

**Cucurbalsaminones A–C, Rearranged Triterpenoids with a 5/6/3/6/5-Fused
Pentacyclic Carbon Skeleton from *Momordica balsamina* as Multidrug Resistance
Reversers**

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X-ray Crystallography

Table S1. Crystal data and structure refinement details for **1** (CCDC 1878164).

Formula	C ₂₇ H ₄₀ O ₃
Fw	412.59
Crystal form, color	Block, colourless
Crystal size (mm)	0.20×0.10×0.04
cryst. syst.	Orthorhombic
space group	<i>P</i> 2 ₁ 2 ₁ 2 ₁
<i>a</i> , Å	6.1959(4)
<i>b</i> , Å	14.0177(8)
<i>c</i> , Å	26.2372(16)
<i>Z</i>	4
<i>V</i> , Å ³	2278.8(2)
<i>T</i> , K	150(2)
<i>D</i> _c , g cm ⁻³	1.203
μ(Mo Kα), mm ⁻¹	0.076
θ range (°)	2.745–26.436
refl. Collected	26968
independent refl.	4682
<i>R</i> _{int}	0.0813
<i>R</i> ₁ ^a , <i>wR</i> ₂ ^b [<i>I</i> ≥ 2σ(<i>I</i>)]	0.0496, 0.1140
GOF on <i>F</i> ²	1.013

$$^a R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}, ^b wR_2 = \left[\frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right]^{1/2}$$

Table S2. Bond lengths [Å] and angles [°] for compound **1**.

O(2)-C(7)	1.214(4)	C(12)-C(13)-C(18)	110.1(2)	C(3)-C(5)-C(4)	119.9(3)
O(3)-C(23)	1.194(4)	C(12)-C(13)-C(17)	117.5(2)	C(6)-C(5)-C(4)	59.2(2)
C(13)-C(12)	1.524(4)	C(18)-C(13)-C(17)	108.1(2)	C(10)-C(5)-C(4)	123.3(3)
C(13)-C(18)	1.544(4)	C(12)-C(13)-C(14)	107.1(2)	C(19)-C(9)-C(11)	106.8(2)
C(13)-C(17)	1.558(4)	C(18)-C(13)-C(14)	112.2(2)	C(19)-C(9)-C(10)	108.6(2)
C(13)-C(14)	1.571(4)	C(17)-C(13)-C(14)	101.6(2)	C(11)-C(9)-C(10)	109.7(3)
C(8)-C(7)	1.532(4)	C(7)-C(8)-C(14)	111.6(2)	C(19)-C(9)-C(8)	106.9(3)
C(8)-C(14)	1.560(4)	C(7)-C(8)-C(9)	114.5(2)	C(11)-C(9)-C(8)	113.2(2)
C(8)-C(9)	1.584(4)	C(14)-C(8)-C(9)	117.1(2)	C(10)-C(9)-C(8)	111.5(2)
C(17)-C(20)	1.534(4)	C(20)-C(17)-C(13)	119.5(2)	O(3)-C(23)-C(24)	121.5(3)
C(17)-C(16)	1.571(4)	C(20)-C(17)-C(16)	112.0(2)	O(3)-C(23)-C(22)	121.8(3)
C(20)-C(21)	1.521(4)	C(13)-C(17)-C(16)	102.7(2)	C(24)-C(23)-C(22)	116.6(3)
C(20)-C(22)	1.535(4)	C(21)-C(20)-C(17)	114.3(2)	C(15)-C(16)-C(17)	107.5(2)
C(7)-C(6)	1.493(4)	C(21)-C(20)-C(22)	108.5(3)	C(2)-C(1)-C(10)	107.6(3)
C(10)-C(5)	1.522(4)	C(17)-C(20)-C(22)	108.3(2)	C(14)-C(15)-C(16)	103.9(2)
C(10)-C(1)	1.558(4)	O(2)-C(7)-C(6)	118.2(3)	C(12)-C(11)-C(9)	118.9(2)
C(10)-C(9)	1.565(4)	O(2)-C(7)-C(8)	120.8(3)	C(23)-C(22)-C(20)	114.6(3)
C(12)-C(11)	1.528(4)	C(6)-C(7)-C(8)	120.8(3)	C(3)-C(2)-C(1)	106.1(3)
C(6)-C(5)	1.506(4)	C(5)-C(10)-C(1)	102.9(2)	O(1)-C(3)-C(5)	126.1(3)
C(6)-C(4)	1.508(4)	C(5)-C(10)-C(9)	110.9(3)	O(1)-C(3)-C(2)	125.1(3)
C(14)-C(30)	1.536(4)	C(1)-C(10)-C(9)	115.3(2)	C(5)-C(3)-C(2)	108.8(3)
C(14)-C(15)	1.537(4)	C(13)-C(12)-C(11)	110.9(3)	C(29)-C(4)-C(6)	122.8(3)
C(5)-C(3)	1.486(4)	C(7)-C(6)-C(5)	118.4(3)	C(29)-C(4)-C(28)	111.8(3)
C(5)-C(4)	1.548(4)	C(7)-C(6)-C(4)	124.3(3)	C(6)-C(4)-C(28)	115.1(3)
C(9)-C(19)	1.543(4)	C(5)-C(6)-C(4)	61.8(2)	C(29)-C(4)-C(5)	120.2(3)
C(9)-C(11)	1.557(4)	C(30)-C(14)-C(15)	106.7(2)	C(6)-C(4)-C(5)	59.0(2)
C(23)-C(24)	1.494(5)	C(30)-C(14)-C(8)	110.2(2)	C(28)-C(4)-C(5)	118.7(3)
C(23)-C(22)	1.504(5)	C(15)-C(14)-C(8)	116.0(2)		
C(16)-C(15)	1.543(5)	C(30)-C(14)-C(13)	112.8(2)		
C(1)-C(2)	1.537(5)	C(15)-C(14)-C(13)	101.2(2)		
O(1)-C(3)	1.211(4)	C(8)-C(14)-C(13)	109.9(2)		
C(2)-C(3)	1.501(5)	C(3)-C(5)-C(6)	120.2(3)		
C(28)-C(4)	1.519(4)	C(3)-C(5)-C(10)	108.9(3)		
C(29)-C(4)	1.507(5)	C(6)-C(5)-C(10)	117.9(2)		

Table S3. Antiproliferative activity of compounds **1 - 3** on *ABCB1*-transfected mouse T-lymphoma (L5178Y) cells.

compound	PAR^a ID₅₀ (μM)	MDR^a ID₅₀ (μM)
cucurbalsaminone A (1)	72.2 ± 1.3	68.3 ± 2.1
cucurbalsaminone B (2)	> 100	> 100
cucurbalsaminone C (3)	20.7 ± 2.7	37.6 ± 3.9

^a Values represent the mean ± SD of three independent experiments

HRMS, IR and NMR spectra of compounds 1-3

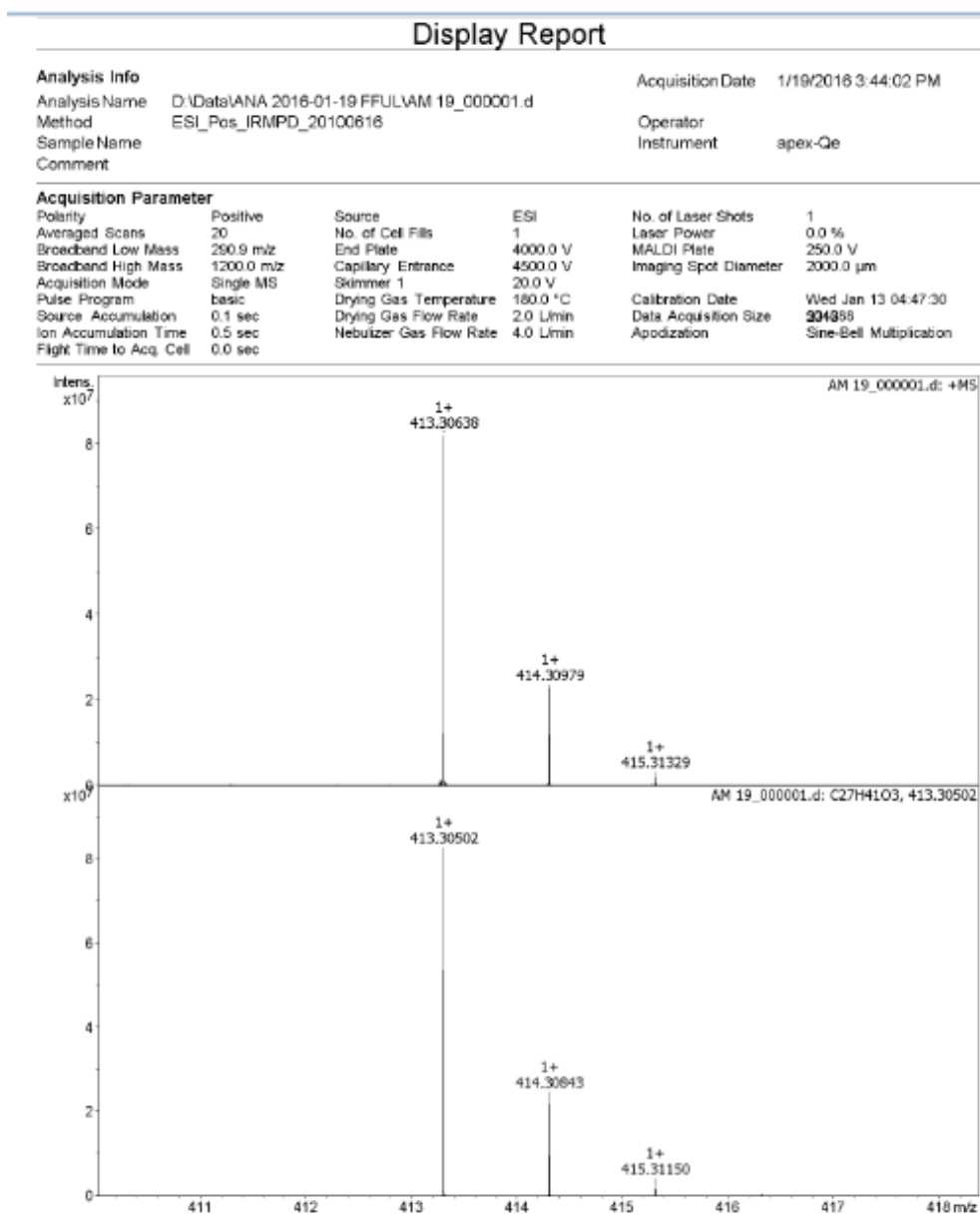


Figure S1. HRMS of compound 1.

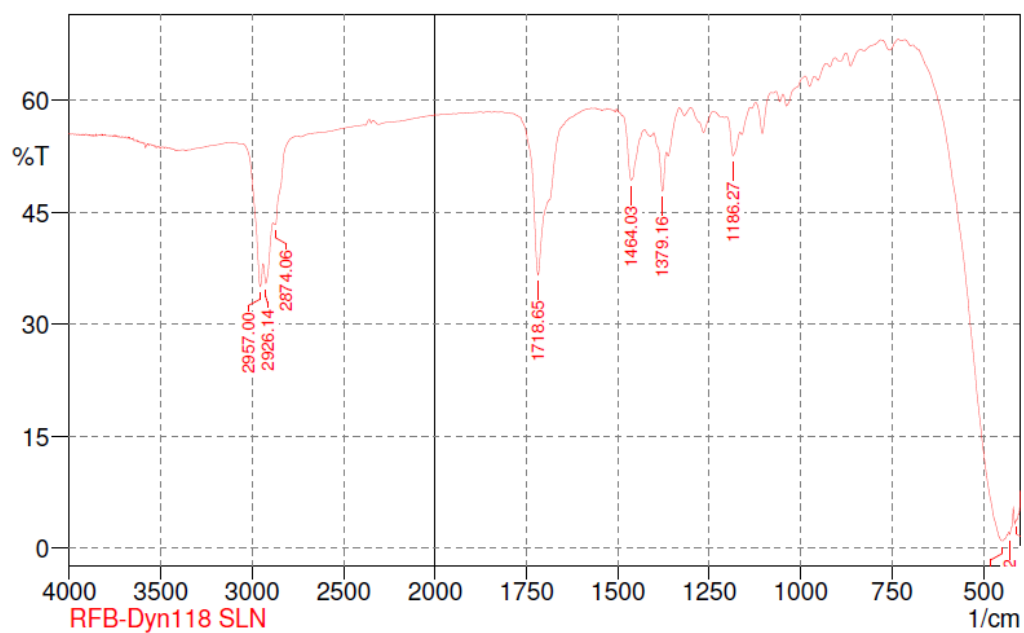


Figure S2. IR of compound **1**.

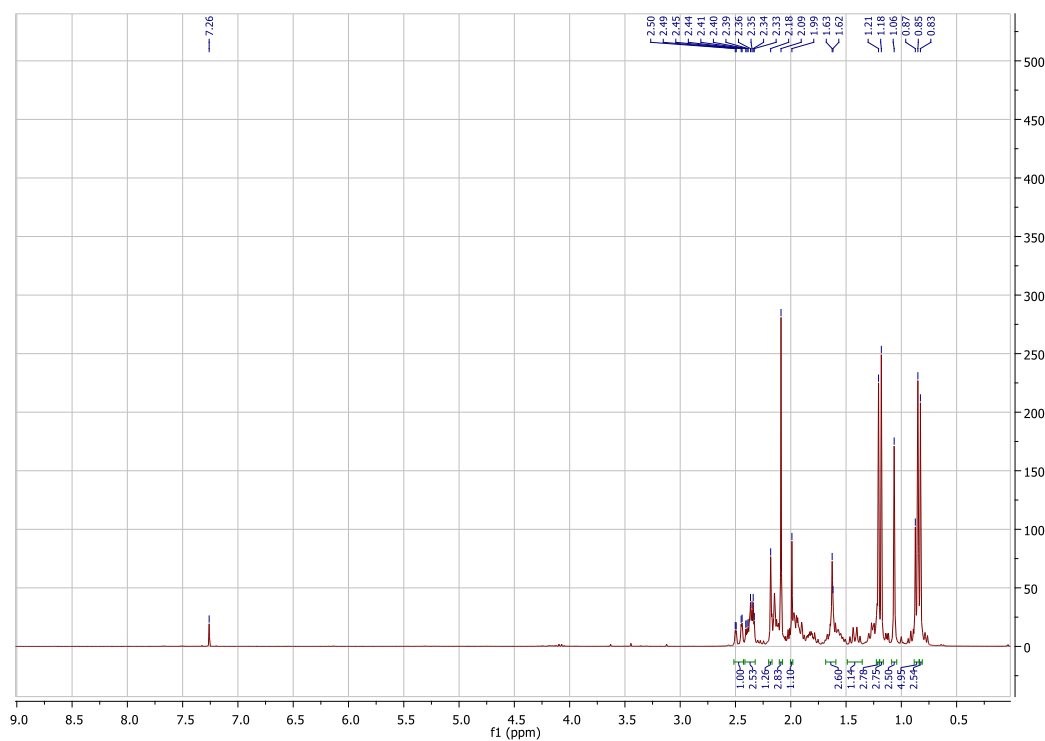


Figure S3. $^1\text{H-NMR}$ spectrum of compound **1**, in CDCl_3 (300 MHz).

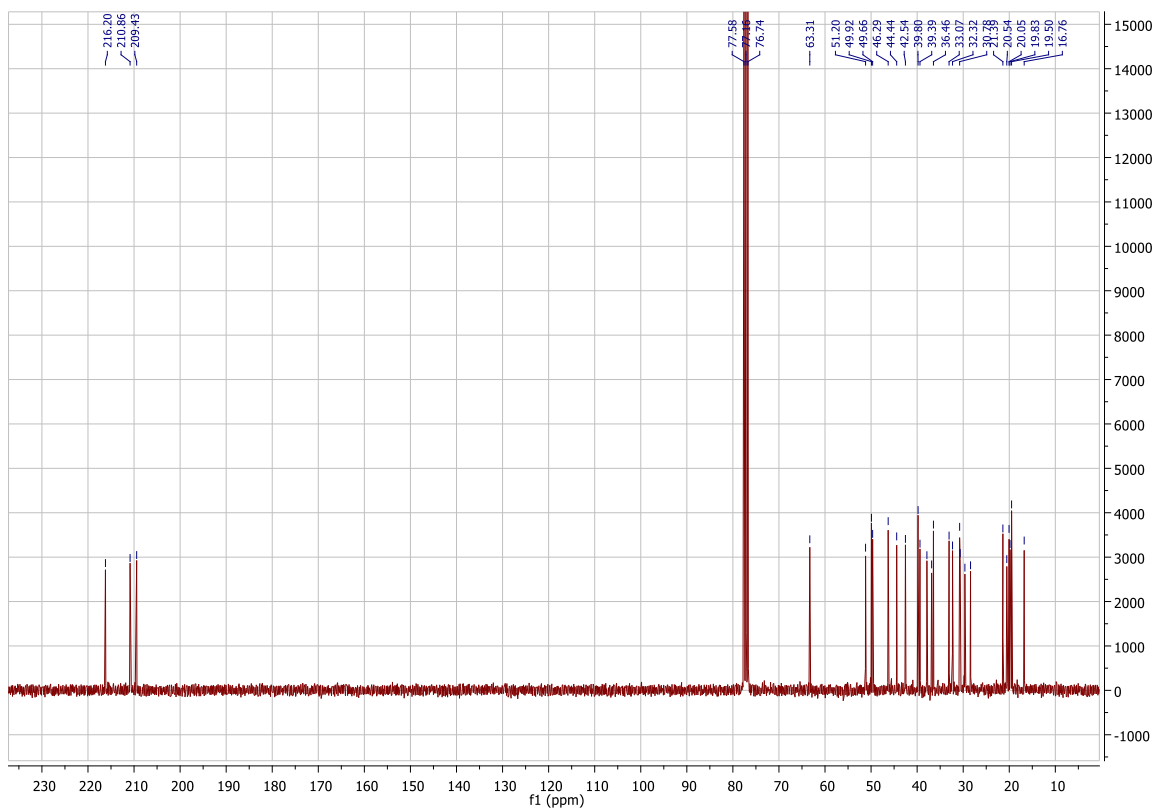


Figure S4. ^{13}C -NMR spectrum of compound **1**, in CDCl_3 (75 MHz).

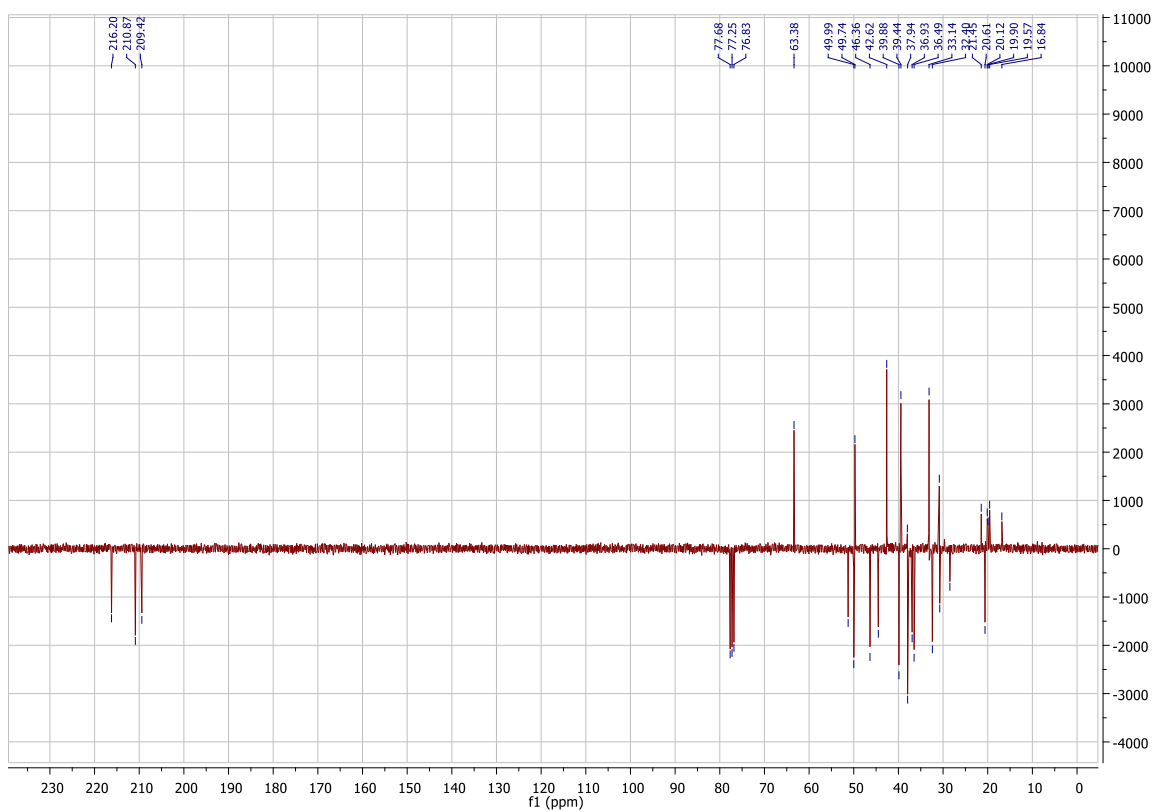


Figure S5. DEPT spectrum of compound **1** in CDCl_3 (75 MHz).

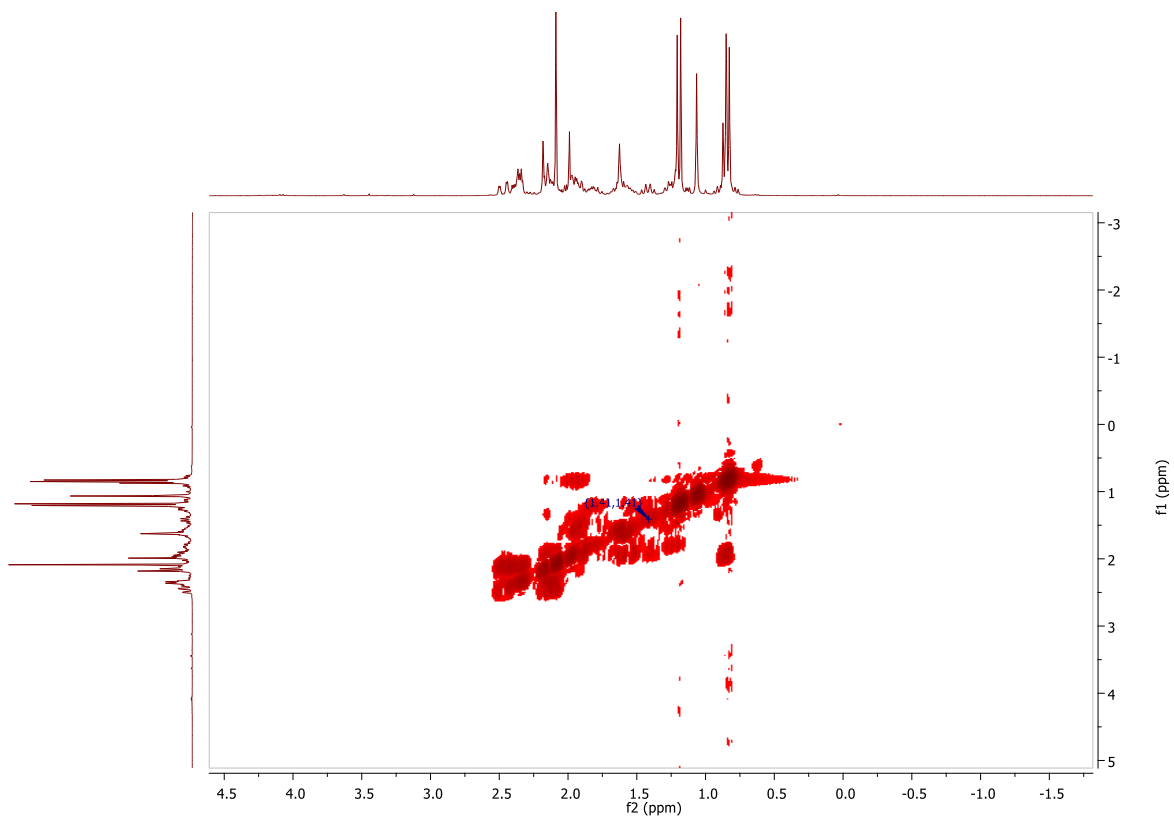


Figure S6. COSY spectrum of compound **1** in CDCl_3 .

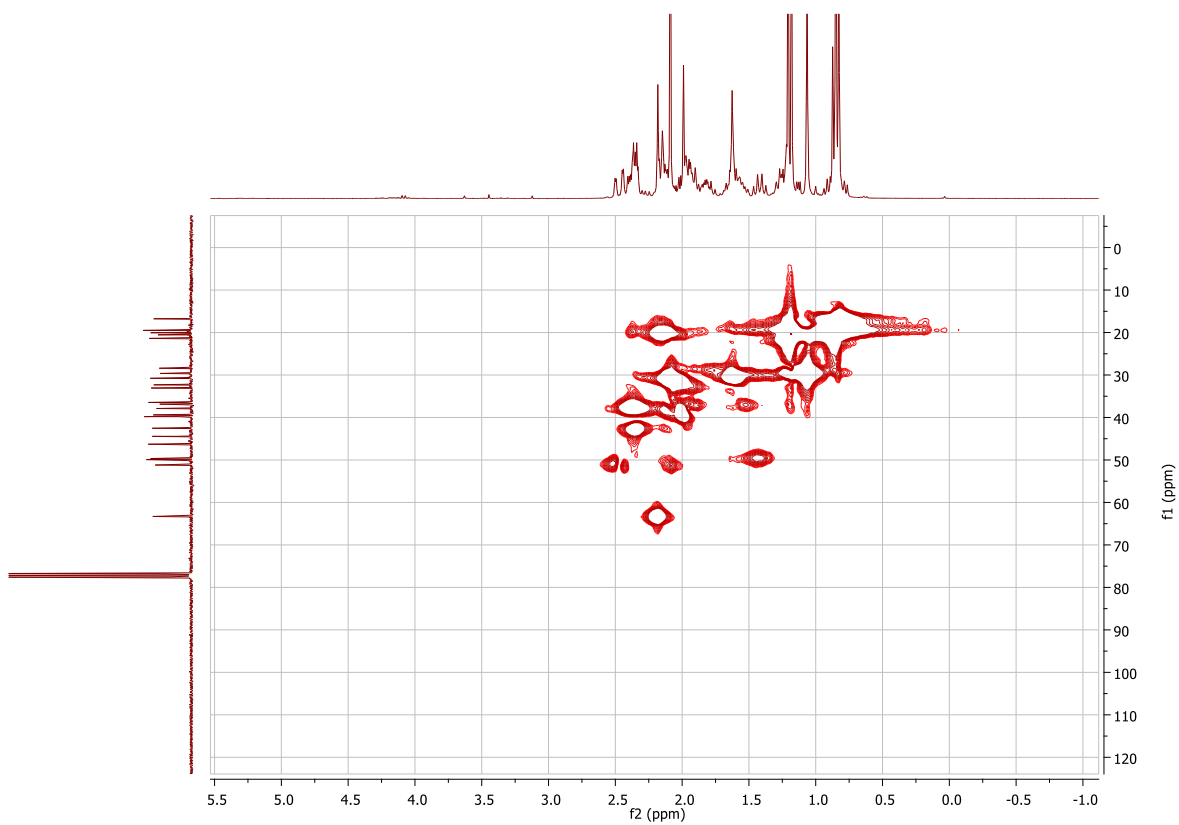


Figure S7. HMQC spectrum of compound **1** in CDCl_3 .

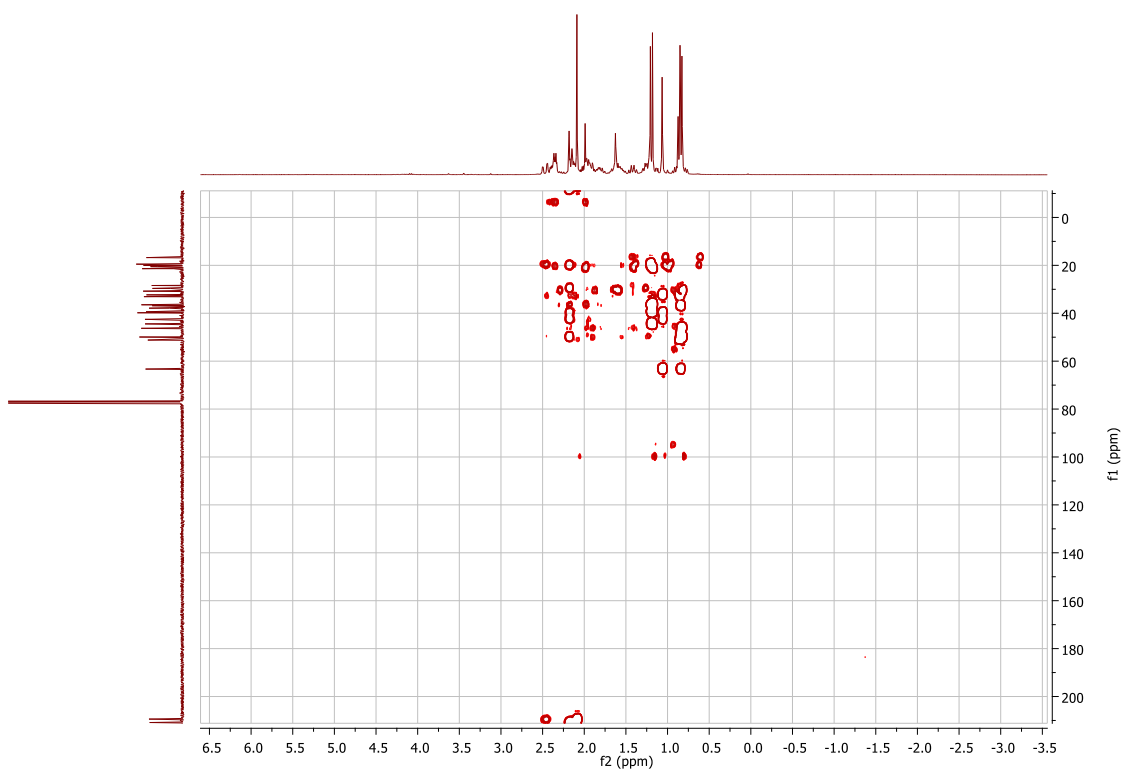


Figure S8. HMBC spectrum of compound **1** in CDCl_3

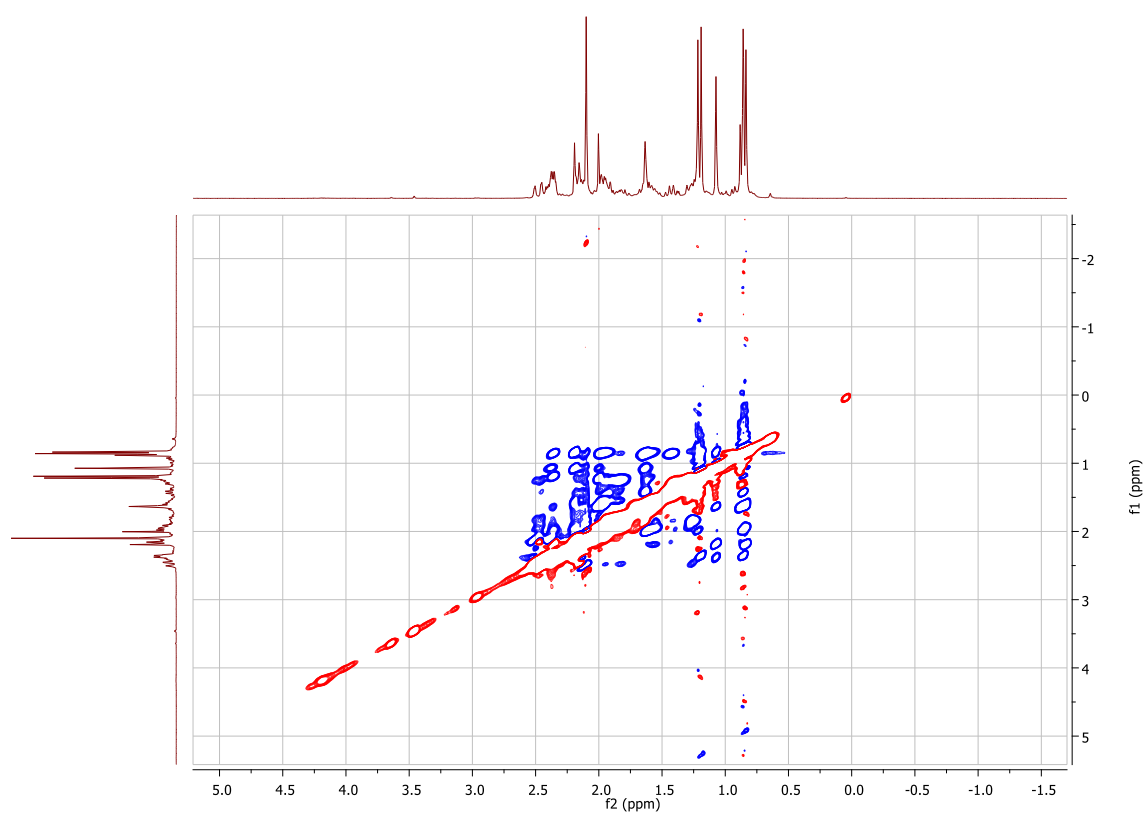
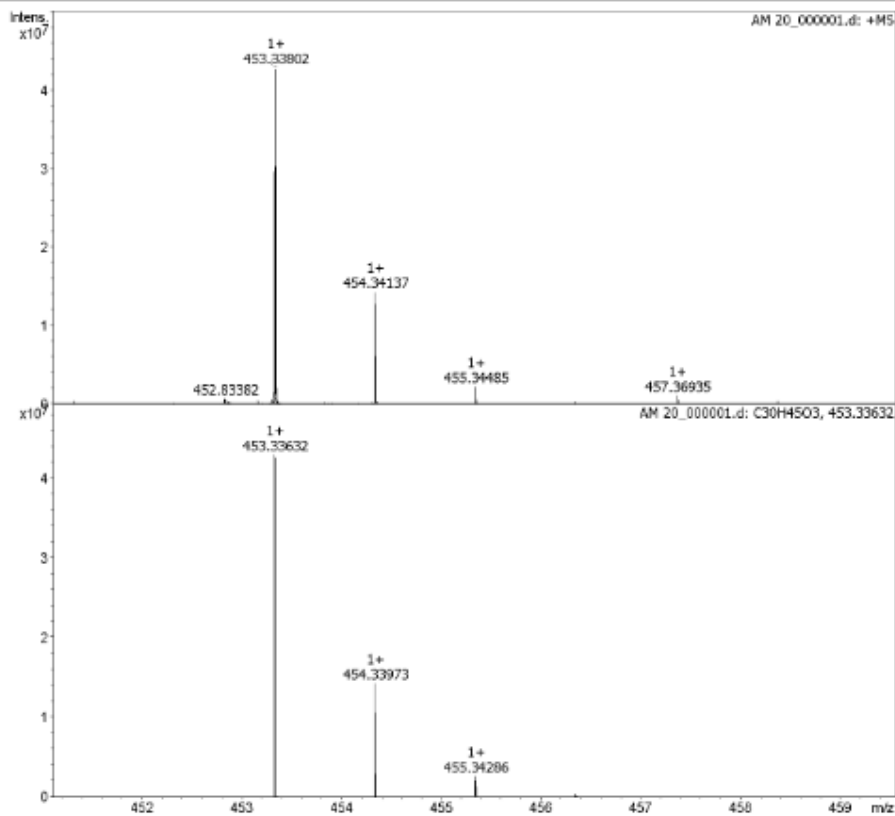


Figure S9. NOESY spectrum of compound **1** in CDCl_3 .

Analysis Info	Acquisition Date	1/19/2016 3:34:37 PM	
Analysis Name	D:\Data\ANA 2016-01-19\FFULVAM 20_000001.d		
Method	ESI_Pos_IRMPD_20100616	Operator	
Sample Name		Instrument	apex-Qe
Comment			

Acquisition Parameter					
Polarity	Positive	Source	ESI	No. of Laser Shots	1
Averaged Scans	20	No. of Cell Fills	1	Laser Power	0.0 %
Broadband Low Mass	250.9 m/z	End Plate	4000.0 V	MALDI Plate	250.0 V
Broadband High Mass	1200.0 m/z	Capillary Entrance	4500.0 V	Imaging Spot Diameter	2000.0 μm
Acquisition Mode	Single MS	Skimmer 1	20.0 V		
Pulse Program	basic	Drying Gas Temperature	180.0 °C	Calibration Date	Wed Jan 13 04:47:30
Source Accumulation	0.1 sec	Drying Gas Flow Rate	2.0 L/min	Data Acquisition Size	304888
Ion Accumulation Time	0.5 sec	Nebulizer Gas Flow Rate	4.0 L/min	Apodization	Sine-Bell Multiplication
Flight Time to Acq. Cell	0.0 sec				



Bruker Compass DataAnalysis 4.1 printed: 1/19/2016 3:38:52 PM Page 1 of 1

Figure S10. HRMS of compound 2.

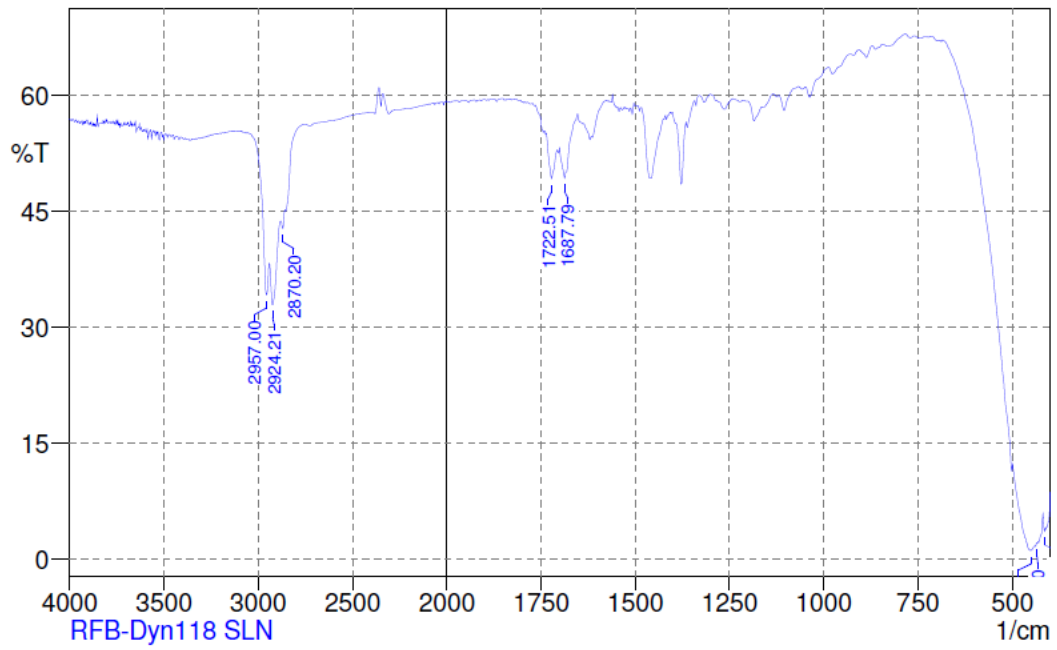


Figure S11. IR of compound 2.

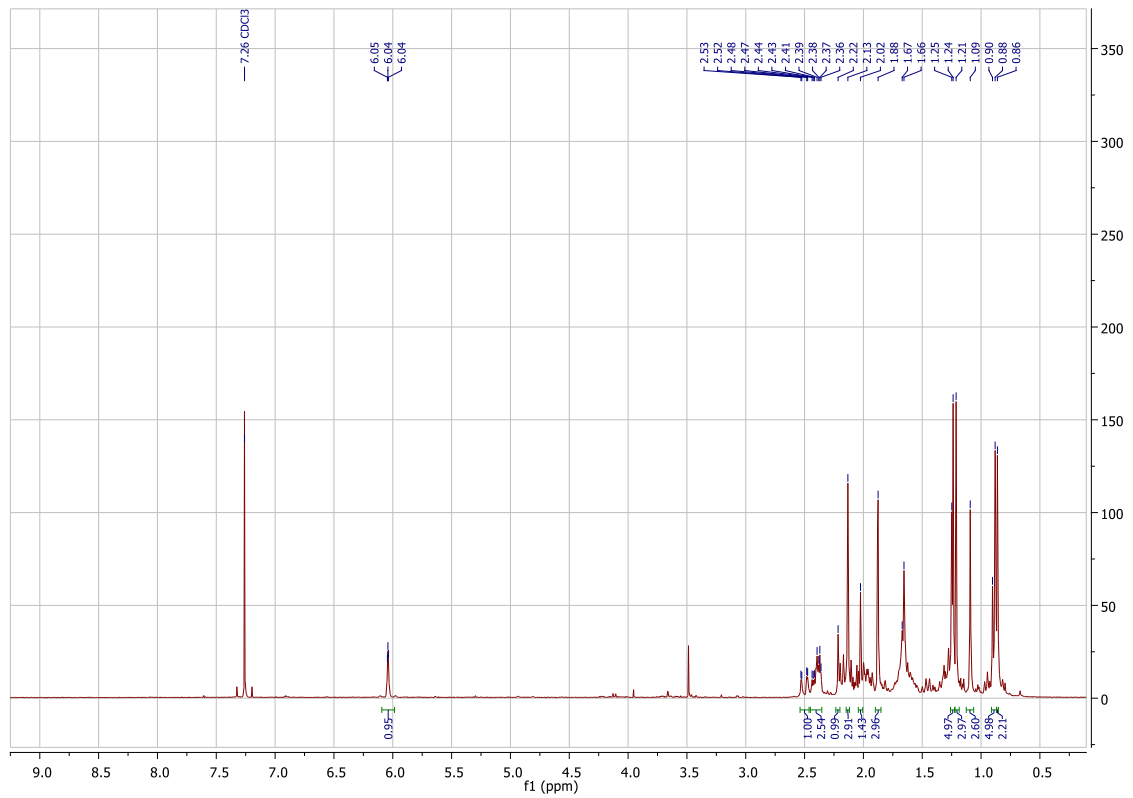


Figure S12. $^1\text{H-NMR}$ spectrum of compound 2 in CDCl_3 (300 MHz)..

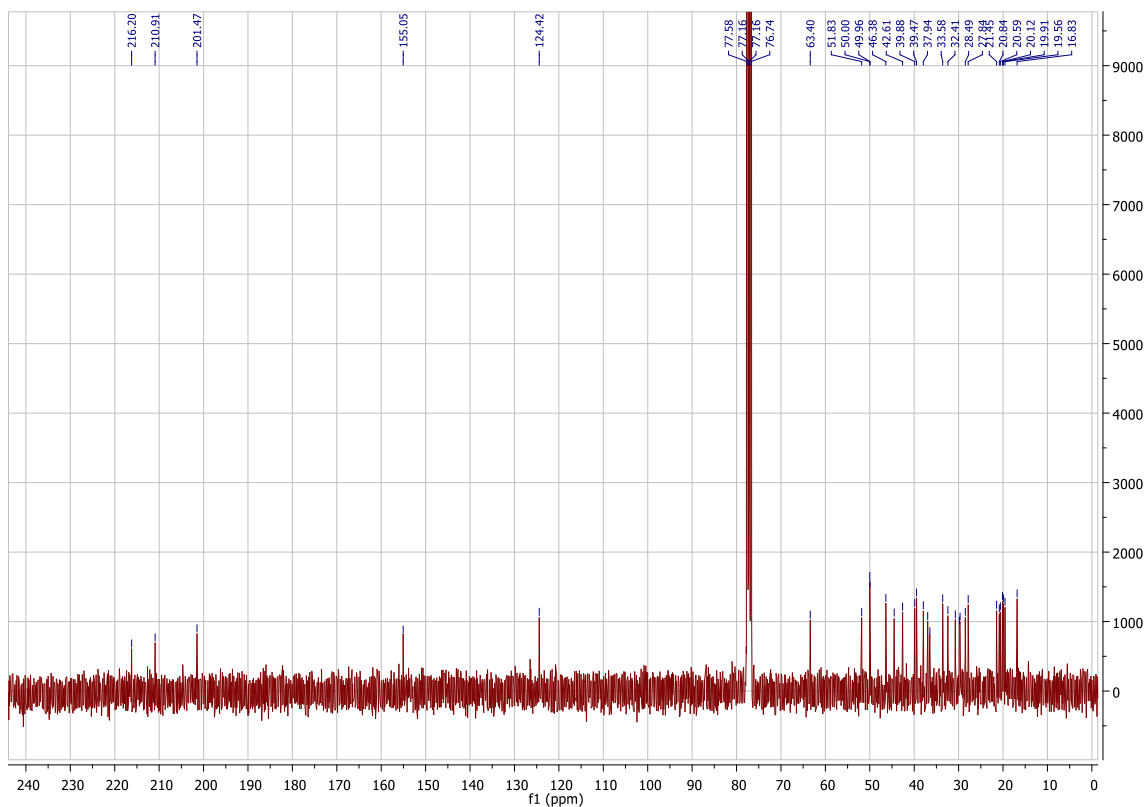


Figure S13. ^{13}C -NMR spectrum of compound **2** in CDCl_3 (75 MHz).

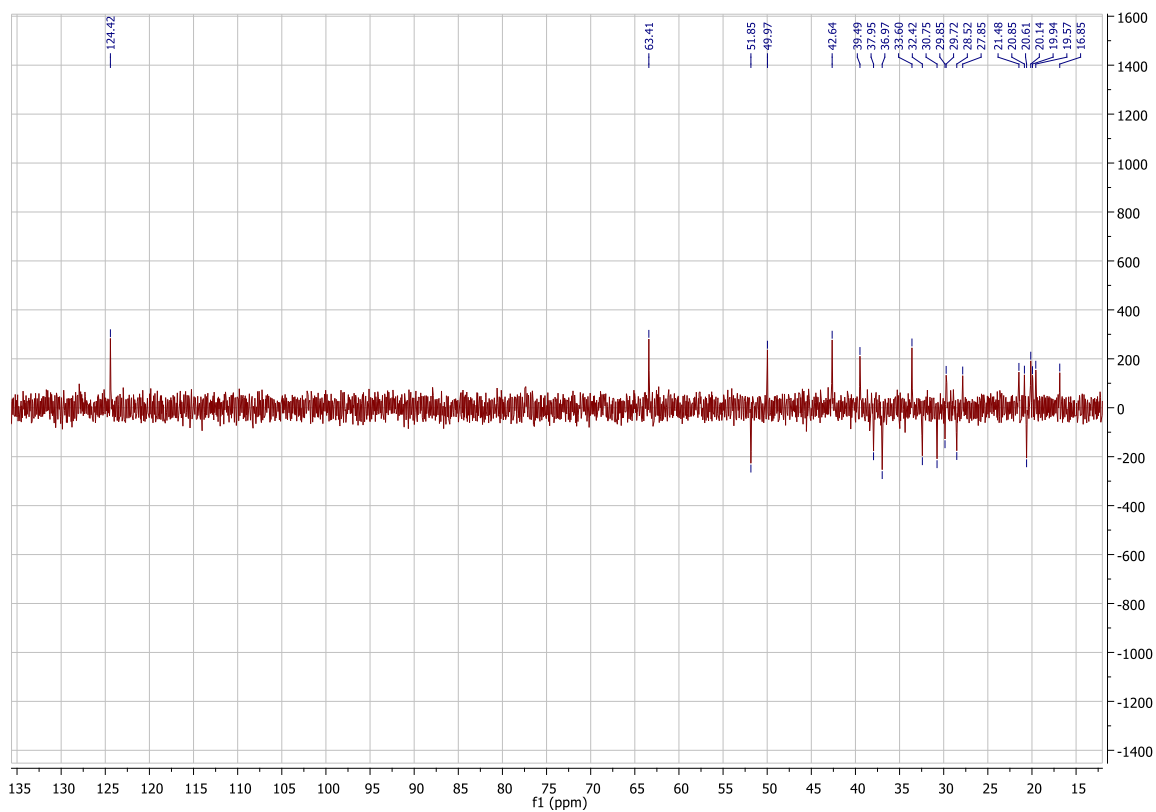


Figure S14. DEPT spectrum of compound **2** in CDCl_3 (75 MHz).

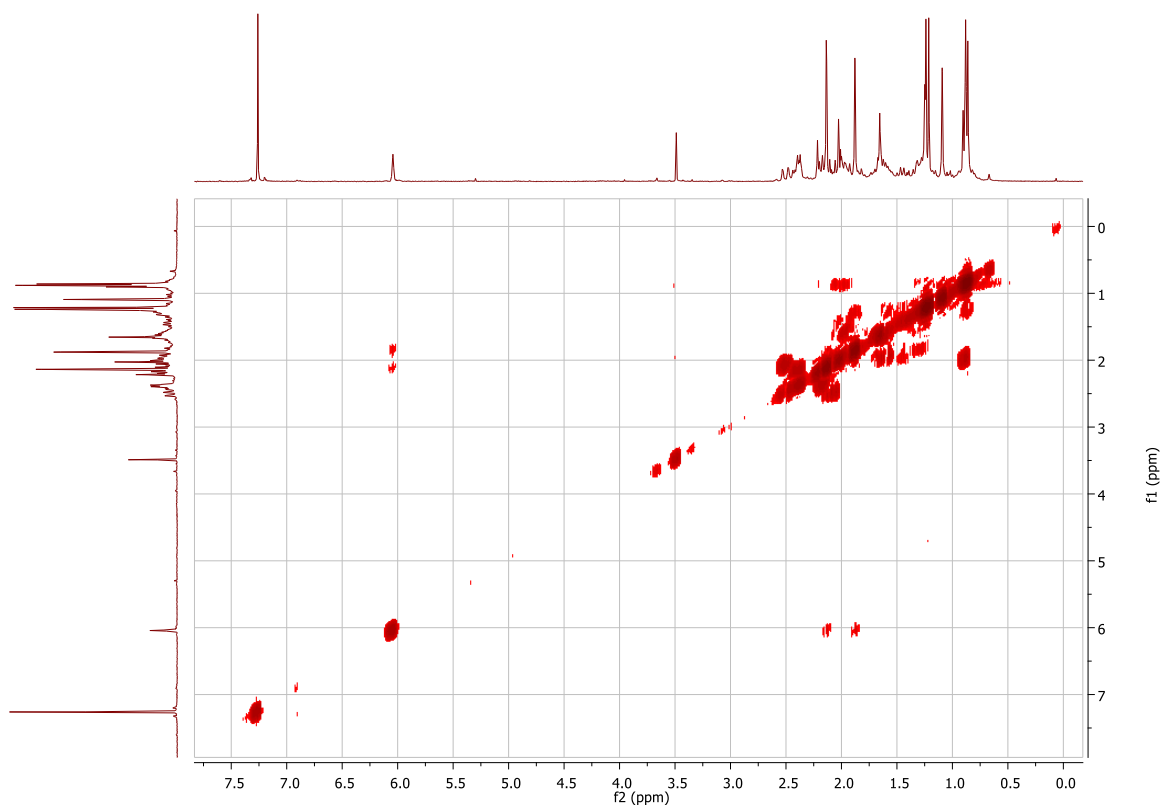


Figure S15. COSY spectrum of compound **2** in CDCl_3 .

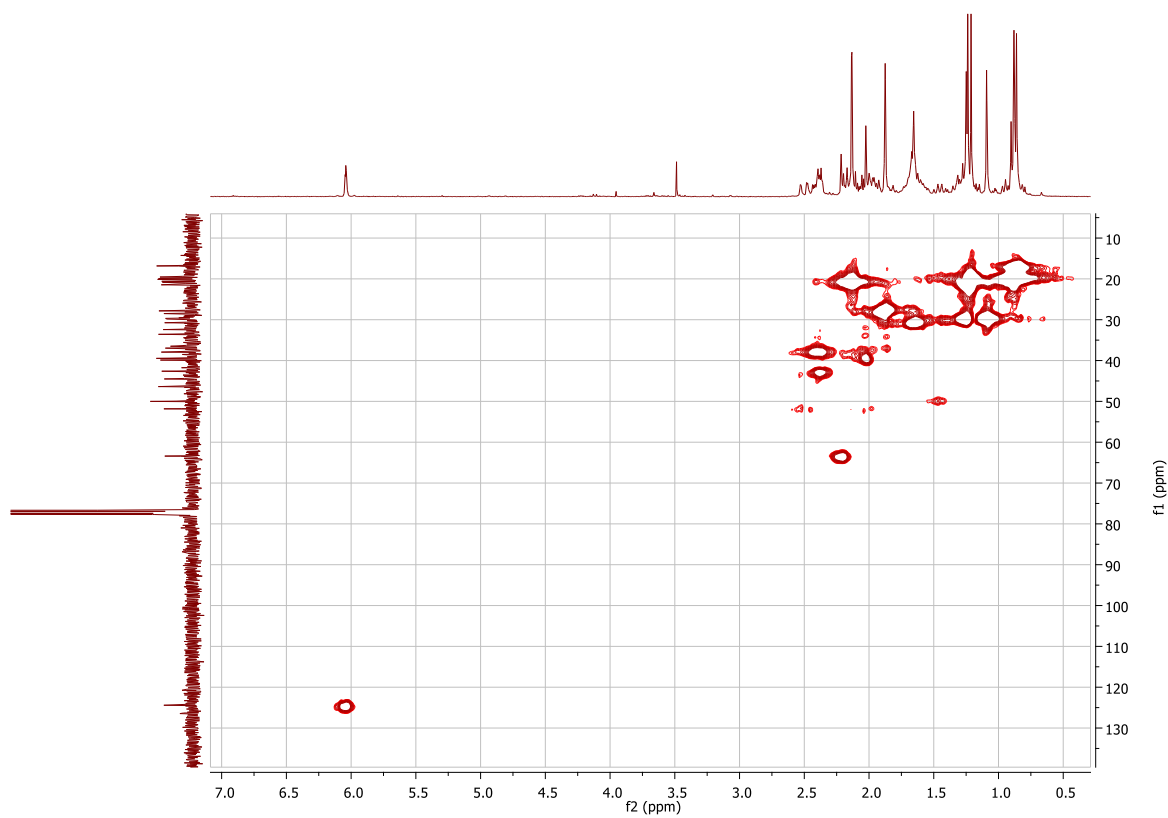


Figure S16. HMBC spectrum of compound **2** in CDCl_3 .

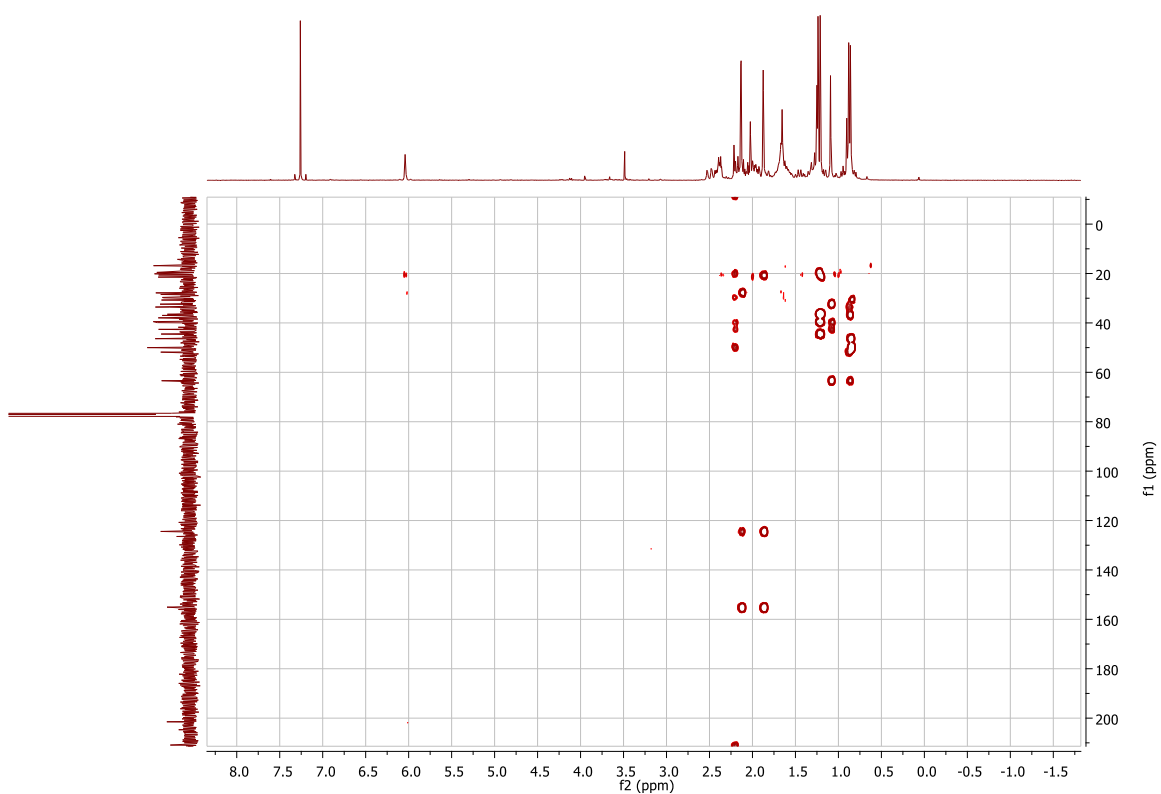


Figure S17. HMBC spectrum of compound **2** in CDCl_3 .

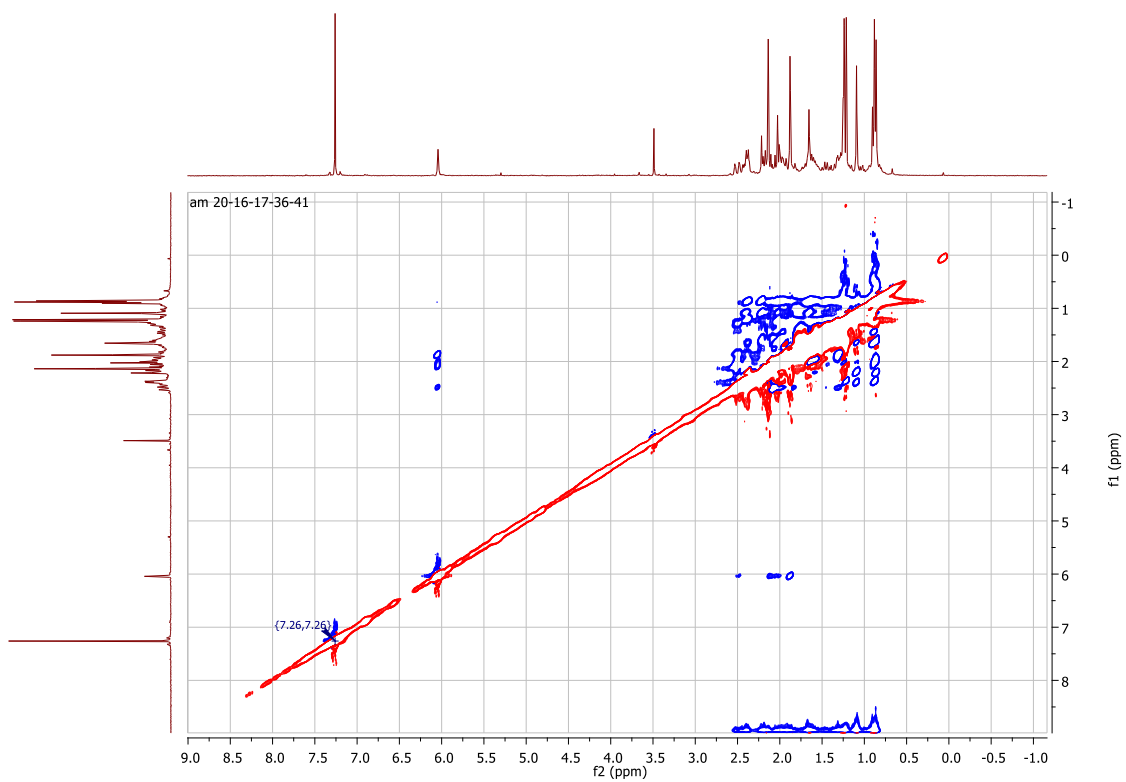
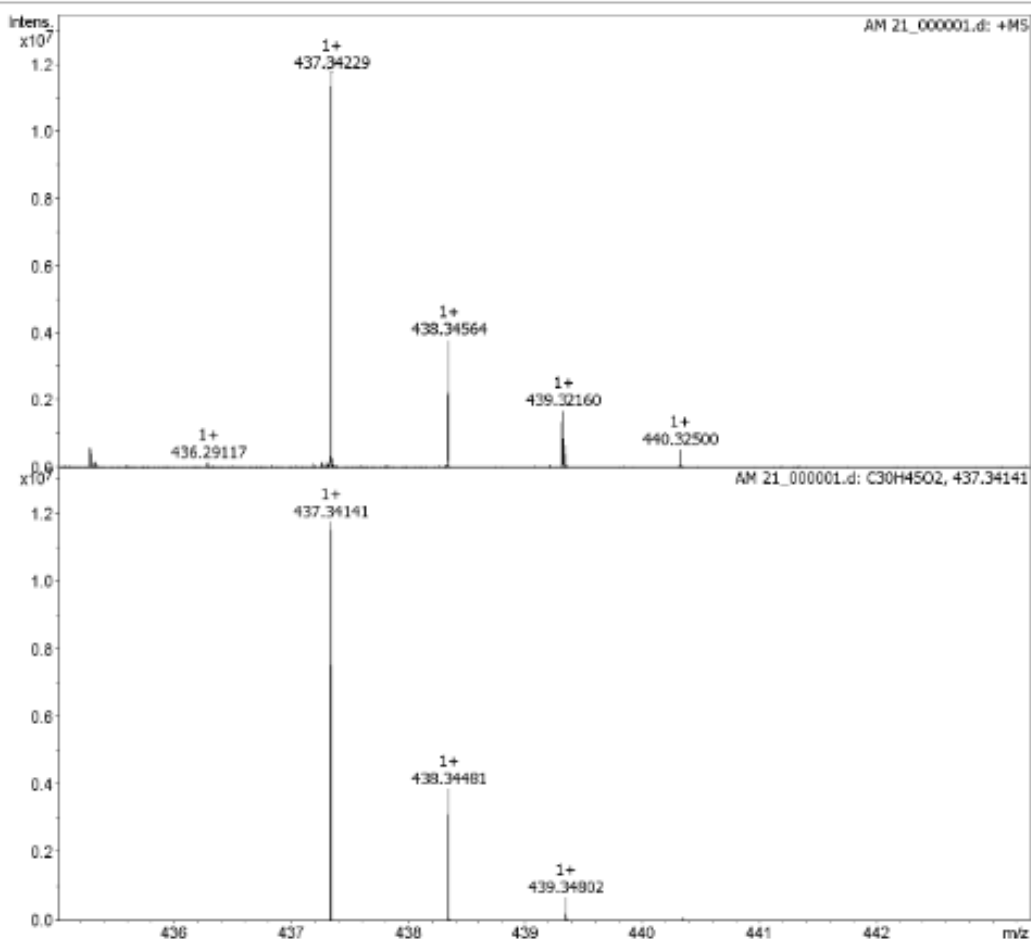


Figure S18. NOESY spectrum of compound **2** in CDCl_3 .

Analysis Info		Acquisition Date	1/19/2016 2:37:22 PM
Analysis Name	D:\Data\ANA 2016-01-19 FFULVAM 21_000001.d	Operator	
Method	ESI_Pos_IRMPD_20100618	Instrument	apex-Qe
Sample Name			
Comment			

Acquisition Parameter			
Polarity	Positive	Source	ESI
Averaged Scans	20	No. of Cell Fills	1
Broadband Low Mass	290.9 m/z	End Plate	4000.0 V
Broadband High Mass	1200.0 m/z	Capillary Entrance	4500.0 V
Acquisition Mode	Single MS	Skimmer 1	20.0 V
Pulse Program	basic	Drying Gas Temperature	180.0 °C
Source Accumulation	0.1 sec	Drying Gas Flow Rate	2.0 L/min
Ion Accumulation Time	0.5 sec	Nebulizer Gas Flow Rate	4.0 L/min
Flight Time to Acq. Cell	0.0 sec	No. of Laser Shots	1
		Laser Power	0.0 %
		MALDI Plate	250.0 V
		Imaging Spot Diameter	2000.0 µm
		Calibration Date	Wed Jan 13 04:47:30
		Data Acquisition Size	304888
		Apodization	Sine-Bell Multiplication



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Page 1 of 1

Figure S19. HRMS of compound 3.

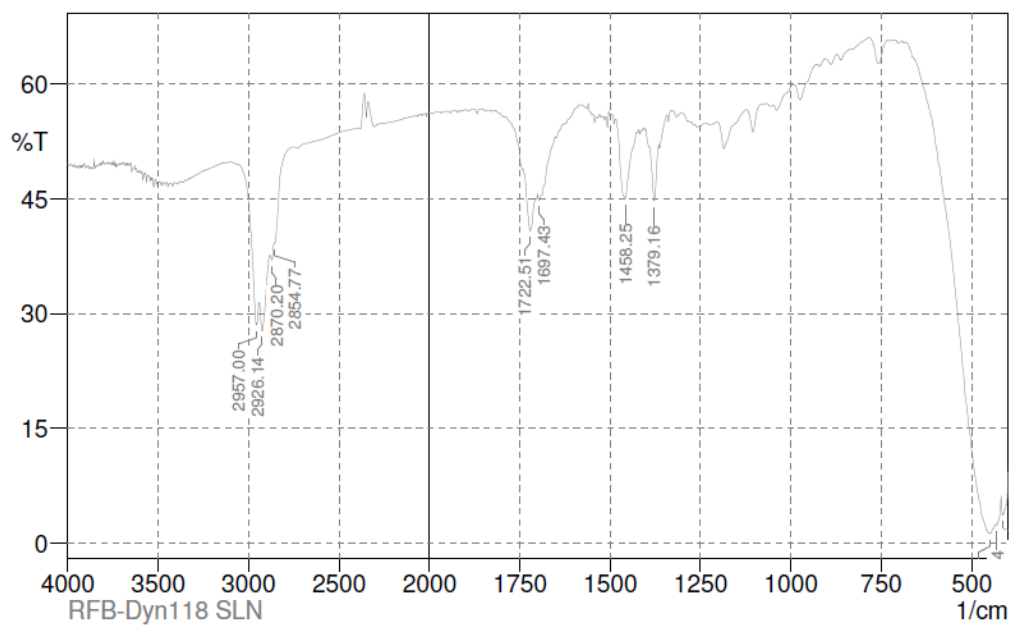


Figure S20. HRMS of compound 3.

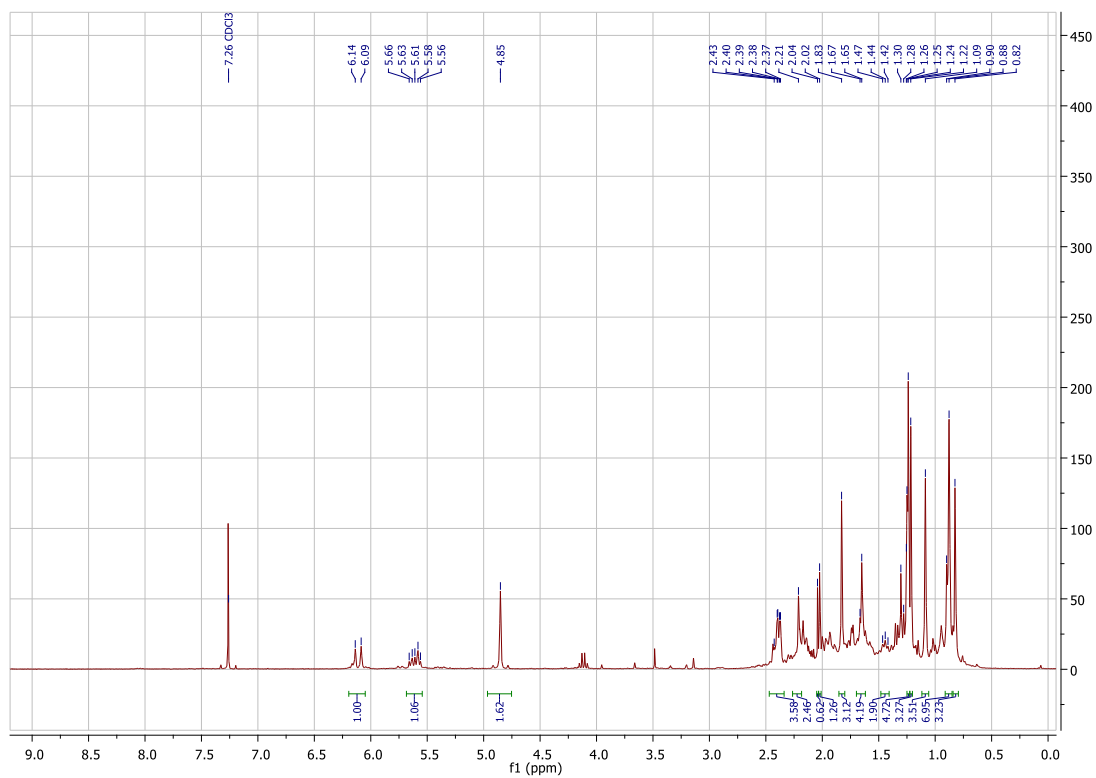


Figure S21 $^1\text{H-NMR}$ spectrum of compound 3 in CDCl_3 (300 MHz)..

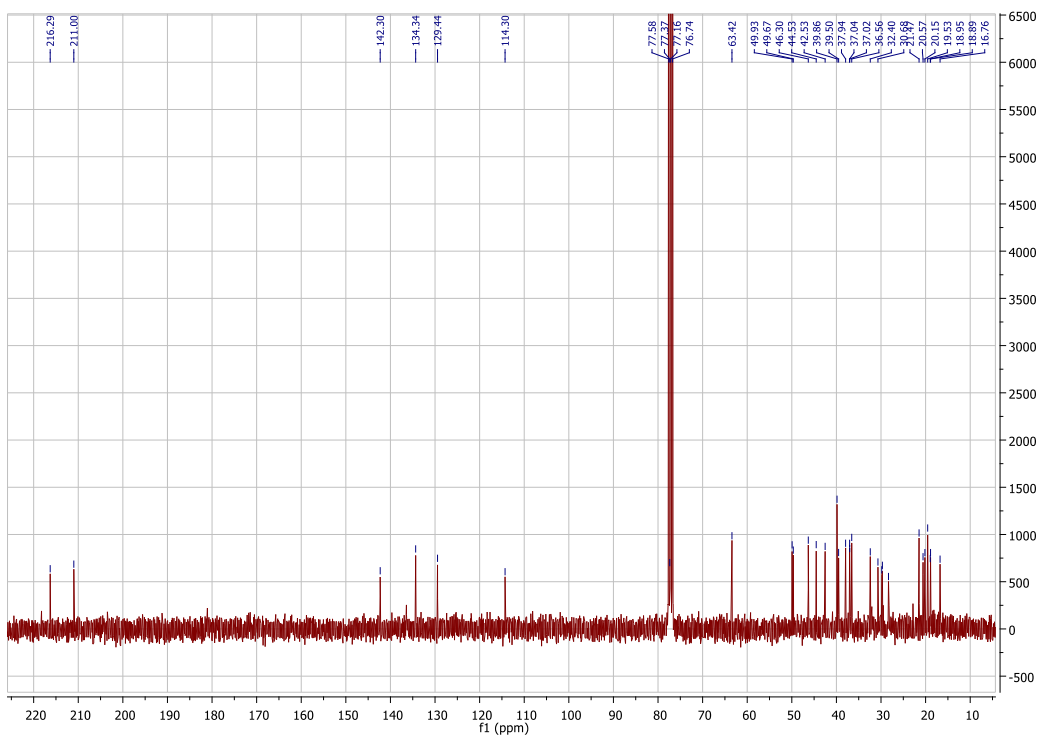


Figure S22. ^{13}C -NMR spectrum of compound **3** in CDCl_3 (75 MHz).

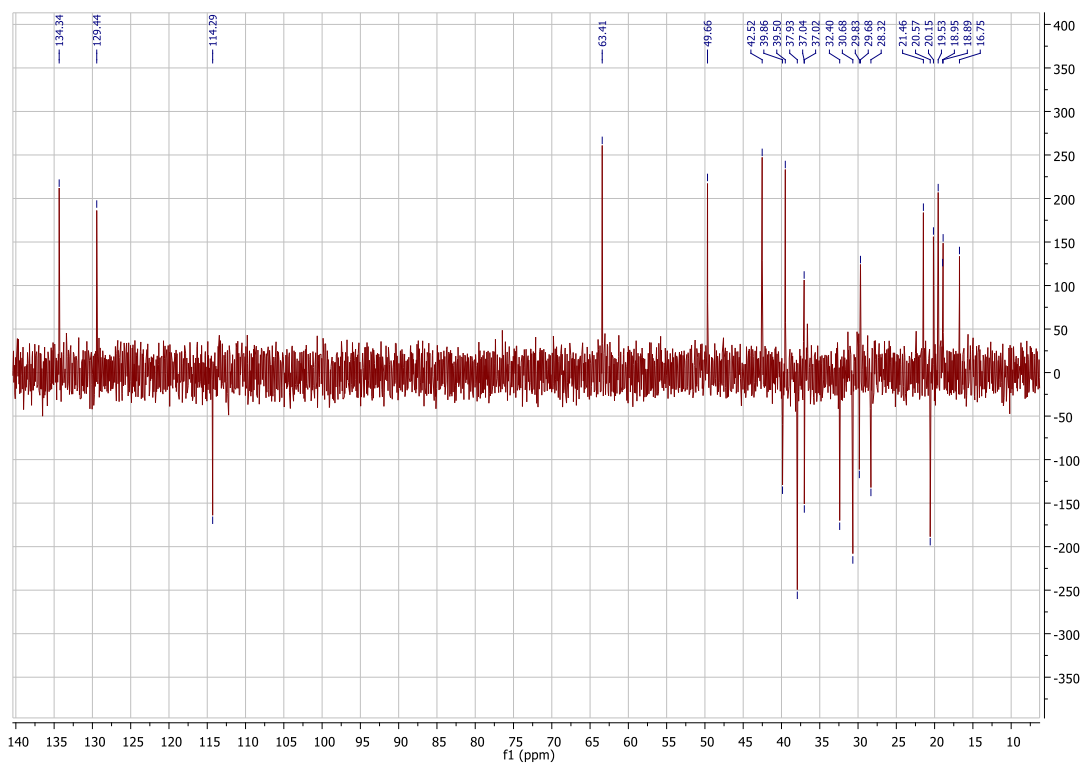


Figure S23. DEPT spectrum of compound **3** in CDCl_3 (75 MHz)

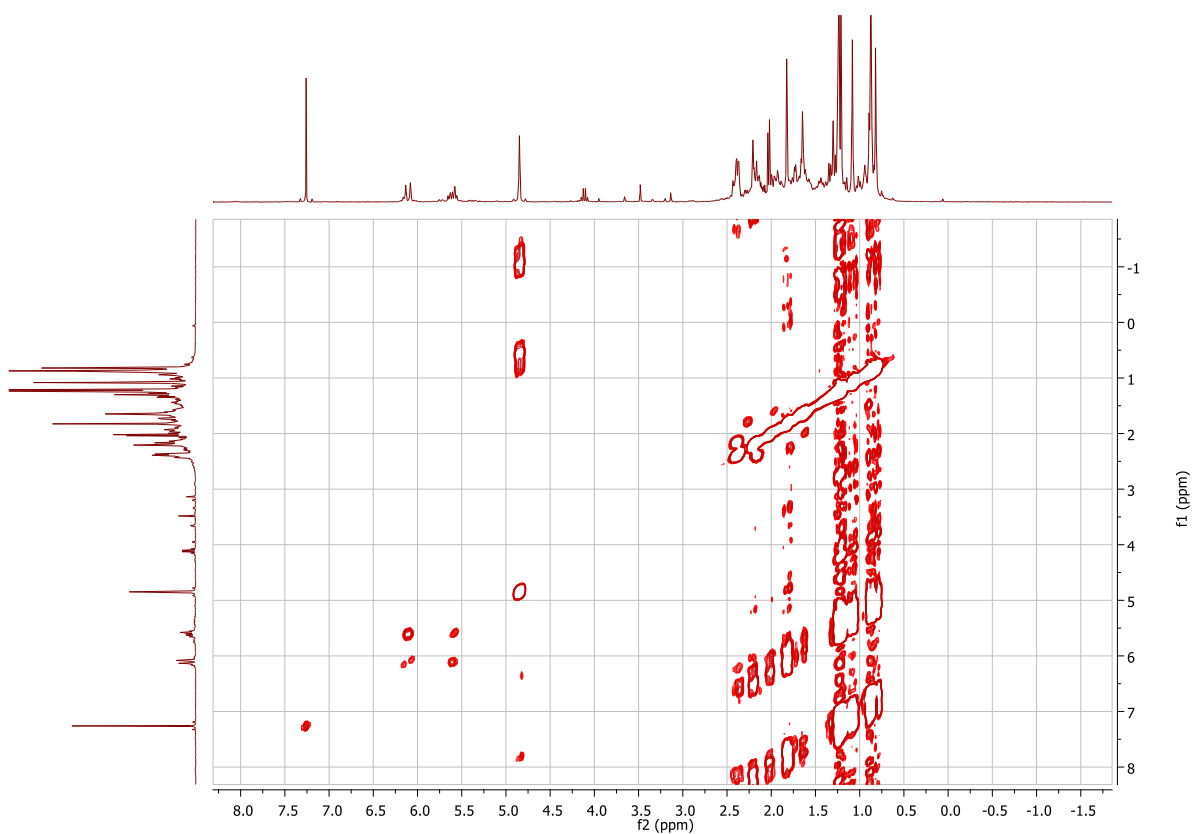


Figure S24. COSY spectrum of compound **3** in CDCl₃.

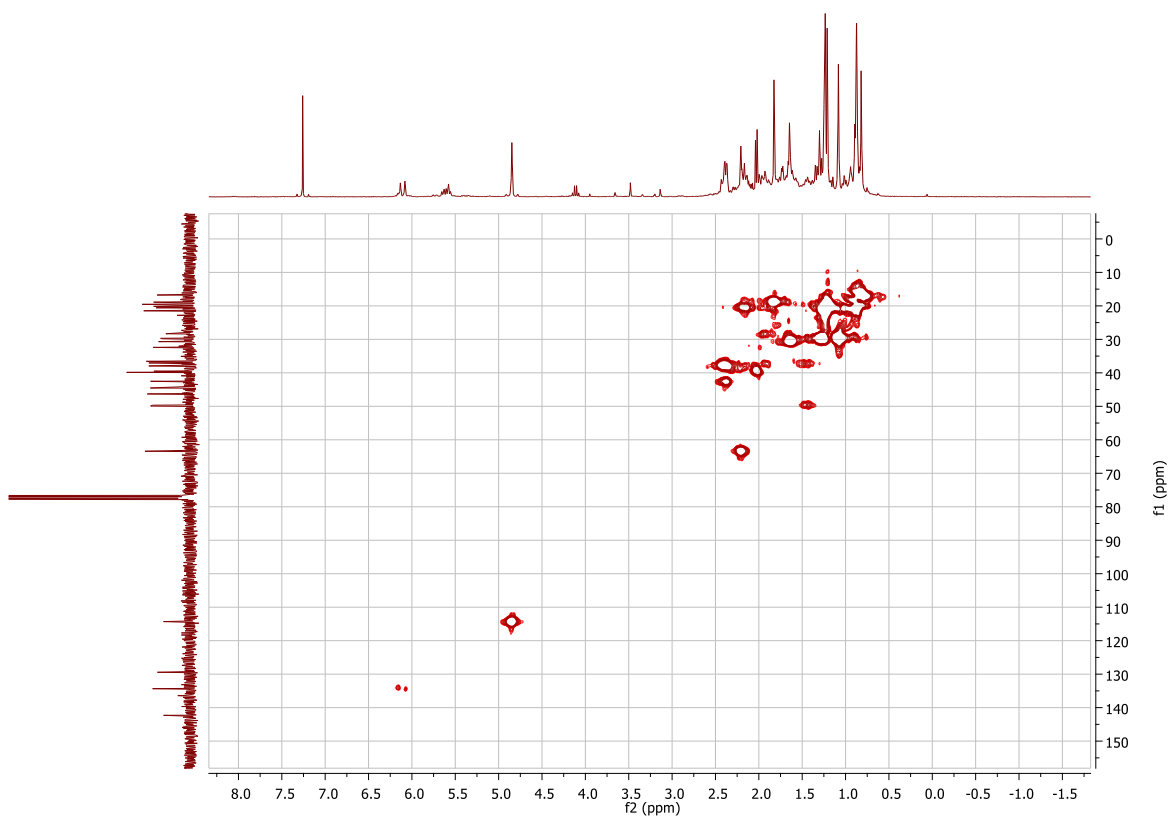


Figure S25. HMQC spectrum of compound **3** in CDCl₃.

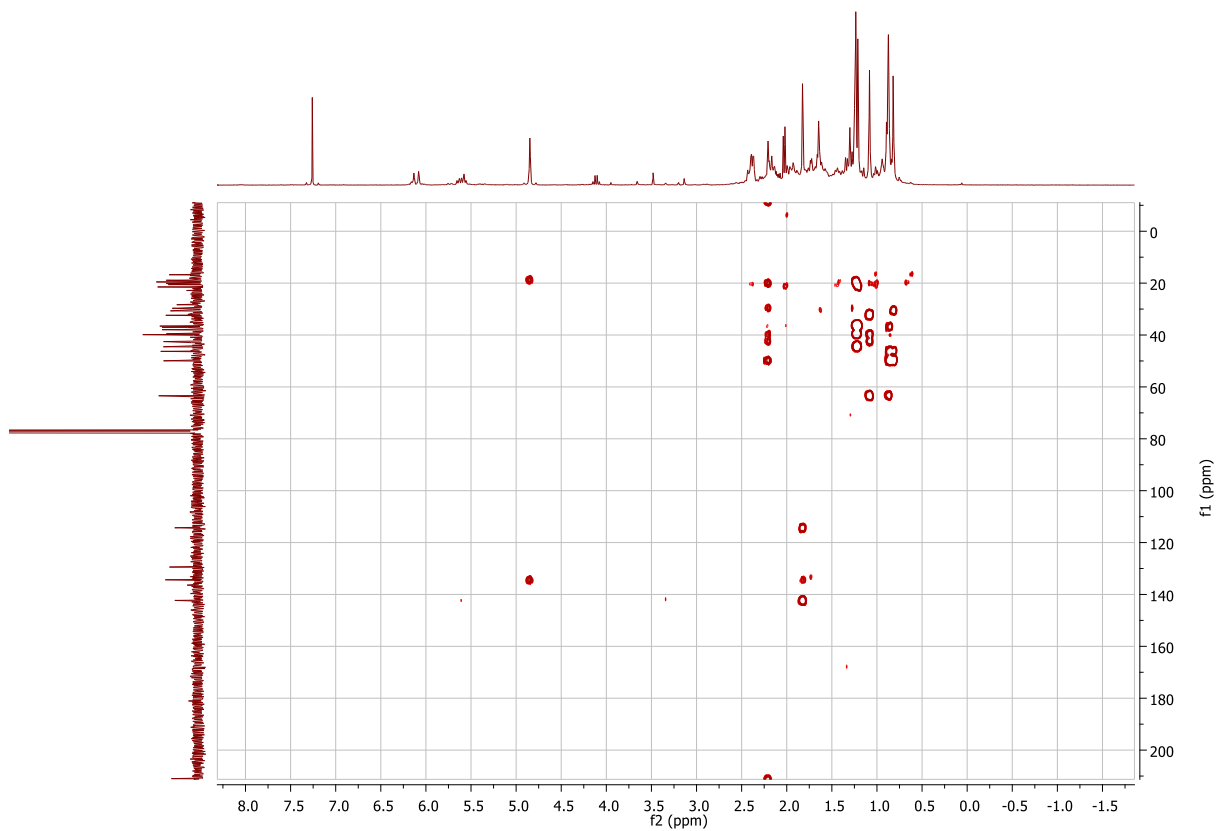


Figure S26. HMBC spectrum of compound **3** in CDCl₃