

Glycoluril Dimers Bearing Hydrogen Atoms on Their Convex Face and Their Self-Assembly in the Solid State – Supporting Information

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General Experimental. Starting materials were purchased from commercial suppliers and were used without further purification.

Table S1. Crystal data and refinement parameters.

	1d	2c	3c
Empirical formula	C ₁₂ H ₂₀ N ₈ O ₄	C ₂₆ H ₂₈ N ₈ O ₆	C ₁₆ H ₂₄ N ₈ O ₆
Formula weight	340.36	548.56	424.43
Crystal system	Orthorhombic	Triclinic	Monoclinic
Space group	Pnn2	P-1	P2 ₁ /c
a [Å]	11.238(2)	11.635(1)	14.237(1)
b [Å]	16.985(3)	14.530(1)	11.571(1)
c [Å]	4.113(1)	16.385(1)	11.872(1)
α [°]	90	67.621(2)	90
β [°]	90	79.274(2)	112.698(9)
γ [°]	90	83.560(2)	90
Volume [Å ³]	785.1(3)	2514.1(1)	1804.3(2)
Z	2	4	4
Calc. density [Mg/m ³]	1.440	1.449	1.562
μ [mm ⁻¹]	0.111	0.108	0.122
Crystal size [mm]	0.25 x 0.10 x 0.07	0.40 x 0.40 x 0.20	0.40 x 0.40 x 0.30
ϑ range [°]	3.63 to 25.00	2.61 to 25.00	3.35 to 25.00
Refl. collected / unique	5548 / 1563	30368 / 8820	15053 / 3174
Data / parameters	1563 / 120	8820 / 771	3174 / 297
Final R indices [I>2σ(I)]	0.0617, 0.1242	0.0524, 0.1507	0.0324, 0.0870
Δρ _{max} /Δρ _{min} [e. Å ⁻³]	0.231 / -0.223	1.811 / -0.650	0.193 / -0.214

Figure S1. ^1H NMR spectrum of **1c** (300 MHz, $\text{DMSO-}d_6$)

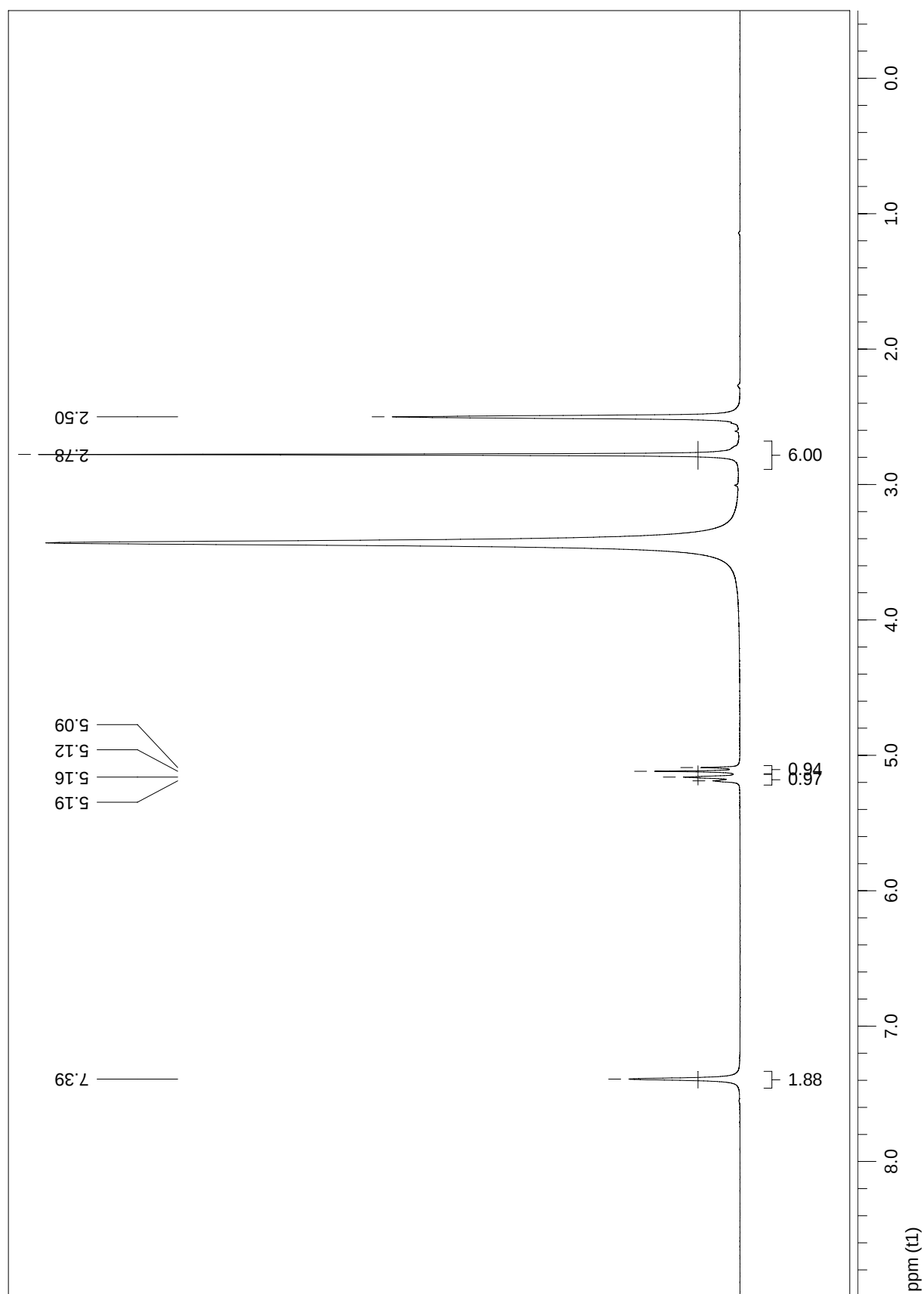


Figure S2. ^{13}C NMR APT spectrum of **1c** (75 MHz, $\text{DMSO-}d_6$)

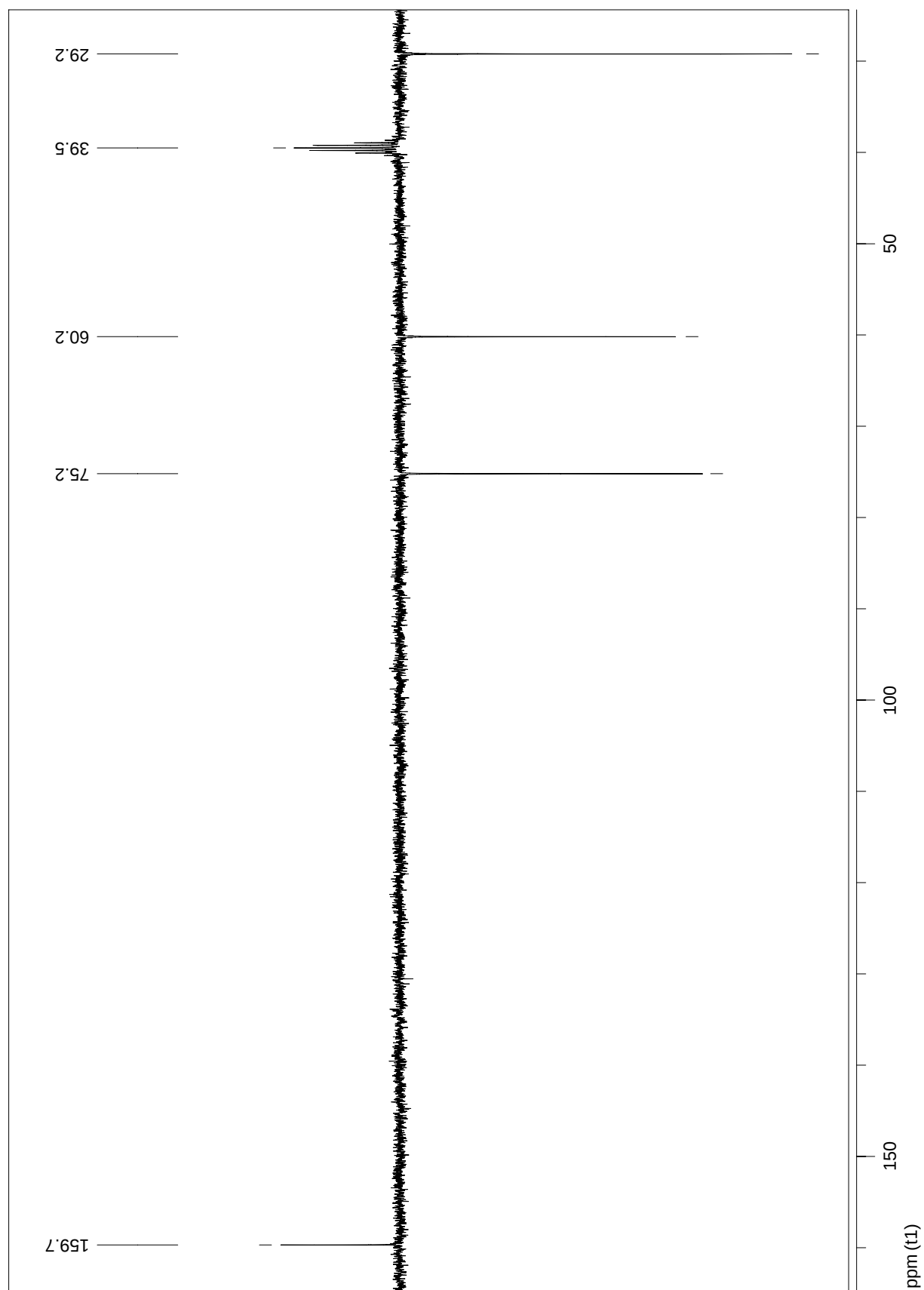


Figure S3. ^1H NMR spectrum of **1d** (300 MHz, $\text{DMSO}-d_6$)

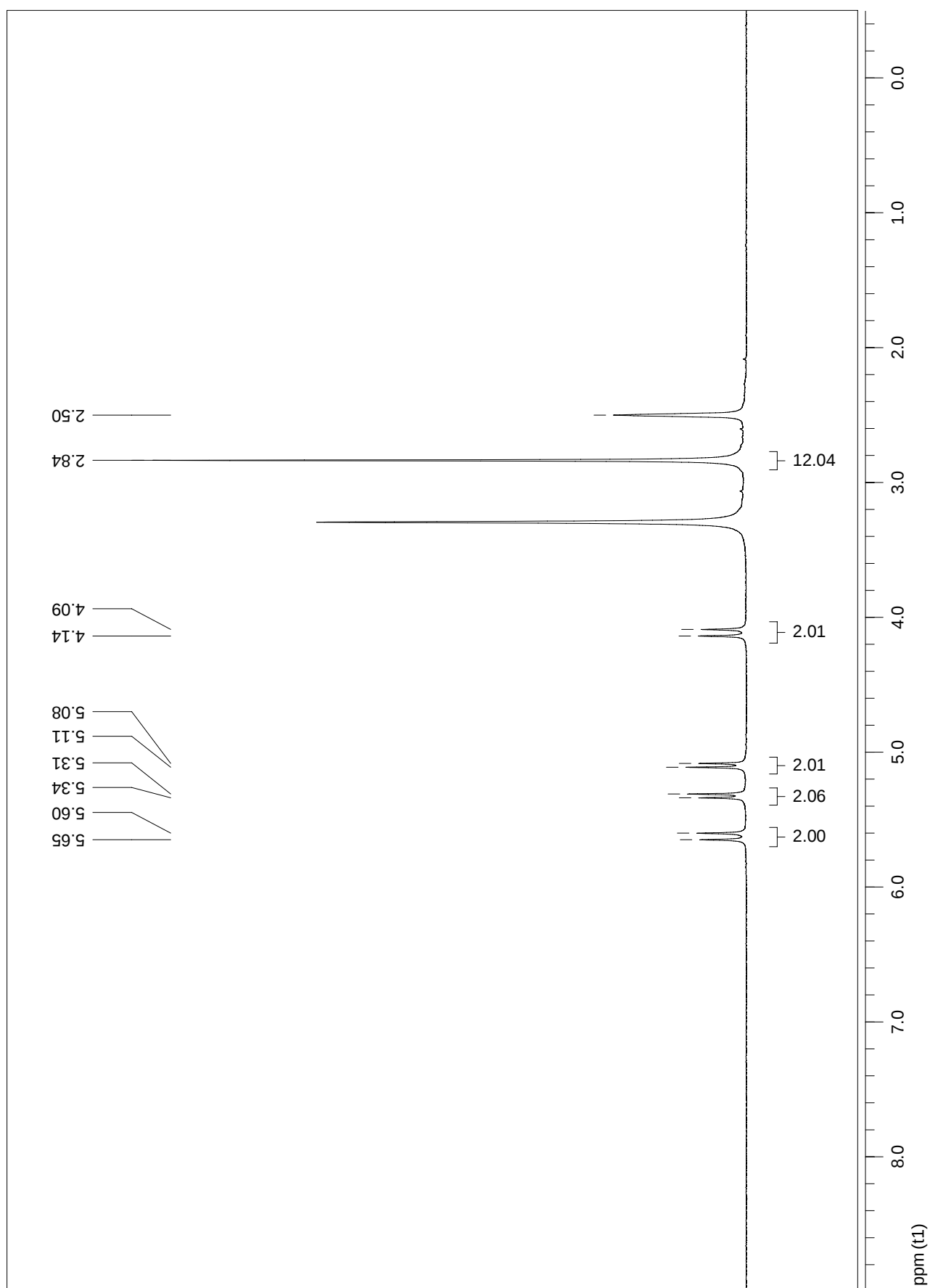


Figure S4. ^{13}C NMR APT spectrum of **1d** (75 MHz, $\text{DMSO-}d_6$)

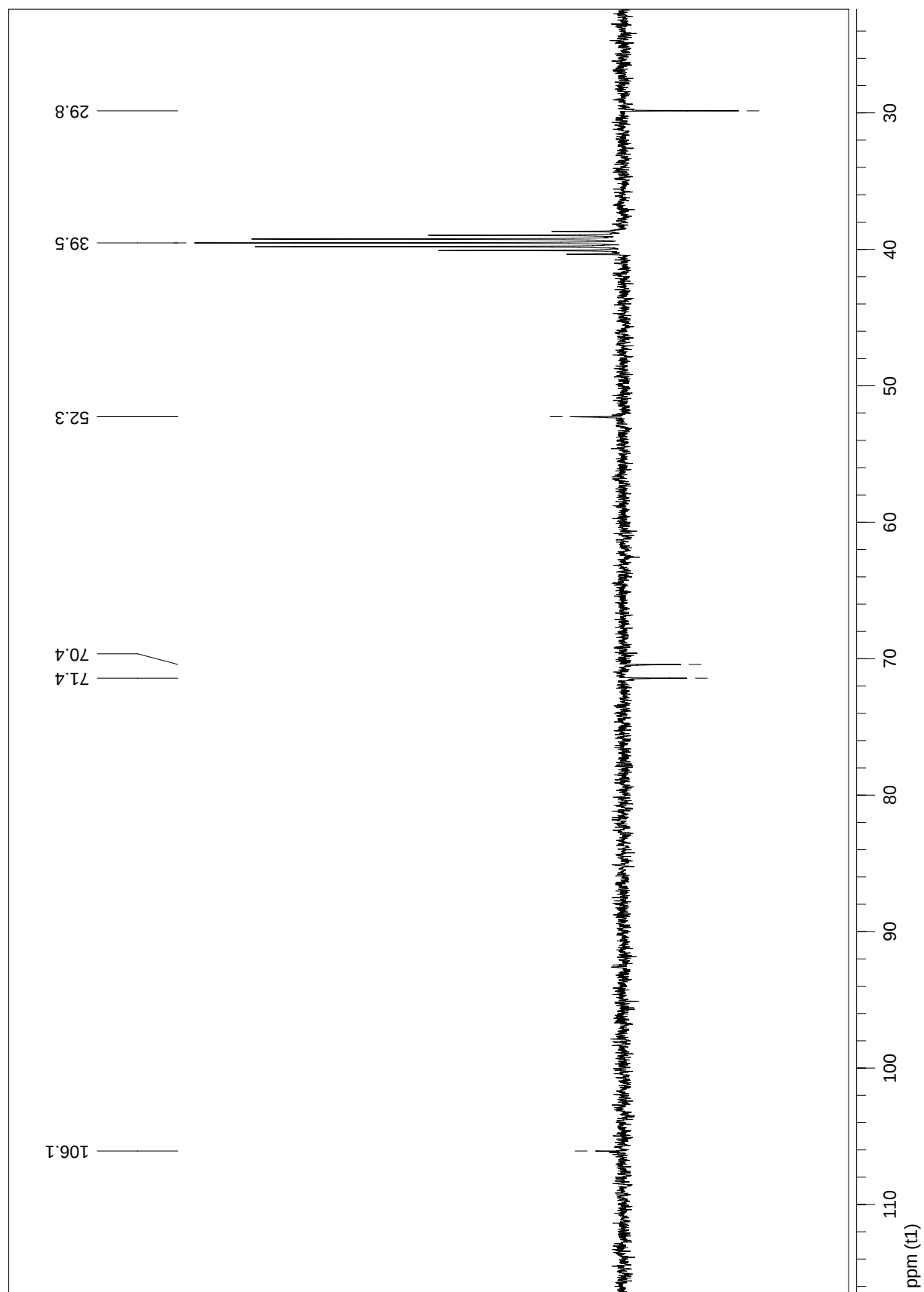


Figure S5. ^1H NMR spectrum of **2a** (300 MHz, $\text{DMSO}-d_6$)

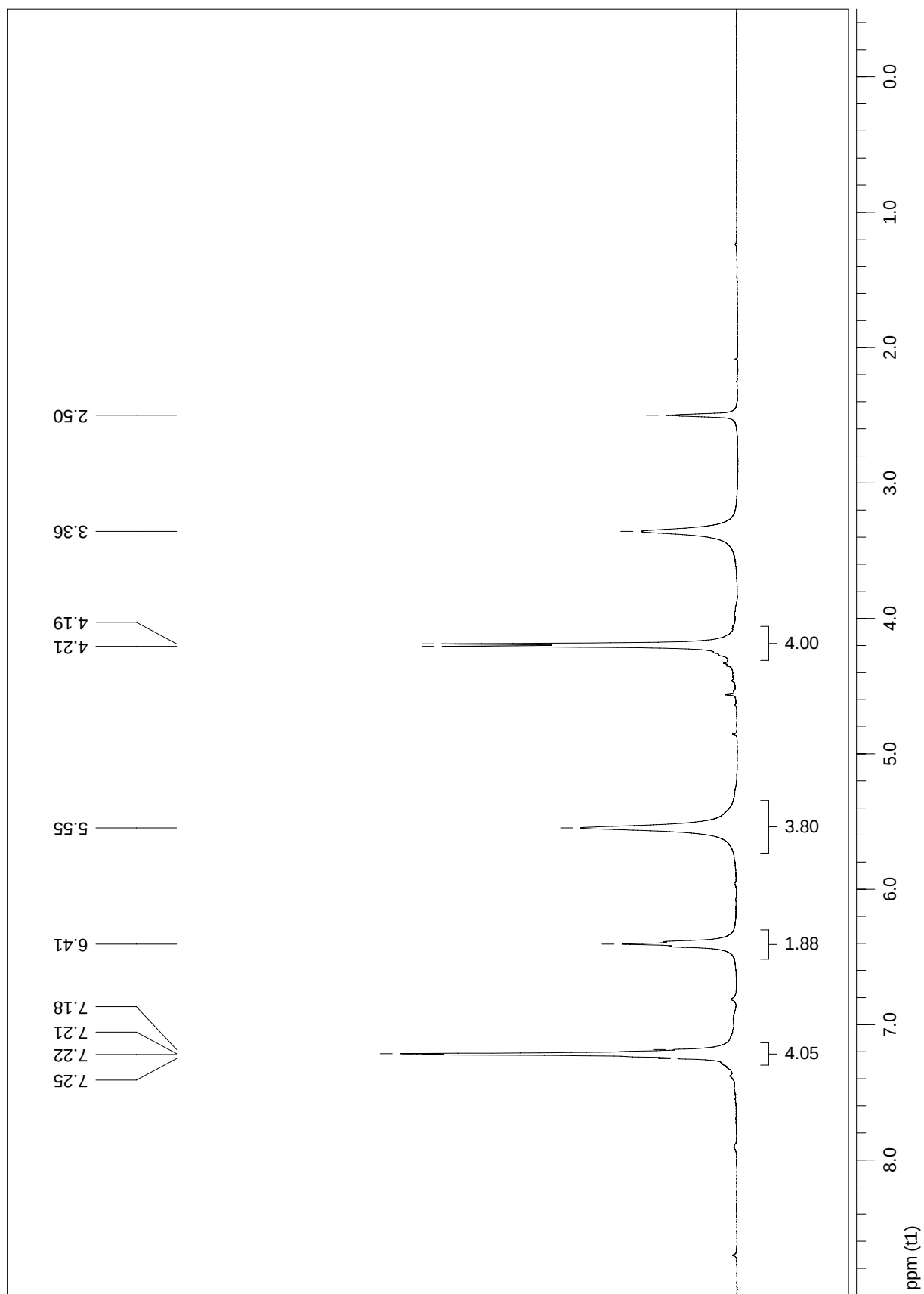


Figure S6. ^{13}C NMR APT spectrum of **2a** (75 MHz, $\text{DMSO-}d_6$)

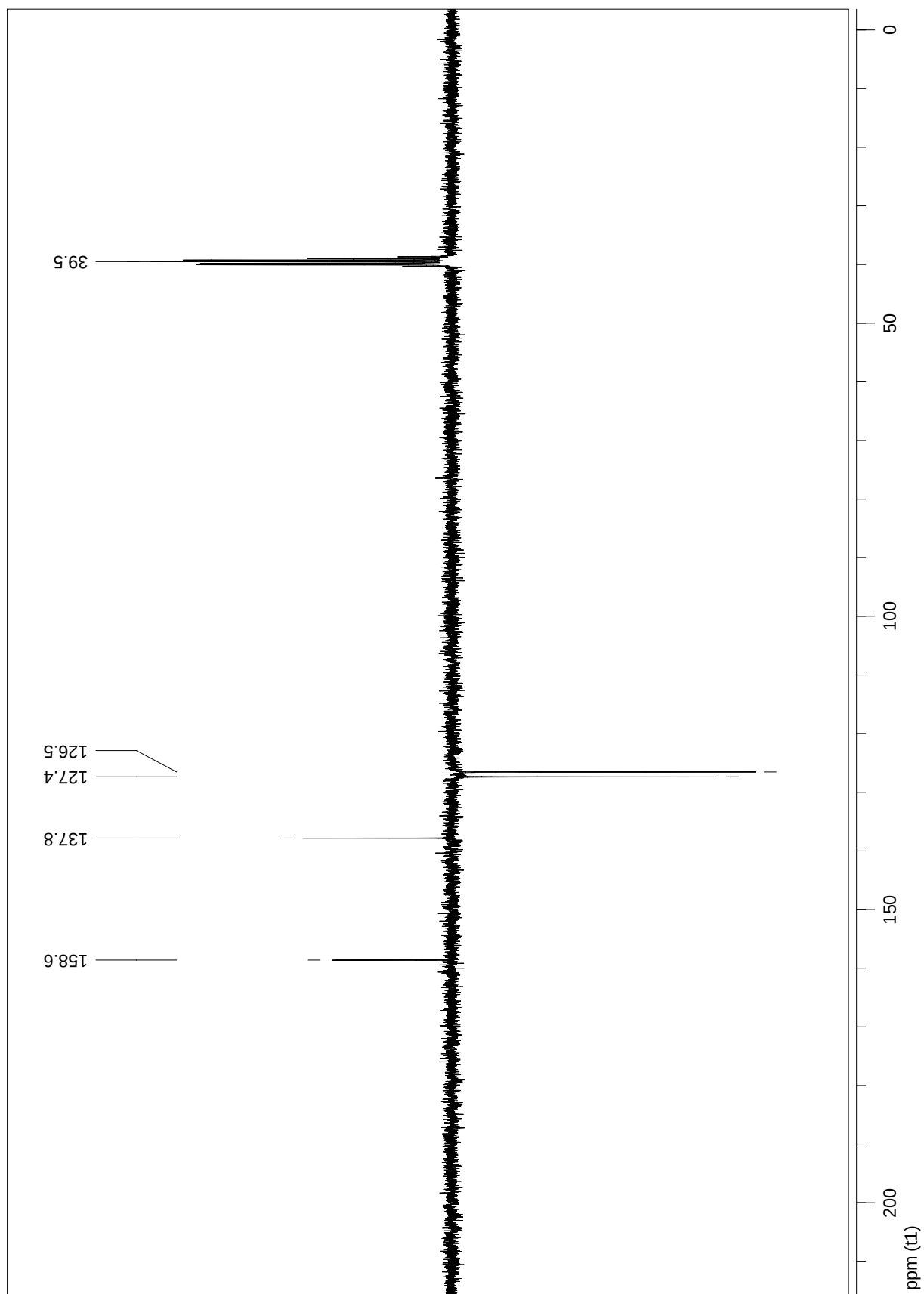


Figure S7. ^1H NMR spectrum of **2b** (300 MHz, $\text{DMSO}-d_6$)

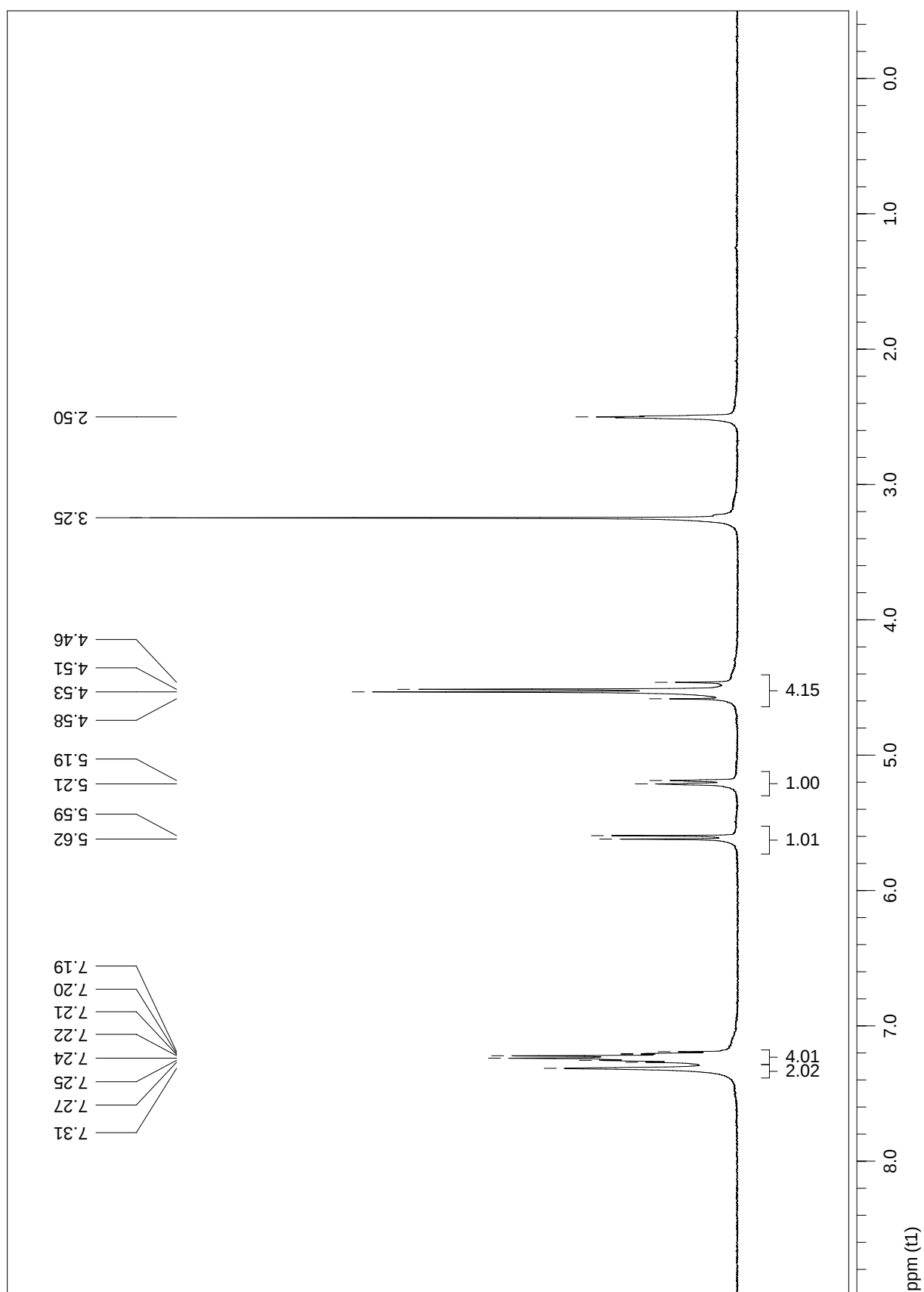


Figure S8. ^{13}C NMR spectrum of **2b** (75 MHz, $\text{DMSO-}d_6$)

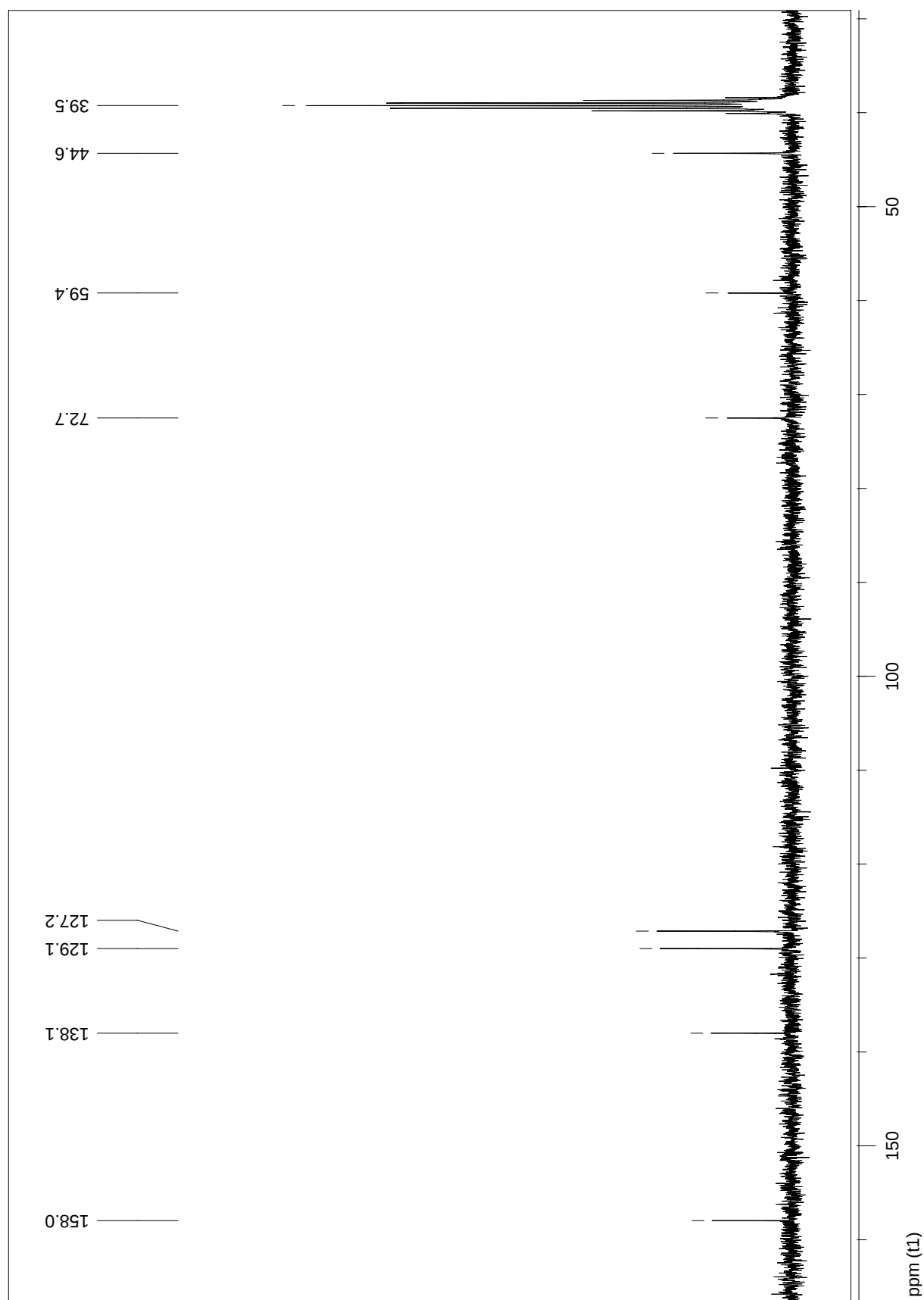


Figure S9. ^1H NMR spectrum of **2c** (300 MHz, $\text{DMSO}-d_6$)

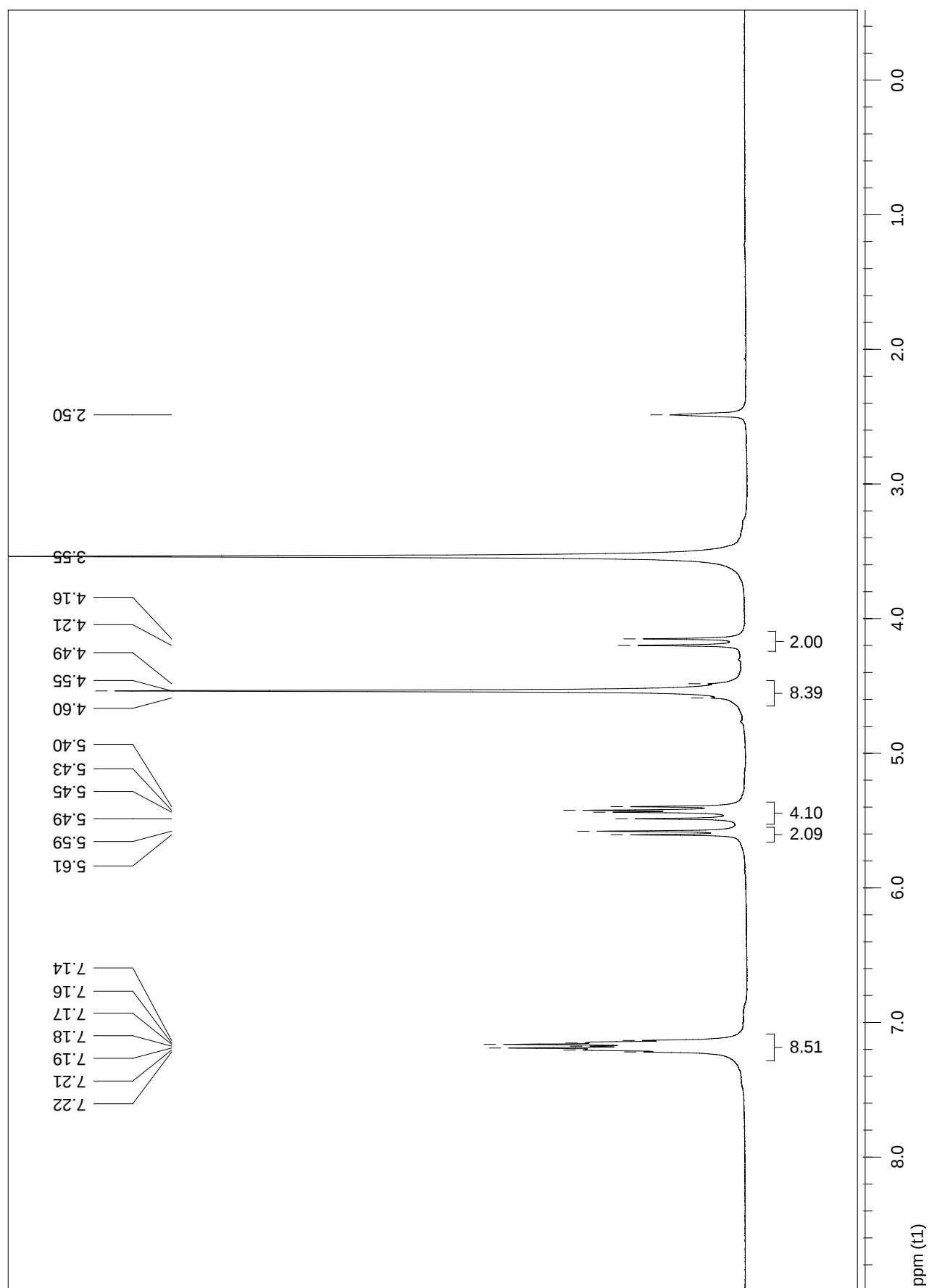


Figure S10. ^{13}C NMR APT spectrum of **2c** (75 MHz, $\text{DMSO-}d_6$)

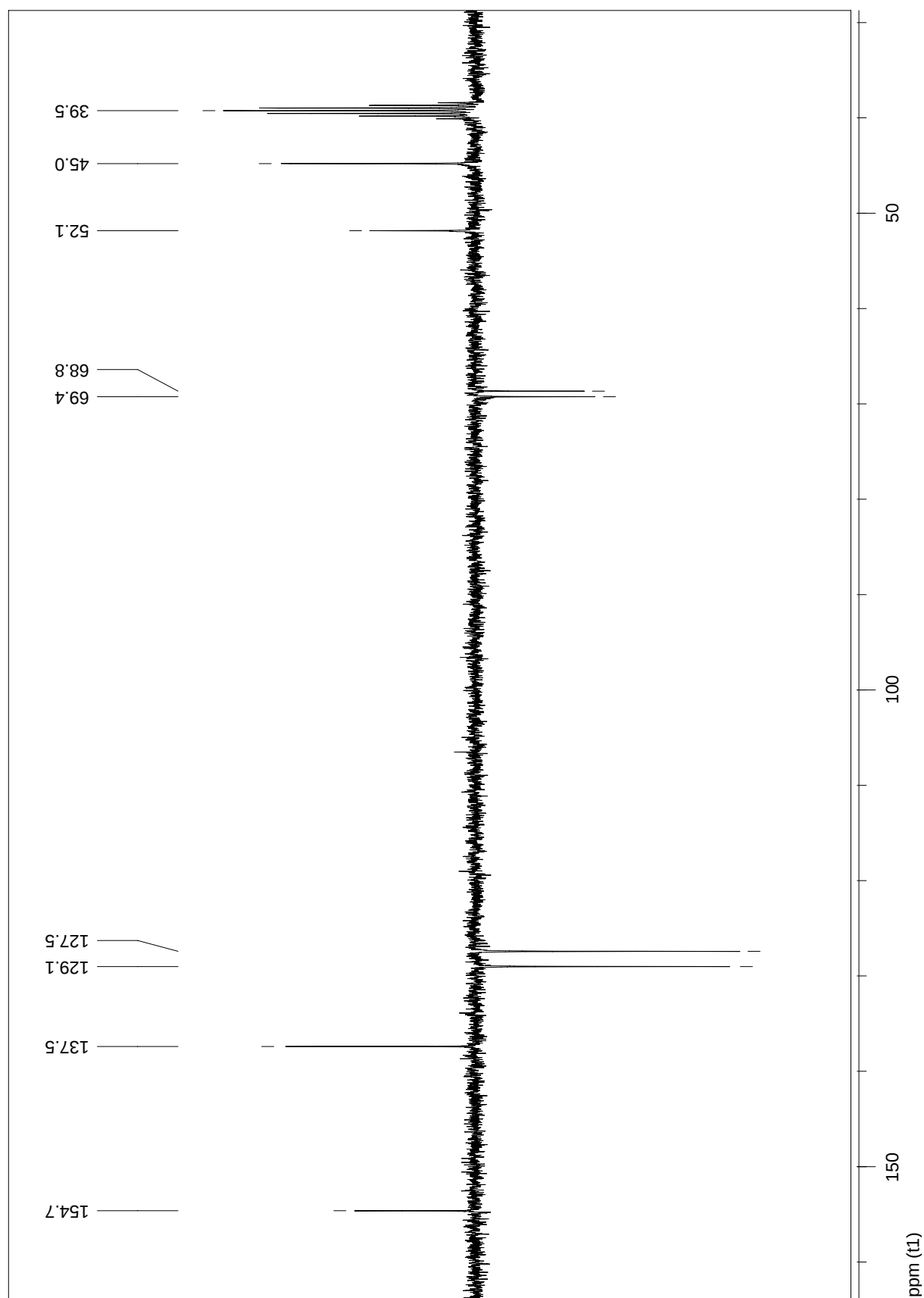


Figure S11. ^1H NMR spectrum of **3a** (300 MHz, $\text{DMSO}-d_6$)

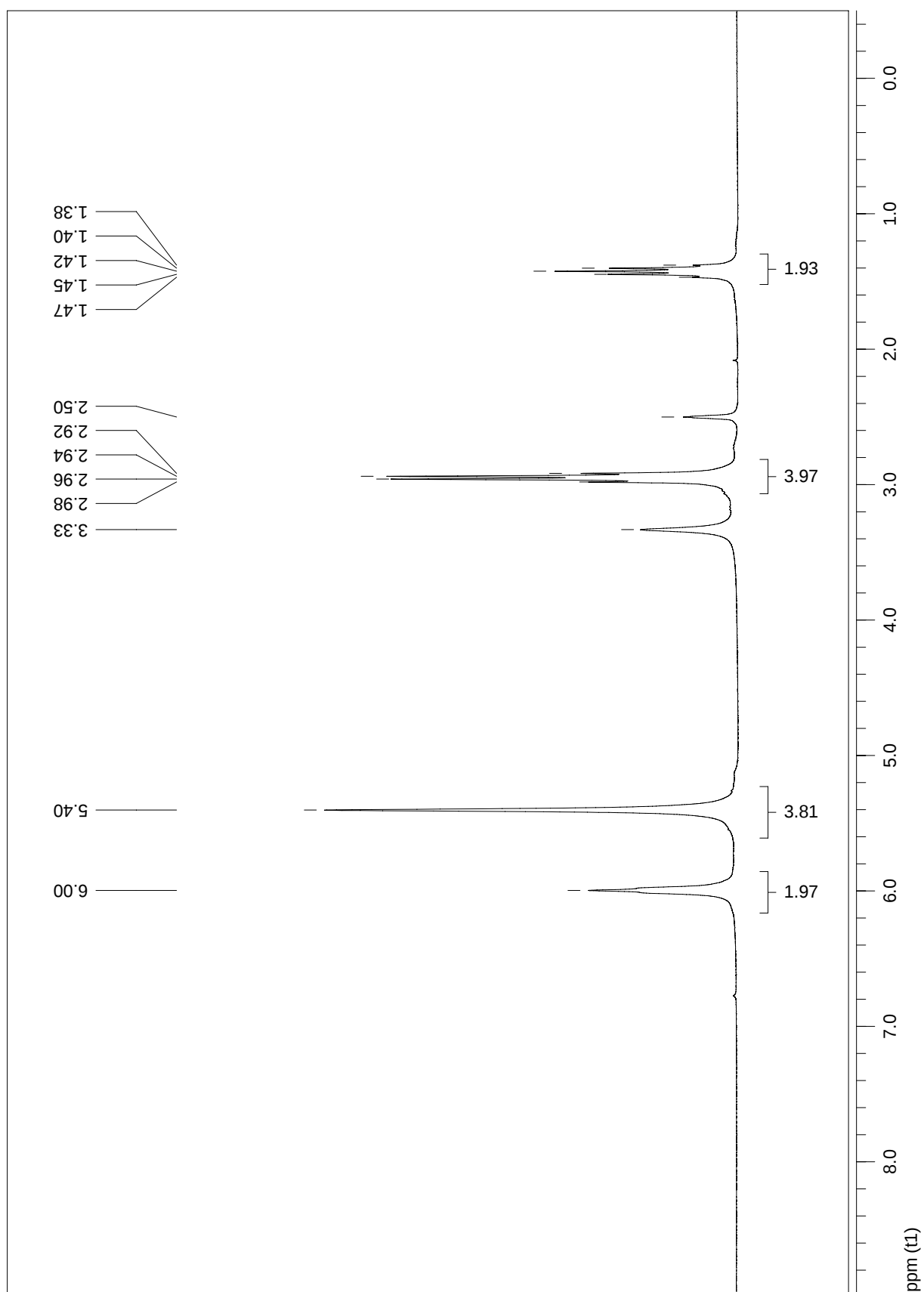


Figure S12. ^{13}C NMR APT spectrum of **3a** (75 MHz, $\text{DMSO}-d_6$)

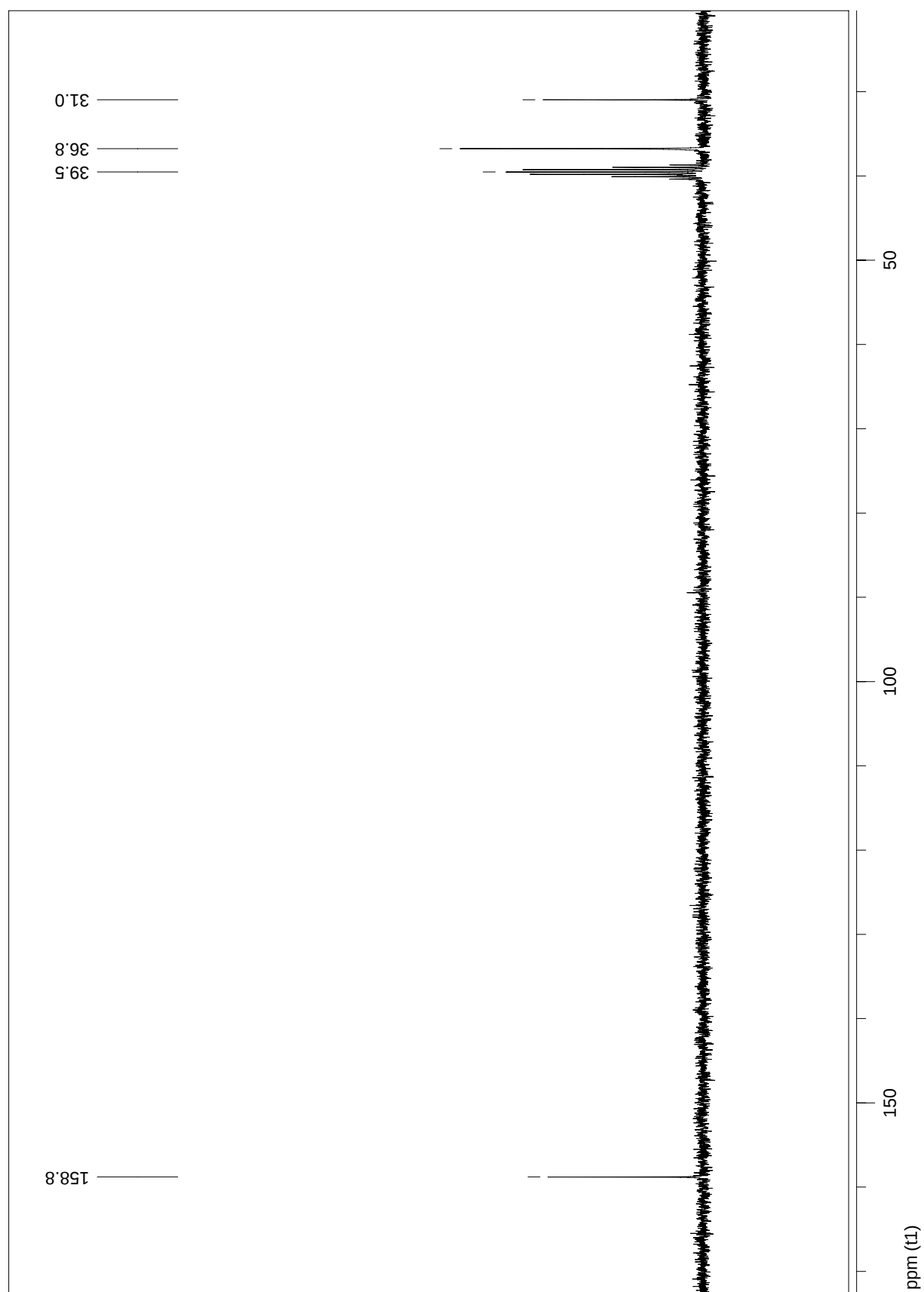


Figure S13. ^1H NMR spectrum of **3b** (300 MHz, $\text{DMSO}-d_6$)

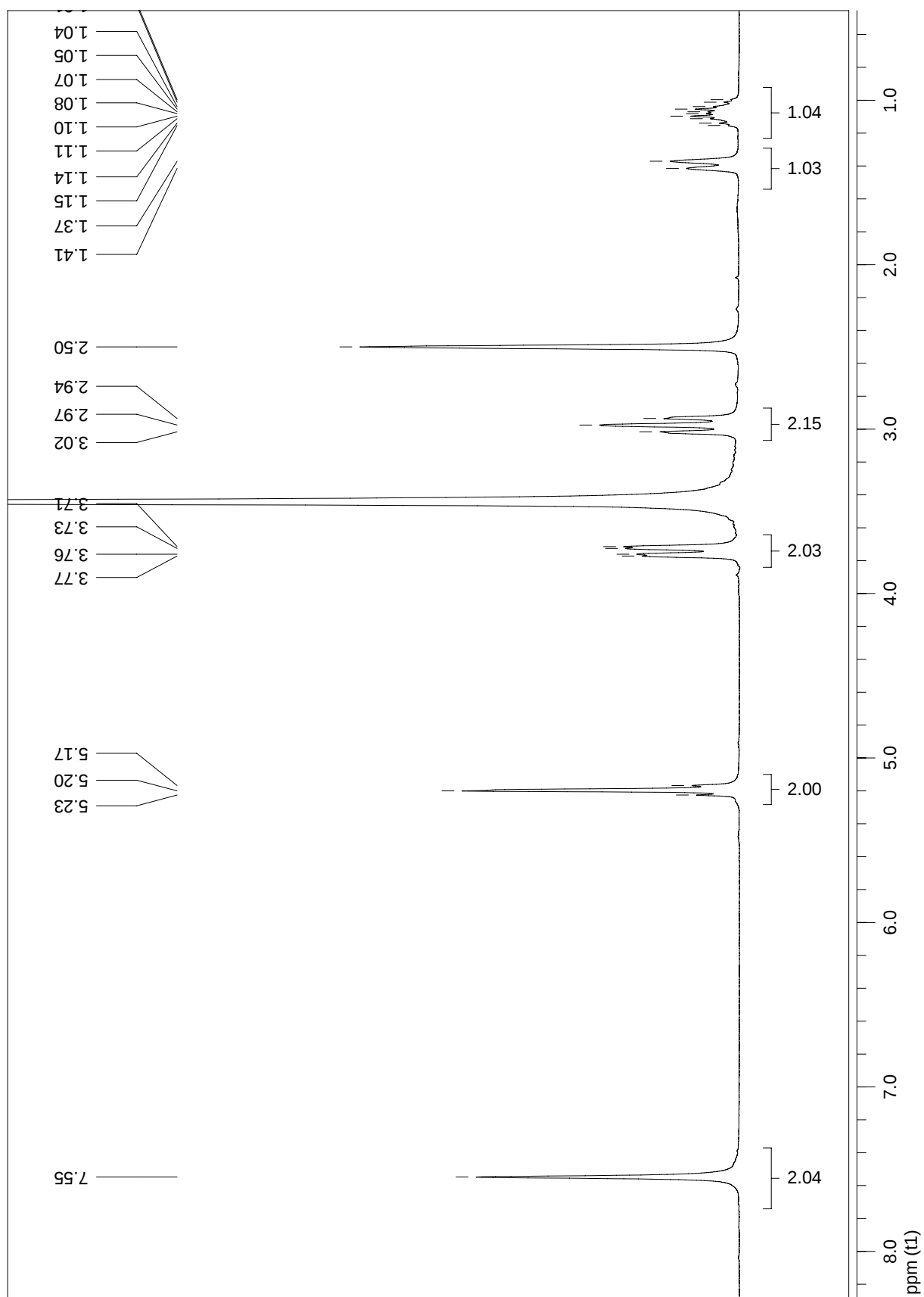


Figure S14. ^{13}C NMR APT spectrum of **3b** (75 MHz, $\text{DMSO-}d_6$)

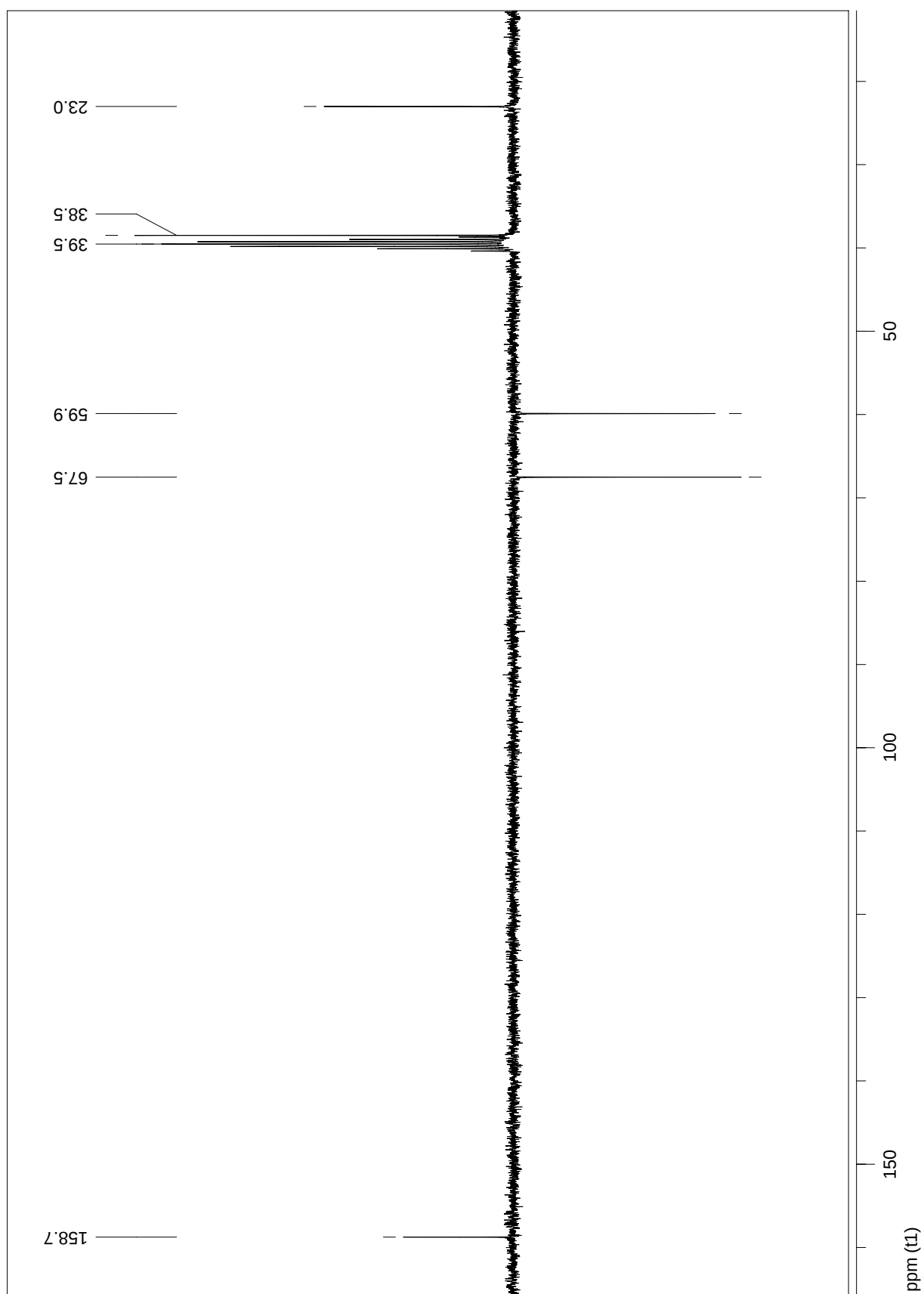


Figure S15. ^1H NMR spectrum of **3c** (300 MHz, $\text{DMSO}-d_6$)

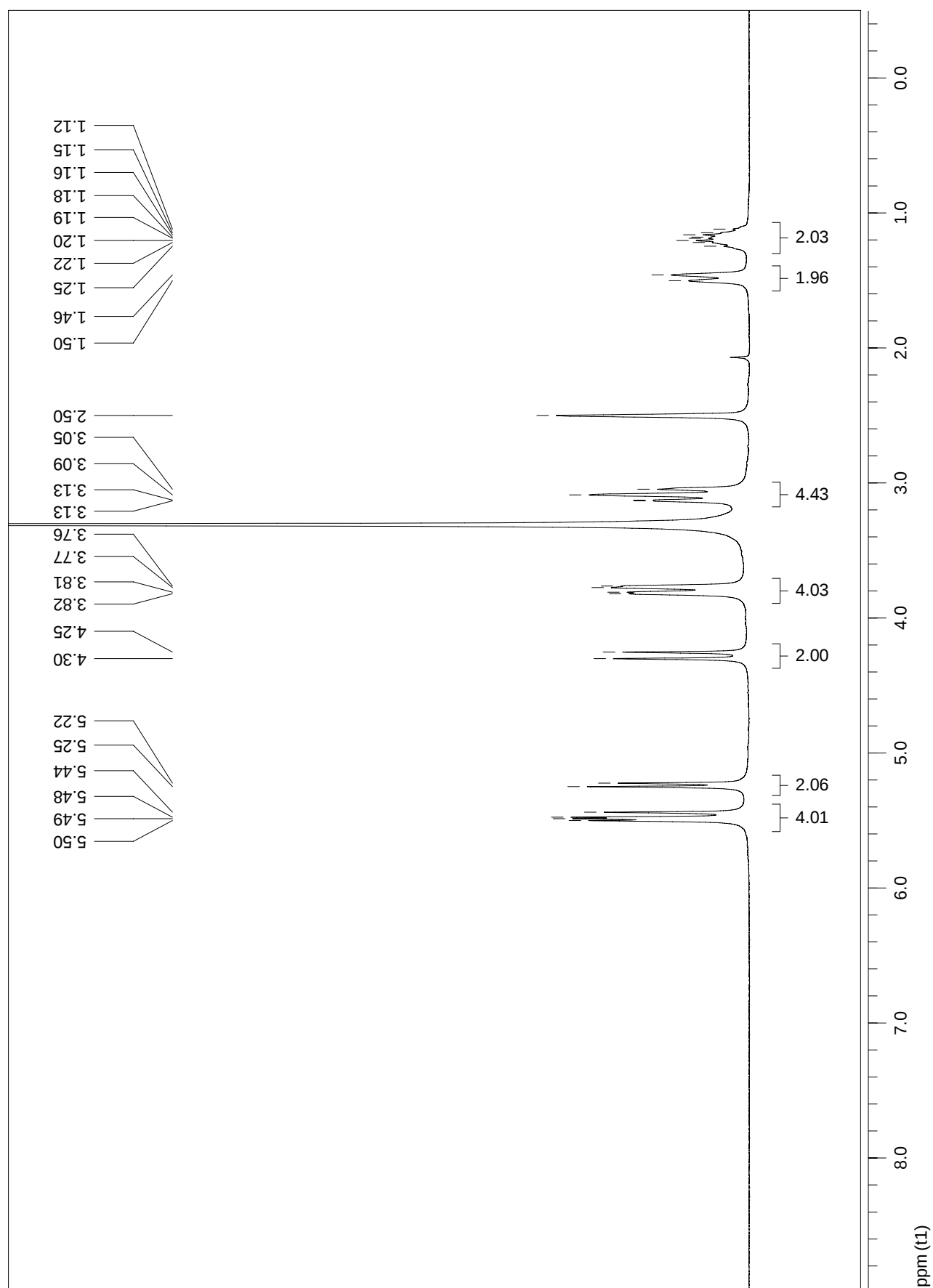


Figure S16. ^{13}C NMR APT spectrum of **3c** (75 MHz, $\text{DMSO-}d_6$)

