Supporting Informatiom

Tetraaryltetraanthra[2,3]porphyrins: synthesis, crystal structure and optical properties

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Table of Contents

NMR spectra

filatov

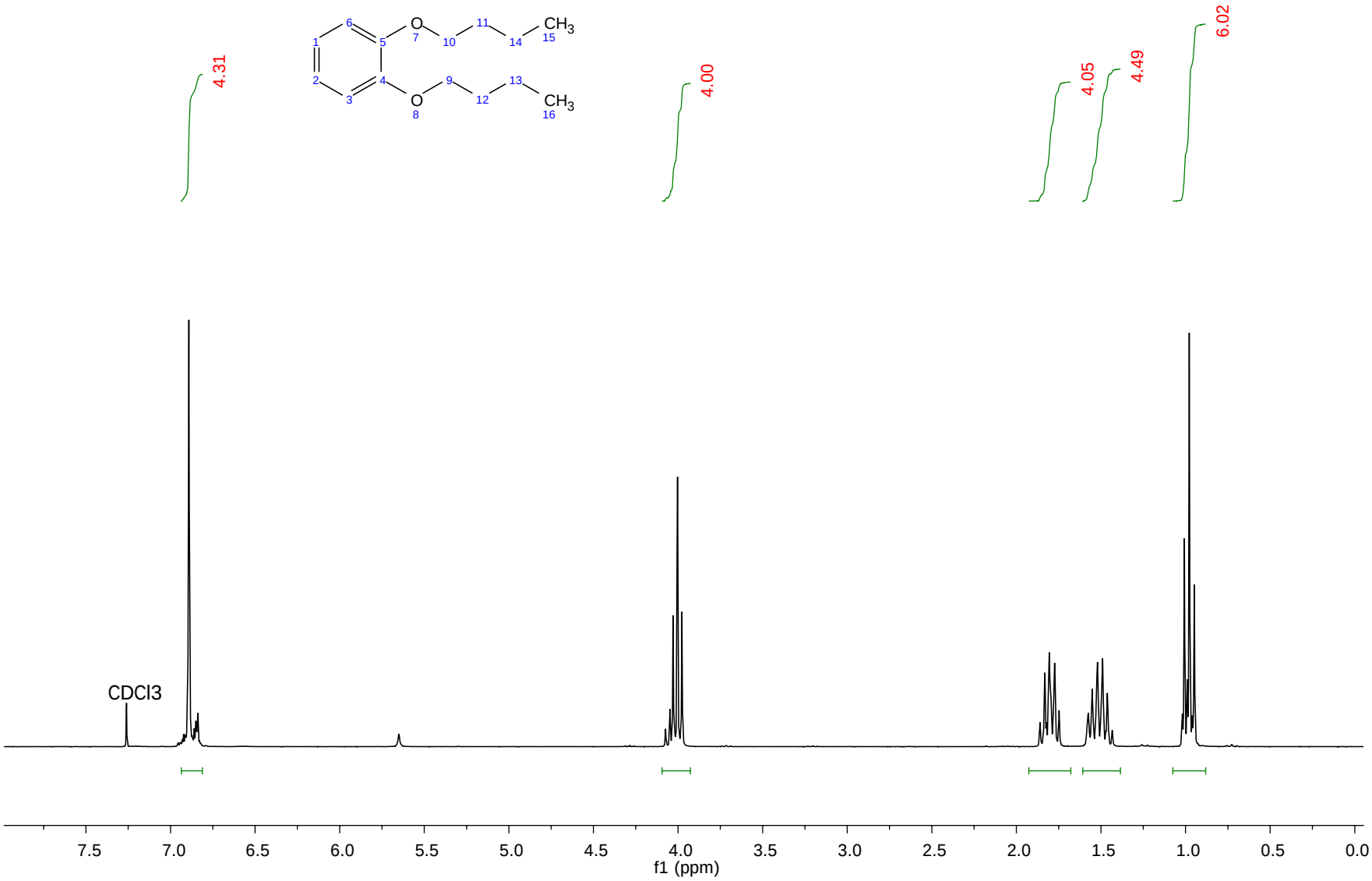


Figure S1. ^{1}H NMR spectrum for compound **7** in $CDCl_3$

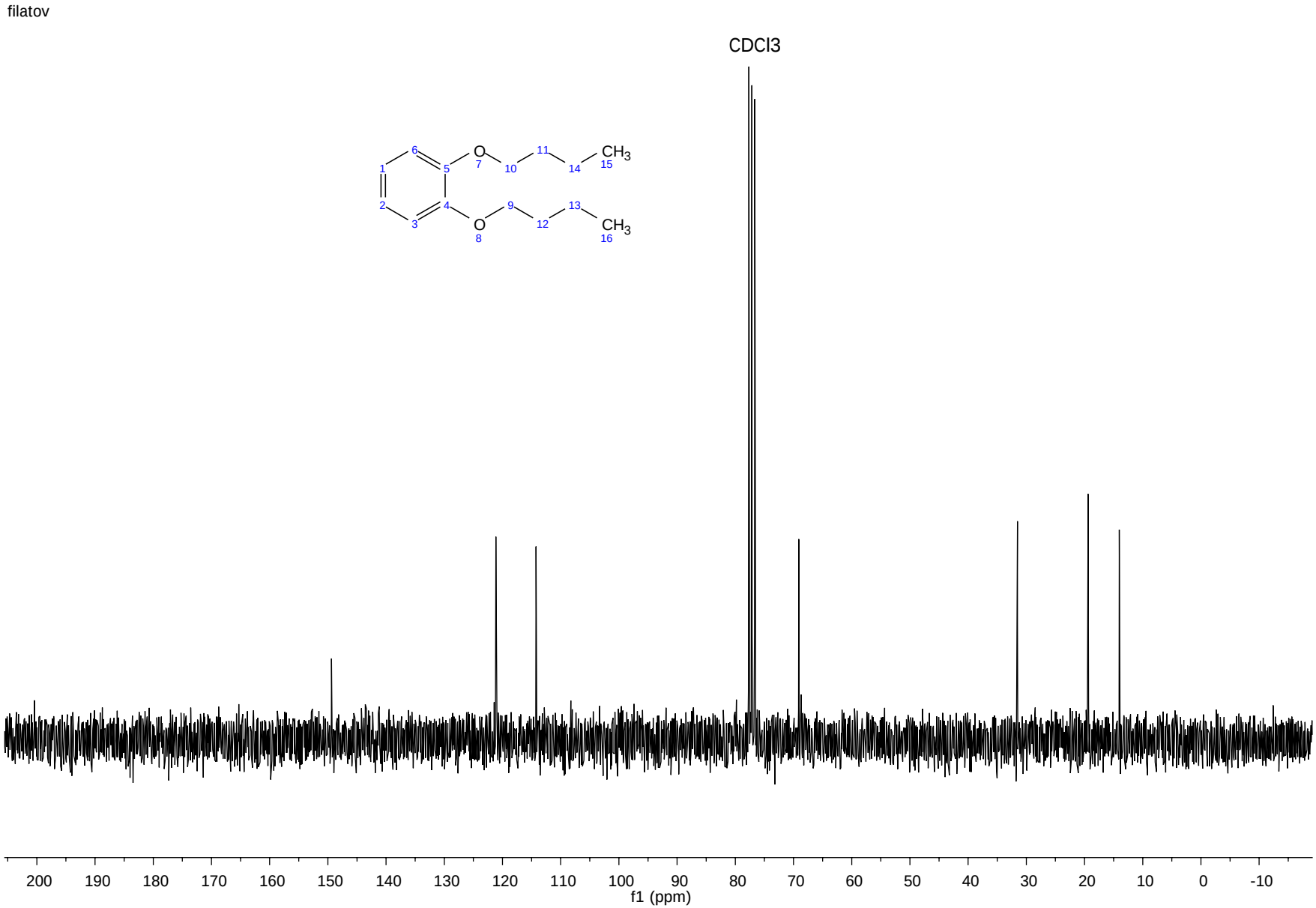


Figure S2. ^{13}C NMR spectrum for compound **7** in $CDCl_3$

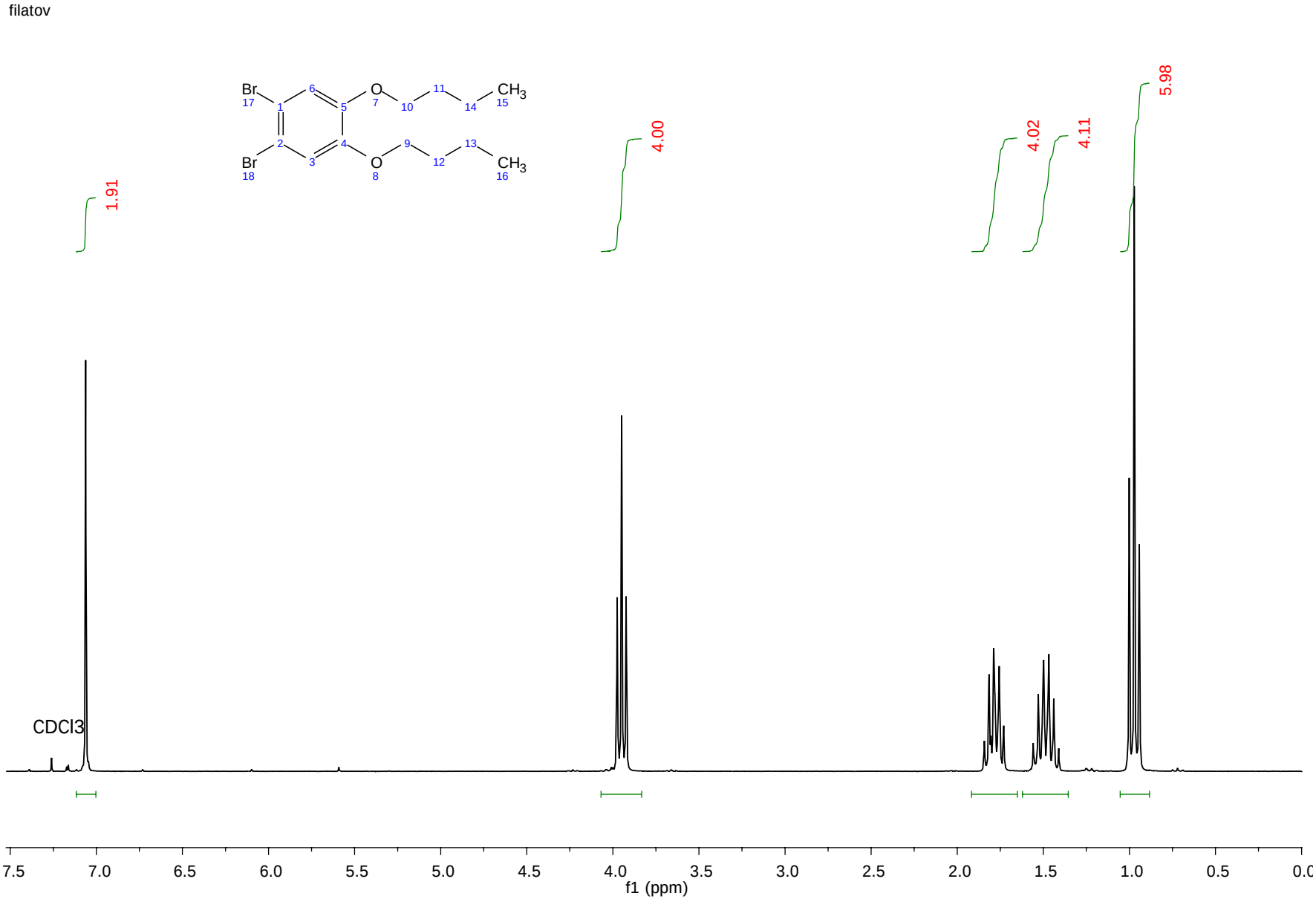


Figure S3. ^{1}H NMR spectrum for compound **8** in $CDCl_3$

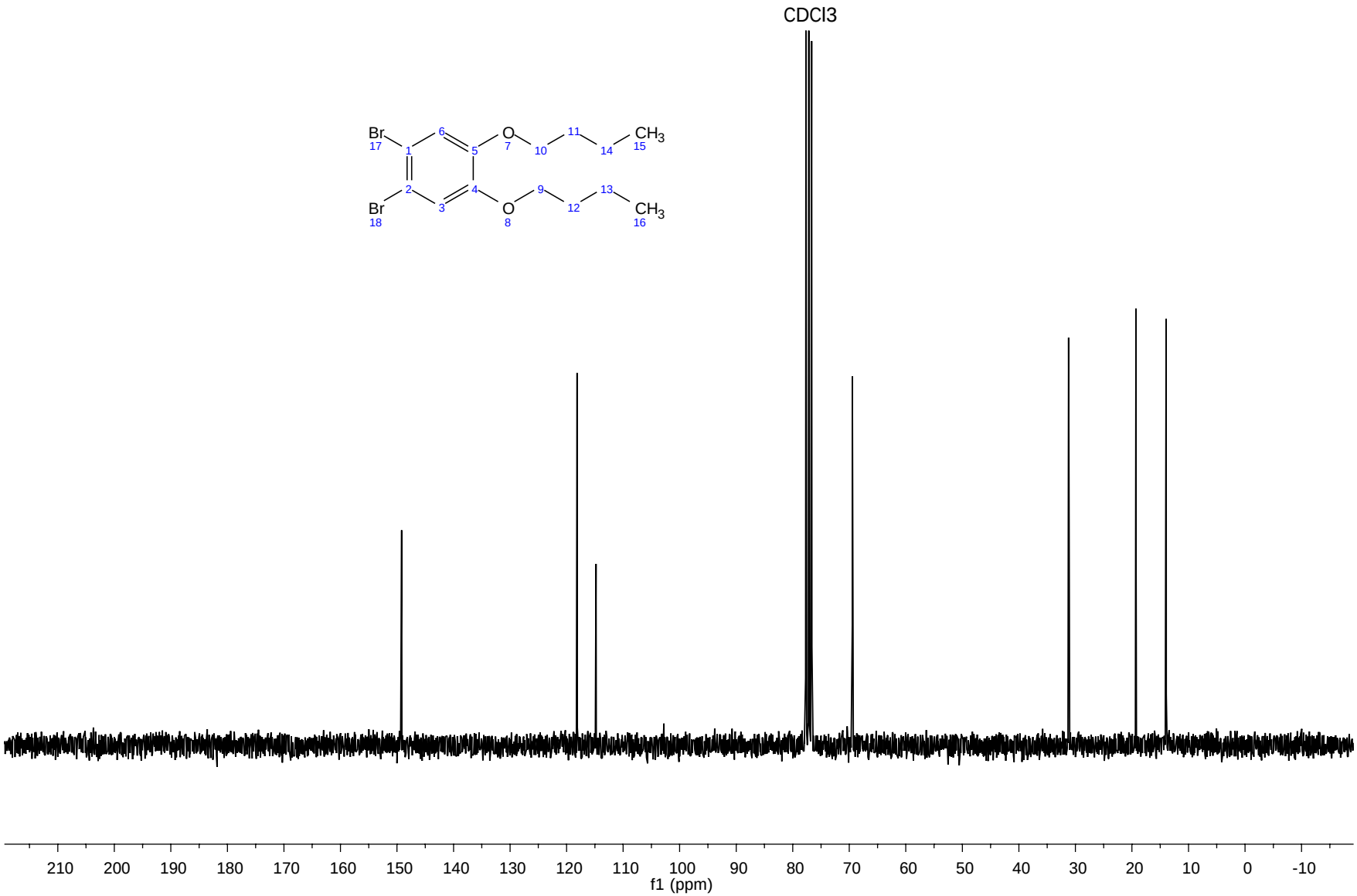


Figure S4. ^{13}C NMR spectrum for compound **8** in $CDCl_3$

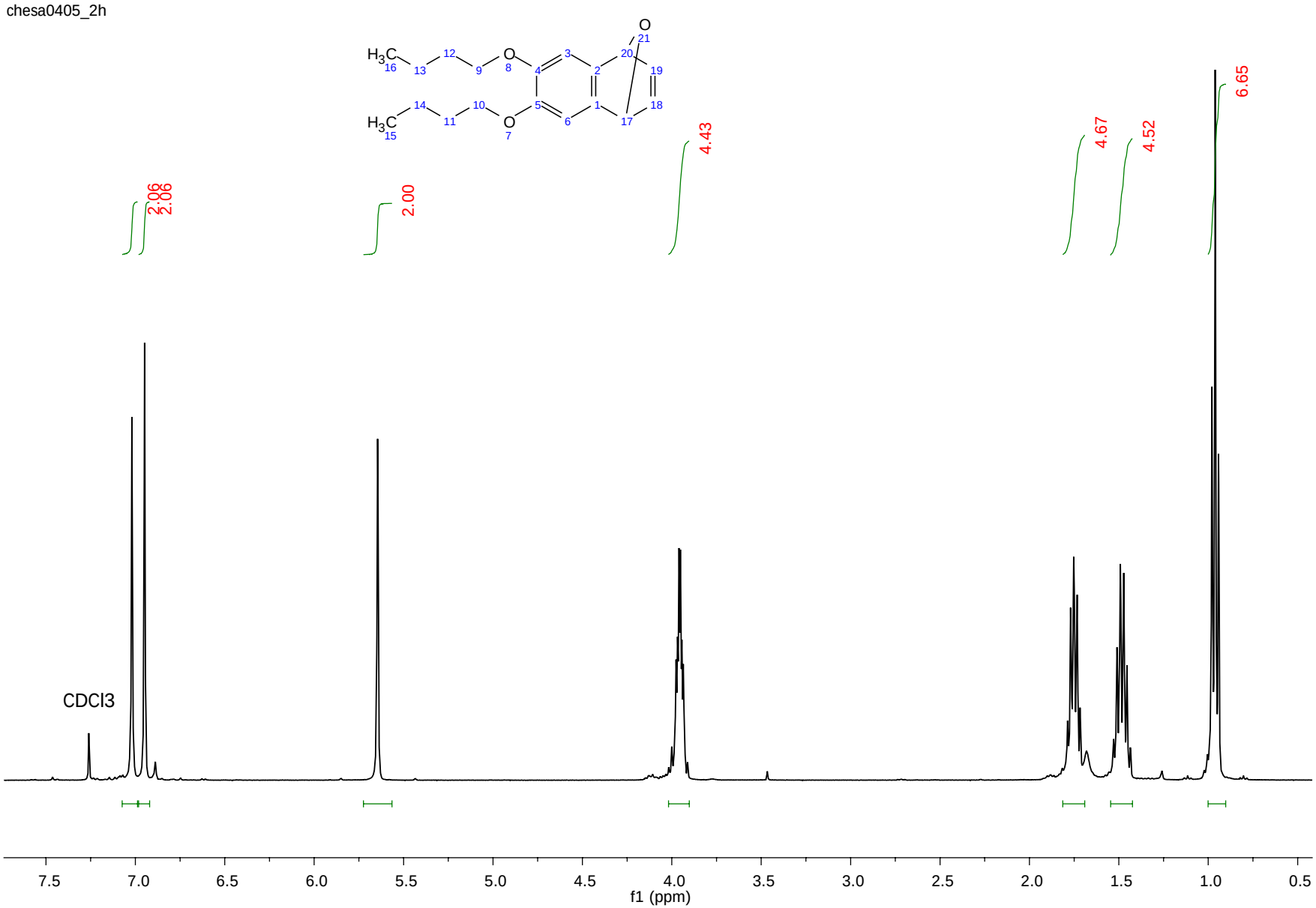


Figure S5. ^{1}H NMR spectrum for compound **9** in $CDCl_3$

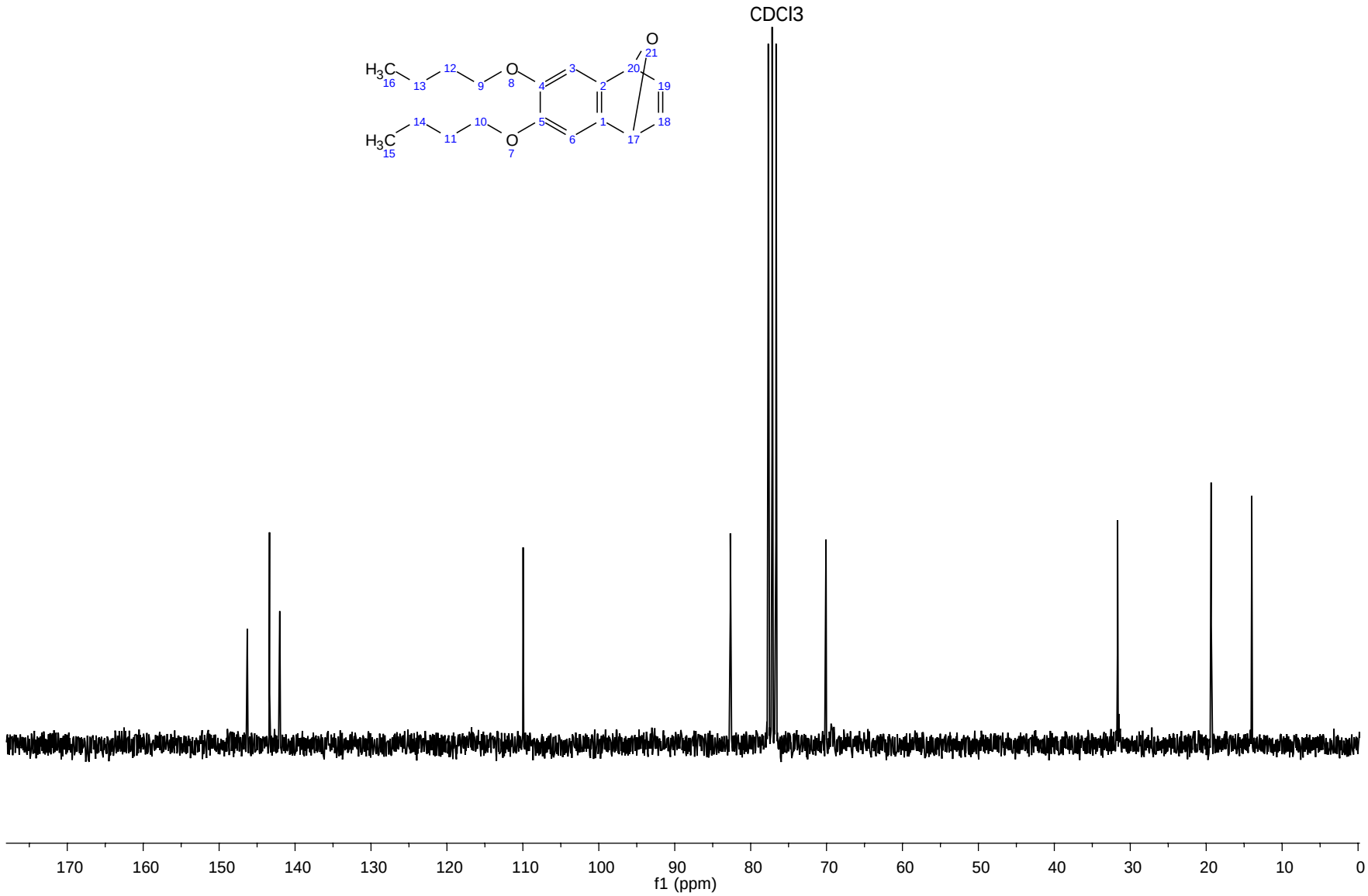


Figure S6. ^{13}C NMR spectrum for compound **9** in $CDCl_3$

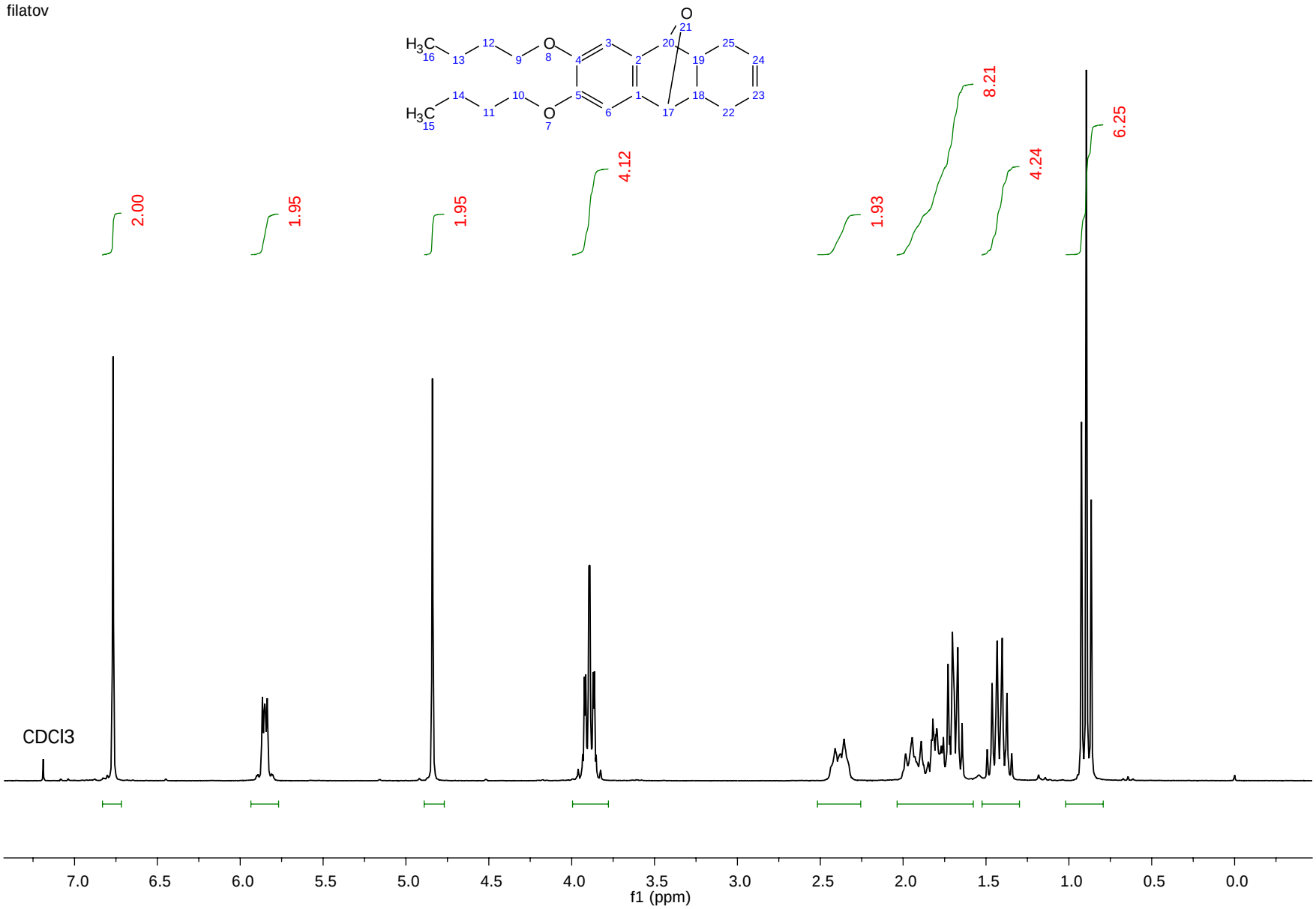


Figure S7. ^{1}H NMR spectrum for compound **10** in $CDCl_3$

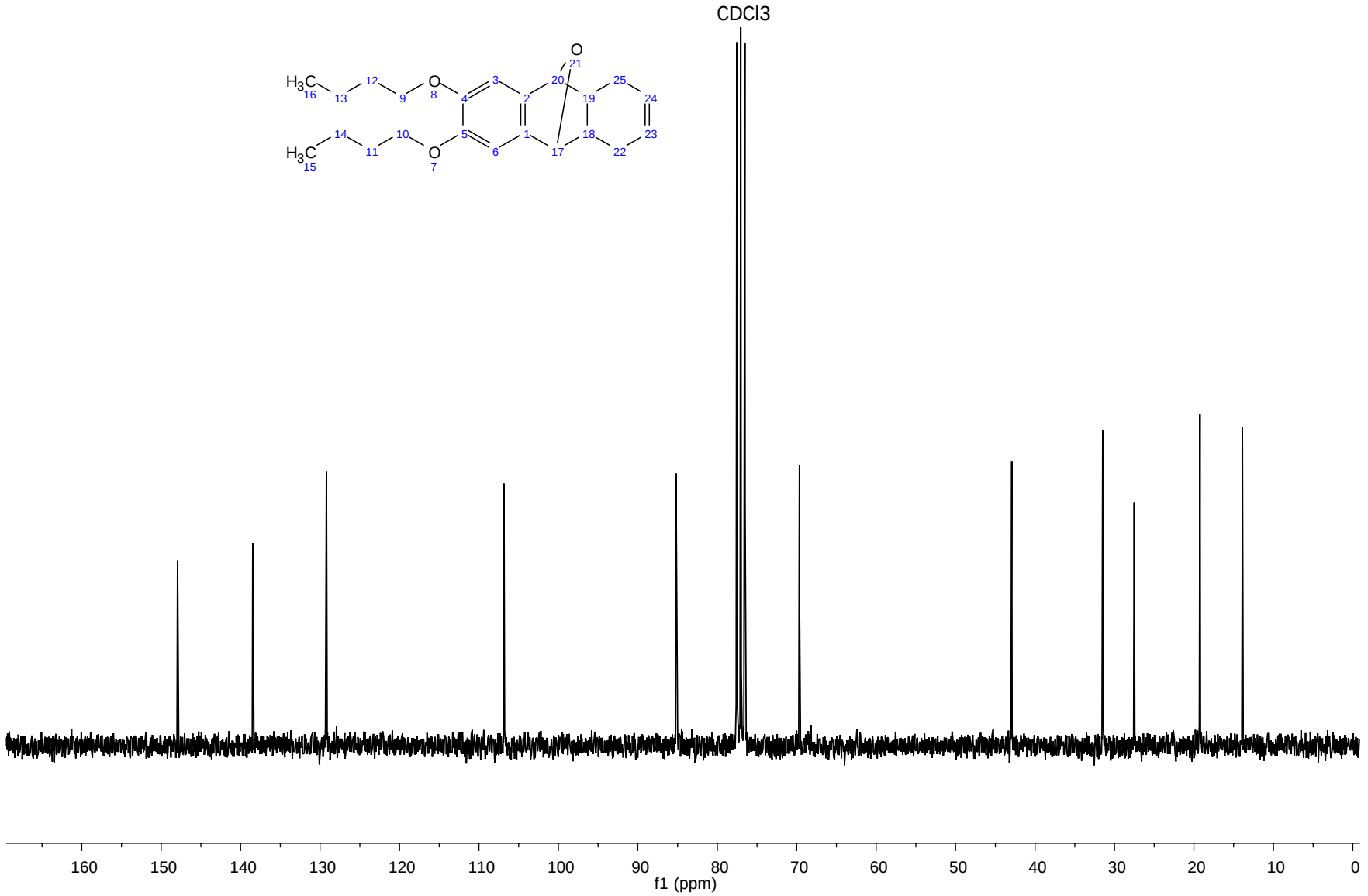


Figure S8. ^{13}C NMR spectrum for compound **10** in $CDCl_3$

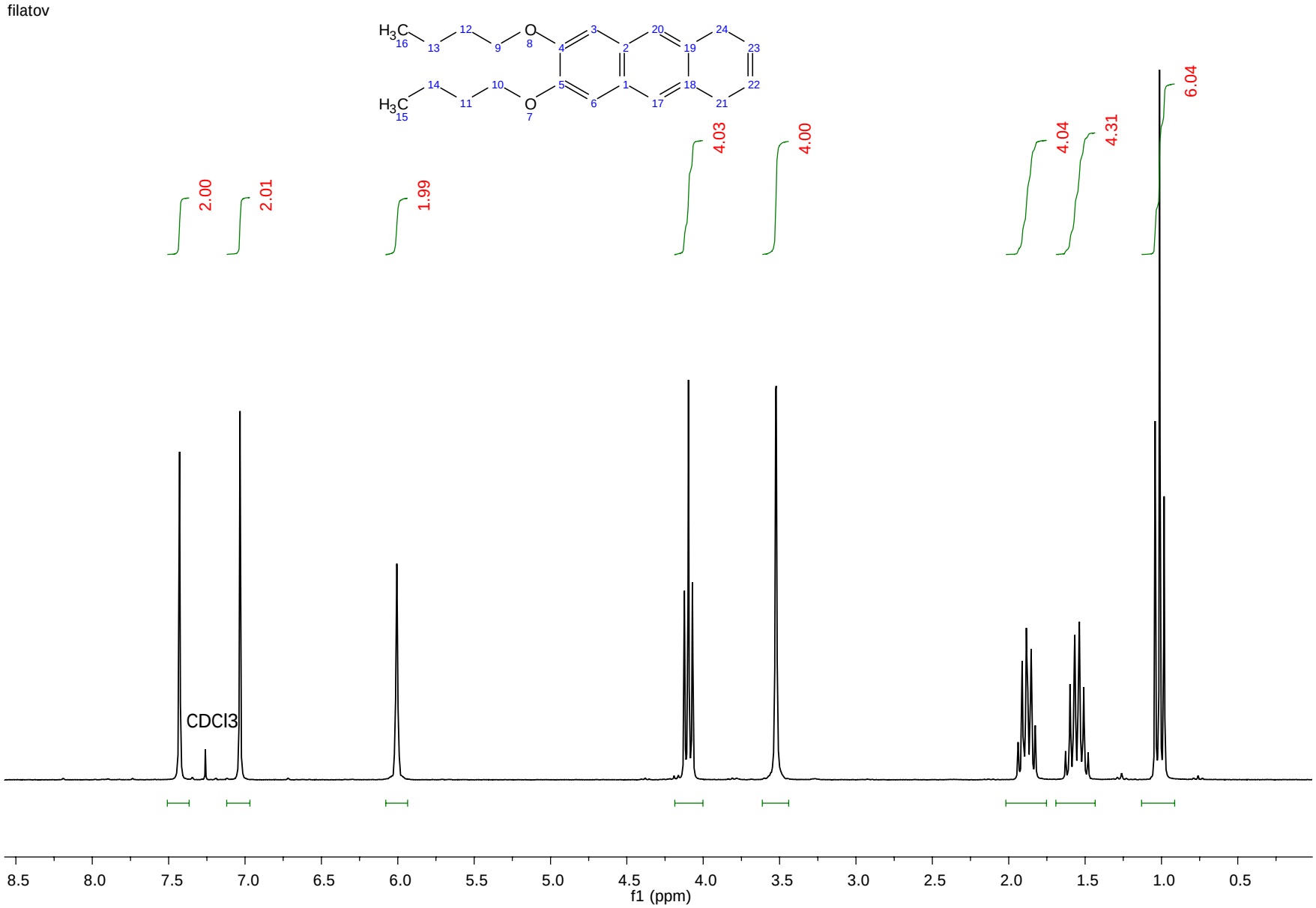


Figure S9. ^{1}H NMR spectrum for compound **11** in $CDCl_3$

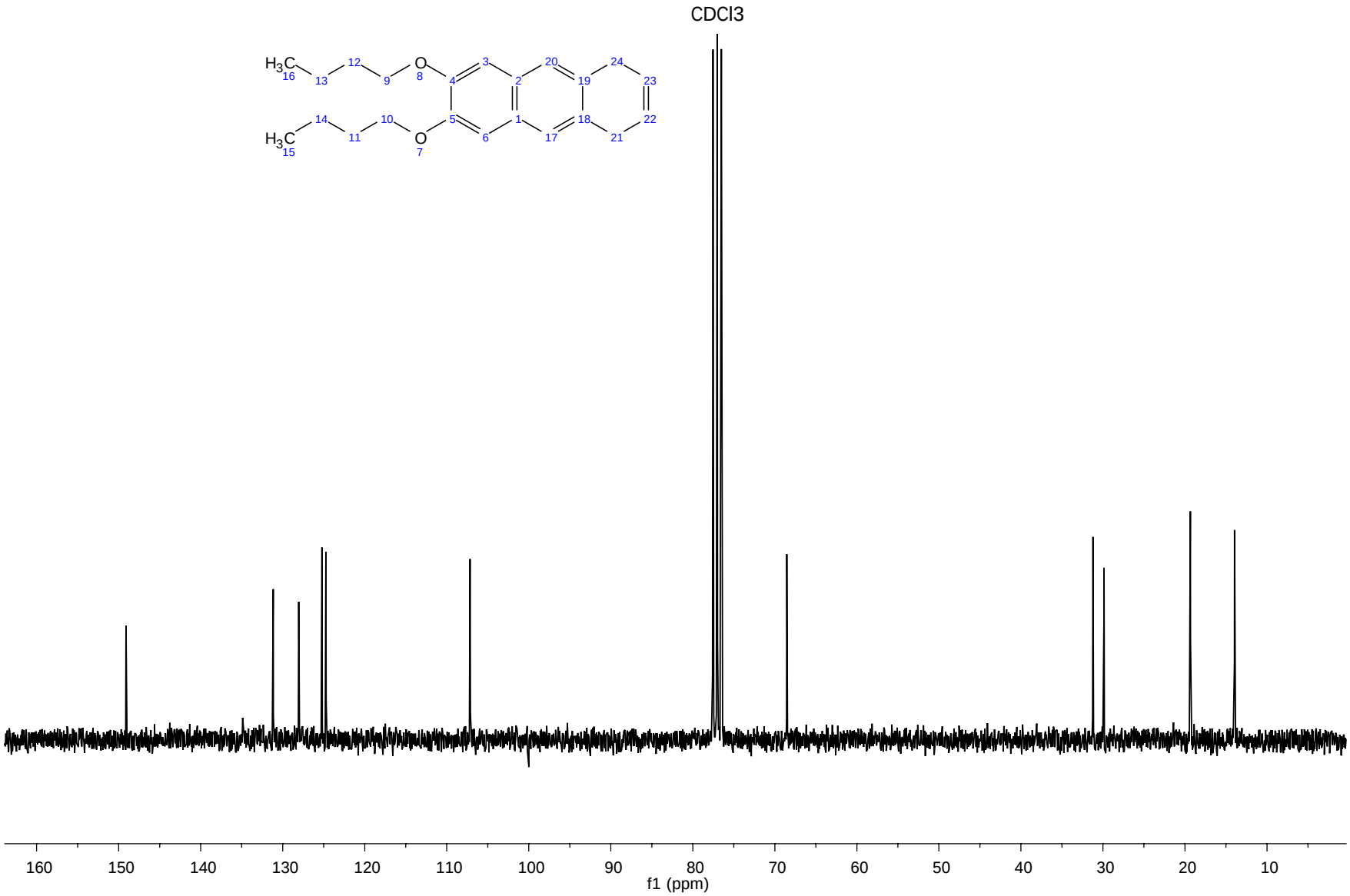


Figure S10. ^{13}C NMR spectrum for compound **11** in $CDCl_3$

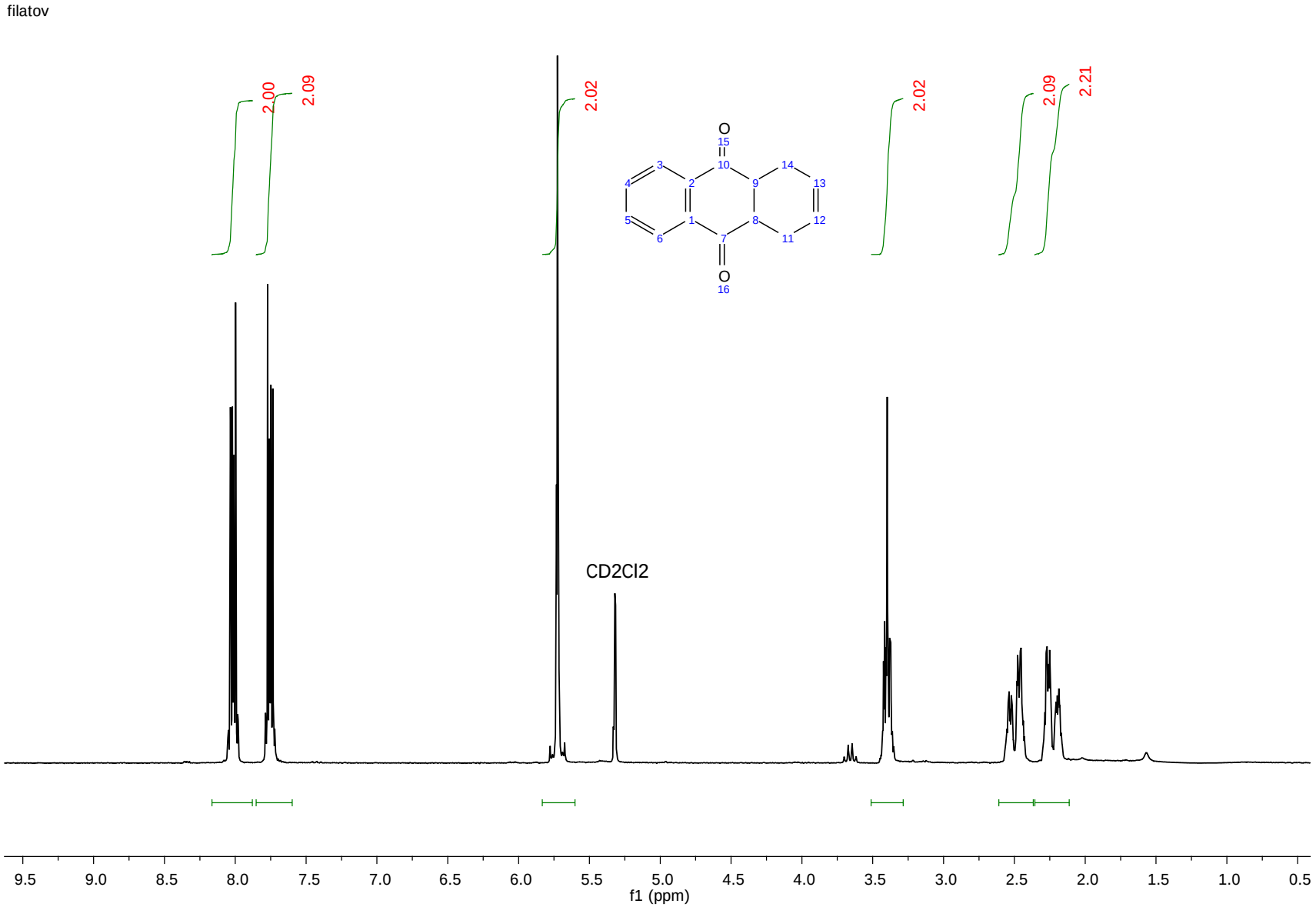


Figure S11. ^{1}H NMR spectrum for compound **18** in CD_2Cl_2

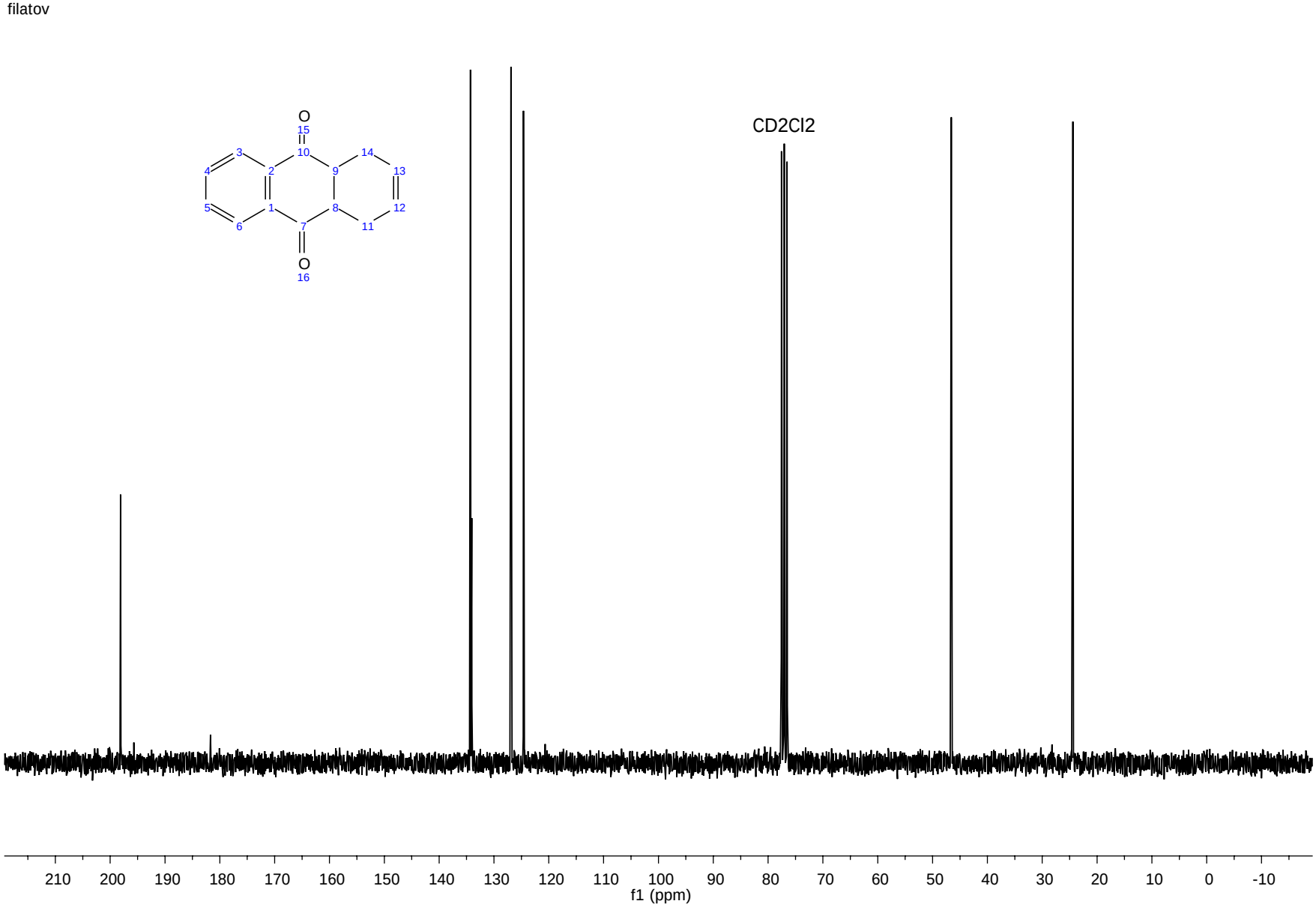


Figure S12. ^{13}C NMR spectrum for compound **18** in CD_2Cl_2

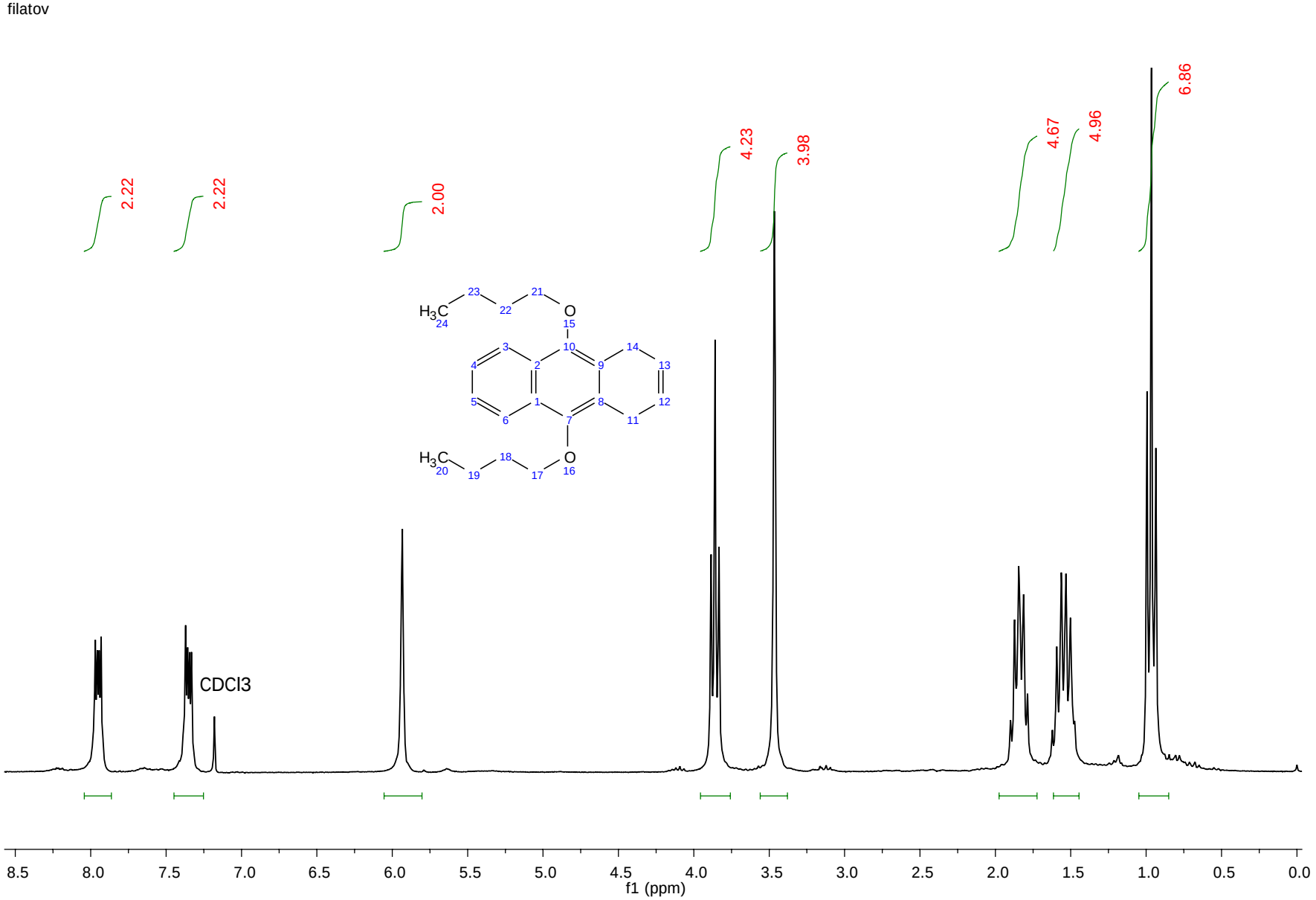


Figure S13. ^{1}H NMR spectrum for compound **19** in $CDCl_3$

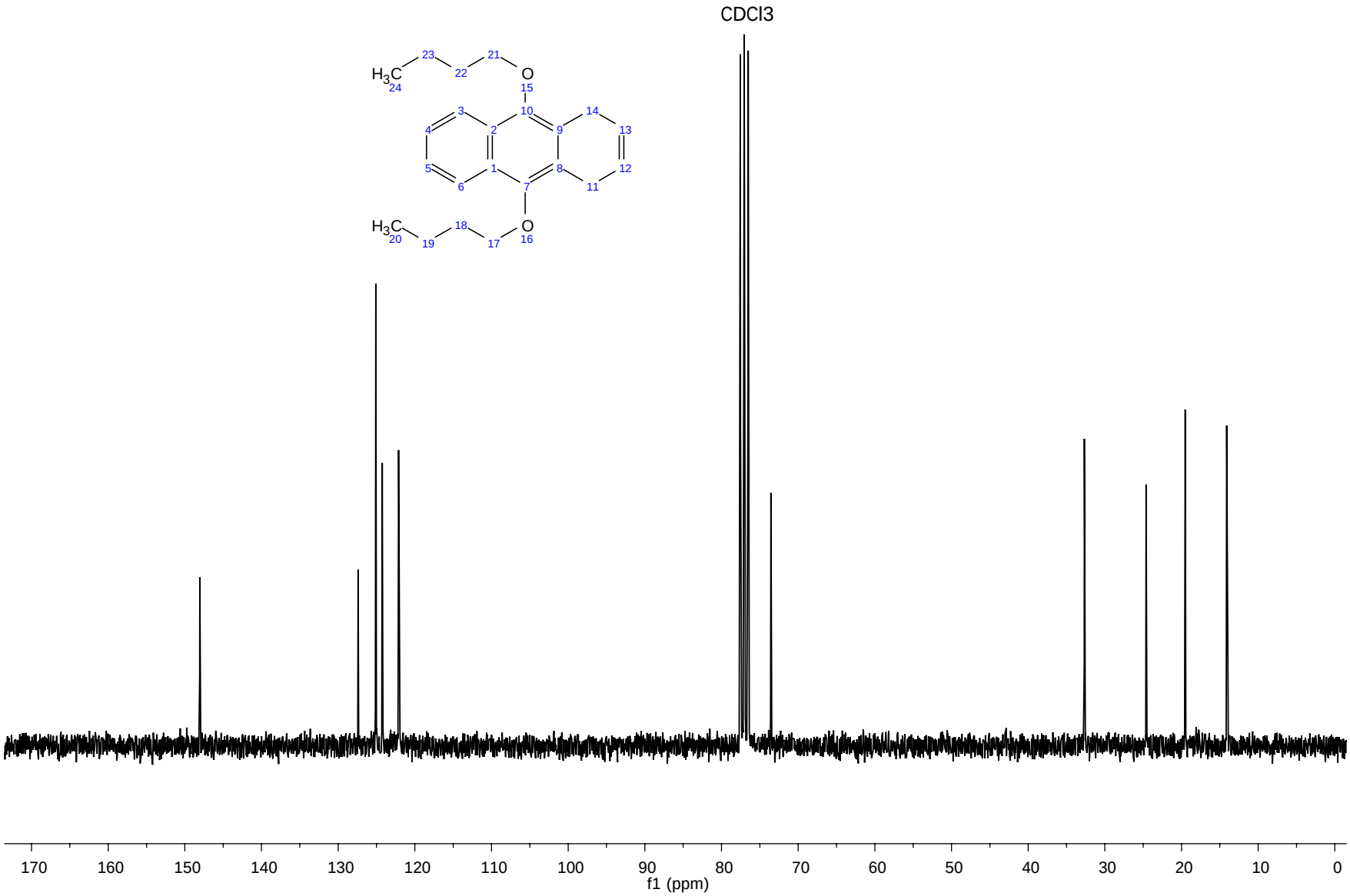


Figure S14. ^{13}C NMR spectrum for compound **19** in $CDCl_3$

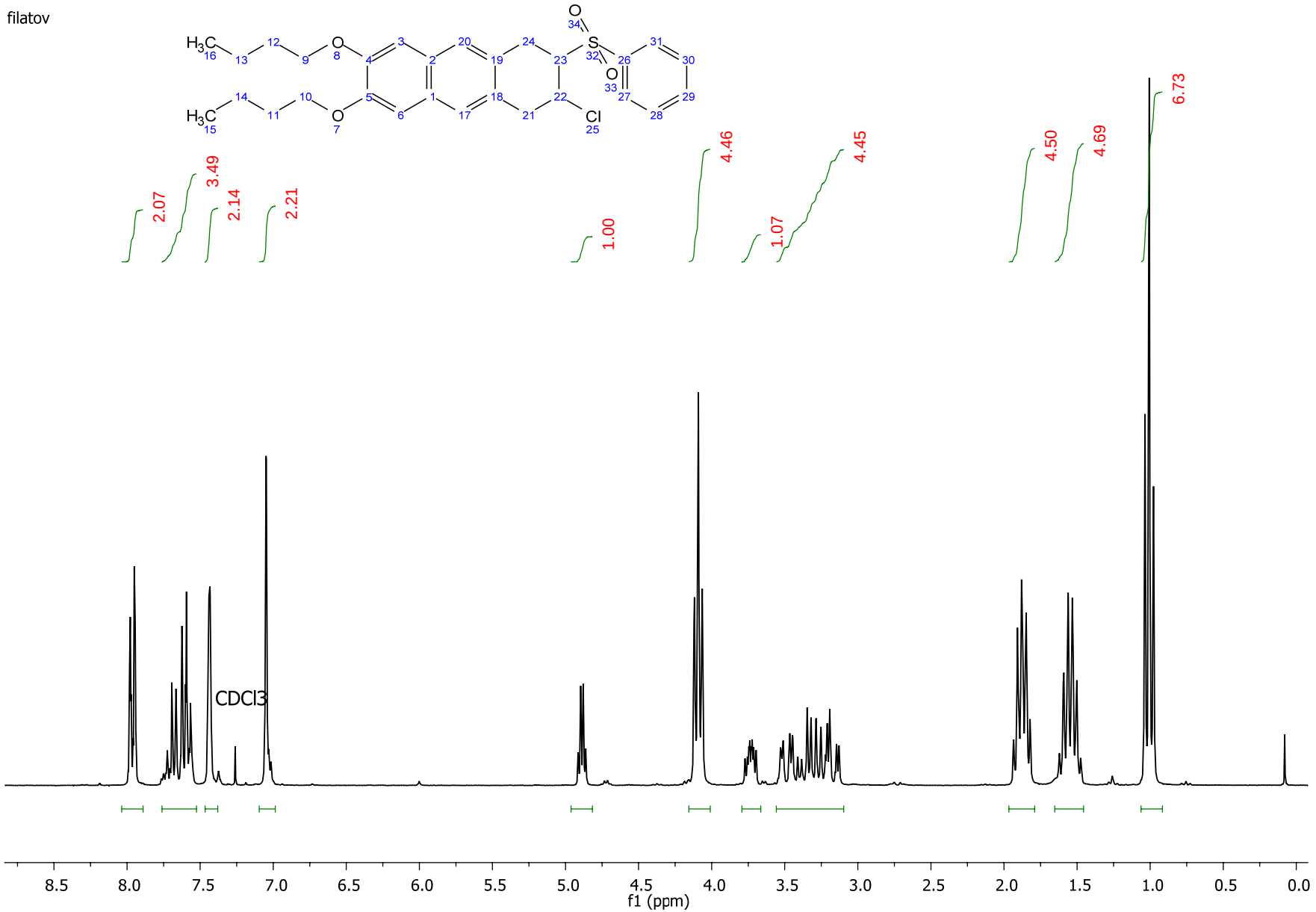


Figure S15. ^{1}H NMR spectrum for compound **12** in $CDCl_3$

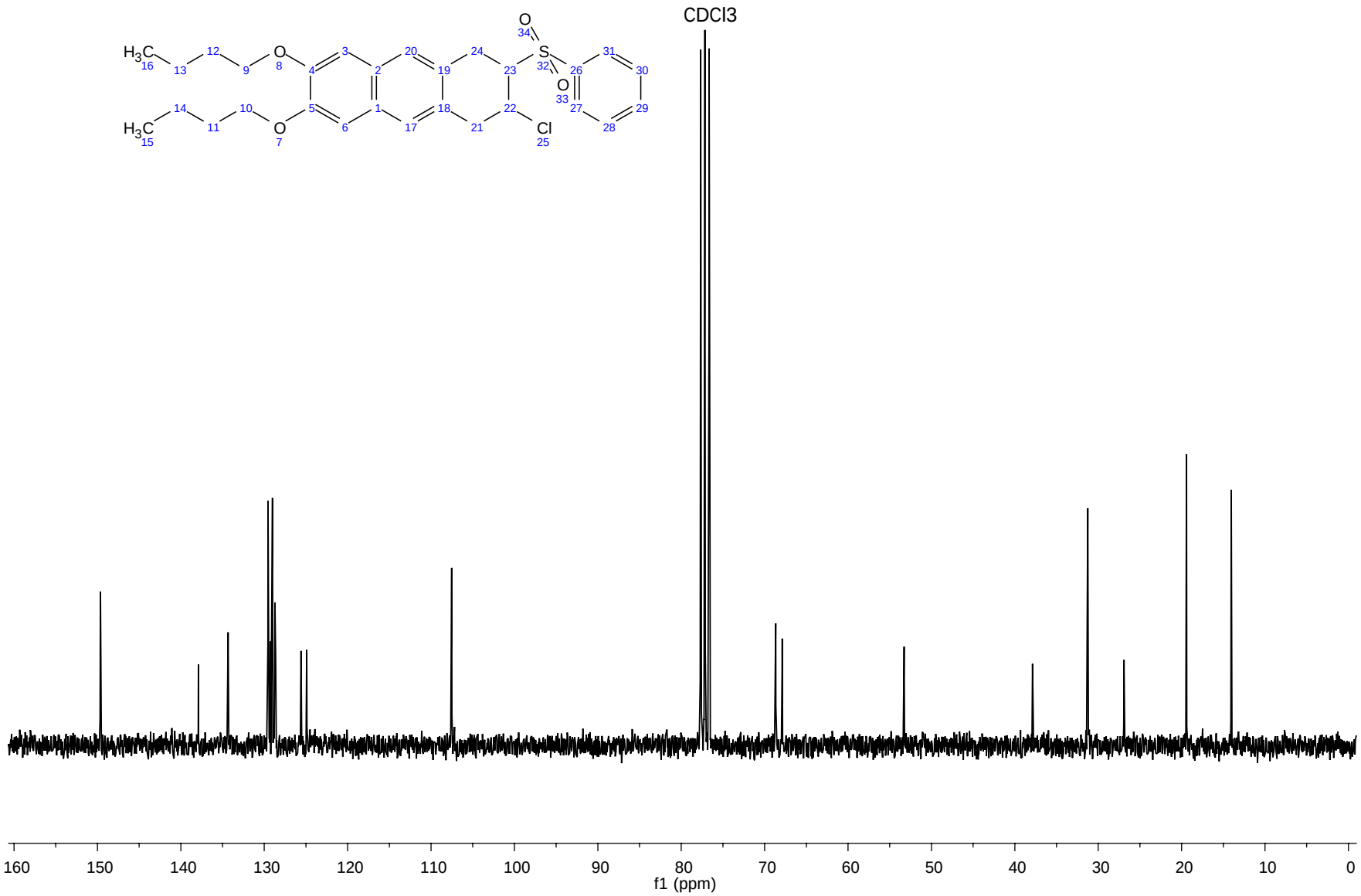
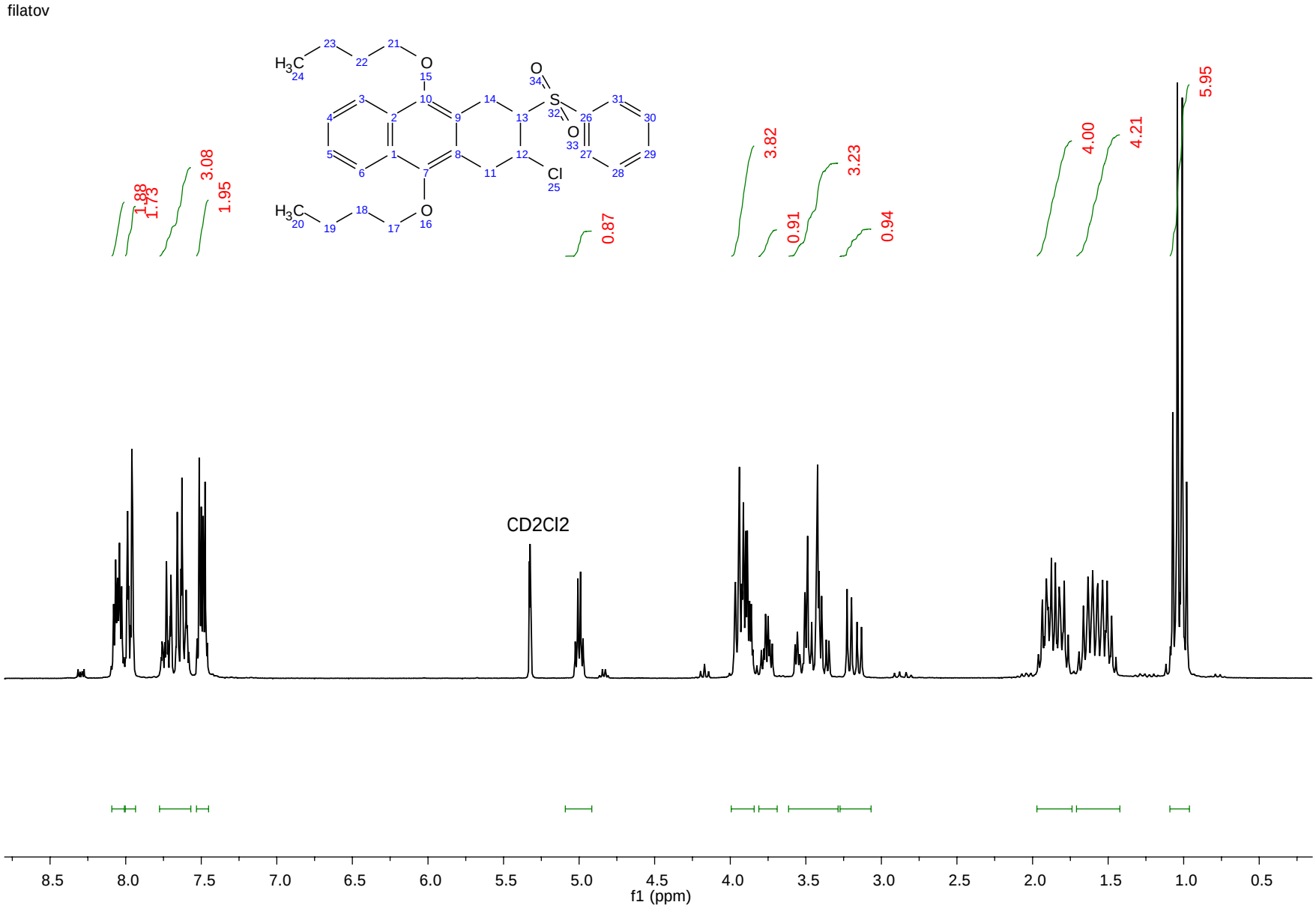


Figure S16. ^{13}C NMR spectrum for compound **12** in $CDCl_3$



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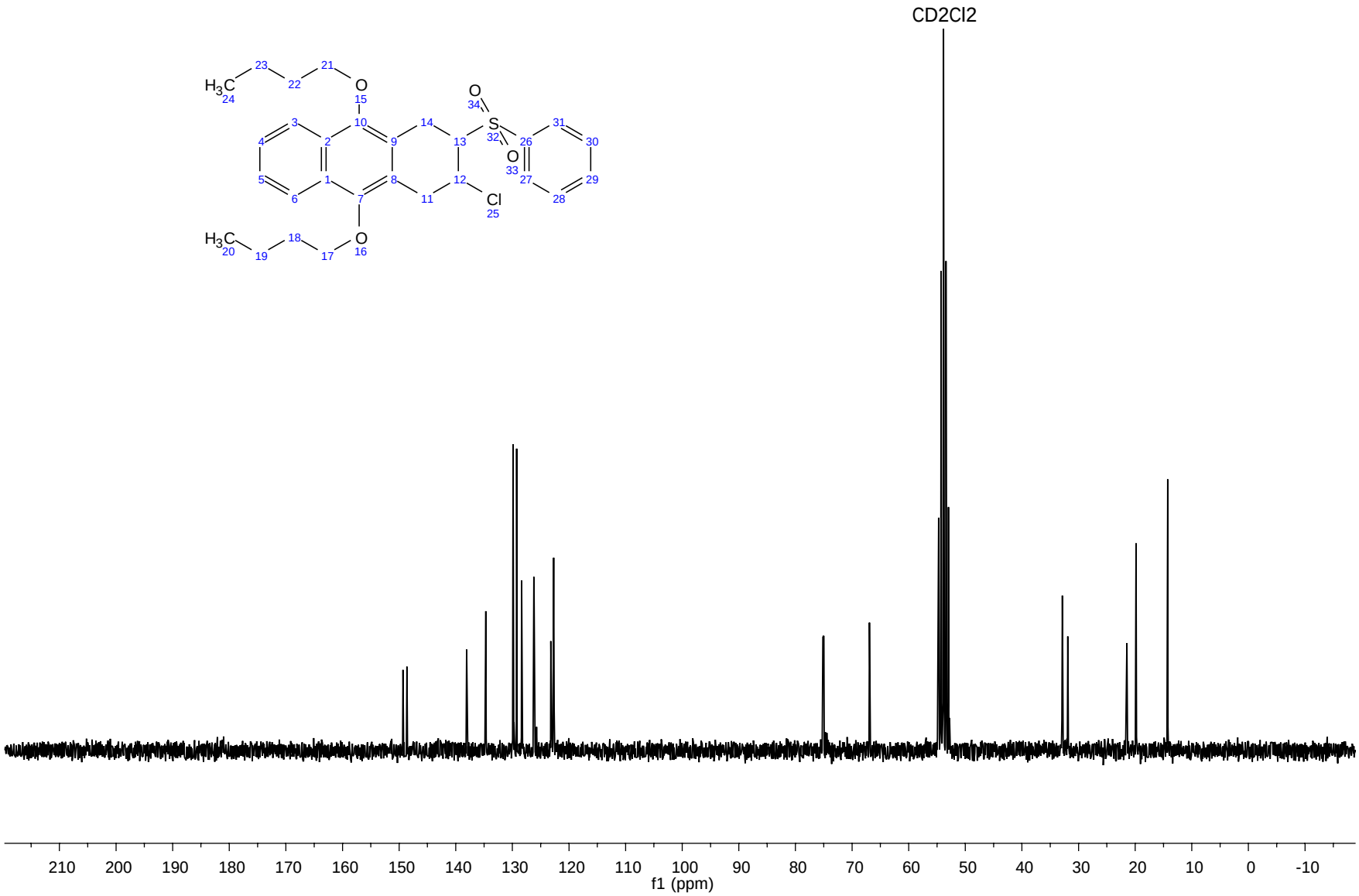

Figure S18. ^{13}C NMR spectrum for compound **20** in CD_2Cl_2

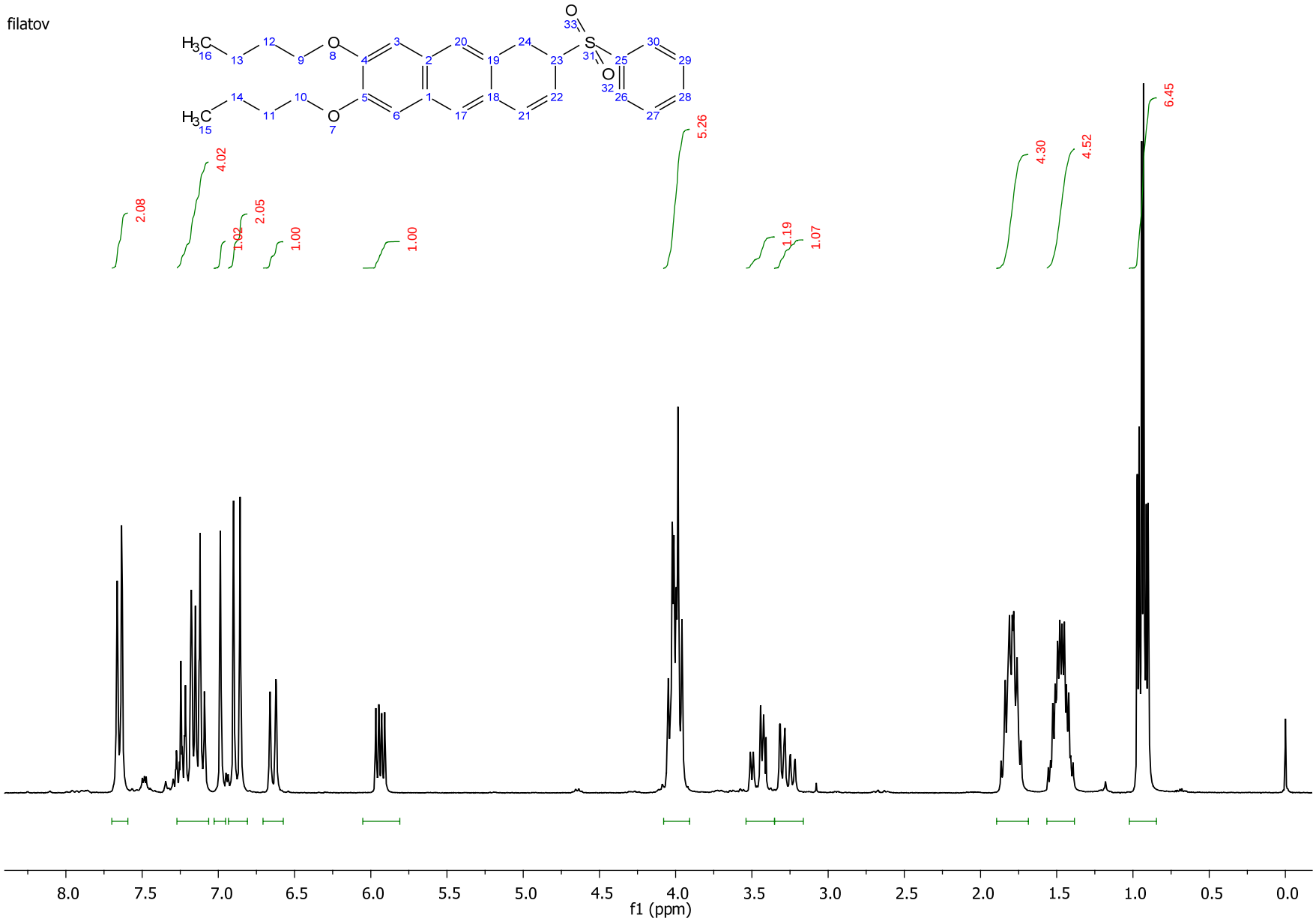

Figure S19. ^{1}H NMR spectrum for compound **13** in $CDCl_3$

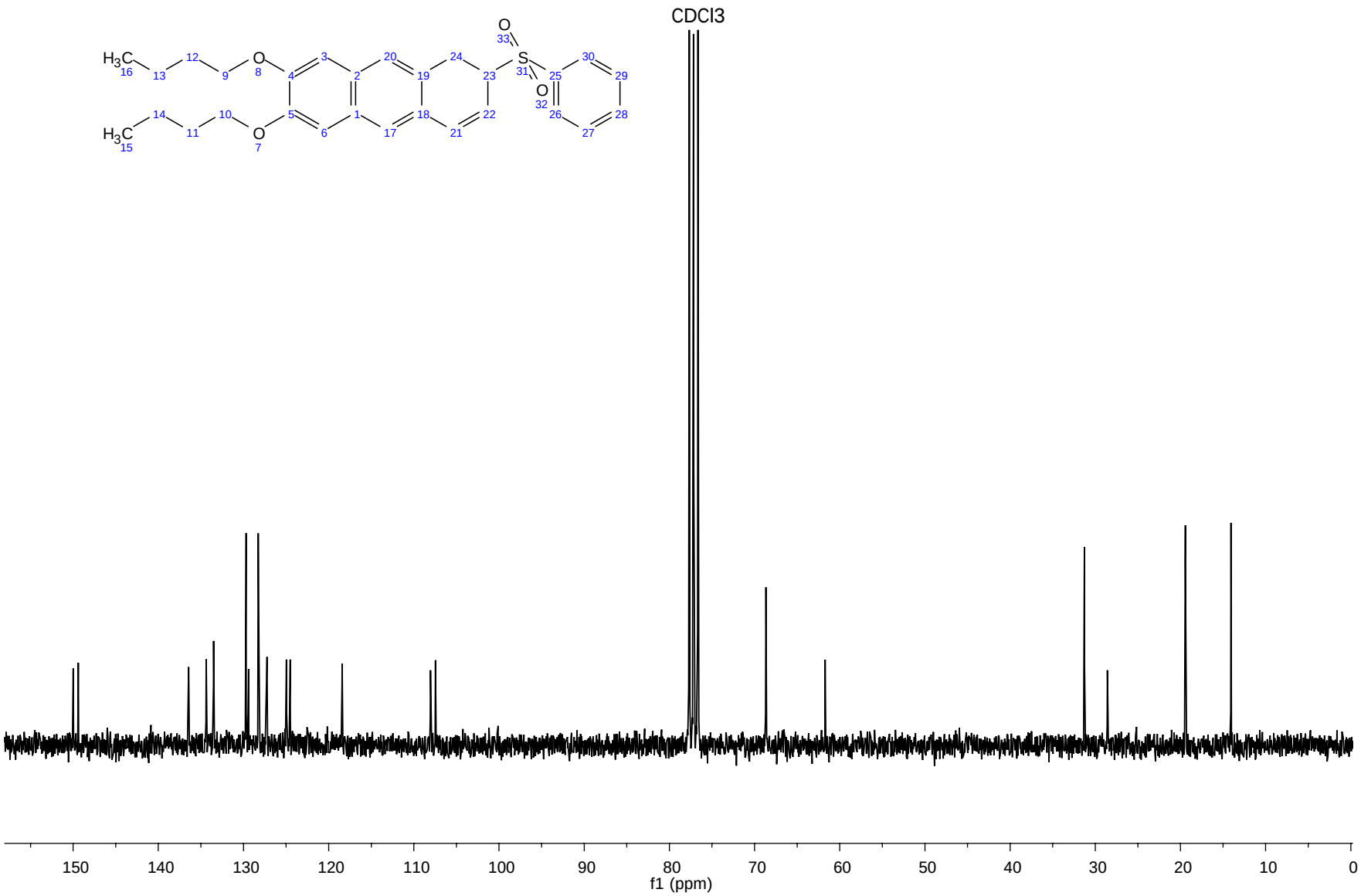

Figure S20. ^{13}C NMR spectrum for compound **13** in $CDCl_3$

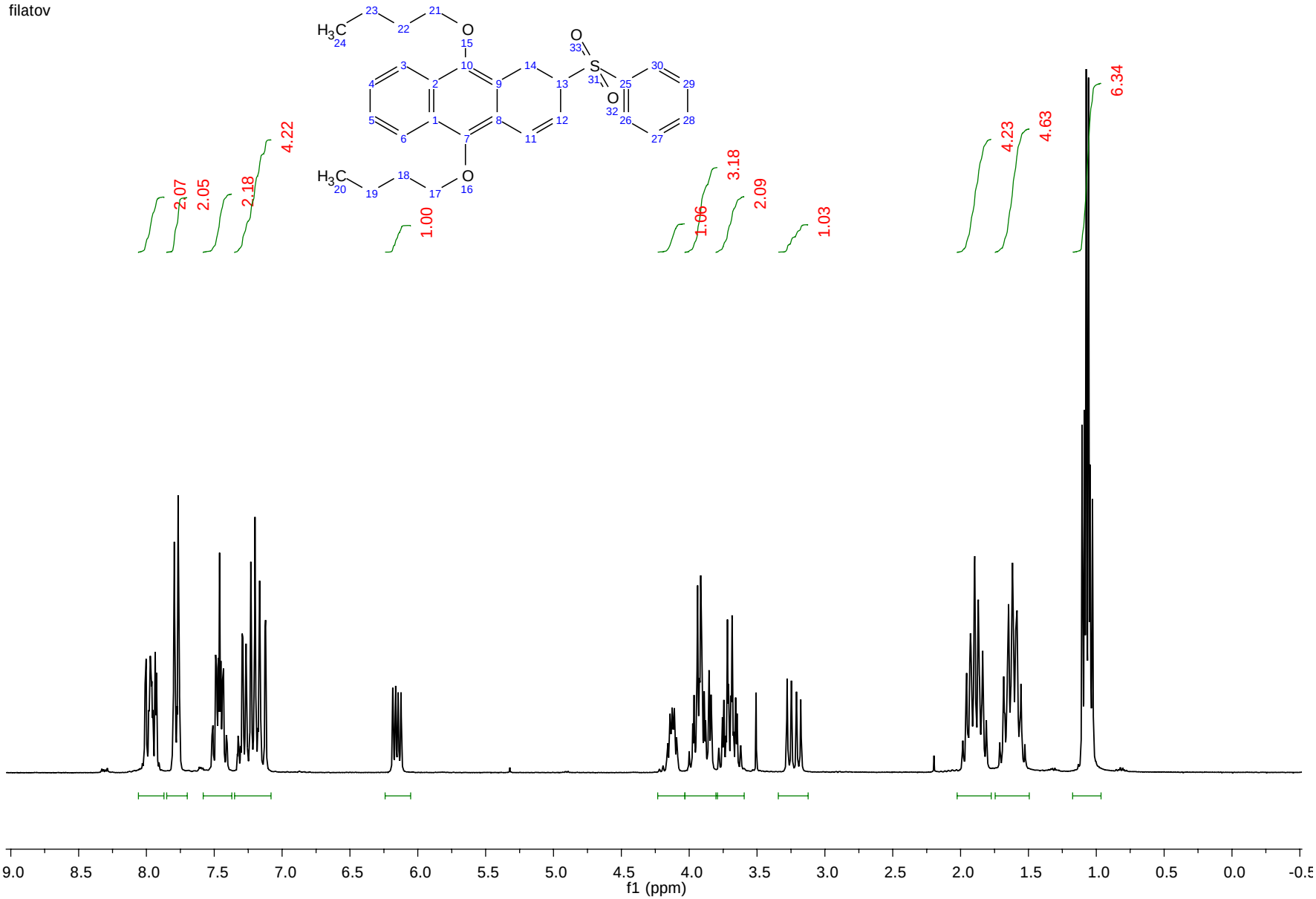

Figure S21. ^{1}H NMR spectrum for compound **21** in $CDCl_3$

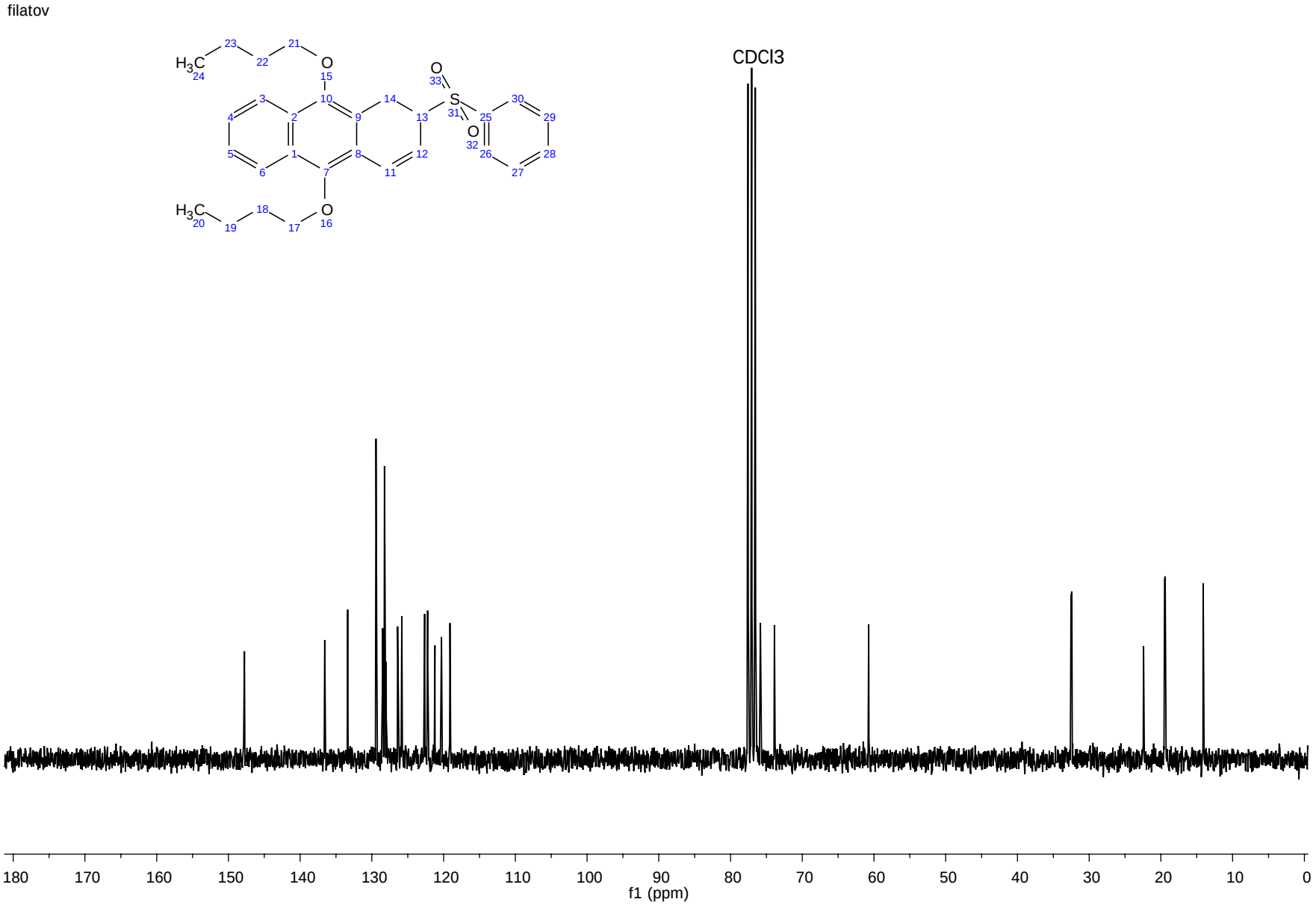

Figure S22. ^{13}C NMR spectrum for compound **21** in $CDCl_3$

25.09.2012 20:28:36

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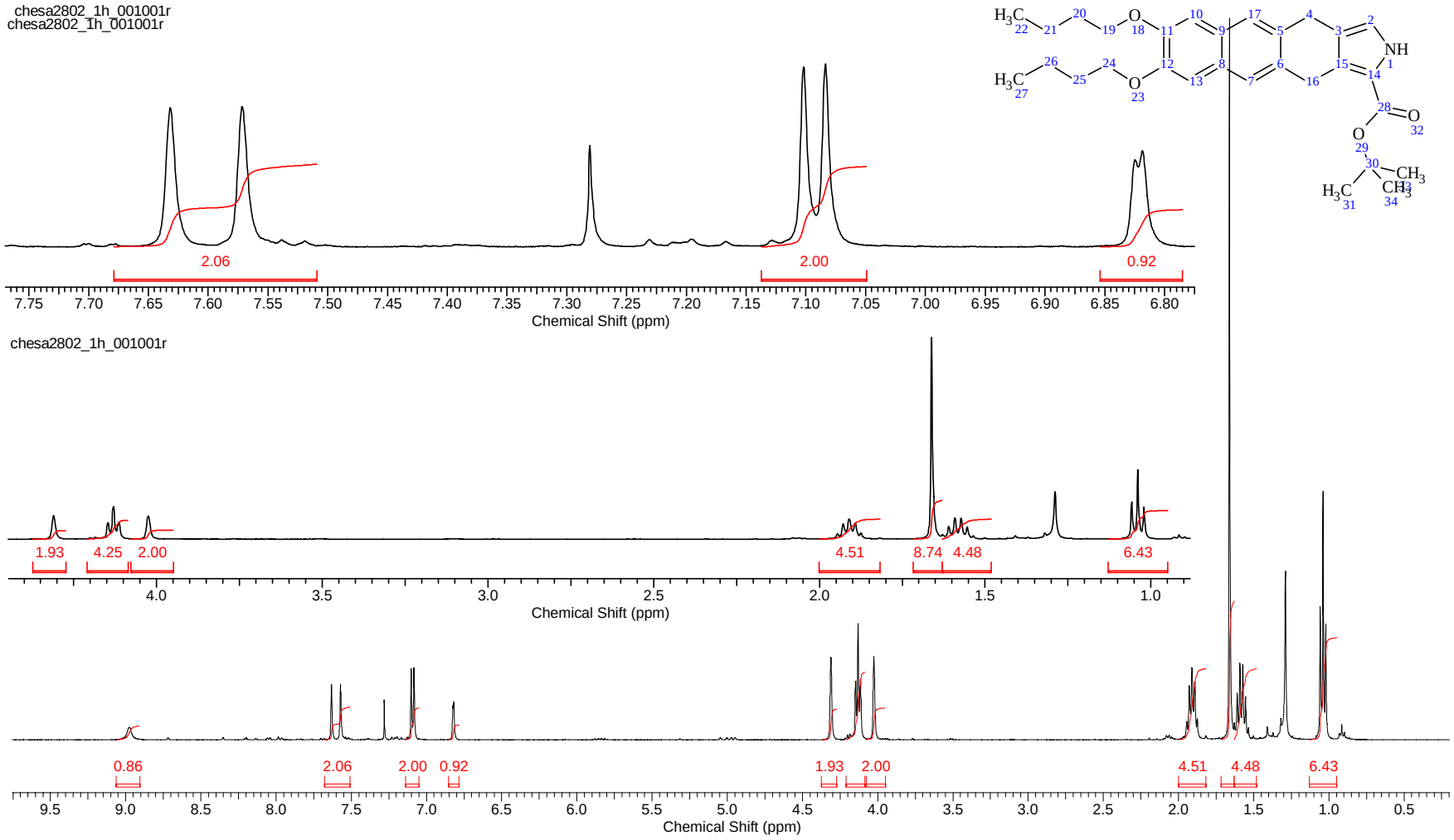

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Figure S23. ^{1}H NMR spectrum for compound **14a** in $CDCl_3$

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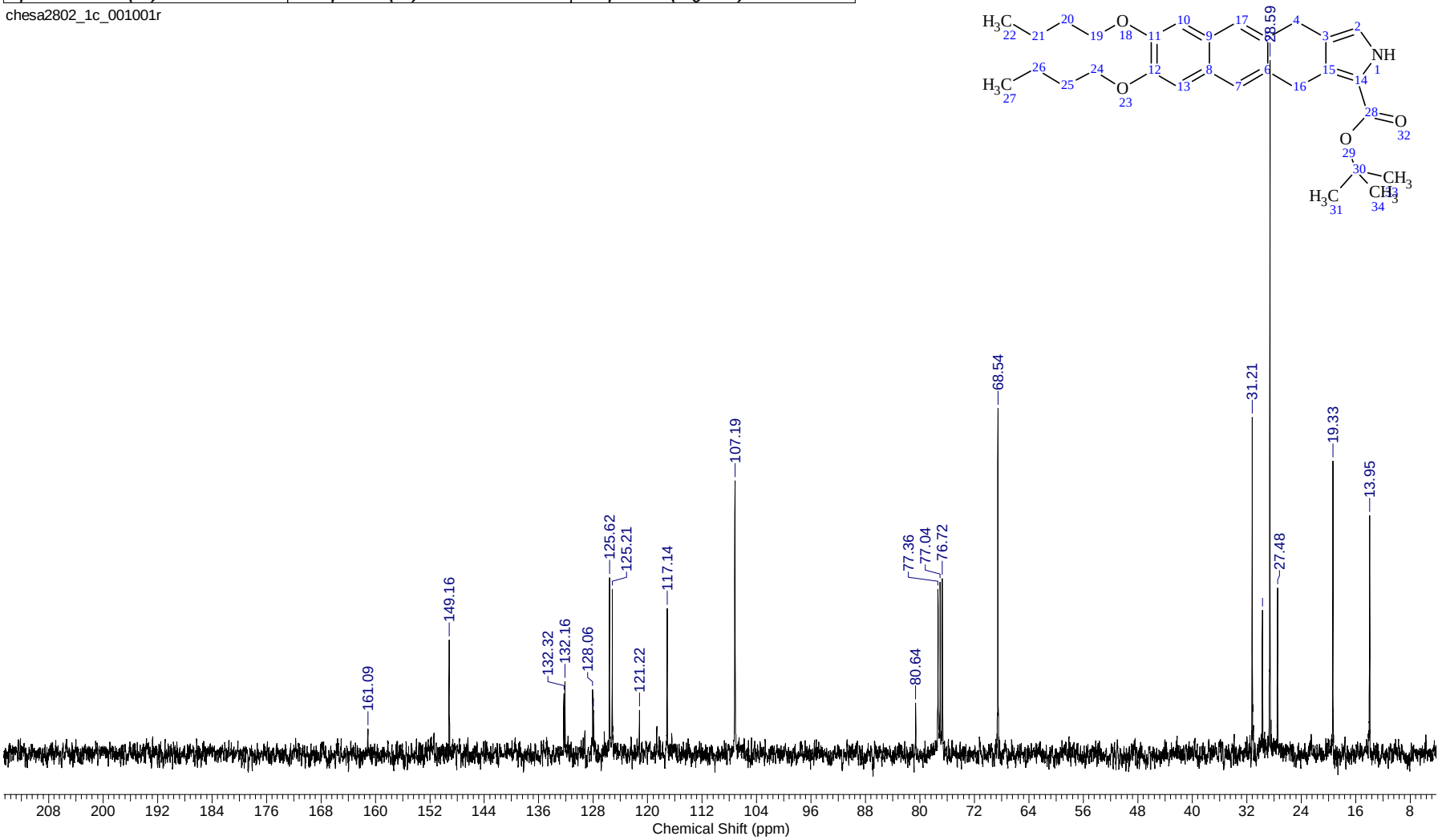

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Figure S24. ^{13}C NMR spectrum for compound **14a** in $CDCl_3$

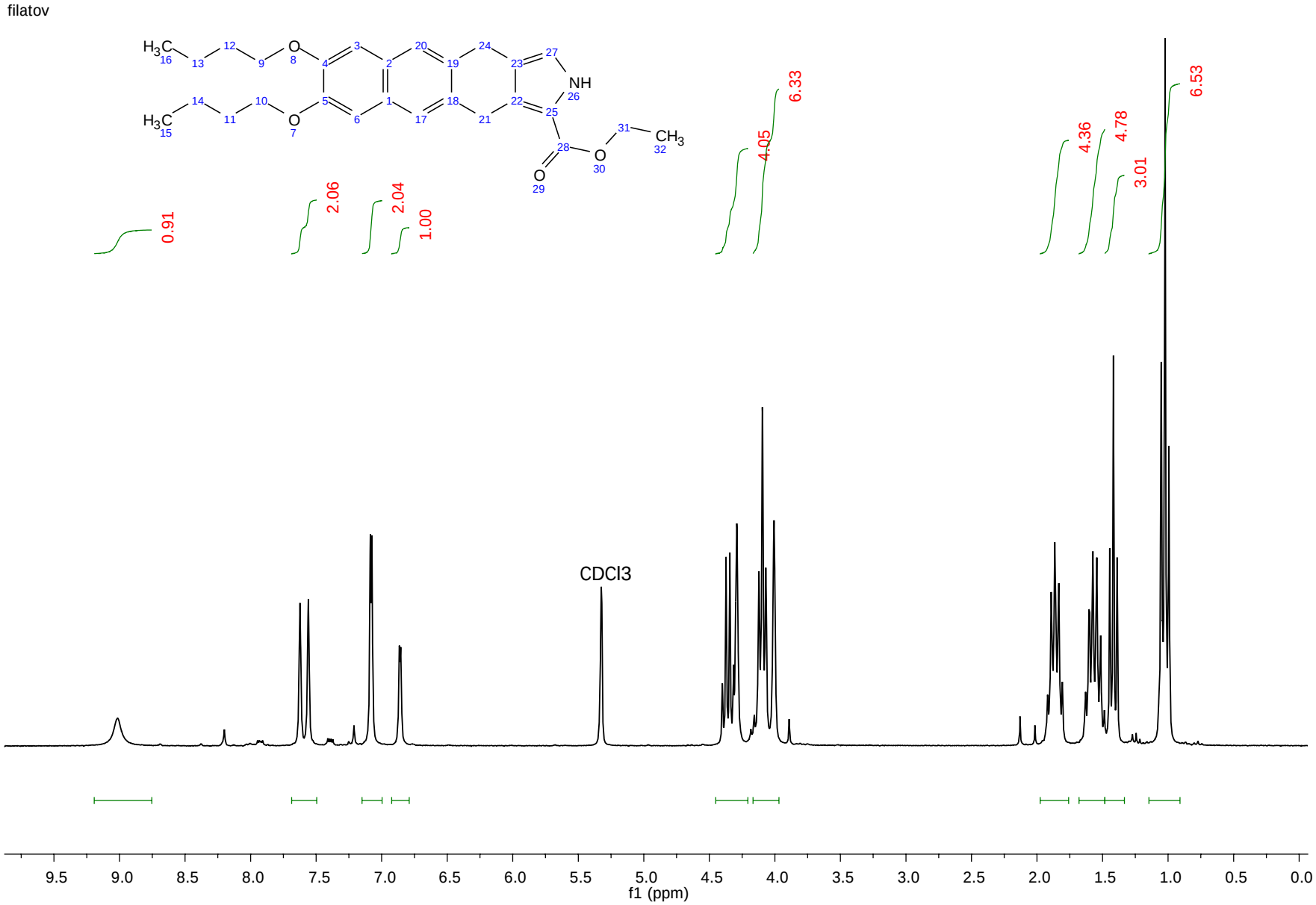

Figure S25. ^{1}H NMR spectrum for compound **14b** in CD_2Cl_2

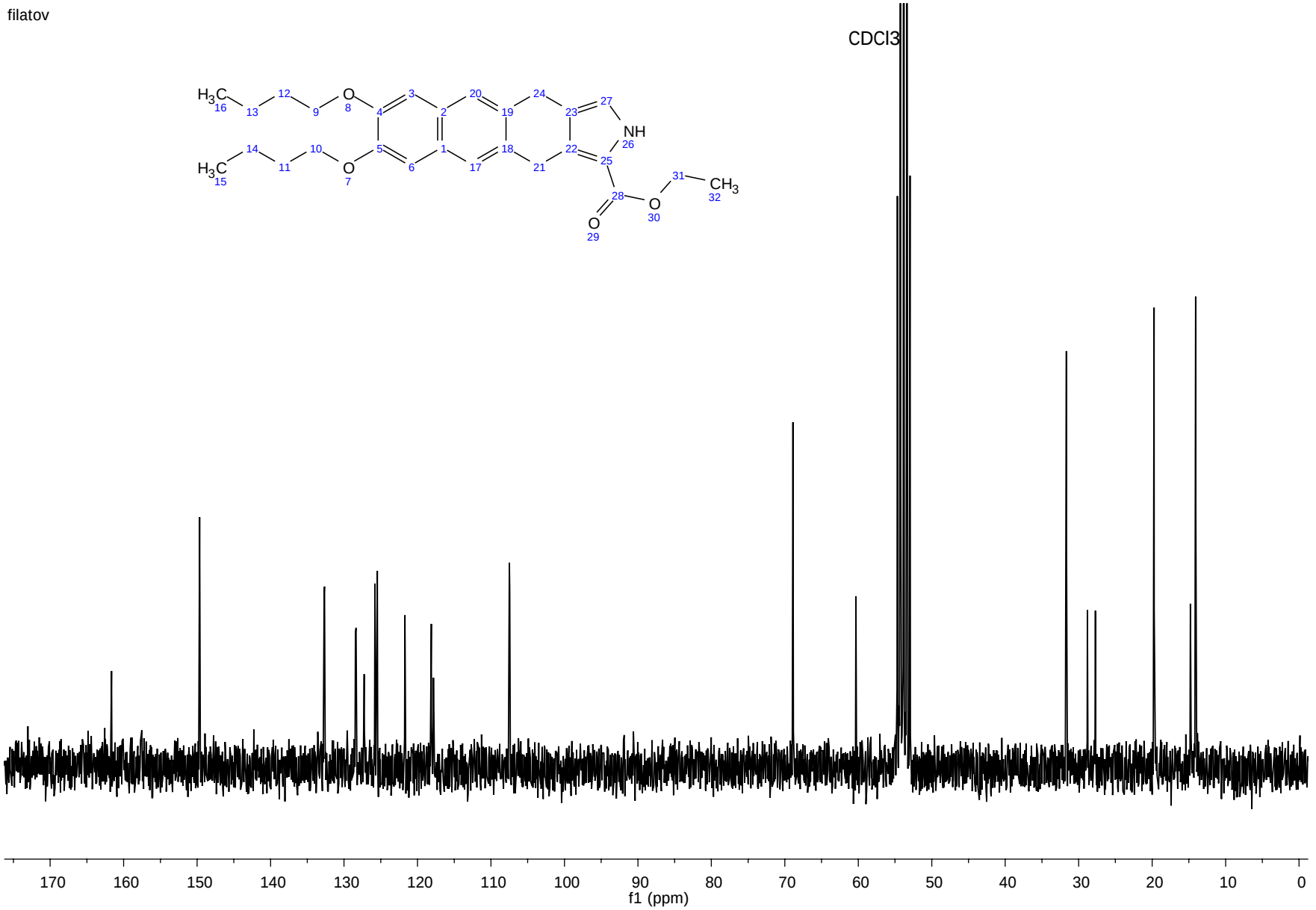

Figure S26. ^{13}C NMR spectrum for compound **14b** in CD_2Cl_2

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Spectrum Offset (Hz)	2470.9668	***Sweep Width (Hz)***	8278.15	***Temperature (degree C)***	25.060				

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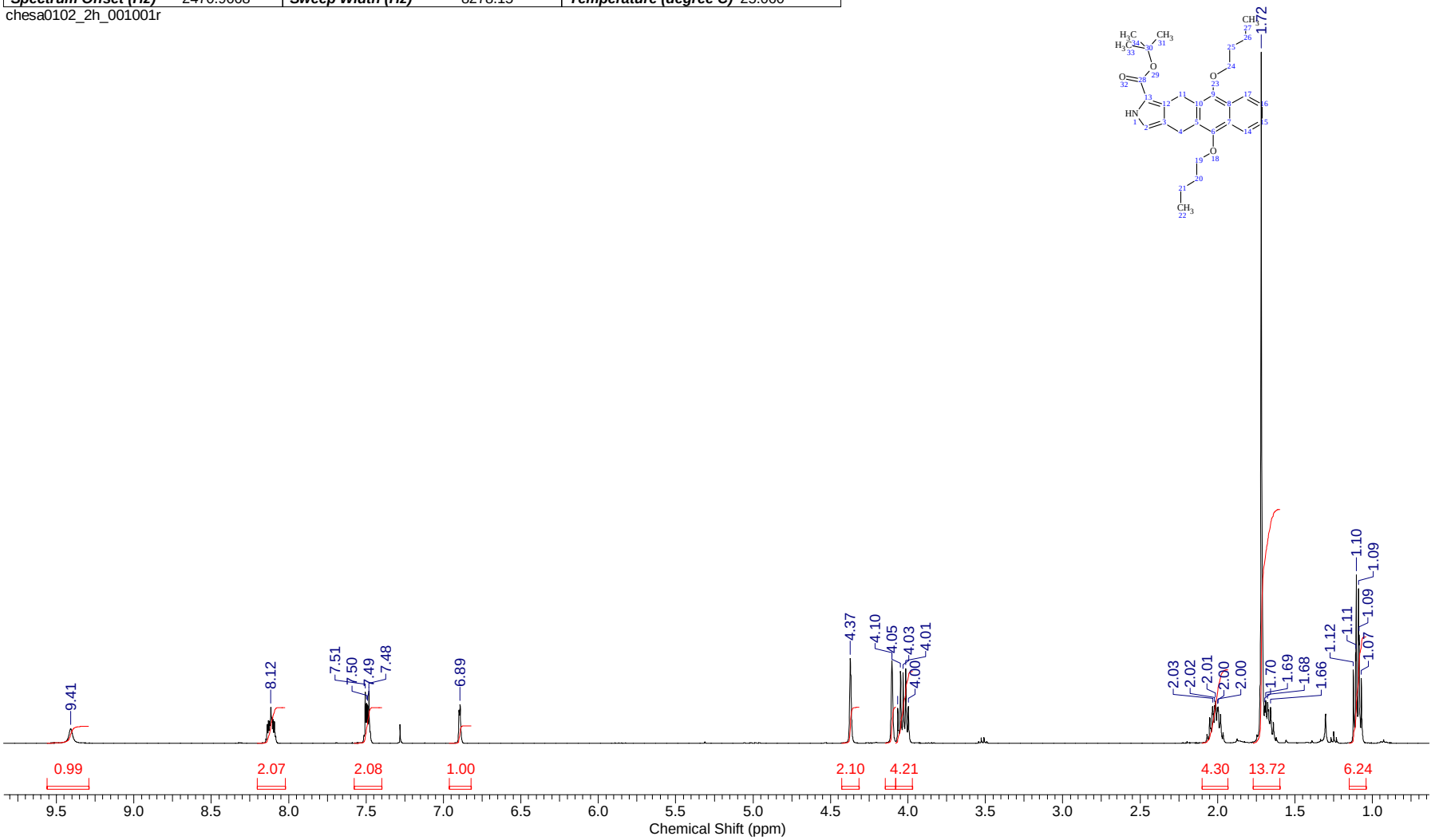

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Figure S27. ^{1}H NMR spectrum for compound **22a** in $CDCl_3$

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Original Points Count	4794	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	10060.8018	***Sweep Width (Hz)***	27624.31	***Temperature (degree C)***	25.160		

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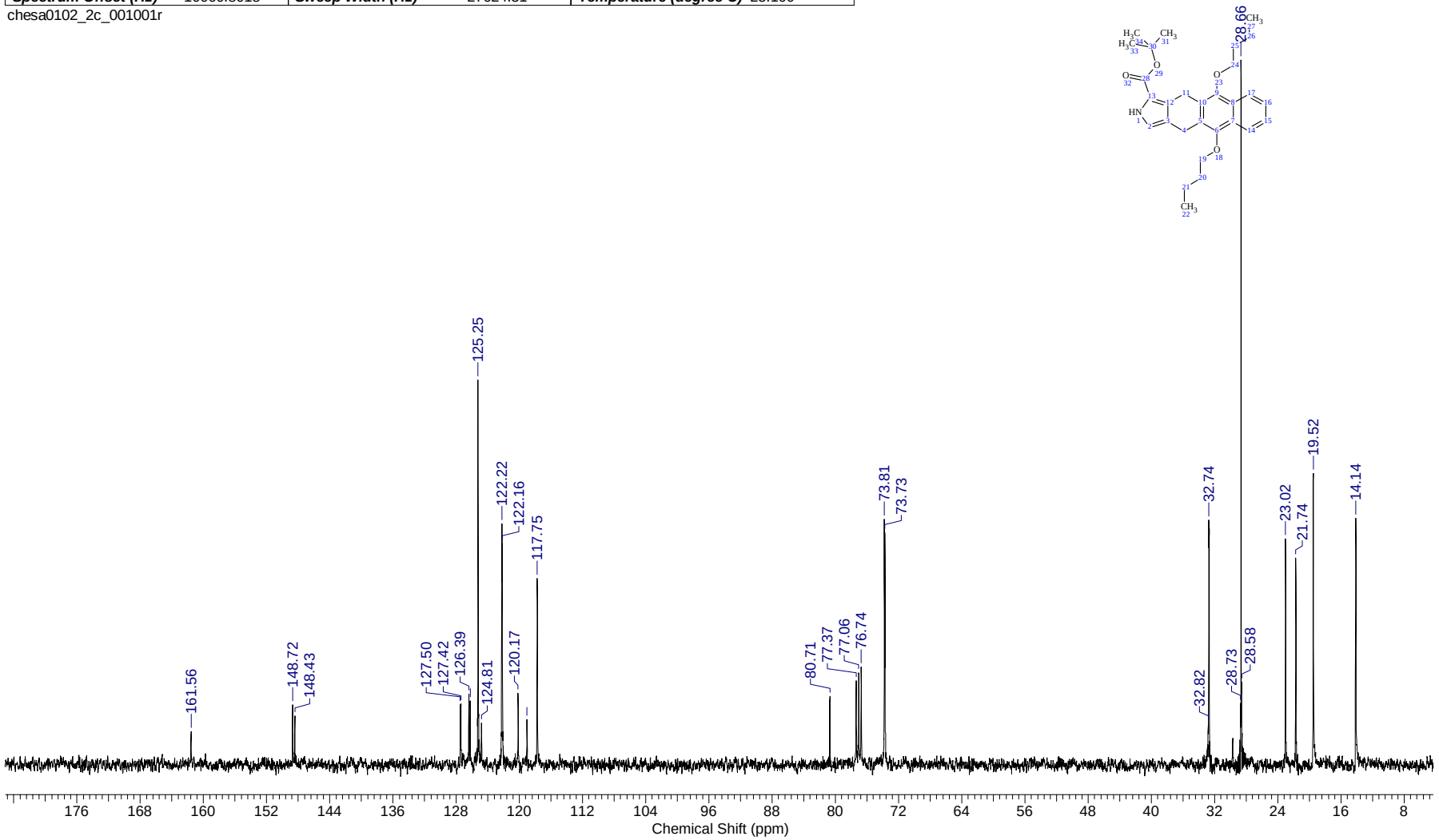

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Figure S28. ^{13}C NMR spectrum for compound **22a** in $CDCl_3$

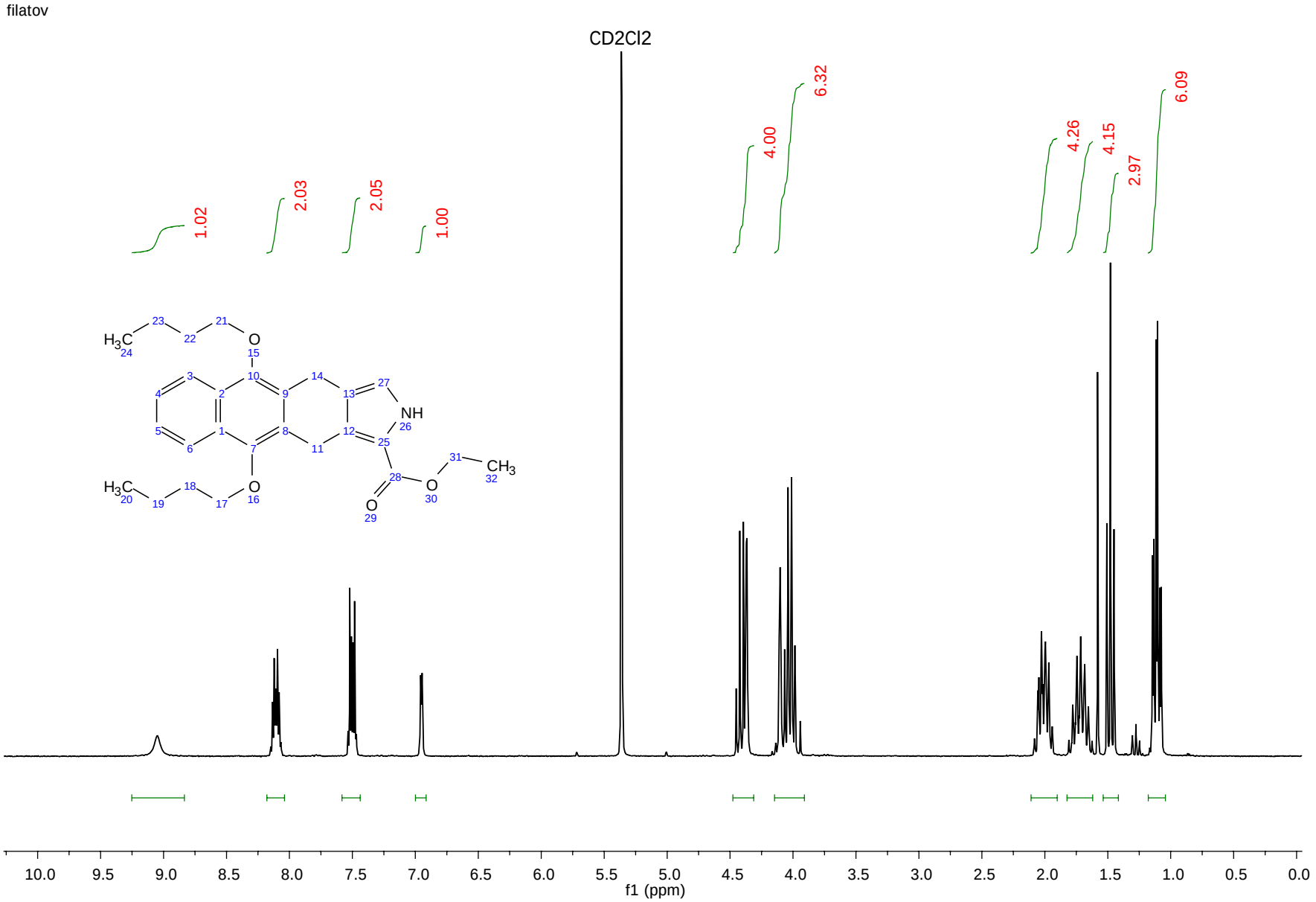

Figure S29. ^{1}H NMR spectrum for compound **22b** in CD_2Cl_2

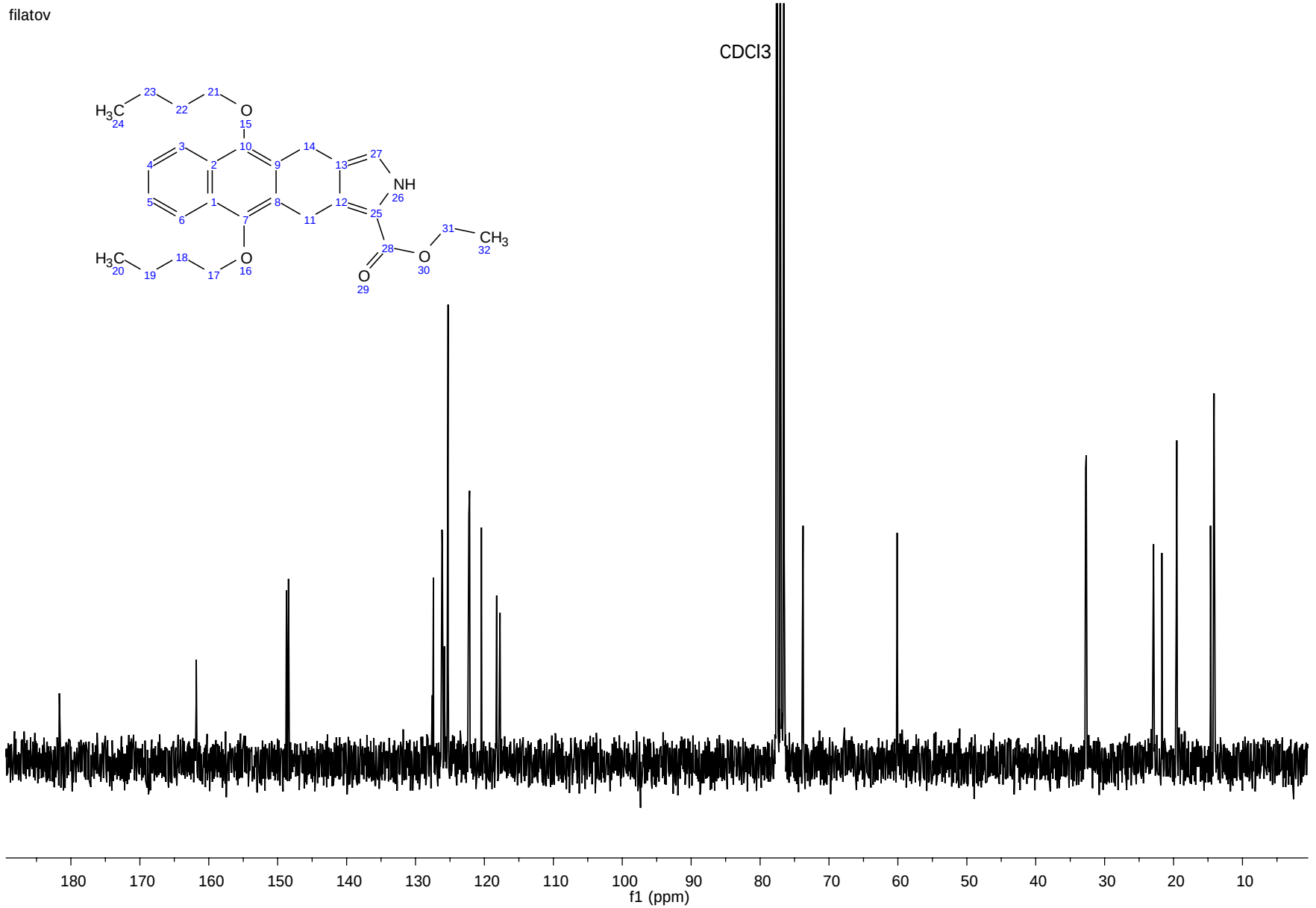

Figure S30. ^{13}C NMR spectrum for compound **22b** in CD_2Cl_2

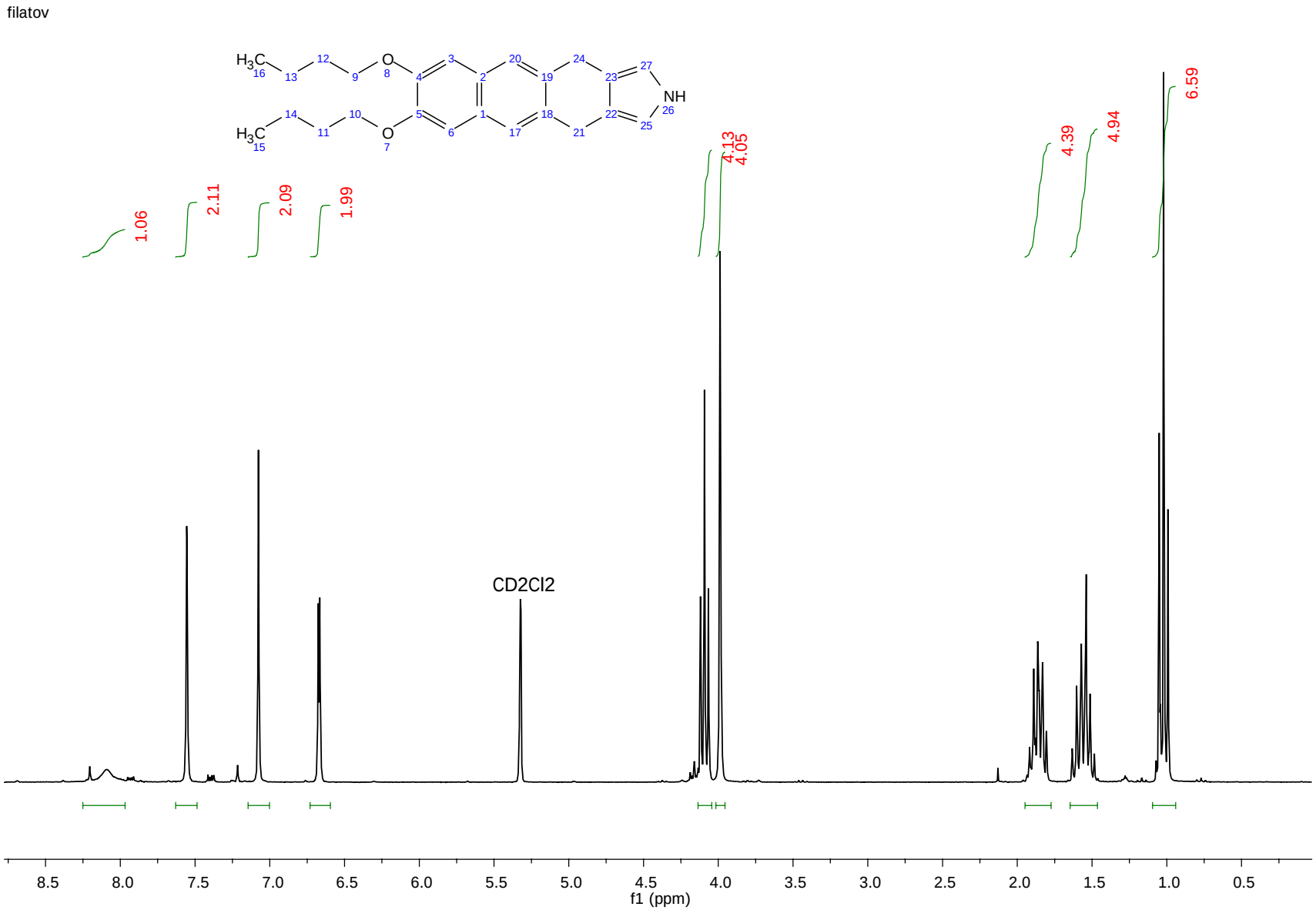

Figure S31. ^{1}H NMR spectrum for compound **15** in CD_2Cl_2

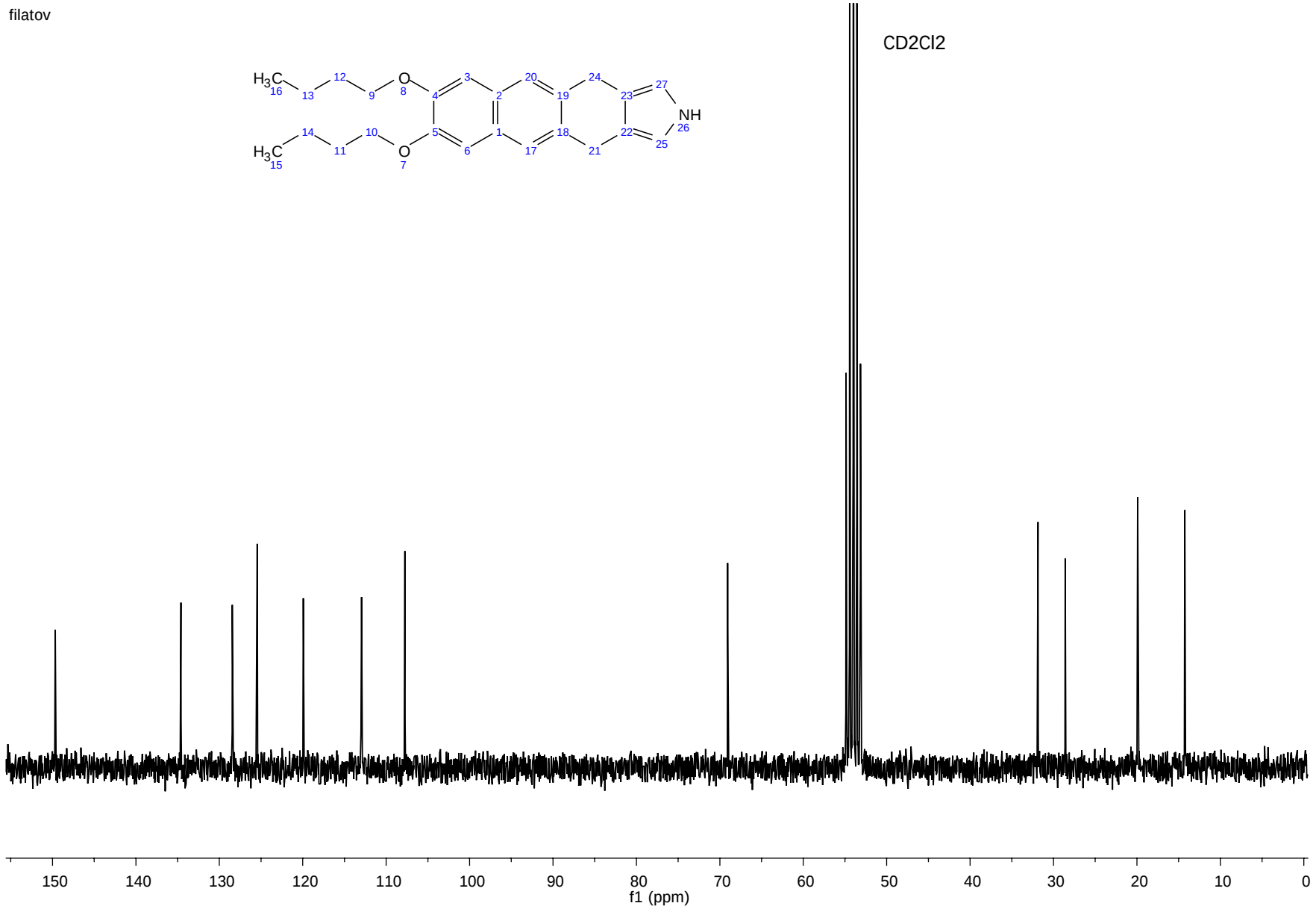

Figure S32. ^{13}C NMR spectrum for compound **15** in CD_2Cl_2

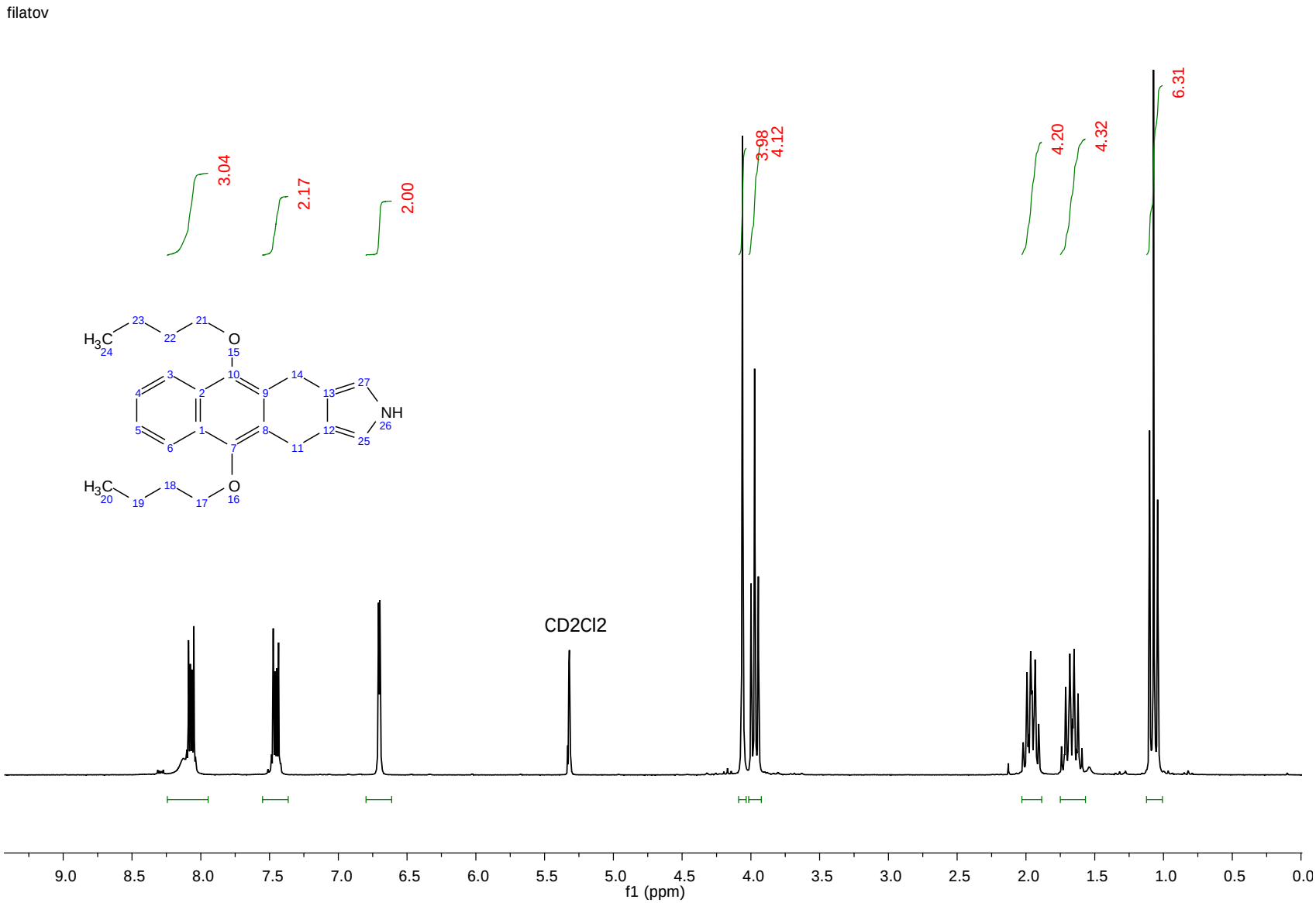

Figure S33. ^{1}H NMR spectrum for compound **23** in CD_2Cl_2

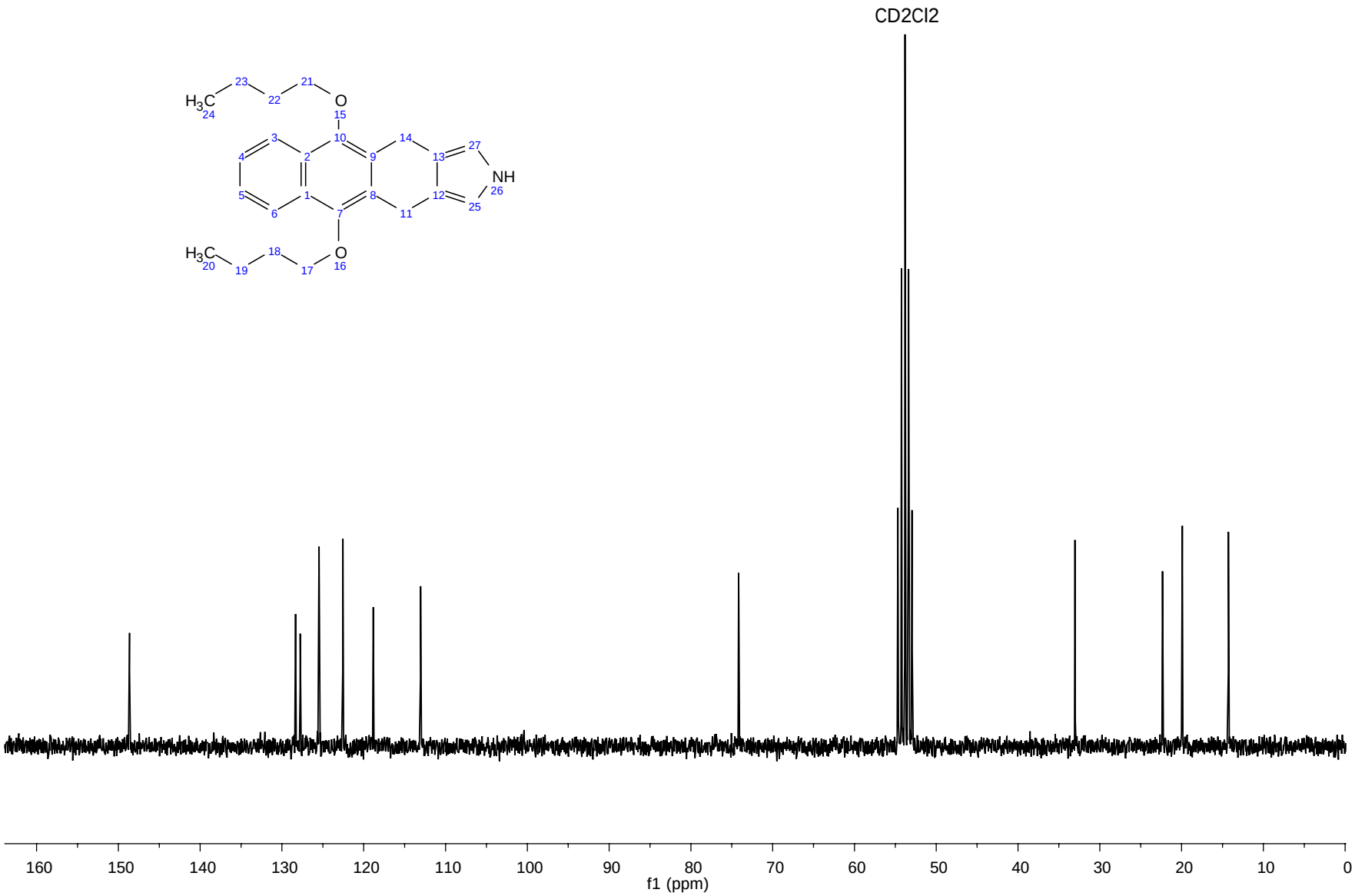

Figure S34. ^{13}C NMR spectrum for compound **23** in CD_2Cl_2

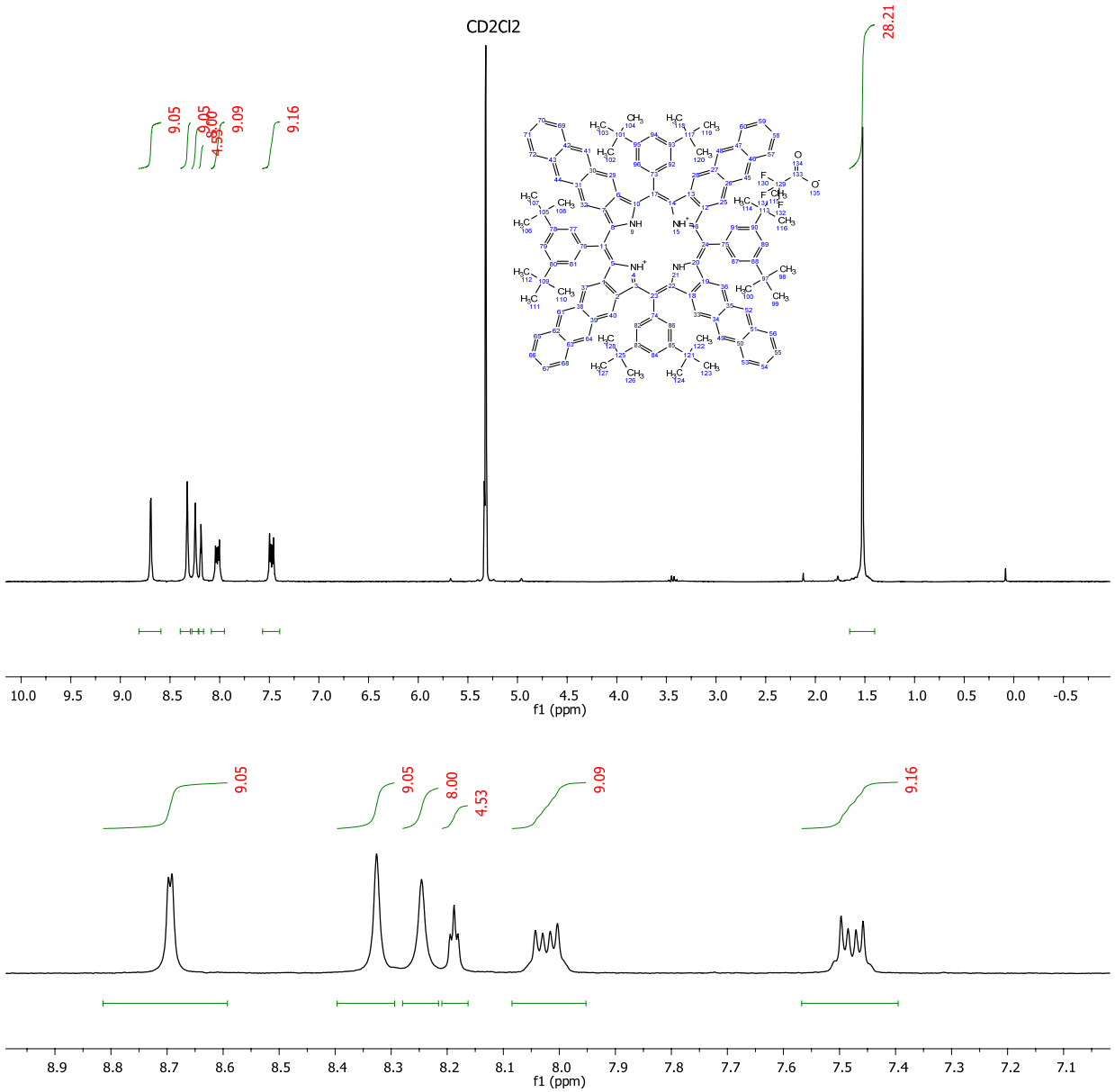

Figure S35. ^{1}H NMR (500 MHz) spectrum for protonated form of porphyrin **5b** in CD_2Cl_2

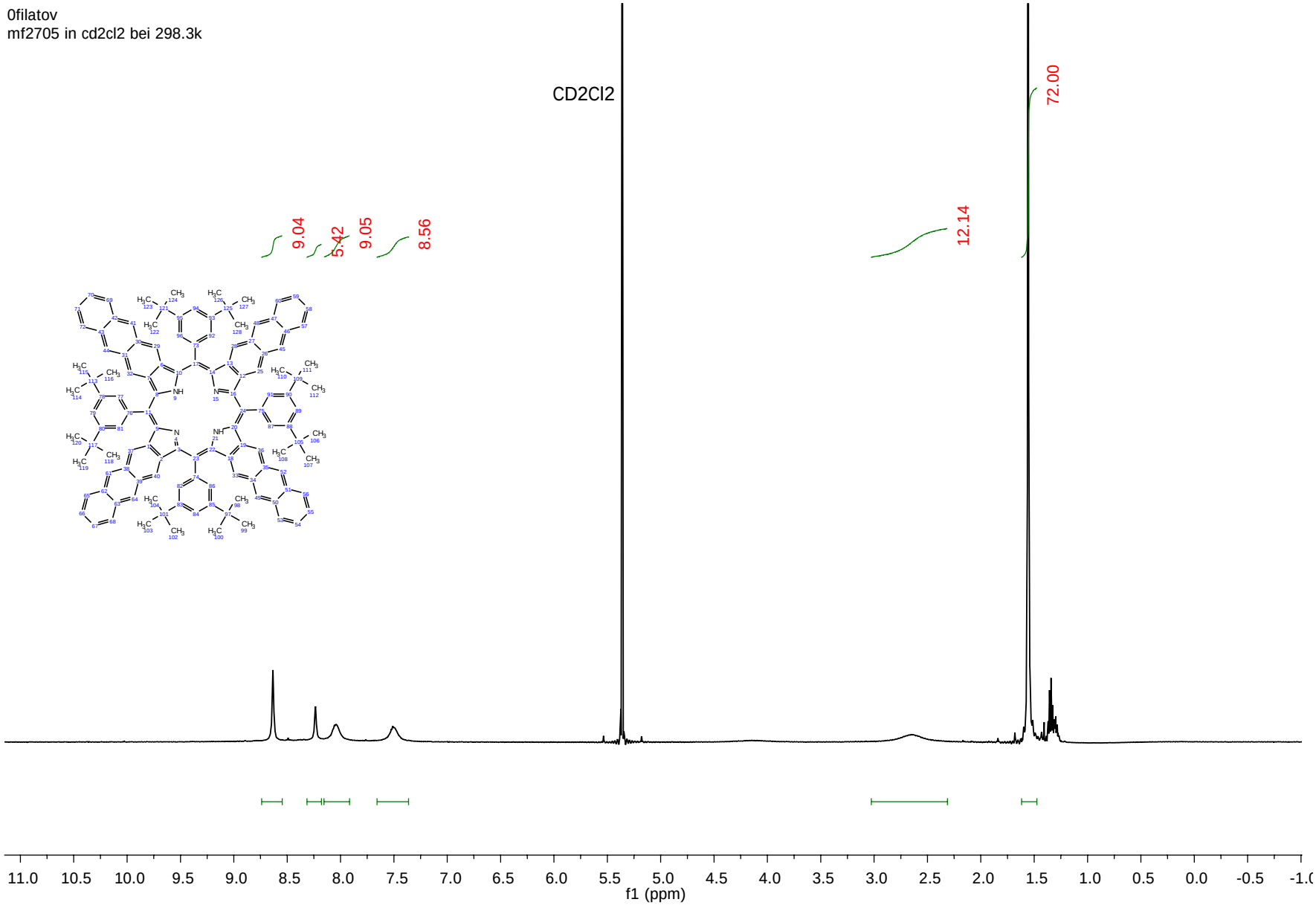

Figure S36. Figure S35. ^{1}H NMR (500 MHz) spectrum for free-base form of porphyrin **5b** in CD_2Cl_2

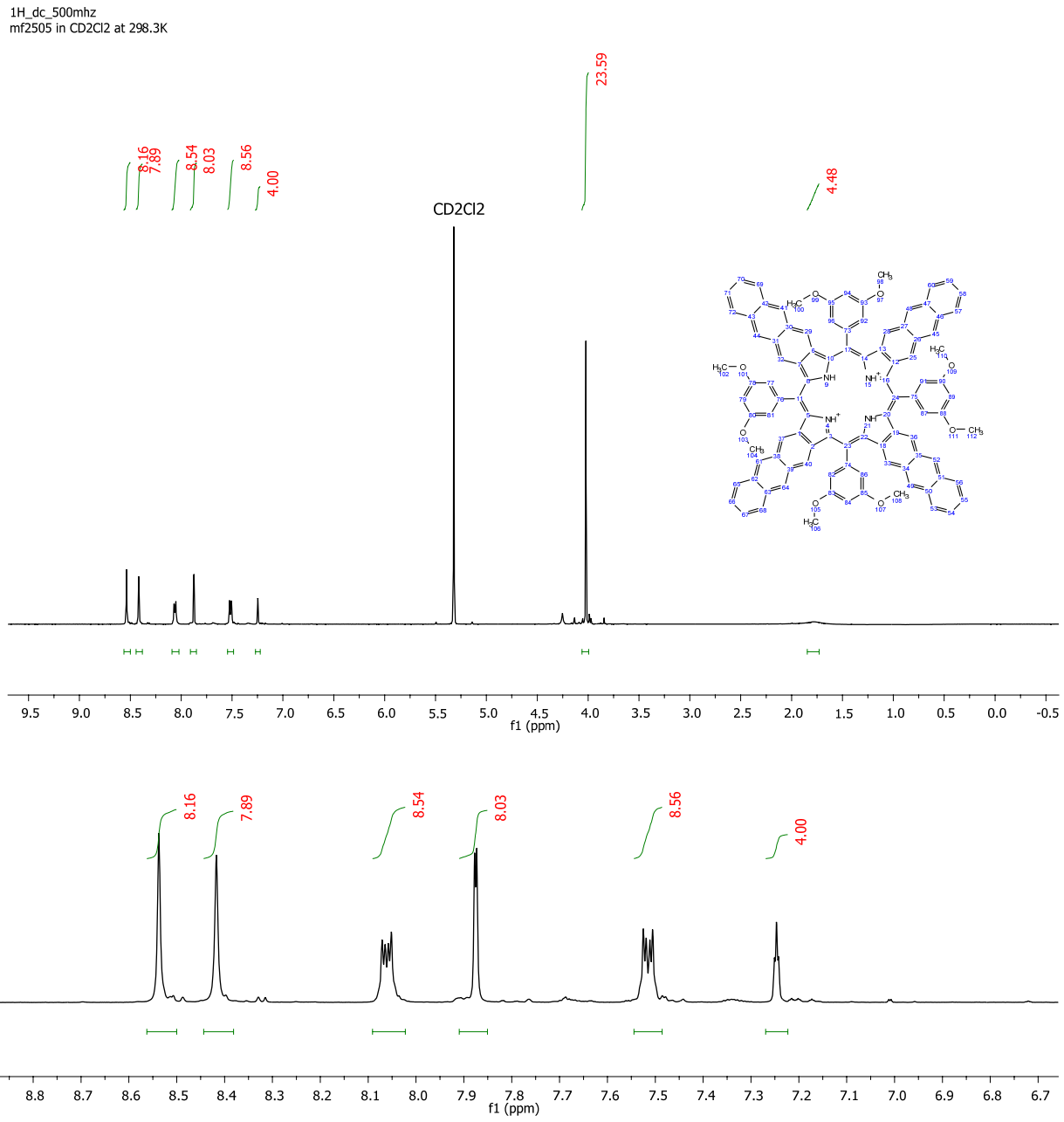

Figure S37. ^{1}H NMR spectrum for protonated form of porphyrin **5c** in CD_2Cl_2

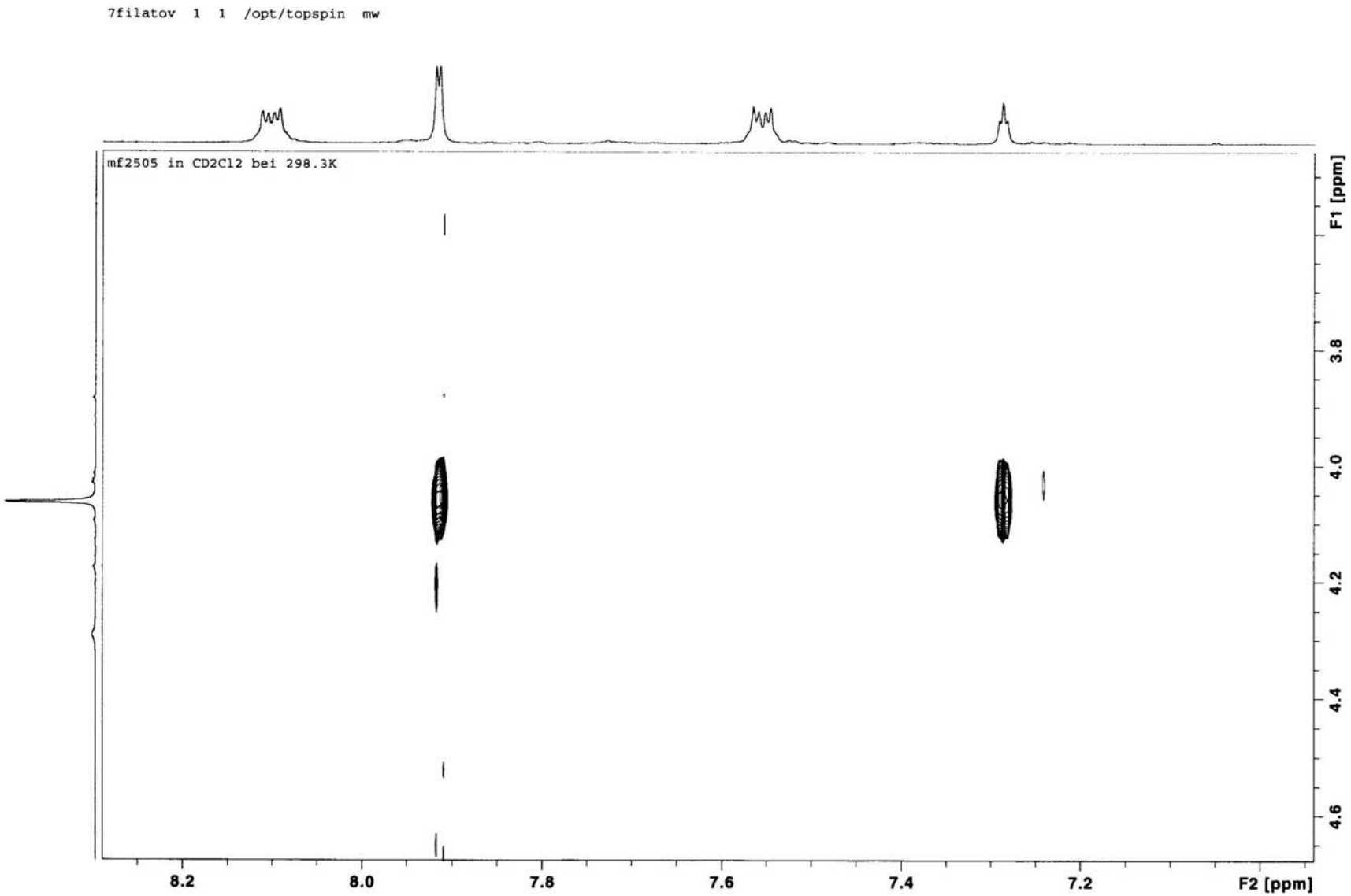

Figure S39. 2D COSY NMR spectrum for protonated form of porphyrin **5c** (correlation of MeO protons and aromatic protons of *meso*-aryl) in CD_2Cl_2

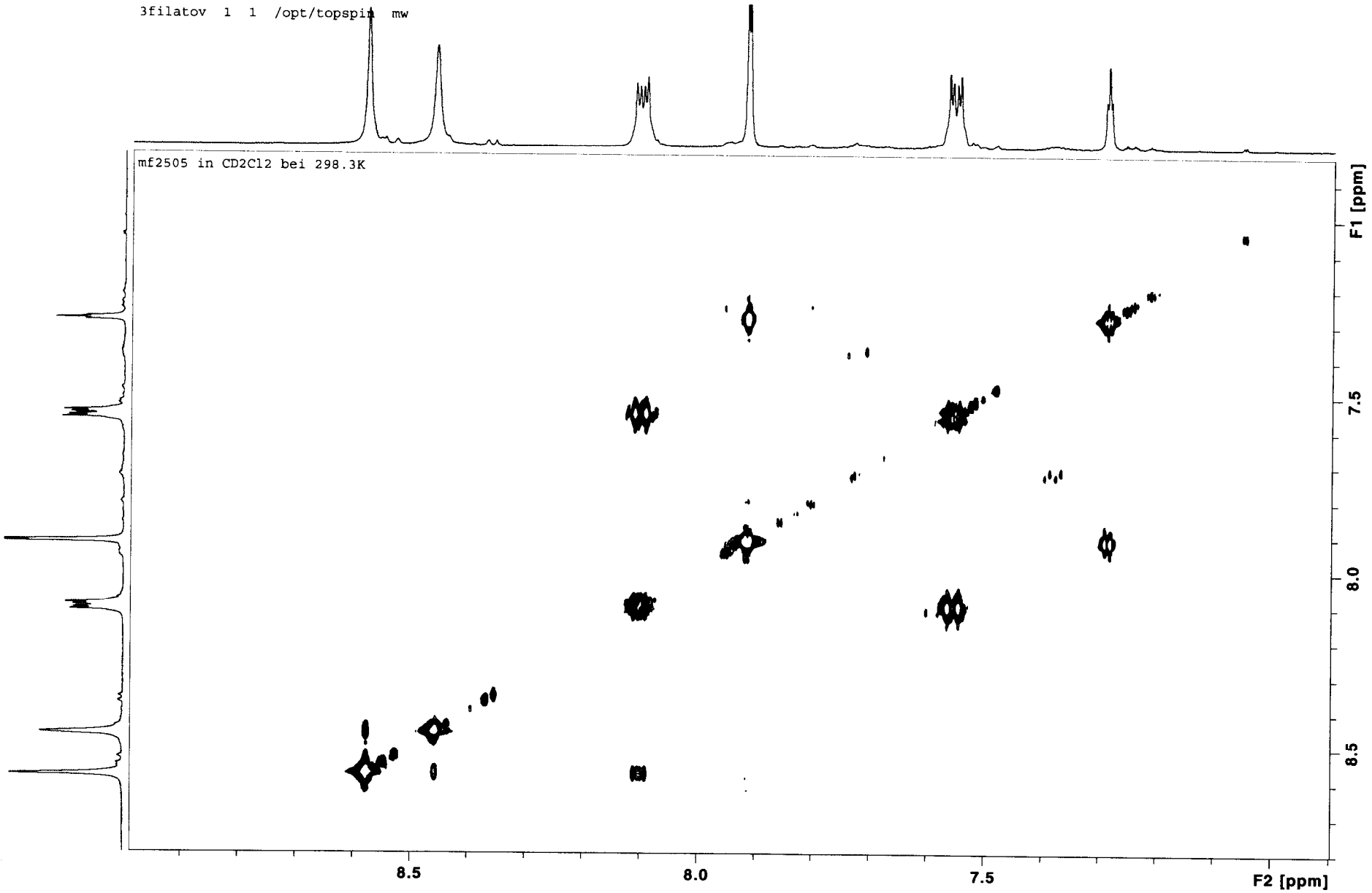

Figure S40. 2D COSY NMR spectrum (aromatic part) for protonated form of porphyrin **5c** in CD_2Cl_2

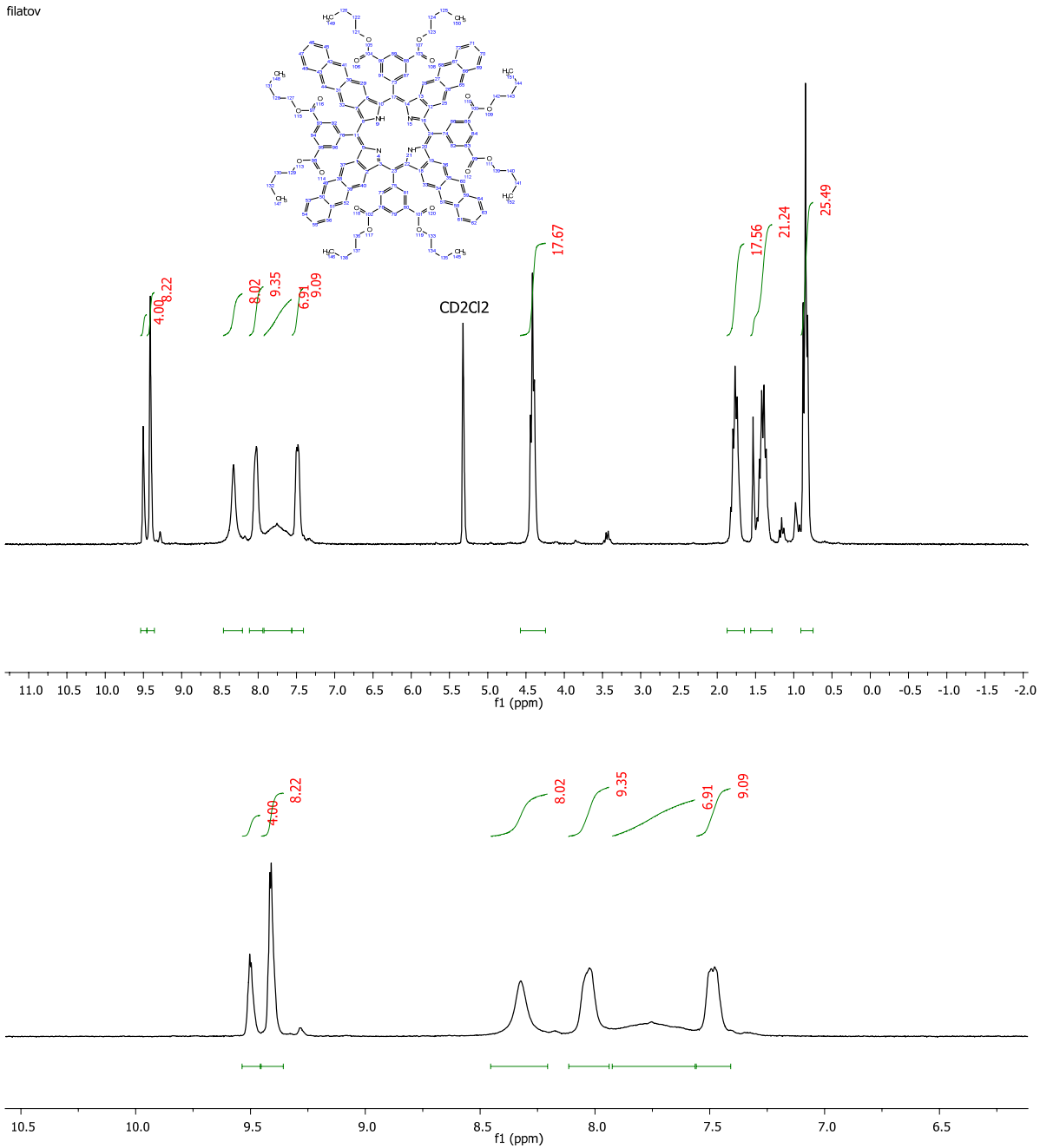

Figure S41. ^{1}H NMR spectrum for protonated form of porphyrin **5d** in CD_2Cl_2

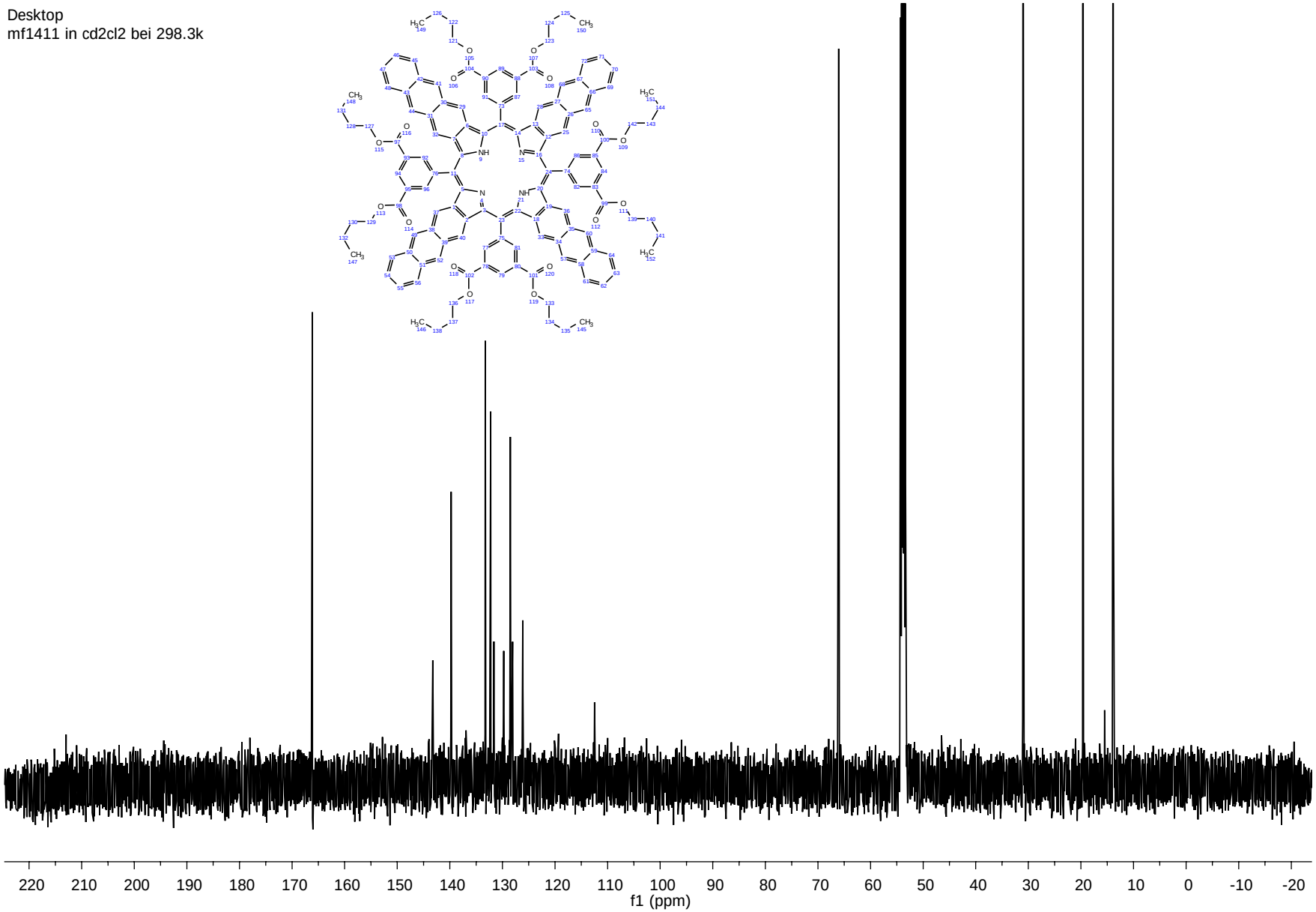

Figure S42. ^{13}C NMR (125 MHz, 10000 scans) spectrum (quaternary carbons partially unresolved) for protonated form of porphyrin **5d** in CD_2Cl_2 (1 mg in 0.6 ml)

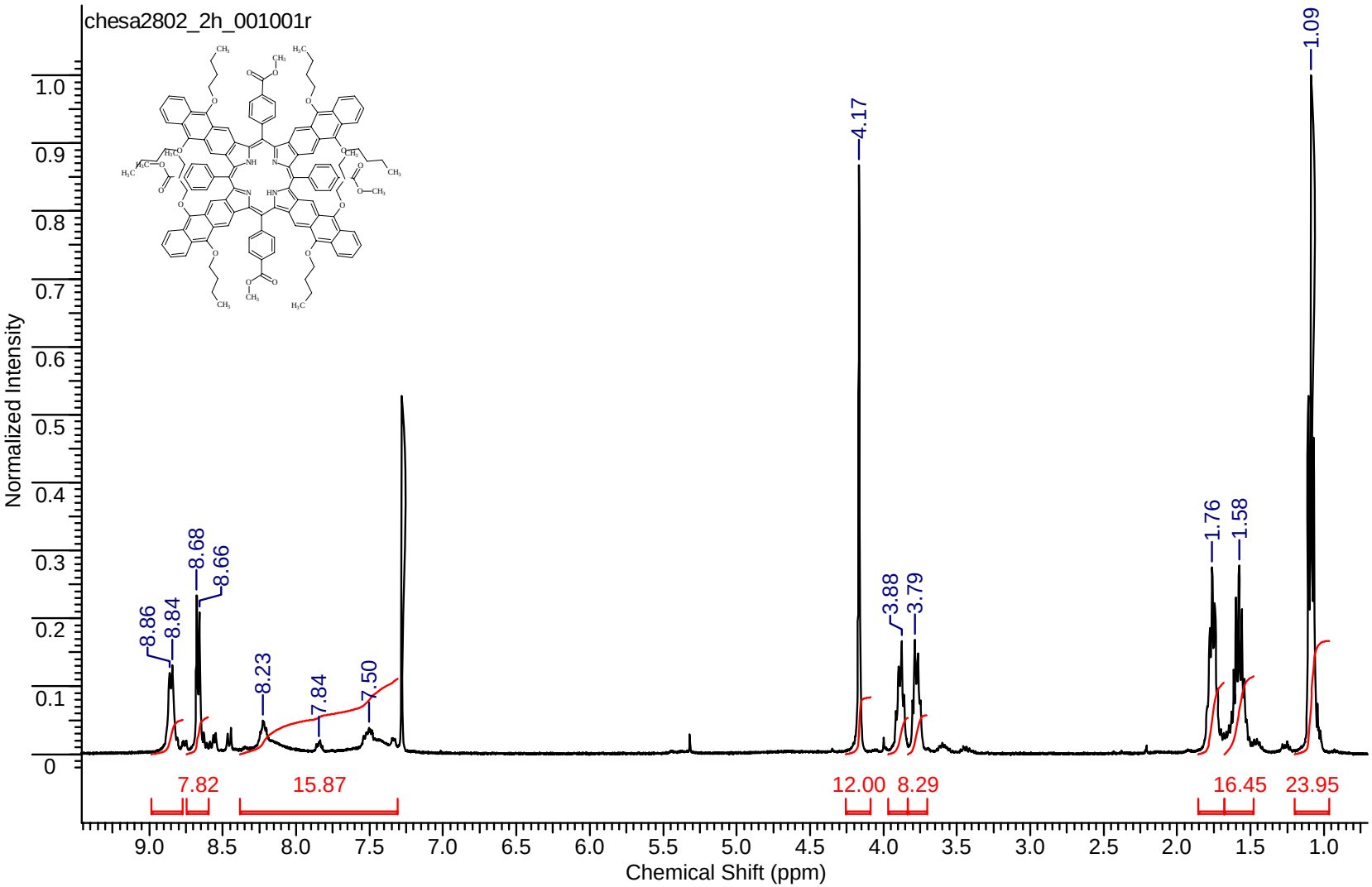

Figure S40. ^{1}H NMR spectrum for protonated form of porphyrin **24** in $CDCl_3$

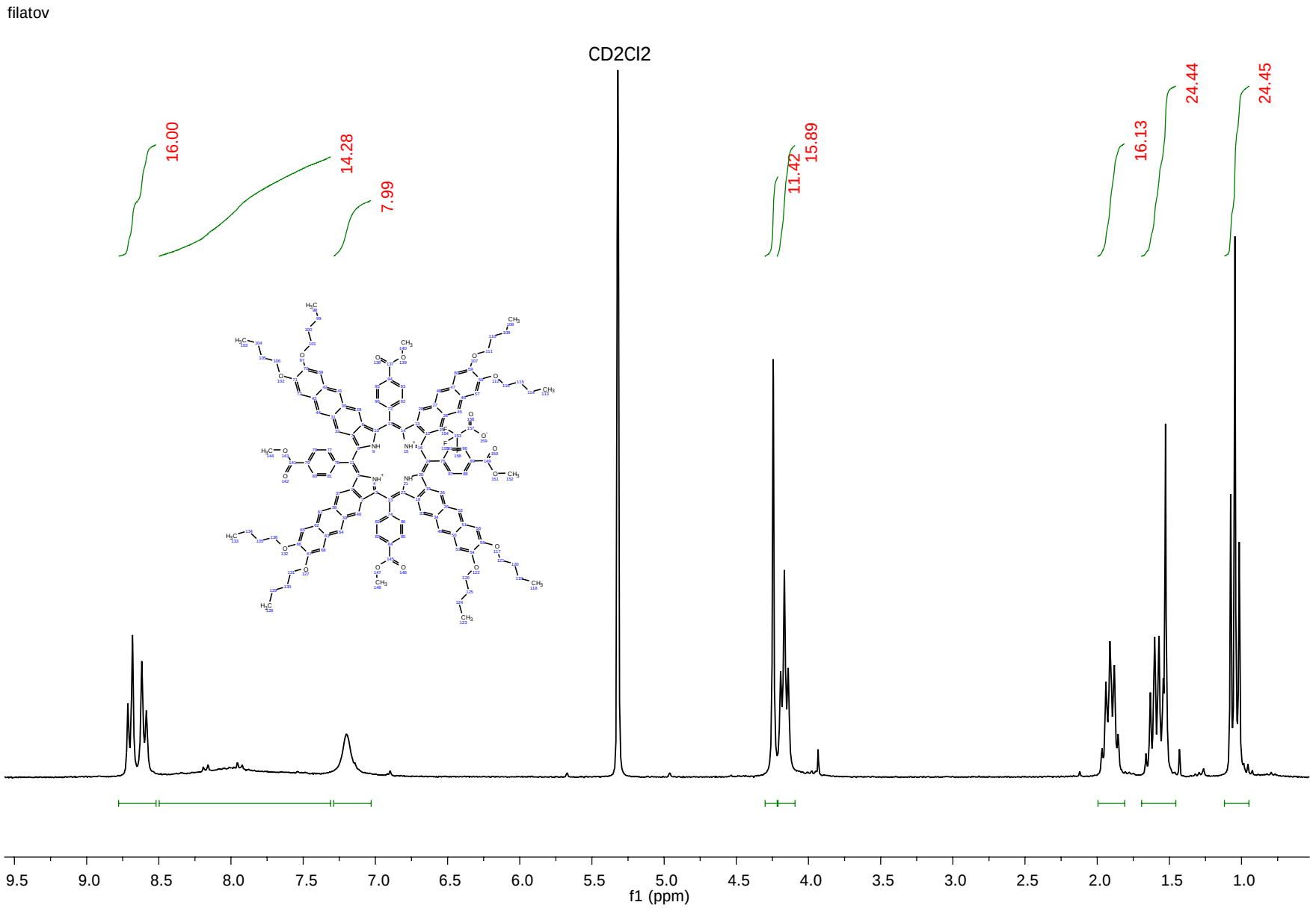

Figure S41. ^{1}H NMR spectrum for protonated form of porphyrin **16** in CD_2Cl_2

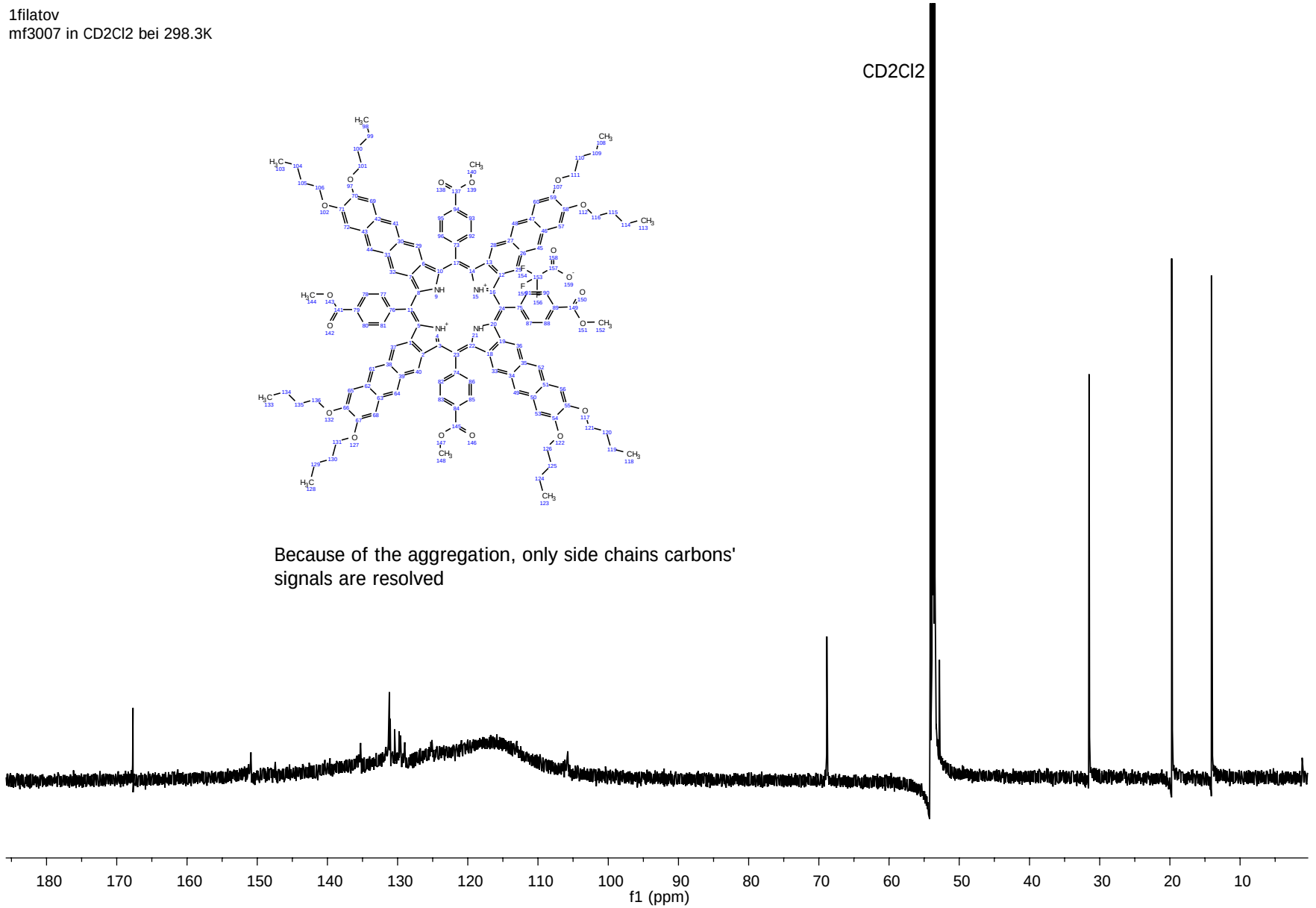

Figure S42. Partially resolved ^{13}C NMR (125 MHz, 10000 scans) spectrum for protonated form of porphyrin **16** in CD_2Cl_2 (1 mg in 0.6 ml)

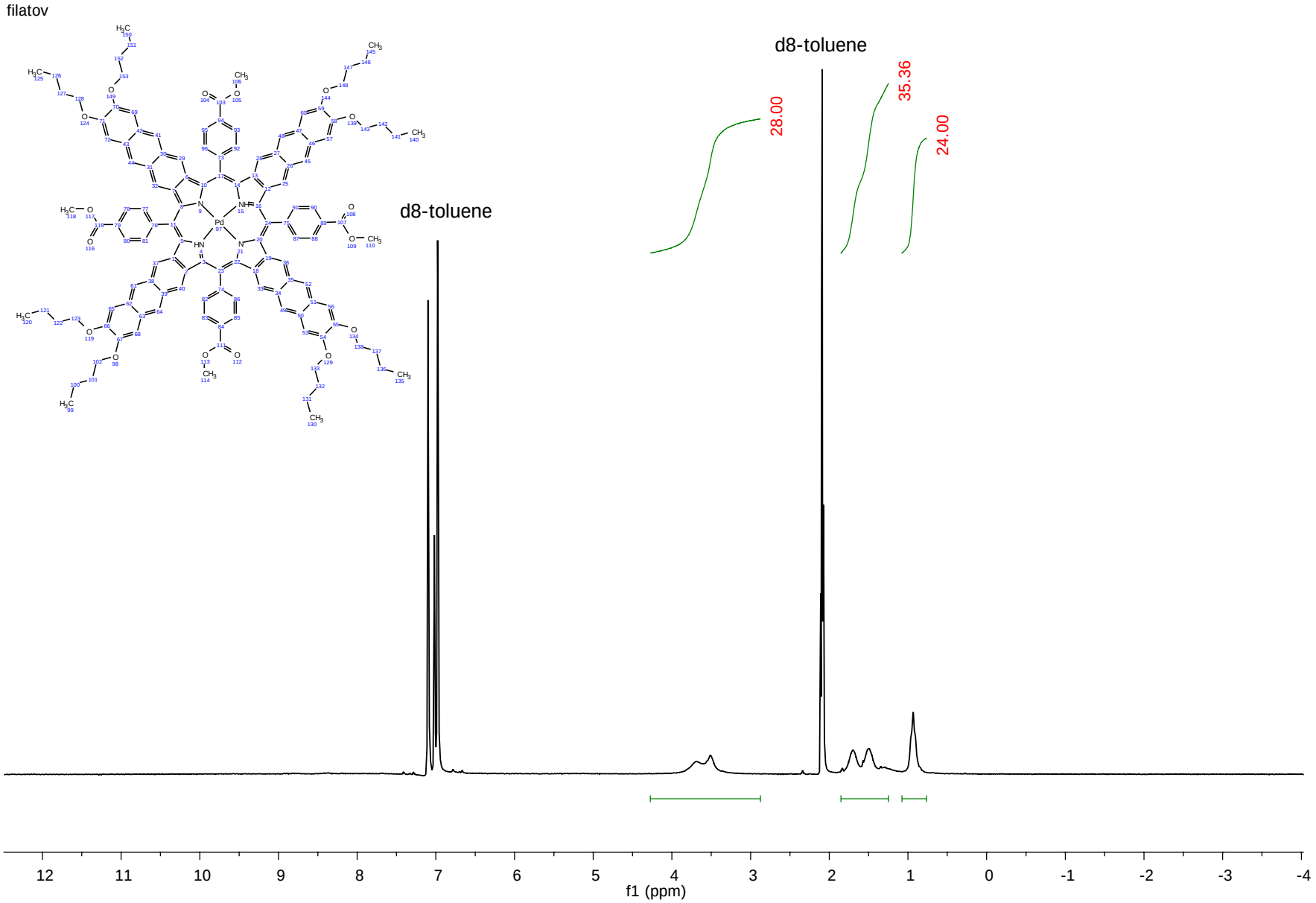

Figure S42. ^{1}H NMR spectrum for of porphyrin **Pd-16** in d^{8}-toluene at 298 K

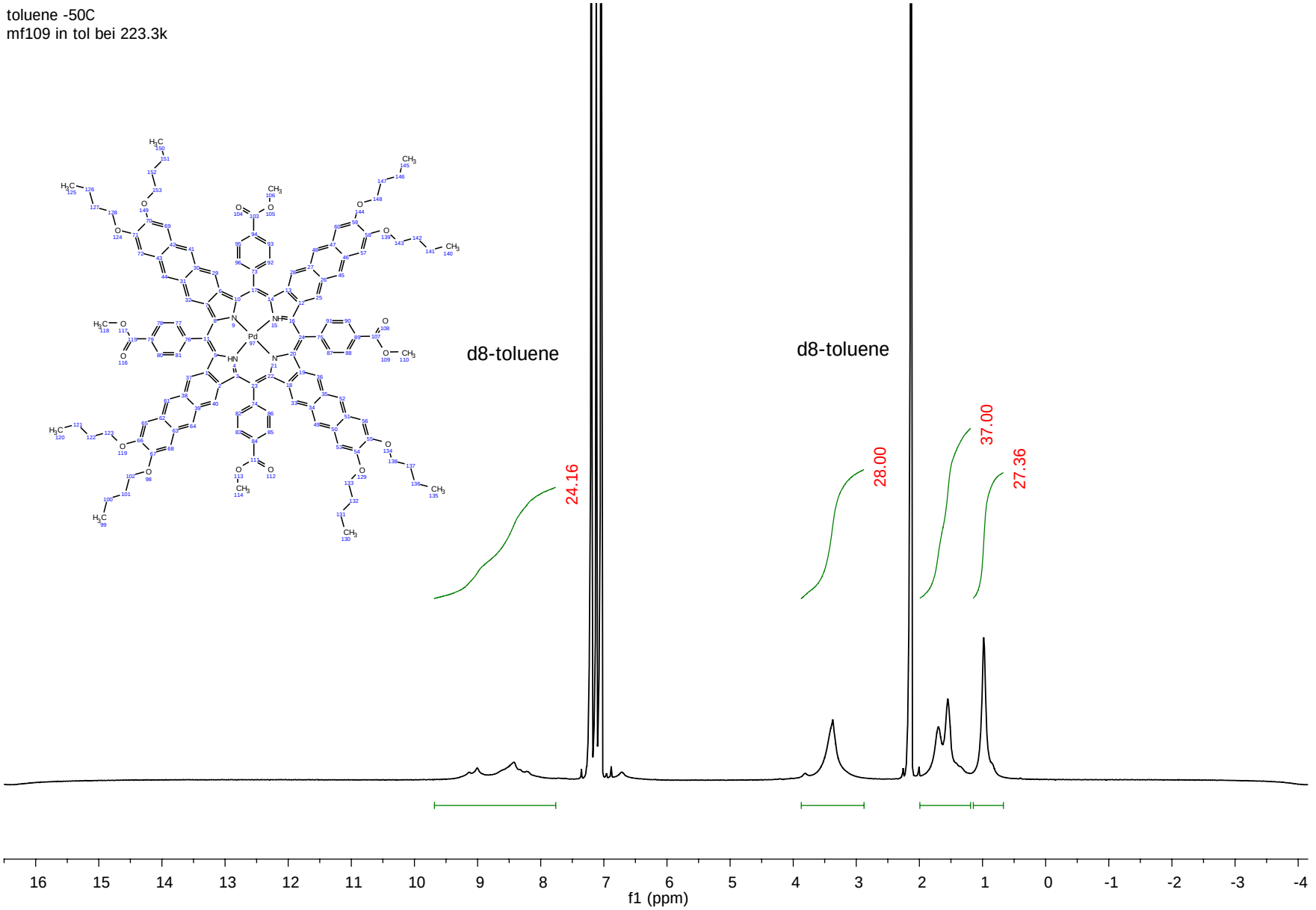

Figure S43. ^{1}H NMR spectrum for of porphyrin **Pd-16** in d^{8}-toluene at 223 K

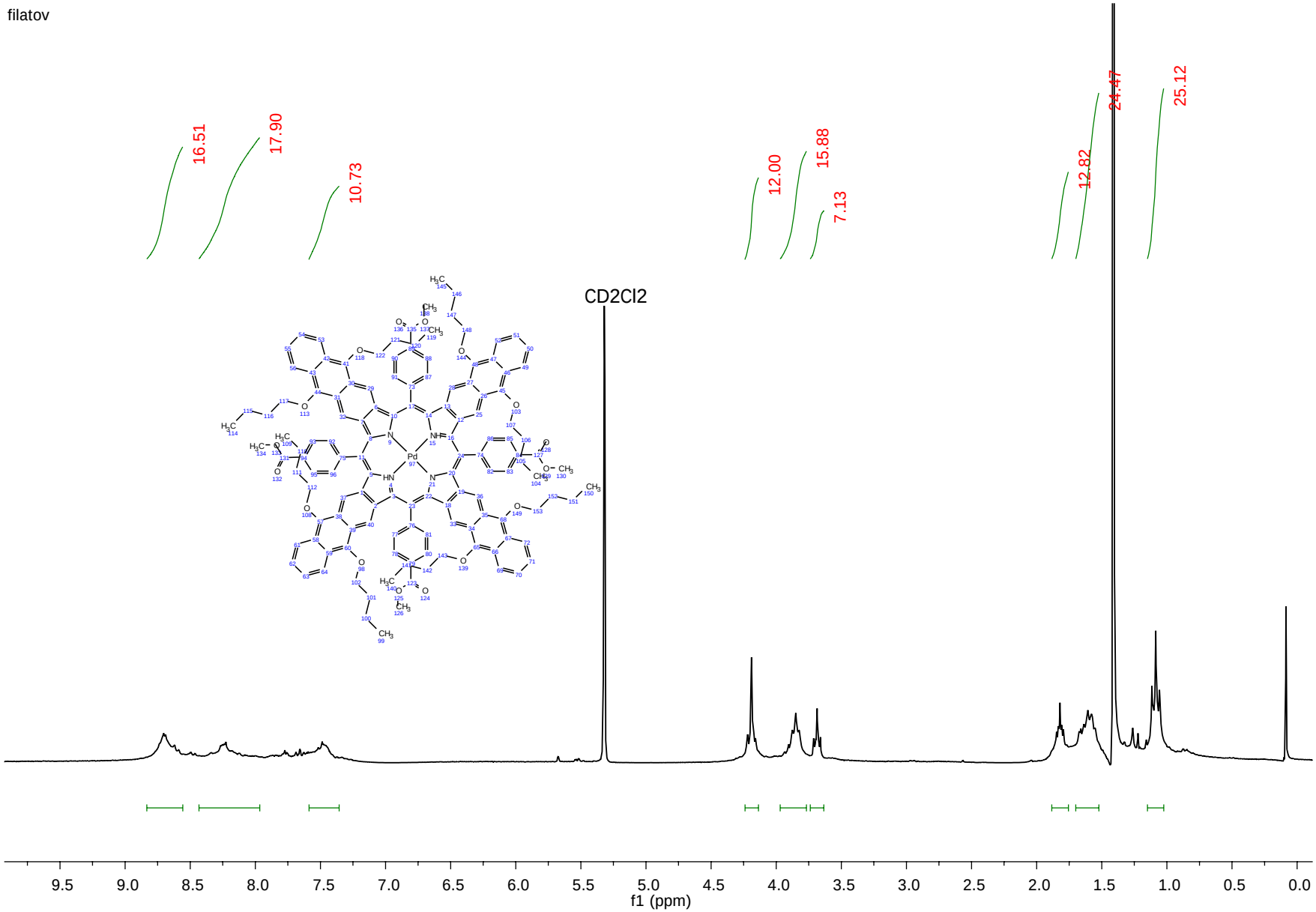

Figure S43. ^{1}H NMR spectrum for protonated form of porphyrin **Pd-24** in CD_2Cl_2

MALDI-TOF spectra

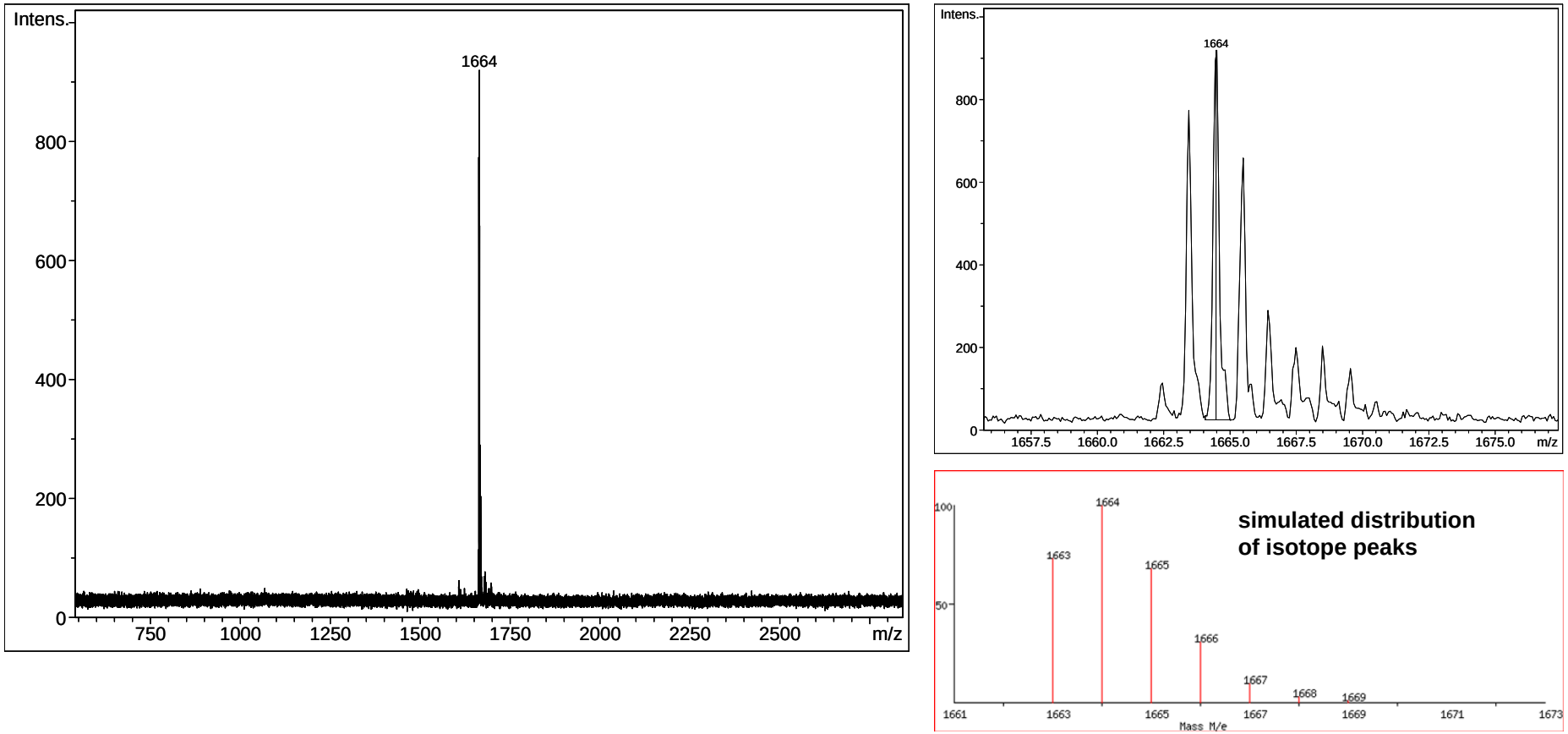

Figure S38. MALDI-TOF spectrum for porphyrin **5b**

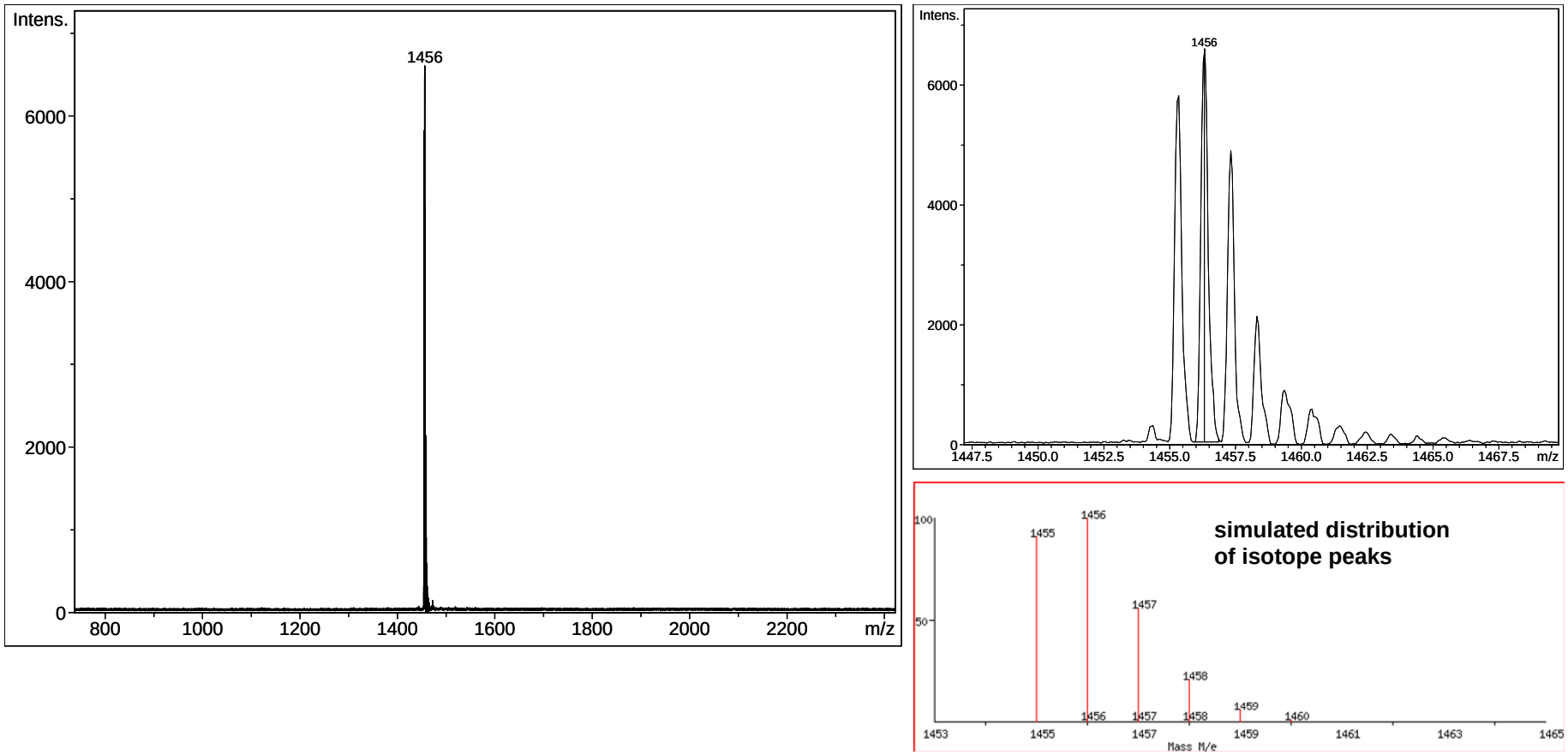

Figure S39. MALDI-TOF spectrum for porphyrin **5c**

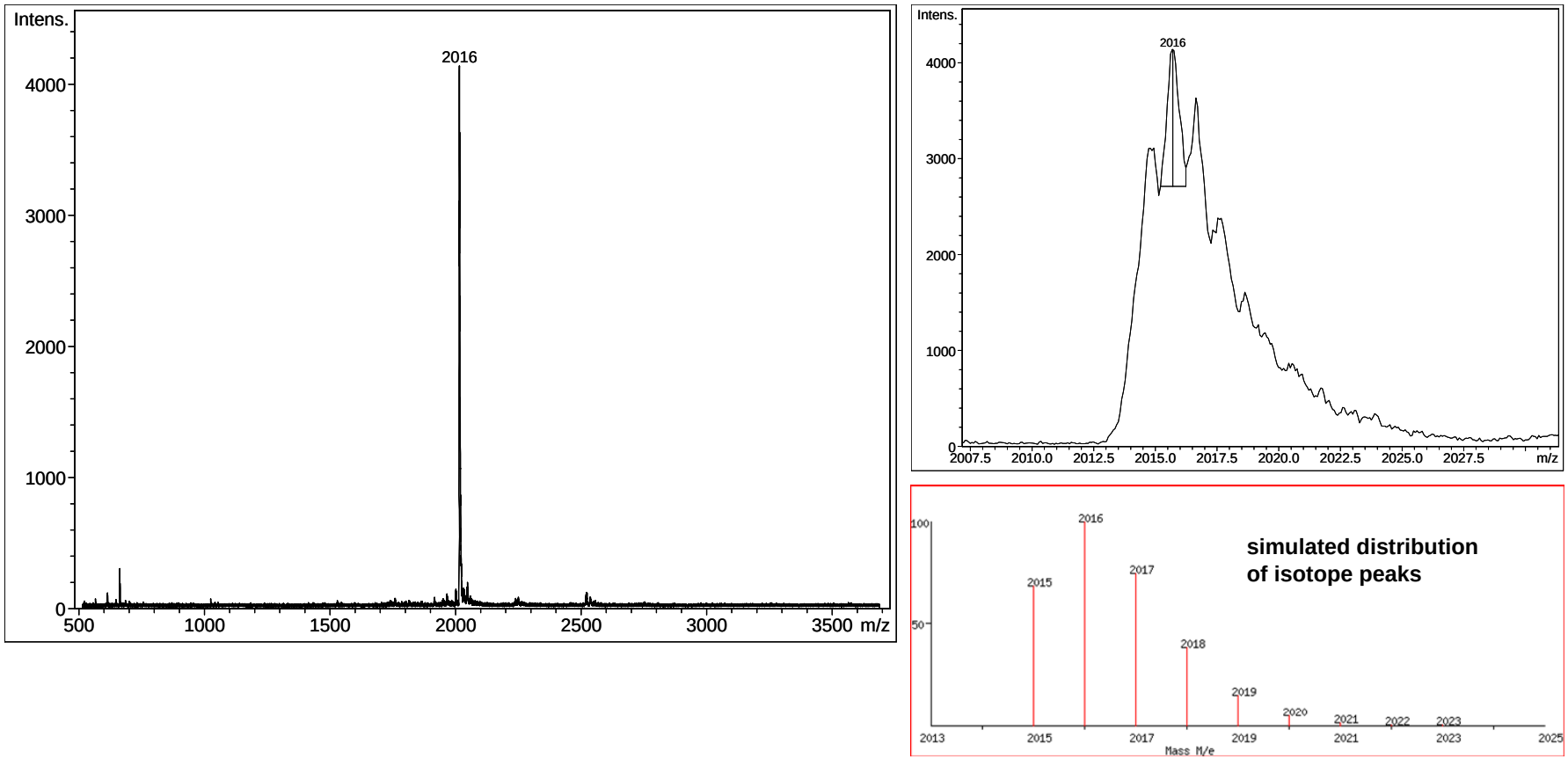

Figure S40. MALDI-TOF spectrum for porphyrin **5d**

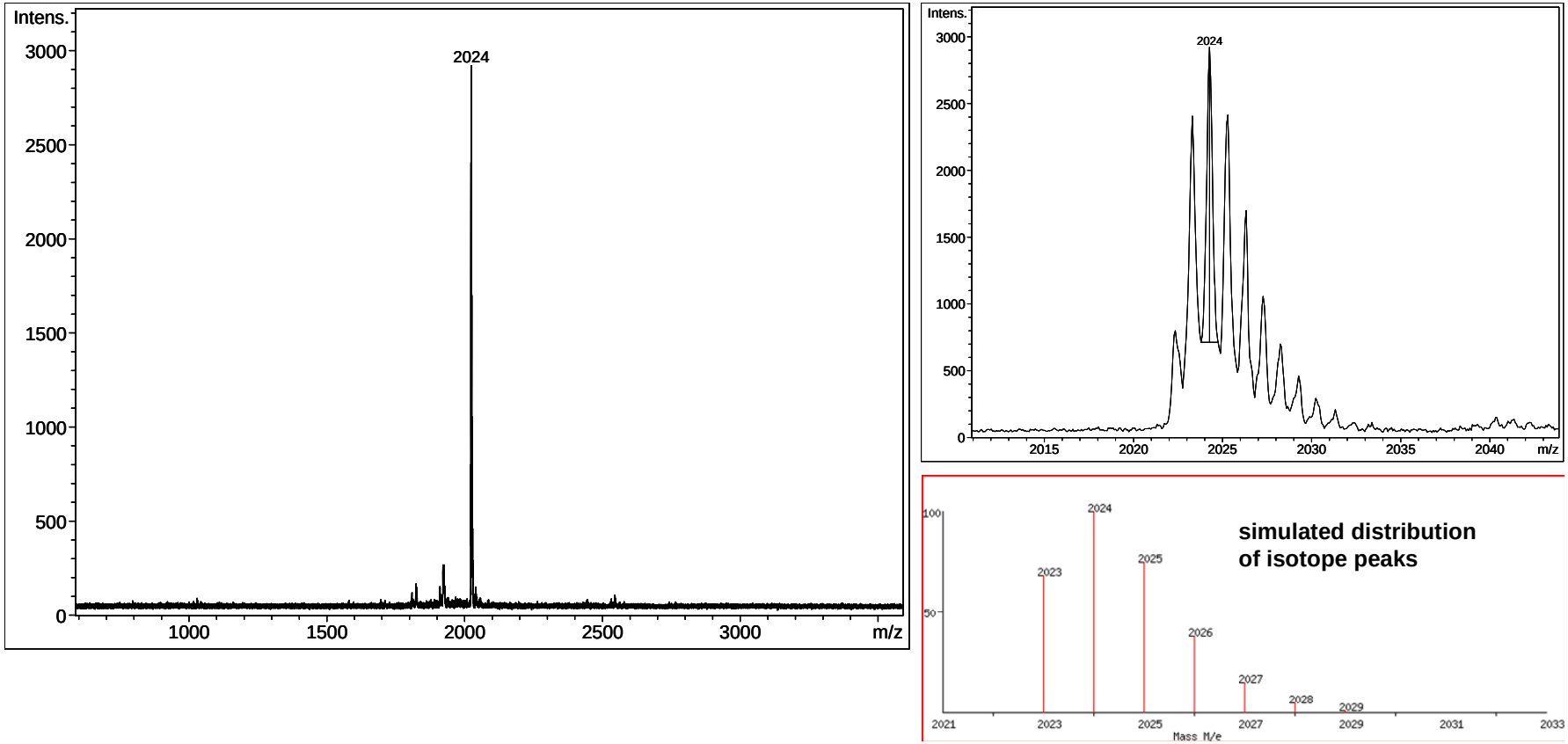

Figure S38. MALDI-TOF spectrum for porphyrin **24**

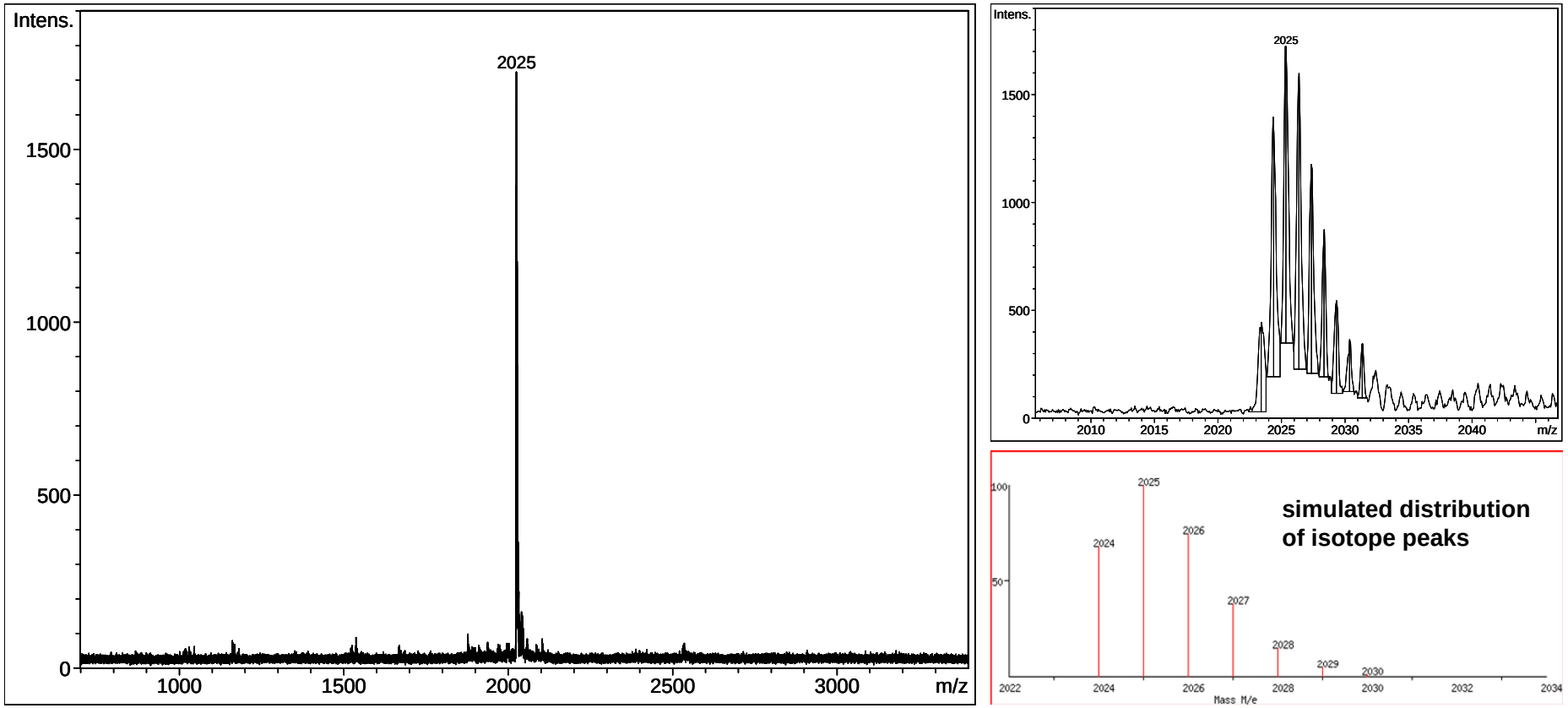

Figure S39. MALDI-TOF spectrum for porphyrin **16**

X-Ray Structure Determination

Crystals of **5d** were obtained by slow evaporation of a CH_2Cl_2 solution. As the crystals contain the solvent, crystals were mounted in glass capillaries and the data collection was carried out at 120K on a Nonius KCCD diffractometer with Mo Kα radiation. **5d** crystallizes in the tetragonal space group I 4_1/a. The structure was solved by direct methods and refined on F by full-matrix least-squares cycles. The unit cell contains 32 CH_2Cl_2 molecules which are disordered and could not be resolved. These have been treated as a diffuse contribution to the overall scattering without specific atom positions by SQUEEZE/PLATON. Crystal data: Tetragonal, I 4_1/a, a = 23.0609(9), c = 27.0102(9) Å,. R = 0.0407, R_w = 0.0584. CCDC 900743 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

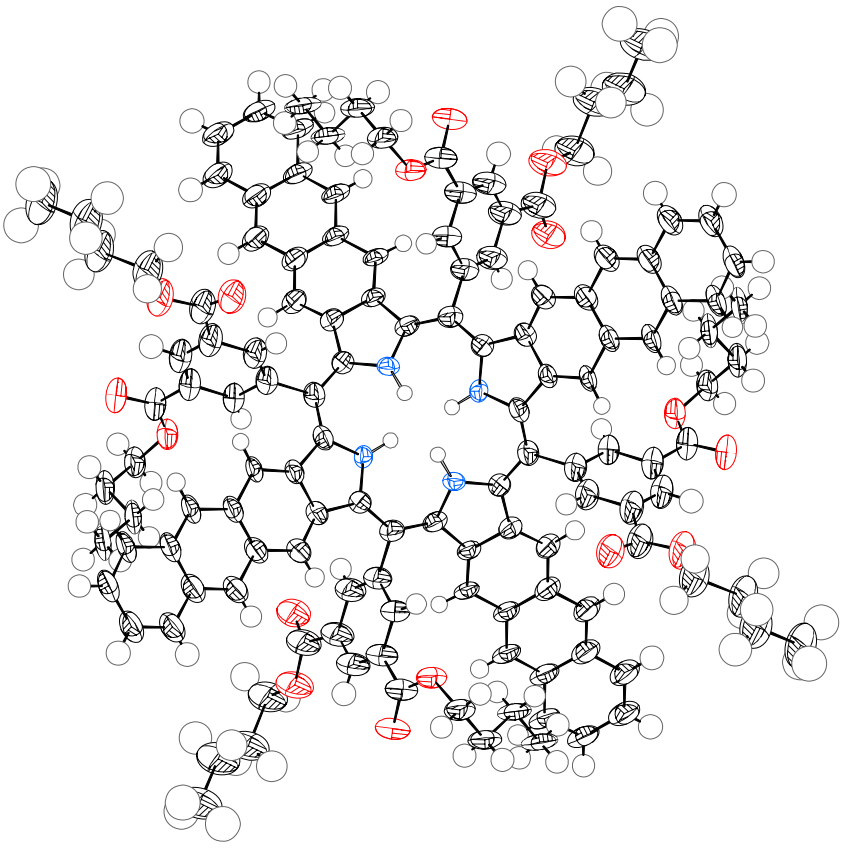

Figure S44. ORTEP drawing of **5d** with xx% probability thermal ellipsoids.

Table 1. Summary of structure determination for compound **5d**

Crystal Class	Tetragonal	Space Group I	41/a
a	23.0609(9)	alpha	90
b	23.0609(9)	beta	90
c	27.0102(9)	gamma	90
Volume	14364.2(9)	Z	16
Radiation type	Mo K\a	Wavelength	0.710730
Dx	0.93	Mr	504.10
Mu	0.061	Temperature (K)	120
Size	0.12x 0.16x 0.42		
Colour	dark blue	Shape	prism
Cell from	0 Reflections	Theta range	0 to 0
Standard Interval	0	Standard Count	0
Diffractometer type	KAPPACCD	Scan type	PHIOMEGA
Absorption type	multi-scan	Transmission range	0.94 0.99
Reflections measured	46100	Independent reflections	8244
Rint	0.0673	Theta max	27.49
Hmin, Hmax	-20 21		
Kmin, Kmax	0 29		
Lmin, Lmax	0 35		
Refinement on F			
R-factor	0.041	Weighted R-factor	0.058
		Max shift/su	0.0005
Delta Rho min	-0.07	Delta Rho max	0.06
Reflections used	3855	sigma(I) limit	2.00
Number of parameters	343	Goodness of fit	0.980