Supporting information for

Unexpected formation of *N*-(1-(2-aryl-hydrazono)isoindolin-2-yl)benzamides and their conversion into 1,2-(bis-1,3,4-oxadiazol-2-yl)benzenes

by

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Contents

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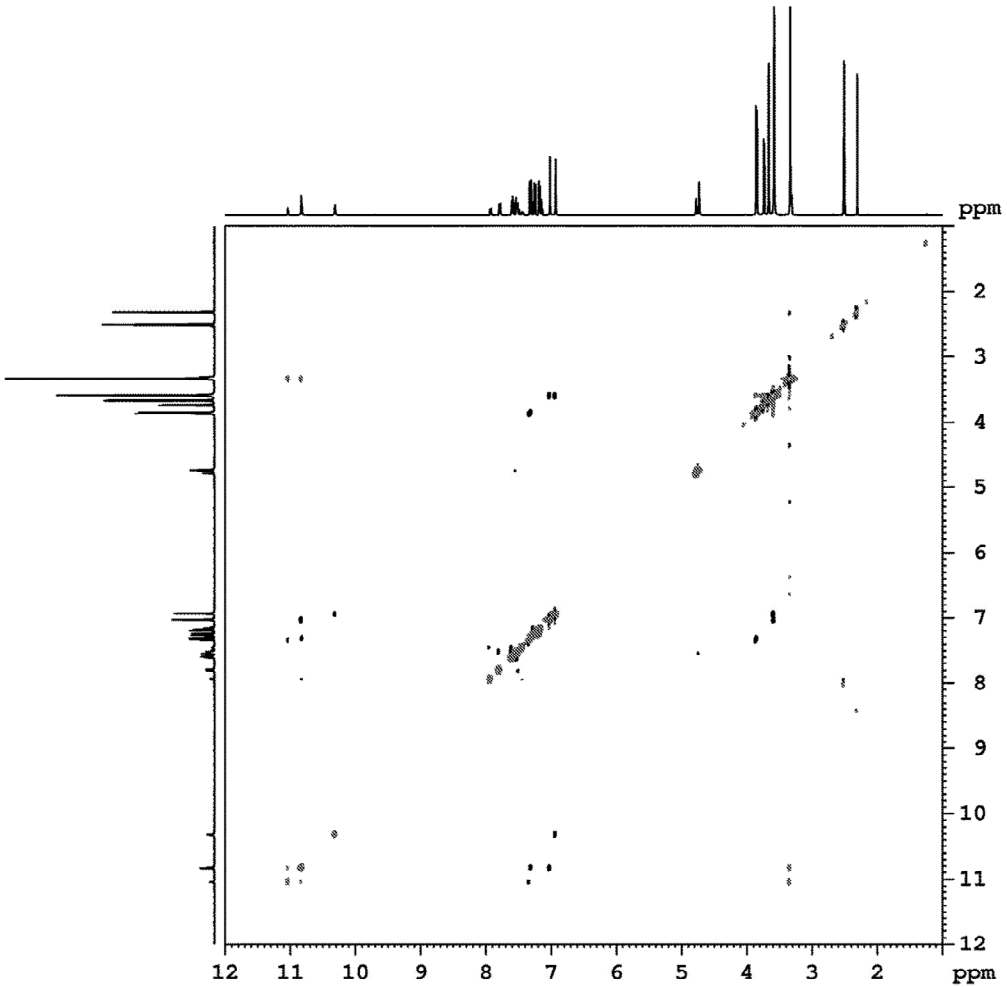
Figure S 1 ROESY full-spectrum of 10Ca



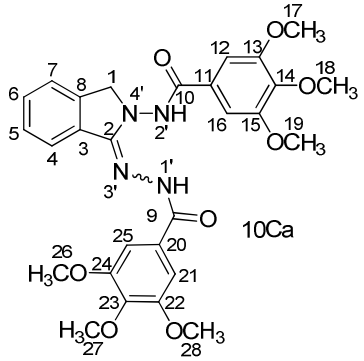
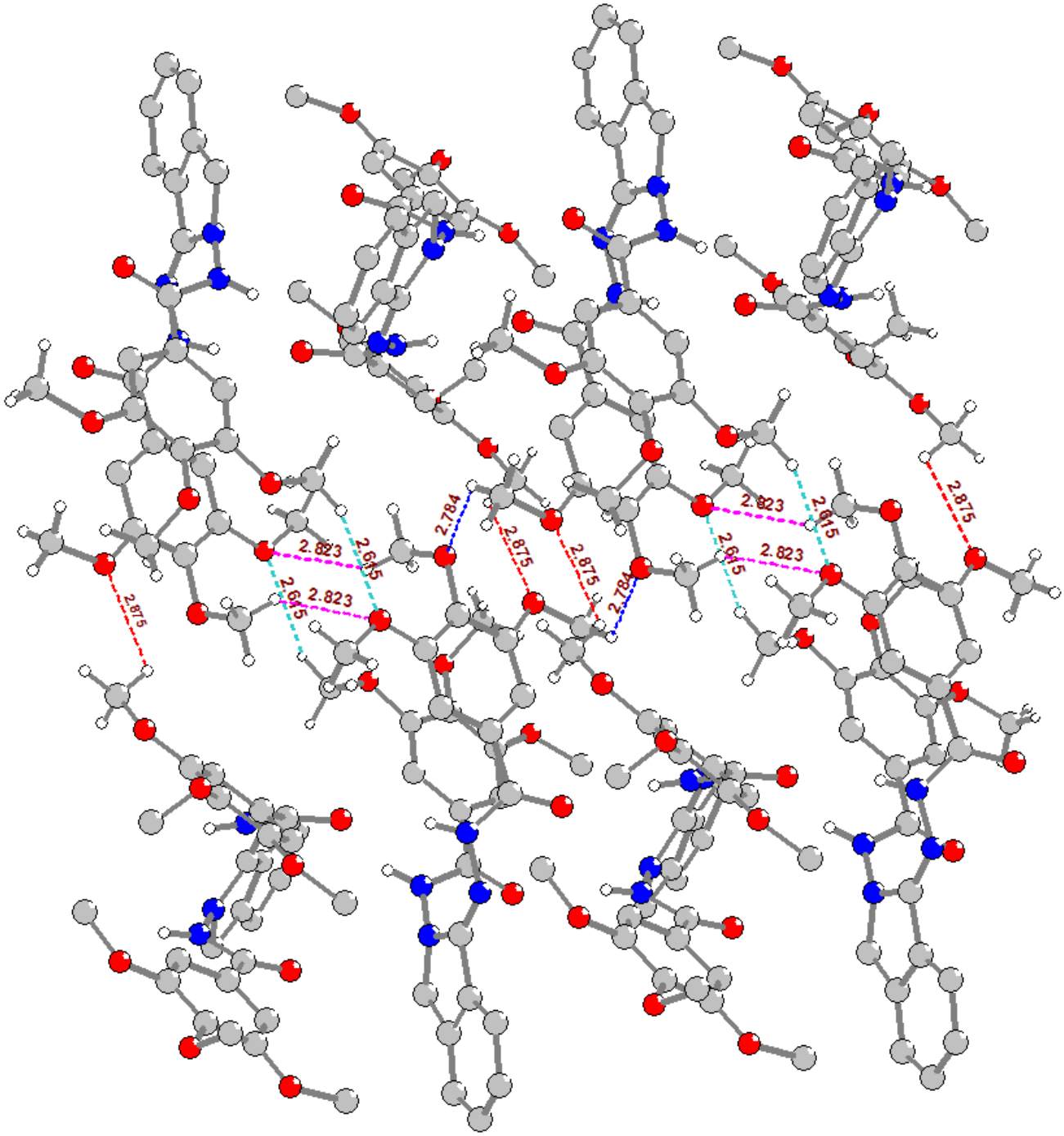


Table S 1 Crystallographic data for 10Ca-*Z*

Formula	**$C_{28}H_{30}N_4O_8$**
FW ($g \cdot mol^{-1}$)	**550.56**
Temperature (K)	**293(2)**
Wavelength (Å)	**0,56080**
Crystal system	**Monoclinic**
Space group	***P*2$_1$/*n***
a (Å)	**10,2036(18)**
b (Å)	**14,2650(15)**
c (Å)	**18,965(3)**
α (º)	**90,00**
β (º)	**97,125(11)**
γ (º)	**90,00**
V ($Å^3$)	**2739,1(7)**
Z	**4**
Density ($g \cdot cm^{-3}$)	**1,335**
μ (mm^{-1})	**0,061**
F (000)	**1160**
Fit quality for F^2	**1,021**
R_1 final, wR^2 [I > 2(I)]	**0,0786; 0,1725**
R_1, wR^2 (for all data)	**0,1378; 0,2002**
Maximum difference peak and hole (e $Å^{-3}$)	**0,457; -0,266**

Figure S 2 Crystal packing of 10C-*Z* (view along *a* axis) with illustration of the C–H---O contacts between the supramolecular chains



General experimental procedures, ^{1}H NMR and MS spectra of crude reactions for condensations of *ortho*-phthalaldehyde and hydrazide 9a

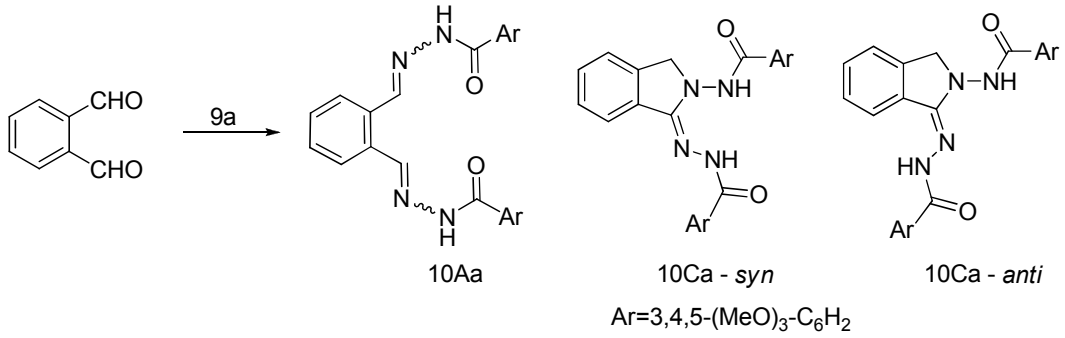


Table S 2 Reaction conditions for condensation of *ortho*-phthaldehyde and hydrazide **9a**

Entry	Solvent	Reaction time (h)	Temperature (ºC)	Catalyst
1	EtOH	24	78	-
2	EtOH	4/12	78/rt	-
3	$CHCl_3$	24	rt	-
4	$CHCl_3$	24	63	AcOH
5	$CHCl_3$	4/12	63/rt	AcOH
6	toluene	1	110	-

Entry 1

Hydrazide **9a** (3 mmol) and *ortho*-phthalaldehyde (1,5 mmol, 0.201 g) were refluxed for 24 h in ethanol (50 mL). The solvent was removed under vacuum, the residue was dried and ^{1}H NMR spectrum recorded (Figure S3).

Entry 2

Hydrazide **9a** (3 mmol) and *ortho*-phthalaldehyde (1,5 mmol, 0.201 g) were refluxed for 4 h in ethanol (50 mL). The mixture was then stirred at room temperature for 12 h. The solvent was removed under vacuum, the residue (denoted R2-**10a**) was dried and ^{1}H NMR spectrum (Figure S4) were recorded.

Entry 3

Hydrazide **9a** (3 mmol) and *ortho*-phthalaldehyde (1.5 mmol, 0.201 g) were stirred at room temperature for 24 h in chloroform (50 mL). The solvent was removed under vacuum, the residue (denoted R3-10a) was washed with cold ethyl ether, filtered and washed again with cold methanol. After drying ^{1}H NMR spectrum was recorded (Figure S5).

Entry 4

Hydrazide **9a** (3 mmol) and *ortho*-phthalaldehyde (1.5 mmol, 0.201 g) were refluxed for 24 h in chloroform (50 mL) and a few drops of acetic acid. The solvent was removed under vacuum, the residue (denoted R4-**10a**) was dried and ^{1}H NMR spectrum recorded (Figure S6).

Entry 5

Hydrazide **9a** (3 mmol) and *ortho*-phthalaldehyde (1.5 mmol, 0.201 g) were refluxed for 4 h in chloroform (50 mL) and a few drops of acetic acid added. The mixture was then stirred at room temperature for 12 h. The solvent was removed under vacuum, the residue was dried and ^{1}H NMR spectra and RP-HPLC performed. The solvent was removed under vacuum, the residue (denoted R5-**10a**) was washed with cold ethyl ether, filtered and washed again with cold methanol. After drying ^{1}H NMR spectrum was recorded (Figure S7).

Entry 6

Hydrazide **9a** (3 mmol) and *ortho*-phthalaldehyde (1.5 mmoli, 0.201 g) were refluxed for 1 h in toluene (50 mL). The solution was concentrated under vacuum, cooled to room temperature and the resulted white residue (denoted R6-**10a**) was dried and ^{1}H NMR spectrum recorded (Figure S8).

Figure S 3 ^{1}H NMR spectrum of crude condensation reaction corresponding to entry 1 in Table S2

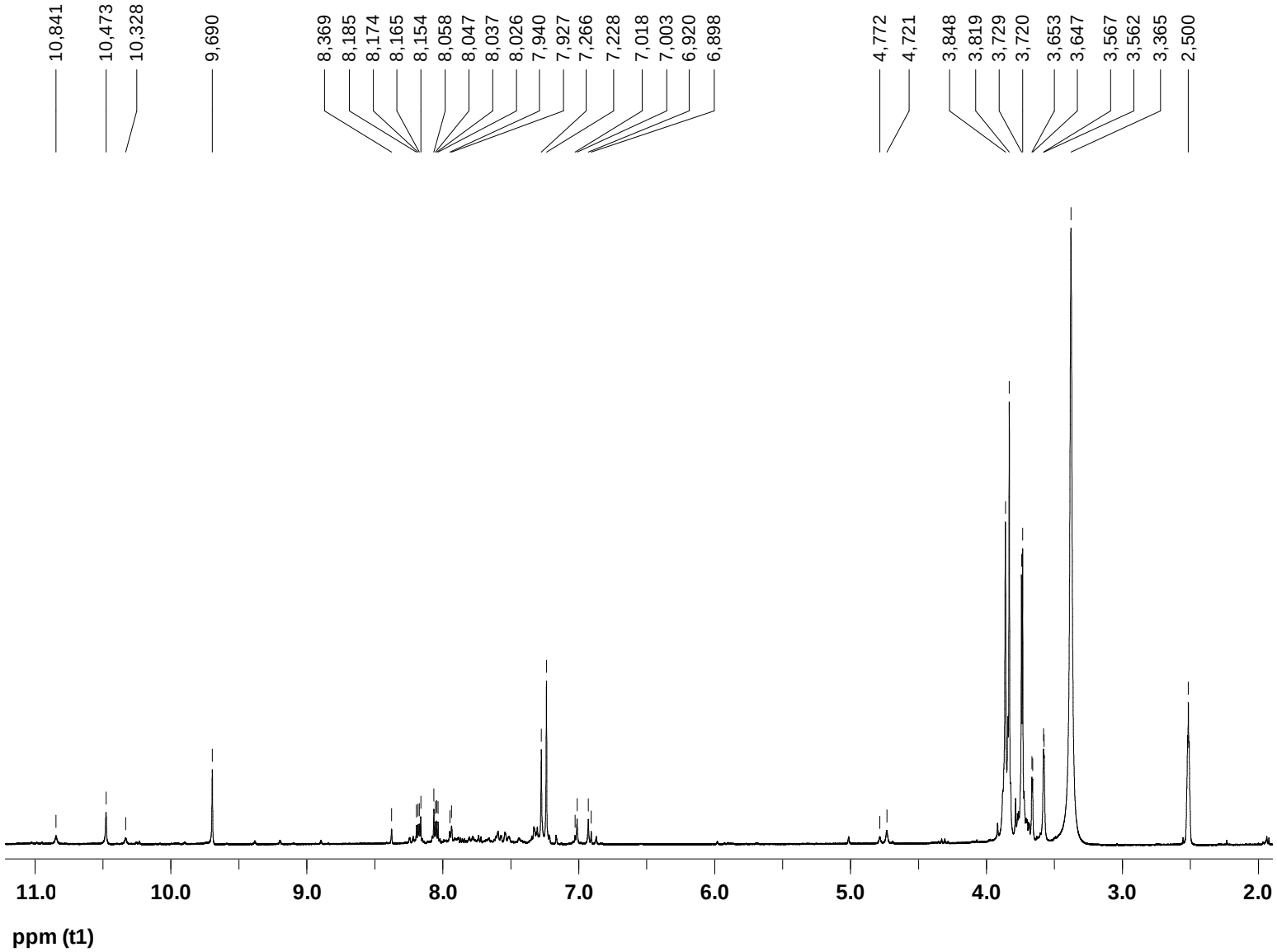


Figure S 4 ^{1}H NMR spectrum of crude condensation reaction corresponding to entry 2 in Table S2

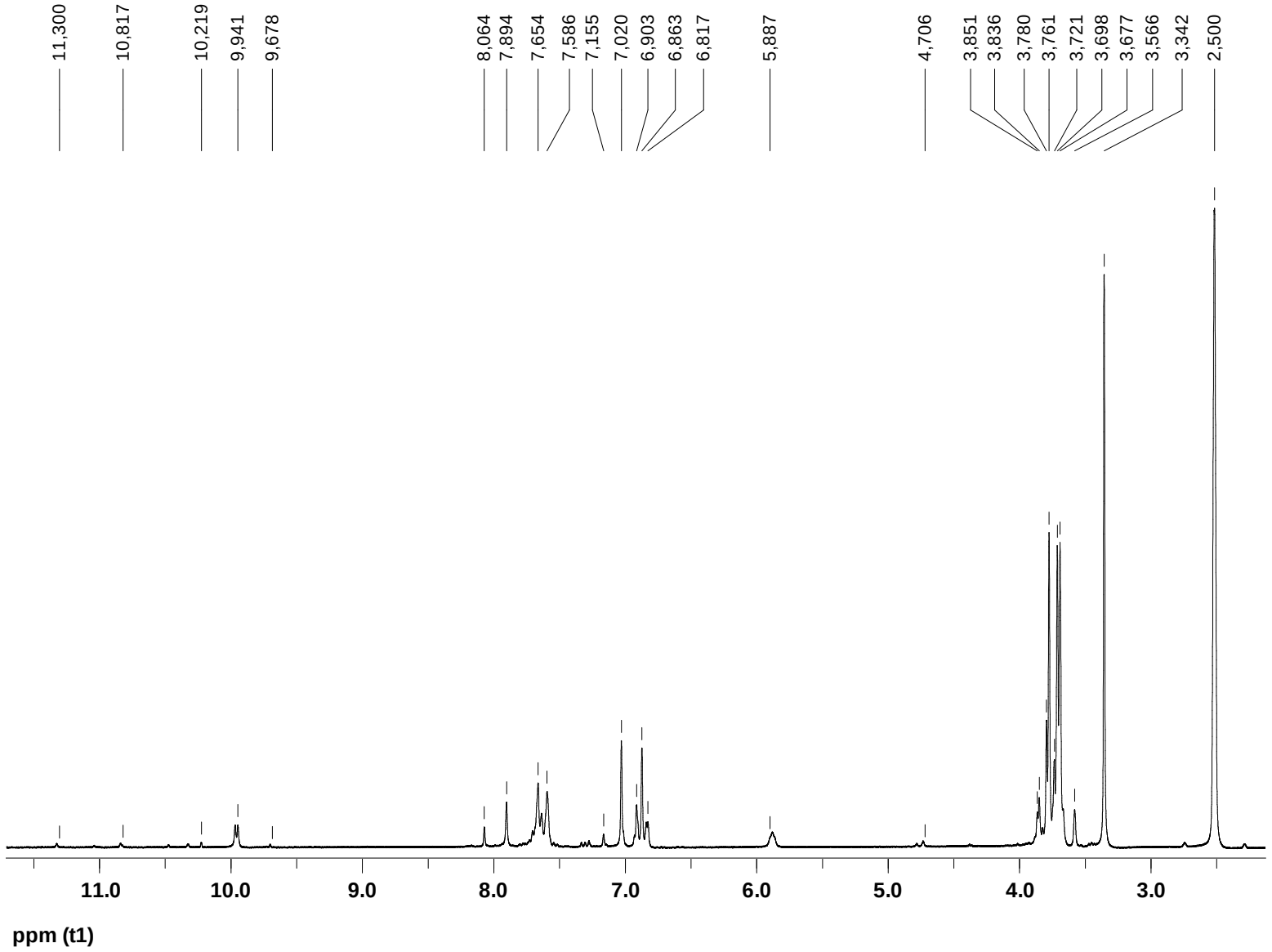


Figure S 5 ^{1}H NMR spectrum of crude condensation reaction corresponding to entry 3 in Table S2

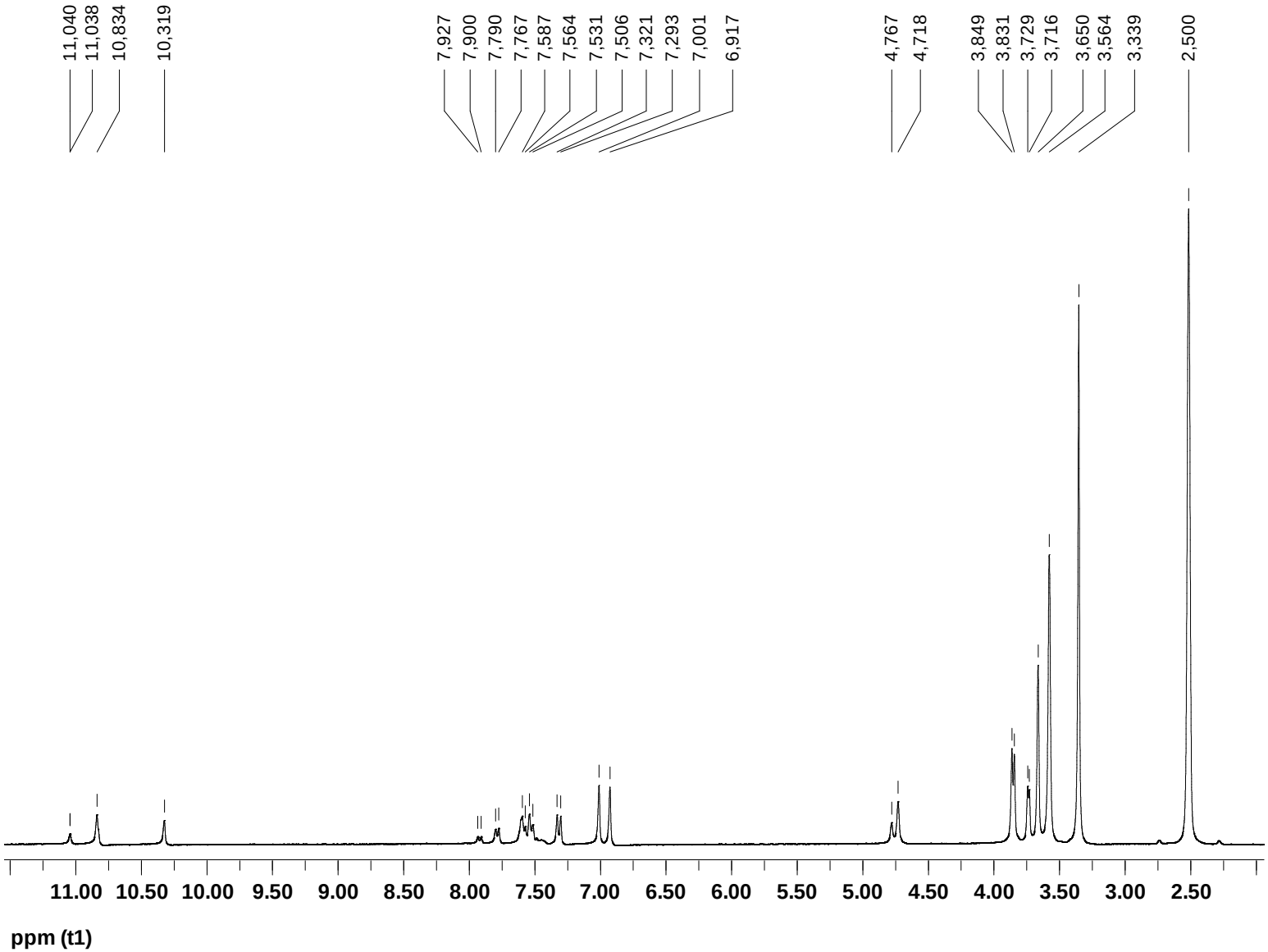


Figure S 6 ^{1}H NMR spectrum of crude condensation reaction corresponding to entry 4 in Table S2

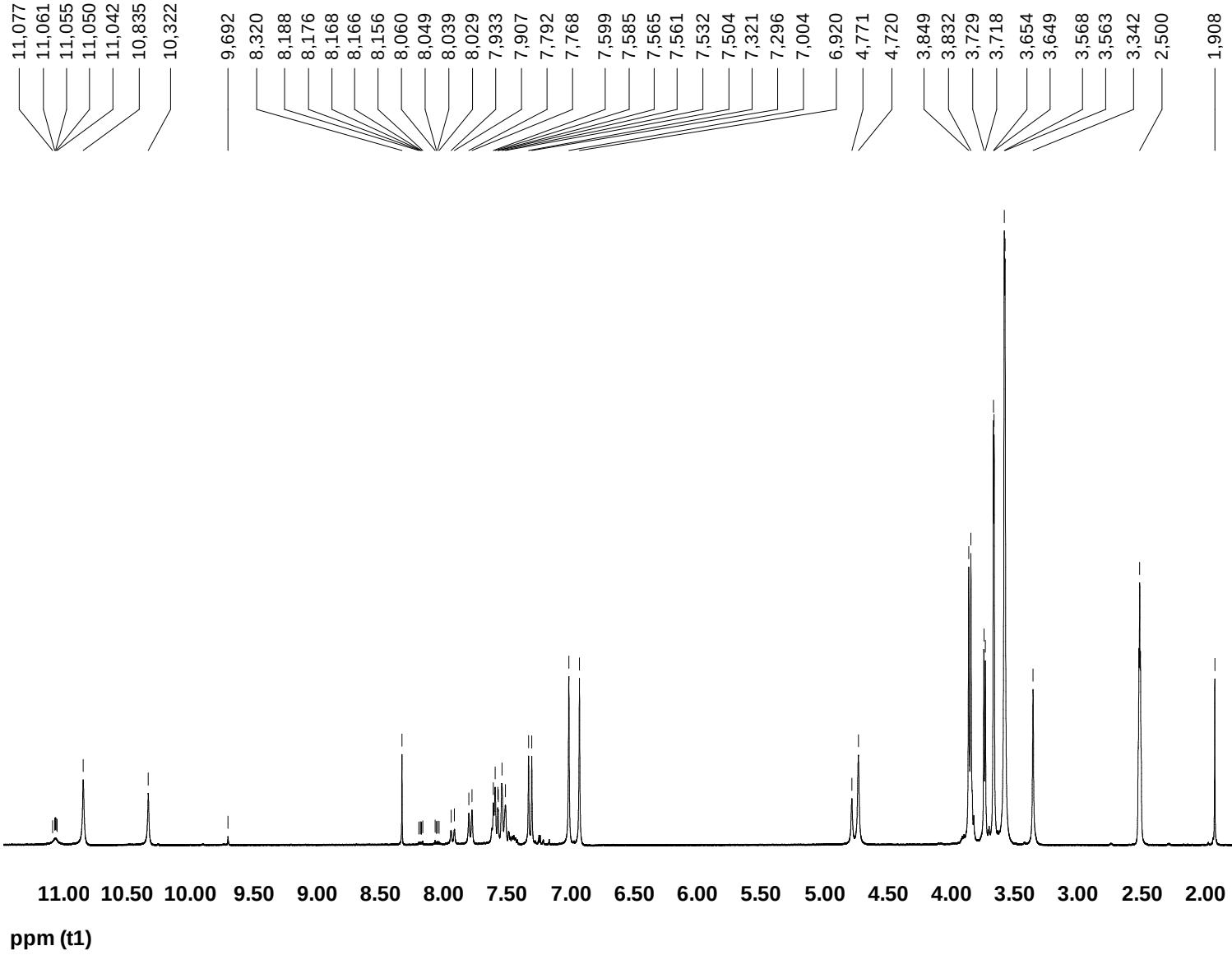


Figure S 7 ^{1}H NMR spectrum of crude condensation reaction corresponding to entry 5 in Table S2

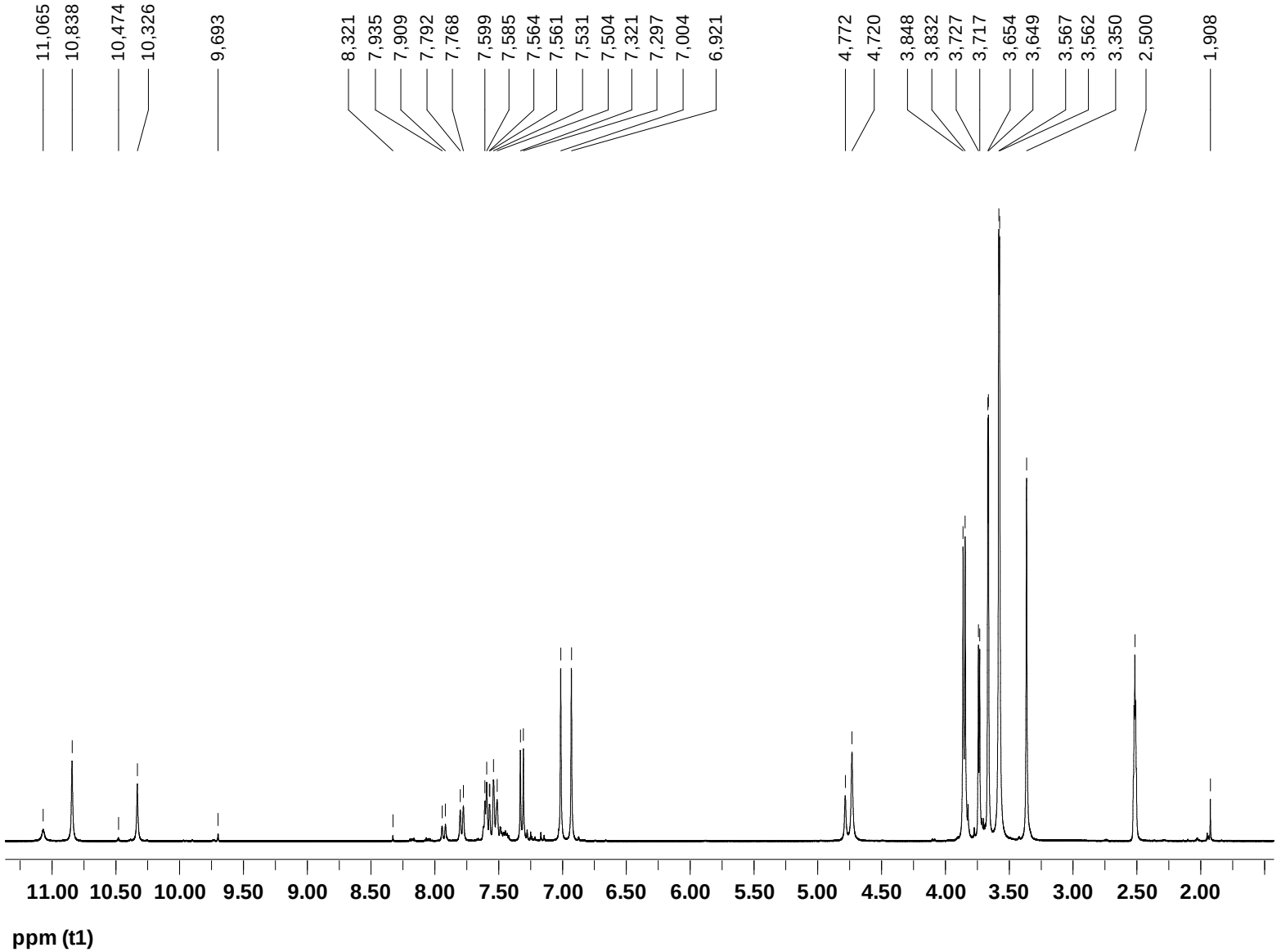
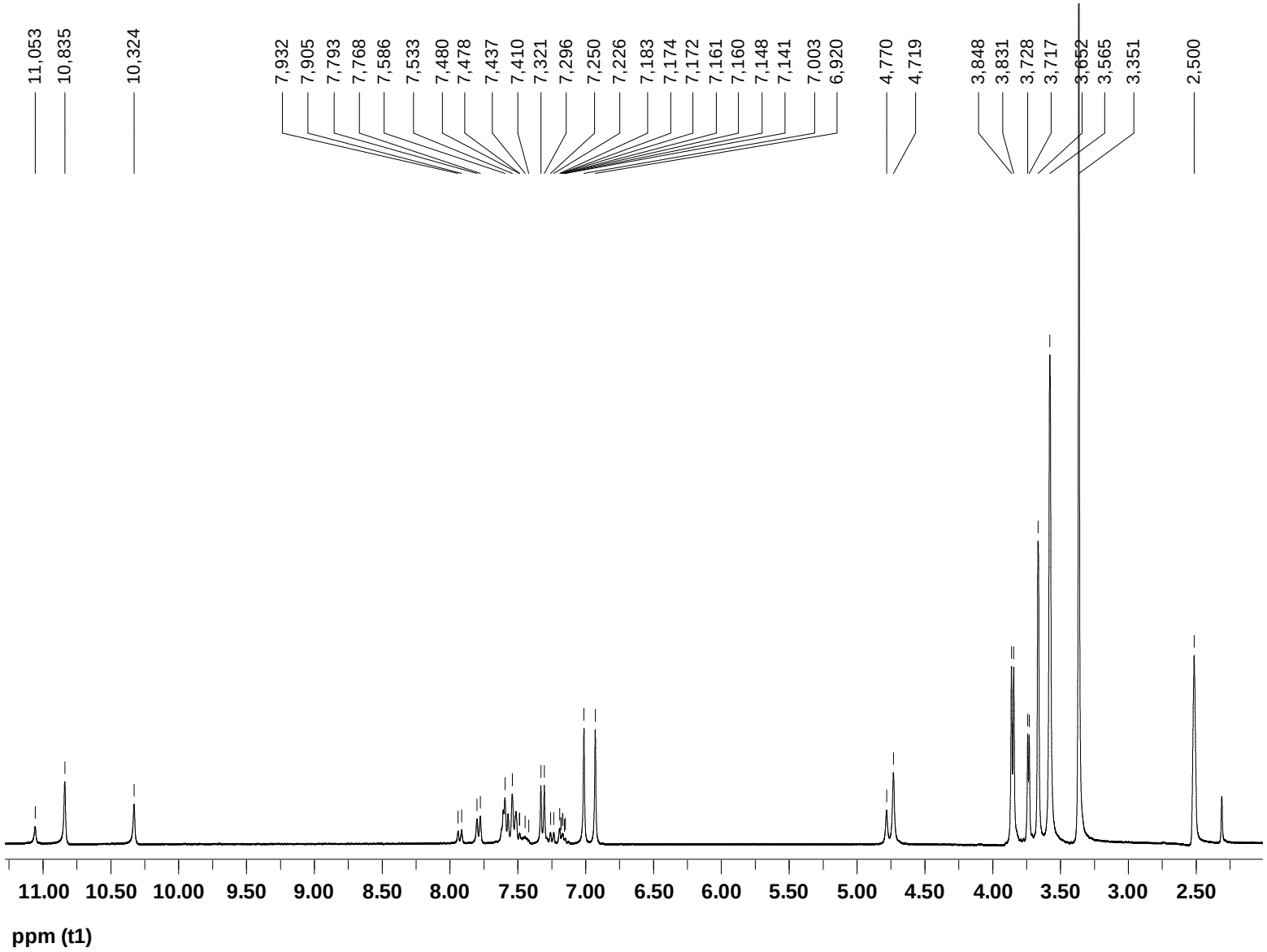


Figure S 8 ^{1}H NMR spectrum of crude condensation reaction corresponding to entry 6 in Table 3



Optimisation of the cyclisation reaction and ^{1}H NMR spectra of the isolated product 5a in each reaction

Table S 3 Reaction conditions used to prepare 10a (various isomers ratios) that were assessed as precursors of the 1,3,4-oxadiazoles

Entry	Starting material	Isolated yield of 5a
1	**10C** resulted from reaction preformed according to entry 6 in Table 3	35%
2	**10A + 10C** resulted from reaction preformed according to entry 5 in Table 3	33%
3	**10A + 10C** resulted from reaction preformed according to entry 4 in Table 3	34%

Figure S 9 ^{1}H NMR spectrum of the product 5a when optimising cyclisation reaction conditions

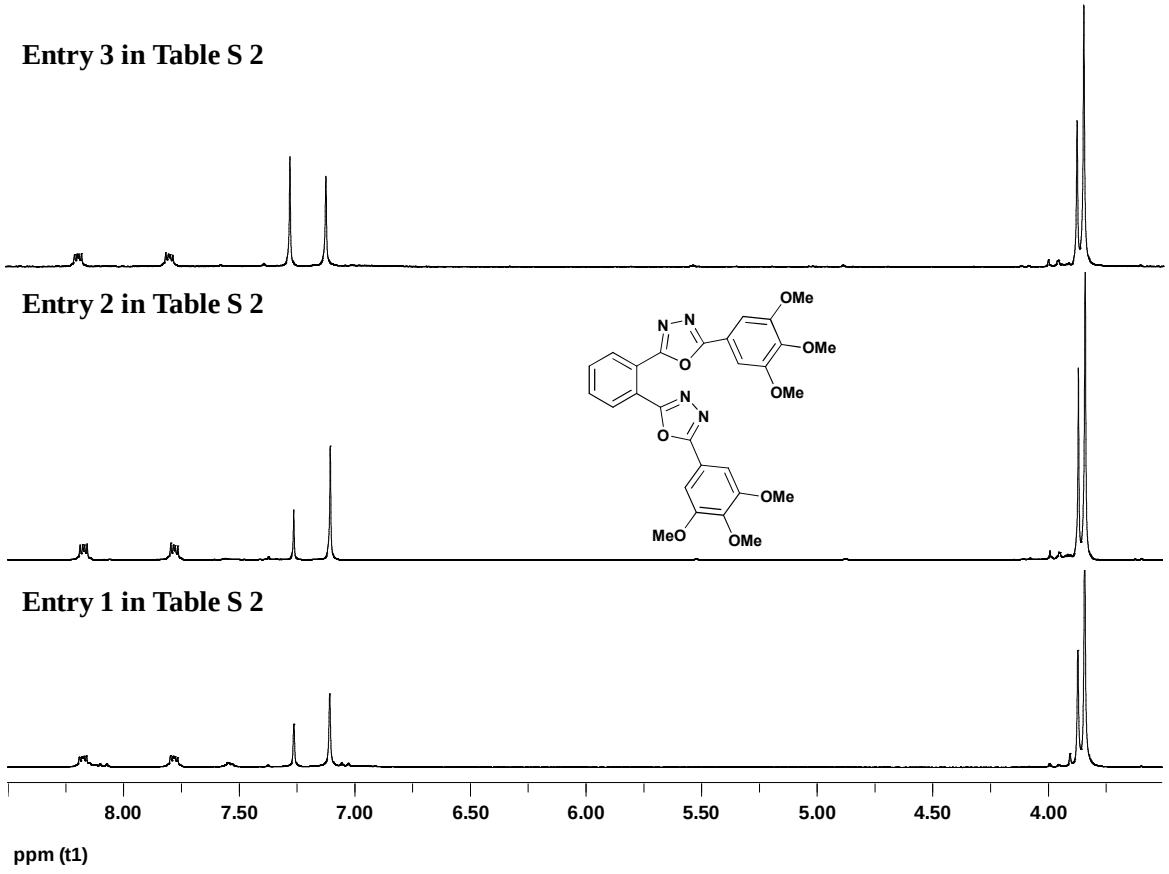
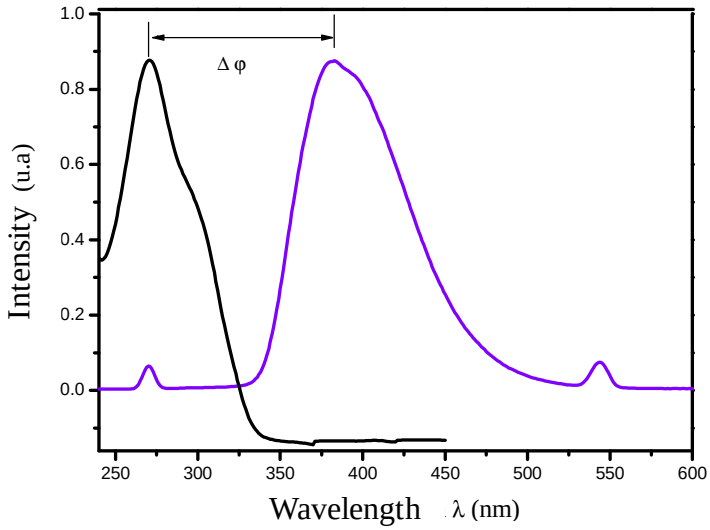
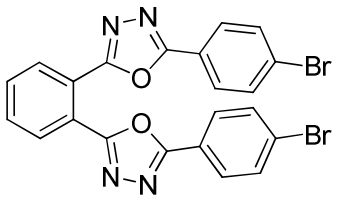


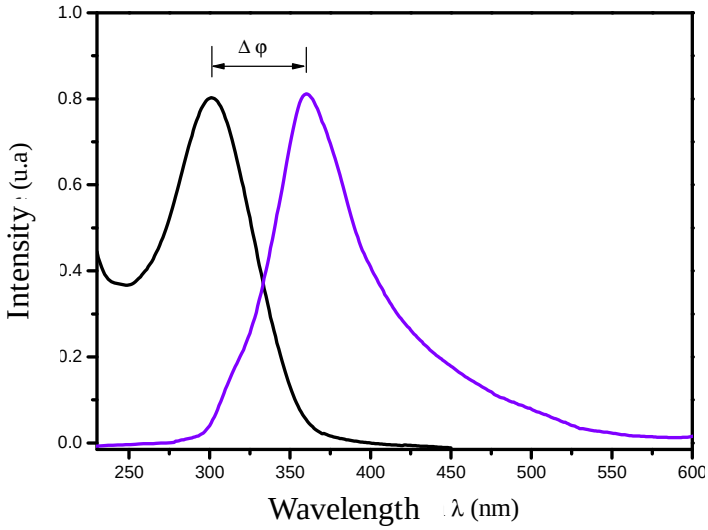
Figure S 10 UV-Vis and fluorescence spectra of compounds 5b Solvent: CH_2Cl_2, λ_{ex}=270 nm, c=8,58·10^{-5} mole/L, A=0,29, λ_A=270 nm, c=2,5·10^{-6} mole/L, λ_F=383 nm, Δφ=113 nm

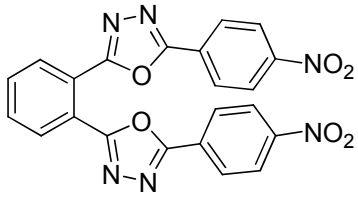




5b

Figure S 11 UV-Vis and fluorescence spectra of compounds 5g Solvent: CH_2Cl_2, λ_{ex}=250 nm, c=2,5·10^{-4} mole/L, A=0,8, λ_A=301 nm, c=2,5·10^{-6} mole/L, λ_F=360 nm, Δφ=59 nm





5g

Figure S 12 ^{1}H NMR spectrum of compound 10Ca

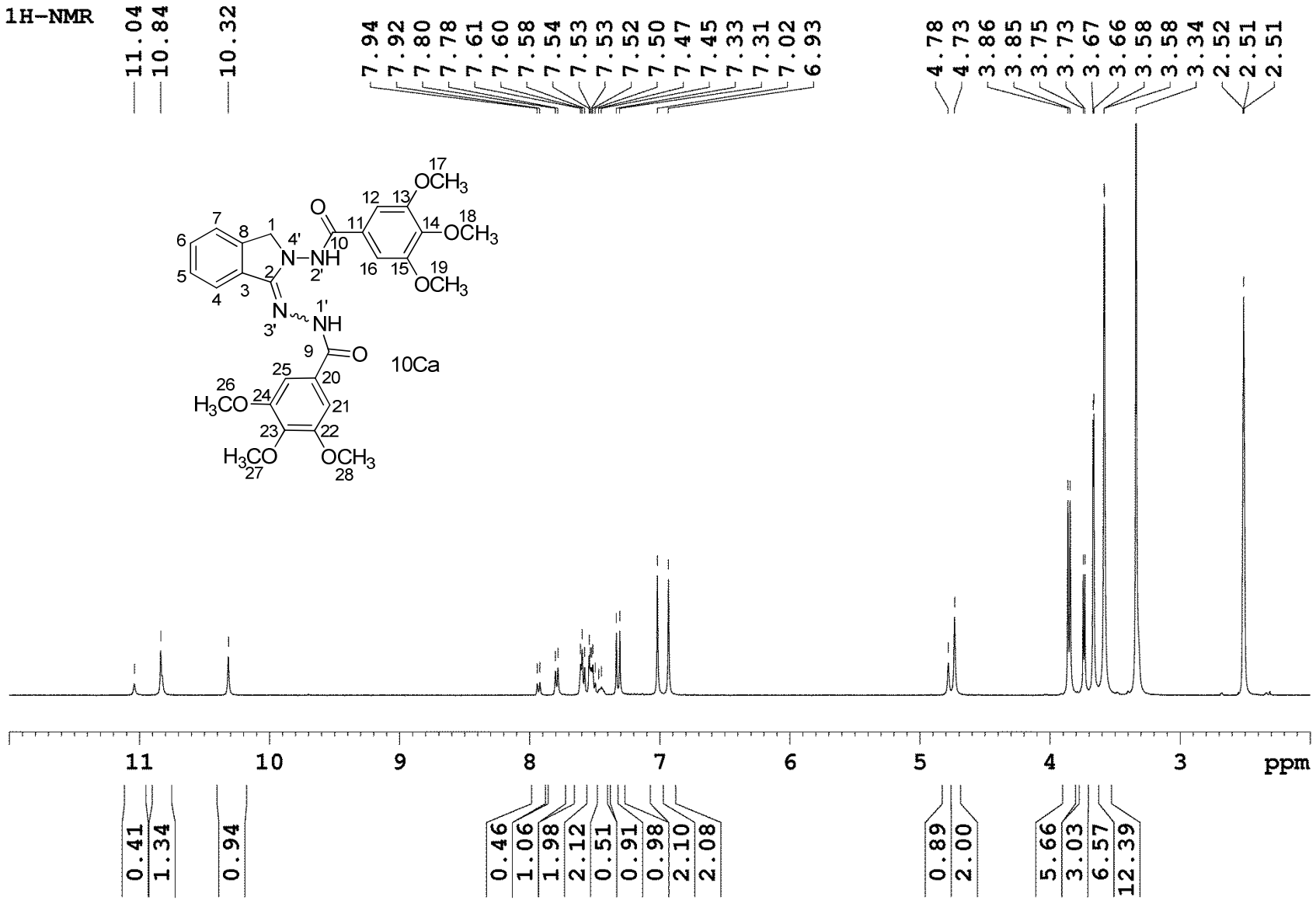


Figure S 13 ^{1}H NMR spectra of compound 10Ca at 40^{o}C, 70^{o}C, 100^{o}C, 120^{o}C and 30^{o}C after cooling down

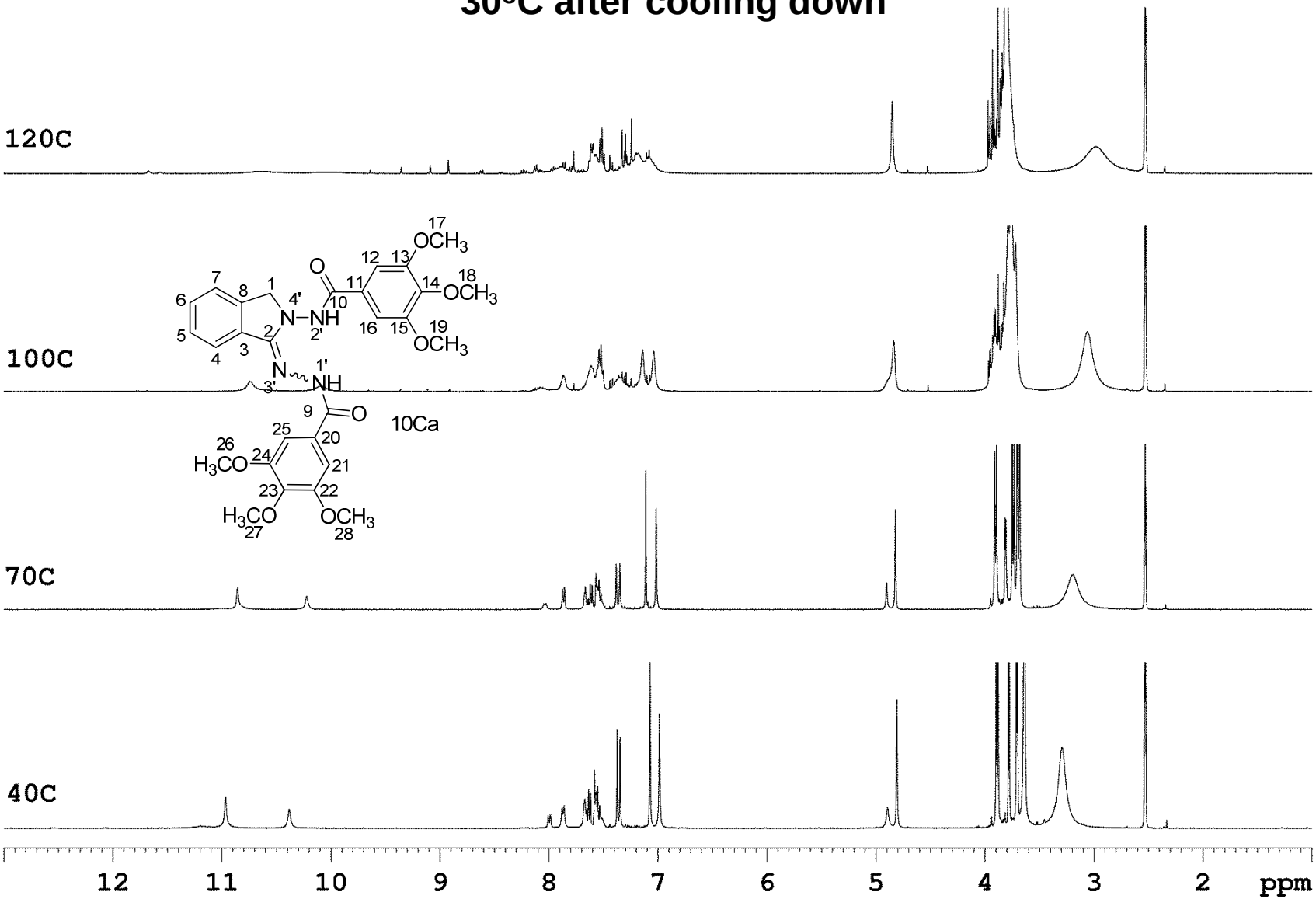


Figure S 14 ^{13}C NMR spectrum of compound 10Ca

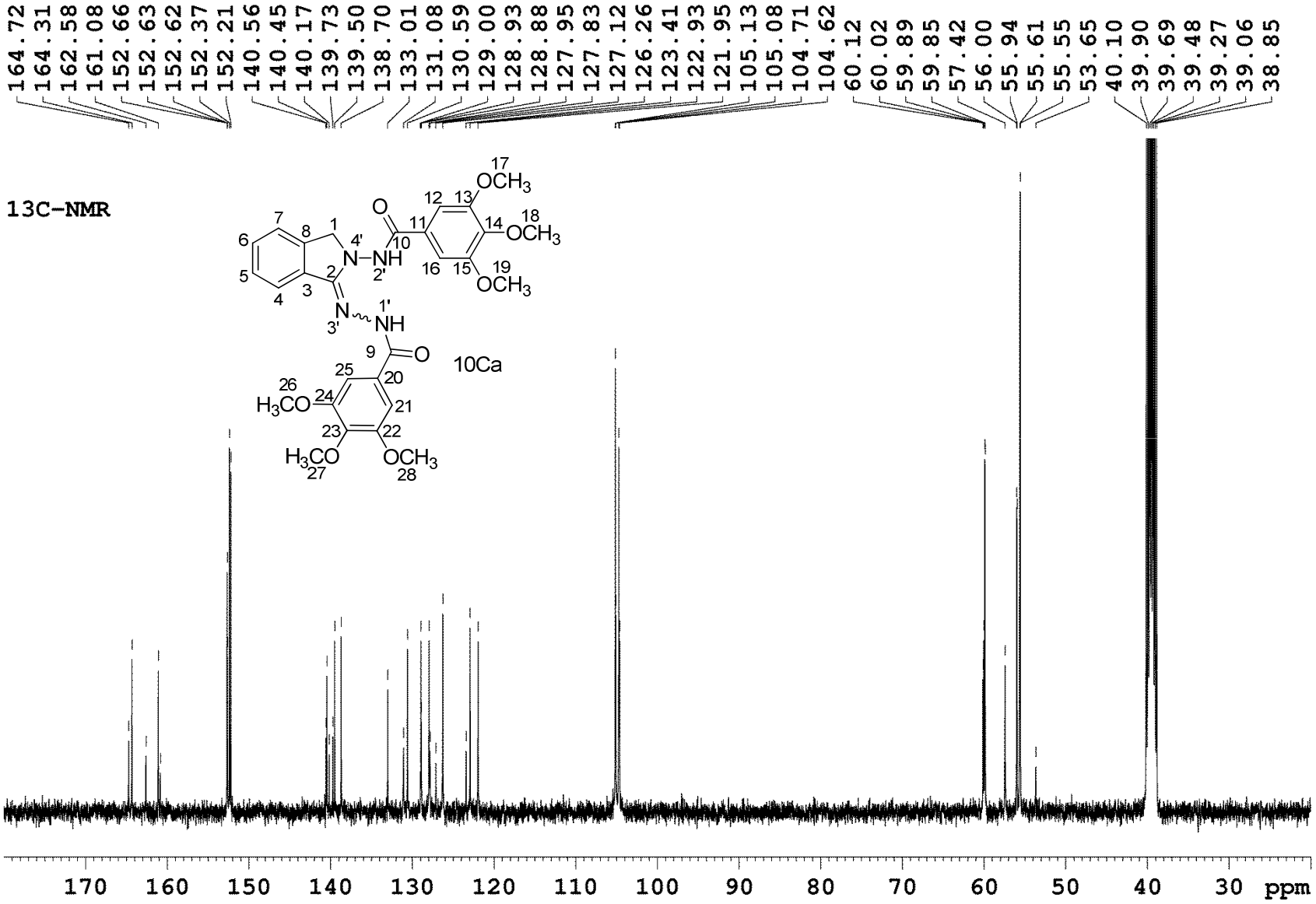


Figure S 15 H-H COSY NMR spectrum of compound 10Ca

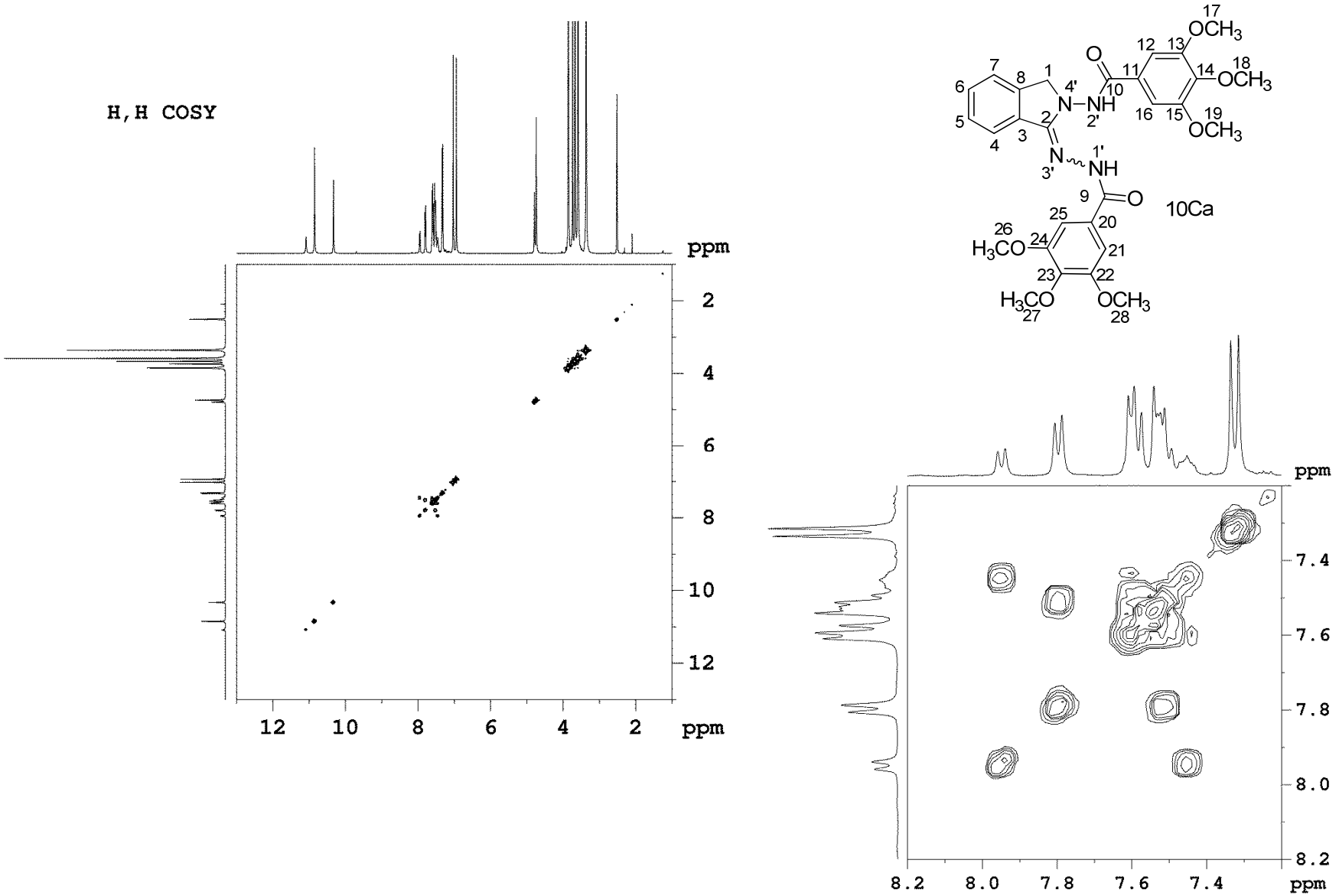


Figure S 16 H-C HMBC NMR spectrum of compound 10Ca

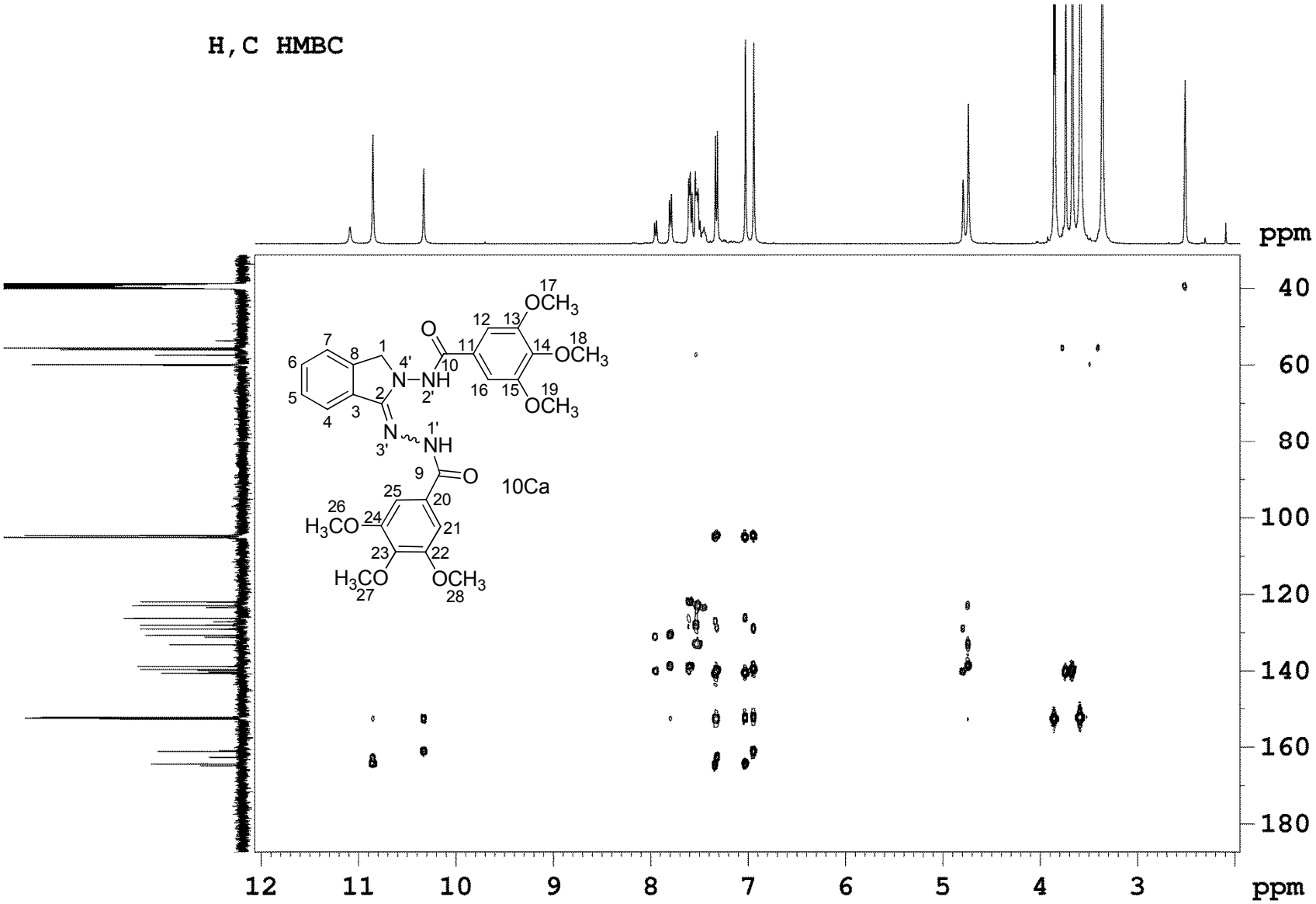


Figure S 17 H-C HSQC NMR spectrum of compound 10Ca

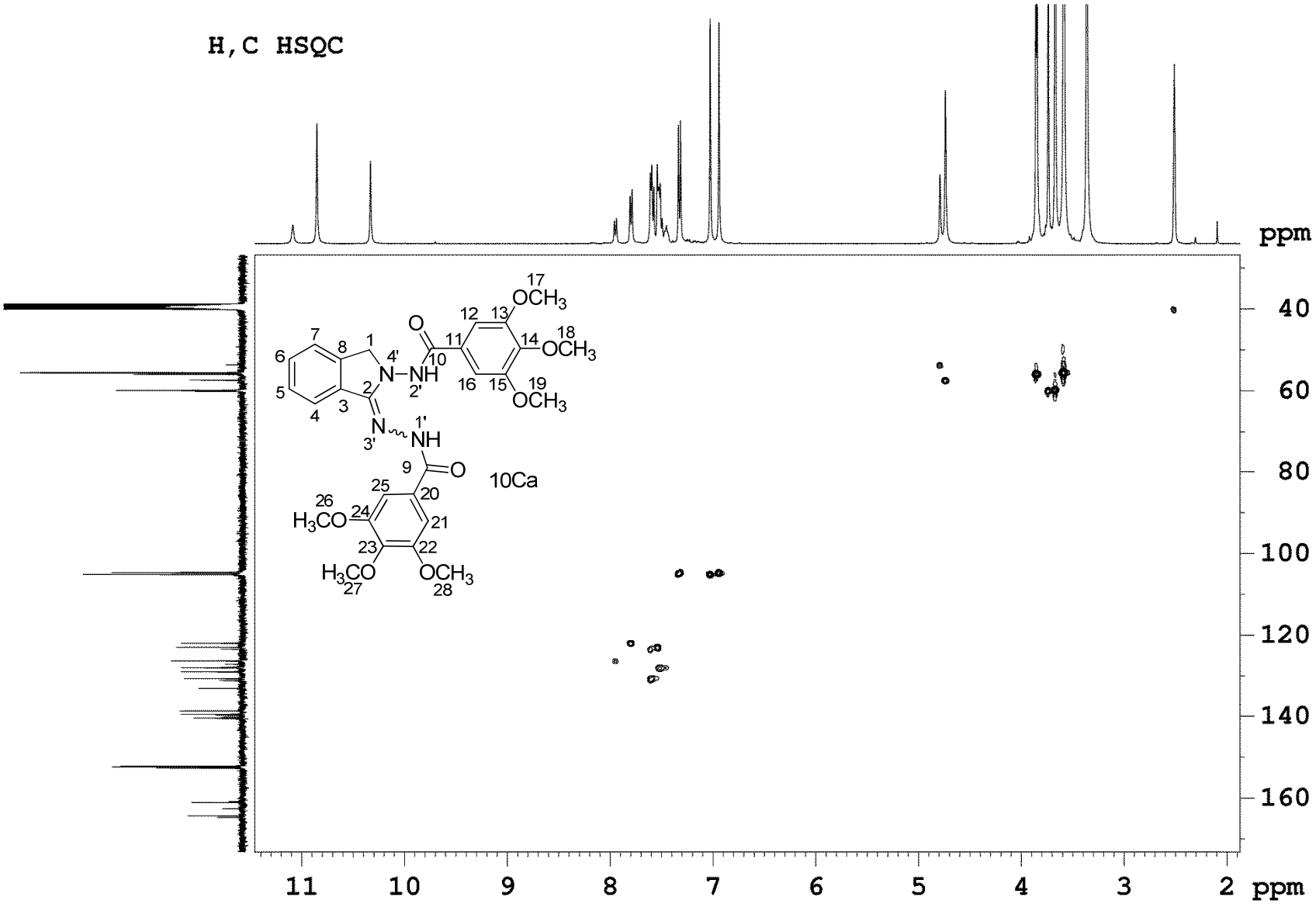
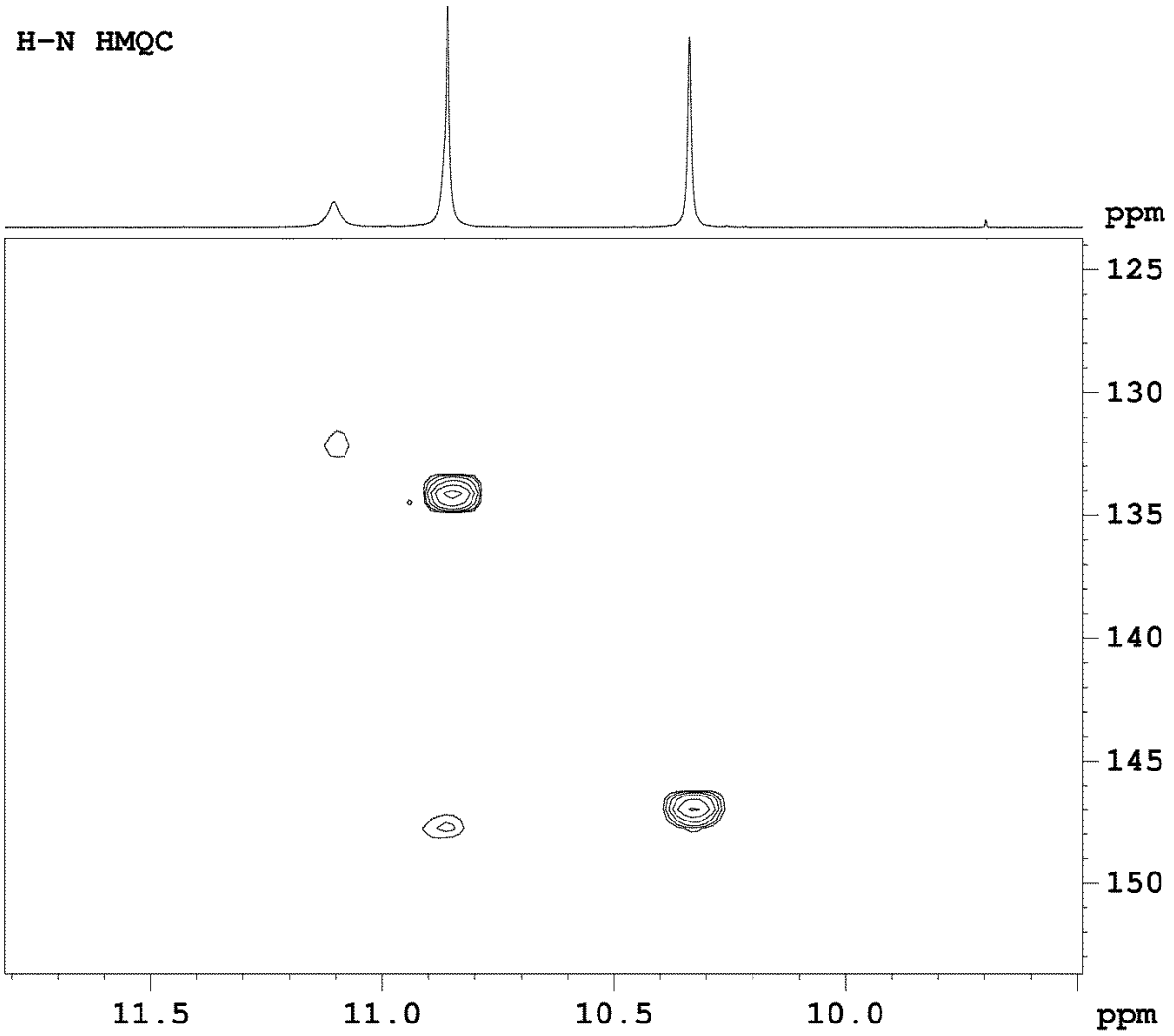


Figure S 18 H-N HMQC NMR spectrum of compound 10Ca



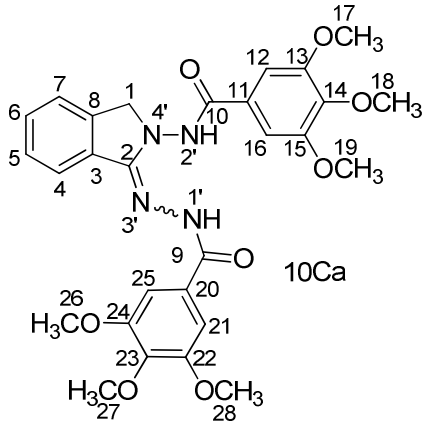
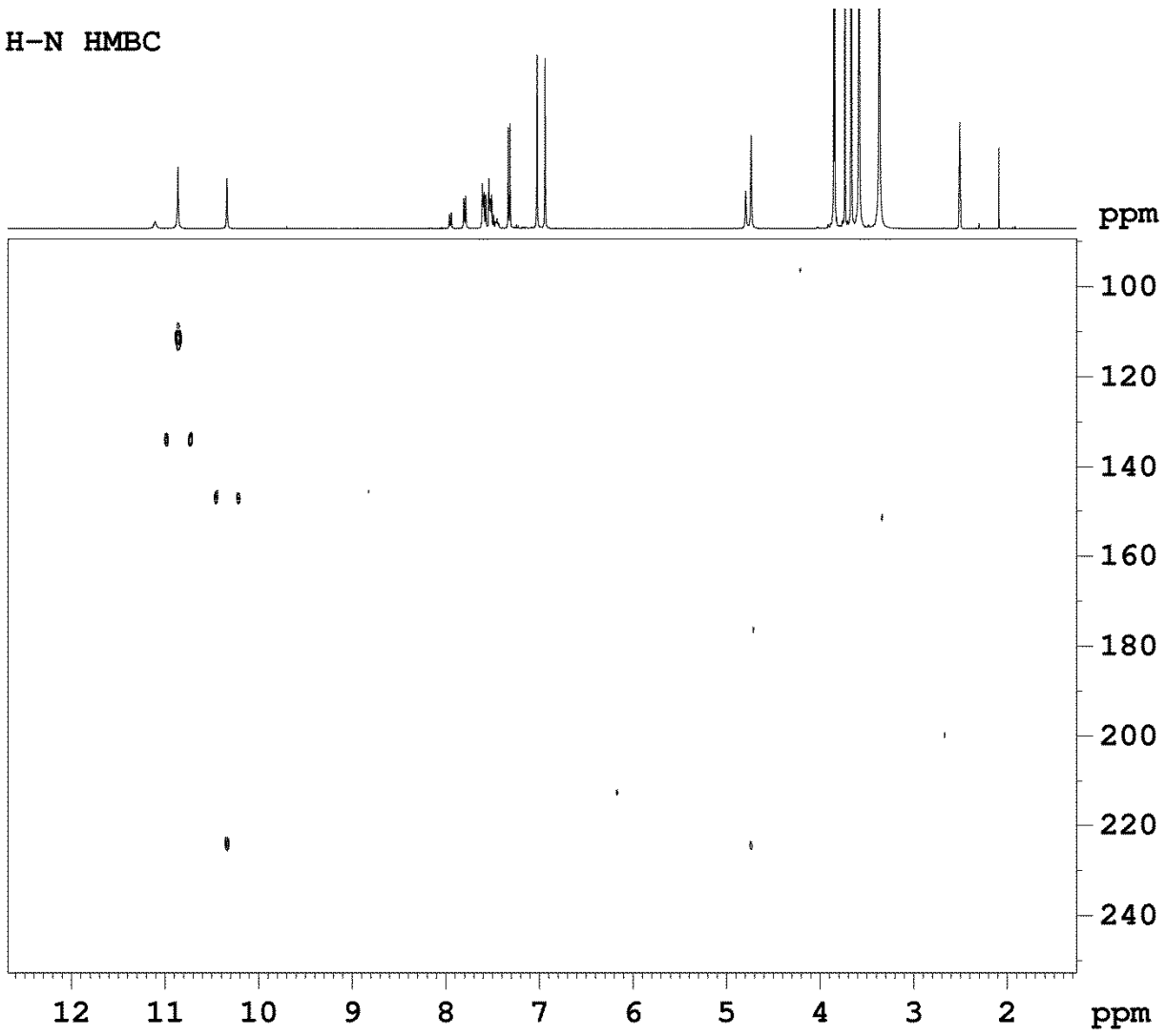


Figure S 19 H-N HMBC NMR spectrum of compound 10Ca



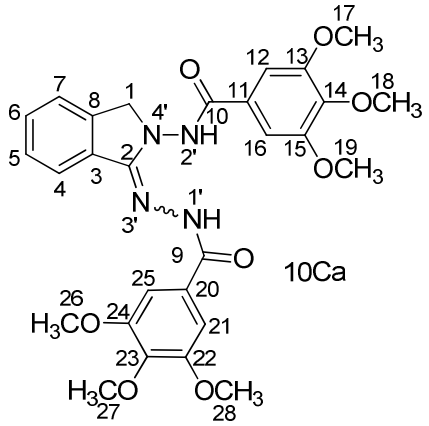


Figure S 20 APCI-HRMS spectrum of compound 10Ca

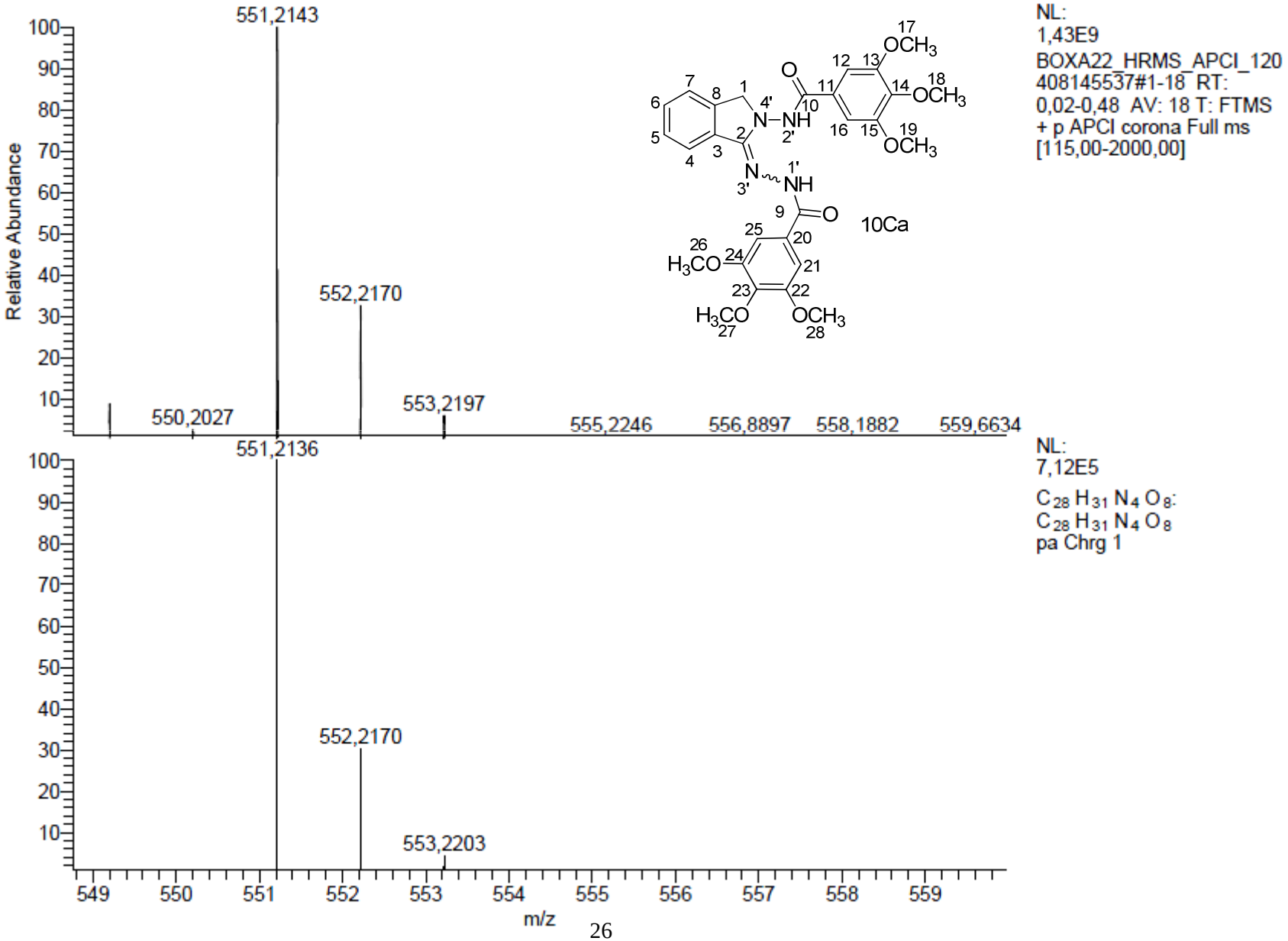


Figure S 21 ^{1}H NMR spectrum of compound 10Cb

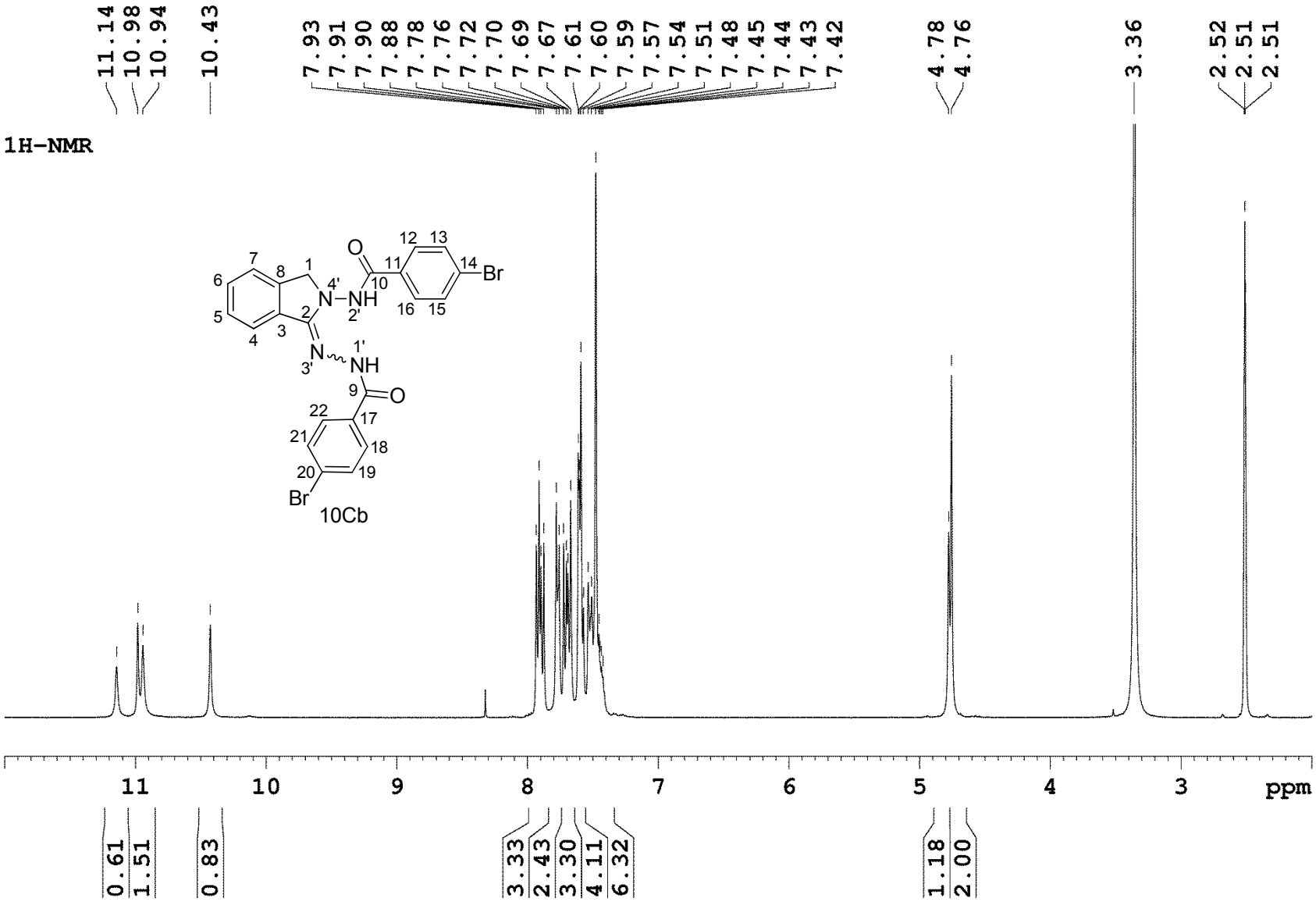


Figure S 22 ^{13}C NMR spectrum of compound 10Cb

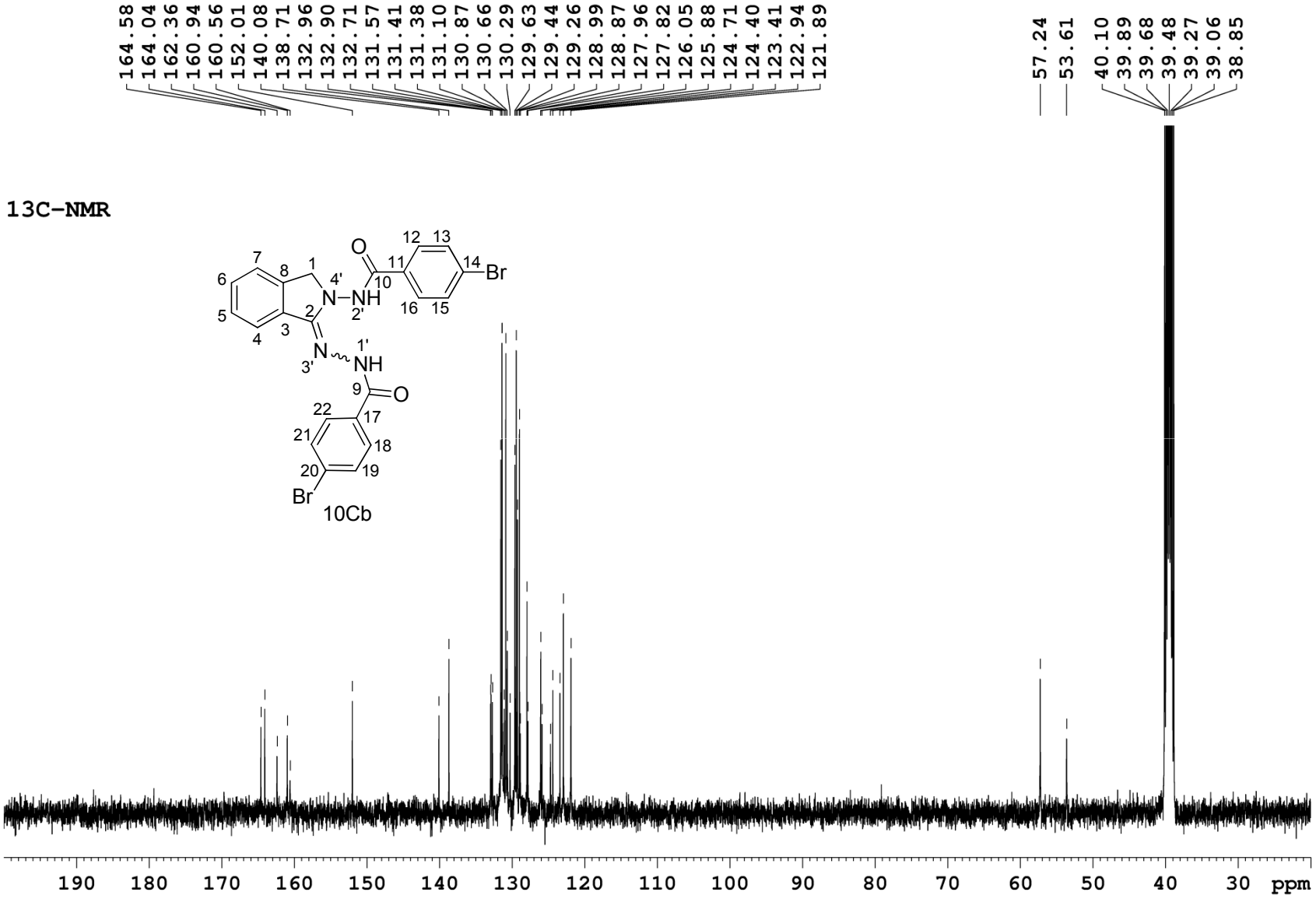
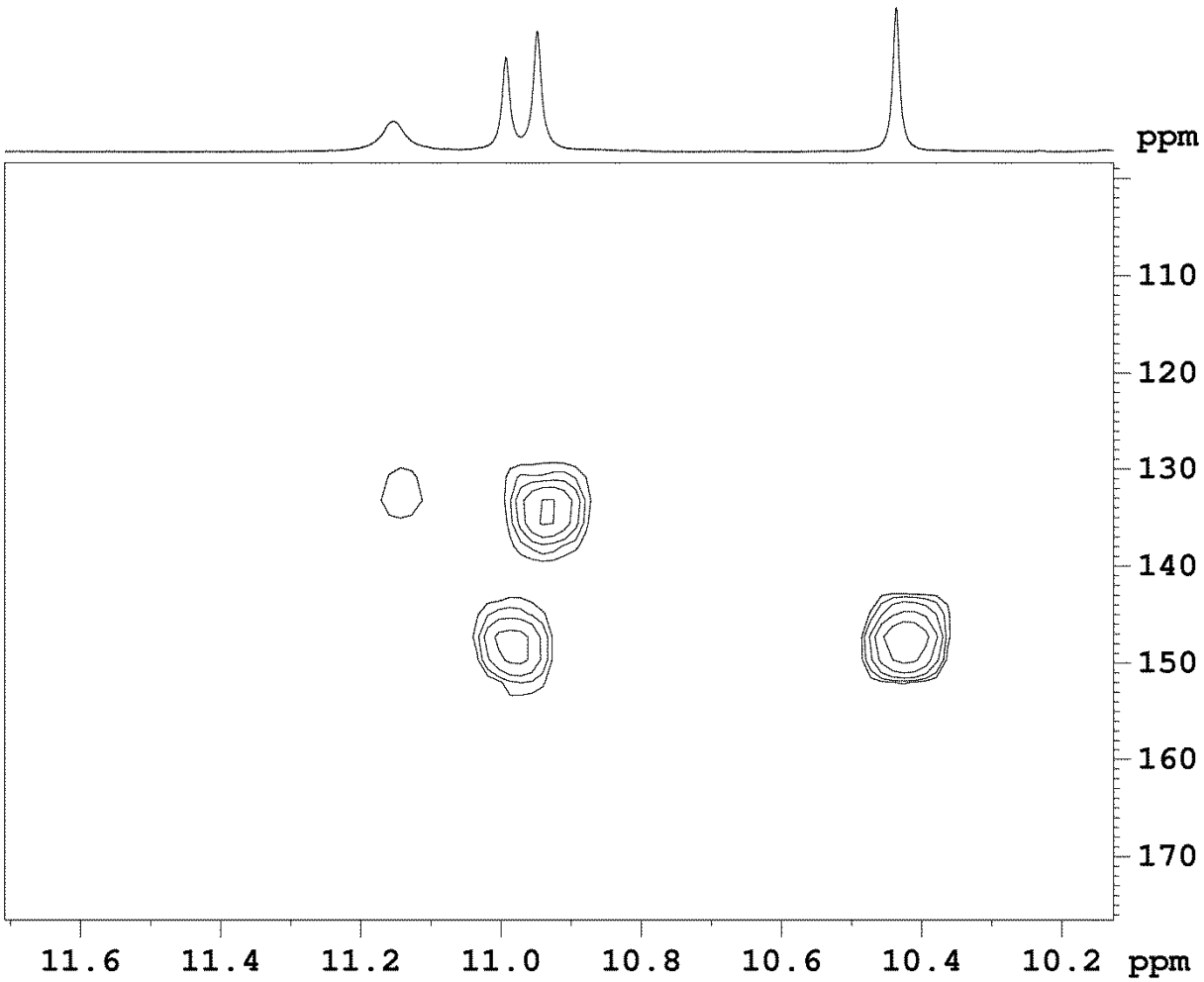
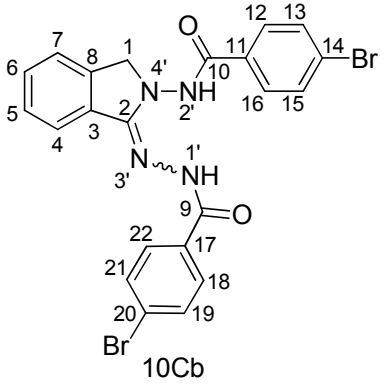


Figure S 23 H-N HMQC NMR spectrum of compound 10Cb





10Cb

Figure S 24 APCI-HRMS spectrum of compound 10Cb

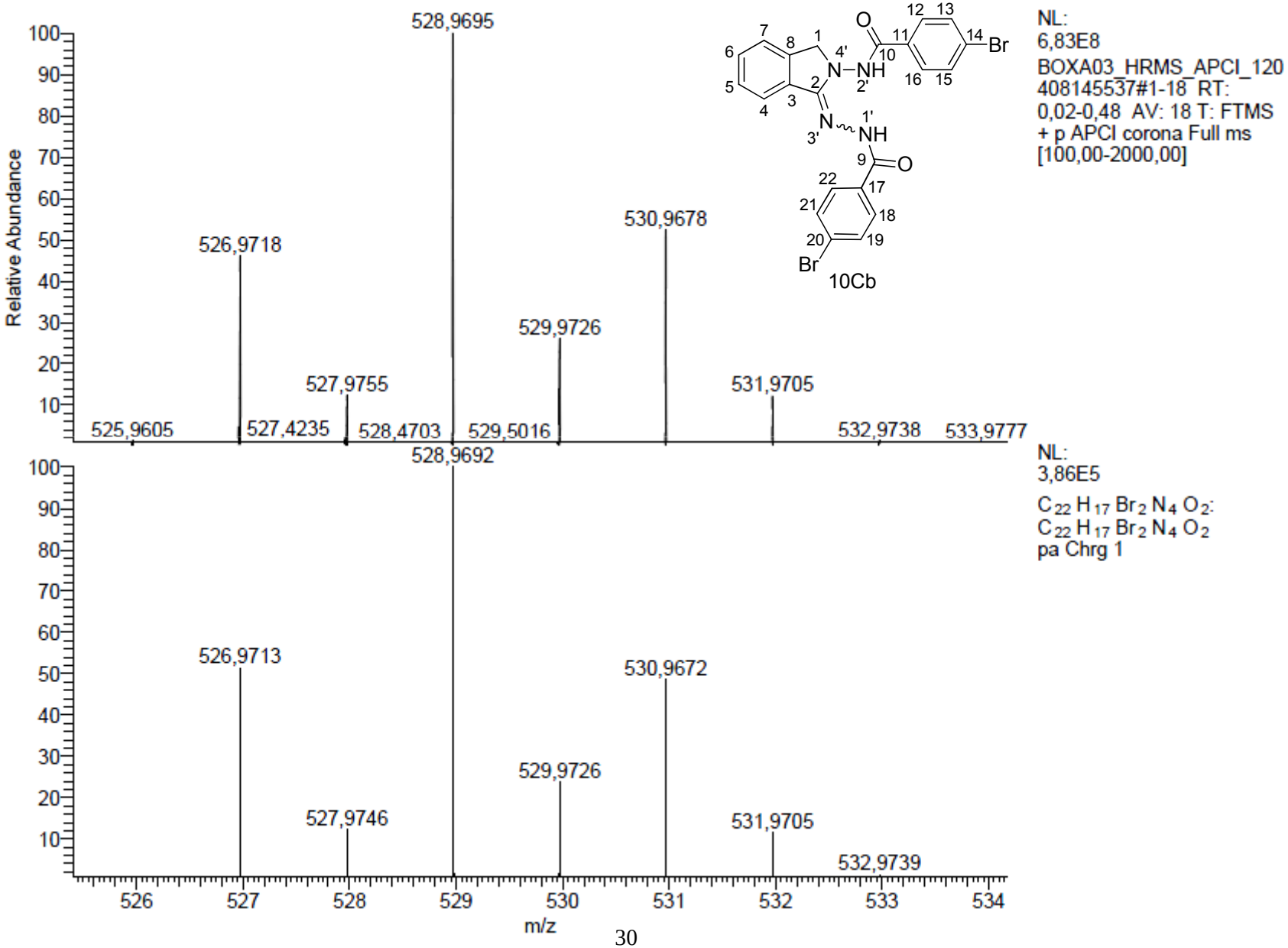


Figure S 25 ^{1}H NMR spectrum of compound 10Cc

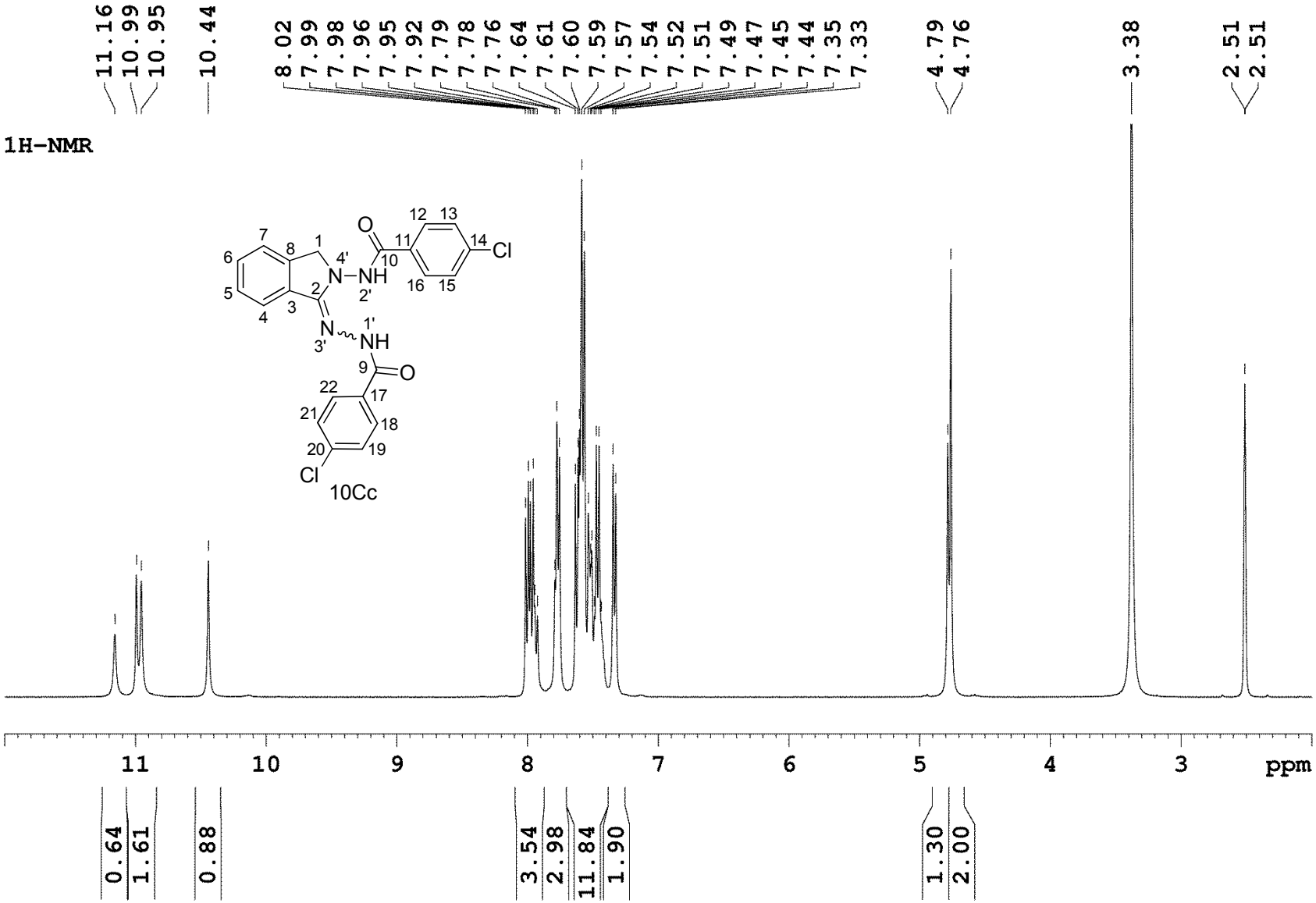


Figure S 26 ^{13}C NMR spectrum of compound 10Cc

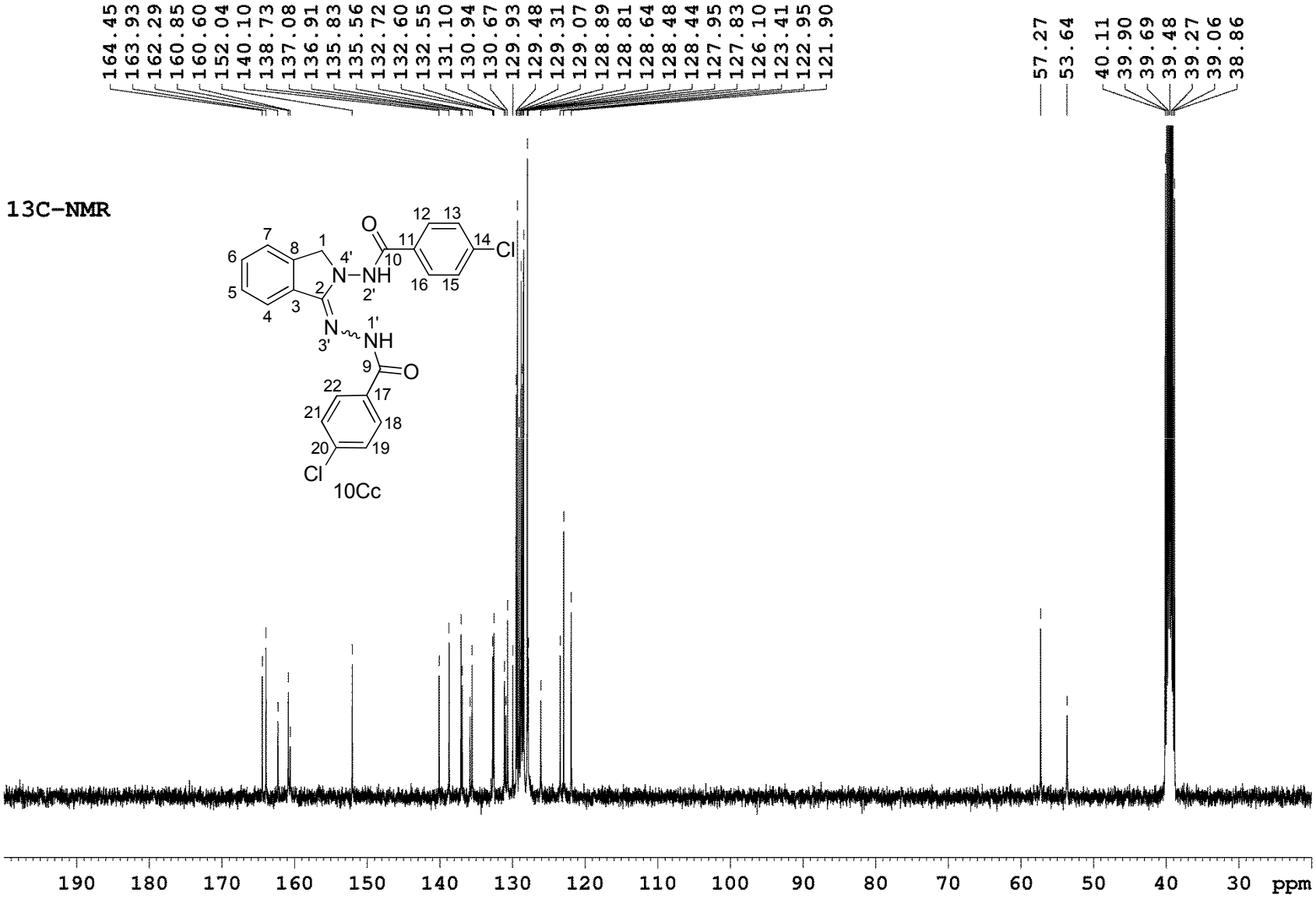
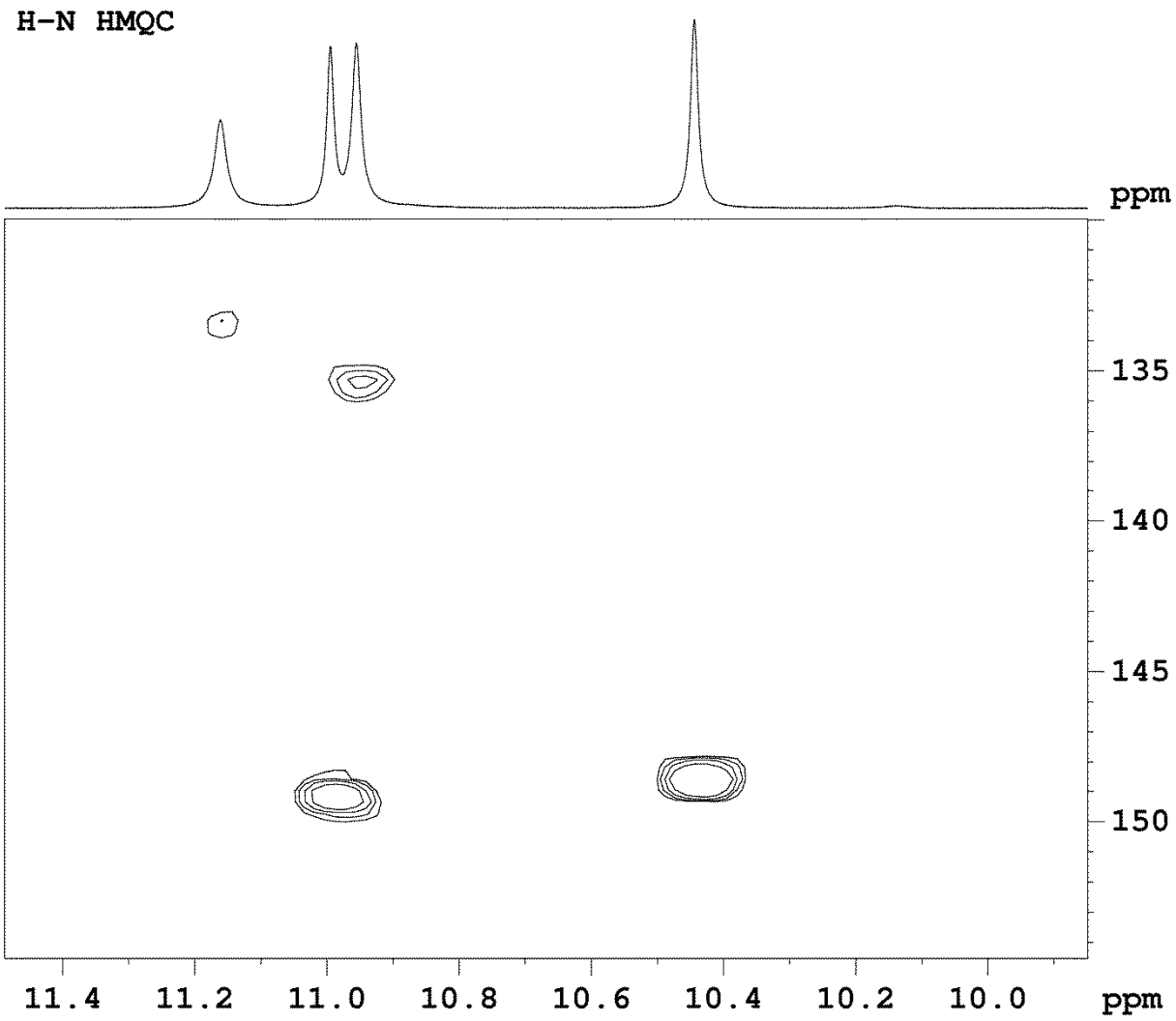
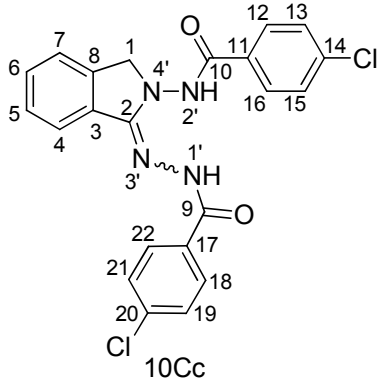


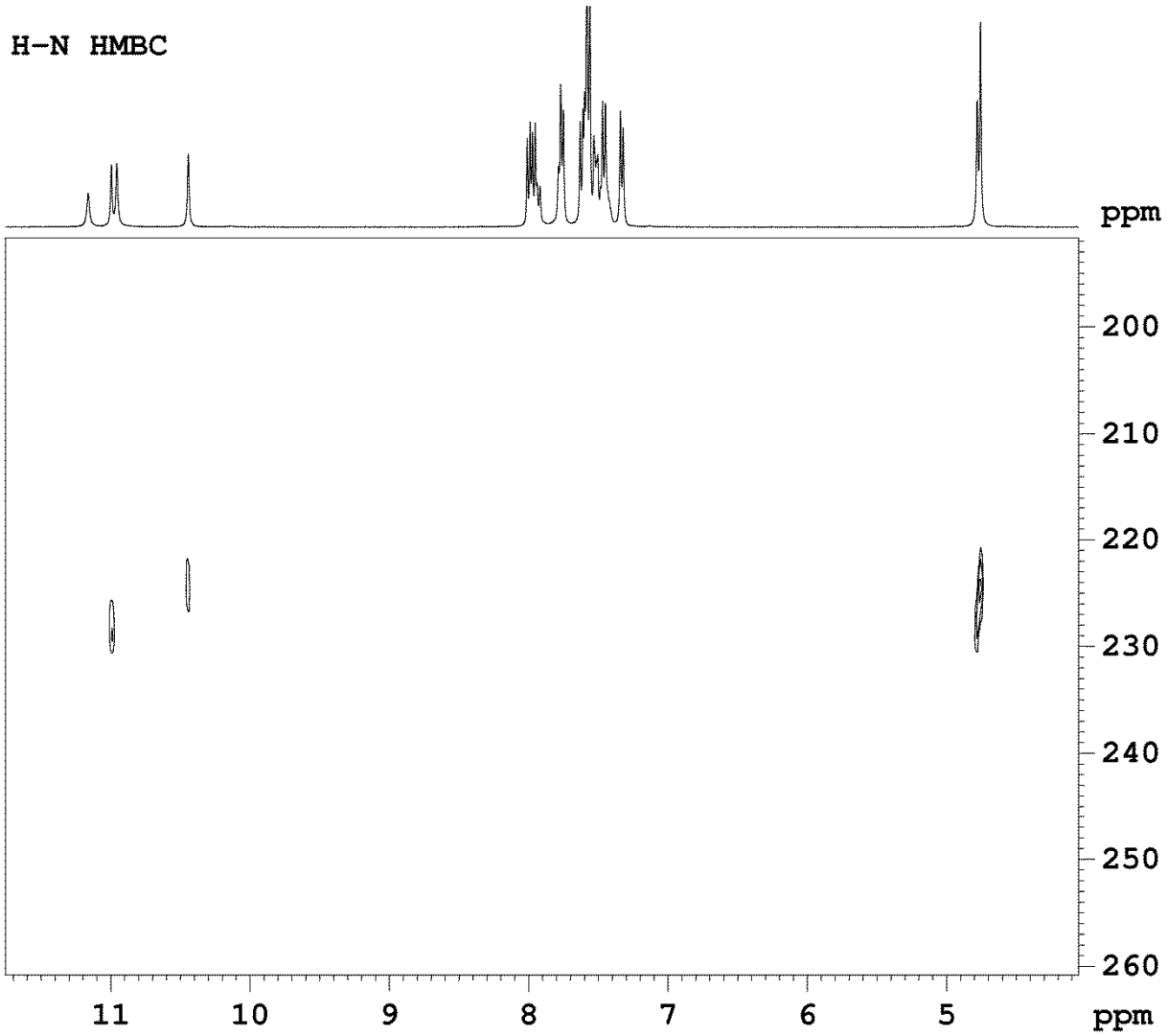
Figure S 27 H-N HMQC NMR spectrum of compound 10Cc

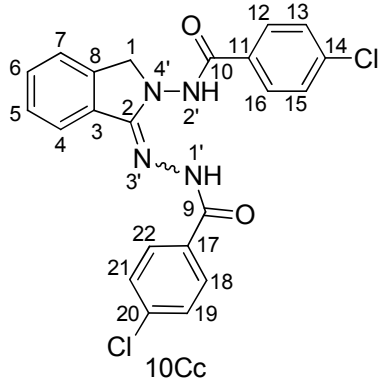




10Cc

Figure S 28 H-N HMBC NMR spectrum of compound 10Cc





10Cc

Figure S 29 APCI-HRMS spectrum of compound 10Cc

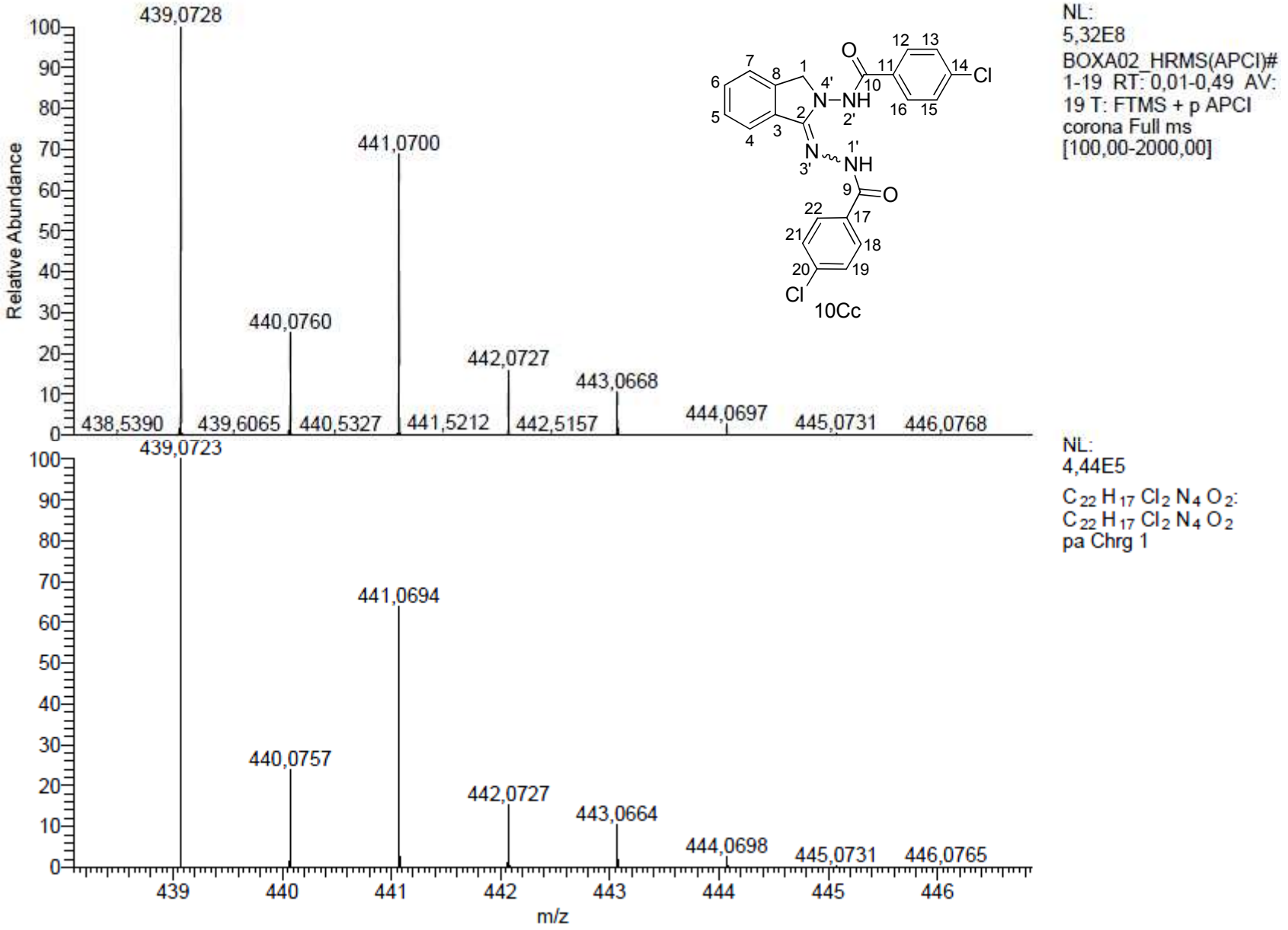


Figure S 30 ^{1}H NMR spectrum of compound 10Cd

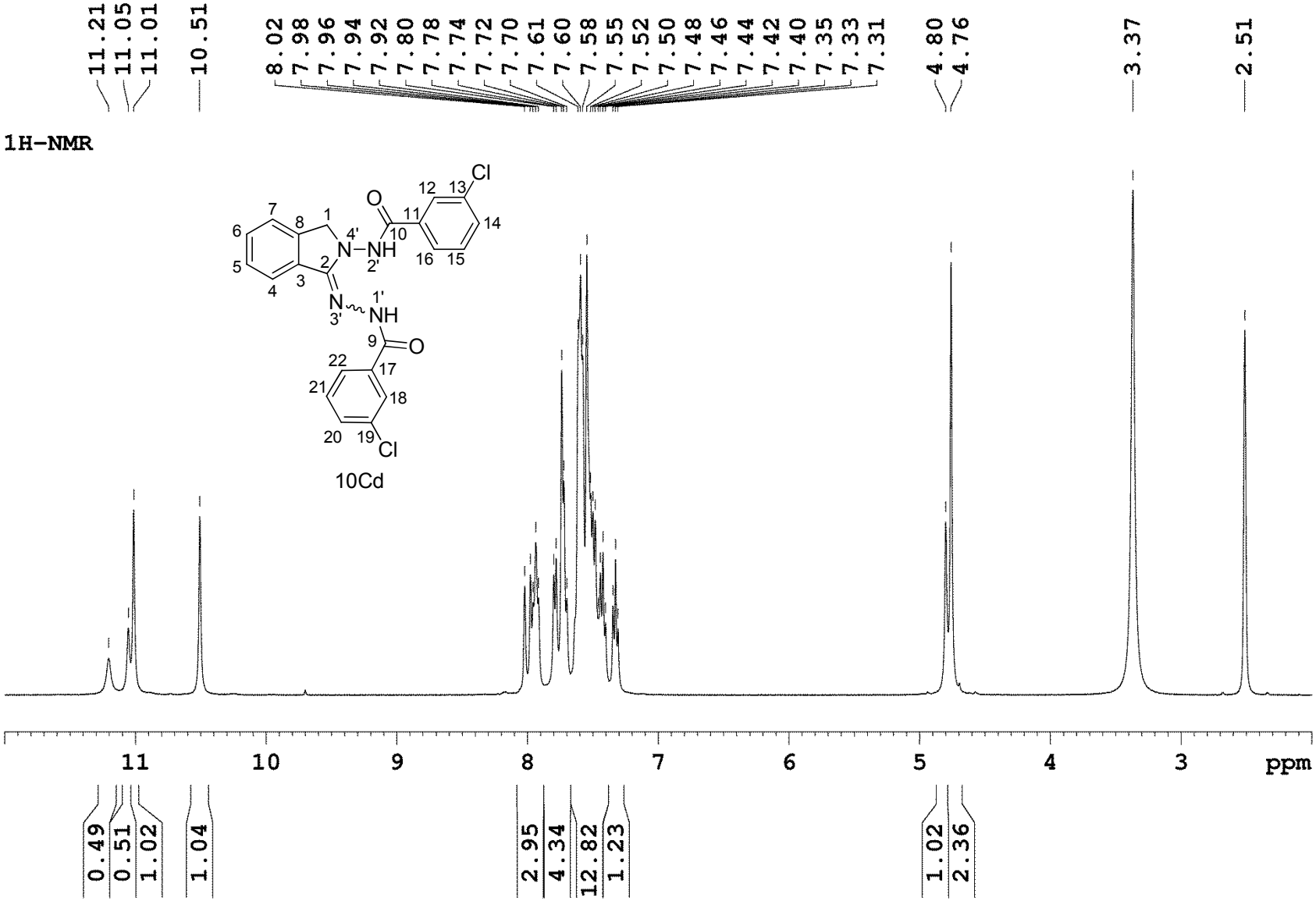


Figure S 31 ^{13}C NMR spectrum of compound 10Cd

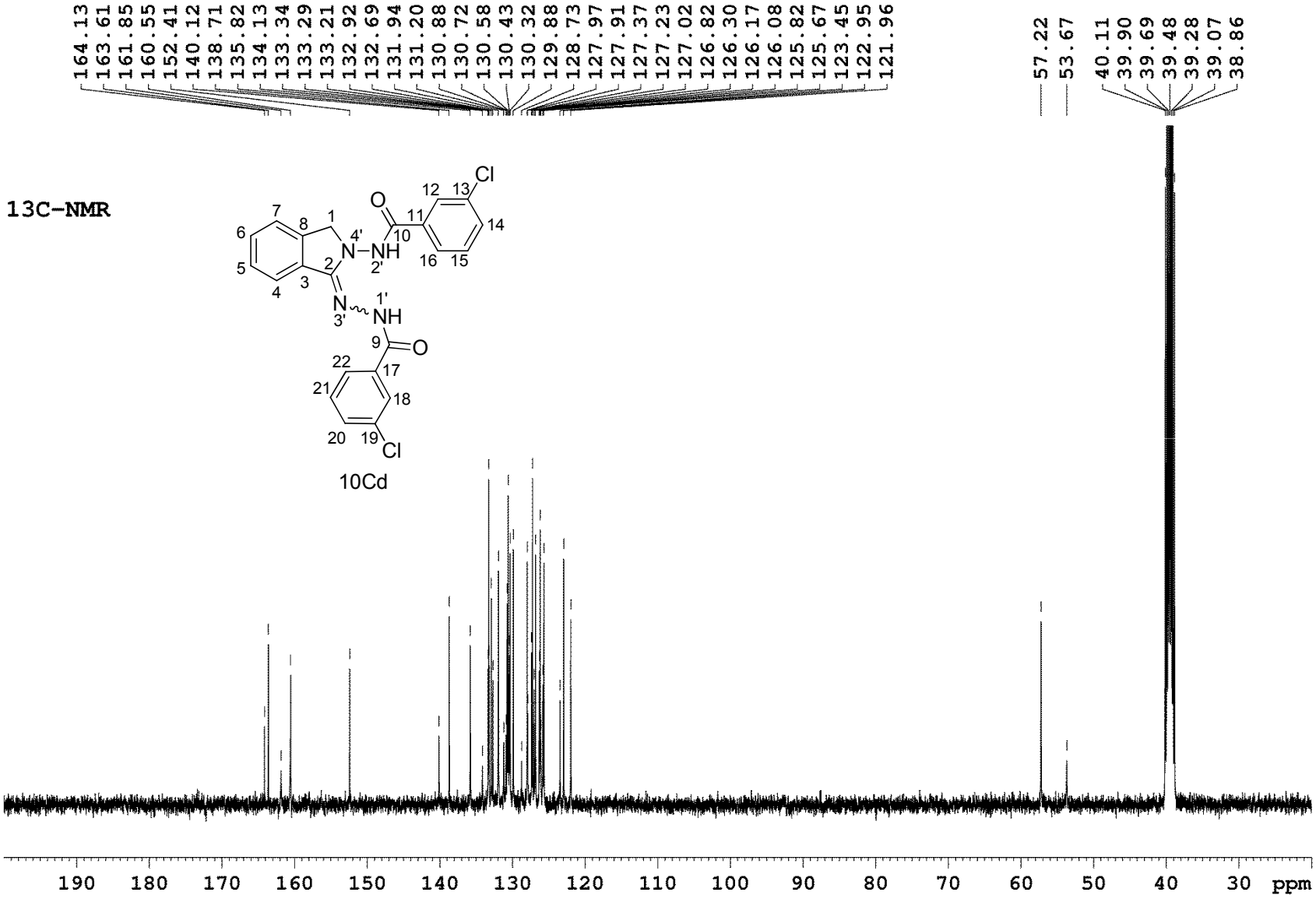
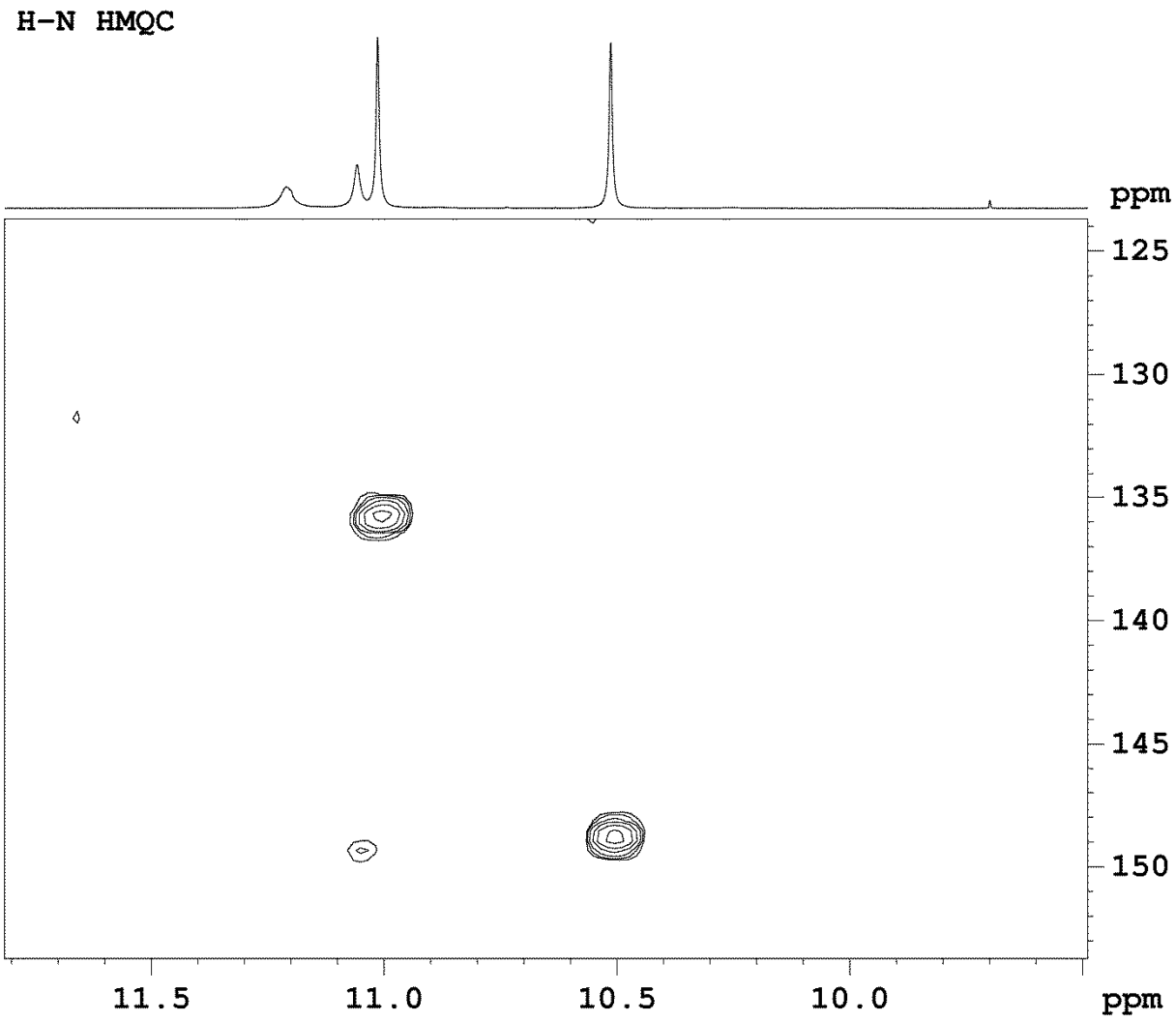
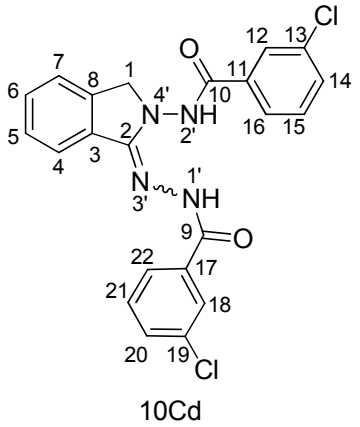


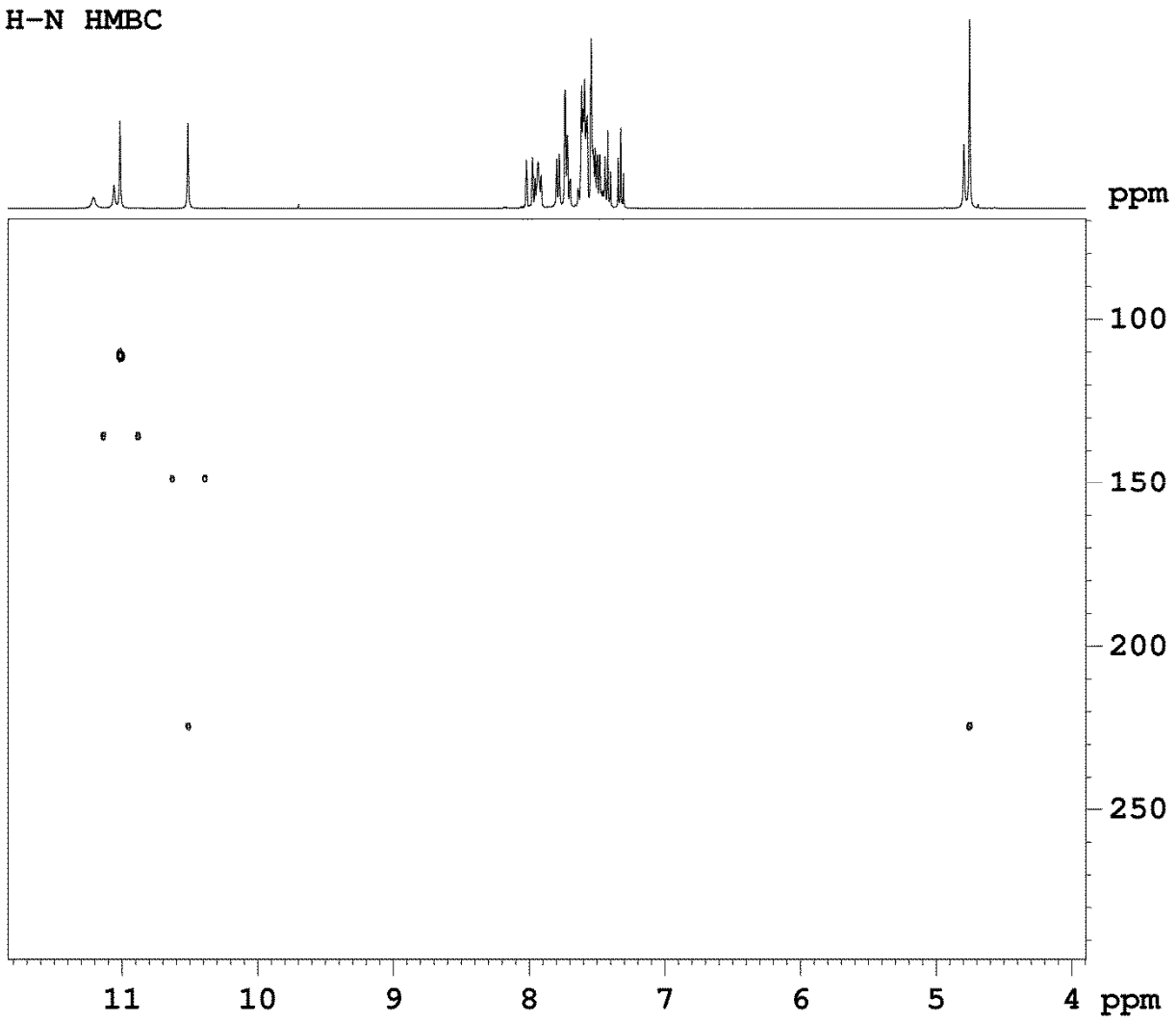
Figure S 32 H-N HMQC NMR spectrum of compound 10Cd

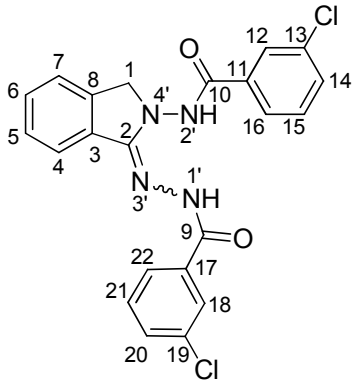




10Cd

Figure S 33 H-N HMBC NMR spectrum of compound 10Cd





10Cd

Figure S 34 APCI-HRMS spectrum of compound 10Cd

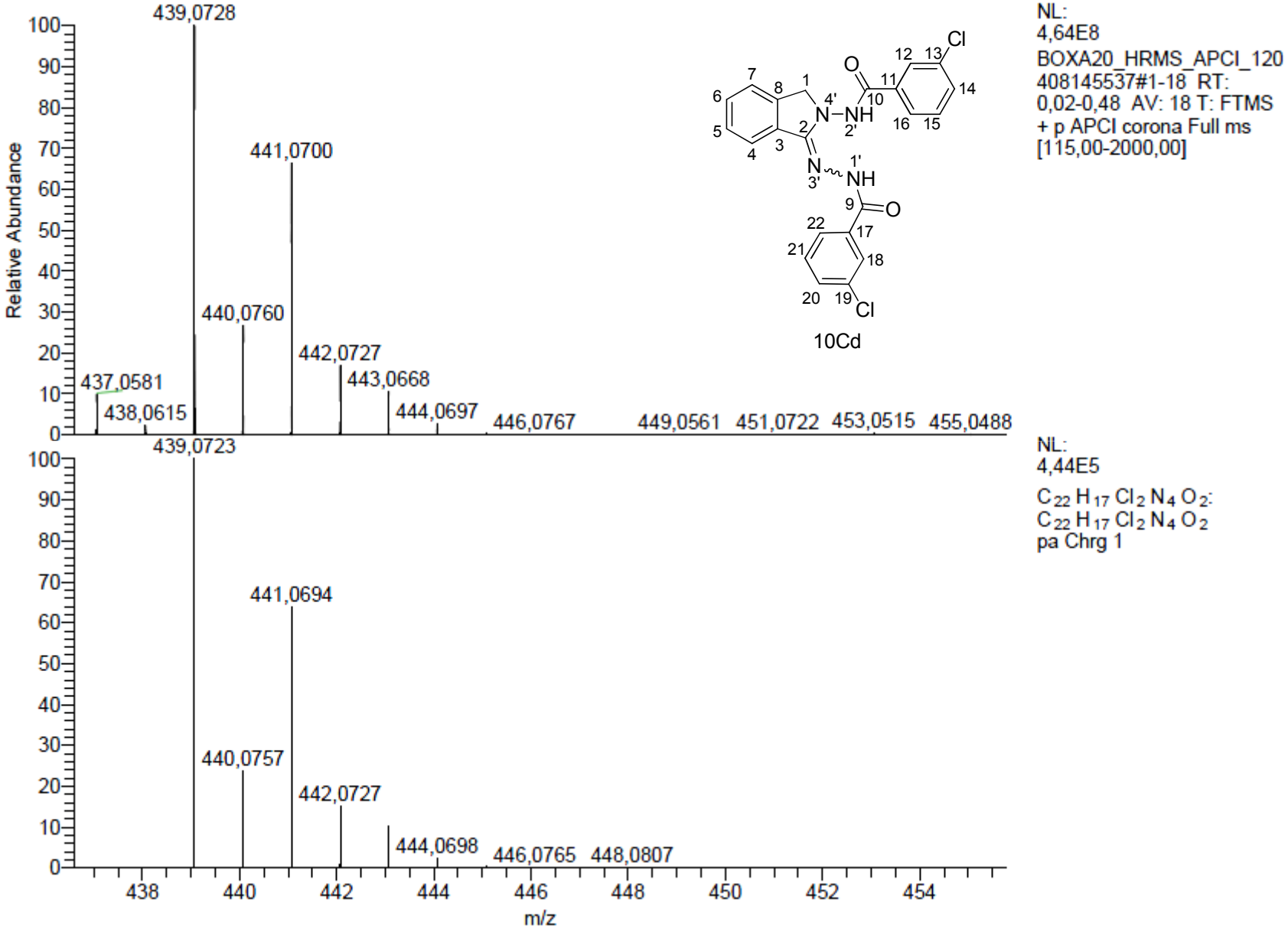


Figure S 35 ^{1}H NMR spectrum of compound 10Ce

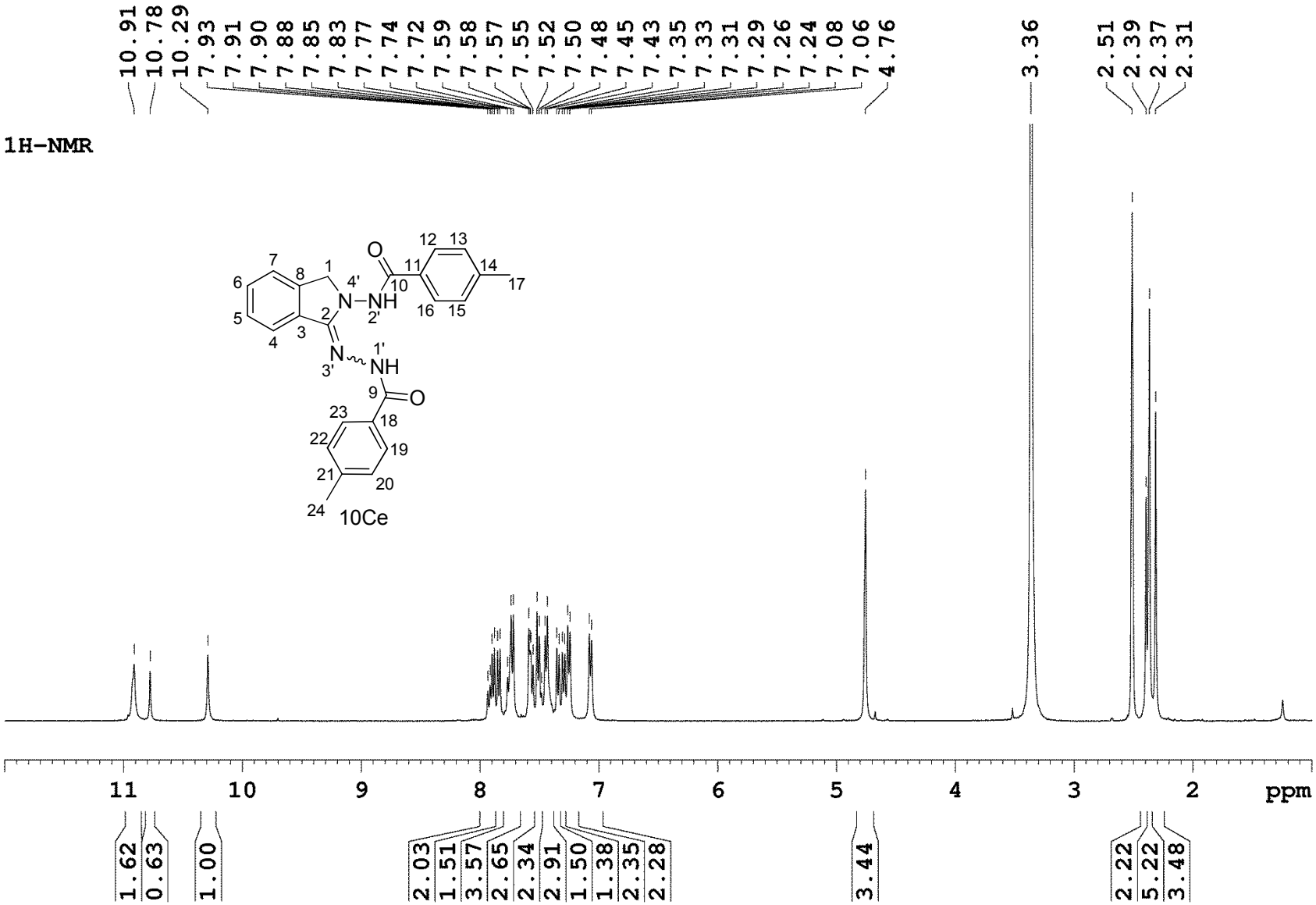


Figure S 36 ^{13}C NMR spectrum of compound 10Ce

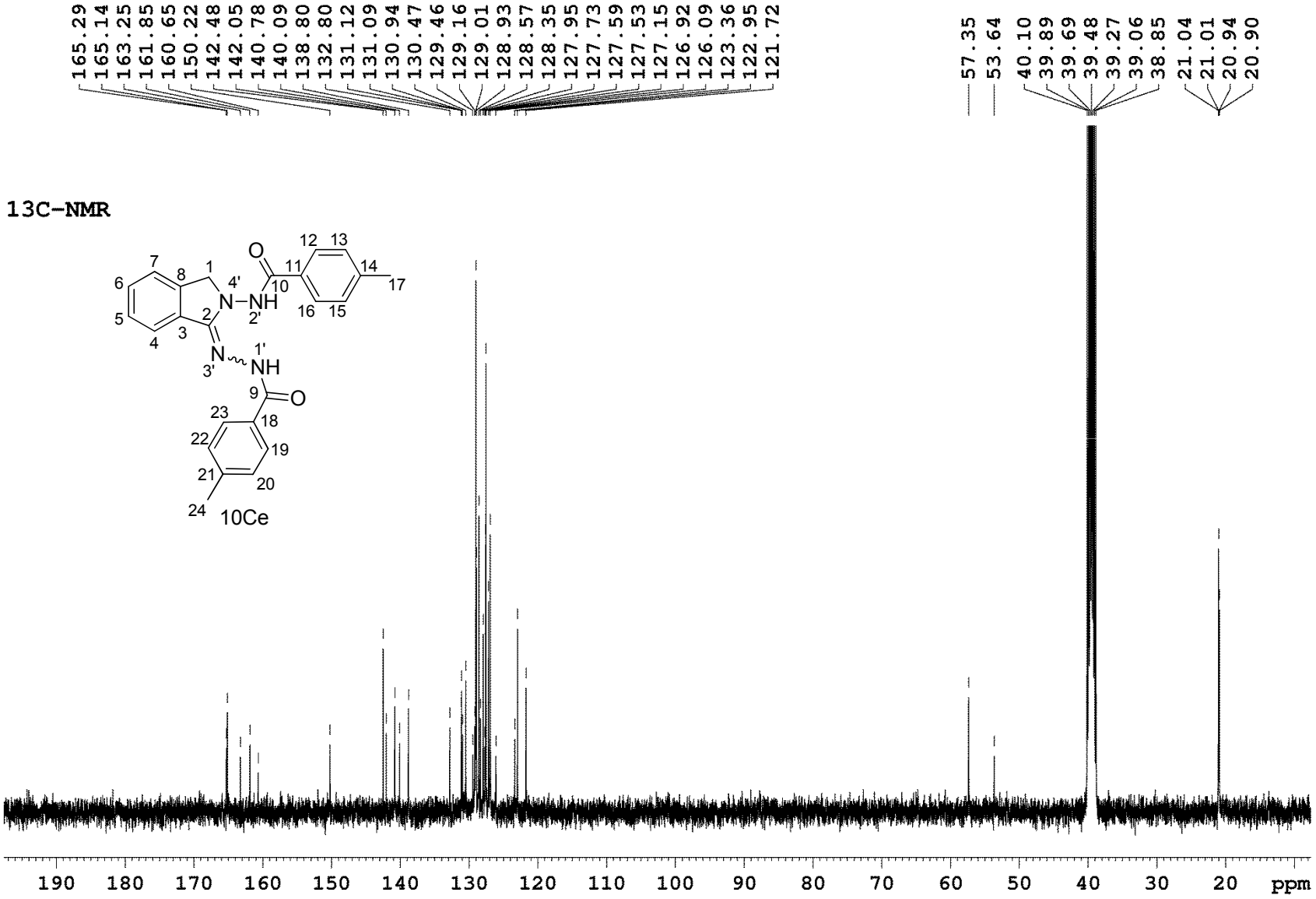
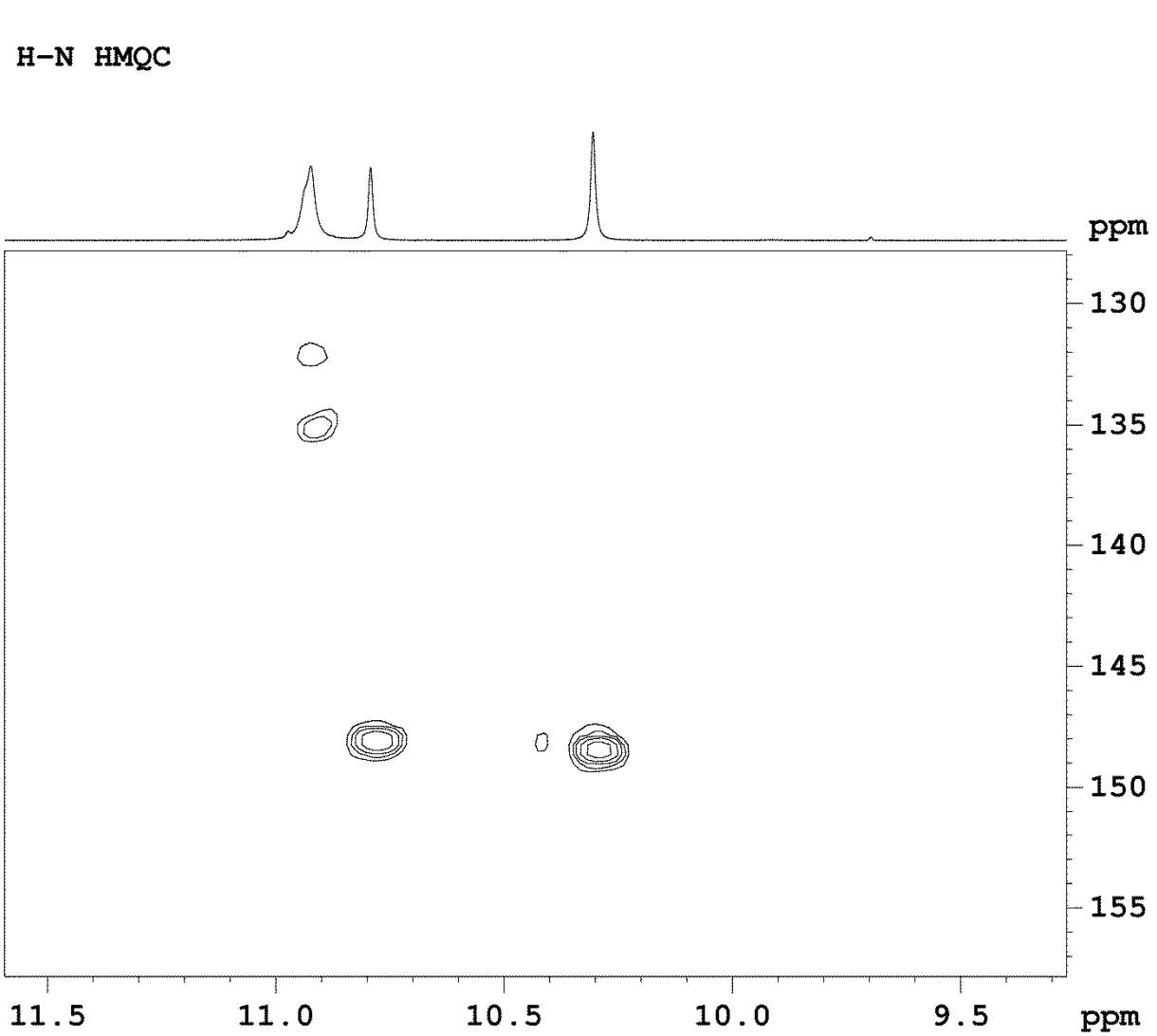


Figure S 37 H-N HMQC NMR spectrum of compound 10Ce



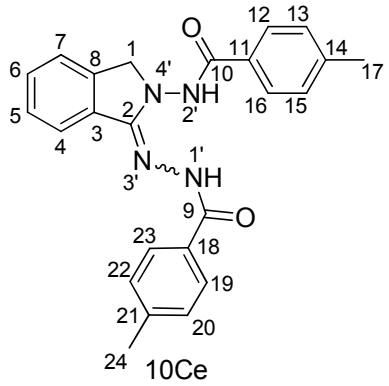
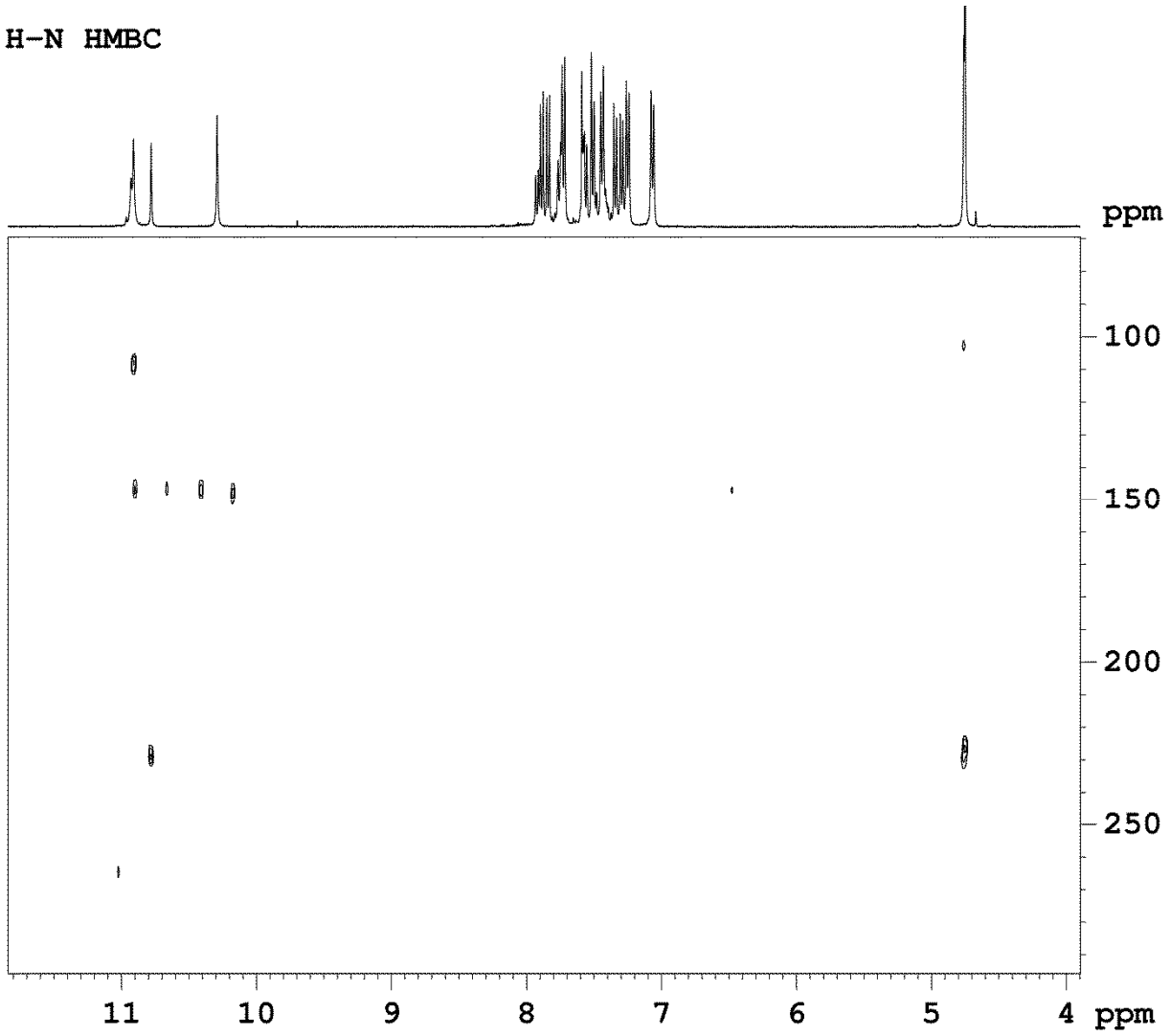
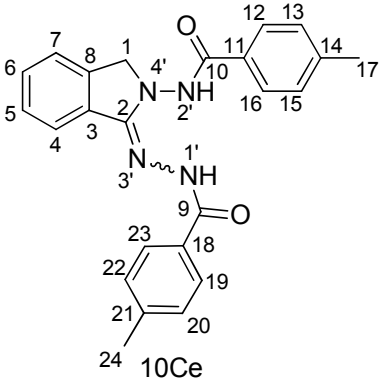


Figure S 38 H-N HMBC NMR spectrum of compound 10Ce





10Ce

Figure S 39 APCI-HRMS spectrum of compound 10Ce

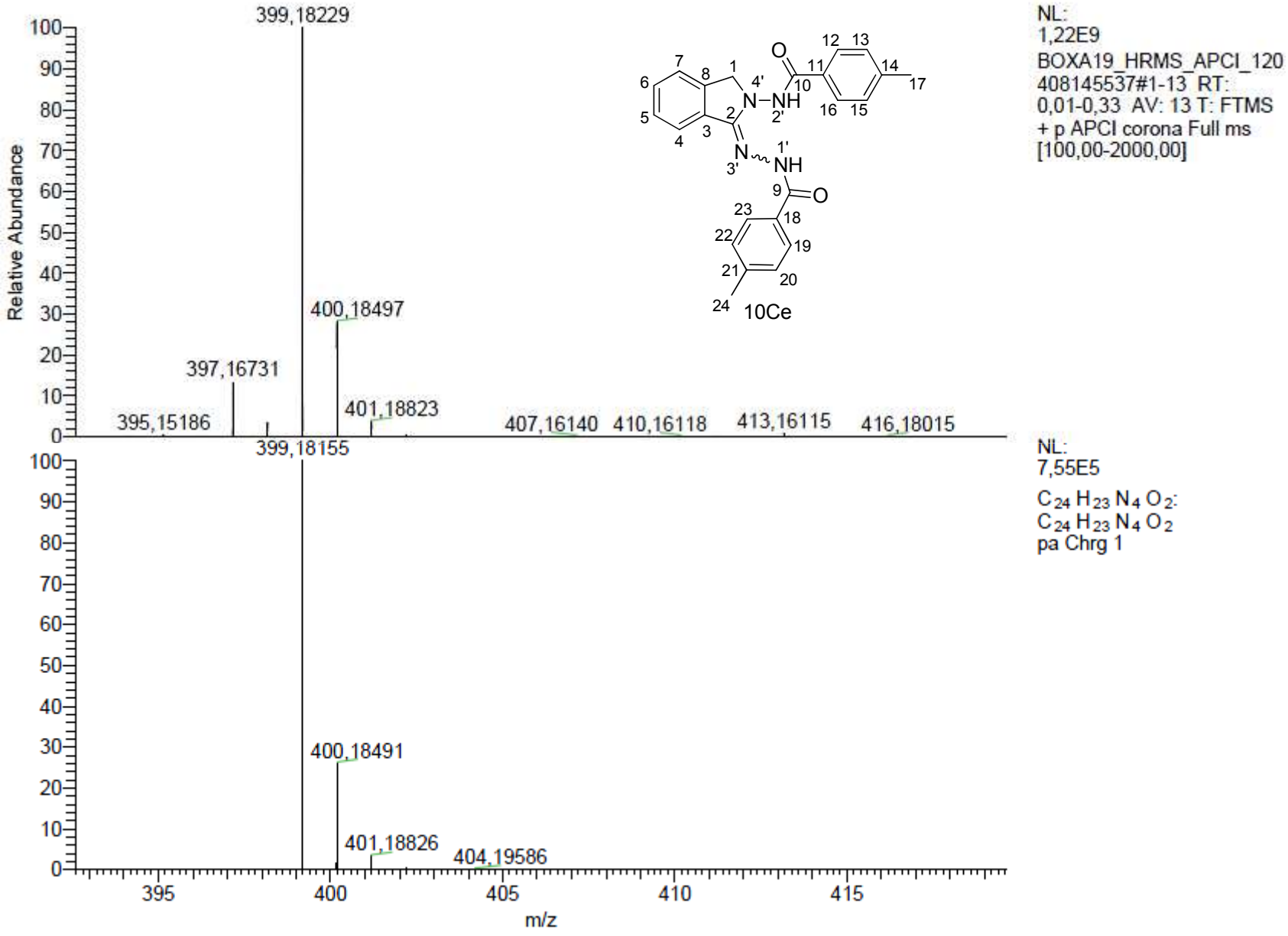


Figure S 40 ^{1}H NMR spectrum of compound 10Cf

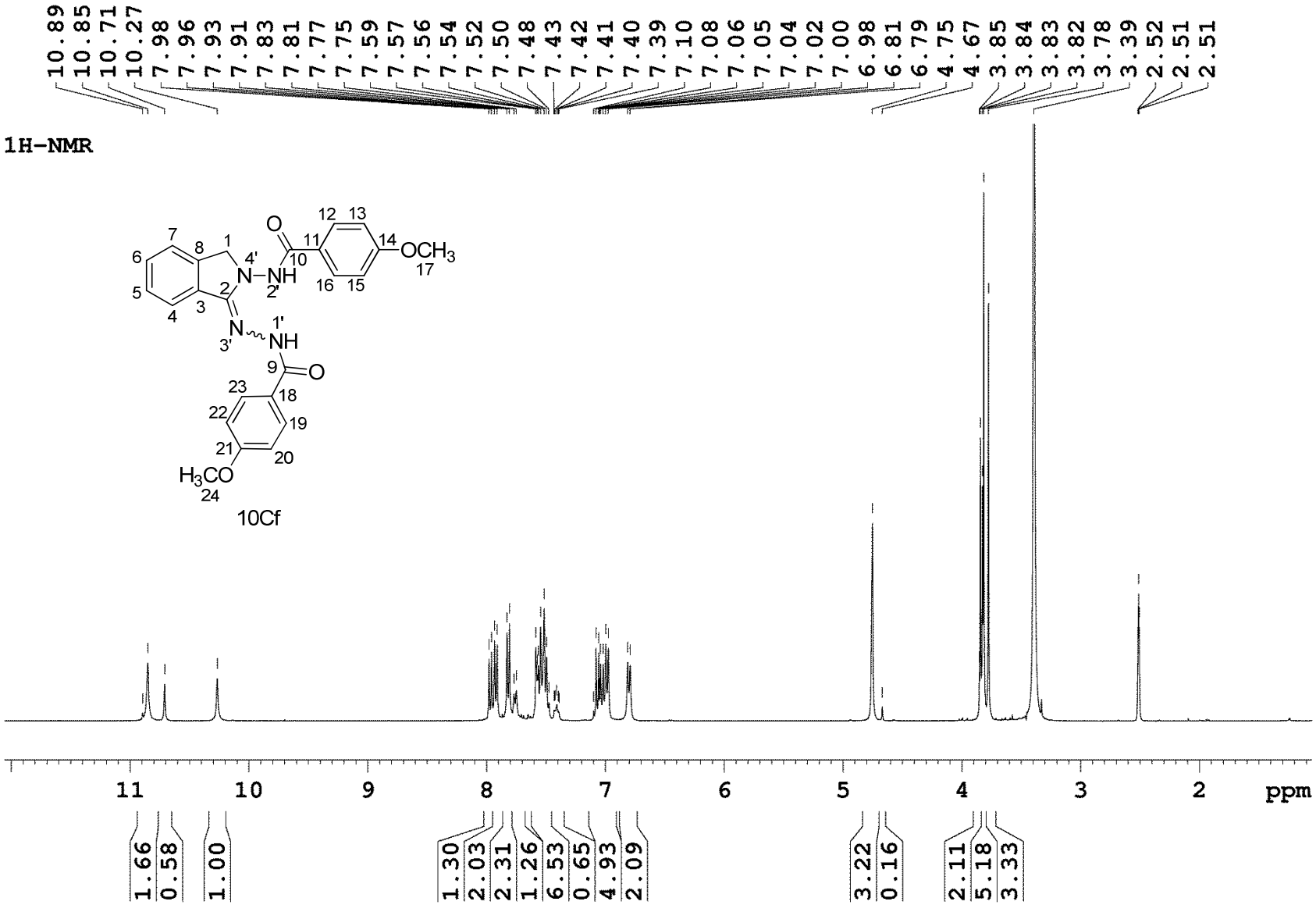


Figure S 41 ^{13}C NMR spectrum of compound 10Cf

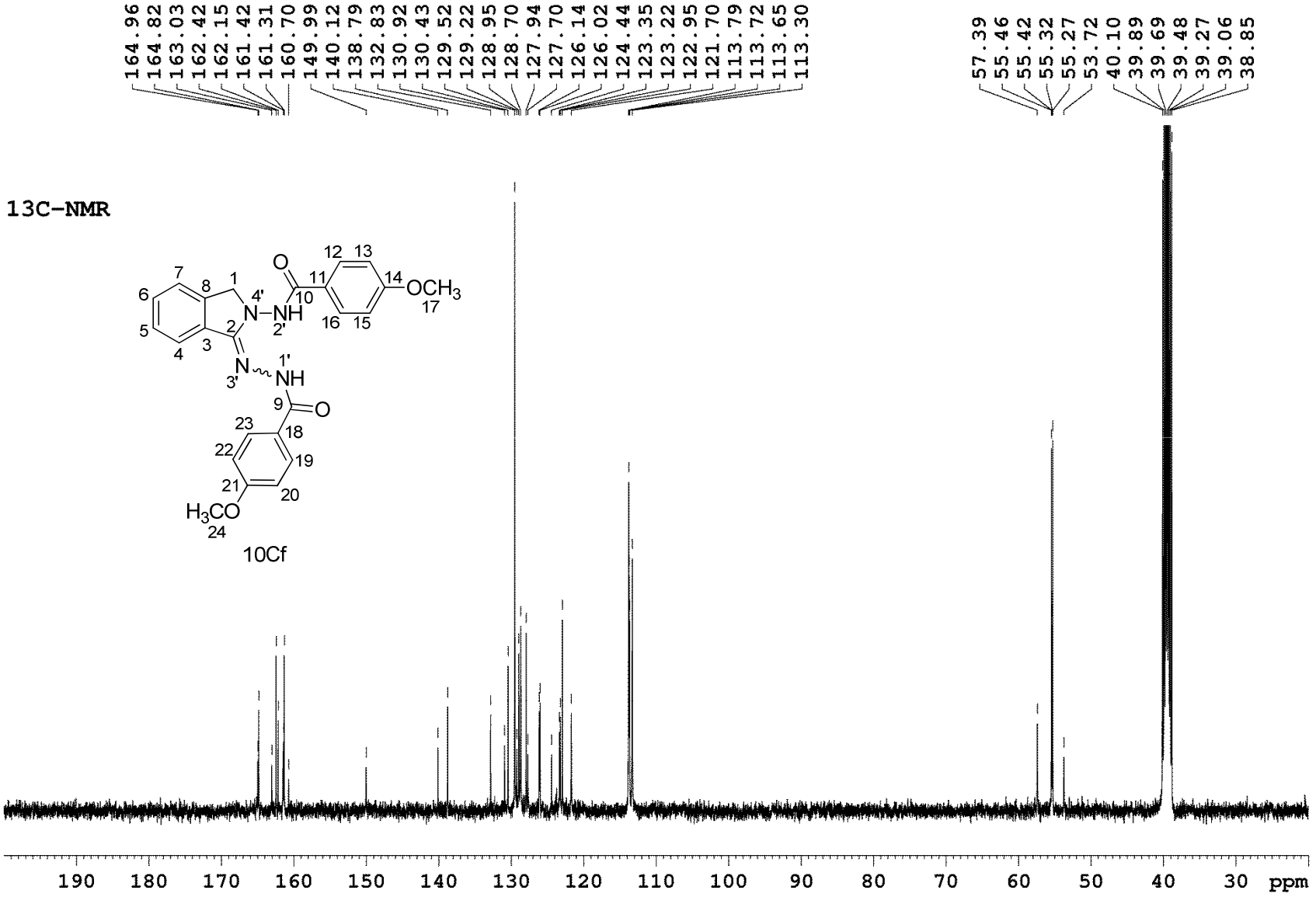
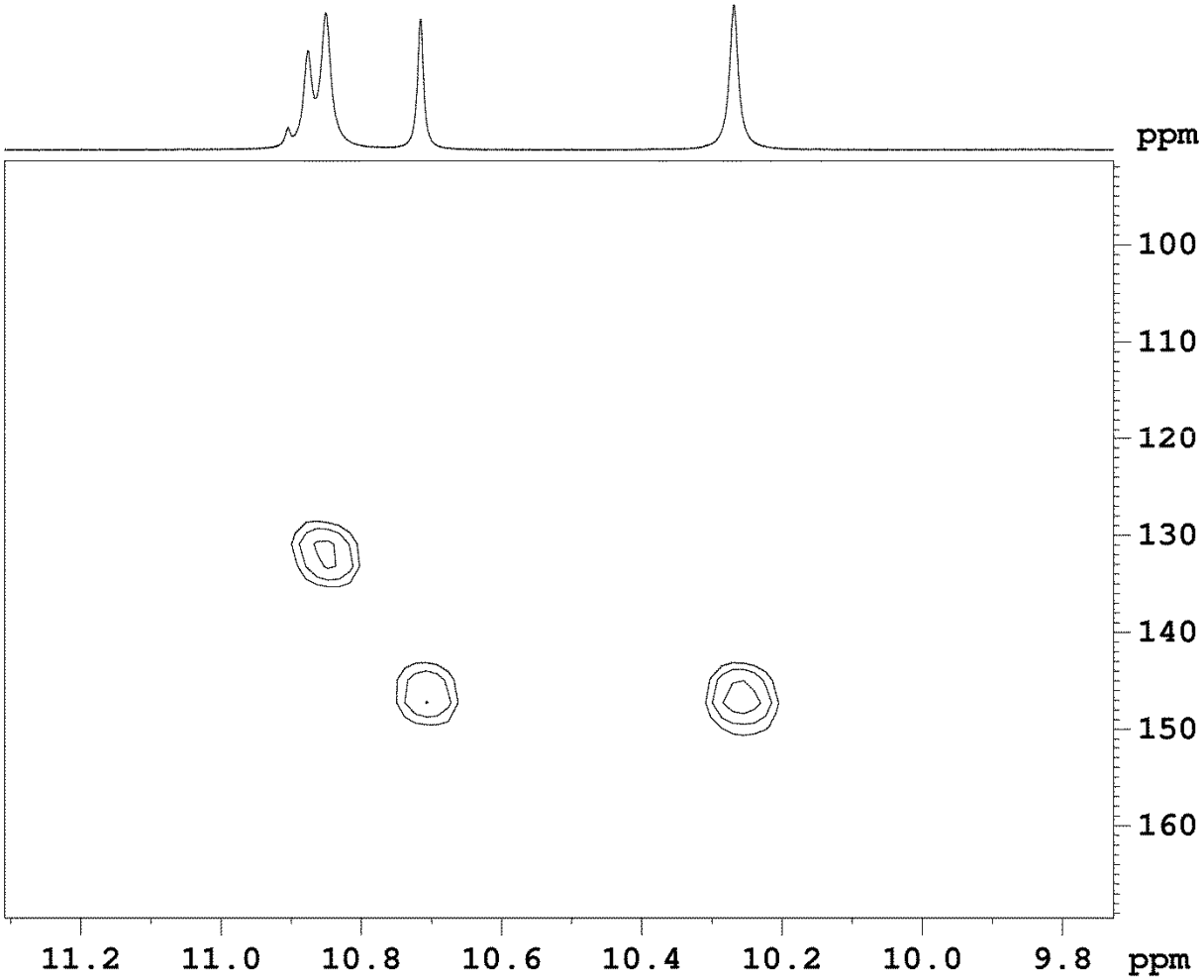
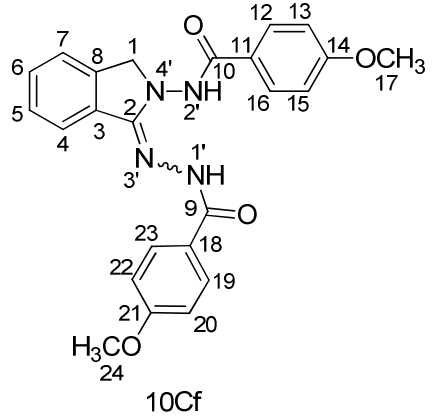


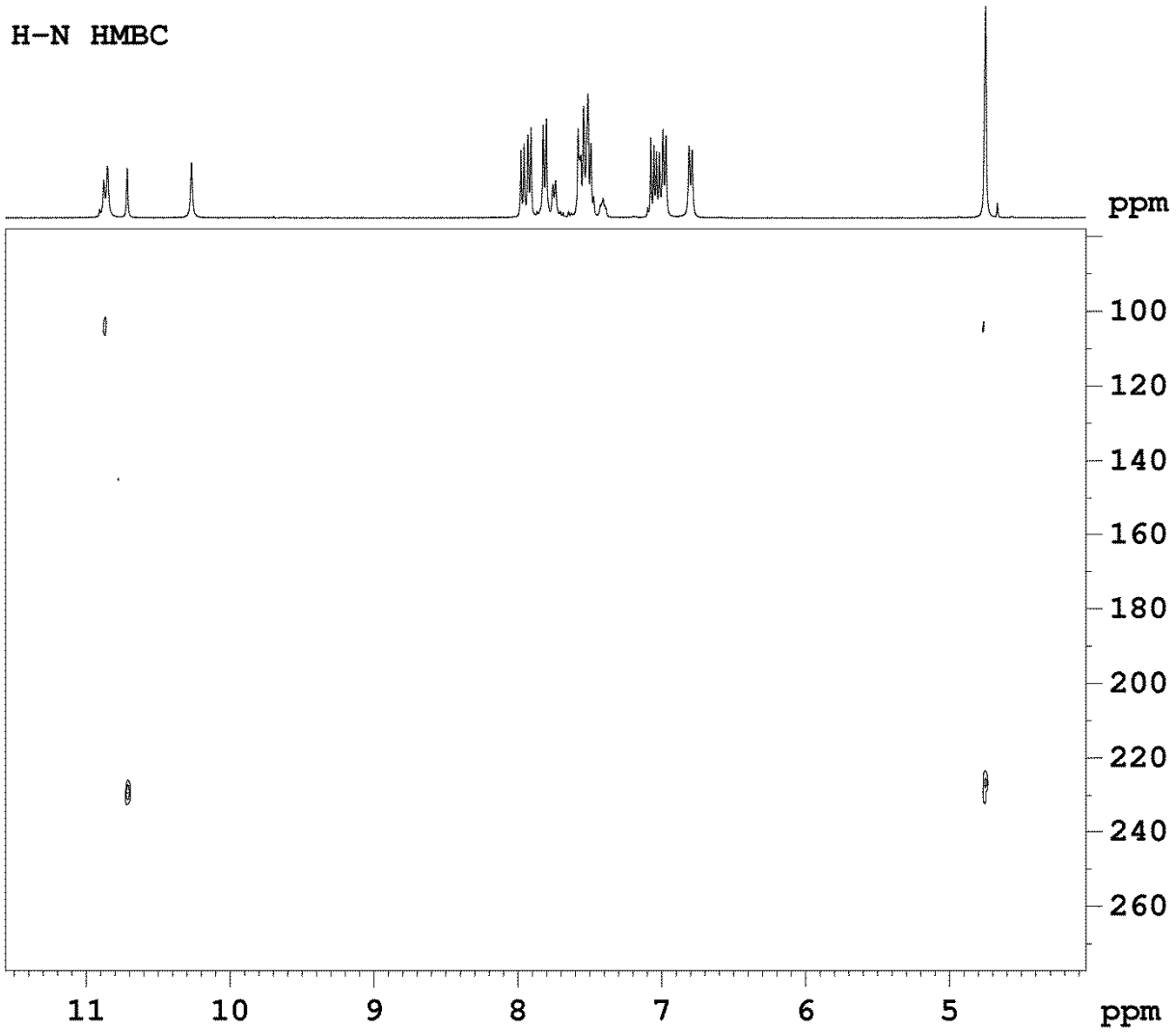
Figure S 42 H-N HMQC NMR spectrum of compound 10Cf

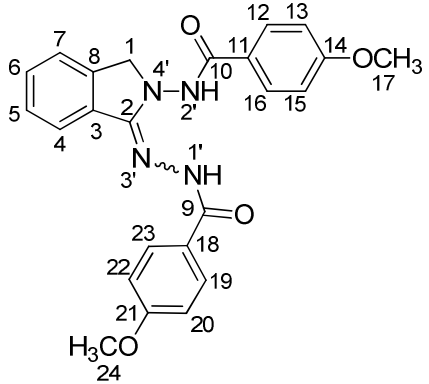




10Cf

Figure S 43 H-N HMBC NMR spectrum of compound 10Cf





10Cf

Figure S 44 APCI-HRMS spectrum of compound 10Cf

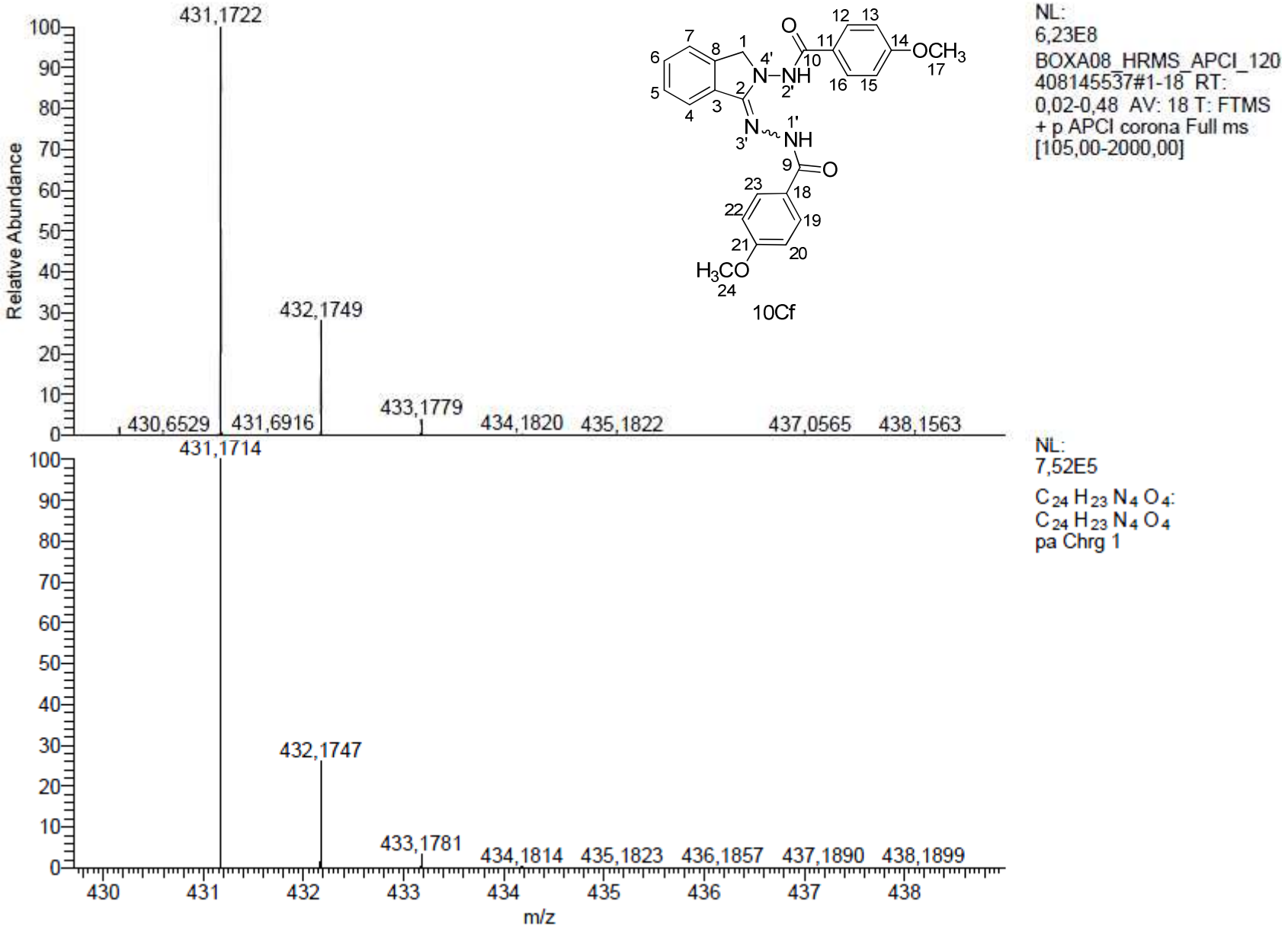


Figure S 45 ^{1}H NMR spectrum of compound 10Cg

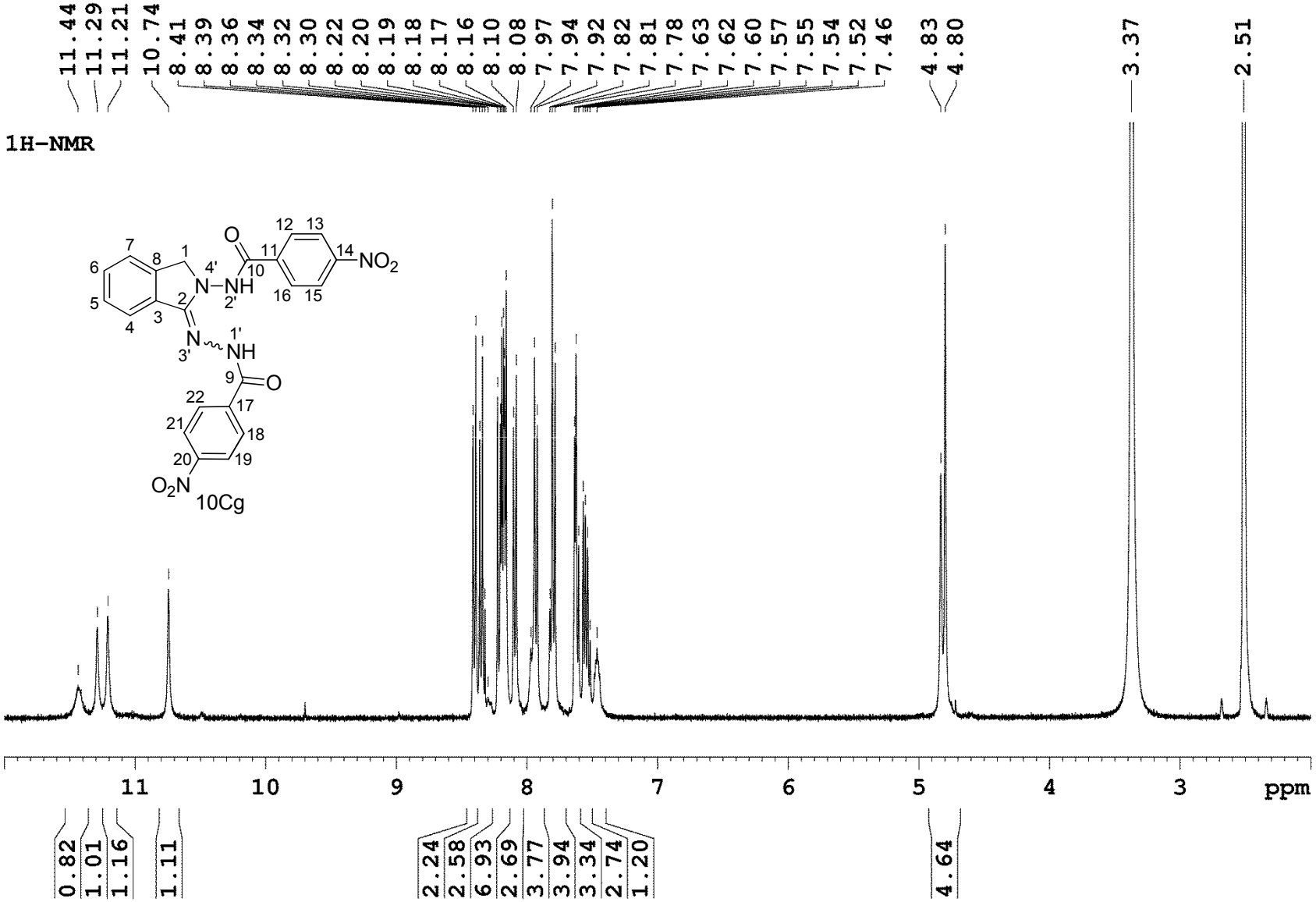


Figure S 46 ^{13}C NMR spectrum of compound 10Cg

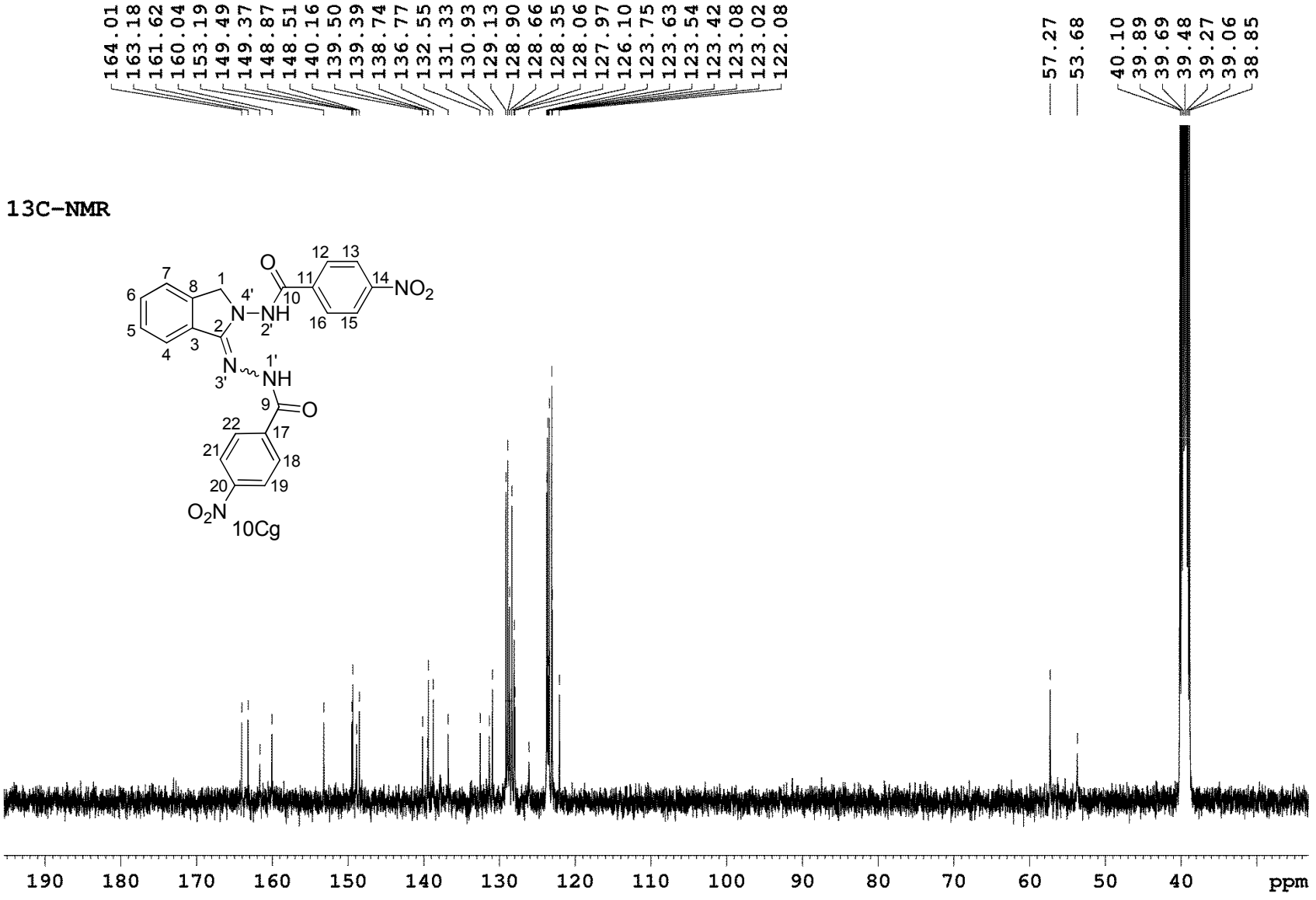


Figure S 47 APCI HR-MS spectrum of compound 10Cg

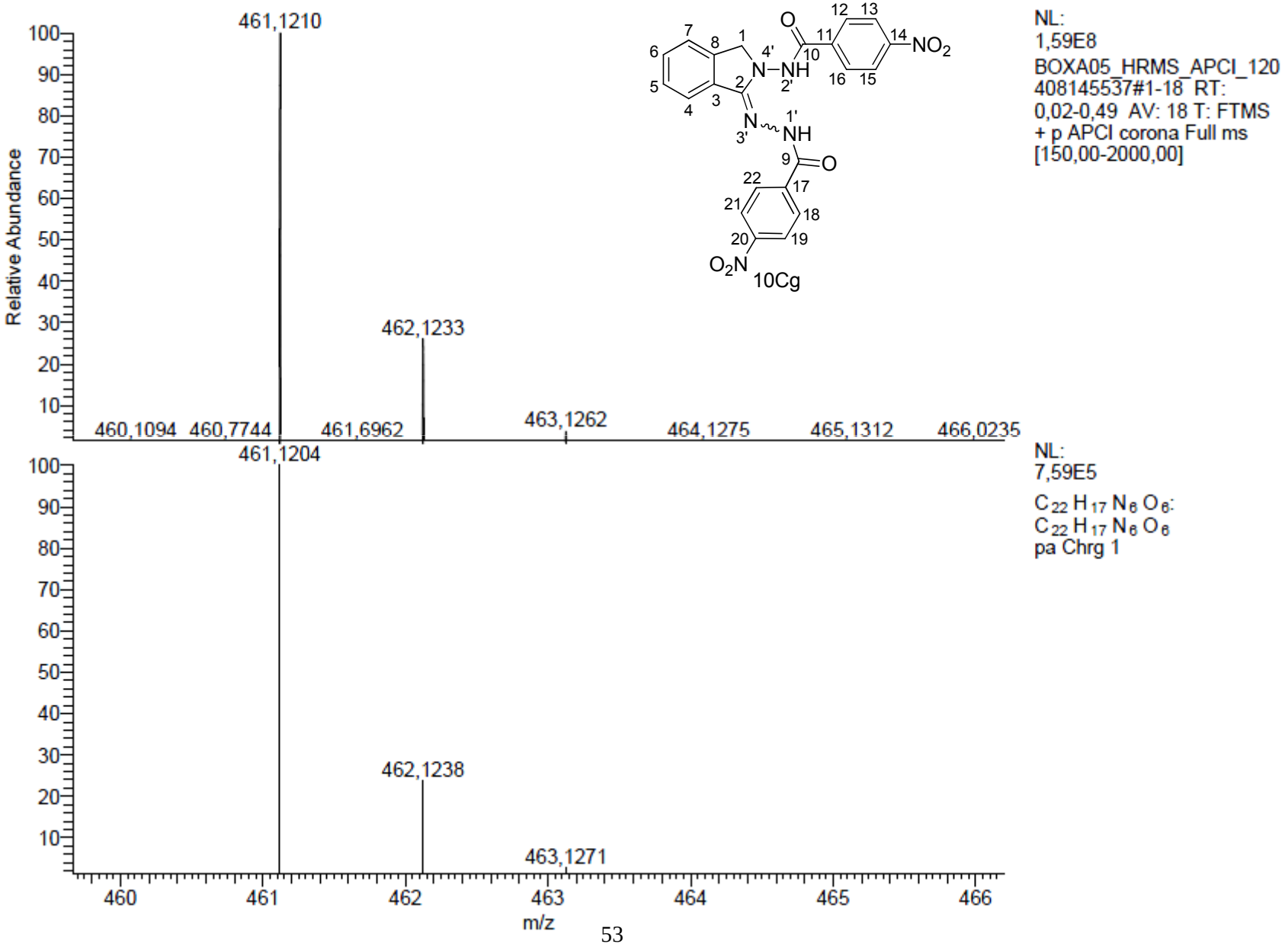


Figure S 48 ^{1}H NMR spectrum of compound 10Ch

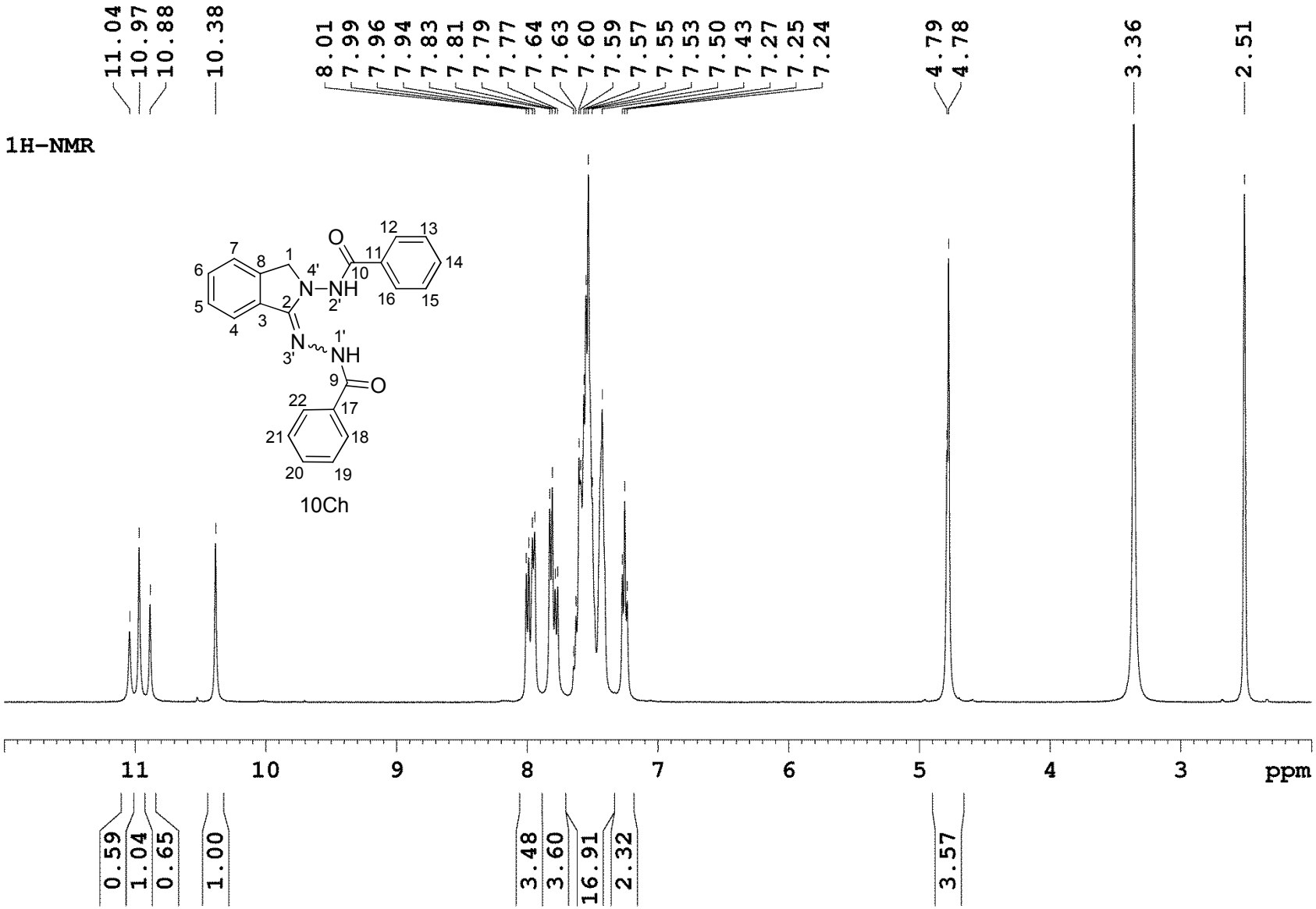


Figure S 49 ^{13}C NMR spectrum of compound 10Ch

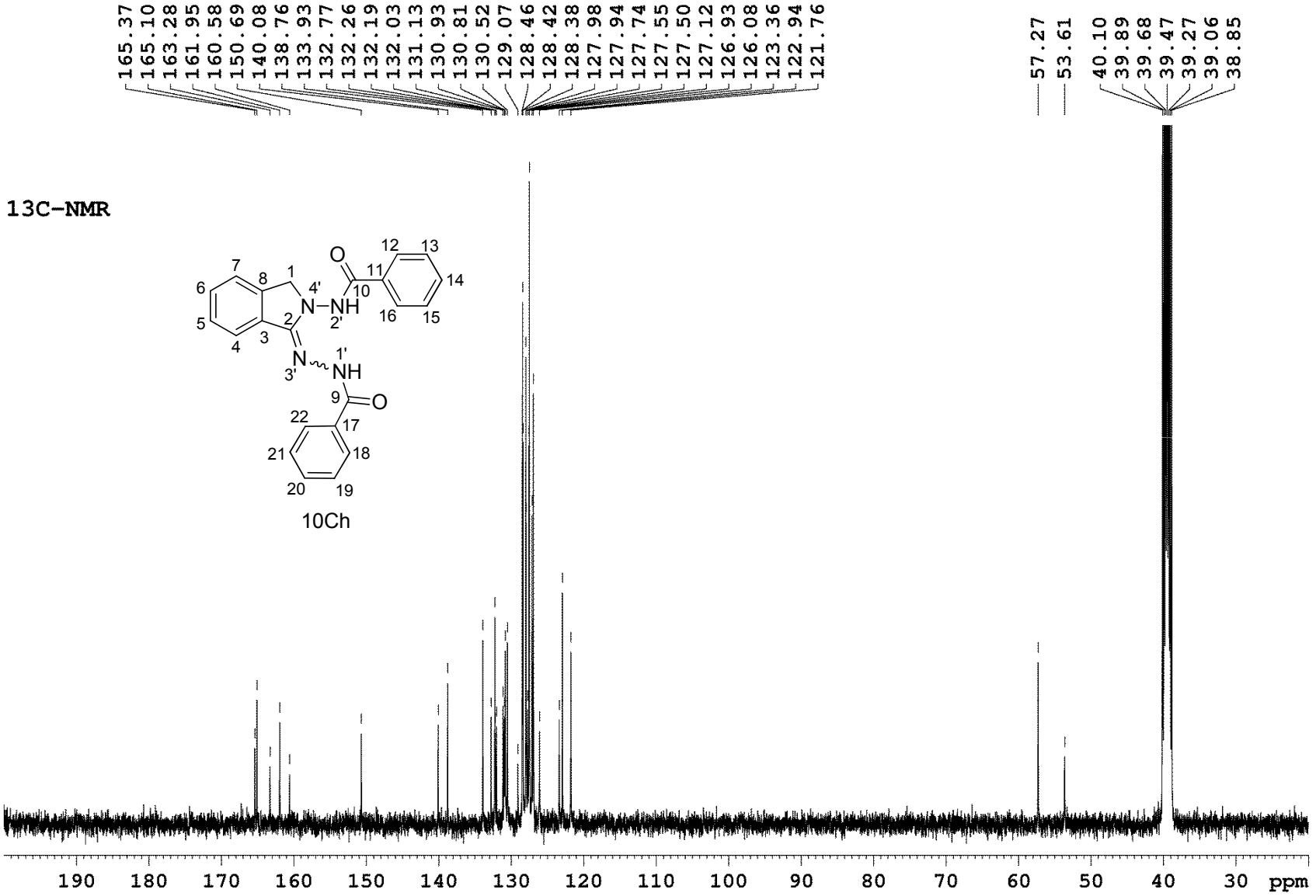
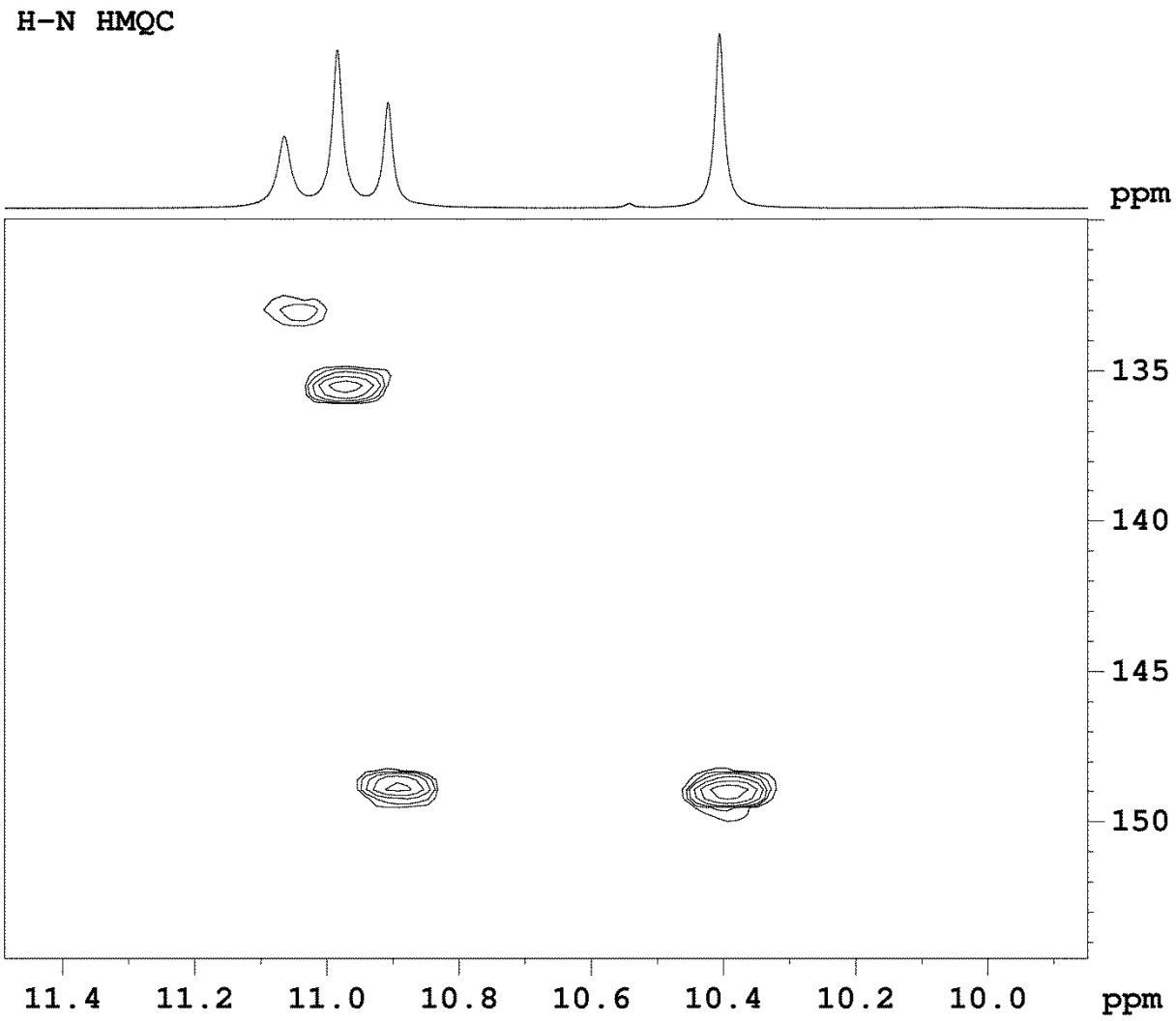
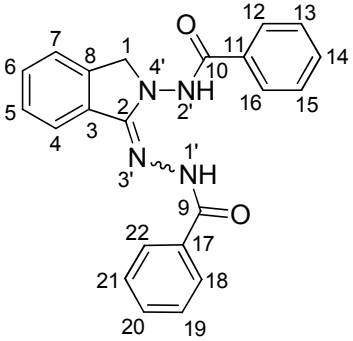


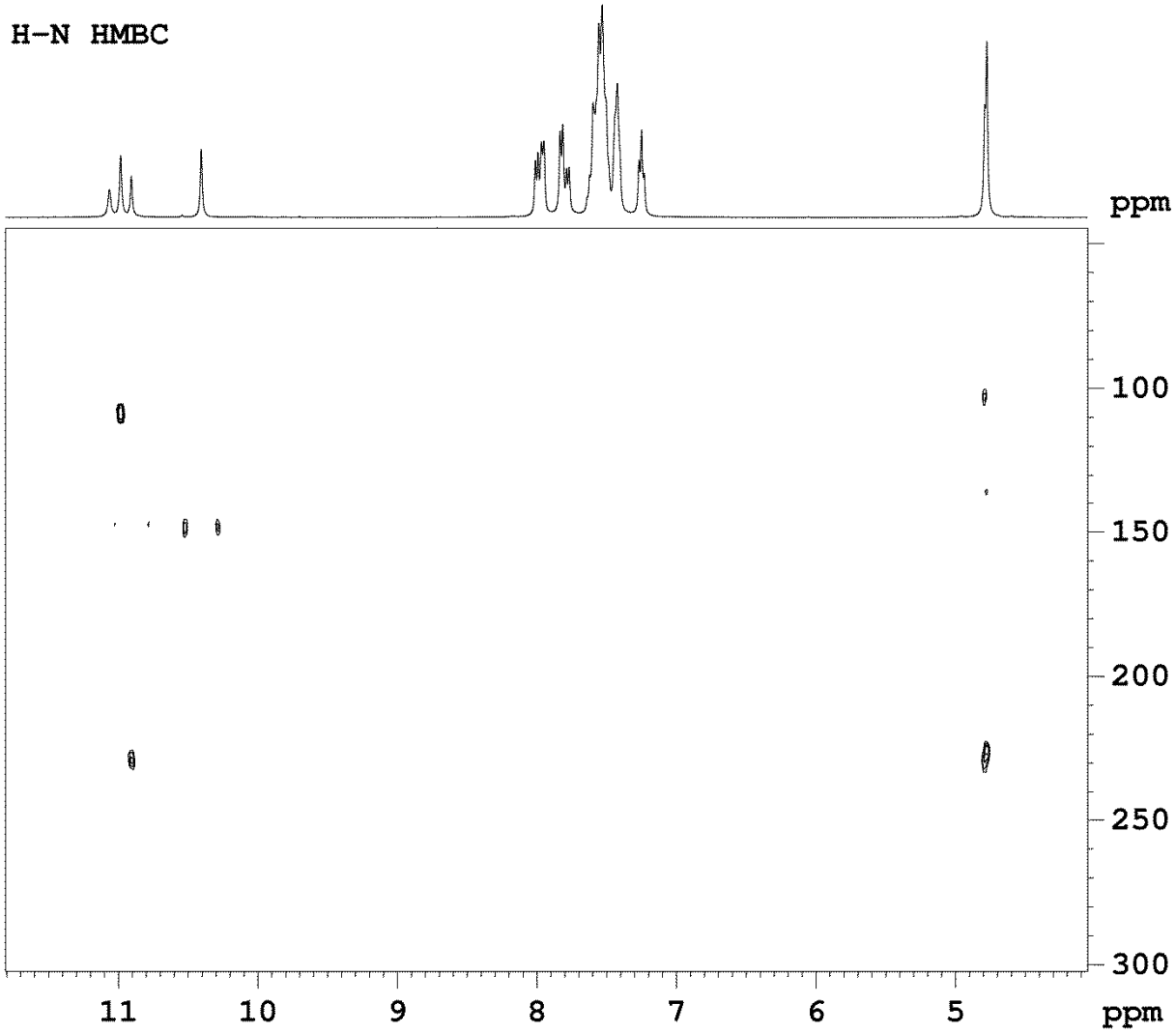
Figure S 50 H-N HMQC NMR spectrum of compound 10Ch

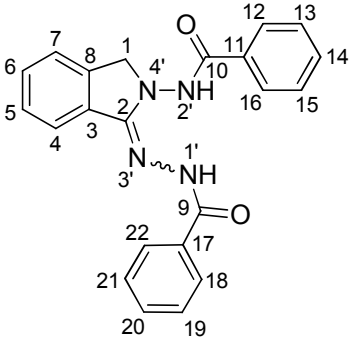




10Ch

Figure S 51 H-N HMBC NMR spectrum of compound 10Ch





10Ch

Figure S 52 APCI-HRMS spectrum of compound 10Ch

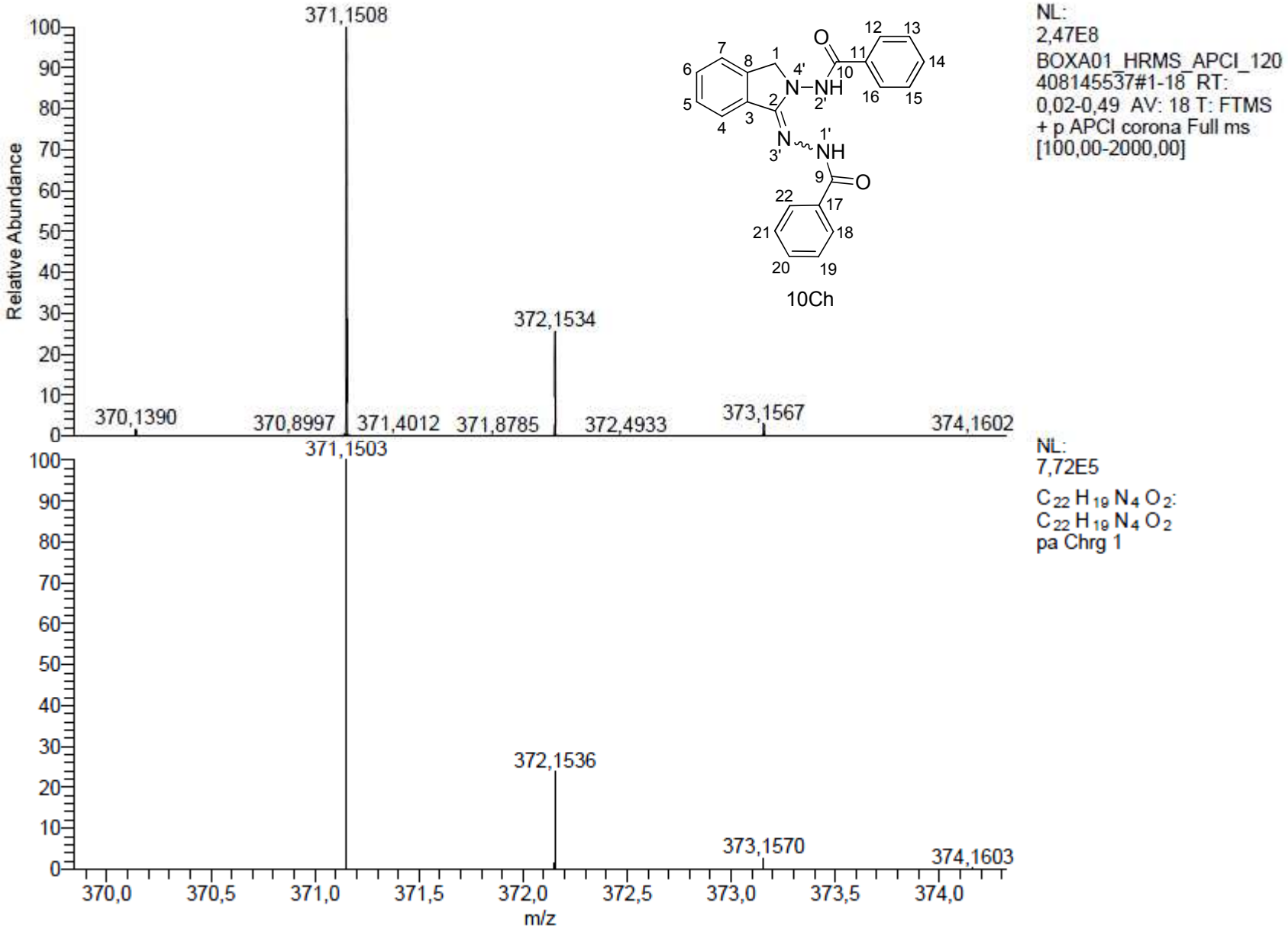


Figure S 53 ^{1}H NMR spectrum of compound 10Ci

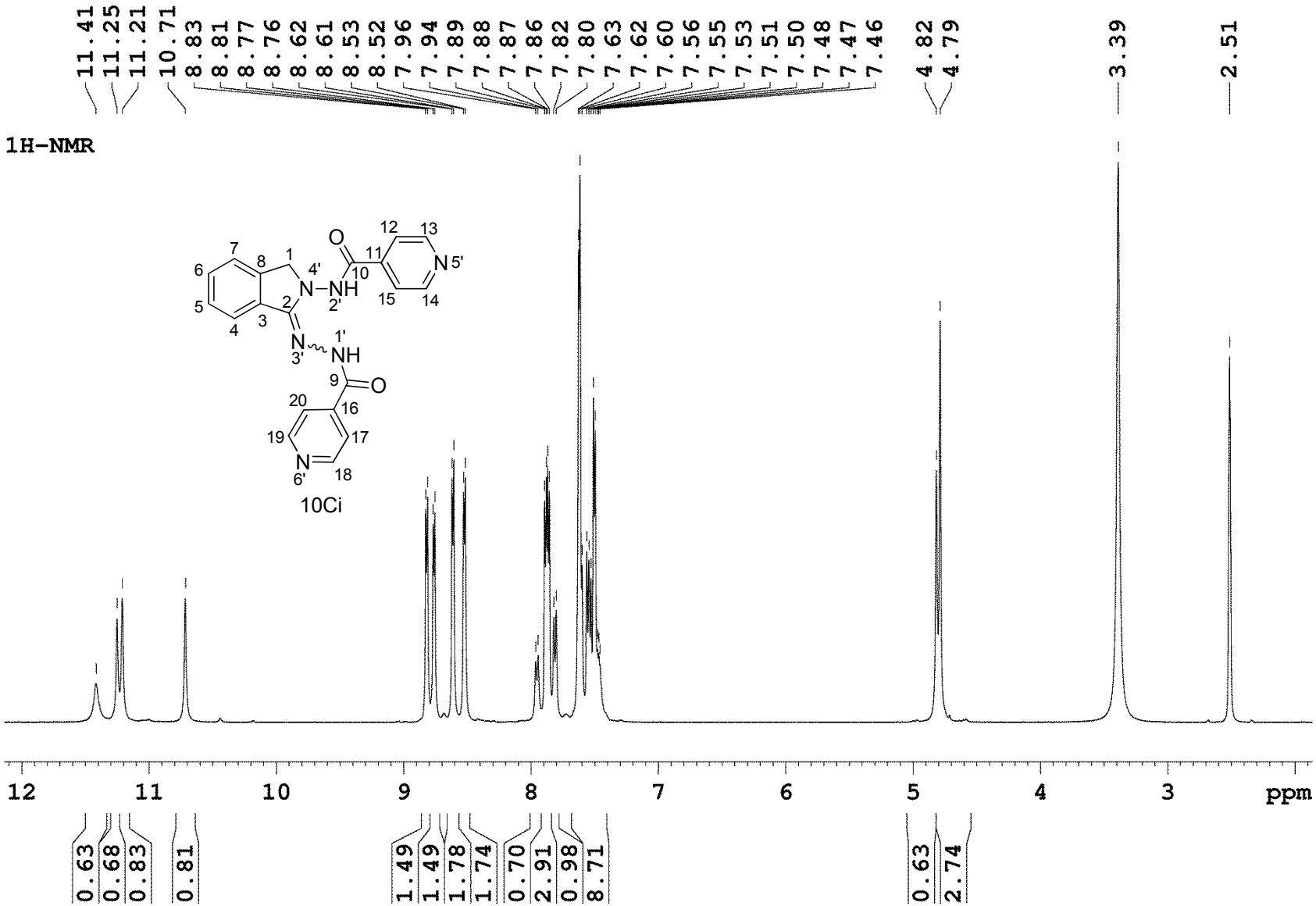


Figure S 54 ^{13}C NMR spectrum of compound 10Ci

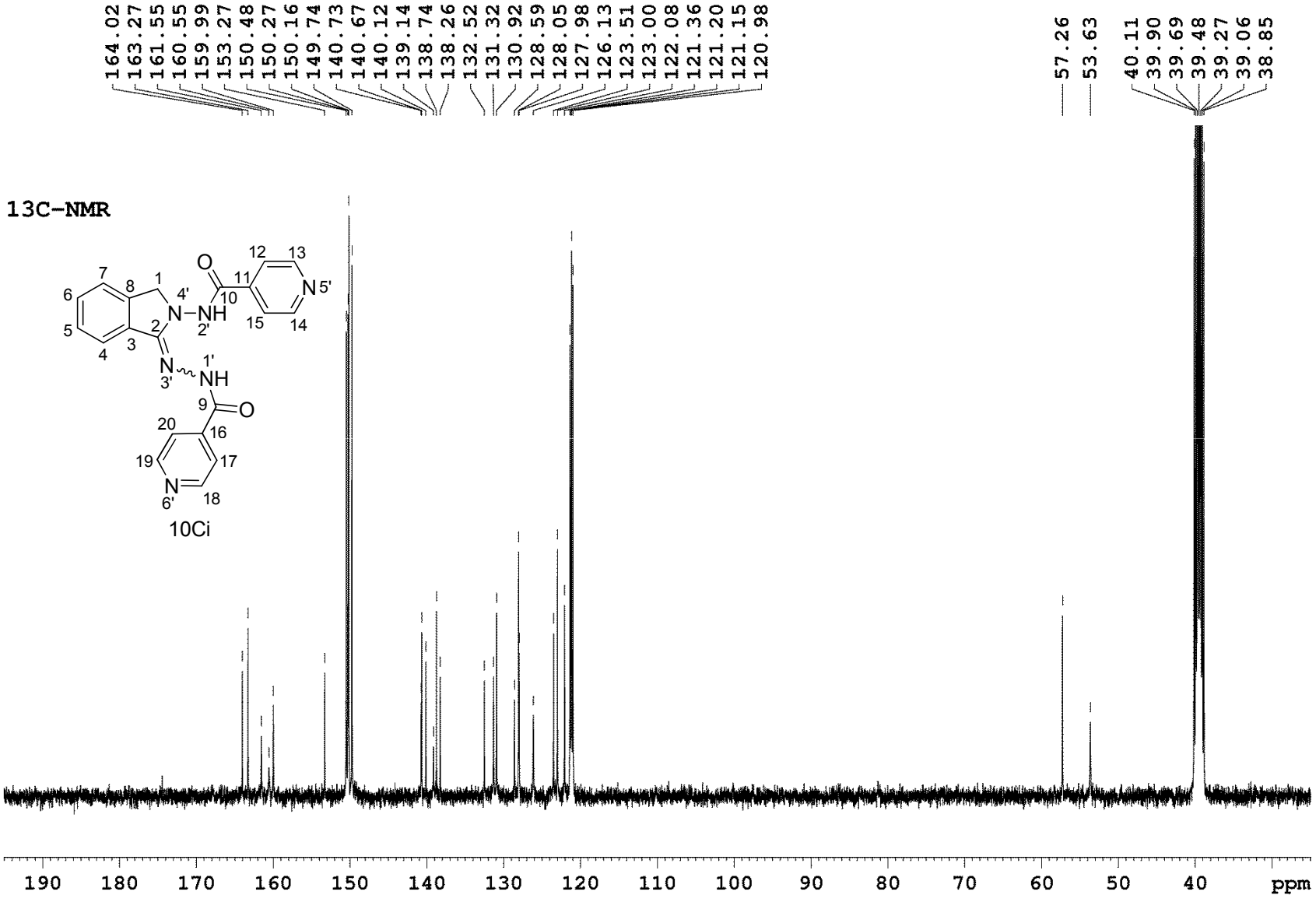
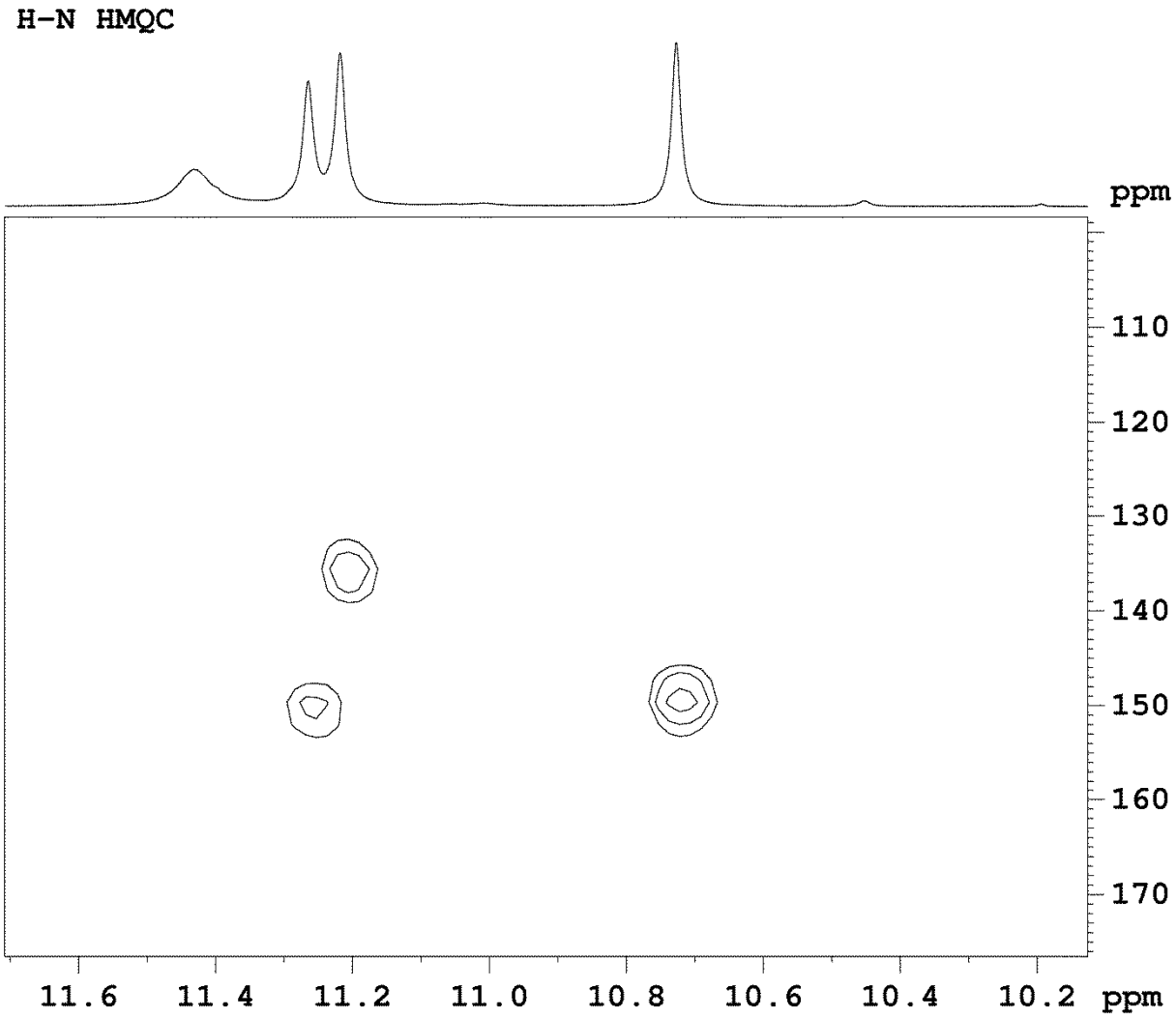
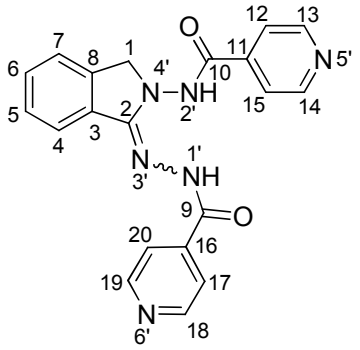


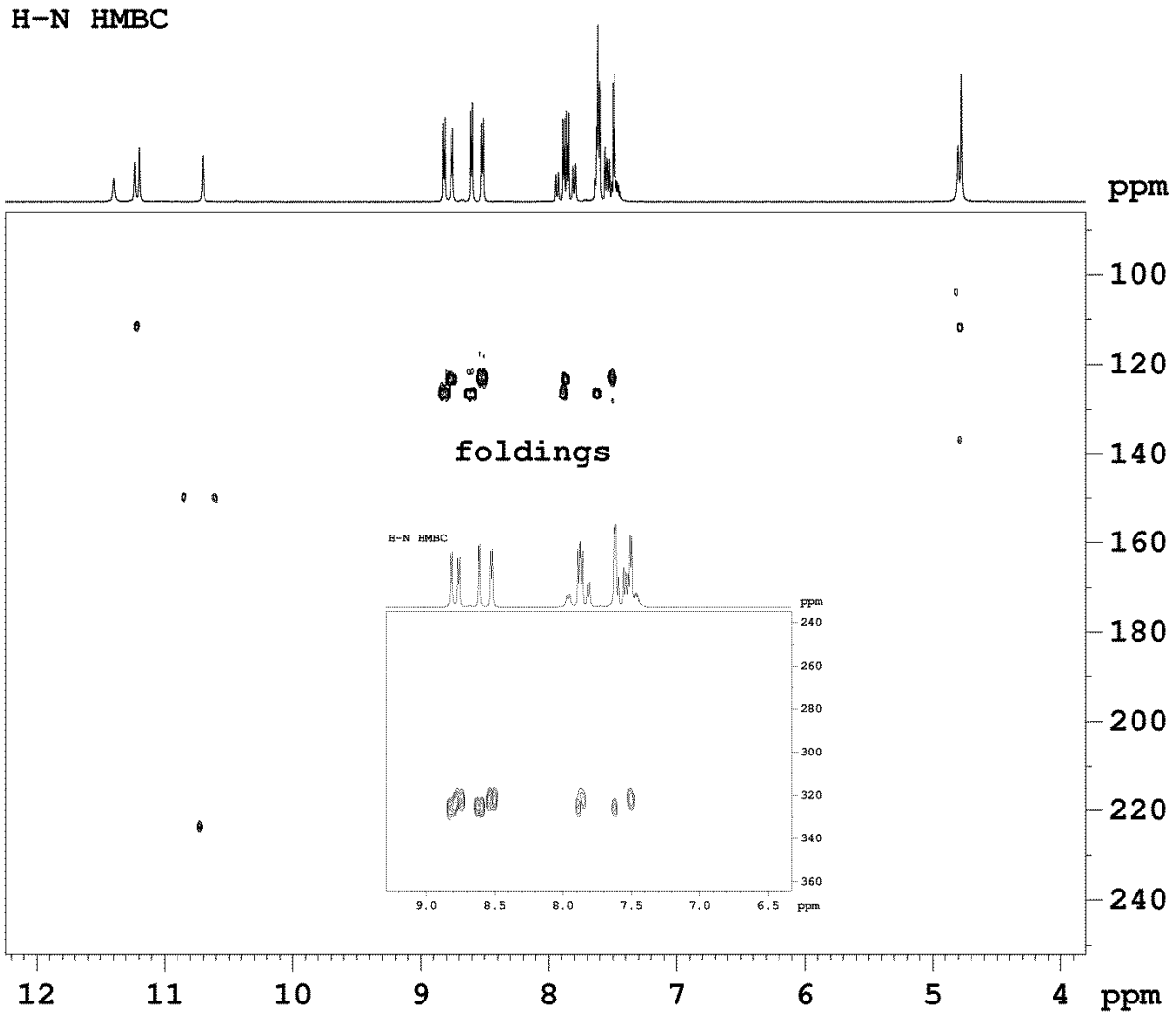
Figure S 55 H-N HMQC NMR spectrum of compound 10Ci

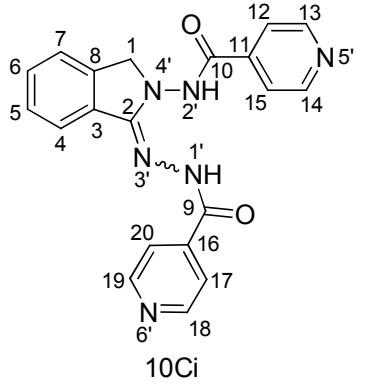




10Ci

Figure S 56 H-N HMBC NMR spectrum of compound 10Ci





10Ci

Figure S 57 APCI-HRMS spectrum of compound 10Ci

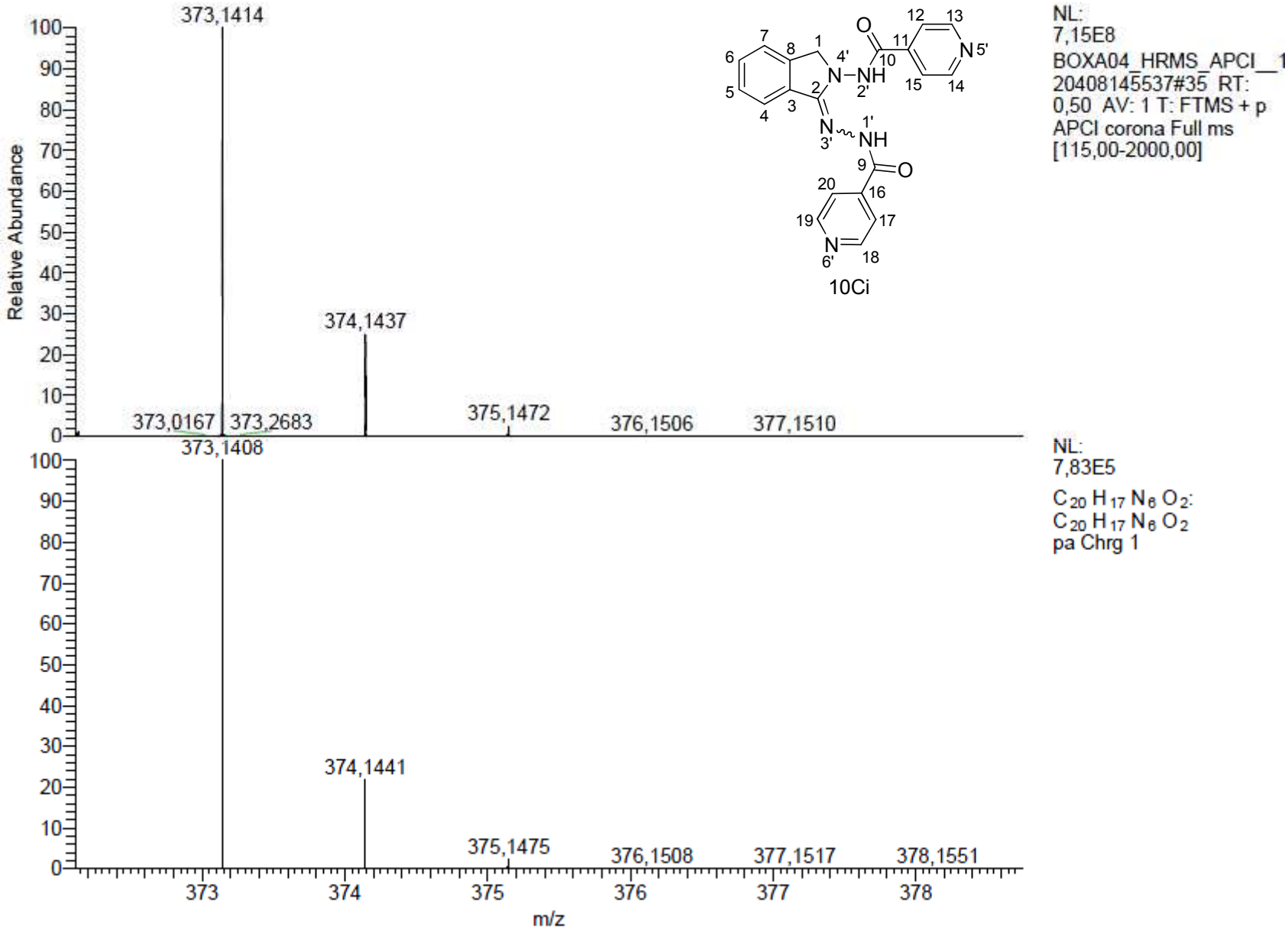


Figure S 58 ^{1}H NMR spectrum of compound 5a

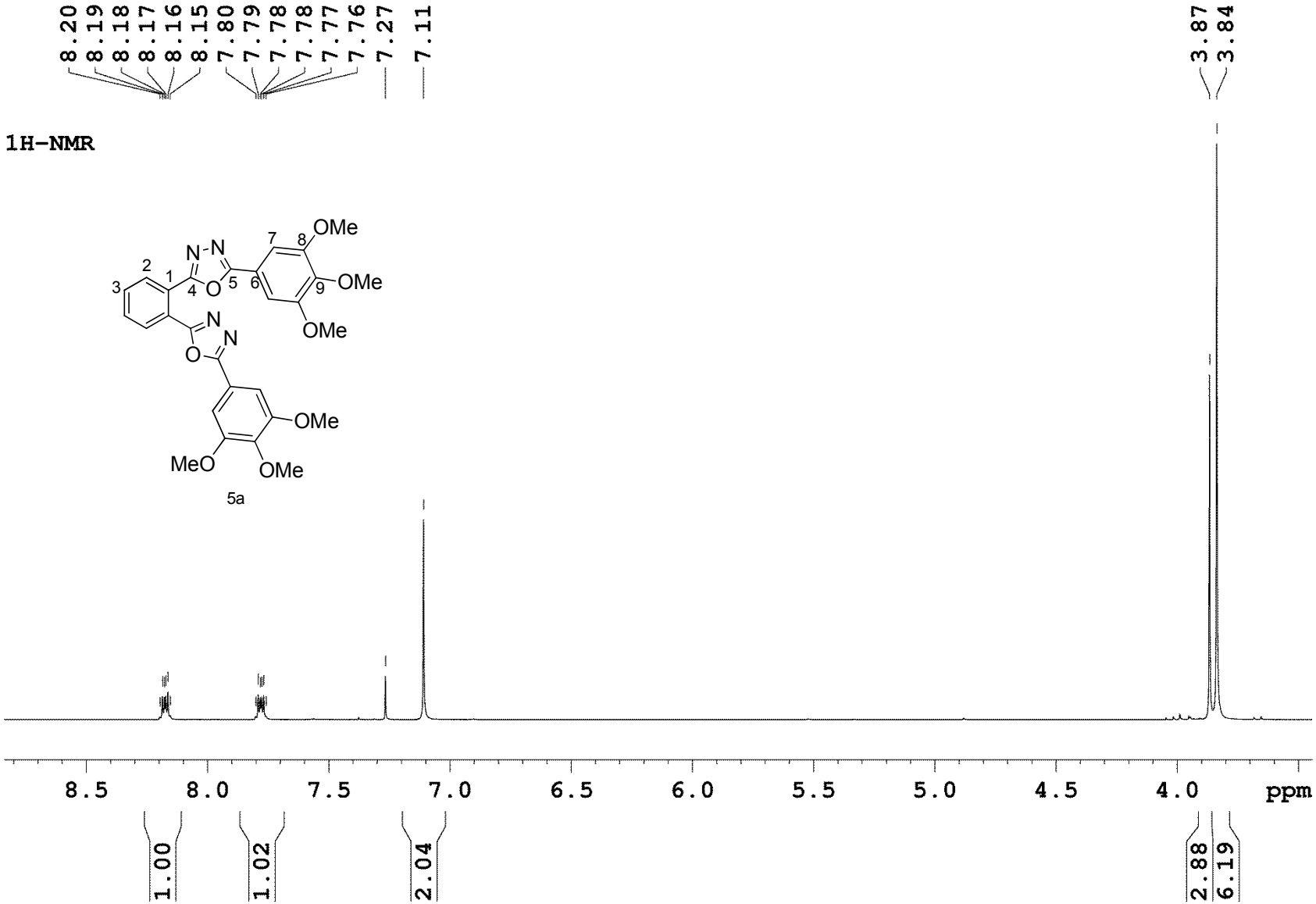


Figure S 59 ^{13}C NMR spectrum of compound 5a

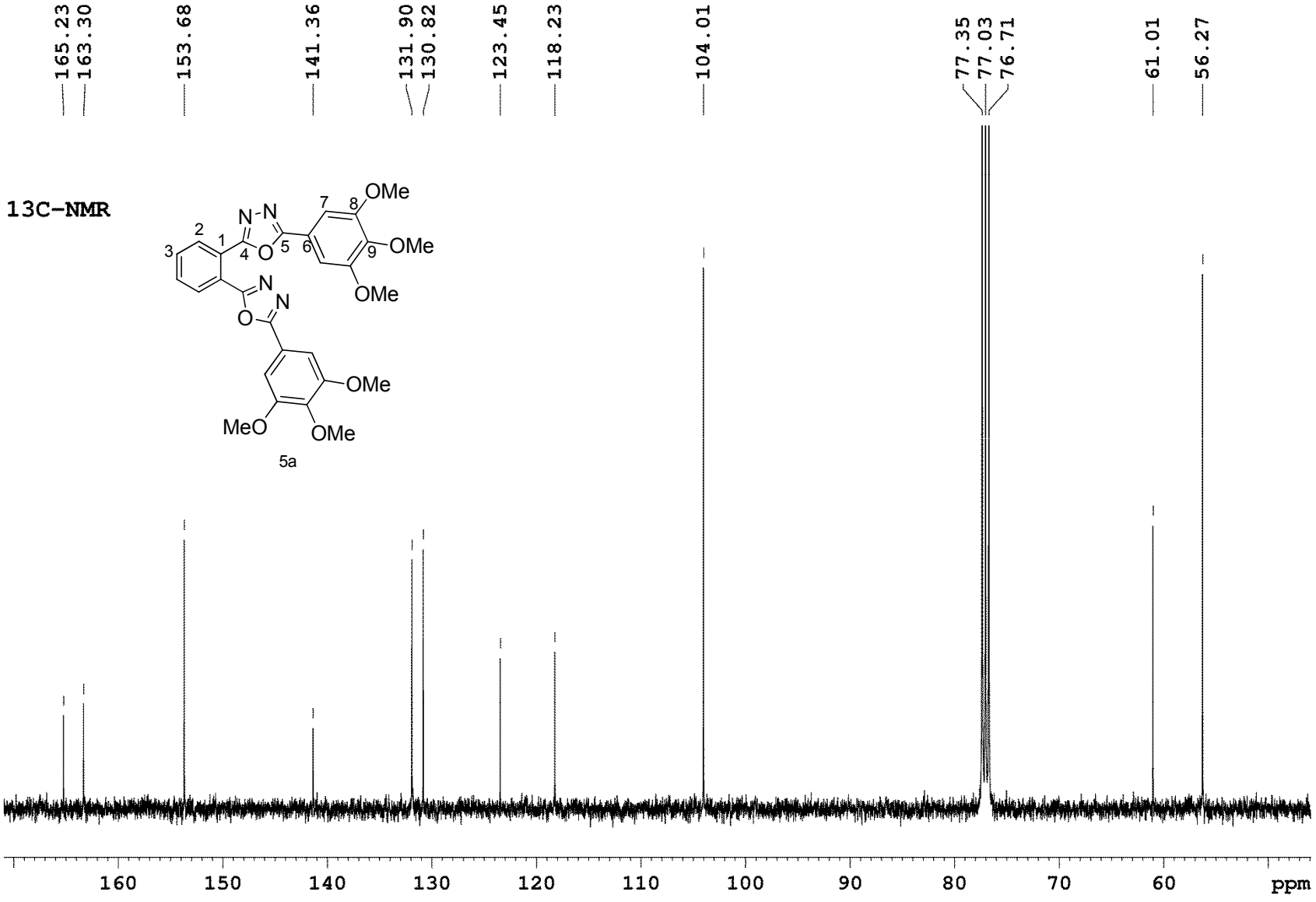


Figure S 60 APCI-HRMS spectrum of compound 5a

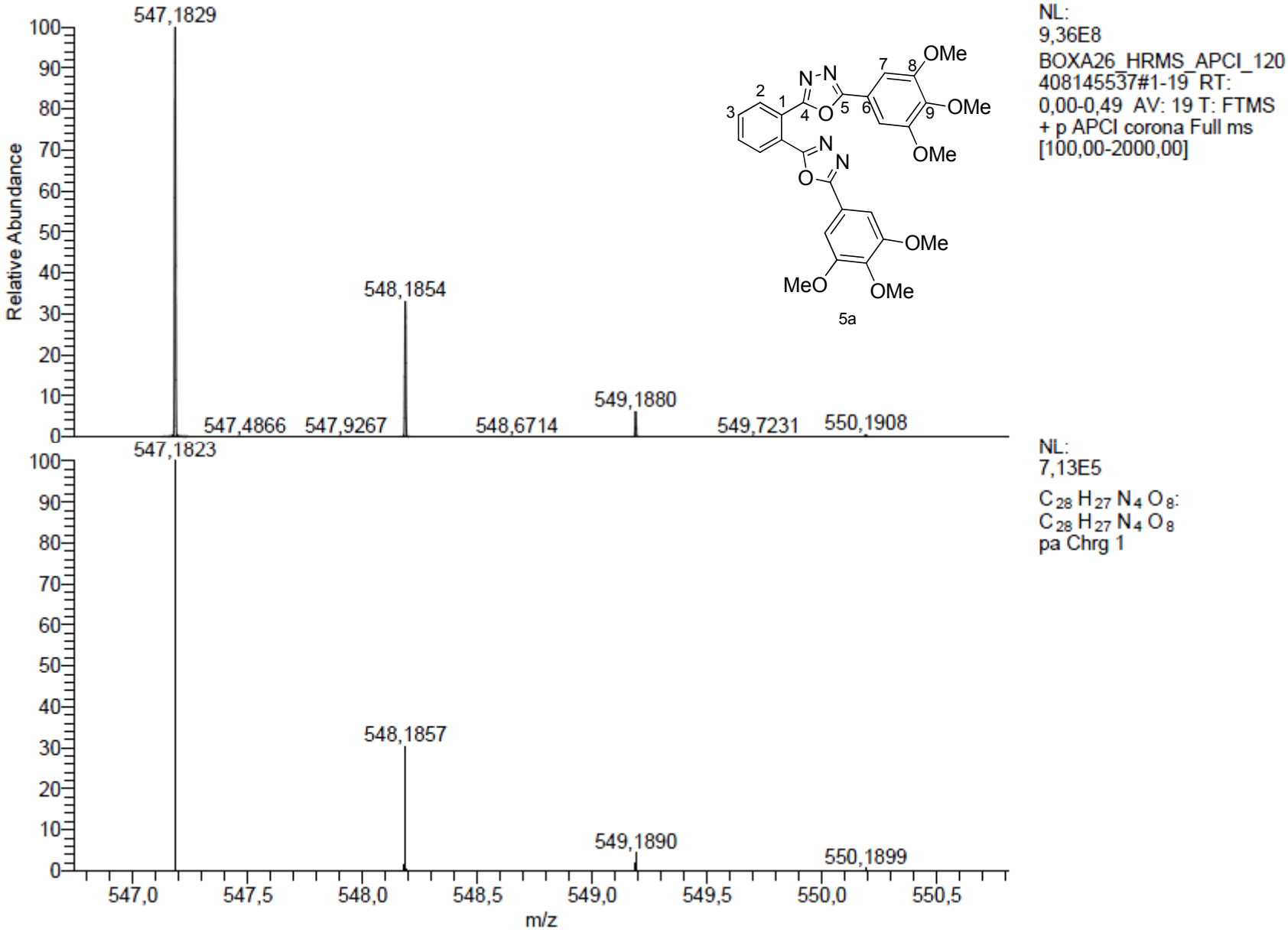


Figure S 61 ^{1}H NMR spectrum of compound 5b

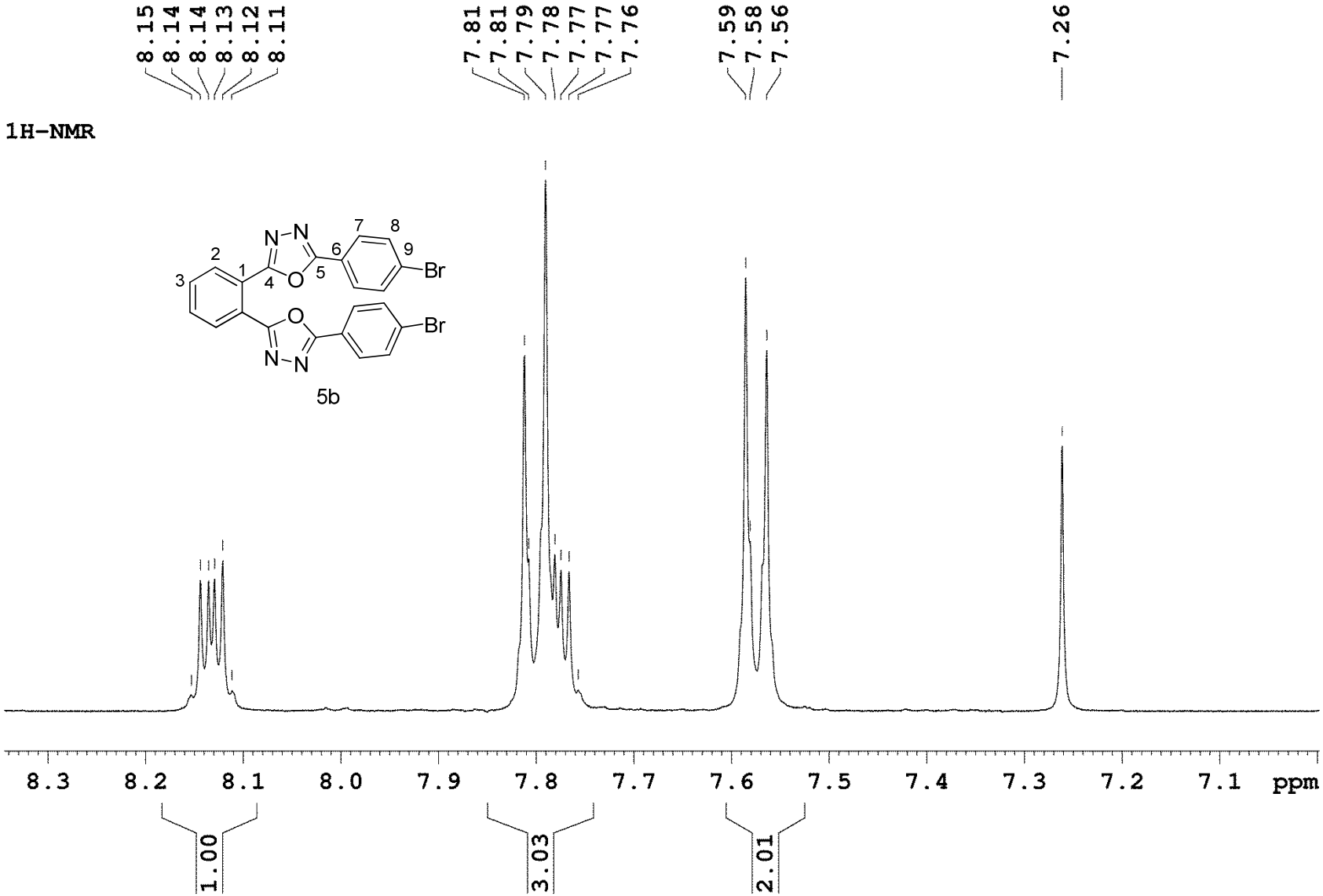


Figure S 62 ^{13}C NMR spectrum of compound 5b

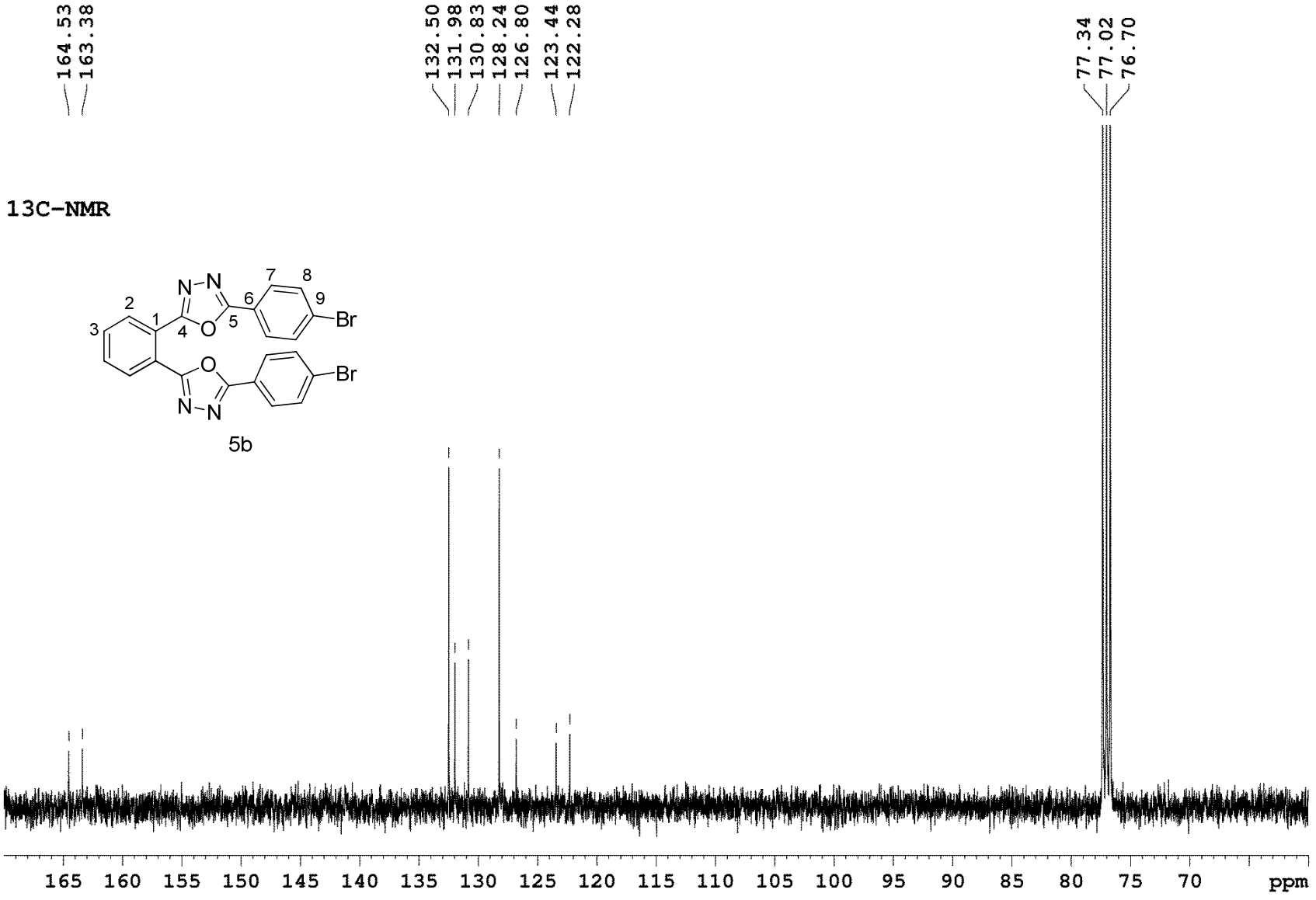


Figure S 63 APCI-HRMS spectrum of compound 5b

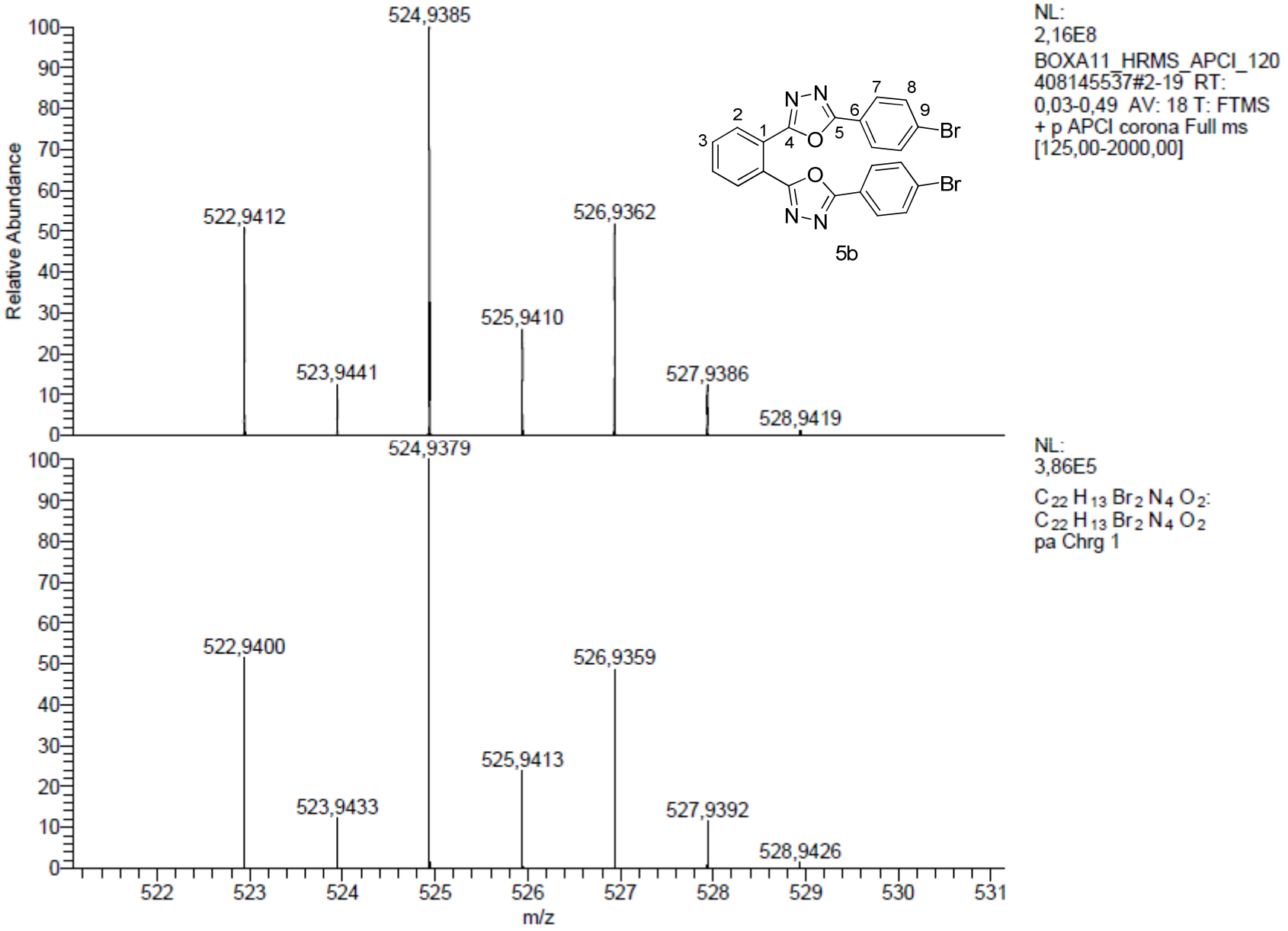


Figure S 64 ^{1}H NMR spectrum of compound 5c

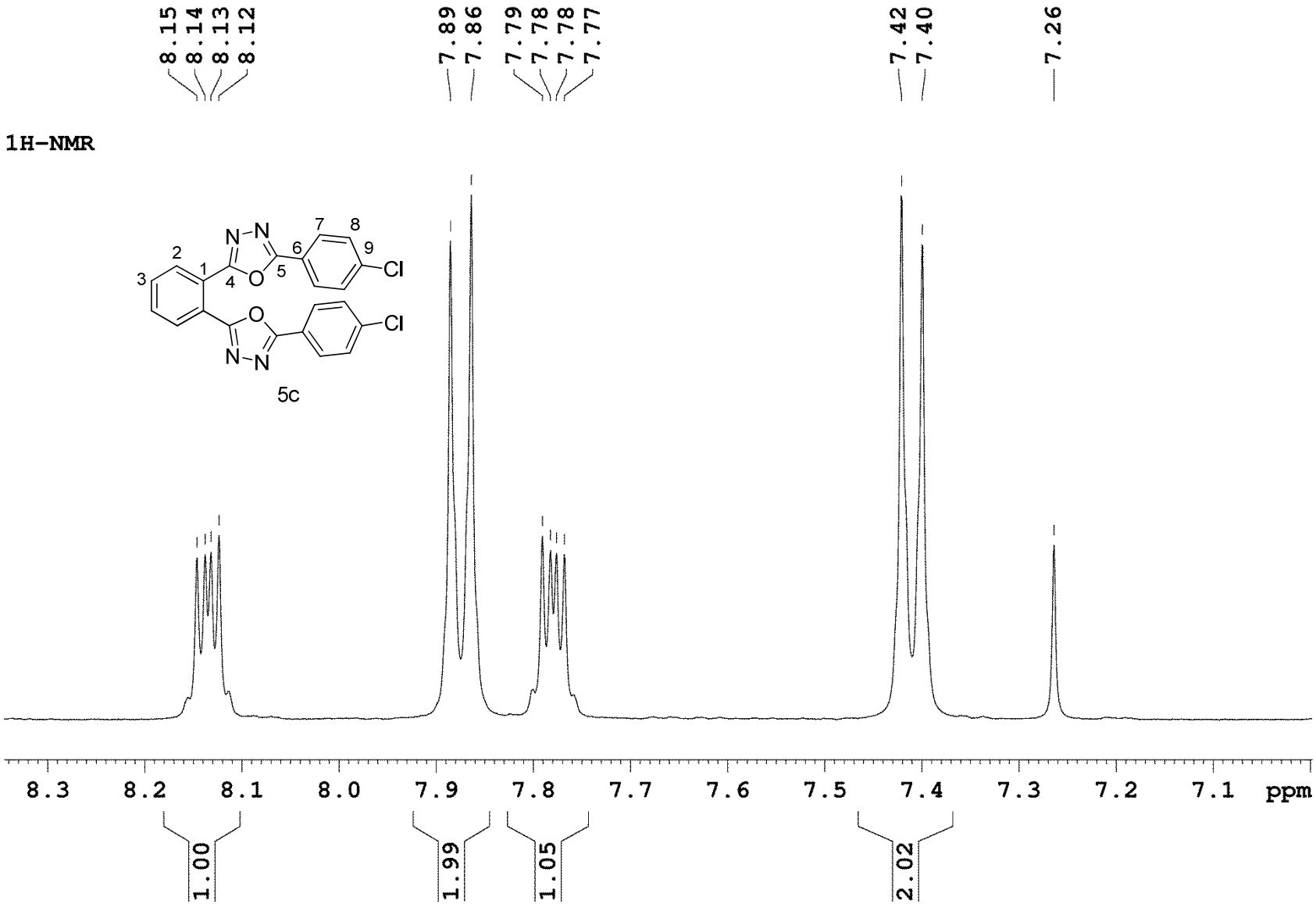


Figure S 65 ^{13}C NMR spectrum of compound 5c

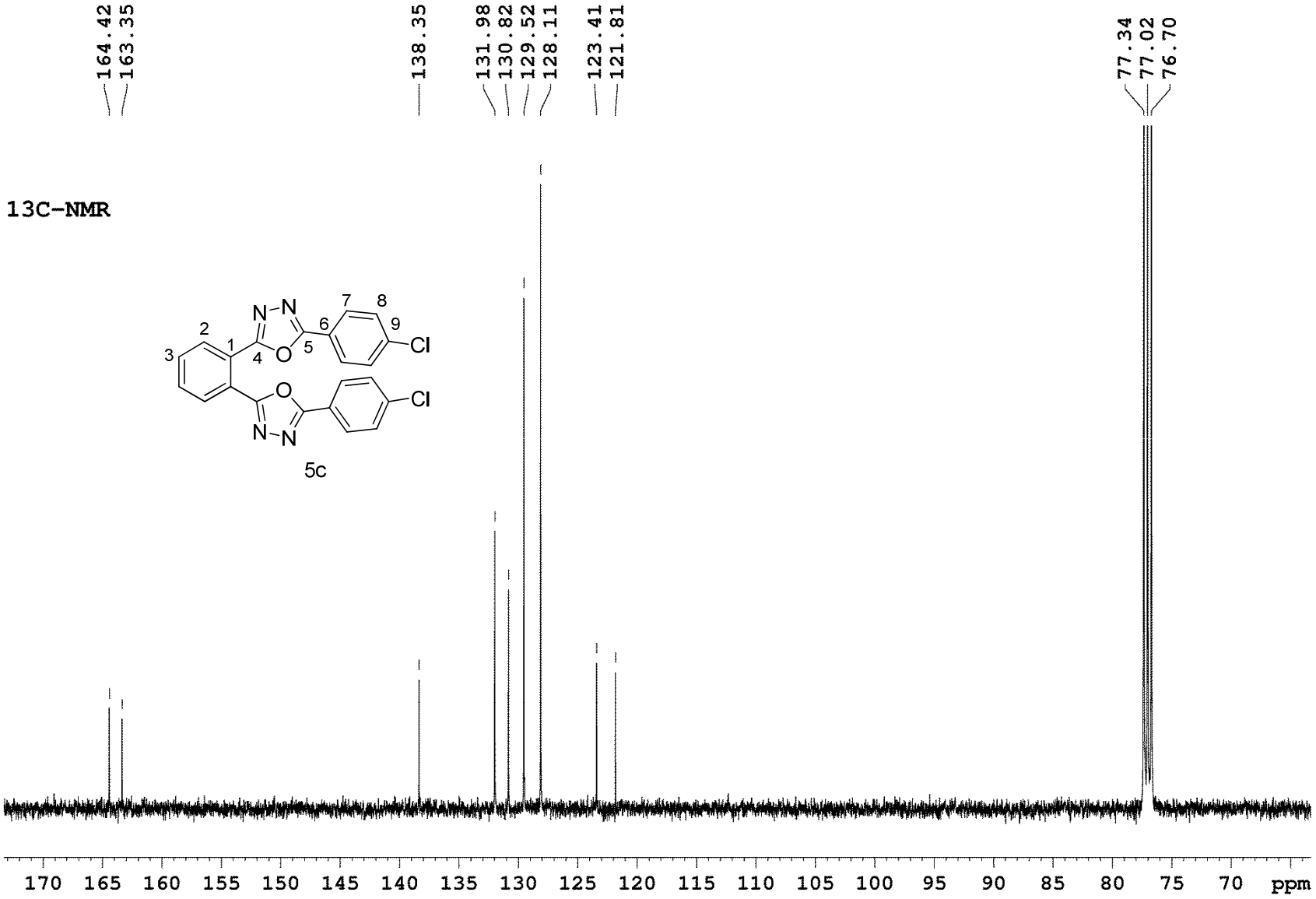


Figure S 66 APCI-HRMS spectrum of compound 5c

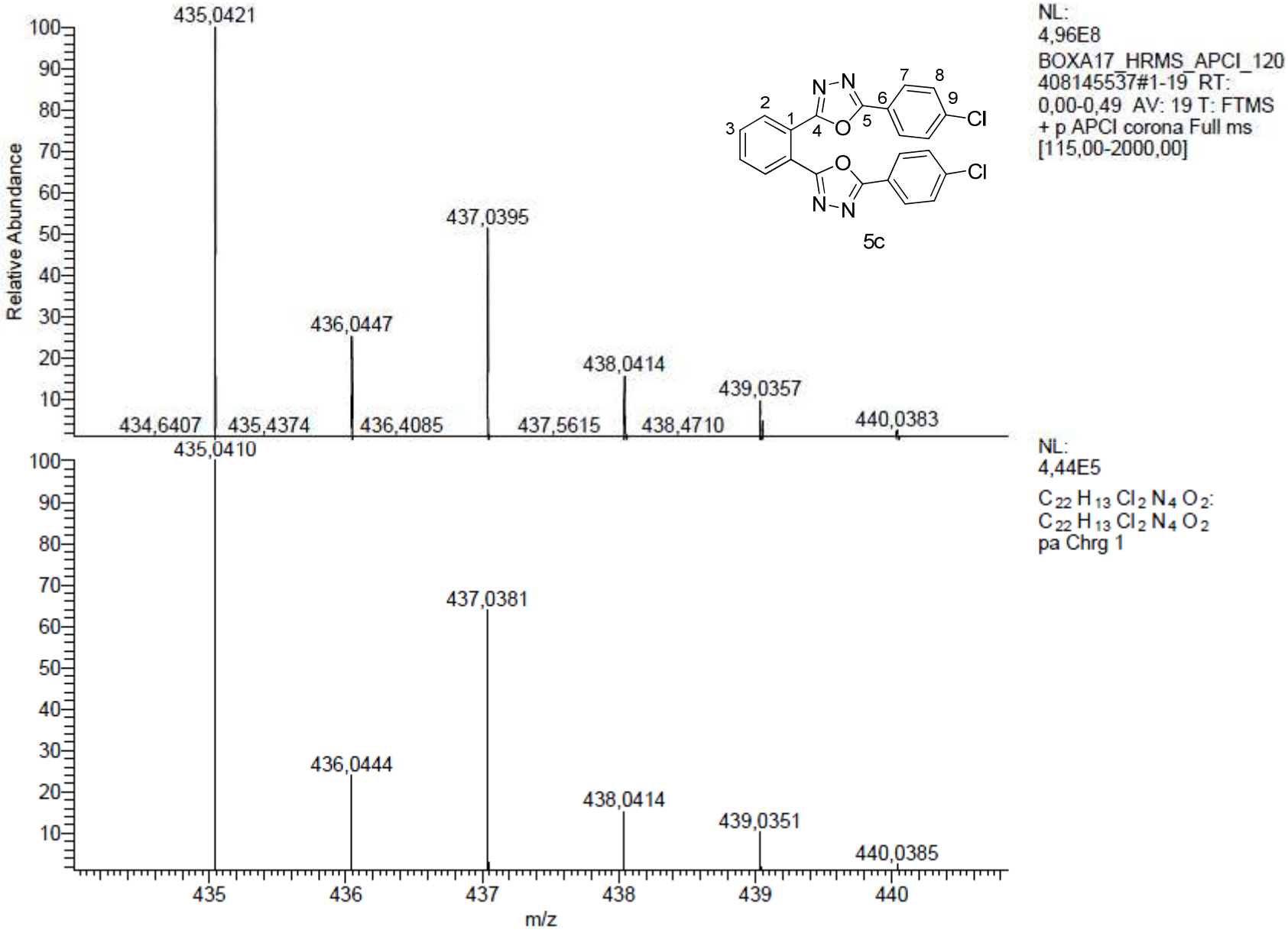


Figure S 67 ^{1}H NMR spectrum of compound 5d

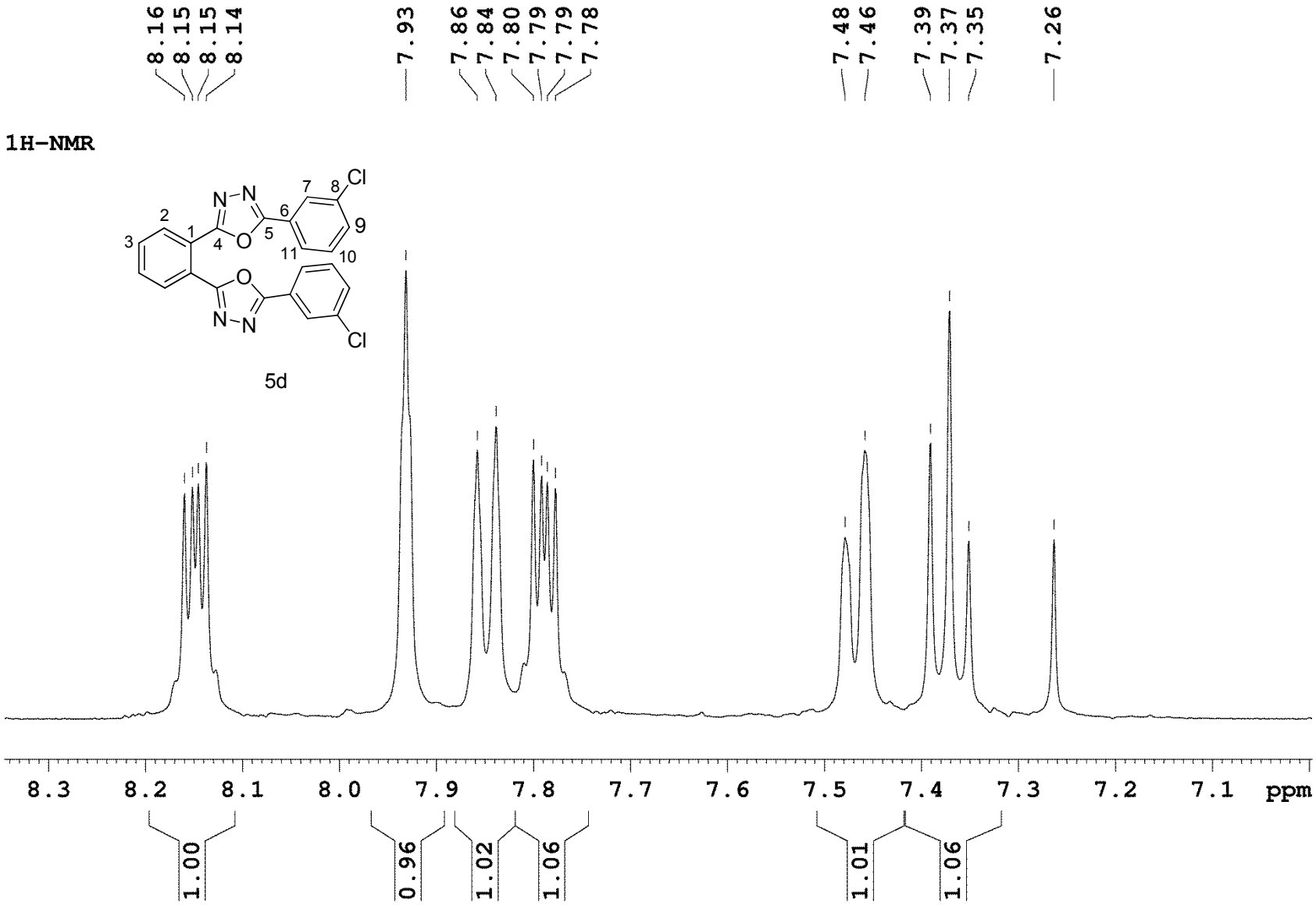


Figure S 68 ^{13}C NMR spectrum of compound 5d

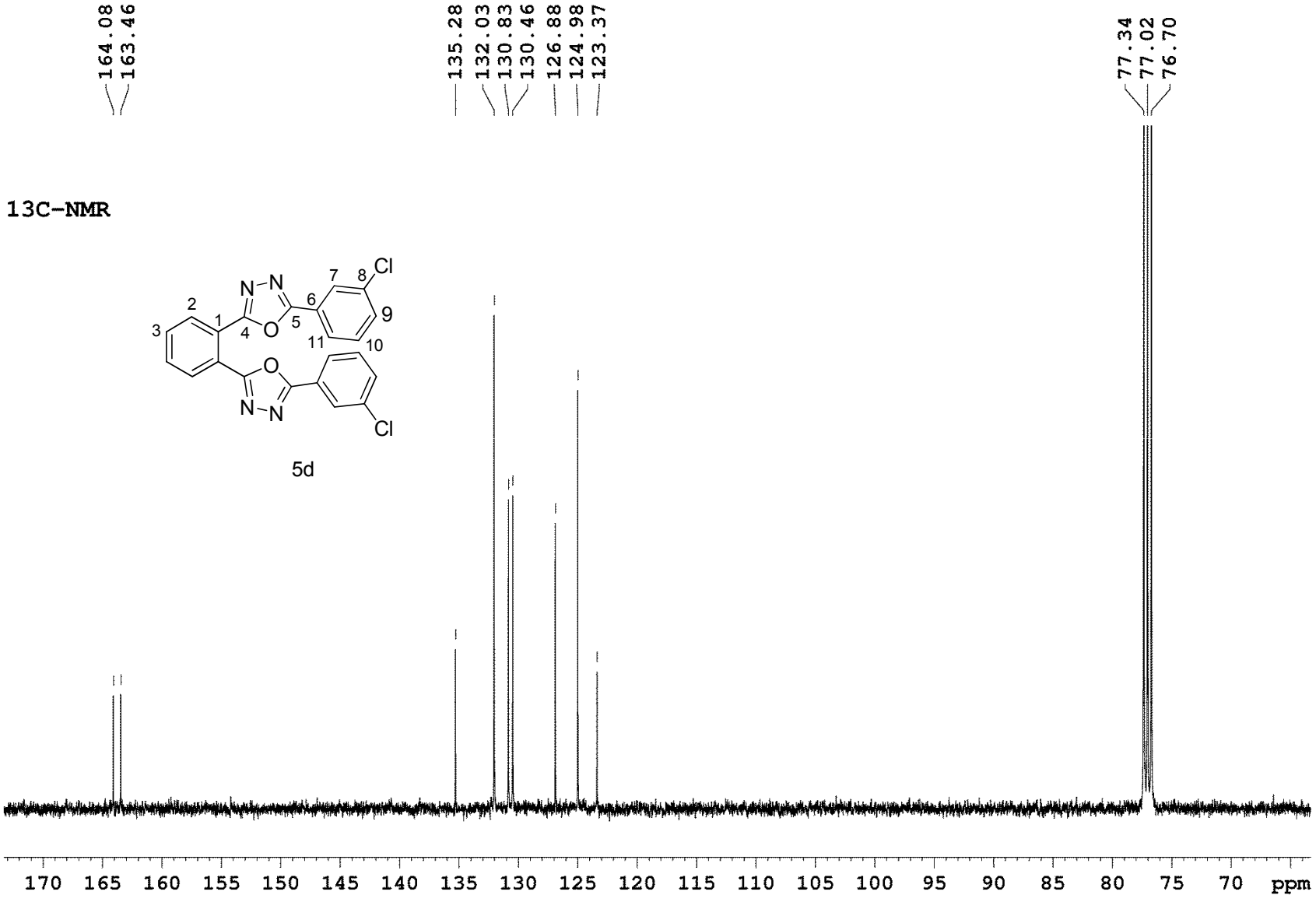
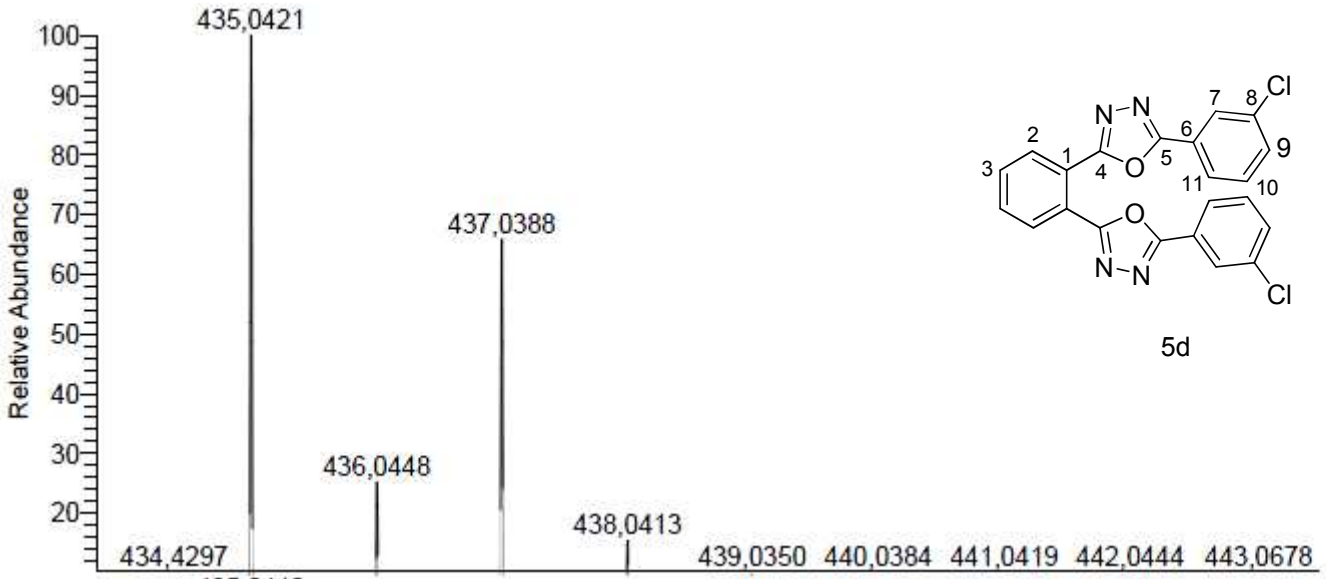


Figure S 69 APCI-HRMS spectrum of compound 5d



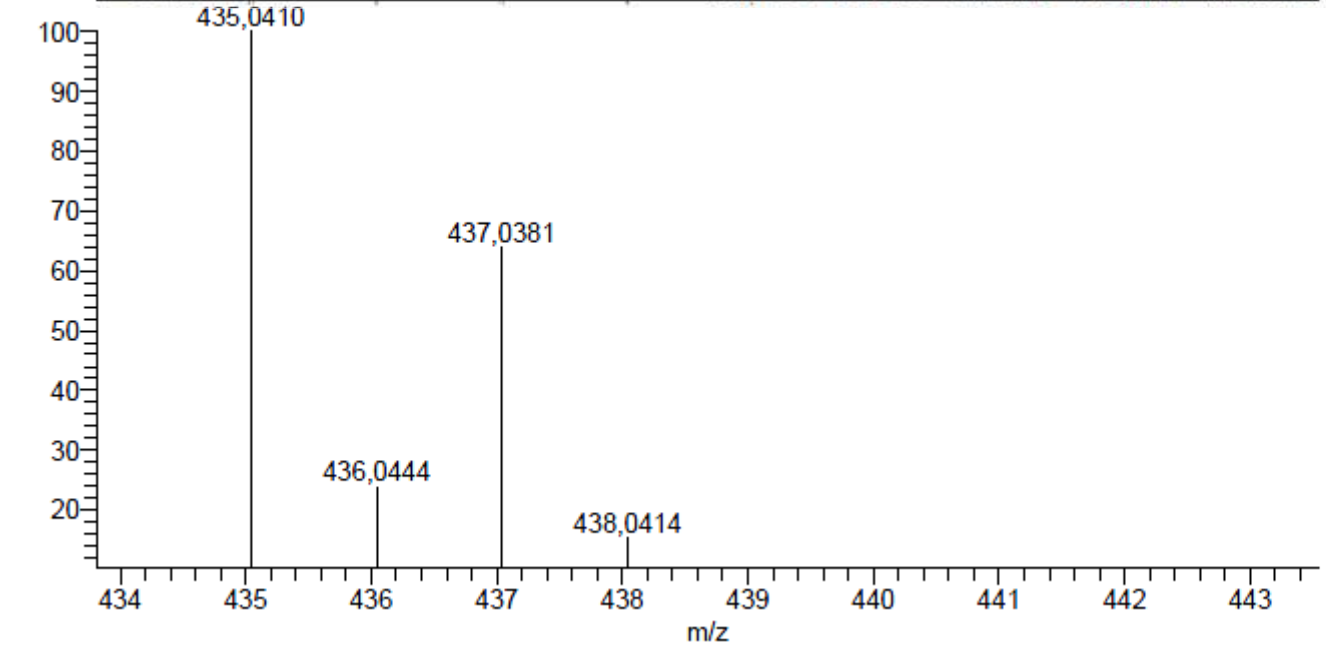


Figure S 70 ^{1}H NMR spectrum of compound 5e

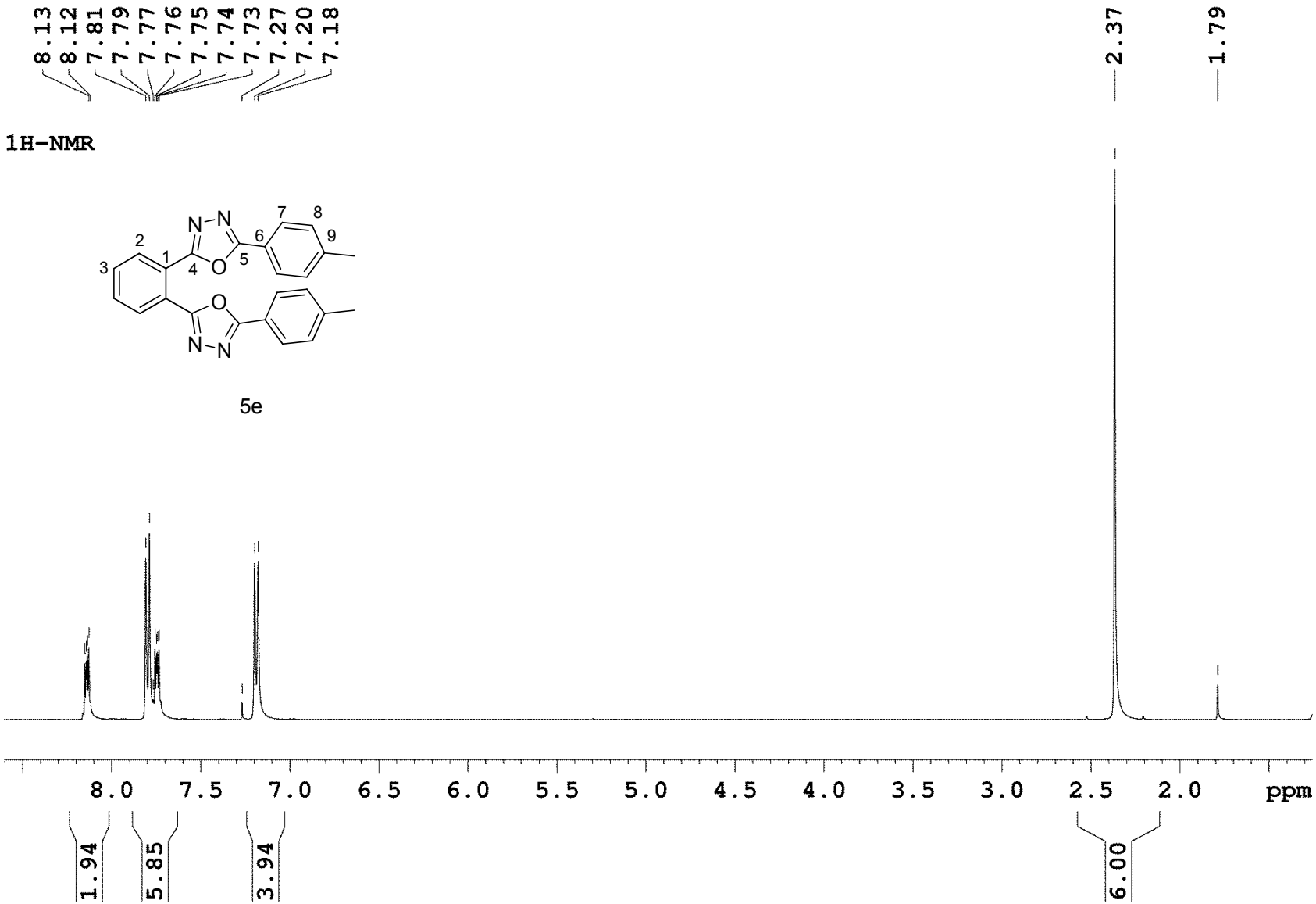


Figure S 71 ^{13}C NMR spectrum of compound 5e

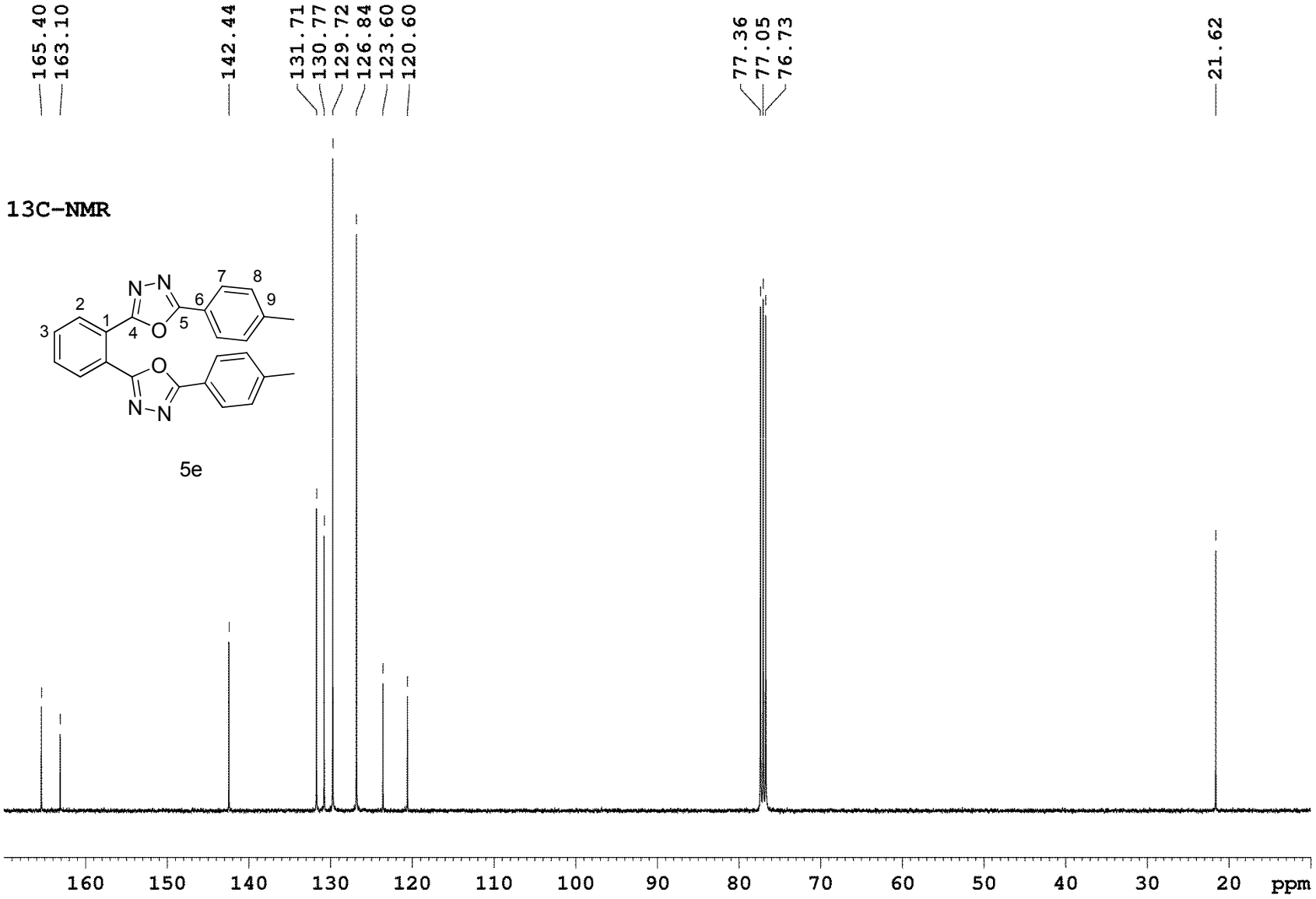


Figure S 72 APCI-HRMS spectrum of compound 5e

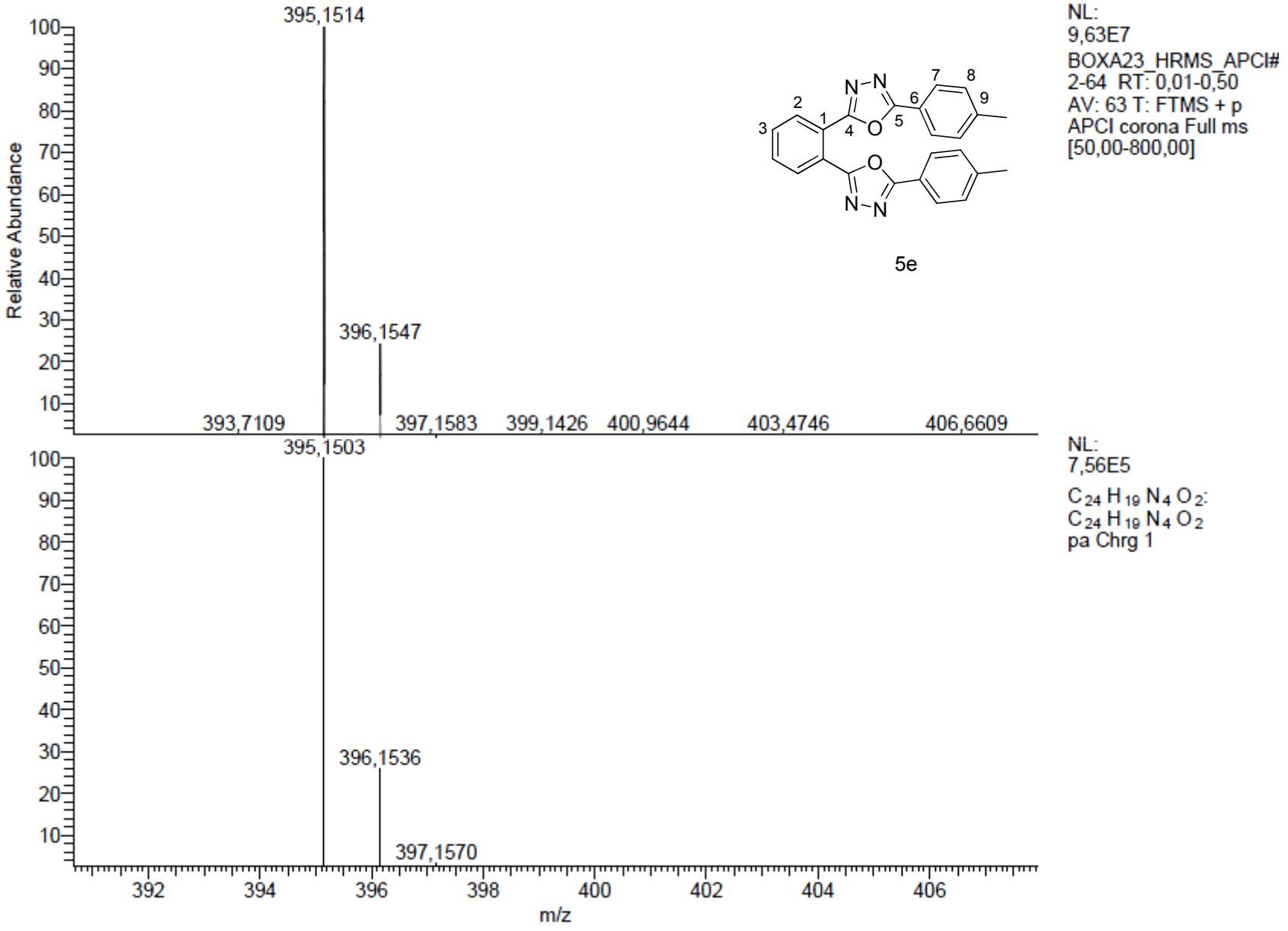


Figure S 73 ^{1}H NMR spectrum of compound 5f

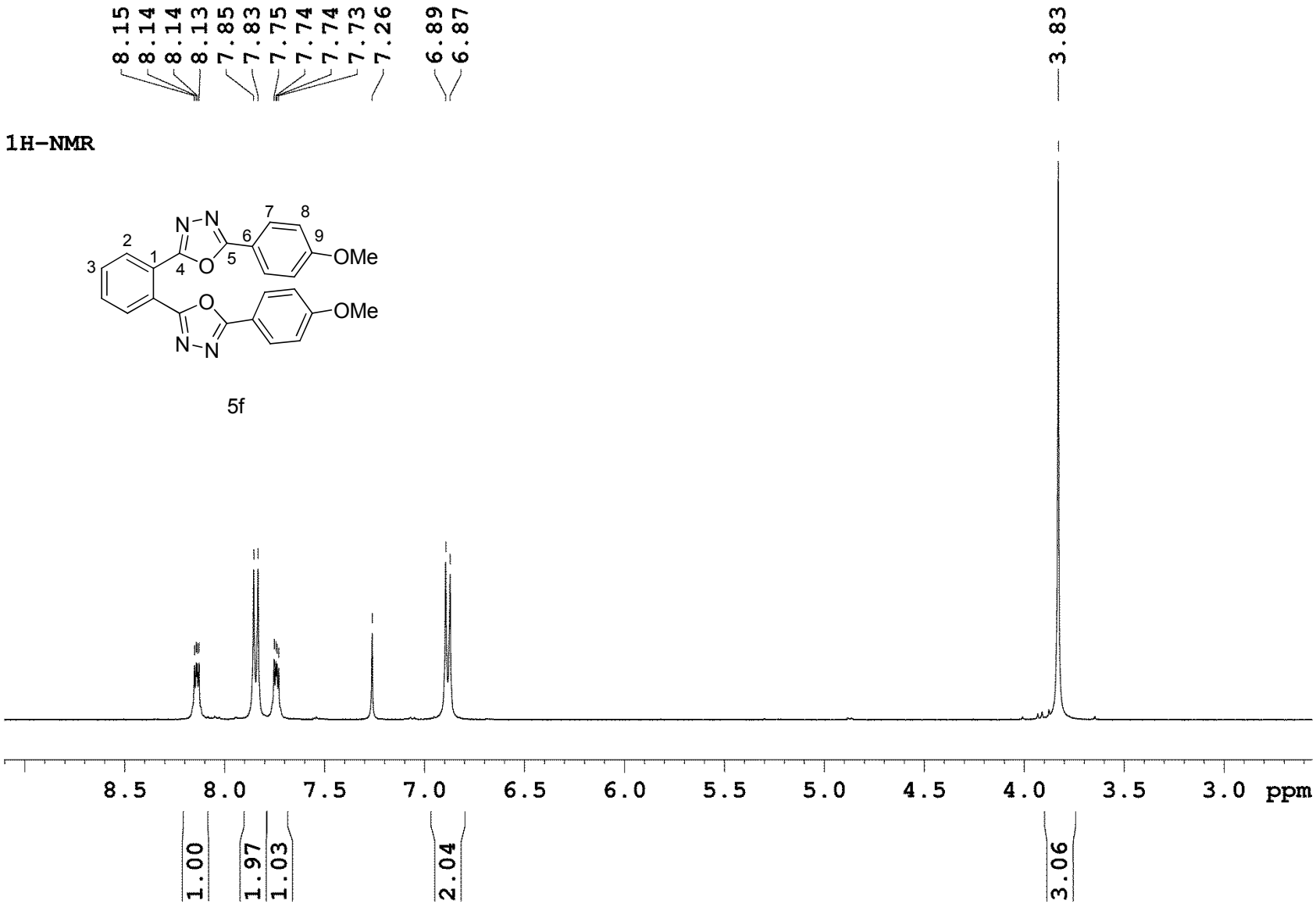


Figure S 74 ^{13}C NMR spectrum of compound 5f

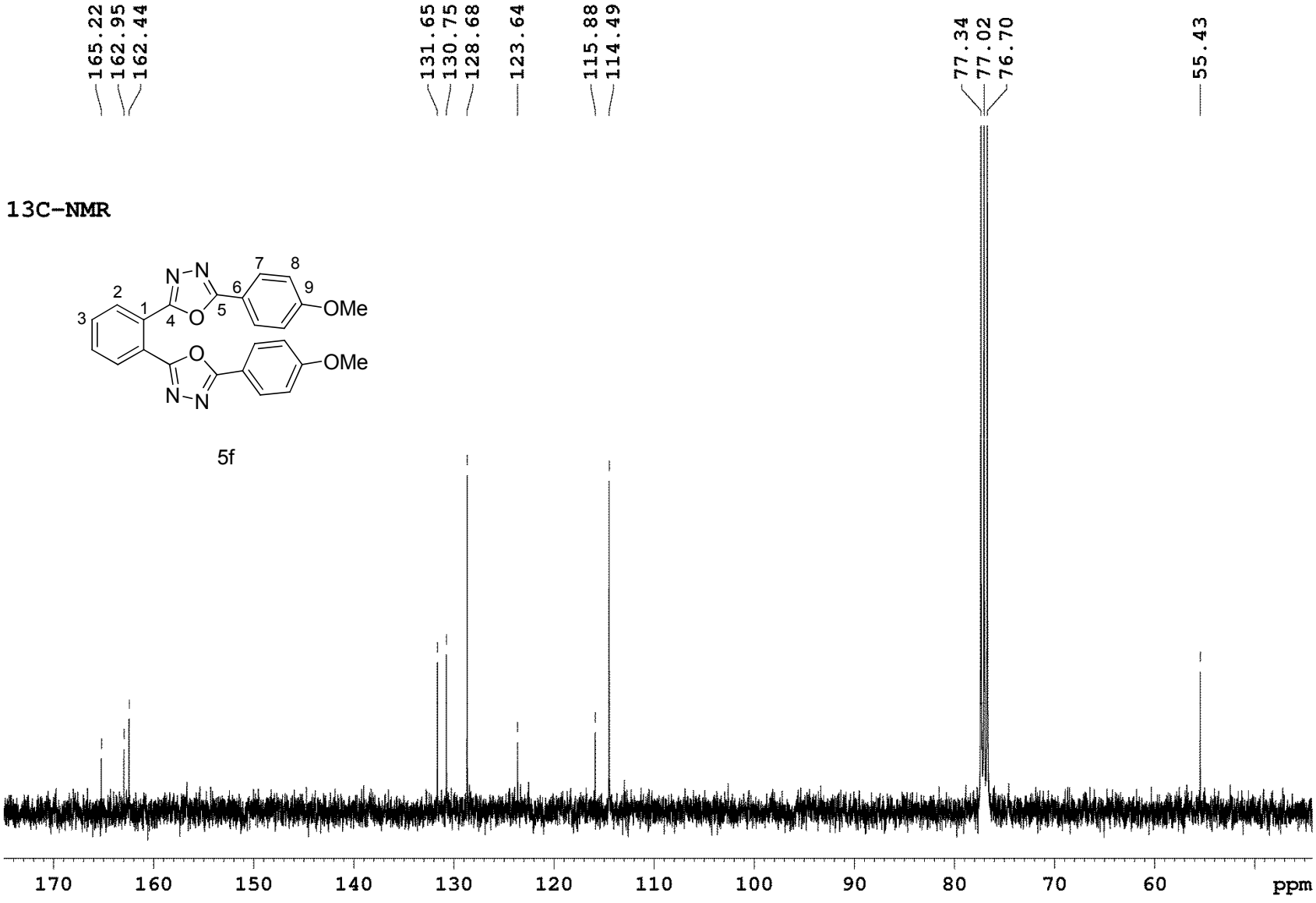


Figure S 75 APCI-HRMS spectrum of compound 5f

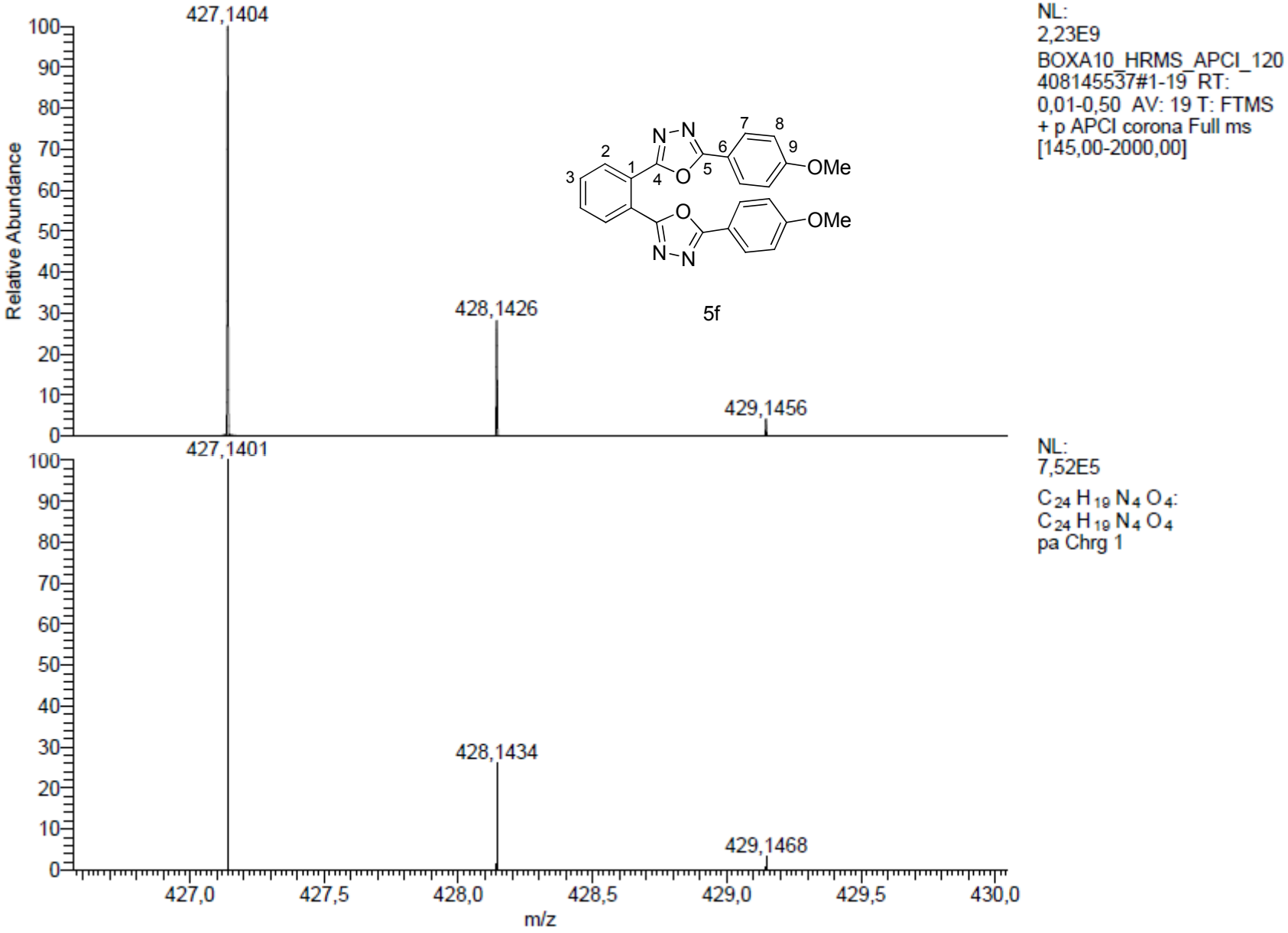


Figure S 76 ^{1}H NMR spectrum of compound 5g

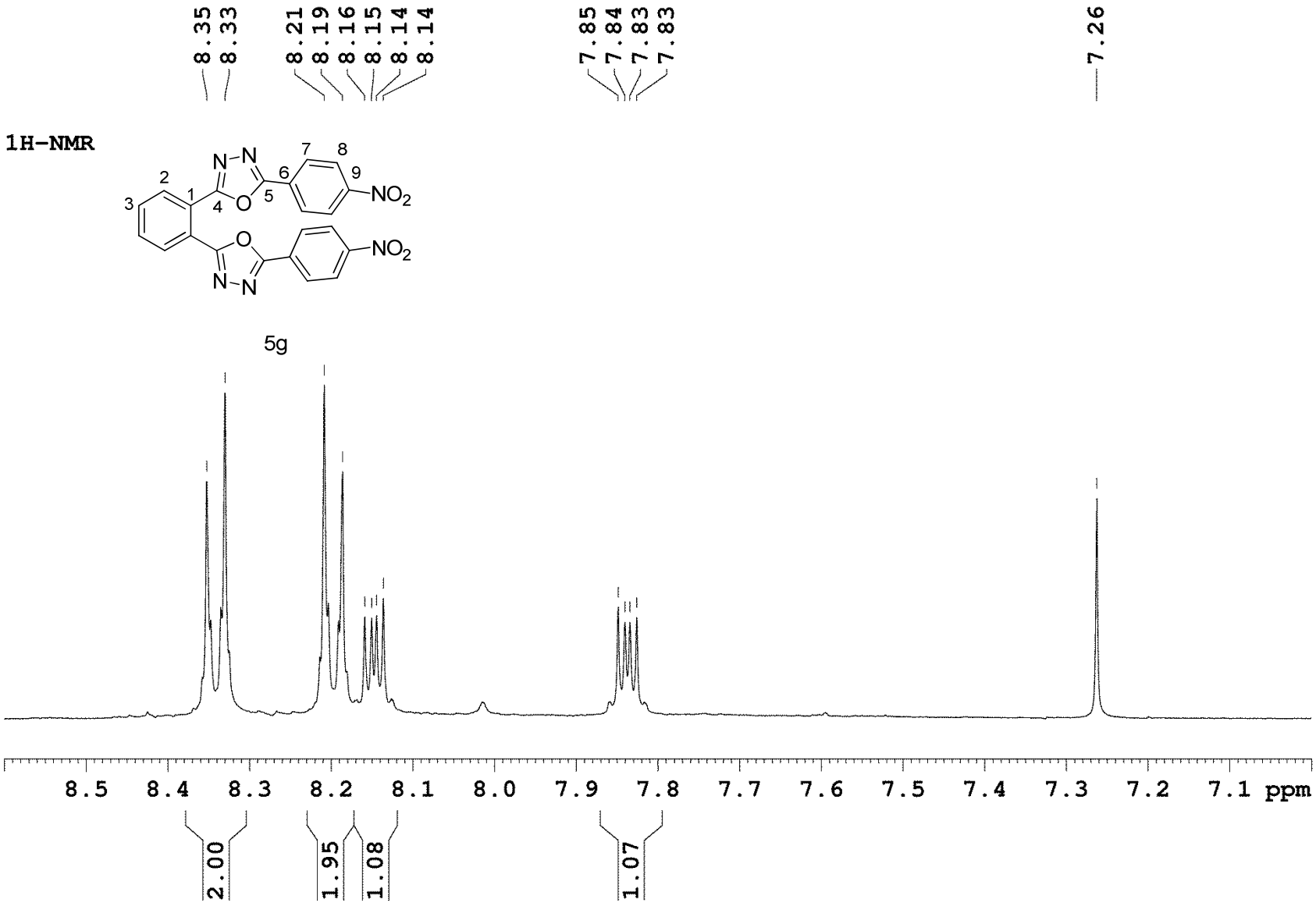


Figure S 77 ^{13}C NMR spectrum of compound 5g

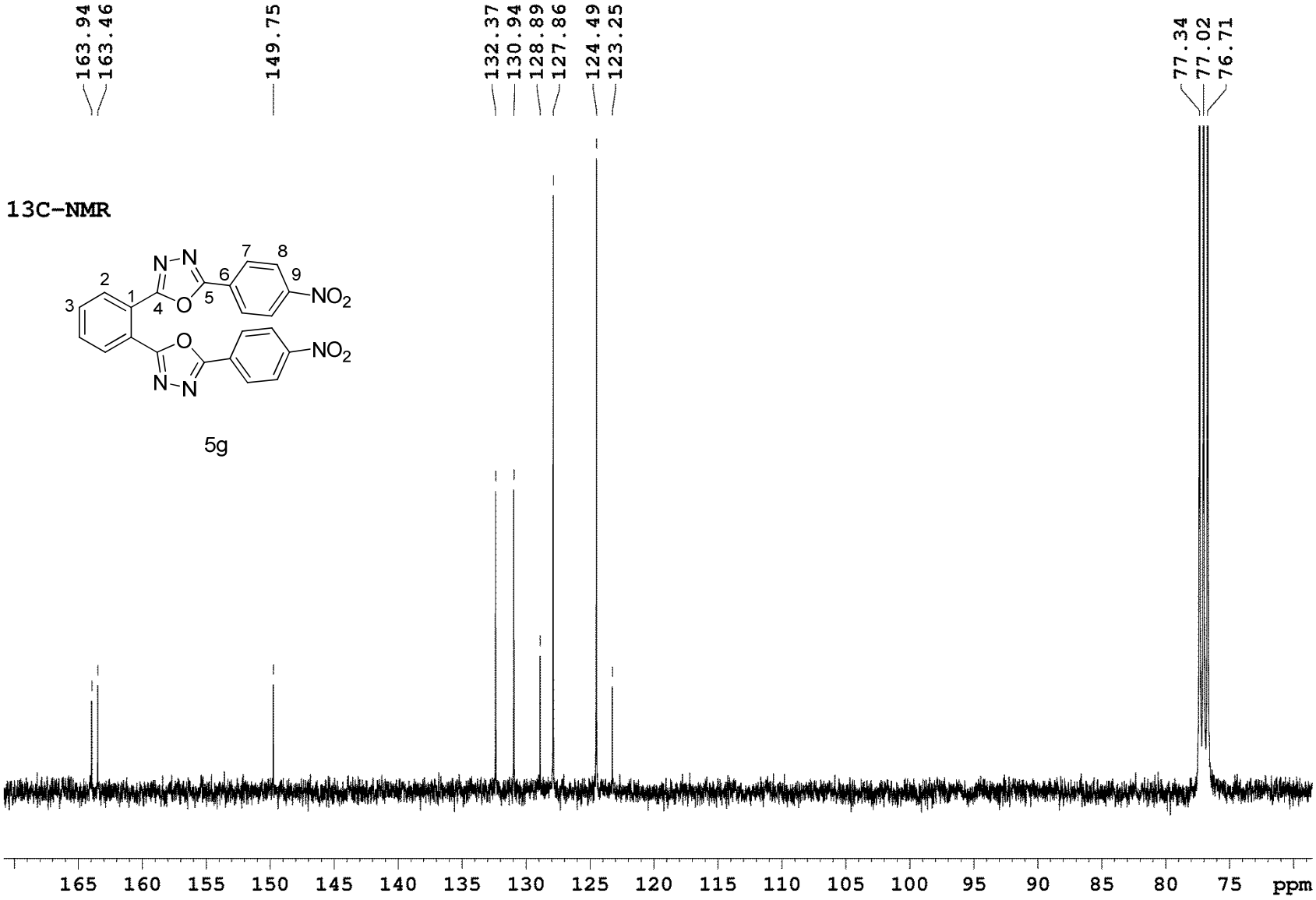


Figure S 78 APCI-HRMS spectrum of compound 5g

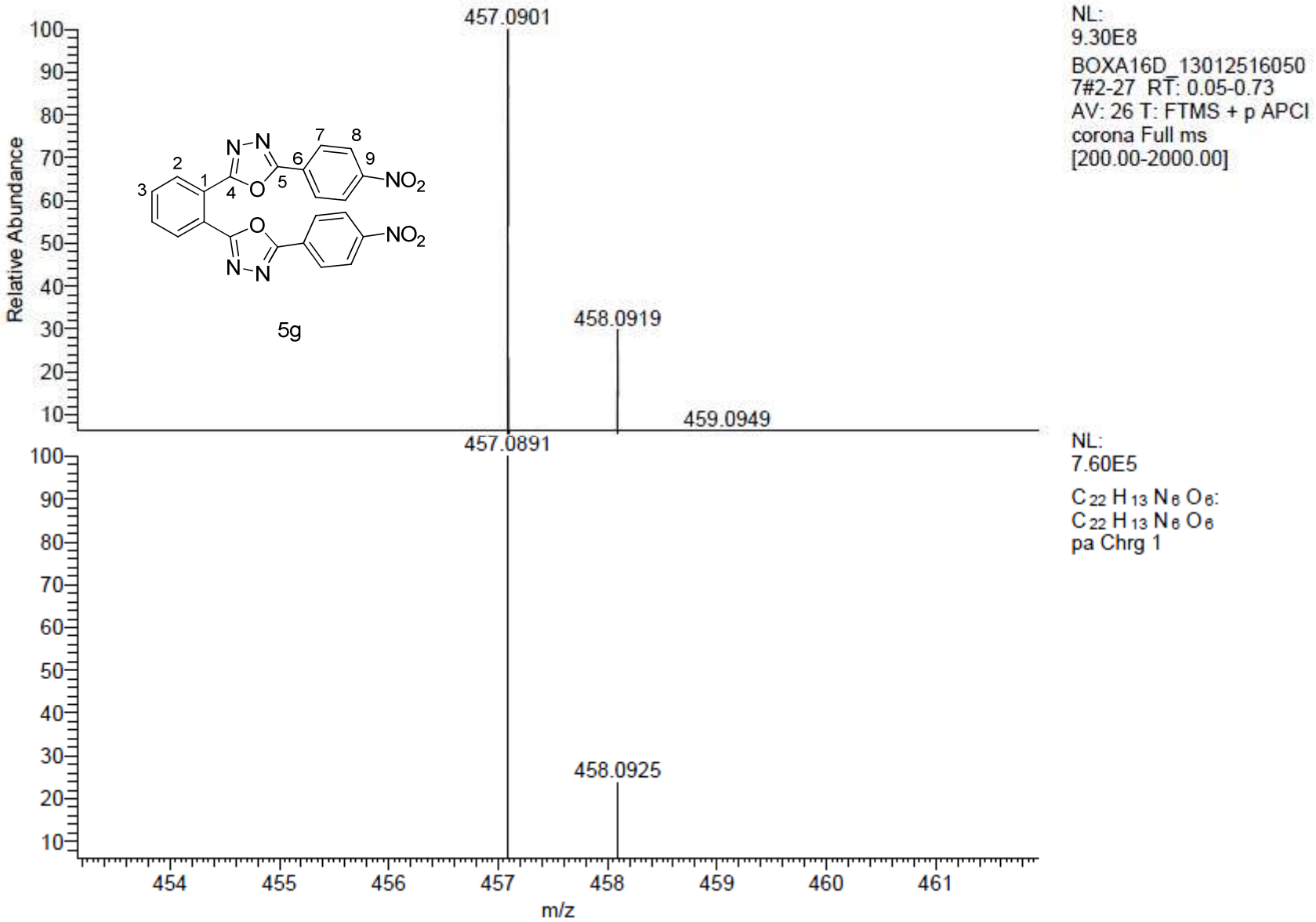


Figure S 79 ^{1}H NMR spectrum of compound 5h

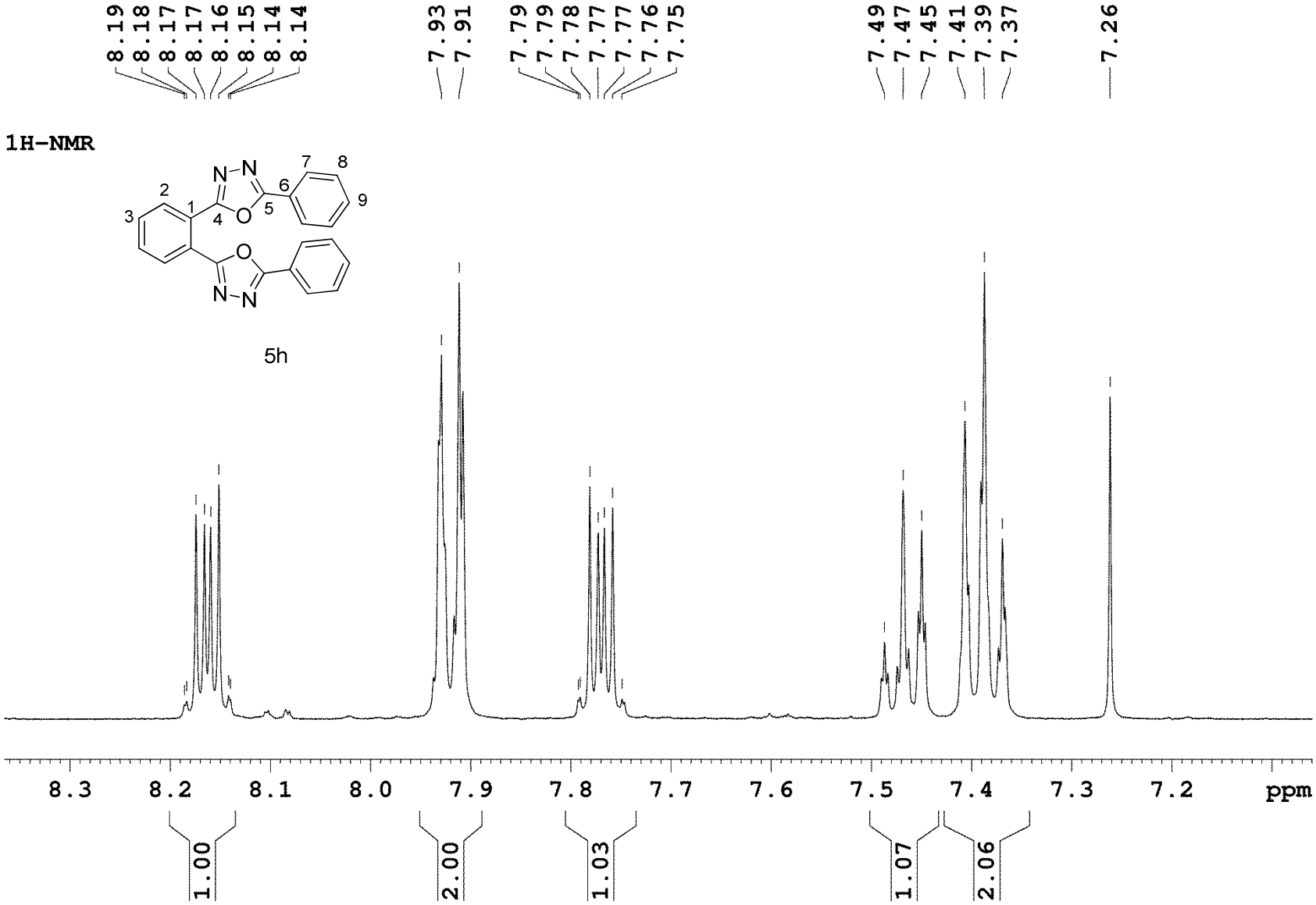


Figure S 80 ^{13}C NMR spectrum of compound 5h

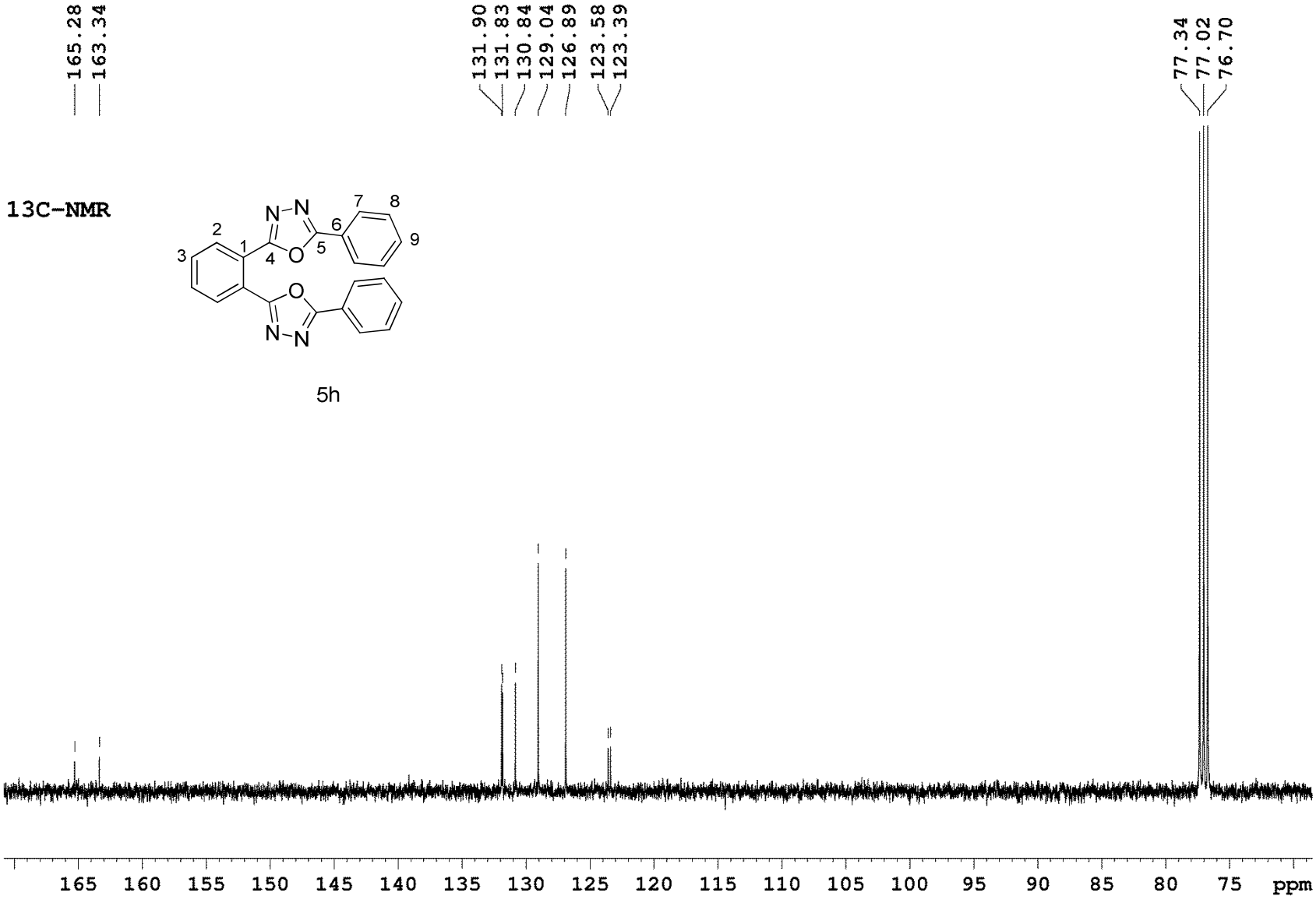


Figure S 81 APCI-HRMS spectrum of compound 5h

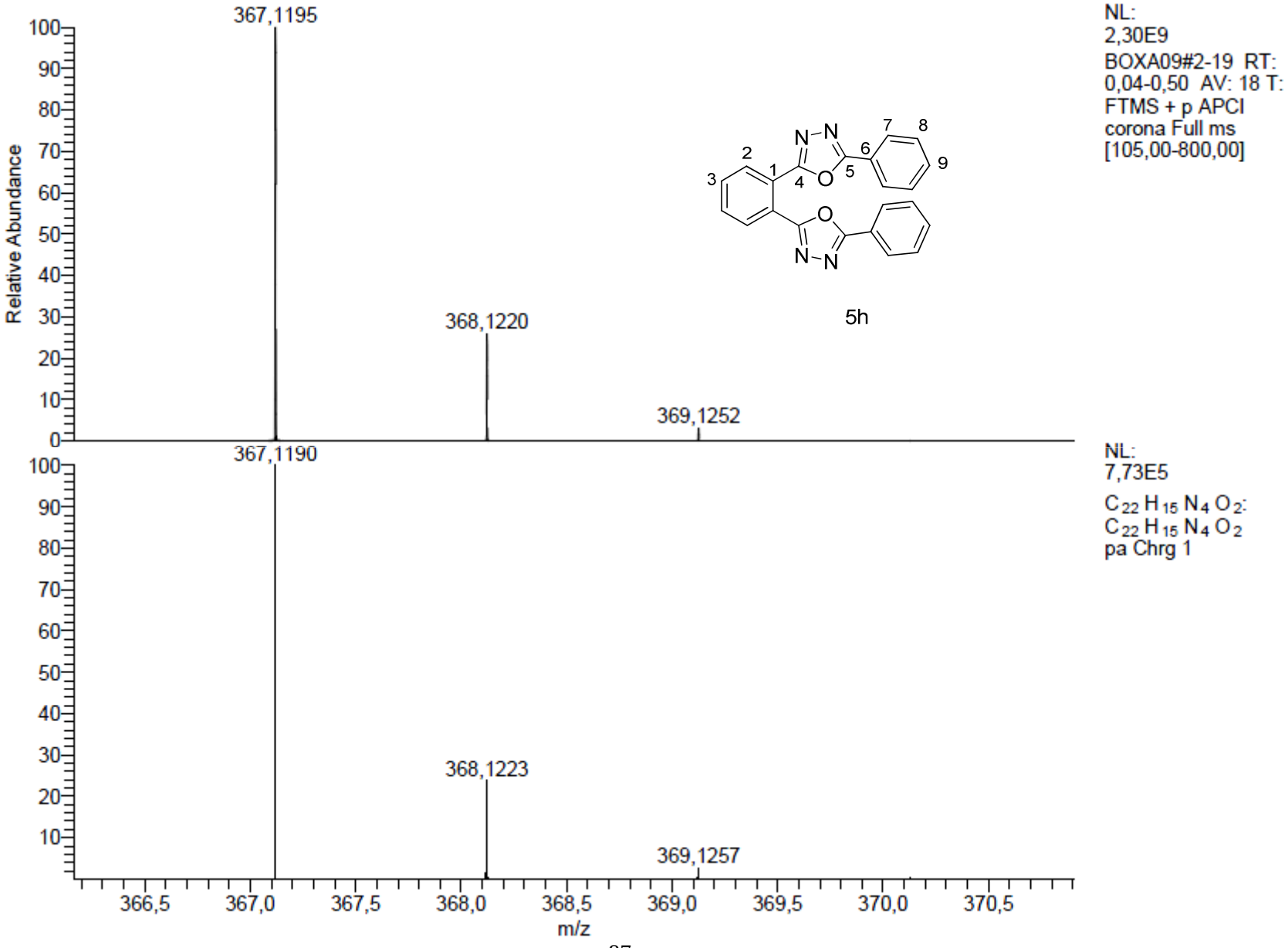


Figure S 82 ^{1}H NMR spectrum of compound 5i

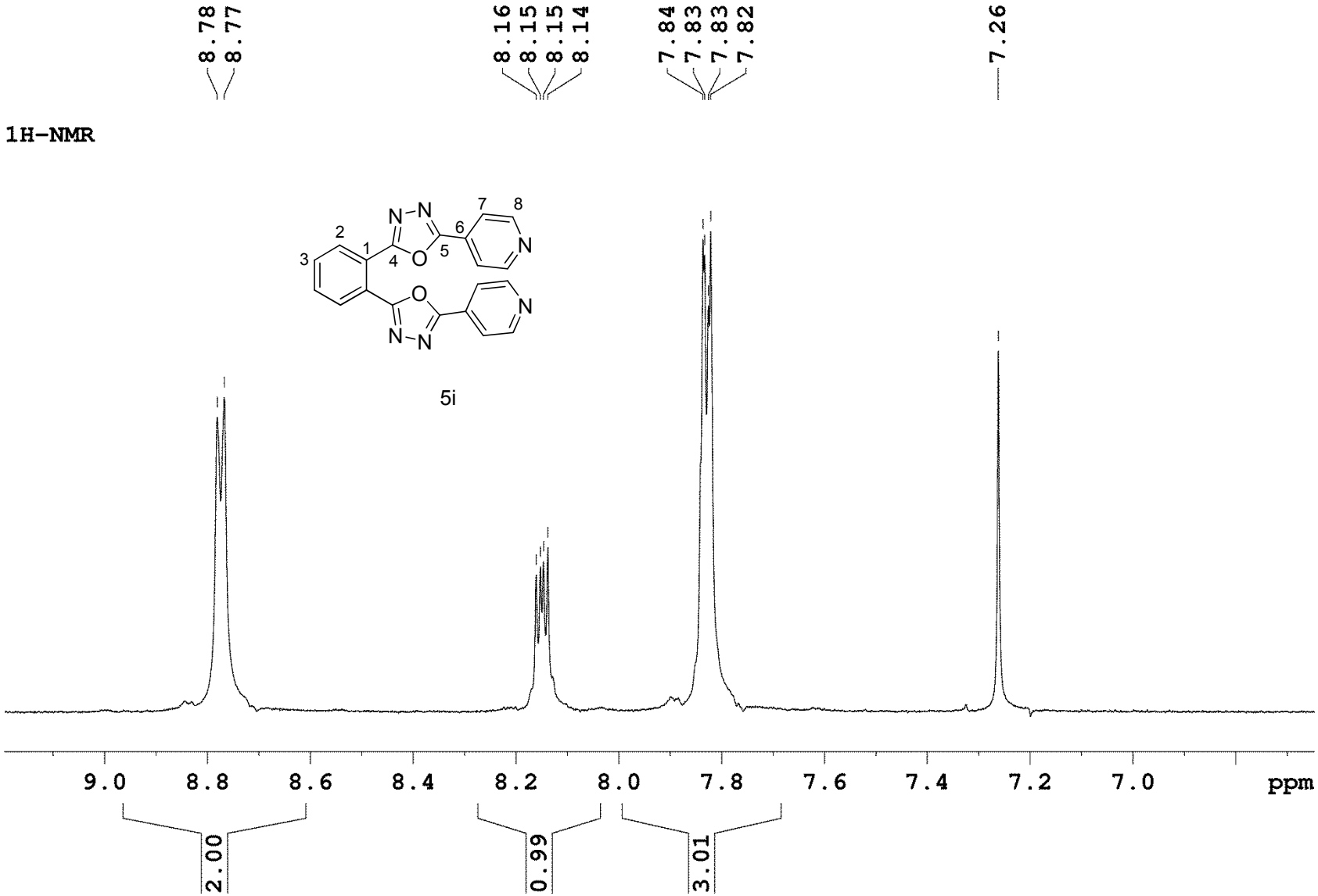


Figure S 83 ^{13}C NMR spectrum of compound 5i

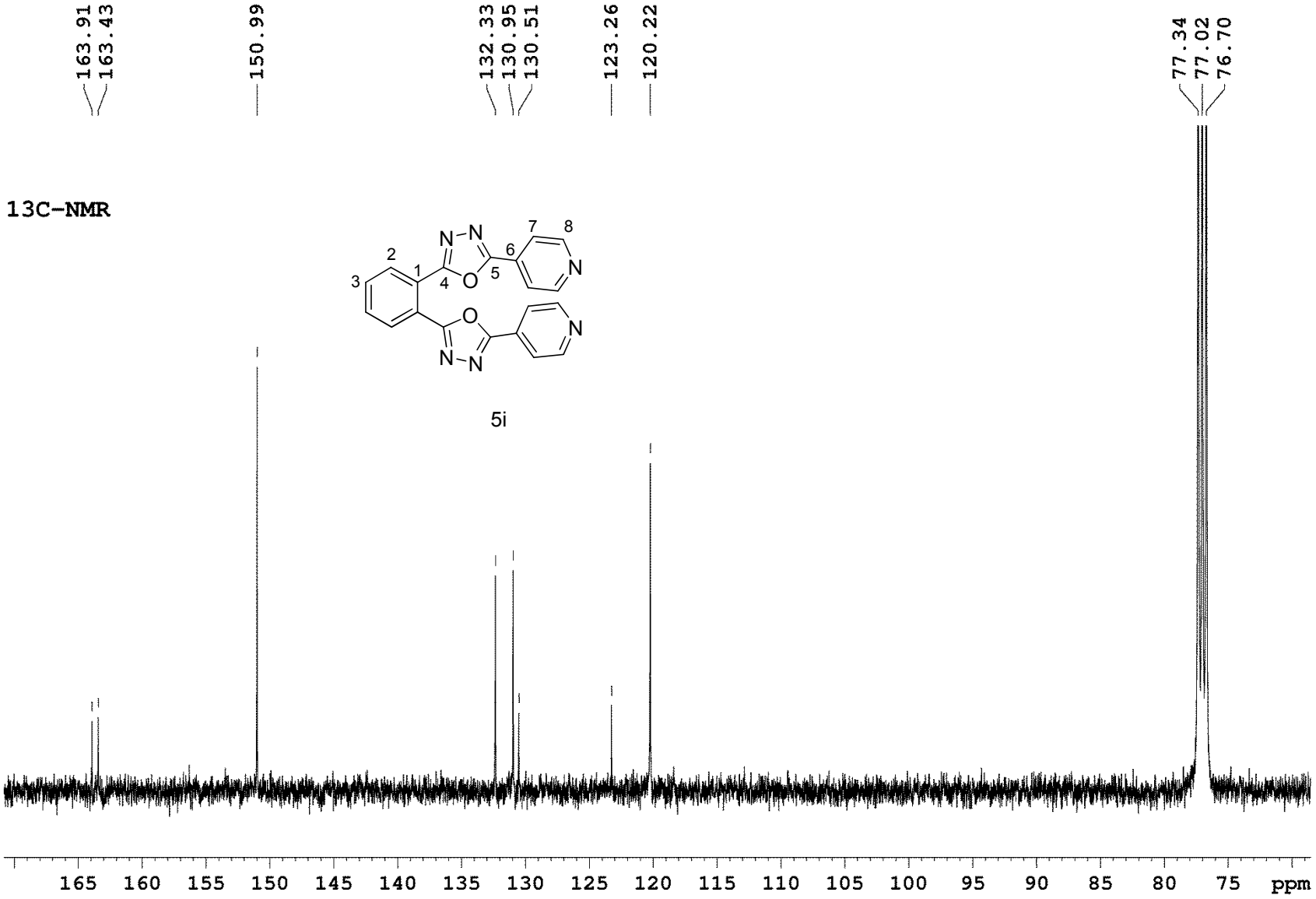


Figure S 84 APCI-HRMS spectrum of compound 5i