Supporting information for:

Synthesis and Solid State Dynamics of a Crystalline Steroid Molecular Rotor Without the Alkyne Axle: Steroid Dimers Based on a 1,4-Di(1,3-dioxan-2-yl)benzene Moiety

Katherine Vargas-Romero, Fátima C. Martínez-Torres, Andrés Aguilar-Granda, Salvador Pérez-Estrada, Marcos Flores-Alamo, Braulio Rodríguez-Molina,* Martín A. Iglesias-Arteaga *

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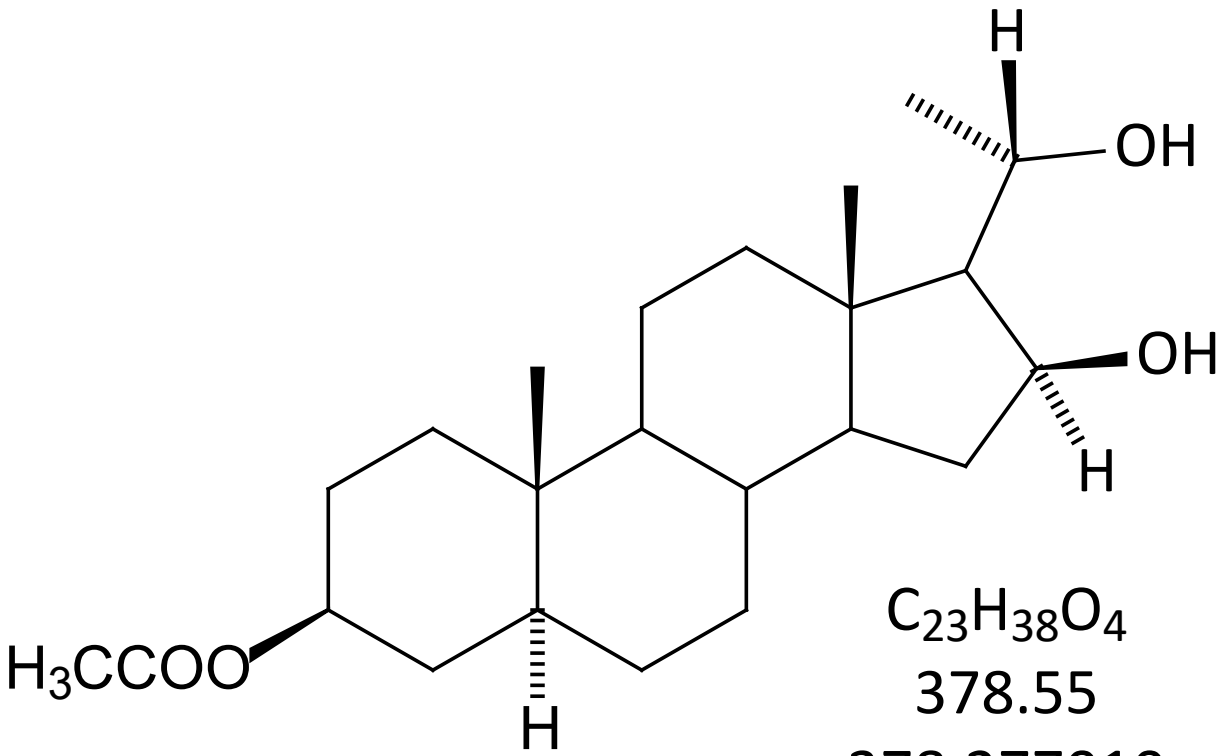
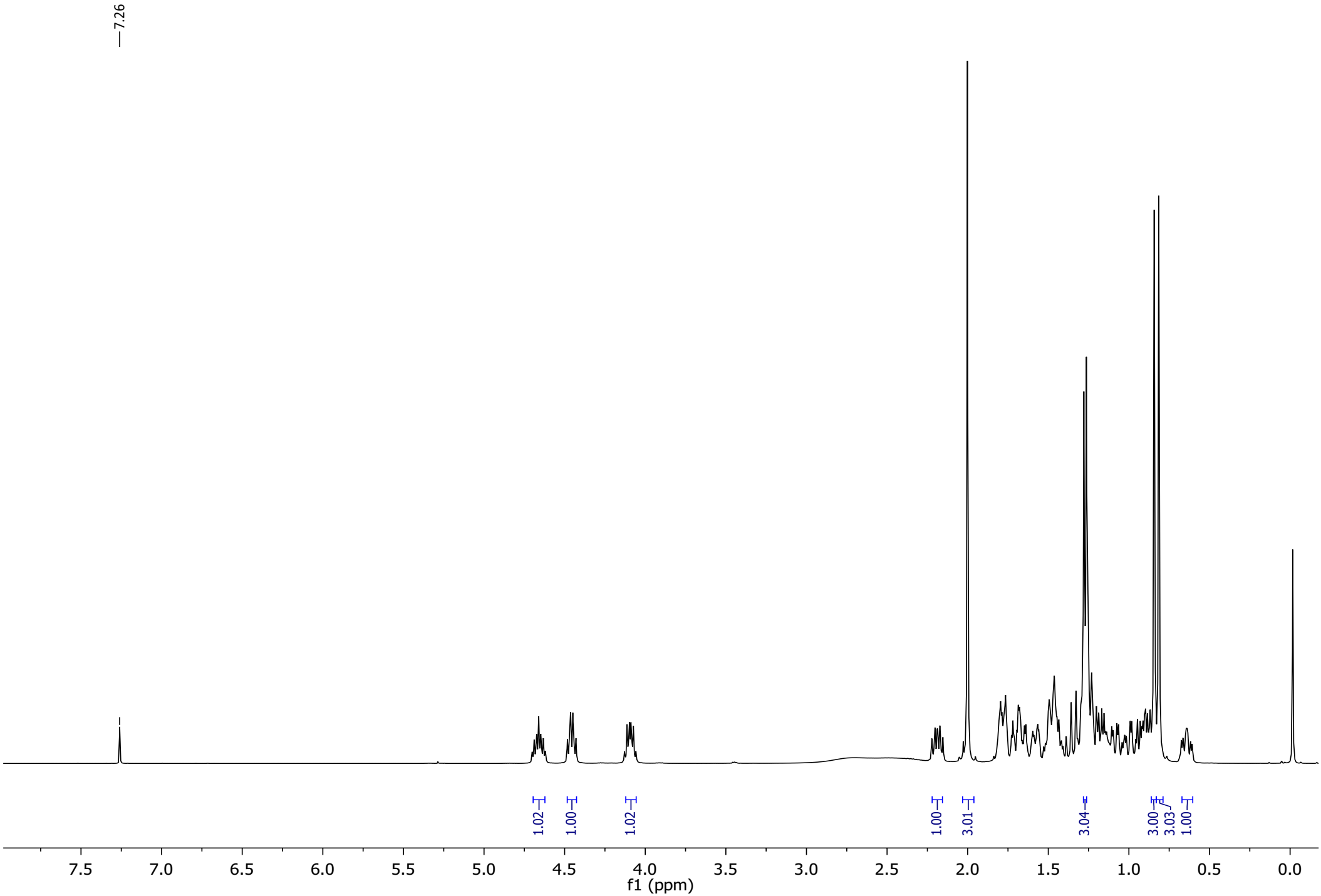
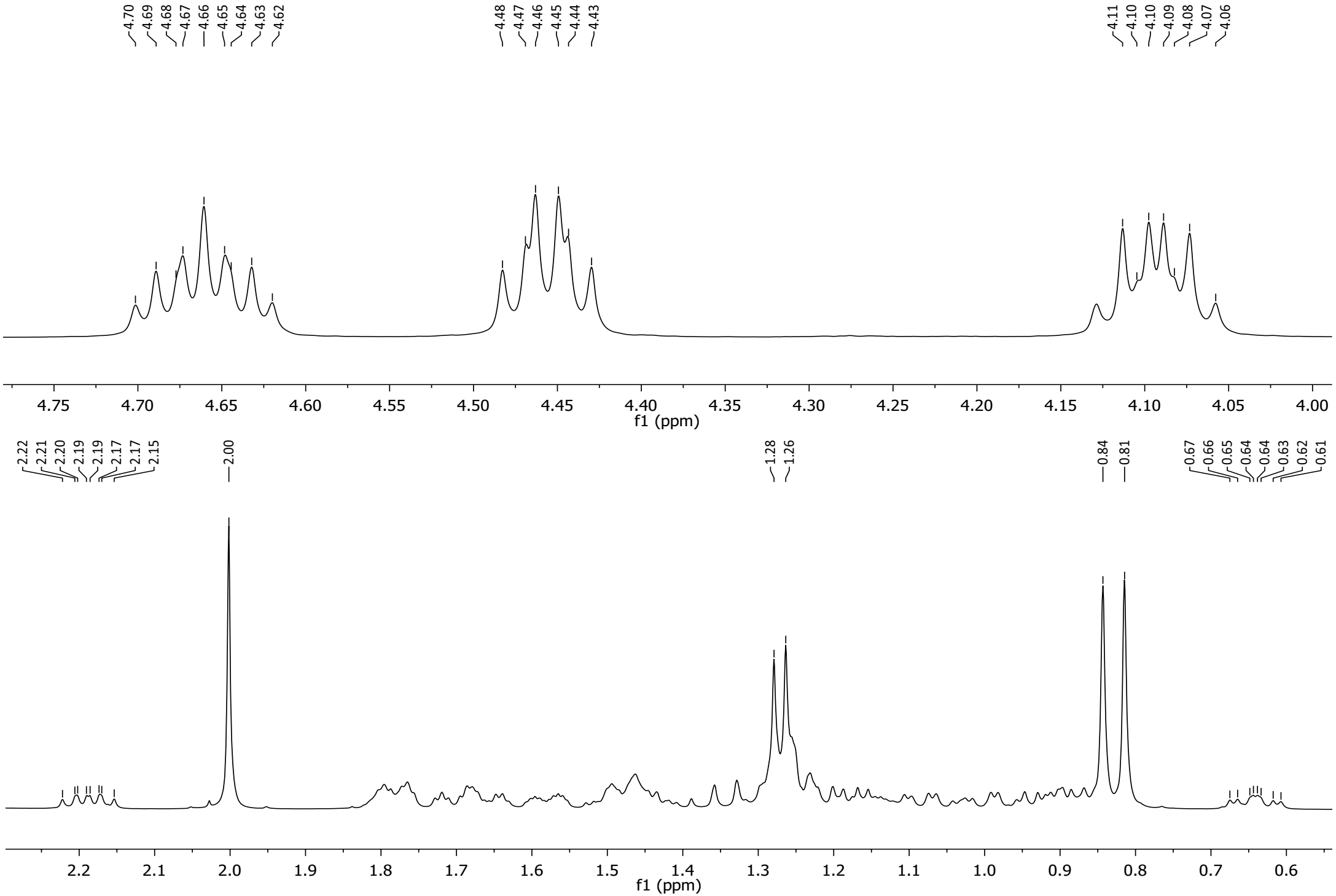


$C_{23}H_{38}O_4$
378.55
378.277010
C 72.98% H 10.12% O 16.91%

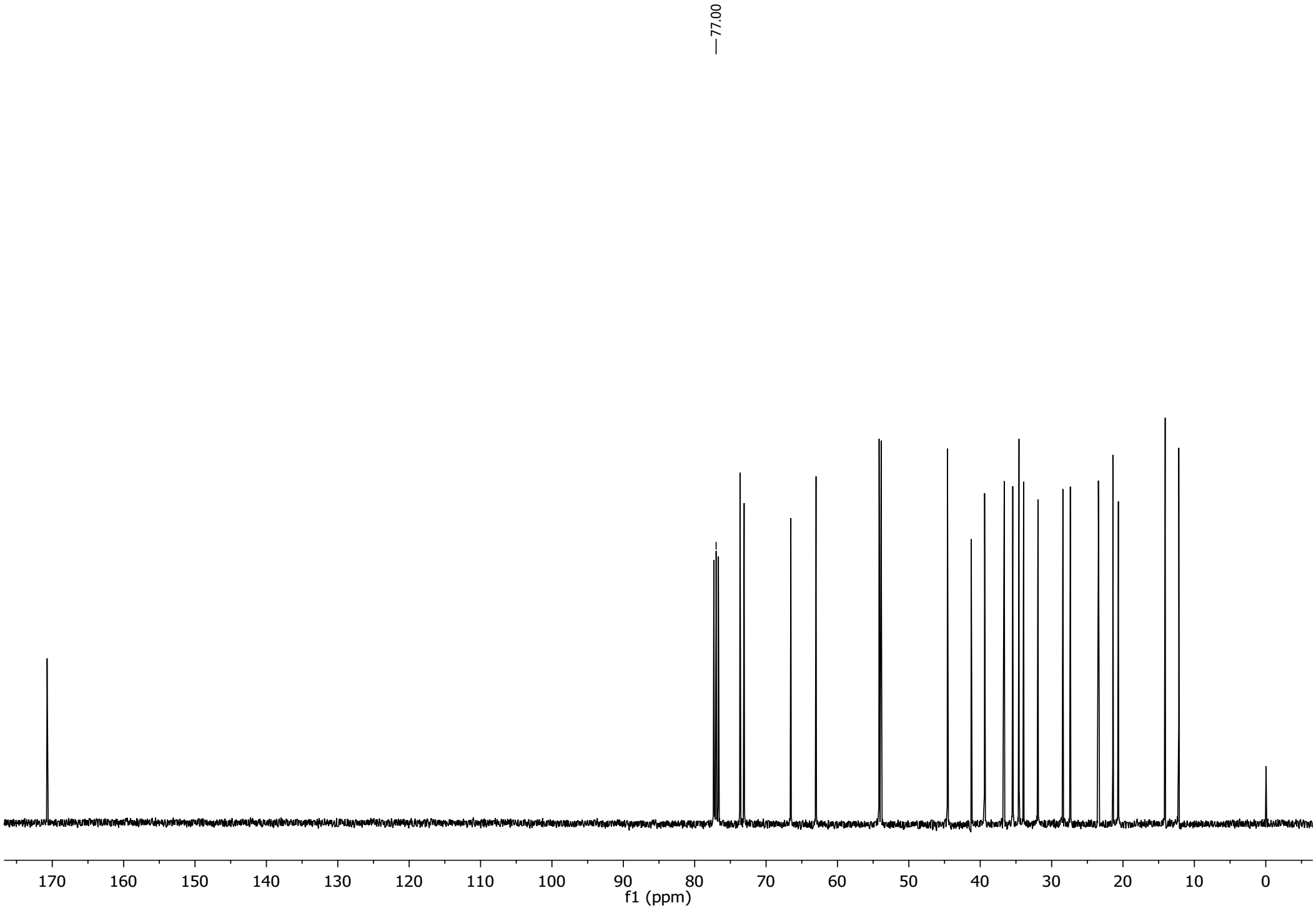
(20*S*)-5α-pregnan-3β,16β,20-triol 3-monoacetate **(17)**



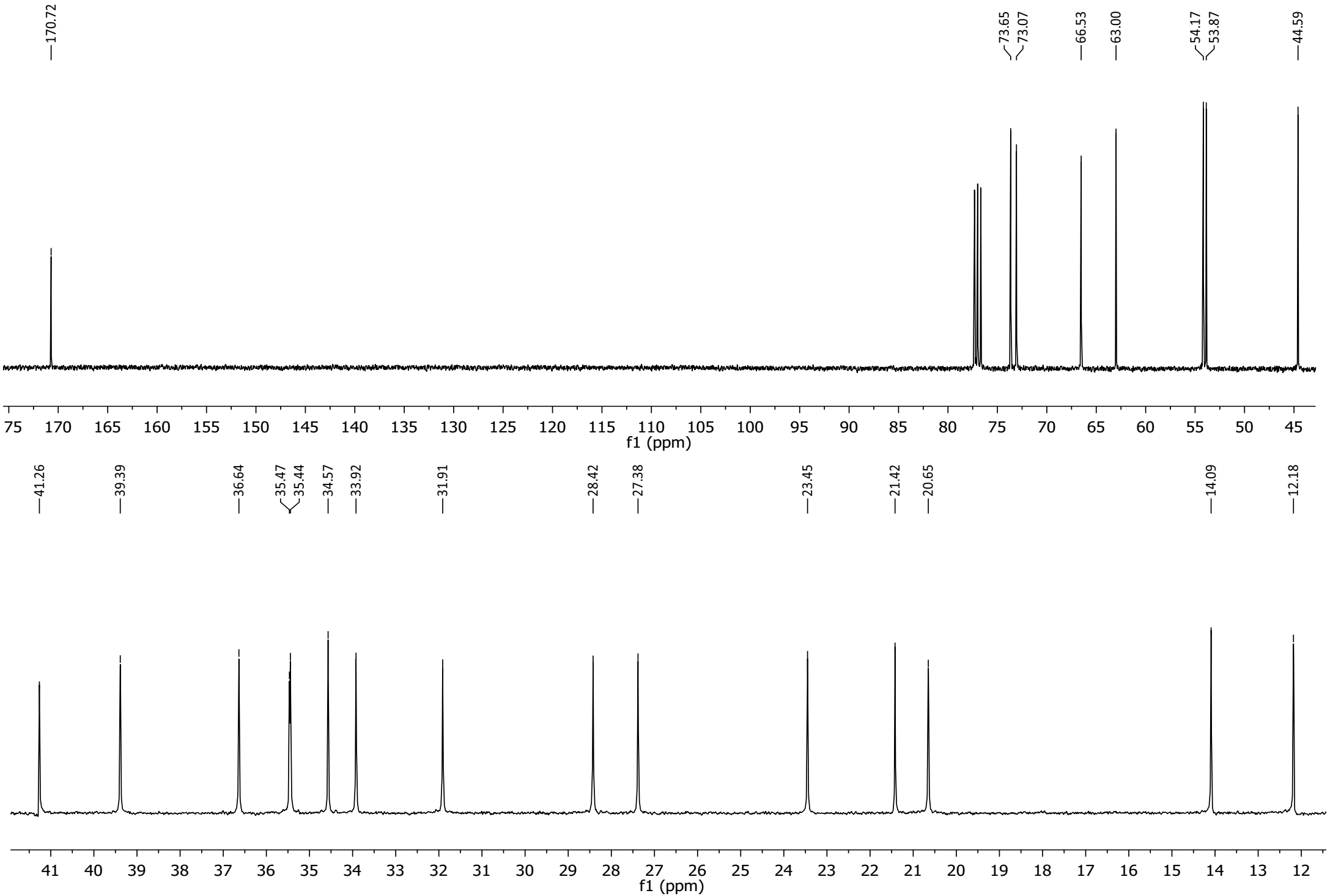
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **17**



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **17**



^{13}C NMR (100.53 MHz, $CDCl_3$) spectrum of compound **17**



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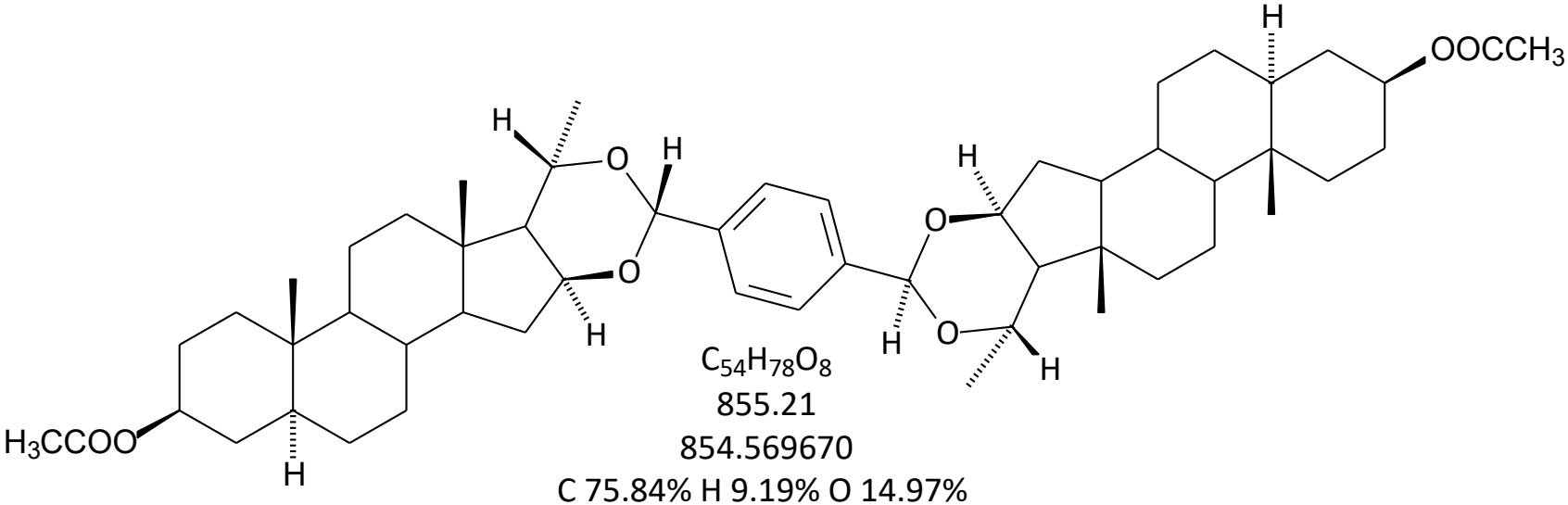
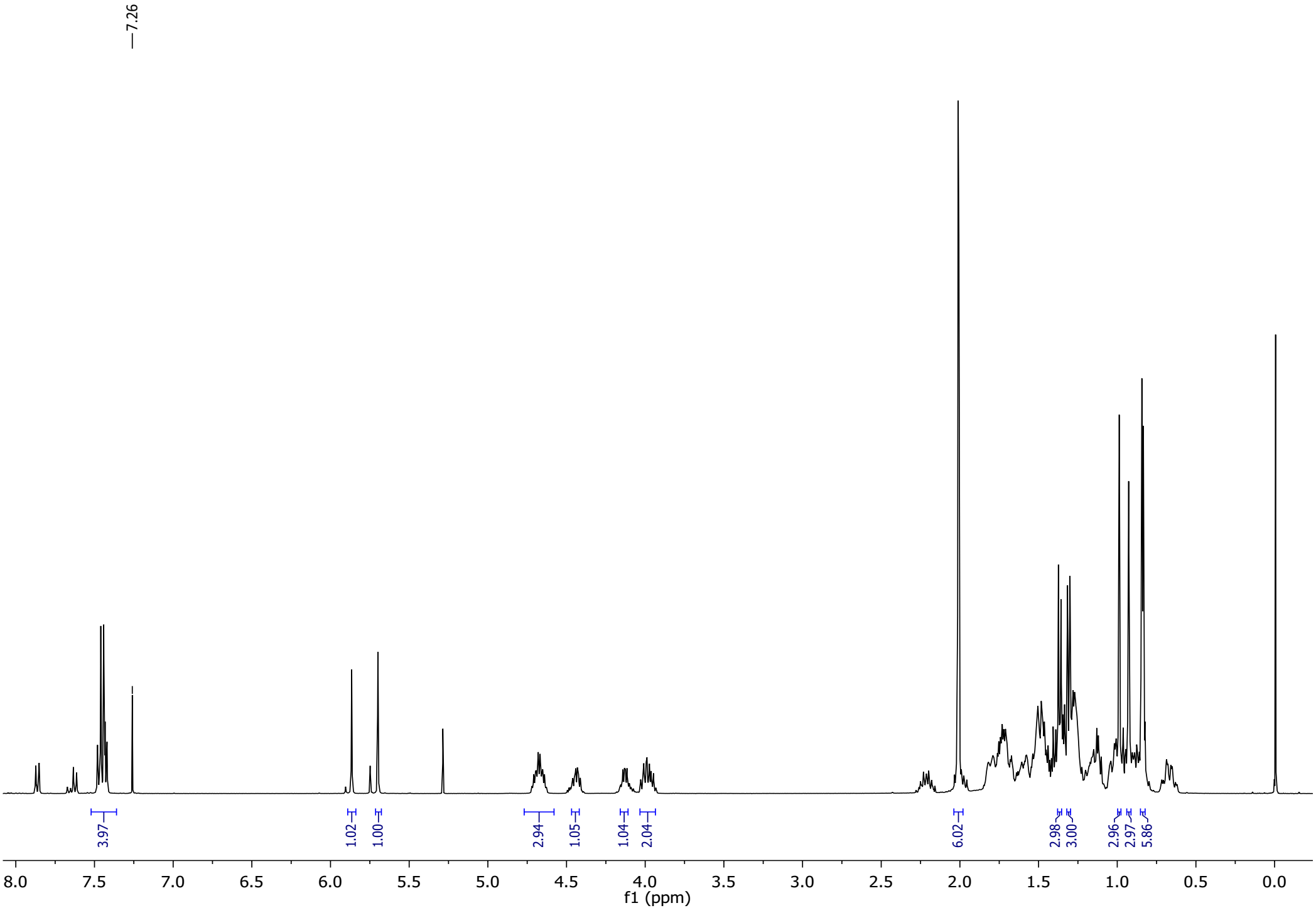
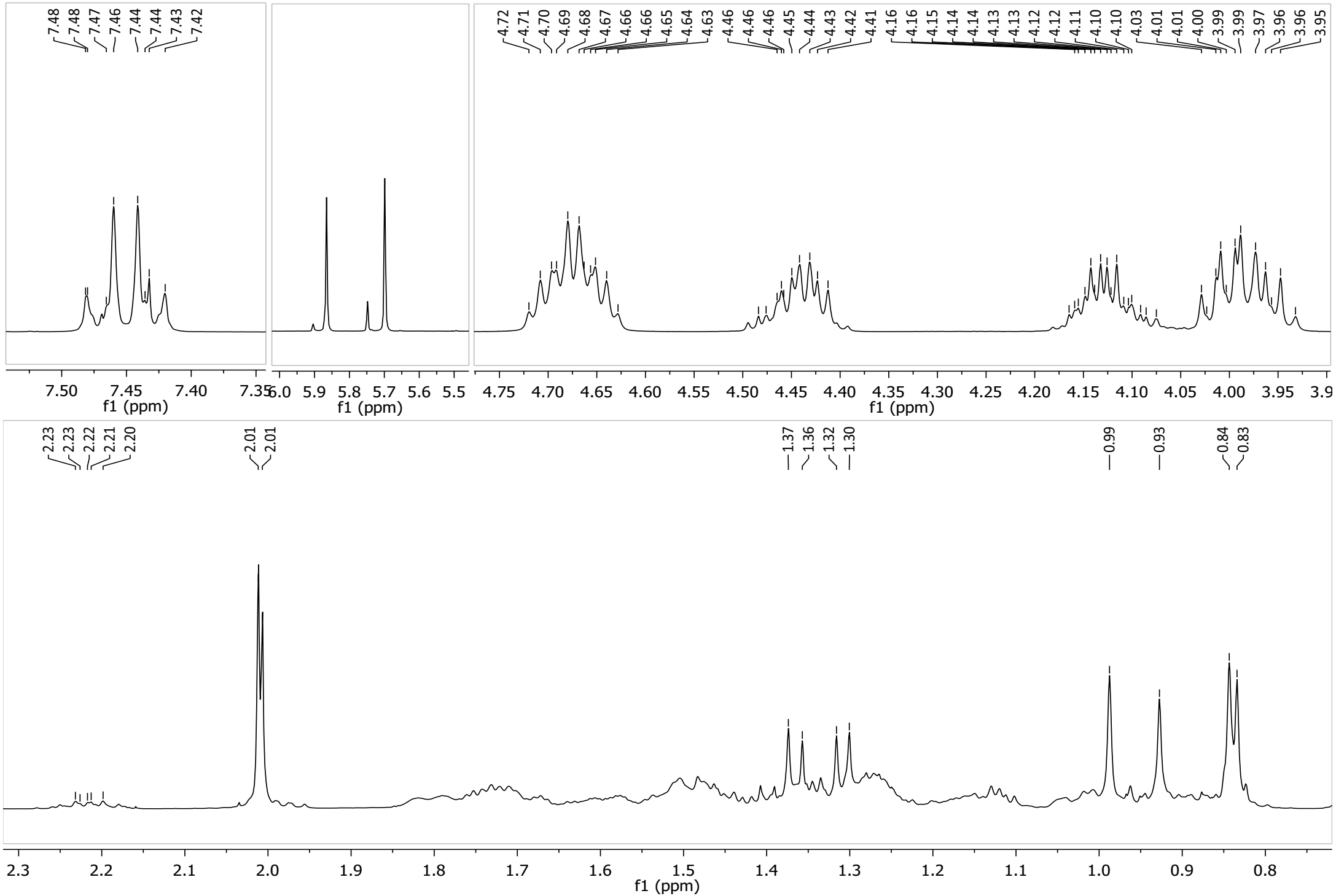


$C_{54}H_{78}O_8$
855.21
854.569670
C 75.84% H 9.19% O 14.97%

Dimer *SR*-18



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound ***SR*-18**



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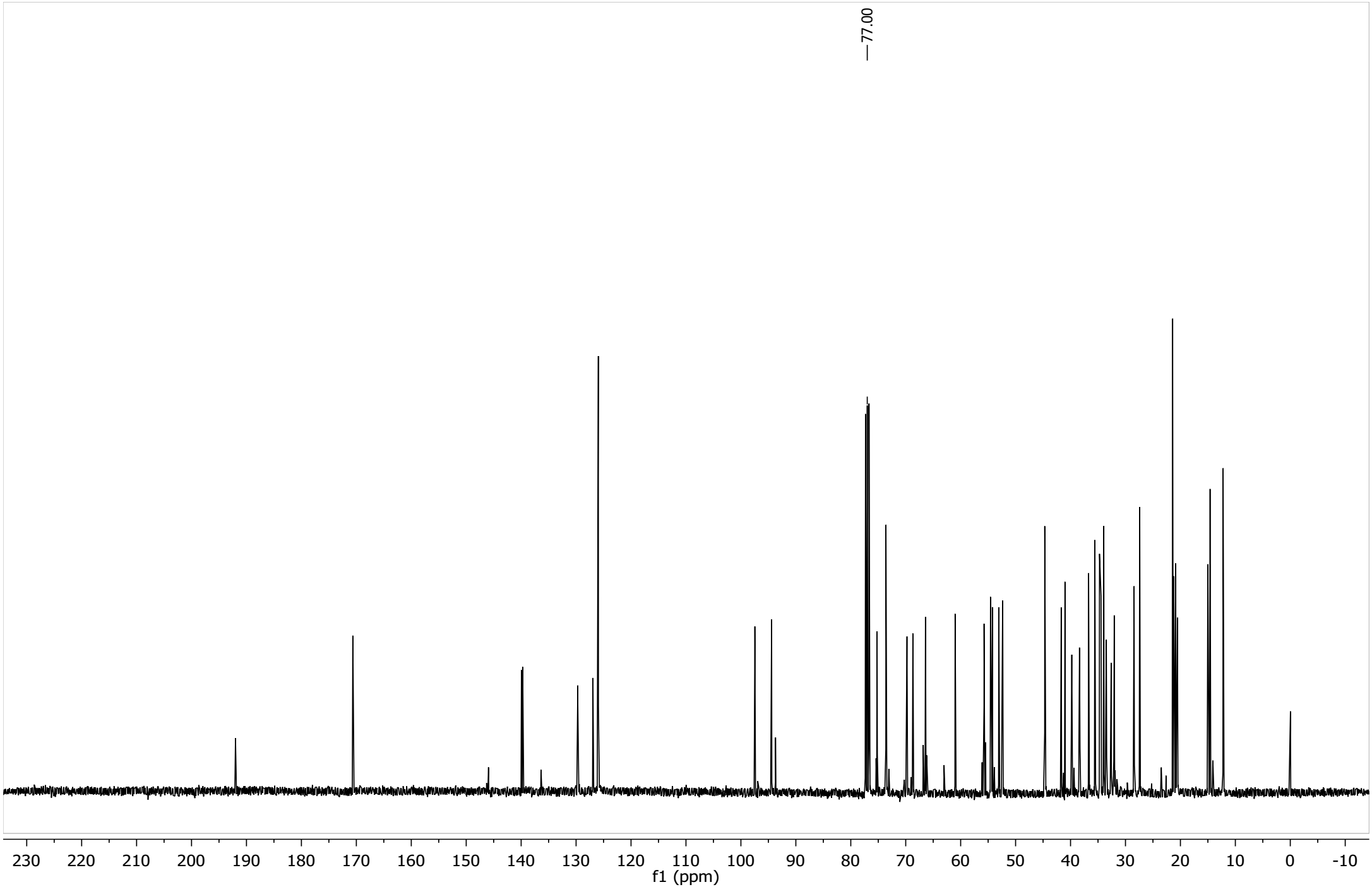

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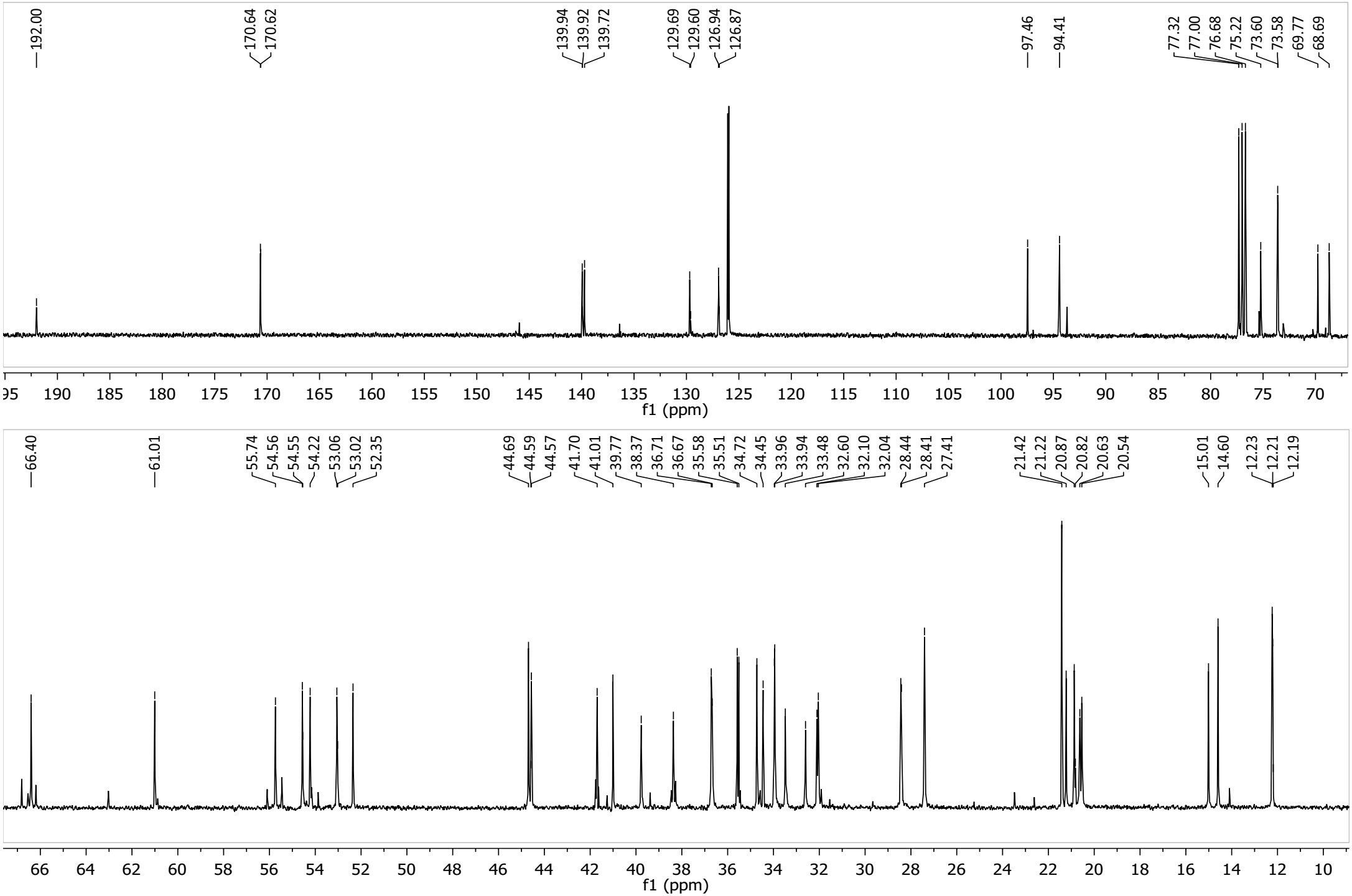
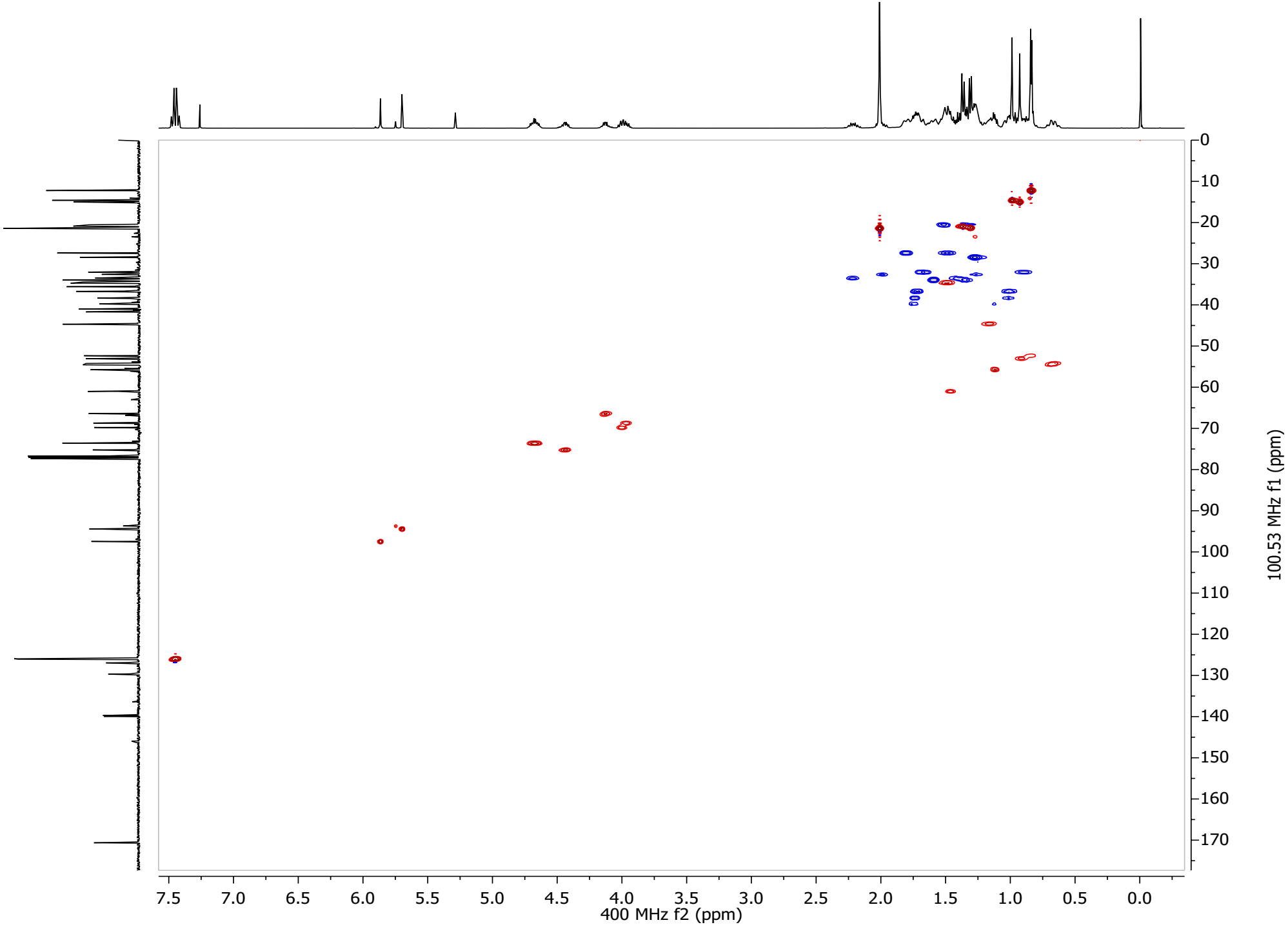
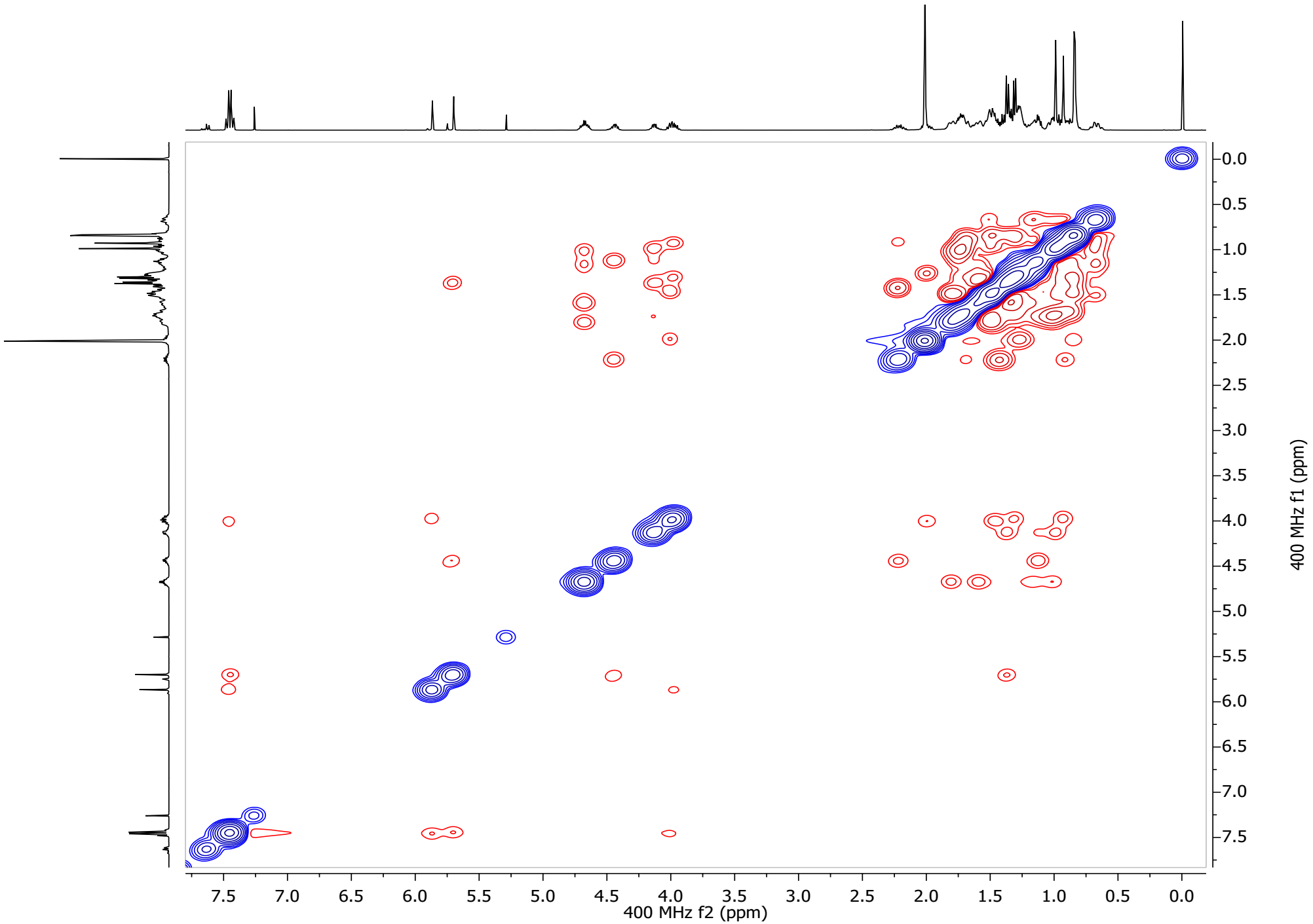


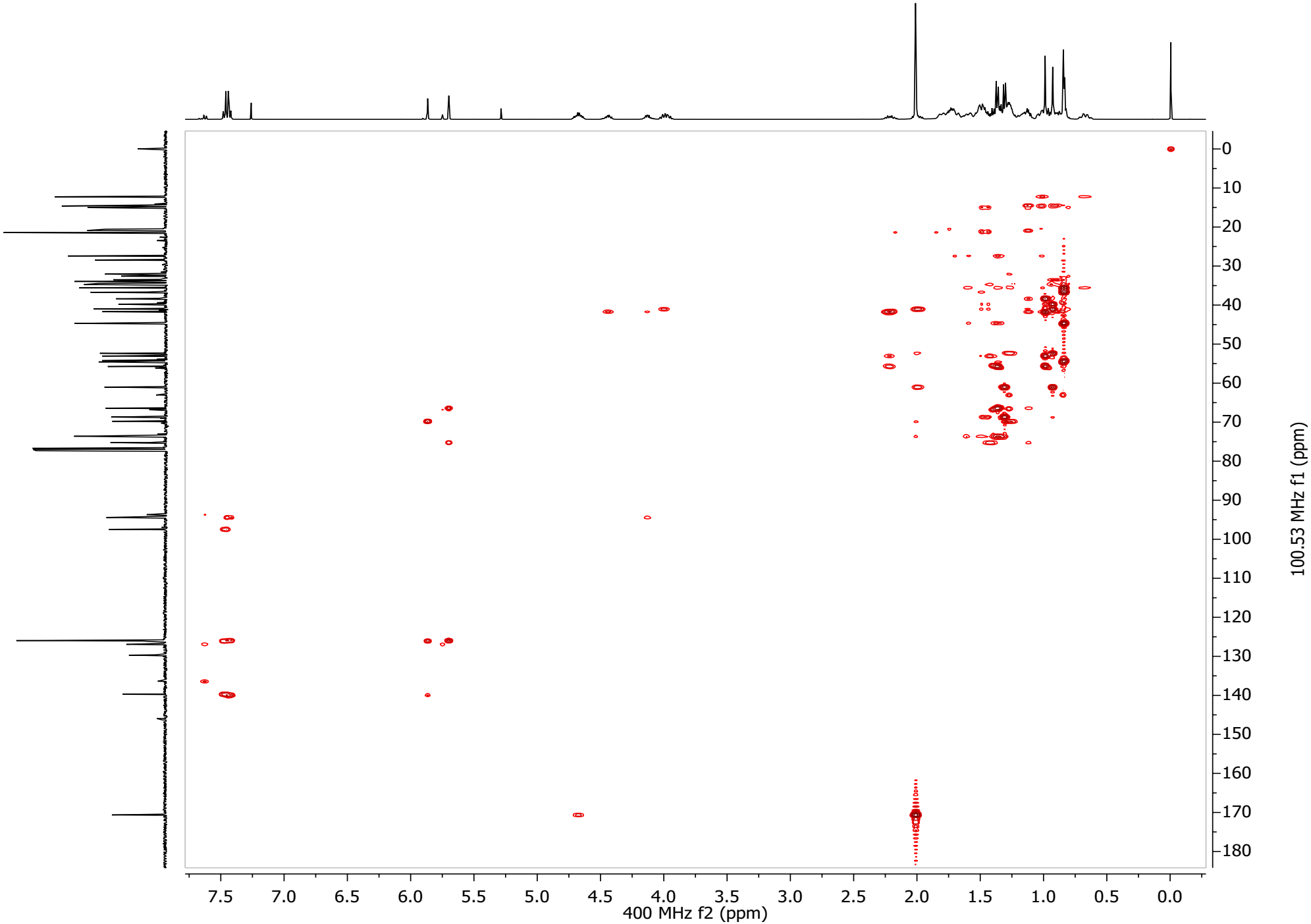
^{13}C NMR (100.53 MHz, $CDCl_3$) spectrum of compound ***SR*-18**



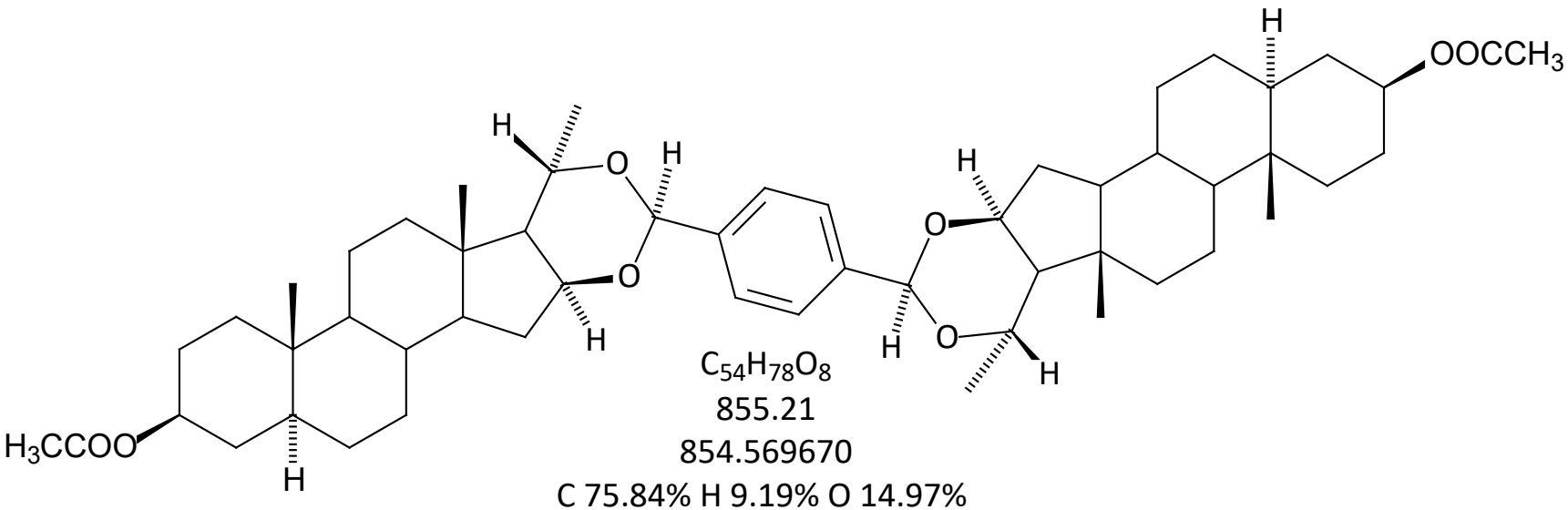
HSQC ($CDCl_3$) spectrum pf compoudn ***SR*-18**



NOESY($CDCl_3$) spectrum pf compoudn ***SR*-18**

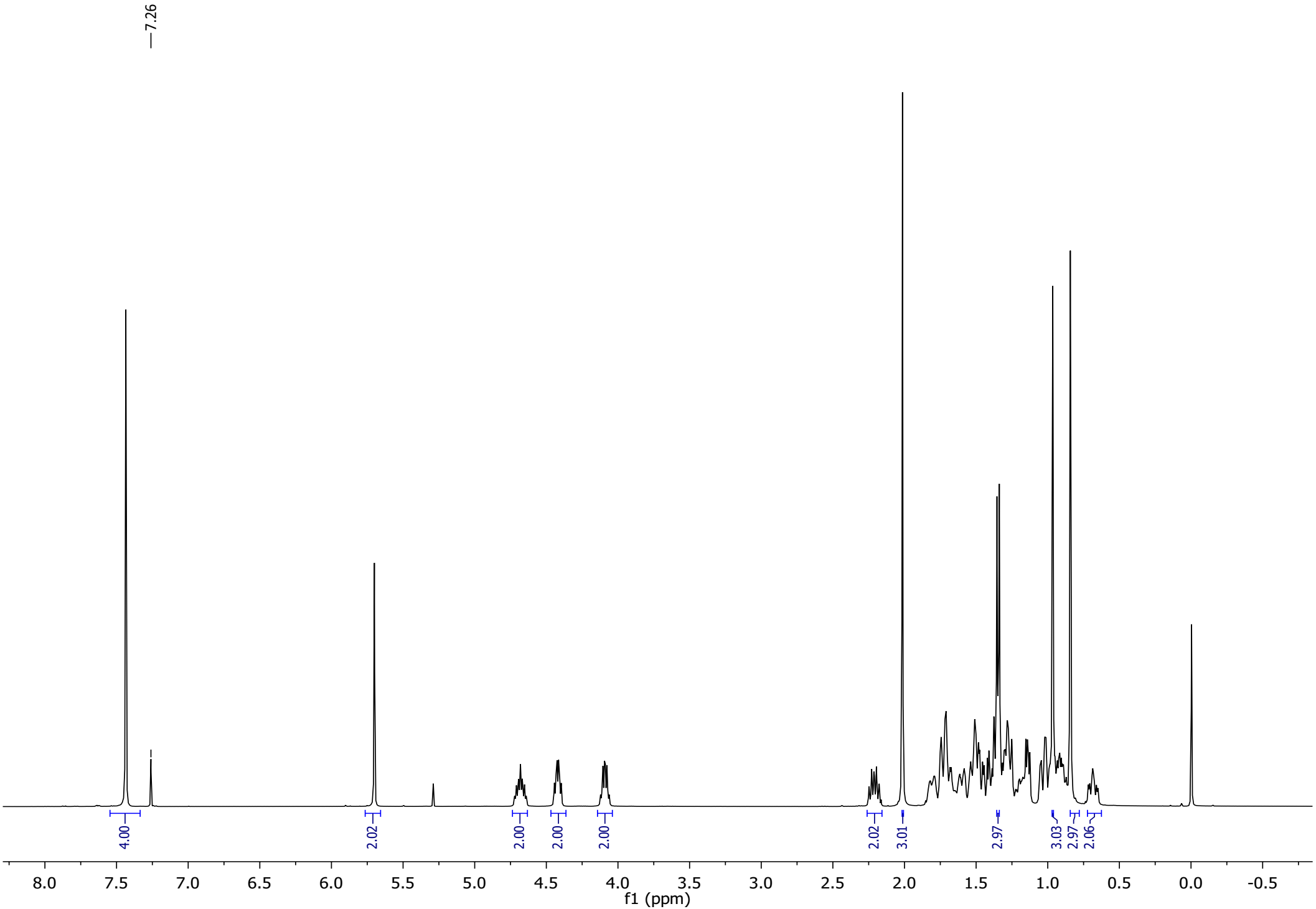


HMBC ($CDCl_3$) spectrum pf compoudn ***SR*-18**

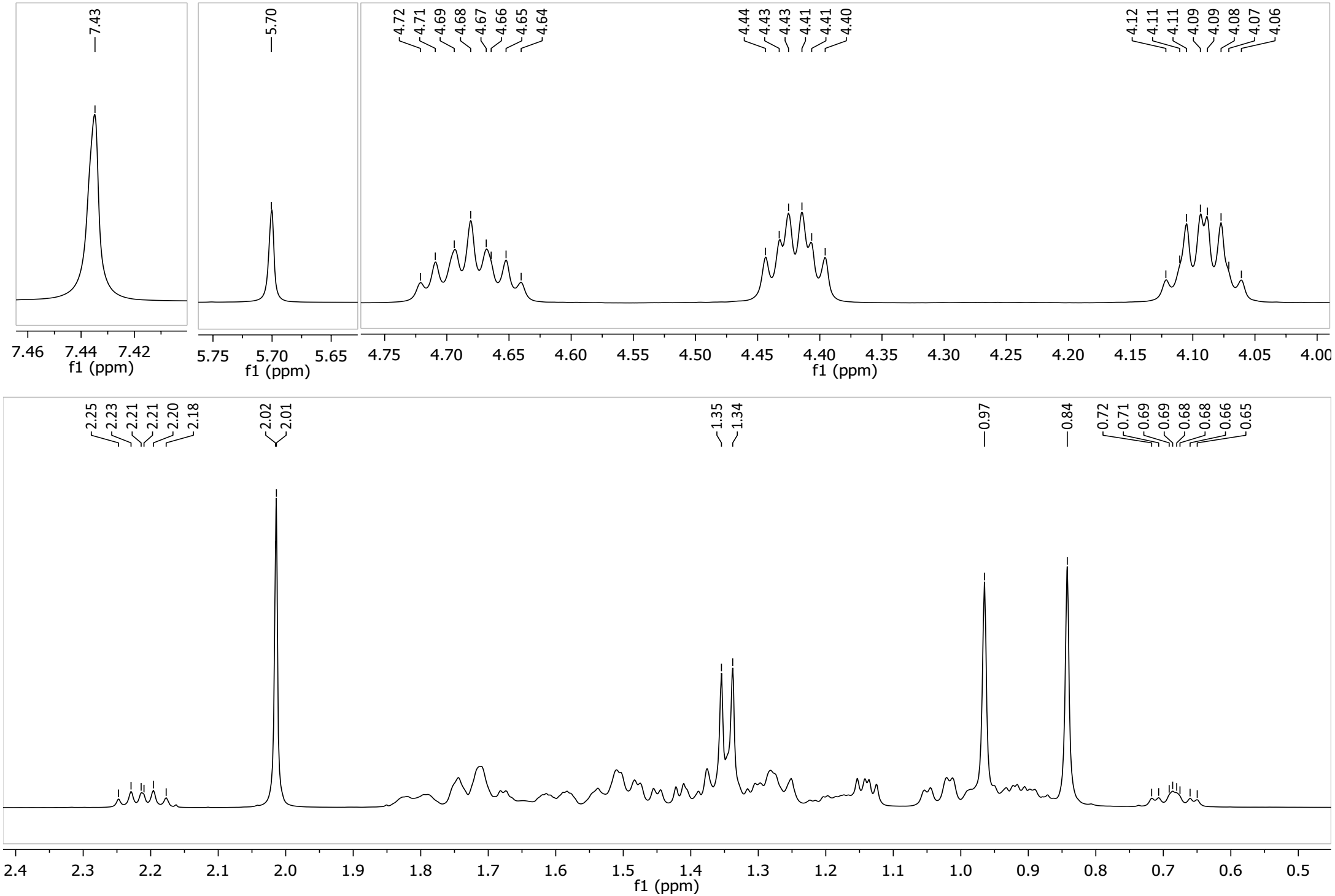


$C_{54}H_{78}O_8$
855.21
854.569670
C 75.84% H 9.19% O 14.97%

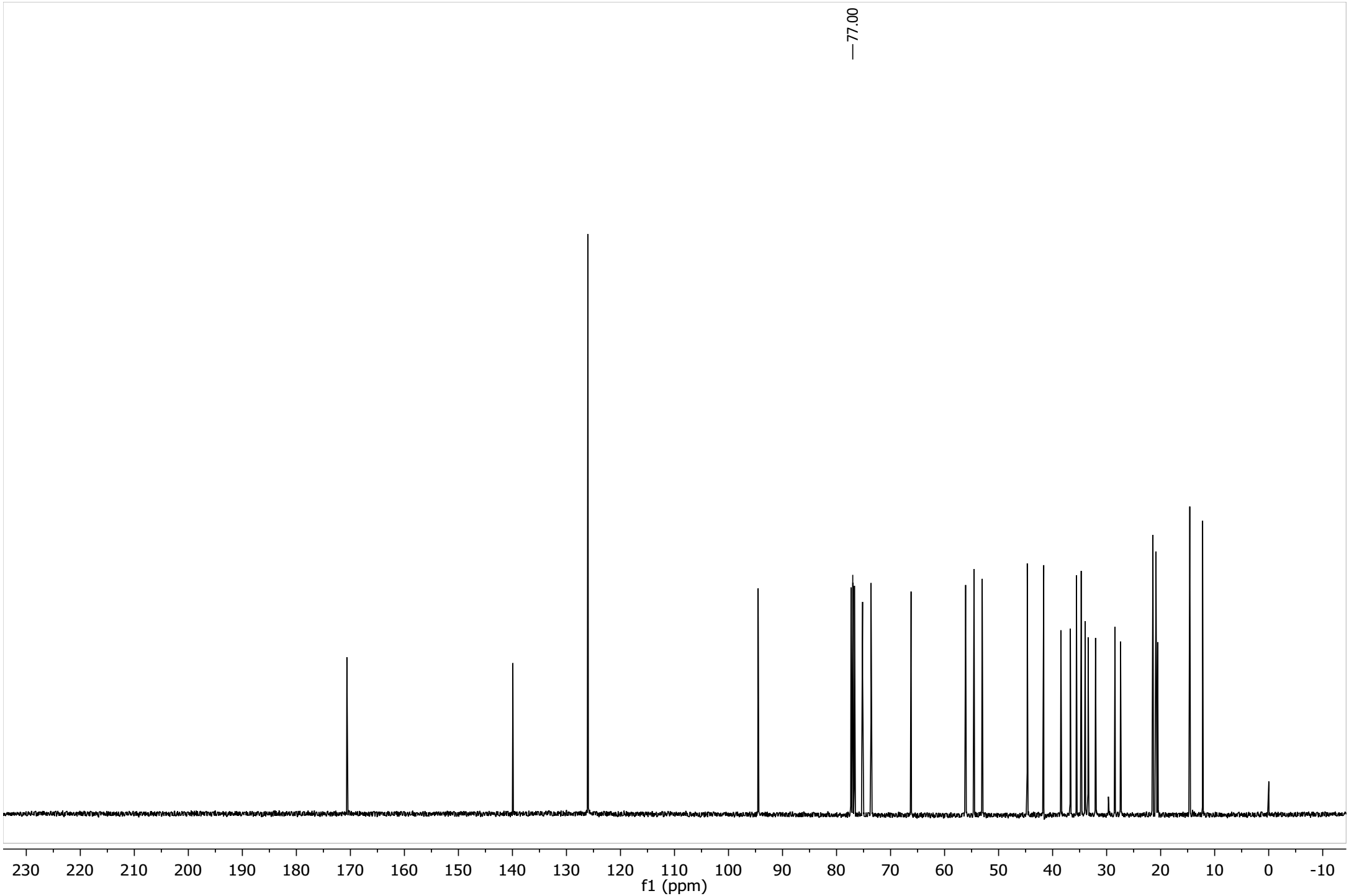
Dimer *RR*-18



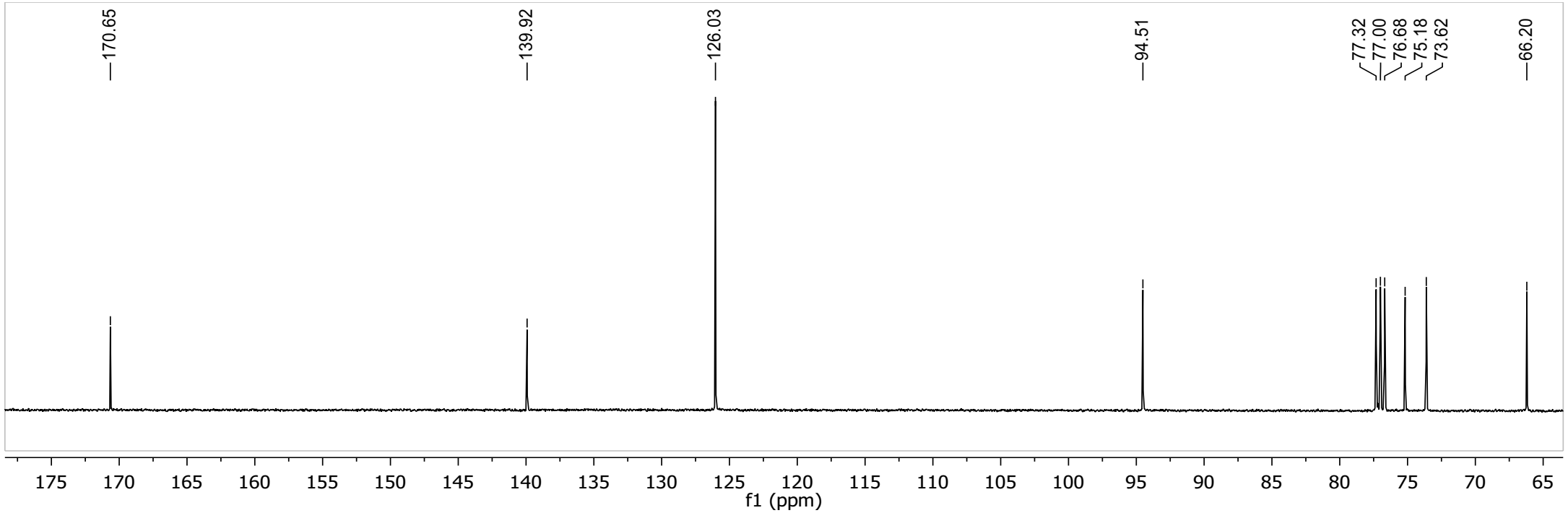
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound ***RR*-18**

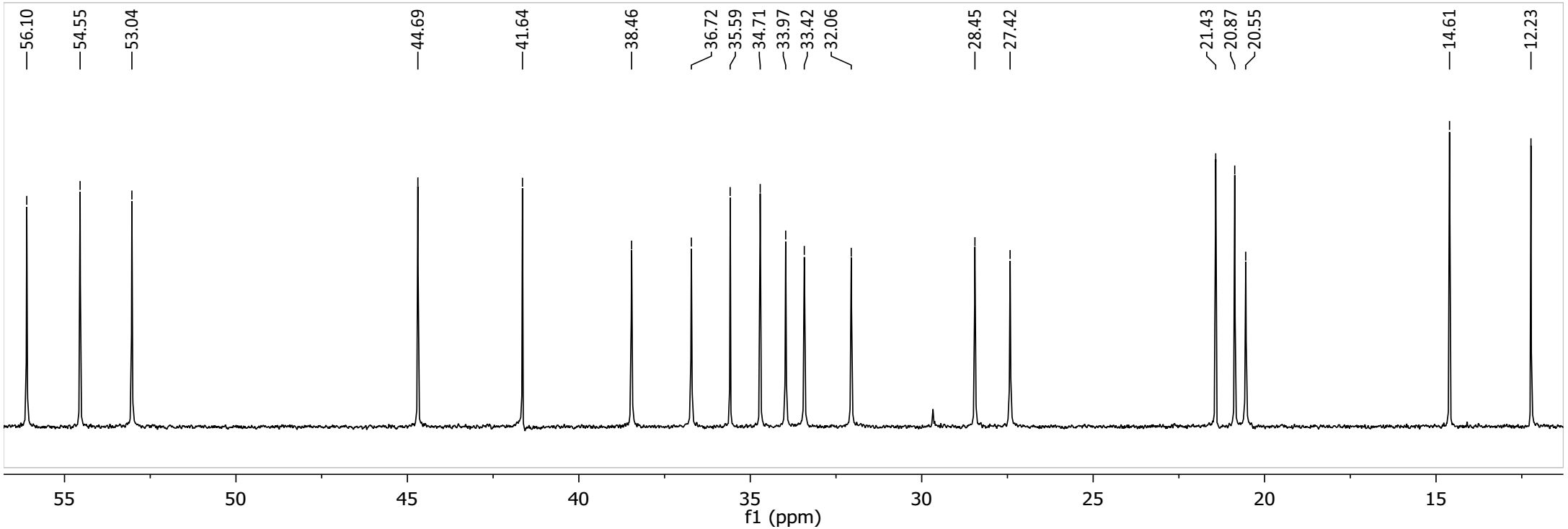


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound ***RR*-18**

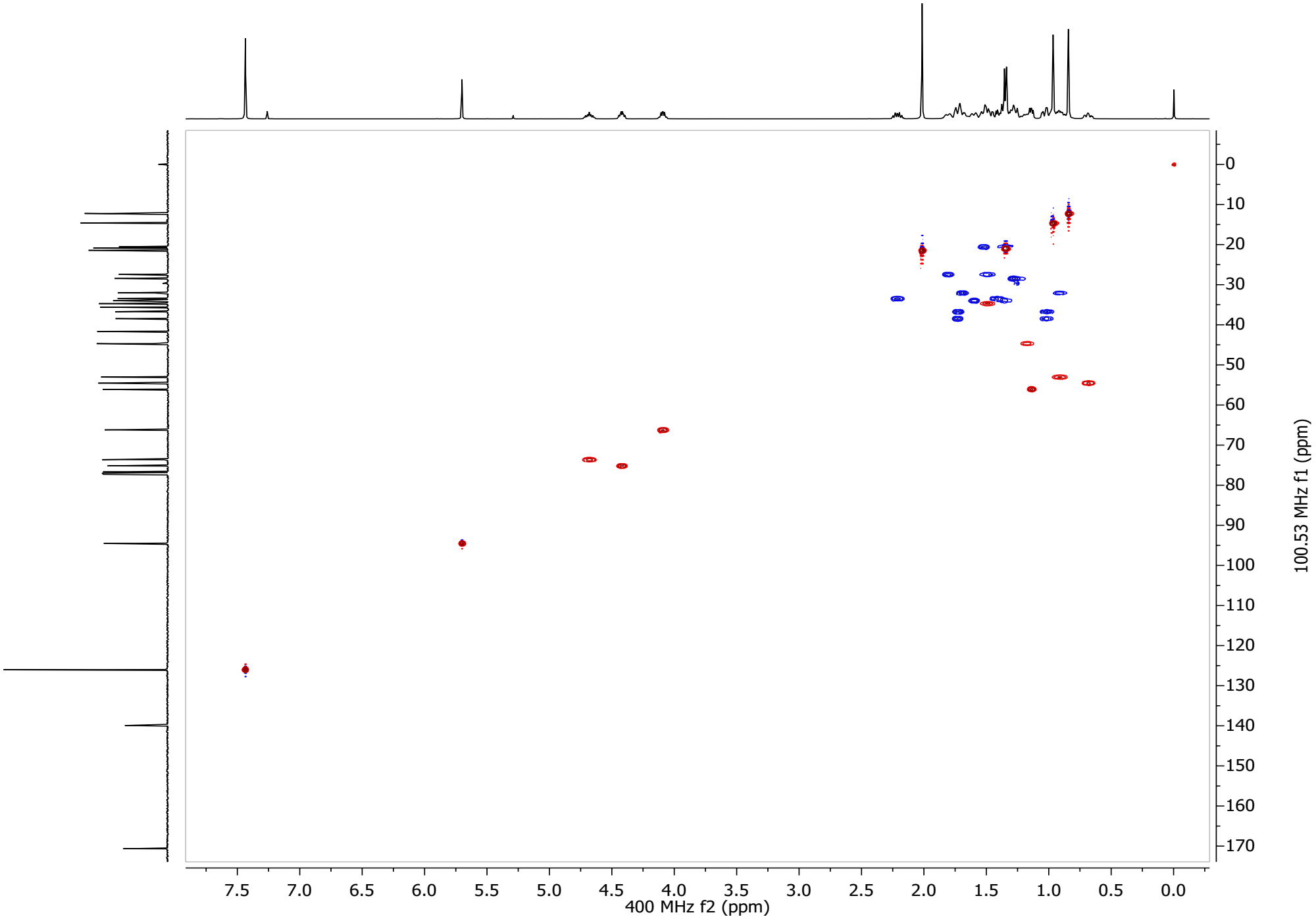


^{13}C NMR (100.53 MHz, $CDCl_3$) spectrum of compound ***RR*-18**

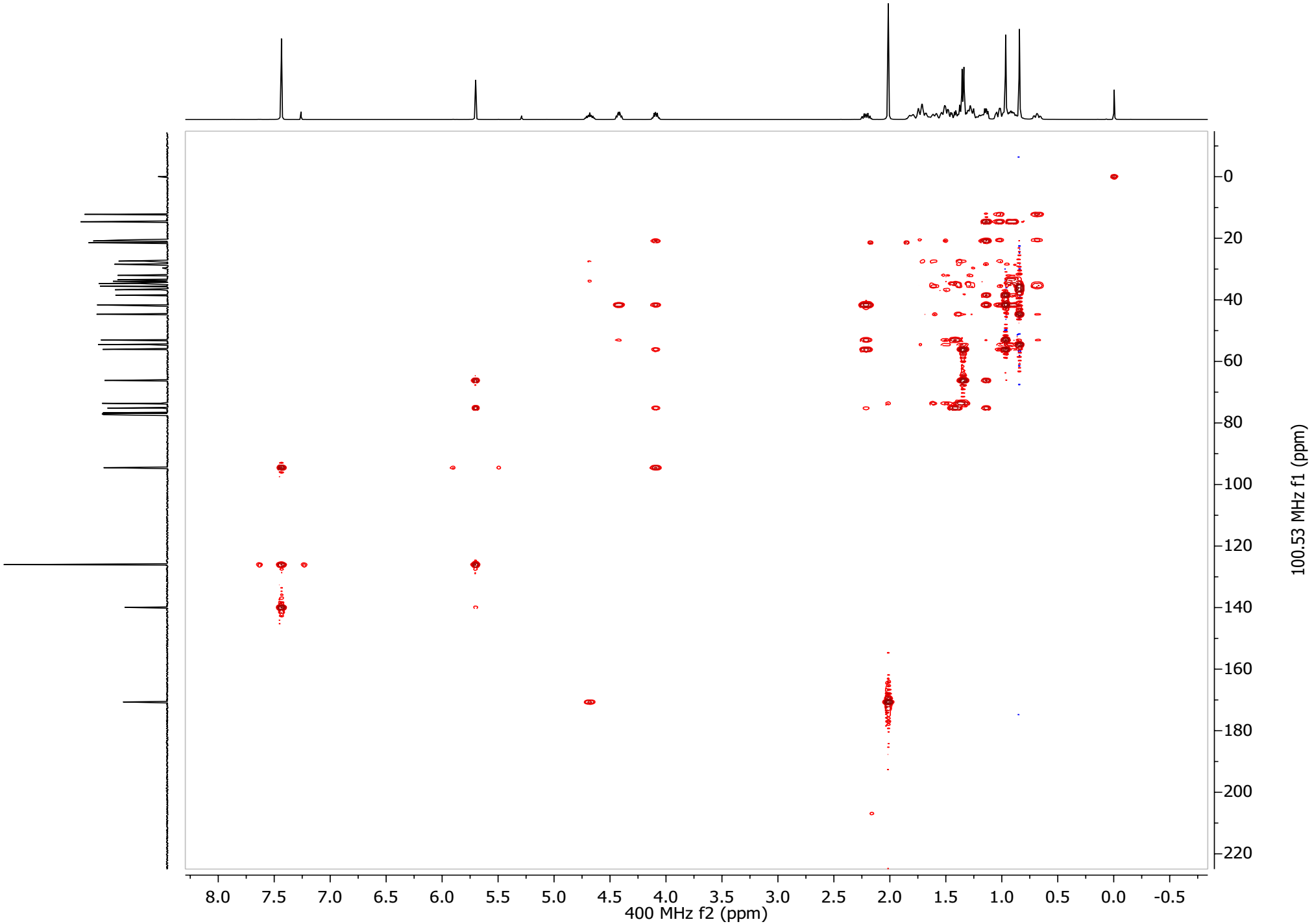




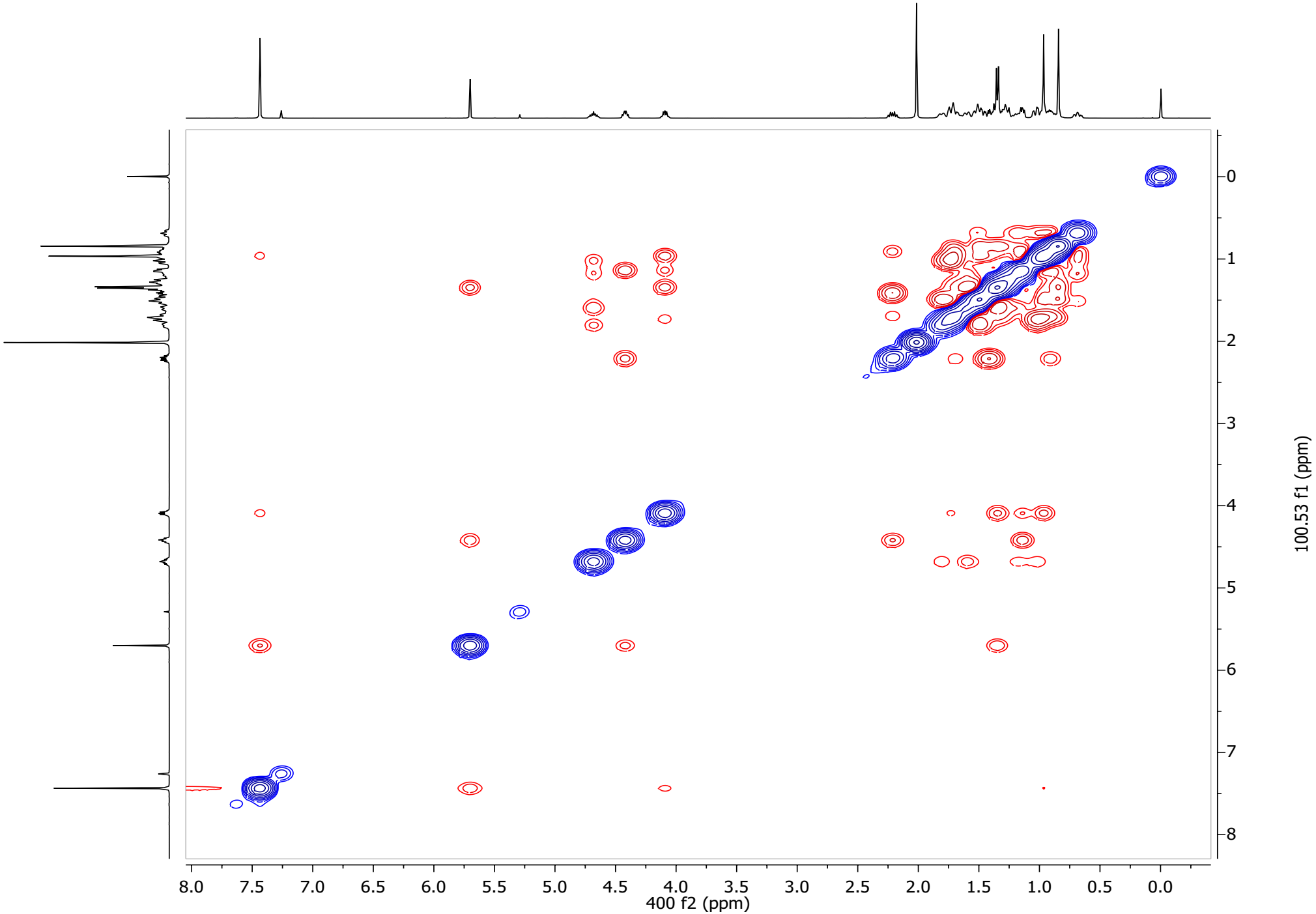
^{13}C NMR (100.53 MHz, $CDCl_3$) spectrum of compound ***RR*-18**



HSQC ($CDCl_3$) spectrum of compound ***RR*-18**

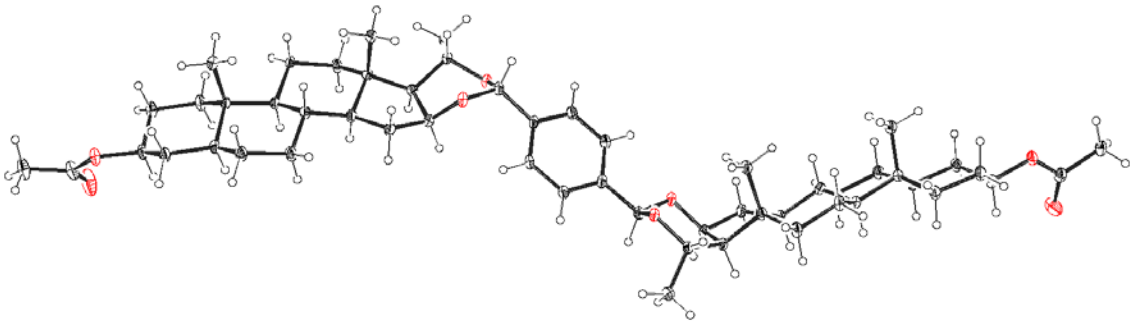


HMBC ($CDCl_3$) spectrum of compound ***RR*-18**



NOESY($CDCl_3$) spectrum of compound ***RR*-18**

A



B

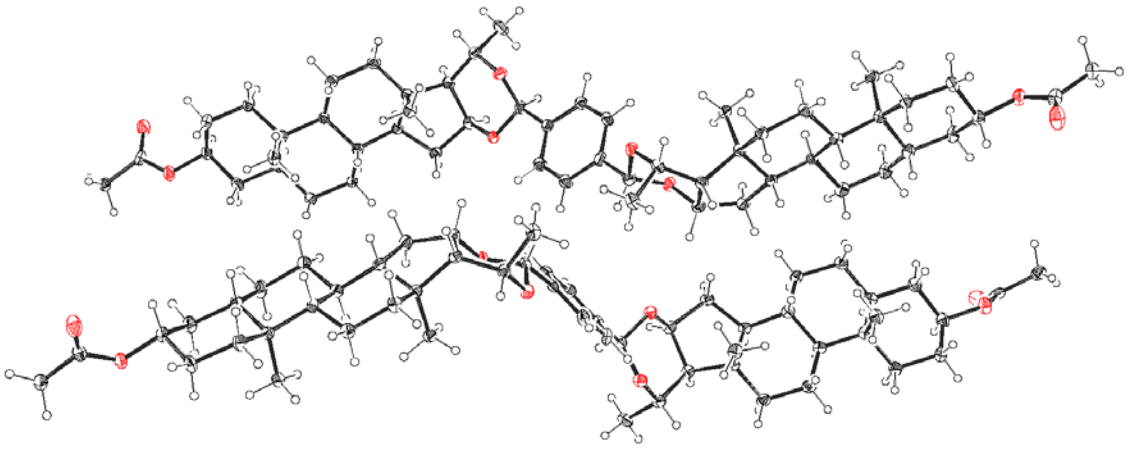


Figure S-1 Crystal structures of steroid dimers ***SR*-18 (A)** and ***RR*-18 (B)** with the thermal ellipsoids drawn at 30% probability.

Table S-1. Crystal data and structure refinement for compounds ***SR*-18** and ***RR*-18**.

Parameter	***SR*-18**	***RR*-18**
Empirical formula	$C_{54}H_{78}O_8$	$C_{54}H_{78}O_8$
Formula weight	855.16	855.16
Temperature	130(2) K	130(2) K
Wavelength	1.54184 Å	1.54184 Å
Crystal system	Monoclinic	Triclinic
Space group	$P\,2_1$	$P1$
Unit cell dimensions	a = 13.7565(7) Å	a = 13.4848(8) Å
	b = 10.3255(6) Å	b = 13.8175(9) Å
	c = 16.8608(7) Å	c = 14.4535(10) Å
	α= 90°	α= 74.959(6)°
	β= 100.716(4)°	β= 88.351(5)°
	γ = 90°	γ = 65.812(6)°
Volume	2353.2(2) $Å^3$	2363.3(3) $Å^3$
Z	2	2
Density (calculated)	1.207 Mg/m^3	1.202 Mg/m^3
Absorption coefficient	0.624 mm^{-1}	0.621 mm^{-1}
F(000)	932	932
Crystal size	0.370 x 0.140 x 0.060 mm^3	0.410 x 0.290 x 0.150 mm^3
Theta range for data collection	3.817 to 77.853°.	3.607 to 78.091°.
Index ranges	-17<=h<=17, -12<=k<=10, -21<=l<=21	-16<=h<=16, -17<=k<=17, -18<=l<=18
Reflections collected	45982	45764
Independent reflections	9330 [R(int) = 0.1087]	18197 [R(int) = 0.0766]
Completeness to theta = 67.684°	100.0 %	100.0 %
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	9330 / 1 / 567	18197 / 3 / 1133
Goodness-of-fit on F^2	1.040	1.065
Final R indices [I>2sigma(I)]	R1 = 0.0563, wR2 = 0.1199	R1 = 0.0595, wR2 = 0.1210
R indices (all data)	R1 = 0.0869, wR2 = 0.1397	R1 = 0.0920, wR2 = 0.1445
Absolute structure parameter	0.13(18)	-0.02(14)
Largest diff. peak and hole	0.200 and -0.263 e.$Å^{-3}$	0.234 and -0.343 e.$Å^{-3}$

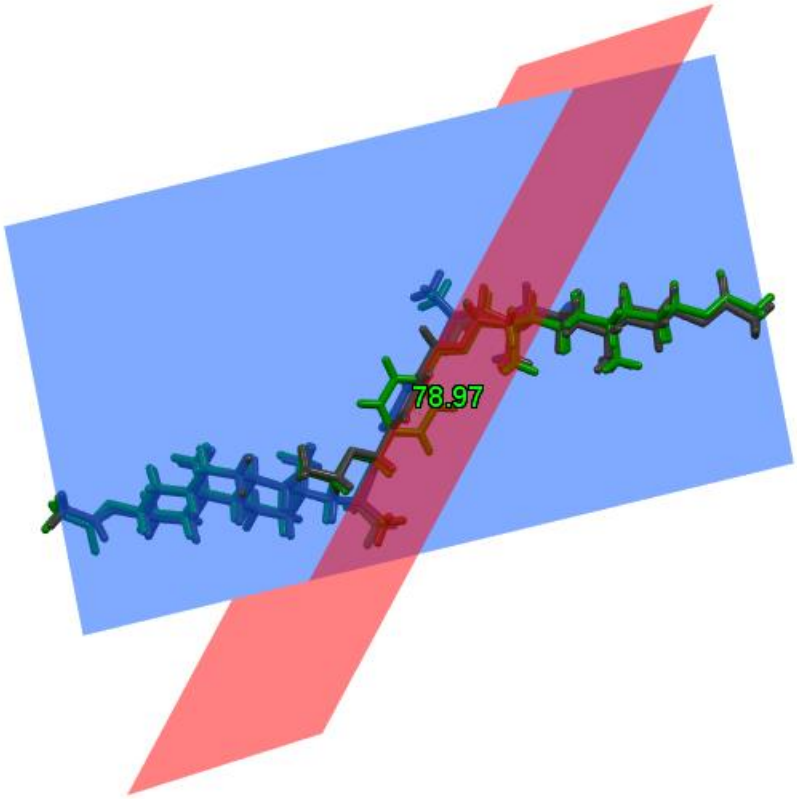


Figure S2. Overlay of the two symmetry independent molecules in the asymmetric unit of ***RR*-18** highlighting the differences in the conformation of the molecules.

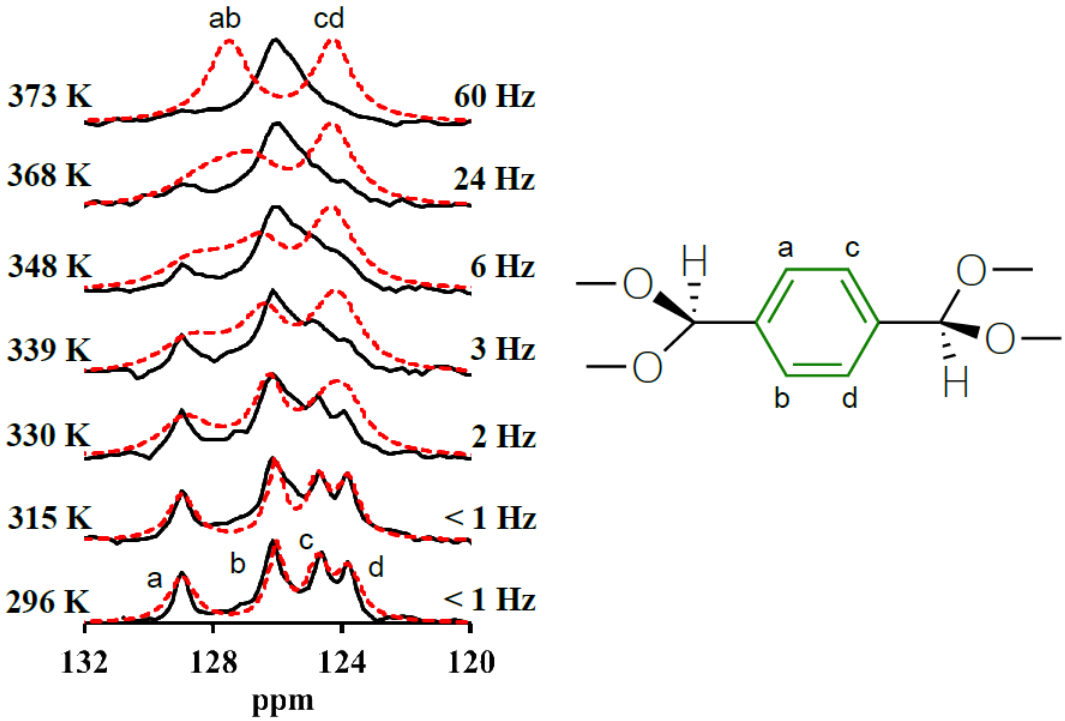


Figure S3. Alternative dynamic model for the motion of ***RR*-18.** The simulated line shapes (dotted red line) do not match the evolution of the experimental ^{13}C CPMAS (75.47 MHz) peaks at higher temperatures (solid black line).