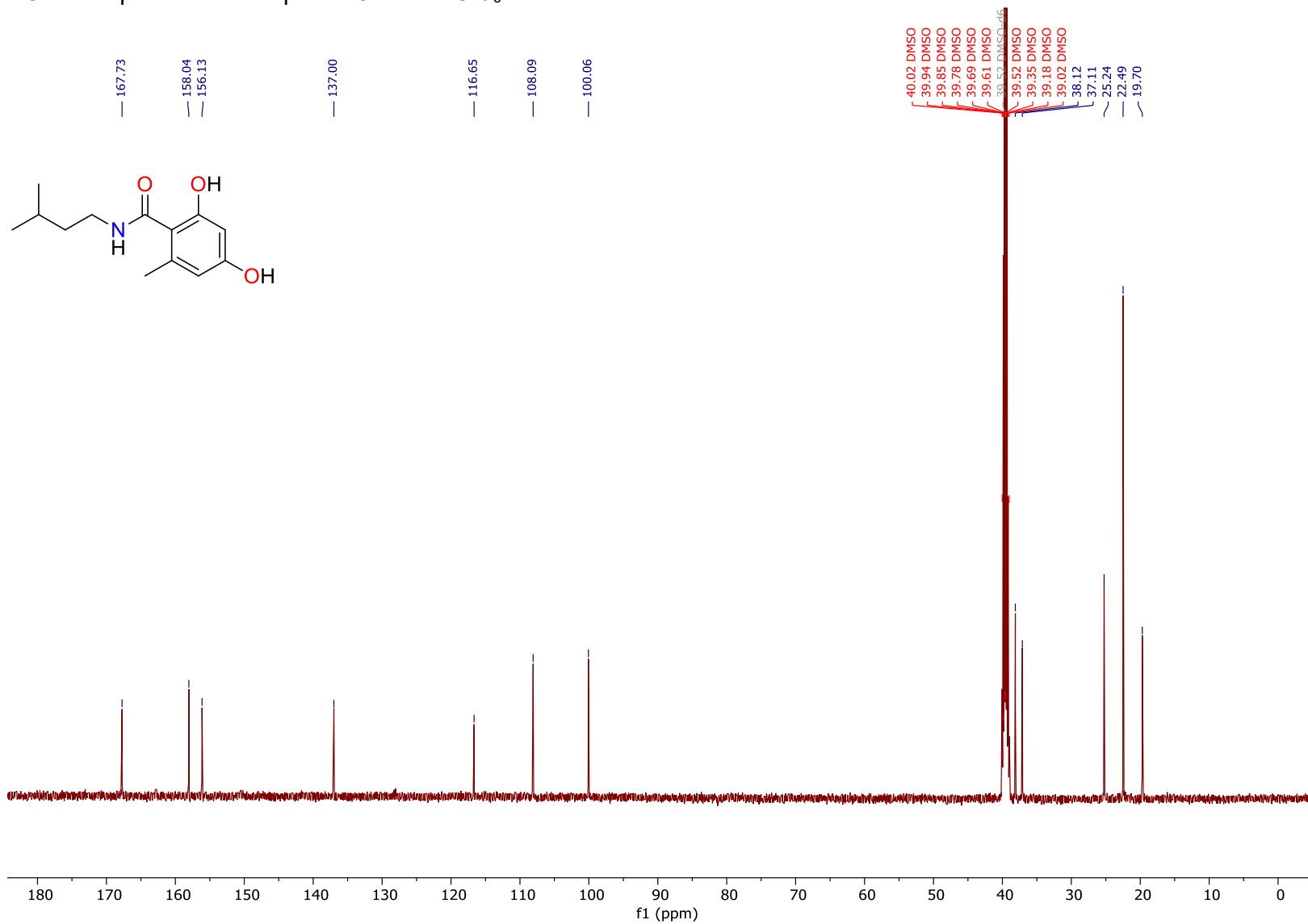
¹H NMR spectrum of compound **6** in DMSO-*d*₆



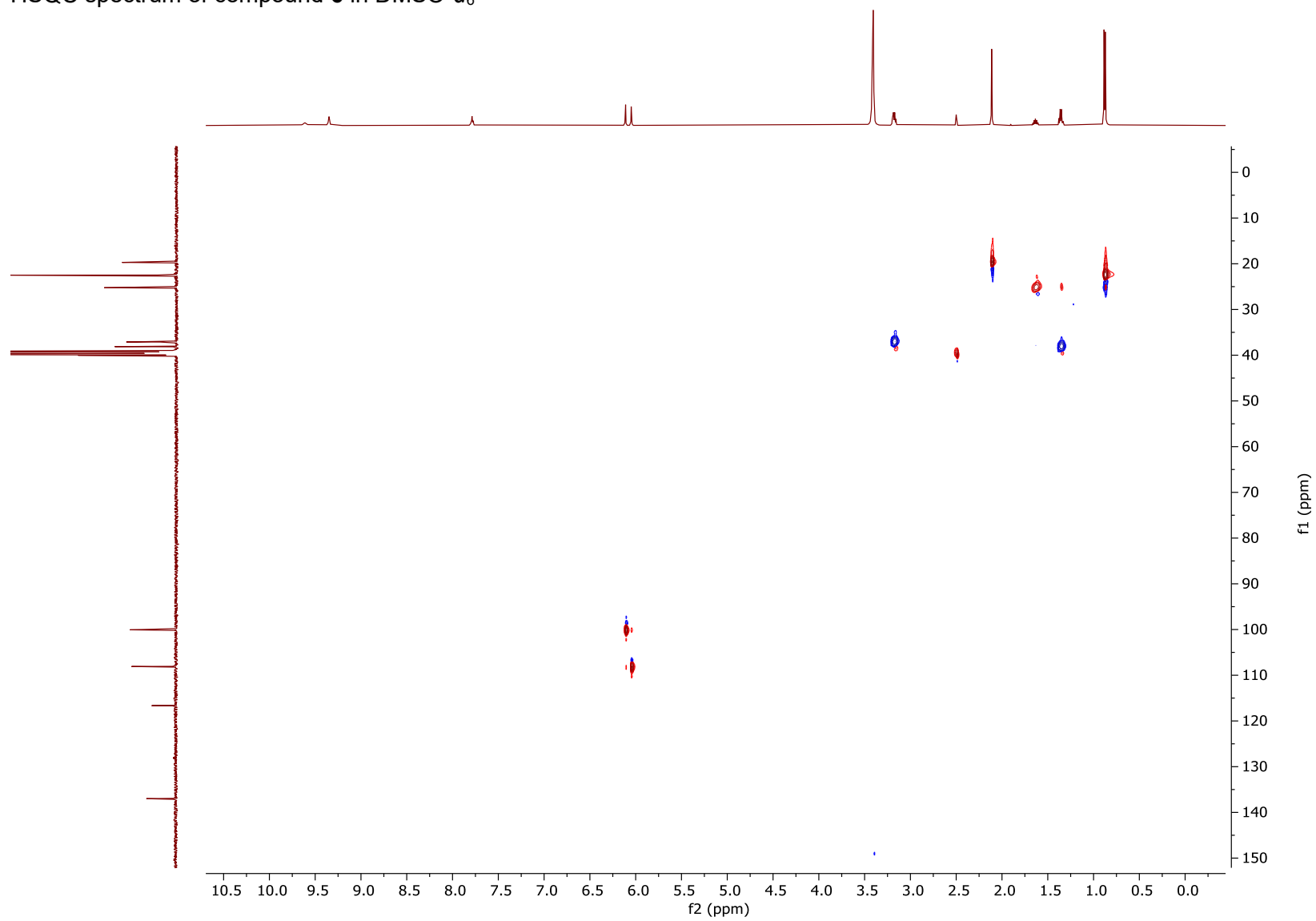
^1H NMR spectrum of compound **6** in $\text{DMSO-}d_6$ and D_2O



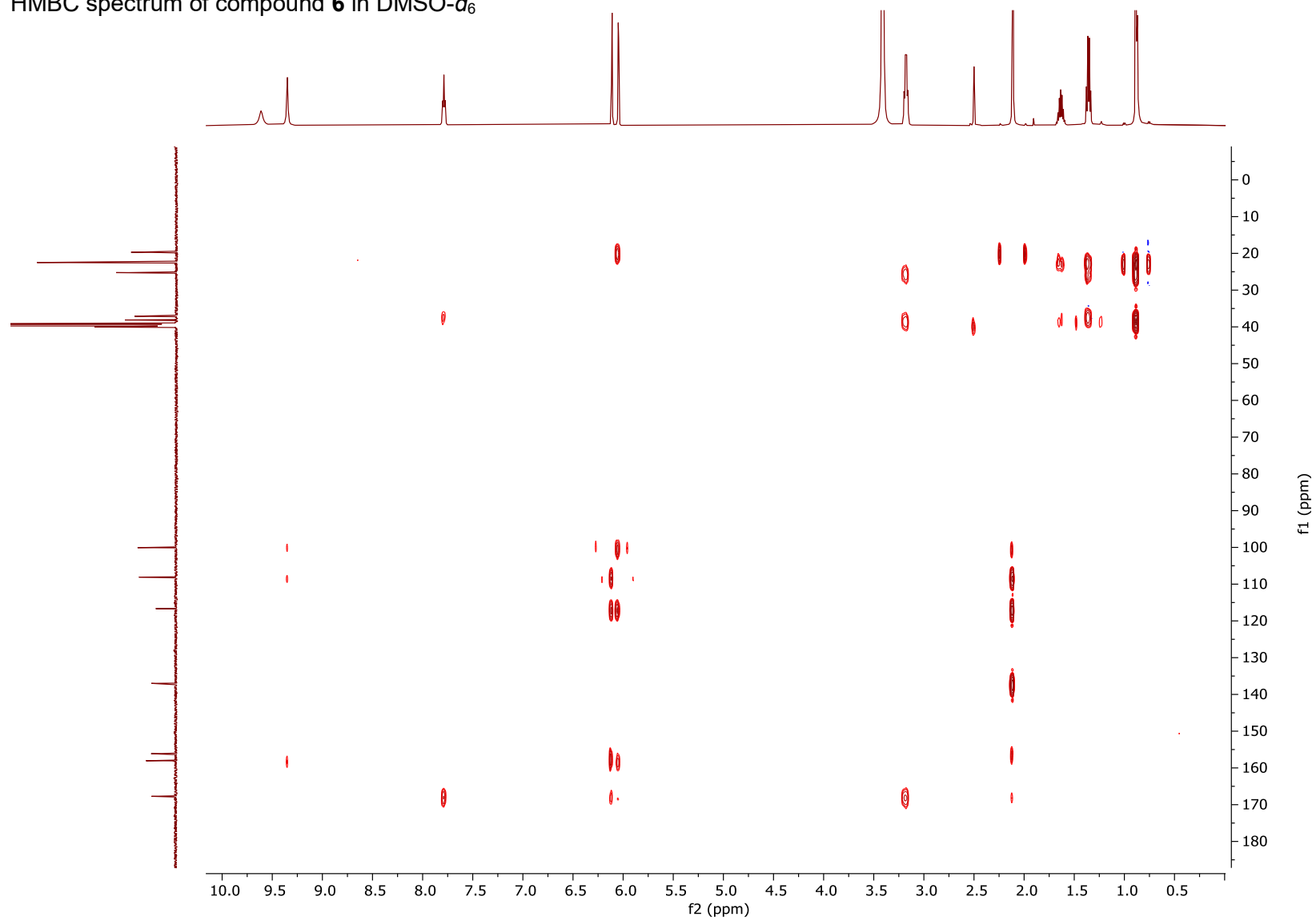
^{13}C NMR spectrum of compound **6** in $\text{DMSO-}d_6$



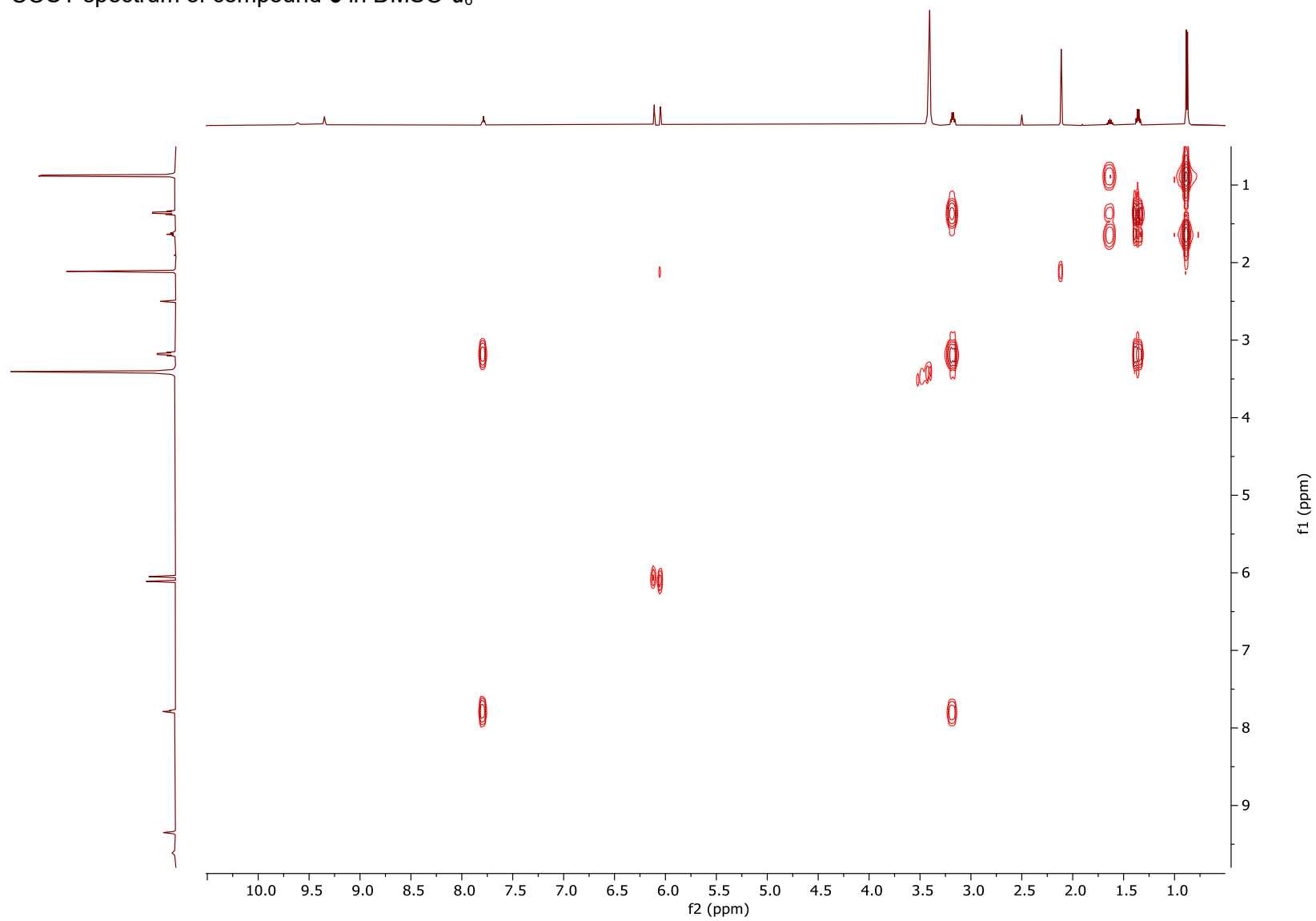
HSQC spectrum of compound **6** in DMSO- d_6



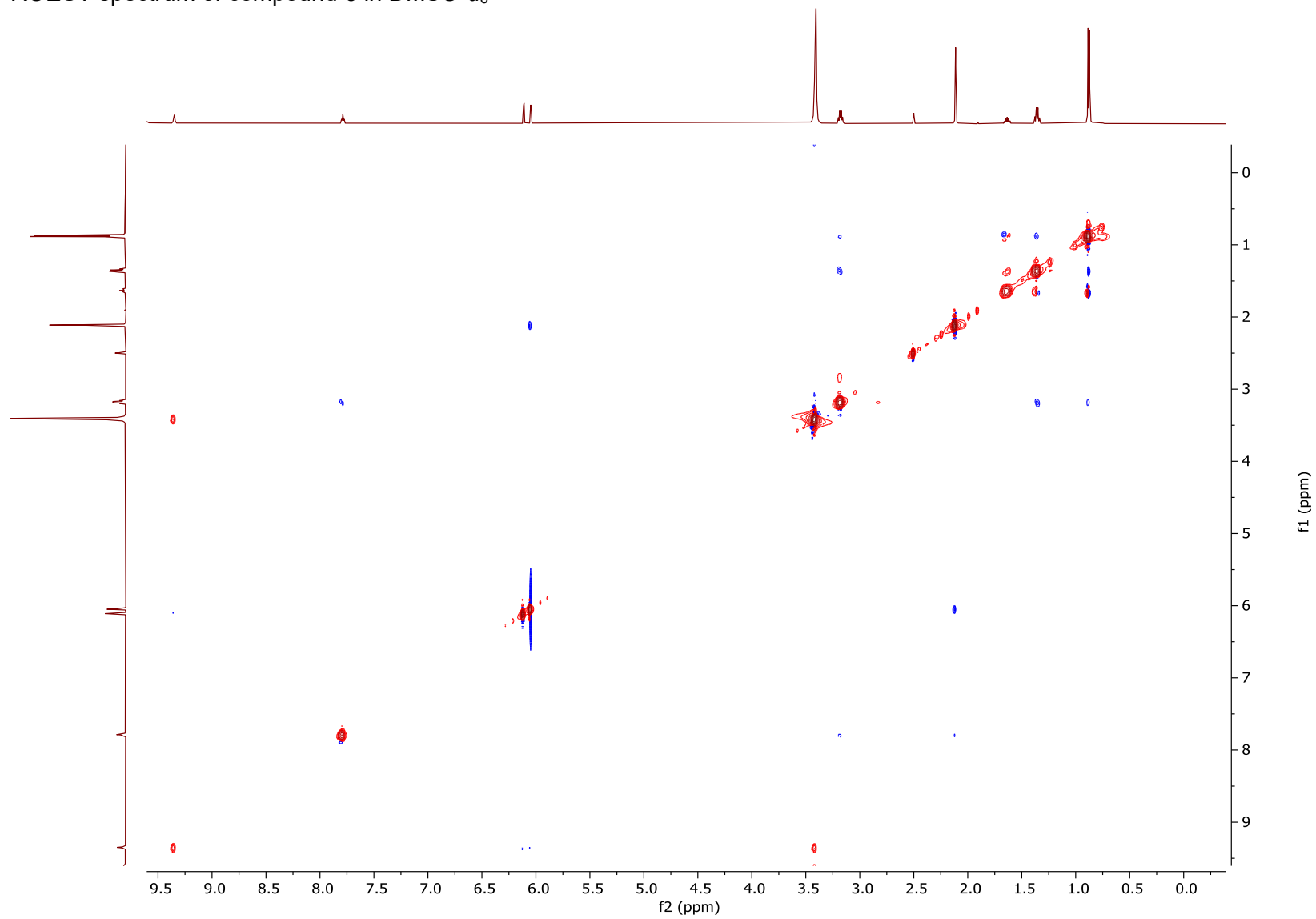
HMBC spectrum of compound **6** in DMSO-*d*₆



COSY spectrum of compound **6** in DMSO- d_6



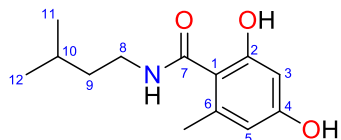
ROESY spectrum of compound **6** in DMSO- d_6



NMR data table for compound **6** in DMSO-*d*₆^a

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type	COSY	HMBC	ROESY
1		116.7, C			
2		156.1, C			
2-OH	9.61 (brs)				3
3	6.11 (d, 2.2)	100.1, CH	5,6-Me ^w	1,4,5,7 ^w	2-OH,4-OH
4		158.0, C			
4-OH	9.35 (brs)			3,4,5	3,5
5	6.04 (d, 2.2)	108.1, CH	3,6-Me ^w	1,3,4,6-Me,7 ^w	4-OH,6-Me
6		137.0, C			
6-Me	2.11 (s)	19.7, CH ₃	3 ^w ,5 ^w	1,2 ^w ,3 ^w ,4,5,6,7 ^w	5,7-NH
7		167.7, C			
7-NH	7.79 (t, 5.6)		8	7,8,9	6-Me,8,9
8	3.17 (dt, 5.6, 6.6)	37.1, CH ₂	7-NH,9	7,8,9,10	7-NH,9,11,12
9	1.35 (dt, 6.6, 7.1)	38.1, CH ₂	8,10	8,10,11,12	8,11,12
10	1.64 (m)	25.2, CH	9,11,12	9,11,12	9,11,12
11	0.87 (d, 6.6)	22.5, CH ₃	10	9,10,12	8,9,10
12	0.89 (d, 6.6)	22.5, CH ₃	10	9,10,11	8,9,10

^aSpectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^wWeak correlation.



High resolution mass spectrum of compound 6

Mass Spectrum SmartFormula Report

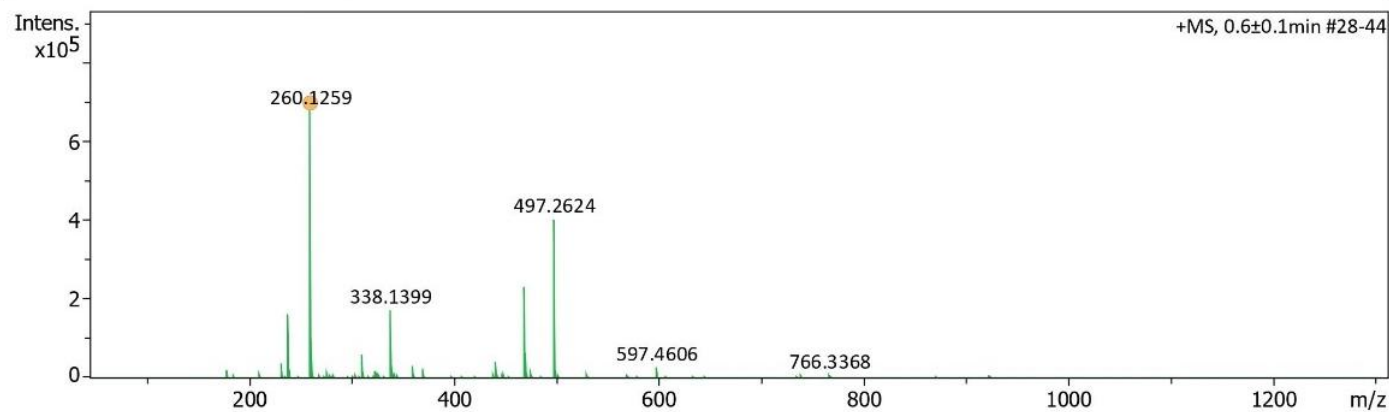
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Comment

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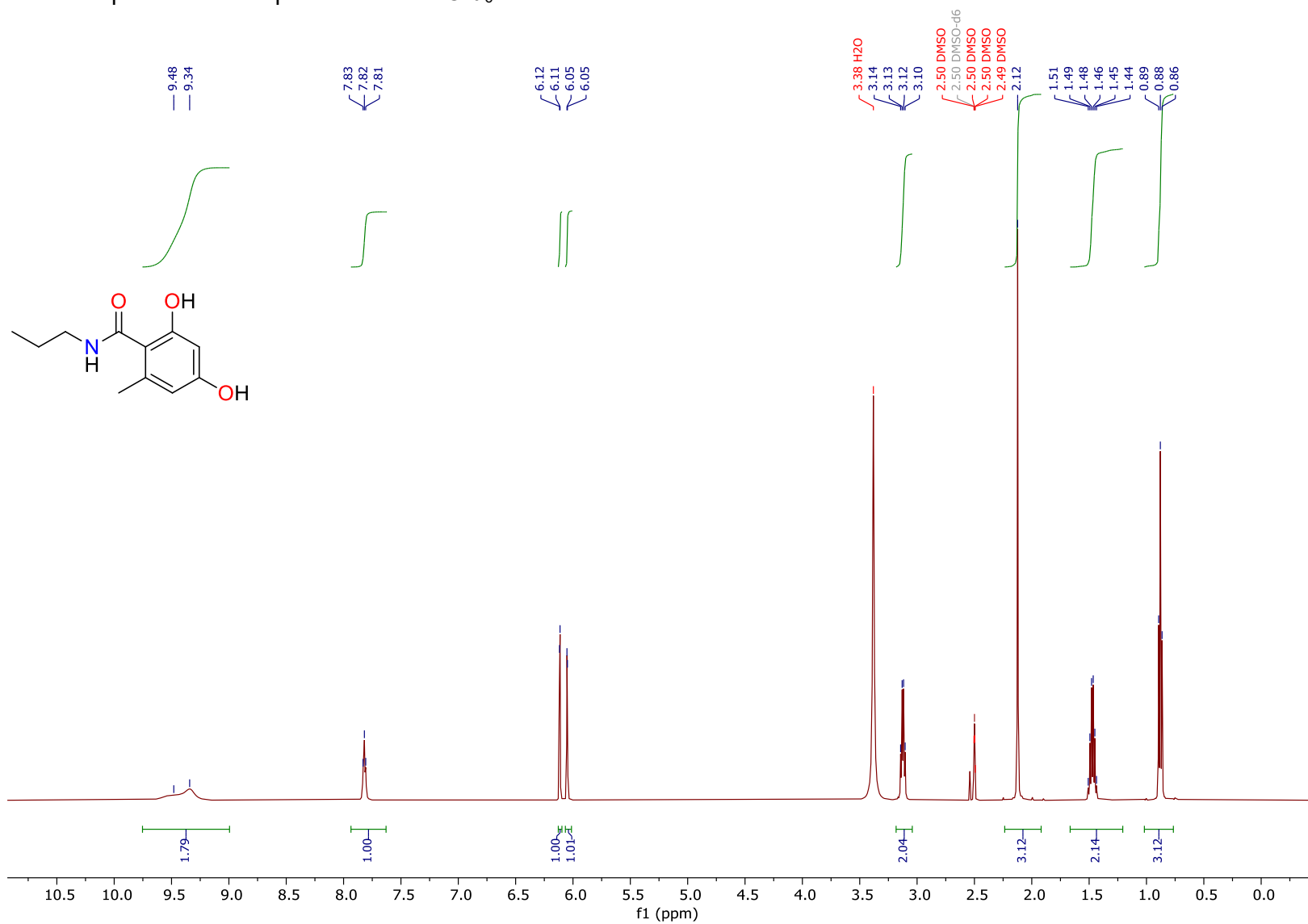
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Instrument maXis II ETD

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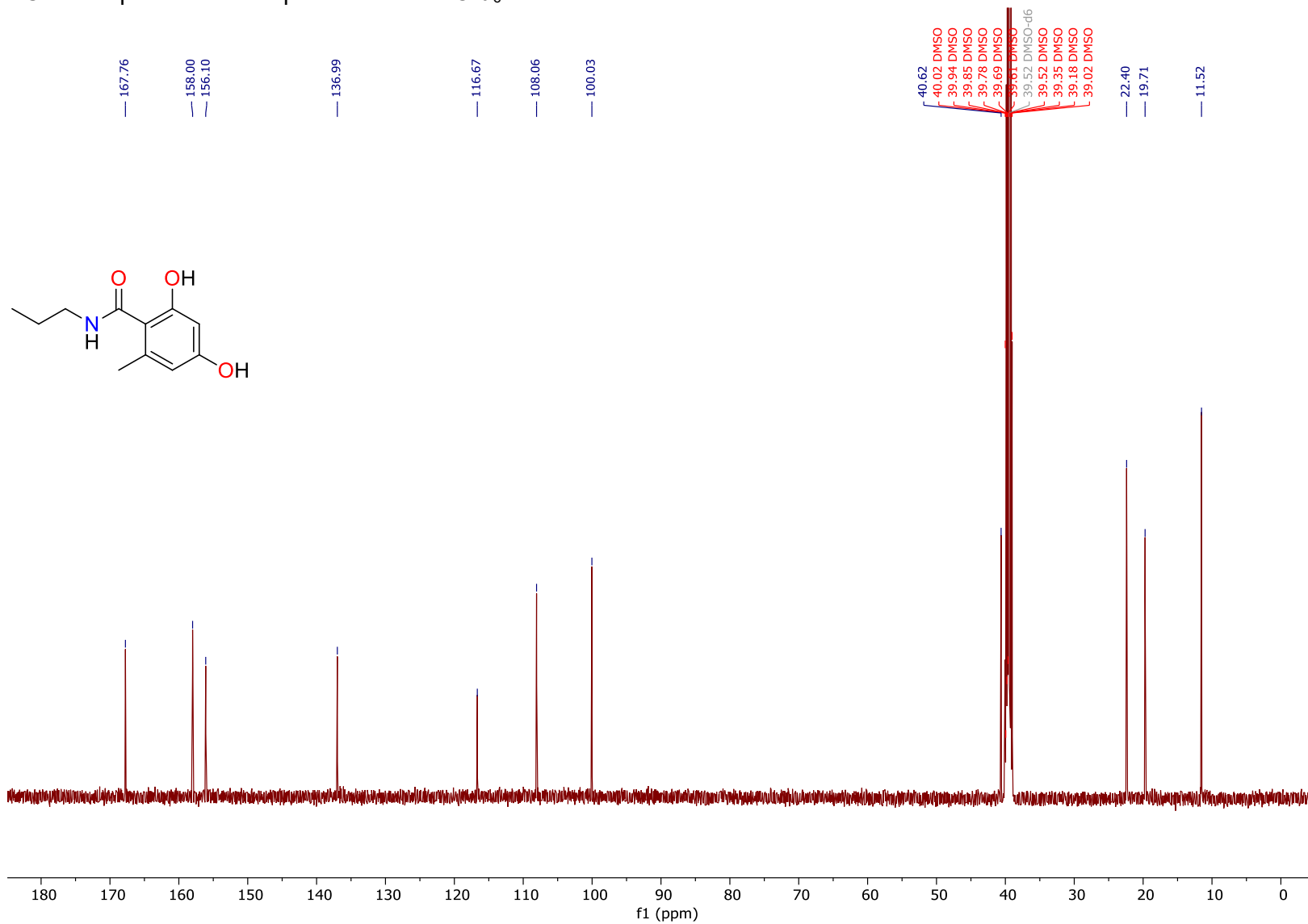


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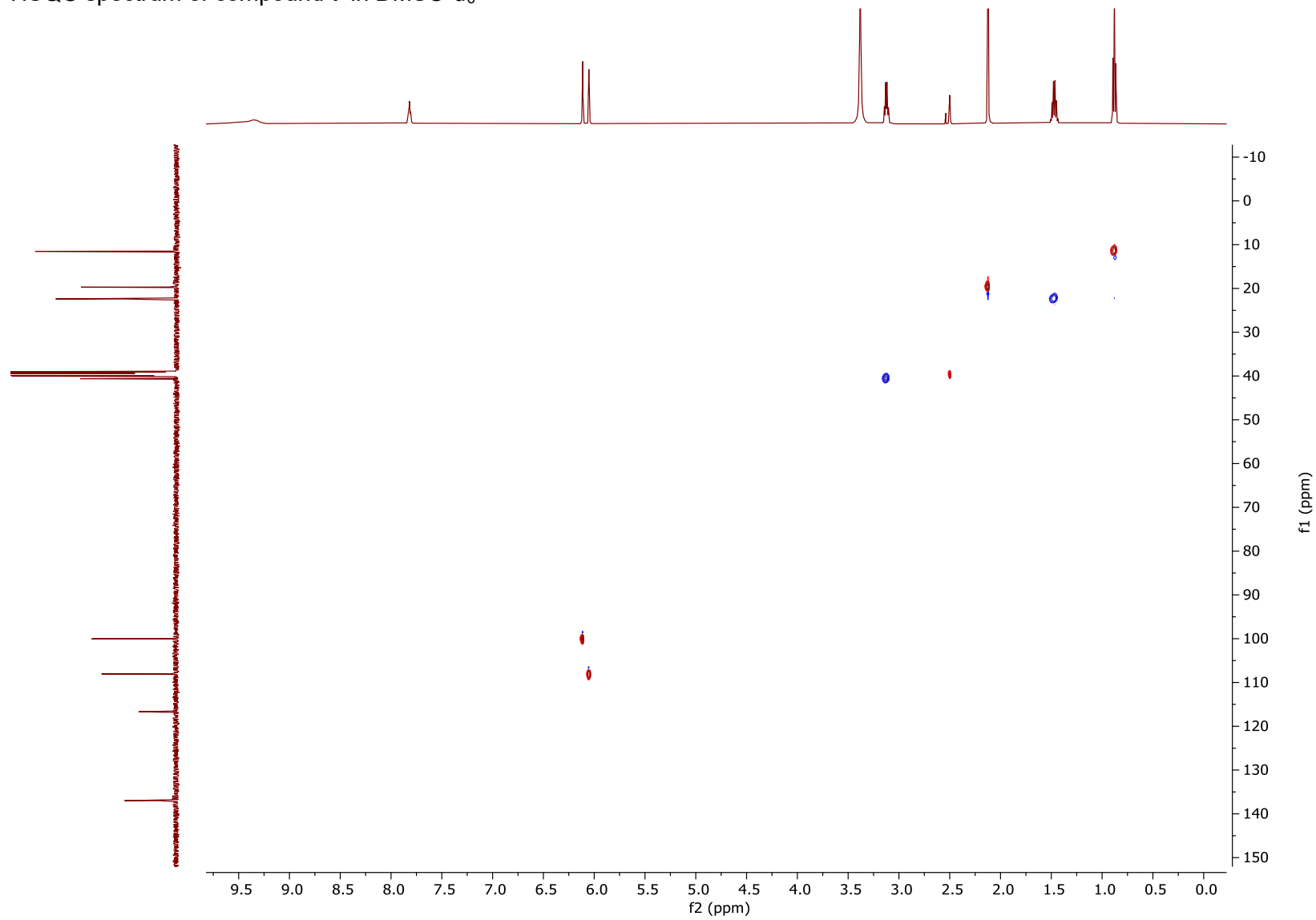
¹H NMR spectrum of compound **7** in DMSO-*d*₆



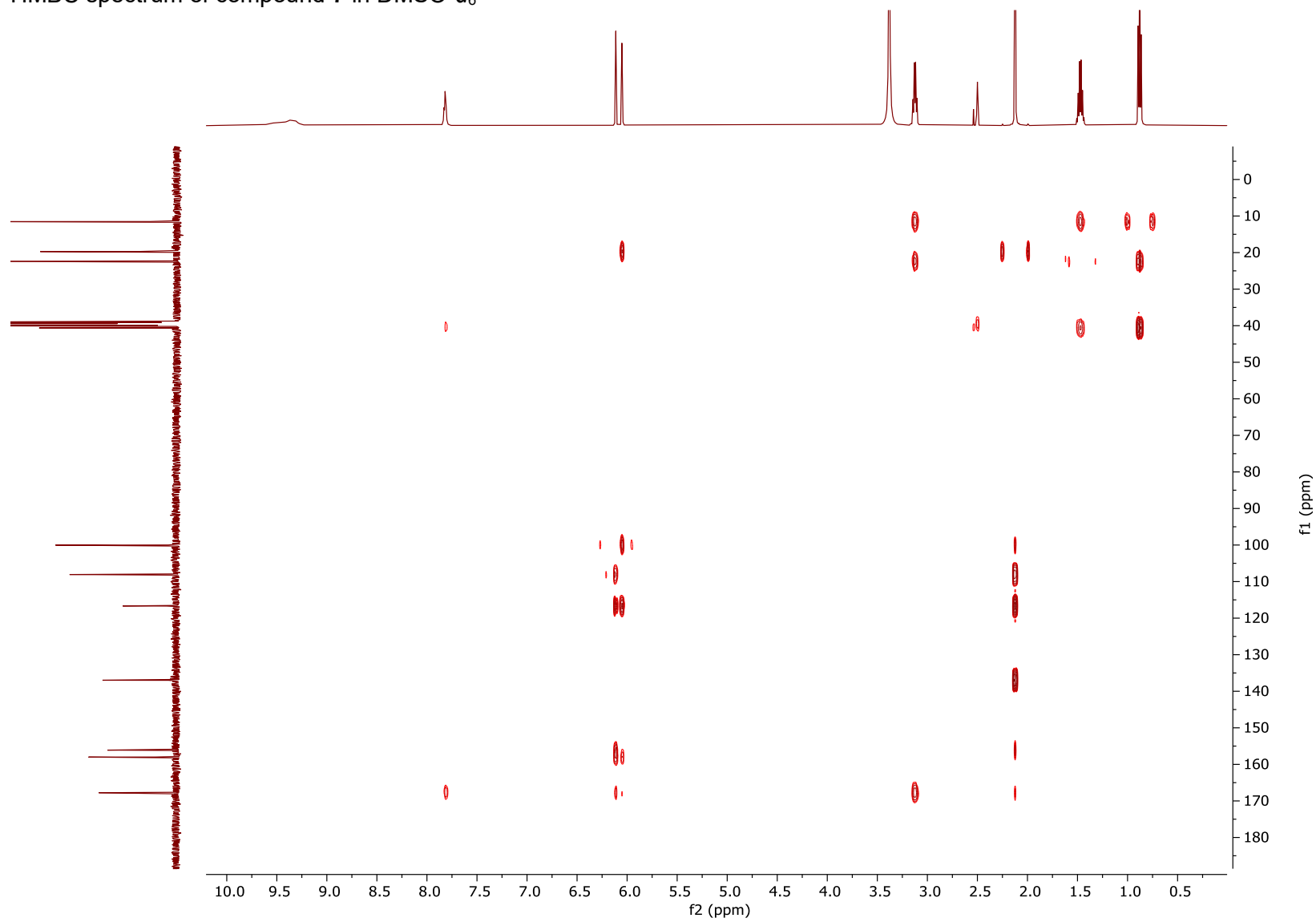
¹³C NMR spectrum of compound **7** in DMSO-*d*₆



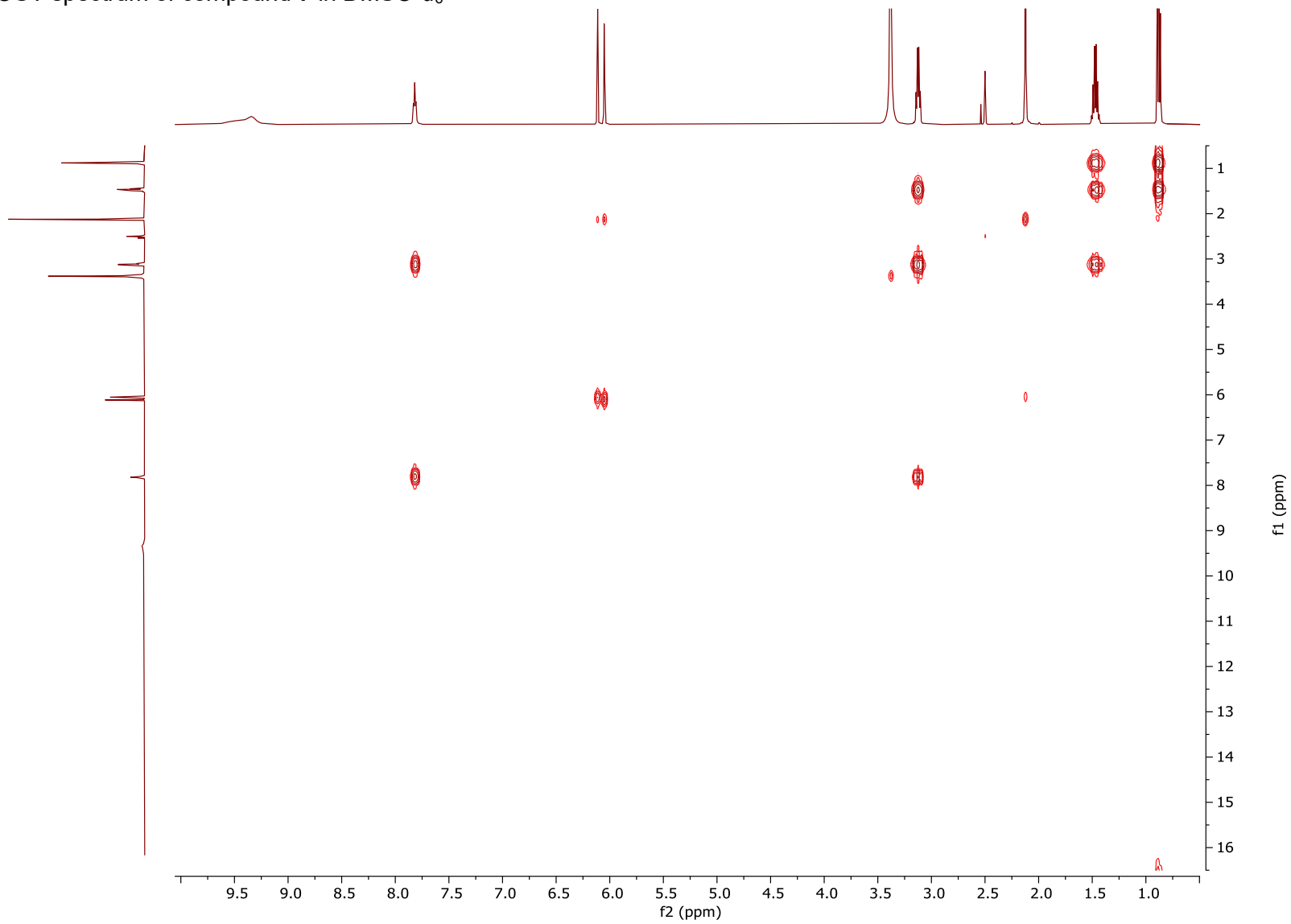
HSQC spectrum of compound 7 in DMSO- d_6



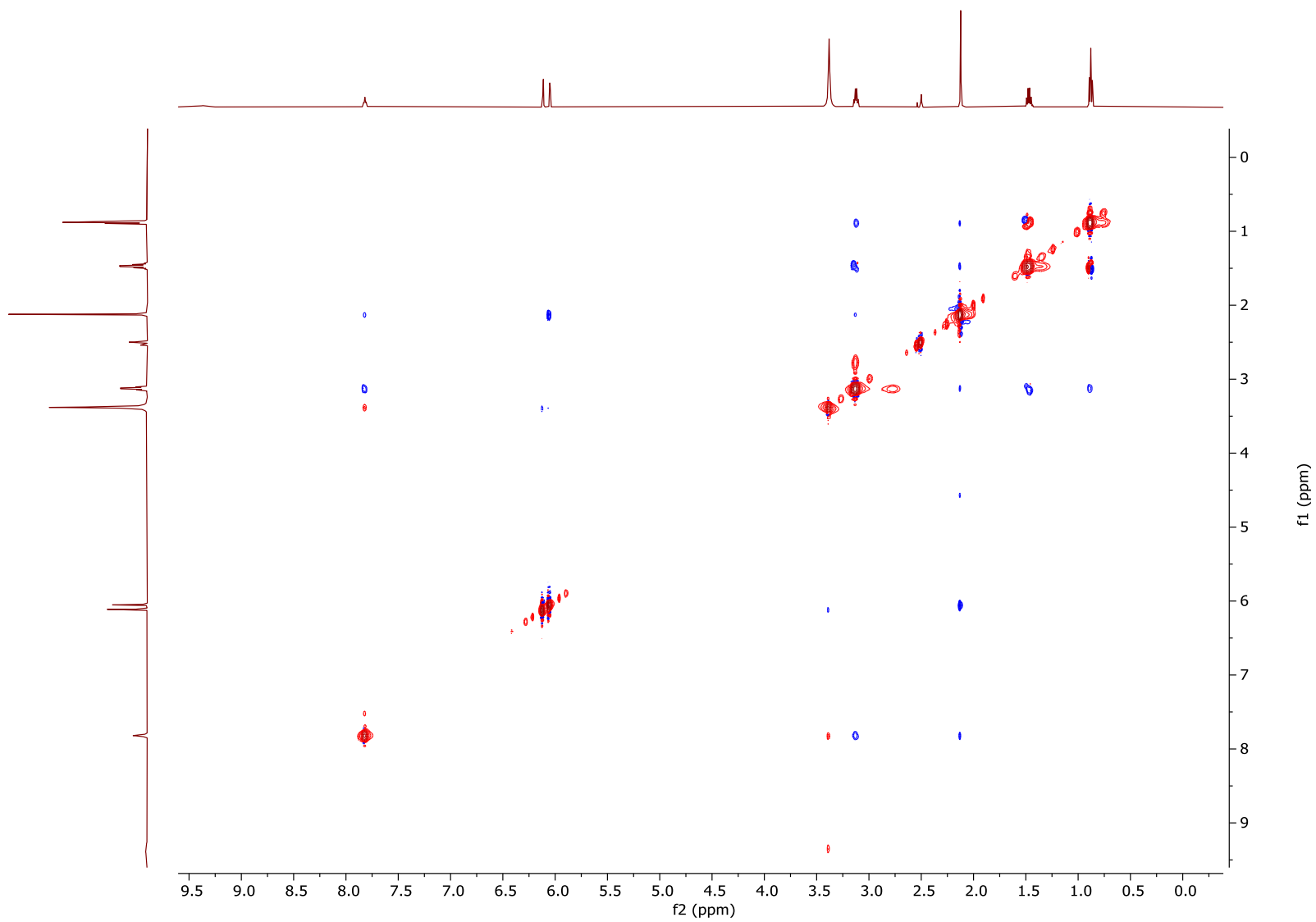
HMBC spectrum of compound 7 in DMSO- d_6



COSY spectrum of compound **7** in DMSO- d_6



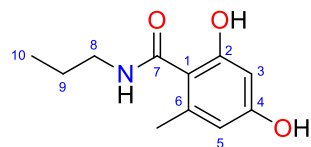
ROESY spectrum of compound **7** in DMSO- d_6



NMR data table for compound **7** in DMSO-*d*₆^a

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type	COSY	HMBC	ROESY
1		116.7, C			
2		156.1, C			
2-OH	9.48 (brs)				
3	6.11 (d, 2.2)	100.0, CH	5,6-Me ^w	1,2,4,5	4-OH,6-Me ^w
4		158.0, C			
4-OH	9.34 (brs)				3 ^w
5	6.05 (d, 2.2)	108.1, CH	3,6-Me ^w	1,3,4,6-Me	4-OH,6-Me
6		137.0, C			
6-Me	2.12 (s)	19.7, CH ₃	3 ^w ,5 ^w	1,2,5,6,7	3,5,7-NH,8 ^w
7		167.8, C			
7-NH	7.82 (t, 5.6)		8	7,8	6-Me,8,9 ^w
8	3.12 (dt, 5.6, 6.6)	40.6, CH ₂	7-NH, 9	7,9,10	6-Me ^w ,7-NH,9,10
9	1.48 (tq, 6.6,7.4)	22.4, CH ₂	8,10	8,10	7-NH ^w ,8,10
10	0.88 (t, 7.4)	11.5, CH ₃	9	8,9	8,9

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^w Weak correlation.



High resolution mass spectrum of compound 7

Mass Spectrum SmartFormula Report

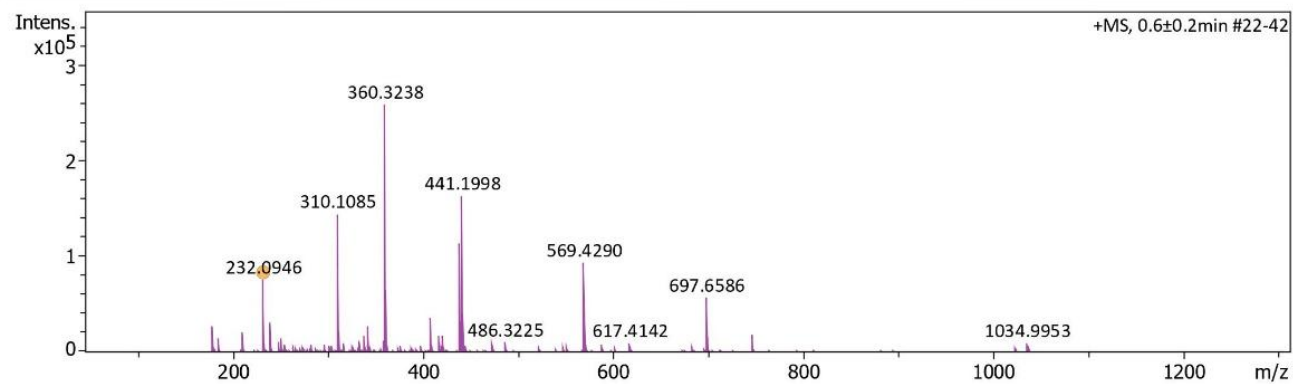
Analysis Info

Analysis Name D:\Data\Sasha\20231004\RAD915D000001.d
Method DirectInfusion_2018_pos.m
Sample Name RAD915D
Comment

Acquisition Date 10/4/2023 2:05:35 PM

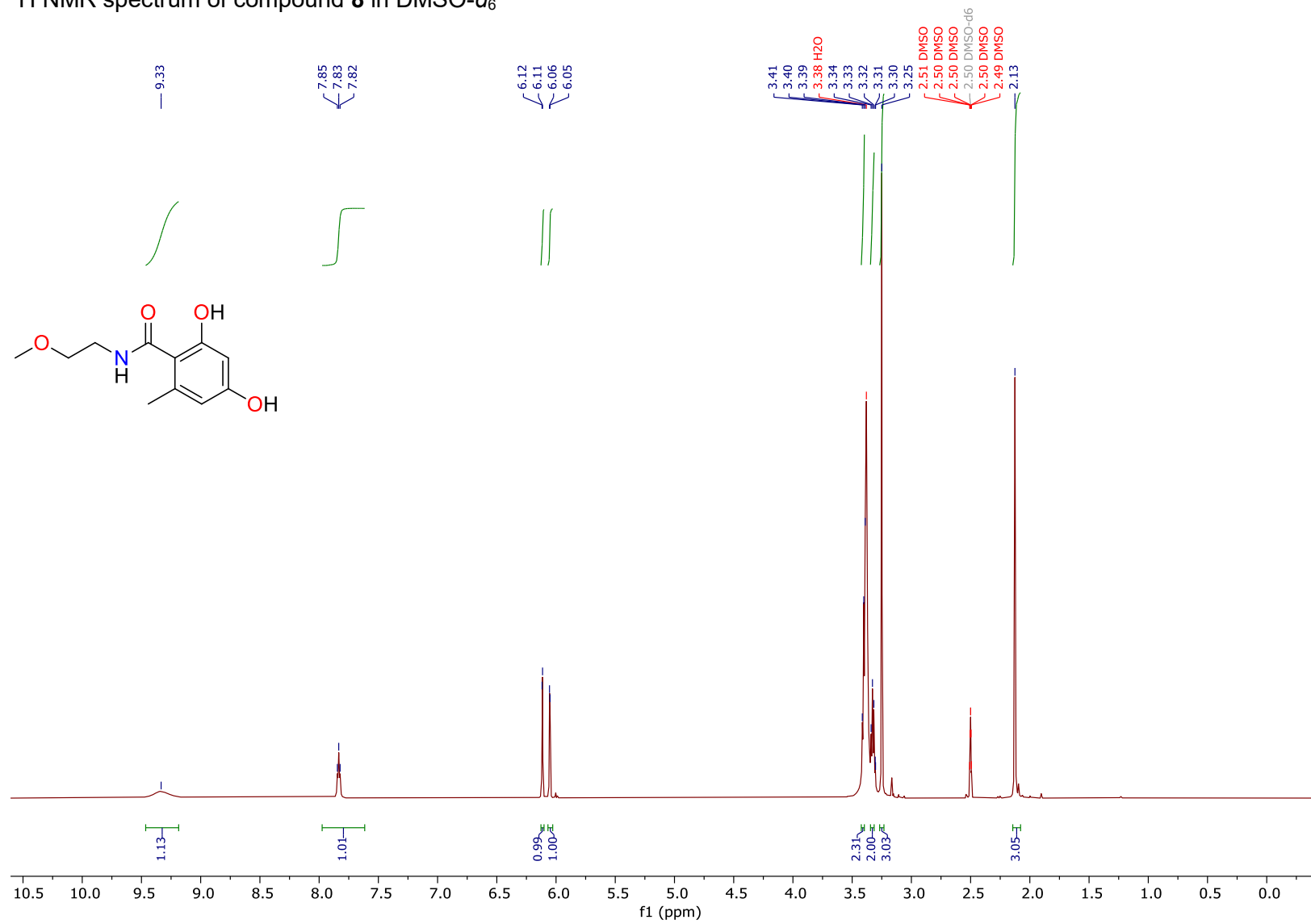
Operator Demo User
Instrument maXis II ETD

Acquisition Parameter

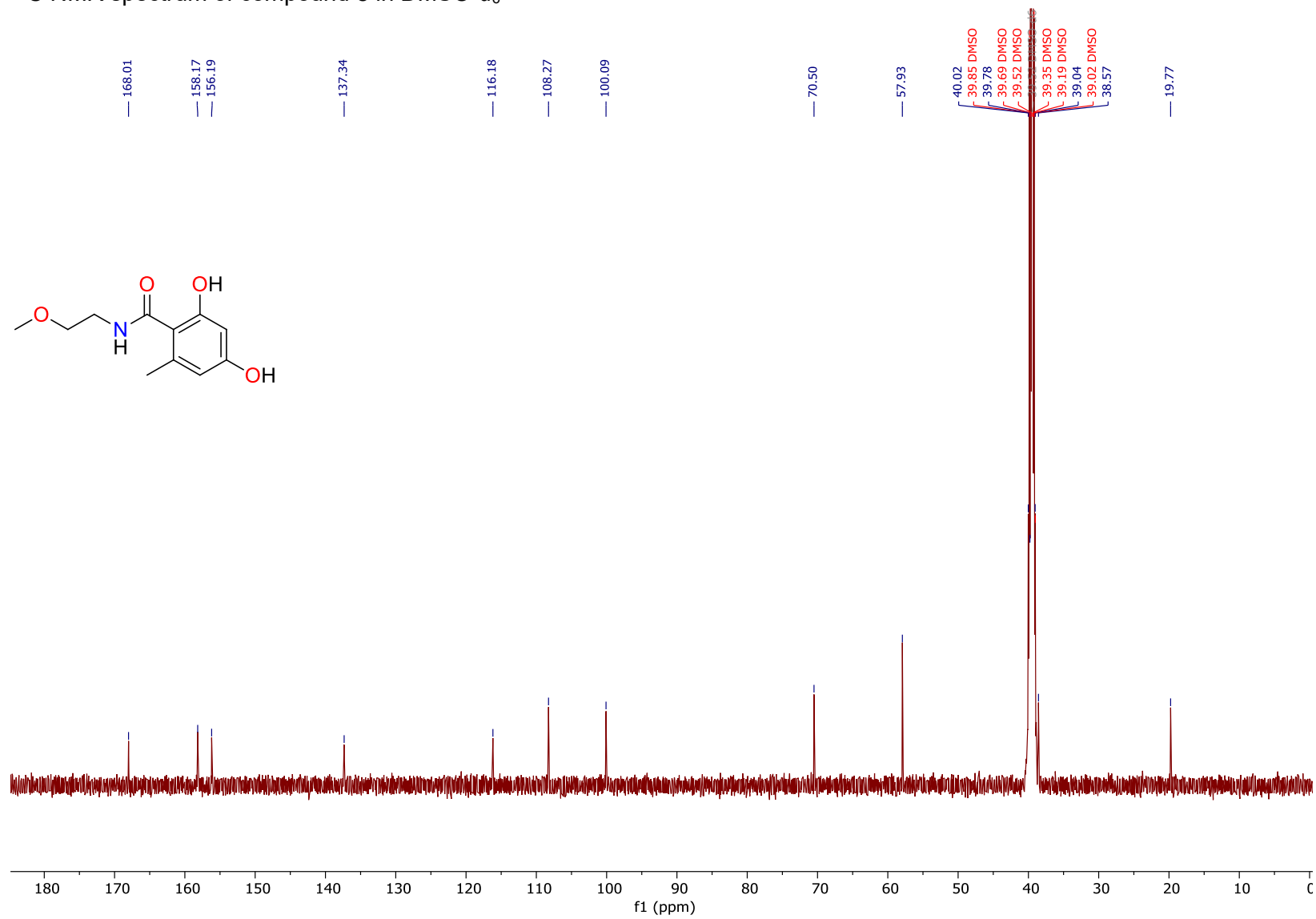


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
232.0946	1	C ₁₁ H ₁₅ NNaO ₃	232.0944	-0.7	7.4	1	100.00	5.0	even	ok

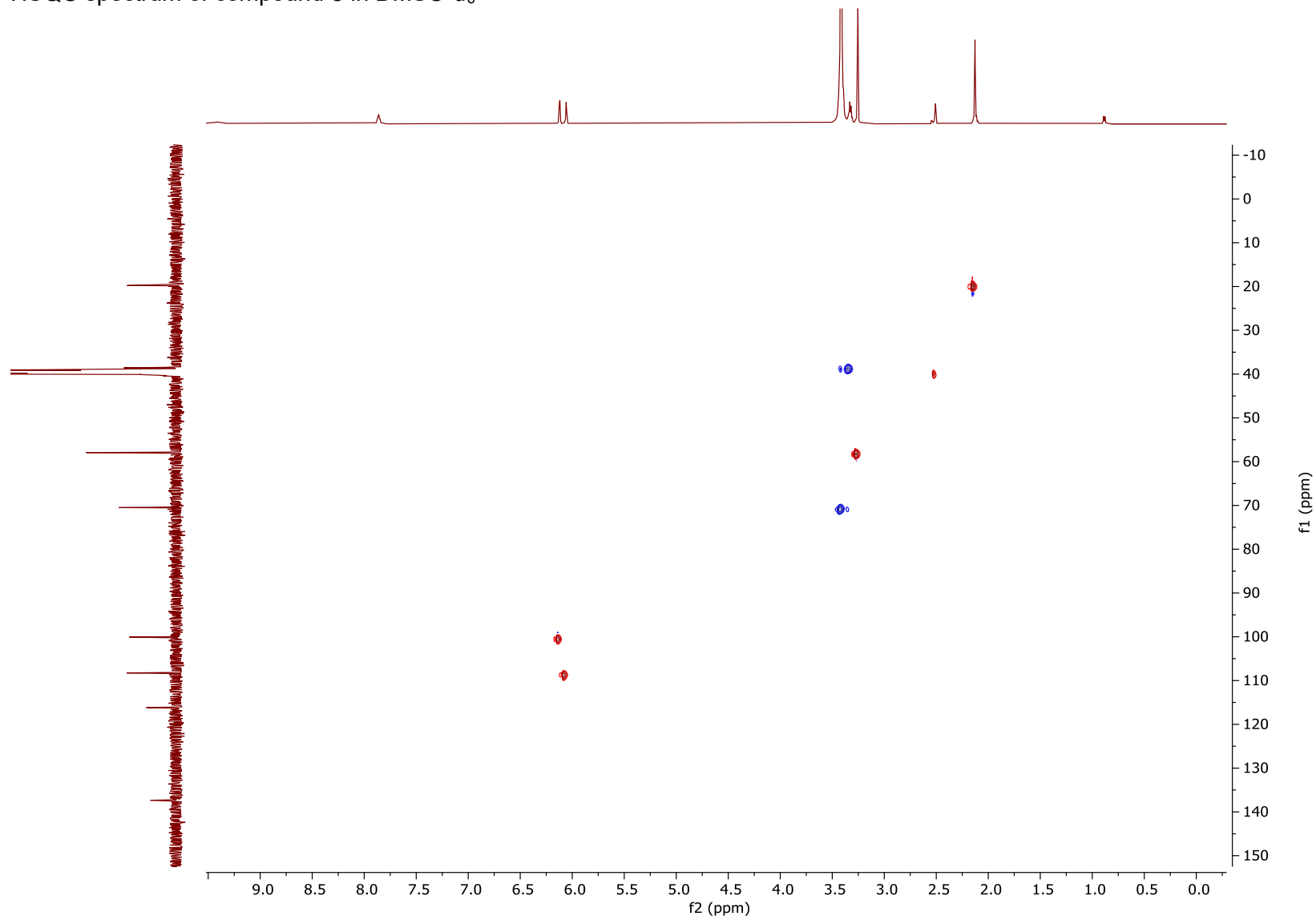
¹H NMR spectrum of compound **8** in DMSO-*d*₆



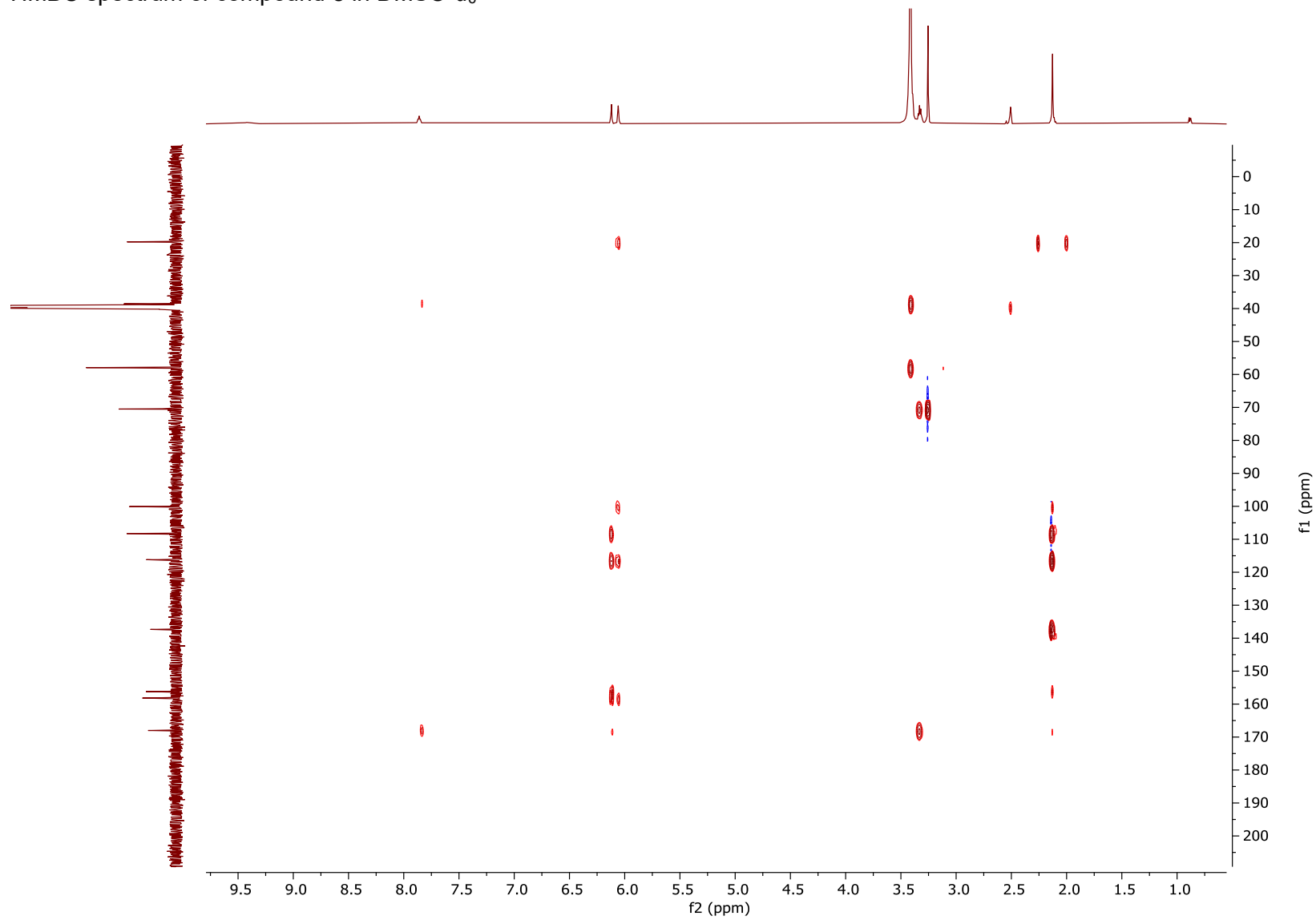
^{13}C NMR spectrum of compound **8** in $\text{DMSO-}d_6$



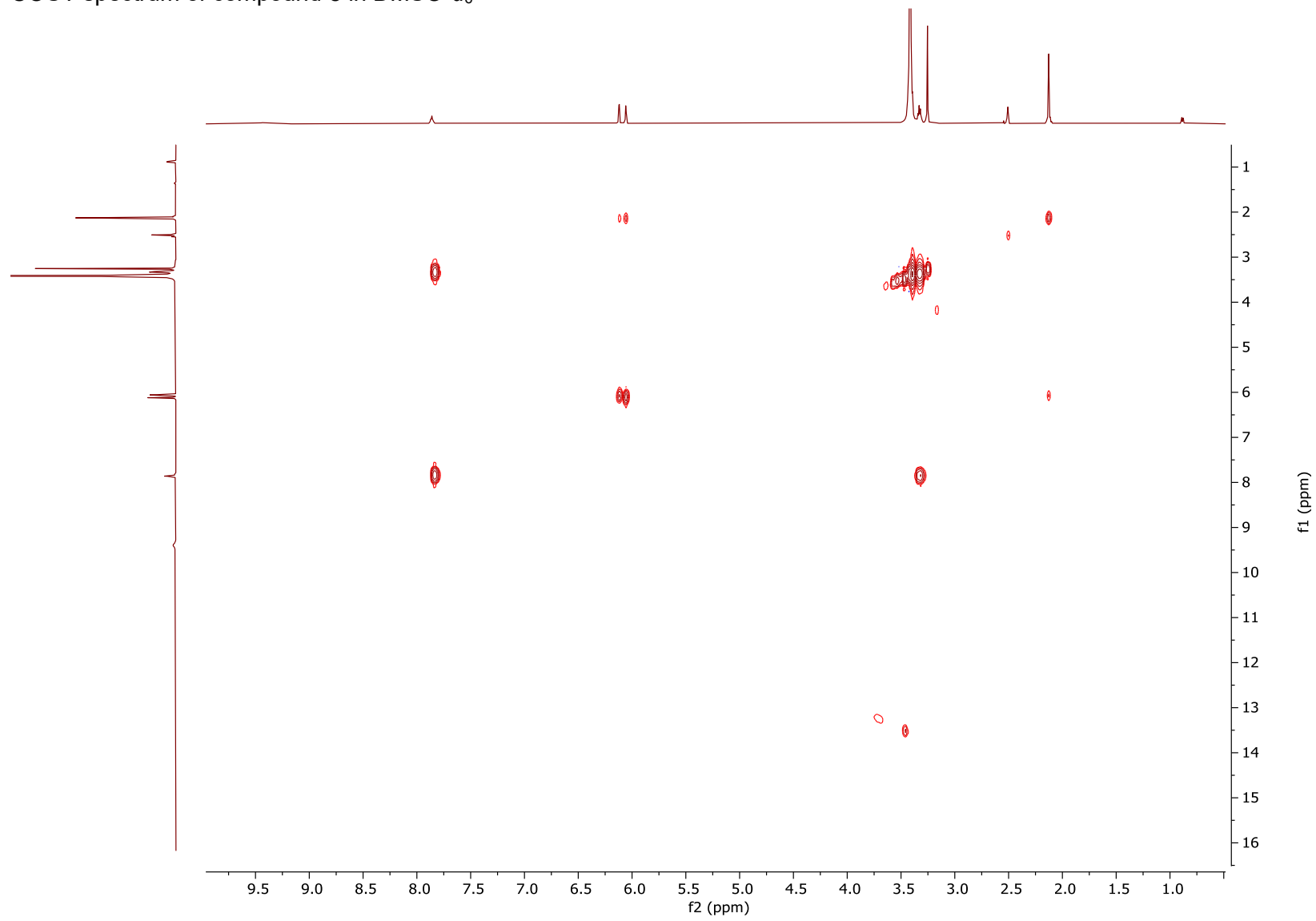
HSQC spectrum of compound **8** in DMSO- d_6



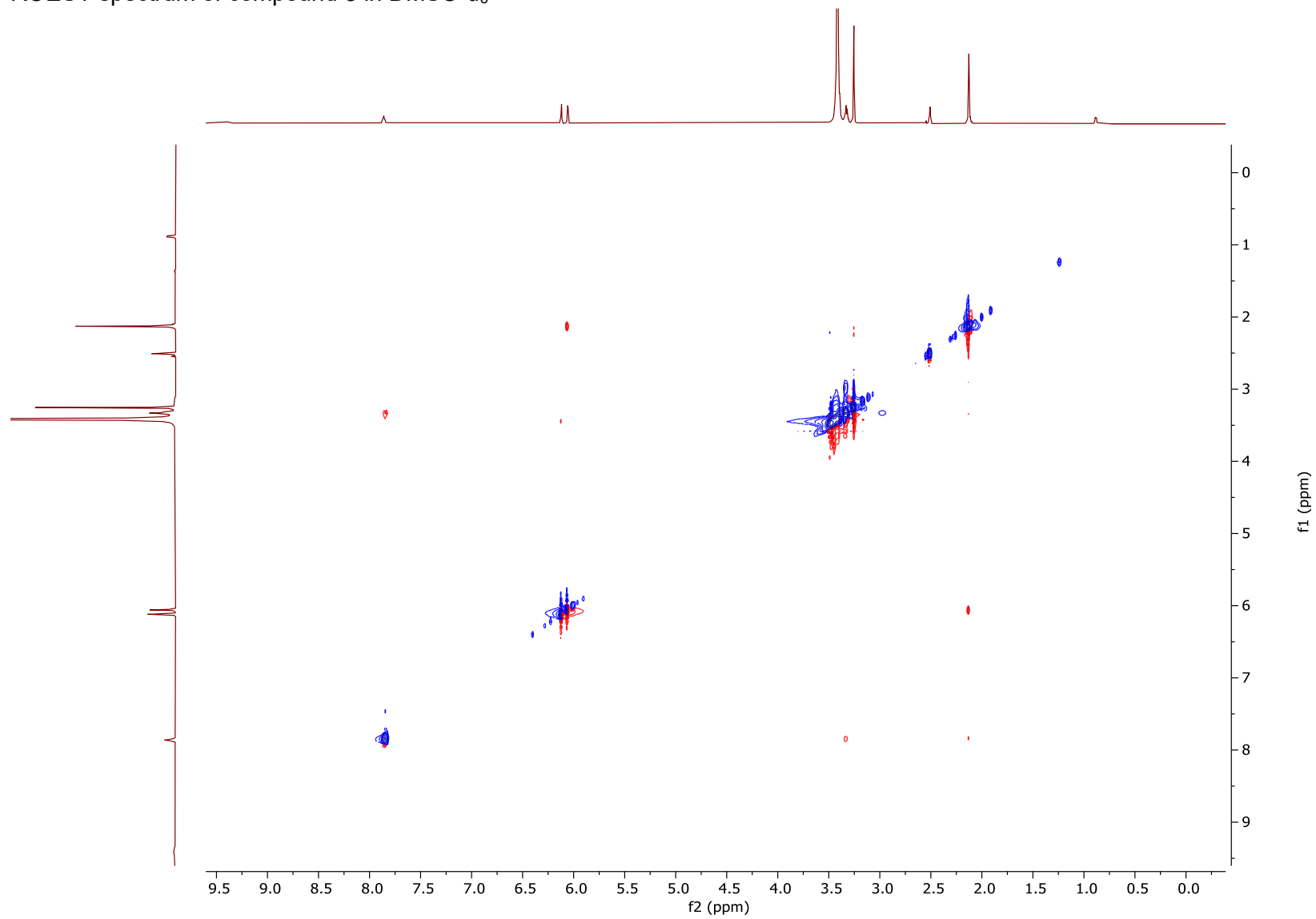
HMBC spectrum of compound **8** in DMSO- d_6



COSY spectrum of compound **8** in DMSO- d_6



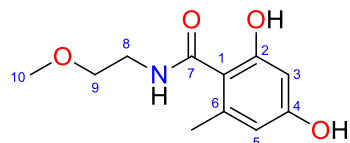
ROESY spectrum of compound **8** in DMSO- d_6



NMR data table for compound **8** in DMSO-*d*₆^a

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type	COSY	HMBC	ROESY
1		116.2, C			
2		156.2, C			
2-OH	^b				
3	6.11 (d, 2.2)	100.1, CH	5,6-Me ^w	1,4,5	6-Me
4		158.2, C			
4-OH	9.33 (brs)				
5	6.05 (d, 2.2)	108.3, CH	3,6-Me ^w	1,3,4,6-Me	6-Me
6		137.3, C			
6-Me	2.13 (s)	19.8, CH ₃	3 ^w ,5 ^w	1,2,5,6,7 ^w	5,7-NH ^w
7		168.0, C			
7-NH	7.83 (t, 5.7)		8	7,8	6-Me ^w ,8,9
8	3.33 (dt, 5.7, 5.8)	38.6, CH ₂	7-NH, 9	7,9	7-NH,9,10
9	3.40 (t, 5.8)	70.5, CH ₂	8	8,10	7-NH ^w ,8,10
10	3.25 (s)	57.9, CH ₃		9	8,9

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Not observed; ^w Weak correlation.



High resolution mass spectrum of compound **8**

Mass Spectrum SmartFormula Report

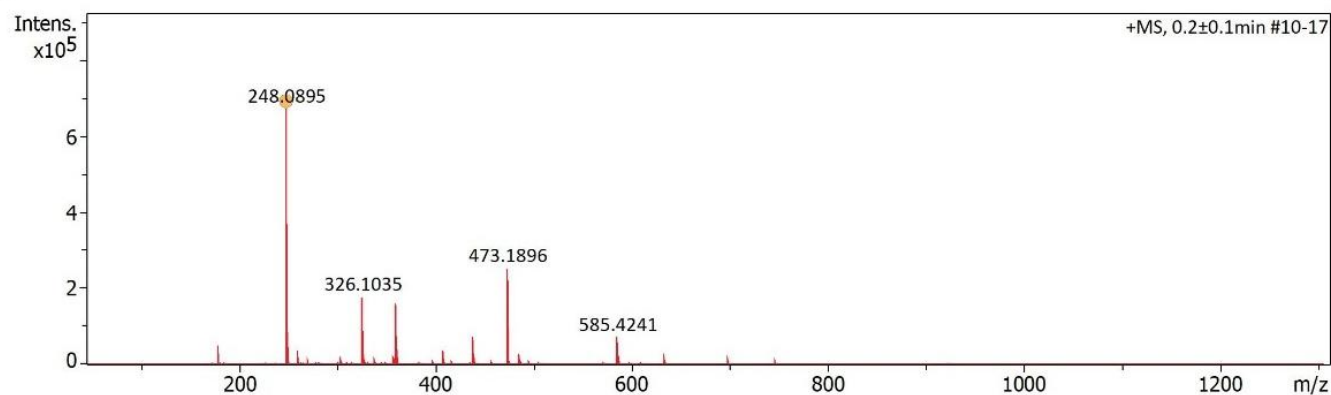
Analysis Info

Analysis Name D:\Data\Sasha\20231004\RAD915A000001.d
 Method DirectInfusion_2018_pos.m
 Sample Name RAD915A
 Comment

Acquisition Date 10/4/2023 1:48:42 PM

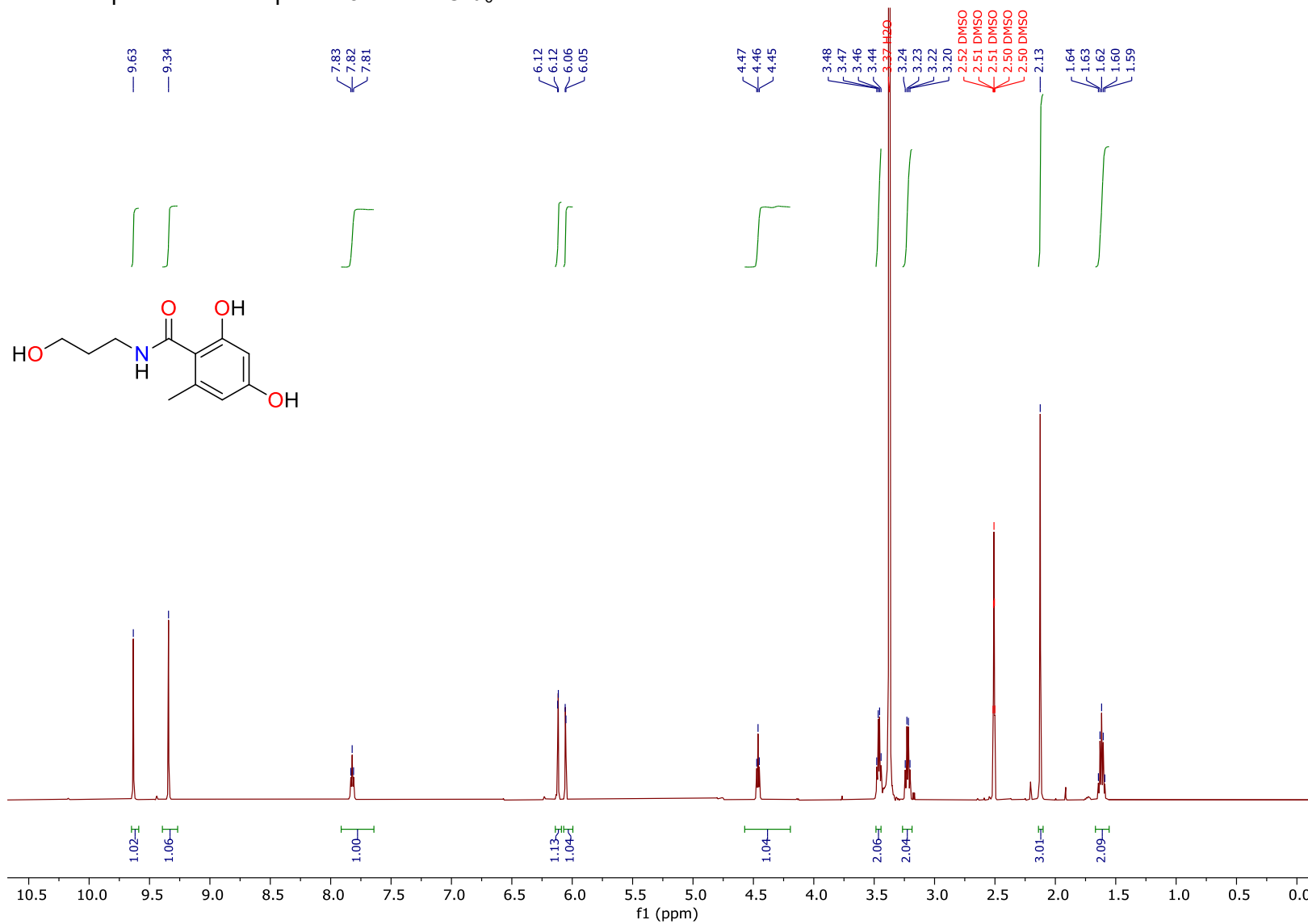
Operator Demo User
 Instrument maXis II ETD

Acquisition Parameter

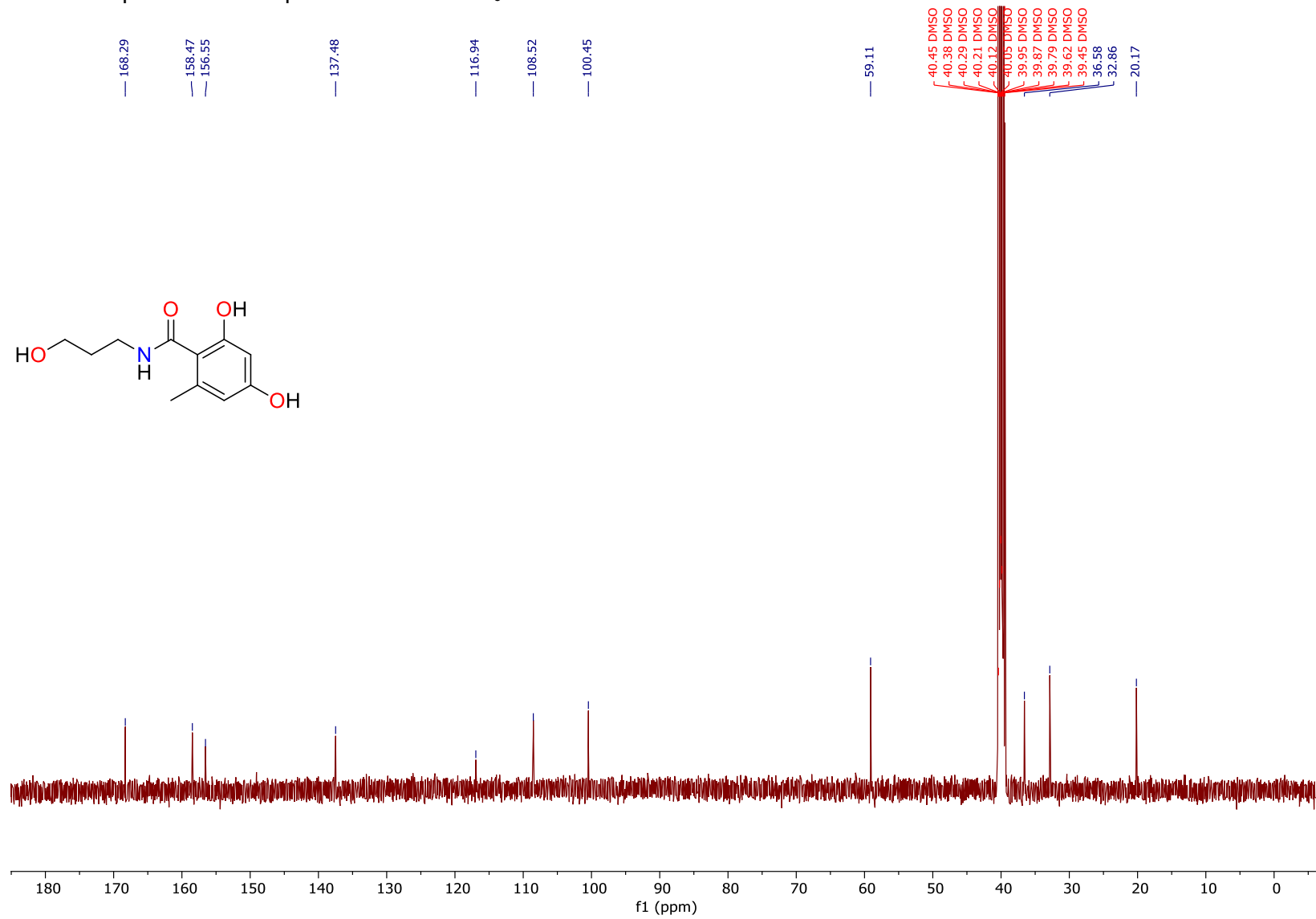


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
248.0895	1	C11H15NNaO4	248.0893	-0.7	0.3	1	100.00	5.0	even	ok
	2	C12H11N5Na	248.0907	4.7	13.6	2	47.59	10.0	even	ok
	3	C7H11N7NaO2	248.0866	-11.5	13.7	3	12.91	6.0	even	ok

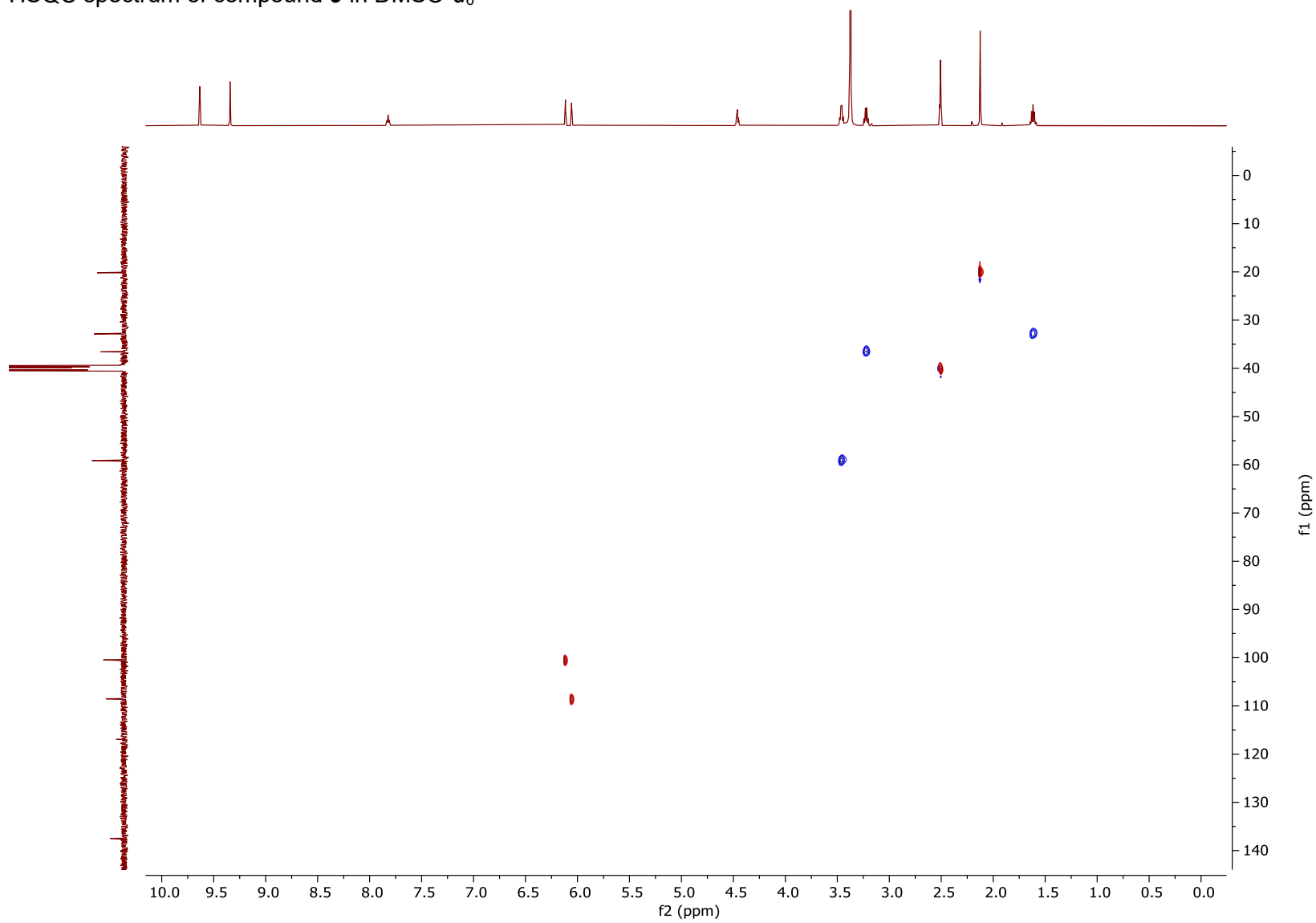
¹H NMR spectrum of compound **9** in DMSO-*d*₆



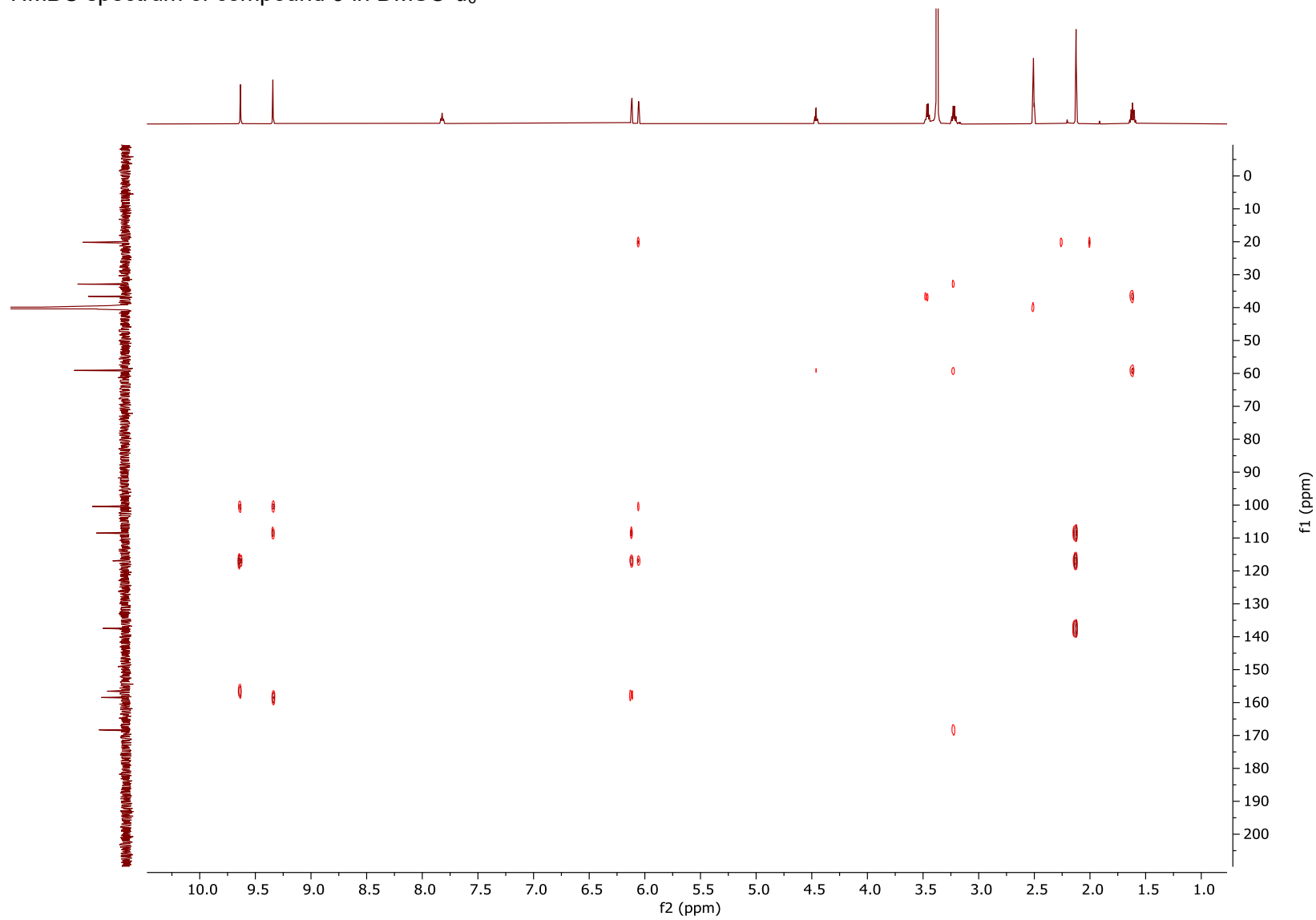
^{13}C NMR spectrum of compound **9** in $\text{DMSO-}d_6$



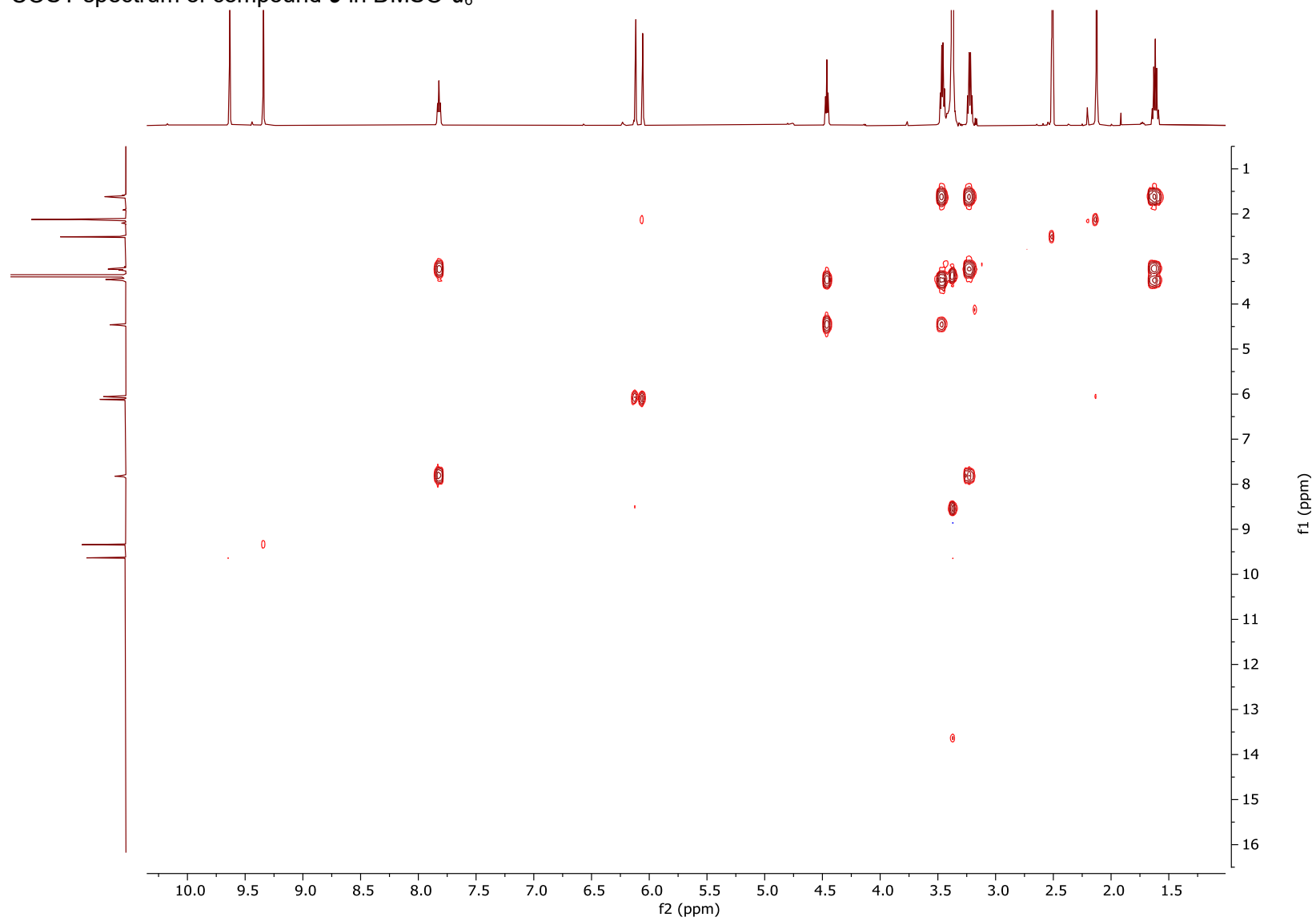
HSQC spectrum of compound **9** in DMSO- d_6



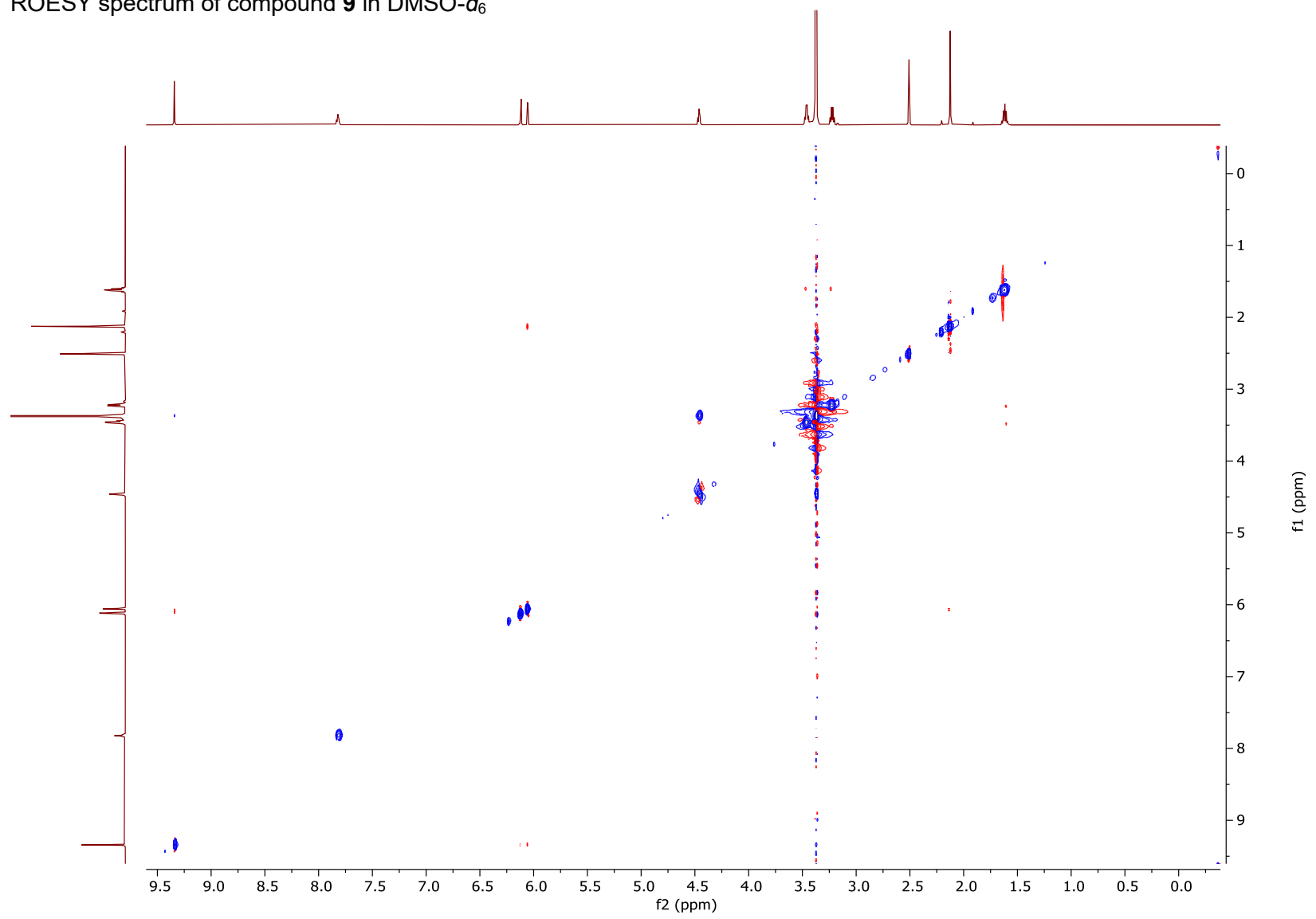
HMBC spectrum of compound **9** in DMSO- d_6



COSY spectrum of compound **9** in DMSO- d_6



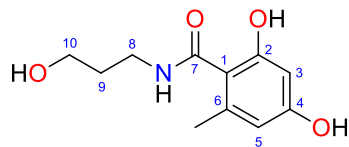
ROESY spectrum of compound **9** in DMSO-*d*₆



NMR data table for compound **9** in DMSO-*d*₆^a

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type	COSY	HMBC	ROESY
1		116.9, C			
2		156.6, C			
2-OH	9.63 (brs)			1,2,3	3
3	6.12 (d, 2.3)	100.5, CH	5 ^w ,6-Me ^w	1,2,4,5,7 ^w	2-OH,4-OH
4		158.5, C			
4-OH	9.34 (brs)			3,4,5	3,5
5	6.05 (d, 2.3)	108.5, CH	3,6-Me ^w	1,3,4,6-Me	4-OH,6-Me
6		137.5, C			
6-Me	2.13 (s)	20.2, CH ₃	3 ^w ,5 ^w	1,2 ^w ,3 ^w ,4 ^w ,5,6	5,7-NH
7		168.3, C			
7-NH	7.82 (t, 5.6)		8	7 ^w	6-Me,8
8	3.22 (dt, 5.6, 6.6)	36.6, CH ₂	7-NH,9,10	9,10	7-NH,9 ^w
9	1.62 (tq, 6.6, 6.2)	32.9, CH ₂	8,10	8,10	8,10
10	3.46 (dt, 5.5, 6.2)	59.1, CH ₂	9,10-OH	8,9	9 ^w ,10-OH
10-OH	4.46 (t, 5.4)		10	9 ^w ,10	10

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^w Weak correlation.



High resolution mass spectrum of compound 9

Mass Spectrum SmartFormula Report

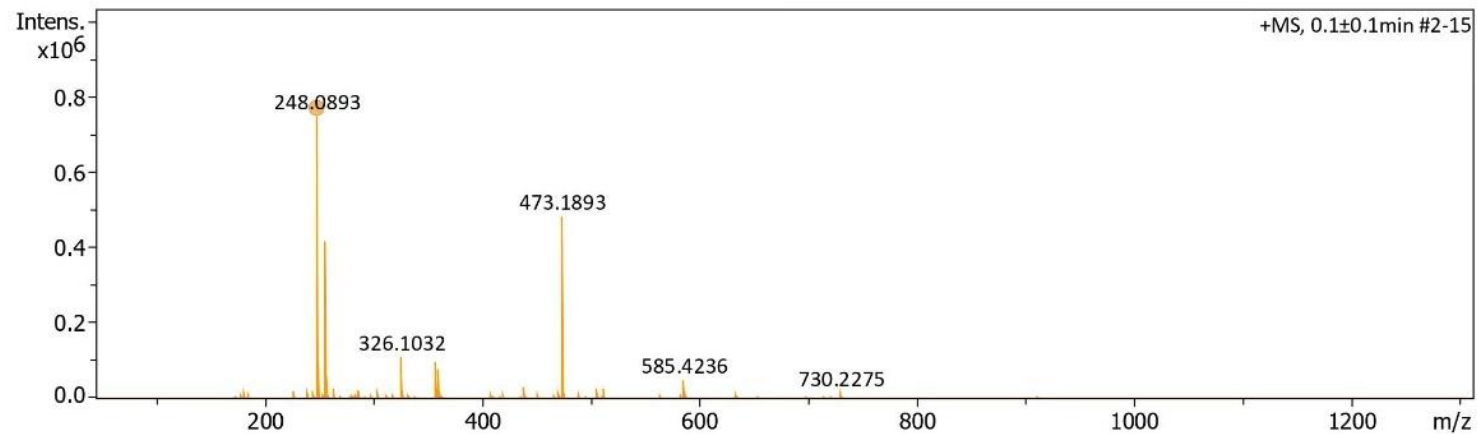
Analysis Info

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Method DirectInfusion_2018_pos.m
Sample Name RAD915H
Comment

Acquisition Date 10/4/2023 2:23:44 PM

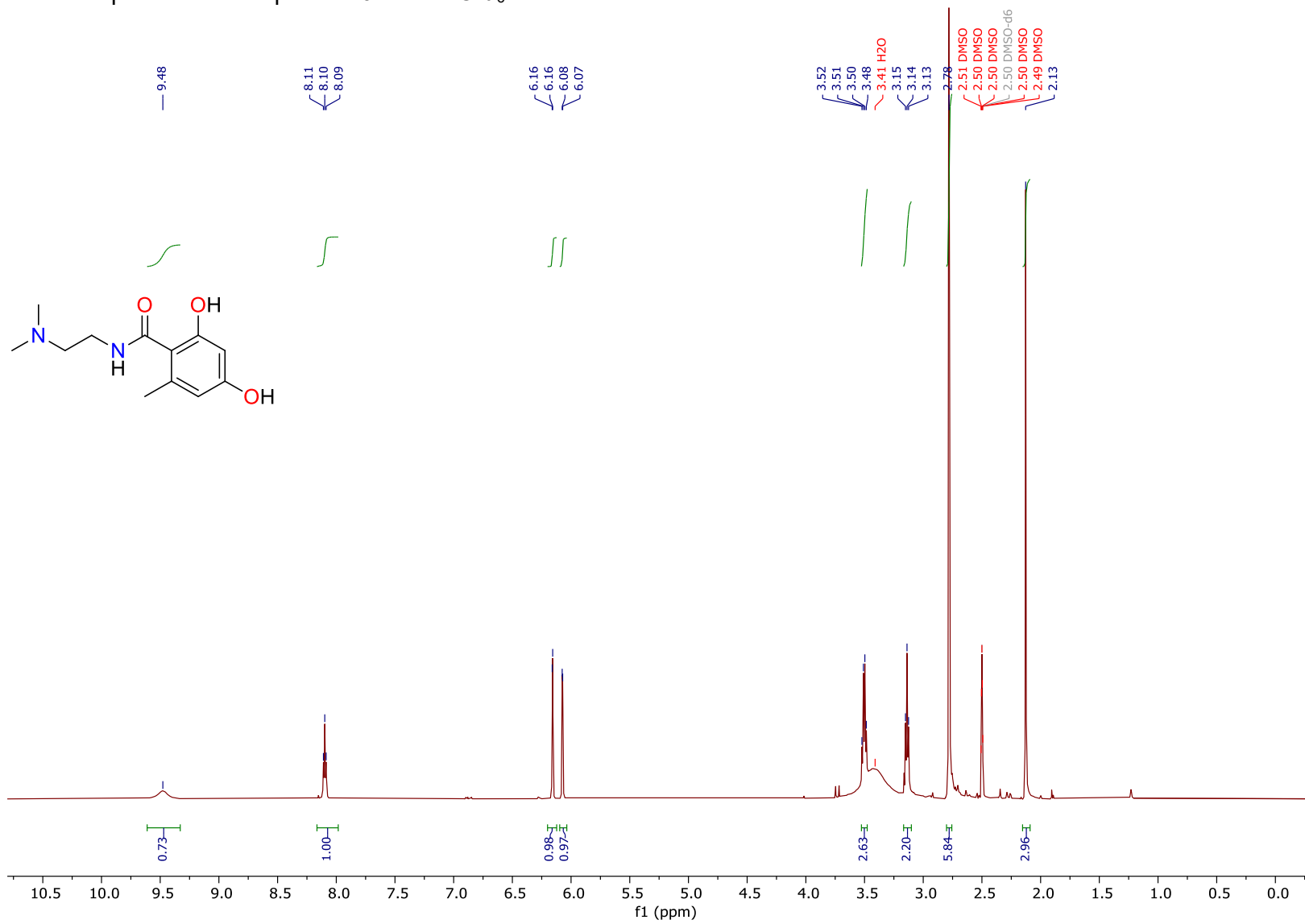
Operator Demo User
Instrument maXis II ETD

Acquisition Parameter

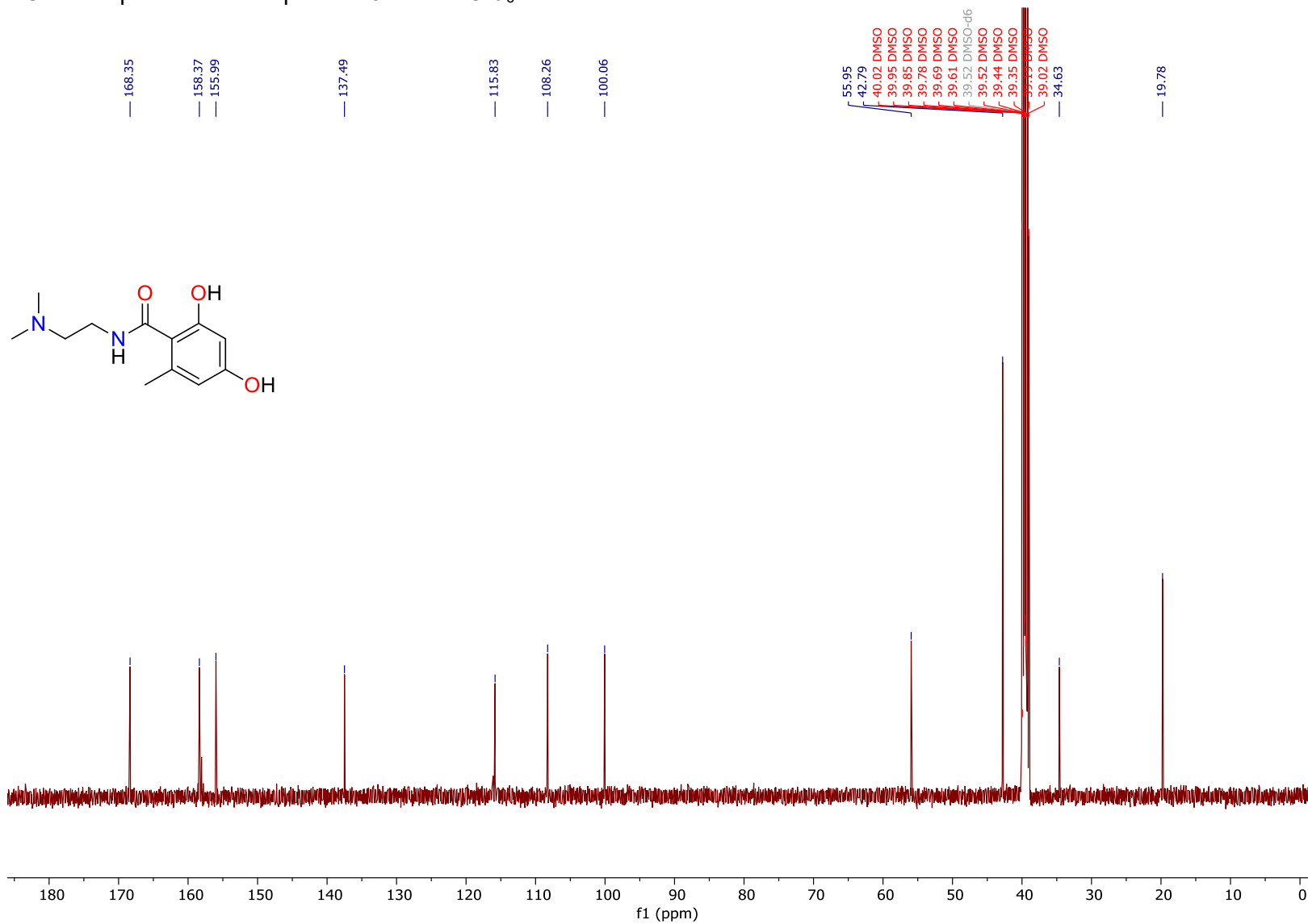


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
248.0893	1	C ₁₁ H ₁₅ NNaO ₄	248.0893	-0.0	0.5	1	100.00	5.0	even	ok

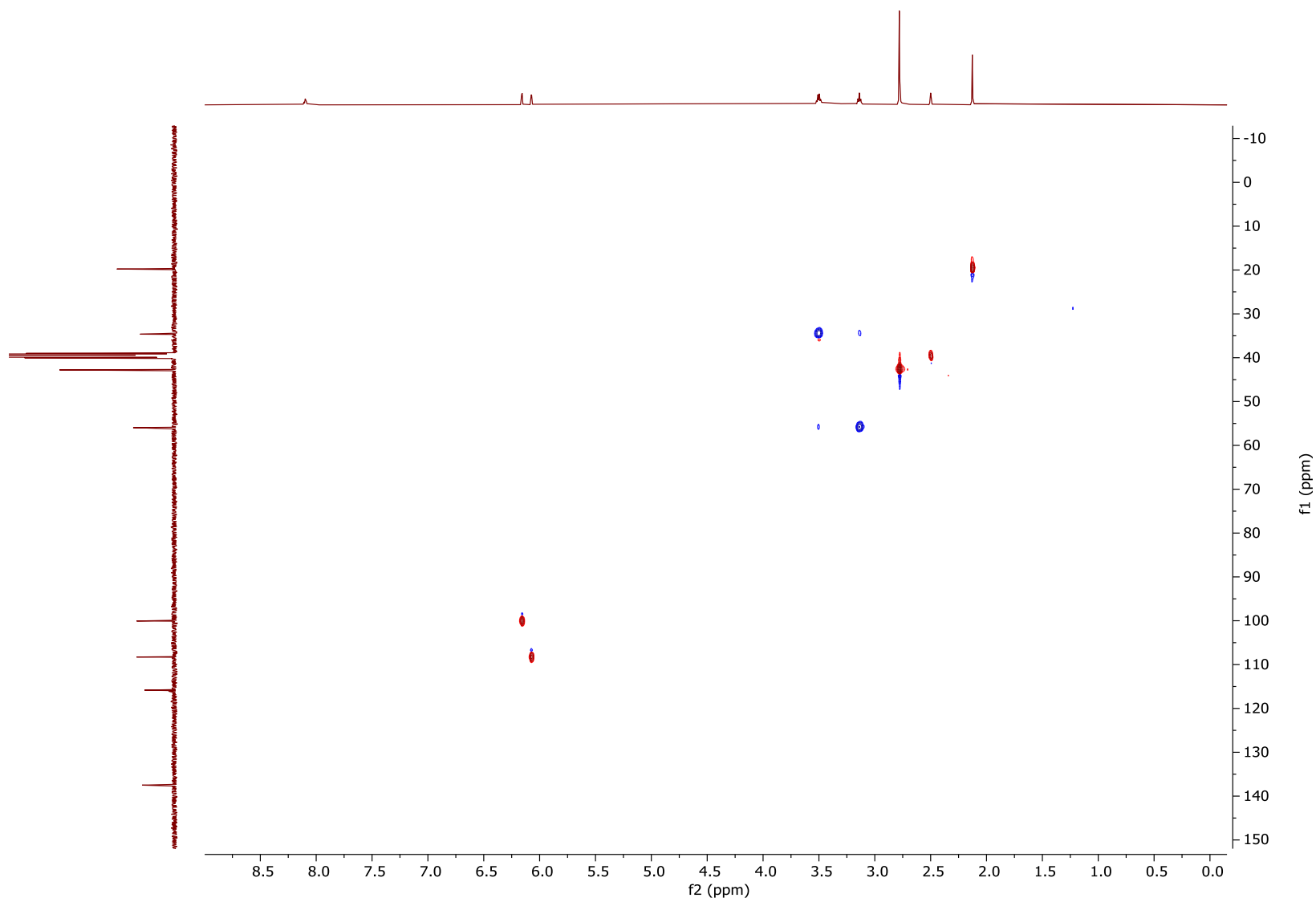
¹H NMR spectrum of compound **10** in DMSO-*d*₆



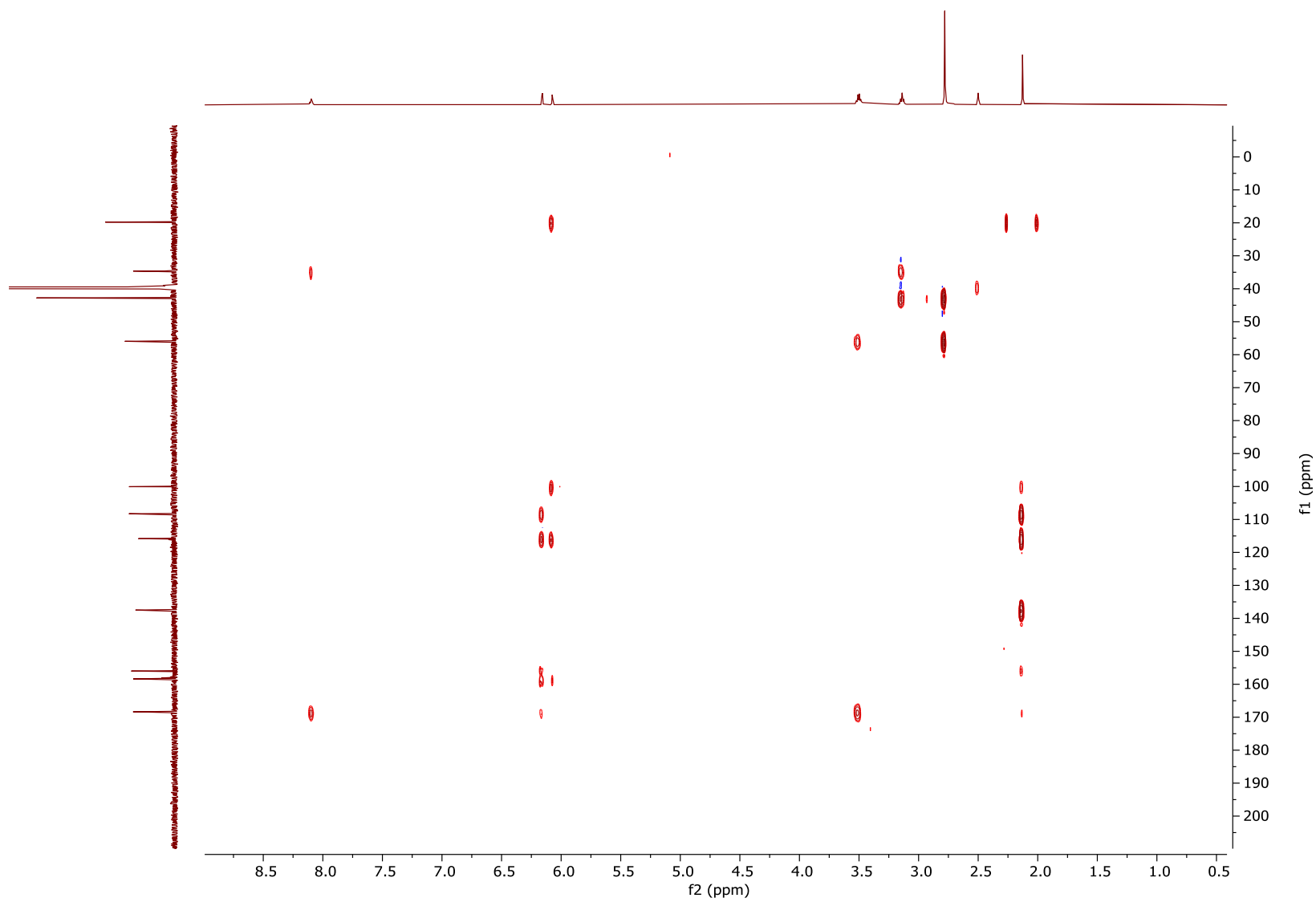
¹³C NMR spectrum of compound **10** in DMSO-*d*₆



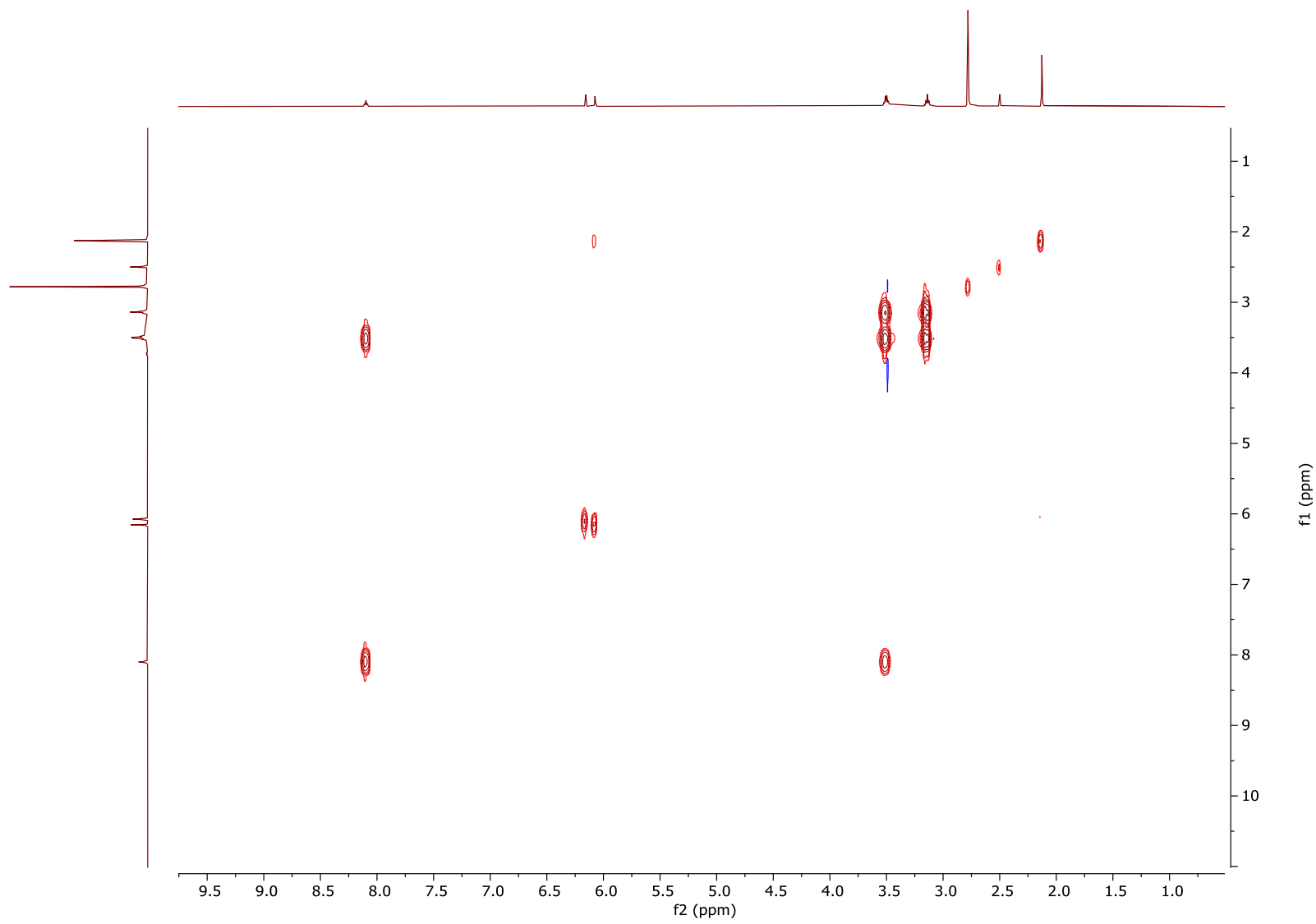
HSQC spectrum of compound **10** in DMSO- d_6



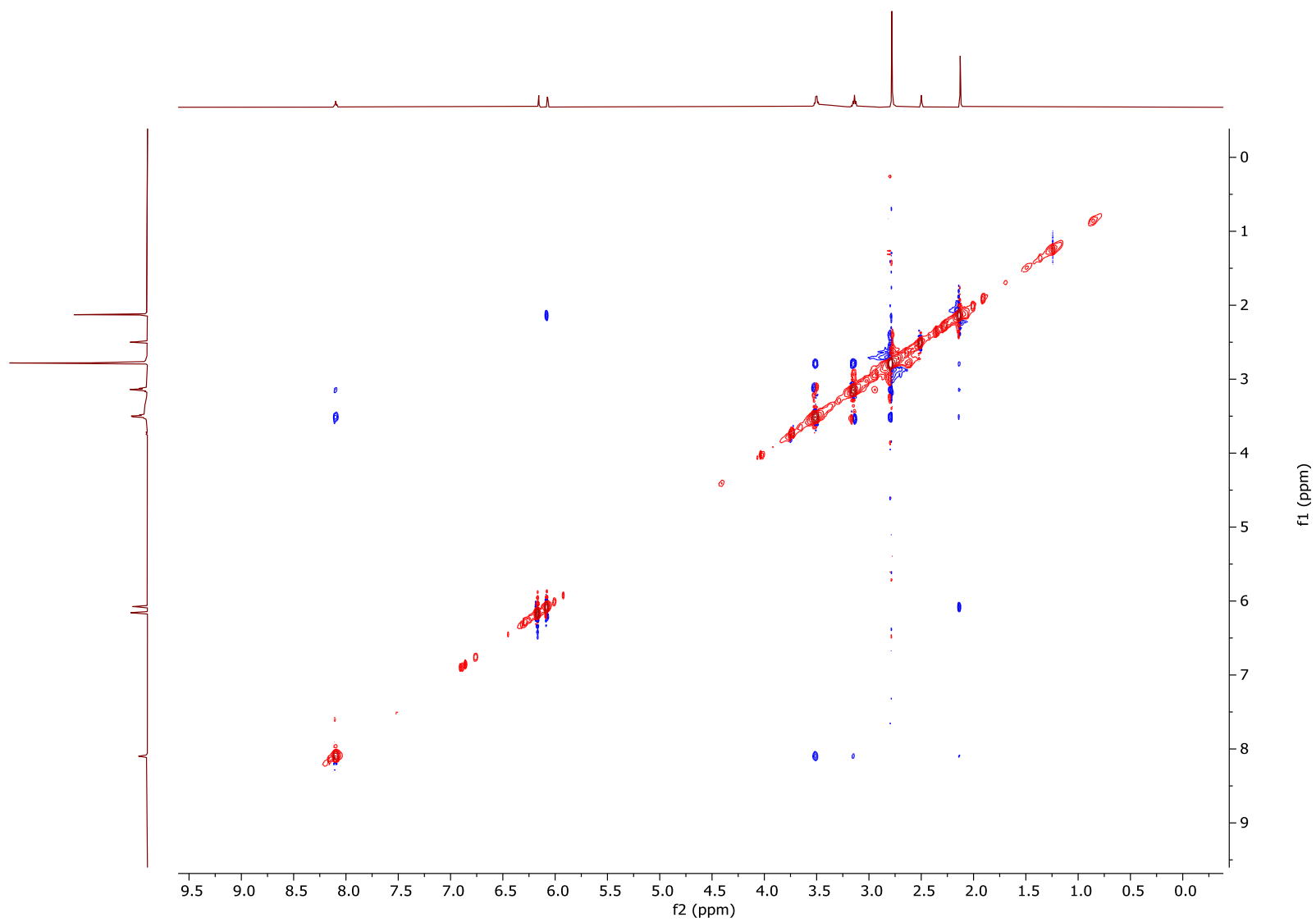
HMBC spectrum of compound **10** in DMSO- d_6



COSY spectrum of compound **10** in DMSO- d_6



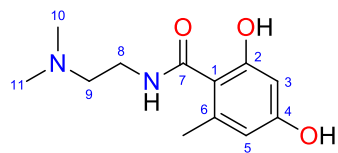
ROESY spectrum of compound **10** in DMSO- d_6



NMR data table for compound **10** in DMSO- d_6 ^{a, b}

Position	δ_H (mult., J in Hz)	δ_C , type	COSY	HMBC	ROESY
1		115.8, C			
2		156.0, C			
2-OH	^c				
3	6.16 (d, 2.0)	100.1, CH	5,6-Me ^w	1,2,4,5,7 ^w	
4		158.4, C			
4-OH	9.48 (brs)				
5	6.08 (d, 2.0)	108.3, CH	3,6-Me ^w	1,3,4,6-Me	6-Me
6		137.5, C			
6-Me	2.13 (s)	19.8, CH ₃	3 ^w ,5 ^w	1,2 ^w ,3 ^w ,4,5,6,7 ^w	5,7-NH,8,9
7		168.4, C			
7-NH	8.10 (t, 5.6)		8	7,8	6-Me ^w ,8,9 ^w ,10 ^w ,11 ^w
8	3.50 (dt, 5.6, 6.4)	34.6, CH ₂	7-NH,9	7,9	6-Me ^w ,7-NH,9,10,11
9	3.14 (t, 6.4)	56.0, CH ₂	8	8,10,11	6-Me ^w ,7-NH ^w ,8,10,11
9-NH	^{b,c}				
10	2.78 (s)	42.8, CH ₃		9,11	8,9
11	2.78 (s)	42.8, CH ₃		9,10	8,9

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Purified as a TFA salt; ^c Not observed; ^w Weak correlation.



High resolution mass spectrum of compound **10**

Mass Spectrum SmartFormula Report

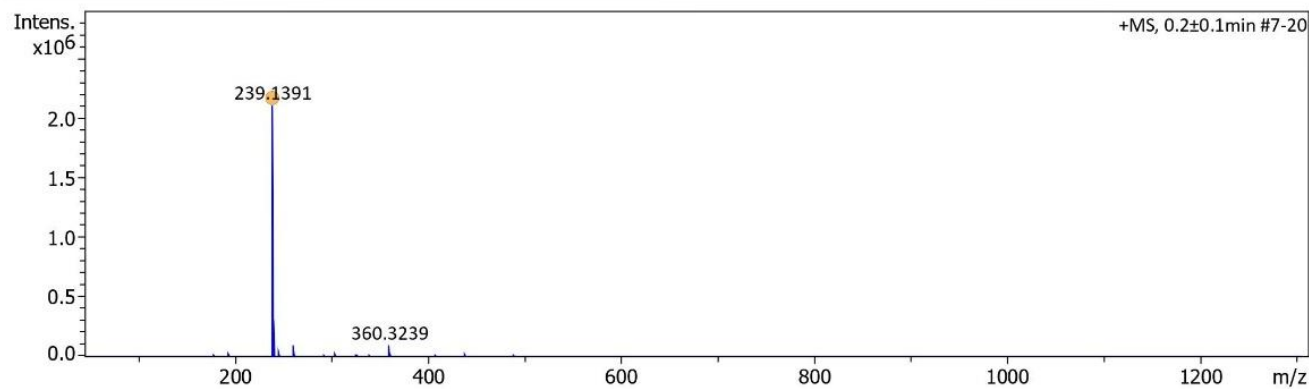
Analysis Info

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Method DirectInfusion_2018_pos.m
Sample Name RAD915E
Comment

Acquisition Date 10/4/2023 2:12:02 PM

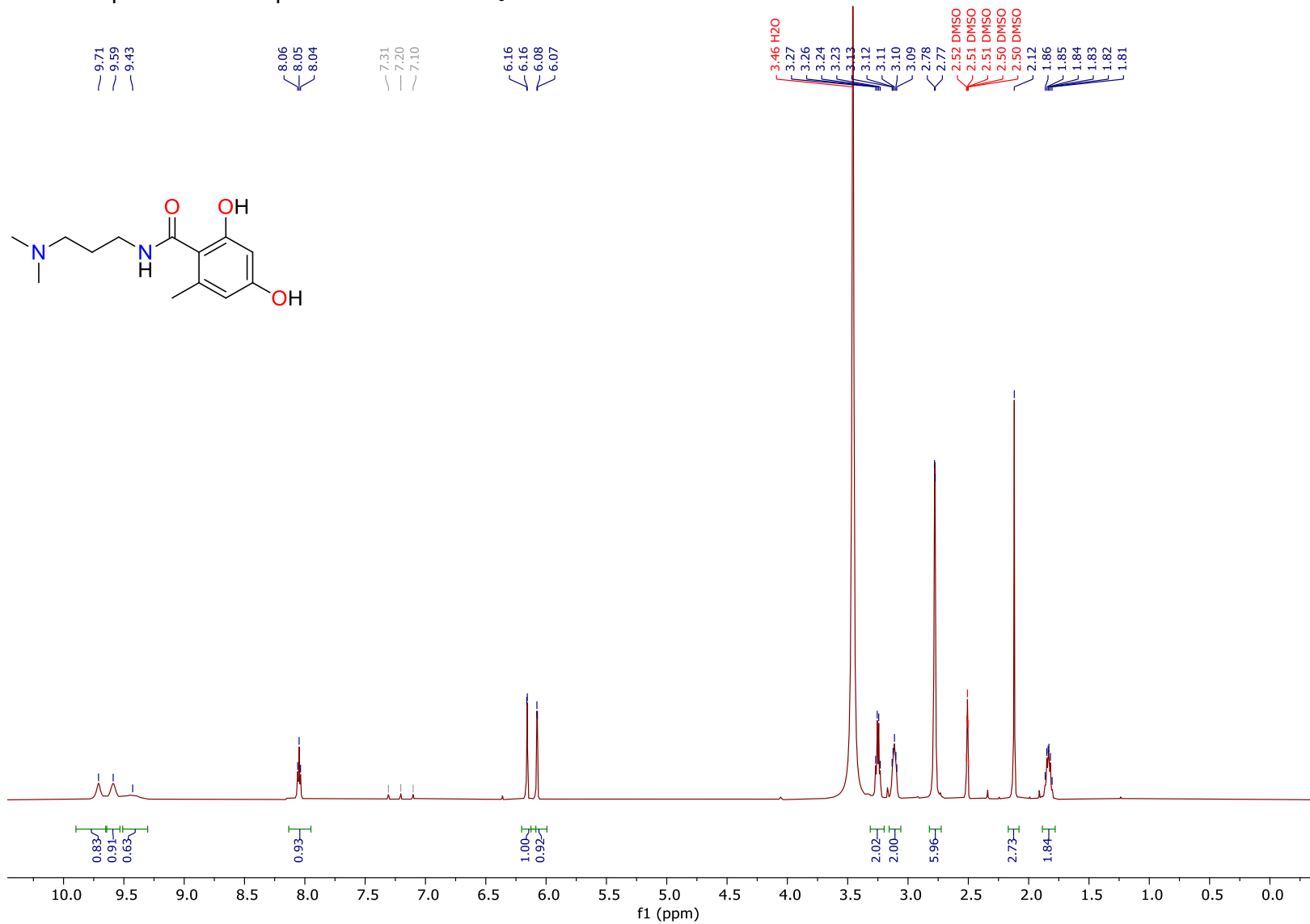
Operator Demo User
Instrument maXis II ETD

Acquisition Parameter

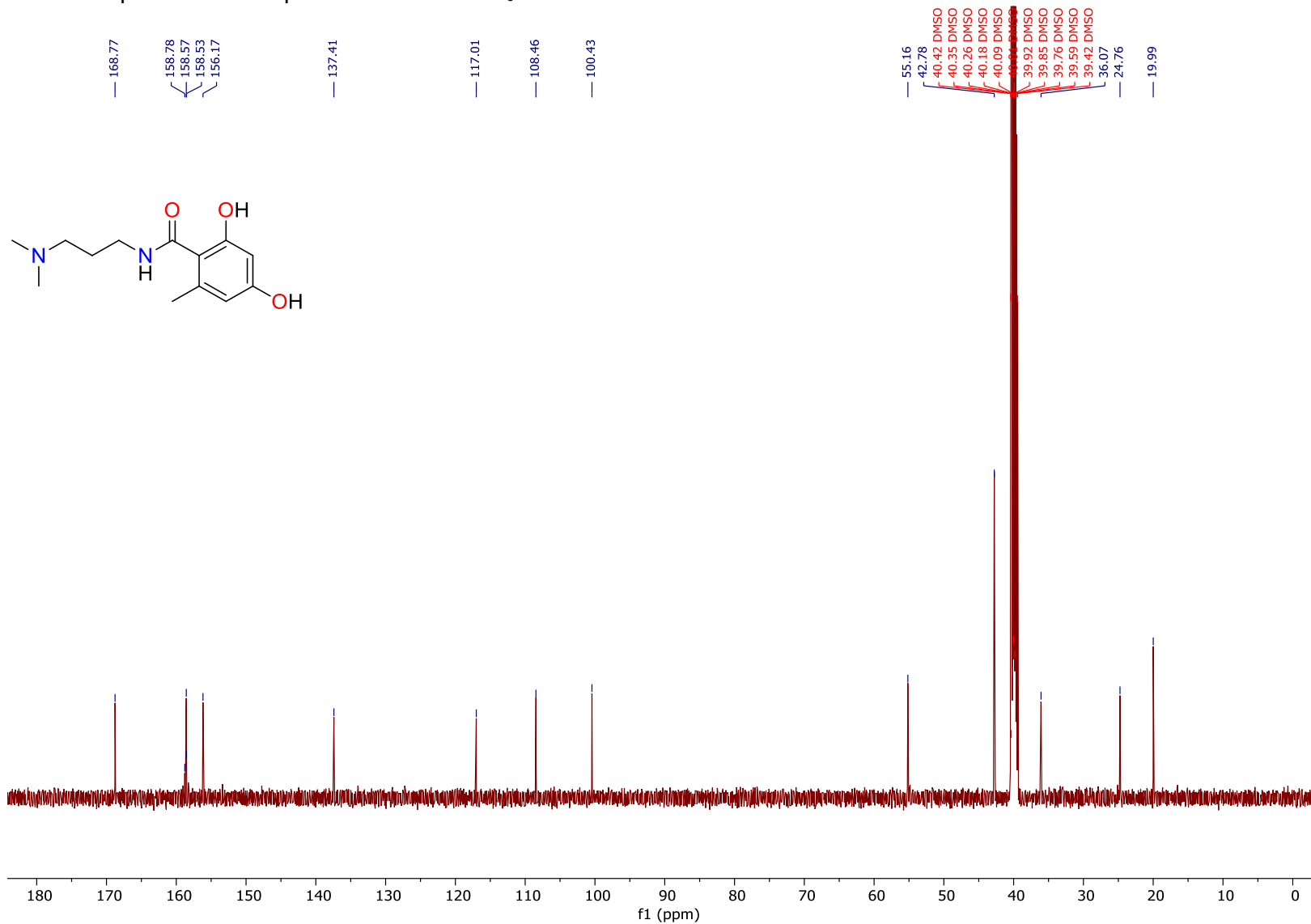


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
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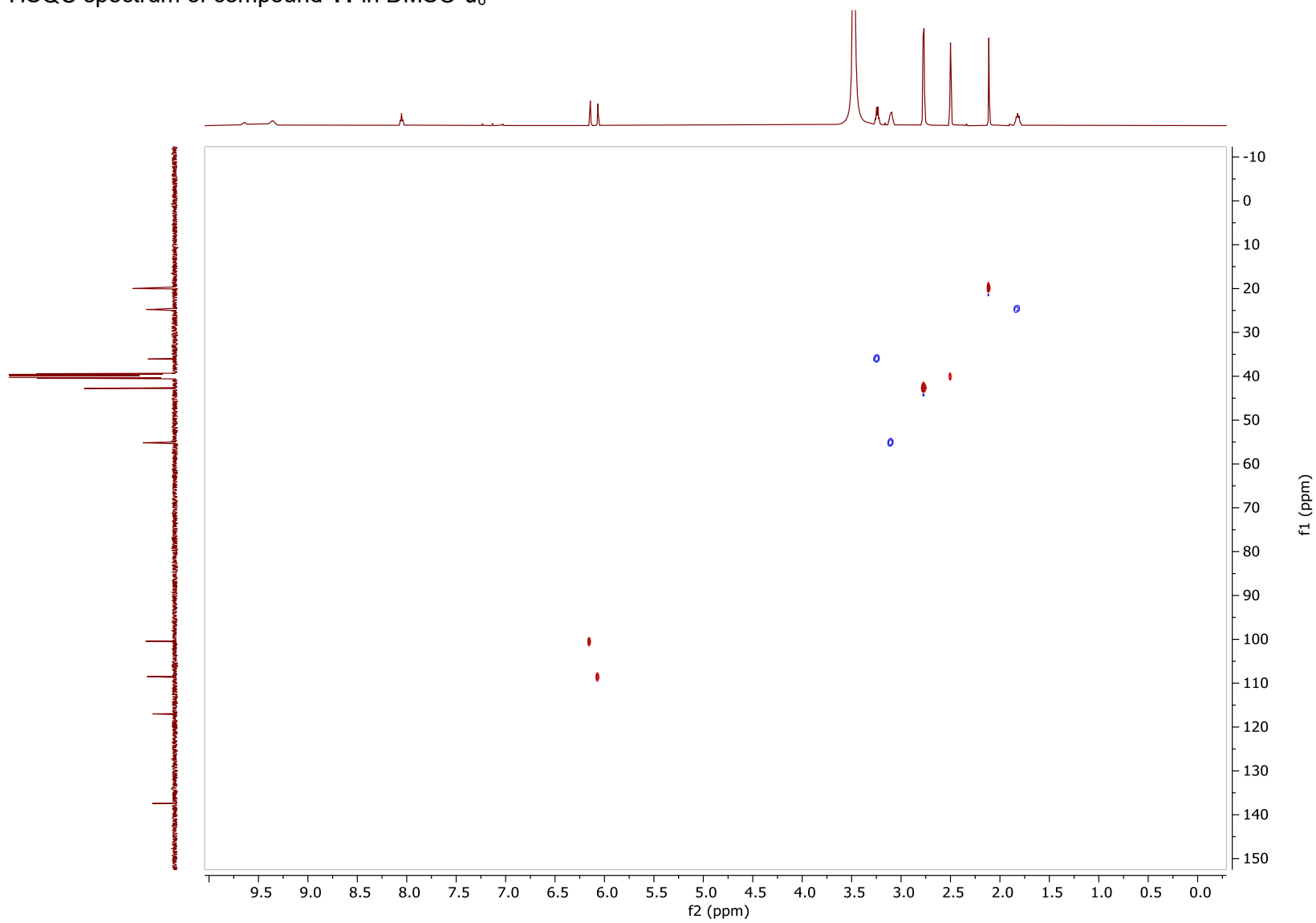
¹H NMR spectrum of compound **11** in DMSO-*d*₆



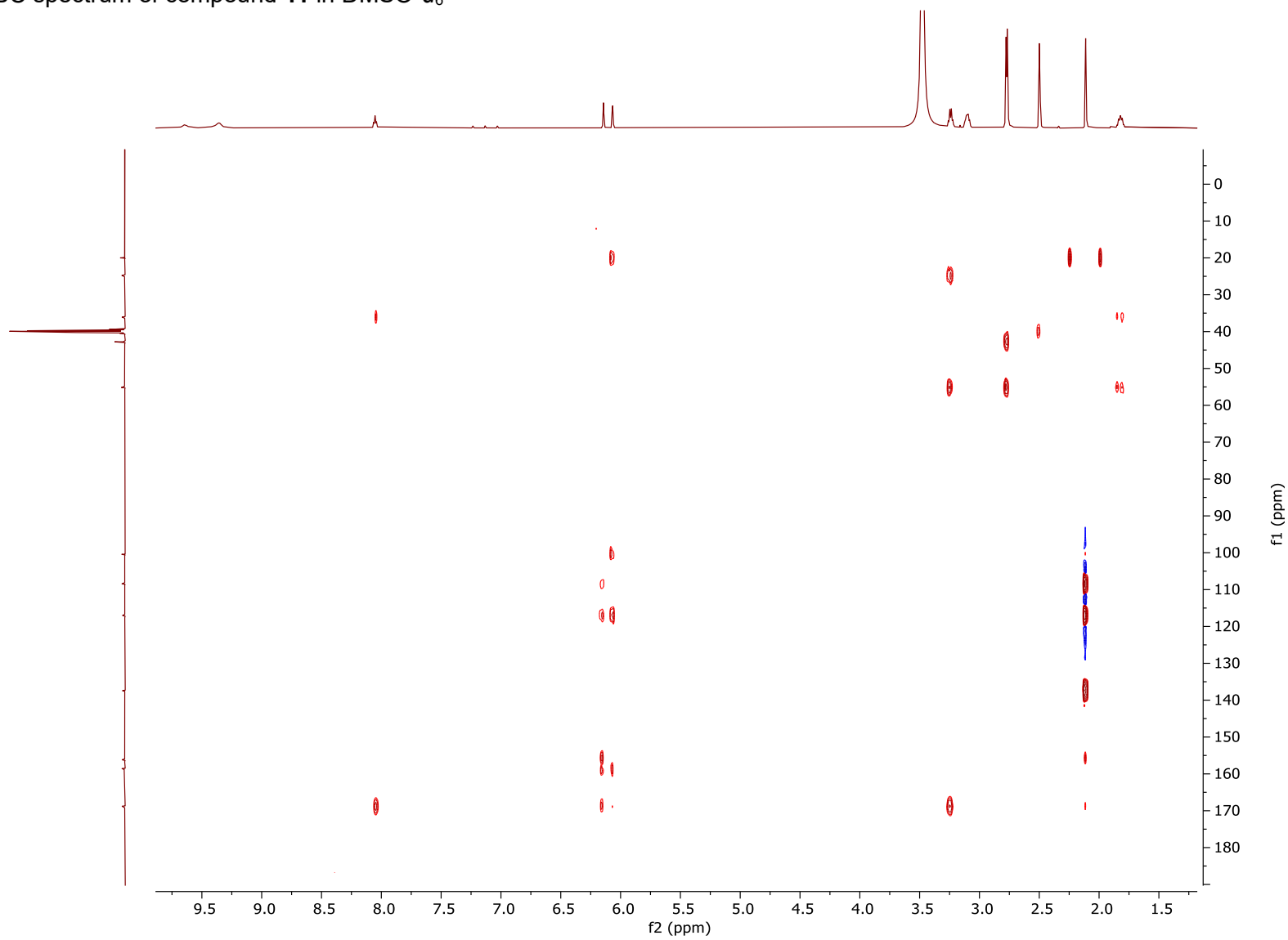
¹³C NMR spectrum of compound **11** in DMSO-*d*₆



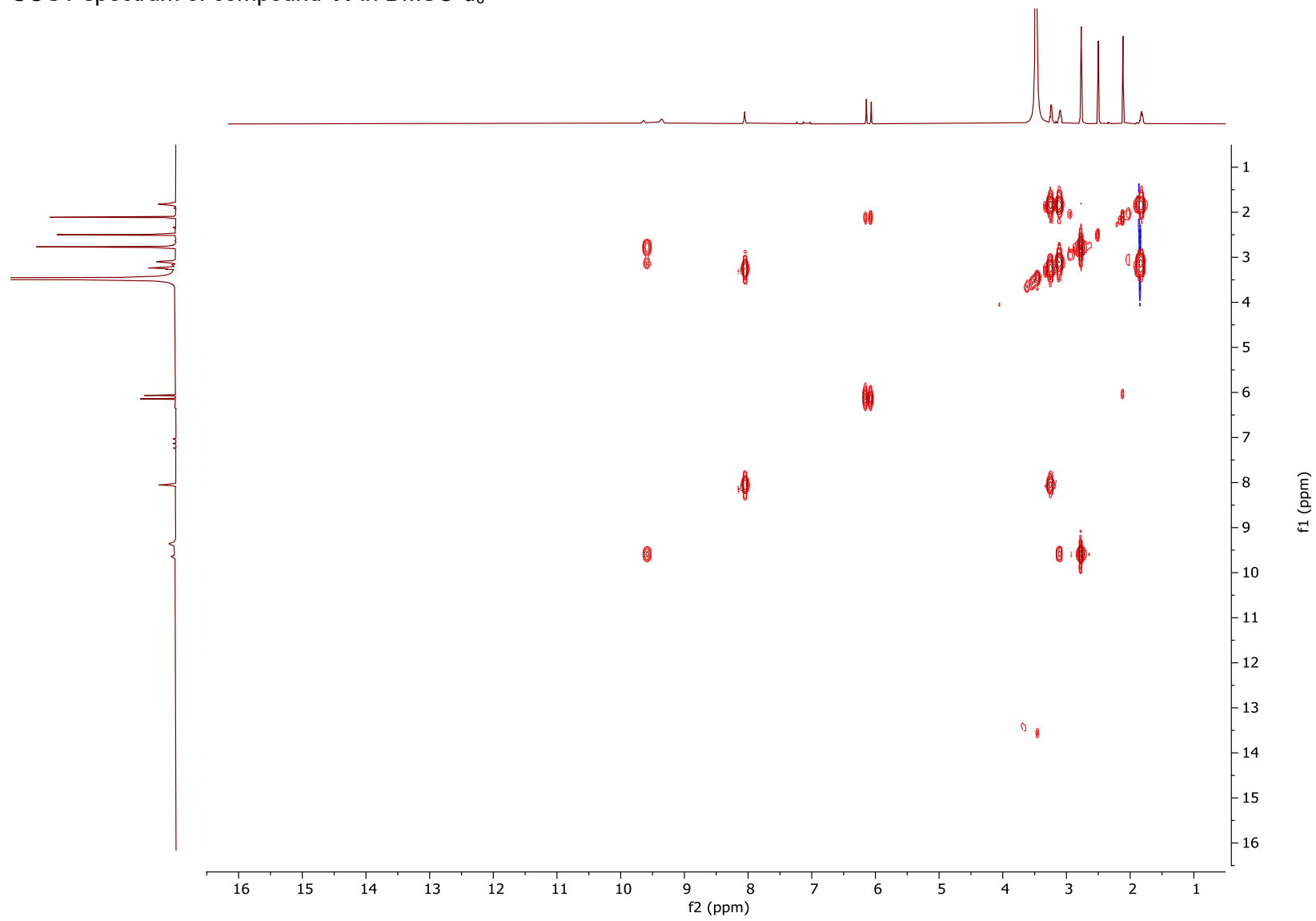
HSQC spectrum of compound **11** in DMSO-*d*₆



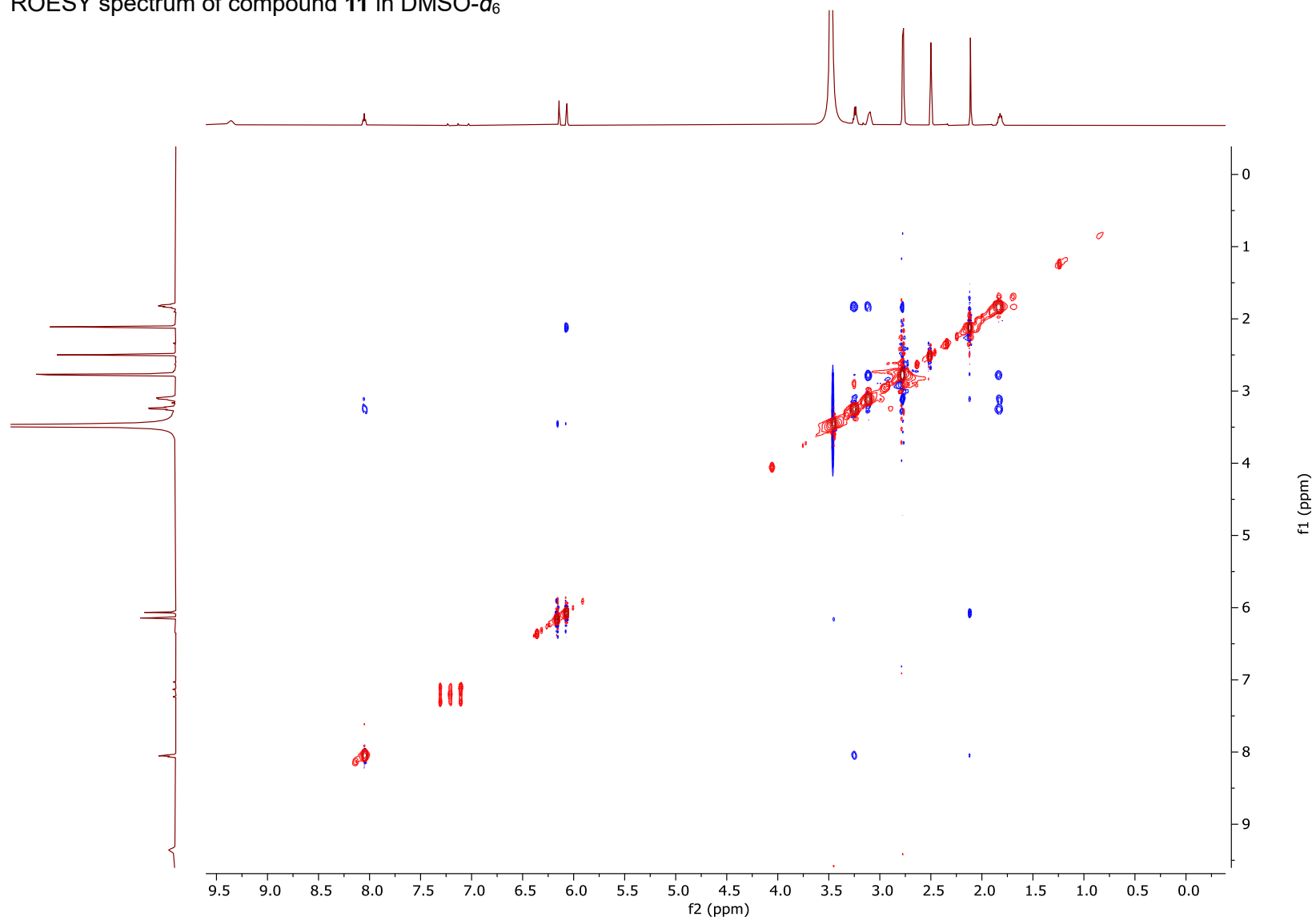
HMBC spectrum of compound **11** in DMSO-*d*₆



COSY spectrum of compound **11** in DMSO- d_6



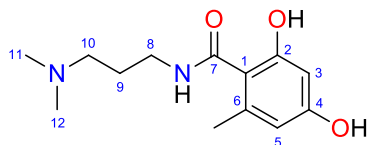
ROESY spectrum of compound **11** in DMSO- d_6



NMR data table for compound **11** in DMSO-*d*₆^{a, b}

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type	COSY	HMBC	ROESY
1		117.0, C			
2		156.2, C			
2-OH	9.71 (brs)		8,10,11,12		
3	6.16 (d, 2.1)	100.4, CH	5,6-Me ^w	1,2,4,5,7 ^w	
4		158.6, C			
4-OH	9.43 (brs)				
5	6.07 (d, 2.1)	108.5, CH	3,6-Me ^w	1,3,4 ^w ,6-Me,7 ^w	6-Me
6		137.4, C			
6-Me	2.12 (s)	20.0, CH ₃	3 ^w ,5 ^w	1,2 ^w ,3 ^w ,5,6,7 ^w	5,7-NH
7		168.8, C			
7-NH	8.05 (t, 5.8)		8	7,8 ^w	6-Me ^w ,8,9,10 ^w
8	3.25 (dt, 5.8, 6.2)	36.1, CH ₂	7-NH,9	7,9,10	7-NH,9,10
9	1.83 (dt, 6.2, 5.6)	24.8, CH ₂	8,10	8 ^w ,10 ^w	7-NH ^w ,8,10,11 ^w ,12 ^w
10	3.11 (tt, 5.6, 5.4)	55.2, CH ₂	9,10-NH		6-Me ^w ,7-NH ^w ,8,9,11 ^w ,12 ^w
10-NH	9.59 (brs) ^b		10,11,12		
11	2.78 (s)	42.8, CH ₃	10-NH	10,12	9,10
12	2.77 (s)	42.8, CH ₃	10-NH	10,11	9,10

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^b Purified as a TFA salt; ^w Weak correlation.



High resolution mass spectrum of compound 11

Mass Spectrum SmartFormula Report

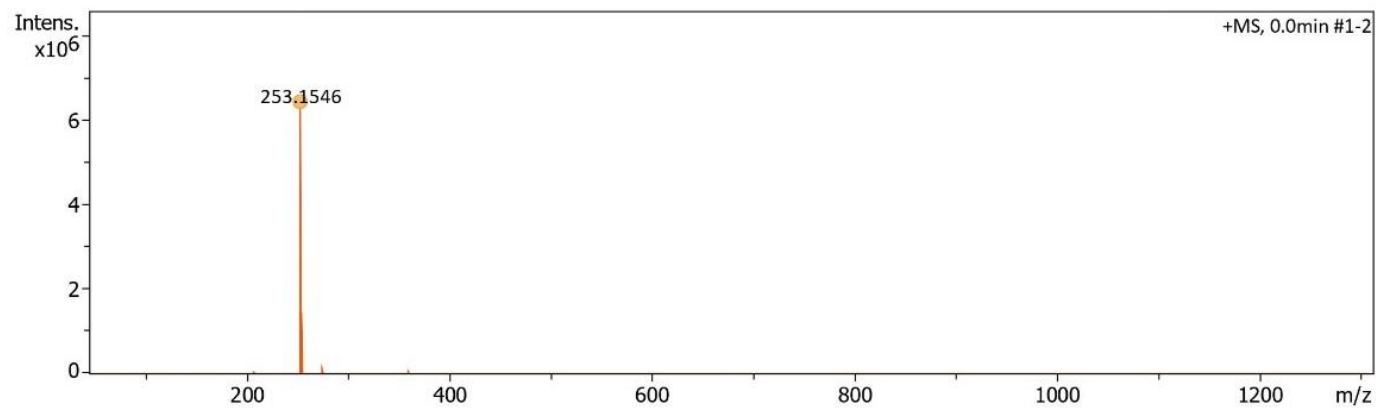
Analysis Info

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Method DirectInfusion_2018_pos.m
Sample Name RAD915J
Comment

Acquisition Date 10/4/2023 2:59:16 PM

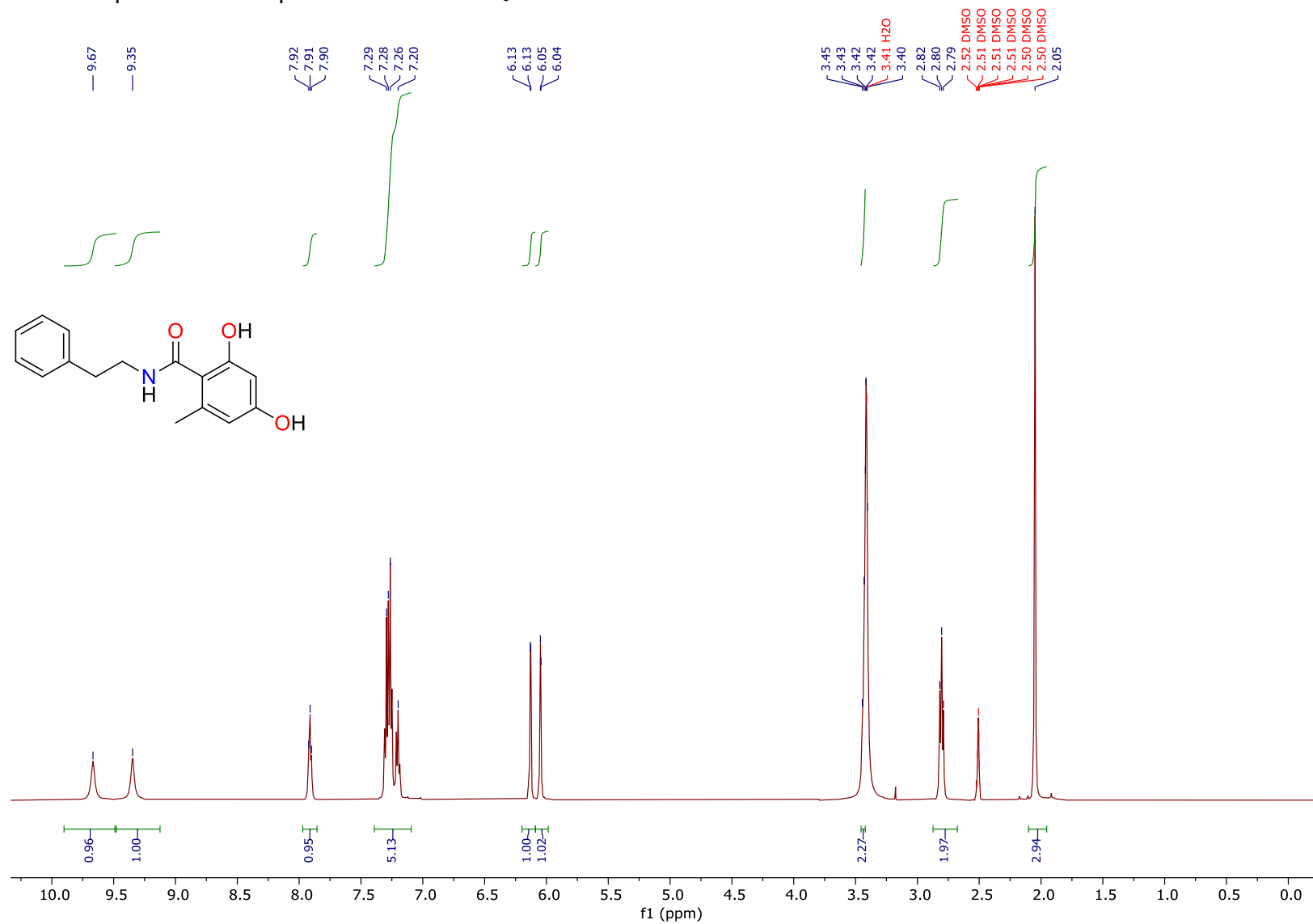
Operator Demo User
Instrument maXis II ETD

Acquisition Parameter

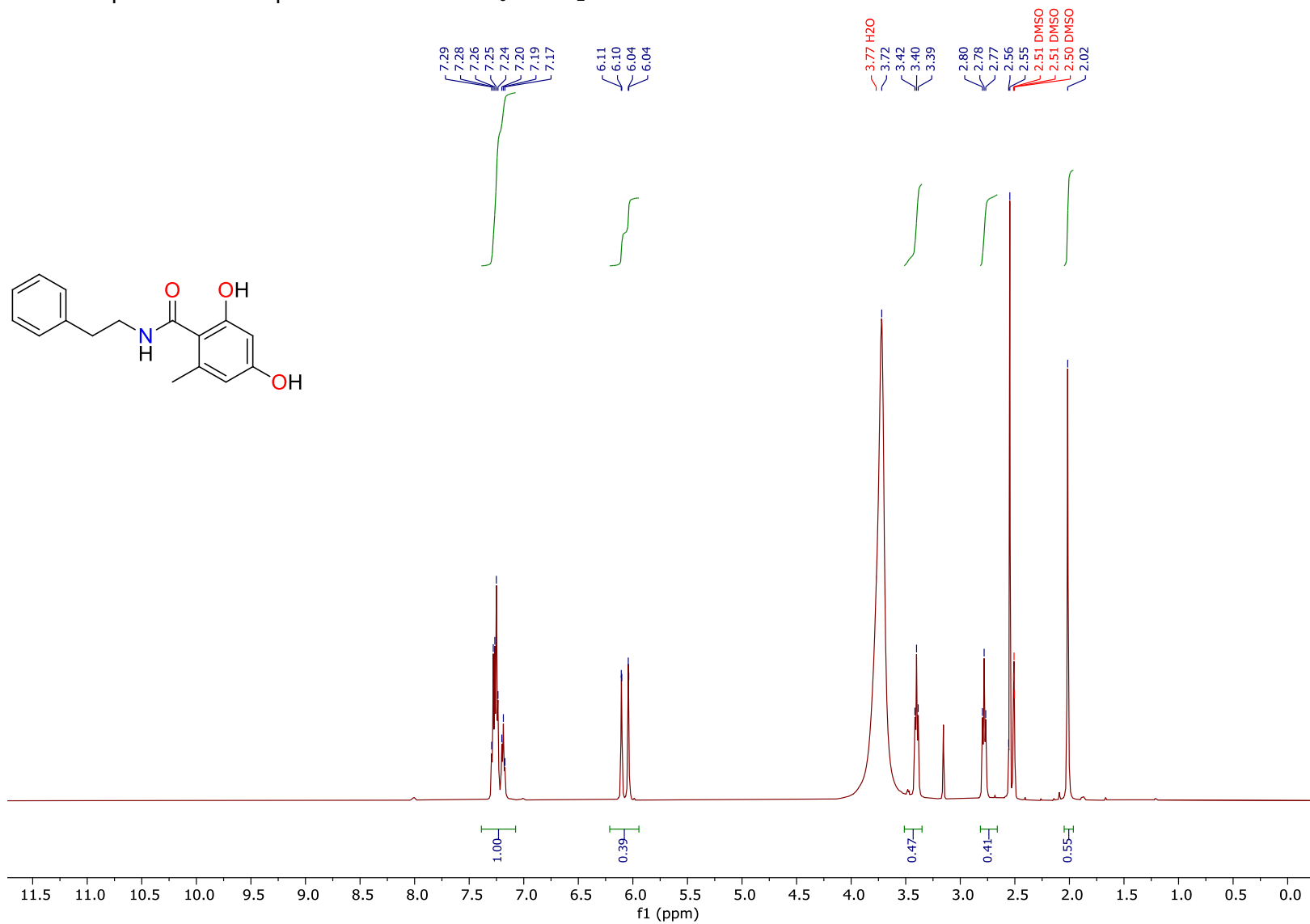


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
253.1546	1	C13H21N2O3	253.1547	0.1	48.6	1	100.00	5.0	even	ok

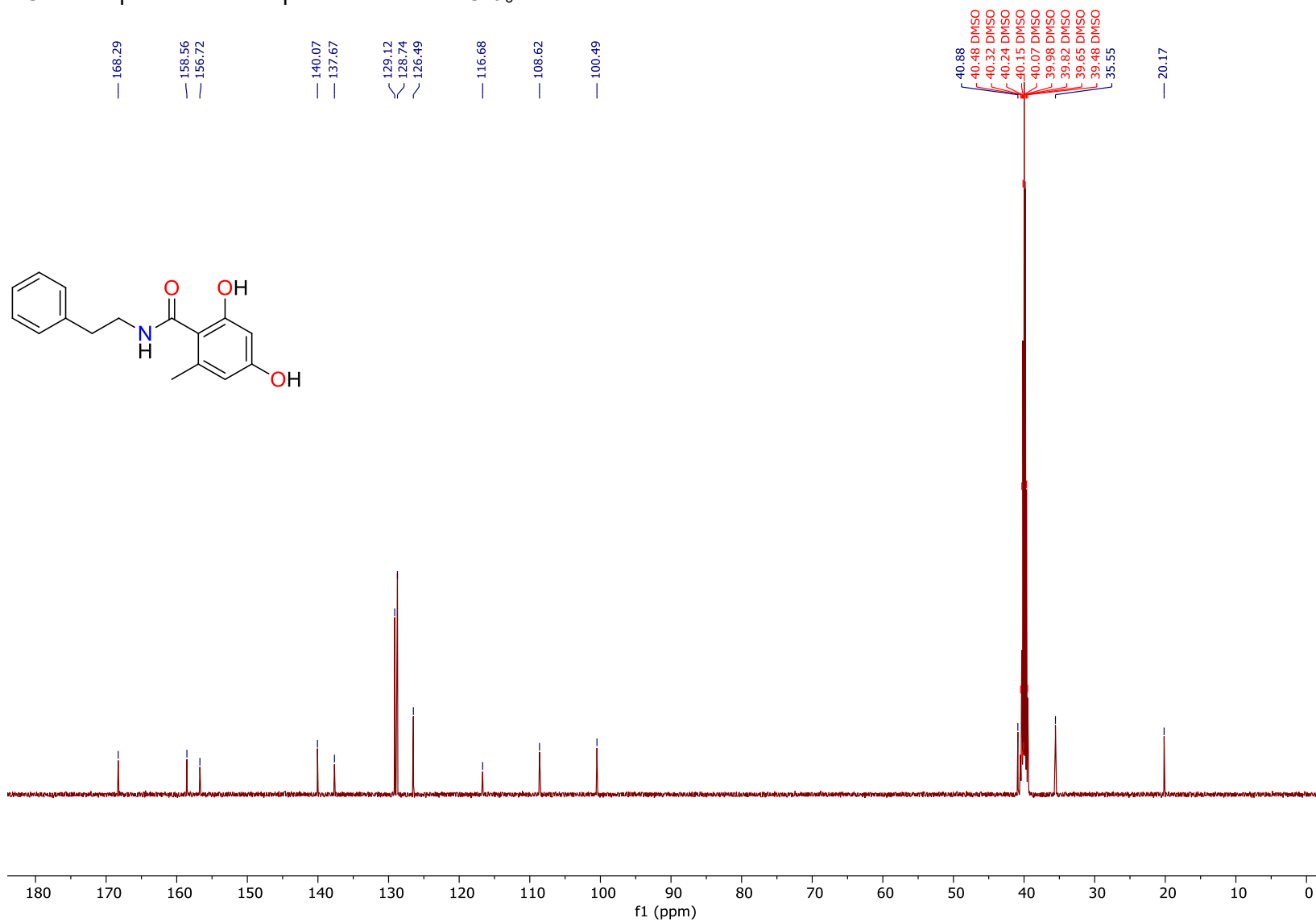
¹H NMR spectrum of compound **12** in DMSO-*d*₆



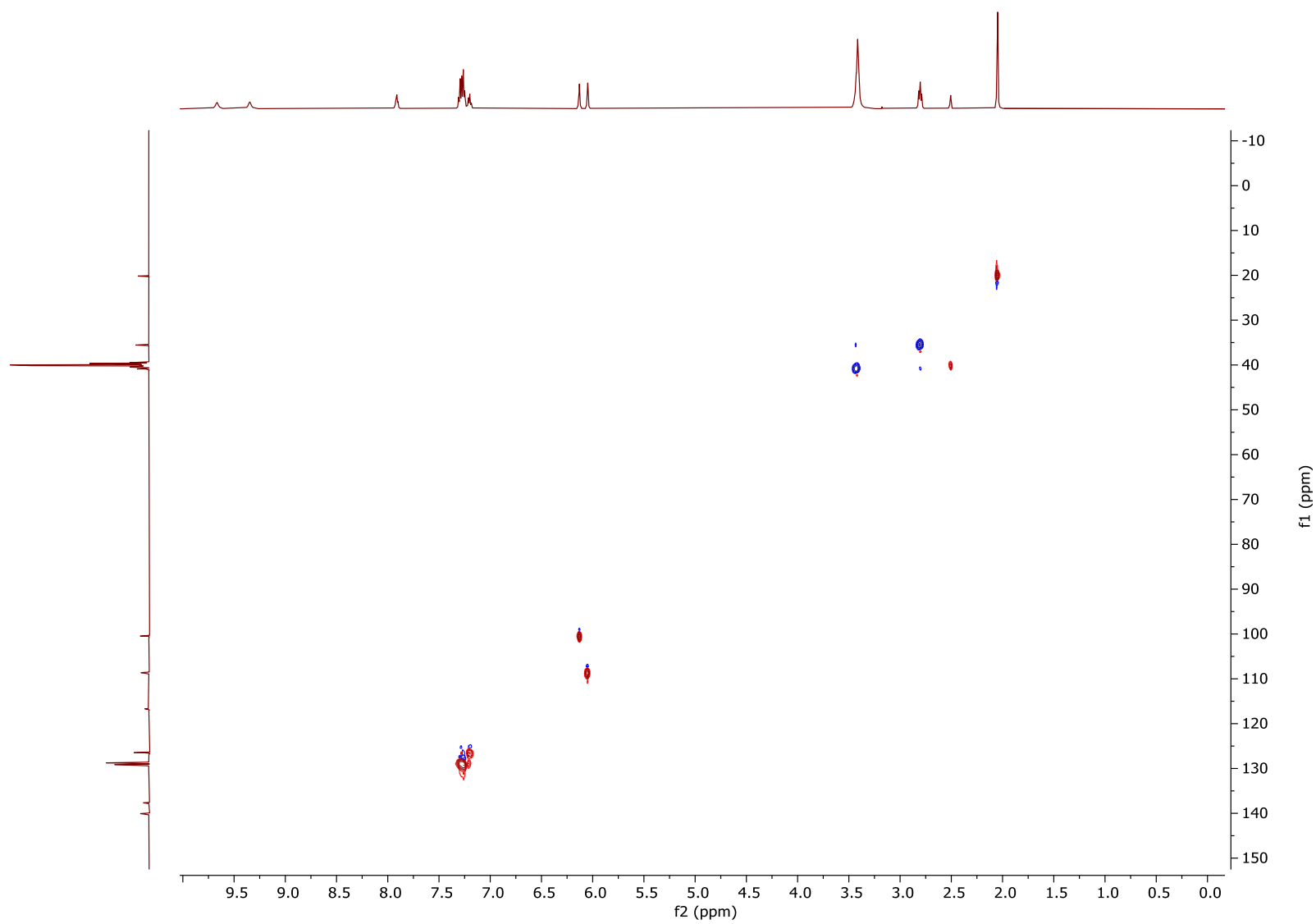
¹H NMR spectrum of compound **12** in DMSO-*d*₆ and D₂O



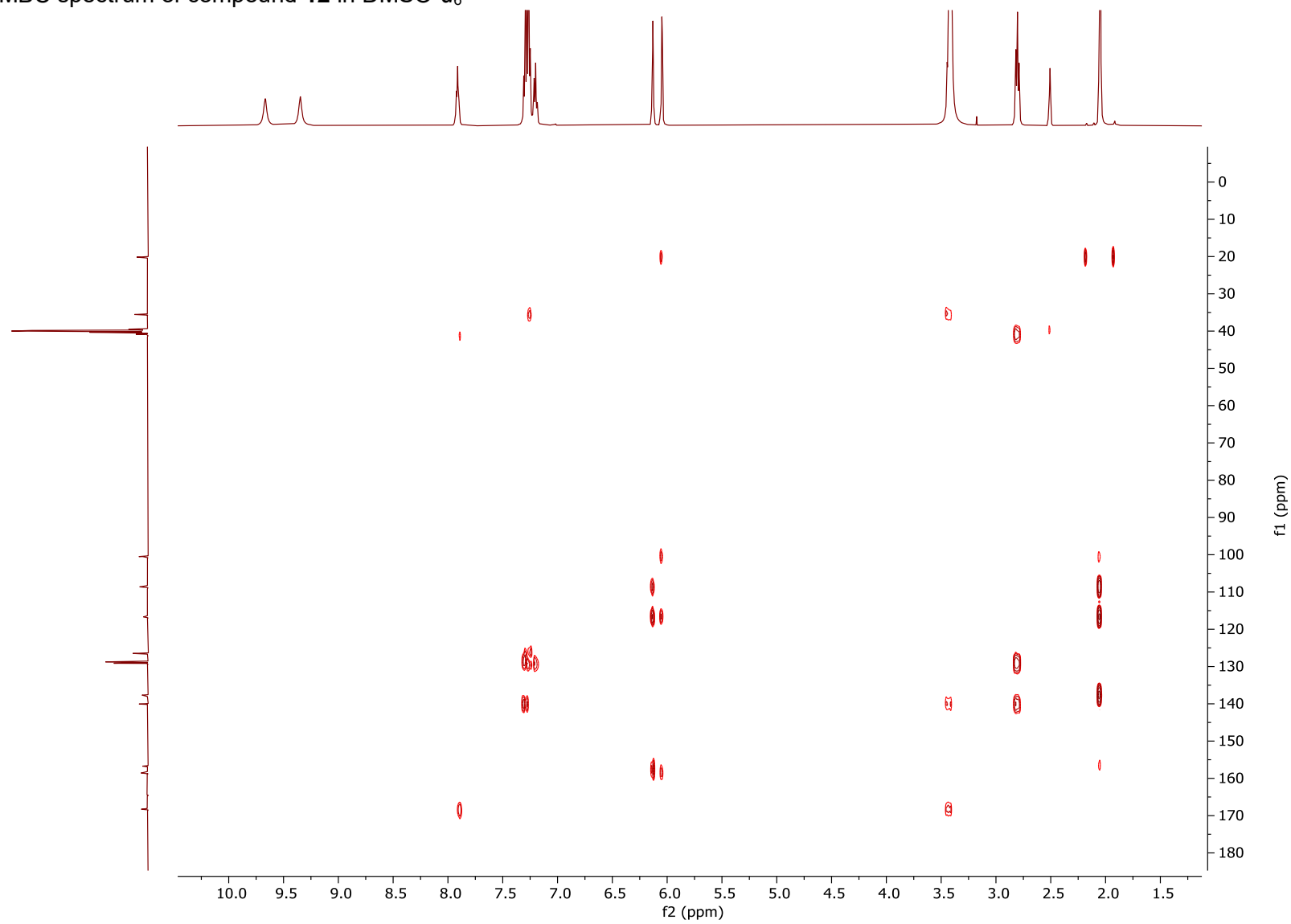
¹³C NMR spectrum of compound **12** in DMSO-*d*₆



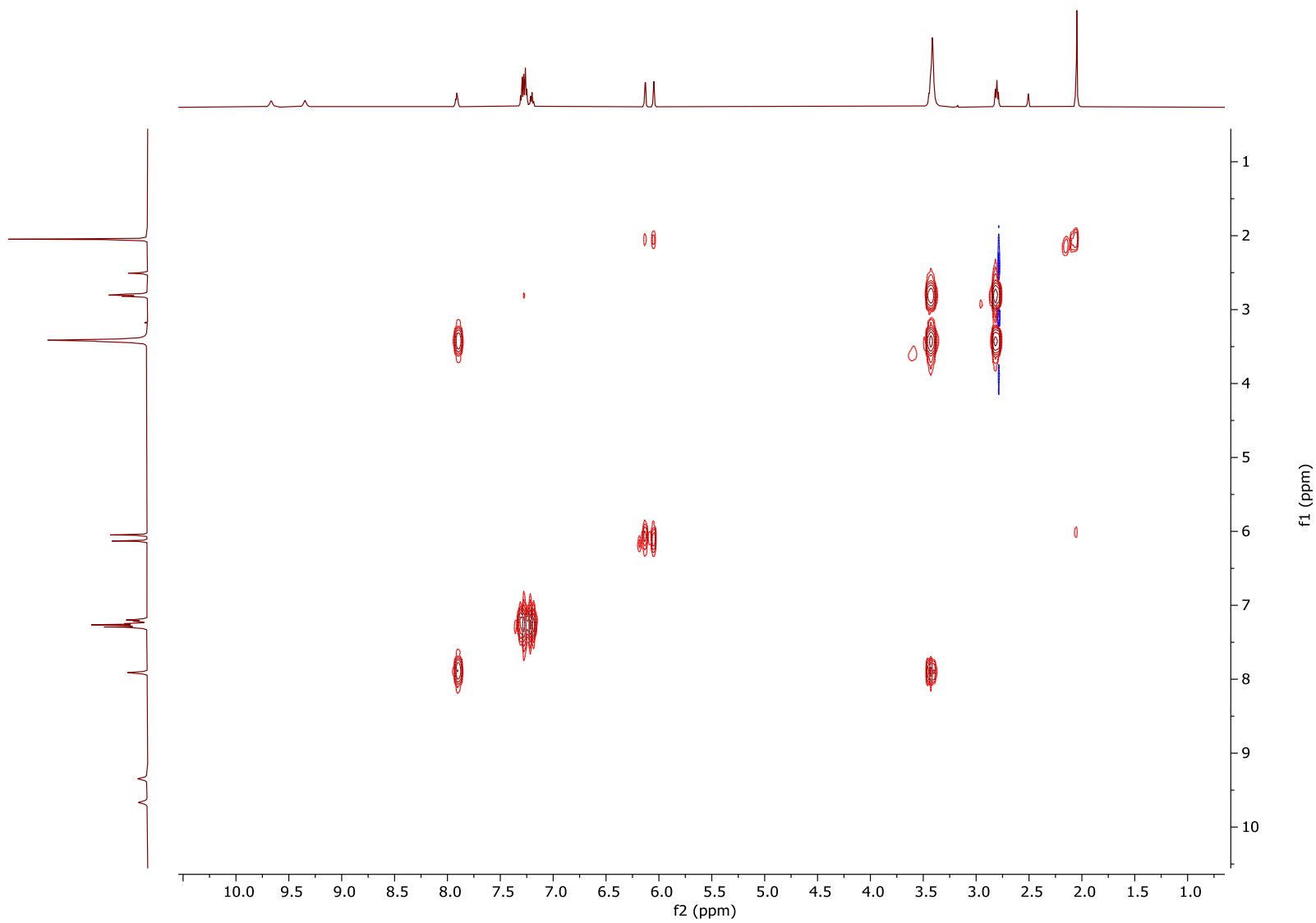
HSQC spectrum of compound **12** in DMSO- d_6



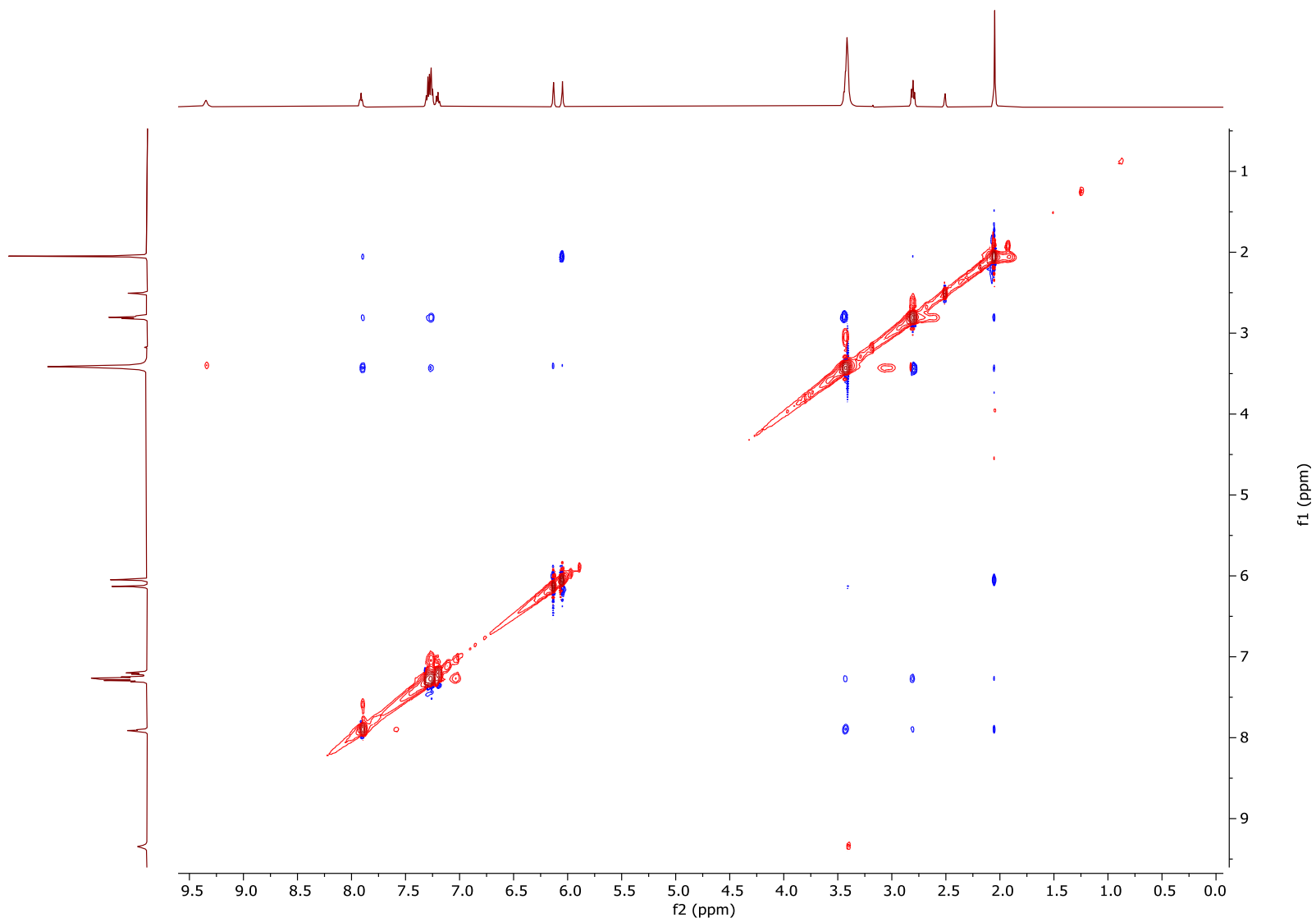
HMBC spectrum of compound **12** in DMSO-*d*₆



COSY spectrum of compound **12** in DMSO- d_6



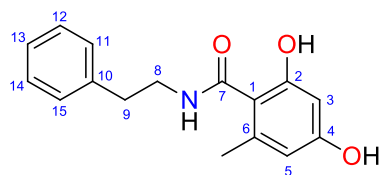
ROESY spectrum of compound **12** in DMSO- d_6



NMR data table for compound **12** in DMSO-*d*₆^a

Position	δ_{H} (mult., <i>J</i> in Hz)	δ_{C} , type	COSY	HMBC	ROESY
1		116.7, C			
2		156.7, C			
2-OH	9.67 (brs)				
3	6.13 (d, 2.0)	100.5, CH	5,6-Me,8 ^w	1,2,4,5,7 ^w	4-OH ^w ,6-Me ^w ,7-NH ^w ,8
4		158.6, C			
4-OH	9.35 (brs)				3,5
5	6.05 (d, 2.0)	108.6, CH	3,6-Me	1,2,3,6-Me,7 ^w	4-OH ^w ,6-Me
6		137.7, C			
6-Me	2.05 (s)	20.2, CH ₃	3,5	1,2,3 ^w ,5,6,7 ^w ,10	5,7-NH,8,9
7		168.3, C			
7-NH	7.91 (t, 5.5)		8	7,8	6-Me,8,9,15 ^w
8	3.42 (dt, 5.5, 7.4)	40.9, CH ₂	7-NH,9	7,9,10	3 ^w ,6-Me ^w ,7-NH,9,11,15
9	2.80 (t, 7.4)	35.6, CH ₂	8,11,13,15	8,10,11,15	6-Me,7-NH ^w ,8,11,15
10		140.1, C			
11	7.26 (br d, 7.3)	129.1, CH	9 ^w ,12	9,12,13,14,15	8,9
12	7.29 (br dd, 7.1, 7.3)	126.5, CH	11,13	10,11,14,15	
13	7.20 (tt, 7.1, 1.6)	128.7, CH	11,12,14,15	11,15	
14	7.29 (br dd, 7.1, 7.3)	126.5, CH	13,15	10,11,12,15	
15	7.26 (br d, 7.3)	129.1, CH	9 ^w ,14	9,11,12,13,14	8,9

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^w Weak correlation.



High resolution mass spectrum of compound **12**

Mass Spectrum SmartFormula Report

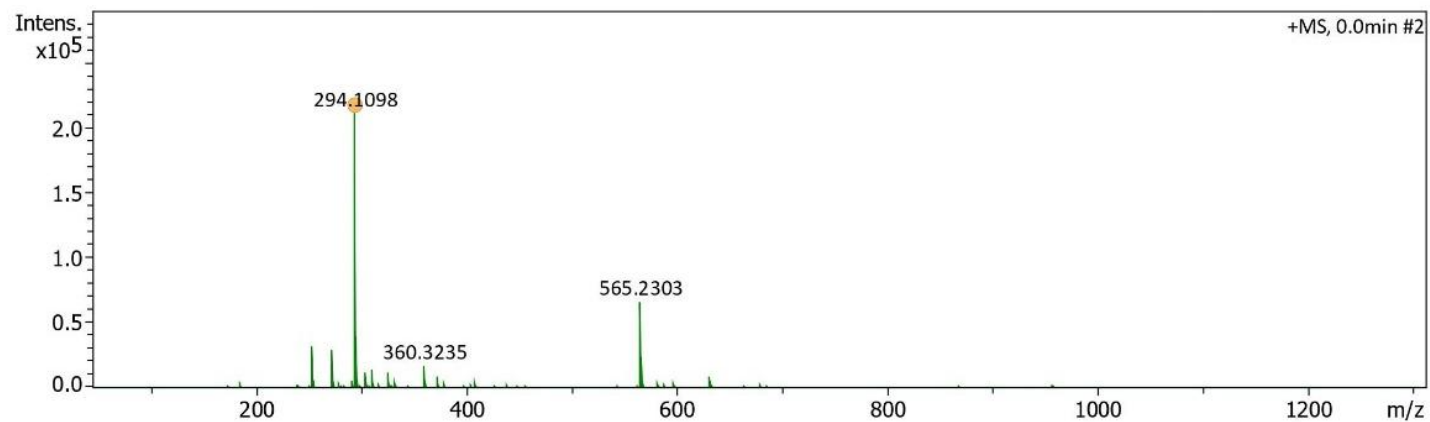
Analysis Info

Analysis Name D:\Data\Sasha\20231004\RAD915M000001.d
Method DirectInfusion_2018_pos.m
Sample Name RAD915M
Comment

Acquisition Date 10/4/2023 2:47:09 PM

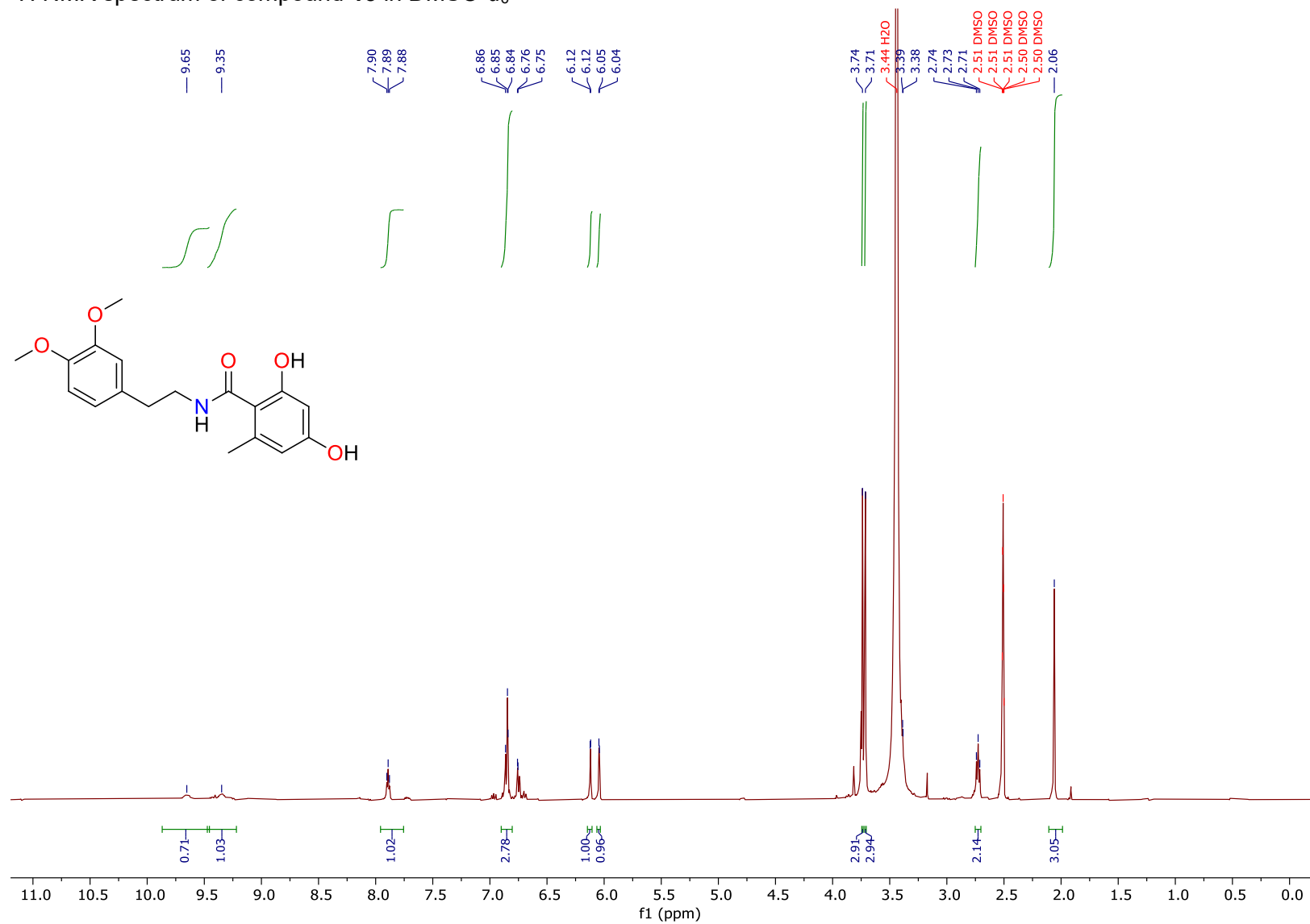
Operator Demo User
Instrument maXis II ETD

Acquisition Parameter

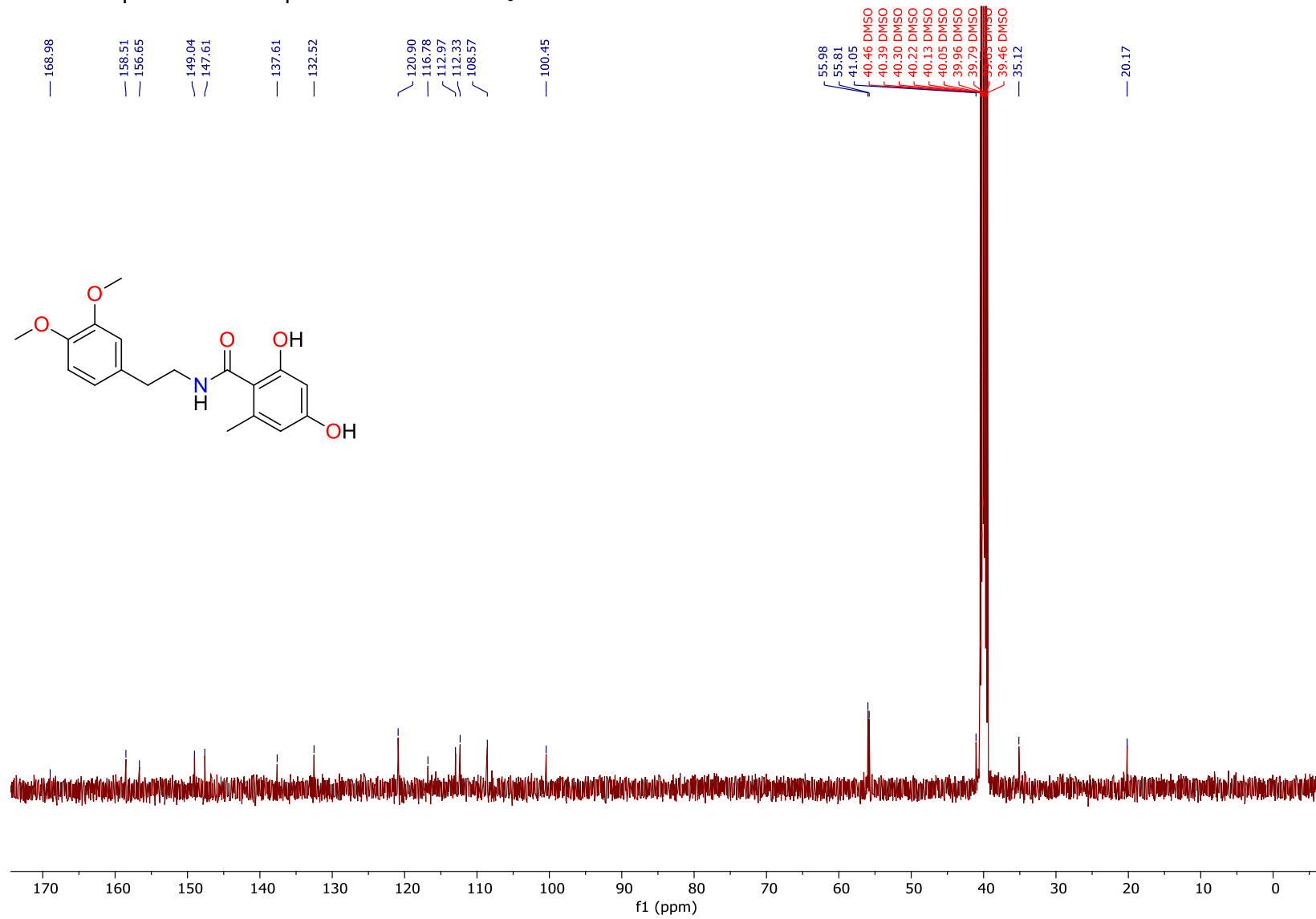


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
294.1098	1	C ₁₆ H ₁₇ NNaO ₃	294.1101	0.8	3.0	1	100.00	9.0	even	ok

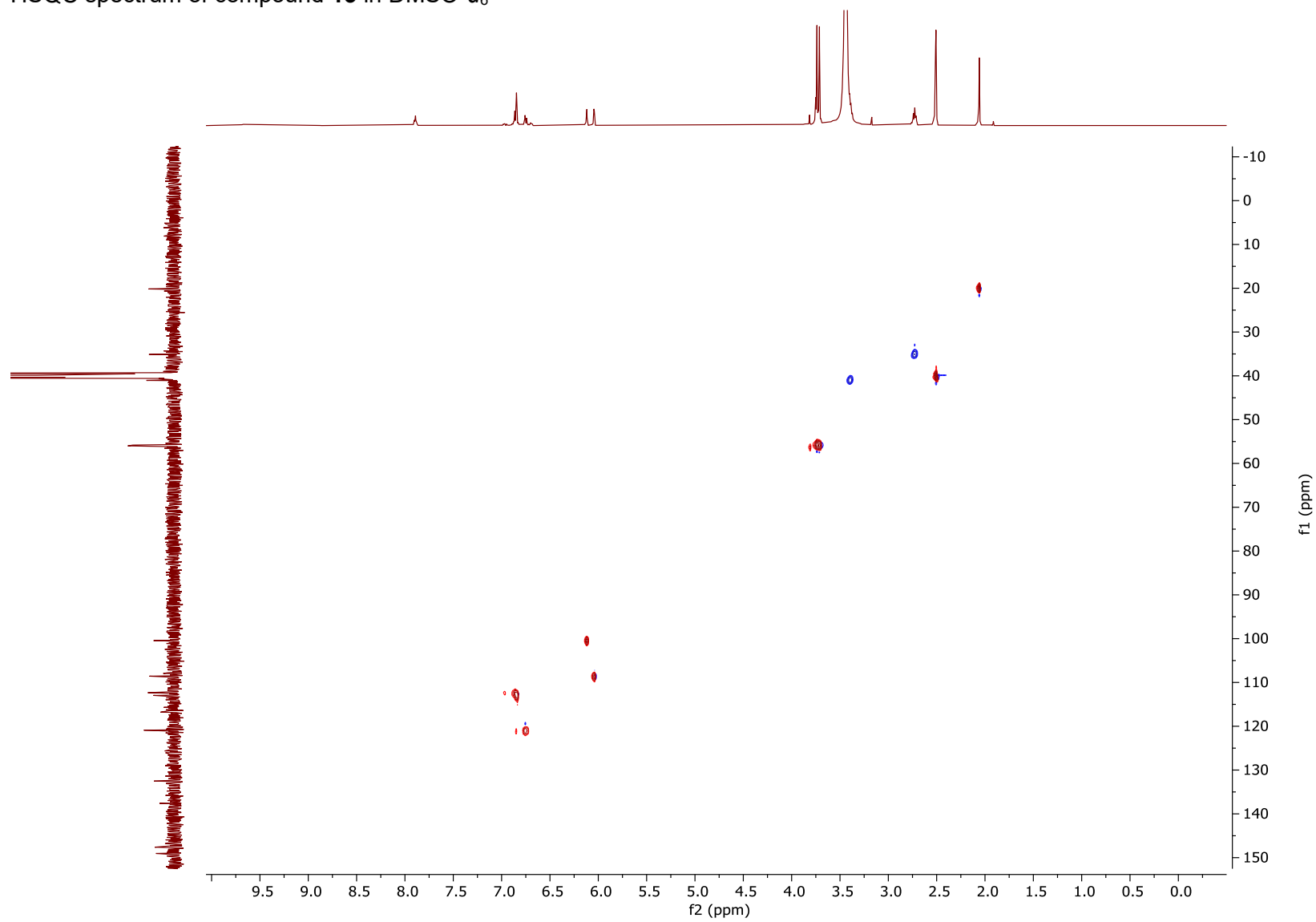
¹H NMR spectrum of compound **13** in DMSO-*d*₆



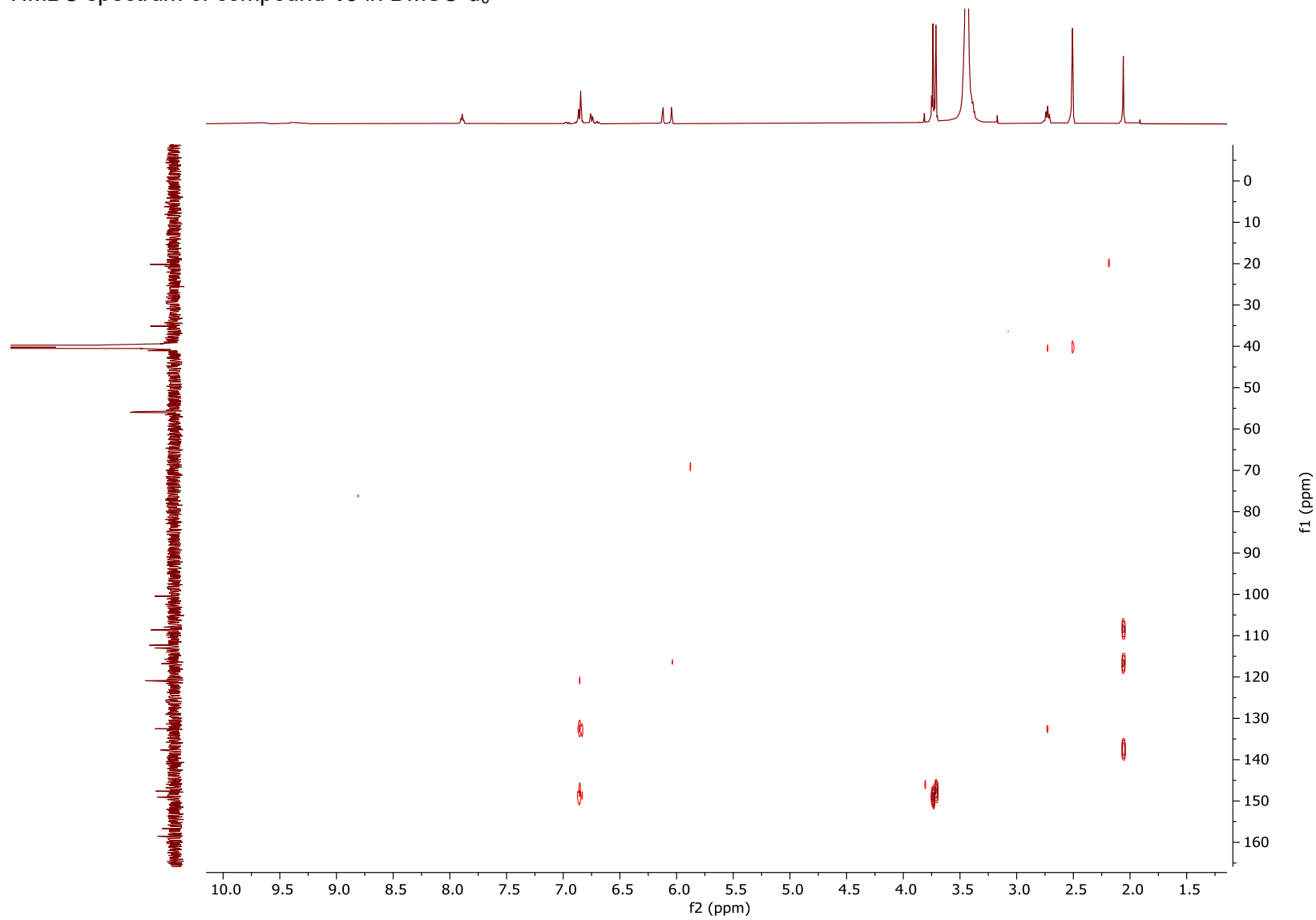
¹³C NMR spectrum of compound **13** in DMSO-d₆



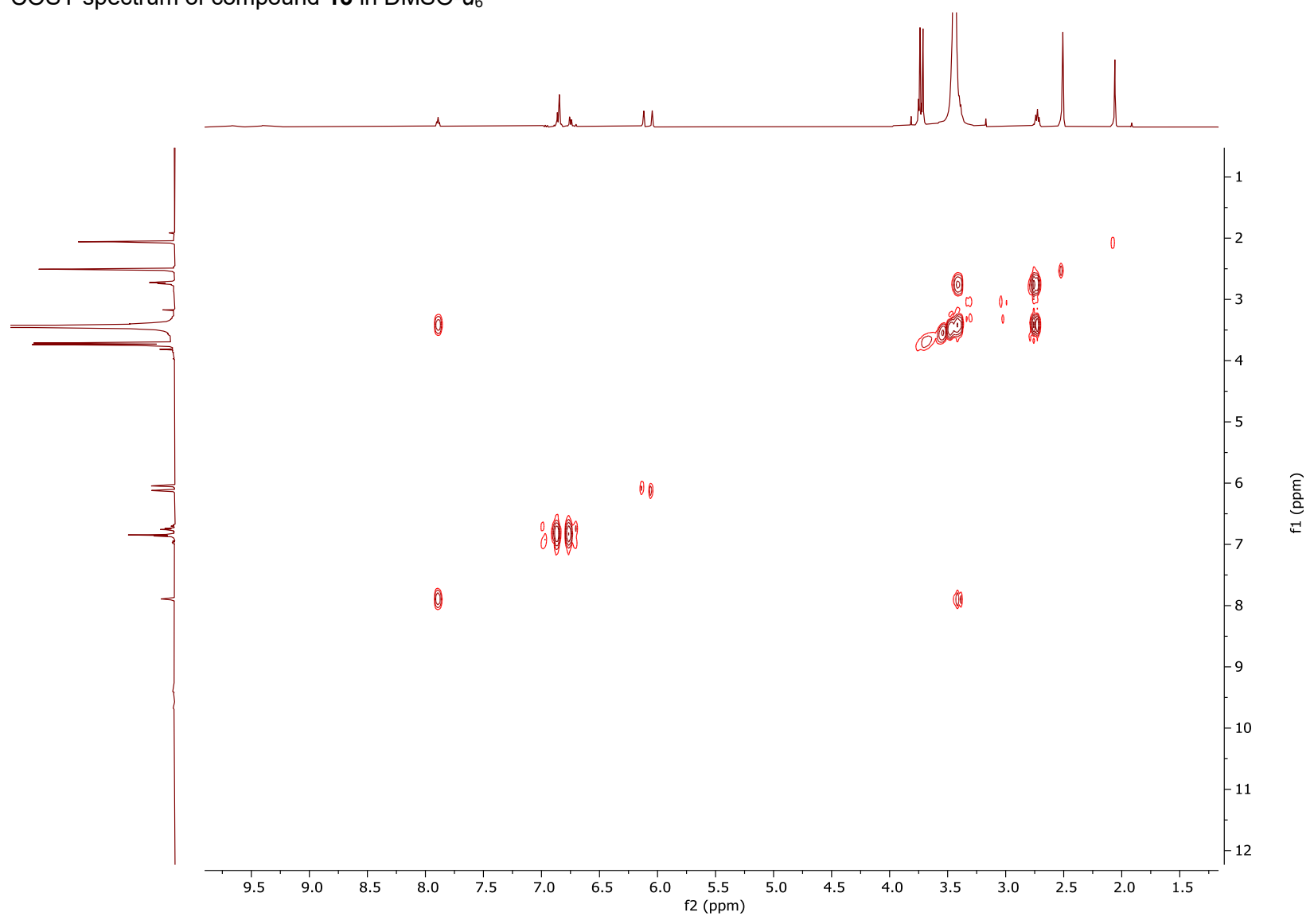
HSQC spectrum of compound **13** in DMSO- d_6



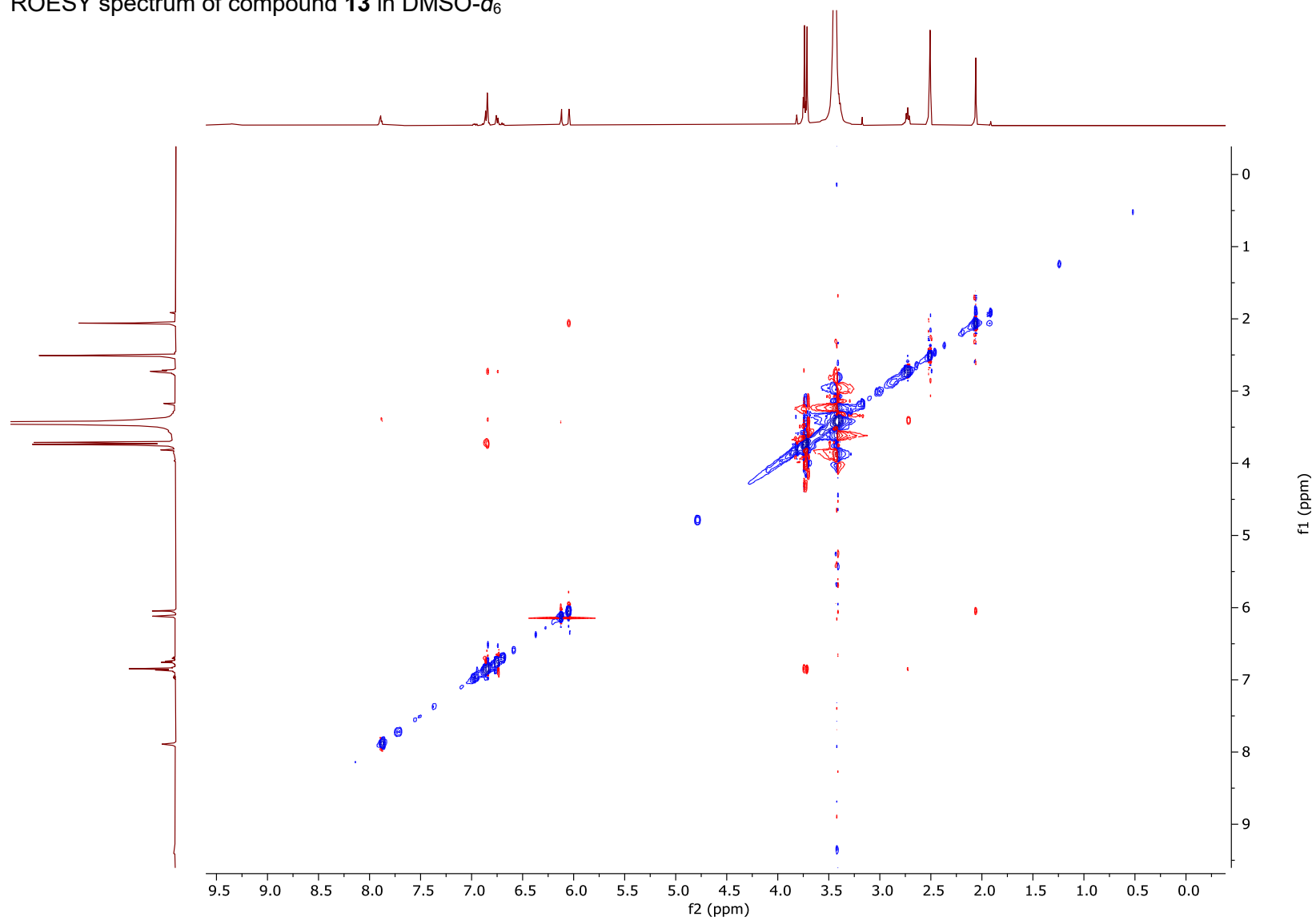
HMBC spectrum of compound **13** in DMSO-*d*₆



COSY spectrum of compound **13** in DMSO-*d*₆



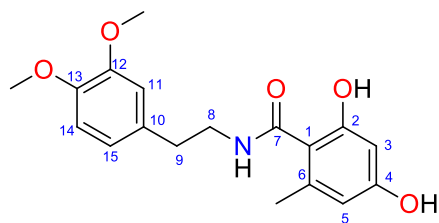
ROESY spectrum of compound **13** in DMSO- d_6



NMR data table for compound **13** in DMSO- d_6^a

Position	δ_H (mult., J in Hz)	δ_C , type	COSY	HMBC	ROESY
1		116.8, C			
2		156.7, C			
2-OH	9.65 (brs)				
3	6.12 (d, 2.0)	100.5, CH	5,6-Me ^w	1	
4		158.5, C			
4-OH	9.35 (brs)				
5	6.04 (d, 2.0)	108.6, CH	3,6-Me ^w	1,6-Me ^w	6-Me
6		137.6, C			
6-Me	2.06 (s)	20.2, CH ₃	5	1,5,6	5
7		169.0, C			
7-NH	7.89 (t, 5.5)	-	8		8
8	3.39 (dt, 5.5, 7.4)	41.1, CH ₂	7-NH,9	9	7-NH,8,9,11,15
9	2.73 (t, 7.4)	35.1, CH ₂	8	8,10,11 ^w ,15	8,11,15
10		132.5, C			
11	6.84 (d, 2.0)	113.0, CH	9 ^w	10,12,15	8,9,12-OMe
12		149.0, C			
12-OMe	3.71 (brs)	56.0, CH ₃	11	12	11
13		147.6, C			
13-OMe	3.74 (brs)	55.8, CH ₃	12-OMe	13	14
14	6.86 (d, 8.0)	112.3, CH	15	10,12 ^w	13-OMe
15	6.75 (dd, 2.0, 8.0)	120.9, CH		14 ^w	9

^a Spectra recorded at 25 °C (500 MHz for ¹H NMR and 125 MHz for ¹³C NMR); ^w Weak correlation.



High resolution mass spectrum of compound 13

Mass Spectrum SmartFormula Report

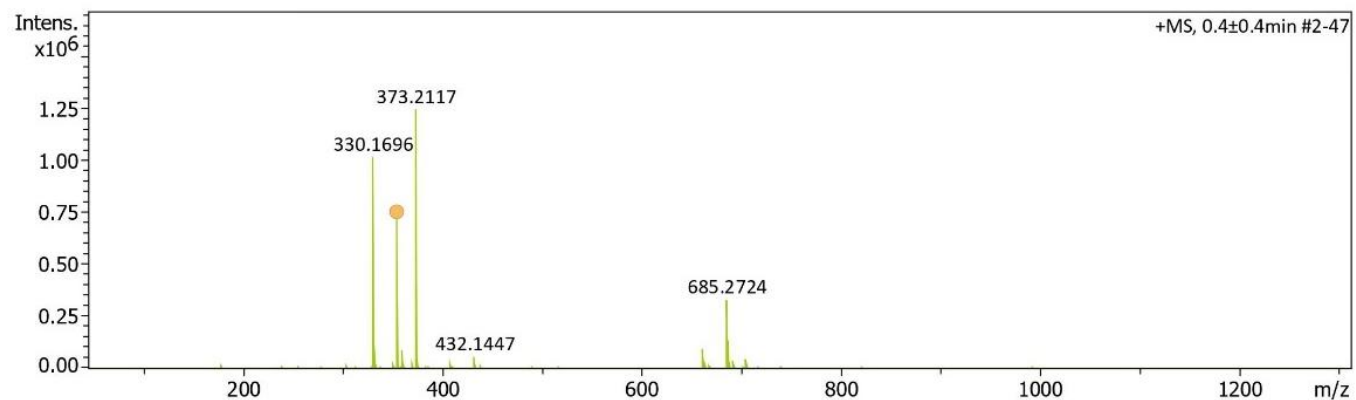
Analysis Info

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Method DirectInfusion_2018_pos.m
Sample Name RAD915I
Comment

Acquisition Date 10/4/2023 2:28:42 PM

Operator Demo User
Instrument maXis II ETD

Acquisition Parameter



Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# mSigma	Score	rdb	e ⁻ Conf	N-Rule
354.1308	1	C ₁₈ H ₂₁ NNaO ₅	354.1312	1.2	0.7	1	100.00	9.0	even	ok
	2	C ₁₉ H ₁₇ N ₅ NaO	354.1325	5.0	14.1	2	35.81	14.0	even	ok