General considerations:

Reagent information: All reactions were carried out under air atmosphere in screw cap reaction tubes. All the solvents were bought from Sigma-Aldrich and Merck in sealed bottles and were used as received. Copper iodide was obtained from S.D. Fine-Chem Limited. Anilines were purchased from Aldrich and Alfa-Aesar. For column chromatography, silica (100–200 mesh) from SRL. A gradient elution using pet–ether and ethyl acetate were performed, based on Merck aluminum TLC sheets (silica gel 60 F_{254}).

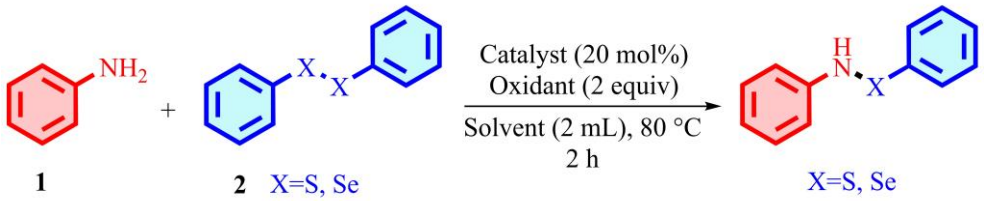
Analytical Information: All isolated compounds were characterized by ^{1}H NMR, ^{13}C NMR spectroscopy, and mass spectrometry (MS). The supporting information can include copies of the 1H NMR and 13C NMR. Unless otherwise stated, all Nuclear Magnetic Resonance spectra were recorded on a Bruker 400 MHz instrument. All ^{1}H NMR experiments were reported in units, parts per million (ppm), and were measured relative to the signals for residual chloroform (7.26 ppm) and DMSO (2.50 ppm) in the deuterated solvent unless otherwise stated. All ^{13}C NMR spectra were reported in ppm relative to deuterochloroform (77.23 ppm) and deuterated DMSO (40.2 ppm). Various temperature single crystal X-ray diffraction data for 3m was collected on Bruker Smart Apex Duo diffractometer using CuKα radiation (λ = 1.5406 Å). Using Olex2 [1], the structure was solved with the olex2.solve structure solution program using Charge Flipping and refined with the XL[2] refinement package using Least Squares minimisation. All the non-hydrogen atoms were refined anisotropically[3]. The structural parameters were retrieved by using mercury software.

[1] Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), *J. Appl. Cryst*. **42**, 339-341.

[2] G. M. Sheldrick, Acta Crystallogr. A: Found. Adv. 2015, **71**, 3-8.

[3] A. L. Spek, Acta Crystallogr. D Biol. Crystallogr. 2009, **65**, 148-155.

Table 1: Optimization of reaction conditions



Entry	Catalyst	Oxidant	Solvent	Time (h)	Isolated Yield
1	CuCl	H_2O_2	MeCN	2	45%
2	CuBr	H_2O_2	MeCN	2	57%
3	CuI	H_2O_2	MeCN	2	80%
4	$Cu(OAc)_2$. H_2O	H_2O_2	MeCN	2	n.d
5	$CuBr_2$	H_2O_2	MeCN	2	n.d
6	$CuCl_2$. $2H_2O$	H_2O_2	MeCN	2	n.d
7	$Cu(OTf)_2$	H_2O_2	MeCN	2	n.d
8	$CuSO_4$. $5H_2O$	H_2O_2	MeCN	2	n.d
9	CuPc	H_2O_2	MeCN	2	n.d
10	CuO	H_2O_2	MeCN	2	n.d
11	$Fe(OAc)_2$	H_2O_2	MeCN	2	n.d
12	$FeCl_3$	H_2O_2	MeCN	2	n.d
13	TBAI	H_2O_2	MeCN	2	50%
14	TBAB	H_2O_2	MeCN	2	n.d
15	I_2	H_2O_2	MeCN	2	n.d
16	CuI	TBHP	MeCN	2	n.d
17	CuI	DTBP	MeCN	2	65%
18	CuI	Bz_2O_2	MeCN	2	n.d
19	CuI	$K_2S_2O_8$	MeCN	2	n.d
20	CuI	$(NH_4)_2S_2O_8$	MeCN	2	n.d
21	CuI	Cumene hydroperoxide	MeCN	2	n.d
22	CuI	Tert-butyl per benzoate	MeCN	2	n.d
23	CuI	Oxone	MeCN	2	n.d
24	CuI	H_2O_2	Toluene	2	n.d
25	CuI	H_2O_2	DMSO	2	30%
26	CuI	H_2O_2	DMF	2	n.d
27	CuI	H_2O_2	DMA	2	25%
28	CuI	H_2O_2	NMP	2	n.d
29	CuI	H_2O_2	*t*-AmOH	2	n.d
30	CuI	H_2O_2	H_2O	2	n.d
31	CuI	H_2O_2	DCE	2	n.d
32	CuI	H_2O_2	1,4 Dioxane	2	n.d
33	CuI	H_2O_2	THF	2	n.d
34	-	H_2O_2	MeCN	2	n.d
35	CuI	-	MeCN	2	n.d
36	-	-	MeCN	2	n.d

Table 4: Catalyst Amount Screening

Entry	Catalyst Amount	Time (h)	Isolated Yield
1	5 mol%	2	25%
2	10 mol%	2	40%

Table 5: Temperature Screening

Entry	Temperature (°C)	Time (h)	Isolated Yield
1	rt	2	7%
2	50	2	55%
3	60	2	62%
4	70	2	71%
5	80	2	80%

General synthesis procedure for N-S/N-Se bond formation:

A clean, oven-dried screw cap reaction tube with a previously placed magnetic stir bar was charged with aniline (0.2 mmol), 1,2 diphenyl disulfide (0.1 mmol), or diphenyl diselenide (0.1 mmol), copper iodide (20 mol %) and H_2O_2 (2 equiv) followed by addition of MeCN (2 mL). The reaction mixture was vigorously stirred for 2 h in a preheated oil bath at 80 °C. After the stipulated time, the reaction mixture was cooled to room temperature and filtered through a celite bed using ethyl acetate as the eluent (25 mL). The ethyl acetate layer was dried under a vacuum. The crude reaction mixture was purified by column chromatography using silica gel and petroleum-ether/ethyl acetate as the eluent to give the desired product.

CRYSTAL DATA

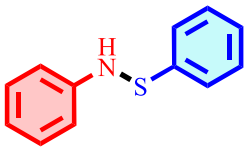
TRY_a

Table 1 Crystal data and structure refinement for TRY_a.

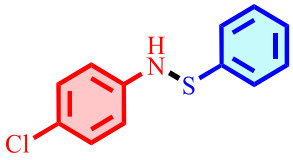
Identification code	TRY_a
Empirical formula	$C_{12}H_{10}N_2O_2S$
Formula weight	246.291
Temperature/K	150.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	8.244(3)
b/Å	20.111(7)
c/Å	7.539(2)
α/°	90
β/°	113.061(15)
γ/°	90
Volume/Å^3	1150.2(6)
Z	4
ρ_{calc}g/cm^3	1.422
μ/mm^{-1}	2.438
F(000)	515.0

Crystal size/mm^3	0.421 × 0.384 × 0.215
Radiation	Cu Kα (λ = 1.54178)
2Θ range for data collection/°	8.8 to 133.02
Index ranges	$-9 \leq h \leq 9$, $-23 \leq k \leq 23$, $-8 \leq l \leq 8$
Reflections collected	6376
Independent reflections	1990 [R_{int} = 0.1523, R_{sigma} = 0.1441]
Data/restraints/parameters	1990/0/130
Goodness-of-fit on F^2	1.092
Final R indexes [I>=2σ (I)]	R_1 = 0.1145, wR_2 = 0.1807
Final R indexes [all data]	R_1 = 0.2053, wR_2 = 0.2219
Largest diff. peak/hole / e $Å^{-3}$	1.08/-0.98

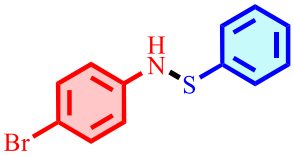
Characterization data of synthesized compounds:



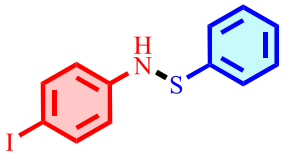
***N, S*-diphenylthiohydroxylamine (Table 2, Entry 3a)**: eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (32 mg, isolated yield: 80%); **^{1}H NMR (400 MHz, $CDCl_3$)** δ 3.70 (s, 1H), 6.56 (d, *J* = 8.5 Hz, 2H), 7.06 – 7.14 (m, 4H), 7.21 (d, *J* = 6.8 Hz, 2H), 7.33 (d, *J* = 8.5 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 146.88, 137.19, 134.15, 130.11, 129.08, 126.06, 116.39, 116.06. **HRMS (ESI)** calcd. for $C_{12}H_{12}NS$ ([M+H]+) is 202.0690 and found 202.0681.



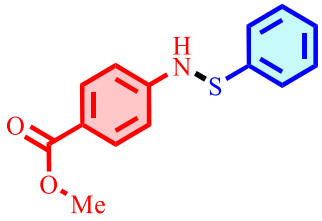
***N*-(4-chlorophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3b)**: eluted in (1% ethyl acetate/petroleum ether, v/v); dark brown liquid (37 mg, isolated yield: 78%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.60 (d, *J* = 8.7 Hz, 1H), 7.10 (d, *J* = 8.7 Hz, 1H), 7.18 – 7.26 (m, 2H), 7.27 – 7.33 (m, 3H), 7.47 – 7.52 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.21, 155.82, 142.85, 137.86, 128.37, 119.58, 119.50, 115.76, 115.01, 114.78. **HRMS (ESI)** calcd. for $C_{12}H_{11}ClNS$ ([M+H]+) is 236.0300 and found 236.0302.



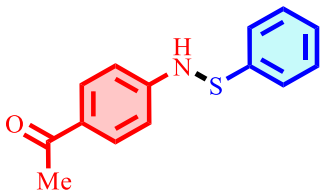
***N*-(4-bromophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3c):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (43 mg, isolated yield: 76%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.59 (s, 1H), 6.82 – 6.91 (m, 3H), 6.97 (d, *J* = 7.6 Hz, 2H), 7.20 (dd, *J* = 7.2, 8.5 Hz, 2H), 7.26 (d, *J* = 8.8 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 112.63, 118.31, 119.04, 121.68, 129.49, 132.21, 142.42, 142.45. **HRMS (ESI)** calcd. for $C_{12}H_{11}BrNS$ ([M+H]+) is 279.9795 and found 279.9750.



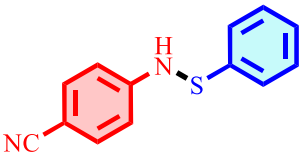
***N*-(4-iodophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3d):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (50 mg, isolated yield: 77%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.20 (s, 1H), 6.82 (d, *J* = 8.8 Hz, 2H), 7.12 – 7.19 (m, 3H), 7.28 (dd, *J* = 7.2, 8.2 Hz, 2H), 7.48 (d, *J* = 8.7 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.62, 140.73, 137.99, 129.02, 127.53, 125.80, 122.58, 116.93, 82.17. **HRMS (ESI)** calcd. for $C_{12}H_{11}INS$ ([M+H]+) is 327.9656 and found 327.9651.



methyl 4-((phenylthio)amino) benzoate (Table 2, Entry 3e): eluted in (7% ethyl acetate/petroleum ether, v/v); light brown solid (35 mg, isolated yield: 68%), Mp. 115-116 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.85 (s, 3H), 5.66 (s, 1H), 7.00 – 7.07 (m, 2H), 7.10 – 7.21 (m, 3H), 7.23 – 7.30 (m, 2H), 7.90 (d, *J* = 8.8 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.00, 151.19, 140.34, 131.40, 129.03, 125.90, 122.71, 122.12, 114.07, 51.79. **HRMS (ESI)** calcd. for $C_{14}H_{14}NO_2S$ ([M+H]+) is 260.0745 and found 260.0743.

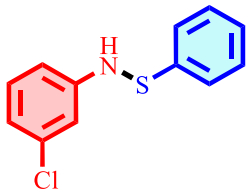


1-(4-((phenylthio)amino)phenyl)ethan-1-one (Table 2, Entry 3f): eluted in (8% ethyl acetate/petroleum ether, v/v); brown solid (34 mg, isolated yield: 70%) Mp. 108-109 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.51 (s, 3H), 5.80 (s, 1H), 7.03 – 7.08 (m, 2H), 7.11 – 7.19 (m, 3H), 7.25 – 7.30 (m, 2H), 7.84 – 7.87 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.82, 151.51, 140.25, 130.54, 130.03, 129.47, 129.10, 126.00, 123.25, 122.80, 114.16, 26.29. **HRMS (ESI)** calcd. for $C_{14}H_{14}NOS$ ([M+H]+) is 244.0796 and found 244.0804.

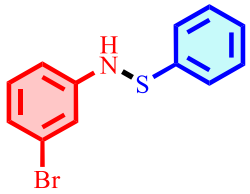


4-((phenylthio)amino) benzonitrile (Table 2, Entry 3g): eluted in (10% ethyl acetate/petroleum ether, v/v); brown solid (29 mg, isolated yield: 63%) Mp. 105-106 °C **^{1}H**

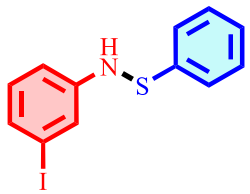
NMR (400 MHz, $CDCl_3$) δ 5.67 (s, 1H), 7.08 (d, *J* = 8.8 Hz, 2H), 7.15 – 7.19 (m, 3H), 7.28 – 7.33 (m, 2H), 7.49 (d, *J* = 8.9 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.90, 139.58, 133.72, 133.37, 129.17, 126.25, 123.75, 122.89, 119.55, 114.94. **HRMS (ESI)** calcd. for $C_{13}H_{11}N_2S$ ([M+H]+) is 227.0642 and found 227.0641.



***N*-(3-chlorophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3h):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (35 mg, isolated yield: 74%) **^{1}H NMR (400 MHz, $CDCl_3$)** δ 5.14 (s, 1H), 6.79 (dd, *J* = 7.4, 21.9 Hz, 2H), 6.97 (t, *J* = 2.2, 2.2 Hz, 1H), 7.02 – 7.13 (m, 4H), 7.19 – 7.24 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.13, 140.72, 135.16, 130.34, 129.06, 125.84, 122.57, 120.67, 114.69, 113.00. **HRMS (ESI)** calcd. for $C_{12}H_{11}ClNS$ ([M+H]+) is 236.0300 and found 236.0302.

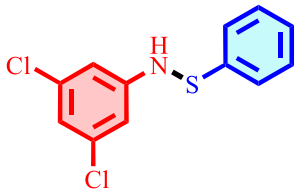


***N*-(3-bromophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3i):** eluted in (2% ethyl acetate/petroleum ether, v/v); yellow liquid (40 mg, isolated yield: 72%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.11 (s, 1H), 6.82 – 6.94 (m, 2H), 6.98 (t, *J* = 7.9, 7.9 Hz, 1H), 7.05 – 7.13 (m, 4H), 7.21 (t, *J* = 7.6, 7.6 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.27, 140.72, 130.64, 129.08, 125.86, 123.59, 122.61, 117.61, 113.43. **HRMS (ESI)** calcd. for $C_{12}H_{11}BrNS$ ([M+H]+) is 279.9795 and found 279.9750.

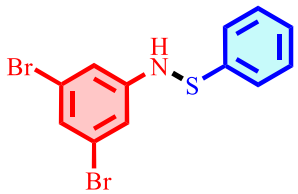


***N*-(3-iodophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3j):** eluted in (1% ethyl acetate/petroleum ether, v/v); light yellow liquid (47 mg, isolated yield: 72%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.10 (s, 1H), 6.85 (t, *J* = 7.9, 7.9 Hz, 1H), 6.93 (ddd, *J* = 1.0, 2.3, 8.2 Hz, 1H), 7.06 – 7.15 (m, 4H), 7.22 (dd, *J* = 7.1, 8.3 Hz, 3H), 7.31 (dd, *J* = 1.6, 2.3 Hz, 1H). **^{13}C NMR**

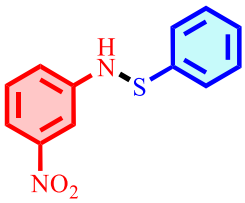
(101 MHz, $CDCl_3$) δ 144.04, 141.77, 129.85, 129.59, 122.36, 119.73, 119.03, 116.91, 113.19. **HRMS (ESI)** calcd. for $C_{12}H_{11}INS$ ([M+H]+) is 327.9656 and found 327.9651.



***N*-(3,5-dichlorophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3k):** eluted in (2% ethyl acetate/petroleum ether, v/v); brown liquid (35 mg, isolated yield: 66 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.28 (s, 1H), 6.85 (t, *J* = 1.8, 1.8 Hz, 1H), 6.93 (d, *J* = 1.8 Hz, 2H), 7.14 – 7.20 (m, 3H), 7.28 – 7.34 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.01, 139.98, 135.68, 129.17, 126.17, 122.80, 120.65, 113.22. **HRMS (ESI)** calcd. for $C_{12}H_{10}Cl_2NS$ ([M+H] +) is 269.9911 and found 269.9909.

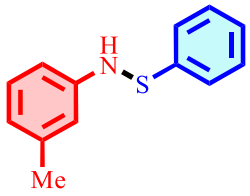


***N*-(3,5-dibromophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3l):** eluted in (2% ethyl acetate/petroleum ether, v/v); yellow liquid (49 mg, isolated yield: 68 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.26 (s, 1H), 6.24 (tt, *J* = 2.3, 2.3, 9.0, 9.0 Hz, 1H), 6.49 (dd, *J* = 2.1, 9.3 Hz, 2H), 7.05 – 7.15 (m, 3H), 7.20 – 7.30 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.45, 142.42, 132.21, 129.49, 121.68, 119.04, 118.31, 112.63. **HRMS (ESI)** calcd. for $C_{12}H_{10}Br_2NS$ ([M+H]+) is 357.8900 and found 357.8902.

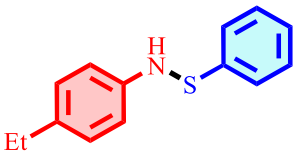


***N*-(3-nitrophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3m):** eluted in (2% ethyl acetate/petroleum ether, v/v); yellow solid (35 mg, isolated yield: 71%) Mp. 102-103 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.51 (s, 1H), 7.14 – 7.24 (m, 3H), 7.28 – 7.37 (m, 4H), 7.71 (dt, *J* = 2.2, 2.2, 7.1 Hz, 1H), 7.88 (td, *J* = 0.7, 1.9, 2.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ

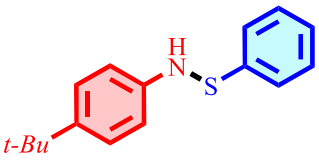
148.60, 138.64, 138.57, 131.69, 131.07, 129.37, 129.25, 126.21, 118.85, 115.05, 112.77. **HRMS (ESI)** calcd. for $C_{12}H_{11}N_2O_2S$ ([M+H]+) is 247.0536 and found 247.0533.



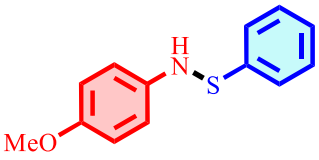
***S*-phenyl-*N*-(m-tolyl)thiohydroxylamine (Table 2, Entry 3n)**: eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (28 mg, isolated yield: 66%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.23 (s, 3H), 5.57 (s, 1H), 6.67 (d, *J* = 7.5 Hz, 1H), 6.80 – 6.86 (m, 3H), 6.98 (ddd, *J* = 1.0, 2.1, 8.6 Hz, 2H), 7.06 (d, *J* = 8.6 Hz, 1H), 7.16 – 7.22 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.25, 139.26, 129.36, 129.22, 121.91, 120.89, 118.51, 117.84, 114.94, 21.59. **HRMS (ESI)** calcd. for $C_{13}H_{14}NS$ ([M+H]+) is 216.0846 and found 216.0838.



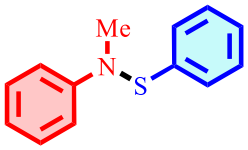
***N*-(4-ethylphenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3o):** eluted in (petroleum ether); orange liquid (28 mg, isolated yield: 62%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.22 (t, *J* = 7.6, 7.6 Hz, 3H), 2.59 (q, *J* = 7.6, 7.6, 7.6 Hz, 2H), 5.57 (s, 1H), 6.86 (tt, *J* = 1.1, 1.1, 7.3, 7.3 Hz, 1H), 6.98 – 7.01 (m, 4H), 7.07 – 7.11 (m, 2H), 7.19 – 7.25 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.97, 140.60, 137.47, 129.38, 128.74, 120.39, 118.88, 117.03, 28.24, 15.87. **HRMS (ESI)** calcd. for $C_{14}H_{16}NS$ ([M+H]+) is 230.1003 and found 230.1009.



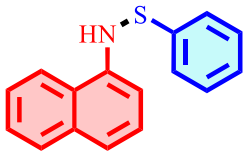
***N*-(4-(tert-butyl)phenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3p):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (32 mg, isolated yield: 63%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.49 (s, 9H), 7.94 (dd, *J* = 2.2, 9.2 Hz, 3H), 8.15 (d, *J* = 2.6 Hz, 5H), 8.18 (s, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.32, 143.30, 142.30, 130.11, 128.73, 123.96, 35.47, 30.79. **HRMS (ESI)** calcd. for $C_{16}H_{20}NS$ ([M+H]+) is 258.1316 and found 258.1318.



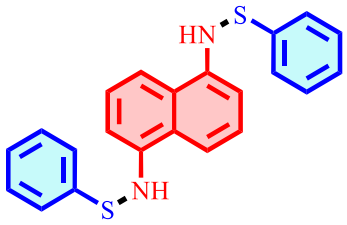
***N*-(4-methoxyphenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3q):** eluted in (5% ethyl acetate/petroleum ether, v/v); brown liquid (28 mg, isolated yield: 60%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.73 (s, 3H), 5.42 (s, 1H), 6.79 (d, *J* = 8.9 Hz, 3H), 6.83 (d, *J* = 7.9 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 2H), 7.14 (t, *J* = 7.7, 7.7 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 155.28, 145.18, 135.72, 129.33, 122.23, 119.57, 115.63, 114.67, 55.60. **HRMS (ESI)** calcd. for $C_{13}H_{14}NOS$ ([M+H]+) is 232.0796 and found 232.0790.



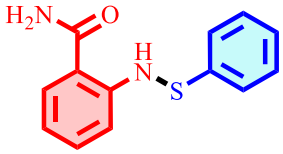
***N*-methyl-*N*,*S*-diphenylthiohydroxylamine (Table 2, Entry 3r)** : eluted in (petroleum ether); light yellow liquid (25 mg, isolated yield: 59%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.82 (s, 3H), 6.65 – 6.72 (m, 2H), 7.01 – 7.06 (m, 2H), 7.07 – 7.13 (m, 1H), 7.20 (dd, *J* = 7.0, 8.3 Hz, 3H), 7.35 (ddd, *J* = 1.7, 7.4, 8.1 Hz, 1H), 7.47 (dd, *J* = 1.6, 7.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.50, 133.95, 132.77, 119.79, 116.58, 111.90, 109.99, 29.83. **HRMS (ESI)** calcd. for $C_{13}H_{14}NS$ ([M+H]+) is 216.0846 and found 216.0838.



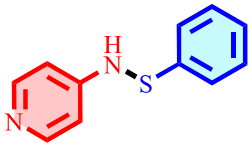
***N*-(naphthalen-1-yl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3s):** eluted in (2% ethyl acetate/petroleum ether, v/v); red solid (33 mg, isolated yield: 65%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.15 (s, 1H), 6.88 – 6.94 (m, 1H), 7.08 – 7.12 (m, 2H), 7.16 (dd, *J* = 2.4, 8.8 Hz, 1H), 7.23 (dddd, *J* = 1.5, 4.9, 6.9, 8.1 Hz, 3H), 7.33 (ddd, *J* = 1.3, 6.8, 8.2 Hz, 1H), 7.37 (d, *J* = 2.3 Hz, 1H), 7.57 (d, *J* = 8.2 Hz, 1H), 7.67 (d, *J* = 8.9 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.76, 137.11, 135.03, 133.63, 128.99, 128.63, 126.97, 126.35, 125.39, 125.33, 123.18, 121.53, 118.60, 108.11. **HRMS (ESI)** calcd. for $C_{16}H_{14}NS$ ([M+H]+) is 252.0847 and found 252.0856.



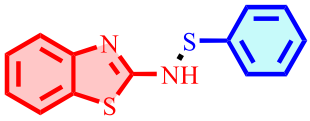
***N, N′*-(naphthalene-1,5-diyl) bis(*S*-phenylthiohydroxylamine) (Table 2, Entry 3t)**: eluted in (10% ethyl acetate/petroleum ether, v/v); black liquid (41 mg, isolated yield: 55%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.65 – 6.73 (m, 2H), 6.93 – 7.02 (m, 3H), 7.07 (ddd, *J* = 1.4, 6.5, 7.4 Hz, 2H), 7.13 – 7.21 (m, 4H), 7.22 – 7.29 (m, 3H), 7.53 (dd, *J* = 5.9, 8.5 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 152.32, 149.90, 137.66, 137.53, 129.90, 120.07, 119.87, 117.47, 111.86, 109.62. **HRMS (ESI)** calcd. for $C_{22}H_{19}N_2S_2$ ([M+H]+) is 375.0989 and found 375.0981.



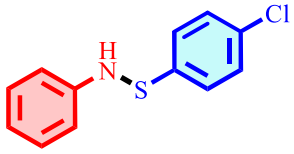
2-((phenylthio)amino) benzamide (Table 2, Entry 3u): eluted in (15% ethyl acetate/petroleum ether, v/v); brown solid (27 mg, isolated yield: 55%) Mp. 110-111 °C; **^{1}H NMR** (401 MHz, $CDCl_3$) δ 5.86 (s, 1H), 6.61 – 6.70 (m, 2H), 7.08 – 7.14 (m, 1H), 7.19 – 7.25 (td, *J* = 2.0, 7.0, 7.7 Hz, 2H), 7.27 – 7.33 (dd, *J* = 6.7, 8.4 Hz, 1H), 7.34 – 7.37 (m, 1H), 7.47 – 7.52 (dd, *J* = 1.8, 7.5 Hz, 1H), 7.54 – 7.60 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.86, 152.65, 140.07, 139.00, 138.47, 131.95, 129.21, 128.60, 119.13, 118.70, 112.10, 106.80. **HRMS (ESI)** calcd. for $C_{13}H_{13}N_2OS$ ([M+H]+) is 245.0748 and found 245.0743.



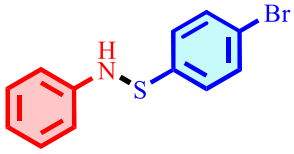
***S*-phenyl-*N*-(pyridin-2-yl)thiohydroxylamine (Table 2, Entry 3v)**: eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (19 mg, isolated yield: 47%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.40 (s, 1H), 6.72 – 6.75 (d, *J* = 8.3 Hz, 1H), 7.15 – 7.25 (m, 5H), 7.28 – 7.32 (m, 1H), 7.42 – 7.47 (dd, *J* = 2.0, 8.3 Hz, 1H), 7.85 – 7.89 (d, *J* = 2.1 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.13, 137.91, 135.47, 116.28, 114.63, 111.22, 108.77. **HRMS (ESI)** calcd. for $C_{11}H_{11}N_2S$ ([M+H]+) is 203.0642 and found 203.0635.



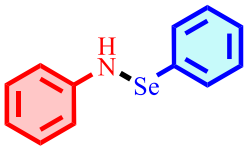
***N*-(benzo[d]thiazol-2-yl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3w)**: eluted in (petroleum ether,); light yellow liquid (26 mg, isolated yield: 51%) **^{1}H NMR** (401 MHz, $CDCl_3$) δ 7.29 – 7.61 (m, 8H), 7.69 – 7.74 (m, 1H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.74, 136.61, 133.64, 132.32, 131.43, 130.93, 129.45, 128.85, 128.81, 127.86, 127.58. **HRMS (ESI)** calcd. for $C_{13}H_{11}N_2S_2$ ([M+H]+) is 259.0358 and found 259.0363.



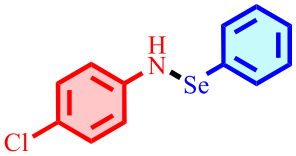
***N*-(4-chlorophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3x)**: eluted in (petroleum ether); dark brown liquid (33 mg, isolated yield: 71%) **^{1}H NMR** (401 MHz, $CDCl_3$) δ 5.16 (s, 1H), 6.86 – 6.91 (td, *J* = 1.1, 7.2 Hz, 1H), 6.98 – 7.02 (d, *J* = 8.1 Hz, 2H), 7.11 – 7.14 (d, *J* = 8.5 Hz, 2H), 7.22 – 7.25 (d, *J* = 8.6 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.26, 140.07, 131.26, 129.39, 129.05, 123.83, 120.84, 114.68. **HRMS (ESI)** calcd. for $C_{12}H_{11}ClNS$ ([M+H]+) is 236.0300 and found 236.0302.



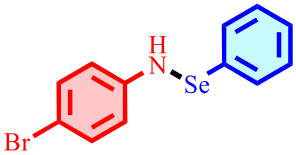
***N*-(4-bromophenyl)-*S*-phenylthiohydroxylamine (Table 2, Entry 3y):** eluted in (petroleum ether); brown liquid (40 mg, isolated yield: 73%) **^{1}H NMR** (401 MHz, $CDCl_3$) δ 5.15 (s, 1H), 6.86 – 6.92 (t, *J* = 7.3 Hz, 1H), 6.98 – 7.02 (m, 2H), 7.05 – 7.09 (m, 2H), 7.20 – 7.25 (m, 2H), 7.36 – 7.40 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.20, 140.79, 131.92, 129.40, 124.05, 120.87, 118.98, 114.67. **HRMS (ESI)** calcd. for $C_{12}H_{11}BrNS$ ([M+H]+) is 279.9795 and found 279.9750.



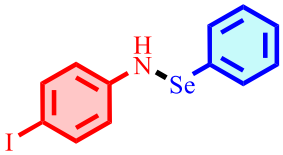
***N, Se*-diphenylselenohydroxylamine (Table 3, Entry 5a):** eluted in (1% ethyl acetate/petroleum ether, v/v); yellow liquid (39 mg, isolated yield: 78 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.21 (s, 1H), 6.63 (td, *J* = 1.3, 7.5, 7.5 Hz, 1H), 6.72 (dd, *J* = 1.3, 8.0 Hz, 1H), 7.06 – 7.19 (m, 7H), 7.50 (dd, *J* = 1.6, 7.7 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.60, 138.64, 138.57, 131.69, 131.07, 129.37, 126.21, 118.85, 115.05, 112.77. **HRMS (ESI)** calcd. for $C_{12}H_{12}NSe$ ([M+H]+) is 250.0134 and found 250.0136.



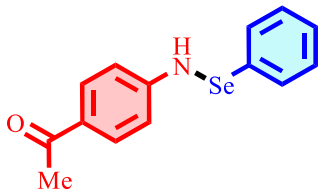
***N*-(4-chlorophenyl)-*Se*-phenylselenohydroxylamine (Table 2, Entry 5b):** eluted in (2% ethyl acetate/petroleum ether, v/v); yellow liquid (42 mg, isolated yield: 75%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.61 (s, 1H), 6.85 – 6.89 (m, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.97 (dd, *J* = 1.2, 8.5 Hz, 2H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.17 – 7.23 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.67, 141.89, 129.46, 129.28, 125.48, 121.51, 118.81, 118.11. **HRMS (ESI)** calcd. for $C_{12}H_{11}ClNSe$ ([M+H]+) is 283.9745 and found 283.9743.



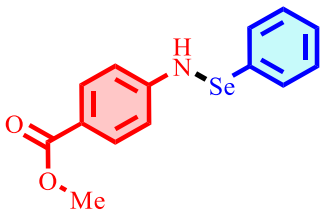
***N*-(4-bromophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5c):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (50 mg, isolated yield: 77 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.29 (s, 1H), 6.67 (d, *J* = 8.6 Hz, 1H), 7.18 – 7.30 (m, 7H), 7.69 (d, *J* = 2.3 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.54, 139.97, 133.64, 130.85, 129.76, 129.43, 126.68, 116.34, 114.47, 109.38. **HRMS (ESI)** calcd. for $C_{12}H_{11}BrNSe$ ([M+H]+) is 327.9154 and found 327.9159.



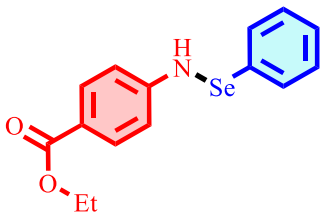
***N*-(4-iodophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5d):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (55 mg, isolated yield: 73 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.58 (s, 1H), 6.70 – 6.75 (m, 2H), 6.85 – 6.91 (m, 1H), 6.95 – 6.99 (m, 2H), 7.15 – 7.22 (m, 2H), 7.40 – 7.44 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.07, 141.12, 137.01, 128.40, 120.75, 118.22, 117.48, 81.05. **HRMS (ESI)** calcd. for $C_{12}H_{11}INSe$ ([M+H]+) is 375.9101 and found 375.9103.



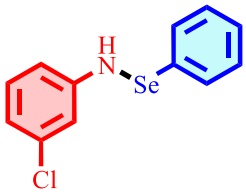
1-(4-((phenylselanyl)amino)phenyl)ethan-1-one (Table 3, Entry 5e): eluted in (12% ethyl acetate/petroleum ether, v/v); brown liquid (34 mg, isolated yield: 58 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.41 (s, 3H), 4.78 (s, 1H), 6.68 (d, *J* = 8.5 Hz, 1H), 7.07 – 7.19 (m, 6H), 7.78 (dd, *J* = 2.1, 8.5 Hz, 1H), 8.16 (d, *J* = 2.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 194.87, 151.77, 139.31, 139.25, 139.18, 130.85, 129.77, 128.36, 127.06, 125.53, 112.78, 110.48, 25.04. **HRMS (ESI)** calcd. for $C_{14}H_{14}NOSe$ ([M+H]+) is 292.0240 and found 292.0246.



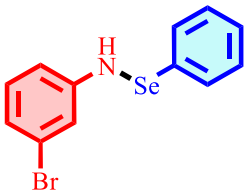
methyl 4-((phenylselanyl)amino)benzoate (Table 3, Entry 5f): eluted in (8% ethyl acetate/petroleum ether, v/v); dark yellow solid (43 mg, isolated yield: 71%) Mp. 115-116 °C; 1**H NMR** (400 MHz, $CDCl_3$) δ 3.84 (s, 3H), 4.77 (s, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 7.13 – 7.25 (m, 6H), 7.89 (dd, *J* = 2.1, 8.5 Hz, 1H), 8.31 (d, *J* = 2.1 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.54, 152.56, 140.95, 140.88, 140.81, 132.89, 130.92, 129.40, 126.48, 119.92, 113.72, 111.57, 51.74. **HRMS (ESI)** calcd. for $C_{14}H_{14}NO_2Se$ ([M+H]+) is 308.0189 and found 308.0187.



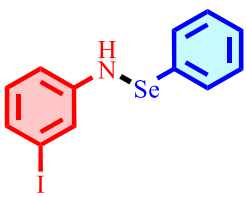
ethyl 4-((phenylselanyl)amino)benzoate (Table 3, Entry 5g): eluted in (8% ethyl acetate/petroleum ether, v/v); brown solid (47 mg, isolated yield: 74%) Mp. 122-123 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.27 (t, *J* = 7.1, 7.1 Hz, 3H), 4.23 (q, *J* = 7.1, 7.1, 7.1 Hz, 2H), 4.68 (s, 1H), 6.66 (d, *J* = 8.4 Hz, 1H), 7.01 – 7.21 (m, 6H), 7.81 (dd, *J* = 2.1, 8.5 Hz, 1H), 8.23 (d, *J* = 2.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.14, 152.55, 140.85, 132.93, 130.99, 129.38, 129.29, 126.50, 120.33, 113.76, 111.51, 60.58, 14.48. **HRMS (ESI)** calcd. for $C_{15}H_{16}NO_2Se$ ([M+H]+) is 322.0346 and found 322.0345.



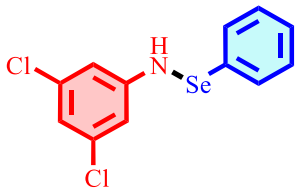
***N*-(3-chlorophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5h):** eluted in (1% ethyl acetate/petroleum ether, v/v); yellow liquid (39 mg, isolated yield: 69%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.75 (s, 1H), 6.45 (dd, *J* = 2.5, 8.4 Hz, 1H), 6.77 (d, *J* = 2.5 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 1H), 7.21 – 7.29 (m, 4H), 7.36 – 7.41 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.53, 139.52, 136.77, 131.21, 129.39, 129.35, 126.46, 118.84, 114.56, 110.94. **HRMS (ESI)** calcd. for $C_{12}H_{11}ClNSe$ ([M+H]+) is 283.9745 and found 283.9743.



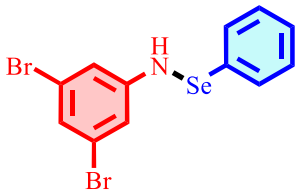
***N*-(3-bromophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5i):** eluted in (2% ethyl acetate/petroleum ether, v/v); yellow liquid (44 mg, isolated yield: 67%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.27 (s, 1H), 6.74 (dd, *J* = 2.1, 8.1 Hz, 1H), 6.86 (d, *J* = 2.1 Hz, 1H), 7.05 – 7.17 (m, 6H), 7.34 (d, *J* = 8.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.64, 138.61, 130.02, 128.45, 128.39, 128.32, 125.44, 123.93, 120.67, 116.41, 110.50. **HRMS (ESI)** calcd. for $C_{12}H_{11}BrNSe$ ([M+H]+) is 327.9154 and found 327.9159.



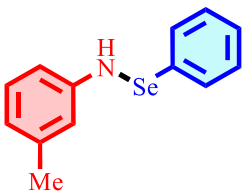
***N*-(3-iodophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5j):** eluted in (2% ethyl acetate/petroleum ether, v/v); orange liquid (47 mg, isolated yield: 63%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.21 (s, 1H), 6.92 (dd, *J* = 1.8, 8.1 Hz, 1H), 7.06 (d, *J* = 1.9 Hz, 1H), 7.08 – 7.20 (m, 7H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.57, 138.57, 129.93, 128.48, 128.32, 126.66, 125.46, 122.35, 111.46, 95.91. **HRMS (ESI)** calcd. for $C_{12}H_{11}INSe$ ([M+H]+) is 375.9101 and found 375.9103.



***N*-(3,5-dichlorophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5k):** eluted in (2% ethyl acetate/petroleum ether, v/v); brown liquid (41 mg, isolated yield: 65%) **^{1}H NMR** (401 MHz, $CDCl_3$) δ 4.59 (s, 1H), 6.61 (d, *J* = 2.1 Hz, 1H), 6.83 (d, *J* = 2.1 Hz, 1H), 7.10 – 7.18 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.85, 137.86, 128.37, 119.58, 119.50, 115.76, 115.01, 114.78. **HRMS (ESI)** calcd. for $C_{12}H_{10}Cl_2NSe$ ([M+H]+) is 317.9355 and found 317.9333.

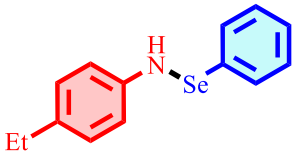


***N*-(3,5-dibromophenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5l):** eluted in (2% ethyl acetate/petroleum ether, v/v); brown liquid (49 mg, isolated yield: 60%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.57 (s, 1H), 6.78 (d, *J* = 2.0 Hz, 1H), 7.09 – 7.19 (m, 7H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 151.39, 138.48, 137.24, 116.95, 115.95, 111.12, 110.48, 97.51. **HRMS (ESI)** calcd. for $C_{12}H_{10}Br_2NSe$ ([M+H]+) is 405.8345 and found 405.8324.

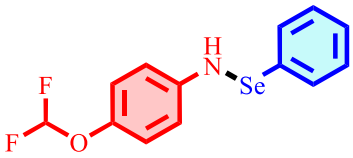


***Se*-phenyl-*N*-(m-tolyl)selenohydroxylamine (Table 3, Entry 5m)**: eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (35 mg, isolated yield: 66%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.10 (s, 3H), 5.67 (s, 1H), 6.80 (ddd, *J* = 2.2, 8.1, 14.3 Hz, 2H), 6.92 (t, *J* = 7.4, 7.4 Hz, 1H), 6.96 (t, *J* = 2.2, 2.2 Hz, 1H), 7.00 – 7.04 (m, 2H), 7.08 (t, *J* = 8.0, 8.0 Hz, 1H), 7.20 –

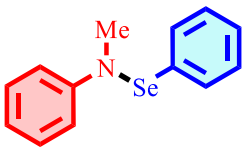
7.25 (m, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 135.40, 135.12, 134.25, 131.73, 129.59, 128.76, 127.02, 120.33, 20.92. **HRMS (ESI)** calcd. for $C_{13}H_{14}NSe$ ([M+H]+) is 264.0291 and found 264.0284.



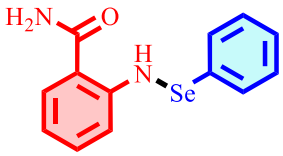
***N*-(4-ethylphenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5n)**: eluted in (2% ethyl acetate/petroleum ether, v/v); brown liquid (33 mg, isolated yield: 60%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.12 (t, *J* = 7.6, 7.6 Hz, 3H), 2.47 (q, *J* = 7.6, 7.6, 7.6 Hz, 2H), 4.08 (s, 1H), 6.67 (d, *J* = 8.1 Hz, 1H), 6.99 (dd, *J* = 2.1, 8.1 Hz, 1H), 7.05 – 7.19 (m, 6H), 7.35 (d, *J* = 2.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.35, 136.50, 133.79, 130.82, 129.59, 128.18, 125.04, 114.11, 111.62, 26.65, 14.76. **HRMS (ESI)** calcd. for $C_{14}H_{16}NSe$ ([M+H]+) is 278.0447 and found 278.0425.



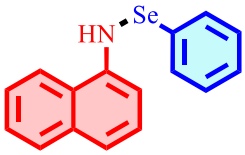
***N*-(4-(difluoromethoxy)phenyl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5o)**: eluted in (5% ethyl acetate/petroleum ether, v/v); brown liquid (39 mg, isolated yield: 62%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.23 (s, 1H), 6.38 (t, *J* = 74.4, 74.4 Hz, 1H), 6.75 (d, *J* = 8.7 Hz, 1H), 7.02 (dd, *J* = 2.8, 8.7 Hz, 1H), 7.17 – 7.29 (m, 6H), 7.37 (d, *J* = 2.7 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 151.39, 138.48, 137.24, 116.95, 115.95, 111.12, 110.48, 97.51. **HRMS (ESI)** calcd. for $C_{13}H_{12}F_2NOSe$ ([M+H]+) is 316.0052 and found 316.0048.



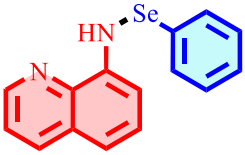
***N*-methyl-*N*,*Se*-diphenylselenohydroxylamine (Table 3, Entry 5p):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (31 mg, isolated yield: 59%) **^{1}H NMR** (401 MHz, $CDCl_3$) δ 2.74 (s, 3H), 6.45 – 6.49 (m, 2H), 7.02 – 7.12 (m, 4H), 7.17 – 7.21 (m, 2H), 7.34 – 7.38 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.51, 136.27, 133.52, 128.73, 127.95, 124.79, 113.43, 112.23, 29.43. **HRMS (ESI)** calcd. for $C_{13}H_{14}NSe$ ([M+H]+) is 264.0291 and found 264.0284.



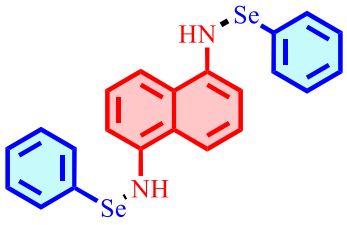
2-((phenylselanyl)amino) benzamide (Table 3, Entry 5q): eluted in (25% ethyl acetate/petroleum ether, v/v); white solid (34 mg, isolated yield: 58%) Mp. 113-114 °C; **^{1}H NMR** (401 MHz, DMSO-d_6) δ 6.74 (d, J = 8.5 Hz, 1H), 6.93 (s, 1H), 7.09 – 7.29 (m, 5H), 7.36 (dd, J = 2.0, 8.5 Hz, 1H), 7.92 (d, J = 2.1 Hz, 1H). **^{13}C NMR** (101 MHz, DMSO) δ 170.95, 151.41, 140.40, 137.68, 137.62, 134.61, 129.74, 129.48, 126.43, 118.36, 114.96, 110.65. **HRMS (ESI)** calcd. for $C_{13}H_{13}N_2OSe$ ([M+H]+) is 293.0193 and found 293.0186.



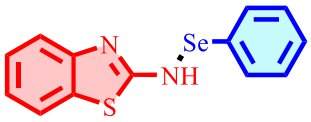
***N*-(naphthalen-1-yl)-*Se*-phenylselenohydroxylamine (Table 3, Entry 5r):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (33 mg, isolated yield: 55%) **^{1}H NMR** (401 MHz, $CDCl_3$) δ 4.91 (s, 1H), 7.05 – 7.17 (m, 7H), 7.37 – 7.46 (m, 2H), 7.57 (d, J = 8.5 Hz, 1H), 7.71 – 7.78 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.20, 134.05, 133.84, 130.92, 128.21, 128.13, 127.56, 125.77, 125.07, 124.35, 121.94, 120.64, 117.63, 105.96. **HRMS (ESI)** calcd. for $C_{16}H_{14}NSe$ ([M+H]+) is 300.0291 and found 300.0264.



***Se*-phenyl-*N*-(quinolin-8-yl) selenohydroxylamine (Table 3, Entry 5s):** eluted in (2% ethyl acetate/petroleum ether, v/v); yellow liquid (32 mg, isolated yield: 54%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.01 (s, 1H), 7.09 (tt, J = 1.2, 1.2, 8.2, 8.2 Hz, 3H), 7.17 – 7.23 (m, 2H), 7.28 (s, 1H), 7.47 – 7.56 (m, 3H), 7.78 – 7.86 (m, 2H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 134.79, 134.19, 132.47, 131.93, 128.82, 128.79, 127.70, 127.28, 126.39, 126.21, 126.06, 125.75, 121.57, 120.97. **HRMS (ESI)** calcd. for $C_{15}H_{13}N_2Se$ ([M+H]+) is 301.0244 and found 301.0238.

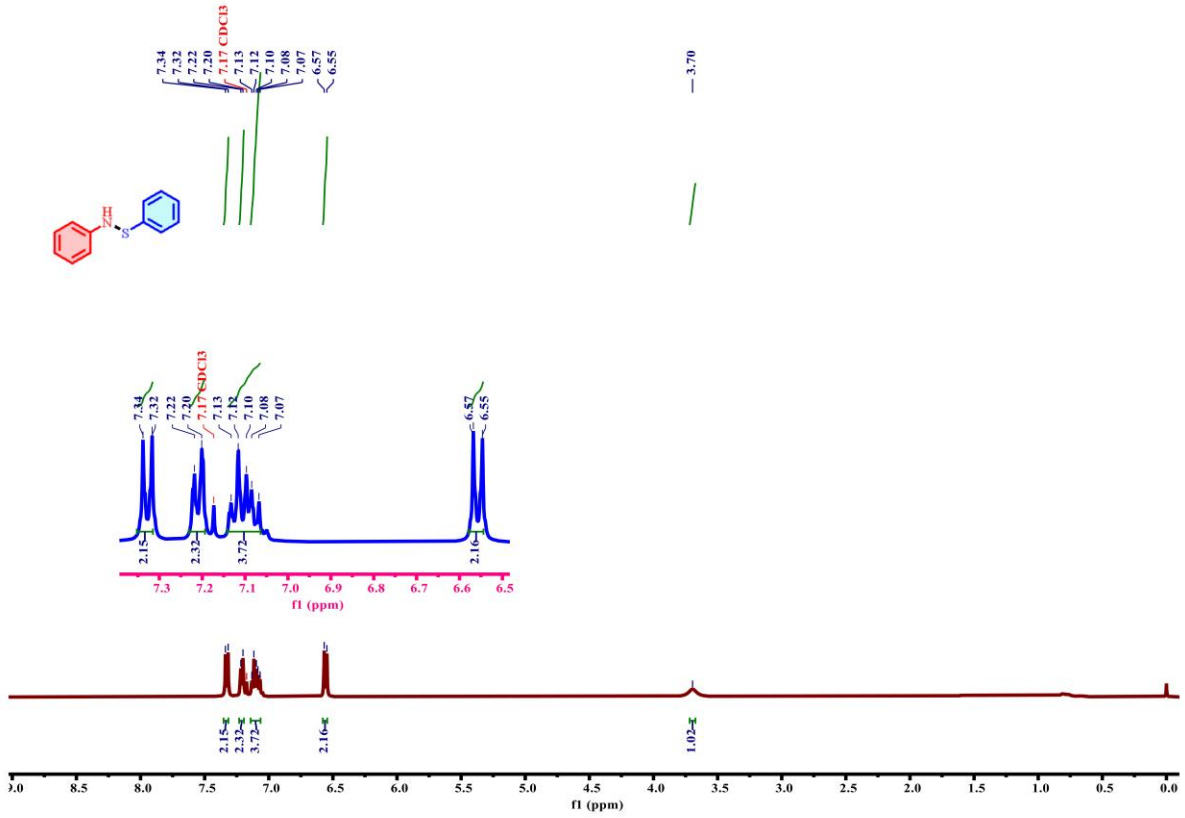


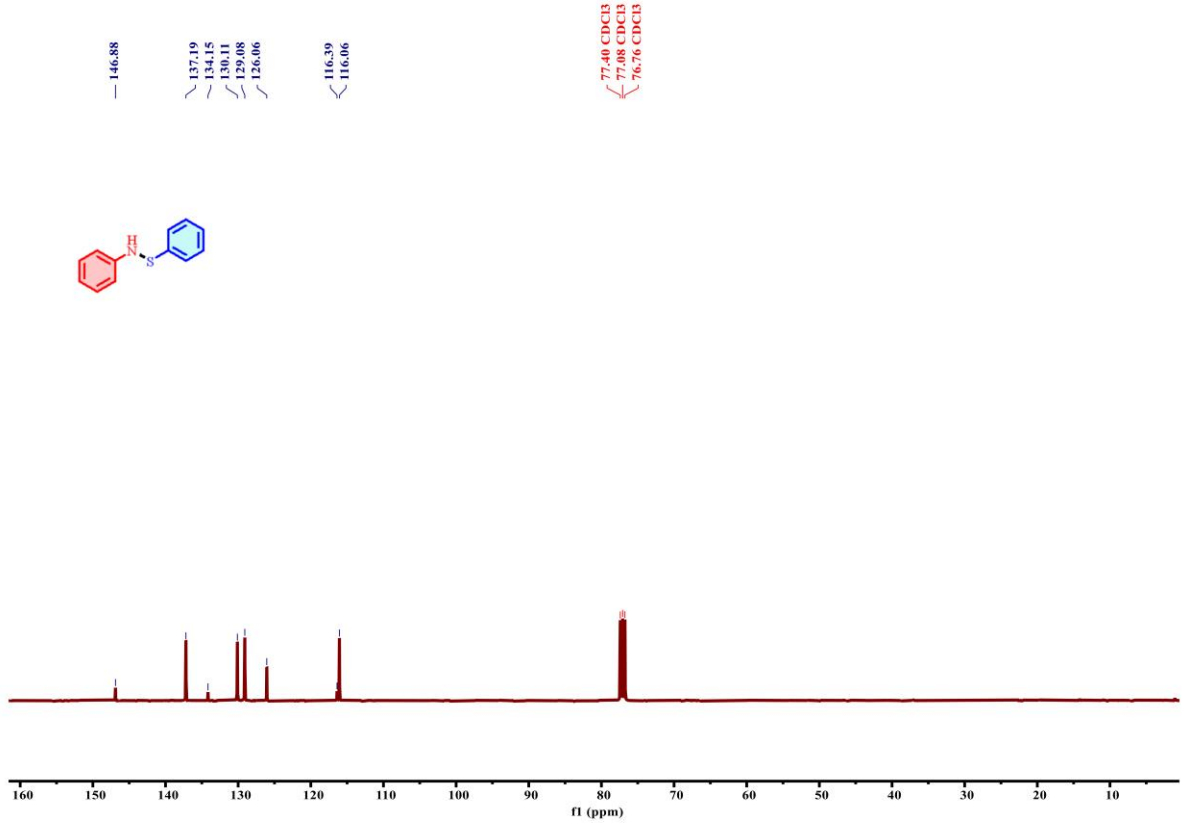
***N,N'*-(naphthalene-1,5-diyl)bis(*Se*-phenylselenohydroxylamine) (Table 3, Entry 5t)**: eluted in (10% ethyl acetate/petroleum ether, v/v); dark red liquid (56 mg, isolated yield: 60 %) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.94 (s, 1H), 6.82 (dd, *J* = 2.6, 5.8 Hz, 1H), 7.13 – 7.30 (m, 13H), 7.61 (d, *J* = 8.7 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.30, 141.10, 134.26, 129.73, 123.25, 123.21, 120.28, 117.29, 112.24, 111.59. **HRMS (ESI)** calcd. for $C_{22}H_{19}N_2Se_2$ ([M+H]+) is 470.9878 and found 470.9875.



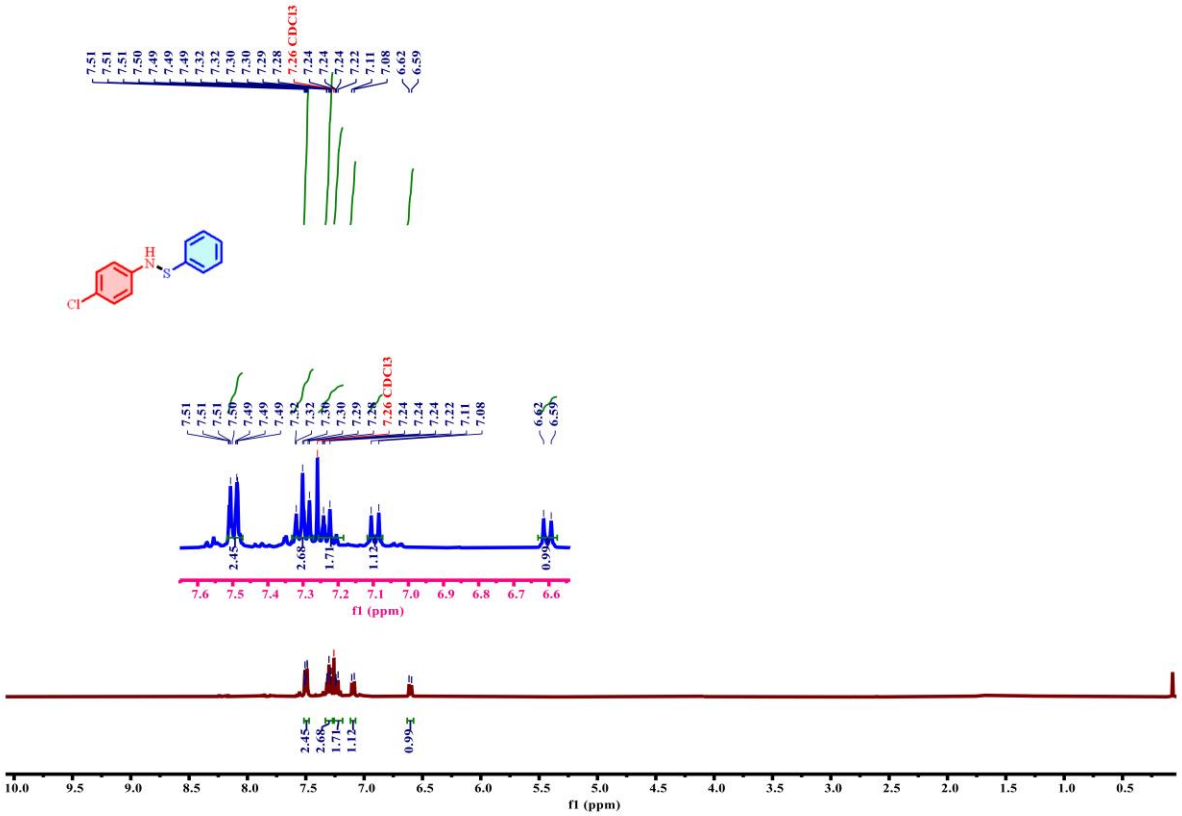
***N*-(benzo[d]thiazol-2-yl)-*Se*-phenylselenohydroxylamine (Table 3, entry 5v):** eluted in (1% ethyl acetate/petroleum ether, v/v); brown liquid (29 mg, isolated yield: 47%) **^{1}H NMR** (401 MHz, DMSO-d_6) δ 7.19 – 7.26 (dd, *J* = 3.1, 6.0 Hz, 2H), 7.48 – 7.53 (m, 1H), 7.54 – 7.60 (dd, *J* = 6.5, 8.3 Hz, 2H), 7.60 – 7.65 (dt, *J* = 3.5, 7.0 Hz, 2H), 8.17 – 8.23 (dd, *J* = 1.8, 7.4 Hz, 2H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 151.84, 144.29, 140.02, 135.41, 129.98, 127.93, 126.85, 122.79, 122.02, 119.17, 111.65. **HRMS (ESI)** calcd. for $C_{13}H_{11}N_2SSe$ ([M+H]+) is 306.9808 and found 306.9810.

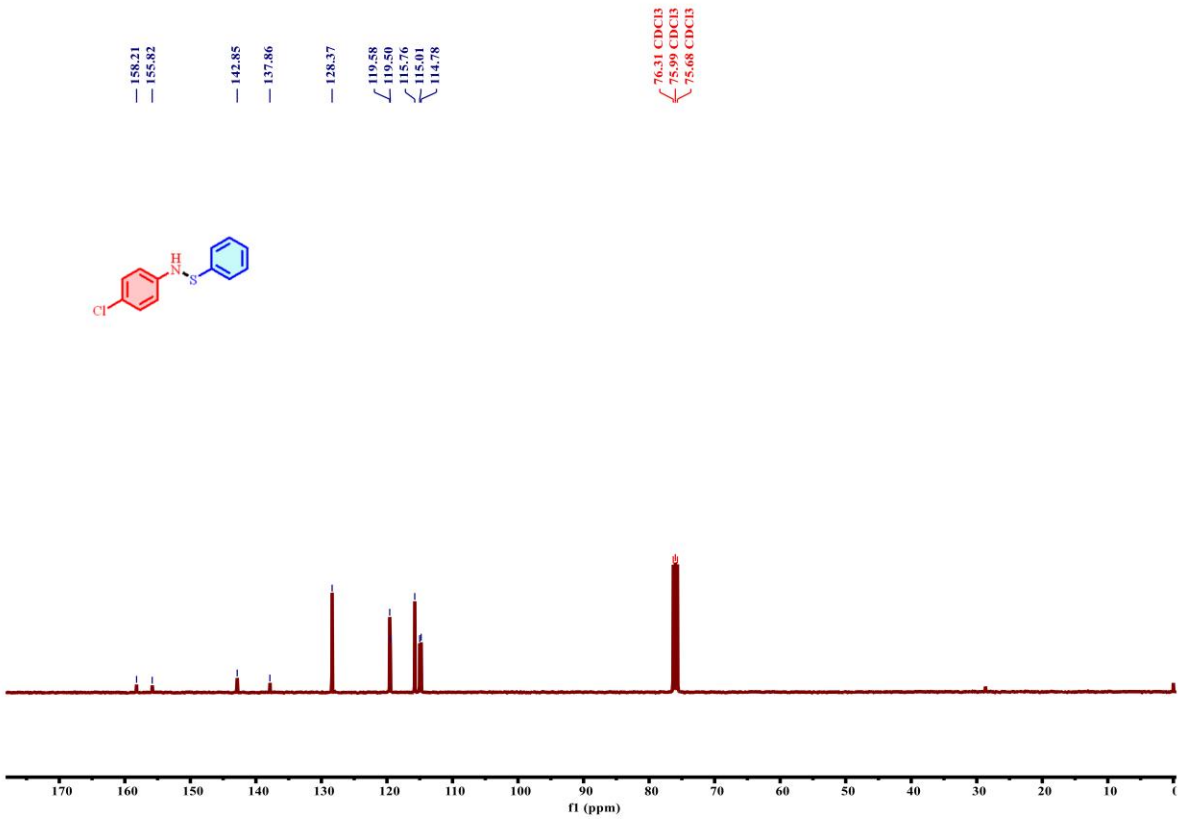
(Table 2, Entry 3a)



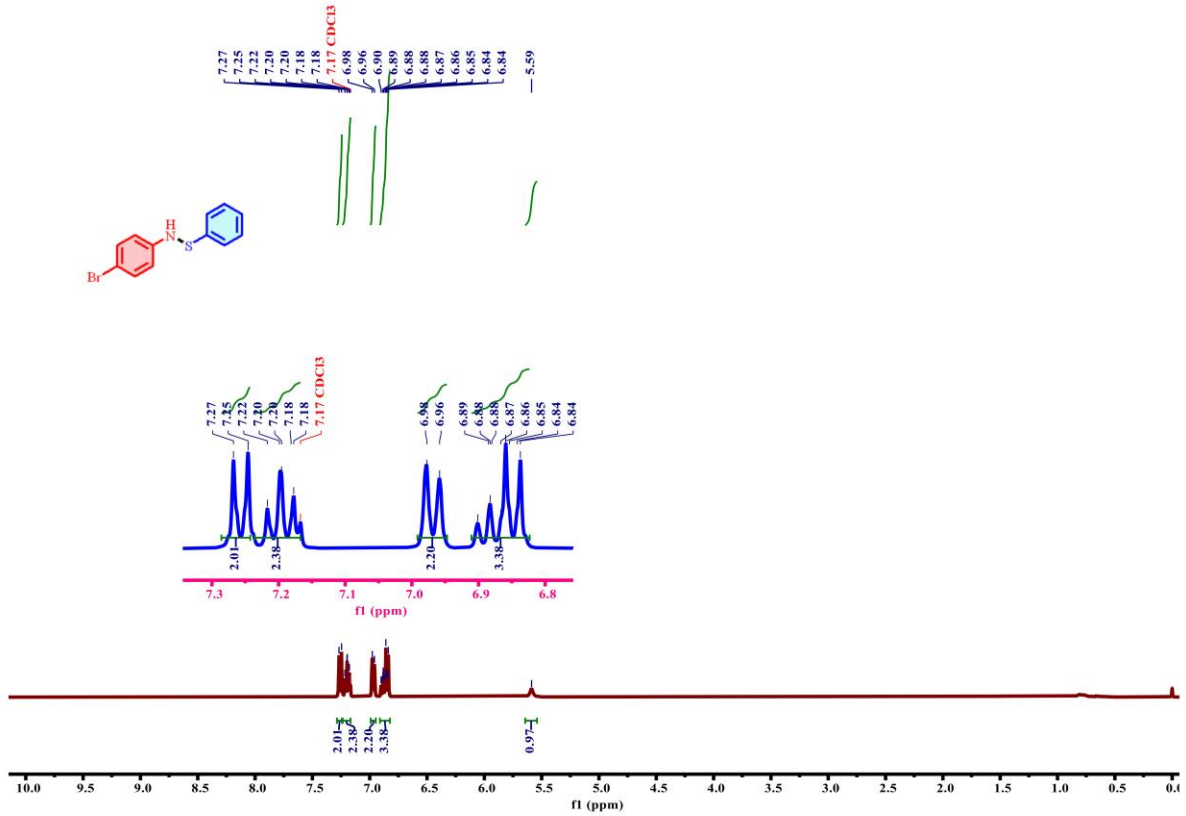


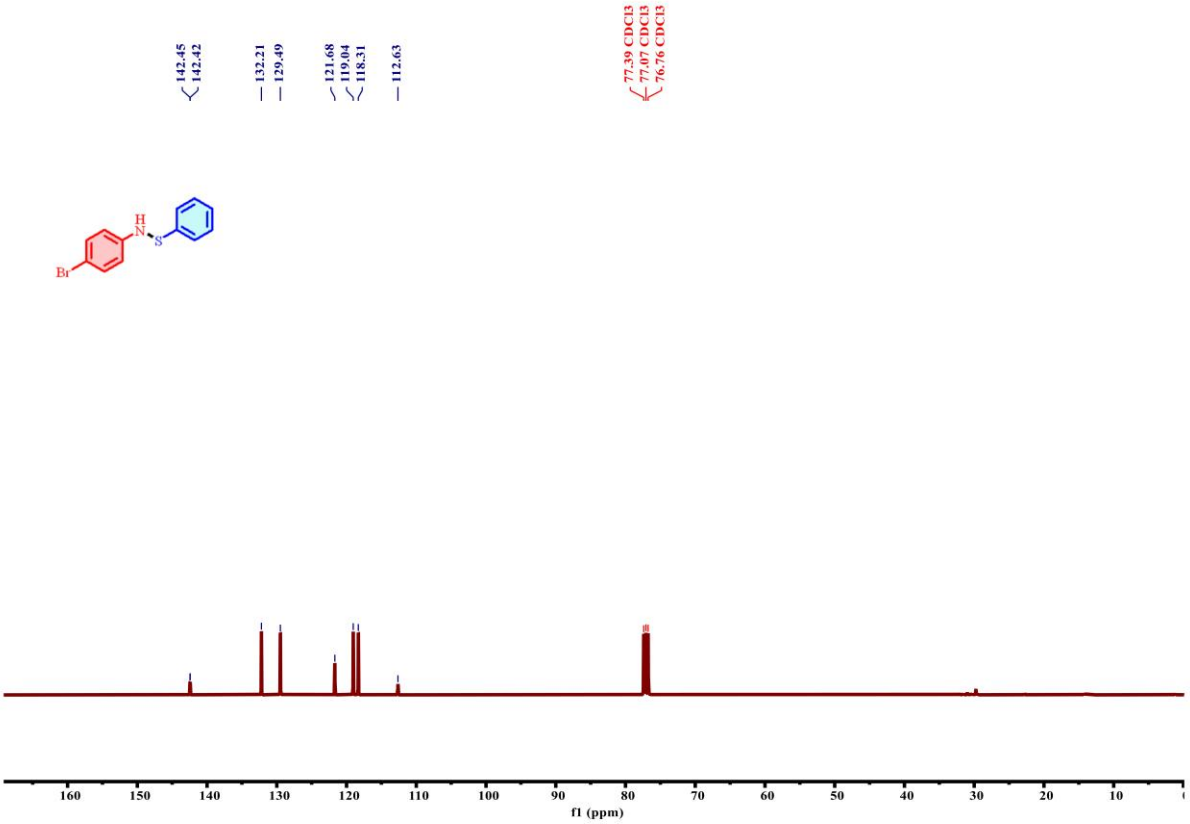
(Table 2, Entry 3b)



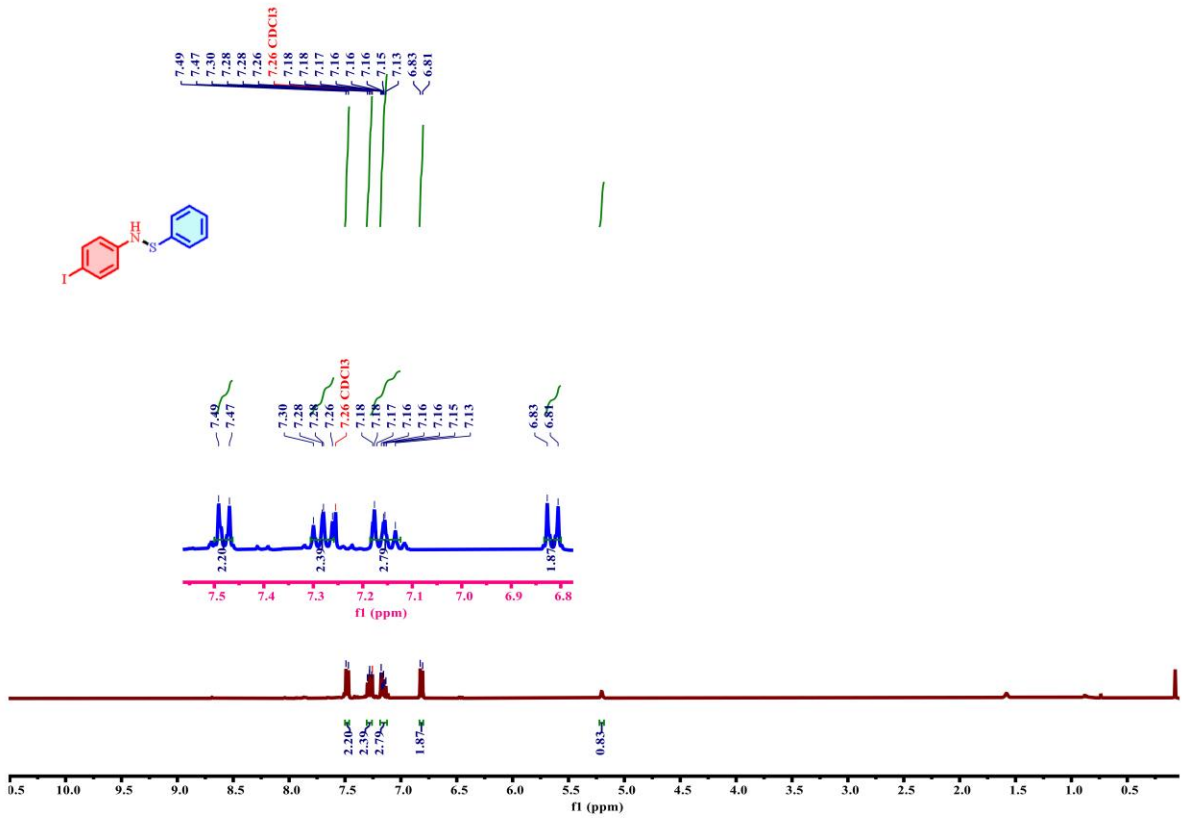


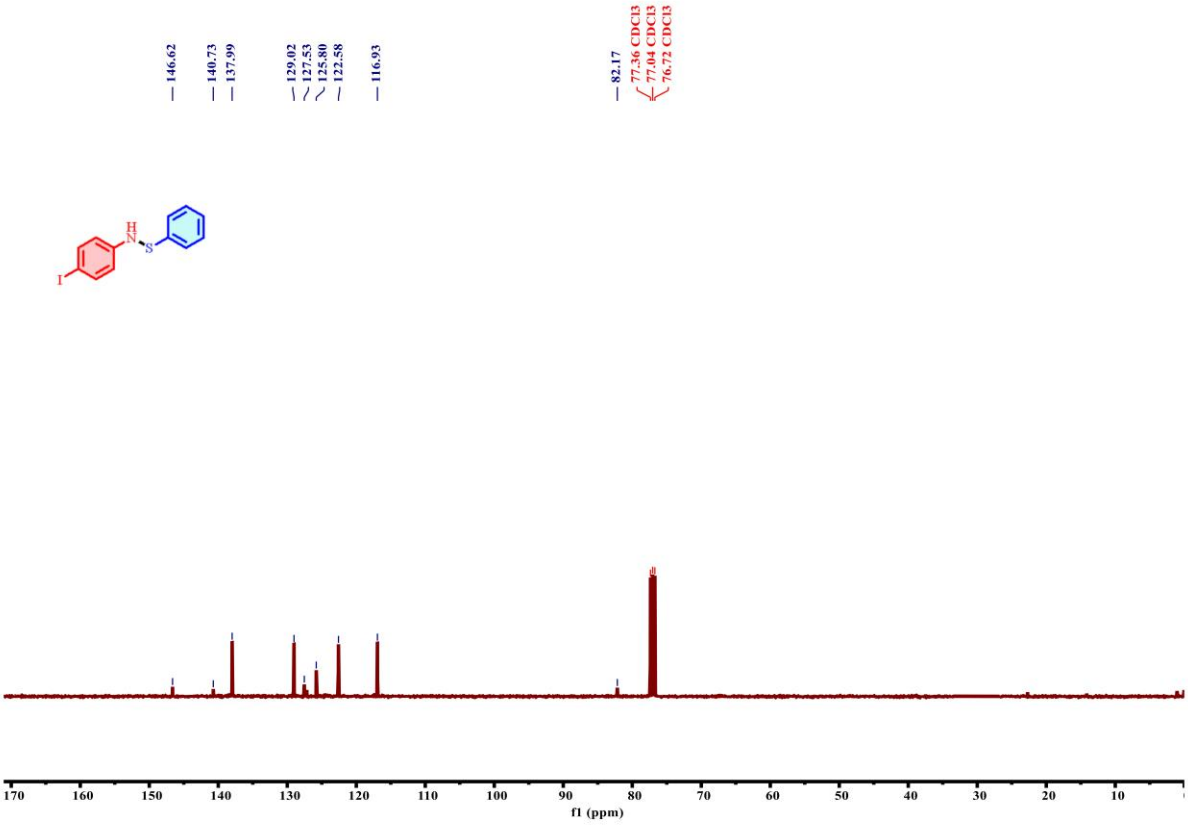
(Table 2, Entry 3c)



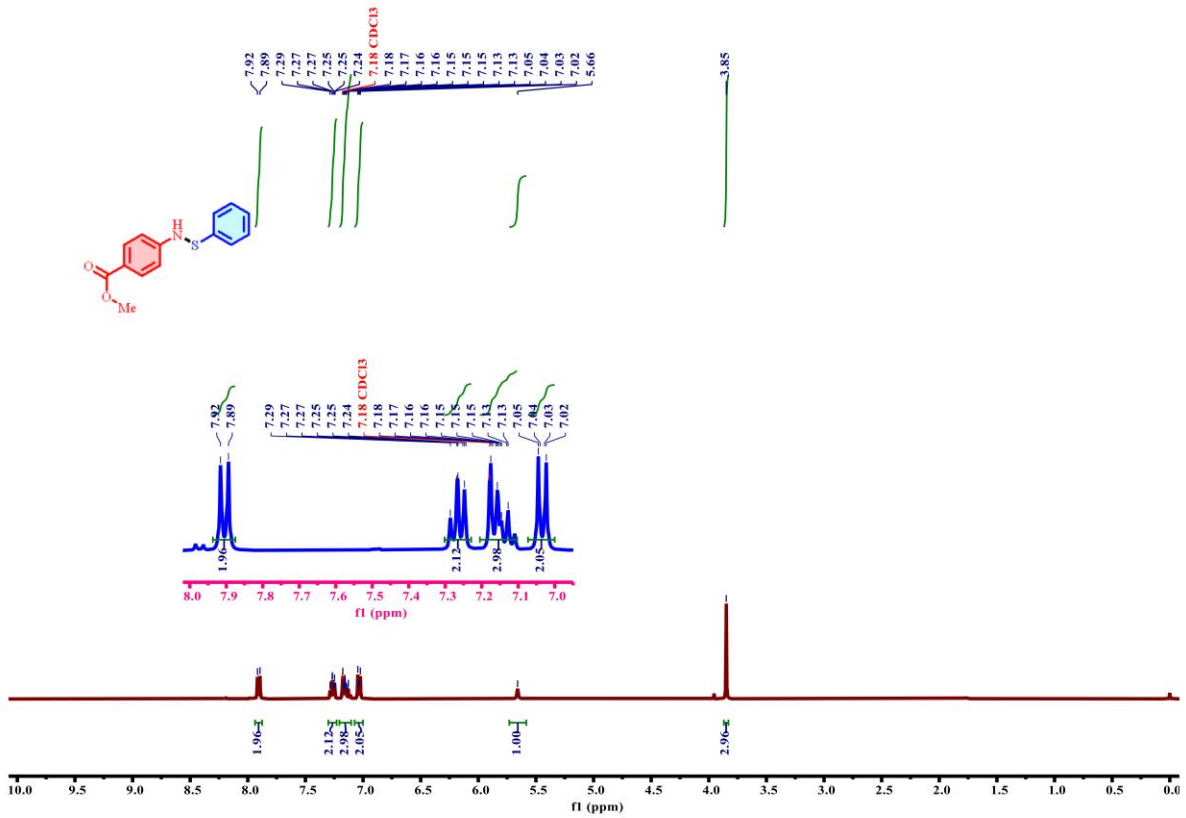


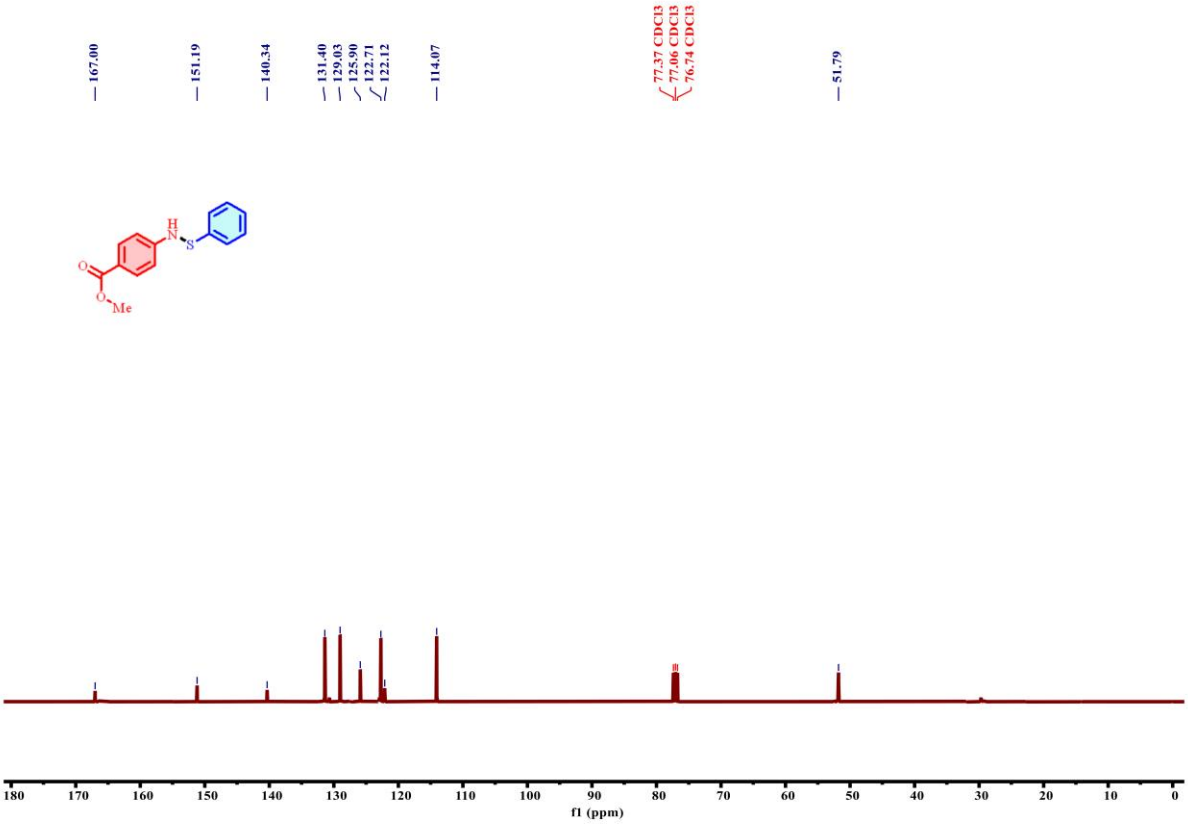
(Table 2, Entry 3d)



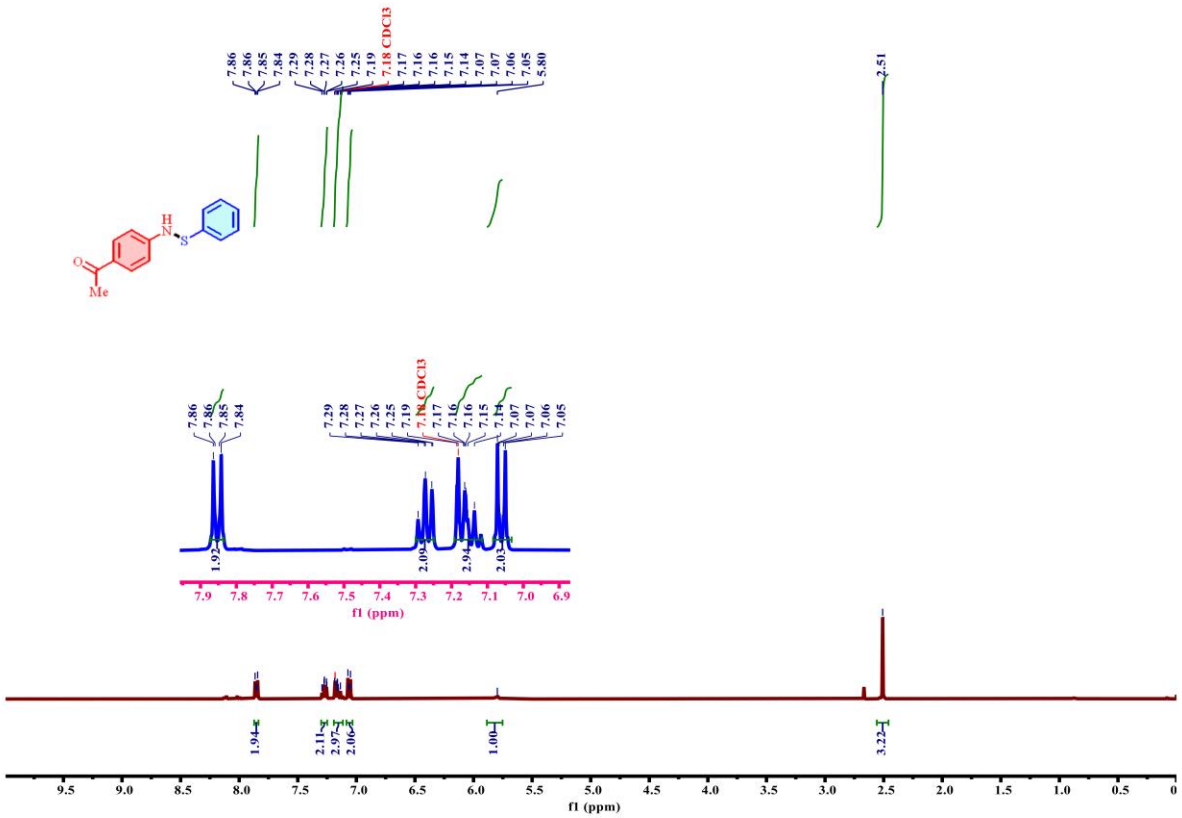


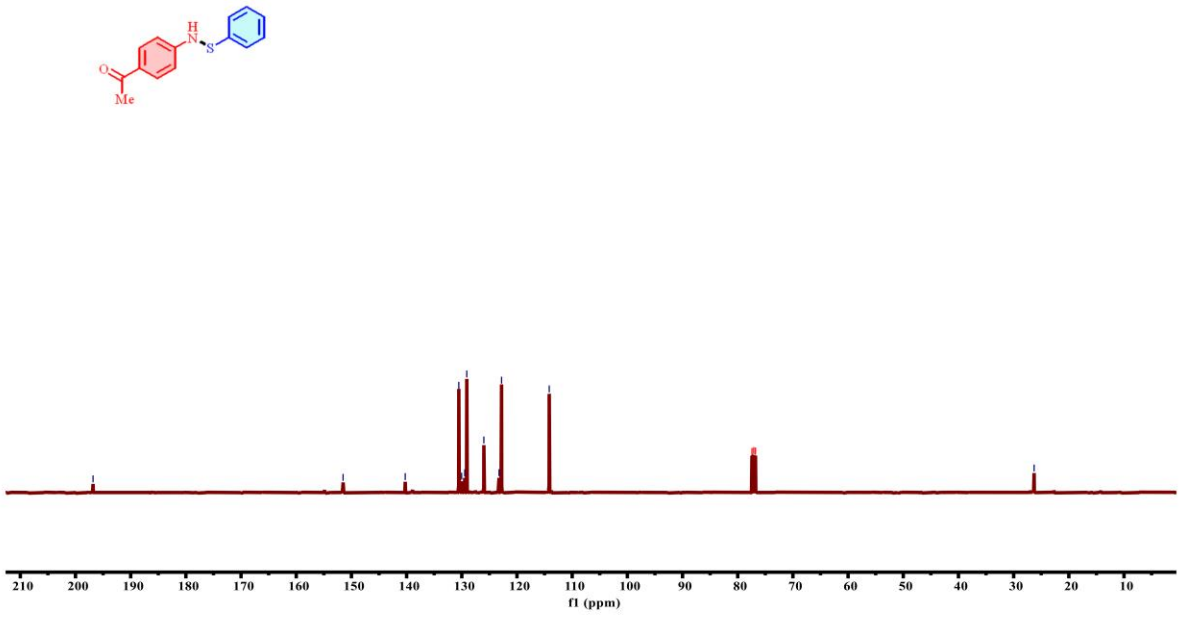
(Table 2, Entry 3e)



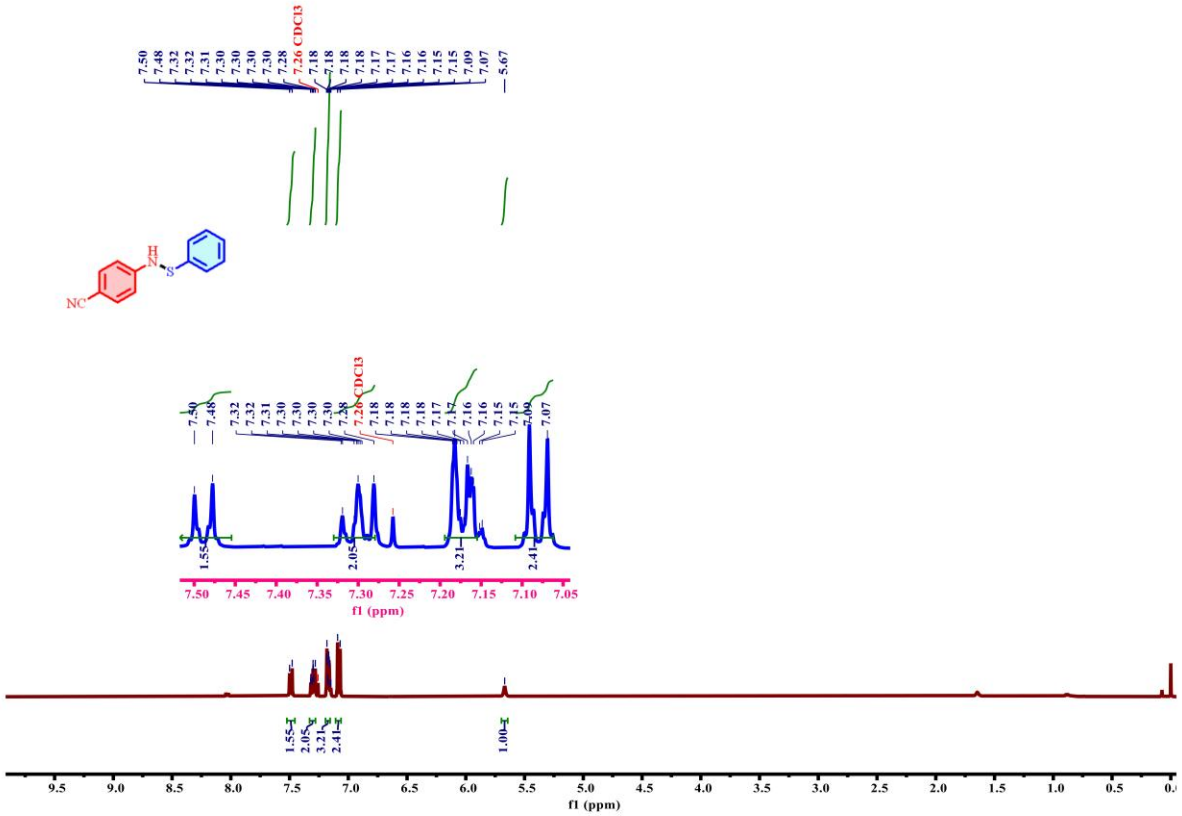


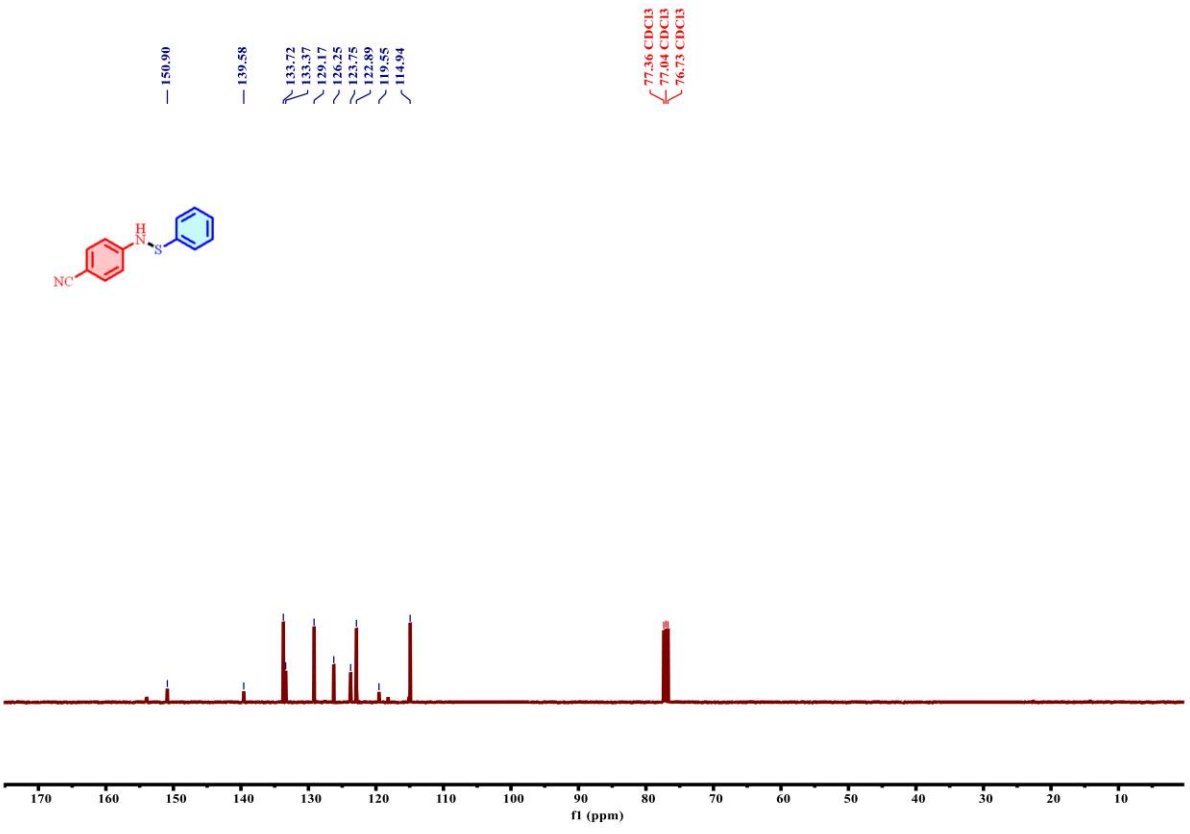
(Table 2, Entry 3f)



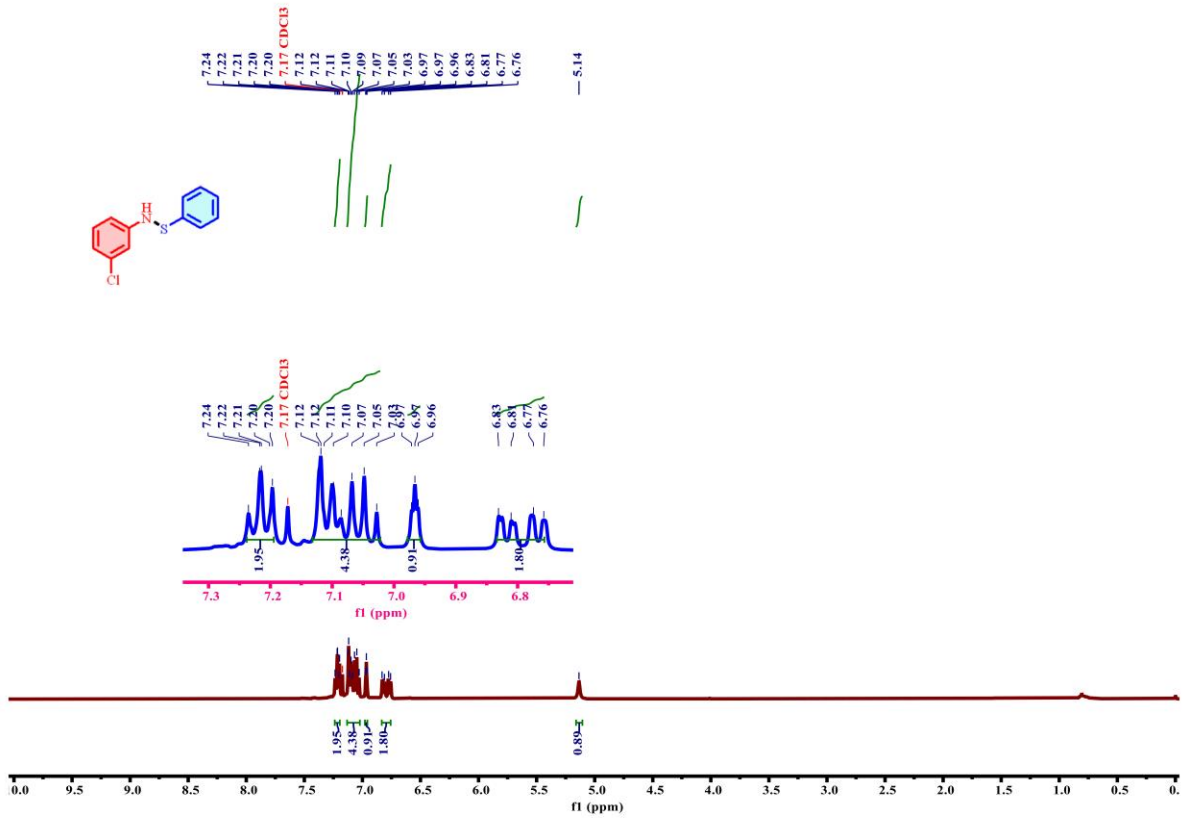


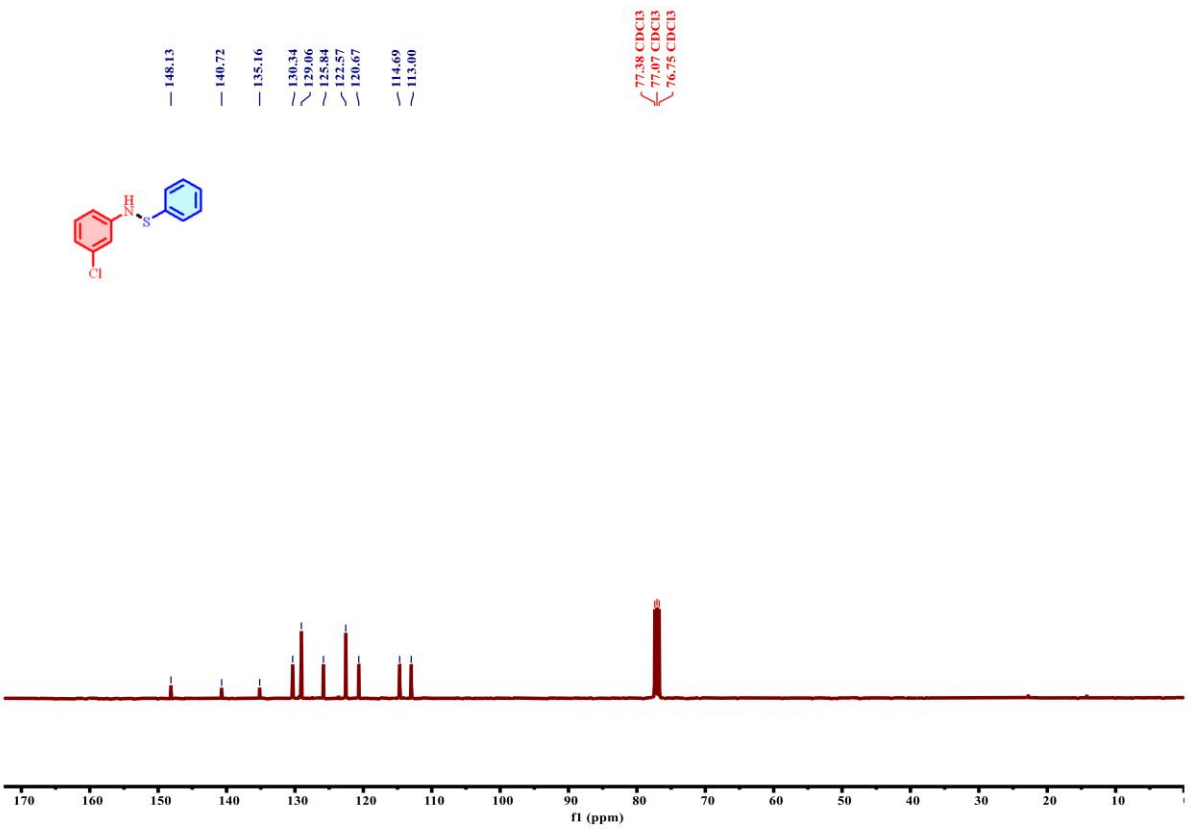
(Table 2, Entry 3g)



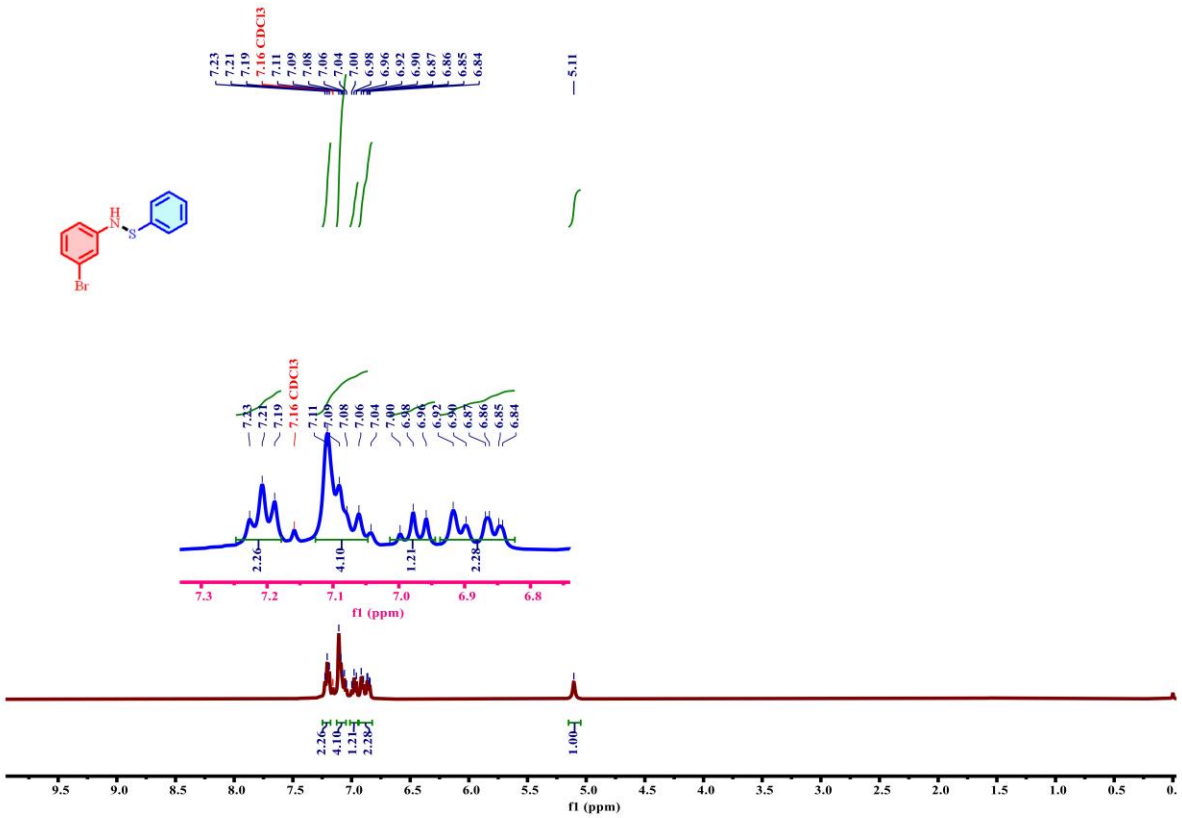


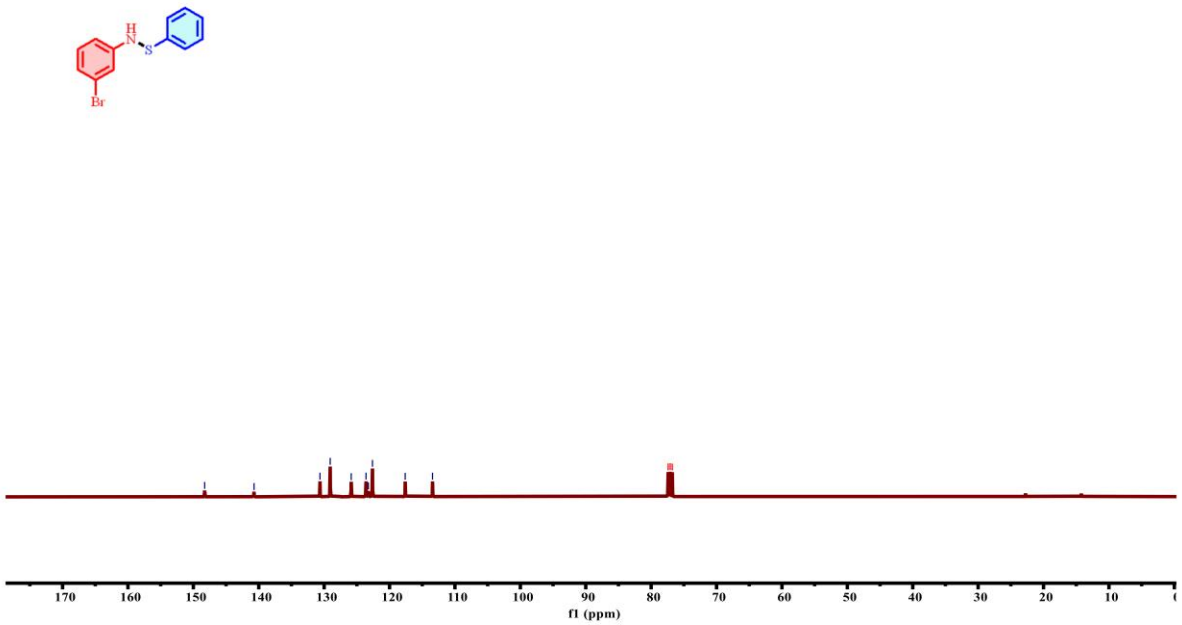
(Table 2, Entry 3h)



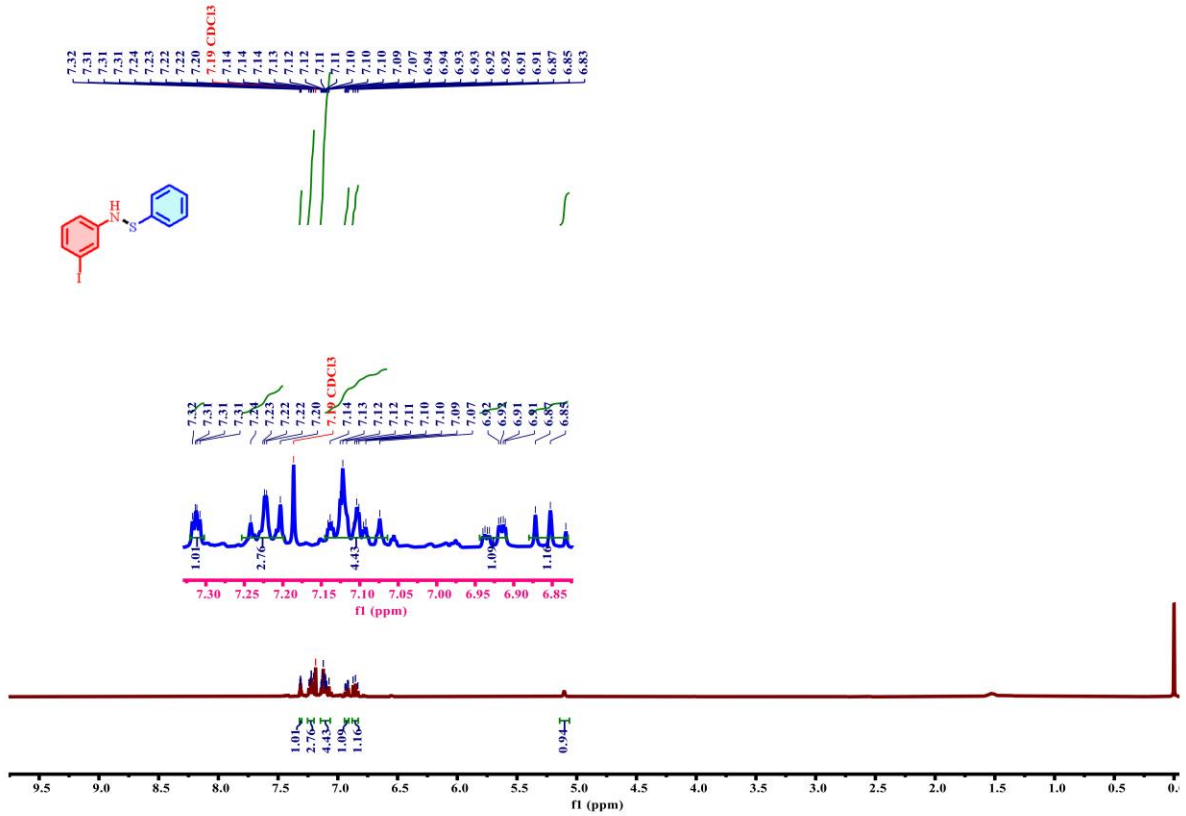


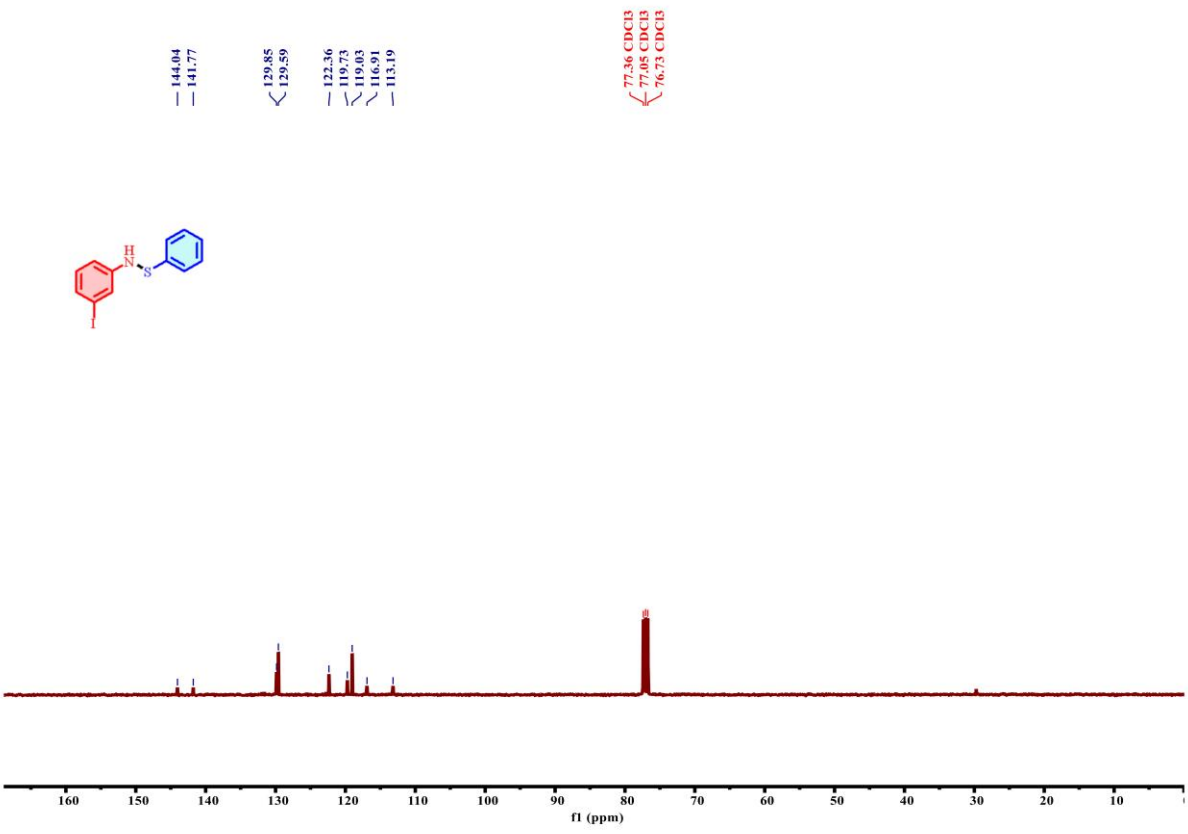
(Table 2, Entry 3i)



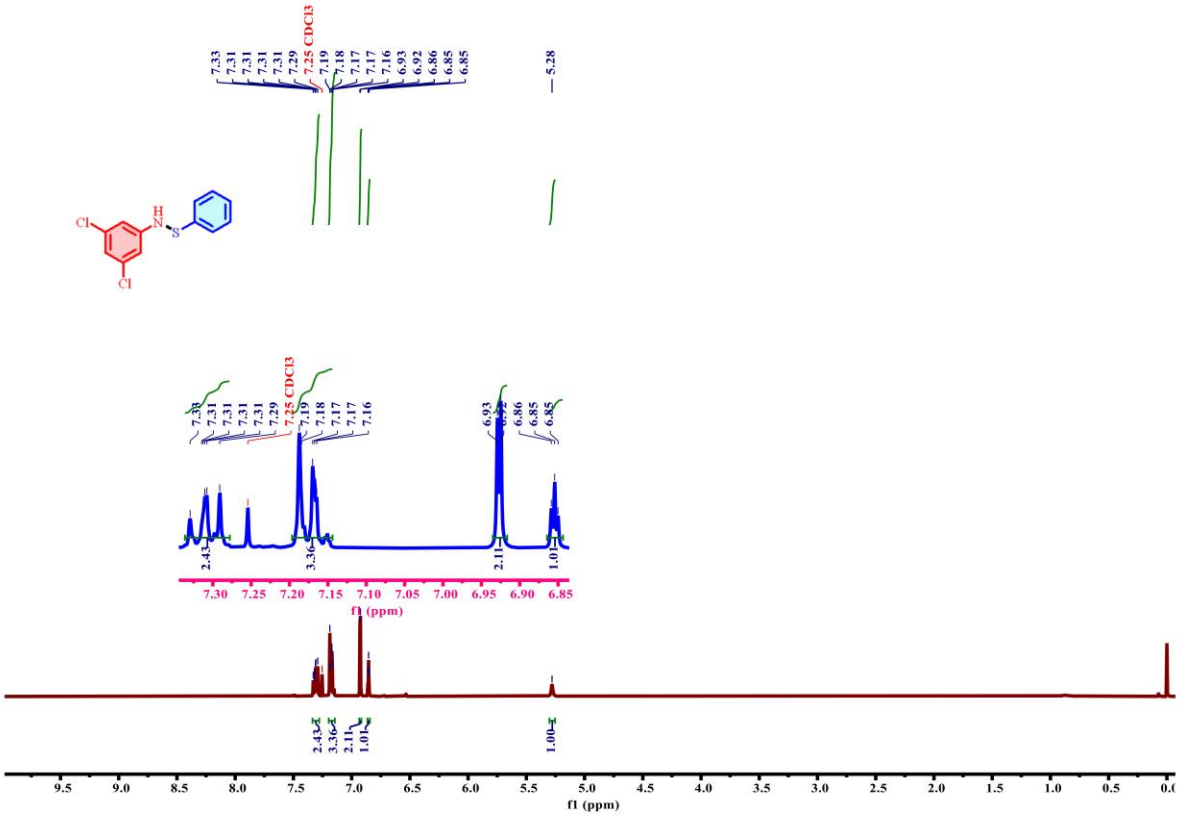


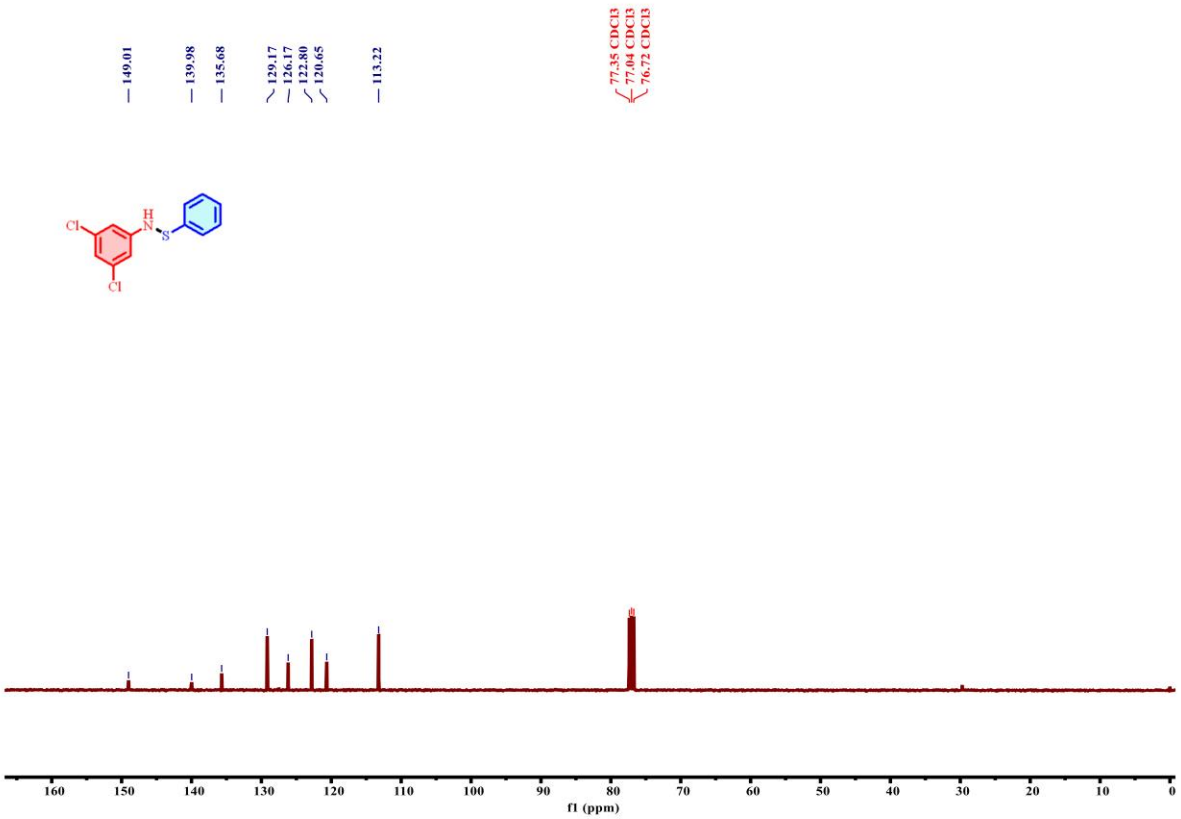
(Table 2, Entry 3j)



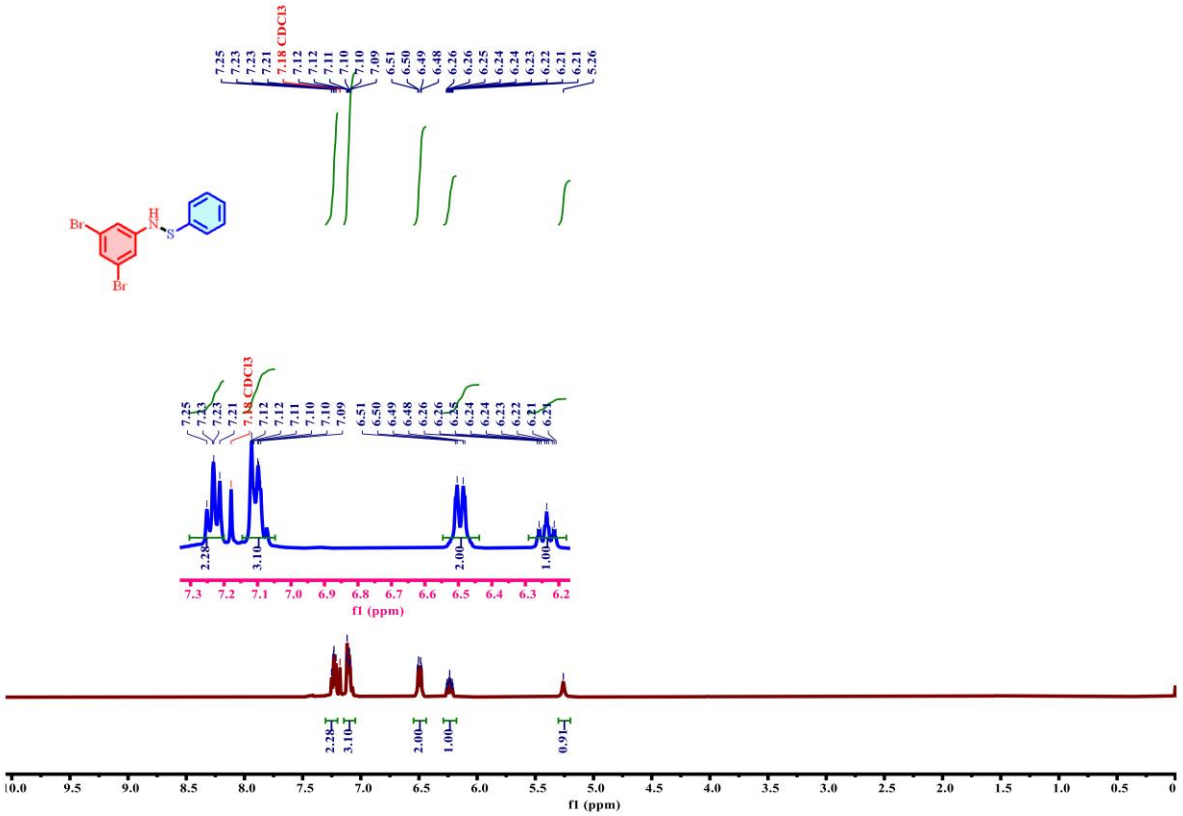


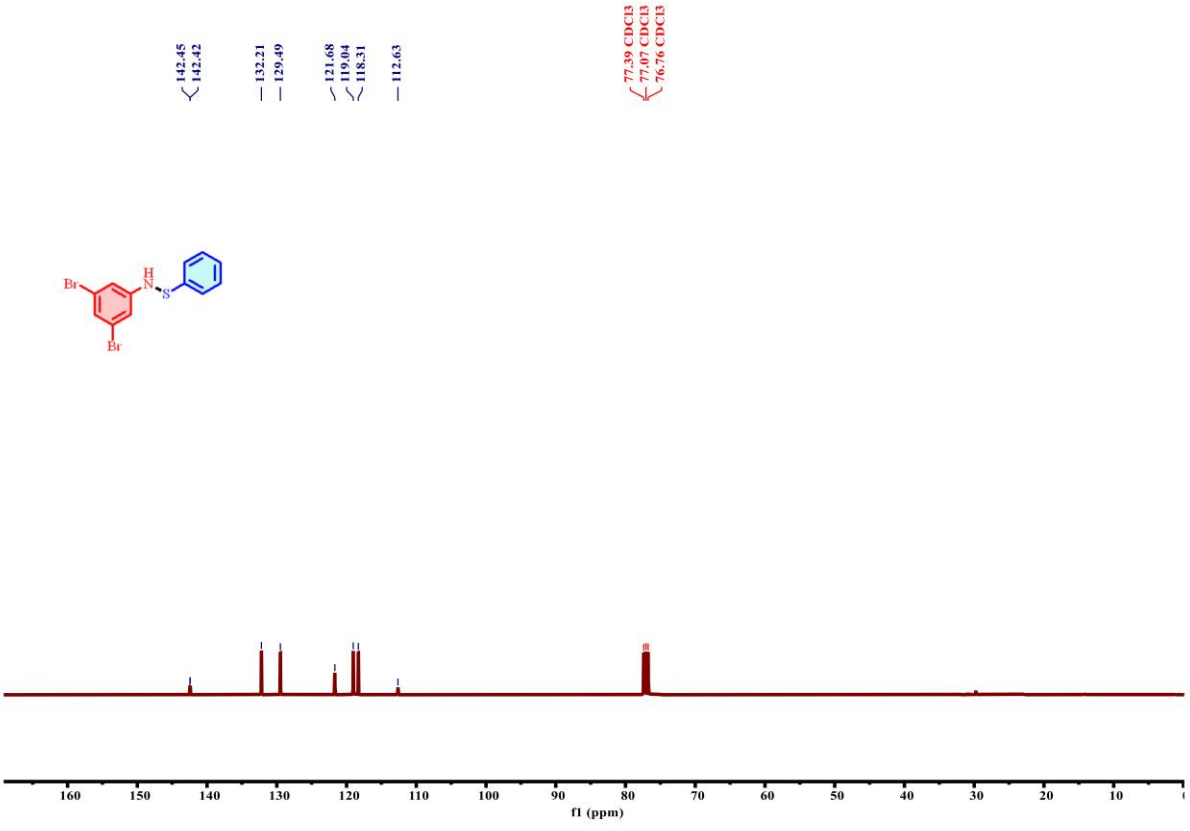
(Table 2, Entry 3k)



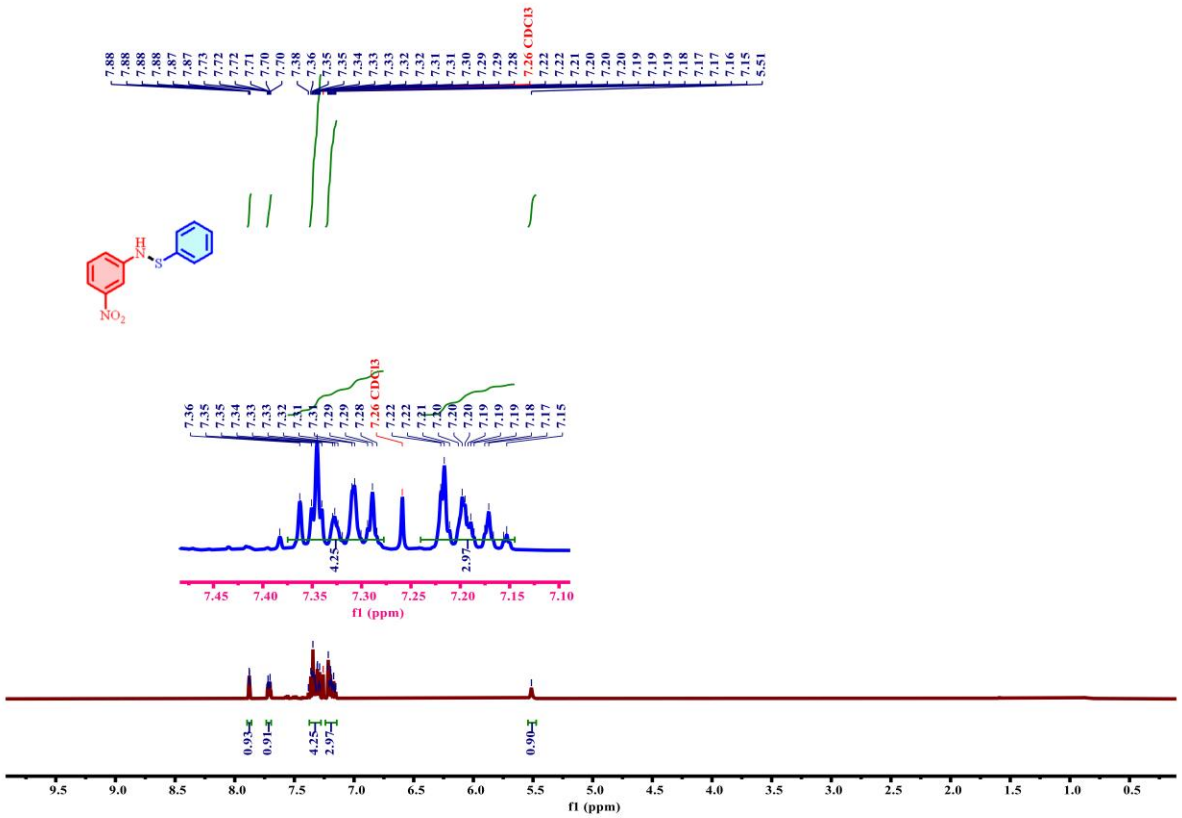


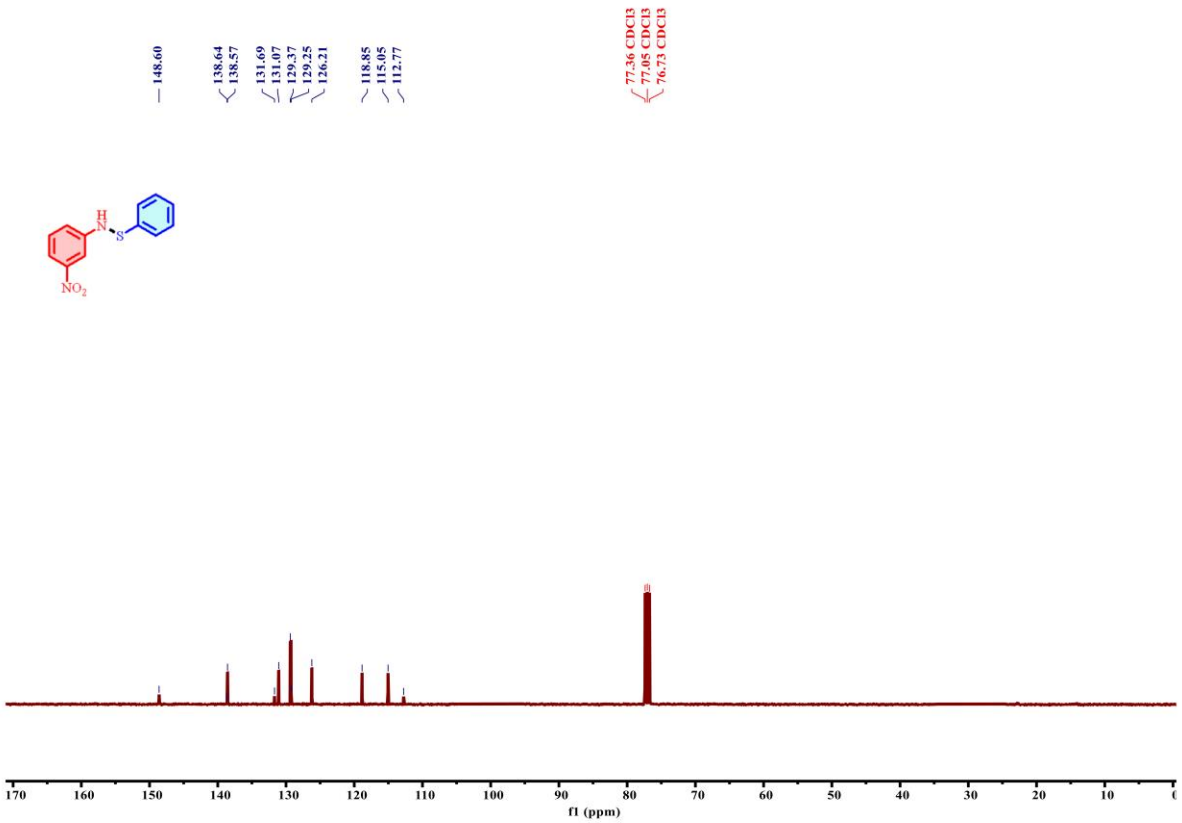
(Table 2, Entry 3l)



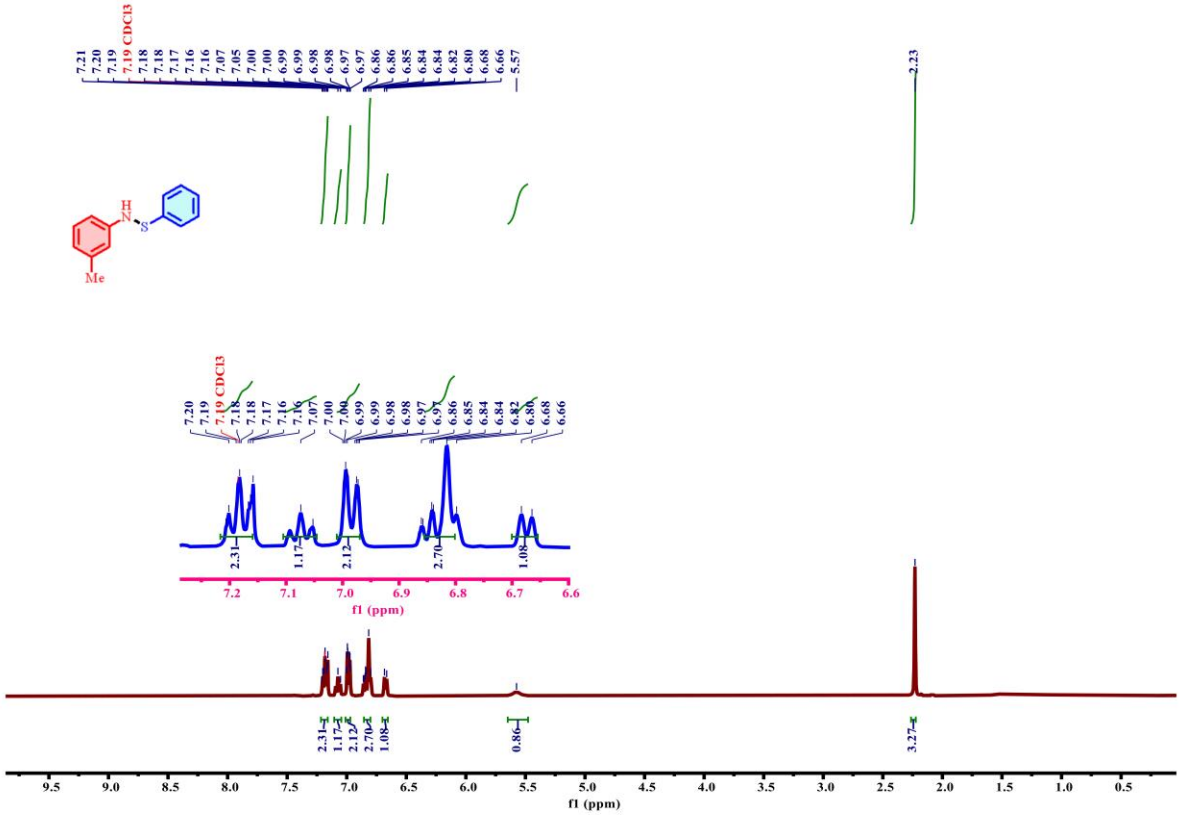


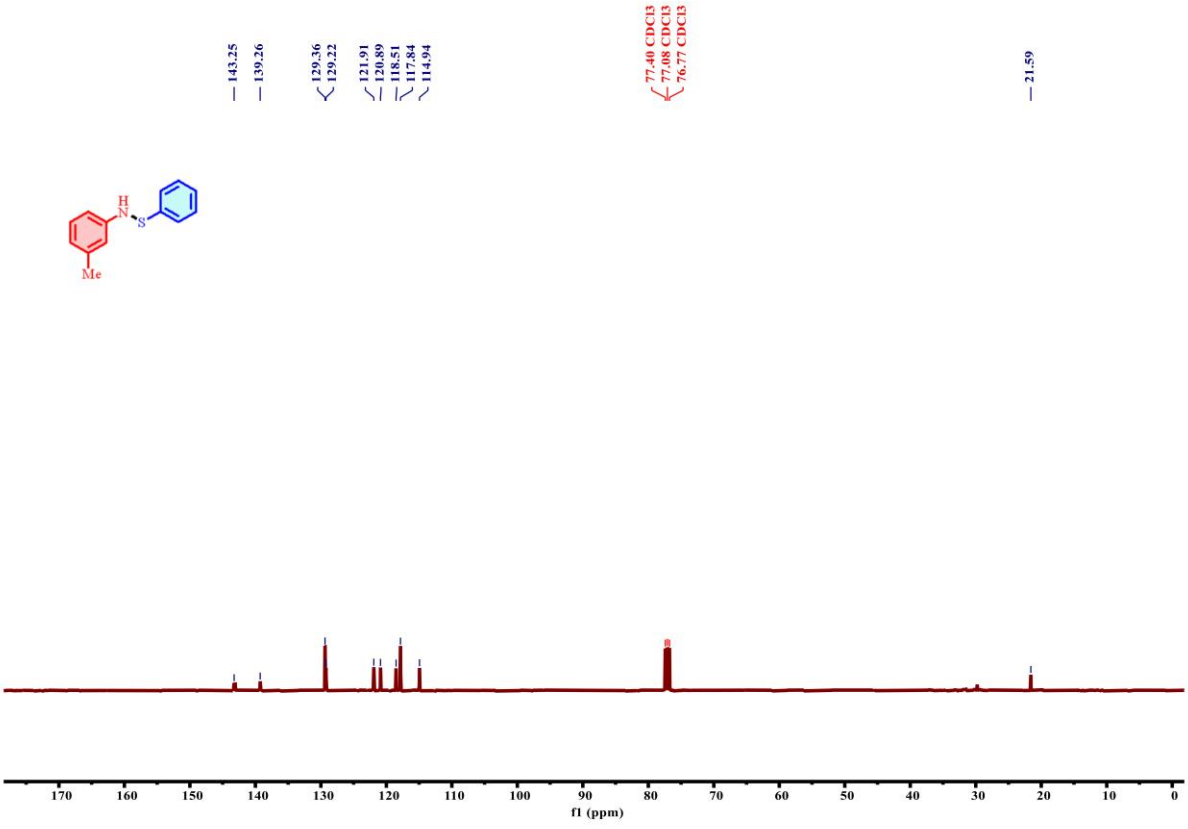
(Table 2, Entry 3m)



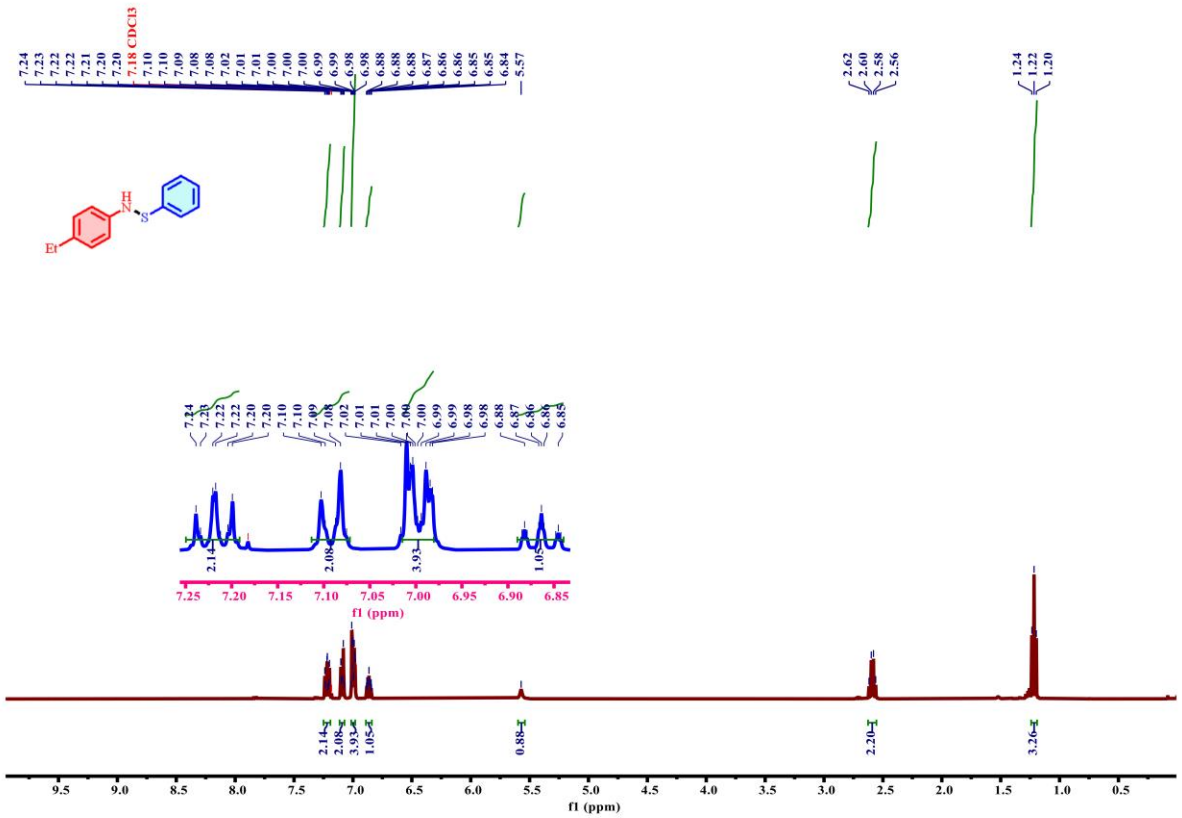


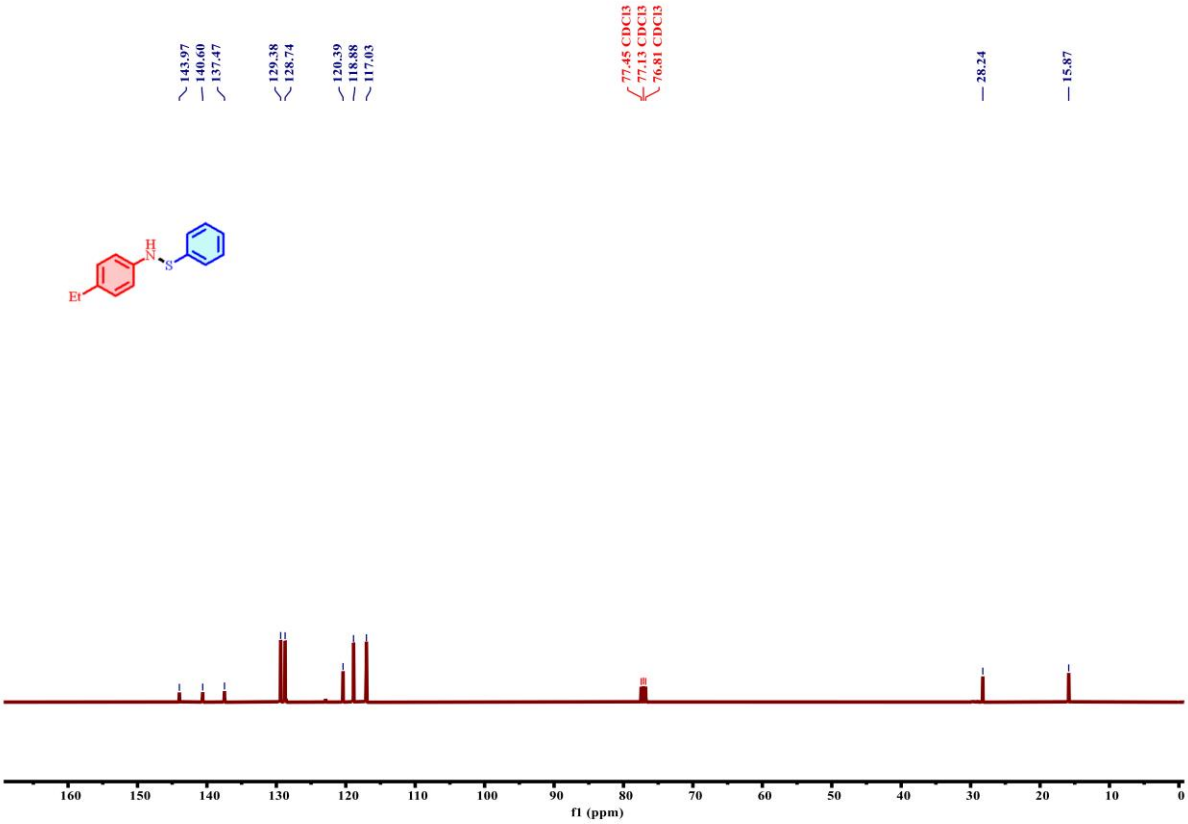
(Table 2, Entry 3n)



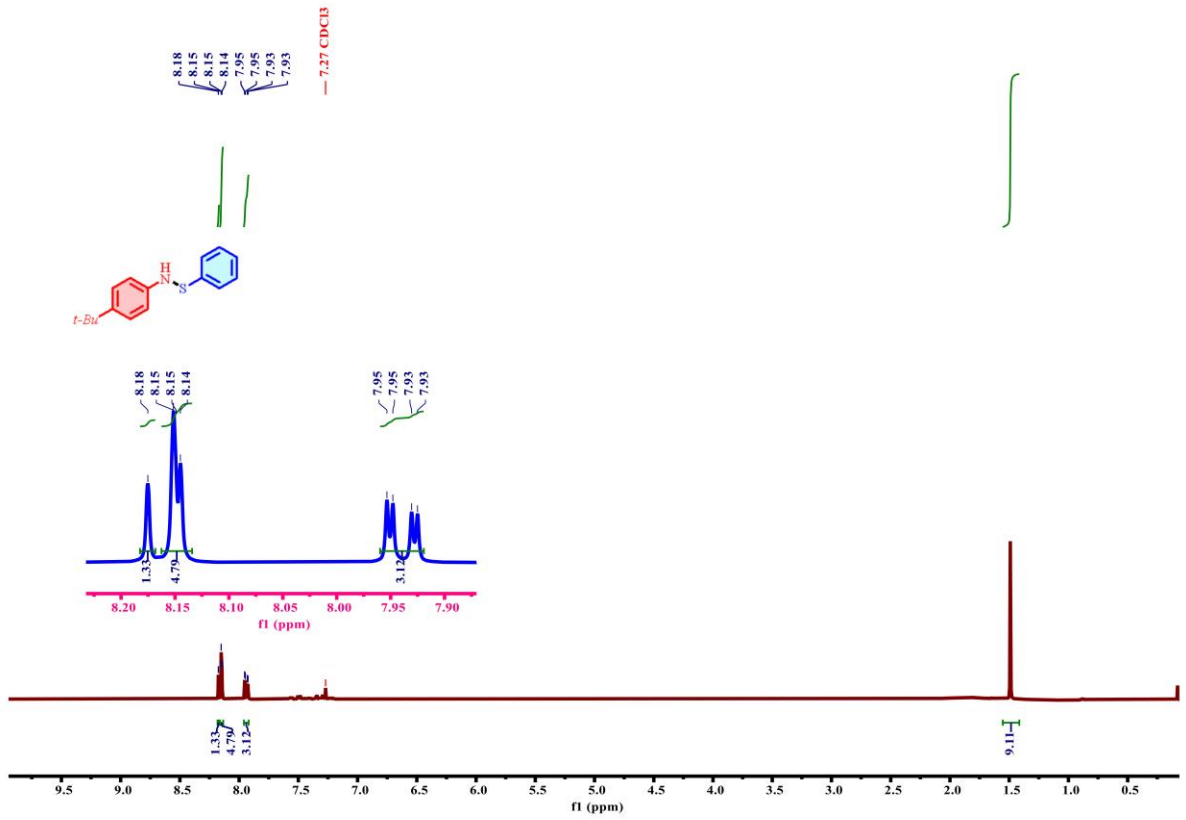


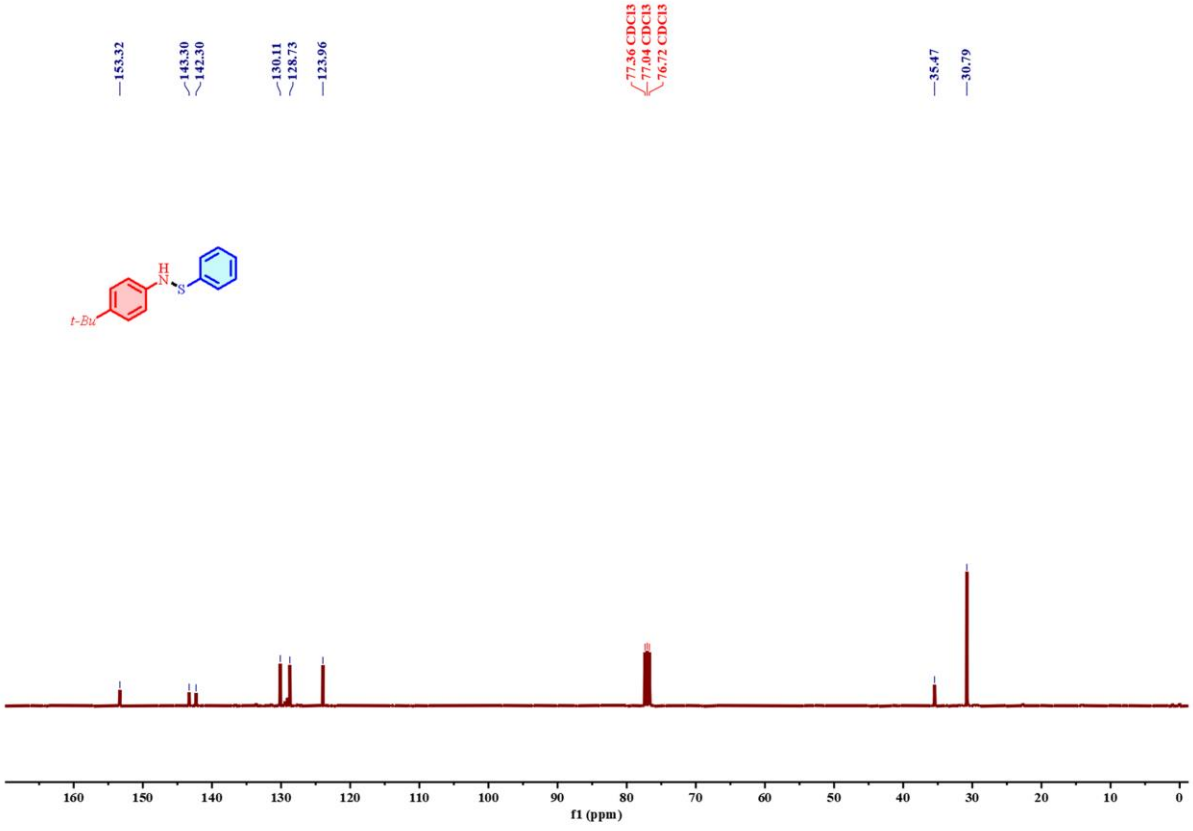
(Table, Entry 3o)





(Table, Entry 3p)





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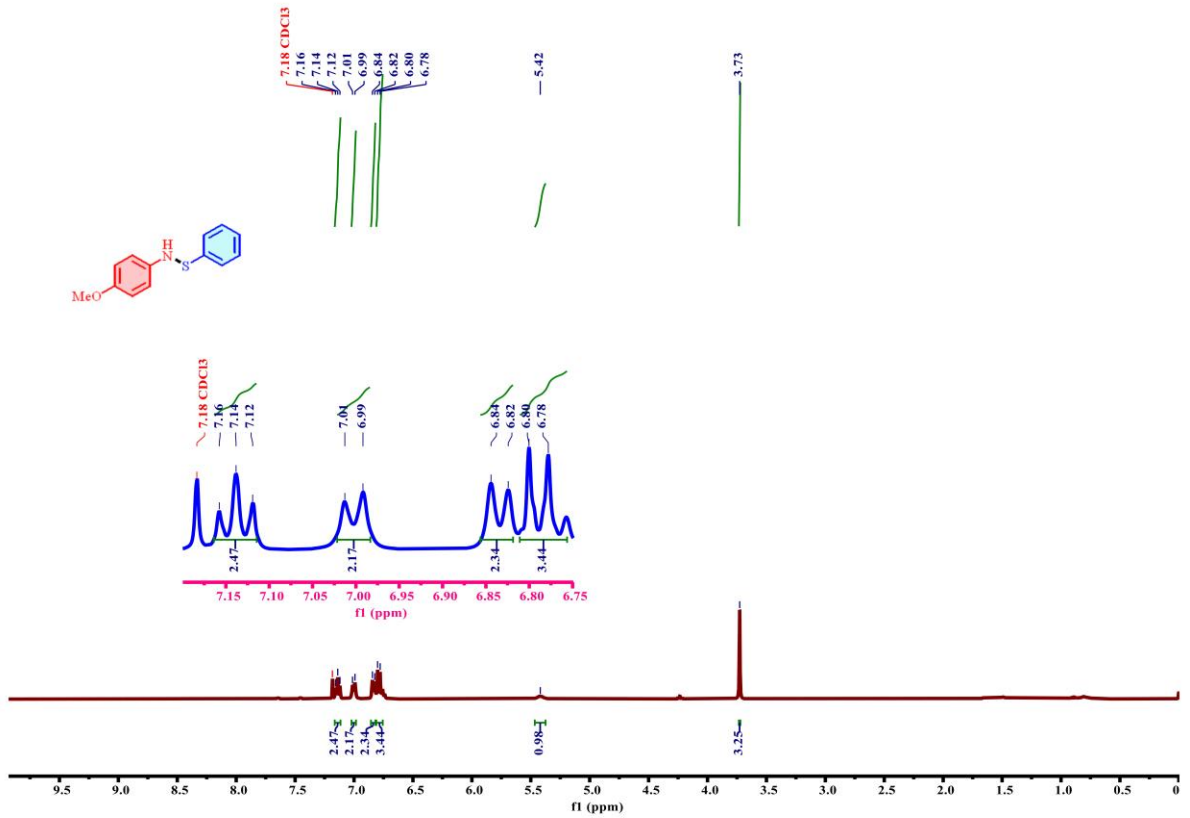
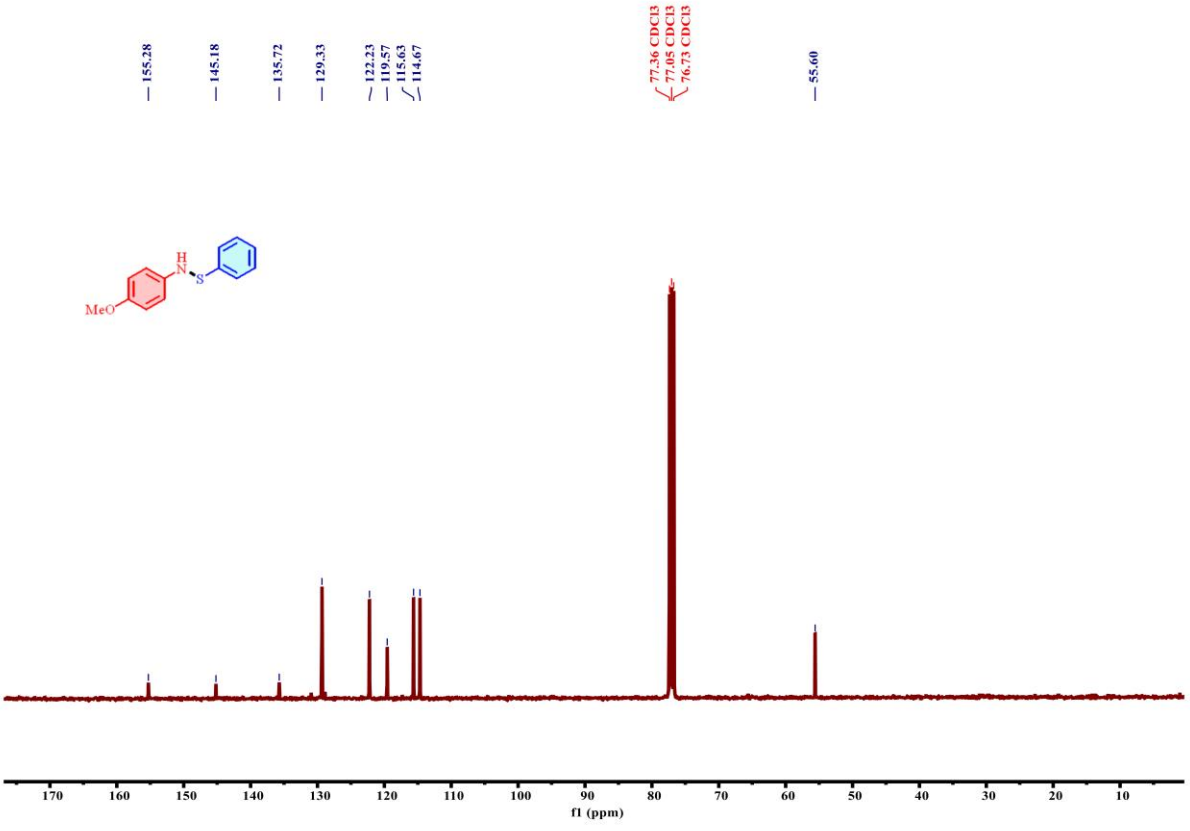
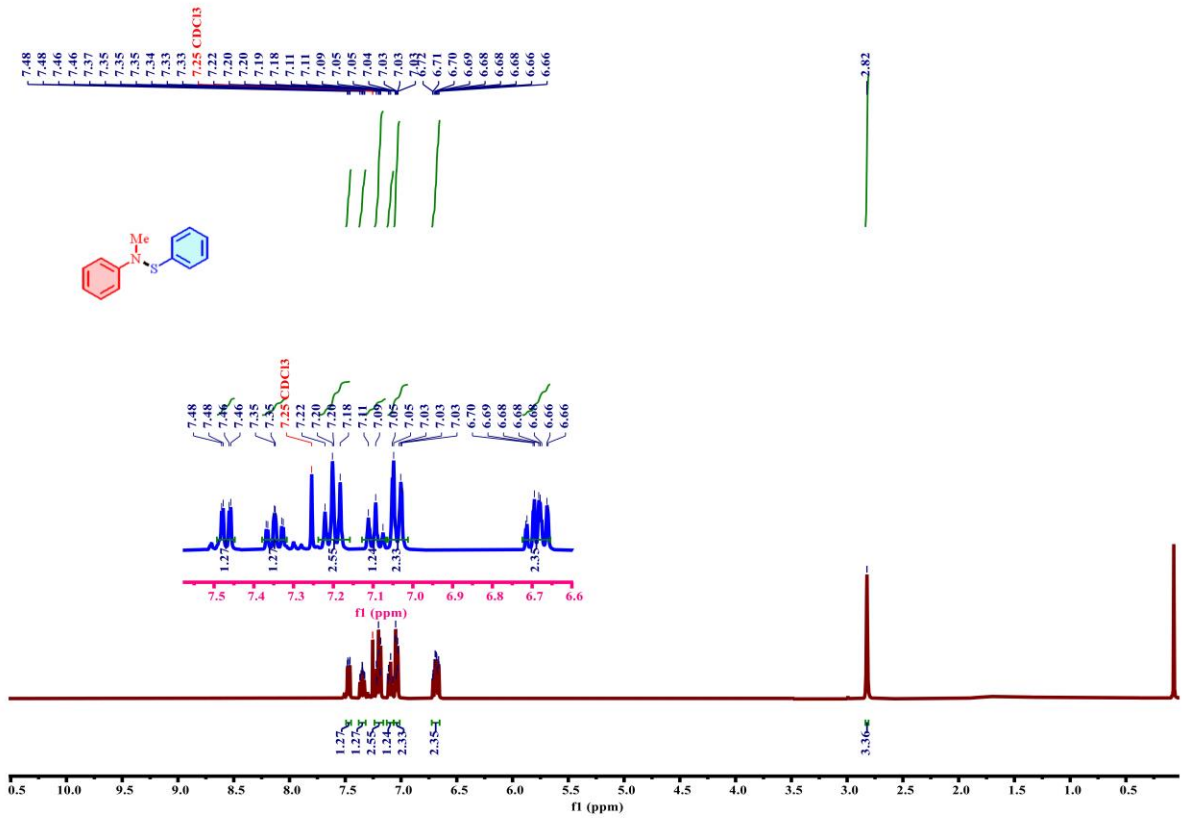
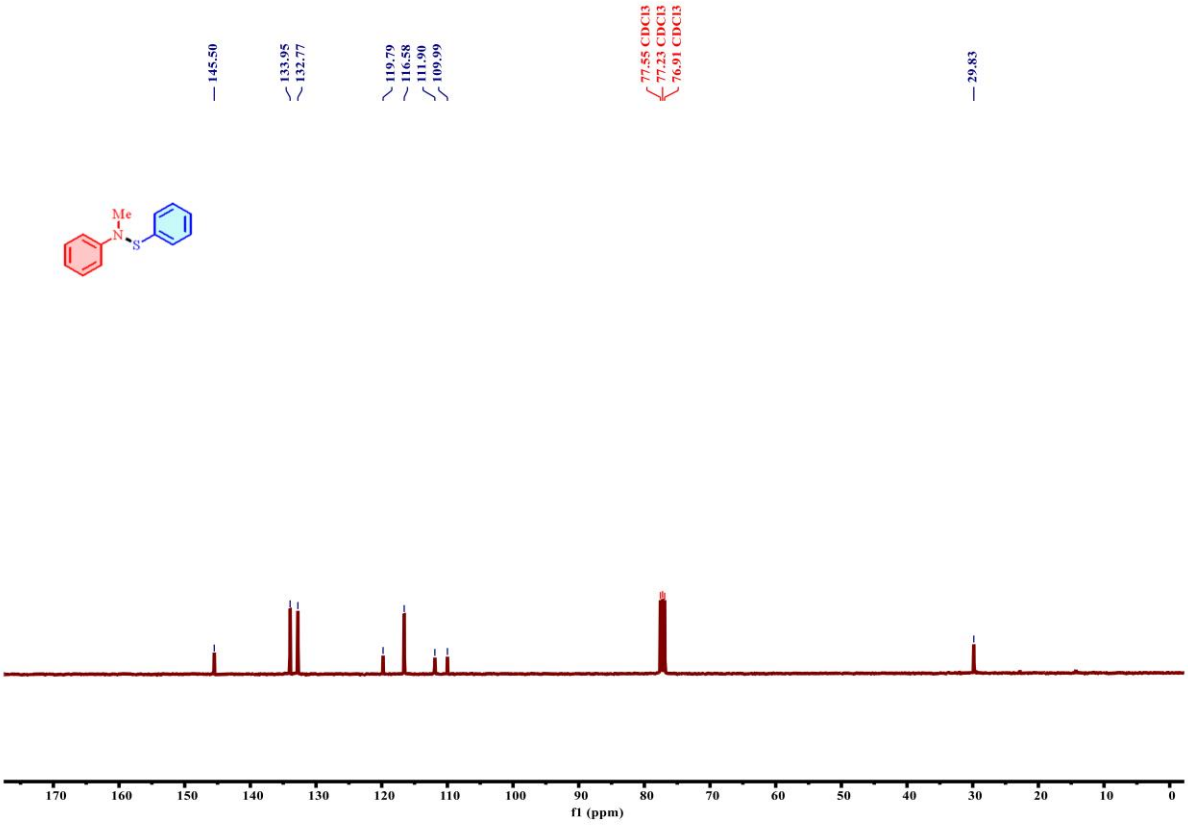


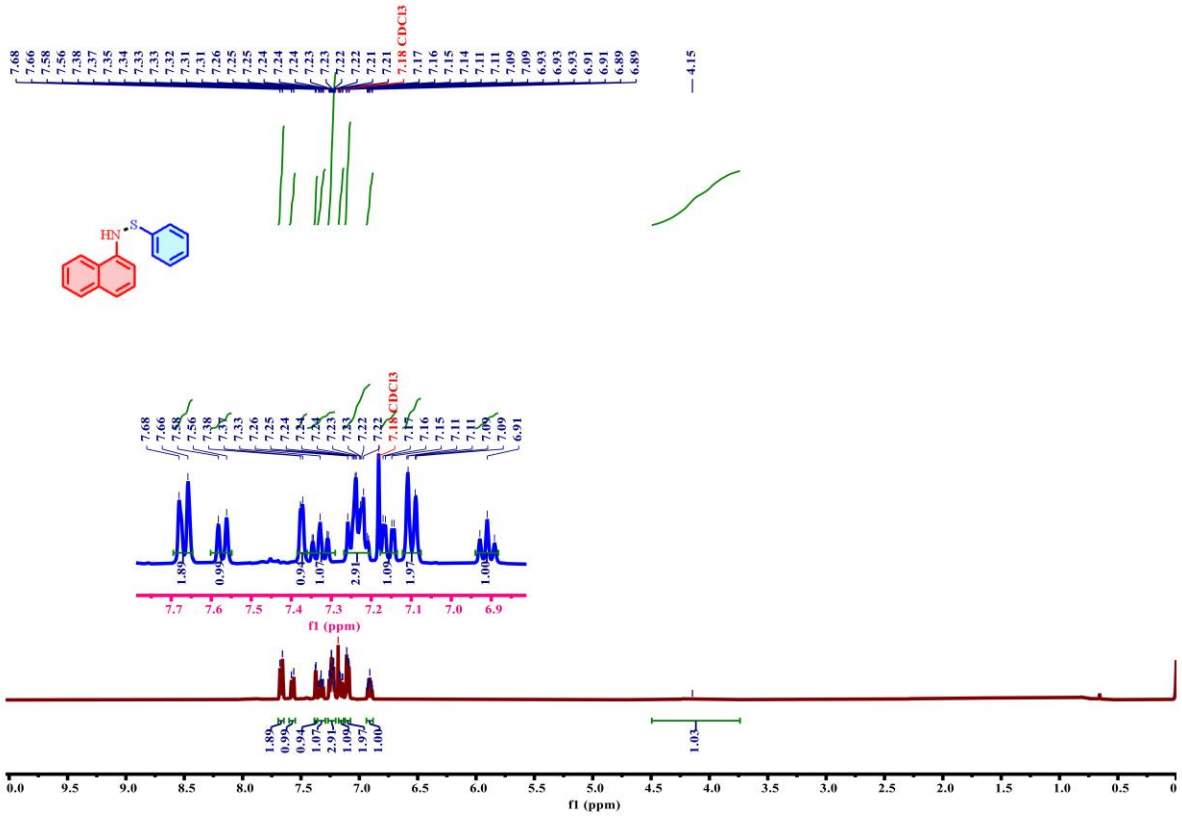


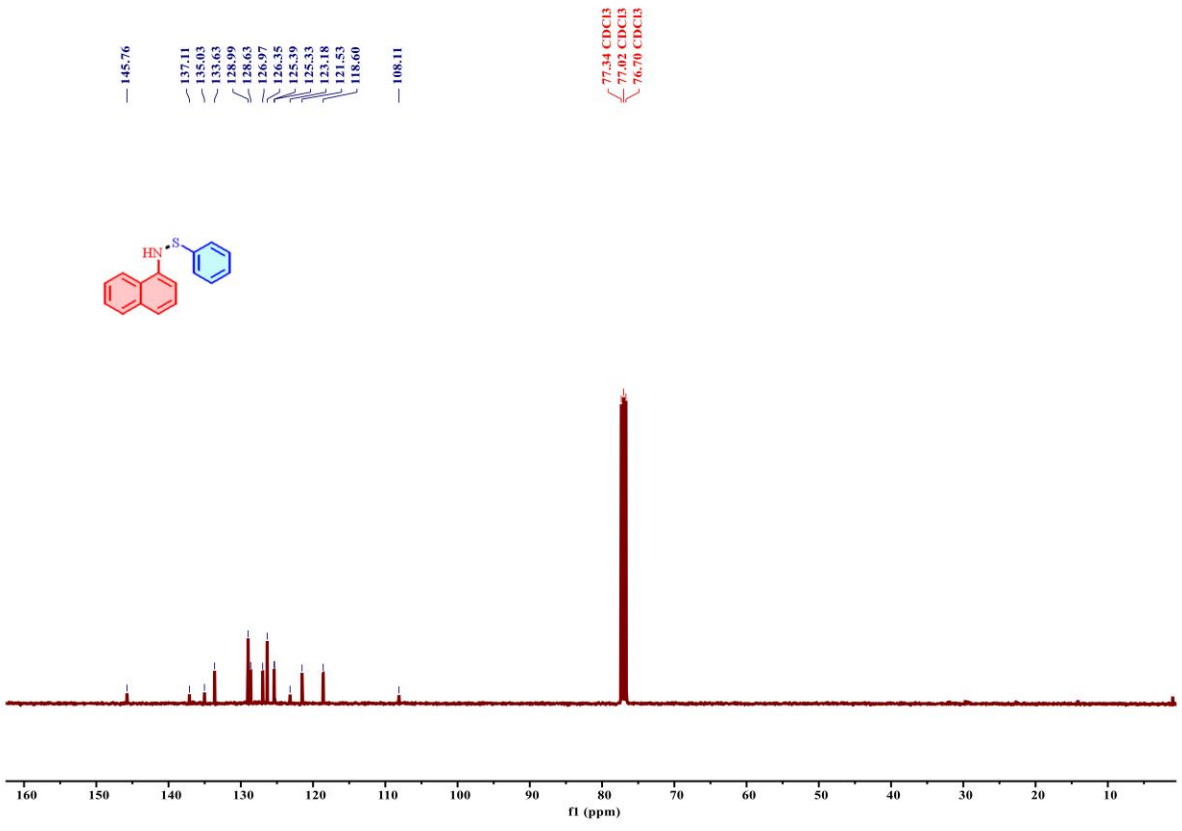
(Table 2, Entry 3r)



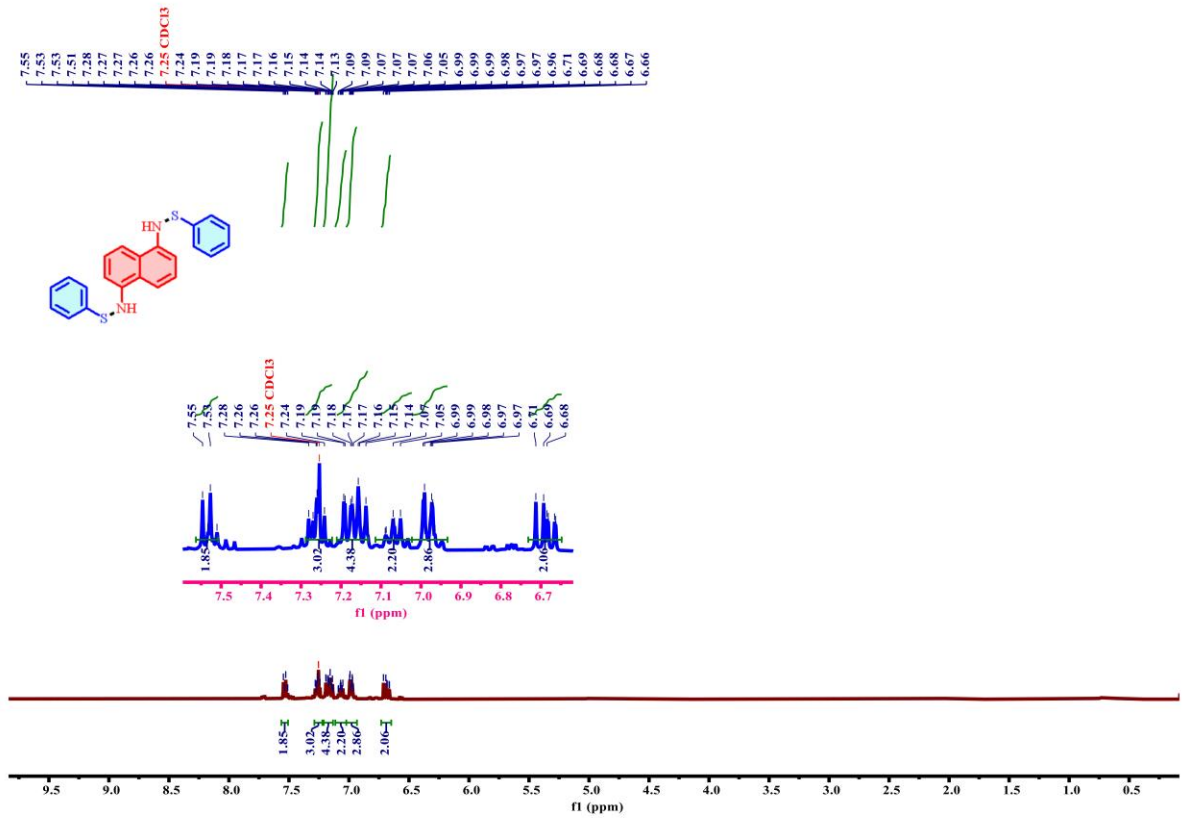


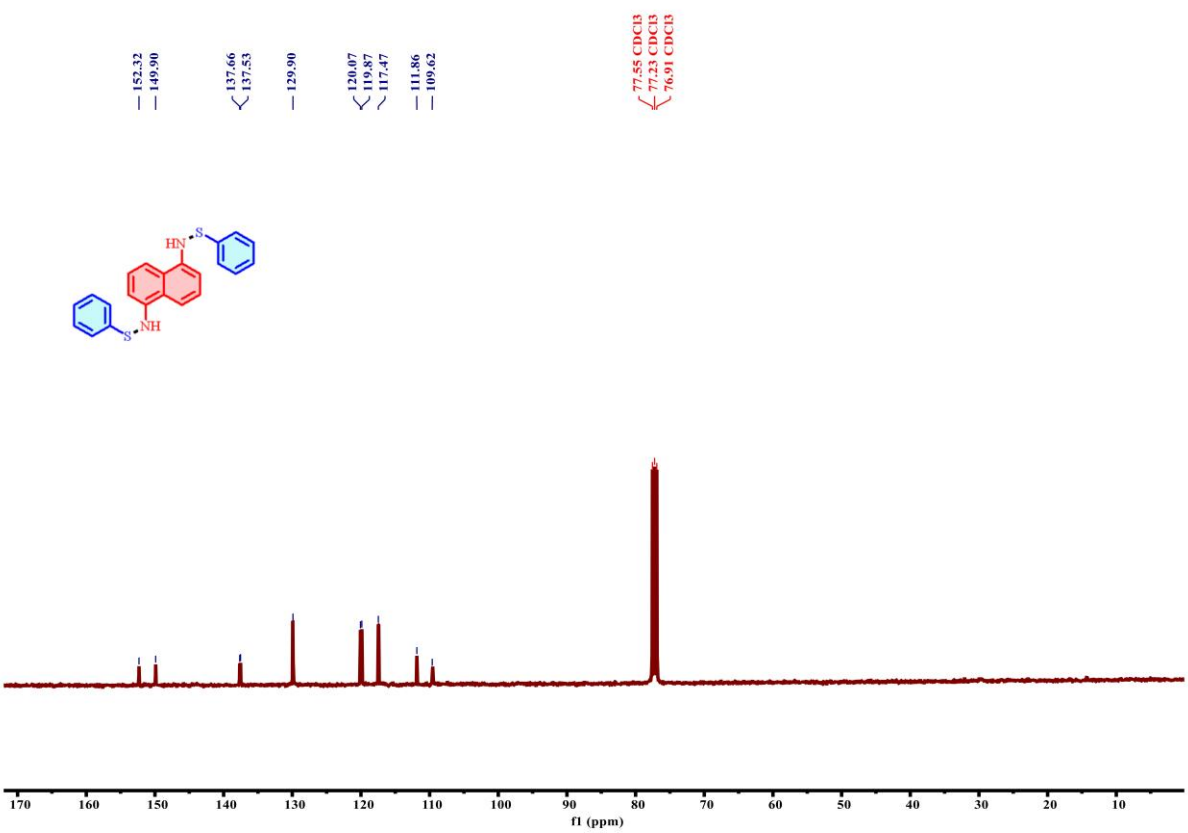
(Table 2, Entry 3s)



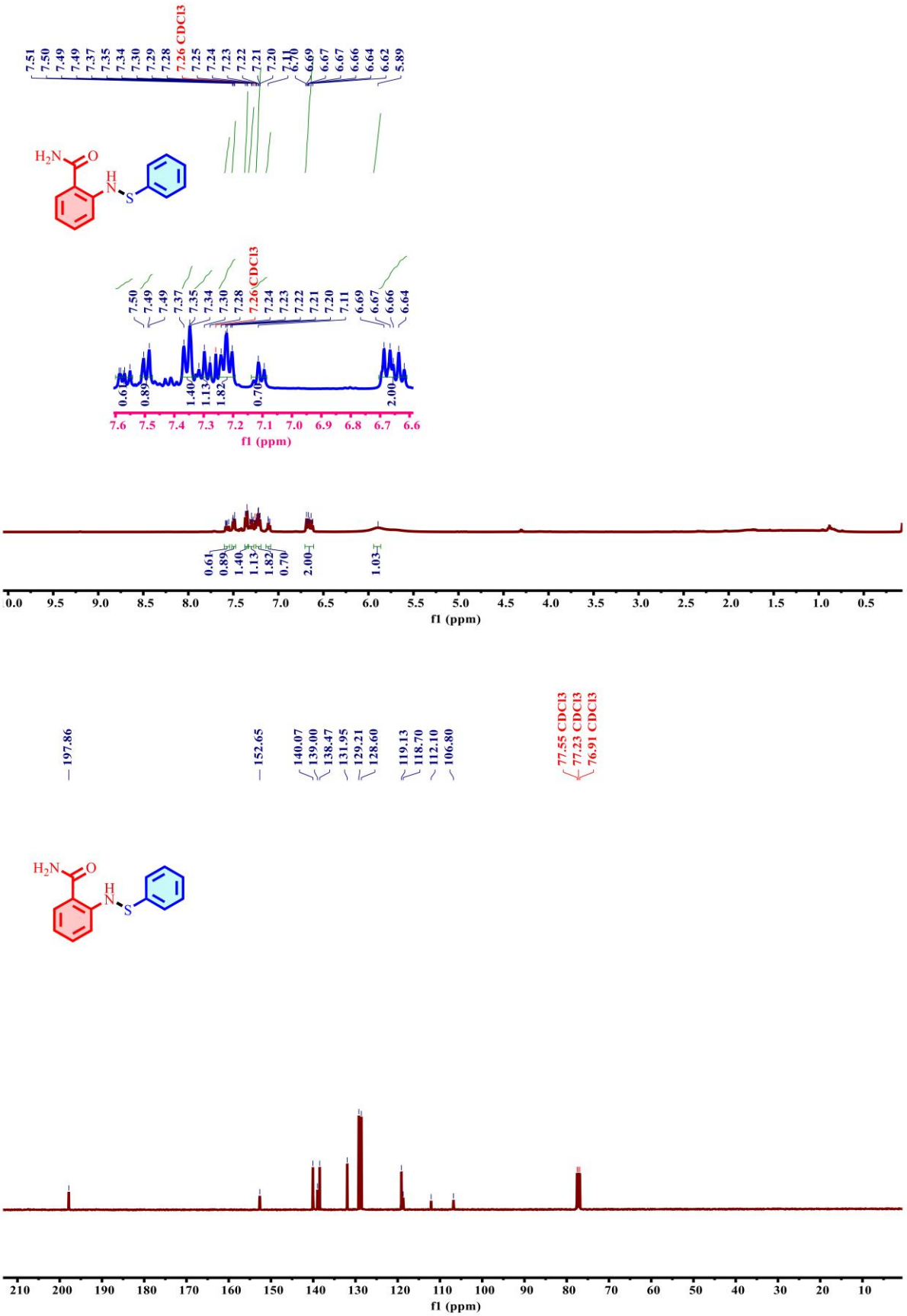


(Table 2, Entry 3t)

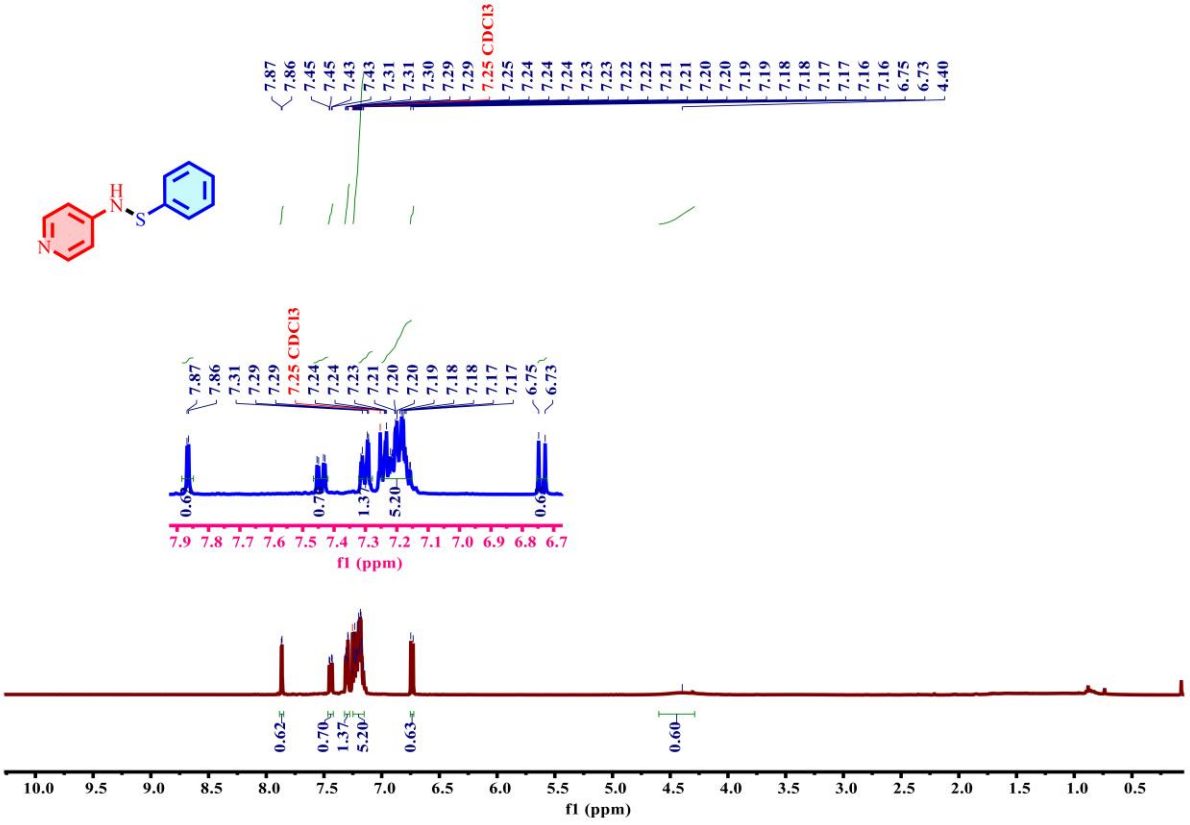


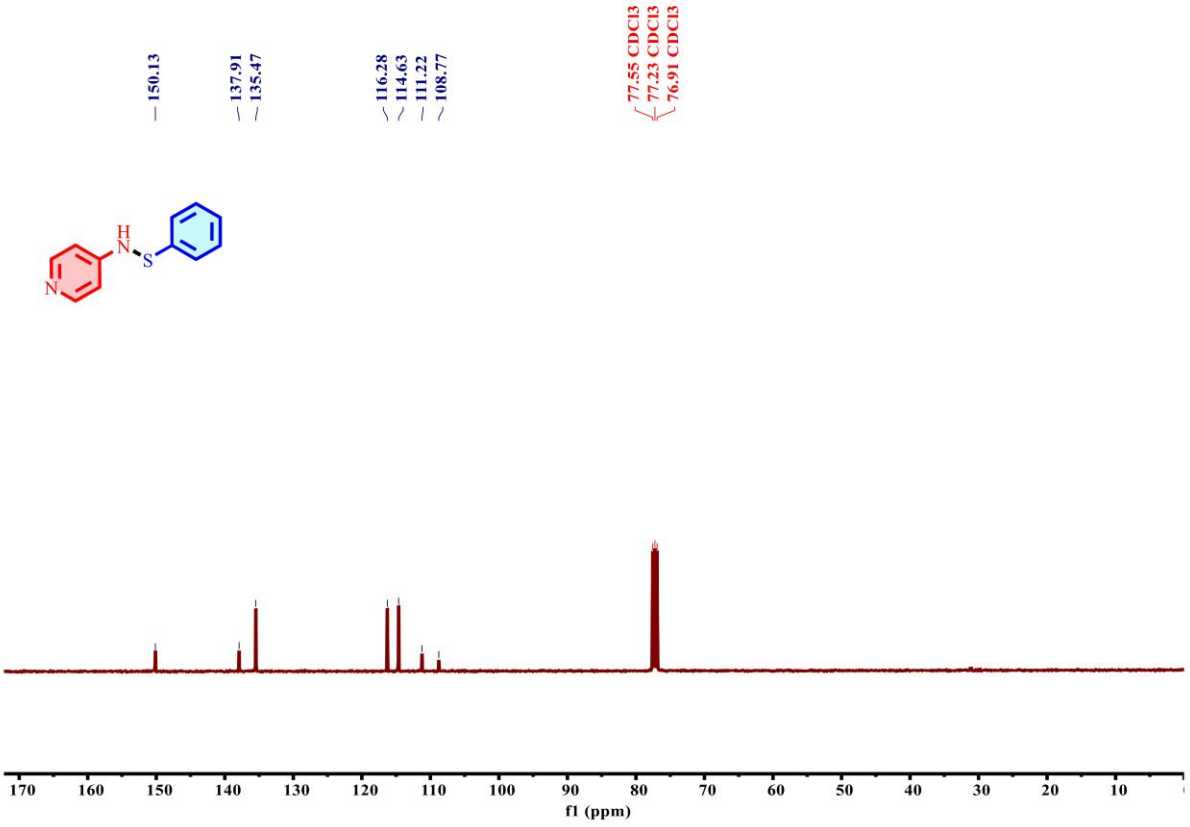


(Table 2, Entry 3u)

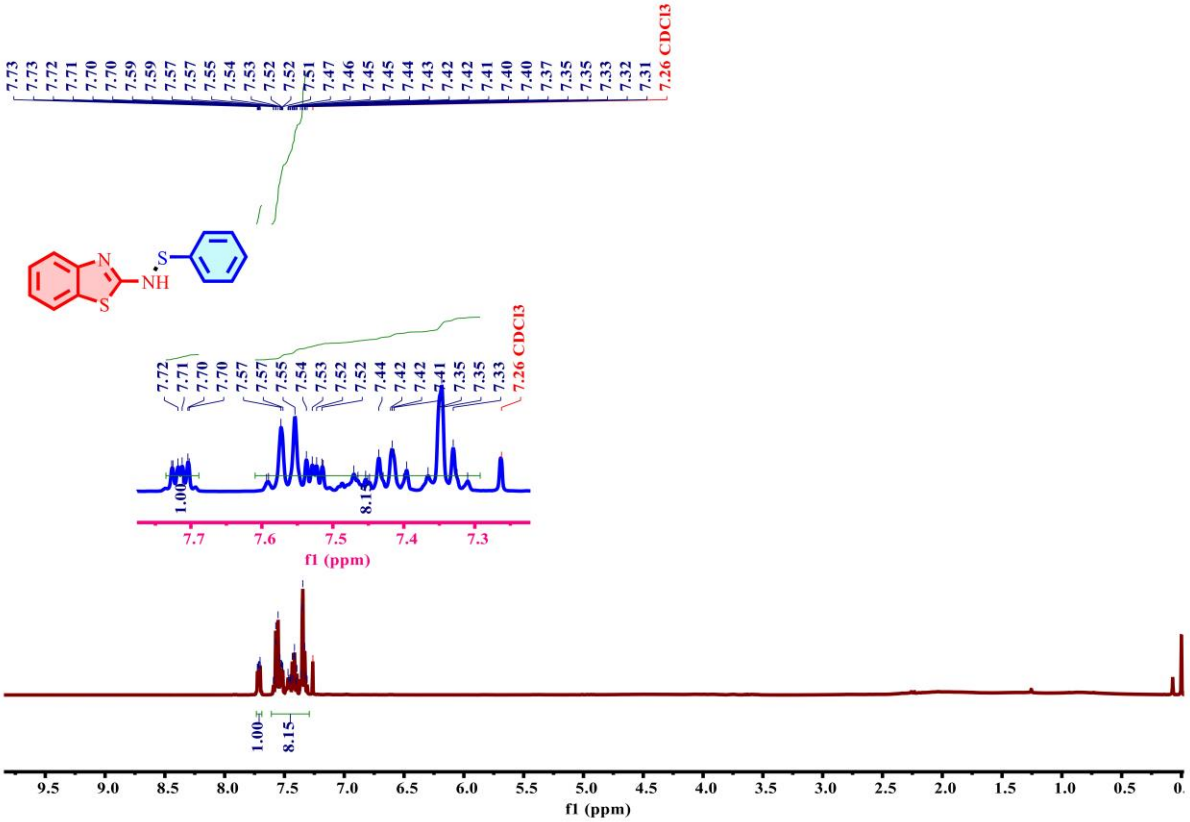


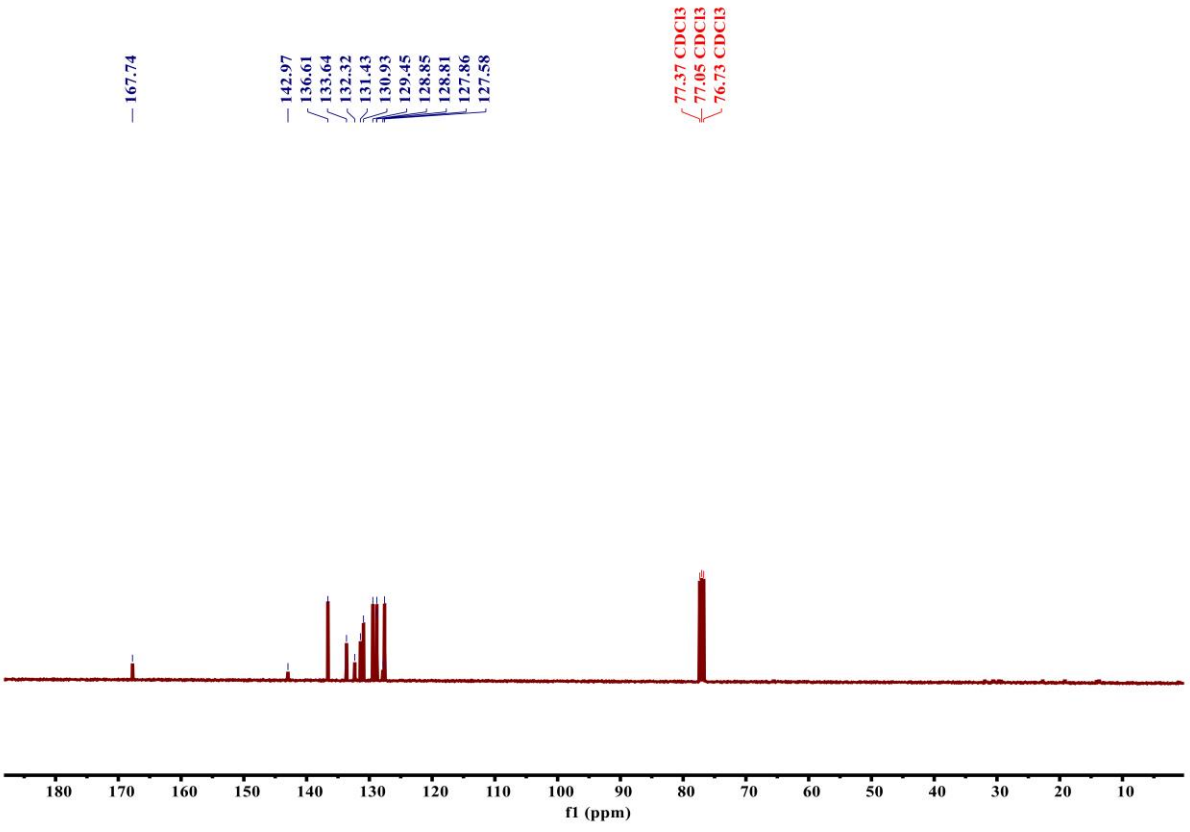
(Table 2, Entry 3v)



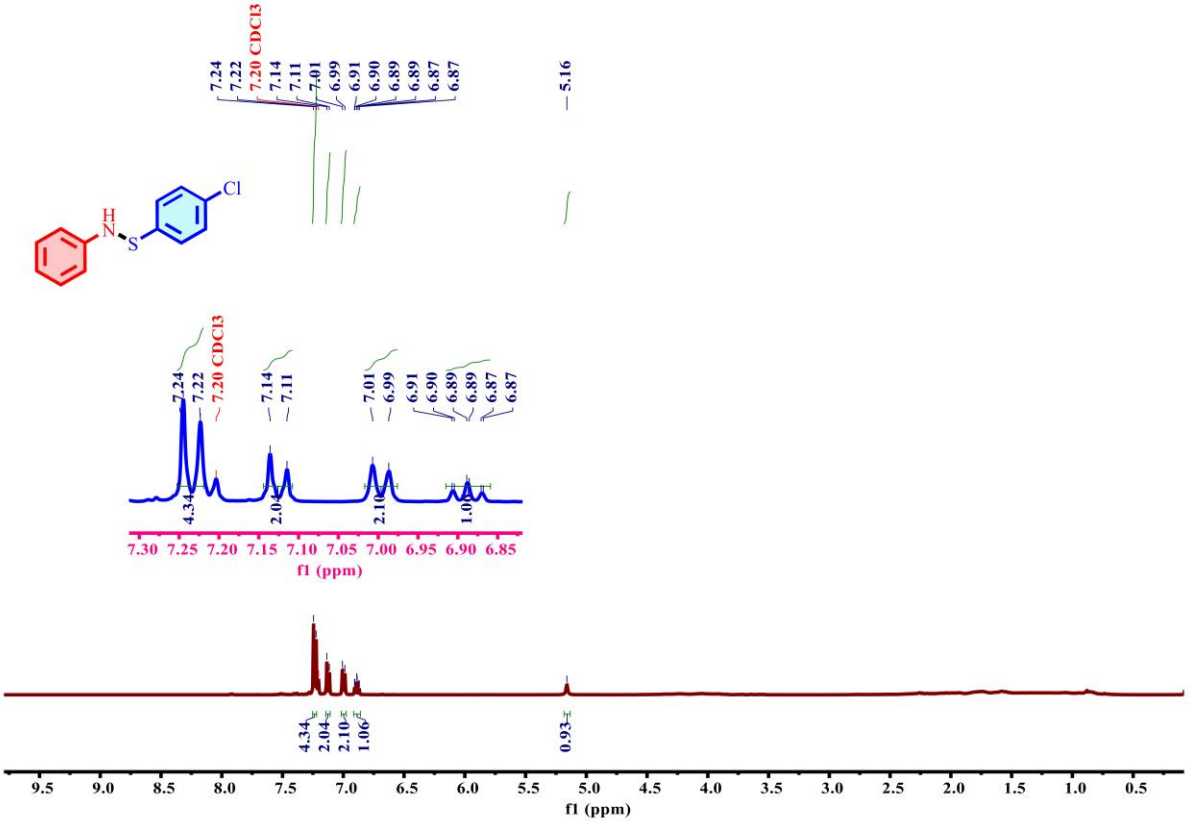


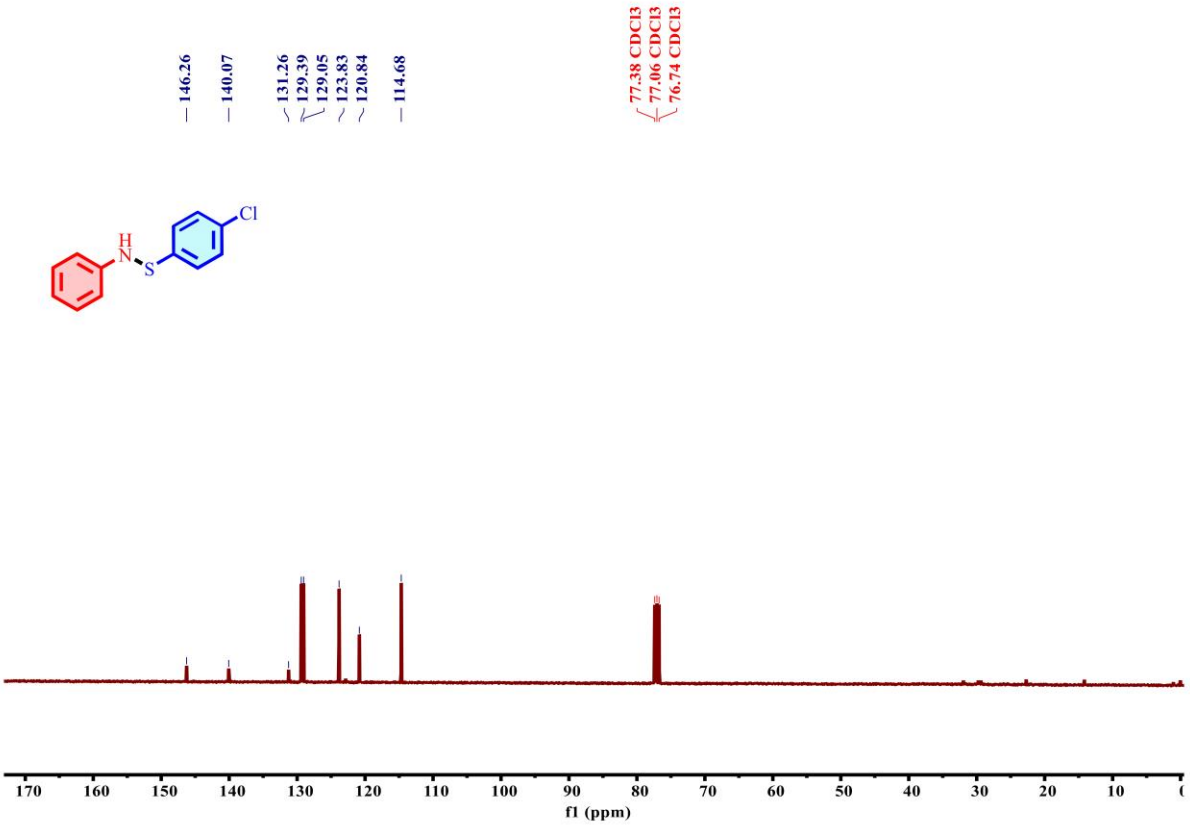
(Table 2, Entry 3w)



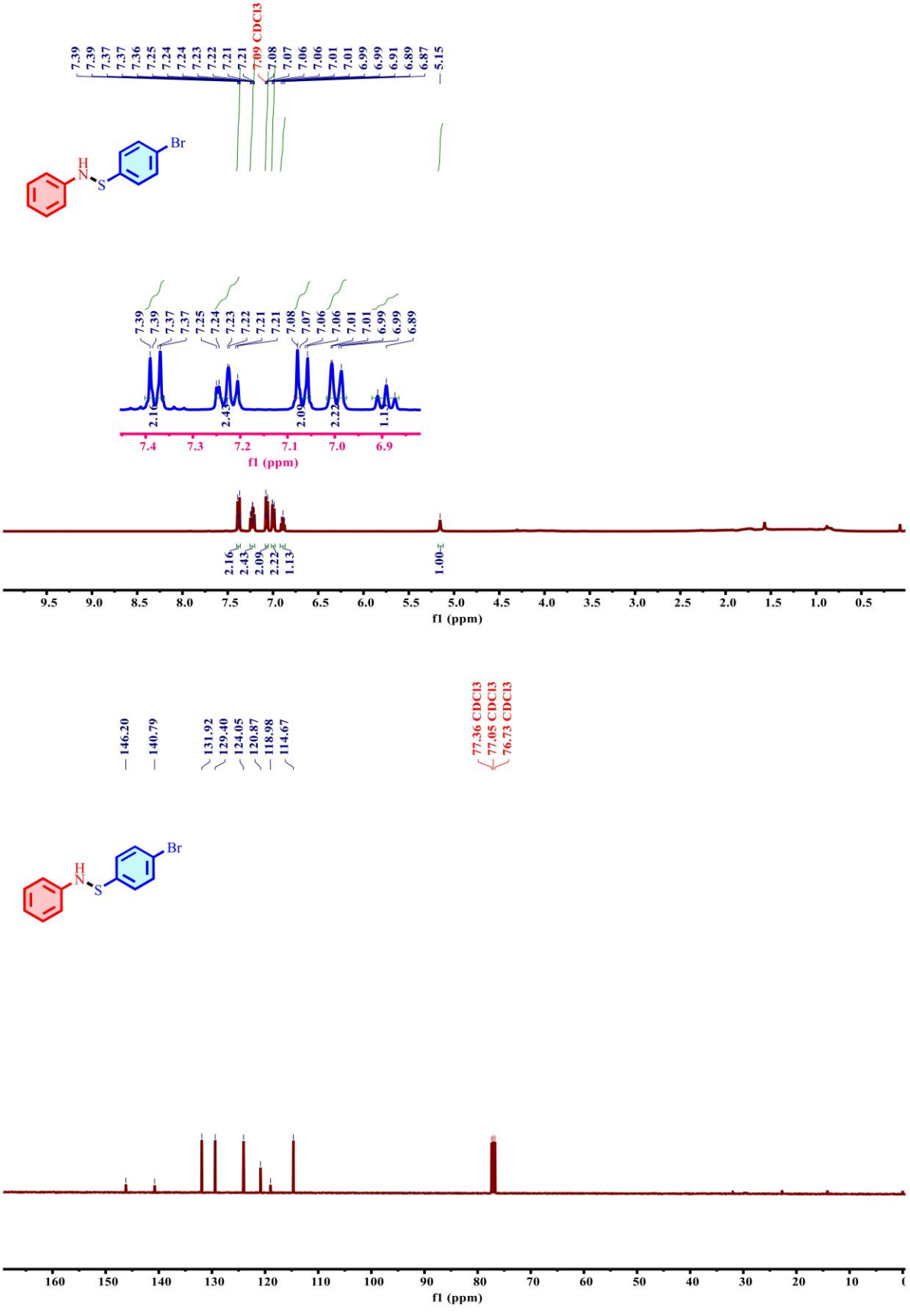


(Table 2, Entry 3x)

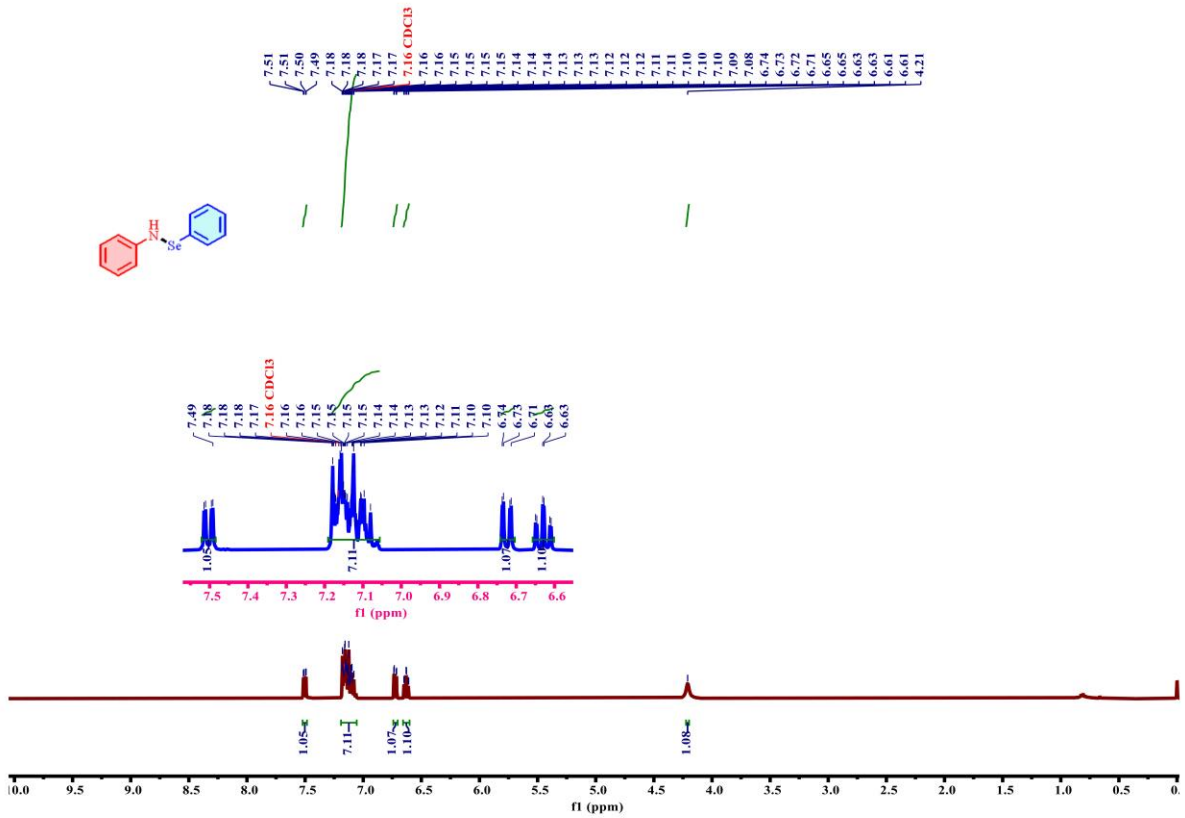


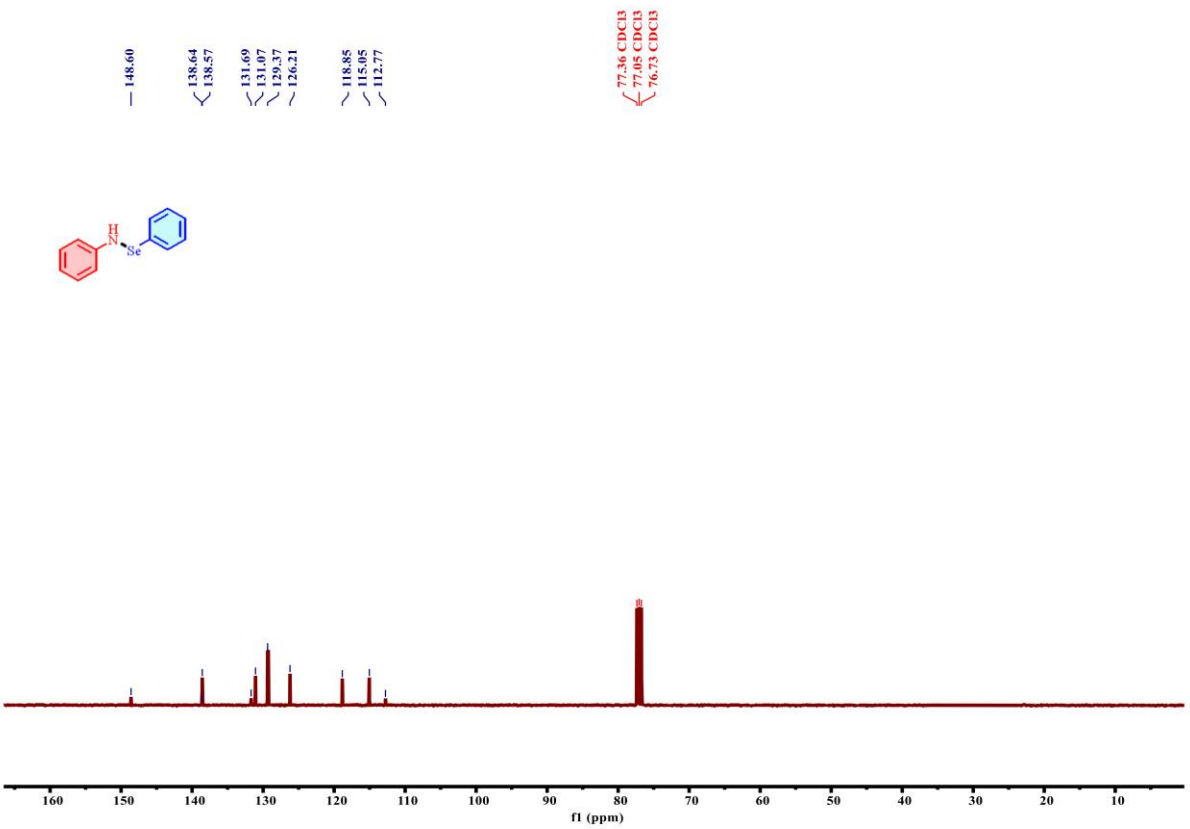


(Table 2, Entry 3y)

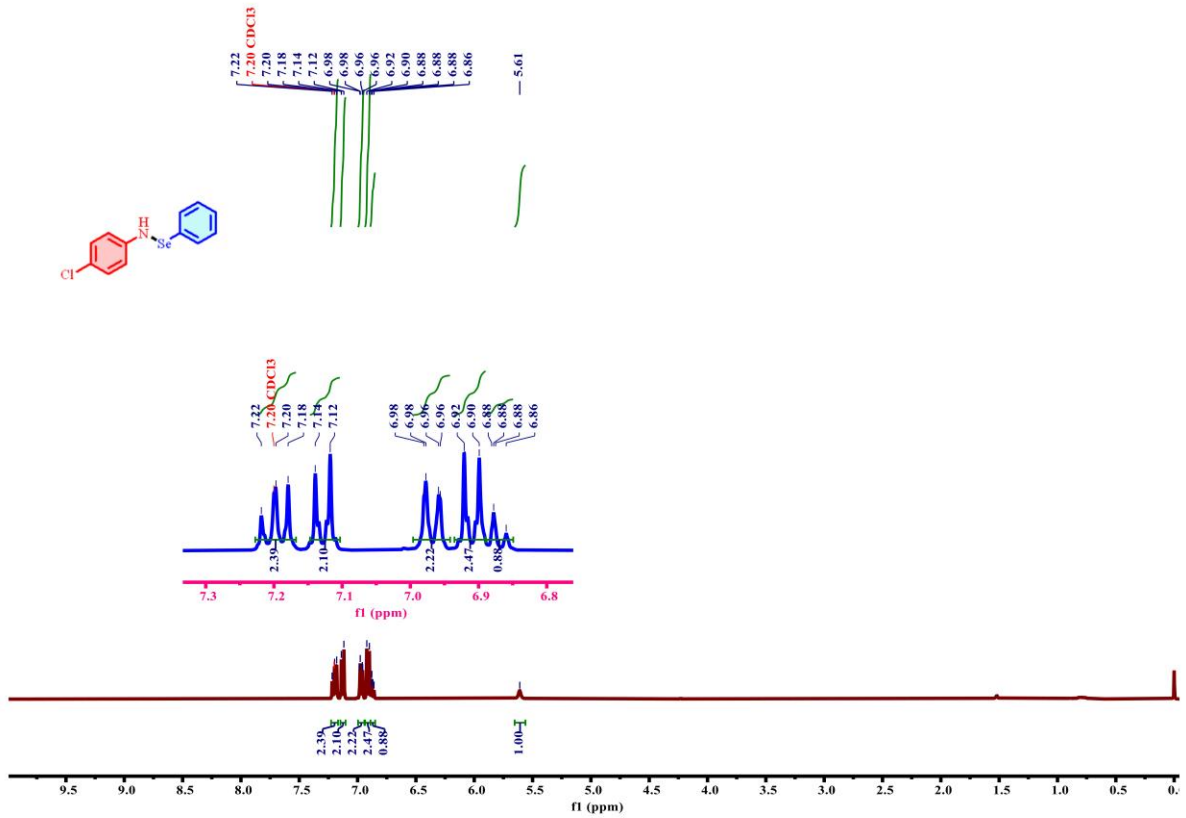


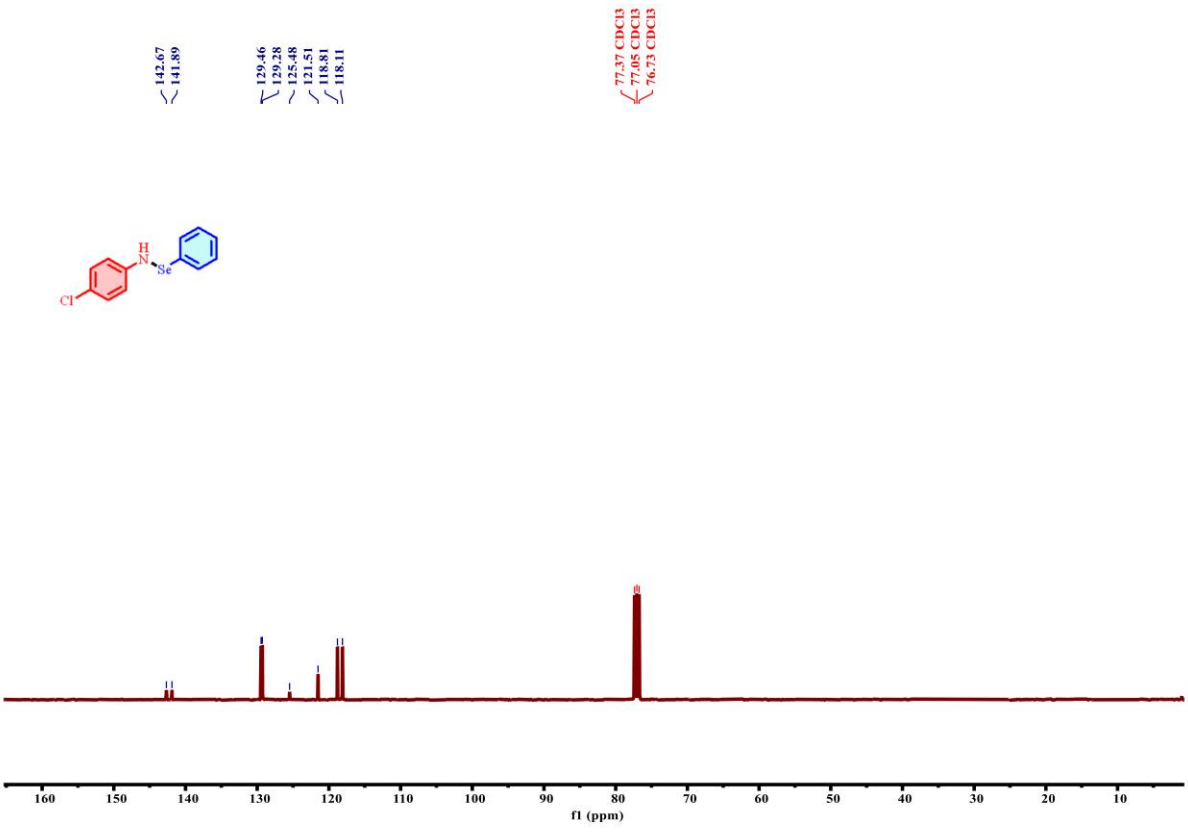
(Table 3, Entry 5a)



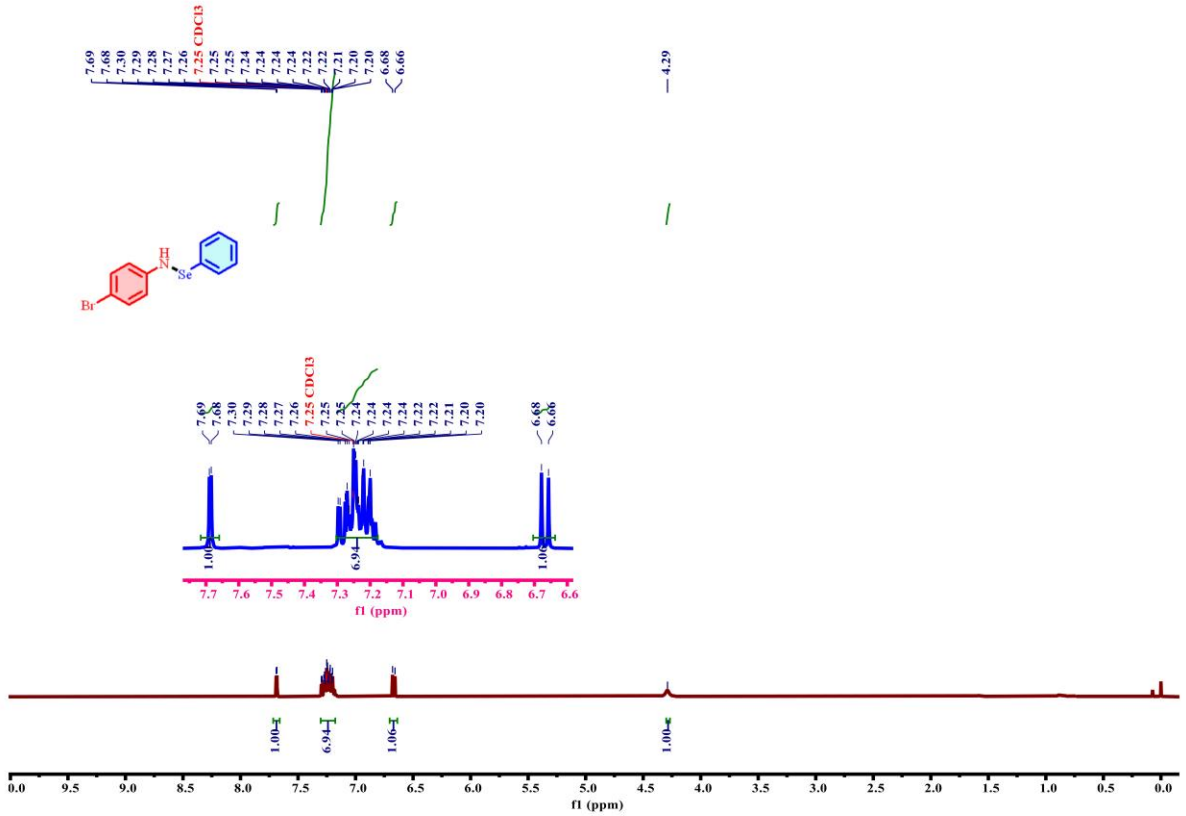


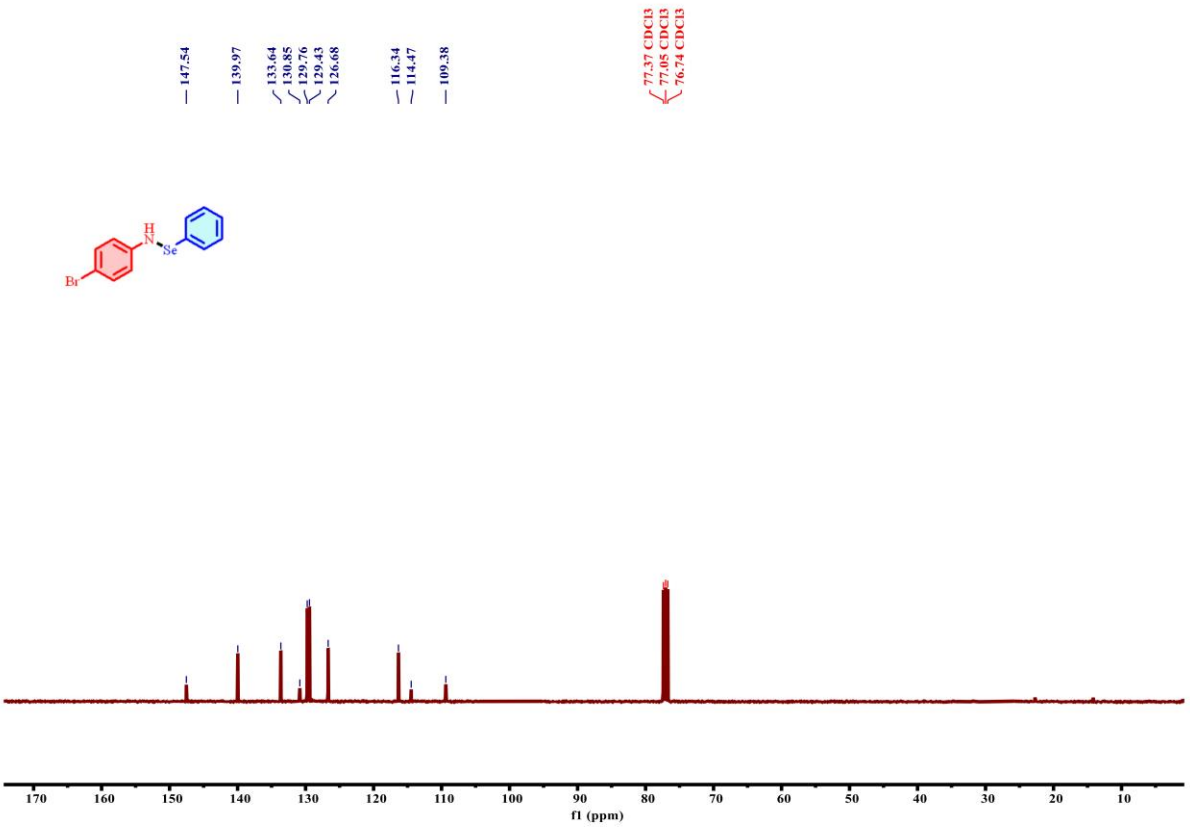
(Table 3, Entry 5b)



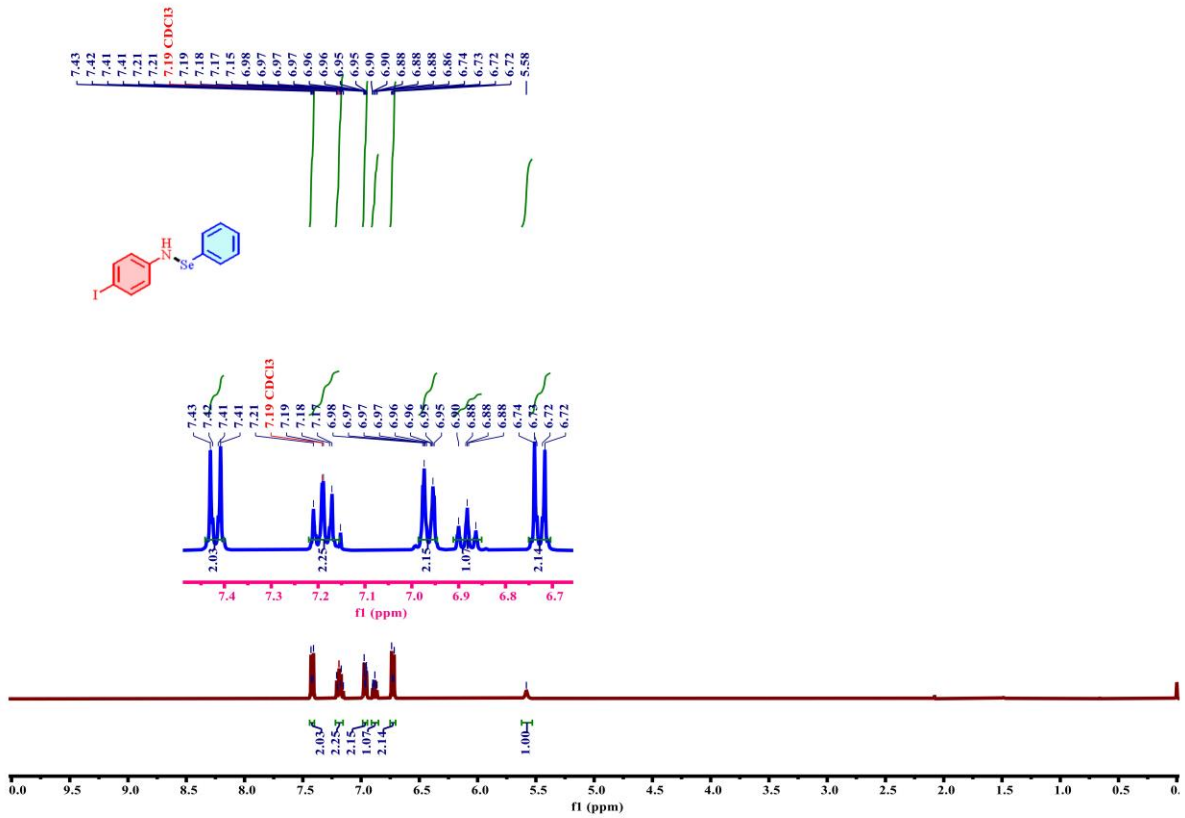


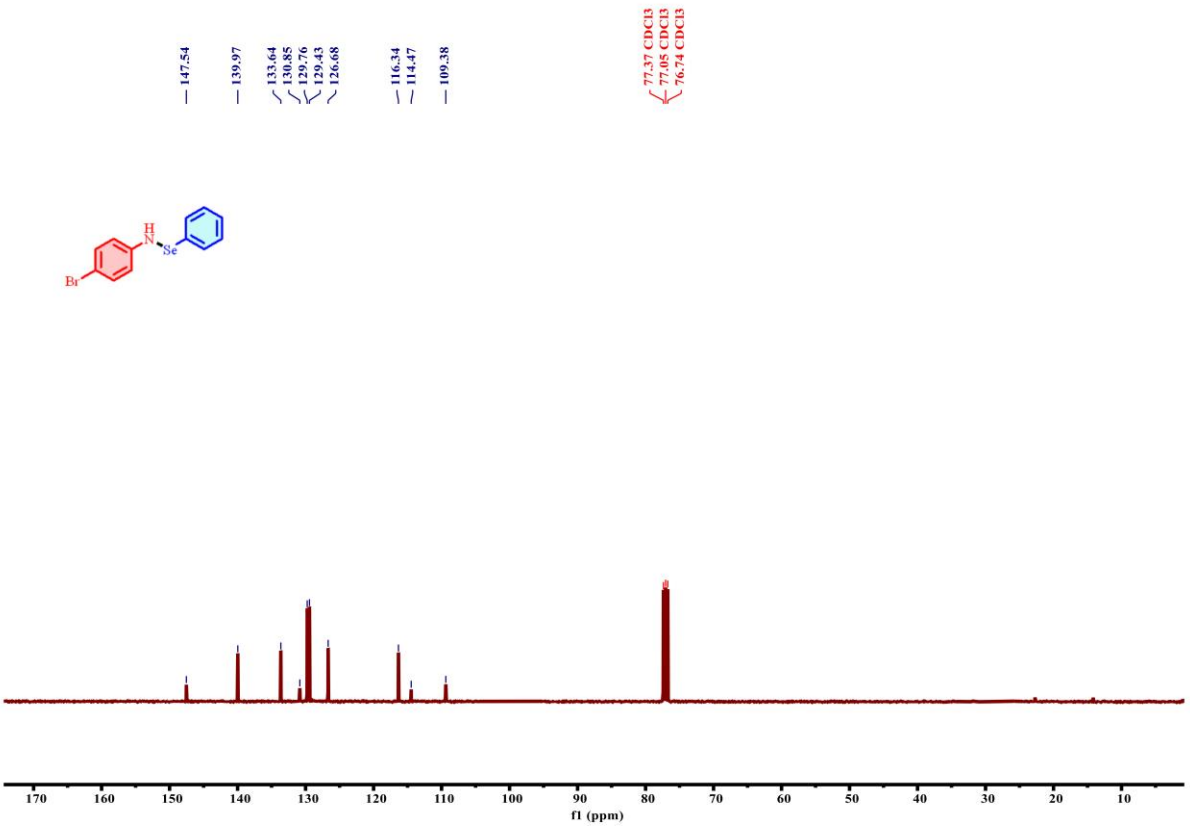
(Table 3, Entry 5c)





(Table 3, Entry 5d)





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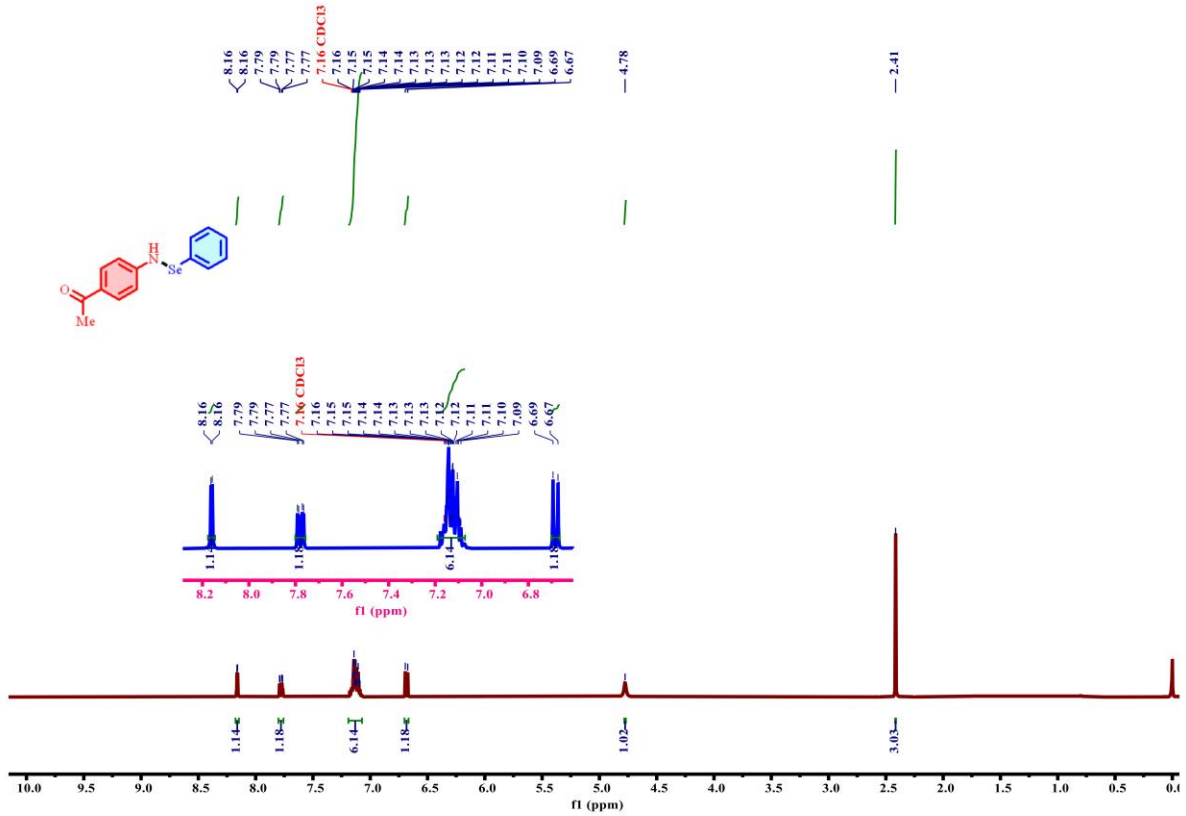
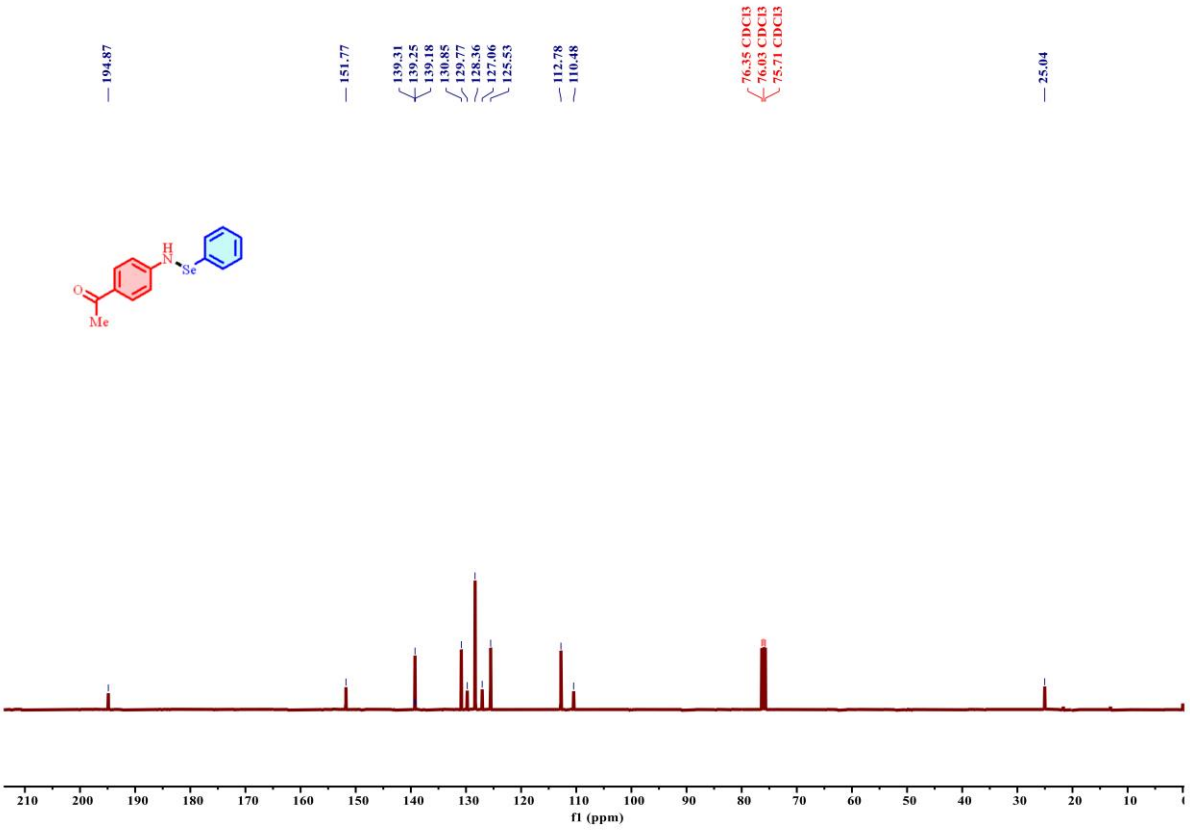
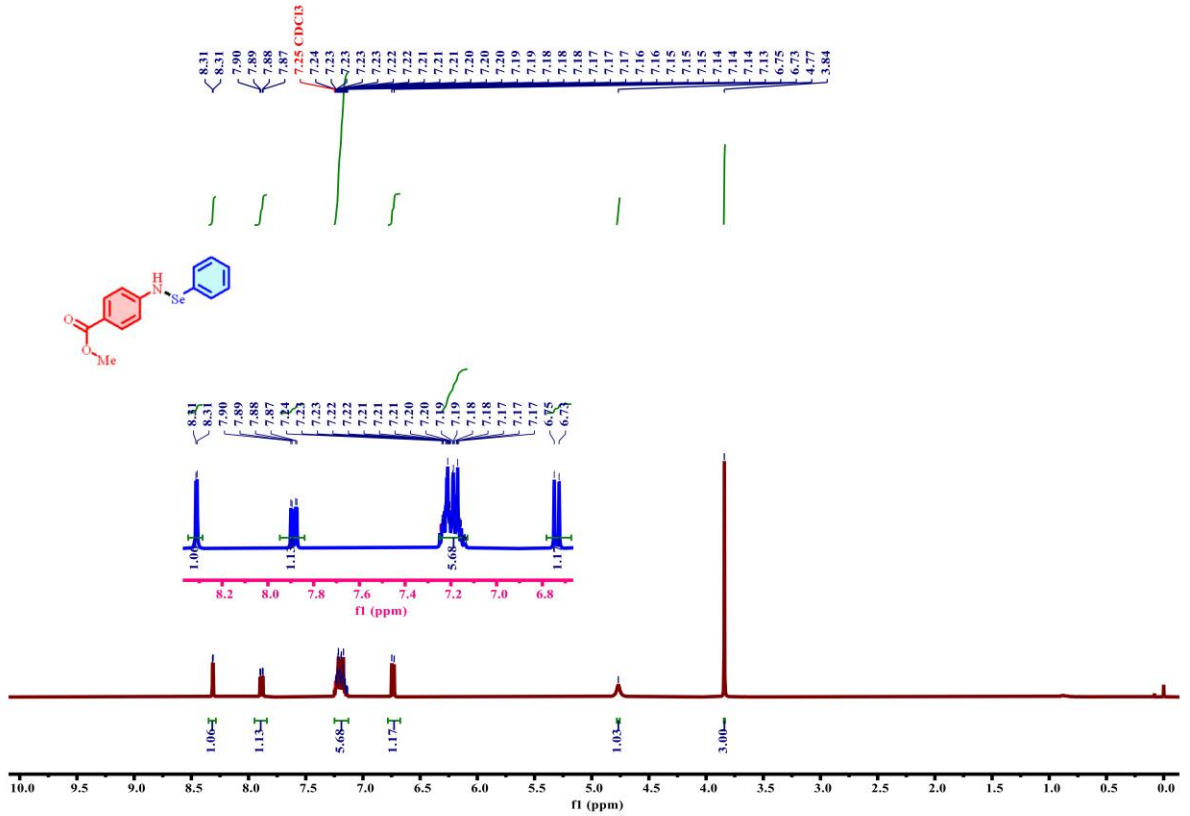
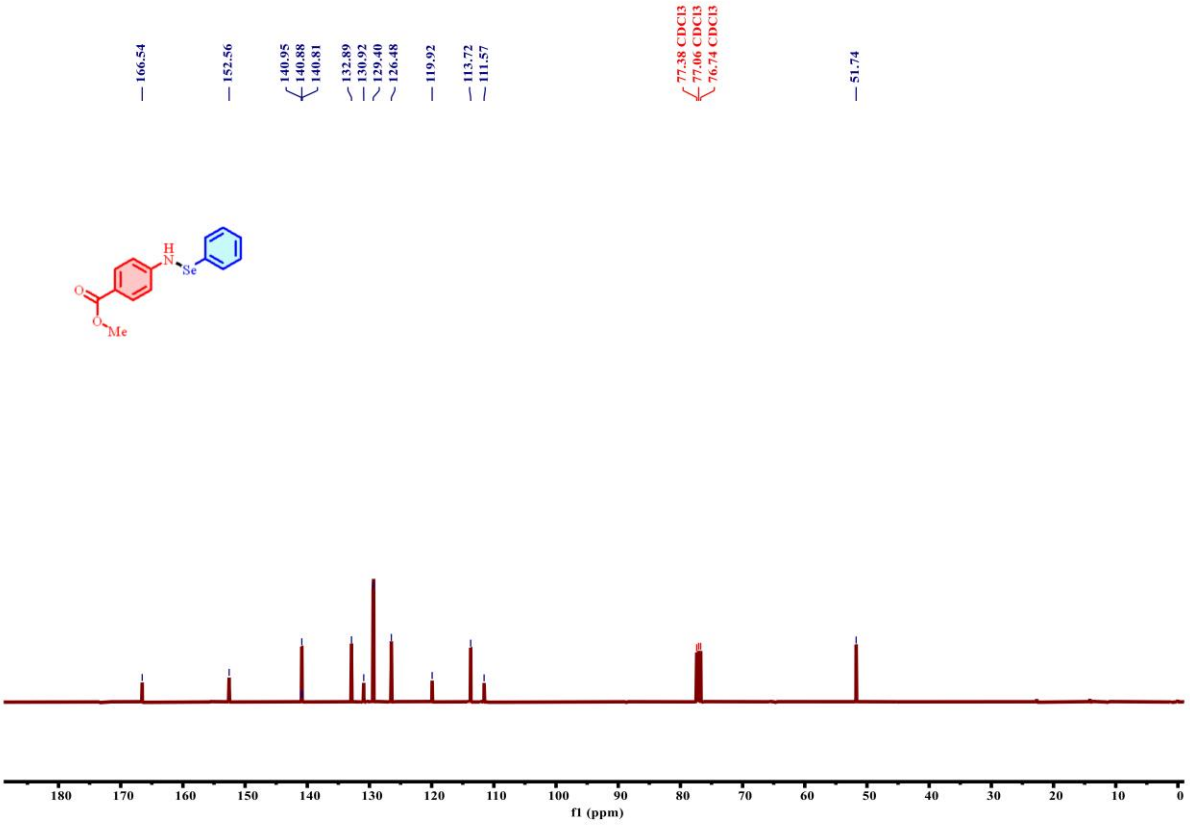


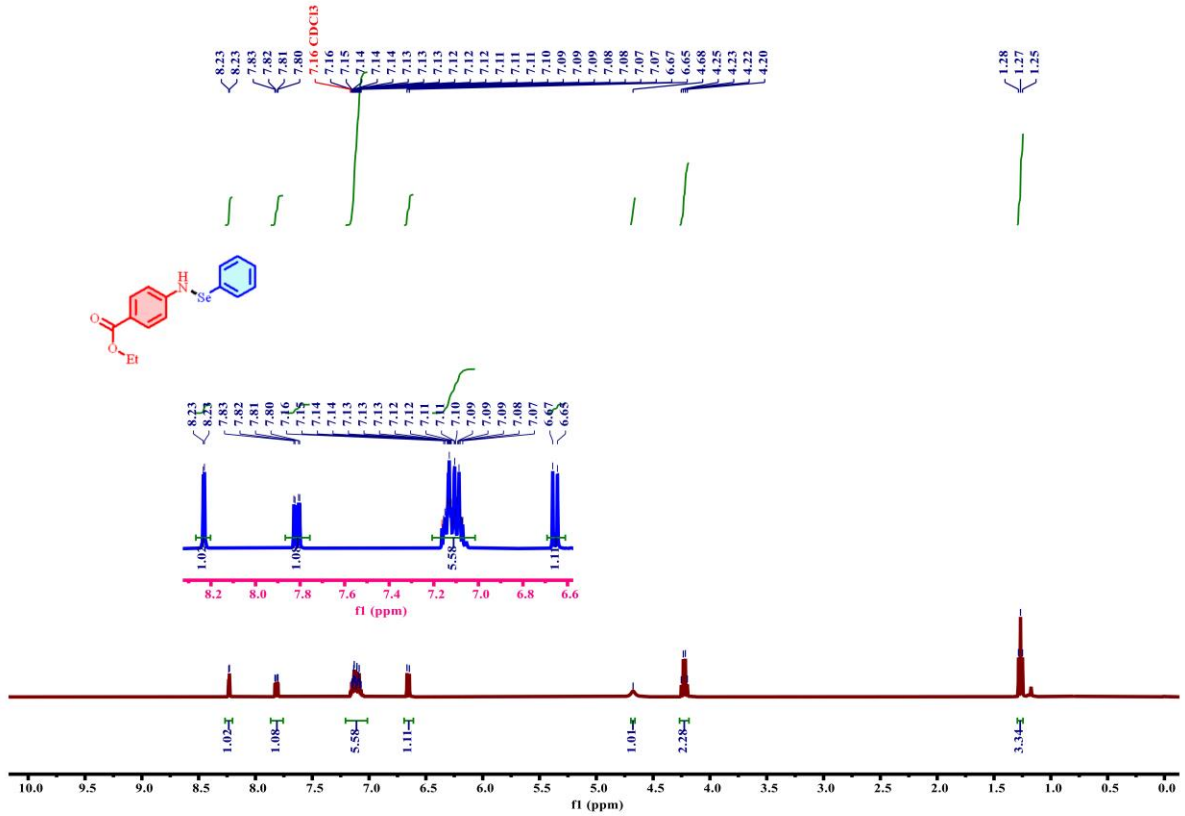


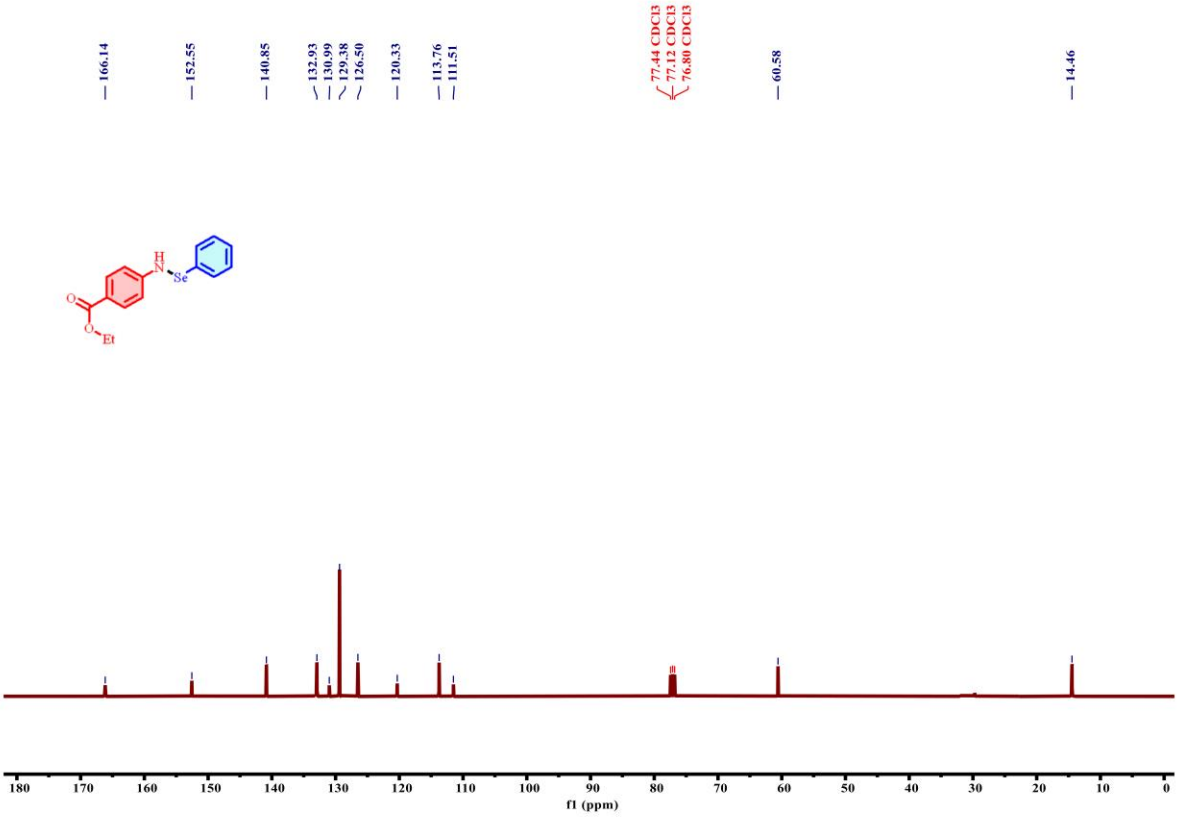
(Table 3, Entry 5f)



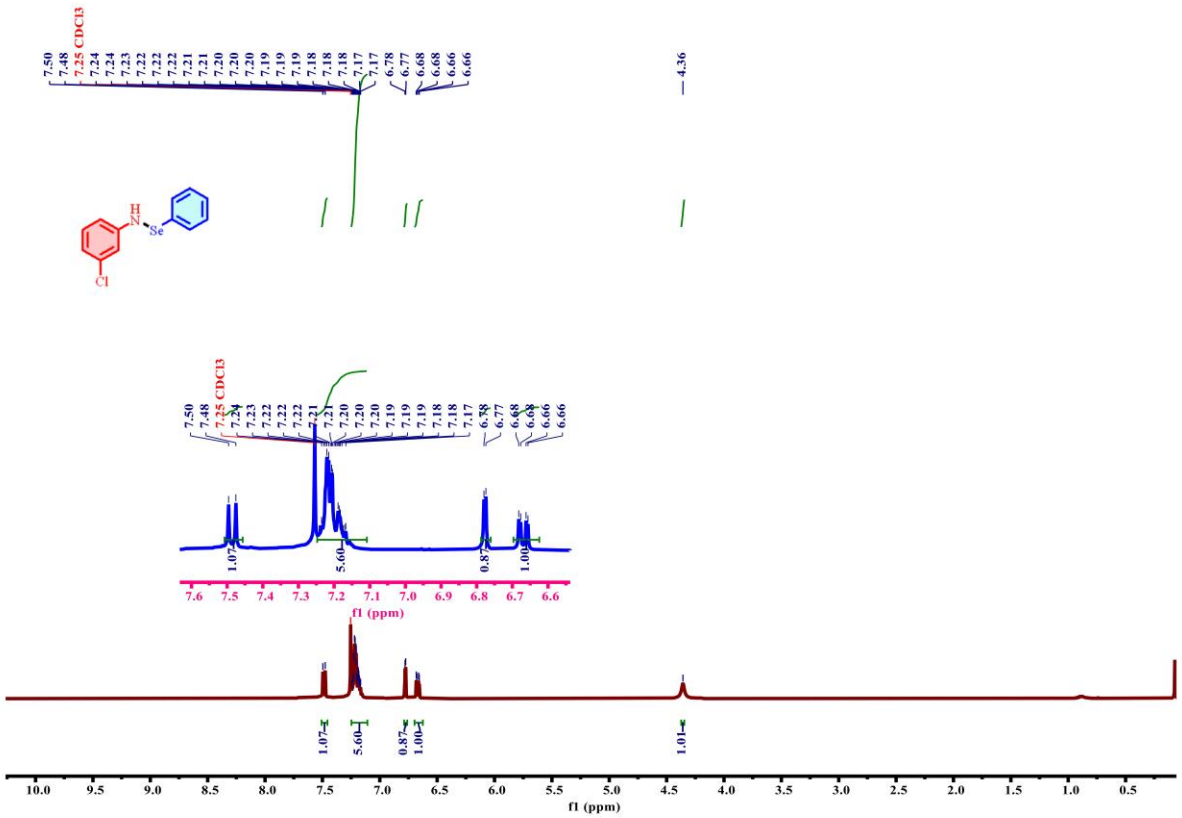


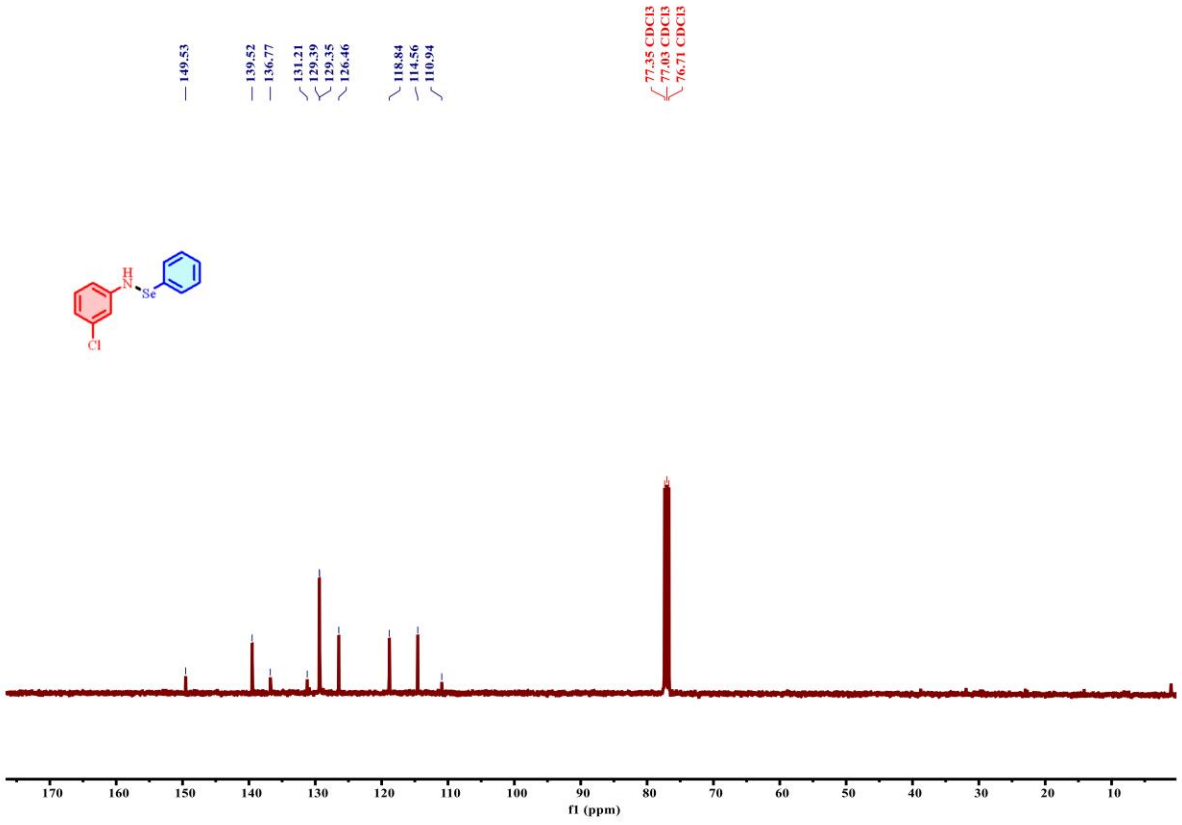
(Table 3, Entry 5g)



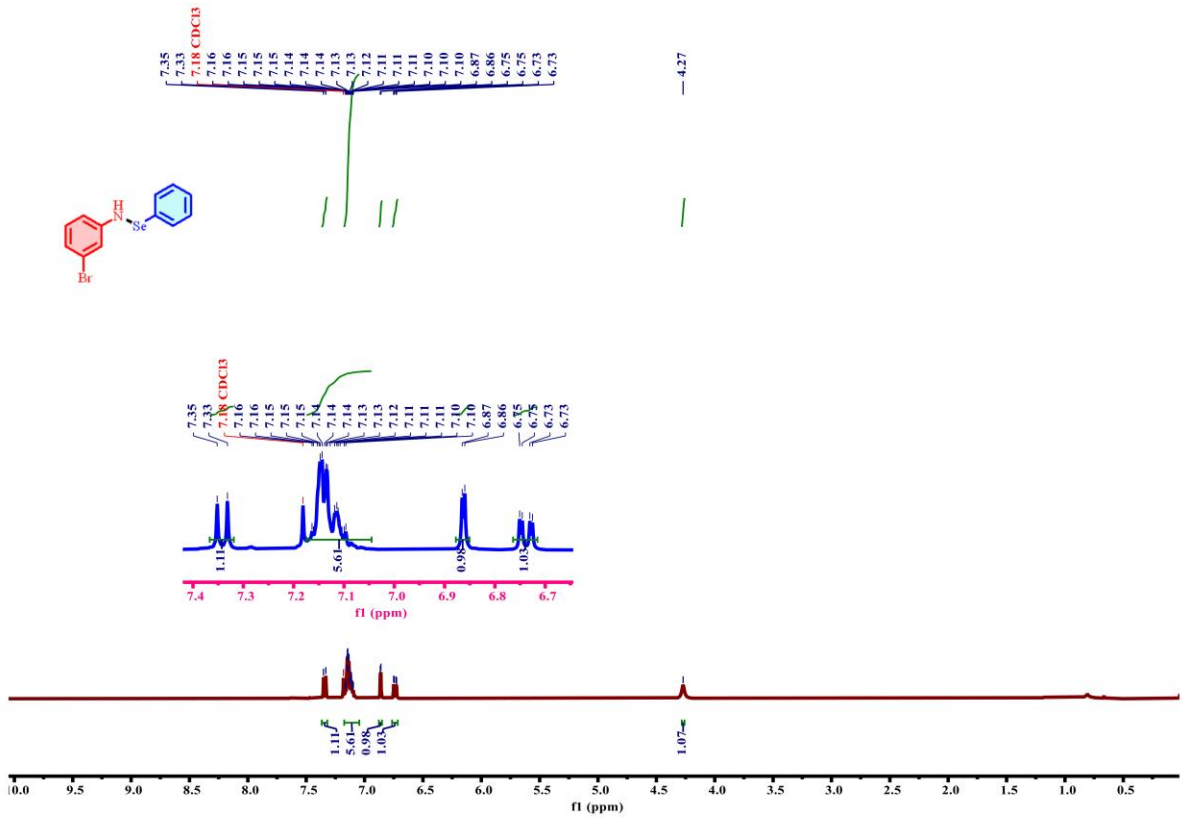


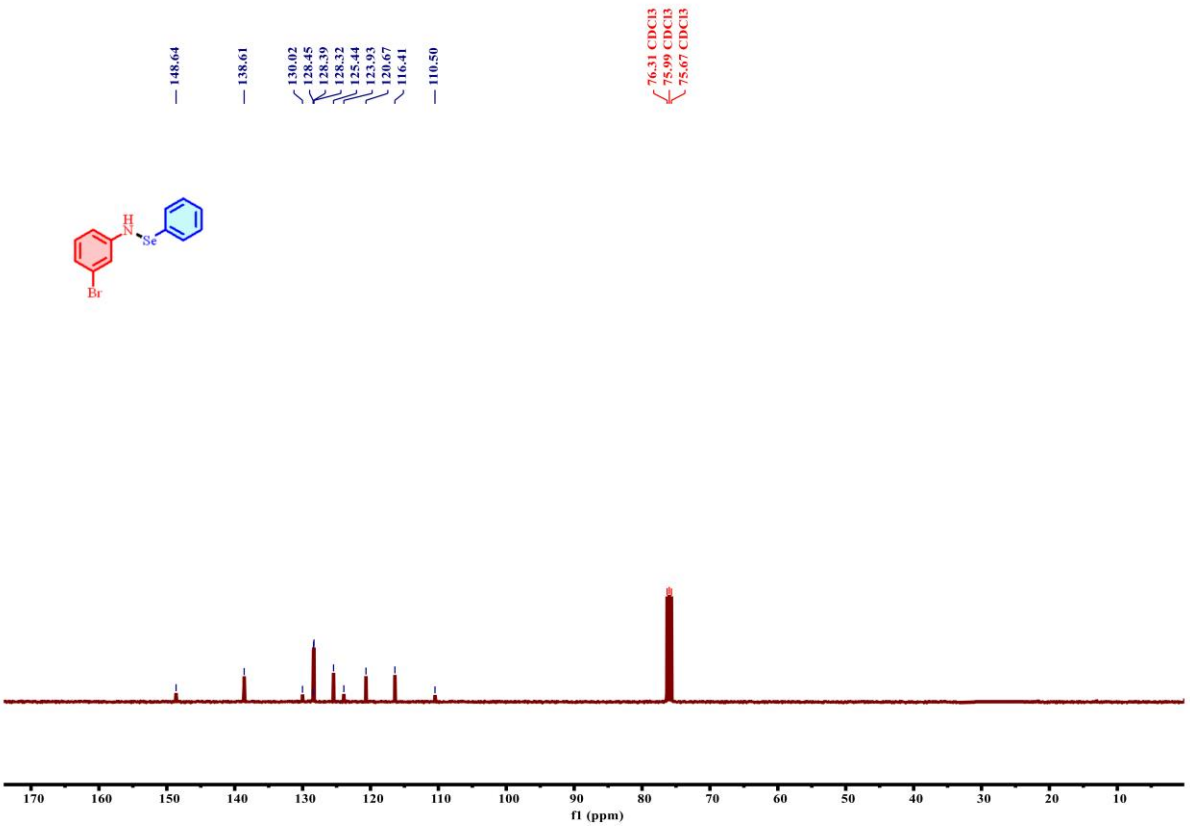
(Table 3, Entry 5h)



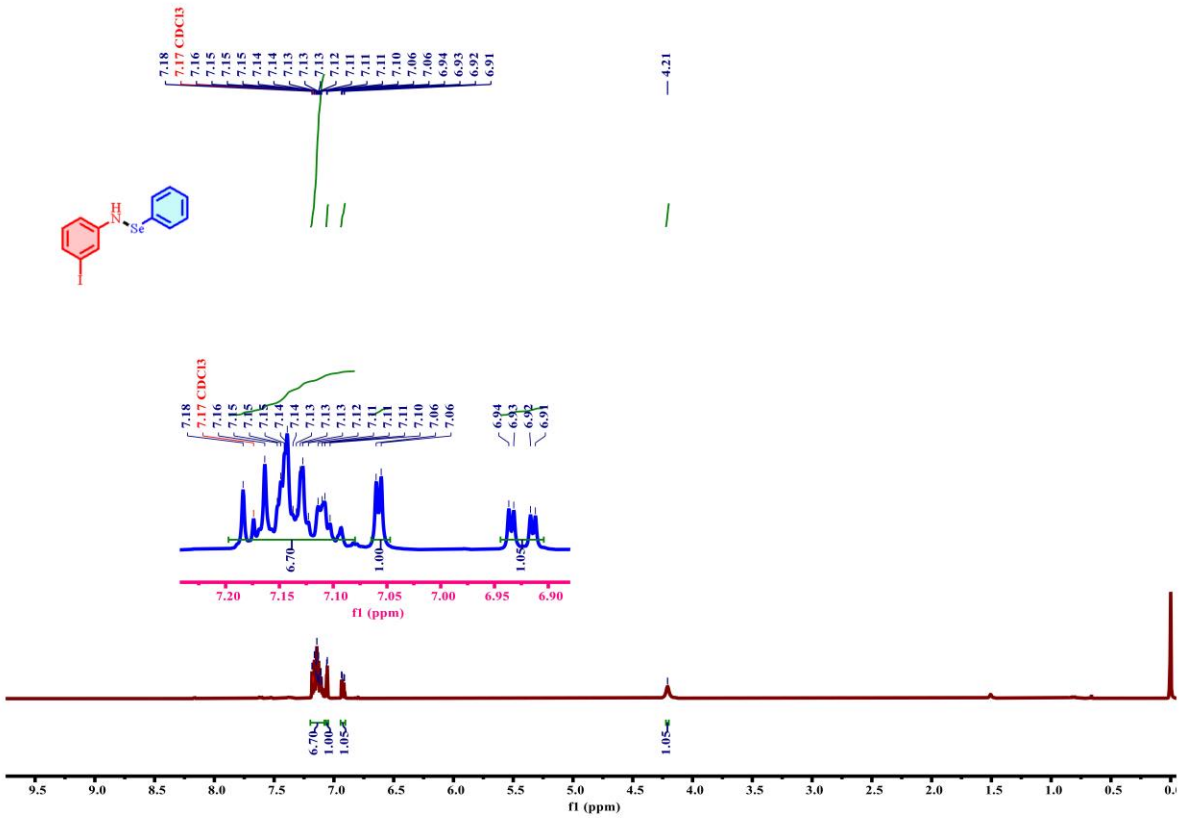


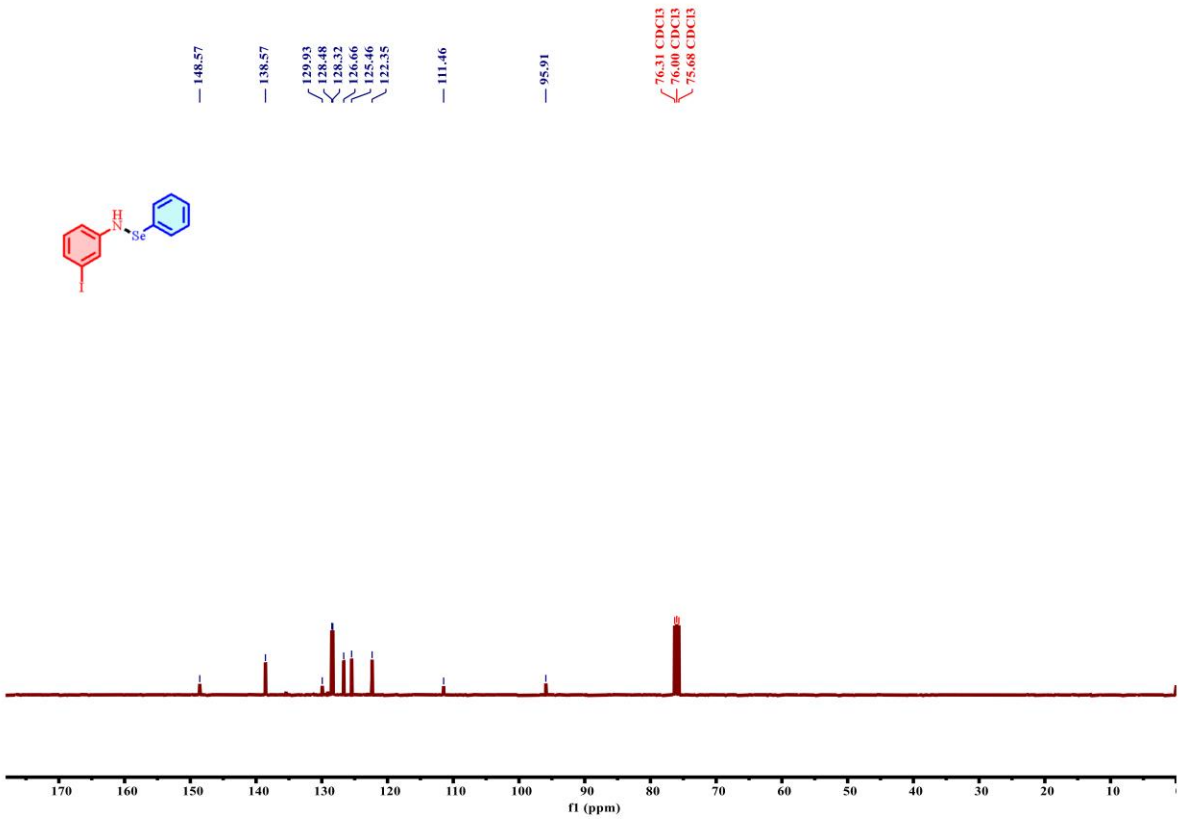
(Table 3, Entry 5i)



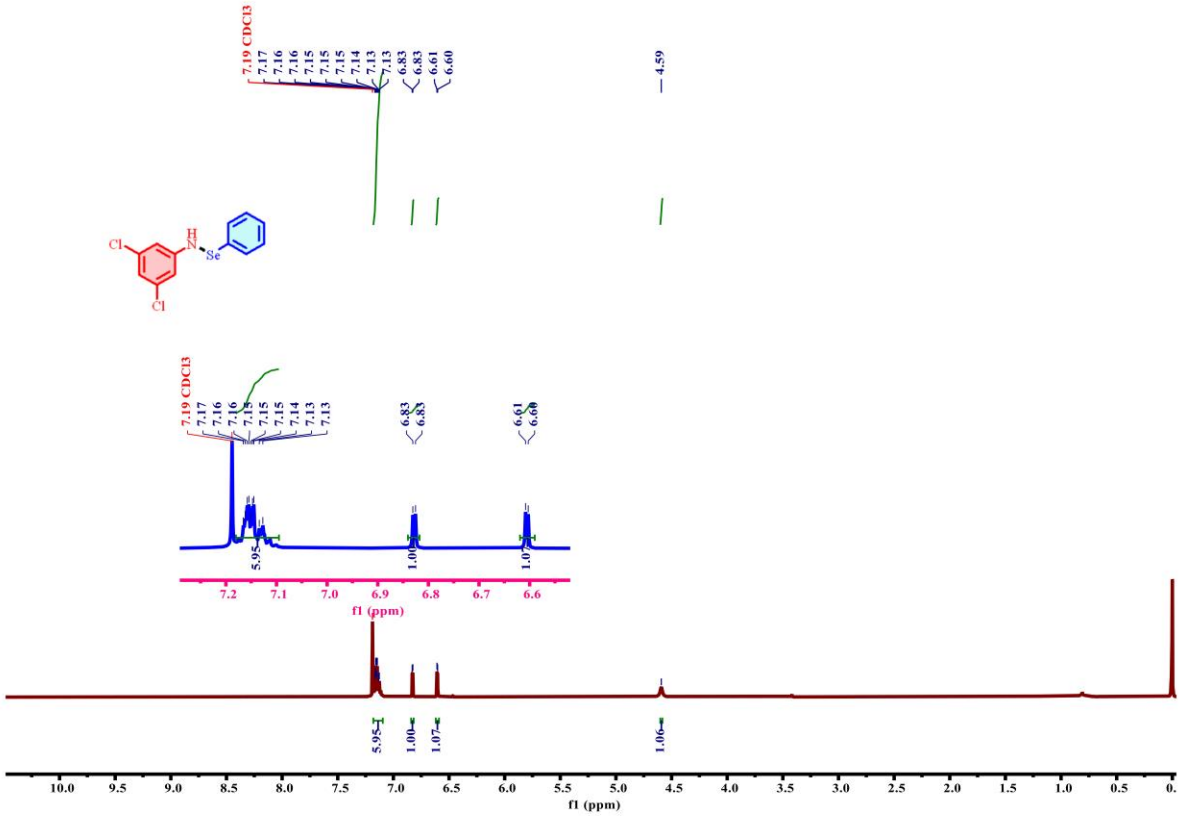


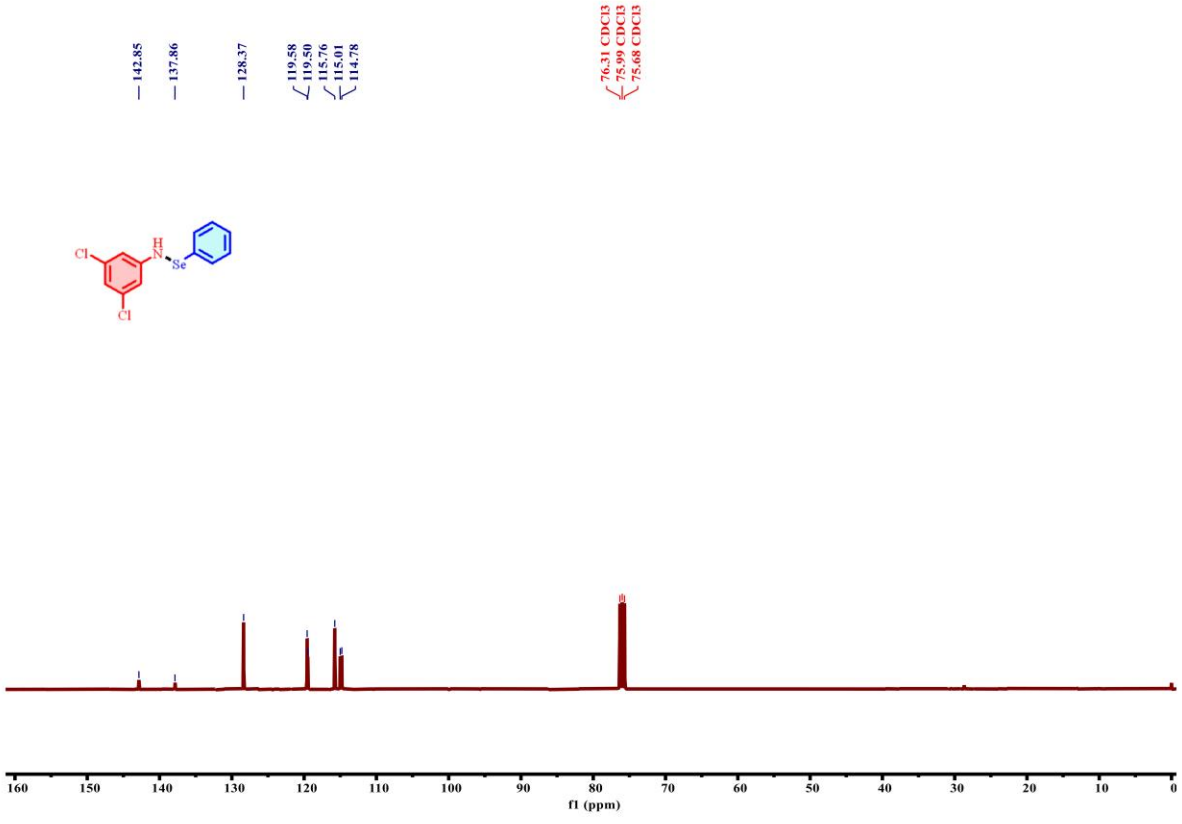
(Table 3, Entry 5j)



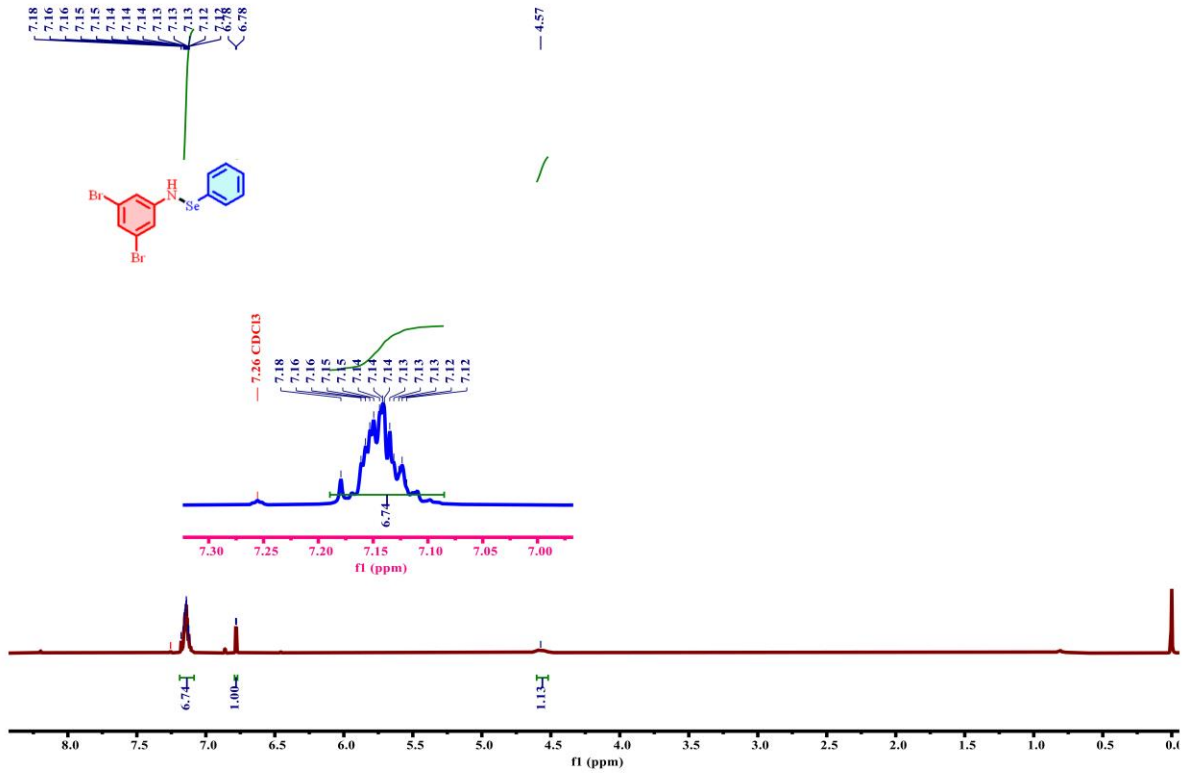


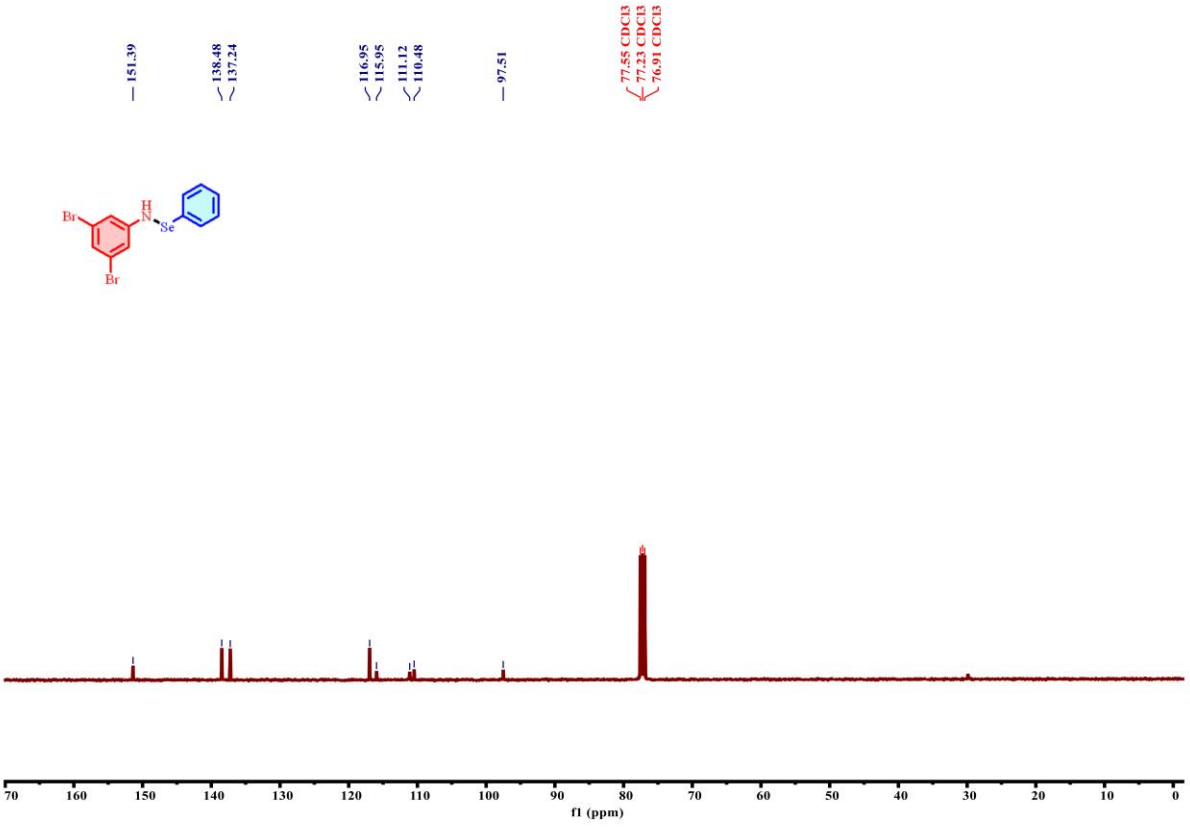
(Table 3, Entry 5k)



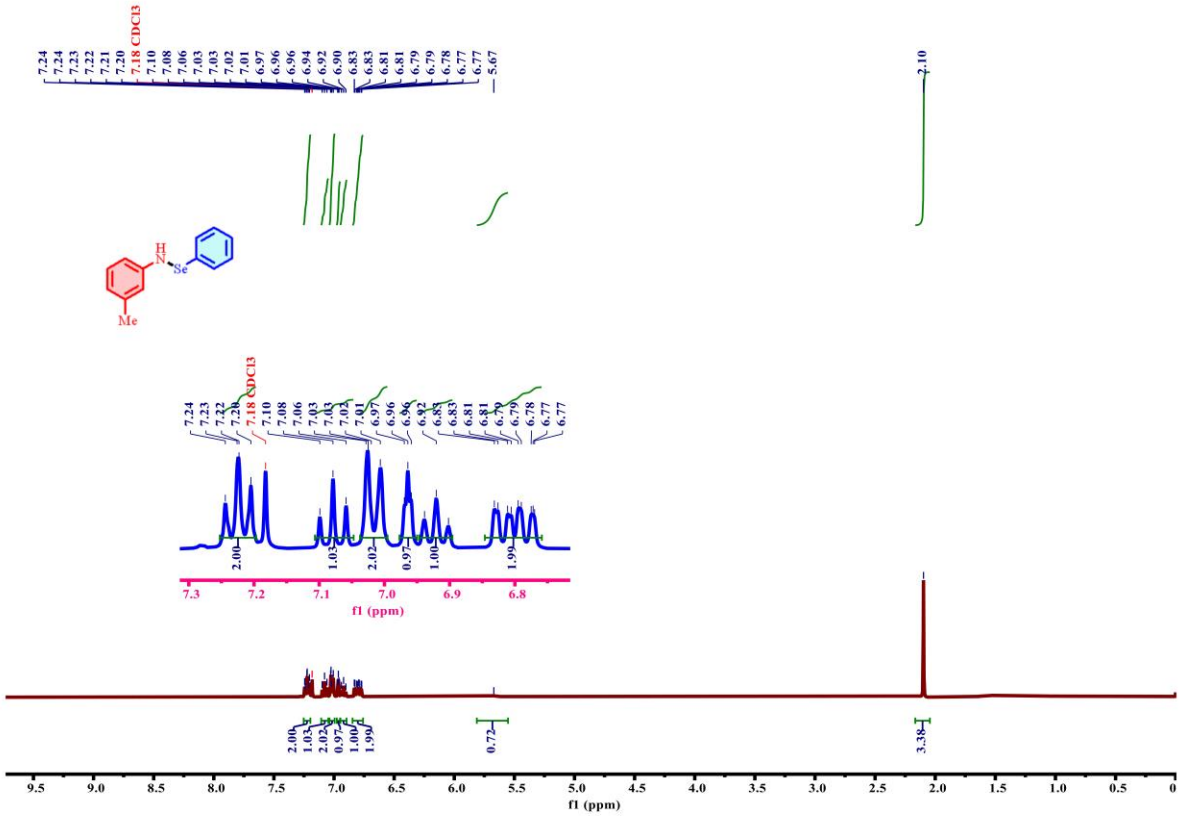


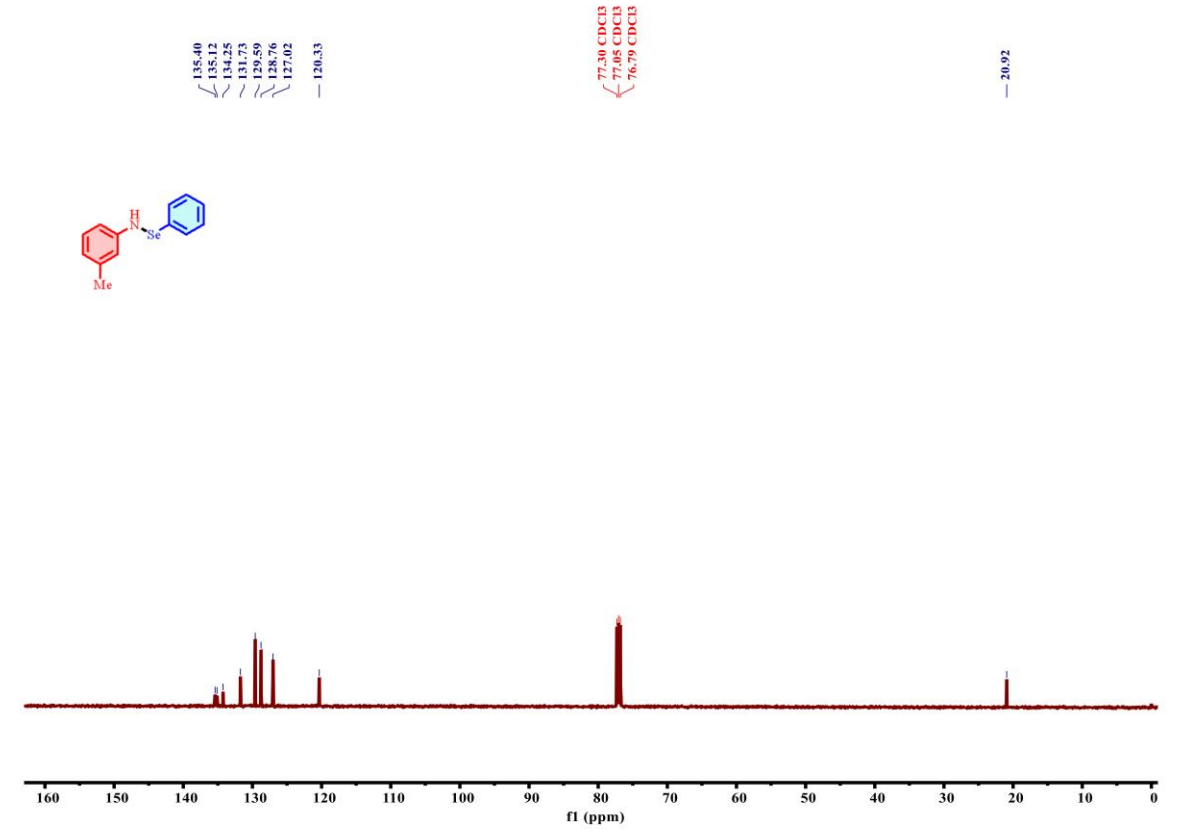
(Table 3, Entry 5l)



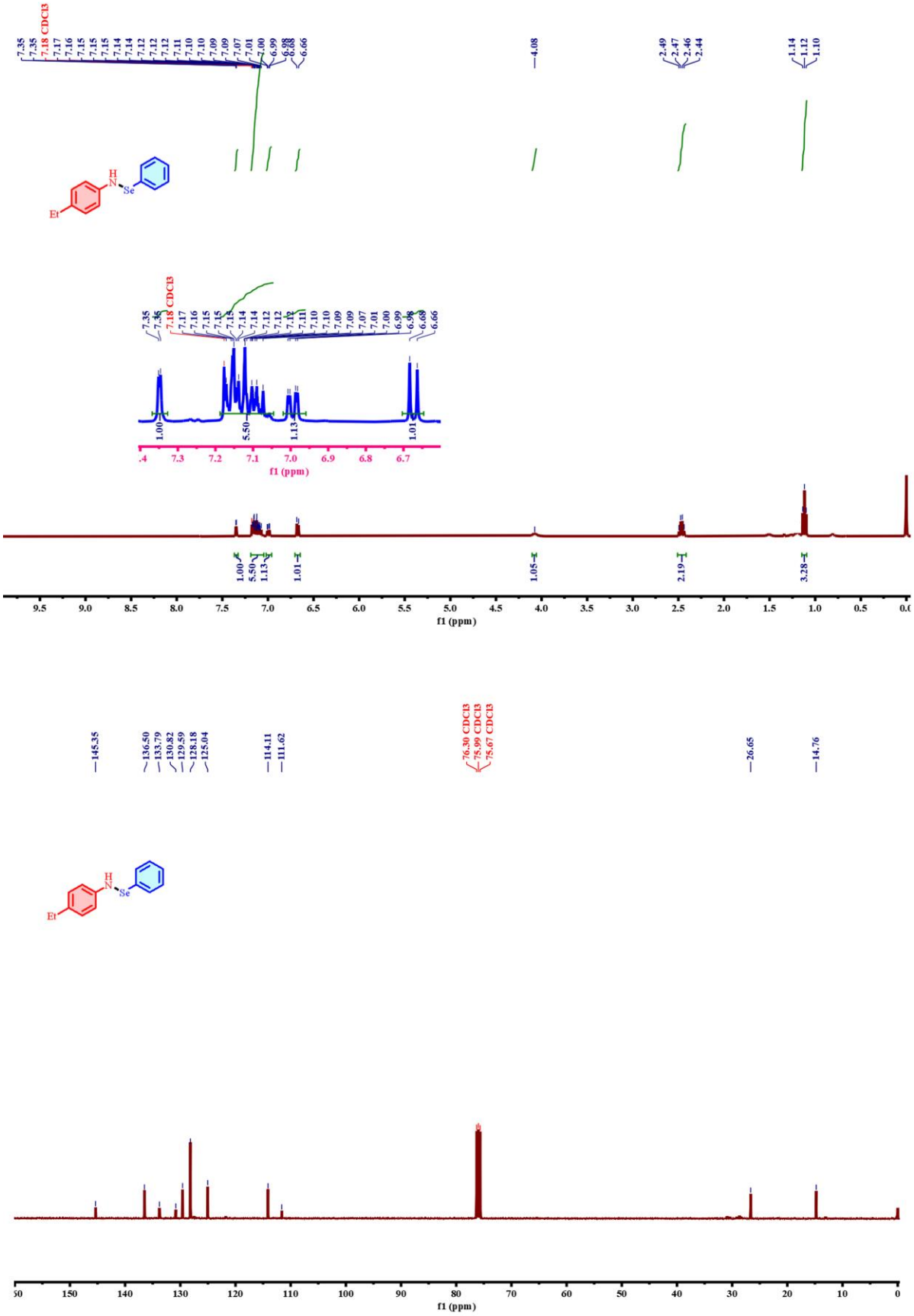


(Table 3, Entry 5m)

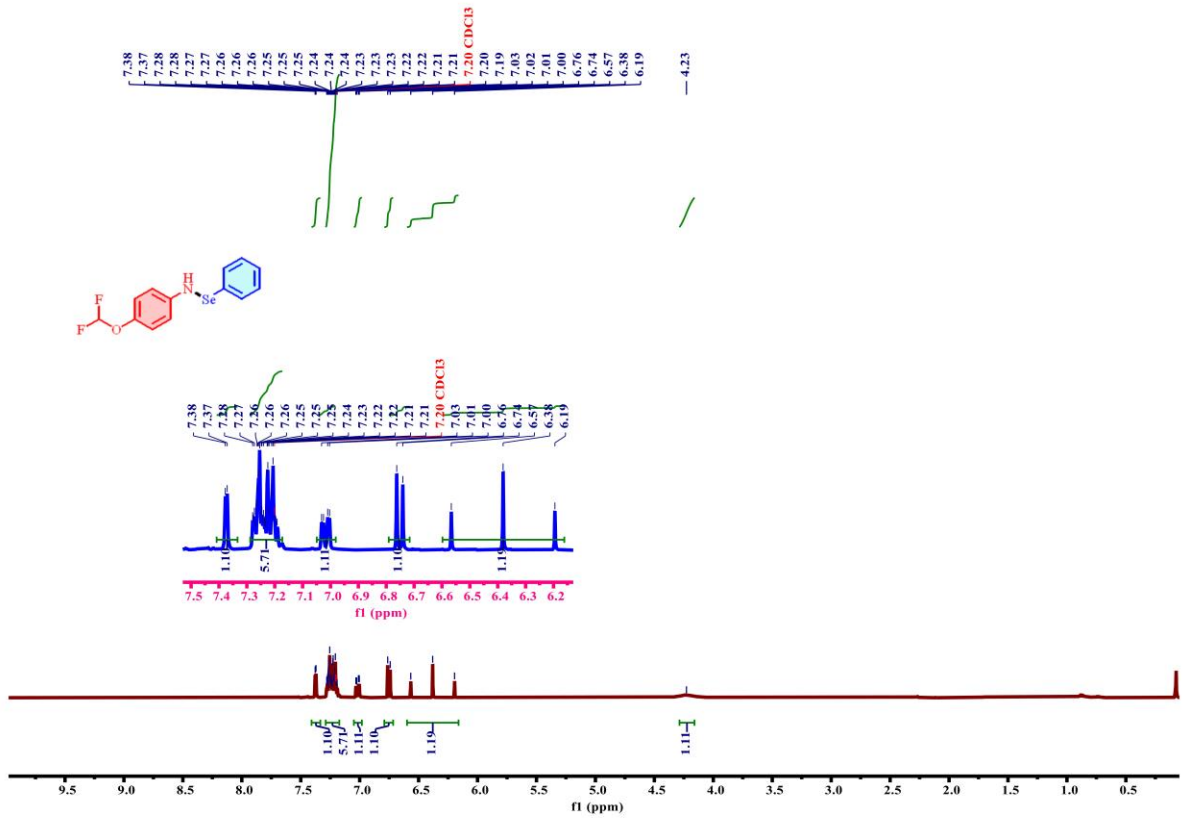


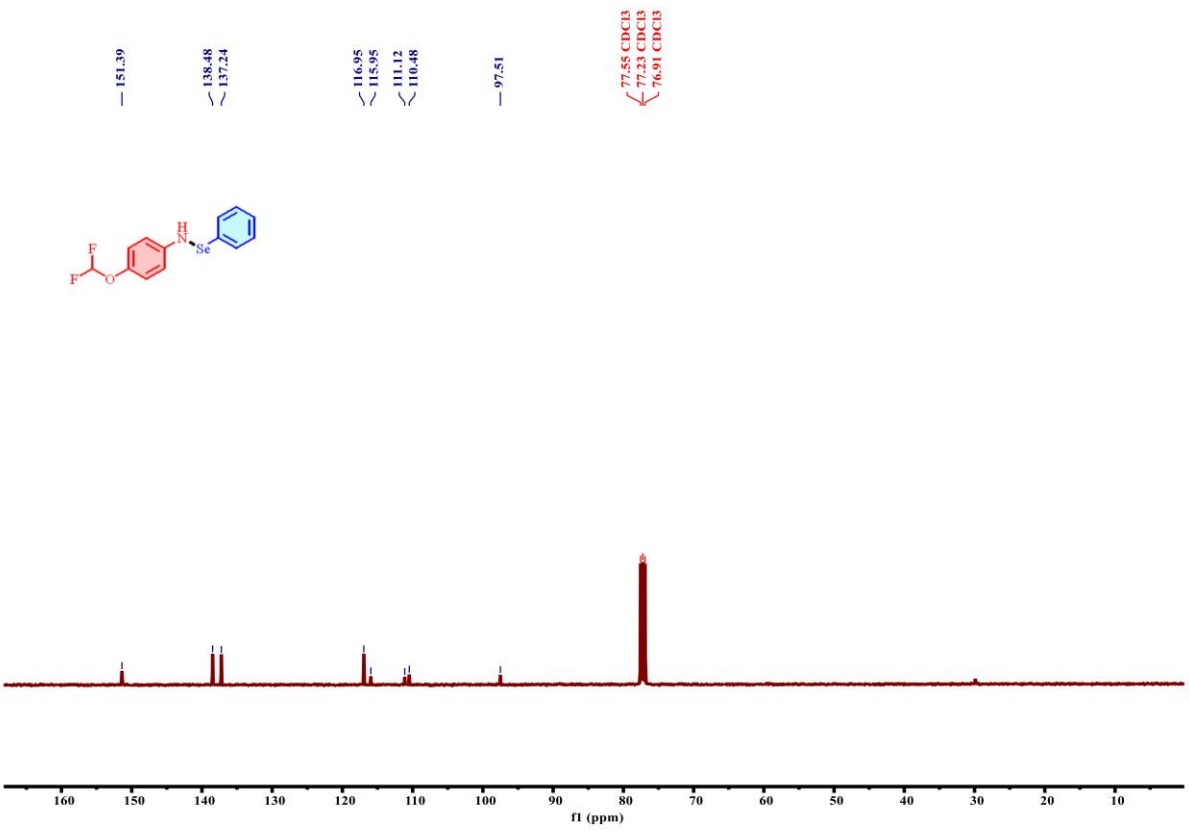


(Table 3, Entry 5n)

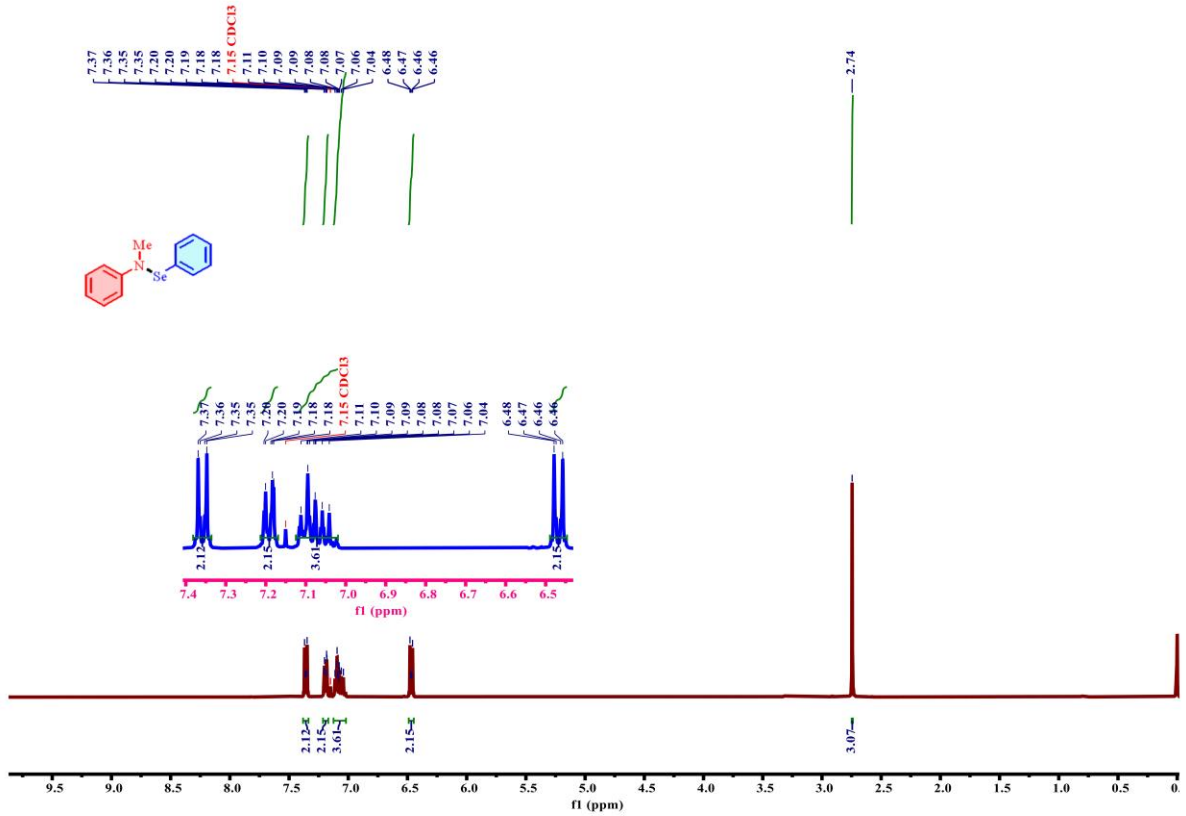


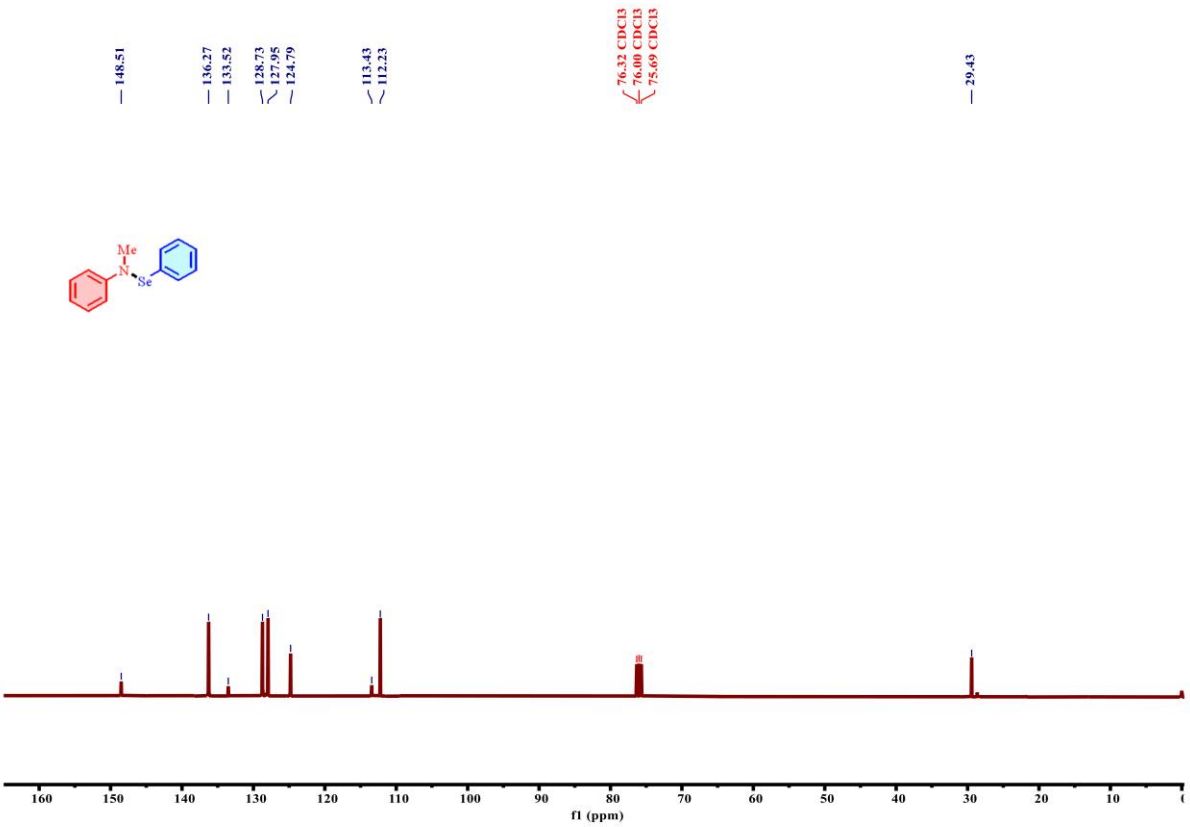
(Table 3, Entry 5o)



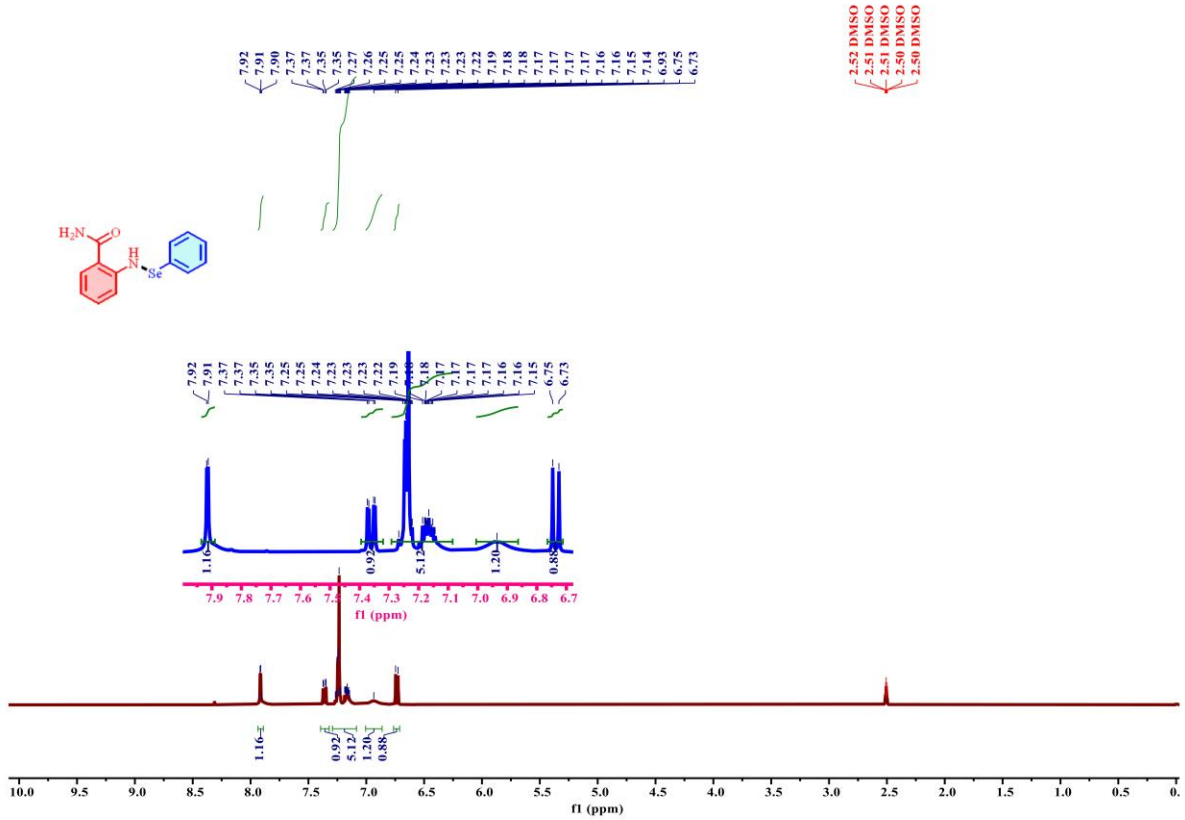


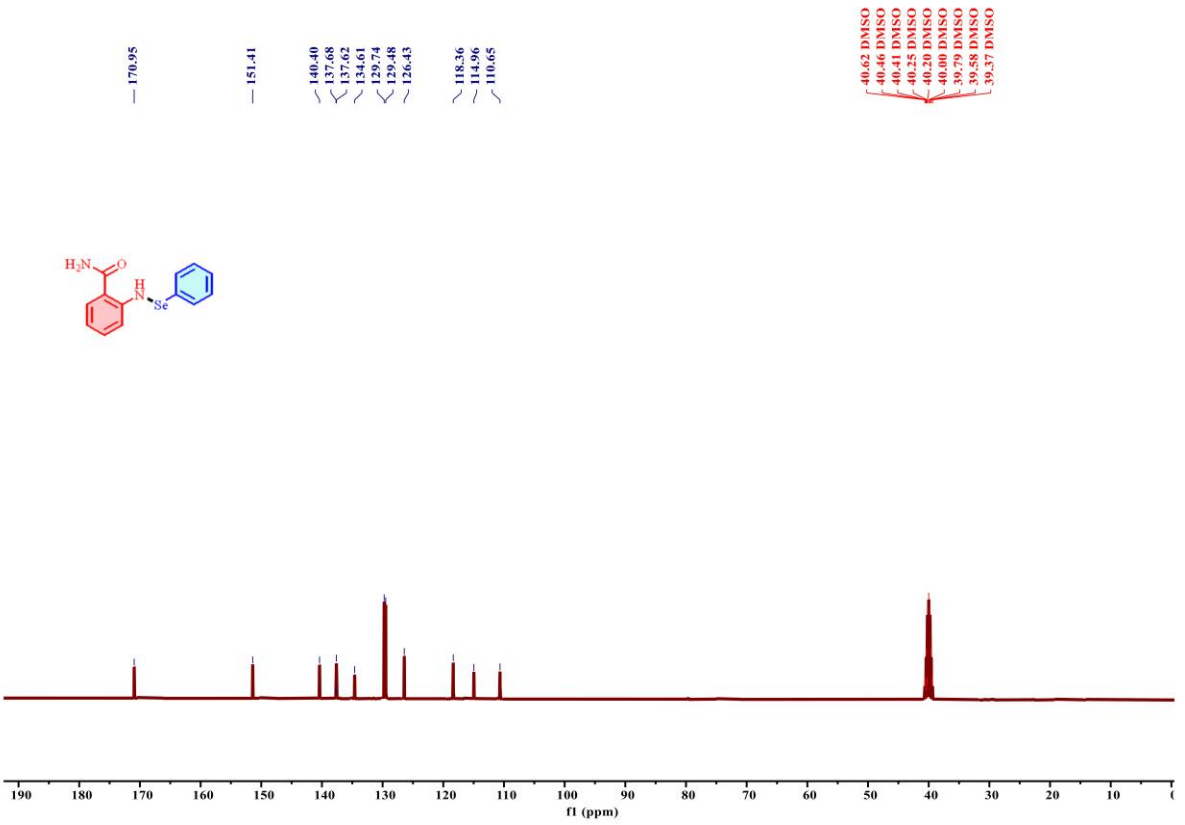
(Table 3, Entry 5p)



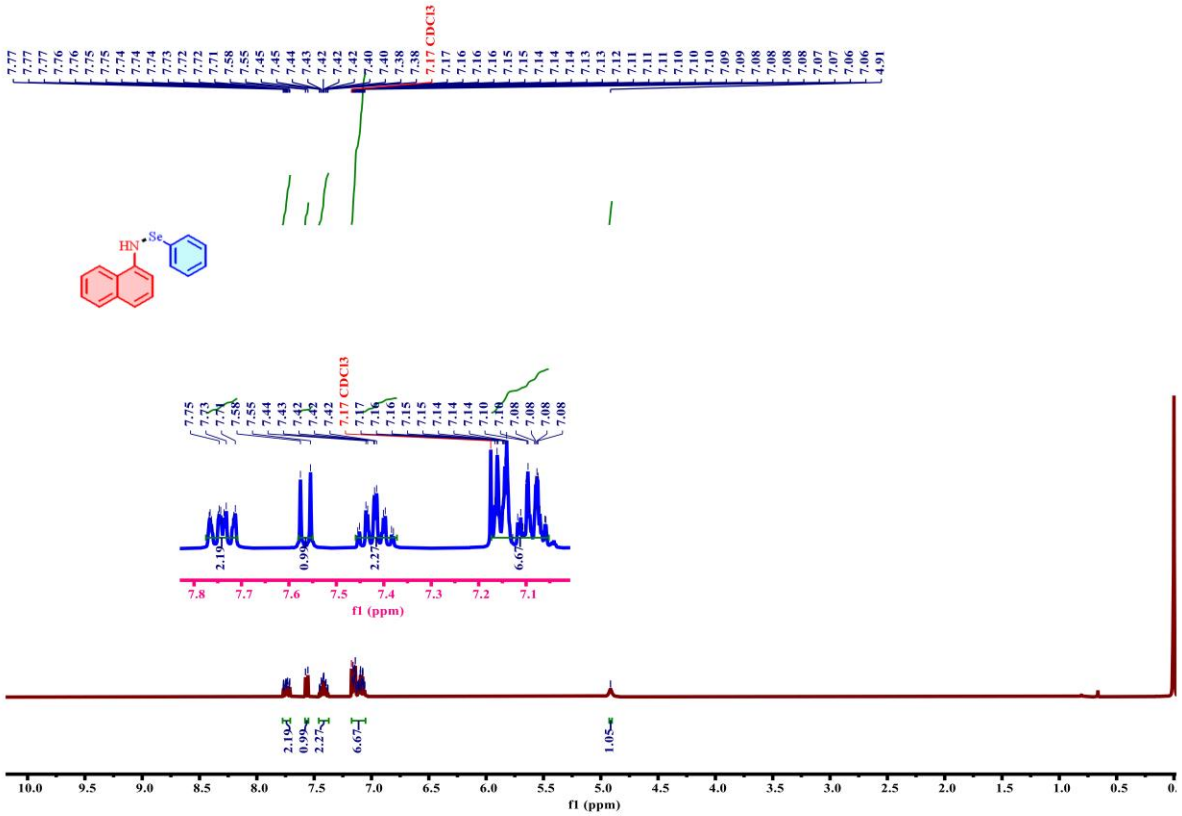


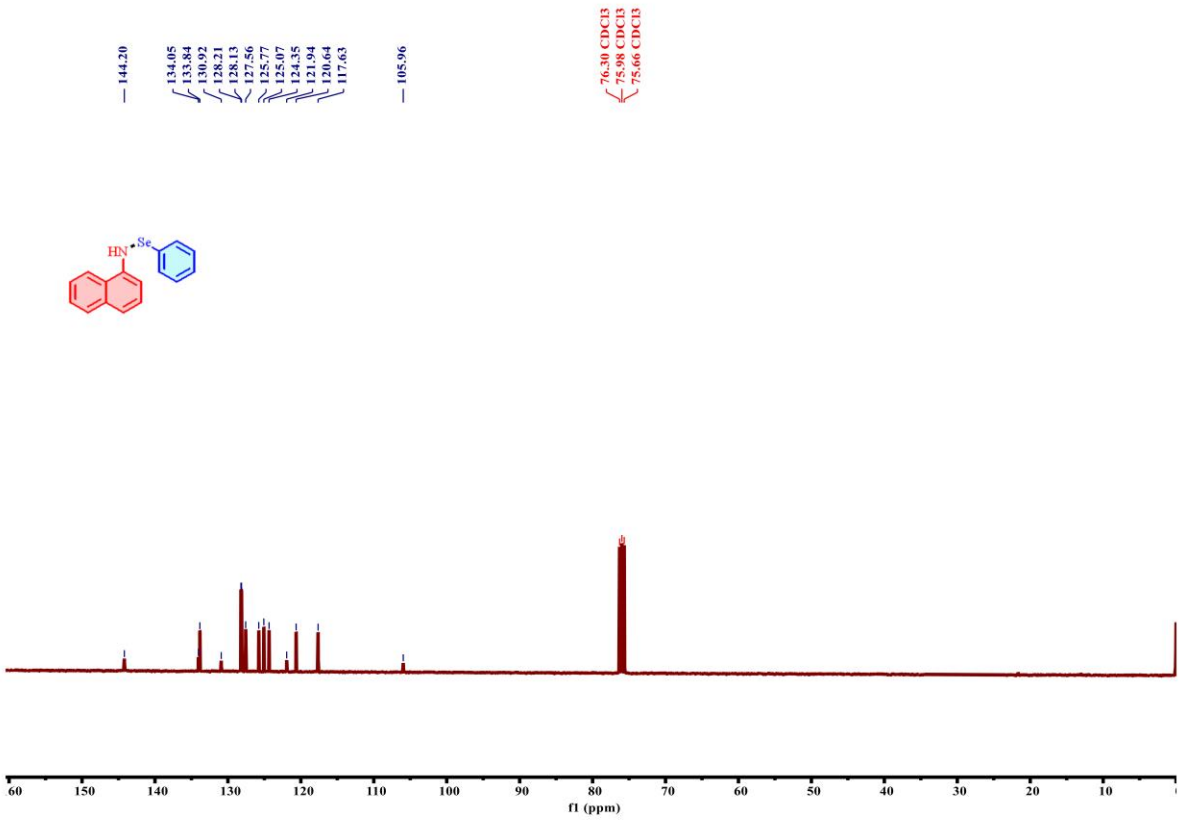
(Table 3, Entry 5q)



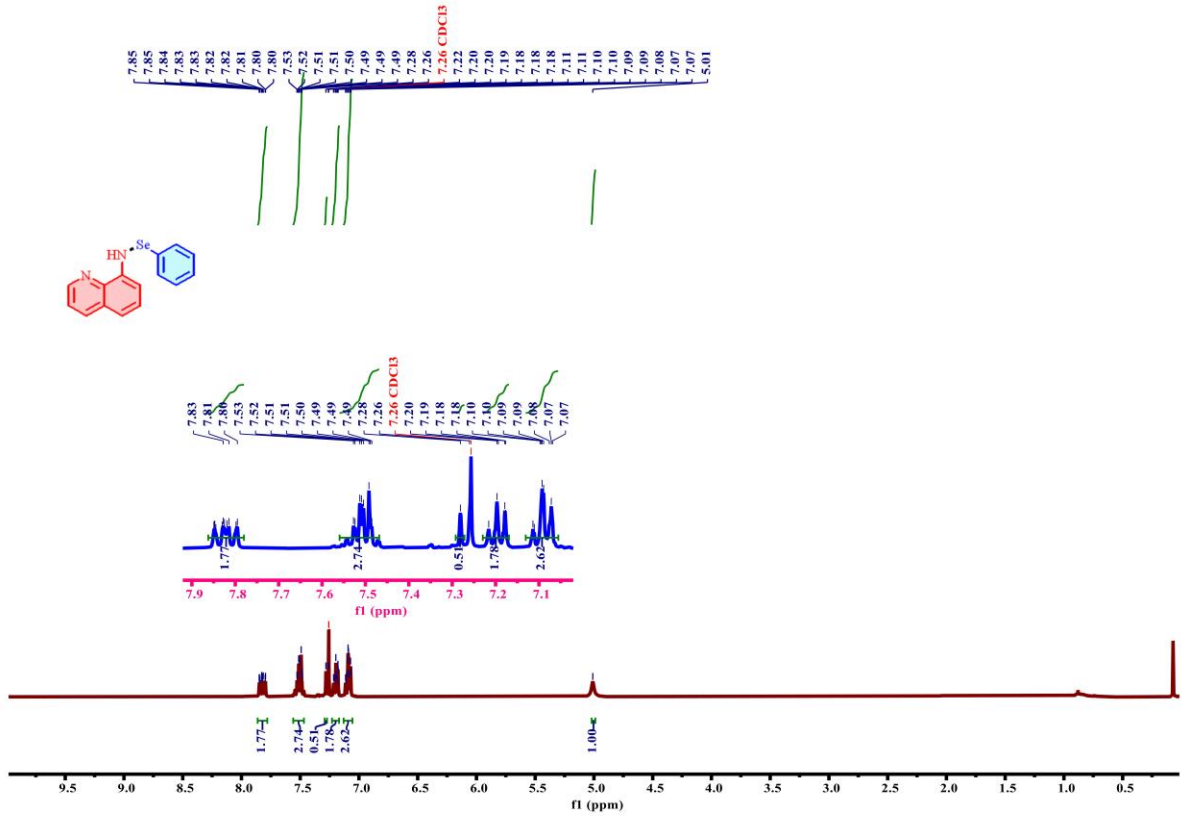


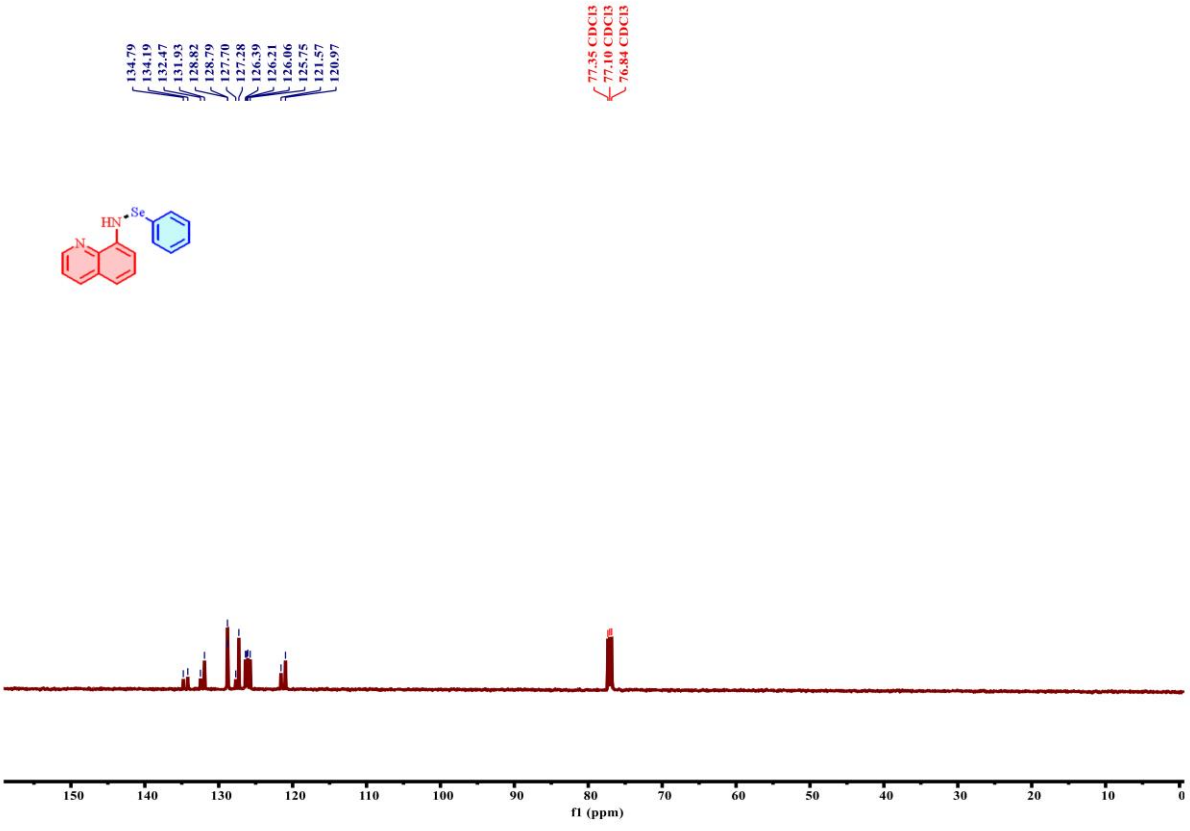
(Table 3, Entry 5r)



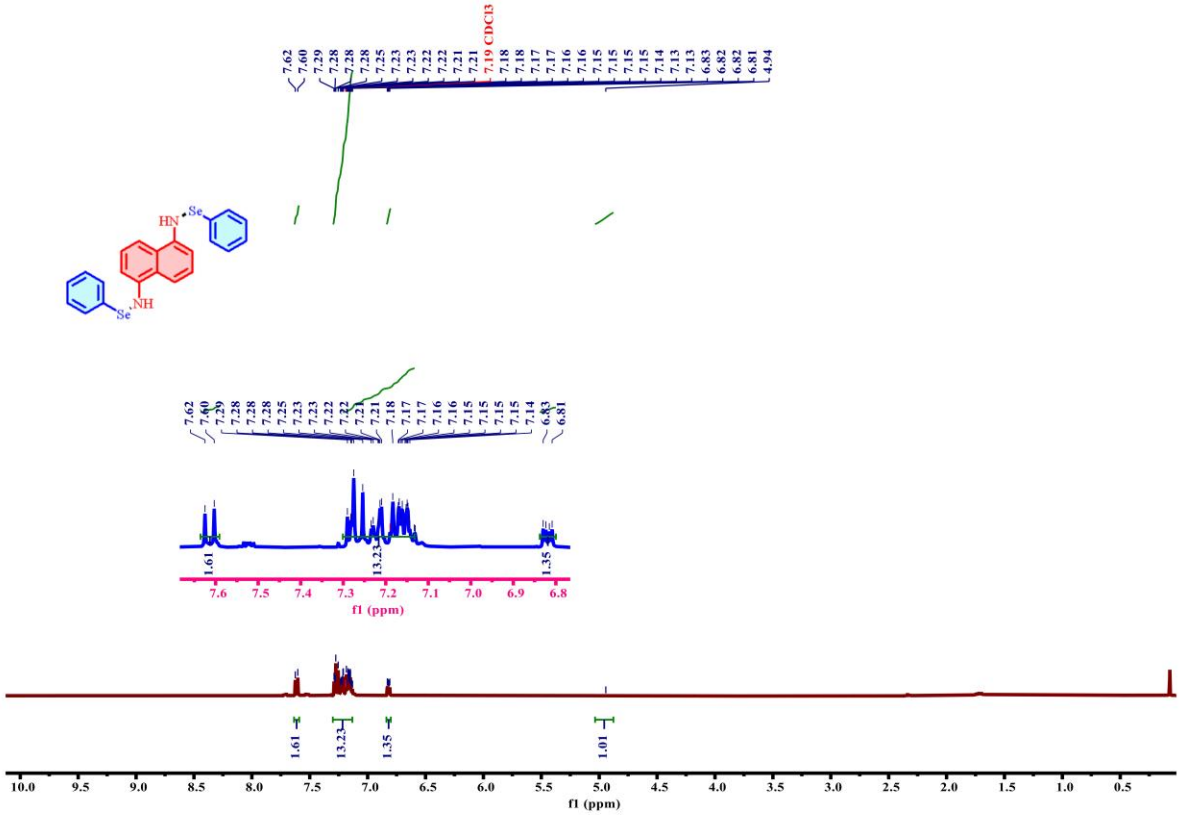


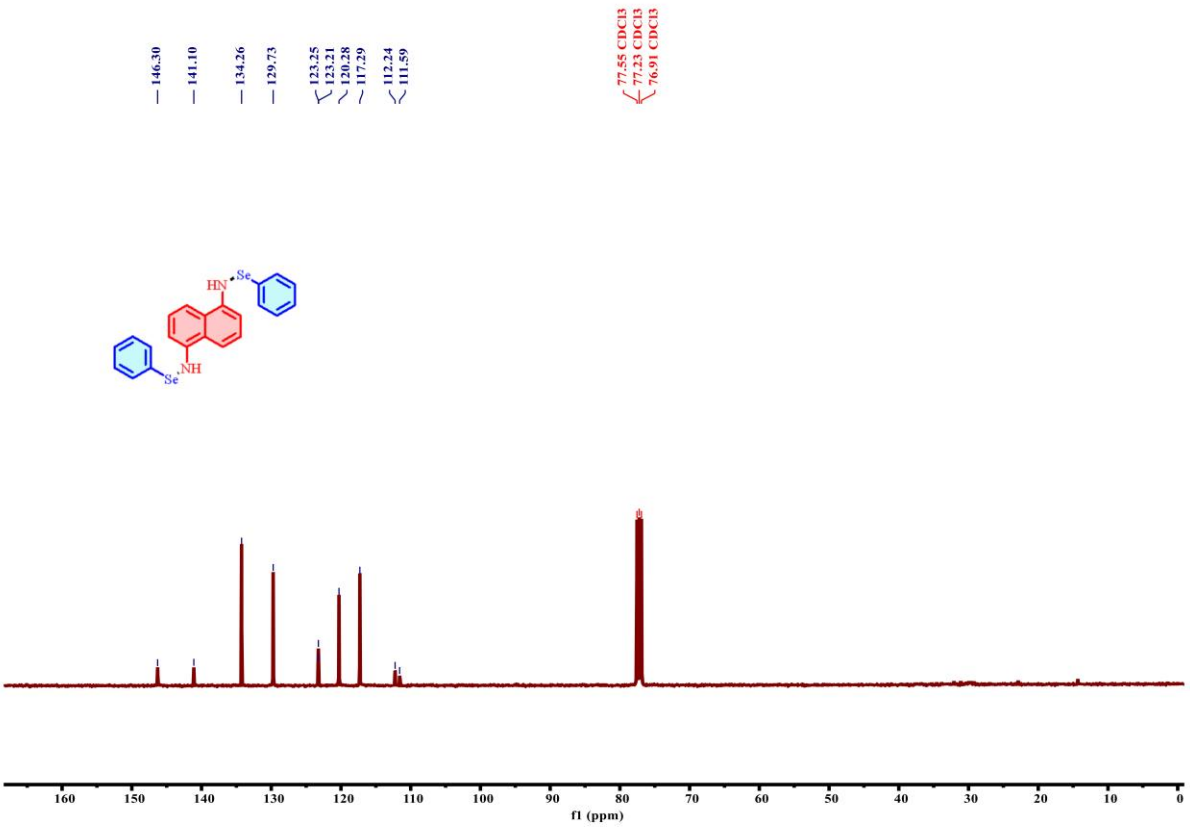
(Table 3, Entry 5s)





(Table 3, Entry 5t)





(Table 3, Entry 5v)