Electronic Supplementary Information (ESI)

Supramolecular assembly and spectroscopic characterization of indolenine - barbituric acid zwitterions

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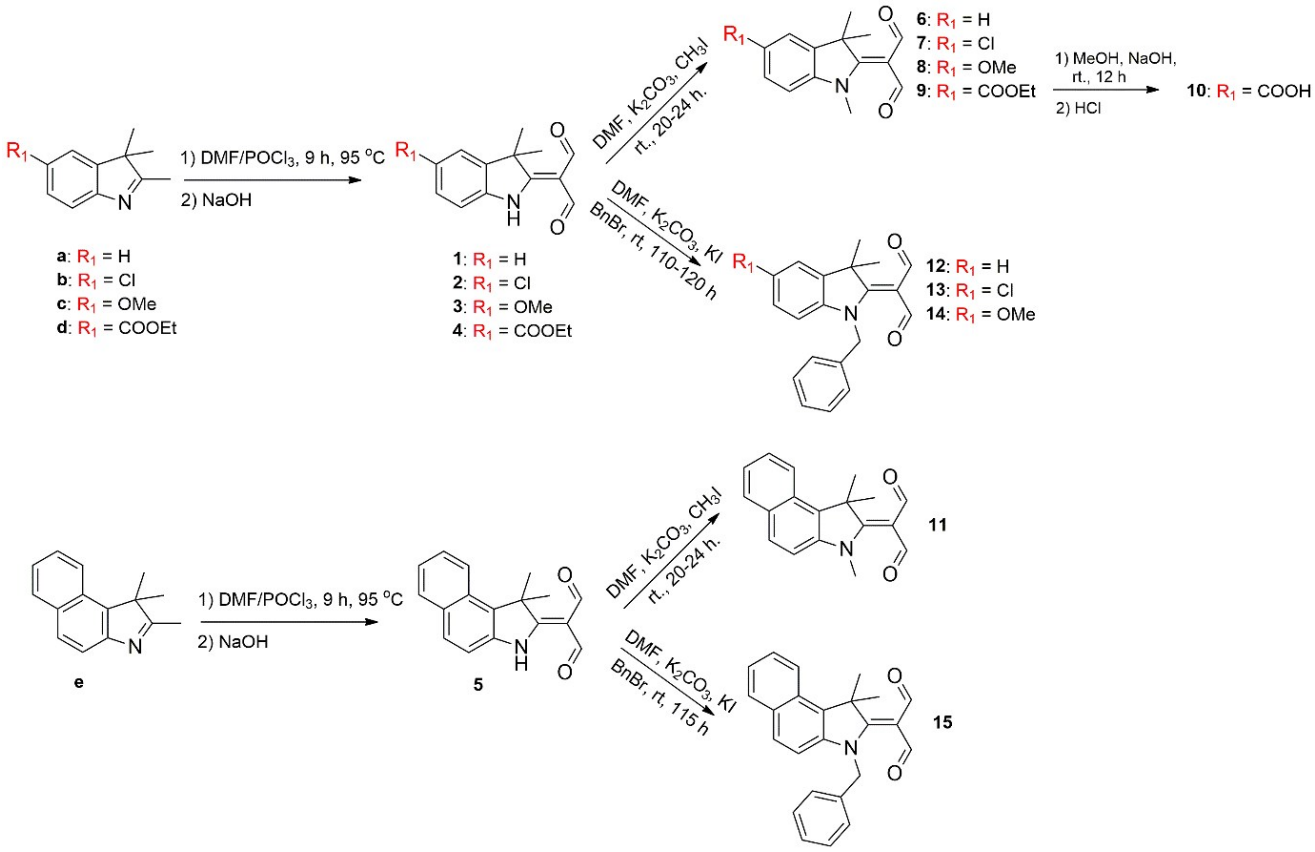


Scheme S1: Synthesis routes of **6 – 15**.

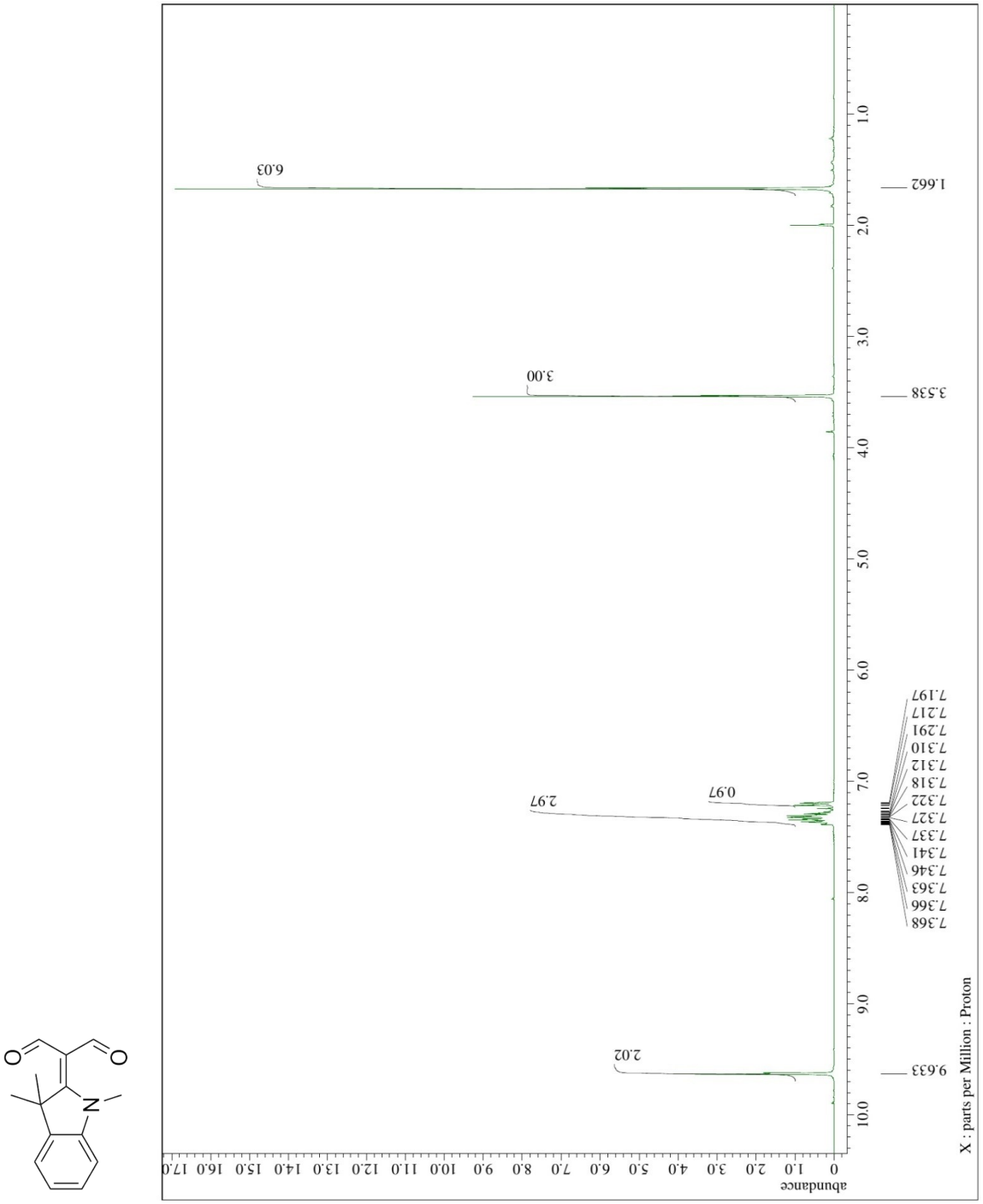


Fig. S1: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **6**.

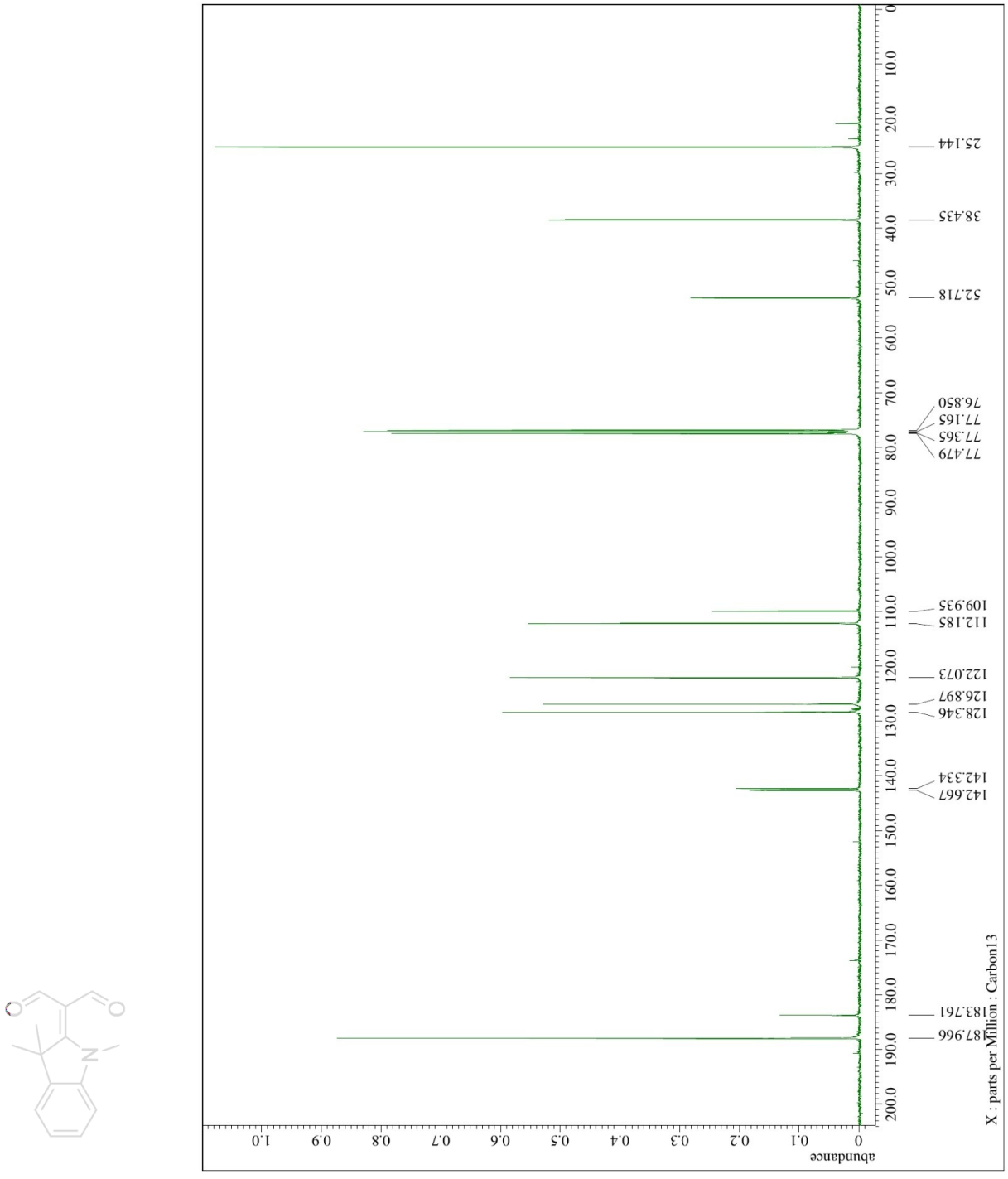


Fig. S2: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **6**.

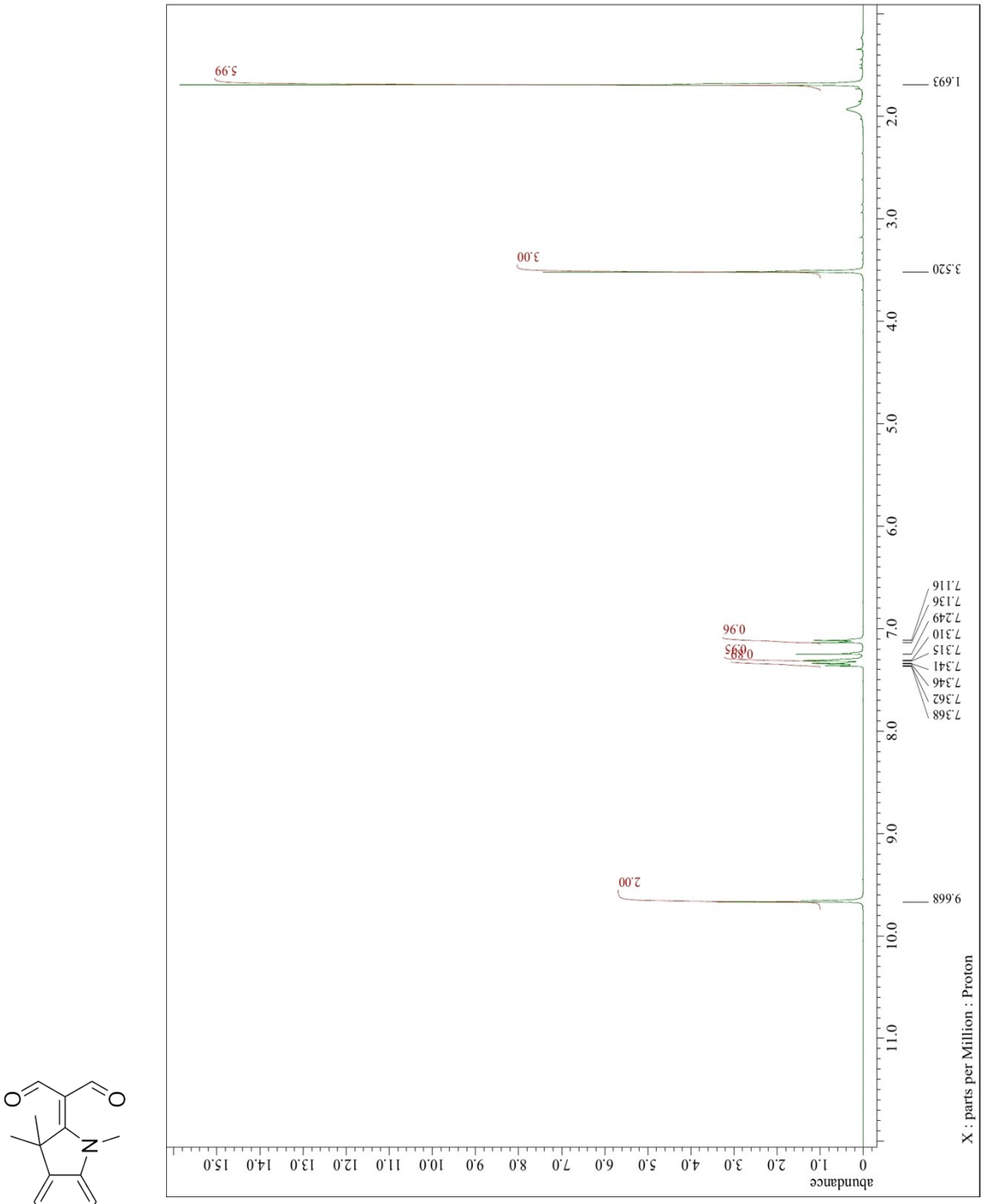


Fig. S3: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **7**.

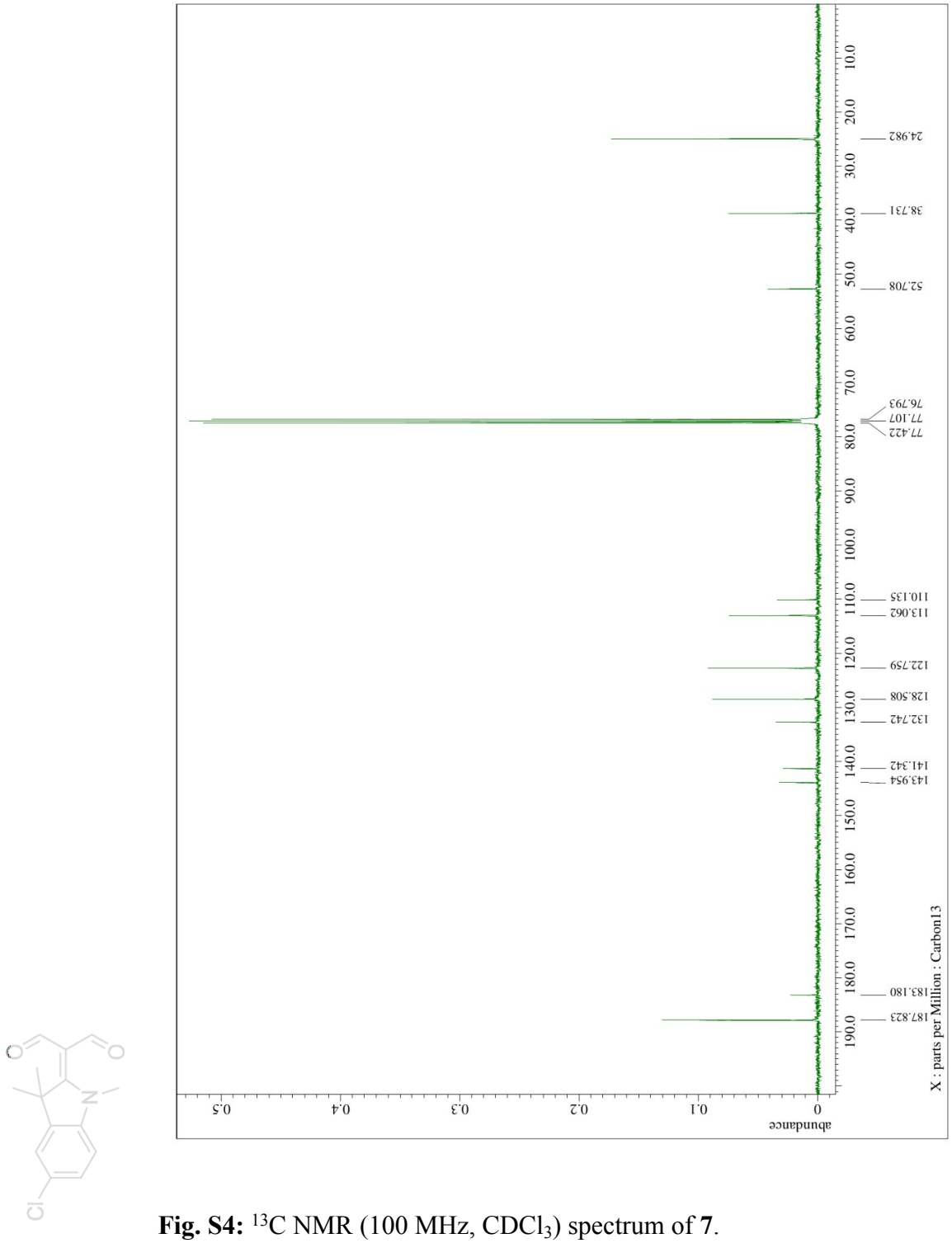


Fig. S4: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **7**.

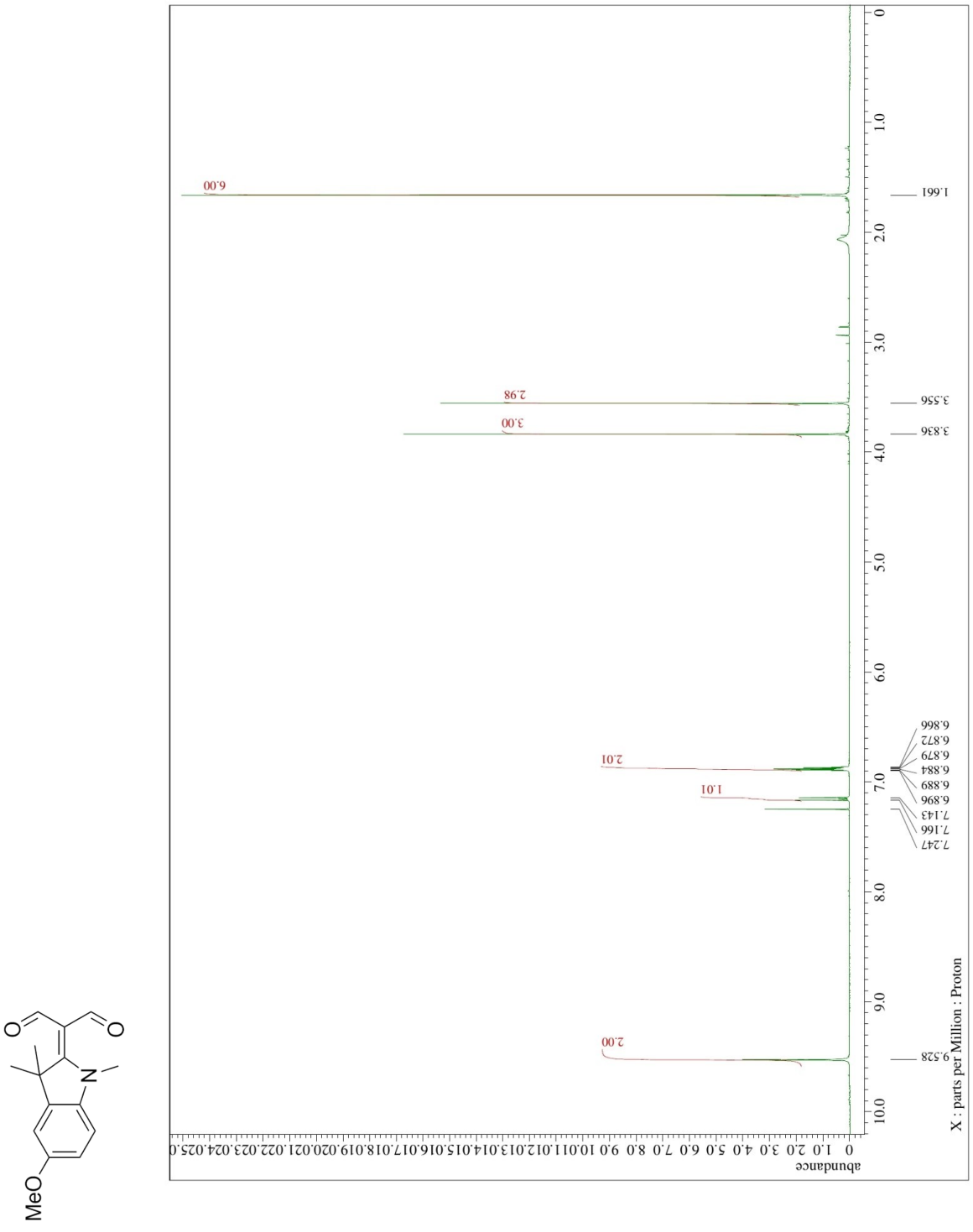


Fig. S5: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **8**.

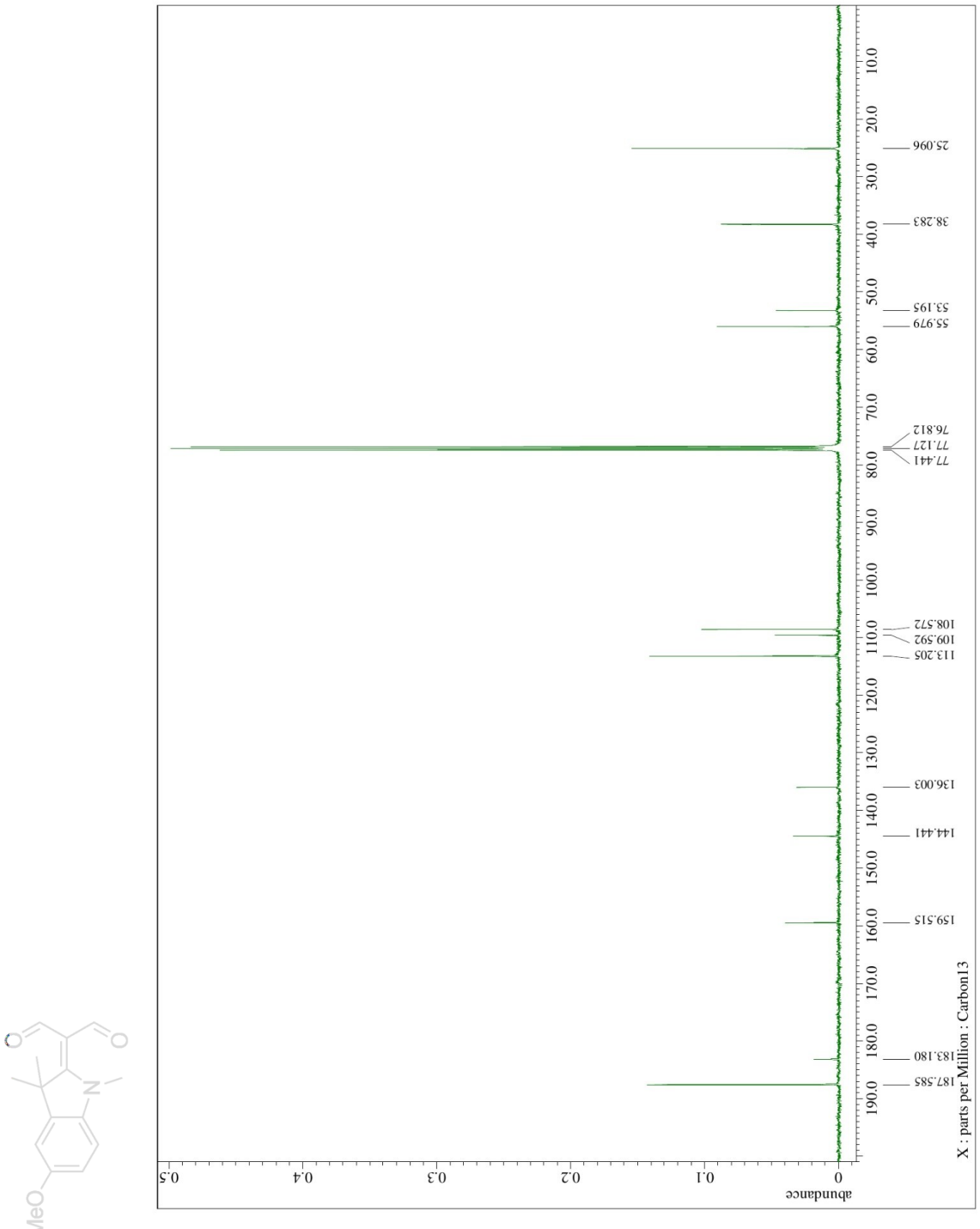


Fig. S6: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **8**.

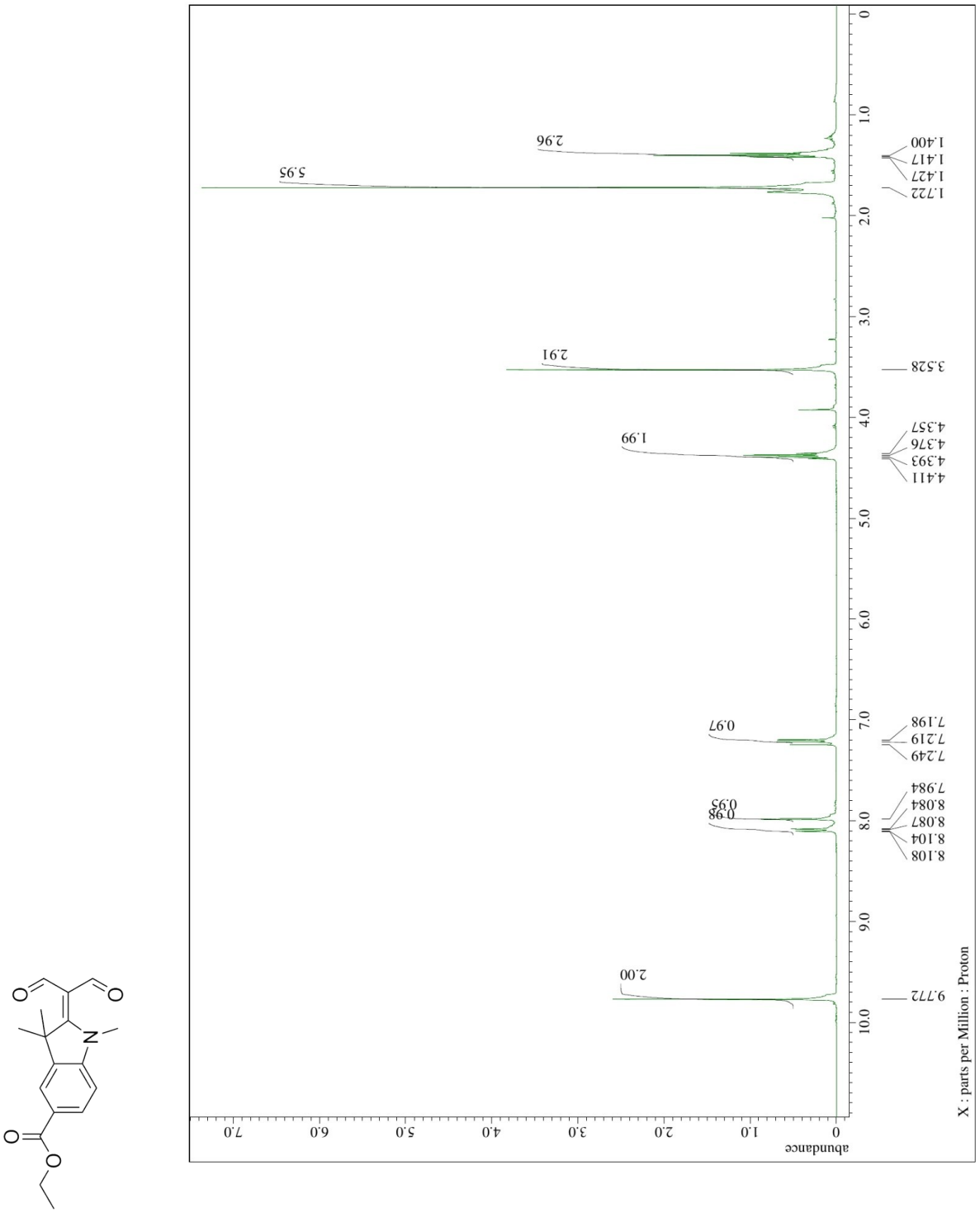


Fig. S7: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **9**.

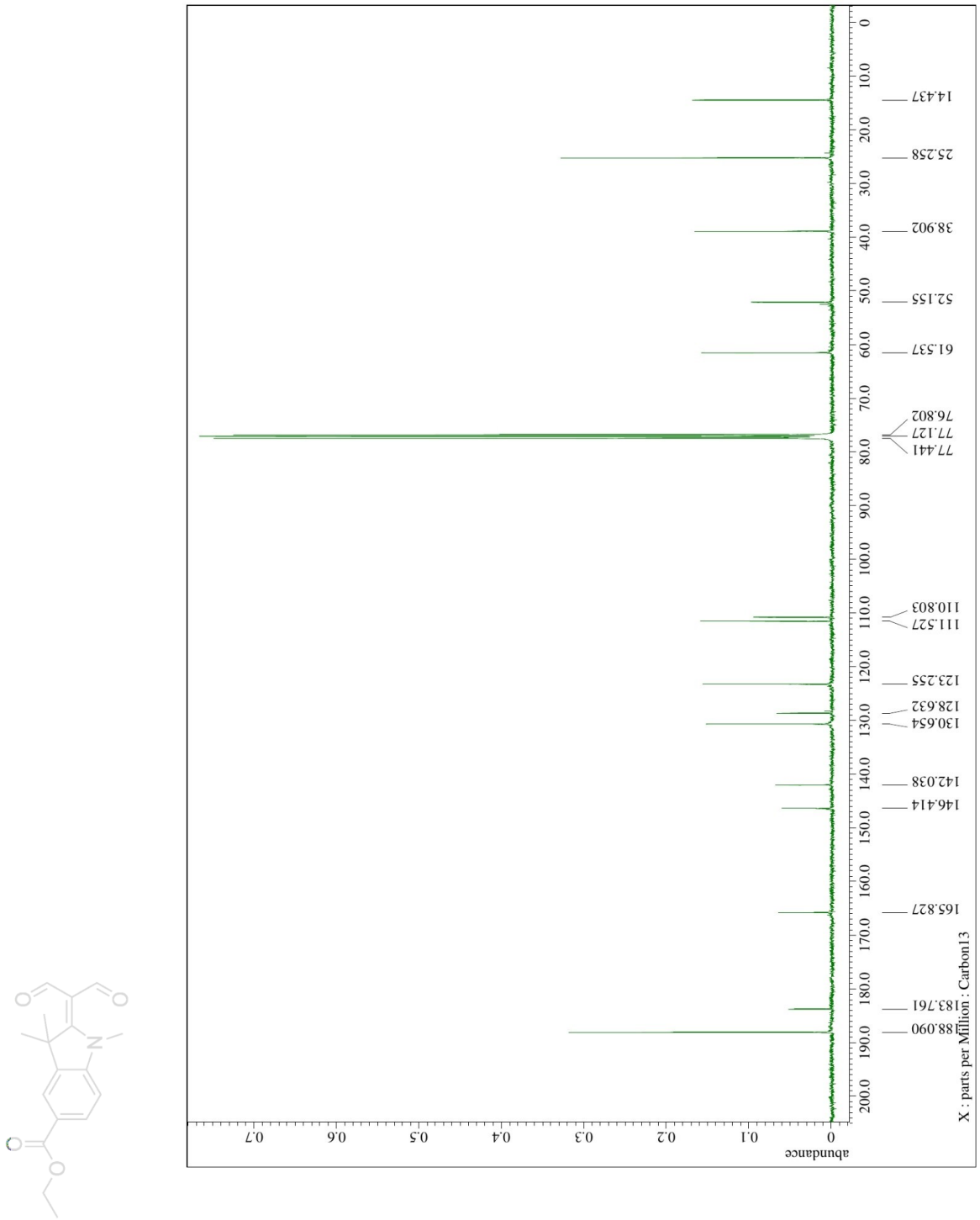


Fig. S8: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **9**.

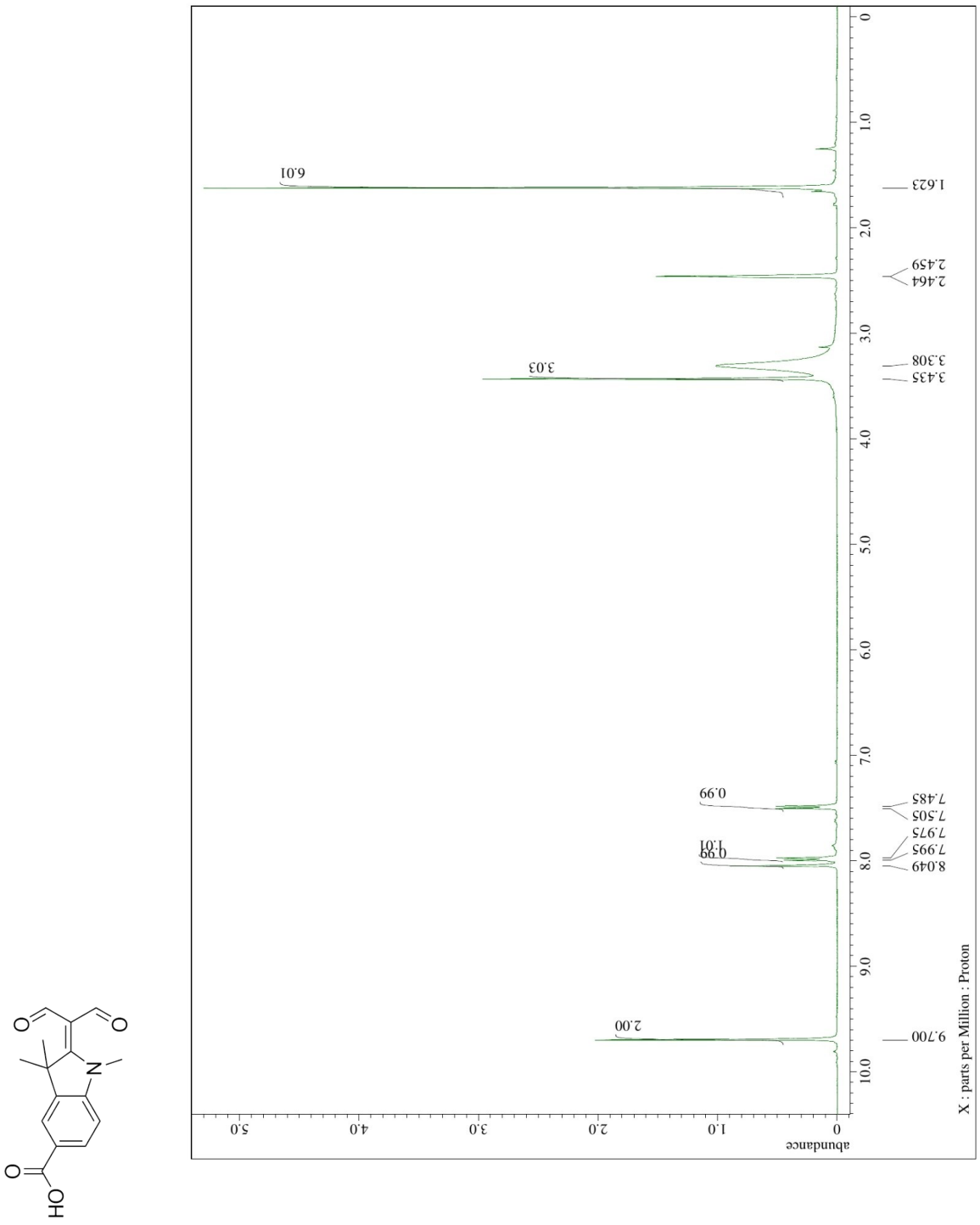


Fig. S9: ^{1}H NMR (400 MHz, DMSO) spectrum of **10**.

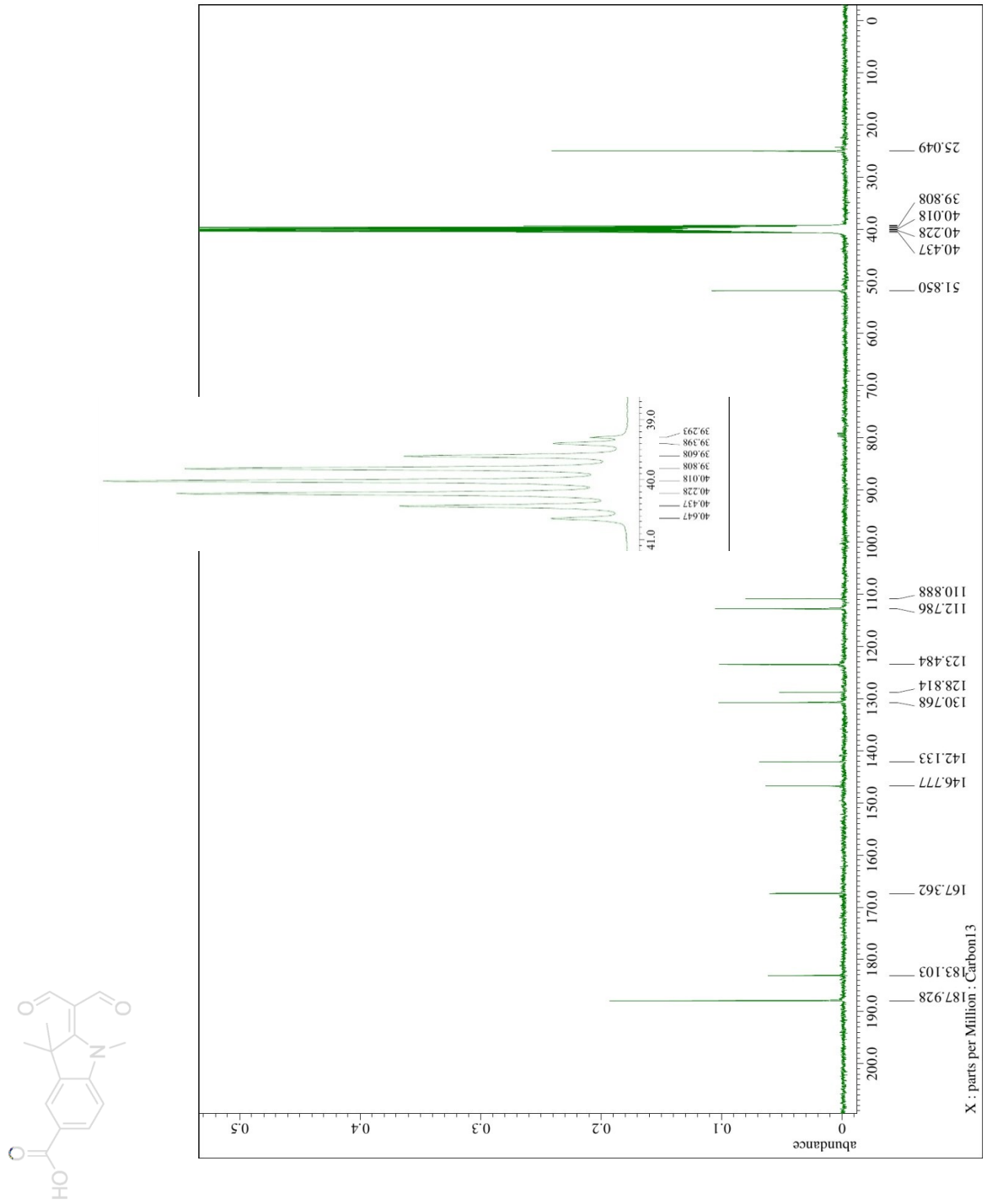


Fig. S10: ^{13}C NMR (100 MHz, DMSO) spectrum of **10**.

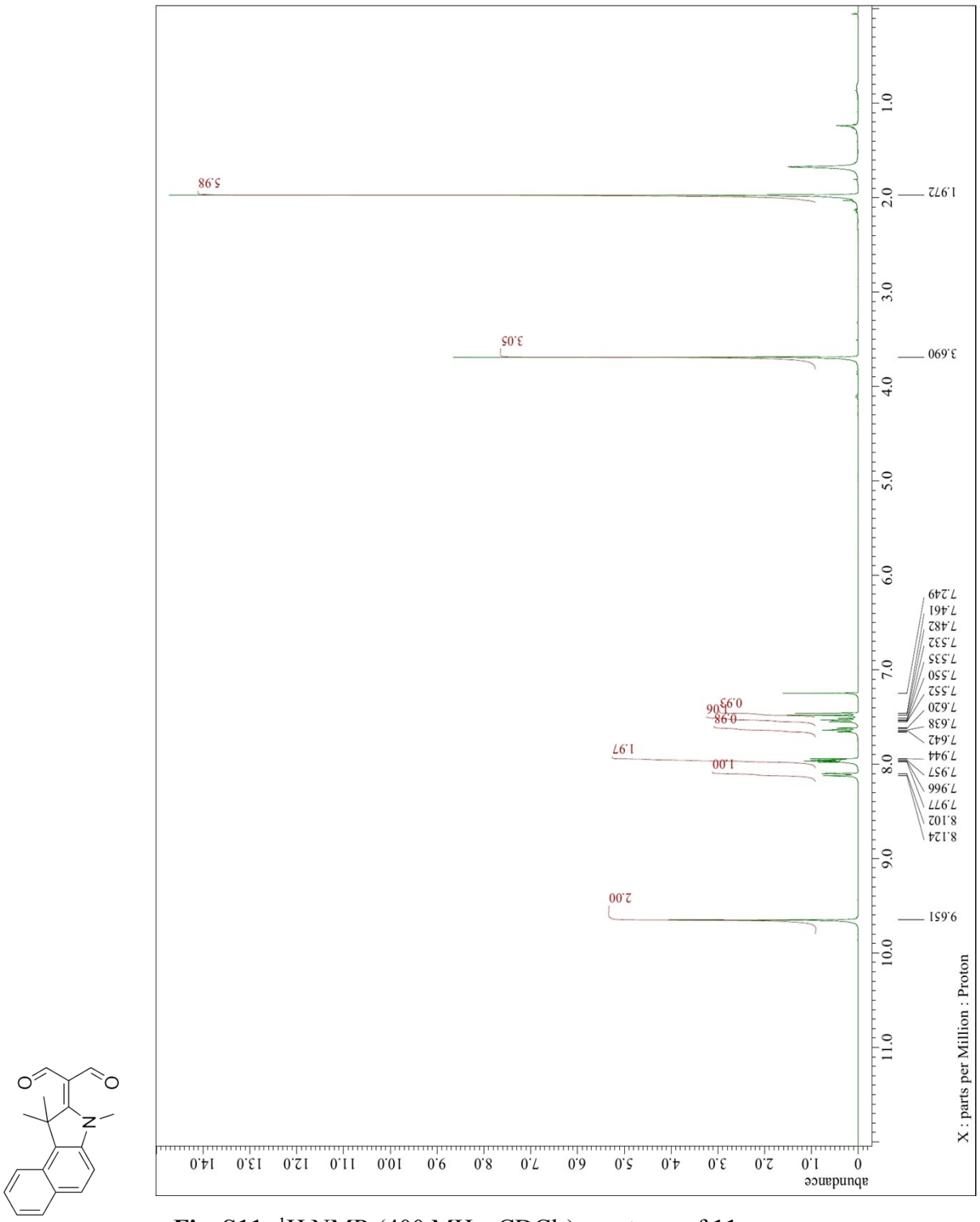


Fig. S11: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **11**.

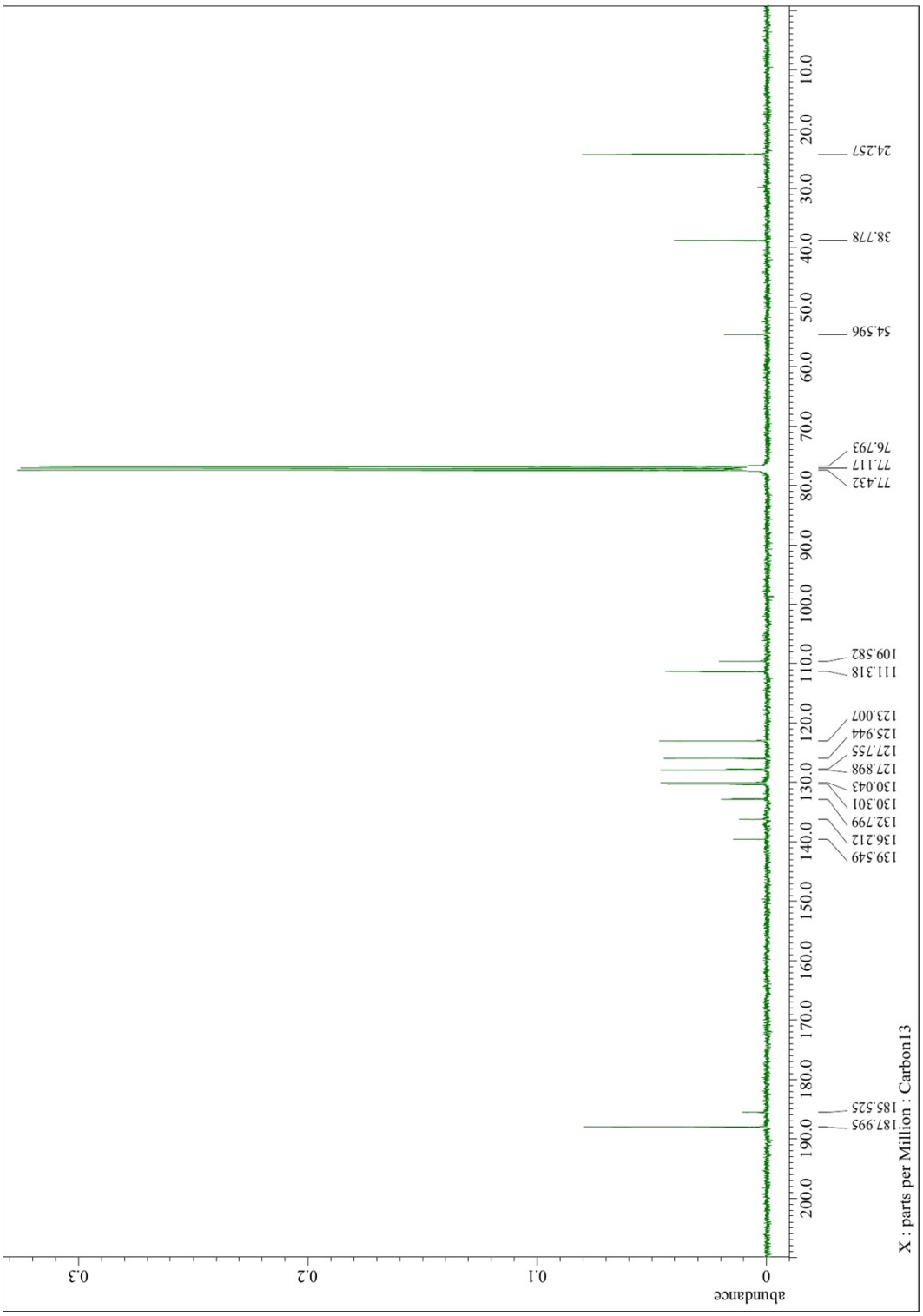


Fig. S12: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **11**.

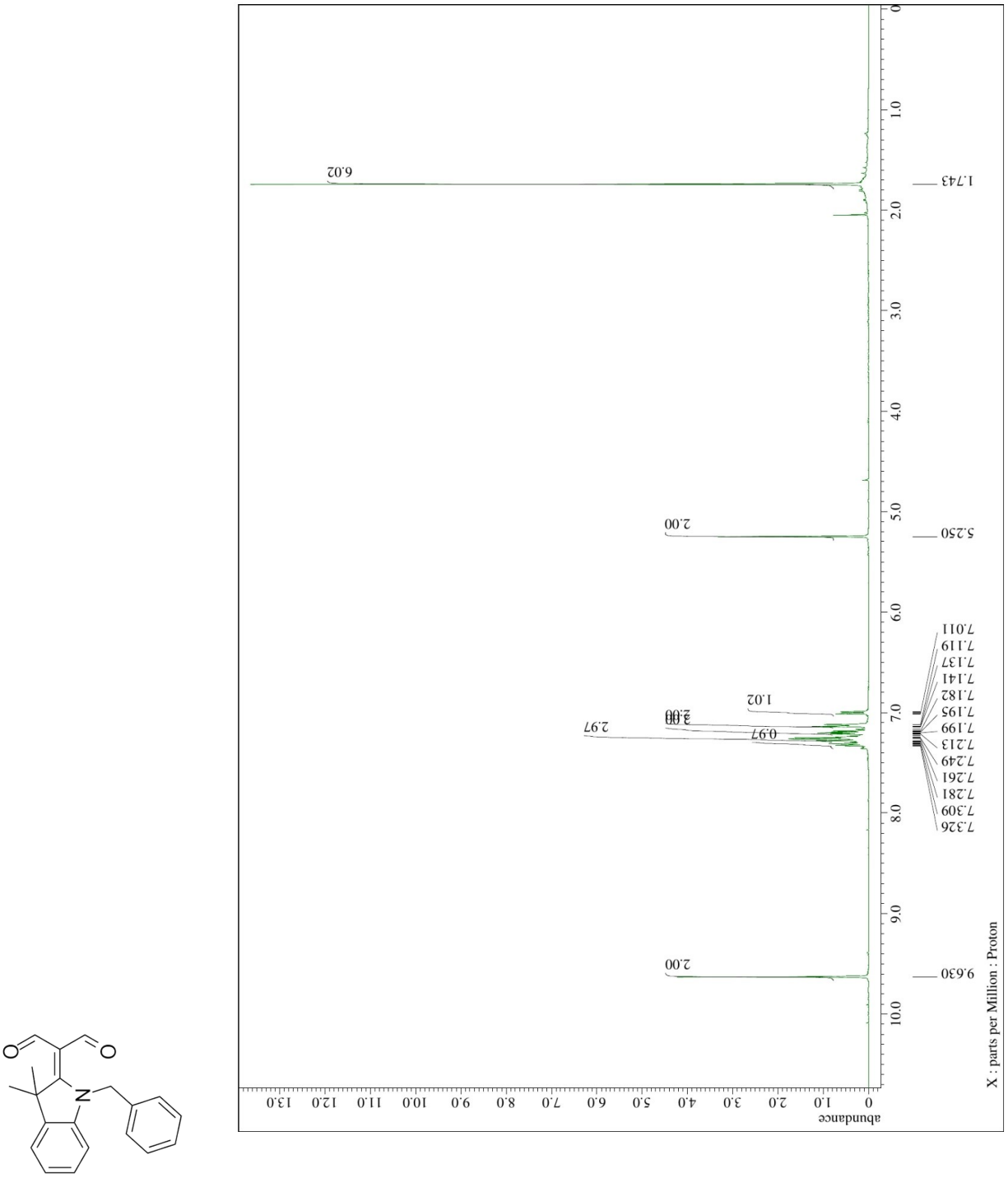


Fig. S13: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **12**.

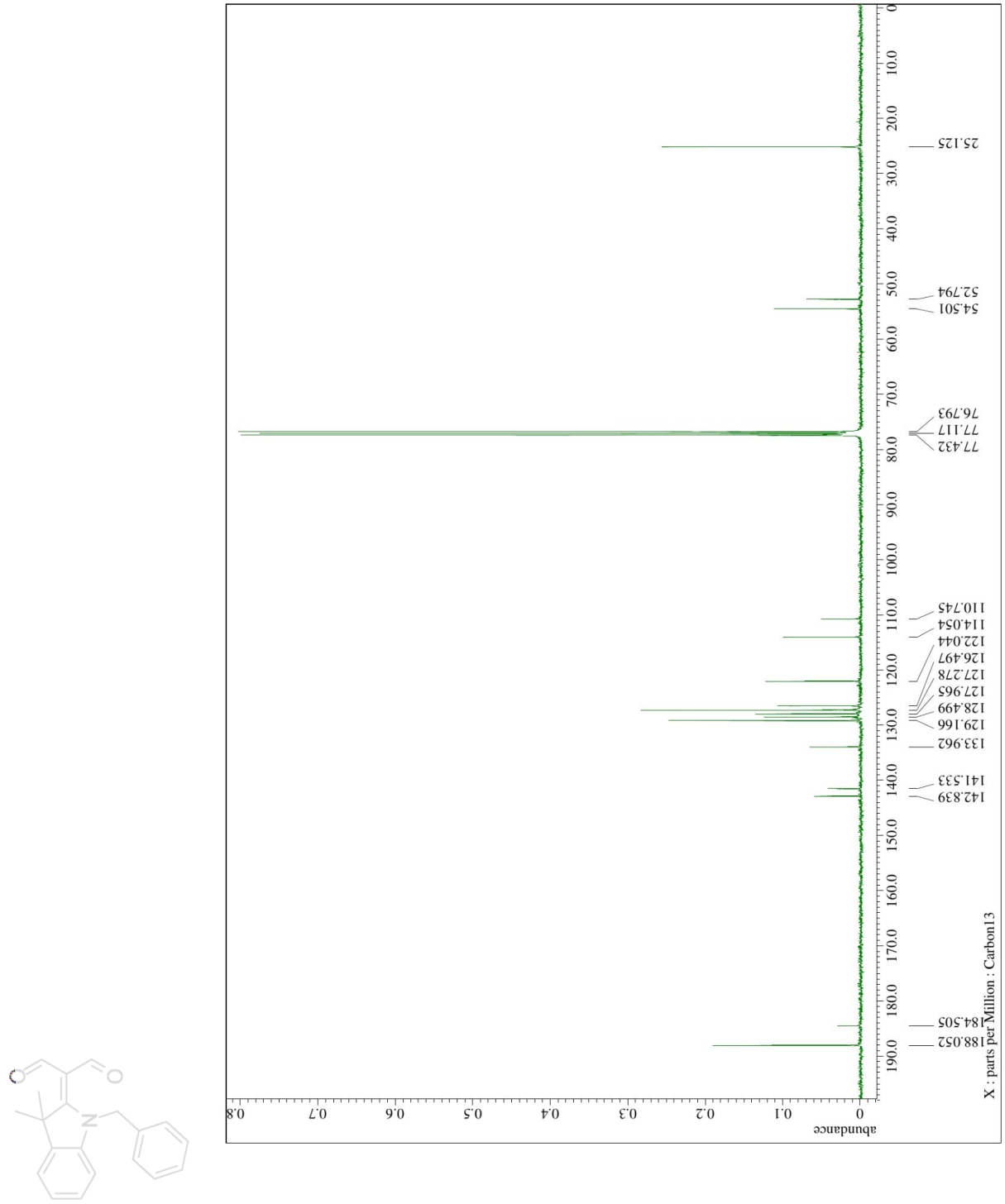


Fig. S14: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **12**.

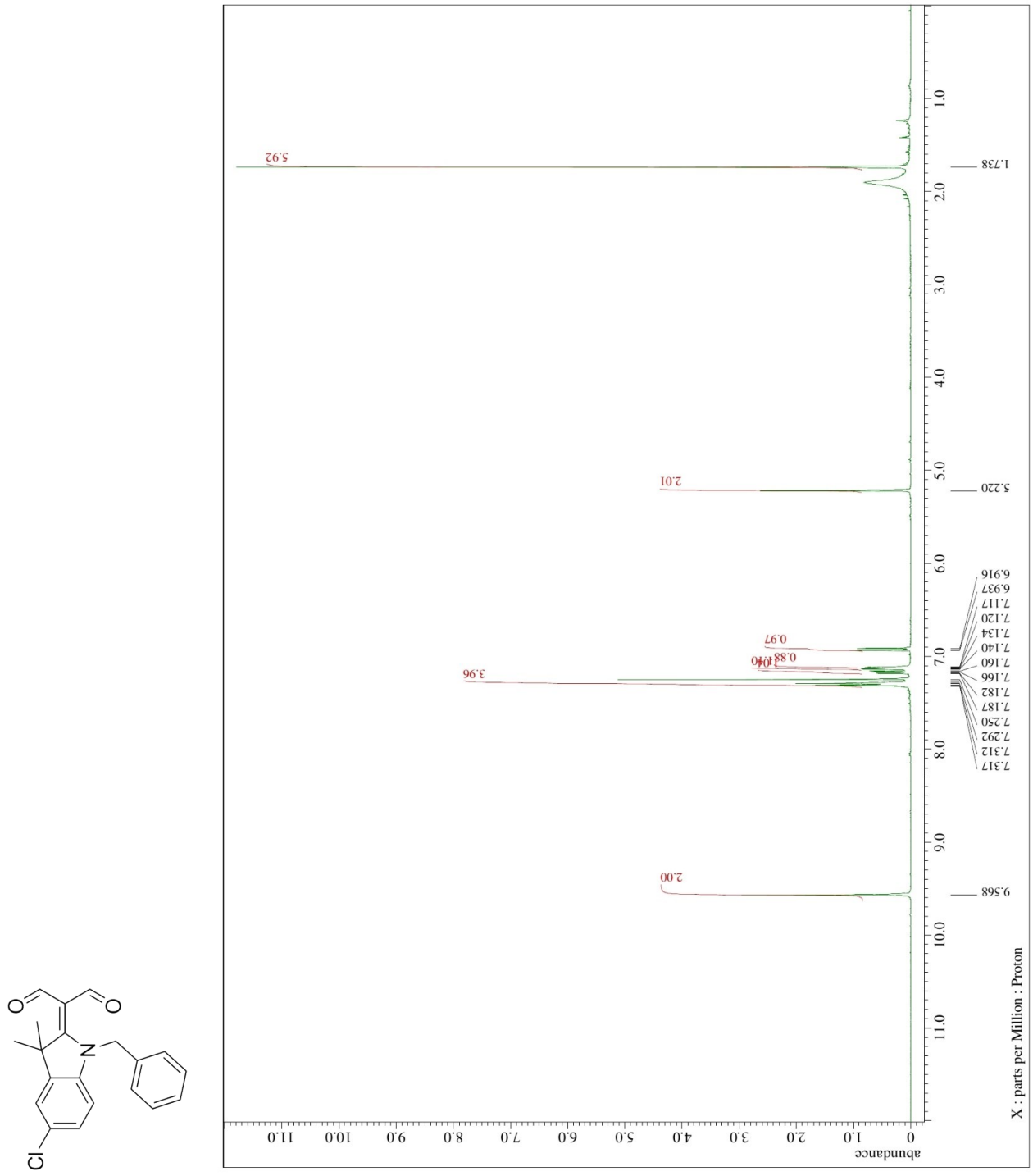


Fig. S15: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **13**.

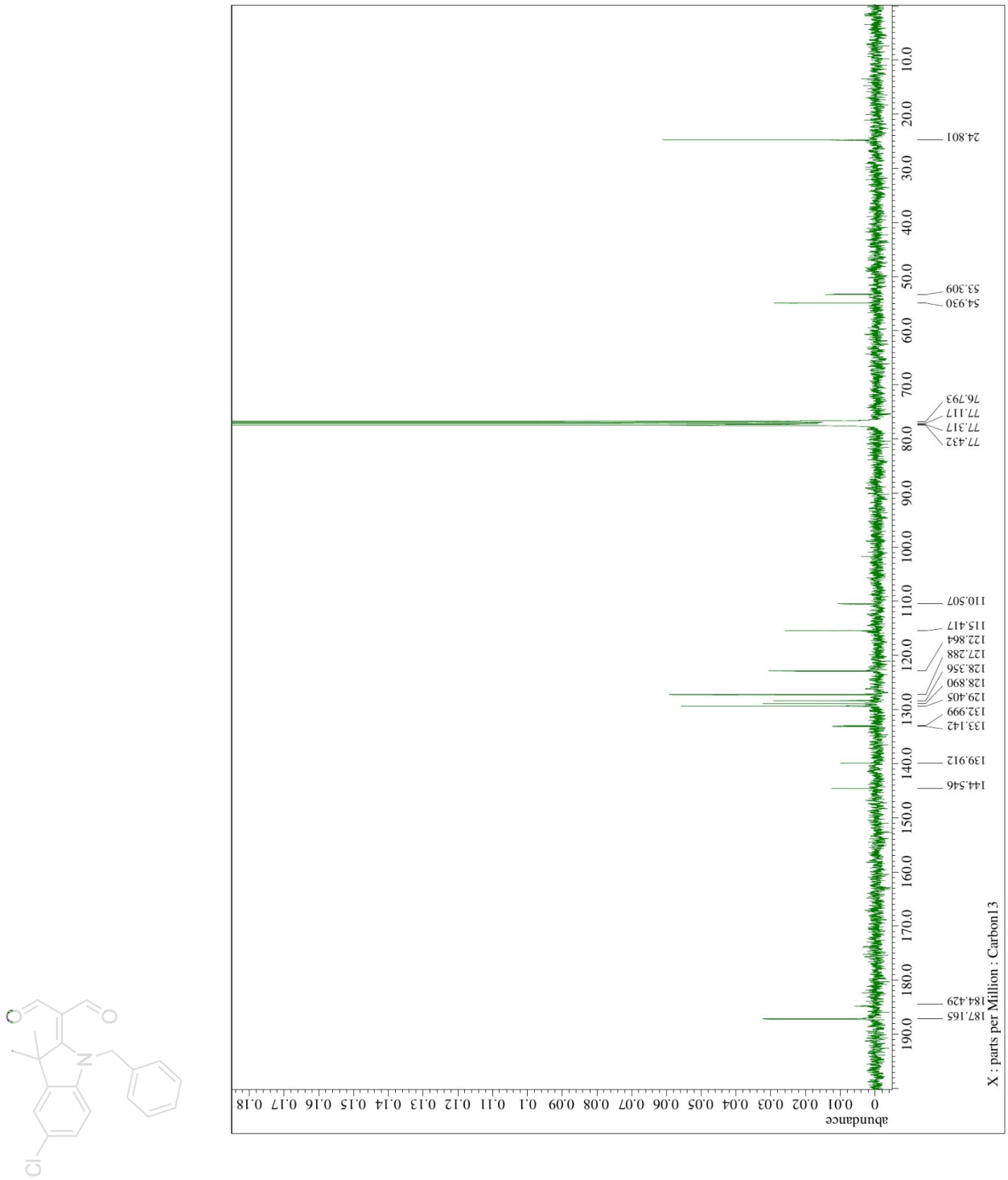


Fig. S16: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **13**.

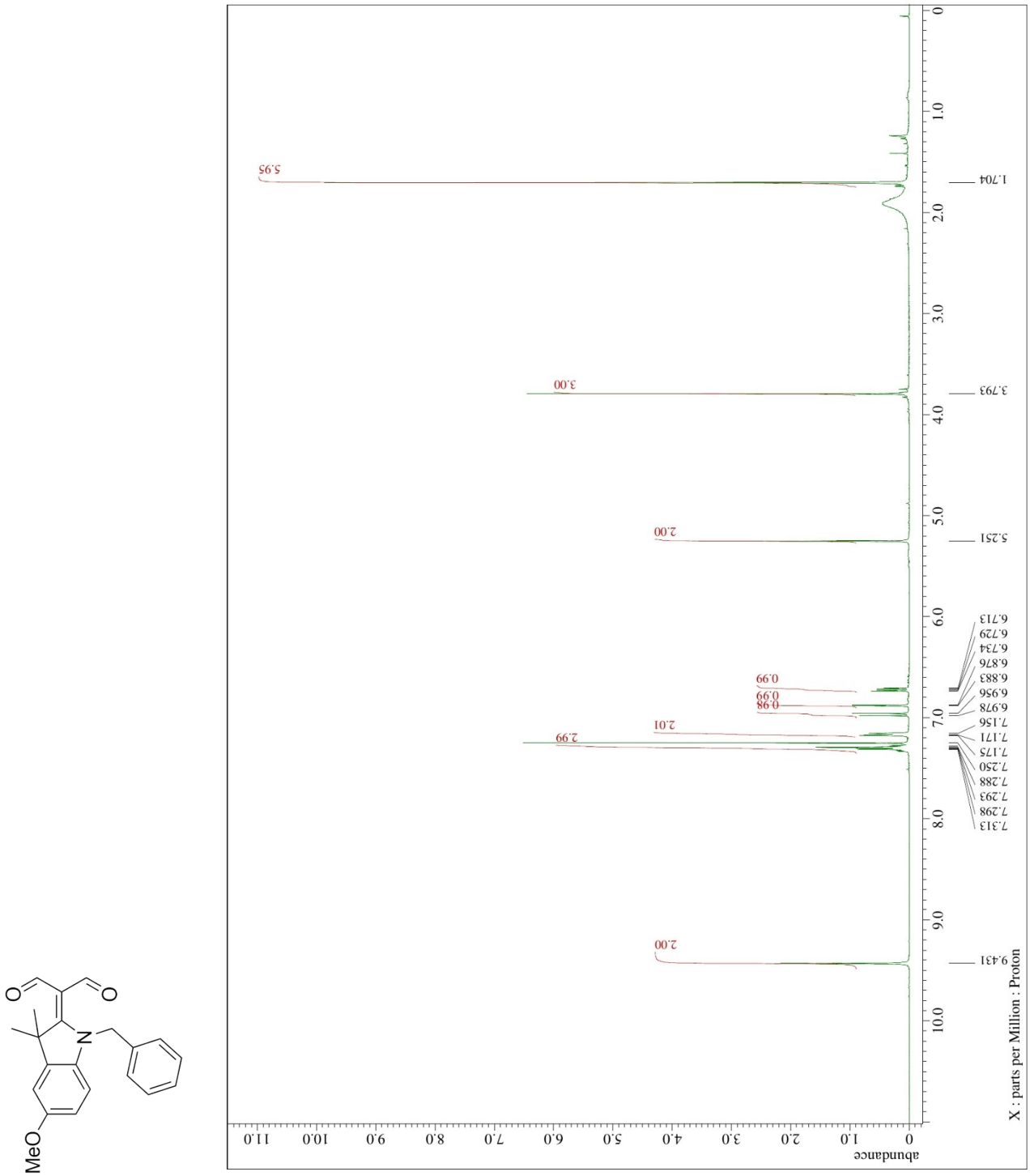


Fig. S17: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **14**.

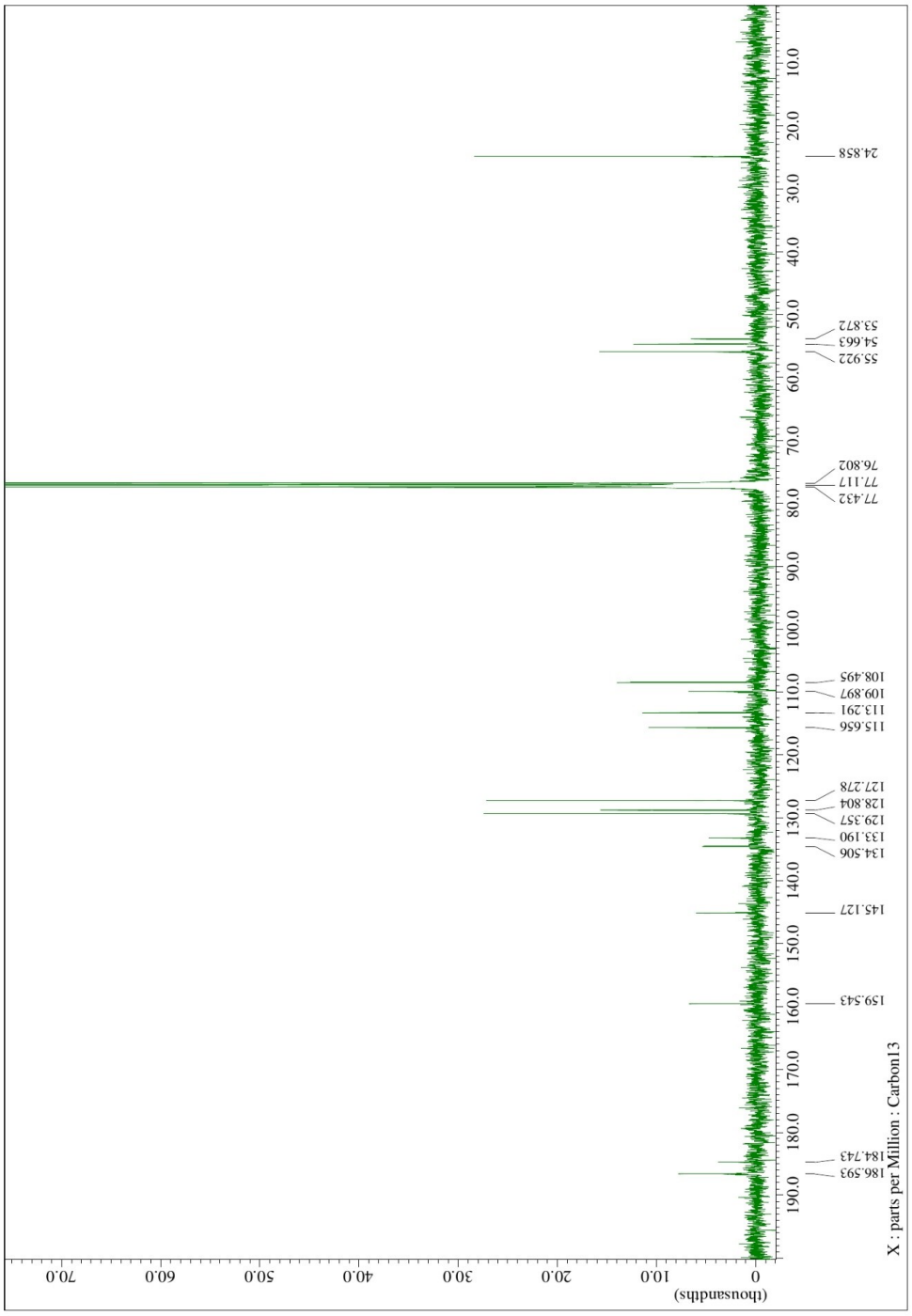
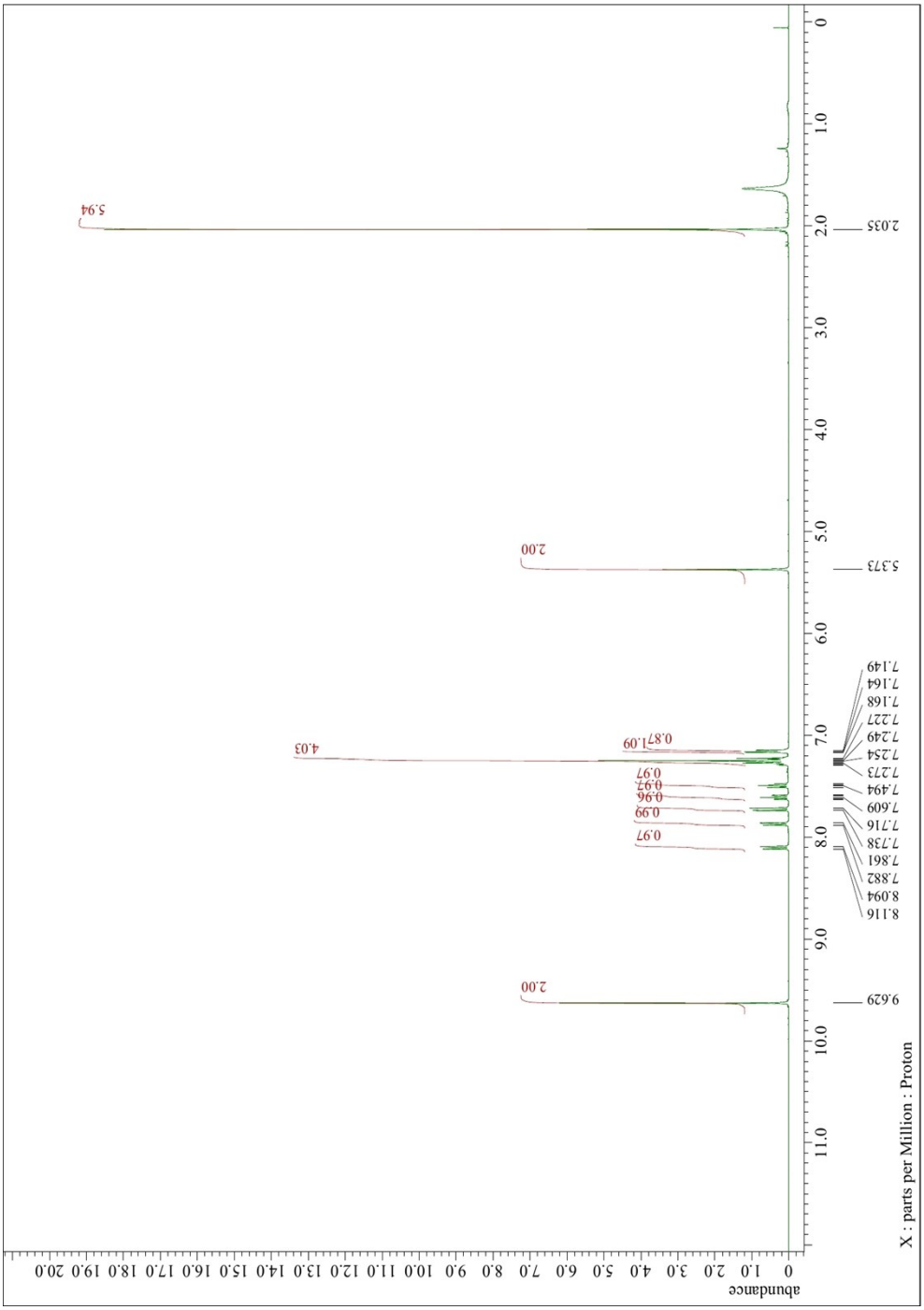


Fig. S18: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **14**.



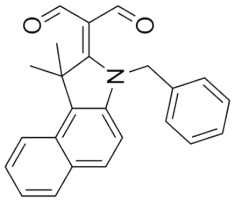


Fig. S19: ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of **15**.

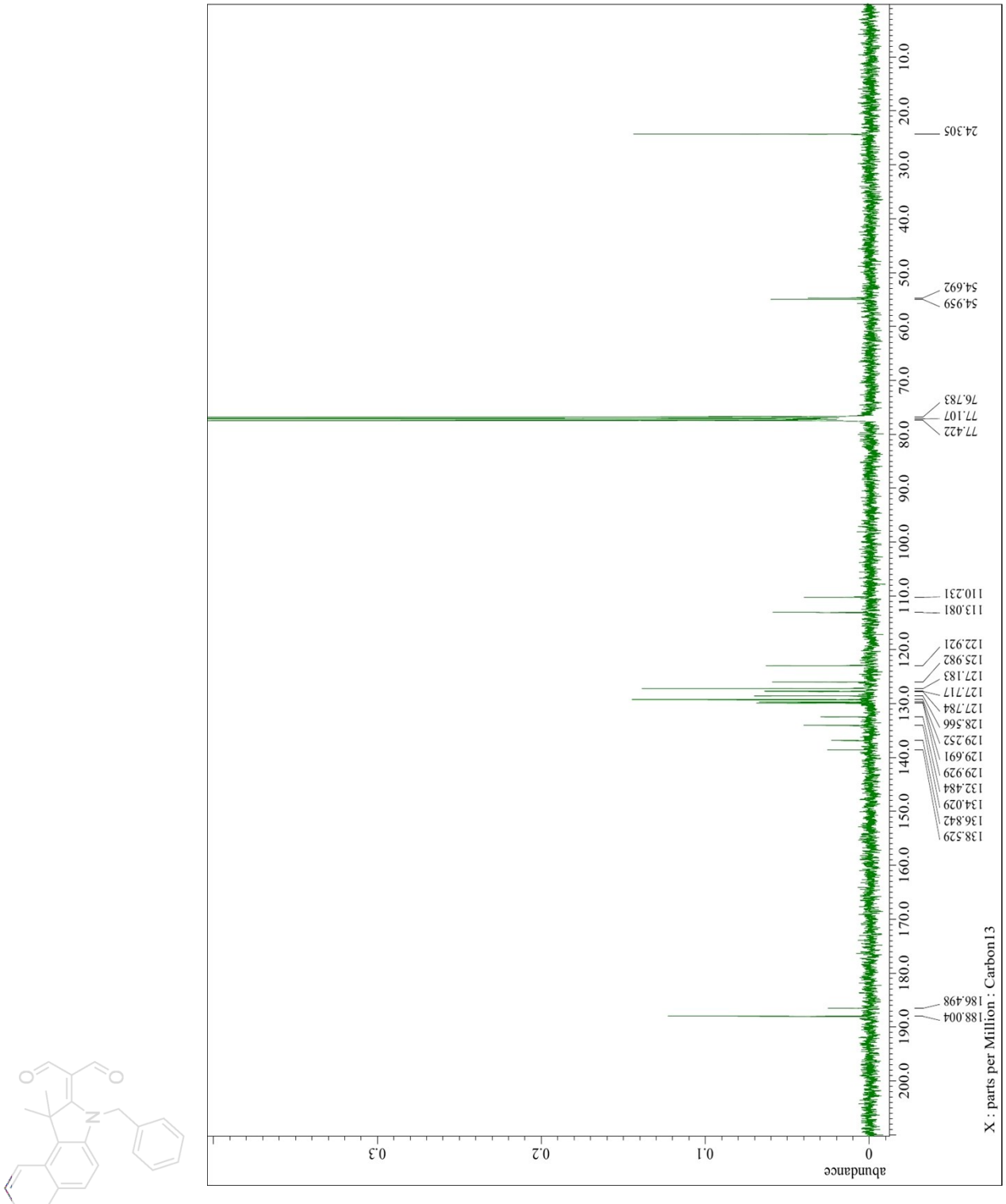


Fig. S20: ^{13}C NMR (100 MHz, $CDCl_3$) spectrum of **15**.

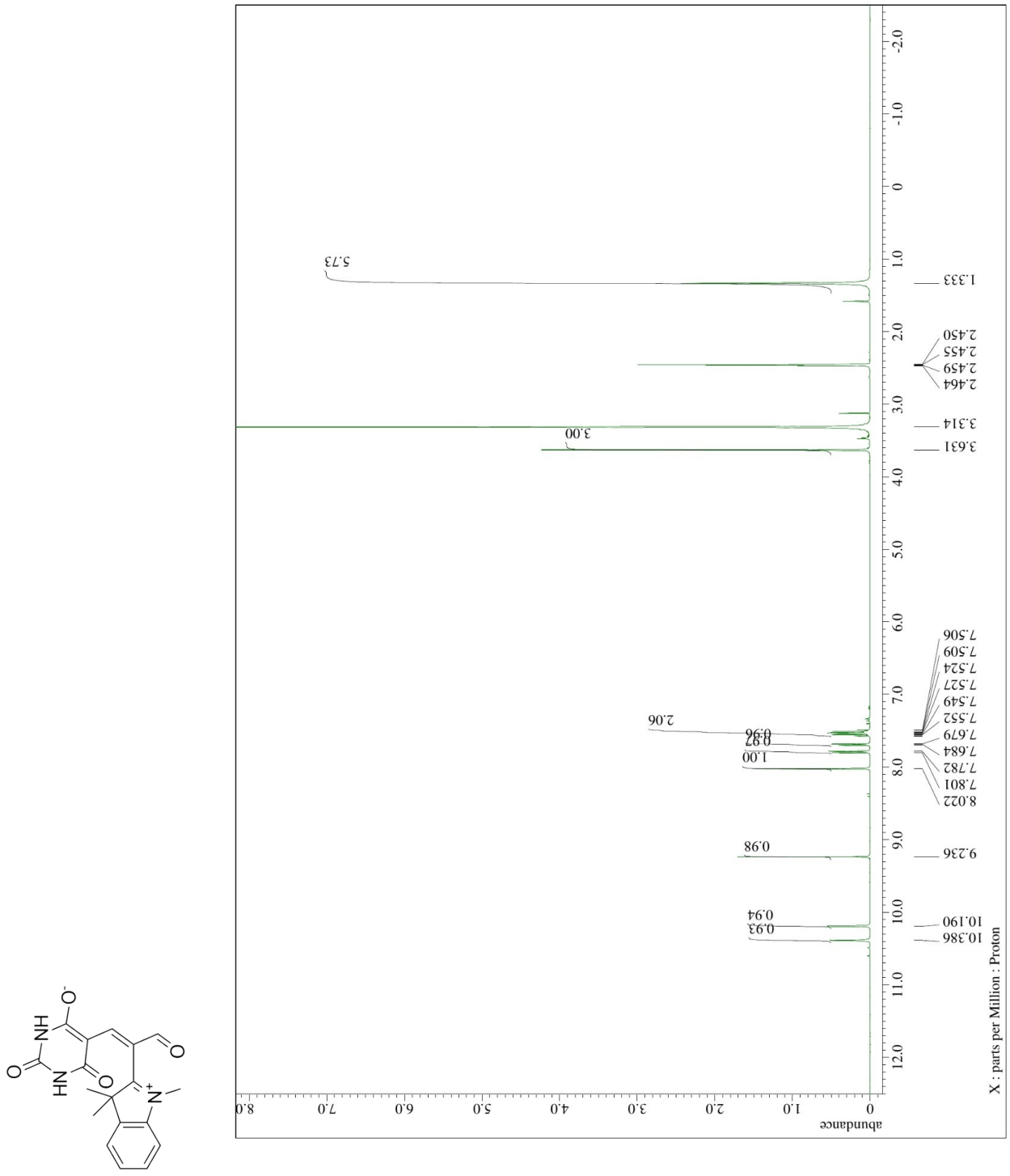


Fig. S21: ^{1}H NMR (400 MHz, DMSO) spectrum of **16**.

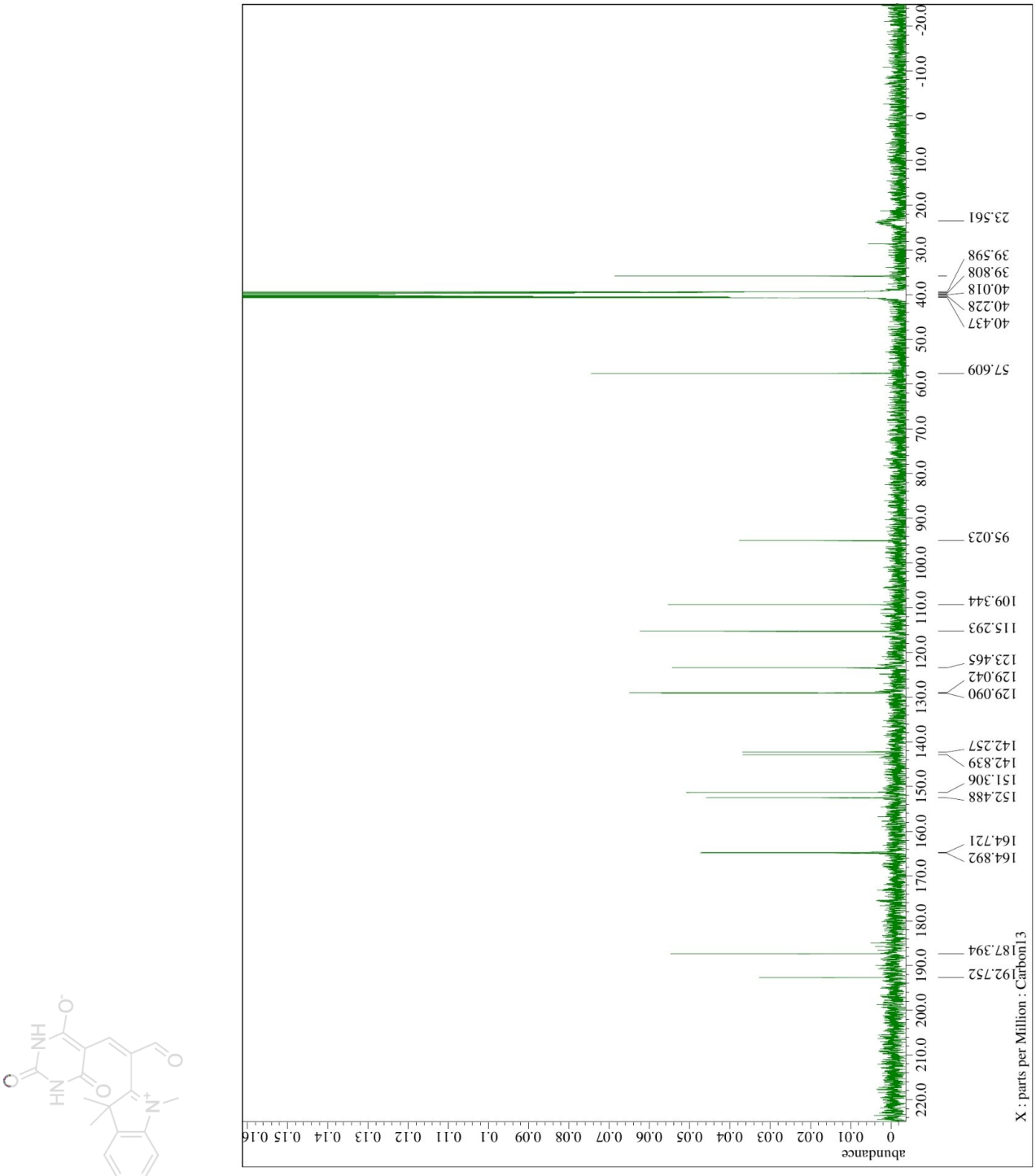


Fig. S22: ^{13}C NMR (100 MHz, DMSO) spectrum of **16**.

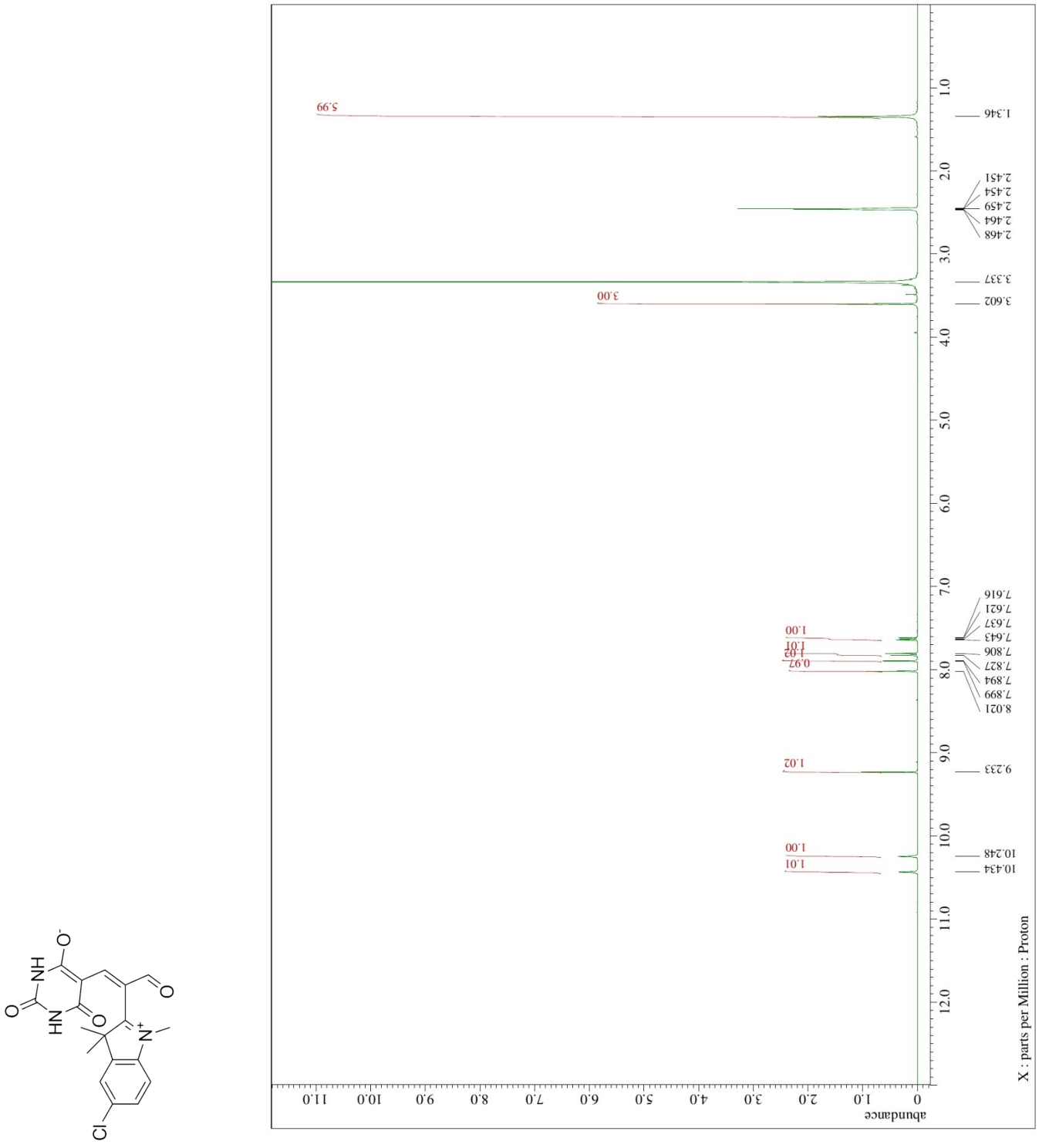


Fig. S23: ^{1}H NMR (400 MHz, DMSO) spectrum of **17**.

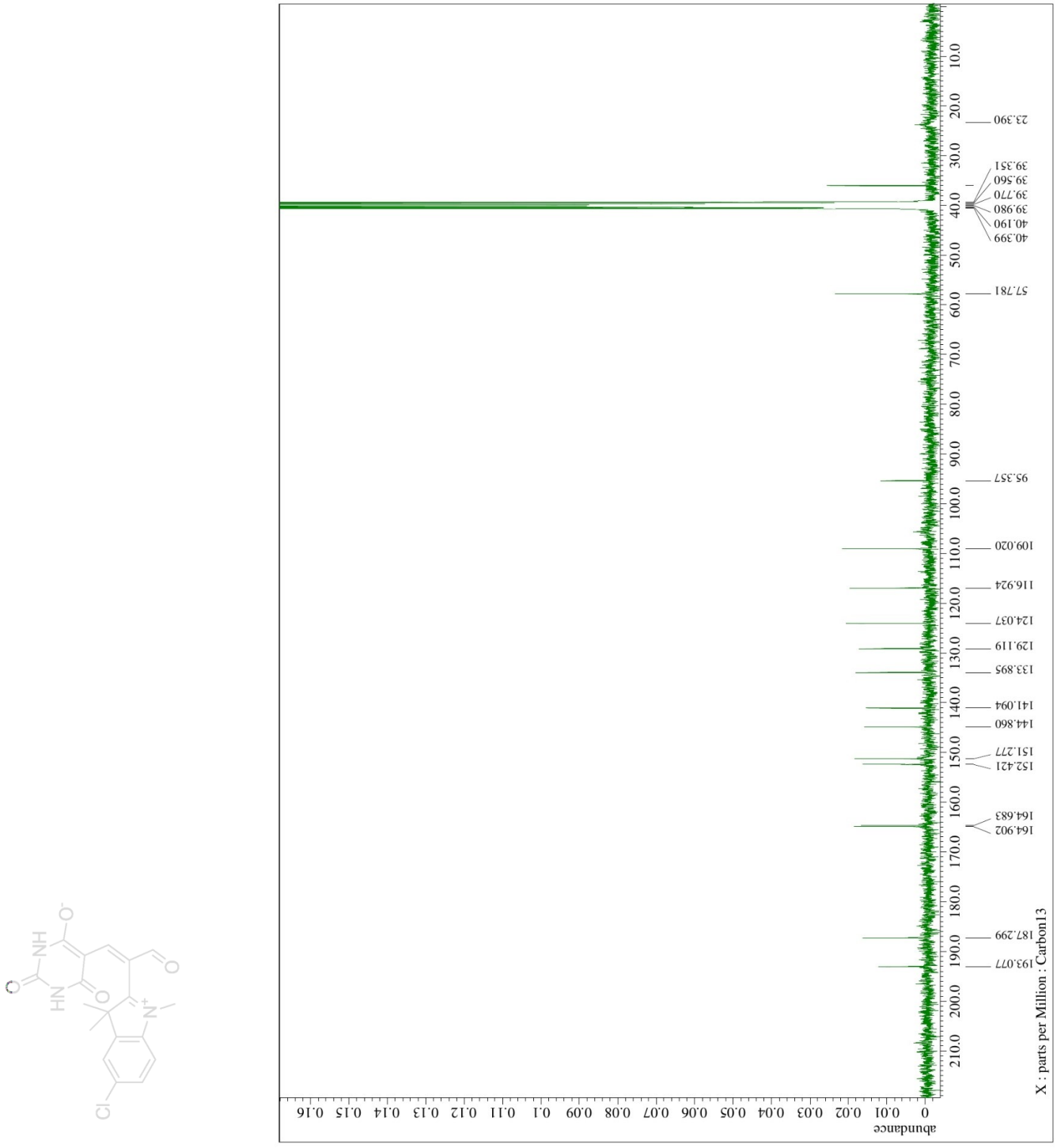


Fig. S24: ^{13}C NMR (100 MHz, DMSO) spectrum of **17**.

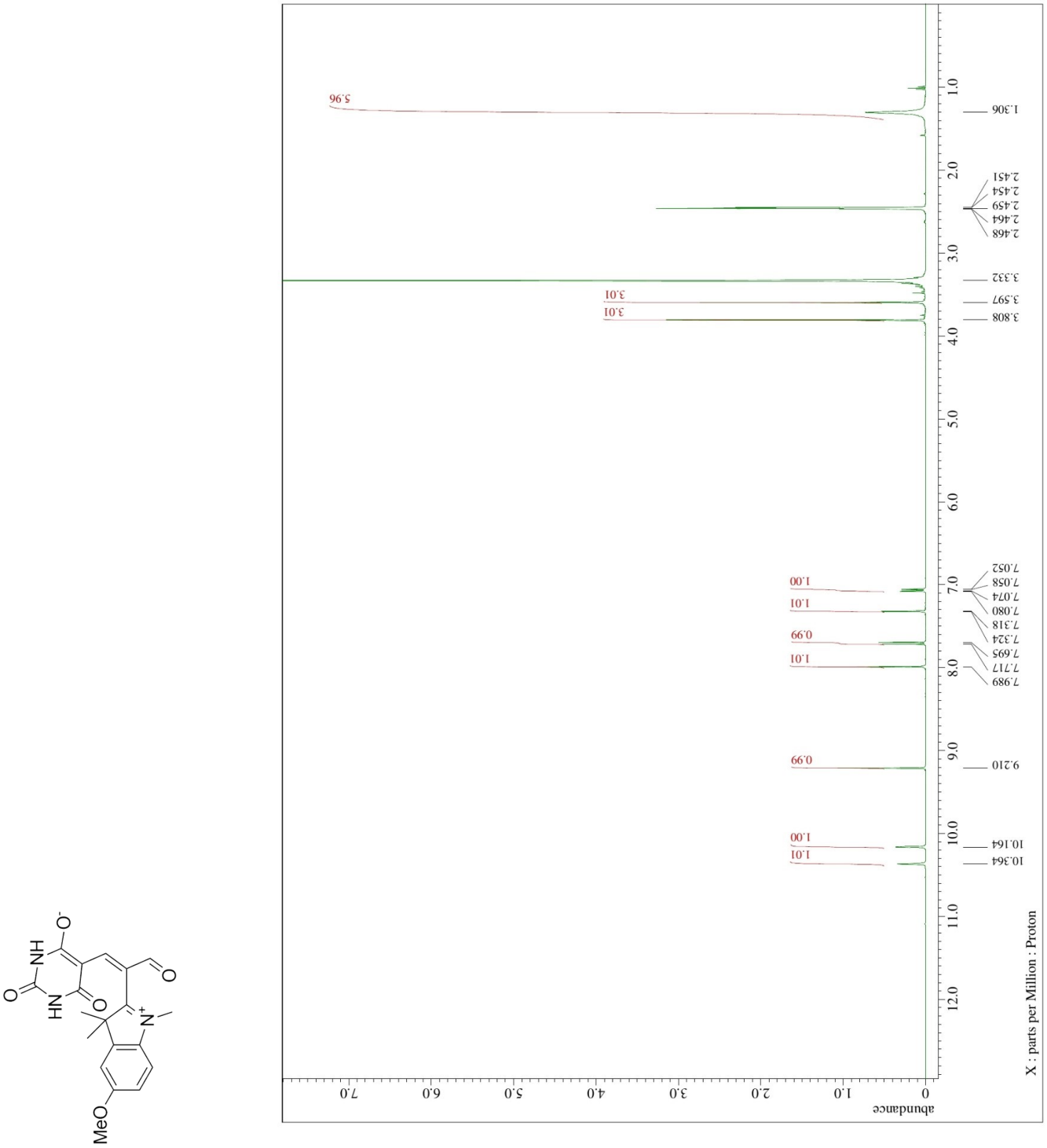


Fig. S25: ^{1}H NMR (400 MHz, DMSO) spectrum of **18**.

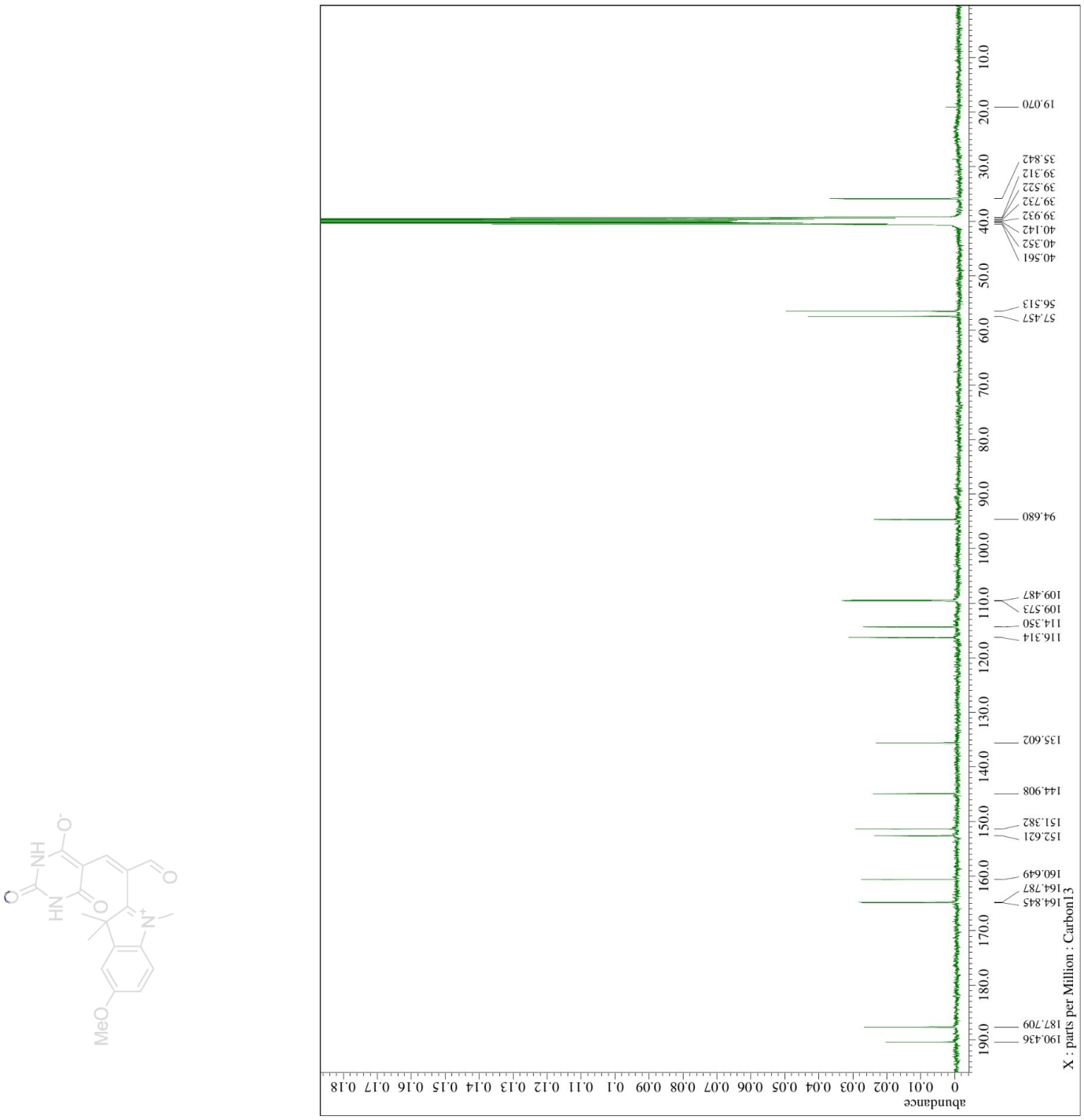


Fig. S26: ^{13}C NMR (100 MHz, DMSO) spectrum of **18**.

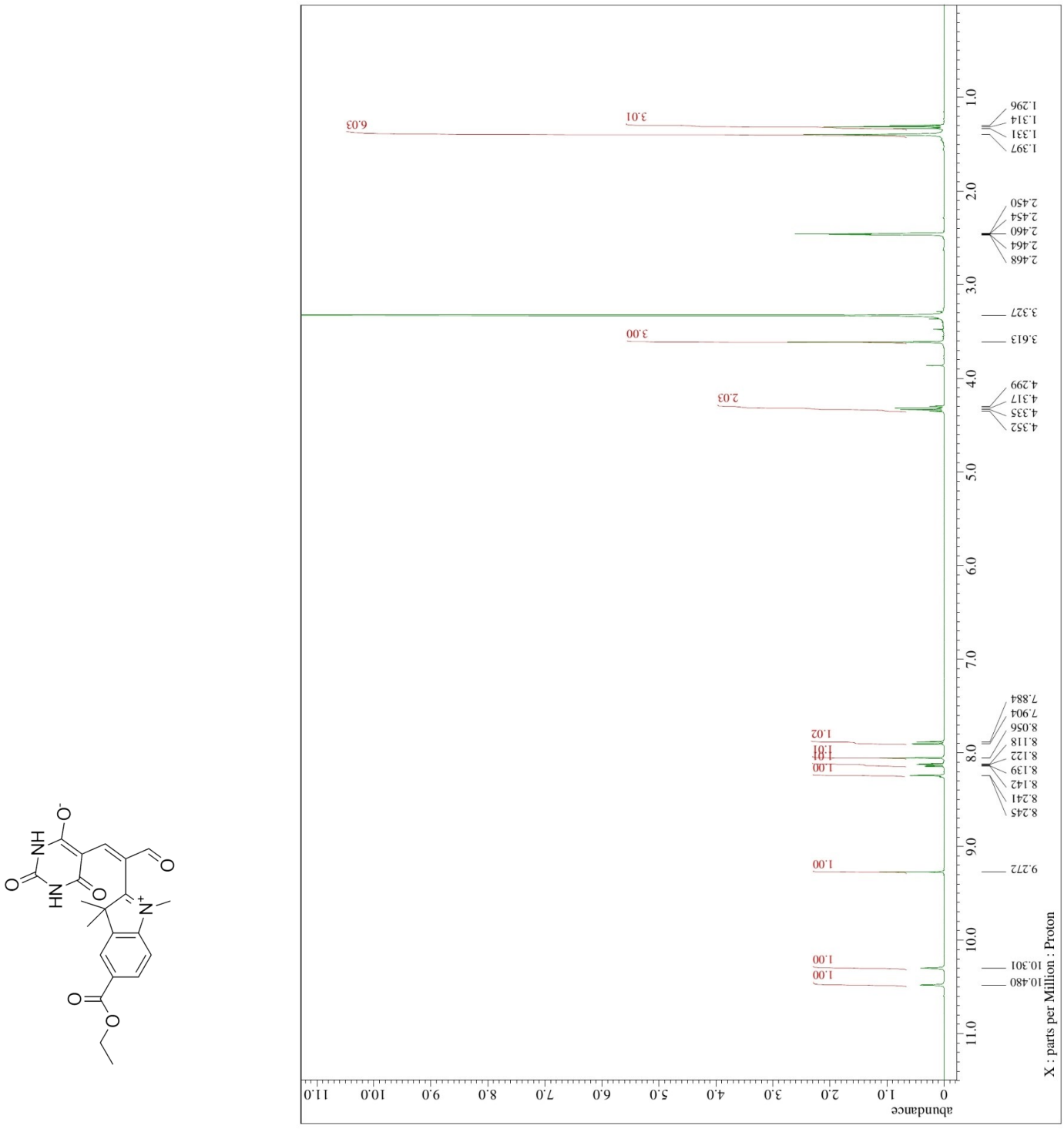


Fig. S27: ^{1}H NMR (400 MHz, DMSO) spectrum of **19**.

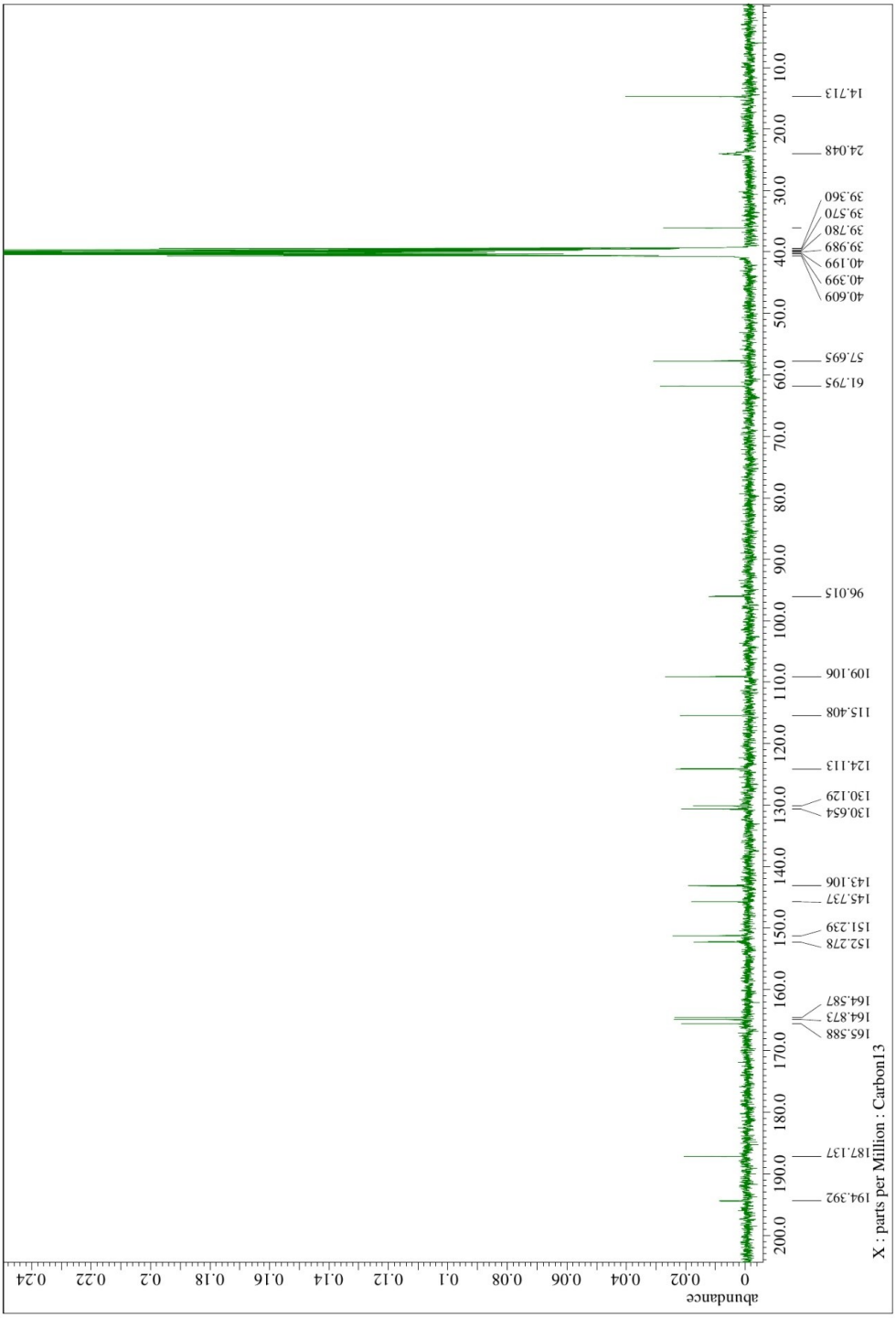


Fig. S28: ^{13}C NMR (100 MHz, DMSO) spectrum of **19**.

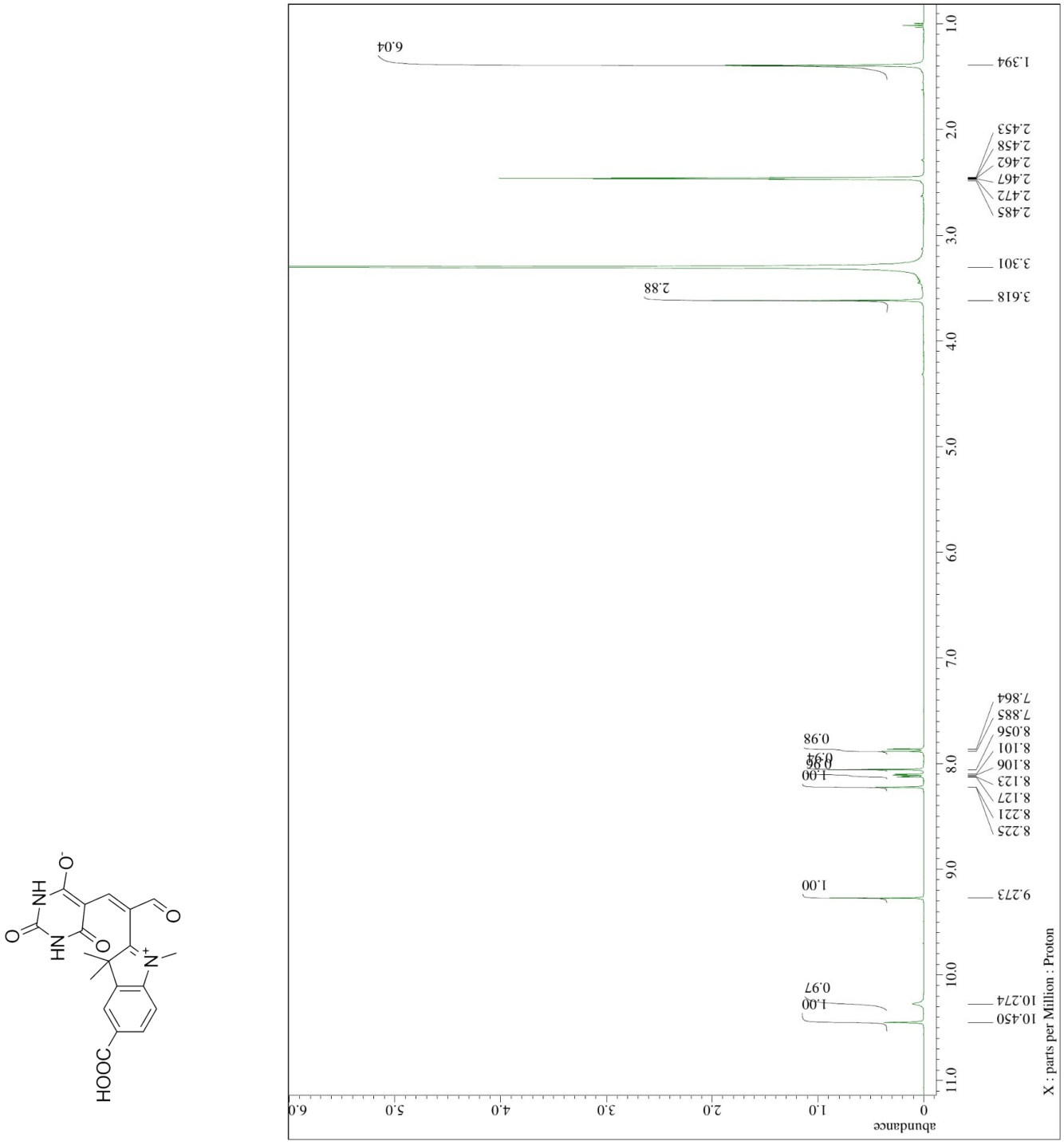


Fig. S29: ^{1}H NMR (400 MHz, DMSO) spectrum of **20**.

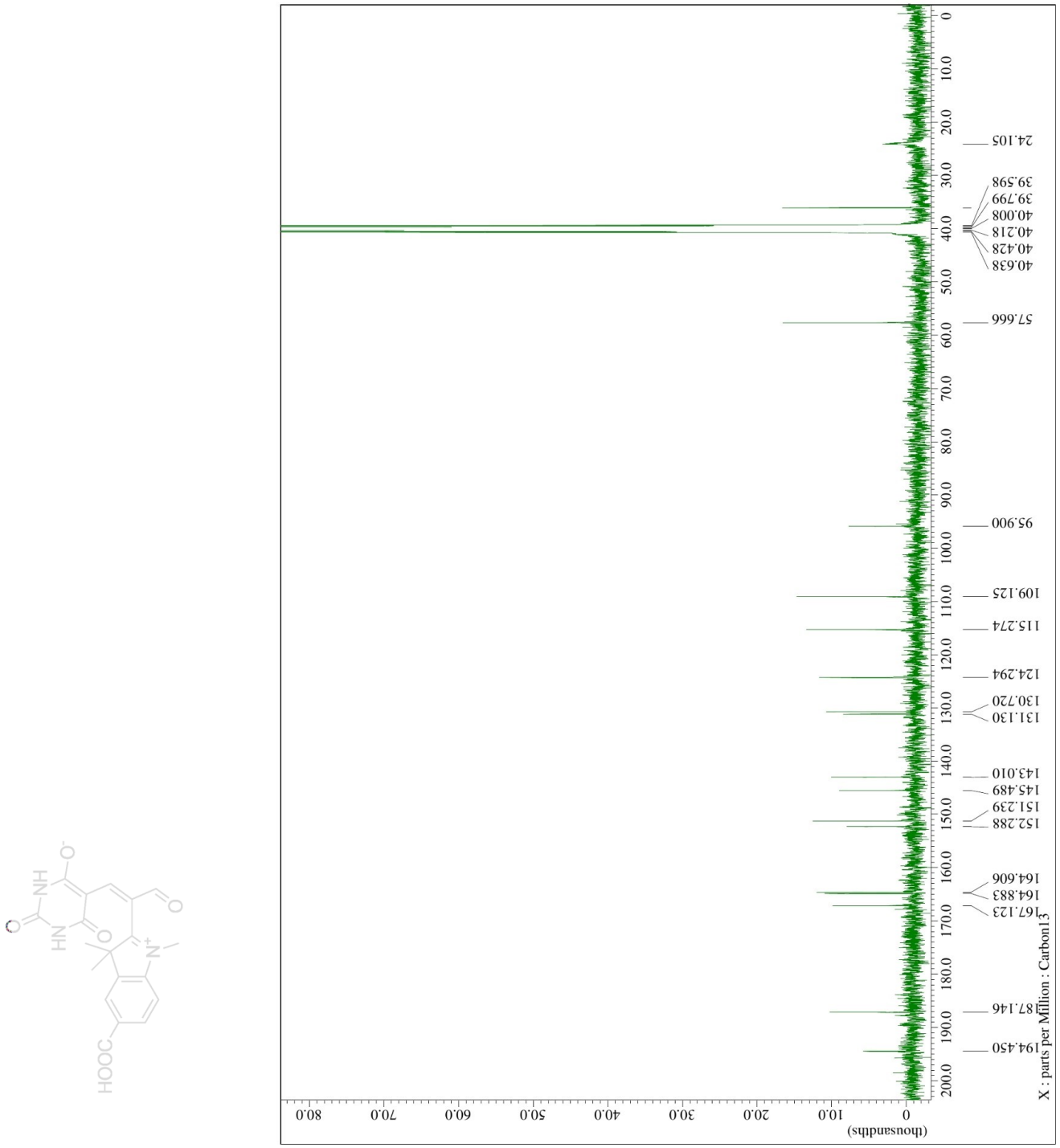
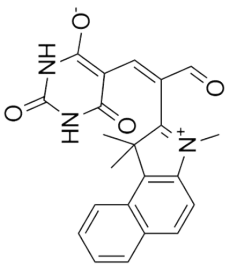


Fig. S30: ^{13}C NMR (100 MHz, DMSO) spectrum of **20**.



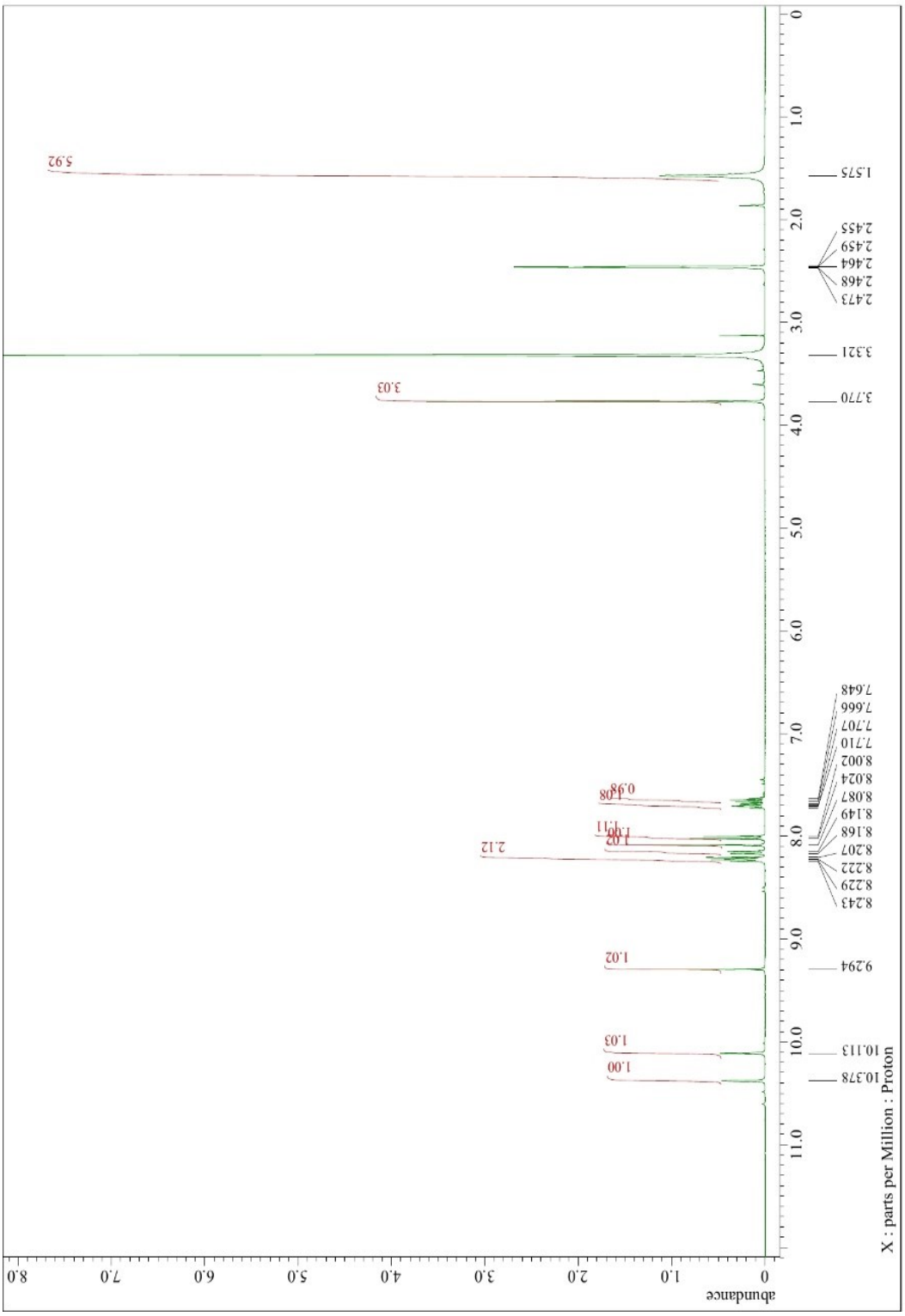


Fig. S31: ^{1}H NMR (400 MHz, DMSO) spectrum of **21**.

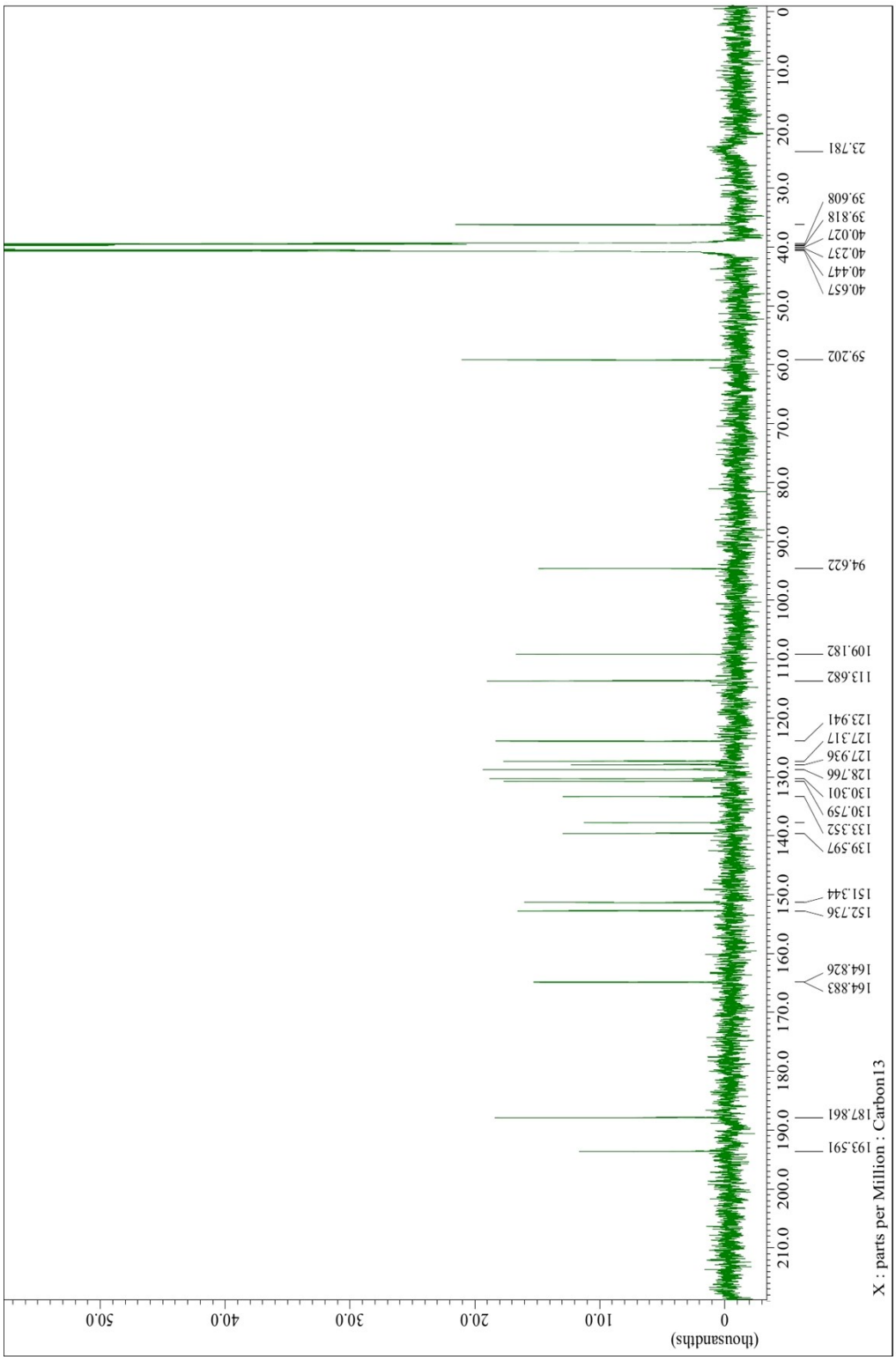


Fig. S32: ^{13}C NMR (100 MHz, DMSO) spectrum of **21**.

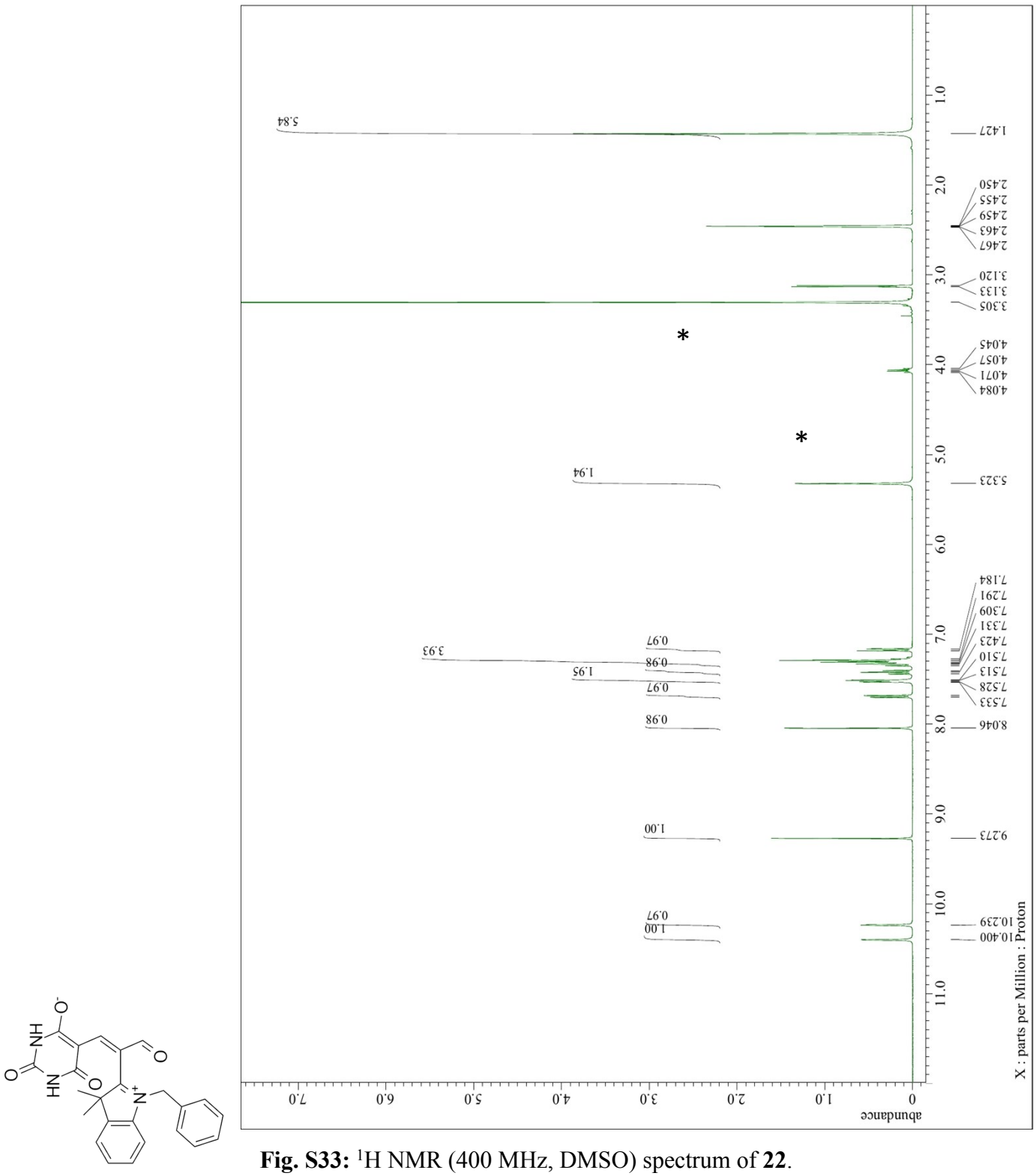


Fig. S33: ^{1}H NMR (400 MHz, DMSO) spectrum of **22**.
* represents MeOH peaks.

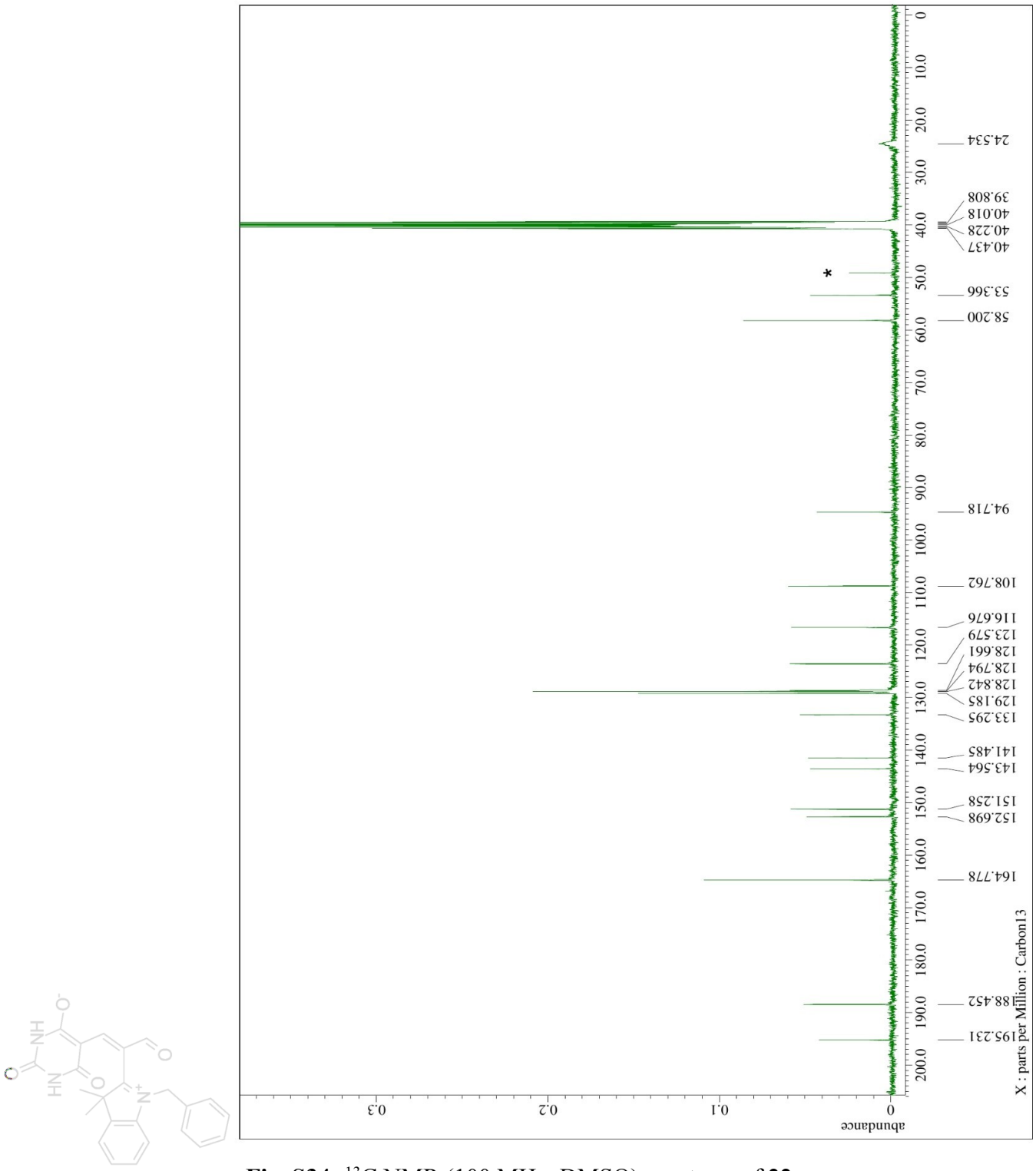


Fig. S34: ^{13}C NMR (100 MHz, DMSO) spectrum of **22**.
* represents MeOH peak.

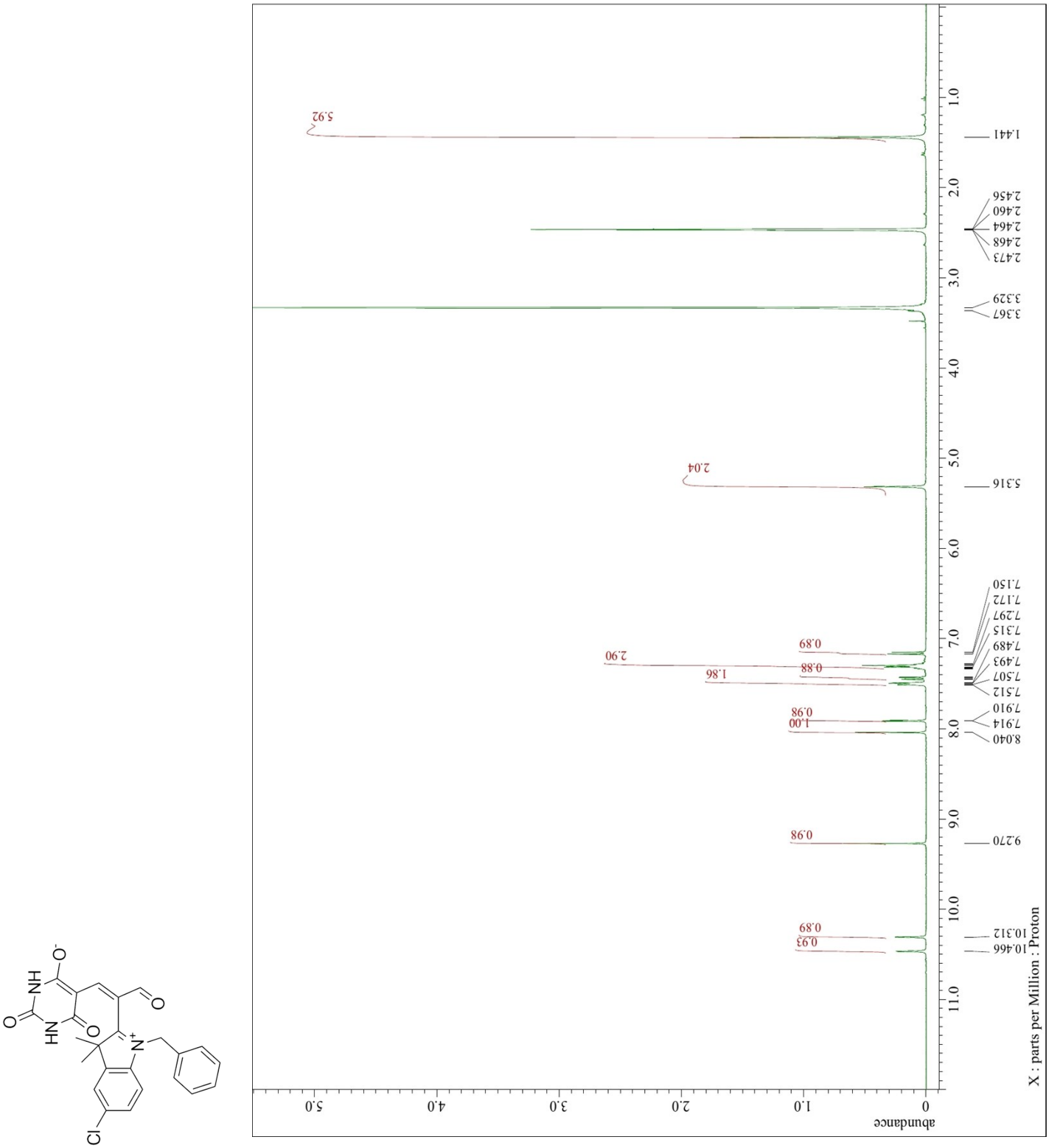


Fig. S35: ^{1}H NMR (400 MHz, DMSO) spectrum of **23**.

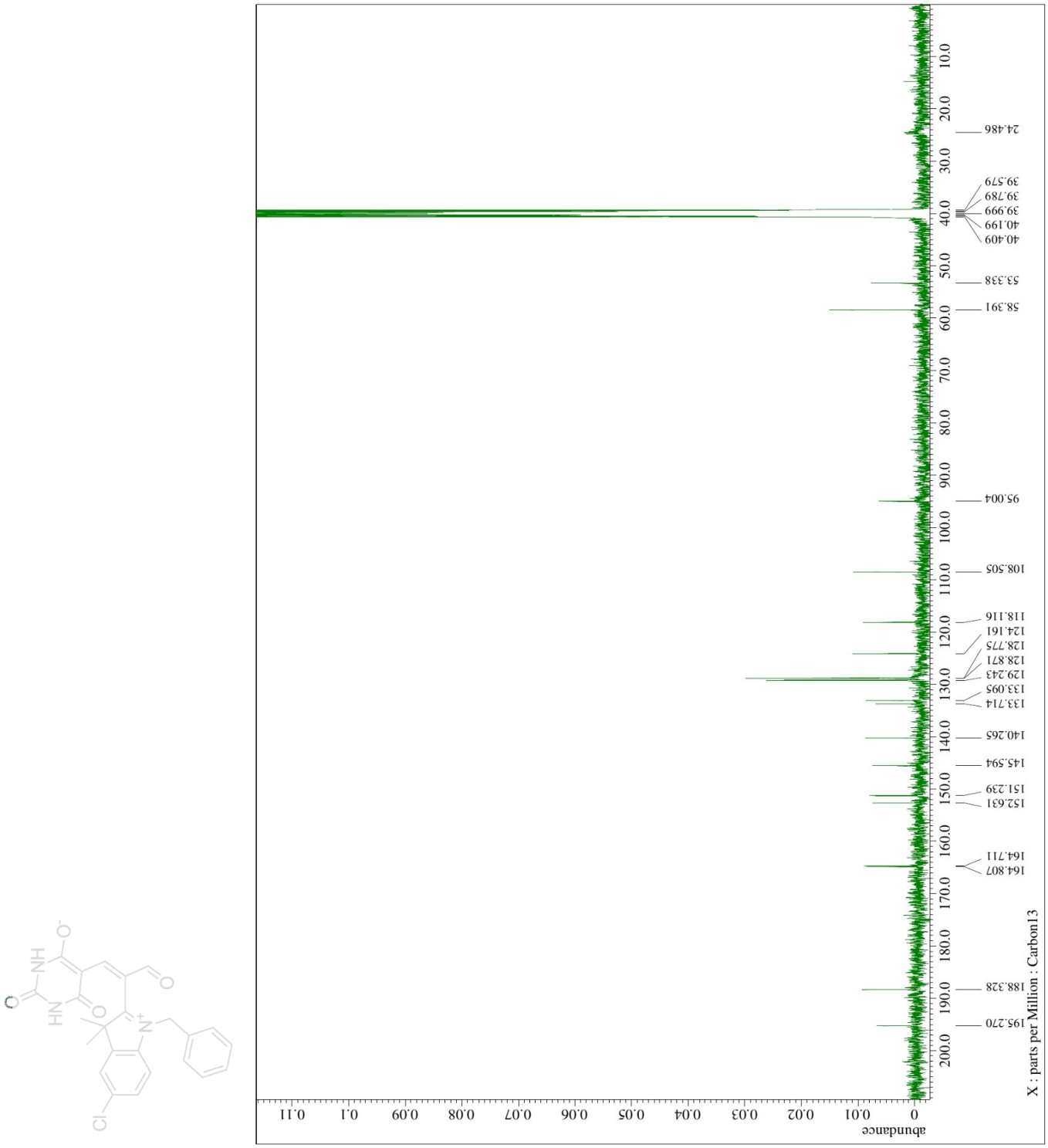


Fig. S36: ^{13}C NMR (100 MHz, DMSO) spectrum of **23**.

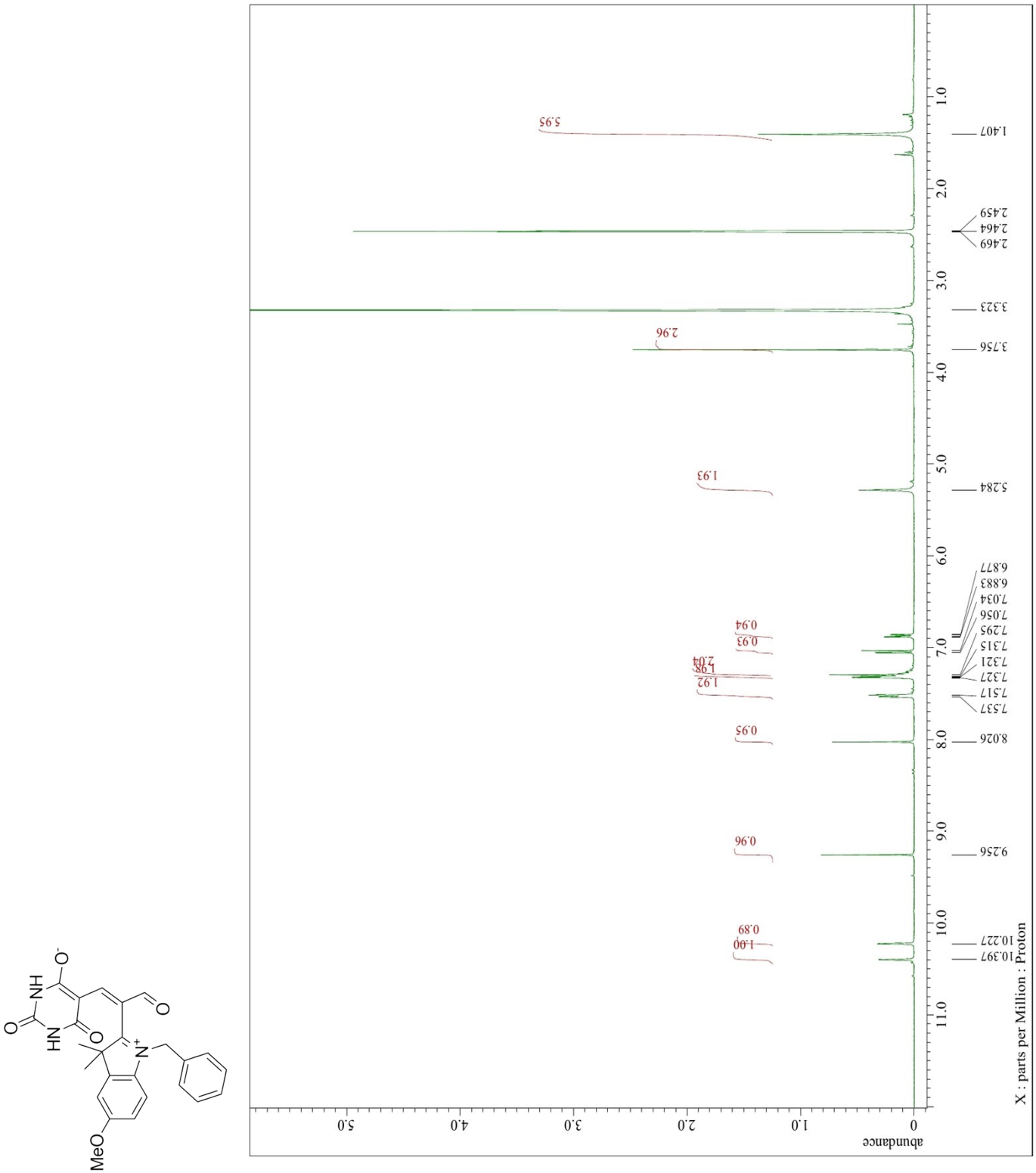


Fig. S37: ^{1}H NMR (400 MHz, DMSO) spectrum of **24**.

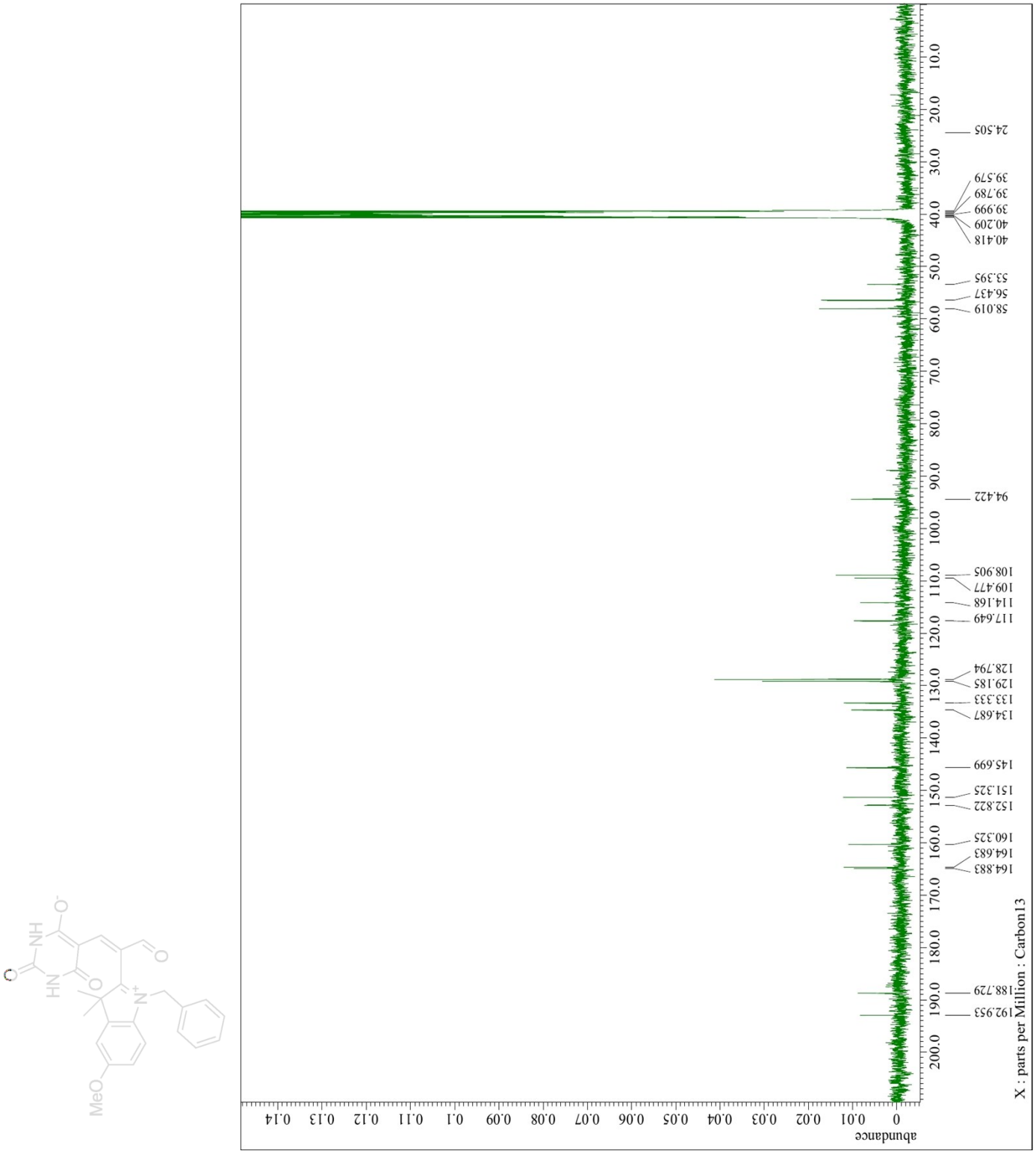


Fig. S38: ^{13}C NMR (100 MHz, DMSO) spectrum of **24**.

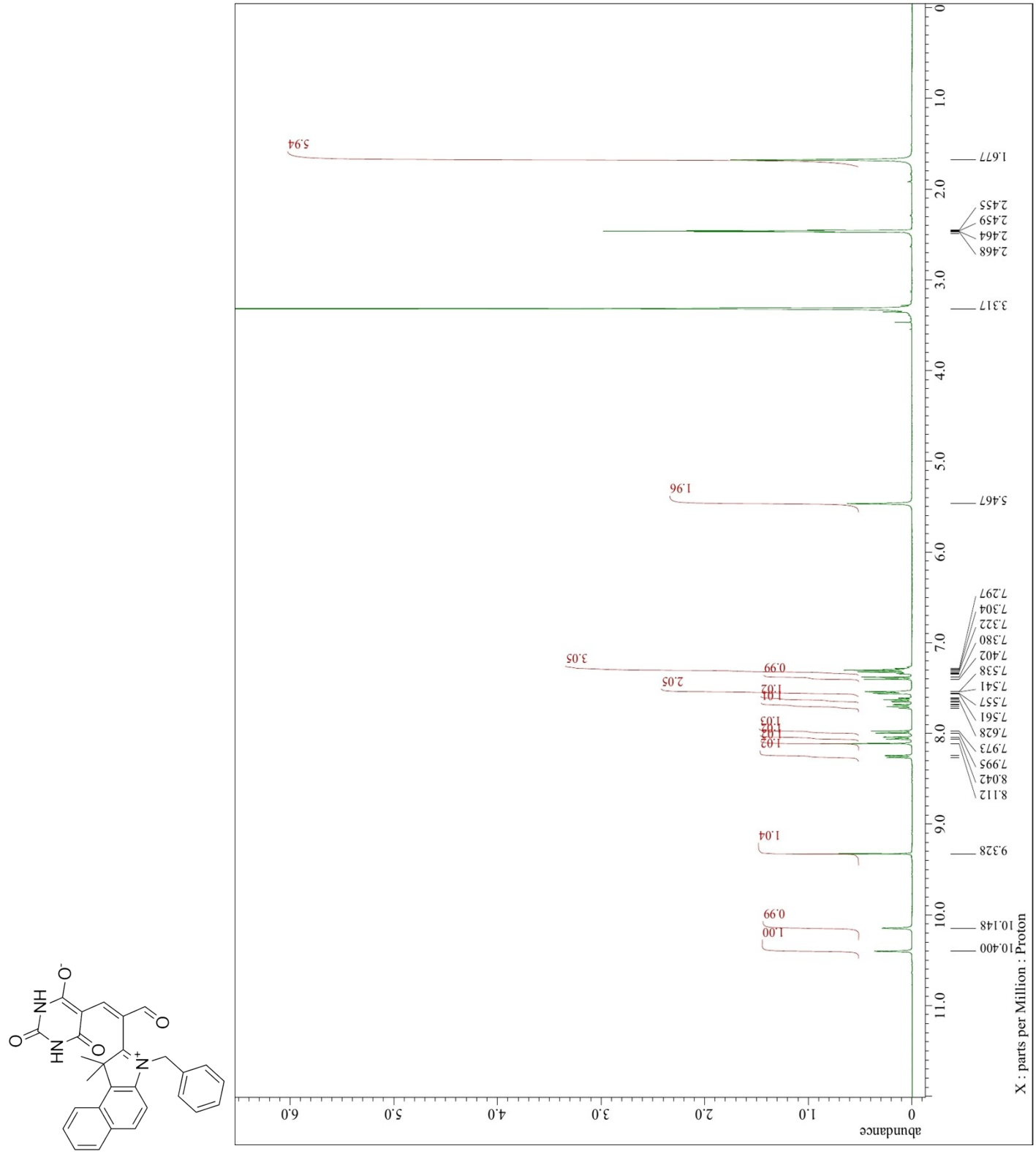


Fig. S39: ^{1}H NMR (400 MHz, DMSO) spectrum of **25**.

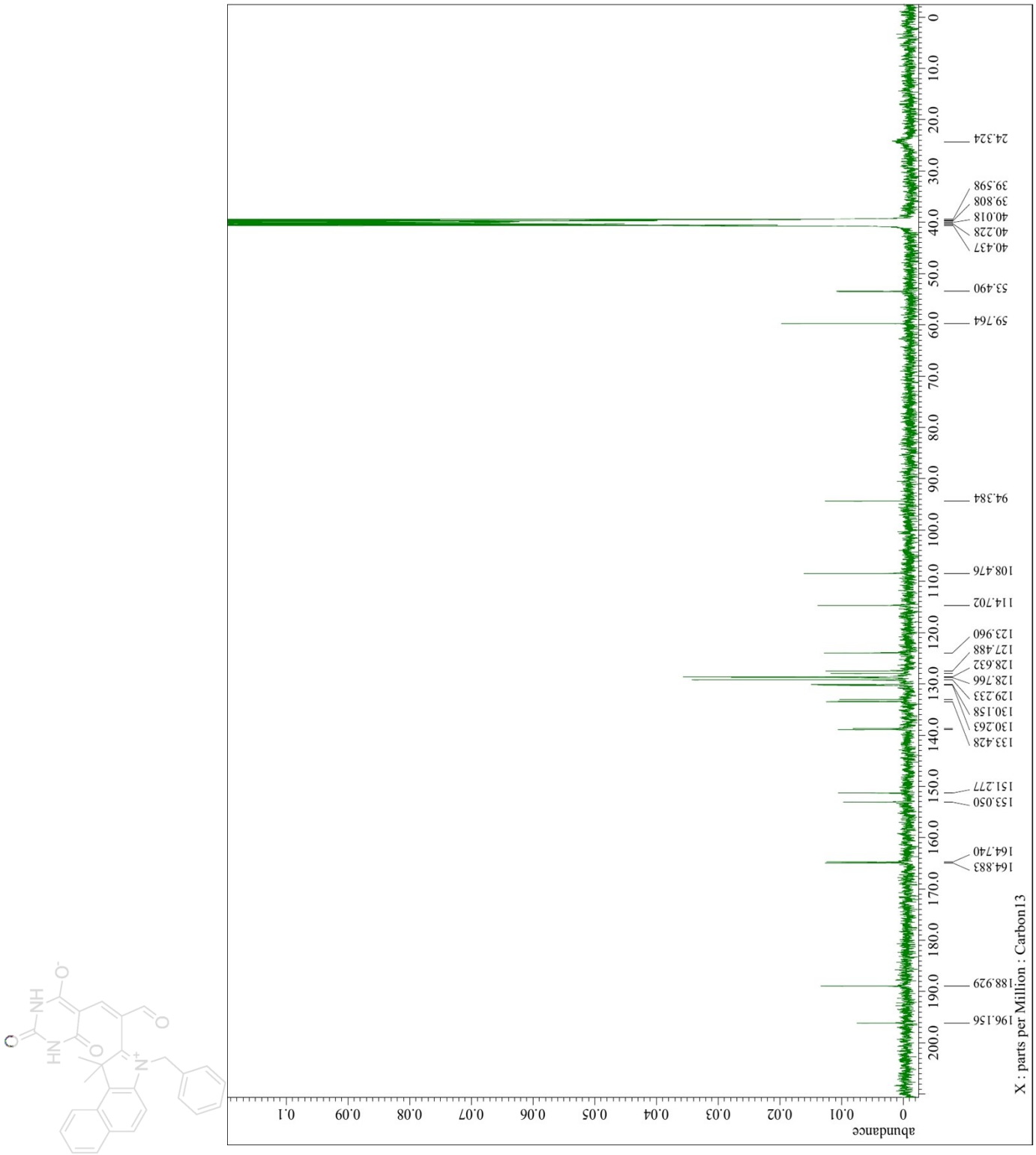


Fig. S40: ^{13}C NMR (100 MHz, DMSO) spectrum of **25**.

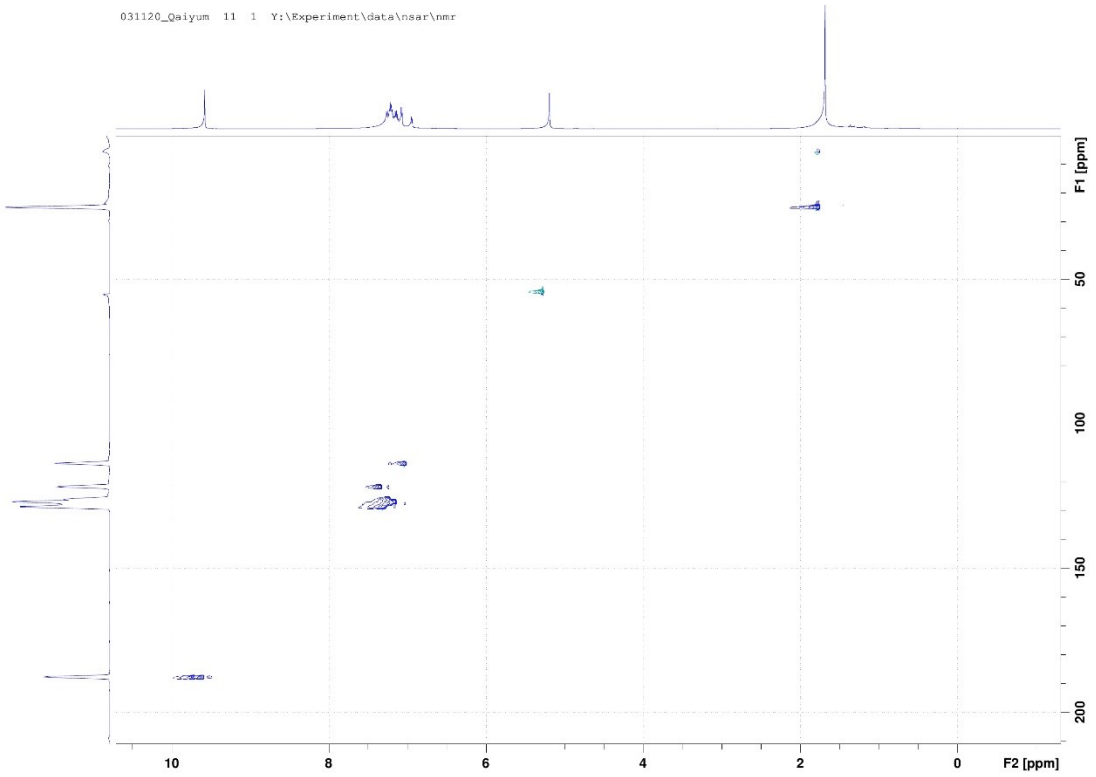


Fig. S41: HSQC spectrum of **12**.

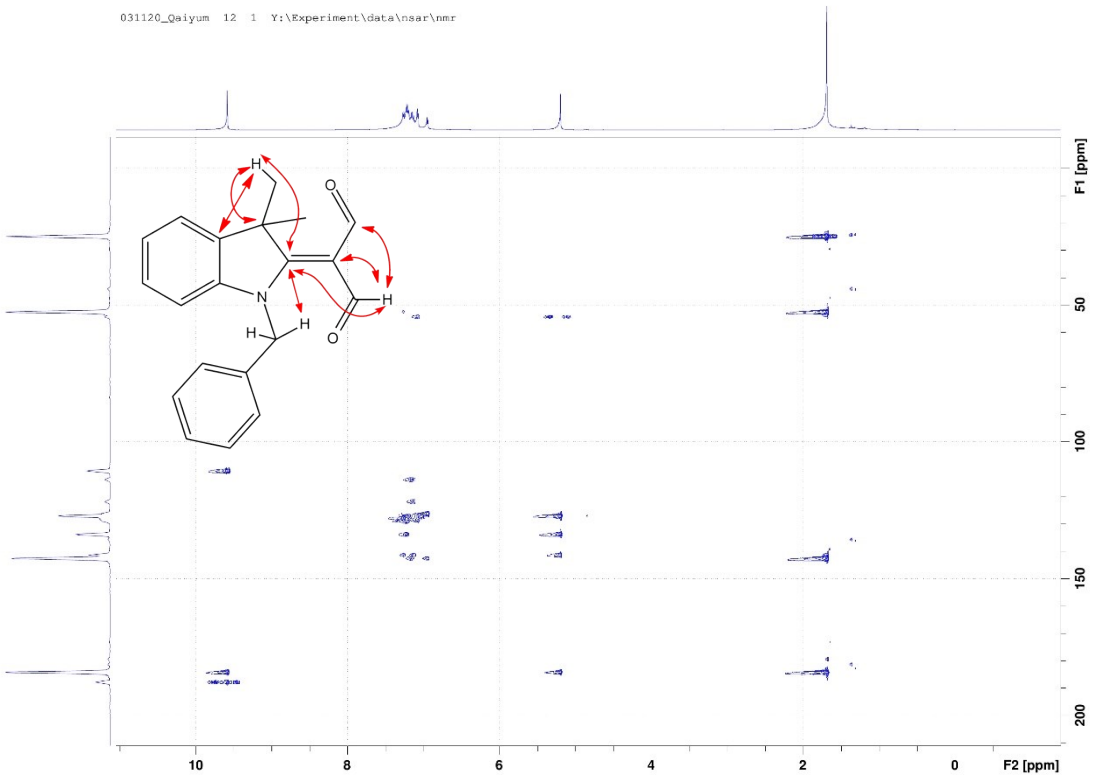


Fig. S42: HMBC spectrum of **12**. The inset image is the crucial HMBC interactions.

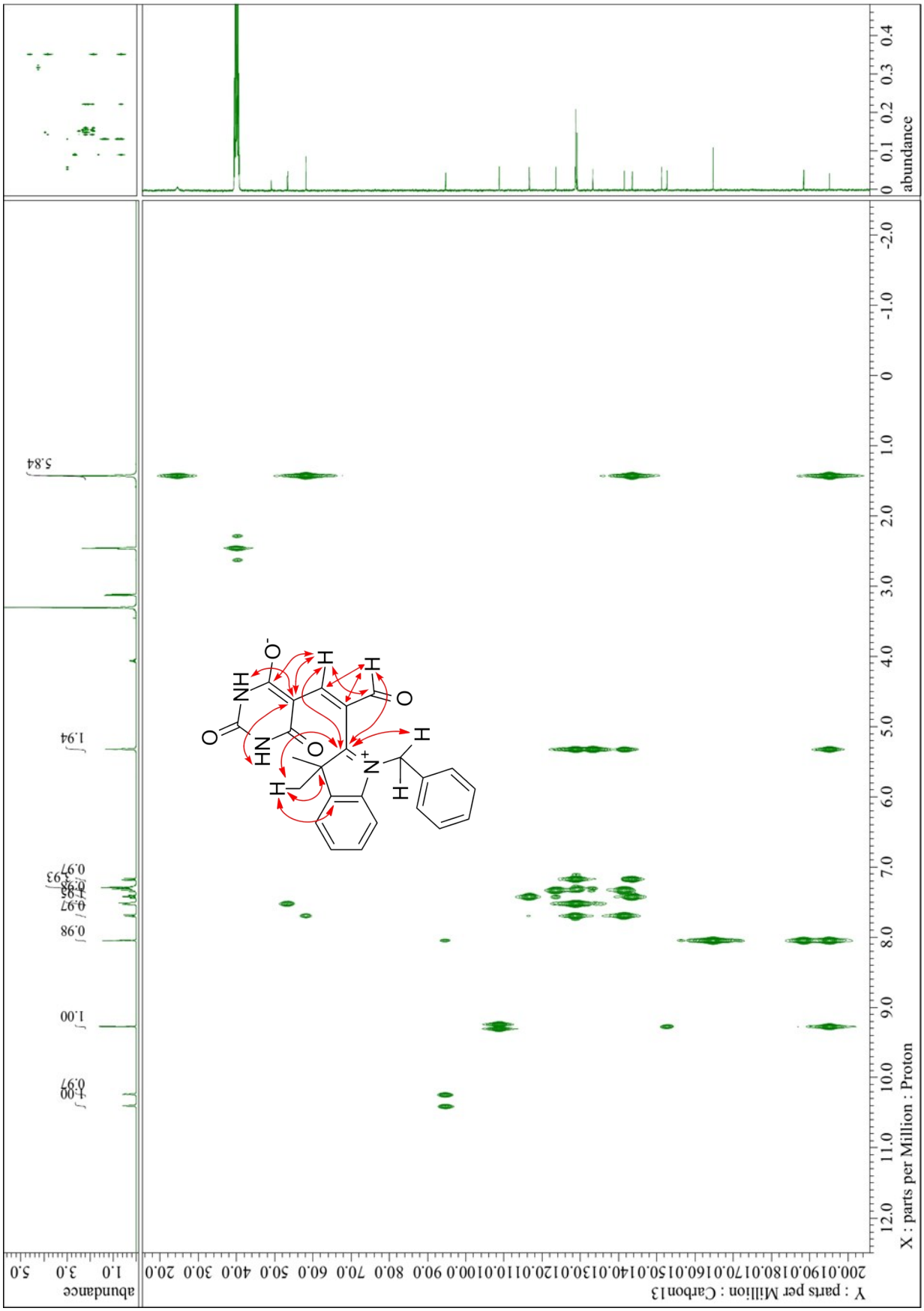


Fig. S43: HMBC spectrum of **22**. The inset image is the crucial HMBC interactions.

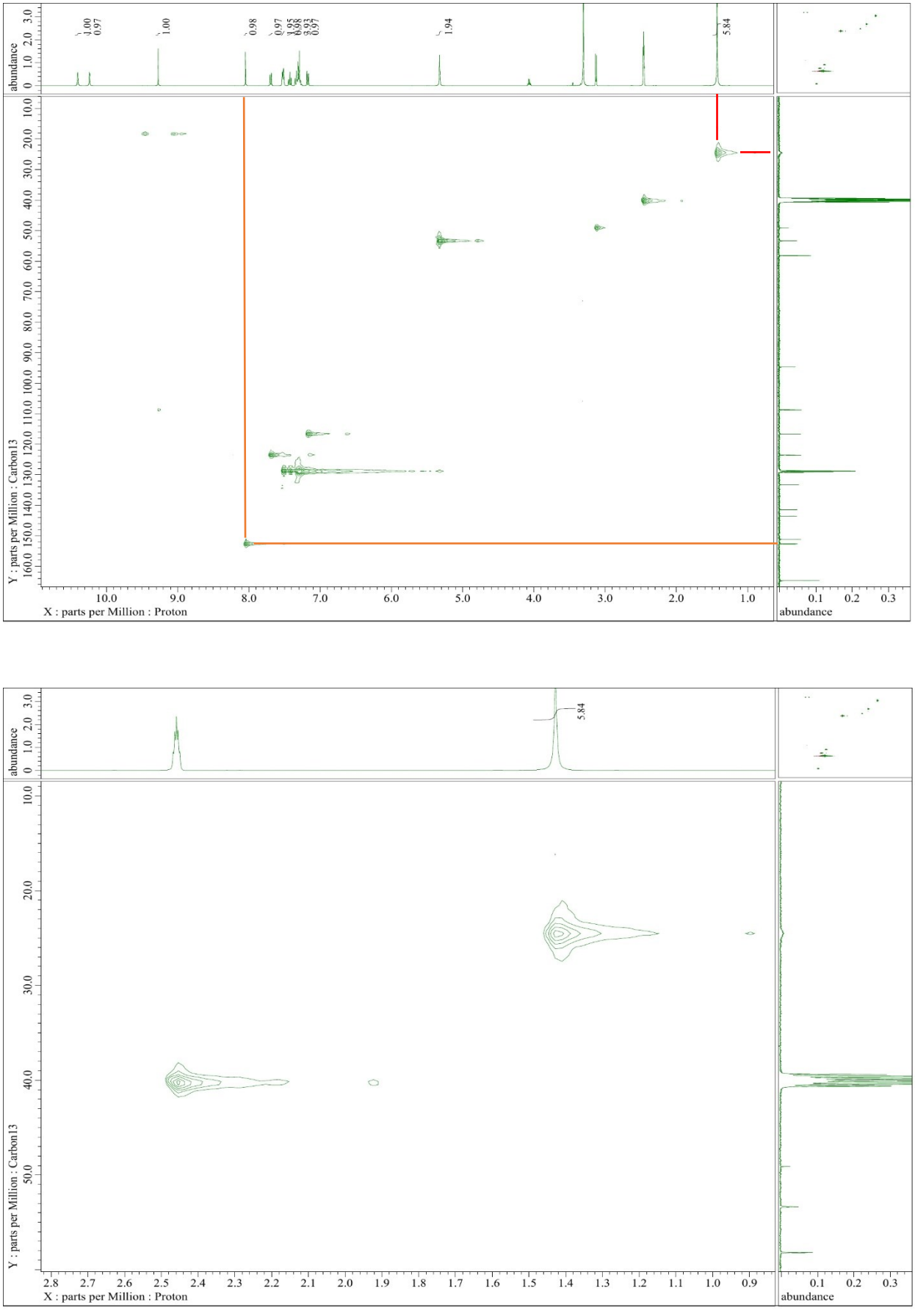


Fig. S44: HSQC spectrum of **22**.

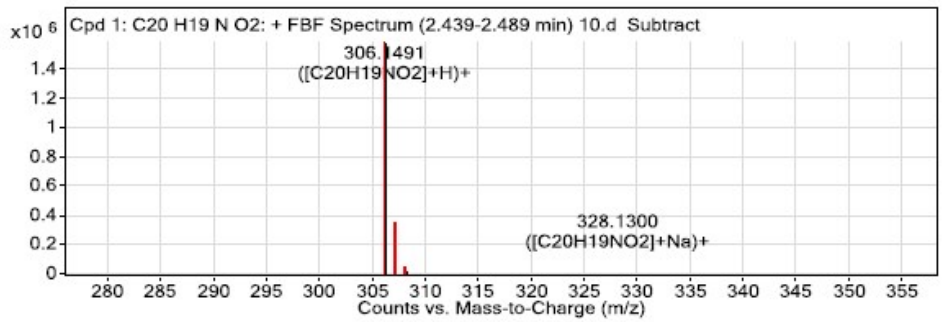


Fig. S45: HRMS spectrum of **12**.

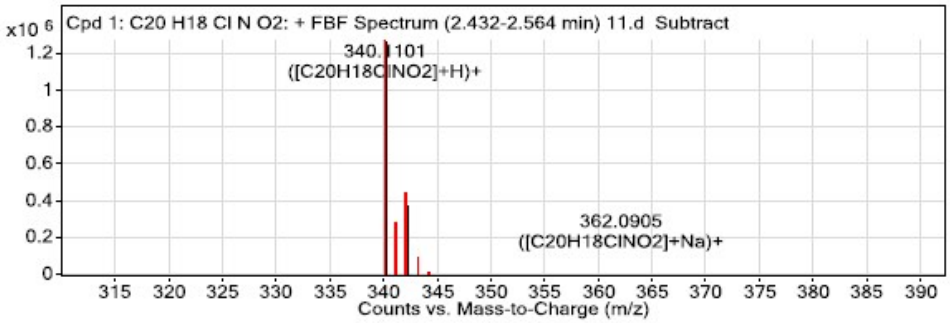


Fig. S46: HRMS spectrum of **13**.

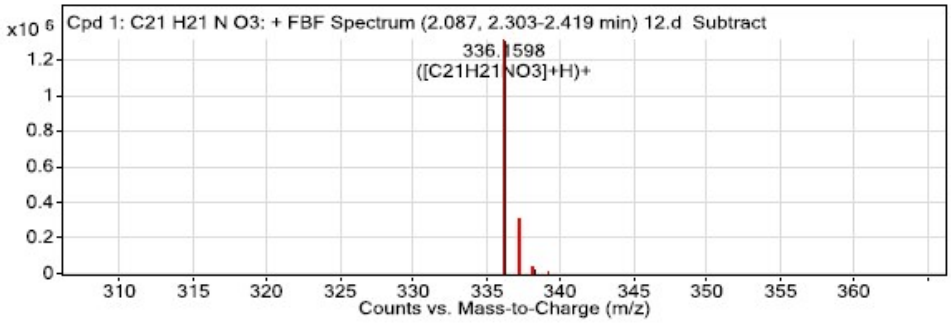


Fig. S47: HRMS spectrum of **14**.

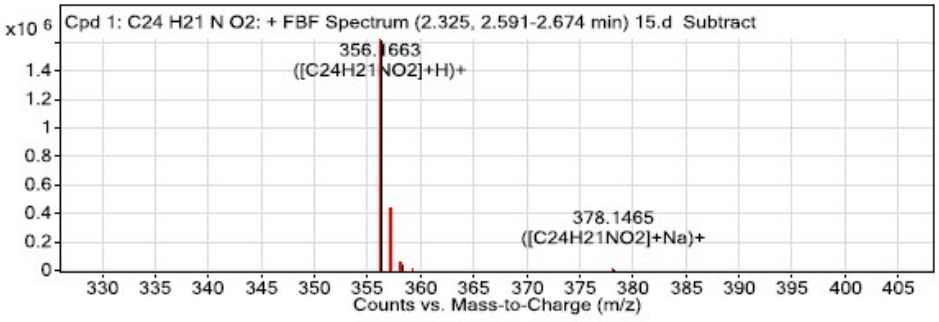


Fig. S48: HRMS spectrum of **15**.

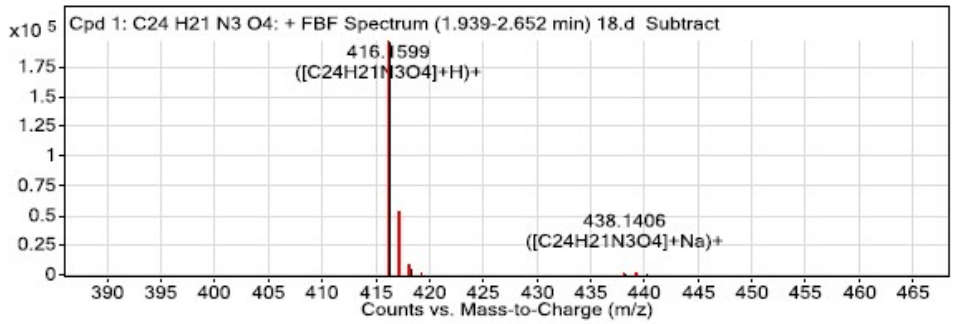


Fig. S49: HRMS spectrum of **22**.

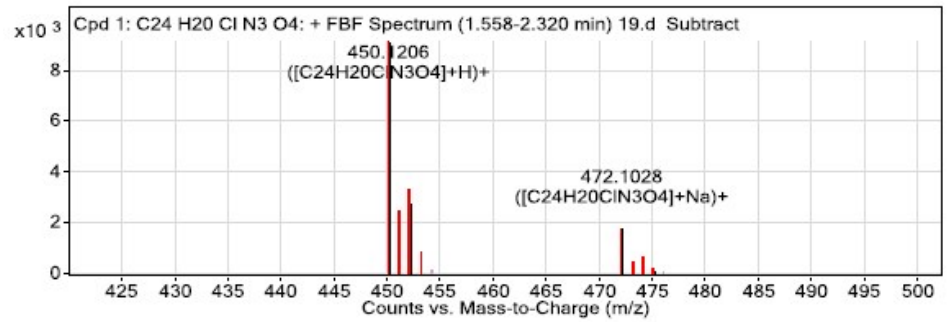


Fig. S50: HRMS spectrum of **23**.

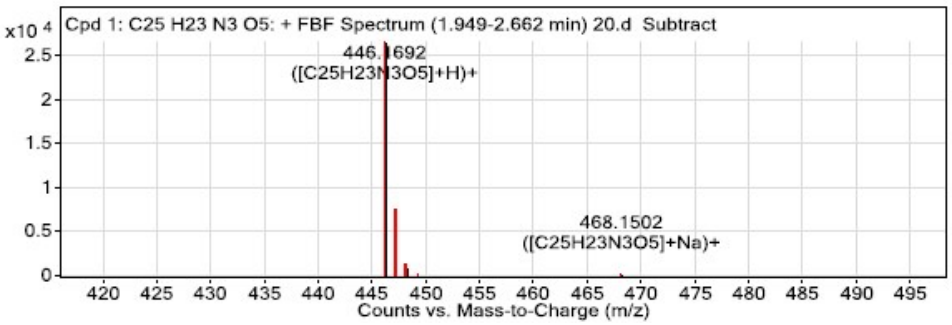


Fig. S51: HRMS spectrum of **24**.

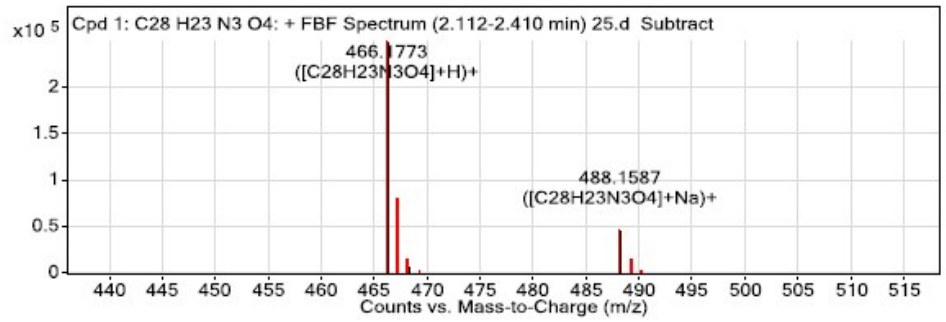


Fig. S52: HRMS spectrum of **25**.

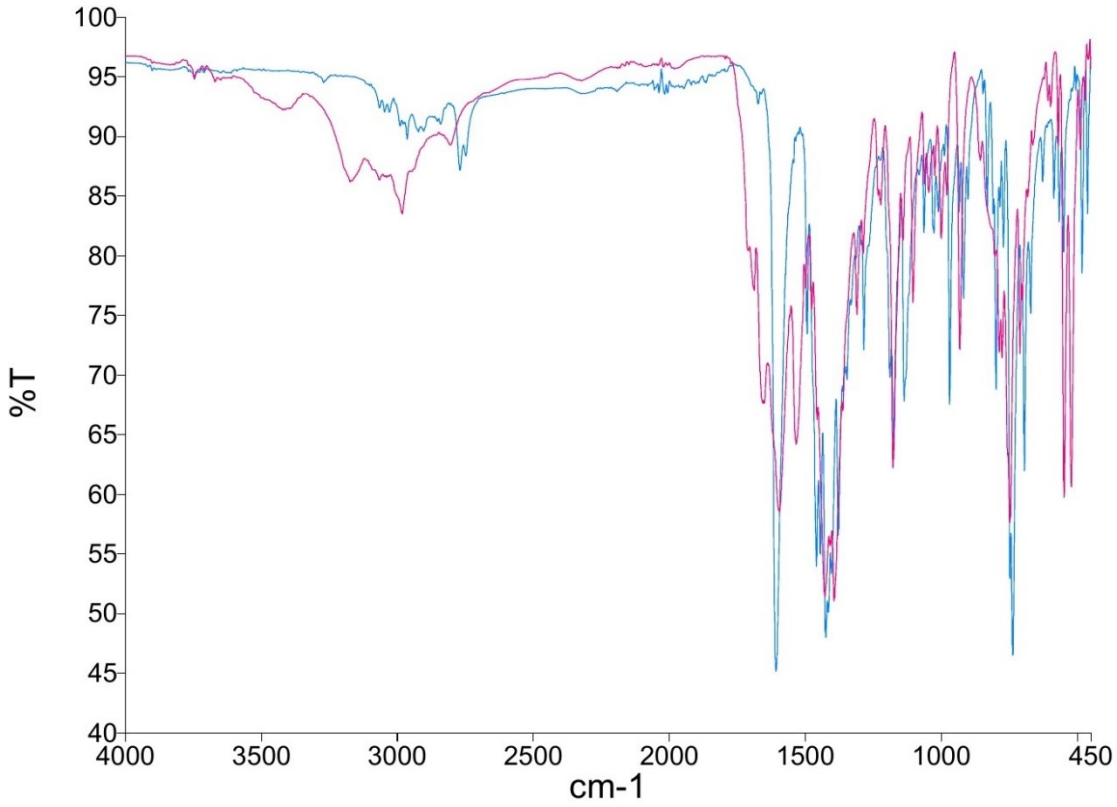


Fig. S53: FT-IR spectra of **12** (blue line) and **22** (purple line).

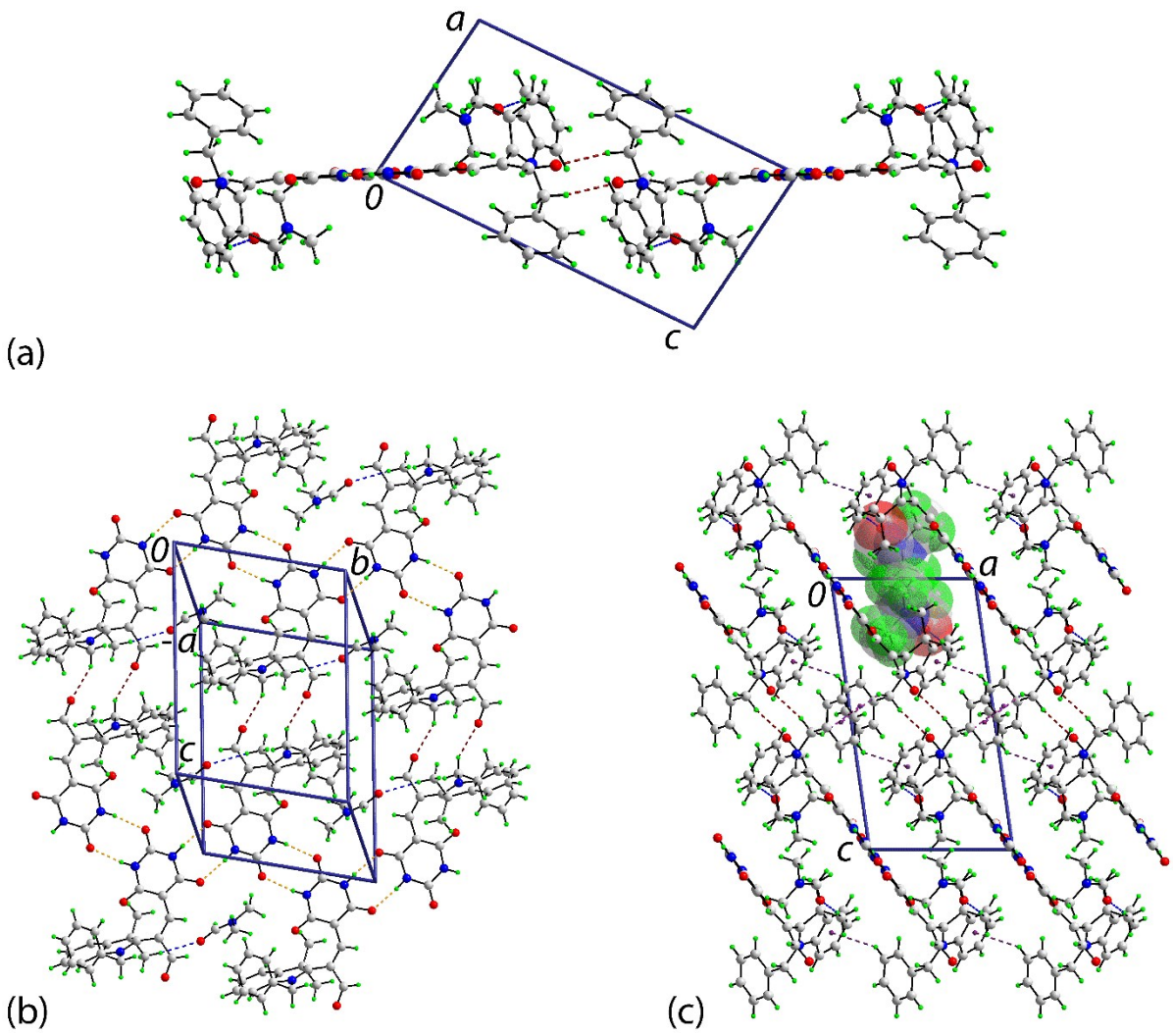


Fig. S54: Molecular packing in the crystal of **22.DMF**: (a) a side-on view of the supramolecular layer, (b) plan view of the supramolecular layer and (c) a view of the unit-cell contents in projection down the *b*-axis with one channel occupied by DMF molecules highlighted in space-filling mode. The methyl-C–H···O(DMF) and methylene-C–H···O(BA) interactions are shown as blue and brown dashed lines, respectively, and the C-H…π(ar.

Table S1: Geometric parameters characterizing the intermolecular points of contact in the crystal of **22.DMF**.

D–H···A	H···A (Å)	D···A (Å)	D – H···A (°)	Symmetry operation
N2–H2n···O3[i]	2.051(14)	2.8965(17)	171.7(16)	2-*x*, 1-*y*, 2-*z*
N3–H3n···O4[ii]	1.984(15)	2.8514(17)	172.6(16)	2-*x*, -*y*, 2-*z*
C9–H9a···O1[iii]	2.43	3.295(2)	148	1-*x*, 1-*y*, 1-*z*
C17–H17b···O5	2.53	3.483(3)	173	*x*, *y*, *z*
C9–H9b···Cg(C3-C8)[iv]	2.91	3.6762(18)	137	2-*x*, 1-*y*, 1-*z*
C14–H14···Cg(C10-C15)[v]	2.83	3.647(3)	148	1+*x*, *y*, *z*

Table S2: Photophysical parameters of **22**.

Solvent	Absorption, λ_{max} (nm) / ε ($\times 10^4$ M^{-1} cm^{-1})	λ_{onset} (nm)	FWHM (nm)	E_{gap} (eV) = 1240 / λ_{onset}
AcOH	349 (2.72)	457	36.5	2.71
Diaxone	351 (2.80), 435 (0.79)	501	27.2, 59.0	2.48
DMSO	354 (2.77)	495	27.9	2.51
MeOH	350 (2.70)	474	31.3	2.62
THF	351 (2.75), 435 (0.78)	501	27.5, 57.2	2.48

Fig. S55: Photograph of zwitterion **19** in dilute DMF solution (left) and after adding 1 equiv. of TFA (right).

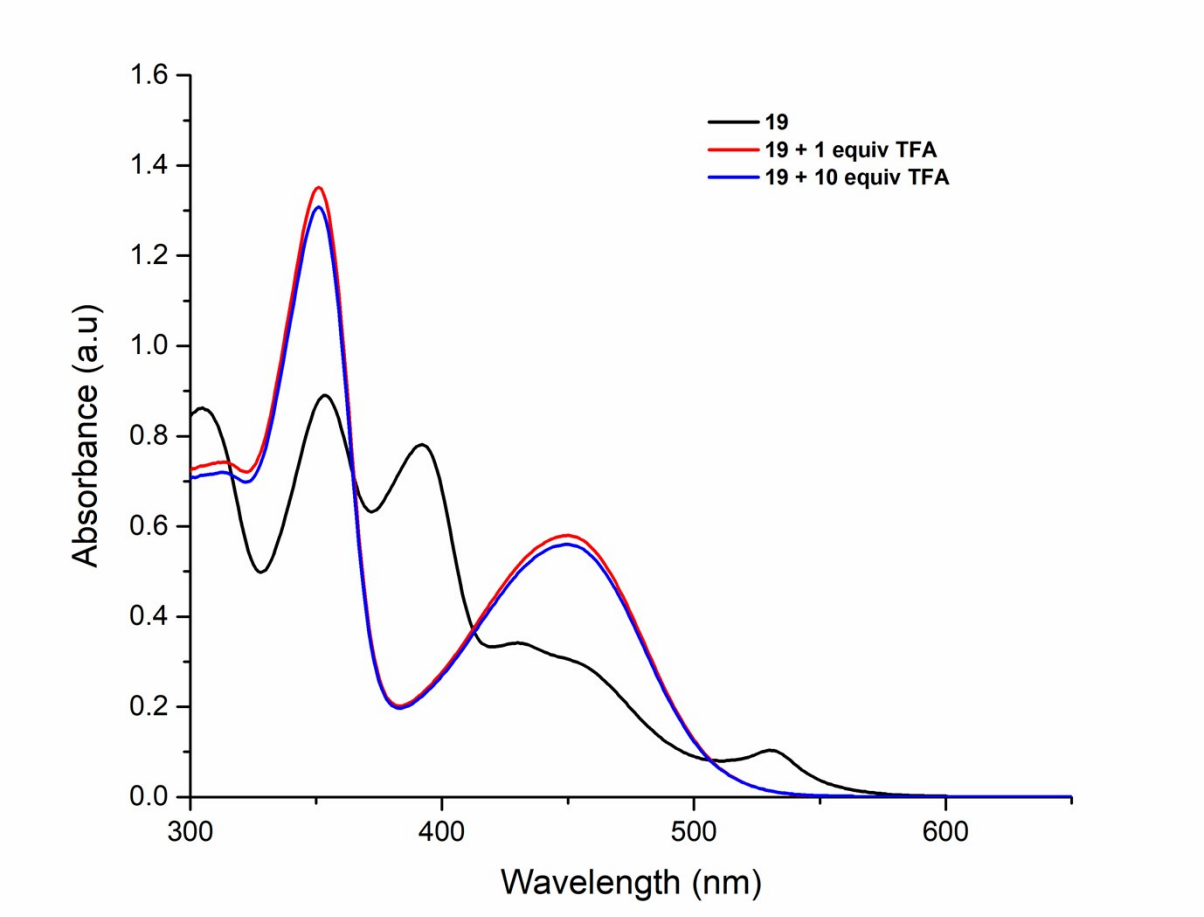


Fig. S56: UV-vis absorption spectra of zwitterion **19** in dilute DMF (50 μM) with different amounts of TFA.

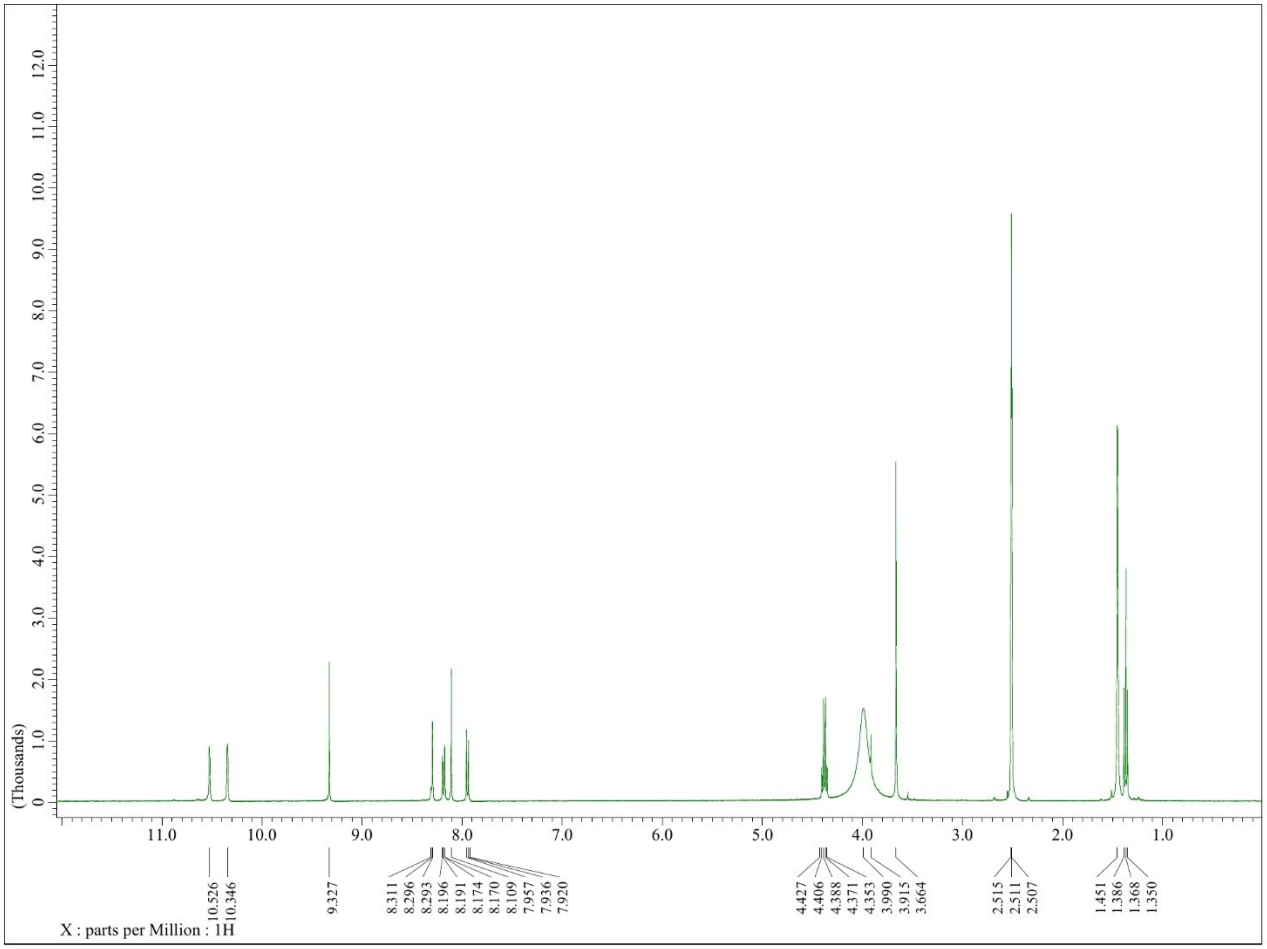


Fig. S57: ^{1}H NMR spectrum (400 MHz, DMSO) of **19** with 10 equiv. of TFA.