Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary Information for

Discovery and Biological Evaluation of Dispirocyclic and Polycyclic *ent*-Clerodane Dimers from *Isodon scoparius* as Novel Inhibitors of Toll-like Receptor Signaling

Xing-Ren Li,[1,‡] Li Chen,[2,3,‡] Kun Hu,[1] Bing-Chao Yan,[1] Xue Du,[1] Xiao-Nian Li,[1] Han-Dong Sun,[1] Shi-Jun He,[2,3,*] and Pema-Tenzin Puno[1,*]

[1]*State Key Laboratory of Phytochemistry and Plant Resources in West China and Yunnan Key Laboratory of Natural Medicinal Chemistry, Kunming Institute of Botany, Chinese Academy of Sciences, Kunming 650201, China;*

[2]*Laboratory of Immunopharmacology, State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica, Chinese Academy of Sciences, Shanghai 201203, China;*

[3]*University of Chinese Academy of Sciences, Beijing 100049, China.*

[*]Corresponding author Tel.: +86-871-65223616; E-mail: punopematenzin@mail.kib.ac.cn (P.-T. Puno); E-mail: heshijun@simm.ac.cn (S. He).

Contents of Supplementary Information

1. General experimental procedures

Melting points were recorded on an RDY-1B micro melting point apparatus. Optical rotation spectra were record on an Autopol VI automatic polarimeter (Manufactured by Rudolph Research Analytical) and an Jasco P-1020 digital polarimeter. UV spectra were obtained on a Shimadzu UV-2401PC spectrophotometer. IR spectra were recorded on BRUKER Tensor-27 Fourier Transform and Thermo NICOLET iS10 mid infrared spectrometers using KBr pellets. Mass spectra were recorded on an Agilent 1290 UPLC/6540 Q-TOF and a Shimadzu UPLC-IT-TOF spectrometers. ECD spectra were recorded on an Applied Photophysics digit circular dichroism spectrometer. X-ray diffraction was realized on Bruker APEX DUO, Bruker D8 Quest and Bruker D8 VENTURE crystallography systems. 1D and 2D NMR spectra were recorded on Bruker Avance III 500, Avance III 600 and AV 800 spectrometers using tetramethylsilane (TMS) as the internal standard. Unless otherwise specified, chemical shifts (δ) were expressed in ppm with reference to solvent signals (pyridine-d_5 δ 7.19 ^{1}H NMR, 123.40 ^{13}C NMR; acetone-d_6 δ 2.05 ^{1}H NMR, 29.92 ^{13}C NMR). Data are reported as follows: chemical shifts (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constants (Hz), and integration.

Column chromatography was performed with silica gel (80–100 mesh; Qingdao Marine Chemical, Inc., Qingdao, People's Republic of China), washed by acidified solvent with formic acid (85% w/w, 0.25 mL HCO_2H/L solvent). Semi-preparative HPLC was performed on an Agilent 1200 liquid chromatograph with a Zorbax SB-C18 (9.4 mm × 250 mm) column. Fractions and reactions were monitored by TLC carried out on glassy TLC plates (silica gel 60 coated with GF254, 250 mm, Qingdao Marine Chemical, Inc., Qingdao, People's Republic of China; Silica gel 60 coated with F254, 10 × 20 cm, Merck, Darmstadt, Germany), and spots were visualized by using UV light and heating silica gel plates sprayed with 10% H_2SO_4 in EtOH or ammonium cerium nitrate/ammonium molybate.

2. X-ray crystal data

Crystal data for **1**: $C_{40}H_{60}O_{10}$•2(CH_4O), M = 764.96, a = 7.90610(10) Å, b = 21.2390(3) Å, c = 24.4951(4) Å, α = 90°, β = 90°, γ = 90°, V = 4113.16(10) Å^3, T = 100(2) K, space group $P212121$, Z = 4, μ(CuKα) = 0.726 mm^{-1}, 26002 reflections measured, 7380 independent reflections (R_{int} = 0.0338). The final R_1 values were 0.0546 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.1471 ($I > 2\sigma(I)$). The final R_1 values were 0.0550 (all data). The final $wR(F^2)$ values were 0.1475 (all data). The goodness of fit on F^2 was 1.097. Flack parameter = 0.07(4).

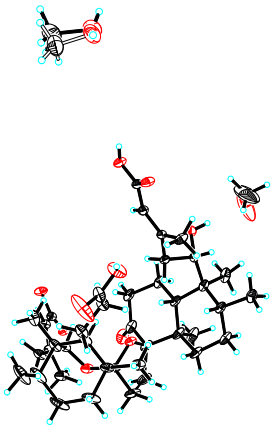


Figure S1. View of the molecules in an asymmetric unit
Displacement ellipsoids are drawn at the 25% probability level.

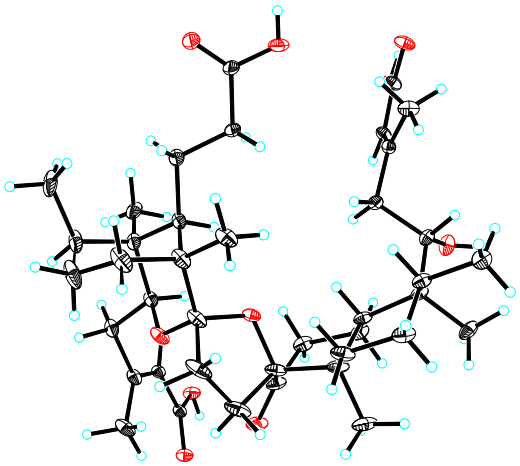


Figure S2. View of a molecule of **1** with the atom-labelling scheme
Displacement ellipsoids are drawn at the 30% probability level.

Table S1. Crystal data and structure refinement for **1**

Identification code	cu_**1**_0m	
Empirical formula	$C_{42}H_{68}O_{12}$	
Formula weight	764.96	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.90610(10) Å	α= 90°.
	b = 21.2390(3) Å	β= 90°.
	c = 24.4951(4) Å	γ= 90°.
Volume	4113.16(10) $Å^3$	
Z	4	
Density (calculated)	1.235 Mg/m^3	
Absorption coefficient	0.726 mm^{-1}	
F(000)	1664	
Crystal size	0.540 x 0.300 x 0.150 mm^3	
Theta range for data collection	2.754 to 70.183°.	
Index ranges	-9<=h<=9, -25<=k<=24, -29<=l<=28	
Reflections collected	26002	
Independent reflections	7380 [R(int) = 0.0338]	
Completeness to theta = 67.679°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7380 / 48 / 524	
Goodness-of-fit on F^2	1.097	
Final R indices [I>2sigma(I)]	R1 = 0.0546, wR2 = 0.1471	
R indices (all data)	R1 = 0.0550, wR2 = 0.1475	
Absolute structure parameter	0.07(4)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.544 and -0.611 $e.Å^{-3}$	

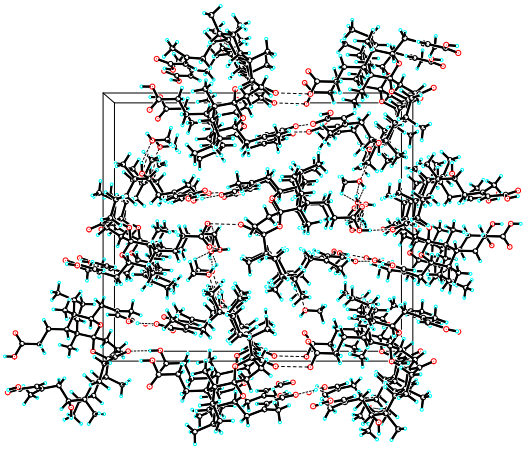


Figure S3. View of the pack drawing of **1**
Hydrogen-bonds are shown as dashed lines.

Crystal data for **2**: $C_{41}H_{62}O_{10}$, M = 714.90, a = 7.8521(3) Å, b = 18.0628(7) Å, c = 27.6909(10) Å, α = 90°, β = 90°, γ = 90°, V = 3927.4(3) Å^3, T = 130(2) K, space group $P212121$, Z = 4, μ(CuKα) = 0.689 mm^{-1}, 68531 reflections measured, 7766 independent reflections (R_{int} = 0.0342). The final R_1 values were 0.0282 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.0760 ($I > 2\sigma(I)$). The final R_1 values were 0.0284 (all data). The final $wR(F^2)$ values were 0.0762 (all data). The goodness of fit on F^2 was 1.026. Flack parameter = 0.00(2).

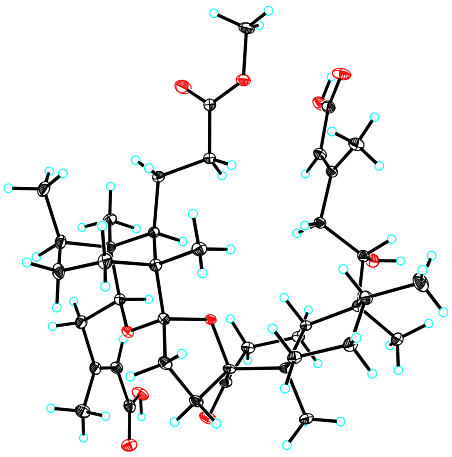


Figure S4. View of a molecule of **2** with the atom-labelling scheme
Displacement ellipsoids are drawn at the 26% probability level.

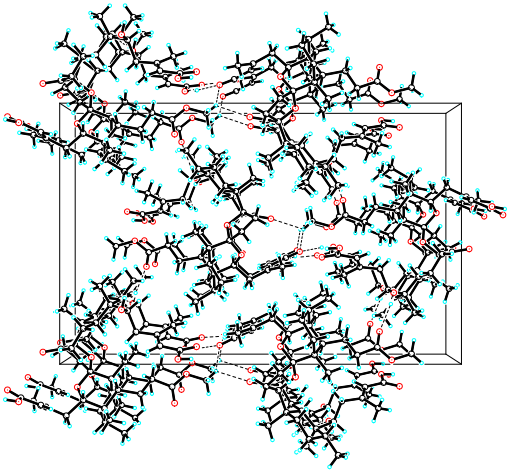


Figure S5. View of the pack drawing of **2**
Hydrogen-bonds are shown as dashed lines.

Table S2. Crystal data and structure refinement for **2**

Identification code	cu_**2**_0m	
Empirical formula	$C_{41}H_{62}O_{10}$	
Formula weight	714.90	
Temperature	130(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.8521(3) Å	α= 90°.
	b = 18.0628(7) Å	β= 90°.
	c = 27.6909(10) Å	γ= 90°.
Volume	3927.4(3) $Å^3$	
Z	4	
Density (calculated)	1.209 Mg/m^3	
Absorption coefficient	0.689 mm^{-1}	
F(000)	1552	
Crystal size	0.210 x 0.170 x 0.080 mm^3	
Theta range for data collection	2.921 to 72.340°.	
Index ranges	-9<=h<=8, -21<=k<=22, -34<=l<=32	
Reflections collected	68531	
Independent reflections	7766 [R(int) = 0.0342]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7766 / 0 / 478	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0282, wR2 = 0.0760	
R indices (all data)	R1 = 0.0284, wR2 = 0.0762	
Absolute structure parameter	0.00(2)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.223 and -0.150 e.$Å^{-3}$	

Crystal data for **10**: $C_{40}H_{54}O_7$•2(H_2O), M = 682.86, a = 7.2339(3) Å, b = 20.5982(8) Å, c = 23.5913(9) Å, α = 90°, β = 90°, γ = 90°, V = 3515.2(2) $Å^3$, T = 100.(2) K, space group $P212121$, Z = 4, μ(Cu Kα) = 0.725 mm^{-1}, 28938 reflections measured, 6645 independent reflections (R_{int} = 0.1138). The final R_1 values were 0.0527 ($I > 2\sigma(I)$). The final $wR(F^2)$ values were 0.1377 ($I > 2\sigma(I)$). The final R_1 values were 0.0761 (all data). The final $wR(F^2)$ values were 0.1452 (all data). The goodness of fit on F^2 was 1.071. Flack parameter = 0.00(10).

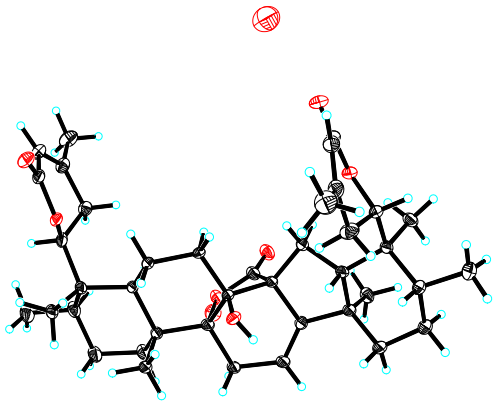


Figure S6. View of the molecules of **10** in an asymmetric unit
Displacement ellipsoids are drawn at the 30% probability level.

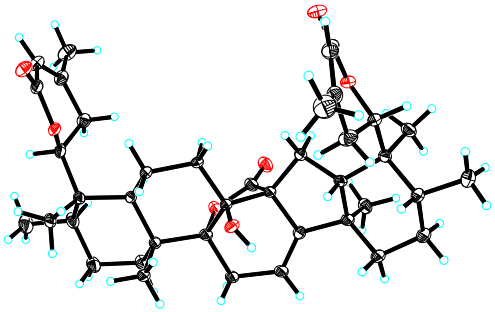


Figure S7. View of a molecule of **10** with the atom-labelling scheme
Displacement ellipsoids are drawn at the 30% probability level.

Table S3. Crystal data and structure refinement for **10**

Identification code	global	
Empirical formula	$C_{40}H_{58}O_9$	
Formula weight	682.86	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.2339(3) Å	α= 90°.
	b = 20.5982(8) Å	β= 90°.
	c = 23.5913(9) Å	γ= 90°.
Volume	3515.2(2) Å3	
Z	4	
Density (calculated)	1.290 Mg/m^3	
Absorption coefficient	0.725 mm^{-1}	
F(000)	1480	
Crystal size	0.340 x 0.110 x 0.070 mm^3	
Theta range for data collection	2.85 to 70.18°.	
Index ranges	-8<=h<=7, -24<=k<=25, -28<=l<=28	
Reflections collected	28938	
Independent reflections	6645 [R(int) = 0.1138]	
Completeness to theta = 70.18°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.95 and 0.80	
Refinement method	Full-matrix least-squares on F2	
Data / restraints / parameters	6645 / 0 / 451	
Goodness-of-fit on F2	1.071	
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1377	
R indices (all data)	R1 = 0.0761, wR2 = 0.1452	
Absolute structure parameter	0.00(10)	
Largest diff. peak and hole	0.345 and -0.577 $e.Å^{-3}$	

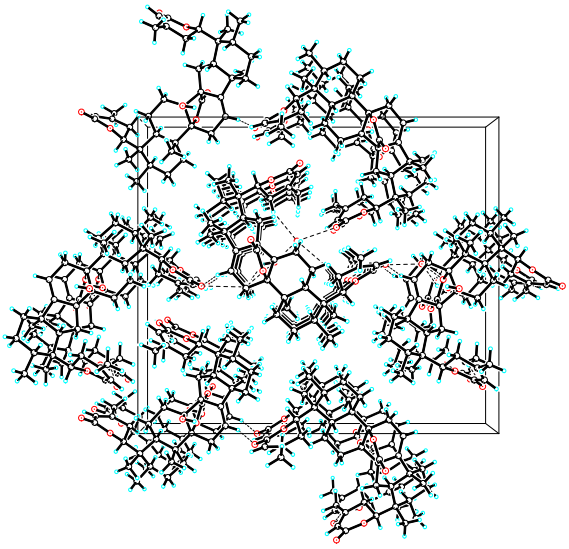


Figure S8. View of the pack drawing of **10**
Hydrogen-bonds are shown as dashed lines.

3. The results of activity assay

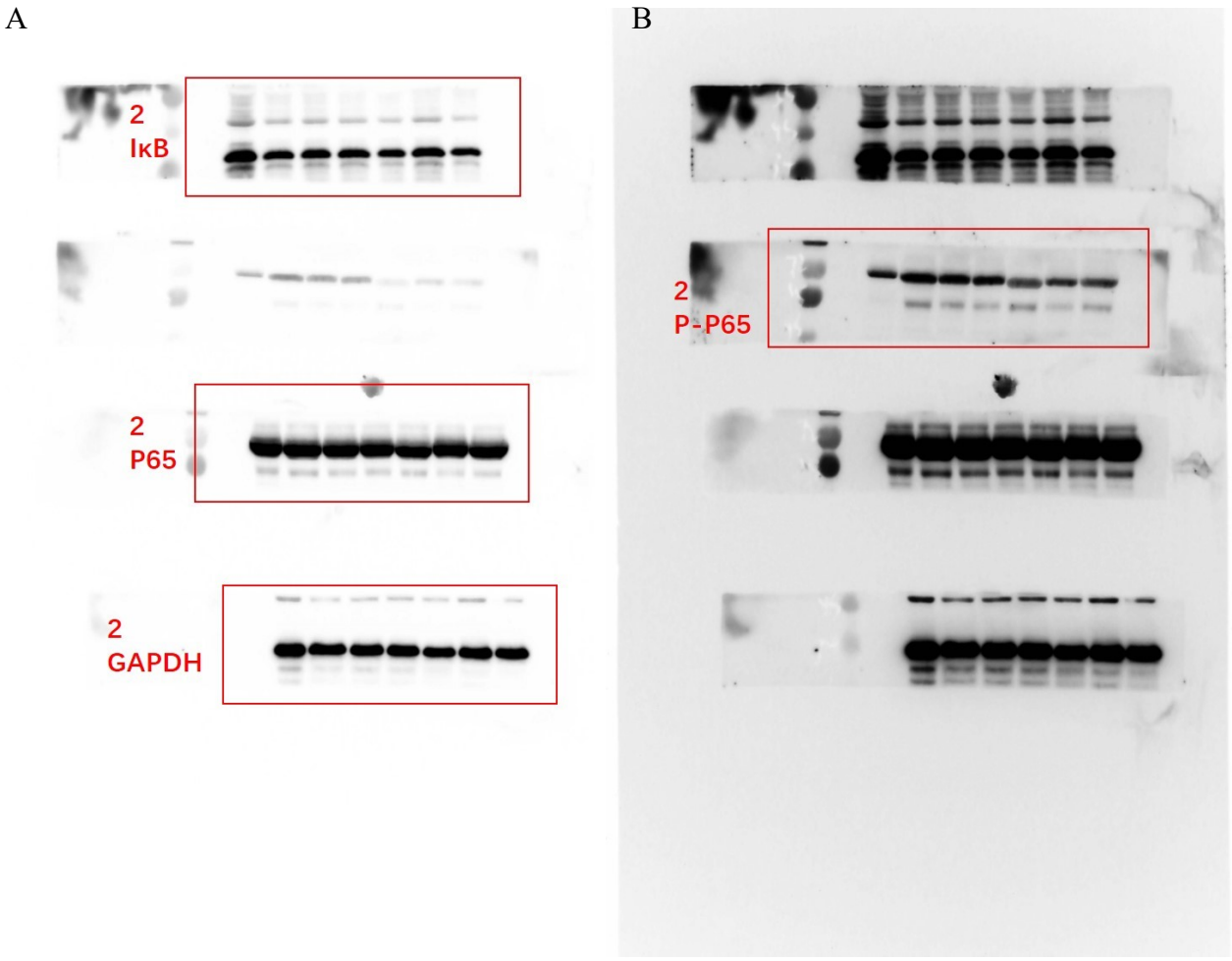


Figure 9. The full raw data of western blots (A and B) of compound **2**.

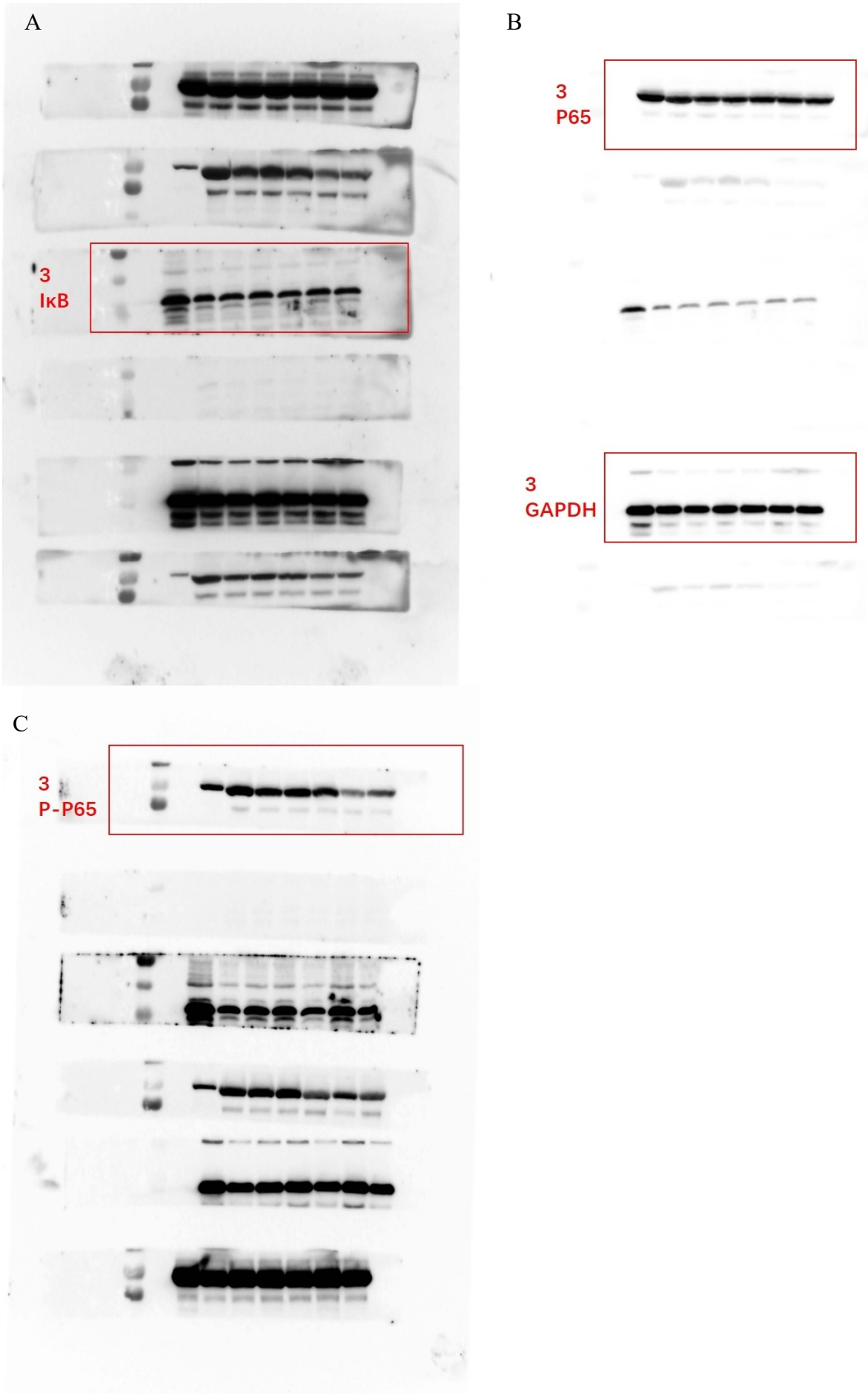


Figure 10. The full raw data of western blots (A–C) of compound **3**.

Table S4. IC_{50} values[a] of cytotoxic activity for compounds **1**–**3** against five human tumour cell lines.

Compd.	HL-60	A549	SMMC-7721	MCF-7	SW480
1	>40	>40	>40	>40	>40
2	14.25±0.28	17.41±0.47	21.06±0.33	>40	>40
3	>40	>40	>40	>40	>40
DDP	4.17±0.19	13.28±0.90	11.33±1.04	-	-
Taxol	<0.008	<0.008	0.219±0.021	-	-

[a] IC_{50} values (μM)

4. NMR, HRESIMS, ECD, UV, OR, and IR spectra of 1–3 and 10

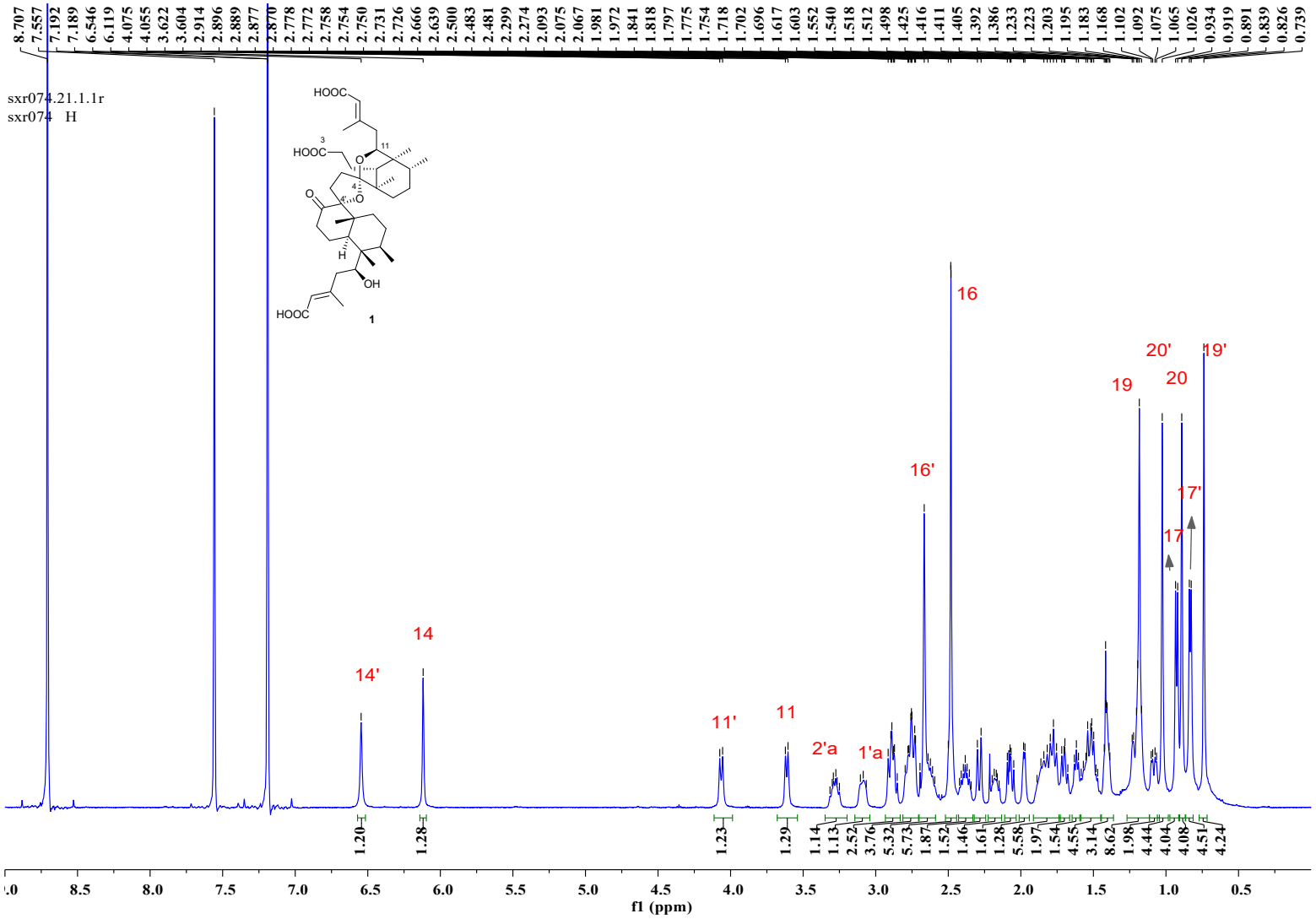


Figure S11. ^{1}H NMR spectrum of scoparicacid A (**1**)

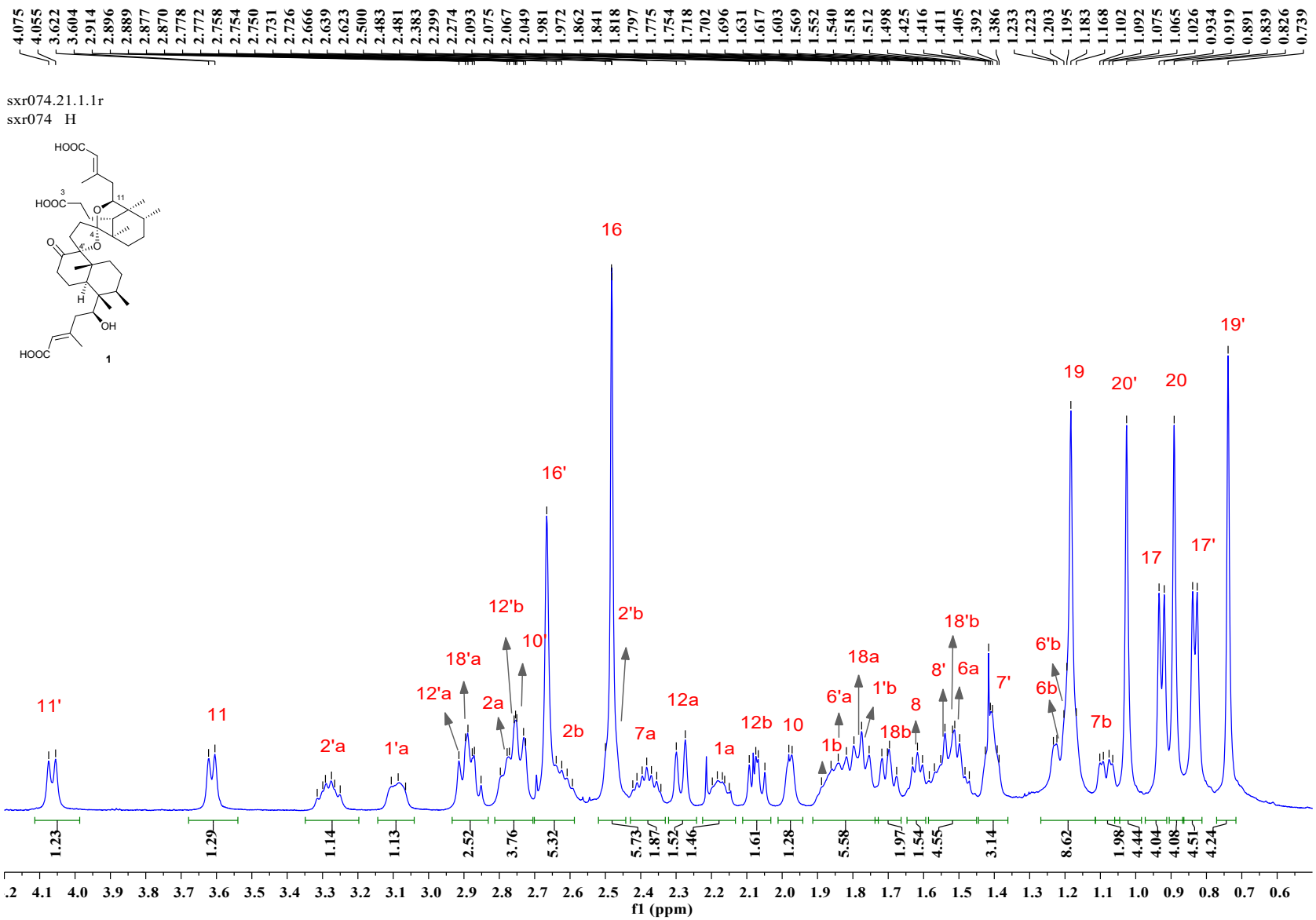


Figure S12. ^{1}H NMR spectrum of scoparicacid A (**1**)

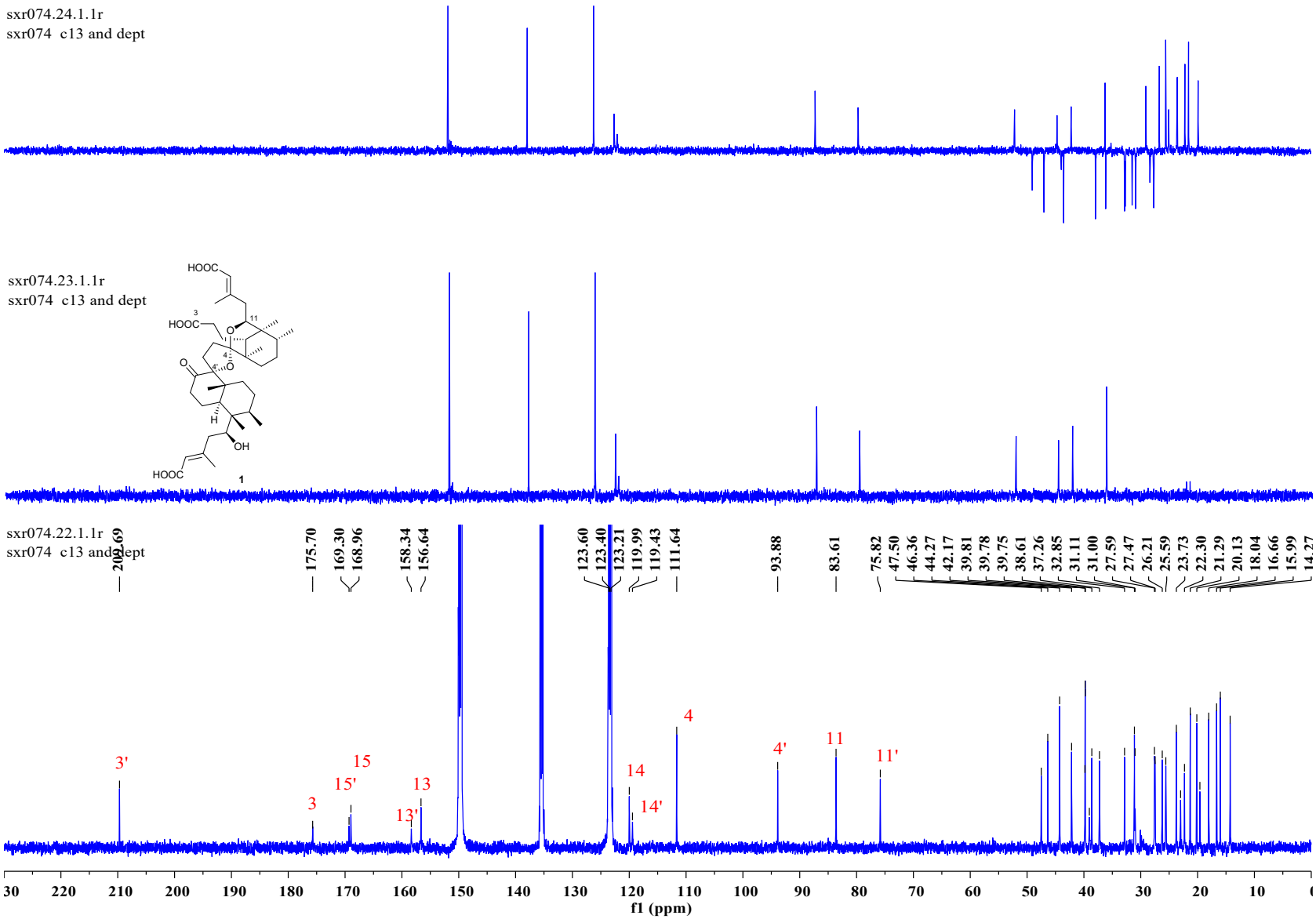


Figure S13. ^{13}C NMR spectrum of scoparicacid A (**1**)

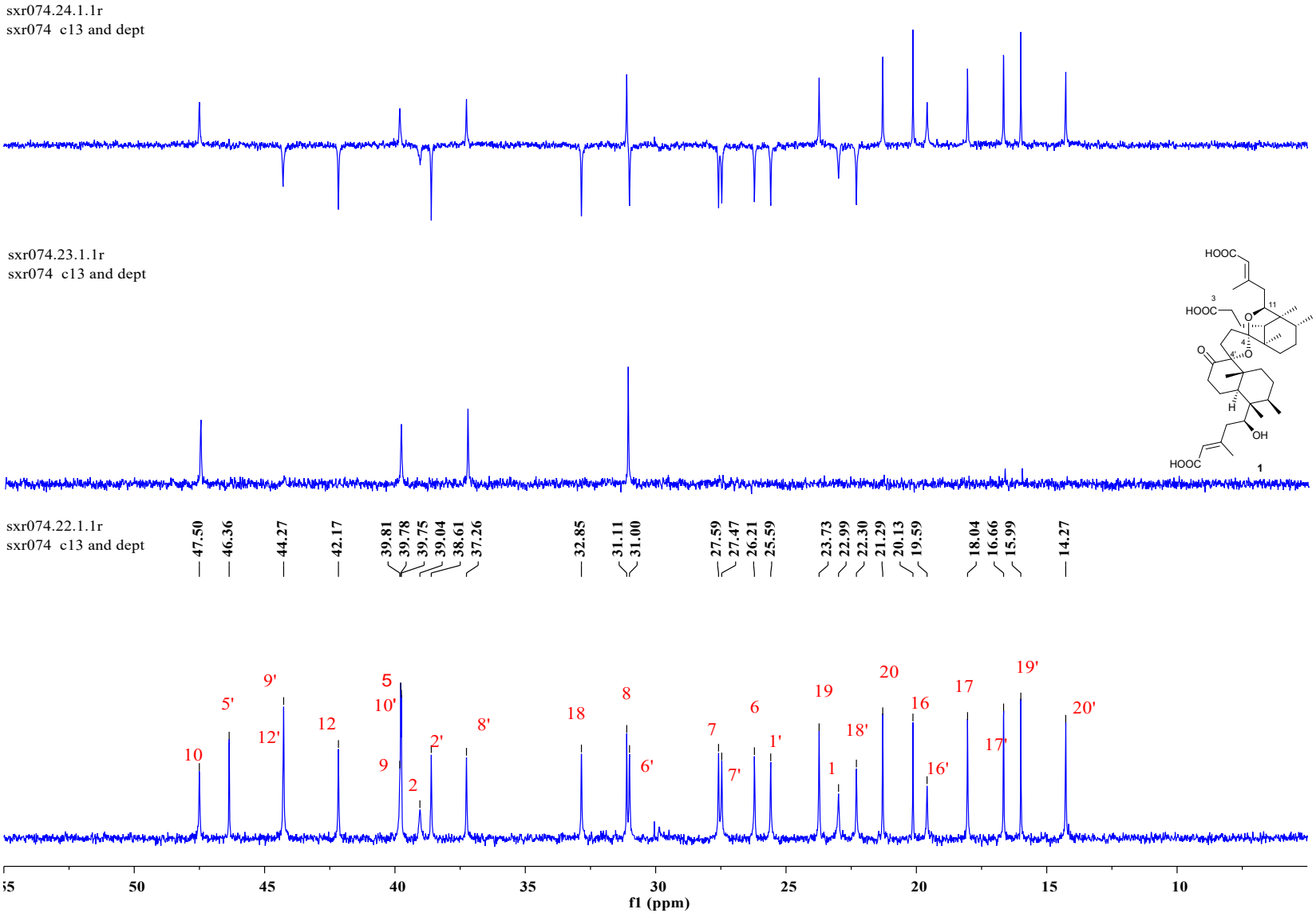


Figure S14. ^{13}C NMR spectrum of scoparicacid A (**1**)

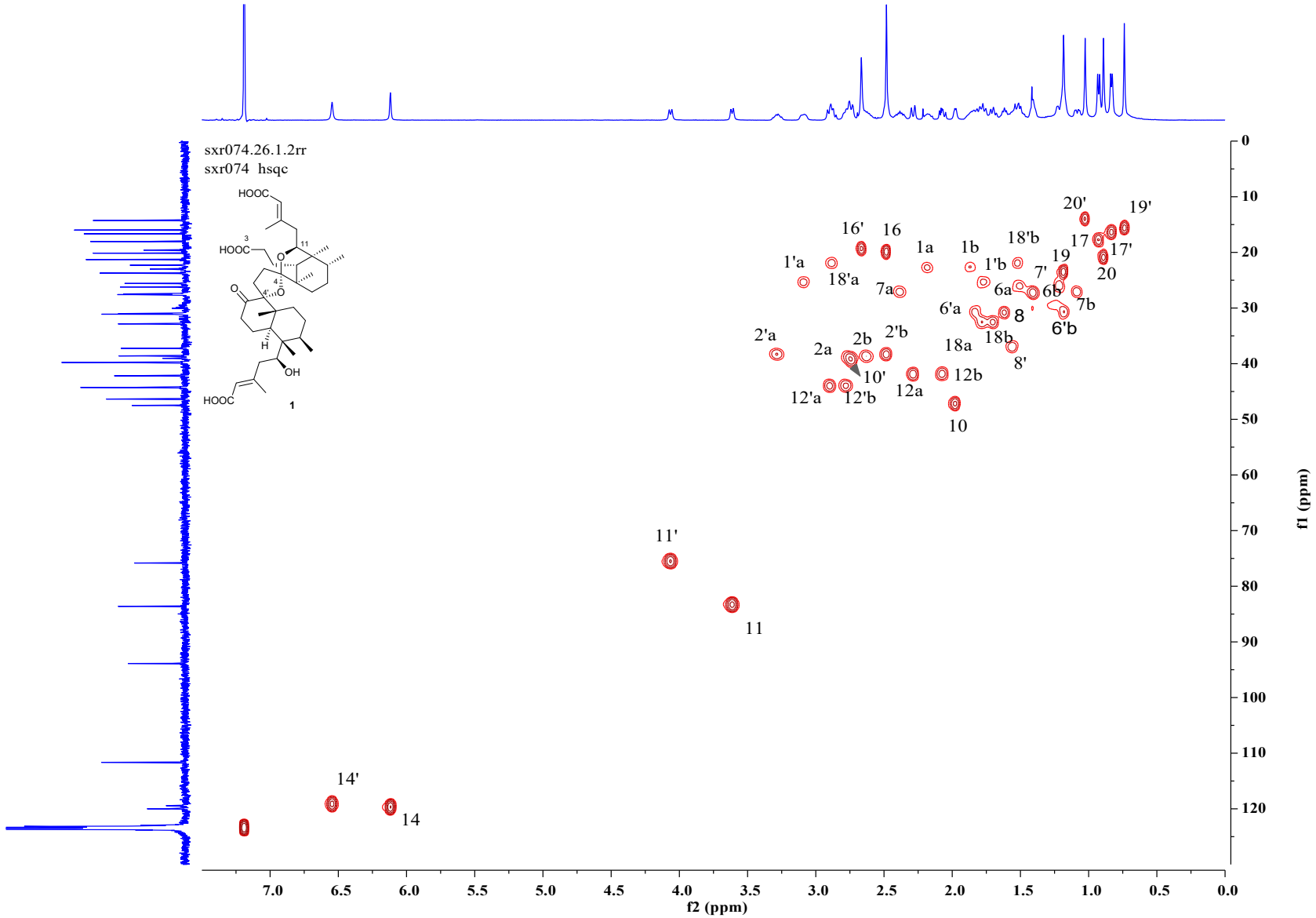


Figure S15. HSQC spectrum of scoparicacid A (**1**)

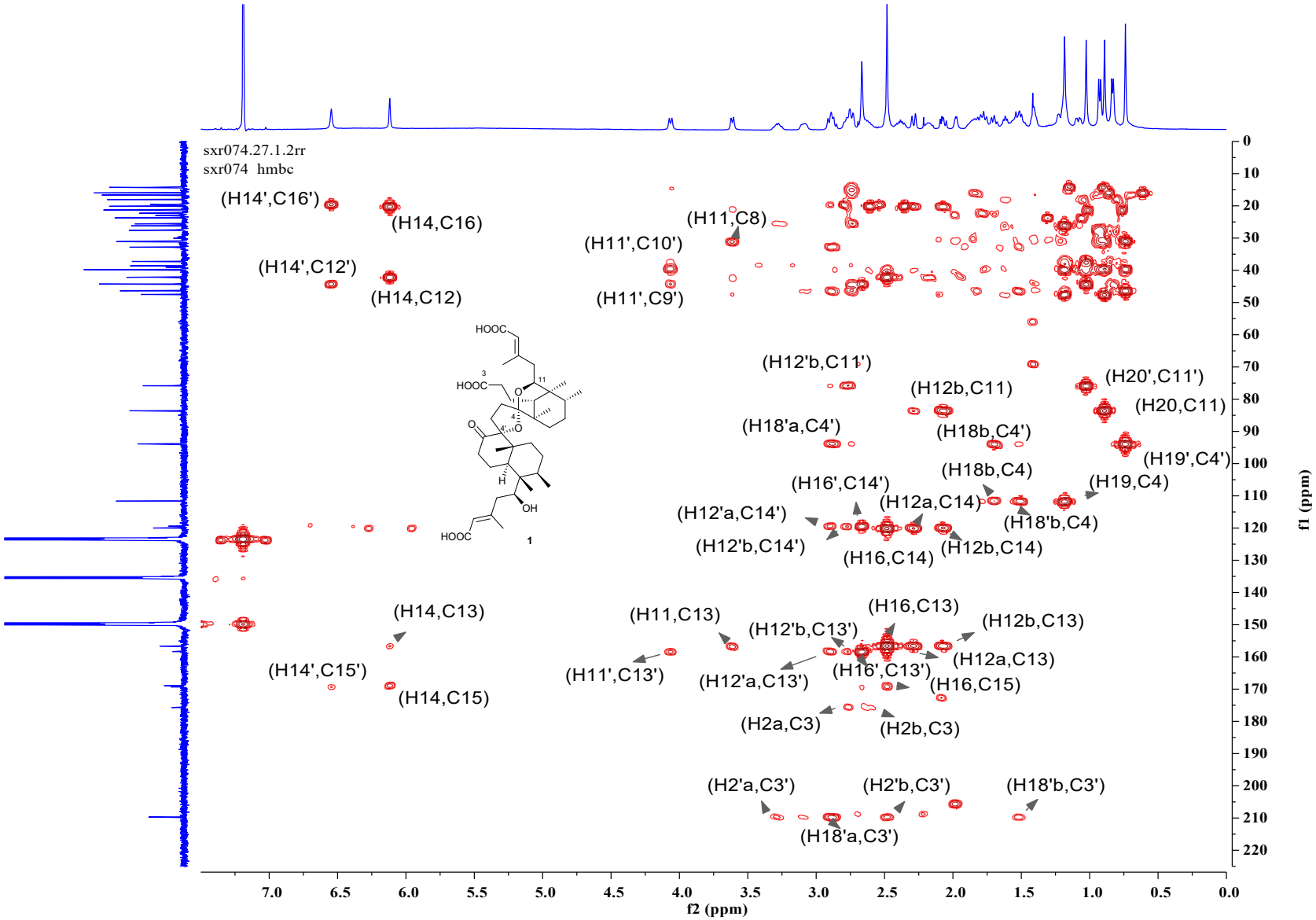


Figure S16. HMBC spectrum of scoparicacid A (**1**)

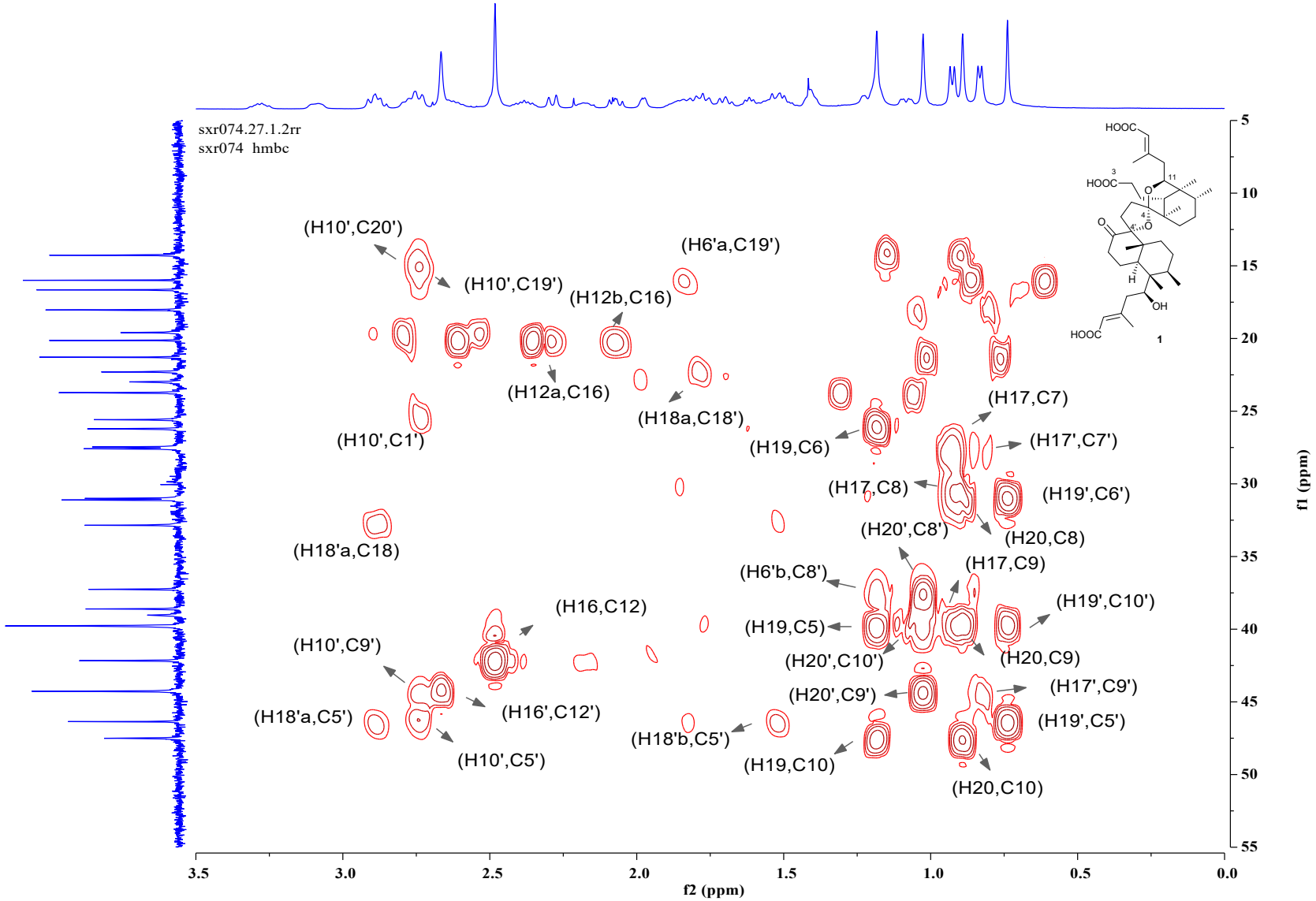


Figure S17. HMBC spectrum of scoparicacid A (**1**)

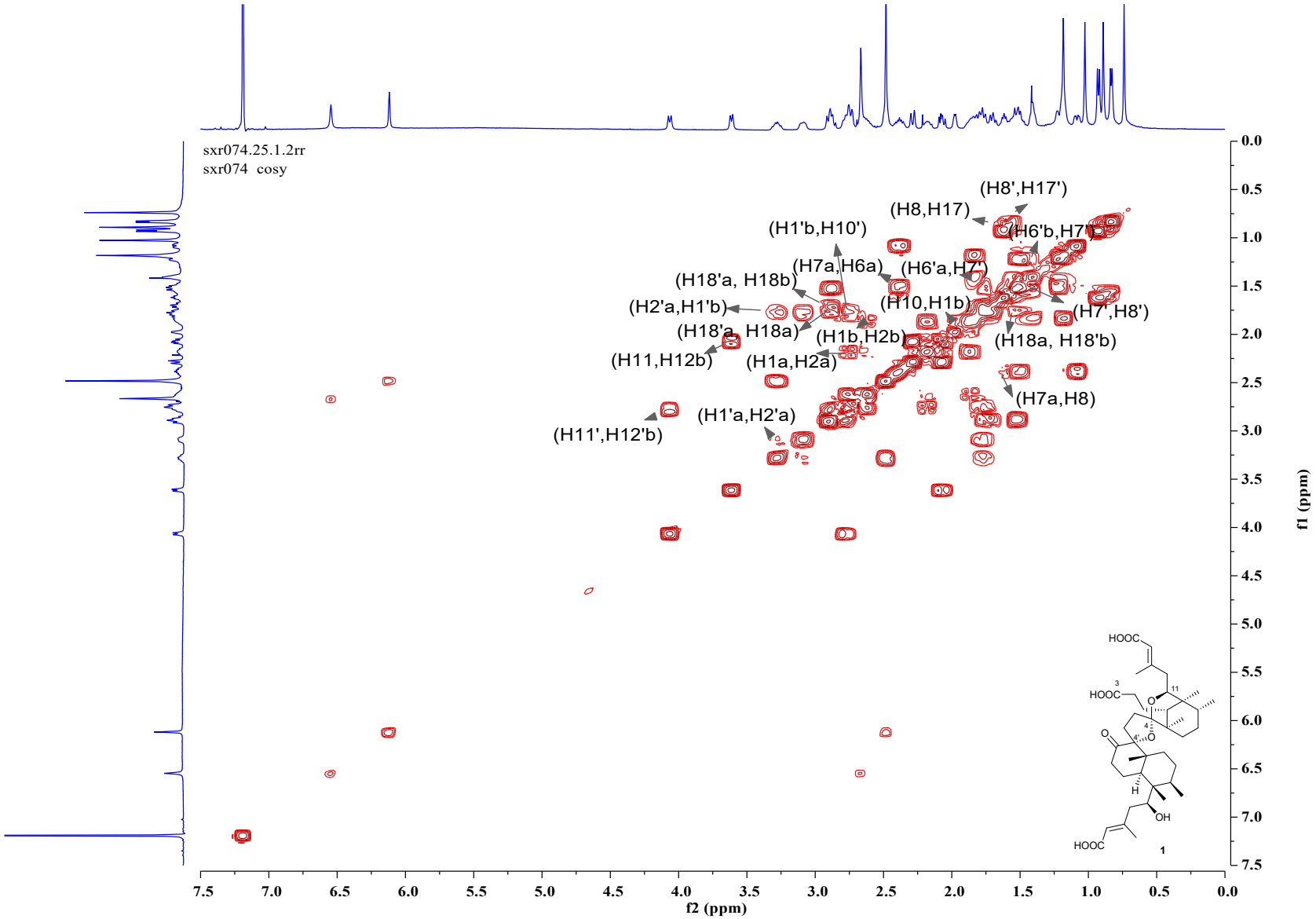


Figure S18. ^{1}H-^{1}H COSY spectrum of scoparicacid A (**1**)

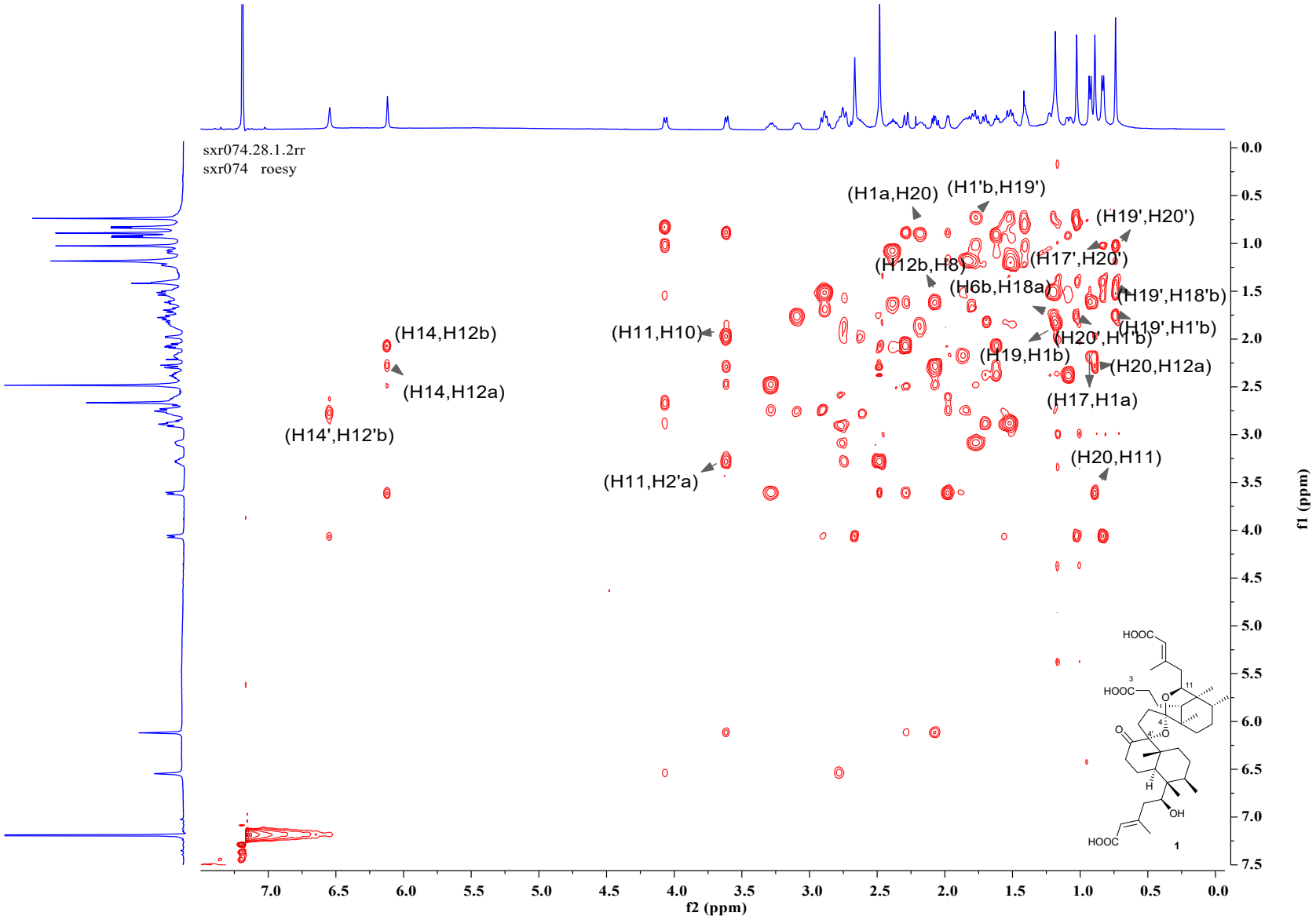


Figure S19. ROESY spectrum of scoparicacid A (**1**)

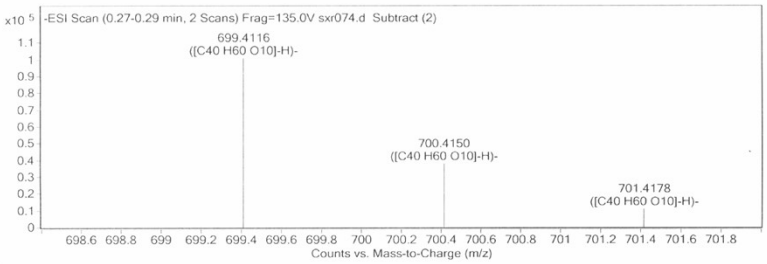
Qualitative Analysis Report

Data Filename	sxr074.d	**Sample Name**	sxr074
Sample Type	Sample	**Position**	P1-A1
Instrument Name	Instrument 1	**User Name**	
Acq Method	s-.m	**Acquired Time**	11/29/2021 2:02:04 PM
IRM Calibration Status	Success	**DA Method**	PCDL.m
Comment			

Sample Group	**Info.**
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
339.233	1	12452.72		
403.3068	1	4092.35		
615.3901	1	4039.76		
699.4116	1	100955.74	C40 H60 O10	(M-H)-
700.415	1	38022.48	C40 H60 O10	(M-H)-
701.4178	1	10911.37	C40 H60 O10	(M-H)-
721.3925	1	8691.88		
722.397	1	3868.84		
735.3877	1	4847.71		
783.3631	1	4352.47		

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C40 H60 O10	700.4187	699.4114	699.4116	-0.20	-0.29	11.0000

--- End Of Report ---

Figure S20. HRESIMS spectrum of scoparicacid A (**1**)

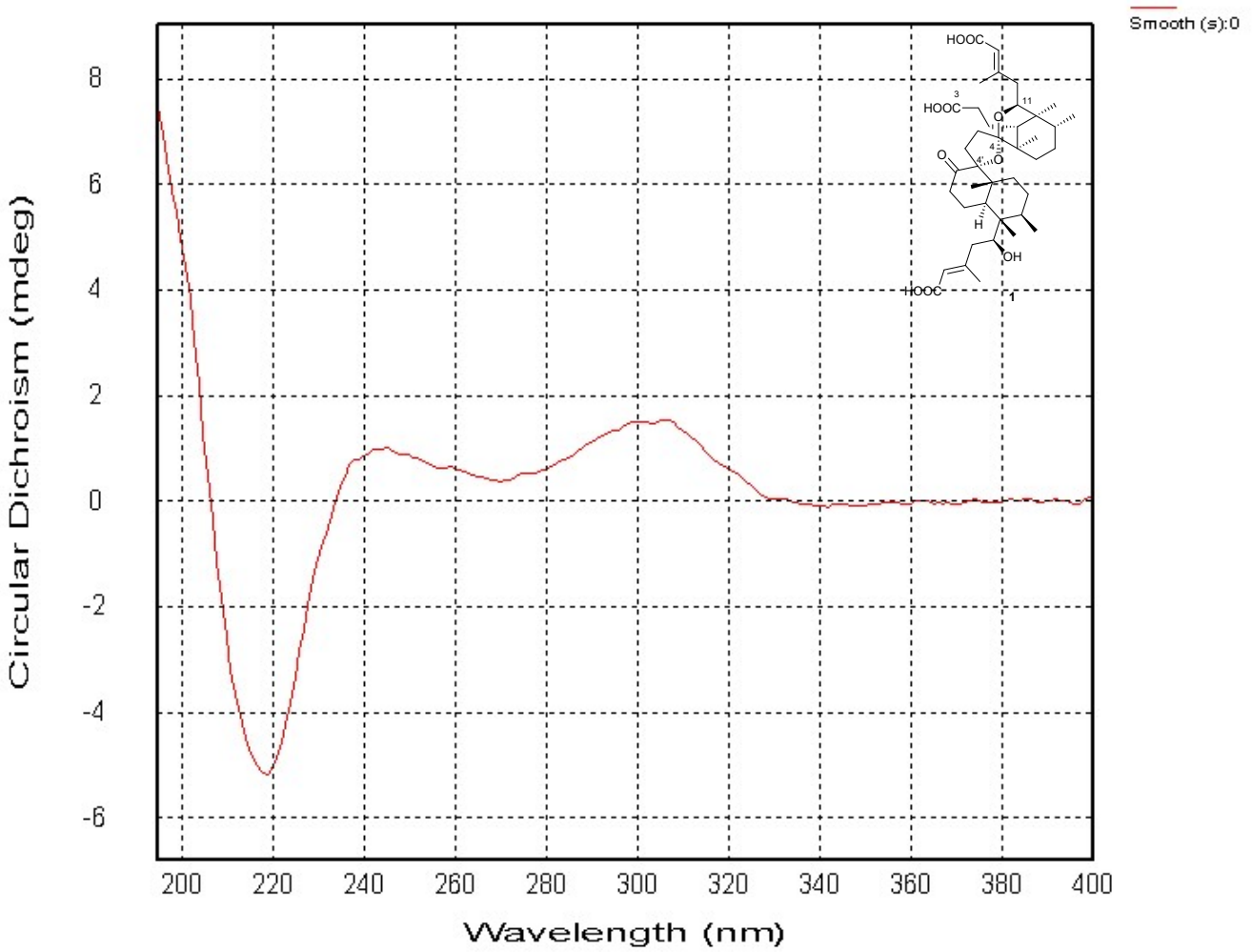


Figure S21. ECD spectrum of scoparicacid A (**1**)

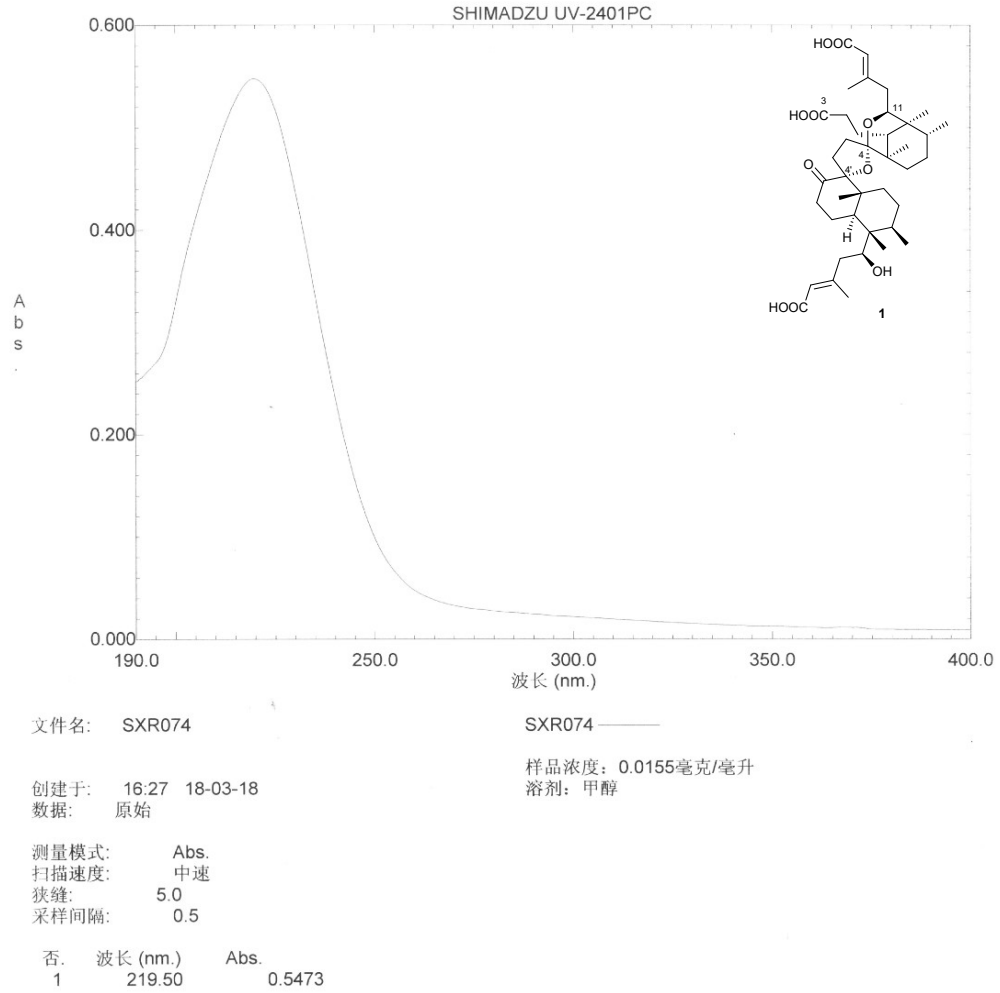


Figure S22. UV spectrum of scoparicacid A (**1**)

Optical rotation measurement

Model : P-1020 (A060460638)

No.	Sample	Mode	Data	Monitor Blank	Temp. Cell Temp Point	Date Comment Sample Name	Light Filter Operator	Cycle Time Integ Time
No.1	4 (1/3)	Sp.Rot	-27.5510	-0.0027 0.0000	17.3 10.00 Cell	Thu Dec 21 01:05:02 2017 0.00098g/mL MeOH SXR074	Na 589nm	2 sec 2 sec
No.2	4 (2/3)	Sp.Rot	-28.5710	-0.0028 0.0000	17.3 10.00 Cell	Thu Dec 21 01:05:08 2017 0.00098g/mL MeOH SXR074	Na 589nm	2 sec 2 sec
No.3	4 (3/3)	Sp.Rot	-24.4900	-0.0024 0.0000	17.3 10.00 Cell	Thu Dec 21 01:05:13 2017 0.00098g/mL MeOH SXR074	Na 589nm	2 sec 2 sec

−26.8707°

Figure S23. OR spectrum of scoparicacid A (**1**)

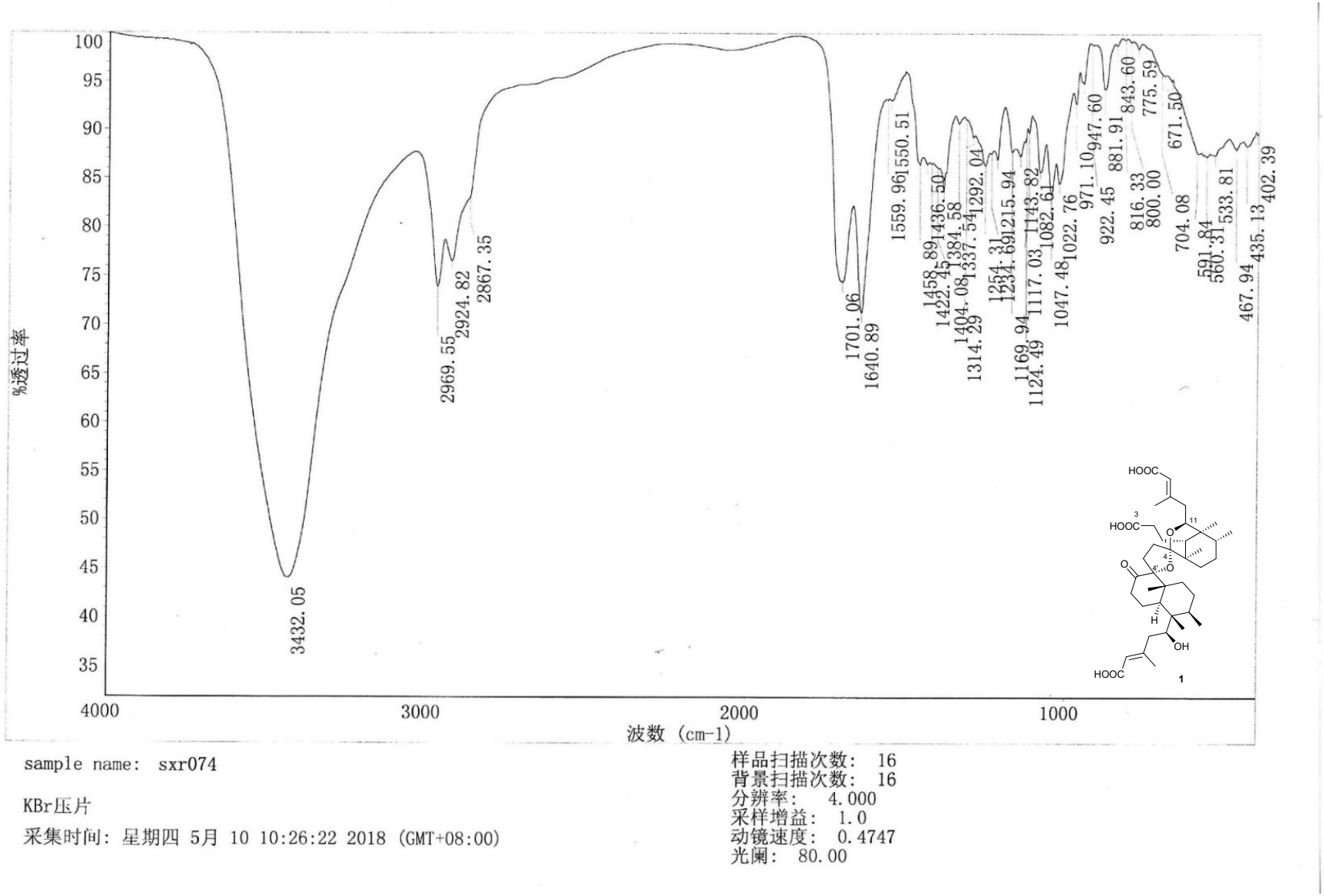


Figure S24. IR spectrum of scoparicacid A (**1**)

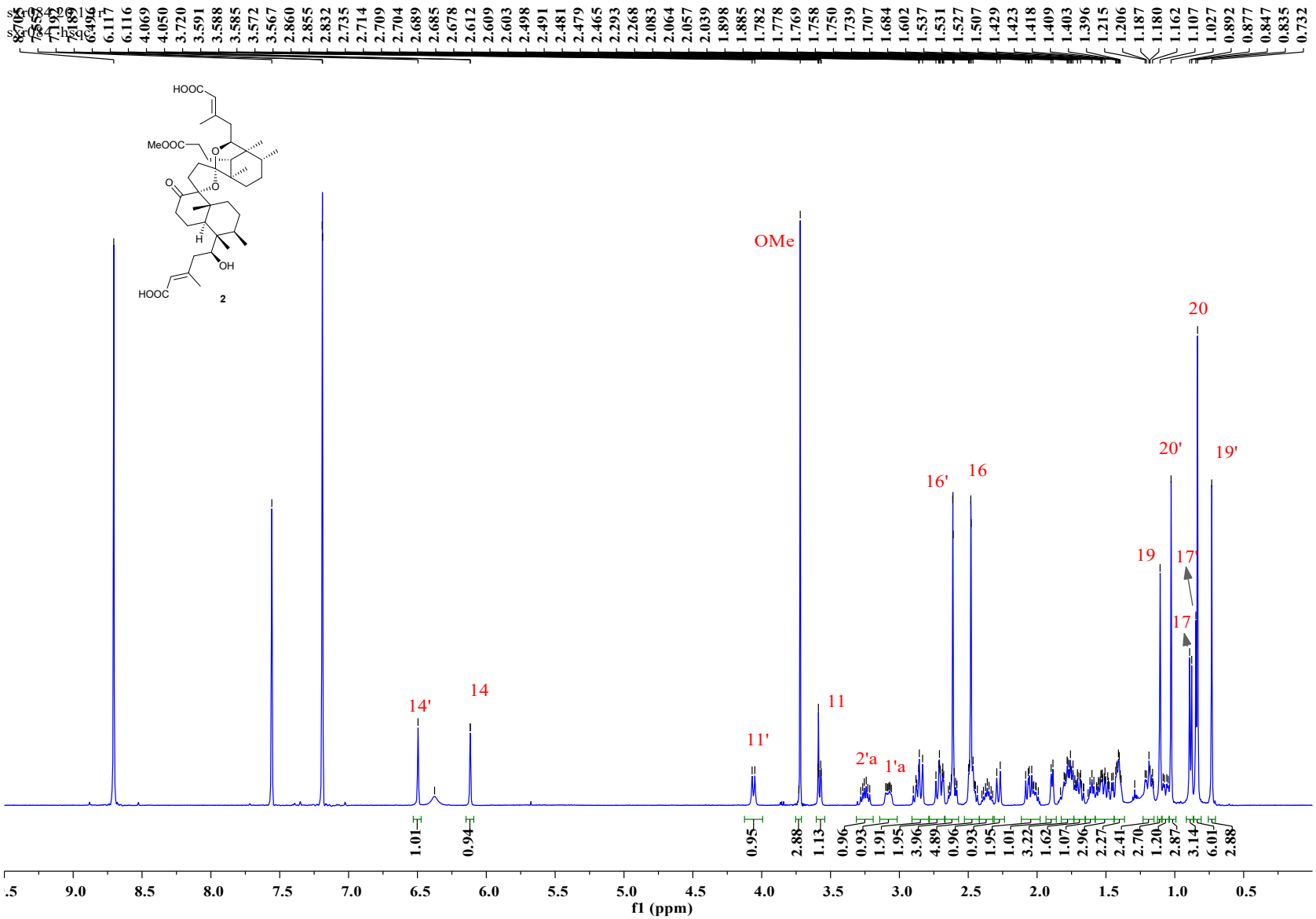


Figure S25. ^{1}H NMR spectrum of scoparicacid B (**2**)

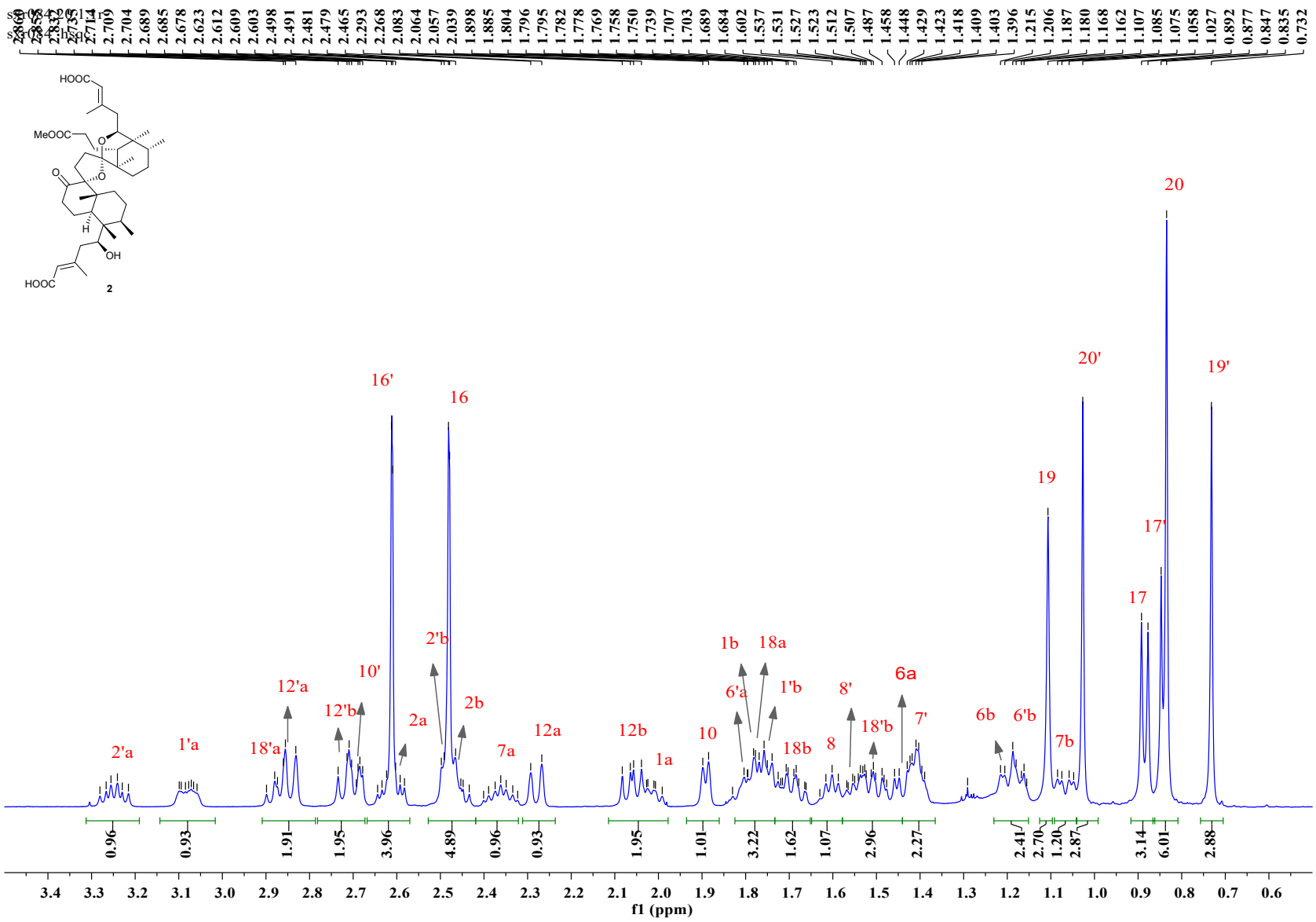


Figure S26. ^{1}H NMR spectrum of scoparicacid B (**2**)

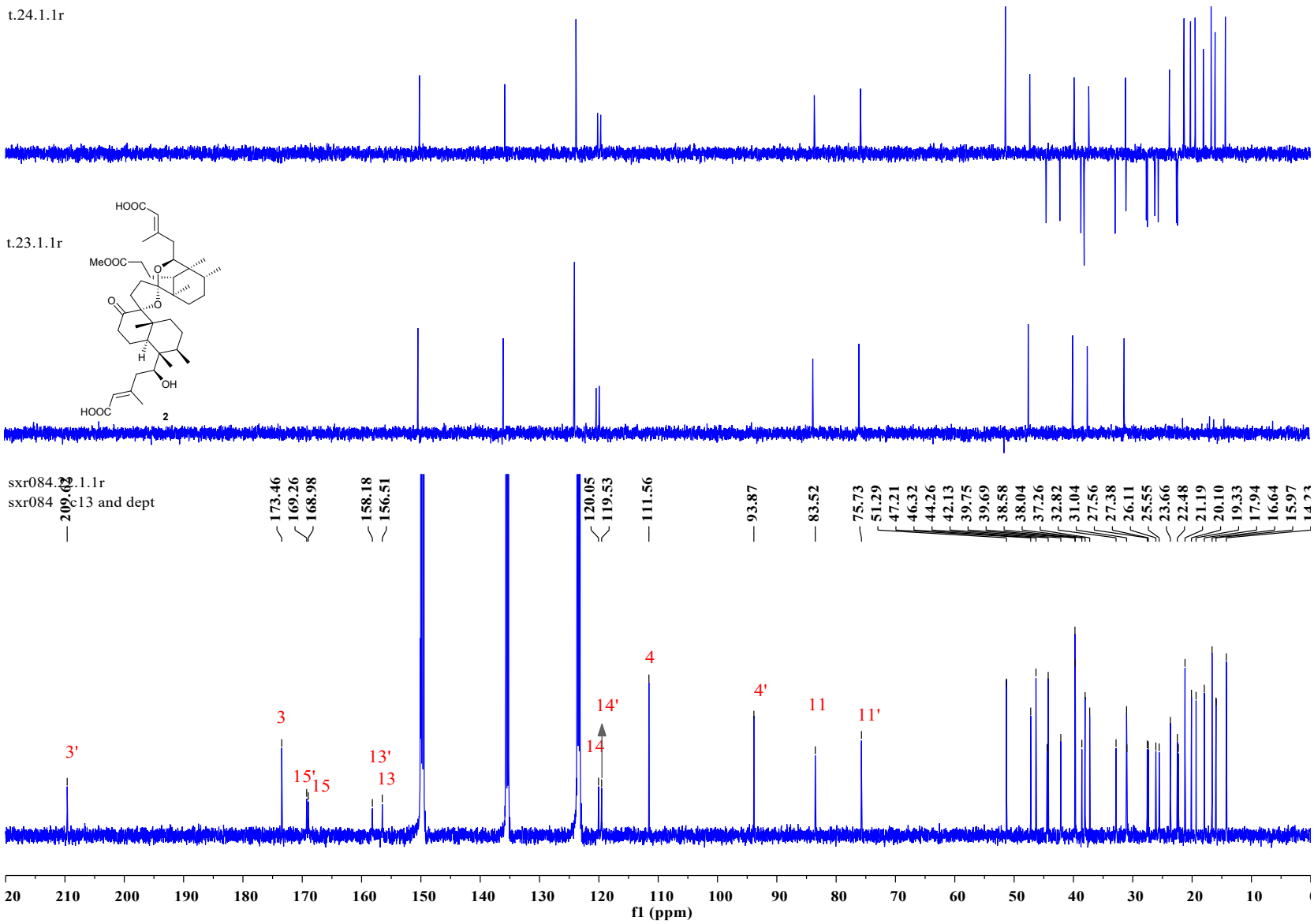


Figure S27. ^{13}C NMR spectrum of scoparicacid B (**2**)

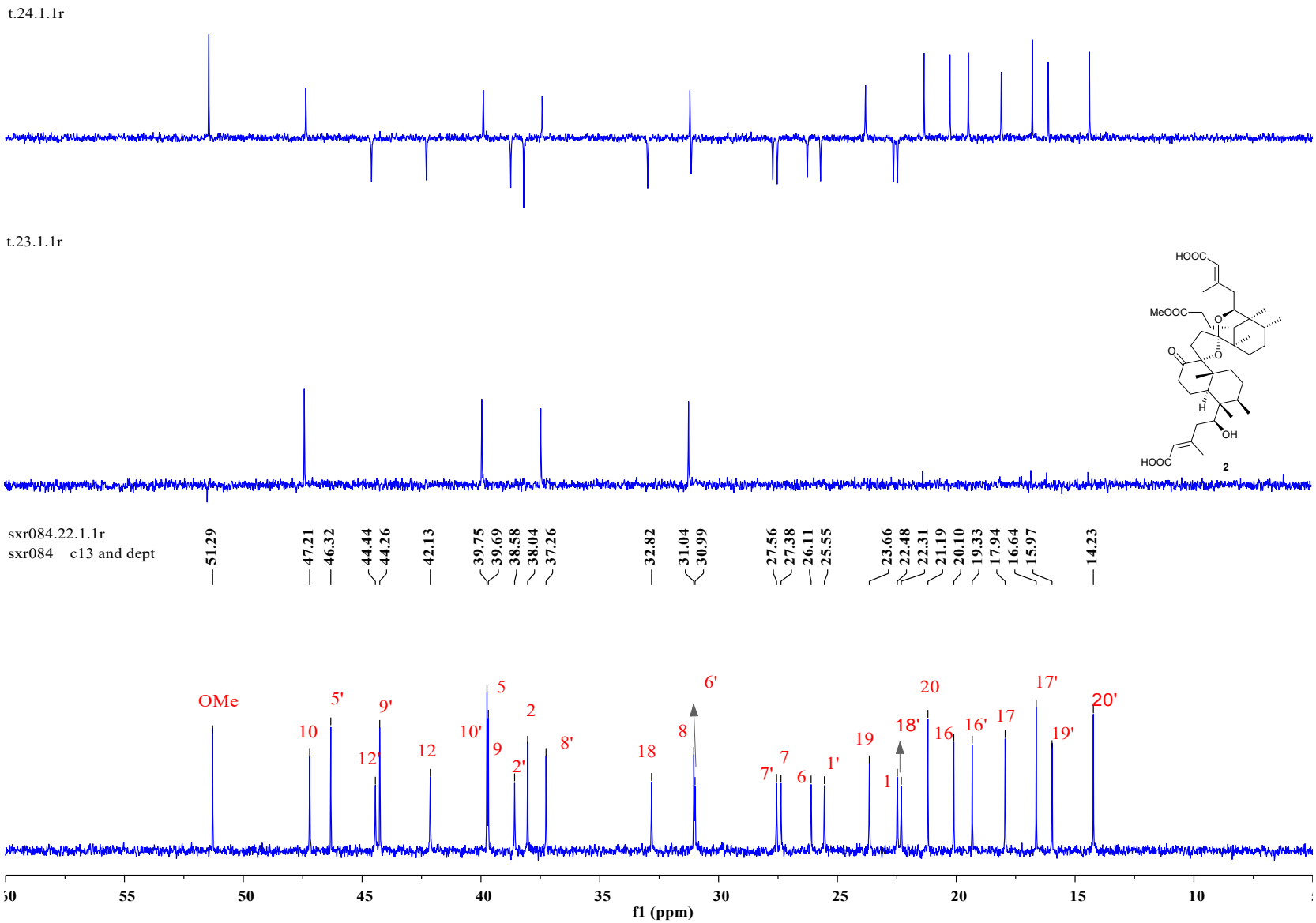


Figure S28. ^{13}C NMR spectrum of scoparicacid B (**2**)

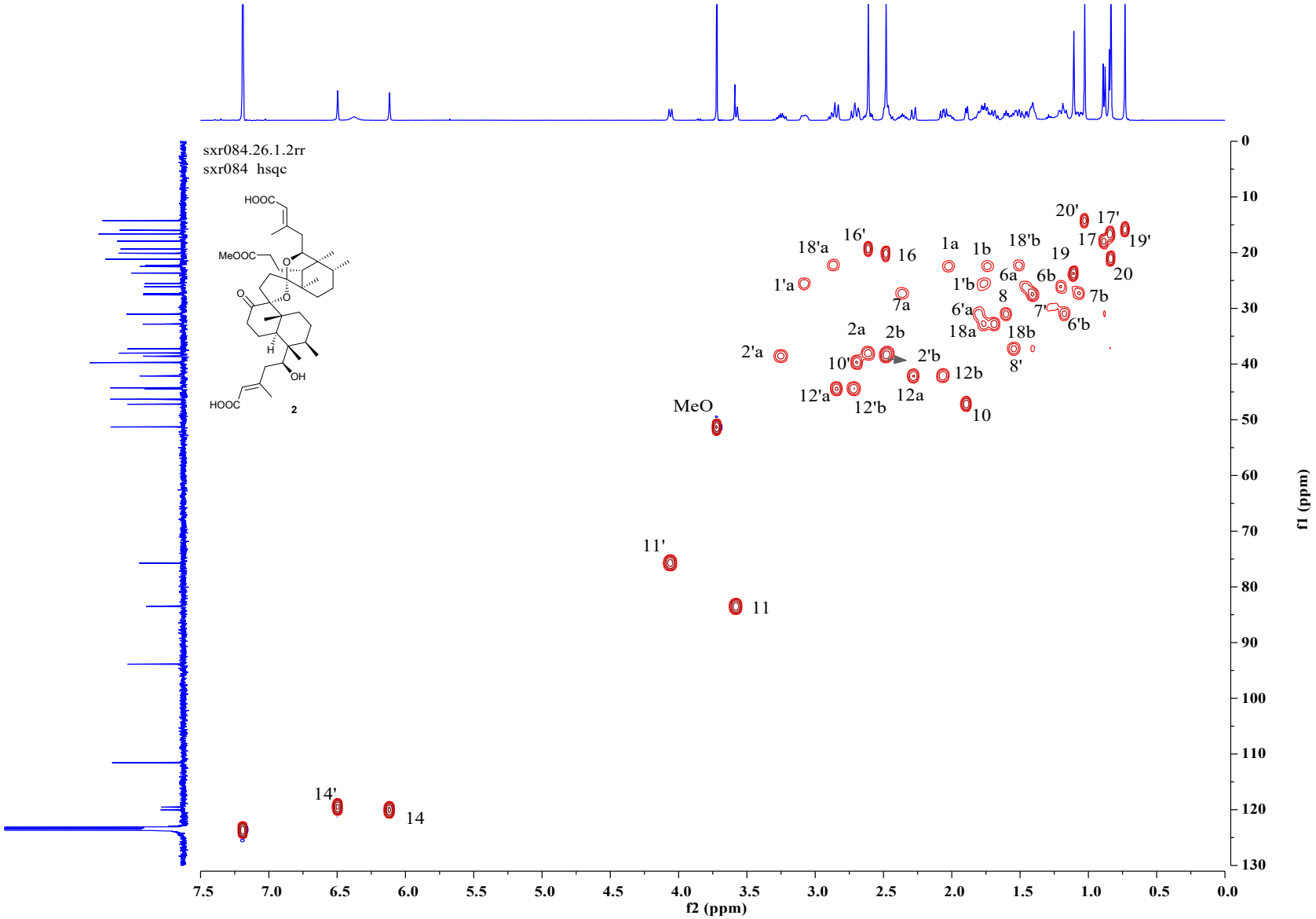


Figure S29. HSQC spectrum of scoparicacid B (**2**)

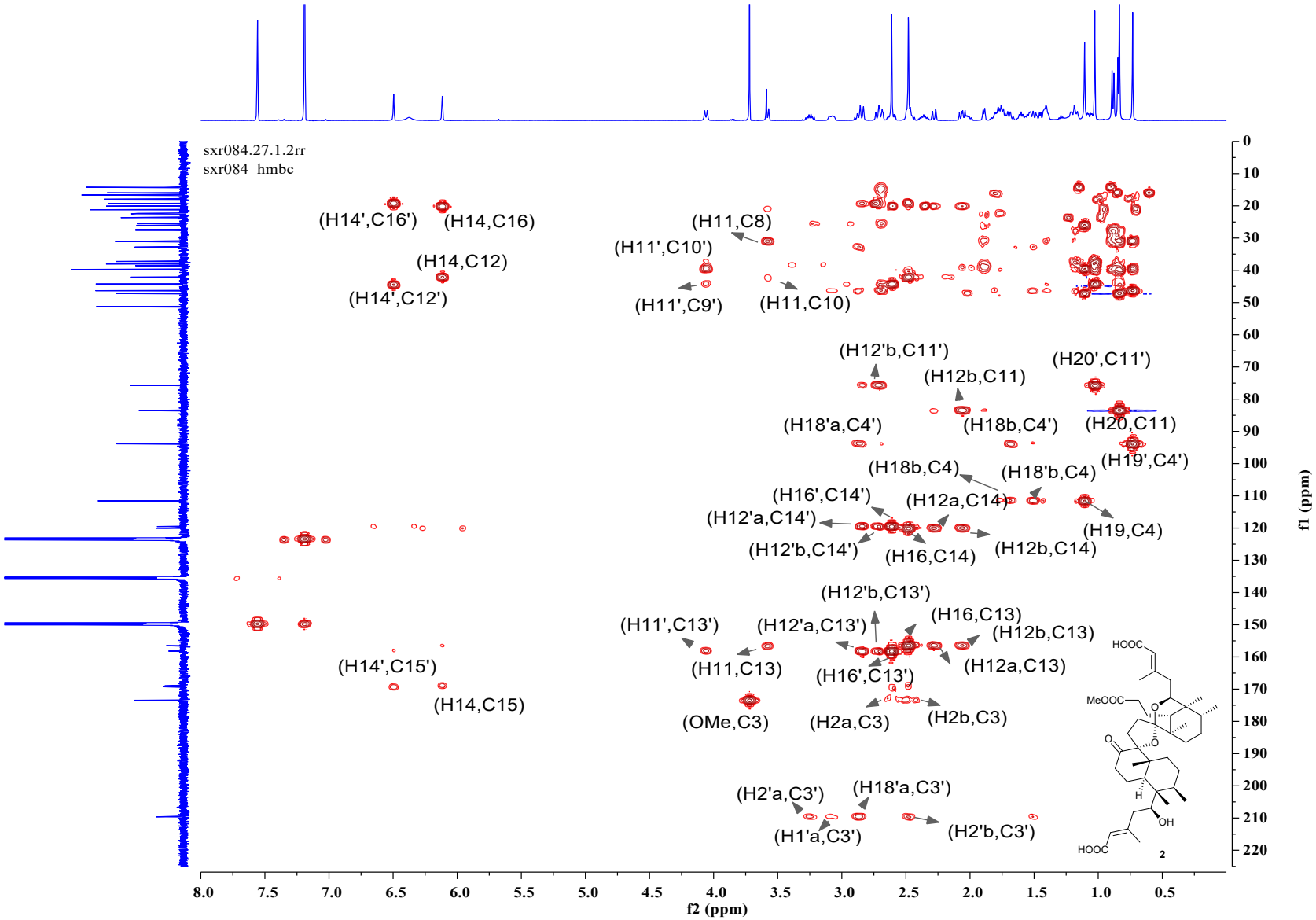


Figure S30. HMBC spectrum of scoparicacid B (**2**)

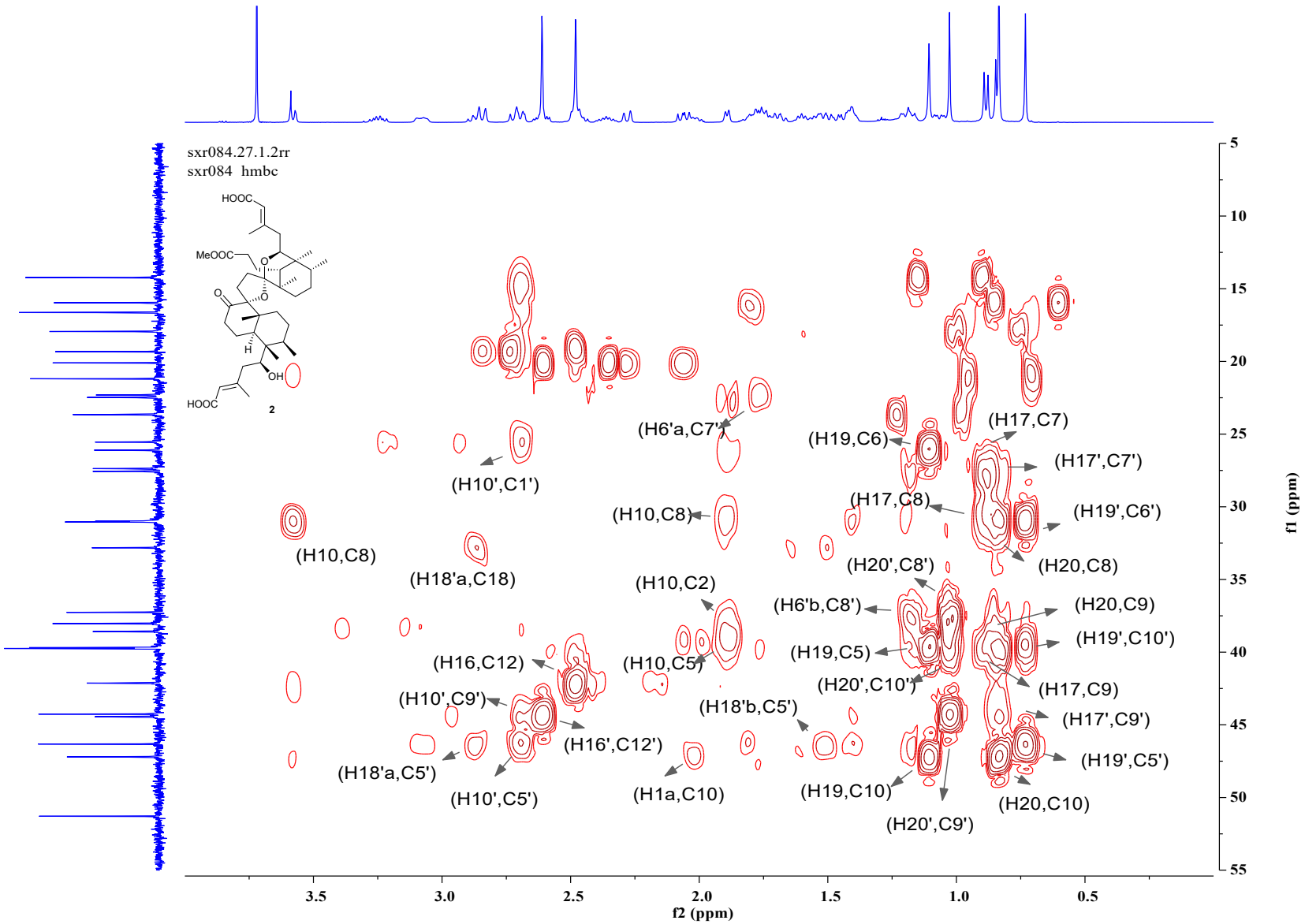


Figure S31. HMBC spectrum of scoparicacid B (**2**)

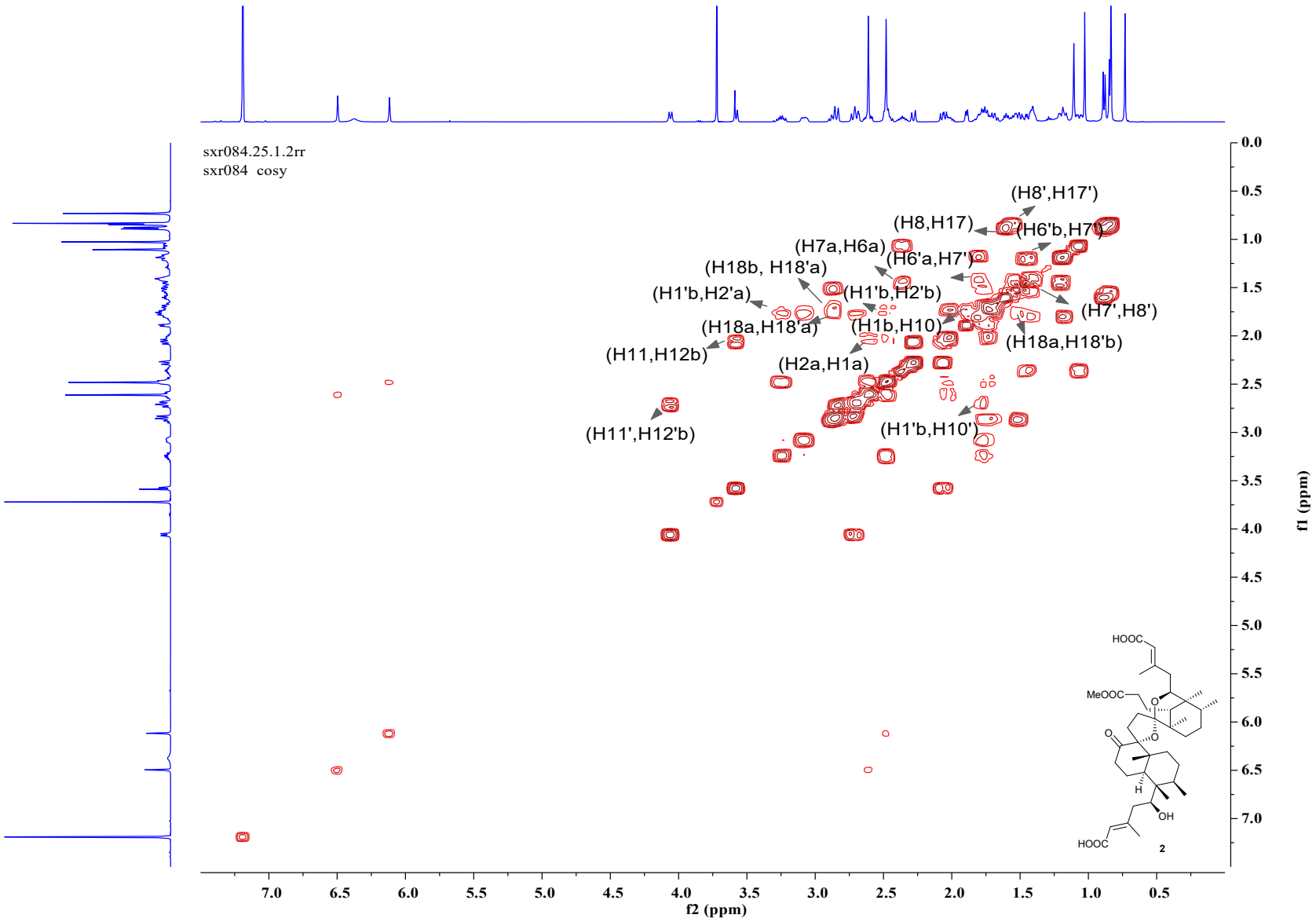


Figure S32. ^{1}H-^{1}H COSY spectrum of scoparicacid B (**2**)

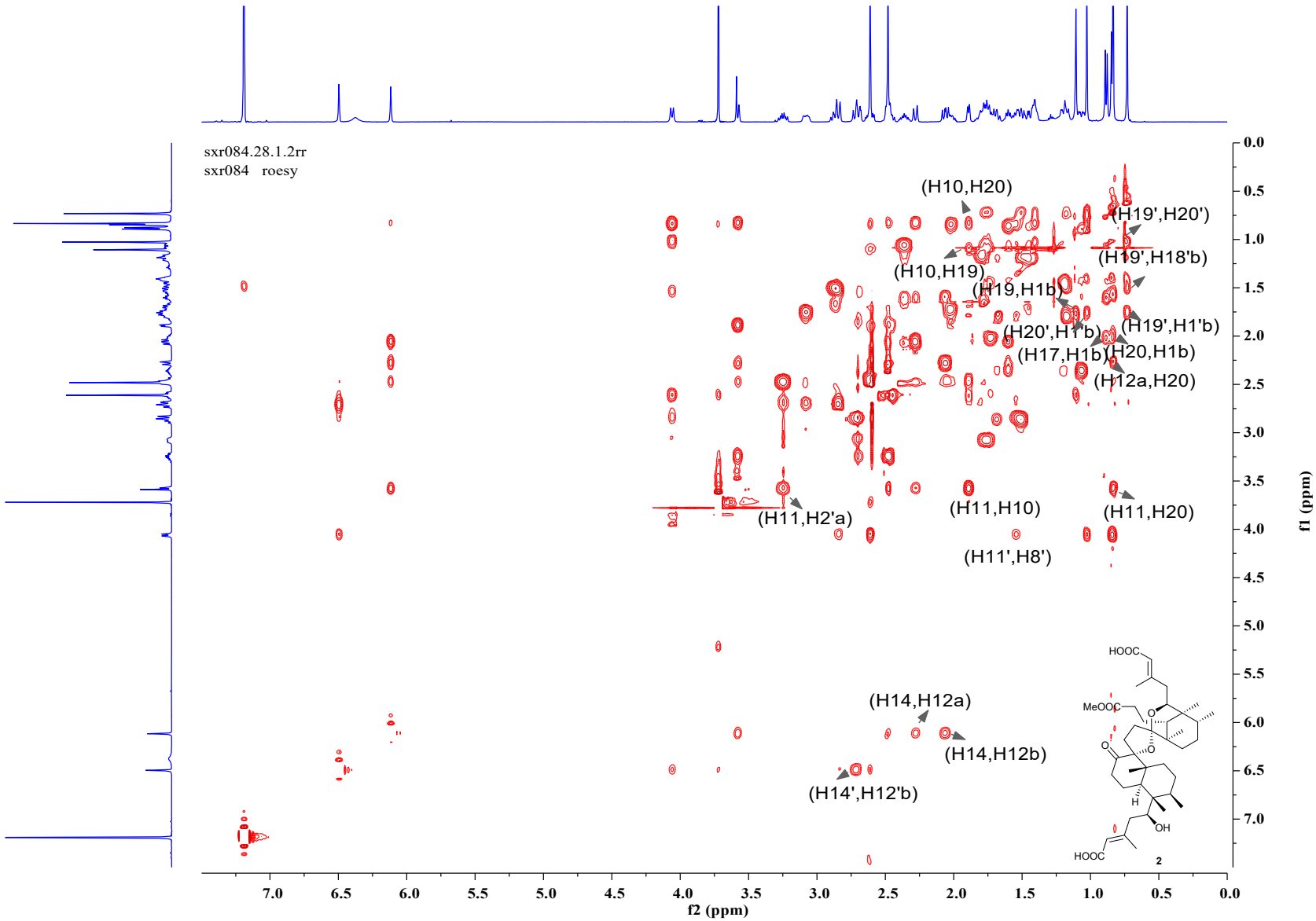


Figure S33. ROESY spectrum of scoparicacid B (**2**)

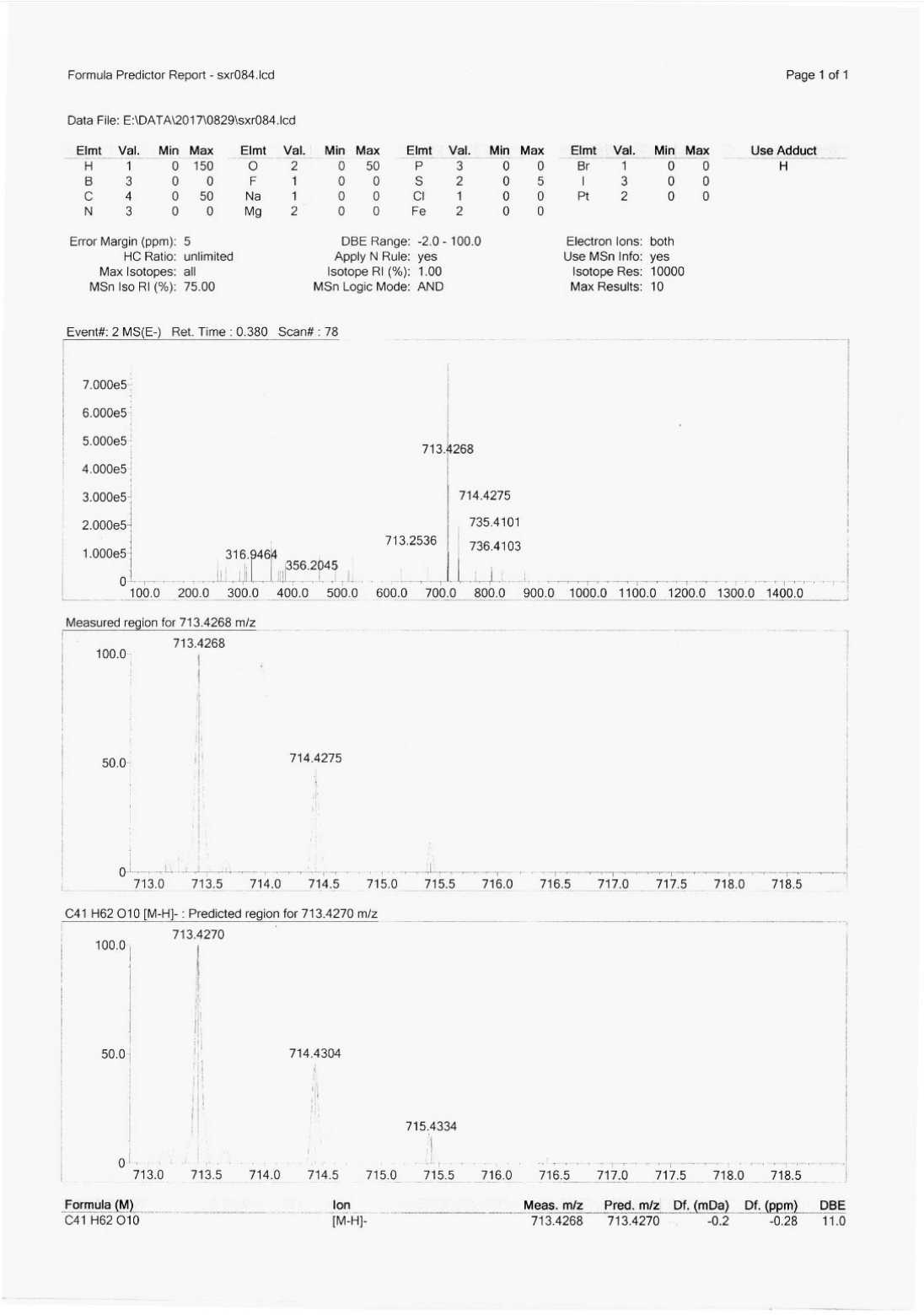


Figure S34. HRESIMS spectrum of scoparicacid B (**2**)

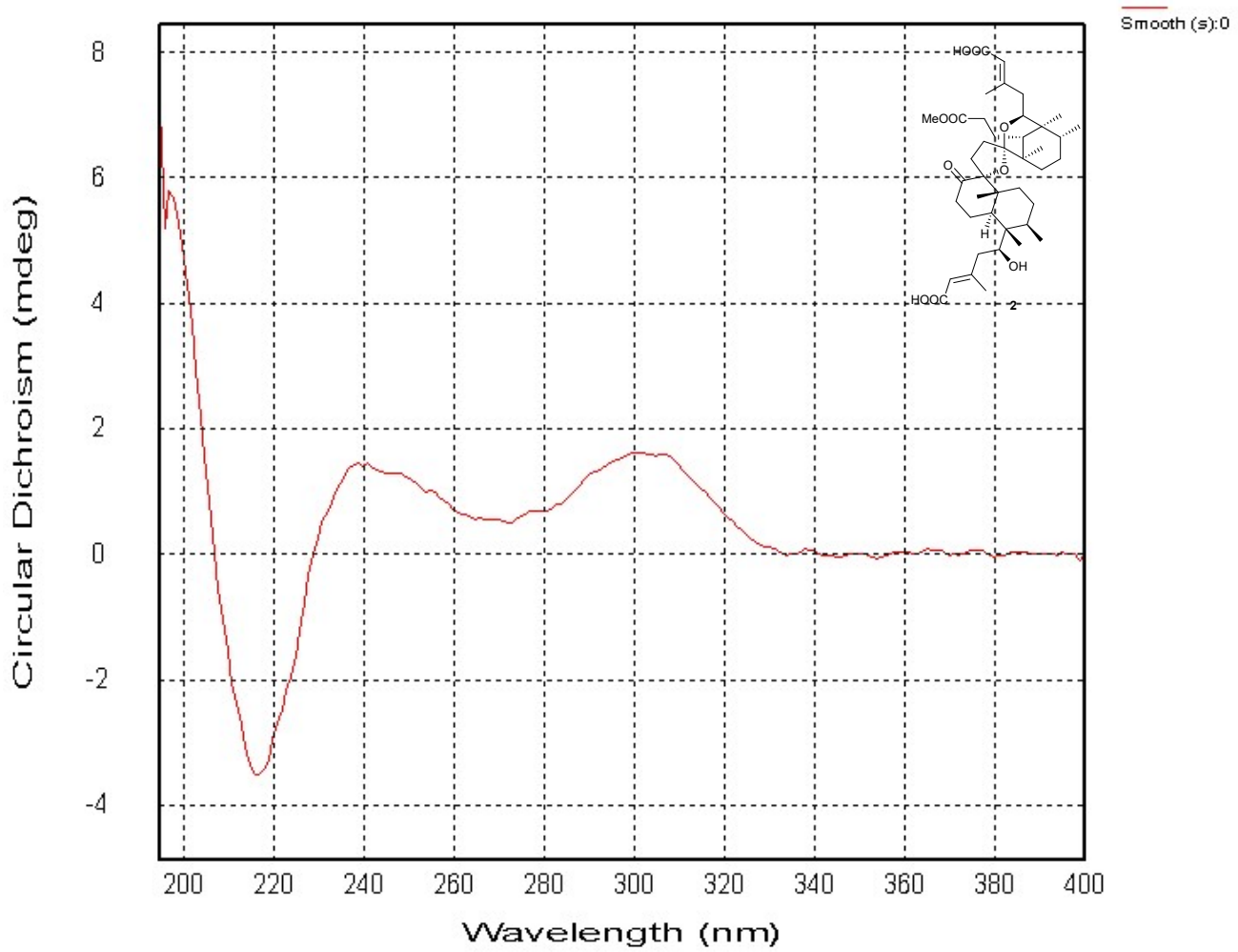


Figure S35. ECD spectrum of scoparicacid B (**2**)

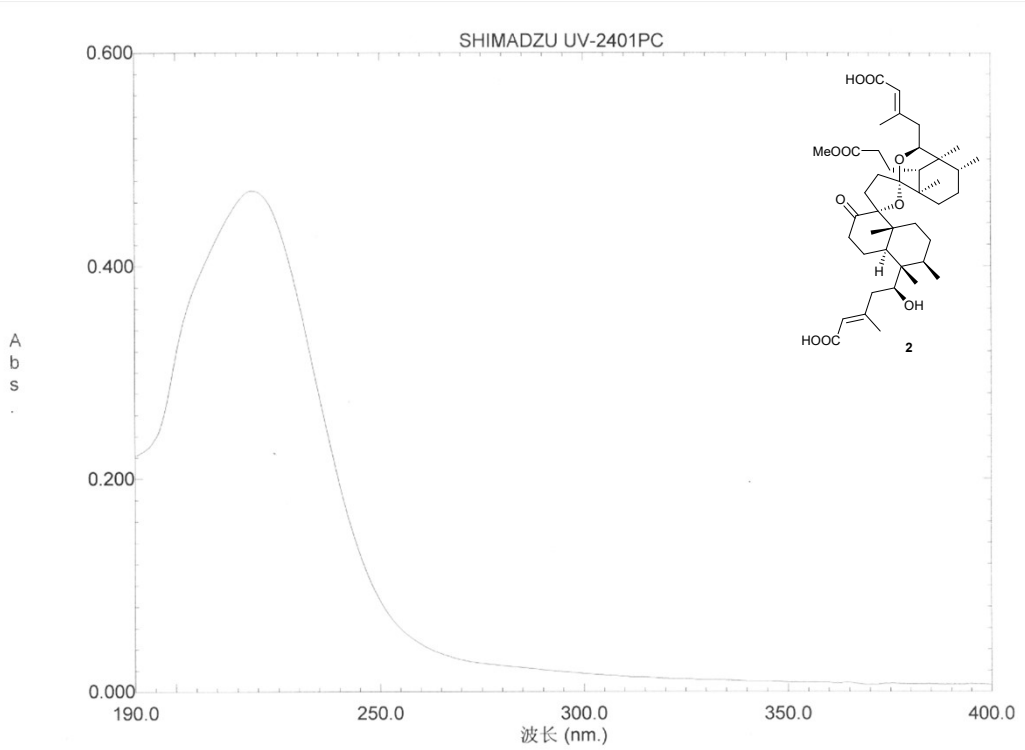


Figure S36. UV spectrum of scoparicacid B (**2**)

Optical rotation measurement

Model : P-1020 (A060460638)

No.	Sample	Mode	Data	Monitor Blank	Temp. Cell Temp Point	Date Comment Sample Name	Light Filter Operator	Cycle Time Integ Time
No.1	35 (1/3)	Sp.Rot	-22.0000	-0.0022	17.6	Thu Dec 21 01:57:15 2017	Na	2 sec
				0.0000	10.00	0.00100g/mL MeOH	589nm	2 sec
					Cell	SXR084		
No.2	35 (2/3)	Sp.Rot	-23.0000	-0.0023	17.5	Thu Dec 21 01:57:20 2017	Na	2 sec
				0.0000	10.00	0.00100g/mL MeOH	589nm	2 sec
					Cell	SXR084		
No.3	35 (3/3)	Sp.Rot	-21.0000	-0.0021	17.5	Thu Dec 21 01:57:26 2017	Na	2 sec
				0.0000	10.00	0.00100g/mL MeOH	589nm	2 sec
					Cell	SXR084		

−22.0000°

Figure S37. OR spectrum of scoparicacid B (**2**)

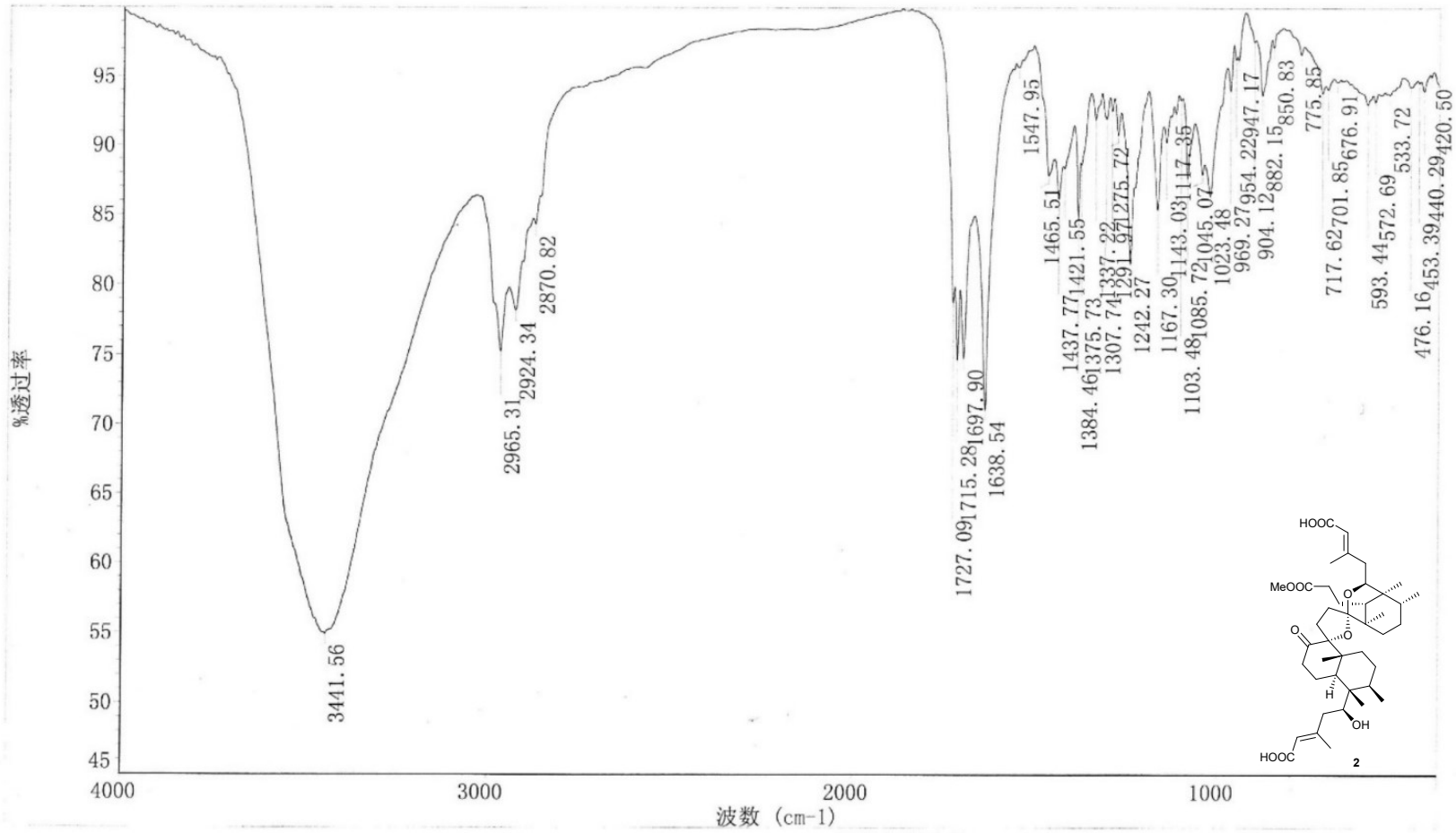


Figure S38. IR spectrum of scoparicacid B (**2**)

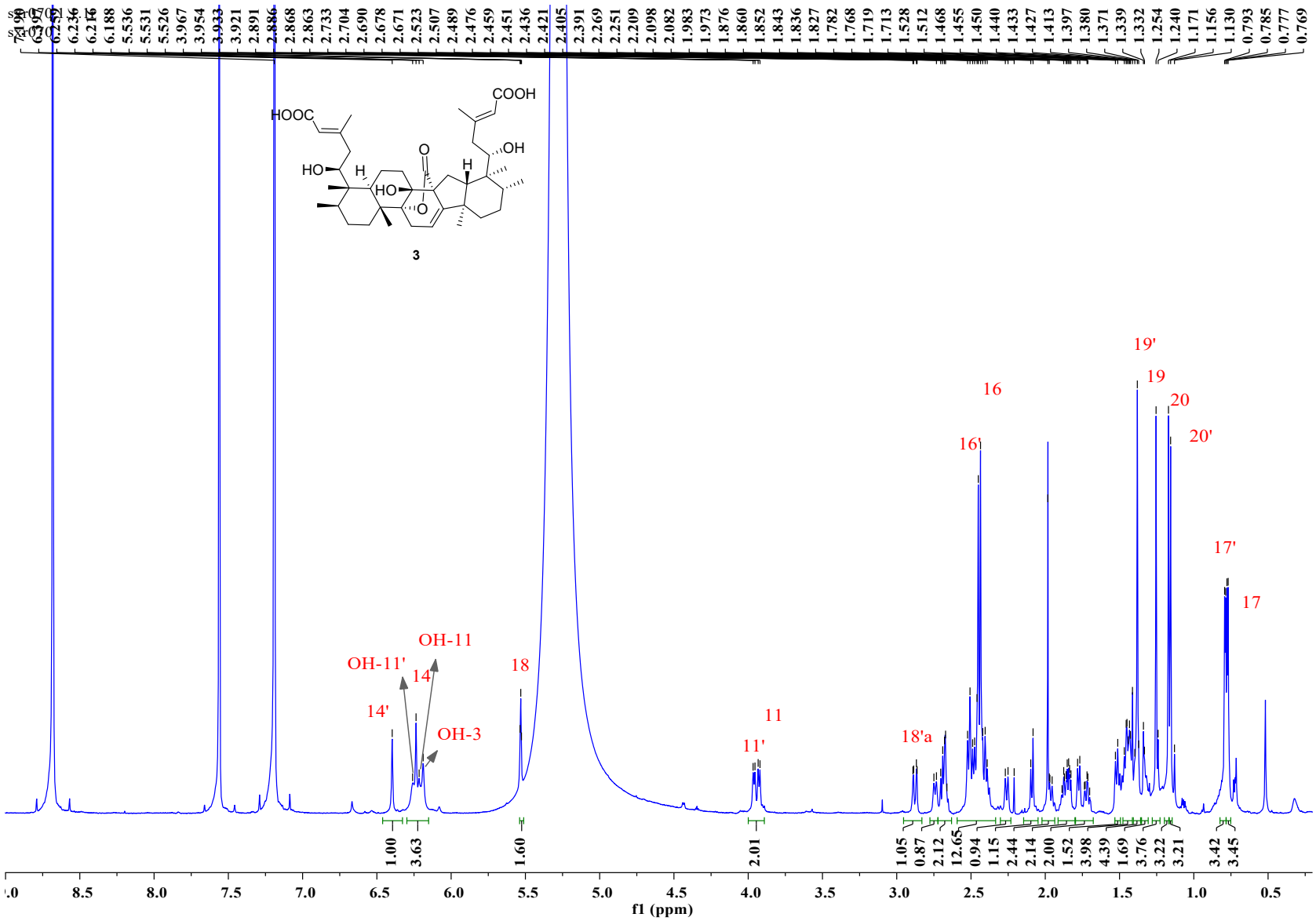


Figure S39. ^{1}H NMR spectrum of scoparicacid C (**3**)

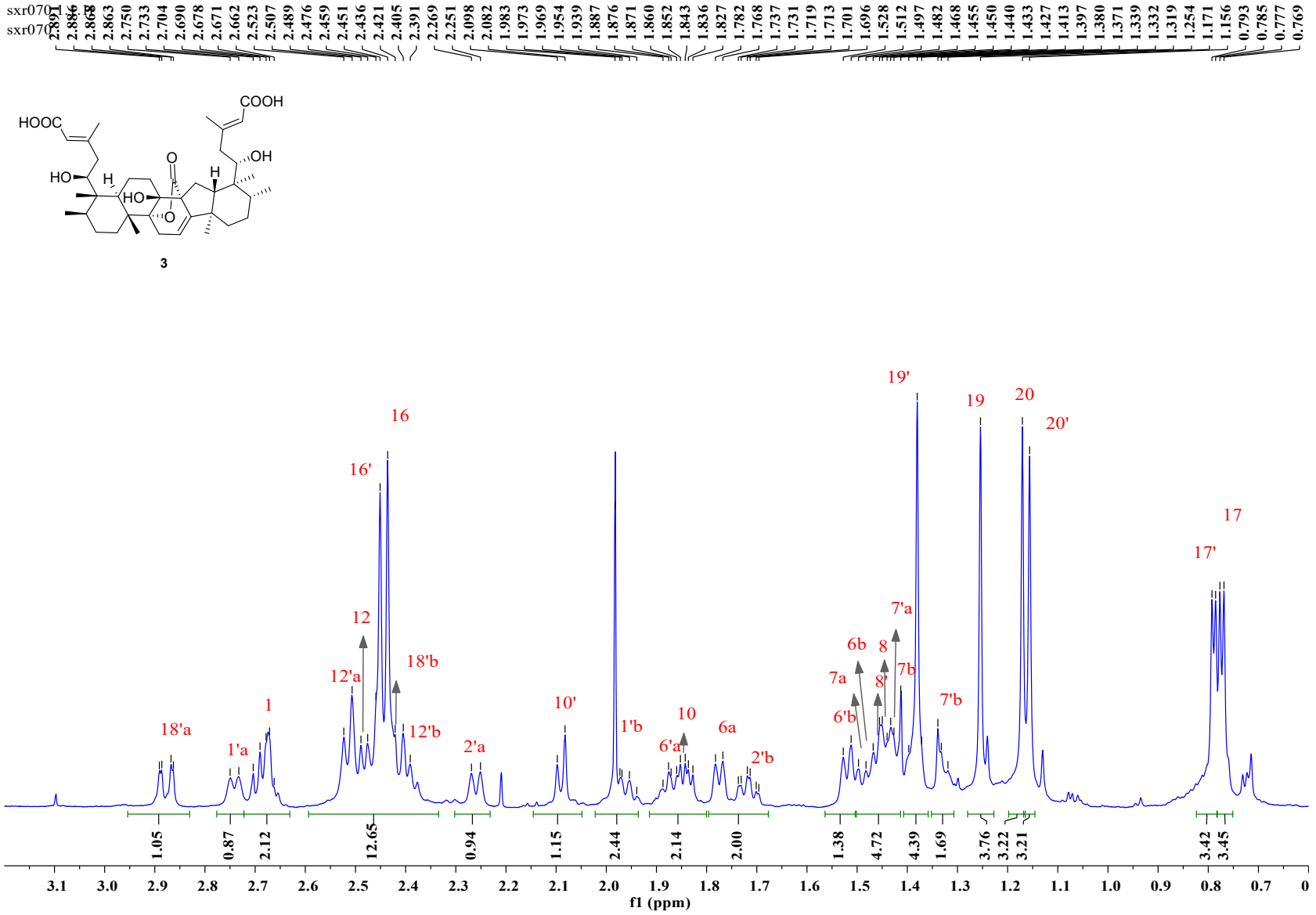


Figure S40. ^{1}H NMR spectrum of scoparicacid C (**3**)

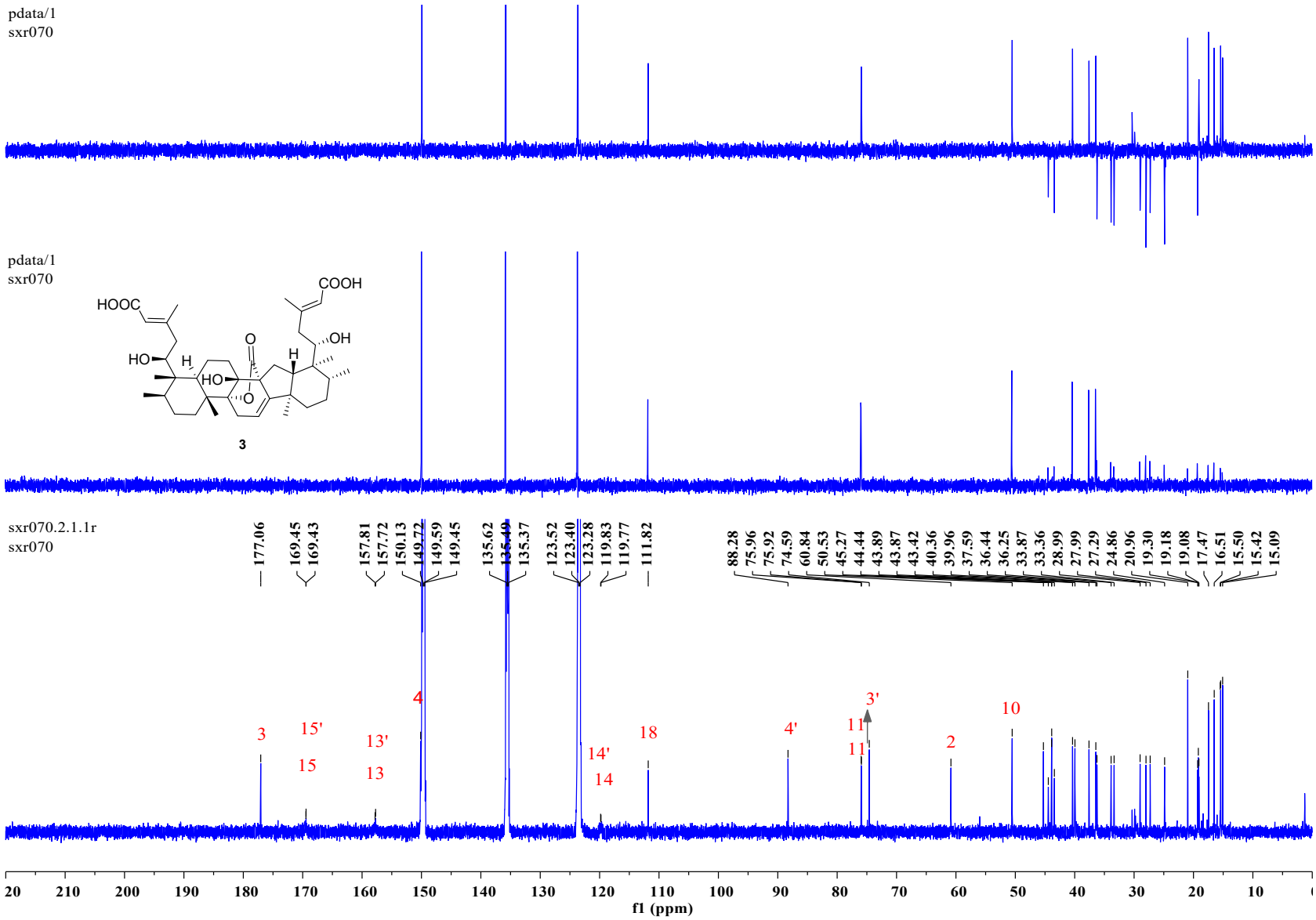


Figure S41. ^{13}C NMR spectrum of scoparicacid C (**3**)

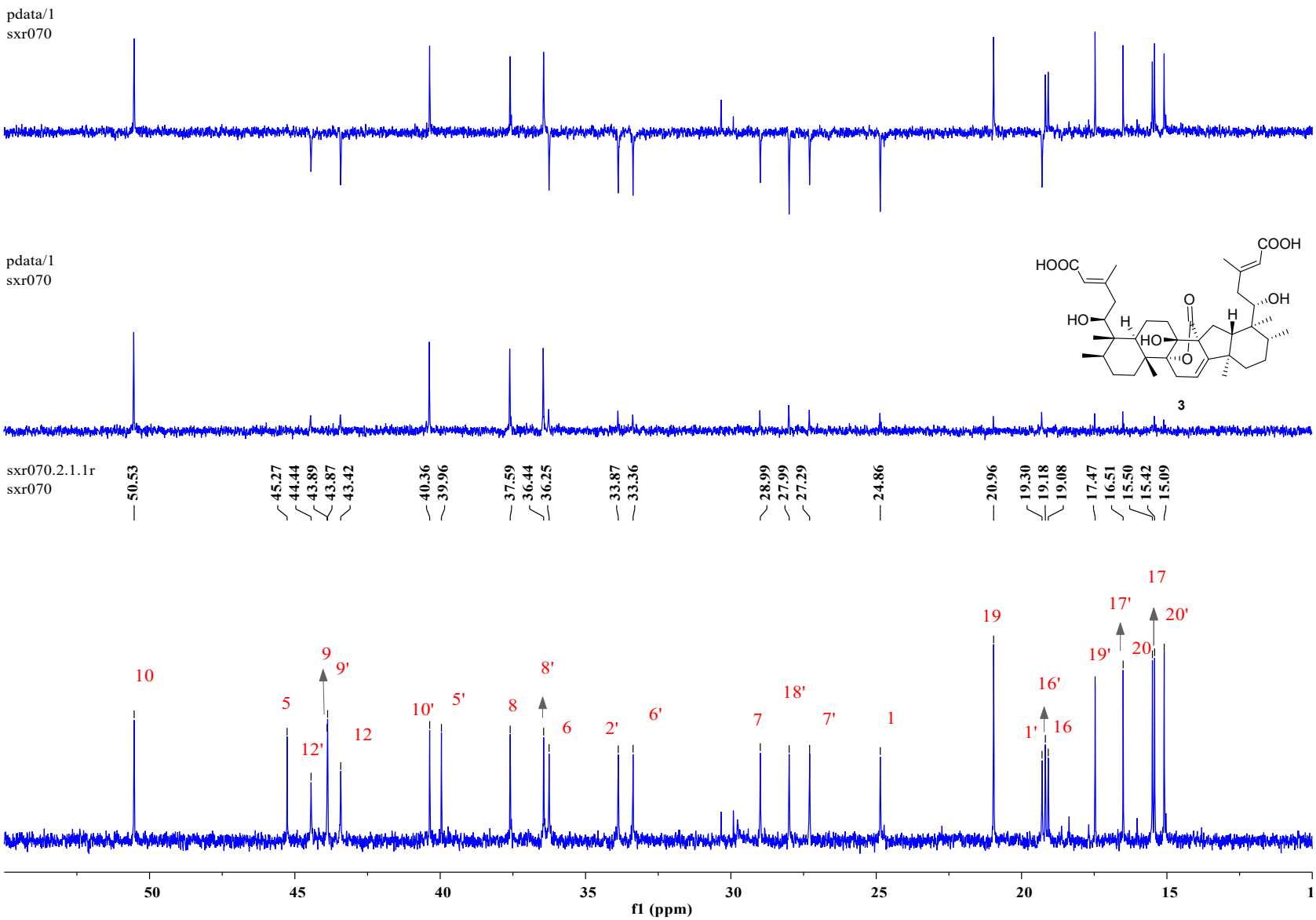


Figure S42. ^{13}C NMR spectrum of scoparicacid C (**3**)

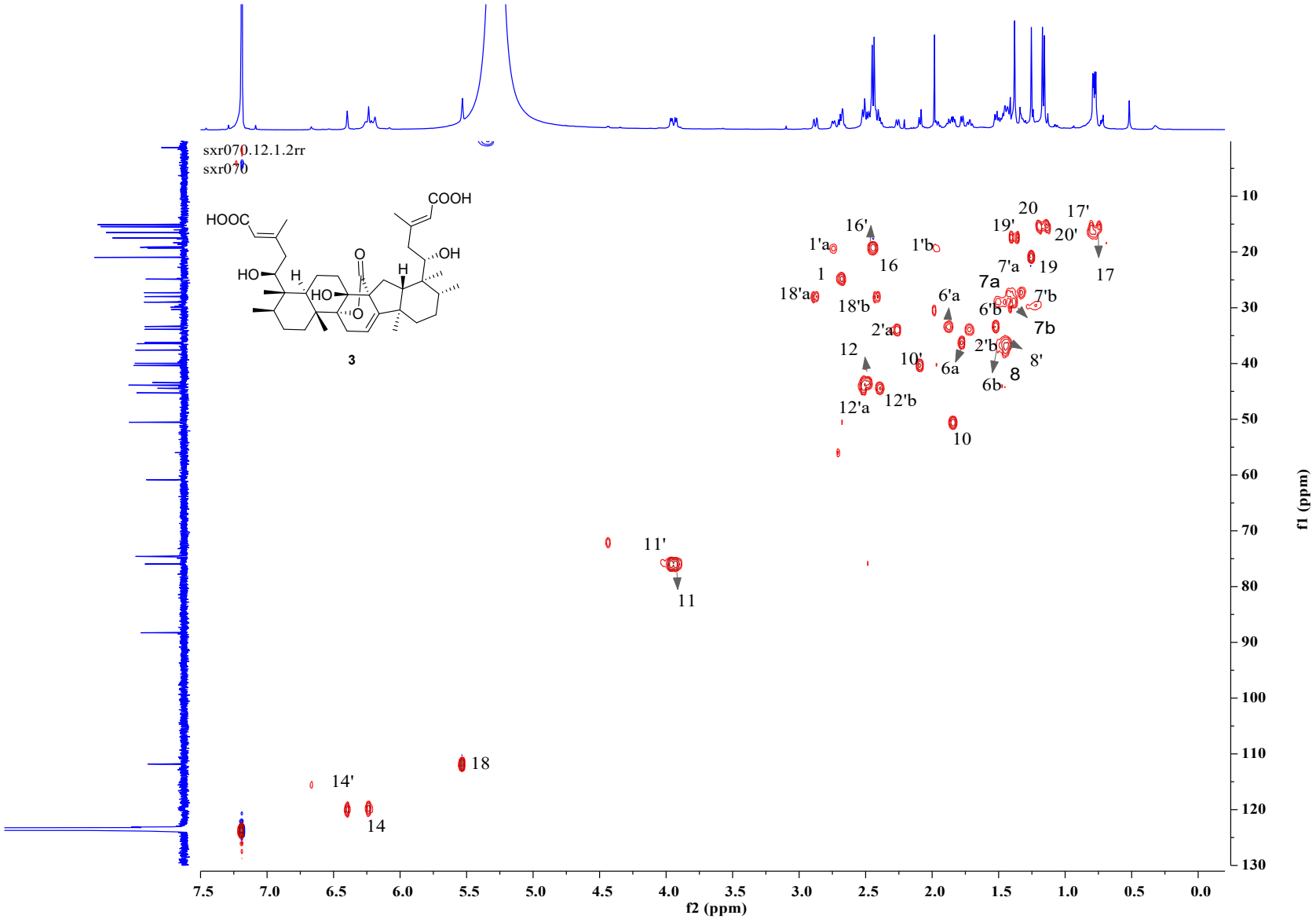


Figure S43. HSQC spectrum of scoparicacid C (**3**)

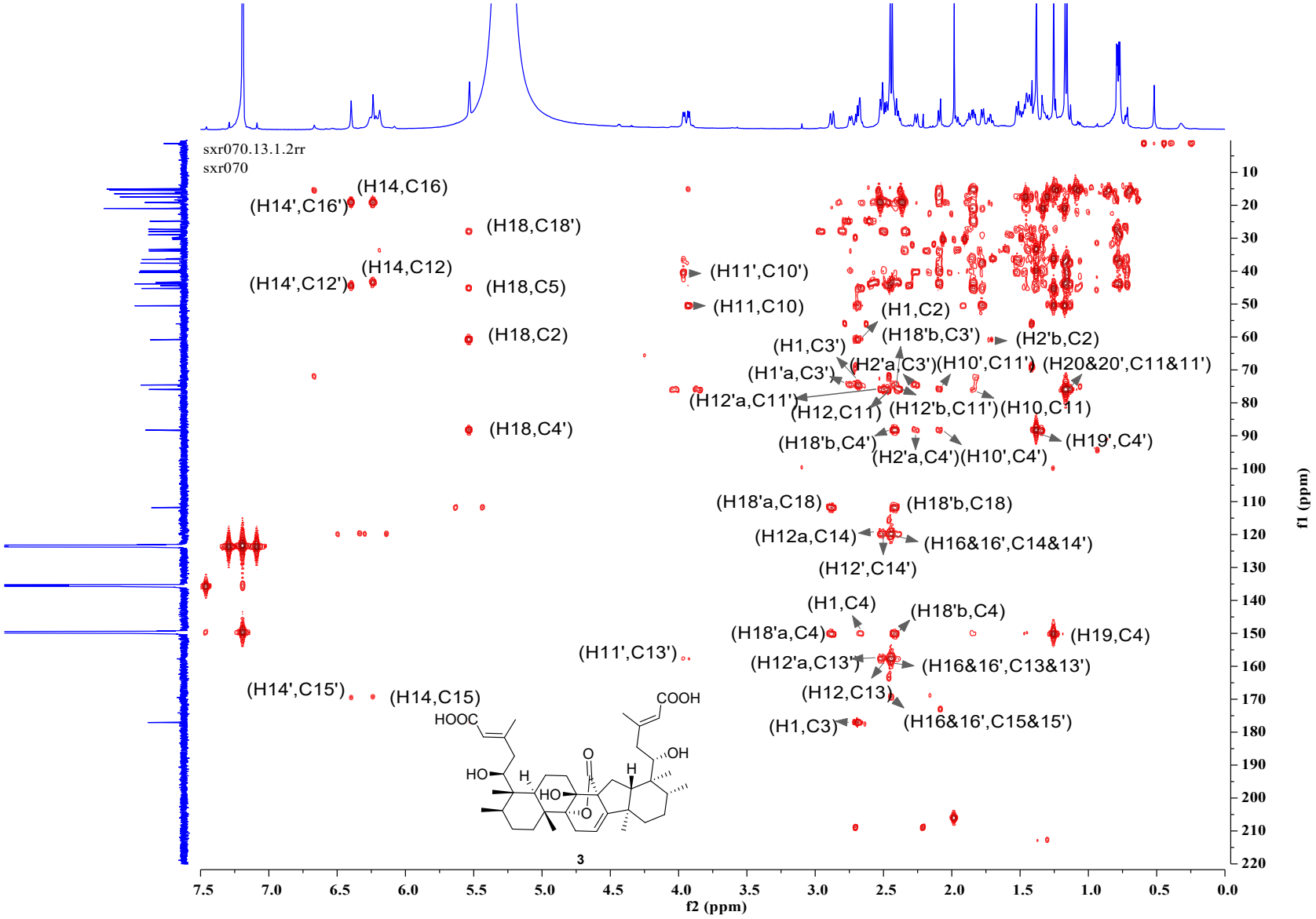


Figure S44. HMBC spectrum of scoparicacid C (**3**)

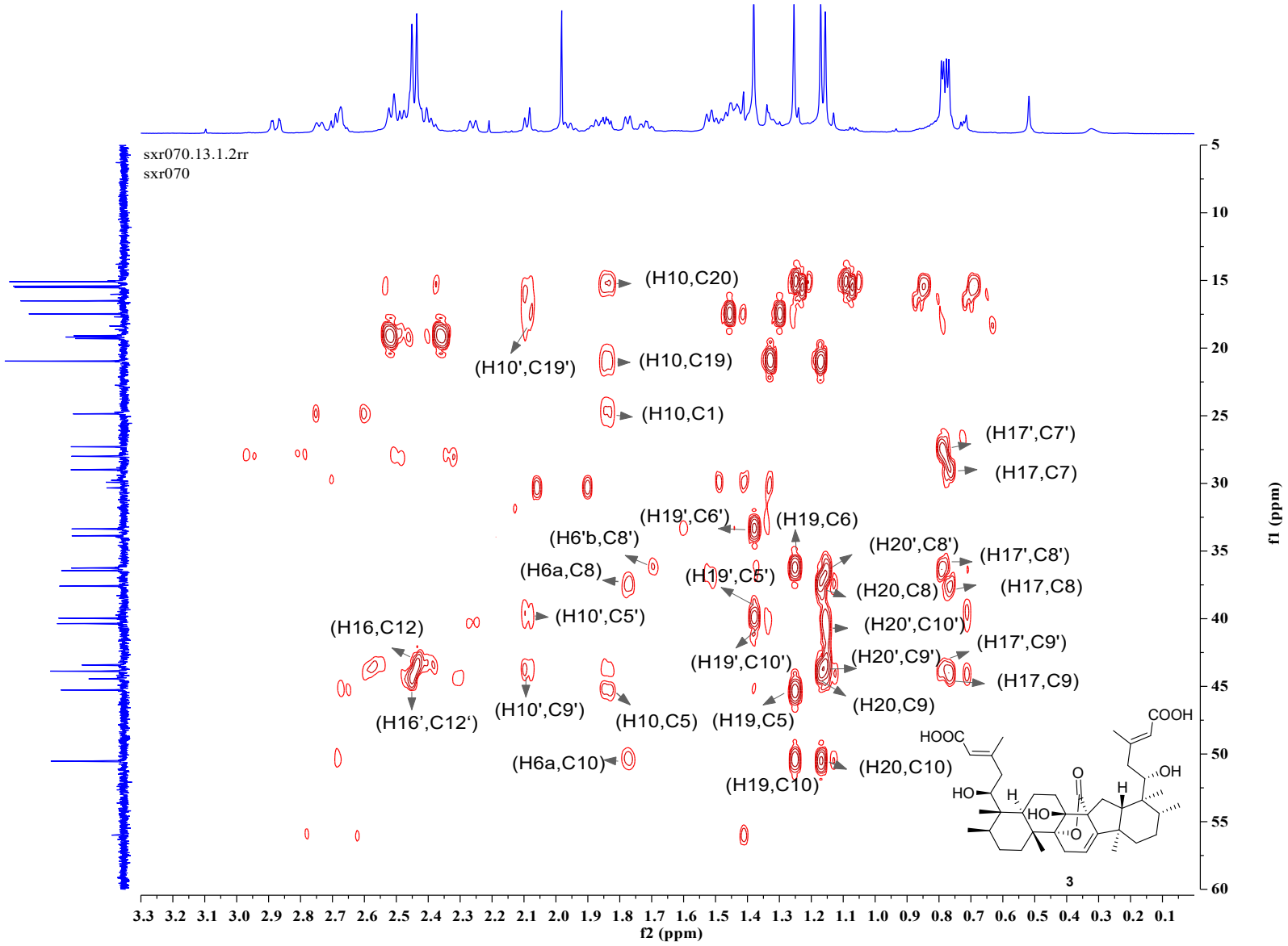


Figure S45. HMBC spectrum of scoparicacid C (**3**)

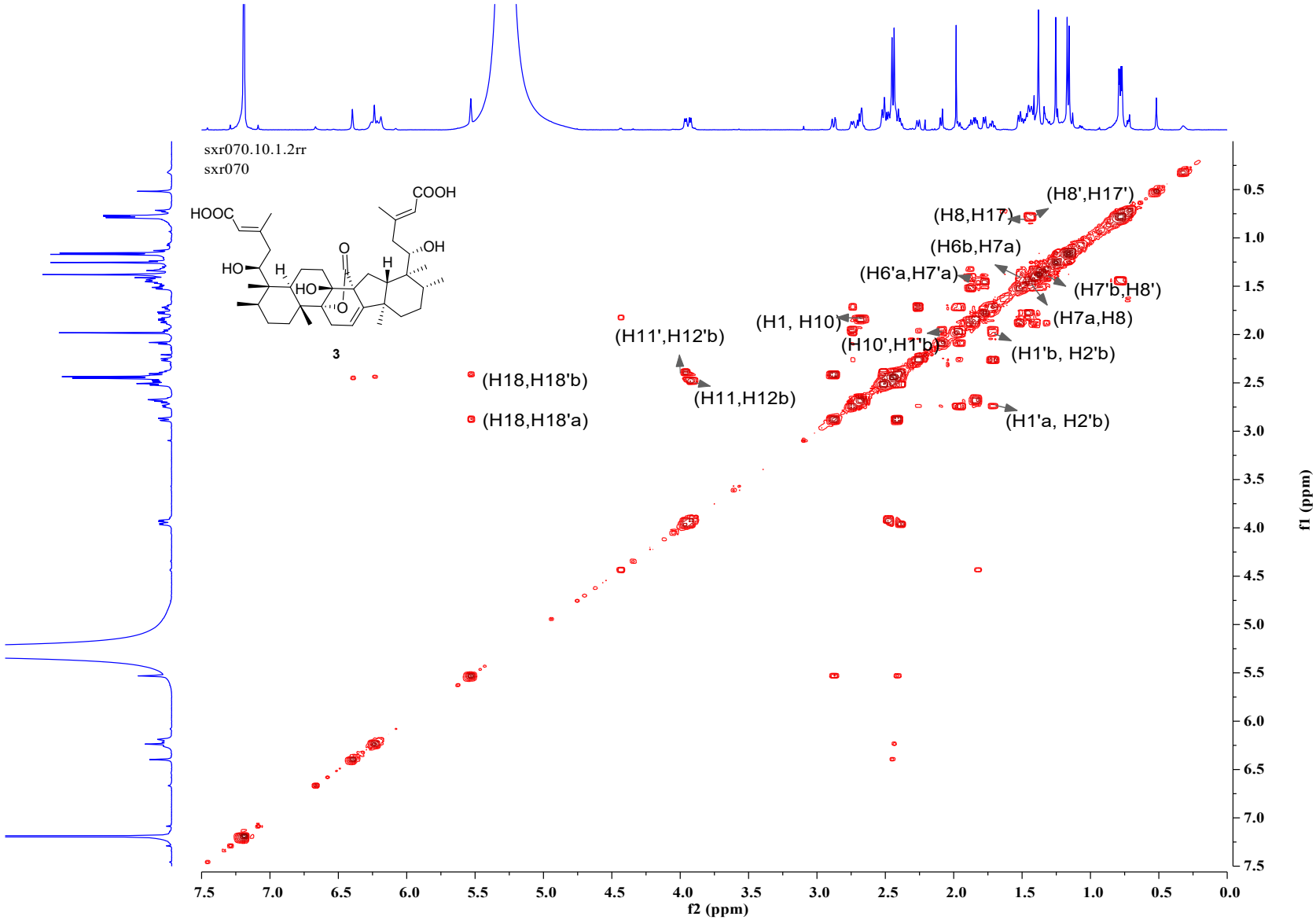


Figure S46. ^{1}H-^{1}H COSY spectrum of scoparicacid C (**3**)

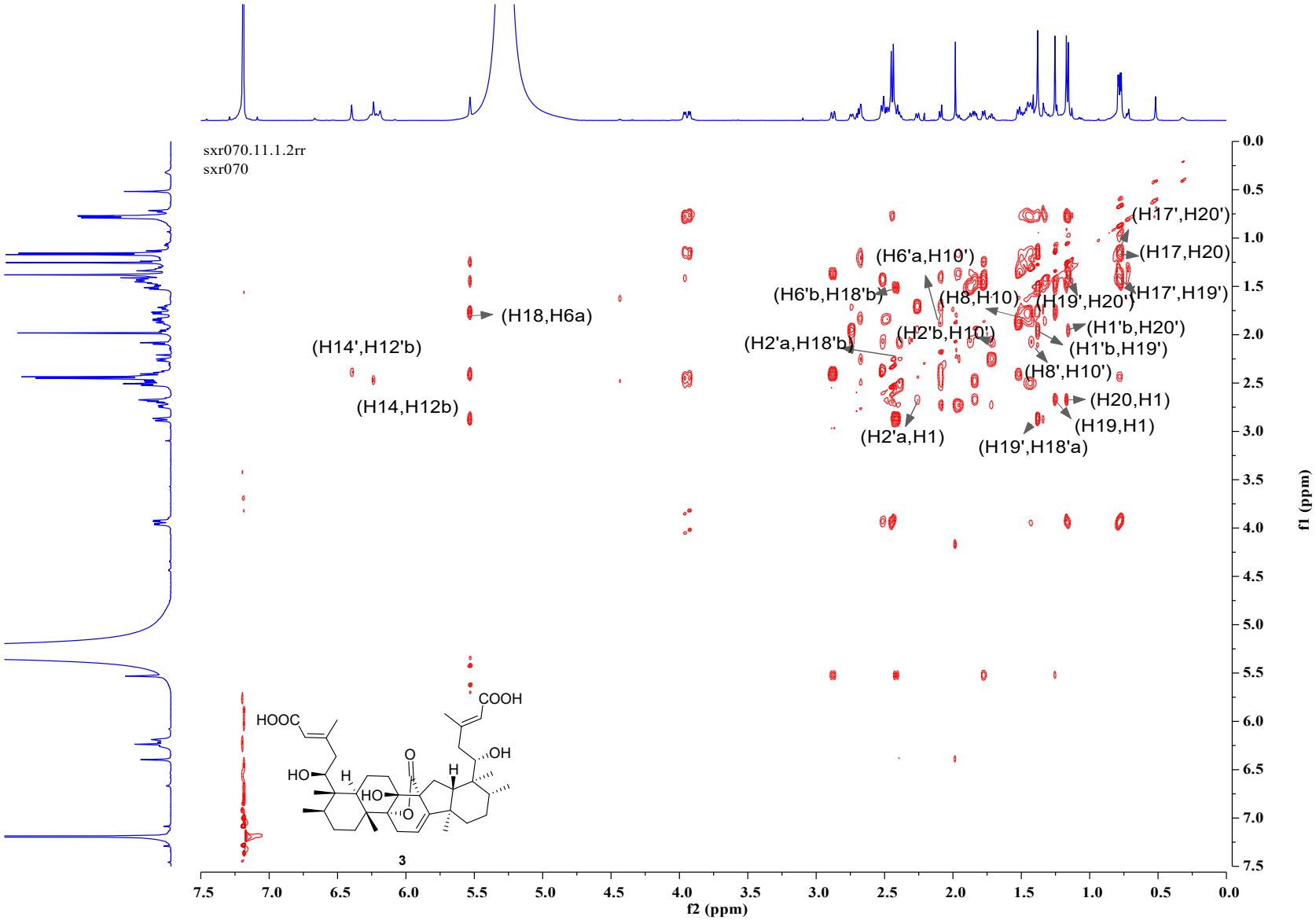


Figure S47. ROESY spectrum of scoparicacid C (**3**)

Formula Predictor Report - sxr070.lcd

Data File: E:\DATA\2017\0801\sxr070.lcd

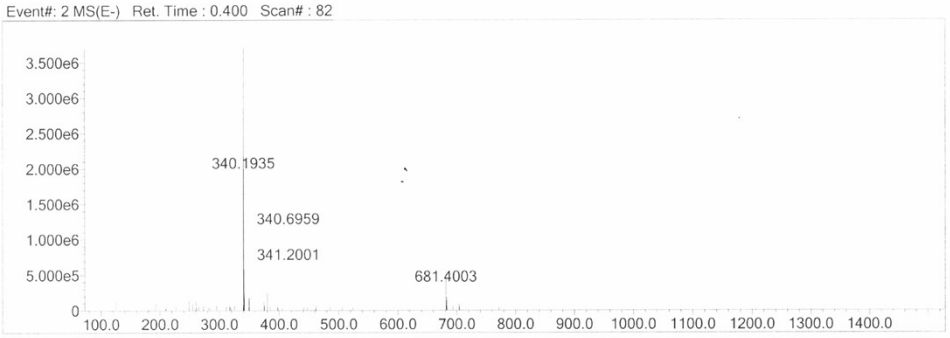
Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Use Adduct
H	1	0	150	O	2	0	50	S	2	0	0	Pt	2	0	0	H
B	3	0	0	F	1	0	0	Cl	1	0	0					
C	4	0	82	Na	1	0	0	Br	1	0	0					
N	3	0	0	Si	4	0	0	I	3	0	0					

Error Margin (ppm): 10
HC Ratio: unlimited
Max Isotopes: all
MSn Iso RI (%): 75.00

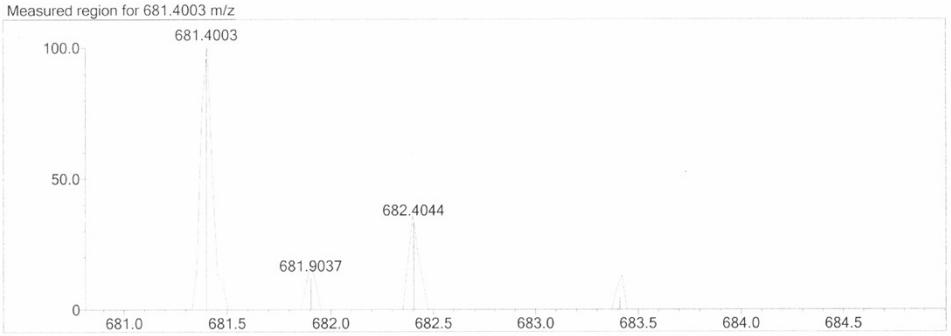
DBE Range: -2.0 - 100.0
Apply N Rule: yes
Isotope RI (%): 1.00
MSn Logic Mode: AND

Electron Ions: both
Use MSn Info: yes
Isotope Res: 10000
Max Results: 10

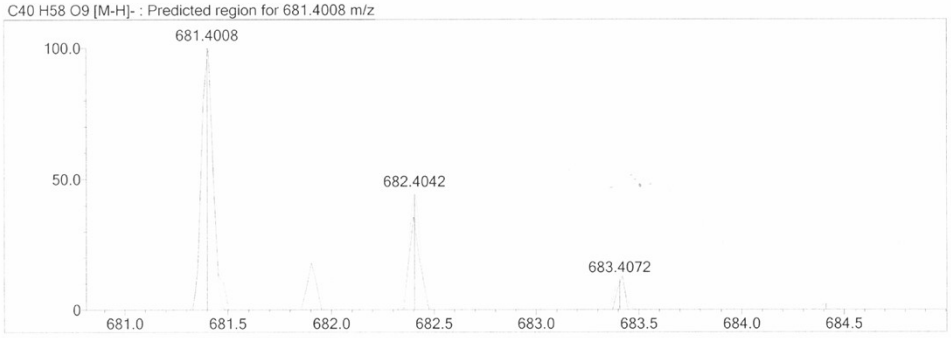
Event#: 2 MS(E-) Ret. Time : 0.400 Scan# : 82



Measured region for 681.4003 m/z



C40 H58 O9 [M-H]- : Predicted region for 681.4008 m/z



Formula (M)	Ion	Meas. m/z	Pred. m/z	Df. (mDa)	Df. (ppm)	DBE
C40 H58 O9	[M-H]-	681.4003	681.4008	-0.5	-0.73	12.0

Figure S48. HRESIMS spectrum of scoparicacid C (**3**)

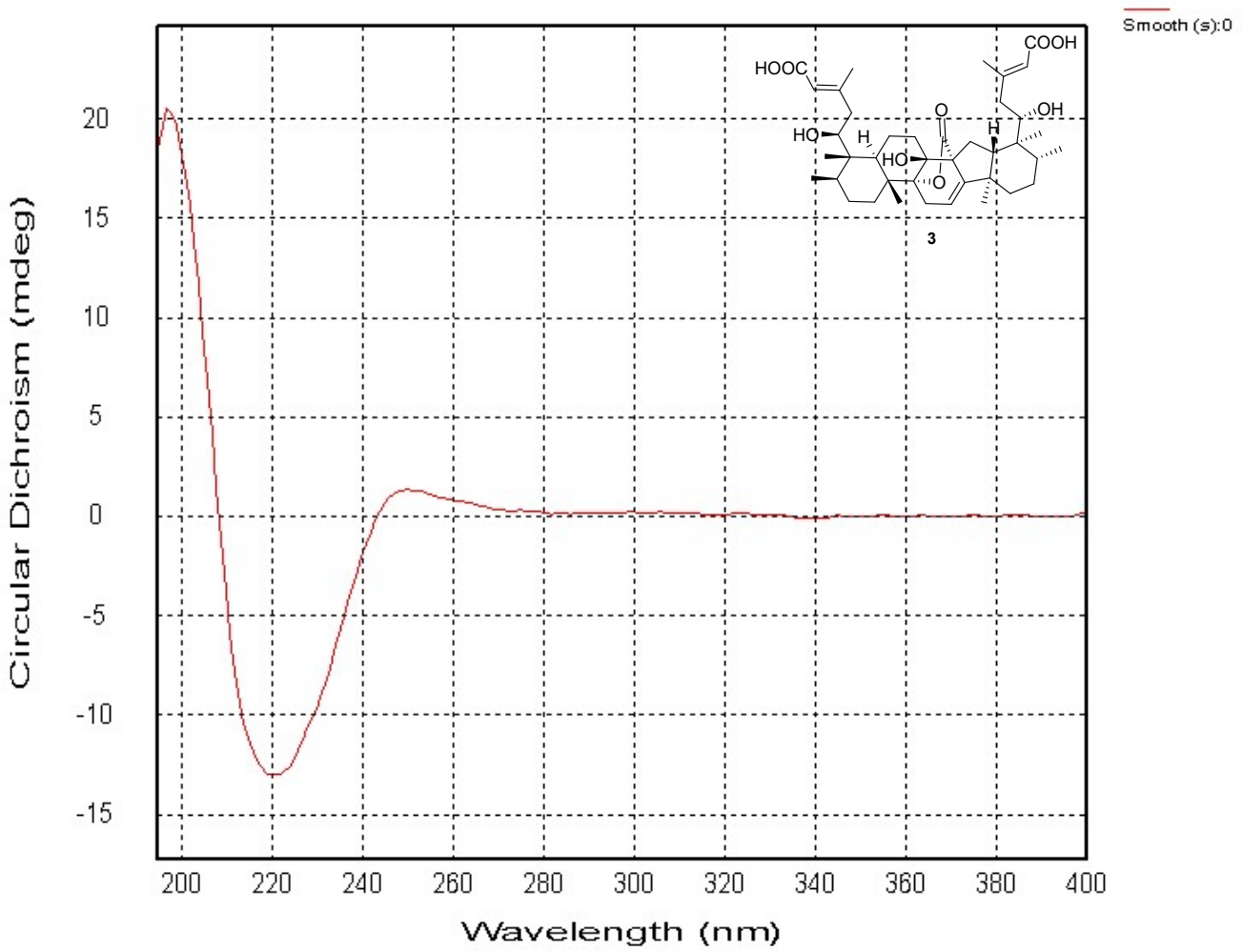


Figure S49. ECD spectrum of scoparicacid C (**3**)

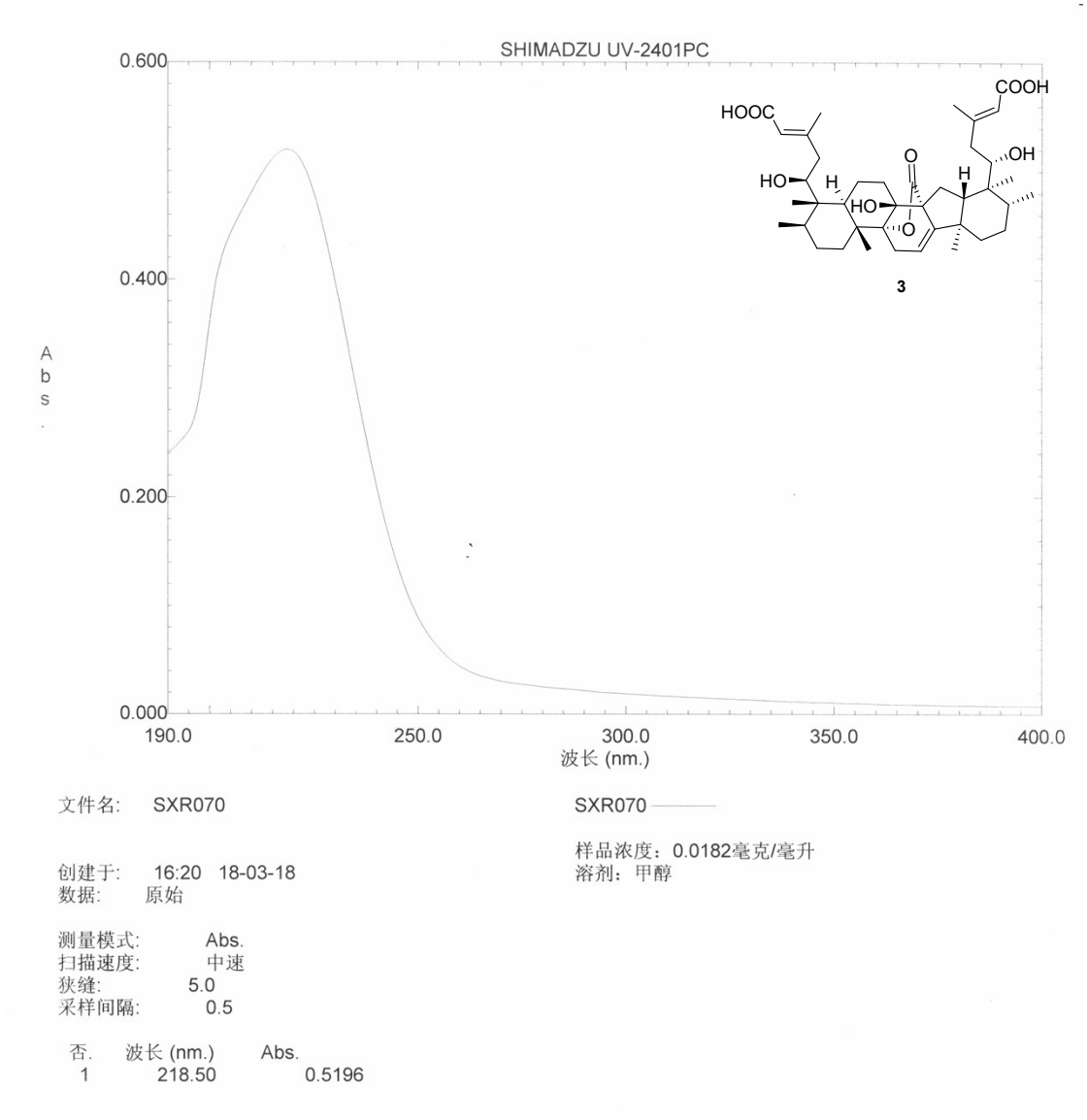


Figure S50. UV spectrum of scoparicacid C (**3**)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Friday, 15-OCT-2021

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

n	Average	Std.Dev.	% RSD	Maximum	Minimum
5	-9.00	1.61	-17.88	-7.25	-11.50

S.No	Sample ID	Time	Result	Scale	OR °Arc	WLG.nm	Lg.mm	Conc.g/100ml	Temp.
1	sxr070	05:46:11 PM	-11.50	SR	-0.0092	589	100.00	0.080	20.9
2	sxr070	05:46:19 PM	-8.50	SR	-0.0068	589	100.00	0.080	20.9
3	sxr070	05:46:27 PM	-7.25	SR	-0.0058	589	100.00	0.080	20.9
4	sxr070	05:46:36 PM	-8.25	SR	-0.0066	589	100.00	0.080	20.9
5	sxr070	05:46:44 PM	-9.50	SR	-0.0076	589	100.00	0.080	20.9

Figure S51. OR spectrum of scoparicacid C (**3**)

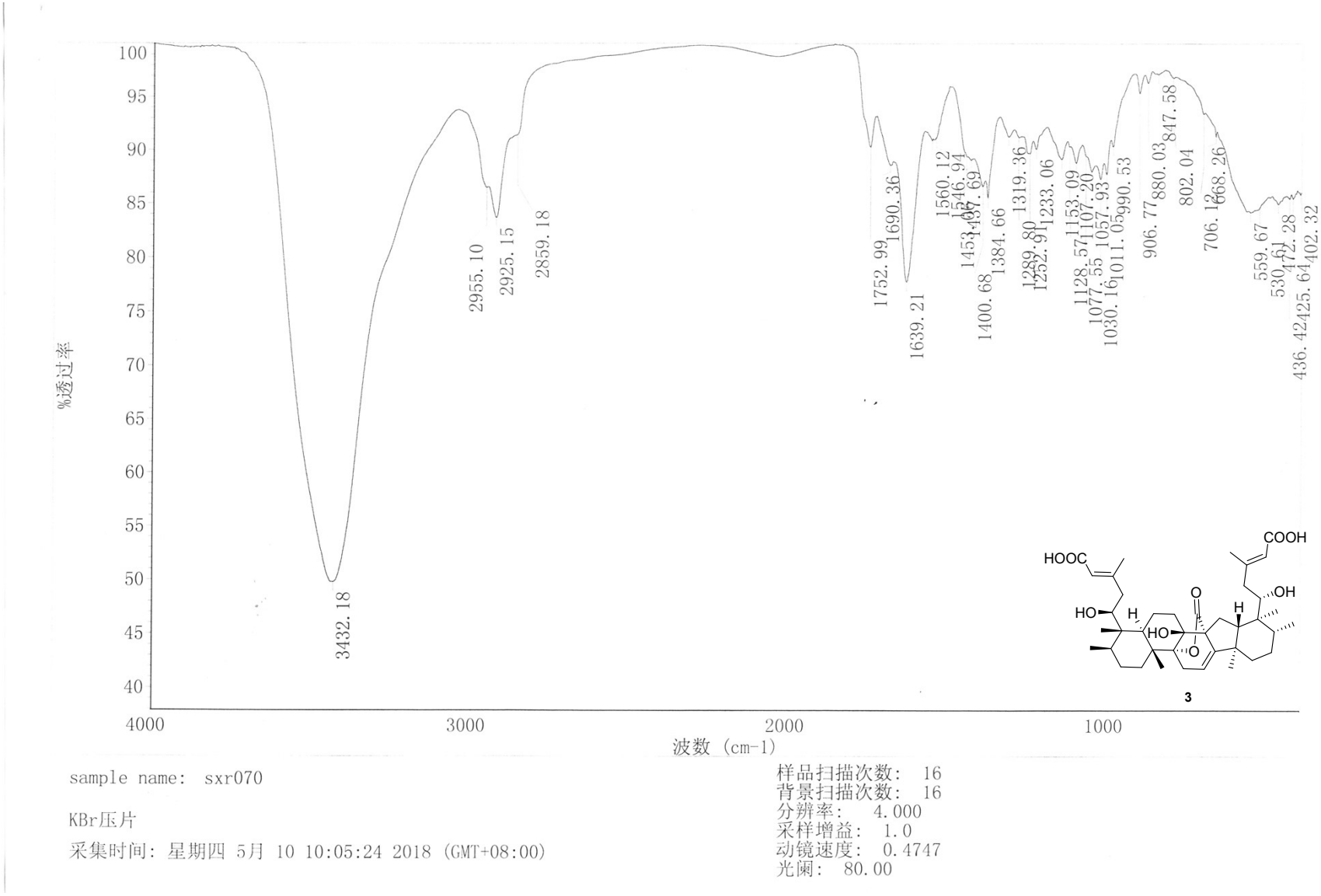


Figure S52. IR spectrum of scoparicacid C (**3**)

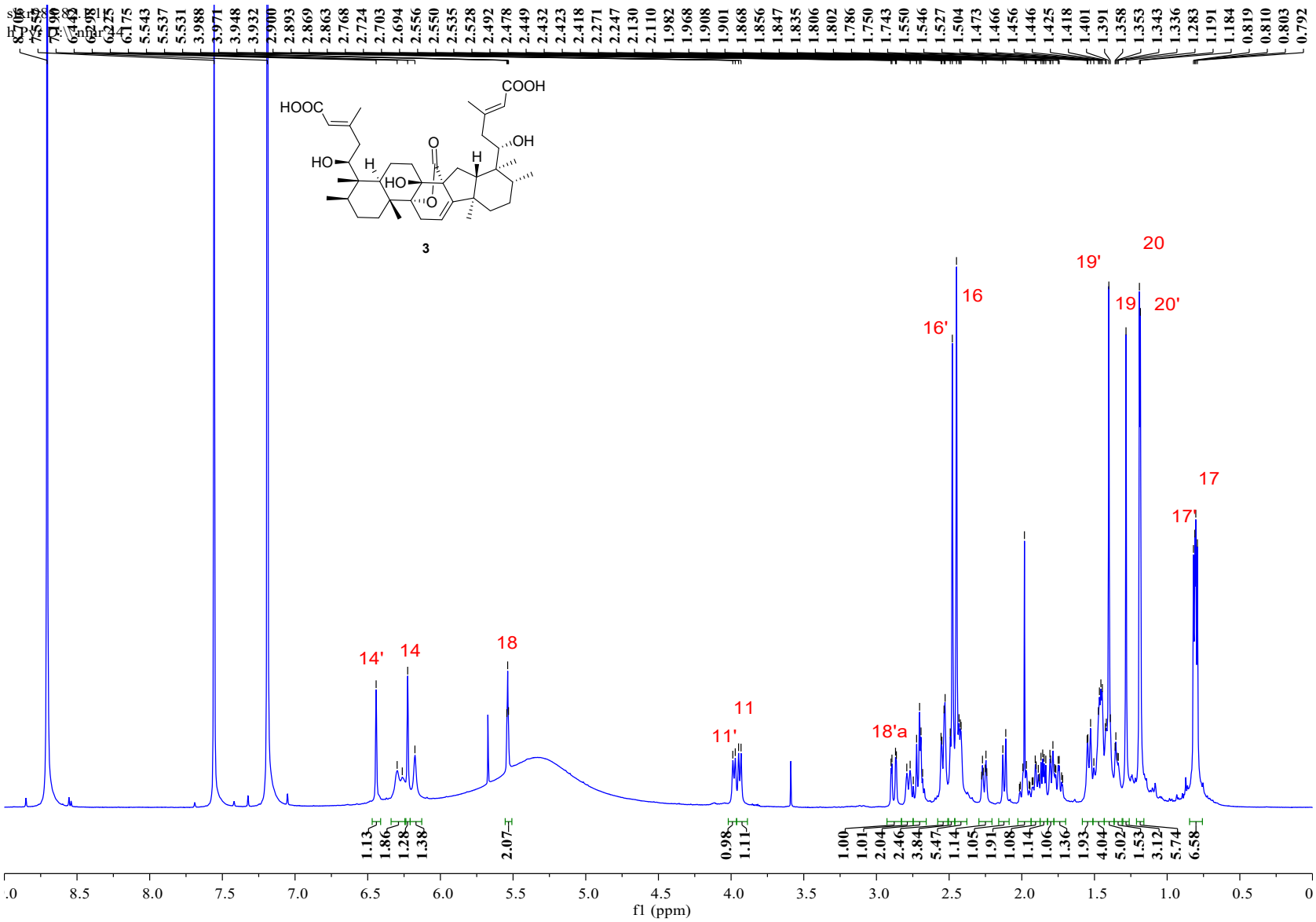


Figure S53. [1]H NMR spectrum of synthetic scoparicacid C (**3**)

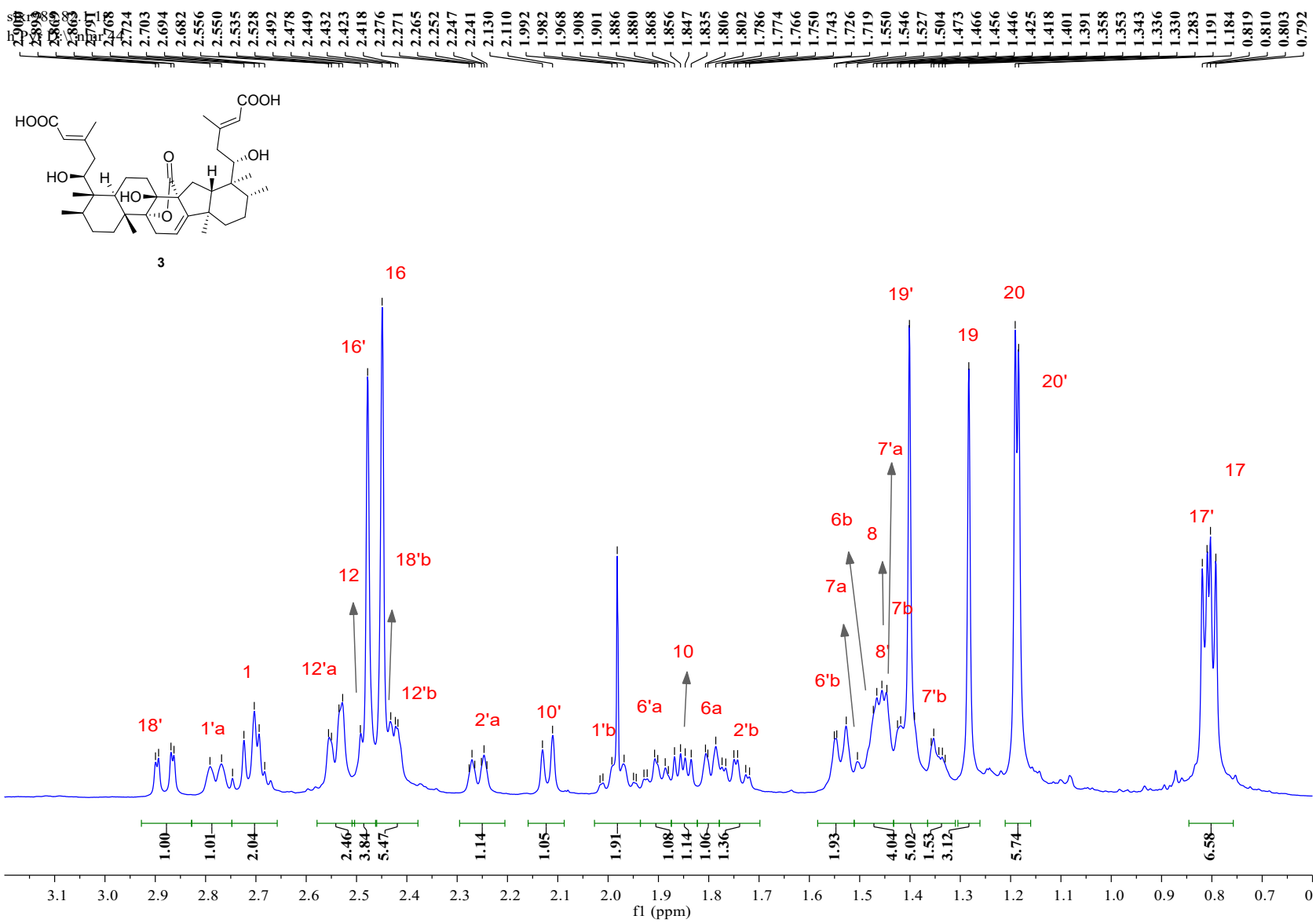


Figure S54. ^{1}H NMR spectrum of synthetic scoparicacid C (**3**)

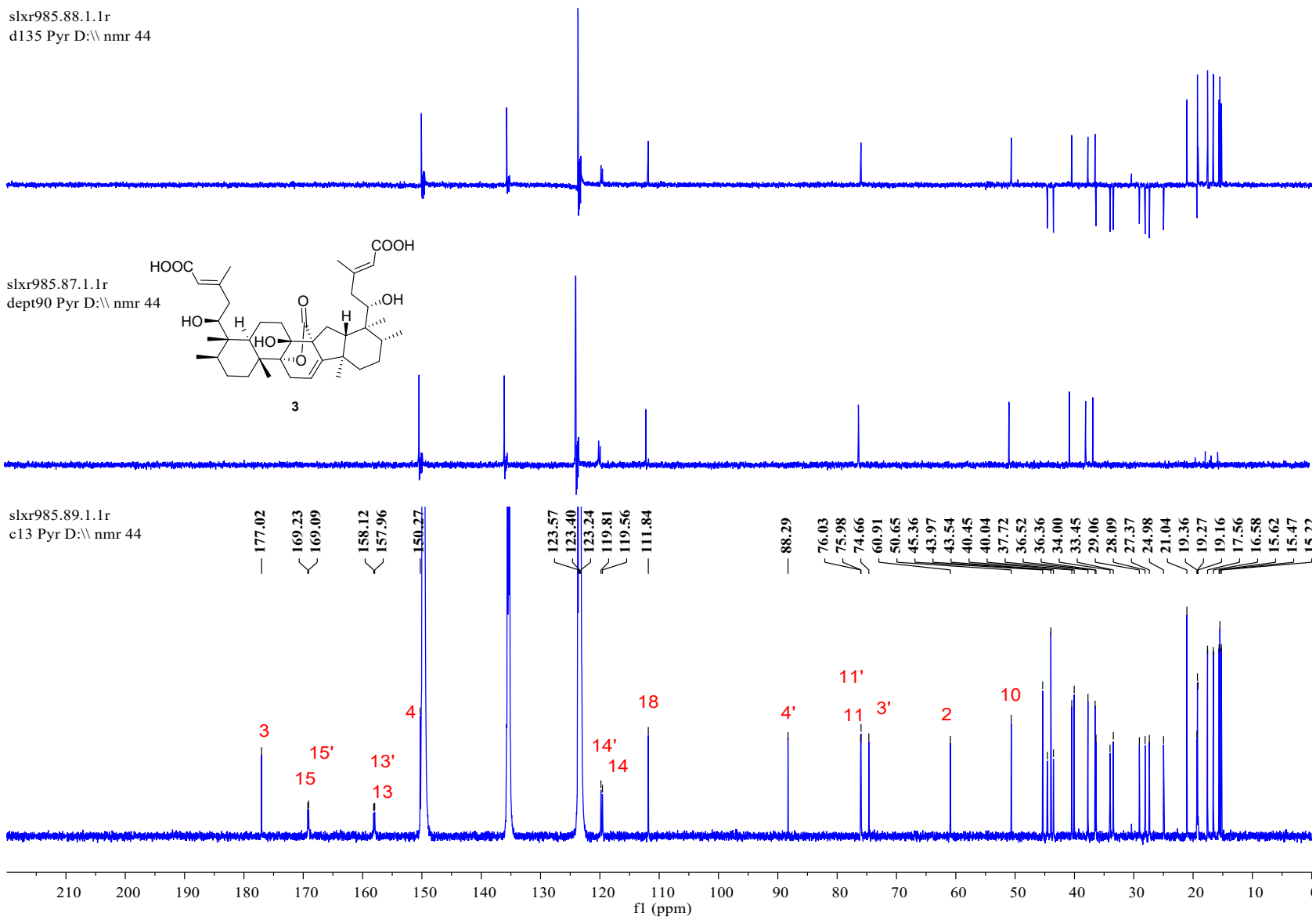


Figure S55. ^{13}C NMR spectrum of synthetic scoparicacid C (**3**)

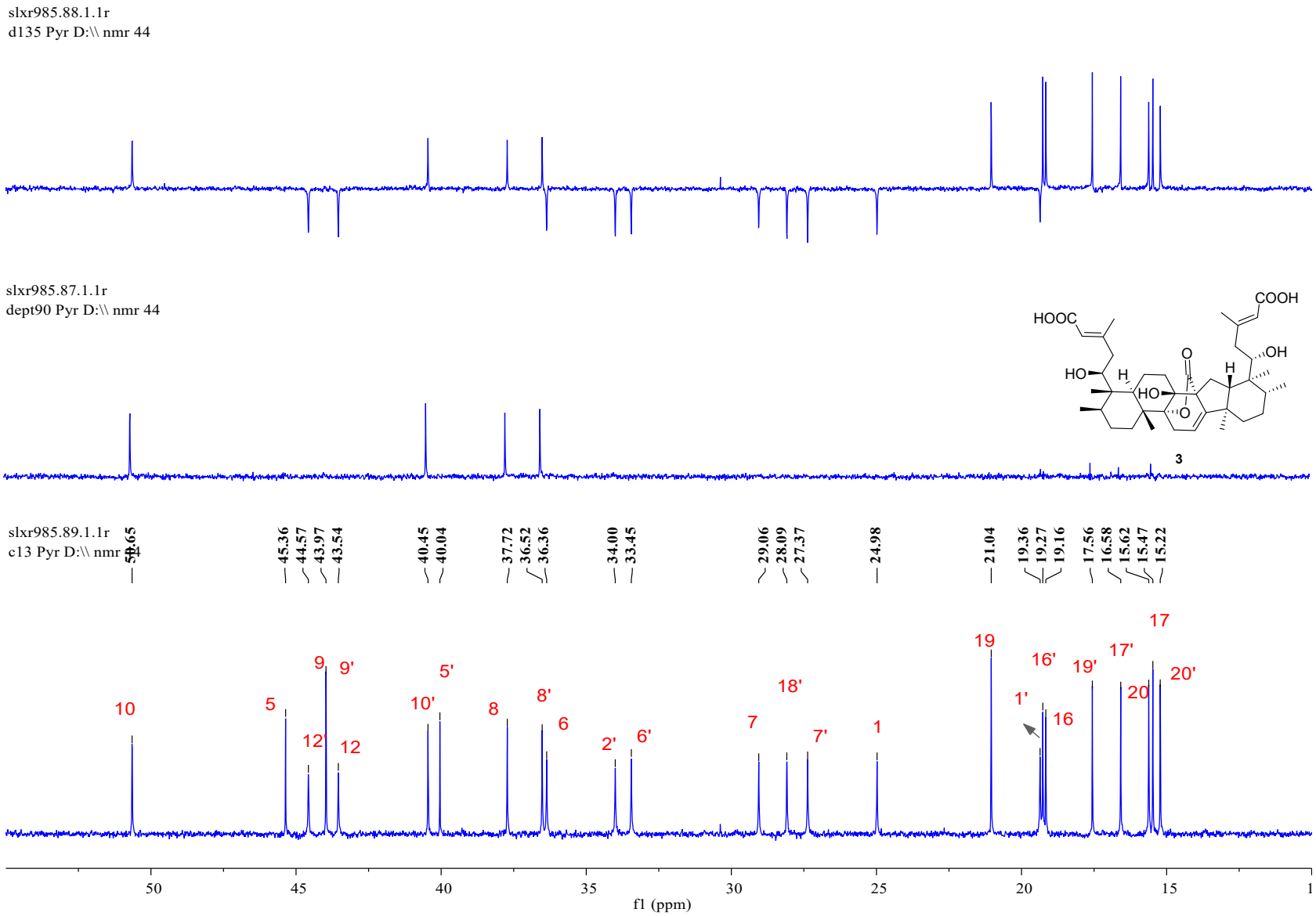


Figure S56. ^{13}C NMR spectrum of synthetic scoparicacid C (**3**)

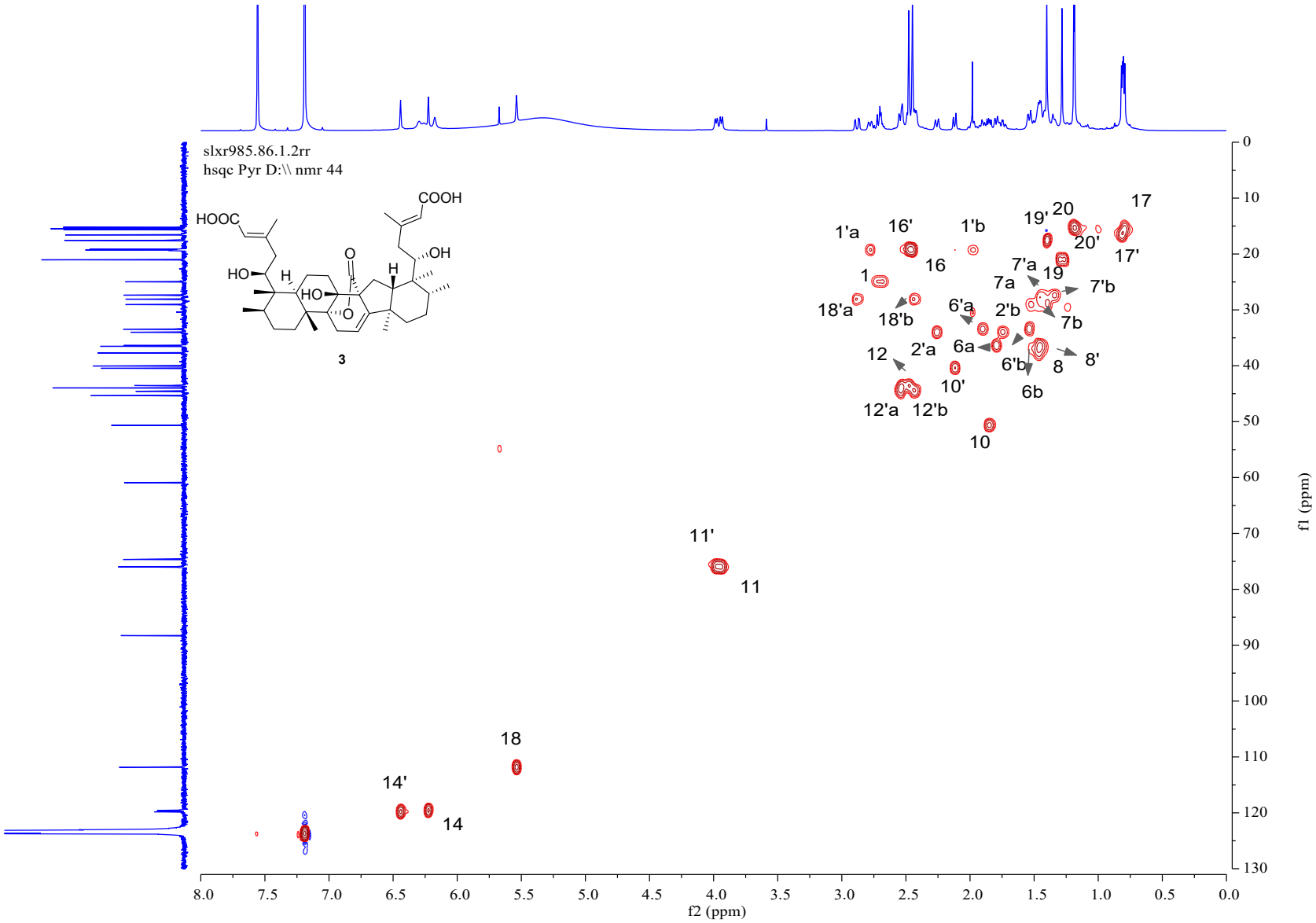


Figure S57. HSQC spectrum of synthetic scoparicacid C (**3**)

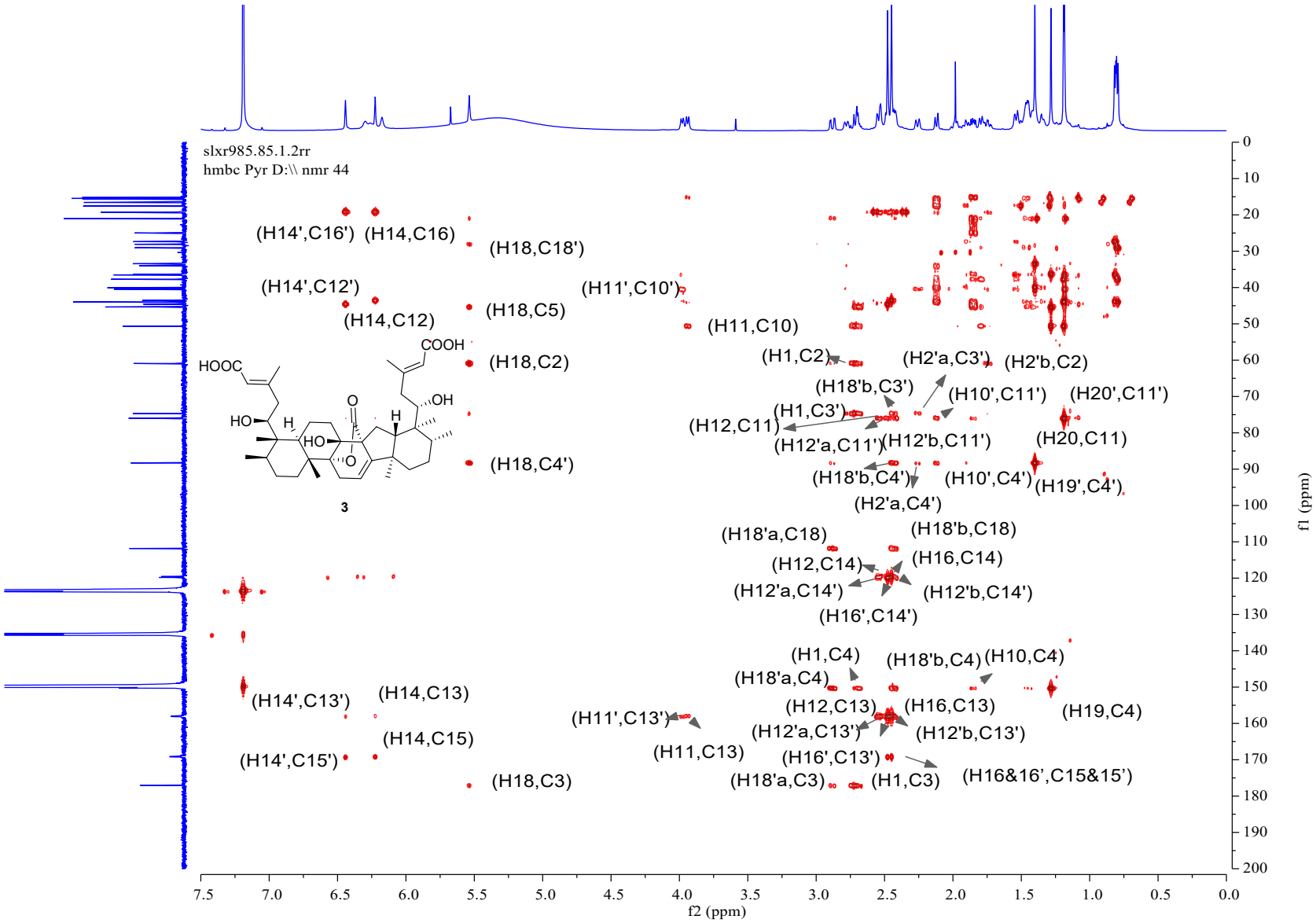


Figure S58. HMBC spectrum of synthetic scoparicacid C (**3**)

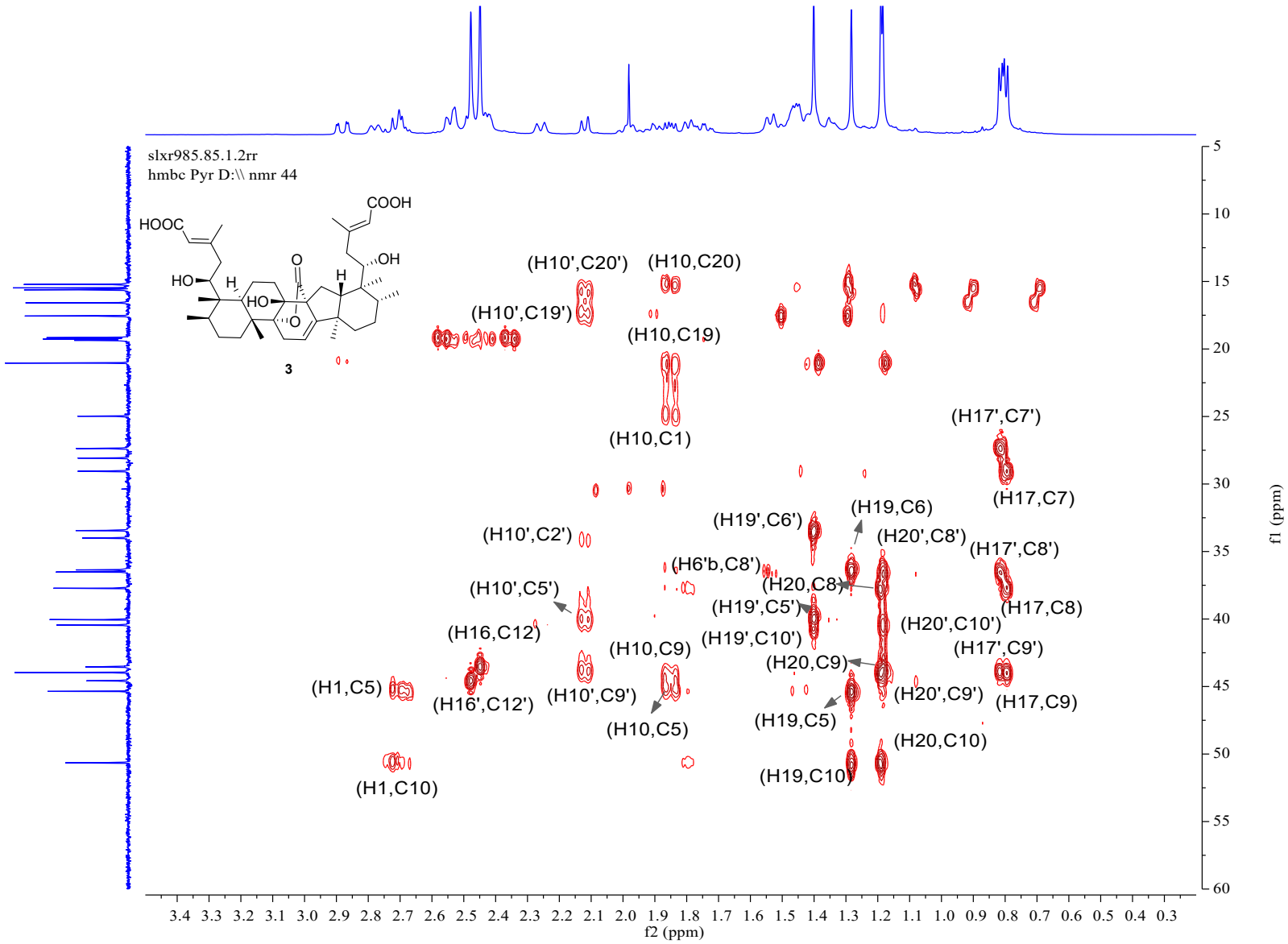


Figure S59. HMBC NMR spectrum of synthetic scoparicacid C (**3**)

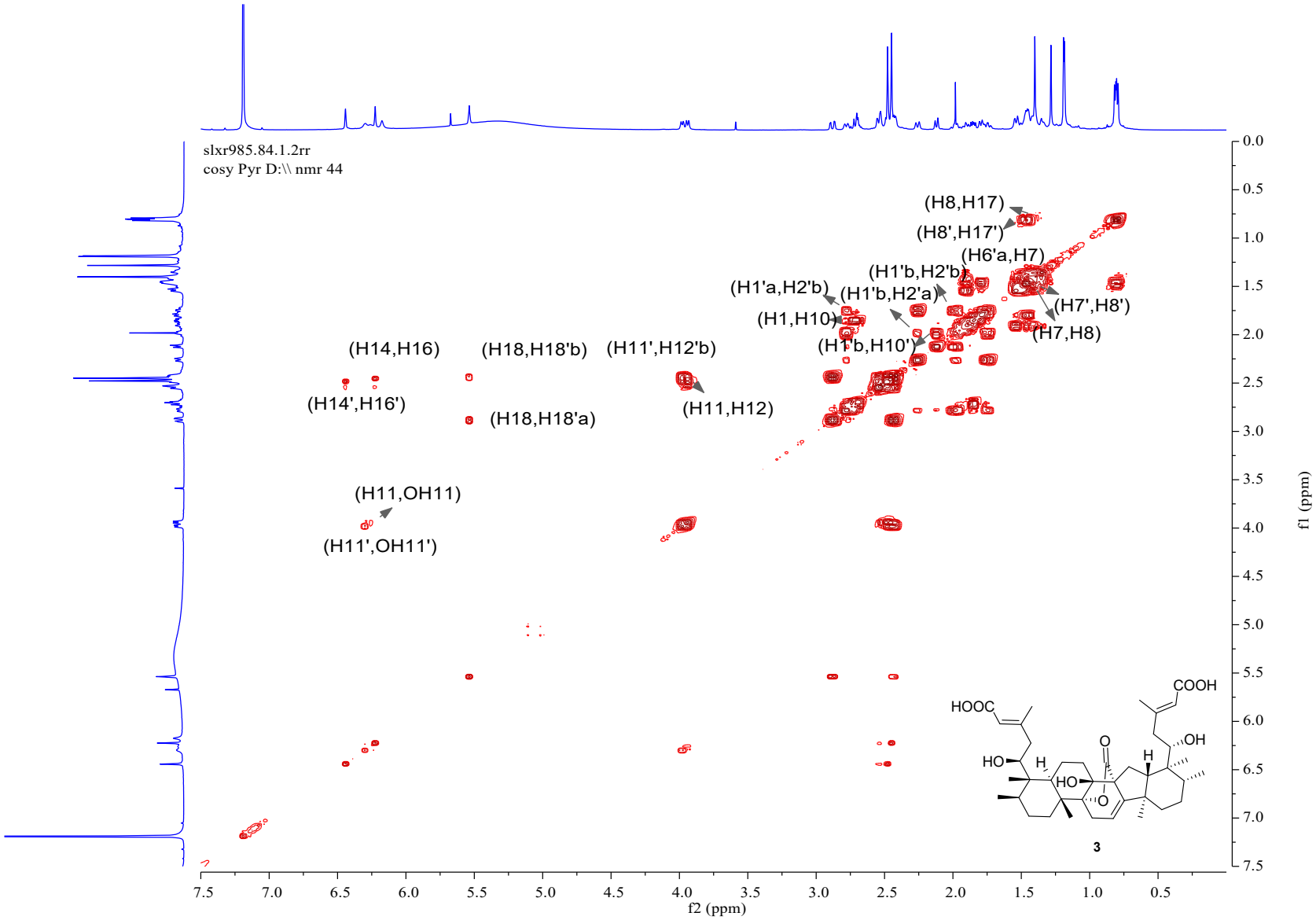


Figure S60. ^{1}H-^{1}H COSY spectrum of synthetic scoparicacid C (**3**)

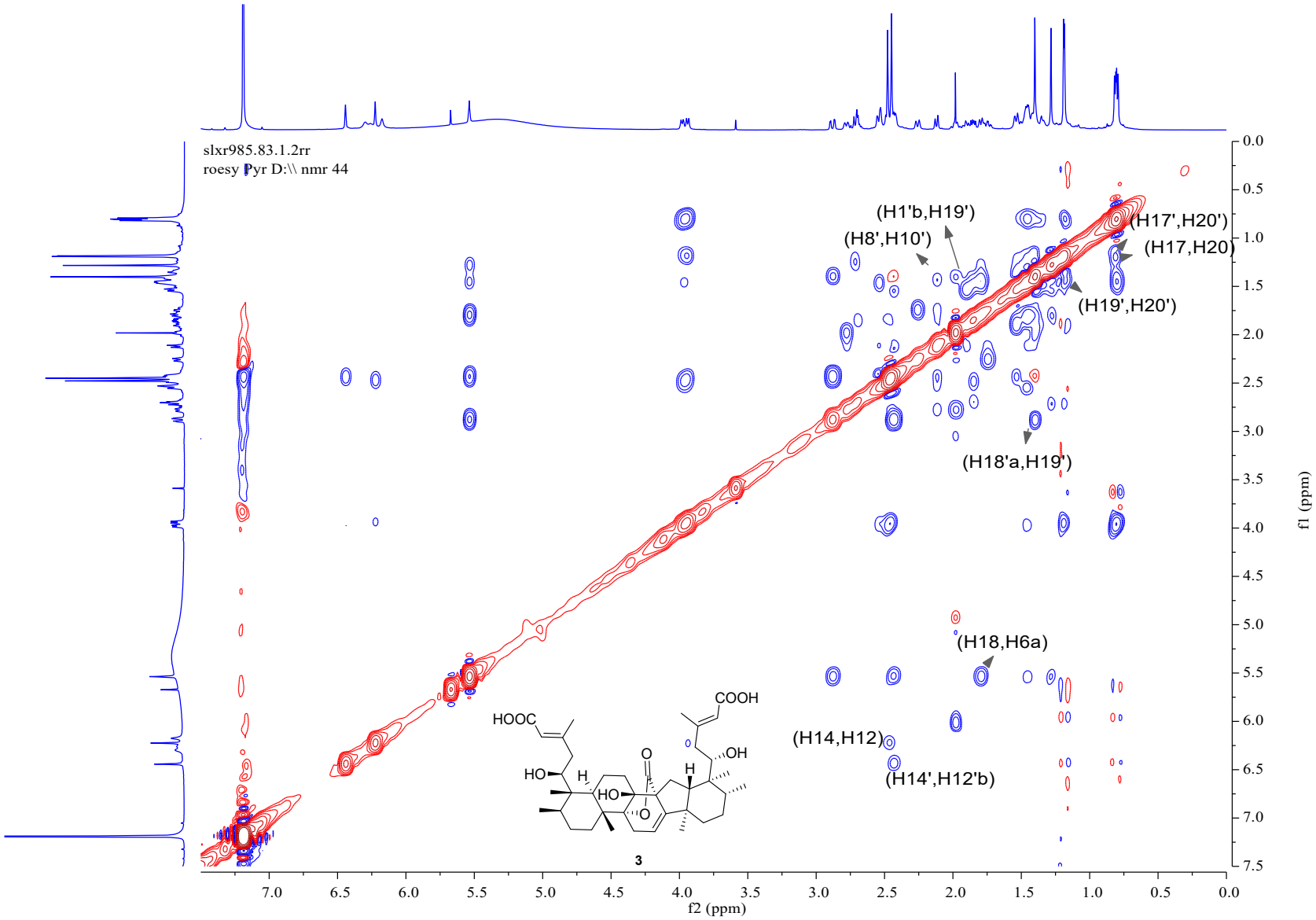


Figure S61. ROESY spectrum of synthetic scoparicacid C (**3**)

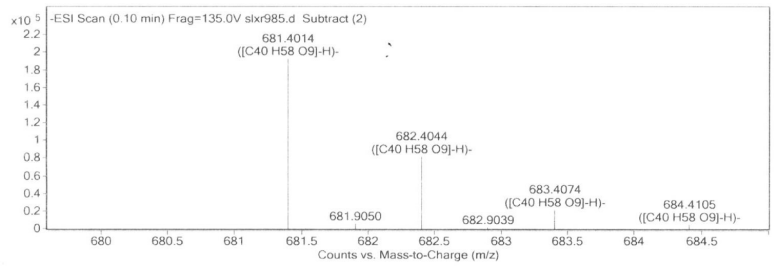
Qualitative Analysis Report

Data Filename	slxr985.d	**Sample Name**	slxr985
Sample Type	Sample	**Position**	P1-A2
Instrument Name	Instrument 1	**User Name**	
Acq Method	s-.m	**Acquired Time**	9/28/2021 11:43:05 AM
IRM Calibration Status	Success	**DA Method**	Default.m
Comment			

Sample Group	**Info.**
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)

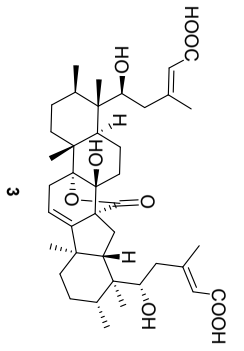
User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
135	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
96.9607		37253.62		
681.4014	1	192619.22	C40 H58 O9	(M-H)-
682.4044	1	81498.82	C40 H58 O9	(M-H)-
683.4074	1	21327.23	C40 H58 O9	(M-H)-
703.3831	1	24197.37		
727.4065	1	99632.31		
728.4096	1	46691.93		
779.3692	1	30536		
1363.8086	1	32705.24		
1364.8111	1	30148.32		



3

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C40 H58 O9	682.4081	681.4008	681.4014	-0.60	-0.88	12.0000

--- End Of Report ---

Figure S62. HRESIMS spectrum of synthetic scoparicacid C (**3**)

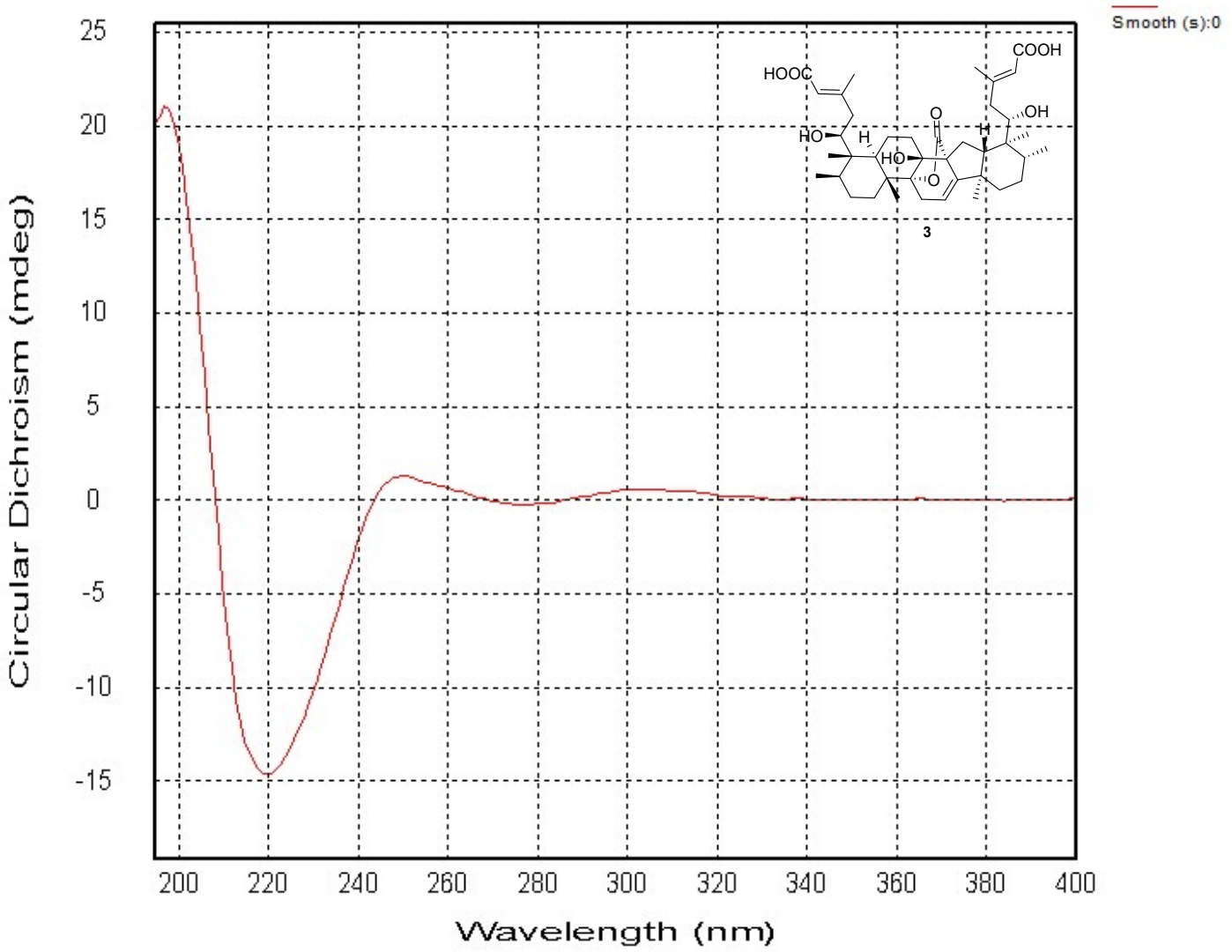
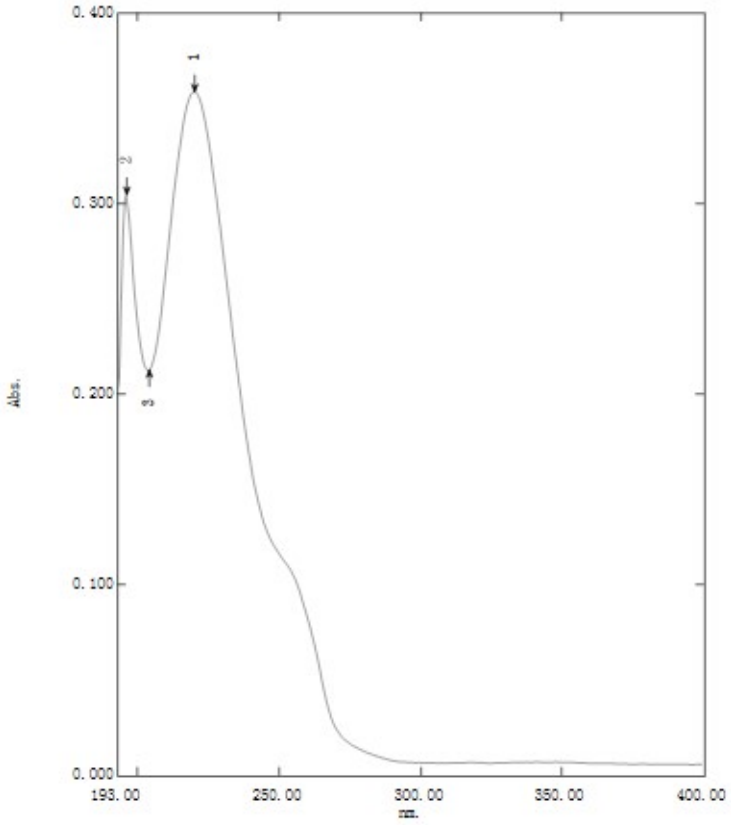


Figure S63. ECD spectrum of synthetic scoparicacid C (**3**)

光谱峰值检测报告

2021/10/08 17:34:03

数据集: SLXR985 - RawData



【测定属性】
波长范围(nm): 190.00 到 600.00
扫描速度: 中速
采样间隔: 0.5
自动采样间隔: 停用
扫描模式: 单个

【仪器属性】
仪器类型: UV-2700 系列
测定方式: 吸收值
狭缝宽: 5.0 nm
积分时间: 0.1 秒
光源转换波长: 323.0 nm
检测器单元: 高级
S/R 转换: 标准
阶梯校正: OFF

【附件属性】
附件: 无

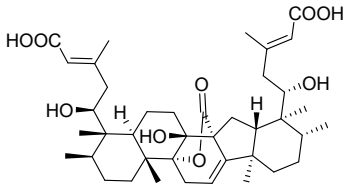
【数据处理参数】
阈值: 0.0010000
点: 4
内插: 停用
平均: 停用

【样品准备属性】
重量:
体积:
稀释:
光程长: 10mm
附加信息: 样品浓度: 0.0124毫克/毫升
溶剂: 甲醇

No.	P/V	波长(nm)	Abs.	描述
1	T	220.00	0.359	
2	↑	196.00	0.305	
3	↓	204.00	0.212	

Figure S64. UV spectrum of synthetic scoparicacid C (**3**)

Rudolph Research Analytical



3

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Thursday, 14-OCT-2021

Set Temperature : OFF

Time Delay : Disabled

Delay between Measurement : Disabled

n	Average	Std.Dev.	% RSD	Maximum	Minimum
5	-6.22	0.61	-9.80	-5.40	-7.00

S.No	Sample ID	Time	Result	Scale	OR °Arc	WLG.nm	Lg.mm	Conc.g/100ml	Temp.
1	slxr985	07:29:15 PM	-5.40	SR	-0.0054	589	100.00	0.100	22.7
2	slxr985	07:29:23 PM	-5.90	SR	-0.0059	589	100.00	0.100	22.7
3	slxr985	07:29:32 PM	-6.30	SR	-0.0063	589	100.00	0.100	22.7
4	slxr985	07:29:40 PM	-6.50	SR	-0.0065	589	100.00	0.100	22.7
5	slxr985	07:29:48 PM	-7.00	SR	-0.0070	589	100.00	0.100	22.7

Figure S65. OR spectrum of synthetic scoparicacid C (**3**)

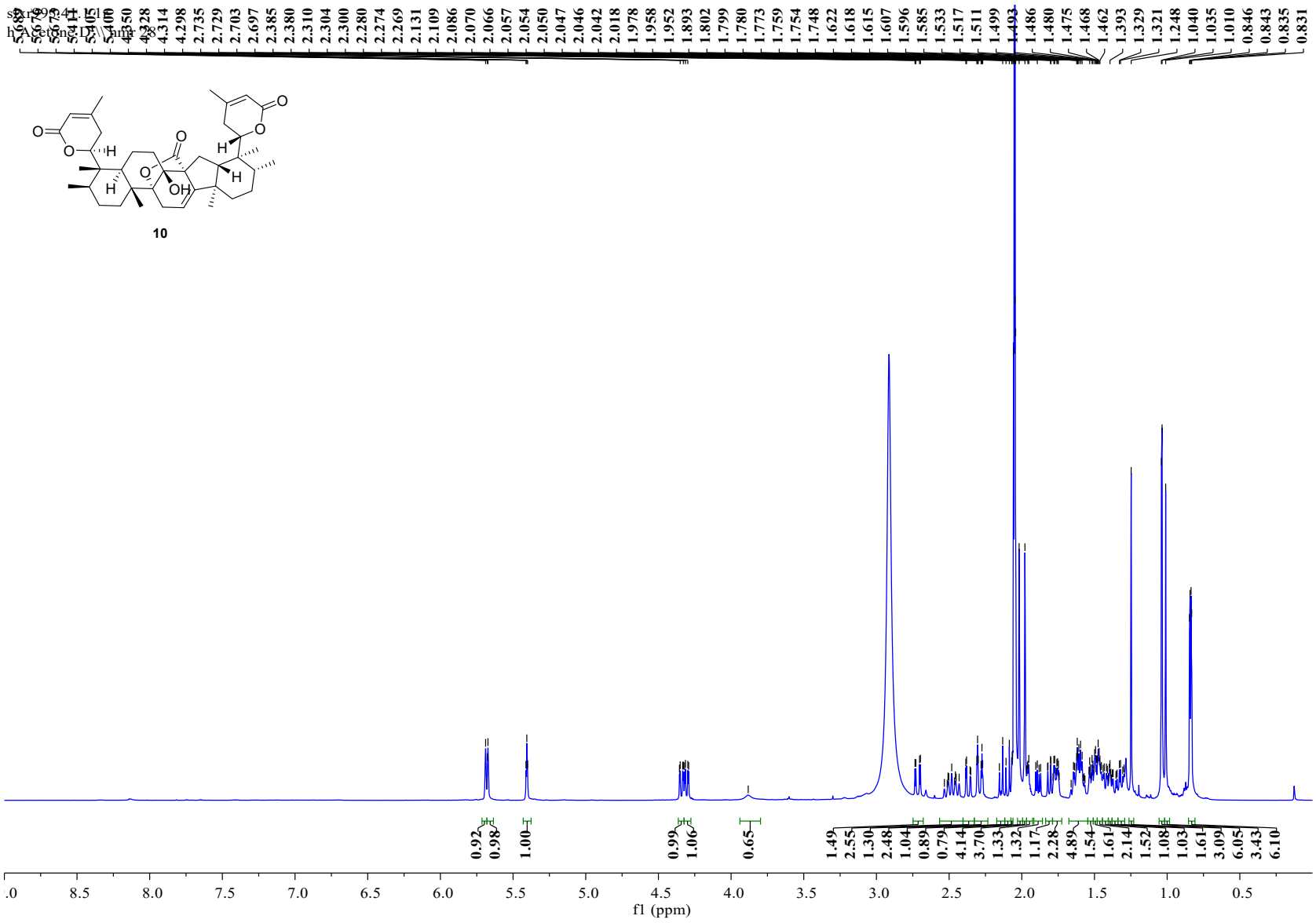


Figure S66. ^{1}H NMR spectrum of scoparitrilactone A (**10**)

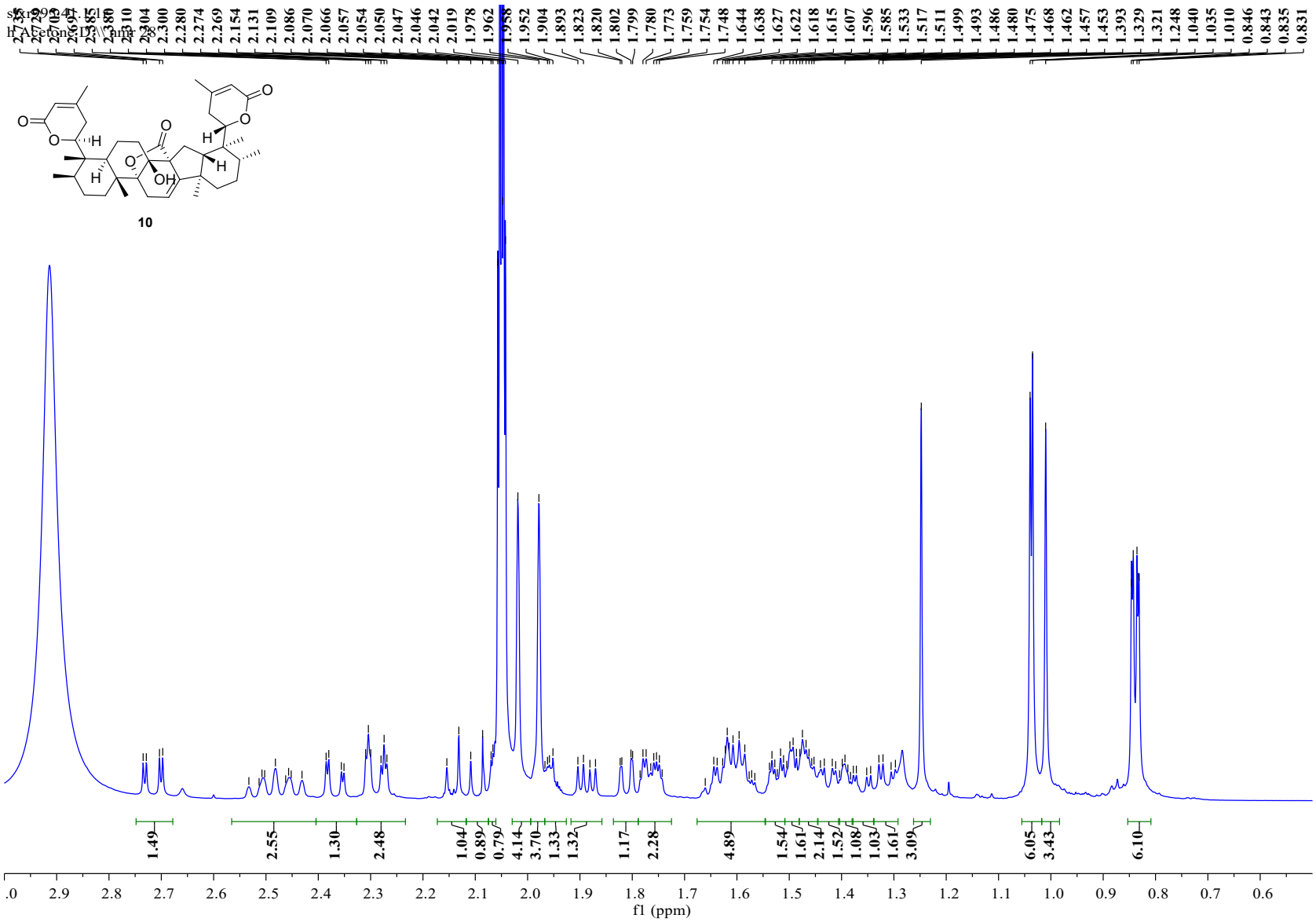


Figure S67. ^{1}H NMR spectrum of scoparitrilactone A (**10**)

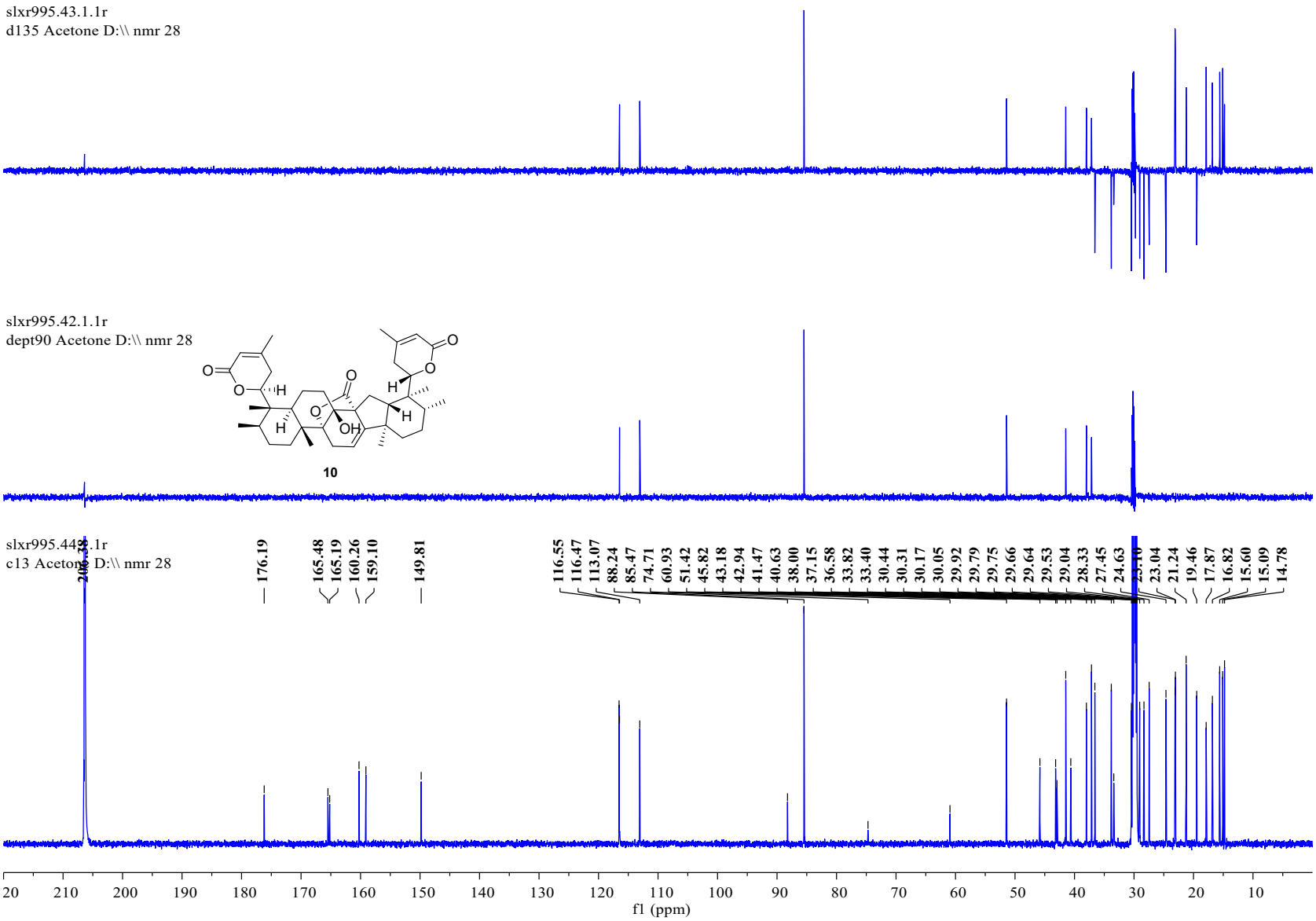


Figure S68. ^{13}C NMR spectrum of scoparitrilactone A (**10**)

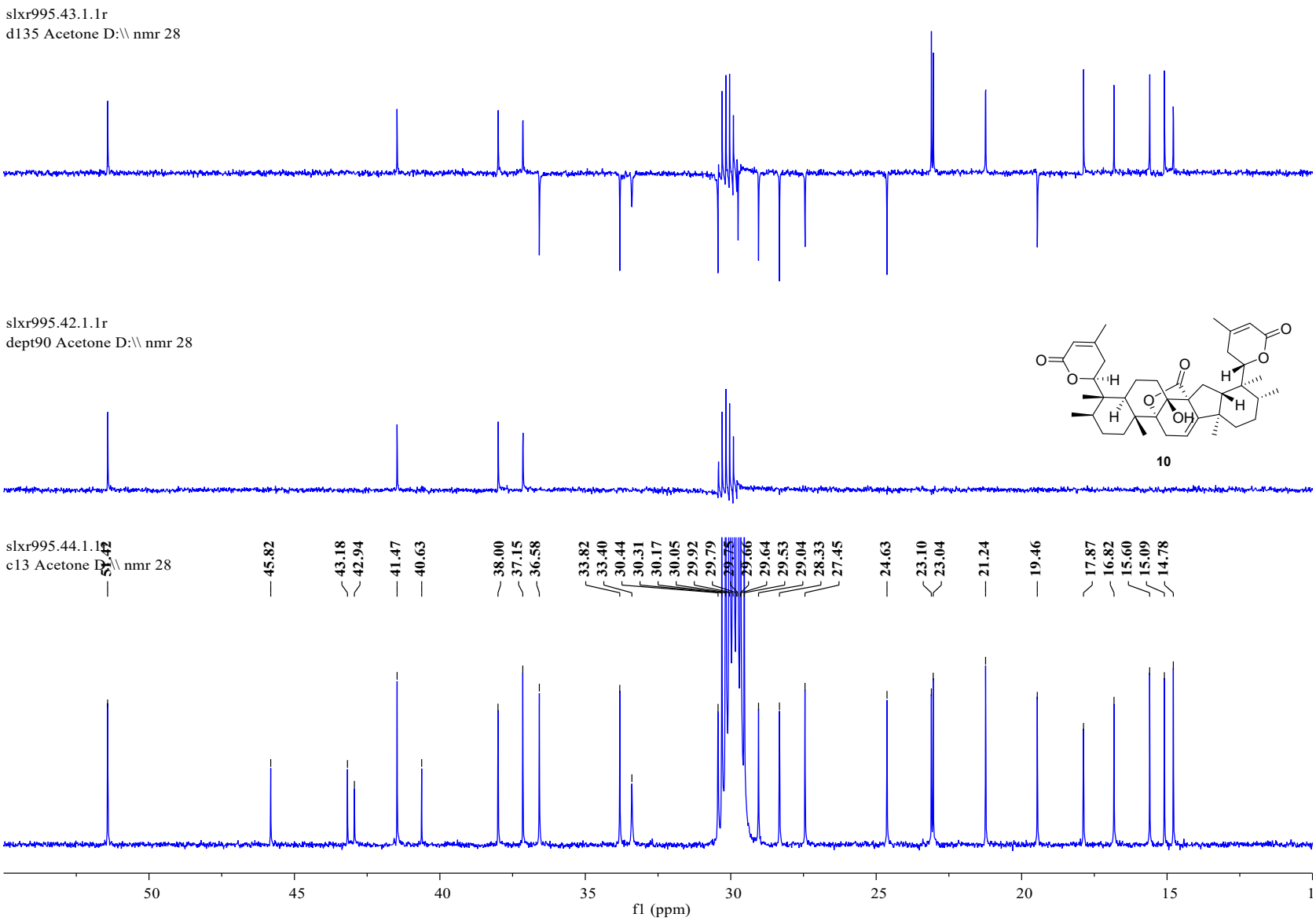


Figure S69. ^{13}C NMR spectrum of scoparitrilactone A (**10**)

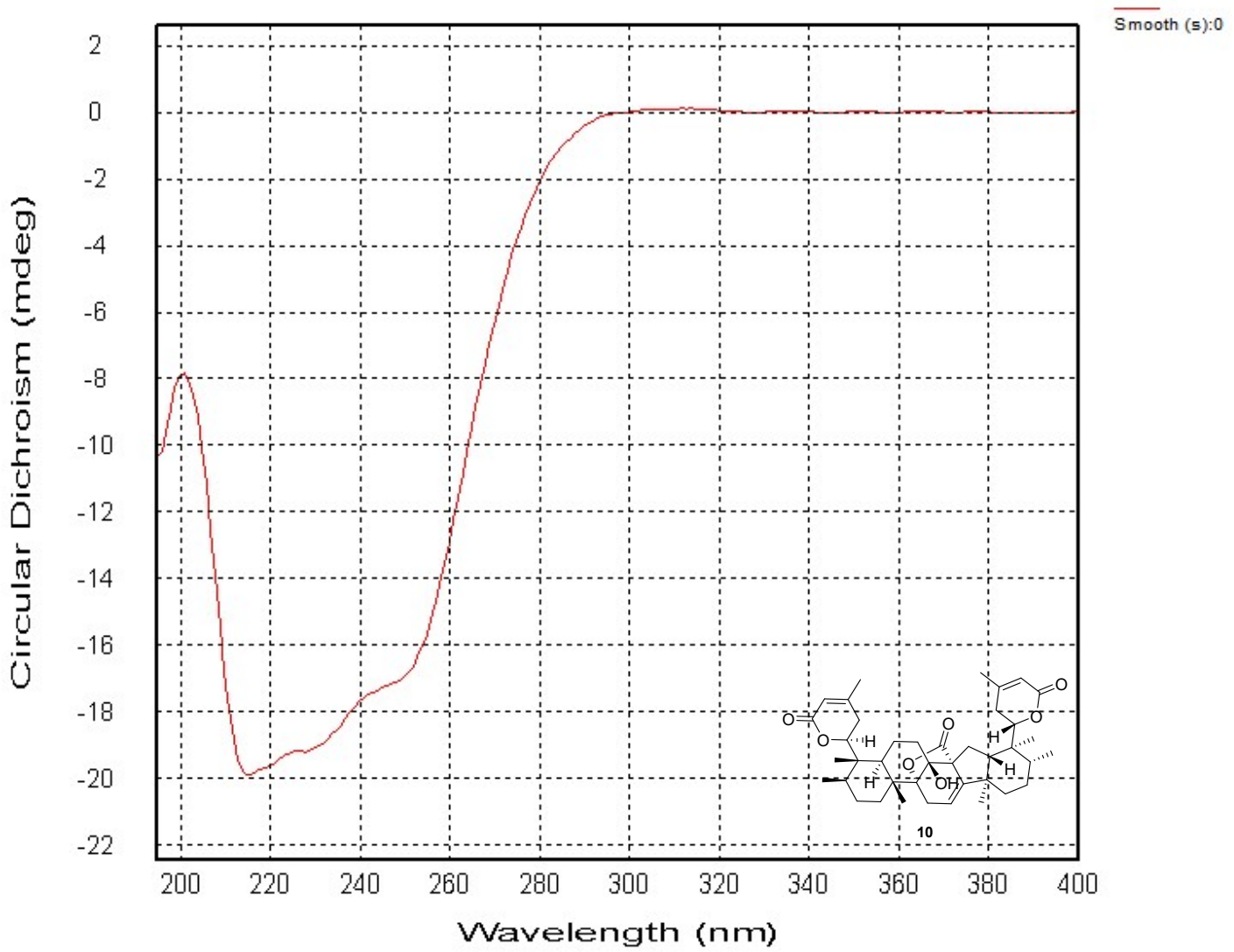


Figure S70. ECD spectrum of scoparitrilactone A (**10**)

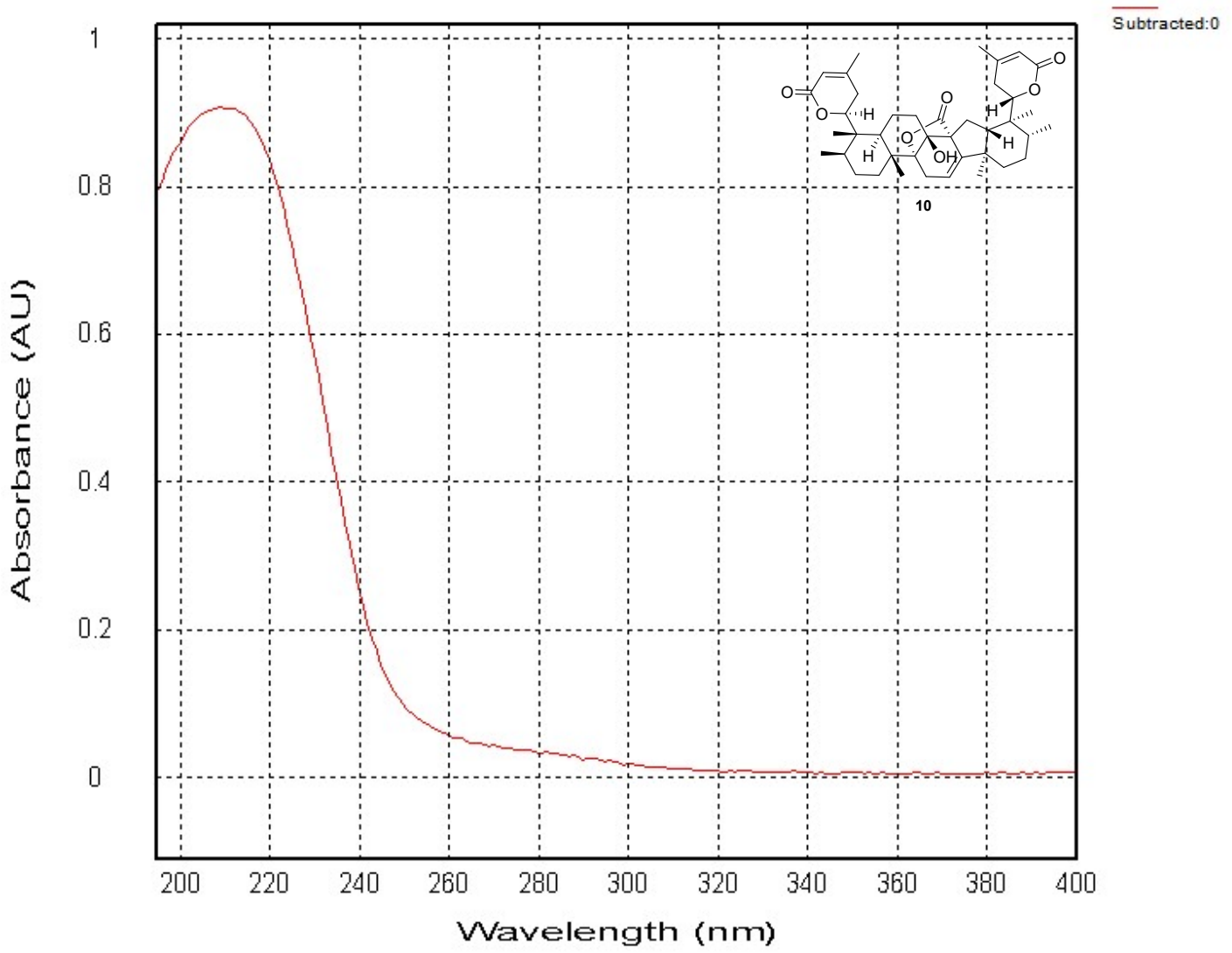


Figure S71. UV spectrum of scoparitrilactone A (**10**)

Qualitative Analysis Report

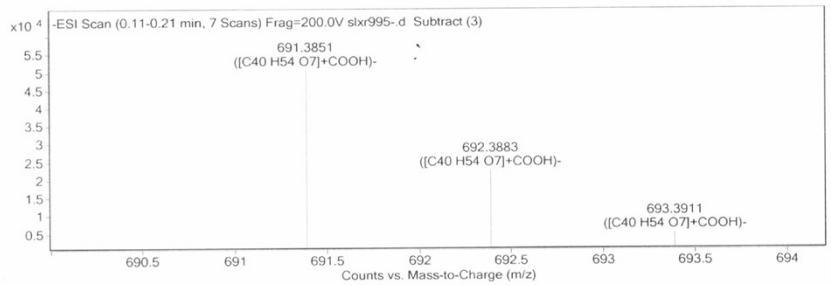
Data Filename	slxr995-.d	**Sample Name**	slxr995
Sample Type	Sample	**Position**	P1-B4
Instrument Name	Instrument 1	**User Name**	
Acq Method	s-.m	**Acquired Time**	10/18/2021 11:17:06 AM
IRM Calibration Status	Success	**DA Method**	PCDL.m
Comment			

Sample Group **Info.**

Acquisition SW Version 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
200	0	ESI



Peak List

m/z	z	Abund	Formula	Ion
645.3794	1	4669.85		
681.3561	1	13377.73		
682.3596	1	6030.29		
683.3559	1	5044.88		
691.3851	1	50798.97	C40 H54 O7	(M+COOH)-
692.3883	1	22685.91	C40 H54 O7	(M+COOH)-
693.3911	1	5285.53	C40 H54 O7	(M+COOH)-
708.375	1	22379.53		
709.3785	1	10273.8		
759.372	1	9078.3		

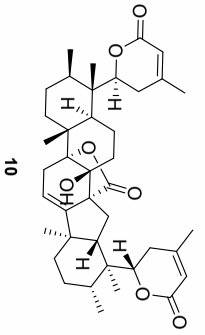
Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	40

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C40 H54 O7	646.3870	691.3852	691.3851	0.10	0.14	14.0000

10



--- End Of Report ---

Figure S72. HRESIMS spectrum of scoparitrilactone A (**10**)

Rudolph Research Analytical

This sample was measured on an Autopol VI, Serial #91058
Manufactured by Rudolph Research Analytical, Hackettstown, NJ, USA.

Measurement Date : Wednesday, 24-NOV-2021

Set Temperature : 20.0

Time Delay : Disabled

Delay between Measurement : Disabled

n	Average	Std.Dev.	% RSD	Maximum	Minimum
5	-116.60	0.89	-0.76	-116.00	-118.00

S.No	Sample ID	Time	Result	Scale	OR °Arc	WLG.nm	Lg.mm	Conc.g/100ml	Temp.
1	SLXR995	03:04:13 PM	-118.00	SR	-0.118	589	100.00	0.100	19.6
2	SLXR995	03:04:21 PM	-117.00	SR	-0.117	589	100.00	0.100	19.7
3	SLXR995	03:04:28 PM	-116.00	SR	-0.116	589	100.00	0.100	19.7
4	SLXR995	03:04:34 PM	-116.00	SR	-0.116	589	100.00	0.100	19.7
5	SLXR995	03:04:40 PM	-116.00	SR	-0.116	589	100.00	0.100	19.8

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Figure S73. OR spectrum of scoparitrilactone A (**10**)

5. Comparison of NMR spectra of synthetic and natural 3

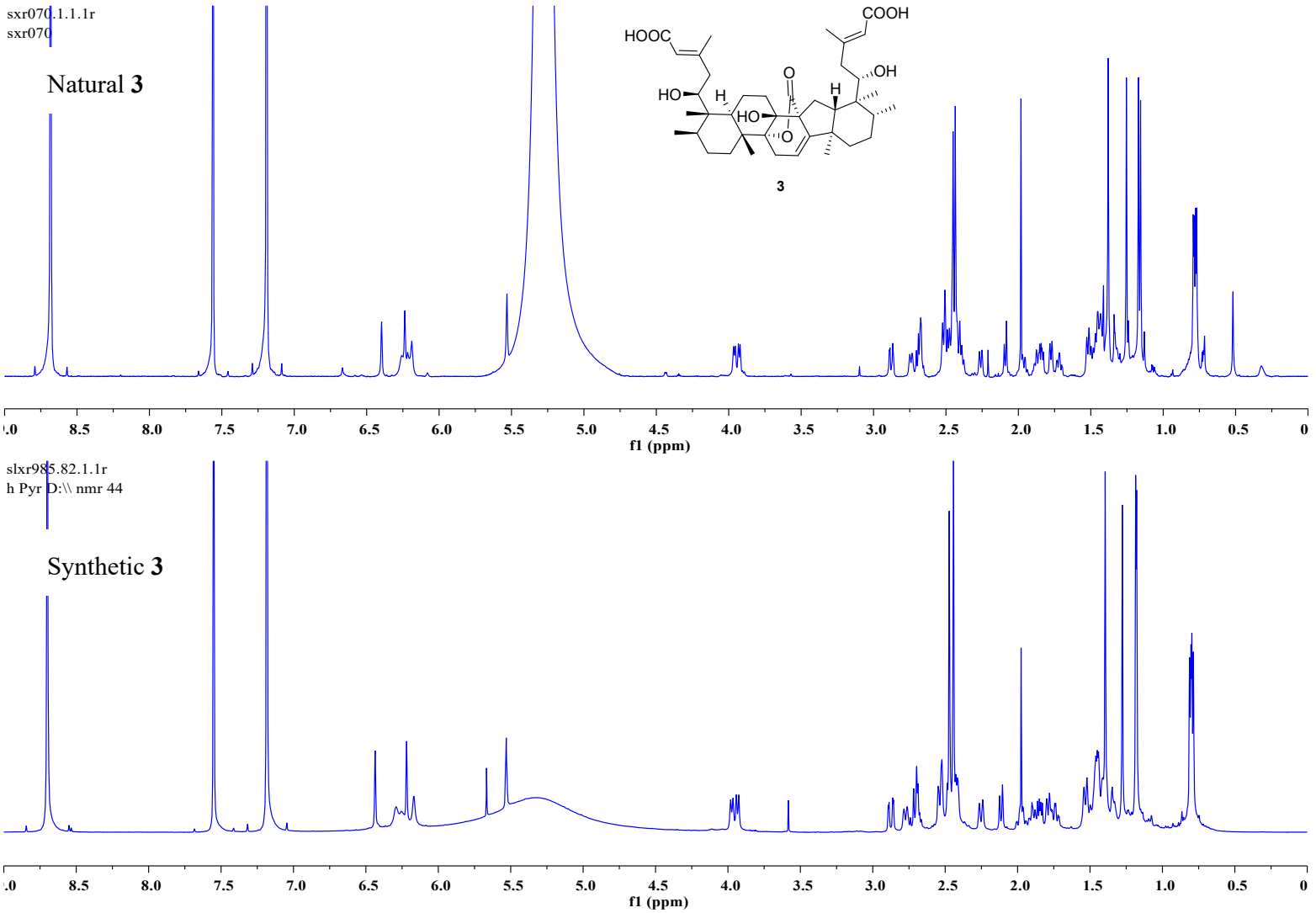

Figure S74. Comparison of ^{1}H NMR spectra of synthetic **3** and natural **3**

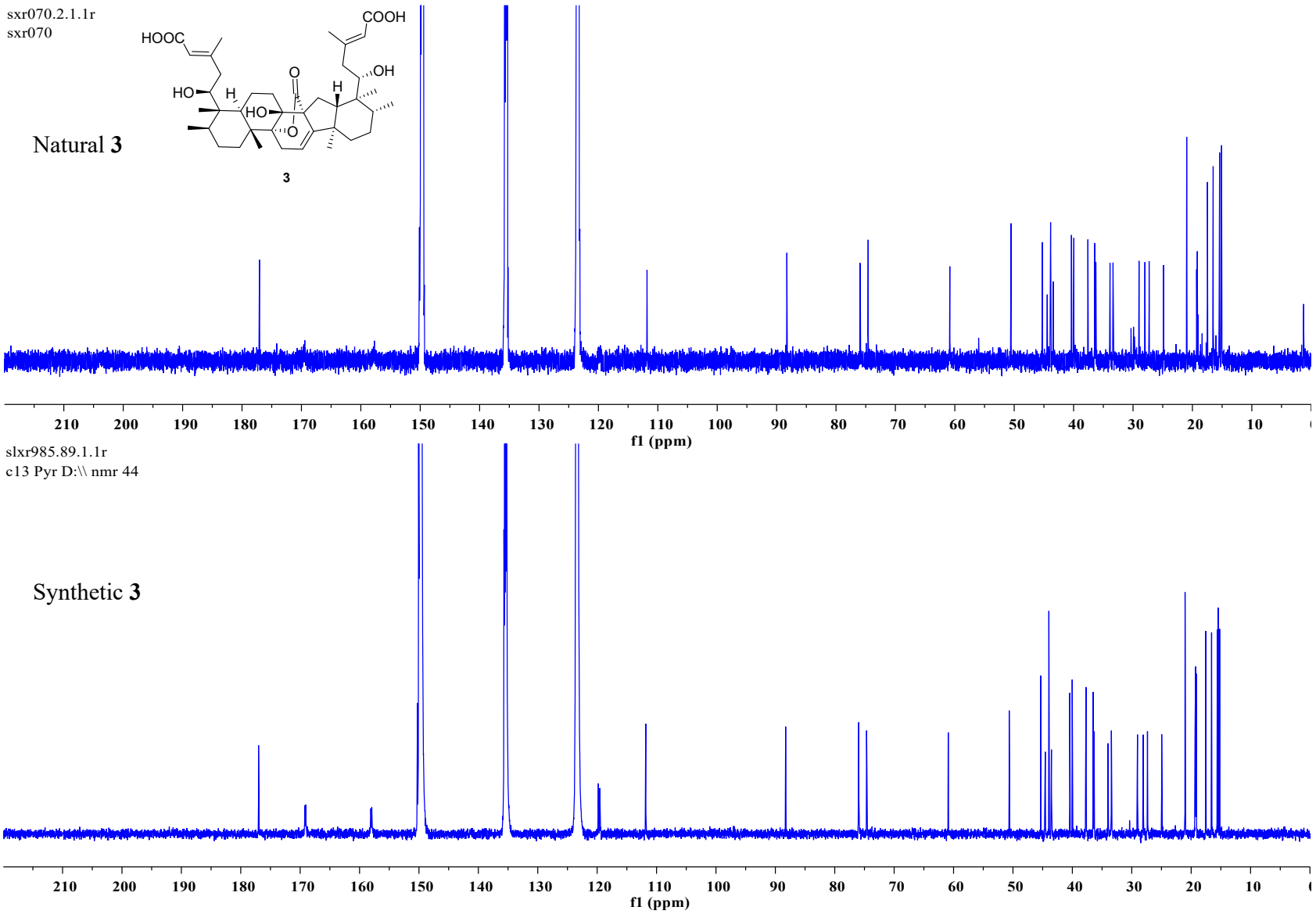

Figure S75. Comparison of ^{13}C NMR spectra of synthetic **3** and natural **3**