New journal of Chemistry

Ligand-free access to Benzisothiazolones and Benzisoselenazolones through $NiFe_2O_4$ catalyzed concomitant annulation of 2-halobenzanilides with chalcogens and their late-stage transformations

Samiran Dhara, Moumita Saha, Asish R. Das*

Department of Chemistry, University of Calcutta, Kolkata-700009, India

*Corresponding author: Tel.: +913323501014, +919433120265; fax: +913323519754;

E-mail: ardchem@caluniv.ac.in, ardas66@rediffmail.com

Electronic Supplementary Information

Contents	Page No.
1. Materials, method, and Experimental section	2-6
2. X-ray analysis data of compound **2u**	7-8
3. ^{1}H and ^{13}C NMR spectra for the products 3, 4, 5 and 6. HRMS spectra of Some compounds.	9-69
4. FT-IR, EDAX, XRD, SEM, TEM, HRTEM and SAED spectra of NNF	70-73
5. ICP-AES analysis data	74

1. Materials, method, and Experimental section

^{1}H-NMR and ^{13}C-NMR spectral analysis were carried out on 300 MHz, 75 MHz instrument where tetramethylsilane (TMS) was used as internal standard. Infrared spectra were recorded in KBr pallets in reflection mode on a FTIR spectrophotometer. High Resolution Mass Spectra were obtained using a mass spectrometer. Elemental analyses were done using an autoanalyzer. Suitable single crystals of compound **2u** was mounted on an X-ray diffractometer equipped with a graphite monochromator. All the reactions were monitored by thin layer chromatography carried out on aluminum-blocked silica gel plates coated with silica gel 60 GF_{254} under UV light and by exposure to iodine vapor for detection. Melting points were recorded on a Köfler Block apparatus and are uncorrected. Synthetic grade chemicals from available companies were used for carrying out the organic reactions. All amides were prepared following literature procedure[1] and for column chromatography 100-200 mesh silica gel used. All the organic solvents, used in the reaction, were properly dried and distilled prior to use.

Procedure for the synthesis of Nano Nickel Ferrite (NNF) catalyst:

25 ml 0.4M aqueous ferric chloride ($FeCl_3$. $6H_2O$) solution and 25 ml 0.2 M aqueous nickel chloride ($NiCl_2.6H_2O$) solution were taken in a 250 ml RB flask set with a magnet bar. To that mixture 3M solution of sodium hydroxide was added drop wise with persistent stirring until the pH of the medium became 12 (monitored by pH paper) along with the complete precipitation. The final volume of the solution was 70 ml and to it 3 drops of oleic acid was added. The mixture was further heated at 80^{o}C for 40 min. Then the product was cooled at room temperature and filtered under vacuum and then the residue was washed with double distilled water repeatedly until it became neutral. The residue was further washed with ethanol (3×5ml) and dried overnight at 80^{o}C. The collected mass was then grinded into fine powder and subjected to calcination for 10 h at 700 ^{o}C. The final product was confirmed to be magnetic nanoparticles of nickel ferrite ($NiFe_2O_4$) with inverse spinel structure through powder X-ray analysis) SEM, EDAX, TEM, HR-TEM, SAED, FT-IR, and XPS analyses**.** Powder X-ray analysis was performed on Rigaku SmartLab Automatic High Resolution Multipurpose PC controlled X-Ray Diffractometer (Volt-20-25KV, Currect-2-50mA). The SEM and EDAX analysis were accomplished on ZEISS EVO MA10, Germany Machine. Also TEM, HR-TEM and SAED characterization were achieved on JEOLJEM 2100. FT-IR analysis was done by Parkin ELMER-spectra2 FT-IR Spectrometer. ICP-AES analysis was completed by

ARCOS 130MV plasma atomic emission spectrometer. X-ray Photoelectron spectroscopy (XPS) measurement was recorded using Omicron X-ray photoelectron Spectrometer (serial number: 0571) with Al kα X-ray source.

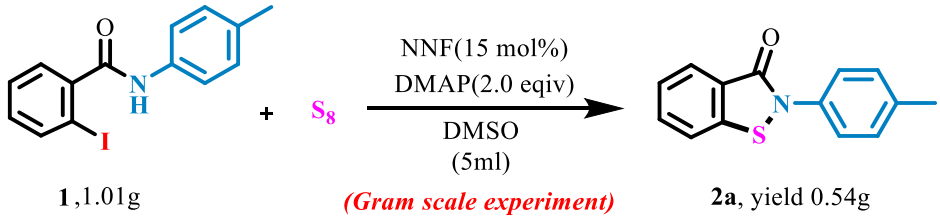
General procedure for the synthesis of products 2 and 3:

In an oven dried 15 mL pressure tube containing a mixture of 0.5 mmol of *o*-halobenzamide, 1.5 mmol of elemental sulfur (24 mg) or selenium (59.5 mg), 15 mol% of NNF (17 mg), 1.0 mmol of DMAP (2.0 equiv., 122 mg) in 2 mL DMSO was stirred at 90^{o}C temperature on a preheated oil bath for 11hr. After completion of the reaction (monitored by TLC), the resulting mixture was cooled to room temperature and quenched by addition of 20 mL of H_2O followed by the extraction with EtOAc (3 × 10 mL). The combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was then removed in vacuum and the residue was purified by column chromatography over silica gel (100-200 mesh) with ethyl acetate and petroleum ether (v/v =1:9-1:4) as the eluent to afford the corresponding benzoisothiazolones and benzoselenazolones (**2a-2z and 3a-3e**).

Gram-scale Synthesis of Benzoisothaizolone (2a):

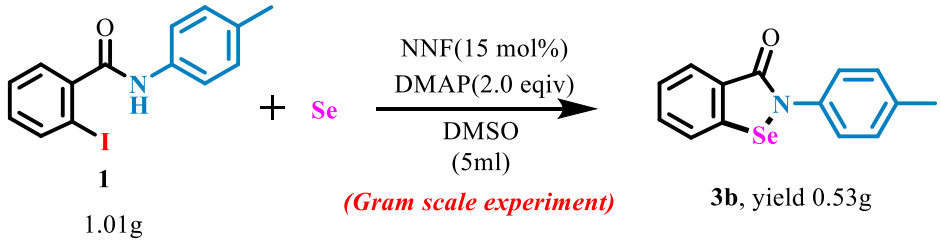
In an oven dried 15 mL pressure tube containing 3 mmol of 2-iodo-*N*-(*p*-tolyl)benzamide (1.01 g), 1.5 mmol of elemental sulfur (144 mg) 15 mol% of NNF (50 mg), 6.0 mmol of DMAP (2.0 equiv., 730 mg) in 5 mL DMSO was stirred at 90^{o}C temperature on a preheated oil bath for 11hr. After completion of the reaction (monitored by TLC), the resulting mixture was cooled to room temperature and quenched by addition of 30 mL of H_2O followed by the extraction with EtOAc (3 × 20 mL). The combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was then removed in vacuum and the residue was purified by column chromatography over silica gel (100-200 mesh) with ethyl acetate and petroleum ether (v/v =1:9) as the eluent to afford the corresponding benzoisothiazolones (**2a**).

Reaction:



Gram-scale Synthesis benzoisoselenazolone (3b):

In an oven dried 15 mL pressure tube containing a mixture of 3 mmol of 2-iodo-*N*-(*p*-tolyl)benzamide (1.01 g), 1.5 mmol of selenium (355 mg) 15 mol% of NNF (50 mg), 6.0 mmol of DMAP (2.0 equiv., 730 mg) in 5 mL DMSO was stirred at 90°C temperature on a preheated oil bath for 11hr. After completion of the reaction (monitored by TLC), the resulting mixture was cooled to room temperature and quenched by addition of 30 mL of H_2O followed by the extraction with EtOAc (3 × 20 mL). The combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was then removed in vacuum and the residue was purified by column chromatography over silica gel (100-200 mesh) with ethyl acetate and petroleum ether (v/v =1:4) as the eluent to afford the corresponding benzoisoselenazolones (**3b**).

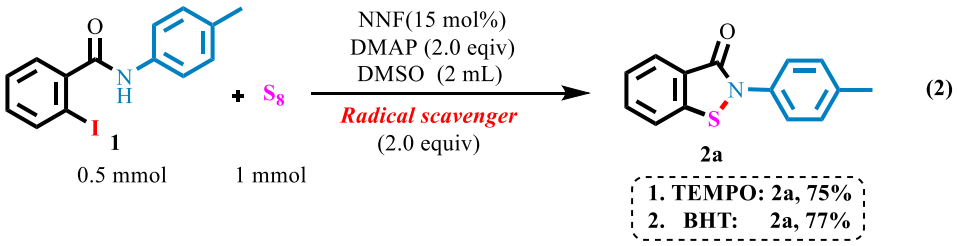


Synthesis of Benzoisothaizolone (2a) in presence of radical scavengers (TEMPO and BHT)

In an oven dried 15 mL pressure tube containing a mixture of 0.5 mmol of 2-iodo-*N*-(*p*-tolyl)benzamide (168 mg), 1.5 mmol of elemental sulfur (24 mg), 15 mol% of NNF (17 mg), 1 mmol of DMAP (2.0 equiv., 122 mg) and 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) (2 equiv.; 156.25 mg) or 2,6-di-tert-butyl-4-methylphenol (BHT) (2 equiv., 220.35 mg) in 2 mL DMSO was stirred at 90°C temperature on a preheated oil bath for 11hr. Reaction monitored by TLC, the resulting mixture was cooled to room temperature and quenched by addition of 20 mL of H_2O followed by the extraction with EtOAc (3 × 10 mL). The combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was then removed in vacuum

and the residue was purified by column chromatography over silica gel (100-200 mesh) with ethyl acetate and petroleum ether (v/v =1.5:10) as the eluent to afford the corresponding benzoisothiazolones (**2a**).

Reaction:



(2)

General procedure for the synthesis of products 4

In an oven dried 25 mL RB flask containing a mixture of 0.5 mmol of benzisothiazolones **2**(alky or aryl) and 3 equiv. of Tert-butyl nitrite (TBN) (155 mg) were taken in 2 mL DCE and stir well, then mixture was then stirred at 70^{o}C temperature on a preheated oil bath for 1.5hr under oxygen atmosphere. After completion of the reaction (monitored by TLC) the mixture was cooled to room temperature and quenched by addition of 20 mL of H_2O followed by the extraction with EtOAc (2 × 10 mL). The combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was then removed in vacuo and the residue was purified by column chromatography over silica gel (100-200 mesh) with ethyl acetate and petroleum ether (v/v=1:4) as the eluent to afford the corresponding Benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide derivatives (**4a-4e)**.

General procedure for the synthesis of products 5

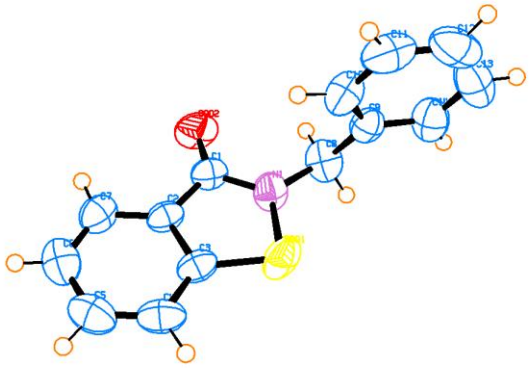
In an oven dried 15 mL pressure tube containing $[RuCl_2(p\text{-cymene})]_2$ (5 mol%, 3 mg) in 0.5 mL DCE, $AgSbF_6$ (20 mol%, 7 mg) was added to it and stirred for 3 min under air. To this solution, **2** (0.1 mmol) and methyl acrylate (0.3 mmol, 3 eq., 26 mg) or Styrene (0.3 mmol, 3 eq., 32 mg) was added and stir well. Finally $Cu(OAc)_2 .H_2O$ (0.12 mmol, 1.2 eq., 24 mg) added to it and another 1mL DCE was added to the above content. Then the pressure tube was sealed under air and heated in preheated oil bath for a stipulated period of time (monitored by TLC). After completion of the reaction, the reaction mixture was diluted with EtOAc and filtered through a pad of celite and

concentrated and directly subjected to column chromatography to get the pure alkenylated product **5 (5a-5e)** as a white solid in % yield.

General procedure for the synthesis of products 6

In an oven 10mL RB flask, 0.1mmol of benzisoselenzoles (**3**) was taken with 0.5mL dry DMF and stir well at rt for few minutes. After that 1.1equiv. (18 mg) of triethyl phosphite added to it at room temperature and the stirring was continued until the full consumption of reactants (monitored by TLC). After the reaction over it was diluted with EtOAc. The combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. The solvent was then removed in vacuum and the residue was purified by column chromatography over silica gel (100-200 mesh) with ethyl acetate and petroleum ether (v/v=1:4) as the eluent to afford the corresponding selenols **(6a-c)**.

2. X-ray analysis data of compound 2u (CCDC: 2165367)



The Ortep diagram of X-ray crystal structure of compound **2u**. The ellipsoid contour percent probability level is 50%. Color code: Red: Oxygen, Pink: Nitrogen, Sky blue: Carbon, Orange: Hydrogen, Yellow: Sulfur. The Crystal was grown from Dichloromethane.

Table 1. Crystallographic data for the compound **2u.**

Compound	**2u**
Empirical formula	$C_{14}H_{11}NOS$
Formula weight	241.30
Crystal system	monoclinic
Space group	P 21/c
a (Å)	9.6609(7)
b(Å)	6.3361(5)
c (Å)	19.6298(14)
α (°)	90
β (°)	96.172(3)
γ (°)	90
V (Å^3)	1194.62(15)
Z	4

T, K	296(2)
Wavelength (Å)	0.71073
2θ (°)	2.087-24.995
μ (mm^{-1})	0.252
ρ_{calcd} (g cm^{-3})	1.342
F (000)	504
Absorption correction	multi-scan
Index ranges	–11≤h≤11
	–7≤k≤7
	–23≤ l≤23
Reflections collected	9949
Independent reflections (R_{int})	9949(0.0461)
Goodness-of-fit on F^2	1.040
$R_1{}^a$ /w$R_2{}^b$ (I> 2σ(I))	0.0645/0.1727
$R_1{}^a$/w$R_2{}^b$ (for all data)	0.0677/0.1767
Largest diff. peak/hole /eÅ^{-3}	0.540/-0.629

$^a R_1 = [\sum||F_o| - |F_c||/\sum|F_o|]$. $^b wR_2 = [\sum w (F_o^2 - F_c^2)^2/\sum wF_o^4]^{1/2}$

References:

1. *APEX-II, SAINT-Plus*, and *TWINABS*; Bruker-Nonius AXS Inc.: Madison, WI, 2004.
2. *SHELXTL*, version 6.10; Bruker AXS Inc.: Madison, WI, 2002.
3. Sheldrick, G. M. *SHELXL-97, Crystal Structure Refinement Program*; University of Göttingen: Göttingen, Germany, 199

3. Characterization data for the Products Table 2, 3, 4, 5 and 6.

2-(*p*-tolyl) benzo[*d*]isothiazol-3(2*H*)-one (2a). white Solid (106 mg, 88%); Mp:132-133^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.12 (d, *J* = 7.9 Hz, 1H), 7.71 – 7.64 (m, 1H), 7.59 (dd, *J* = 8.2, 3.5 Hz, 3H), 7.49 – 7.42 (m, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 2.41 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.19, 139.97, 137.25, 134.52, 132.25, 129.96, 127.14, 125.76, 124.83, 124.77, 120.11, 21.14; IR (KBr):1650,1470,1309,1025,740cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NOS$]: C 69.68; H 4.59; N 5.80, found: C, 69.66; H, 4.60; N, 5.81%.

2-phenylbenzo[*d*]isothiazol-3(2*H*)-one (2b). white Solid (98 mg, 86%); Mp:140-141^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.02 (d, *J* = 7.9 Hz, 1H), 7.66 – 7.53 (m, 3H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.37 (ddd, *J* = 16.1, 7.8, 1.5 Hz, 3H), 7.23 (t, *J* = 7.4 Hz, 1H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.15, 139.89, 137.23, 132.38, 129.40, 127.19, 127.09, 125.84, 124.84, 124.62, 120.13; IR (KBr):1652,1455,1323,995,742,cm^{-1}; Anal. Calcd for [$C_{13}H_9NOS$]: C, 68.70; H, 3.99; N, 6.16, found: C, 68.71; H, 3.98; N, 6.17%.

2-(4-fluorophenyl) benzo[*d*]isothiazol-3(2*H*)-one (2c). White Solid (109 mg, 89%); Mp:100-101^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.03 (d, *J* = 7.9 Hz, 1H), 7.66 – 7.47 (m, 4H), 7.43 – 7.35 (m, 1H), 7.15 – 7.04 (m, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.25, 161.26 ($^{1}J_{C-F}$ =246 Hz), 139.84, 132.48, 127.25, 126.86 (d, $^{3}J_{C-F}$ = 8.5 Hz), 125.95, 124.49, 120.14, 116.30 (d, $^{2}J_{C-F}$ = 23.0 Hz); IR (KBr): 1660,1464,1305,1005,739cm^{-1}; Anal. Calcd for [$C_{13}H_8FNOS$]: C, 63.66; H, 3.29; N, 5.71, found: C, 63.68; H, 3.28; N, 5.70%.

2-(4-chlorophenyl) benzo[*d*]isothiazol-3(2*H*)-one (2d). white Solid (110 mg, 85%); Mp:153-154^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.02 (d, *J* = 7.9 Hz, 1H), 7.64 – 7.54 (m, 3H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.42 – 7.31 (m, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.13, 139.64,

135.83, 132.61, 132.49, 129.48, 127.26, 126.00, 125.63, 124.60, 120.15; IR (KBr): 1655,1473,1290,998,735cm^{-1}; Anal. Calcd for [$C_{13}H_8ClNOS$]: C, 59.66; H, 3.08; N, 5.35, found: C, 59.67; H, 3.08; N, 5.34%.

2-(4-bromophenyl)benzo[*d*]isothiazol-3(2*H*)-one (2e). white Solid (130 mg, 87%); Mp:129-130^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.02 (d, *J* = 7.8 Hz, 1H), 7.65 – 7.46 (m, 6H), 7.43 – 7.34 (m, 1H).; ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 163.95, 139.44, 136.25, 132.51, 132.30, 127.11, 125.89, 125.66, 124.48, 120.17, 120.05; IR (KBr): 1658,1479,1315,1020,742cm^{-1}; Anal. Calcd for [$C_{13}H_8BrNOS$]: C, 51.00; H, 2.63; N, 4.57, found: C, 51.02; H, 2.61; N, 4.57%.

2-(4-methoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one (2f). white Solid (107 mg, 83%); Mp:105-106^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.02 (d, *J* = 7.8 Hz, 1H), 7.62 – 7.53 (m, 1H), 7.52 – 7.44 (m, 3H), 7.40 – 7.33 (m, 1H), 6.95 – 6.87 (m, 2H), 3.76 (s, 3H).; ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.25, 158.66, 139.98, 132.11, 129.54, 127.05, 126.78, 125.68, 124.52, 120.01, 114.52, 55.50; IR (KBr):1648,1468,1305,1005,744cm^{-1}; HRMS (ESI-TOF) m/z Calcd for [$C_{14}H_{11}NO_2S$+H]$^+$: 258.0583, found: 258.0574.

2-(4-(*tert*-butyl)phenyl)benzo[*d*]isothiazol-3(2*H*)-one (2g). white Solid (120 mg, 85%); Mp:124-125^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.07 – 7.98 (m, 1H), 7.61 – 7.46 (m, 4H), 7.44 – 7.32 (m, 3H), 1.27 (s, 9H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.11, 150.24, 139.91, 134.38, 132.17, 127.10, 126.28, 125.68, 124.79, 124.34, 120.02, 34.62, 31.24; IR (KBr): 1652,1465,1300,1020,730cm^{-1}; Anal. Calcd for [$C_{17}H_{17}NOS$]: C, 72.05; H, 6.05; N, 4.94, found: C, 72.06; H, 6.05; N, 4.93%.

2-(*m*-tolyl)benzo[*d*]isothiazol-3(2*H*)-one (2h). white Solid (99 mg, 82%); Mp:120-121^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.03 (d, *J* = 7.9 Hz, 1H), 7.63 – 7.55 (m, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.47 – 7.33 (m, 3H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.06 (d, *J* = 7.6 Hz, 1H), 2.34 (s, 3H); ^{13}C-

NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.09, 139.89, 139.38, 137.01, 132.22, 129.11, 127.94, 127.11, 125.71, 125.31, 124.83, 121.70, 120.01, 21.39; IR (KBr): 1655,1471,1306,1025,735cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NOS$]: C, 69.68; H, 4.59; N, 5.80, found: C, 69.67; H, 4.60; N, 5.81%.

2-(3-chlorophenyl)benzo[*d*]isothiazol-3(2*H*)-one (2i). white Solid (114 mg, 87%); Mp:135-136^oC; ^1H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.03 (d, *J* = 7.9 Hz, 1H), 7.73 (t, *J* = 2.0 Hz, 1H), 7.66 – 7.49 (m, 3H), 7.43 – 7.28 (m, 2H), 7.22 (ddd, *J* = 8.1, 1.9, 1.0 Hz, 1H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.11, 139.64, 138.43, 135.00, 132.70, 130.30, 127.30, 127.01, 126.03, 124.61, 124.39, 122.26, 120.14; IR (KBr): 1658,1465,1310,1021,732cm^{-1}; Anal. Calcd for [$C_{13}H_8ClNOS$]: C, 59.66; H, 3.08; N, 5.35, found: C, 59.67; H, 3.07; N, 5.36%.

2-(4-bromo-3-methylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (2j). white Solid (134 mg, 84%); Mp:125-126^oC; ^1H NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.02 (d, *J* = 7.9 Hz, 1H), 7.63 – 7.56 (m, 1H), 7.52 (dd, *J* = 11.6, 5.3 Hz, 3H), 7.42 – 7.28 (m, 2H), 2.37 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.06, 139.65, 139.20, 136.27, 133.03, 132.48, 127.18, 126.58, 125.90, 124.60, 123.23, 123.00, 120.07, 23.10; IR (KBr): 1650,1479,1309,1025,740cm^{-1}; HRMS (ESI-TOF) m/z Calcd for $[C_{14}H_{10}BrNOS+H]^+$: 319.9739 and 321.9719, found: 319.9702 and 321.9598.

2-(3,5-dimethoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one (2k). white Solid (114 mg, 79%); Mp:99-100^oC; ^1H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.01 (d, *J* = 7.9 Hz, 1H), 7.59 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.41 – 7.32 (m, 1H), 6.86 (d, *J* = 2.2 Hz, 2H), 6.34 (t, *J* = 2.2 Hz, 1H), 3.75 (s, 6H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.15, 161.13, 139.86, 138.84, 132.45, 127.13, 125.85, 124.99, 120.04, 102.70, 99.42, 55.59; IR (KBr): 1640,1466,1320,1027,730cm^{-1}; Anal. Calcd for [$C_{15}H_{13}NO_3S$]: C, 62.70; H, 4.56; N, 4.87, found: C, 62.72; H, 4.56; N, 4.85%.

2-(*o*-tolyl)benzo[*d*]isothiazol-3(2*H*)-one (2l). white Solid (98 mg, 81%); Mp:108-109^oC; ^1H-

NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.15 (d, *J* = 7.9 Hz, 1H), 7.70 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.52 – 7.44 (m, 1H), 7.43 – 7.29 (m, 4H), 2.28 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si) : δ 164.55, 141.25, 137.78, 134.80, 132.19, 131.31, 129.59, 129.08, 127.27, 127.00, 125.73, 123.93, 120.28, 18.06; IR (KBr): 1653,1471,1310,1020,743cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NOS$]: C, 69.68; H, 4.59; N, 5.80, found: C, 69.67; H, 4.58; N, 5.81%.

4-(3-oxobenzo[*d*]isothiazol-2(3*H*)-yl)benzonitrile (2m). white Solid (102 mg, 81%); Mp:145-146ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.04 (d, *J* = 7.9 Hz, 1H), 7.95 – 7.84 (m, 2H), 7.74 – 7.59 (m, 3H), 7.53 (d, *J* = 8.1 Hz, 1H), 7.46 – 7.35 (m, 1H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.23, 141.65, 139.22, 133.33, 133.23, 127.48, 126.33, 124.55, 123.31, 120.21, 118.33, 109.49; IR (KBr):2228, 1655,1329,1138,740cm^{-1}; Anal. Calcd for [$C_{14}H_8N_2OS$]: C, 66.65; H, 3.20; N, 11.10, found: C, 66.66; H, 3.21; N, 11.09%.

2-(pyridin-3-yl)benzo[*d*]isothiazol-3(2*H*)-one (2n). white Solid (90 mg, 79%); Mp:96-98ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.90 (d, *J* = 1.8 Hz, 1H), 8.49 (d, *J* = 4.2 Hz, 1H), 8.16 (ddd, *J* = 8.3, 2.5, 1.4 Hz, 1H), 8.04 (d, *J* = 7.9 Hz, 1H), 7.64 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.45 – 7.35 (m, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.36, 146.84, 144.19, 139.57, 132.90, 131.86, 127.28, 126.14, 124.00, 120.25; IR (KBr): 1655,1490,1319,1125,742cm^{-1}; HRMS (ESI-TOF) m/z Calcd for [$C_{12}H_8N_2OS$+H]$^+$: 229.0430, found: 229.0451.

2-cyclohexylbenzo[*d*]isothiazol-3(2*H*)-one (2o). white Solid (106 mg, 91%); Mp:85-86ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.97 (d, *J* = 7.9 Hz, 1H), 7.55 – 7.44 (m, 2H), 7.31 (ddd, *J* = 8.0, 6.1, 2.0 Hz, 1H), 4.62 – 4.40 (m, 1H), 1.98 (d, *J* = 10.3 Hz, 2H), 1.81 (d, *J* = 12.0 Hz, 2H), 1.66 (d, *J* = 13.1 Hz, 1H), 1.55 – 1.32 (m, 4H), 1.24 – 1.05 (m, 1H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.65, 140.16, 131.30, 126.34, 125.28, 125.17, 120.23, 53.06, 32.77, 25.47, 25.09; IR

(KBr): 2905,1660,1472,1300,925,cm^{-1}; Anal. Calcd for [$C_{13}H_{15}NOS$]: C, 66.92; H, 6.48; N, 6.00, found: C, 66.90; H, 6.49; N, 6.02%.

2-cyclopropylbenzo[*d*]isothiazol-3(2*H*)-one (2p). yellow liquid (86 mg, 90%); ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.02 (d, *J* = 7.9 Hz, 1H), 7.66 – 7.56 (m, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.44 – 7.36 (m, 1H), 3.22 – 3.08 (m, 1H), 1.19 – 1.02 (m, 4H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 166.43, 140.40, 131.74, 126.38, 125.32, 125.20, 120.08, 26.42, 7.85; IR (KBr): 3100,1662,1473,1290,925cm^{-1}; Anal. Calcd for [$C_{10}H_9NOS$]: C, 62.80; H, 4.74; N, 7.32, found: C, 62.81; H, 4.72; N, 7.33%.

2-octylbenzo[*d*]isothiazol-3(2*H*)-one (2q). yellow liquid (121 mg, 92%); ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.06 (d, *J* = 7.9 Hz, 1H), 7.67 – 7.52 (m, 2H), 7.42 (ddd, *J* = 8.0, 6.8, 1.3 Hz, 1H), 3.99 – 3.82 (m, 2H), 1.96 – 1.68 (m, 3H), 1.34 (dd, *J* = 23.6, 7.8 Hz, 10H), 0.89 (t, *J* = 6.7 Hz, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.13, 140.01, 131.46, 126.47, 125.24, 124.68, 120.17, 43.83, 31.60, 29.41, 29.01, 28.97, 26.44, 22.47, 13.94; IR (KBr): 2830,1655,1370,1200,925cm^{-1}; Anal. Calcd for [$C_{15}H_{21}NOS$]: C, 68.40; H, 8.04; N, 5.32, found: C, 68.42; H, 8.03; N, 5.31%.

6-chloro-2-phenylbenzo[*d*]isothiazol-3(2*H*)-one (2r). white Solid (103 mg, 79%); Mp:155-156^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.77 – 7.66 (m, 2H), 7.62 (d, *J* = 1.4 Hz, 1H), 7.50 (t, *J* = 7.8 Hz, 2H), 7.44 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.36 (t, *J* = 7.4 Hz, 1H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 163.21, 141.09, 138.98, 136.84, 129.43, 128.21, 127.30, 126.75, 124.57, 123.27, 119.88; IR (KBr): 1654,1430,1300,1003,730cm^{-1}; Anal. Calcd for [$C_{13}H_8ClNOS$]: C, 59.66; H, 3.08; N, 5.35, found: C, 59.67; H, 3.07; N, 5.36%.

6-chloro-2-(p-tolyl)benzo[*d*]isothiazol-3(2*H*)-one (2s). white Solid (103 mg, 75%); Mp:142-143^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.04 (d, *J* = 8.4 Hz, 1H), 7.58 (dd, *J* = 14.4, 4.9 Hz,

3H), 7.42 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.30 (d, *J* = 7.3 Hz, 2H), 2.41 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 163.24, 141.16, 138.81, 137.50, 134.12, 129.98, 128.13, 126.65, 124.71, 123.25, 119.85, 21.08; IR (KBr): 1655,1472,1296,990,724cm^{-1}; Anal. Calcd for [$C_{14}H_{10}ClNOS$]: C, 60.98; H, 3.66; N, 5.08, found: C, 60.99; H, 3.67; N, 5.07%.

2-benzyl-6-chlorobenzo[*d*]isothiazol-3(2*H*)-one (2t). white Solid (101 mg, 73%); Mp:97-98^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.00 (d, *J* = 8.4 Hz, 1H), 7.51 (d, *J* = 1.6 Hz, 1H), 7.44 – 7.32 (m, 6H), 5.06 (s, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.42, 141.59, 138.42, 135.79, 128.85, 128.40, 127.75, 126.41, 122.93, 120.13, 47.64.; IR (KBr): 1656,1440,1300,1015,735cm^{-1}; Anal. Calcd for [$C_{14}H_{10}ClNOS$]: C, 60.98; H, 3.66; N, 5.08, found: C, 60.99; H, 3.67; N, 5.07%.

2-benzylbenzo[*d*]isothiazol-3(2*H*)-one (2u). white Solid (107 mg, 89%); Mp:85-86^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.14 – 8.06 (m, 1H), 7.60 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.54 – 7.47 (m, 1H), 7.46 – 7.40 (m, 1H), 7.40 – 7.30 (m, 5H), 5.08 (s, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.37, 140.41, 136.17, 131.90, 128.87, 128.47, 128.34, 126.84, 125.55, 124.46, 120.44, 47.59; IR (KBr): 1656,1500,1310,995,742cm^{-1}; HRMS (ESI-TOF) m/z Calcd for [$C_{14}H_{11}NOS$+Na]$^{+}$: 264.0454, found: 264.0462.

2-(4-methylbenzyl)benzo[*d*]isothiazol-3(2*H*)-one (2v). white Solid (107 mg, 84%); Mp:121-122^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.10 (d, *J* = 7.9 Hz, 1H), 7.65 – 7.56 (m, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.46 – 7.39 (m, 1H), 7.30 – 7.24 (m, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 5.04 (s, 2H), 2.37 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.27, 140.37, 138.16, 133.11, 131.74, 129.49, 128.49, 126.75, 125.43, 124.58, 120.34, 47.33, 21.16; IR (KBr): 1655,1440,1298,1002,743cm^{-1}; Anal. Calcd for [$C_{15}H_{13}NOS$]: C, 70.56; H, 5.13; N, 5.49, found: C, 70.55; H, 5.14; N, 5.49%.

2-(4-chlorobenzyl)benzo[*d*]isothiazol-3(2*H*)-one (2w). white Solid (121 mg, 88%); Mp:94-

95ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.09 (d, *J* = 7.8 Hz, 1H), 7.62 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.52 (d, *J* = 8.1 Hz, 1H), 7.47 – 7.39 (m, 1H), 7.38 – 7.28 (m, 4H), 5.04 (s, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.38, 140.29, 134.70, 134.23, 132.06, 129.78, 129.05, 126.90, 125.68, 124.28, 120.48, 46.86; IR (KBr): 1655,1450,1300,1035,747cm^{-1}; Anal. Calcd for [$C_{14}H_{10}ClNOS$]: C, 60.98; H, 3.66; N, 5.08, found: C, 60.97; H, 3.65; N, 5.09%.

(S)-2-(1-phenylethyl)benzo[*d*]isothiazol-3(2*H*)-one (2x). white Solid (109 mg, 85%); Mp:80-81ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.07 (d, *J* = 7.8 Hz, 1H), 7.59 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.52 – 7.31 (m, 7H), 6.09 (q, *J* = 7.0 Hz, 1H), 1.82 (d, *J* = 7.0 Hz, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.93, 140.36, 140.12, 131.56, 128.63, 128.15, 127.25, 126.56, 125.31, 124.99, 120.34, 52.11, 19.10; IR (KBr): 1655,1398,1209,1120,742cm^{-1}; Anal. Calcd for [$C_{15}H_{13}NOS$]: C, 70.56; H, 5.13; N, 5.49, found: C, 70.58; H, 5.12; N, 5.48%.

2-(furan-2-ylmethyl)benzo[*d*]isothiazol-3(2*H*)-one (2y). white Solid (83 mg, 72%); Mp:102-103ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 7.98 (d, *J* = 7.9 Hz, 1H), 7.57 – 7.47 (m, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.38 – 7.27 (m, 2H), 6.36 (d, *J* = 3.1 Hz, 1H), 6.29 (dd, *J* = 3.1, 1.9 Hz, 1H), 4.98 (s, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.07, 149.28, 143.19, 140.47, 131.92, 126.81, 125.50, 124.41, 120.39, 110.64, 109.81, 40.08; IR (KBr): 1660,1473,1319,1125,770cm^{-1}; Anal. Calcd for [$C_{12}H_9NO_2S$]: C, 62.32; H, 3.92; N, 6.06, found: C, 62.31; H, 3.90; N, 6.08%.

2-benzylisothiazolo[5,4-*b*]pyridin-3(2*H*)-one (2z). light yellow gummy liquid (63 mg, 52%); ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.73 – 8.64 (m, 1H), 8.25 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.38 – 7.27 (m, 6H), 5.04 (s, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 163.63, 162.33, 153.61, 135.75, 134.85, 128.94, 128.49, 128.47, 120.73, 119.27, 47.45. IR (KBr): 1661,1495, 1350, 1134, 800cm^{-1}; Anal. Calcd for [$C_{13}H_{10}NO_2S$]: C, 64.44; H, 4.16; N, 11.56, found: C, 62.42; H, 4.15; N, 11.19%.

2-phenylbenzo[*d*][1,2]selenazol-3(2*H*)-one (3a). white Solid (78 mg, 57%); Mp: 182-183^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.14 (d, *J* = 7.7 Hz, 1H), 7.67 (m, 4H), 7.53 – 7.40 (m, 3H), 7.31 (dd, *J* = 9.4, 3.2 Hz, 1H).; ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.75, 139.14, 137.68, 132.55, 129.40, 129.34, 127.59, 126.77, 126.56, 125.45, 123.78; IR (KBr): 1650,1460,1200,745cm^{-1}; Anal. Calcd for [$C_{13}H_9NOSe$]: C, 56.95; H, 3.31; N, 5.11, found: C, 56.94; H, 3.32; N, 5.10%.

2-(*p*-tolyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (3b). white Solid (91 mg, 63%); Mp:170-171^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.13 (d, *J* = 7.7 Hz, 1H), 7.73 – 7.61 (m, 2H), 7.55 – 7.44 (m, 3H), 7.26 (t, *J* = 7.7 Hz, 2H), 2.39 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.81, 137.83, 136.88, 136.41, 132.43, 129.91, 129.33, 127.55, 126.49, 125.52, 123.83, 21.11; IR (KBr): 1653,1464,1220,744cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NOSe$]: C, 58.34; H, 3.85; N, 4.86, found: C, 58.35; H, 3.84; N, 4.87%.

2-(4-fluorophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (3c). white Solid (98 mg, 67%); Mp:176-177^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.13 (d, *J* = 7.8 Hz, 1H), 7.73 – 7.64 (m, 2H), 7.58 (ddd, *J* = 10.3, 5.1, 2.8 Hz, 2H), 7.49 (ddd, *J* = 8.0, 6.4, 1.8 Hz, 1H), 7.20 – 7.09 (m, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.93, 161.05 (d, $^{1}J_{C\text{-}F}$ = 247.3 Hz), 137.68, 134.87, 132.68, 129.43, 127.60 (d, $^{3}J_{C\text{-}F}$ = 8.4 Hz), 127.10, 126.68, 123.85, 116.21 (d, $^{2}J_{C\text{-}F}$ = 22.8 Hz); IR (KBr): 1655,1475,1210,743cm^{-1}; HRMS (ESI-TOF) m/z Calcd for [$C_{13}H_8FNOSe$+H]$^{+}$: 293.9828, found: 293.9829.

2-(3,5-dimethylphenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (3d). white Solid (92 mg, 61%); Mp:165-66^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.13 (d, *J* = 7.9 Hz, 1H), 7.73 – 7.60 (m, 2H), 7.48 (ddd, *J* = 8.0, 6.6, 1.7 Hz, 1H), 7.25 (s, 2H), 6.94 (s, 1H), 2.37 (s, 6H); ^{13}C-NMR (75

MHz; $CDCl_3$; Me_4Si): δ 165.78, 139.12, 138.77, 137.88, 132.41, 129.33, 128.75, 127.66, 126.47, 123.77, 123.33, 21.30; IR (KBr): 1653,1463,1245,743cm^{-1}; Anal. Calcd for [$C_{15}H_{13}NOSe$]: C, 59.61; H, 4.34; N, 4.63, found: C, 59.60; H, 4.35; N, 4.62%.

2-benzylbenzo[*d*][1,2]selenazol-3(2*H*)-one (3e). white Solid (102 mg, 71%); Mp:131-133^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.10 (d, *J* = 7.7 Hz, 1H), 7.67 – 7.52 (m, 2H), 7.50 – 7.41 (m, 1H), 7.33 (d, *J* = 26.6 Hz, 5H), 5.03 (s, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 167.21, 138.14, 137.22, 132.04, 128.92, 128.88, 128.56, 128.35, 127.40, 126.25, 124.05, 48.66; IR (KBr): 1654,1465,1230,740cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NOSe$]: C, 58.34; H, 3.85; N, 4.86, found: C, 58.33; H, 3.84; N, 4.87%.

2-phenylbenzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4a). white Solid (117 mg, 90%); Mp:151-152^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.16 – 8.08 (m, 1H), 7.99 (d, *J* = 7.1 Hz, 1H), 7.87 (dtd, *J* = 16.0, 7.4, 1.2 Hz, 2H), 7.61 – 7.43 (m, 5H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.52, 145.53, 134.63, 133.92, 133.46, 129.76, 128.91, 128.25, 127.39, 126.77, 125.24; IR (KBr):1680,1250,1007,748cm^{-1}; Anal. Calcd for [$C_{13}H_9NO_3S$]: C, 60.22; H, 3.50; N, 5.40, found: C, 60.20; H, 3.51; N, 5.40%.

2-(*p*-tolyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4b). white Solid (126 mg, 92%); Mp:148-149^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.10 – 8.03 (m, 1H), 7.94 (dd, *J* = 6.9, 1.2 Hz, 1H), 7.81 (dtd, *J* = 16.9, 7.4, 1.3 Hz, 2H), 7.42 – 7.34 (m, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 2.40 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.64, 145.58, 139.15, 134.55, 133.40, 131.08, 130.40, 128.29, 127.42, 126.70, 125.23, 21.28; IR (KBr):1682,1256,1000,750cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NO_3S$]: C, 61.53; H, 4.06; N, 5.13, found: C, 61.52; H, 4.05; N, 5.14%.

2-(4-fluorophenyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4c). Light yellow Solid (130 mg,

94%); Mp:120-121°C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.18 – 8.06 (m, 1H), 7.99 (d, *J* = 7.1 Hz, 1H), 7.87 (dtd, *J* = 17.6, 7.3, 1.2 Hz, 2H), 7.57 – 7.44 (m, 2H), 7.27 – 7.18 (m, 2H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.61, δ 162.65 (d, $^{1}J_{C-F}$ = 249.3 Hz), 145.48, 134.73, 133.53, 129.61 (d, $^{3}J_{C-F}$ = 8.9 Hz), 127.99, 126.79, 125.29, 116.78 (d, $^{2}J_{C-F}$ = 22.9 Hz); IR (KBr):1682,1245, 1020,746cm^{-1}; Anal. Calcd for [$C_{13}H_8FNO_3S$]: C, 56.31; H, 2.91; N, 5.05, found: C, 56.30; H, 2.92; N, 5.06%.

2-(4-methoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4d). Light yellow Solid (132 mg, 91%); Mp:126-127°C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.16 – 8.06 (m, 1H), 7.98 (d, *J* = 6.9 Hz, 1H), 7.92 – 7.79 (m, 2H), 7.49 – 7.38 (m, 2H), 7.09 – 7.00 (m, 2H), 3.88 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 164.70, 160.00, 145.54, 134.43, 133.28, 129.22, 128.17, 126.60, 125.98, 125.13, 114.95, 55.50; IR (KBr):1715,1297,1097,825cm^{-1}; HRMS (ESI-TOF) m/z Calcd for [$C_{14}H_{11}NO_4S$+K]$^{+}$: 328.0040, found: 328.0064.

2-benzylbenzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4e). white Solid (120 mg, 88%); Mp:89-90°C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.11 – 8.00 (m, 1H), 7.95 – 7.87 (m, 1H), 7.86 – 7.74 (m, 2H), 7.45 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.41 – 7.30 (m, 3H), 5.33 (d, *J* = 15.3 Hz, 1H), 4.76 (d, *J* = 15.3 Hz, 1H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.22, 145.66, 135.82, 134.26, 133.28, 128.90, 128.66, 128.27, 128.22, 126.33, 125.23, 44.31; IR (KBr):1687,1302,1257,1010,748cm^{-1}; Anal. Calcd for [$C_{14}H_{11}NO_3S$]: C, 61.53; H, 4.06; N, 5.13, found: C, 61.55; H, 4.05; N, 5.11%.

Methyl (*E*)-3-(2-(3-oxobenzo[*d*]isothiazol-2(3*H*)-yl)phenyl)acrylate (5a). white Solid (26 mg, 82%); Mp:100-102°C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.13 (d, *J* = 7.8 Hz, 1H), 7.84 – 7.76 (m, 1H), 7.68 (tt, *J* = 9.9, 4.6 Hz, 3H), 7.55 – 7.42 (m, 4H), 6.49 (d, *J* = 16.0 Hz, 1H), 3.74 (s, 3H);

^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 166.88, 165.20, 141.18, 139.34, 135.44, 133.79, 132.58, 131.03, 129.87, 129.69, 127.56, 127.47, 126.00, 123.35, 120.71, 120.33, 51.79; IR (KBr):1720,1655,1390,1210,745cm^{-1}; HRMS (ESI-TOF) m/z Calcd for $[C_{17}H_{13}NO_3S+Na]^+$: 334.0508, found: 334.0518 and $[C_{17}H_{13}NO_3S+H]^+$:312.0689, found: 312.0693.

Methyl (*E*)-3-(5-methyl-2-(3-oxobenzo[*d*]isothiazol-2(3*H*)-yl)phenyl)acrylate (5b). Pale yellow solid (27 mg, 85%); Mp:97-98ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.12 (d, *J* = 7.9 Hz, 1H), 7.65 (ddd, *J* = 22.6, 12.1, 6.9 Hz, 4H), 7.47 (t, *J* = 7.2 Hz, 1H), 7.32 (s, 2H), 6.47 (d, *J* = 16.0 Hz, 1H), 3.72 (s, 3H), 2.44 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 166.91, 165.28, 141.18, 139.88, 139.42, 133.40, 132.88, 132.48, 131.89, 129.65, 127.98, 127.39, 125.91, 123.42, 120.44, 120.32, 51.74, 21.33; IR (KBr): 1725,1650,1388,1206,742cm^{-1}; Anal. Calcd for $[C_{18}H_{15}NO_3S]$: C, 66.44; H, 4.65; N, 4.30, found: C, 66.45; H, 4.66; N, 4.29%.

Methyl (*E*)-3-(3-methyl-2-(3-oxobenzo[*d*]isothiazol-2(3*H*)-yl)phenyl)acrylate (5c). Pale yellow solid (26 mg, 80%); Mp:92-94ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.07 (d, *J* = 7.8 Hz, 1H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.58 – 7.49 (m, 2H), 7.38 (m, 3H), 7.19 (s, 1H), 6.37 (d, *J* = 15.9 Hz, 1H), 3.63 (s, 3H), 2.12 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 166.85, 164.93, 141.42, 139.42, 139.28, 134.68, 132.82, 132.52, 130.02, 127.55, 125.91, 125.10, 123.54, 121.04, 120.55, 51.76, 17.95; IR (KBr): 1724,1655,140,1200,740cm^{-1}; Anal. Calcd for $[C_{18}H_{15}NO_3S]$: C, 66.44; H, 4.65; N, 4.30; found: C, 66.43; H, 4.66; N, 4.31%.

(*E*)-2-(4-methyl-2-styrylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (5d). Pale yellow solid (25 mg, 72%); Mp:80-81ºC; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.18 (d, *J* = 7.1 Hz, 1H), 7.79 – 7.57 (m, 3H), 7.52 – 7.01 (m, 10H), 2.47 (s, 3H); ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si): δ 165.30, 141.32, 139.77, 137.05, 136.24, 132.30, 131.60, 131.39, 129.57, 129.40, 128.62, 128.00, 127.40,

127.06, 126.88, 125.76, 123.77, 123.33, 120.31, 21.48; IR (KBr): 1655,1370,1105,744cm^{-1}; Anal. Calcd for [$C_{22}H_{17}NOS$]: C, 76.94; H, 4.99; N, 4.08, found: C, 76.95; H, 4.98; N, 4.07%.

(*E*)-2-(4-methoxy-2-styrylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (5e). Pale yellow solid (27 mg, 75%); Mp:89-90^{o}C; ^{1}H-NMR (300 MHz; $CDCl_3$; Me_4Si): δ 8.17 (d, *J* = 7.8 Hz, 1H), 7.76 – 7.66 (m, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.43 – 7.24 (m, 7H), 7.14 (d, *J* = 16.2 Hz, 1H), 7.06 – 6.92 (m, 2H), 3.93 (s, 3H). ^{13}C-NMR (75 MHz; $CDCl_3$; Me_4Si) δ 165.43, 160.34, 141.29, 137.89, 136.84, 132.30, 132.02, 131.01, 128.63, 128.14, 127.40, 126.93, 126.74, 125.75, 123.74, 123.25, 120.29, 114.41, 111.12, 55.68; IR (KBr): 1655,1400,1280,740cm^{-1}; Anal. Calcd for [$C_{18}H_{15}NO_3S$]: C, 73.51; H, 4.77; N, 3.90, found: C, 73.52; H, 4.76; N, 3.91%.

***N*-(3, 5-dimethylphenyl)-2-hydroselenobenzamide (6a).** White Solid (22 mg, 72%); Mp:120^{o}C; ^{1}H-NMR (300 MHz; DMSO-d_6): δ 10.42 (s, 1H), 7.94 (d, *J* = 7.0 Hz, 1H), 7.79 (d, *J* = 7.1 Hz, 1H), 7.44 (dd, *J* = 14.9, 6.8 Hz, 4H), 6.80 (s, 1H), 2.29 (s, 6H). ^{13}C-NMR (75 MHz; DMSO-d_6): δ 166.71, 138.97, 138.18, 134.33, 132.47, 130.60, 129.08, 126.89, 126.15, 118.77, 21.60; IR (KBr): 1660, 1225, 1095, 745cm^{-1}; Anal. Calcd for [$C_{15}H_{15}NOSe$]: C, 59.22; H, 4.97; N, 4.60, found: C, 59.23; H, 4.96; N, 4.61%.

2-hydroseleno-*N*-(*p*-tolyl)benzamide (6b). White Solid (22 mg, 75%); Mp:125^{o}C; ^{1}H-NMR (300 MHz; DMSO-d_6): δ 10.49 (s, 1H), 8.00 – 7.90 (m, 1H), 7.78 (d, *J* = 7.5 Hz, 1H), 7.66 (d, *J* = 8.3 Hz, 2H), 7.44 (tt, *J* = 13.1, 6.6 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 2.30 (s, 3H); ^{13}C-NMR (75 MHz; DMSO-d_6): δ 166.63, 136.60, 134.29, 133.66, 132.45, 130.63, 129.59, 129.04, 126.88, 121.06, 21.01; IR (KBr):1665, 1230, 1105, 750cm^{-1} ; HRMS (ESI-TOF) m/z Calcd for [$C_{14}H_{13}NOSe+H$]$^{+}$: 292.0235, found: 292.0235.

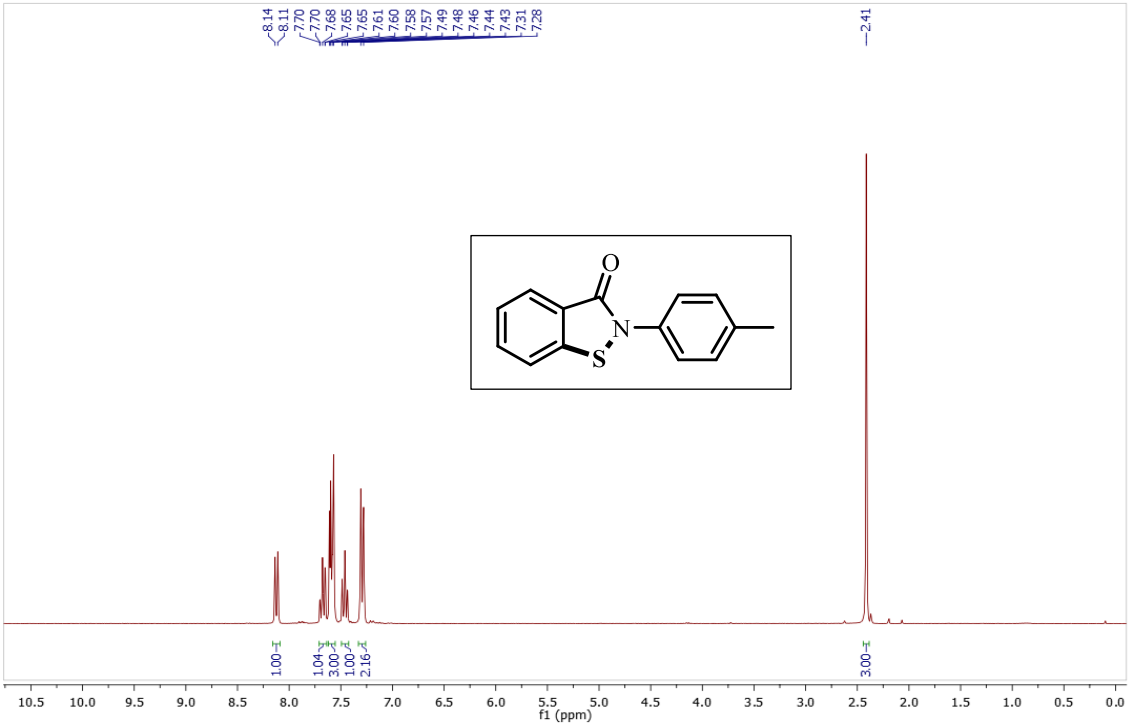
***N*-(4-fluorophenyl)-2-hydroselenobenzamide (6c).** White Solid (21 mg, 76%); Mp:122^{o}C; ^{1}H-

NMR (300 MHz DMSO-d_6): δ 10.63 (s, 1H), 7.96 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.85 – 7.74 (m, 3H), 7.45 (dq, *J* = 7.3, 5.9 Hz, 2H), 7.24 (t, *J* = 8.9 Hz, 2H).; ^{13}C-NMR (75 MHz; DMSO-d_6): δ 166.72, 160.57, 157.44, 135.47, 134.04, 132.59, 132.46, 130.66, 129.10, 126.94, 122.94 (d, $^{3}J_{C\text{-}F}$ =8.0 Hz), 115.83(d, $^{2}J_{C\text{-}F}$=22.3 Hz); IR (KBr): 1668, 1255, 1100, 742cm^{-1}; Anal. Calcd for [$C_{13}H_{10}FNOSe$]: C, 53.08; H, 3.43; N, 4.76, found: C, 53.10; H, 3.41; N, 4.77%.

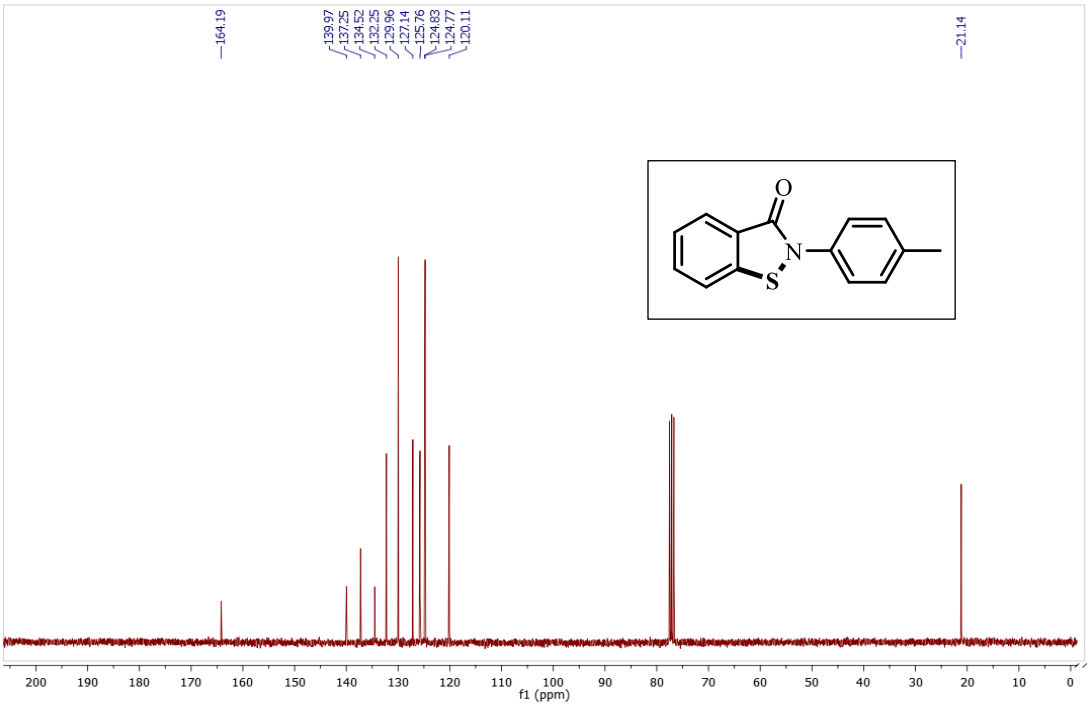
Reference:

1. (a) K. Sukata, *Bull. Chem. Soc. Jpn.* **1985**, *58*, 838. (b) K. Dev, R. Maurya, *RSC Adv.* **2015**, *5*, 13102.

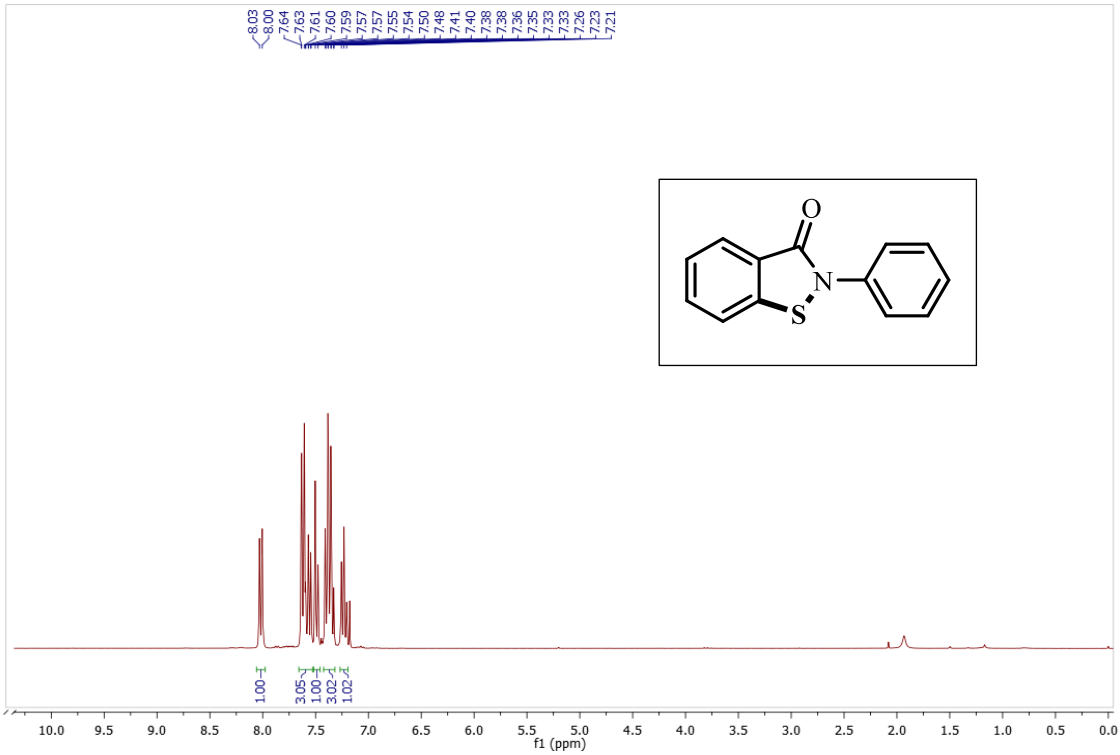
^{1}H-NMR of Compound 2a



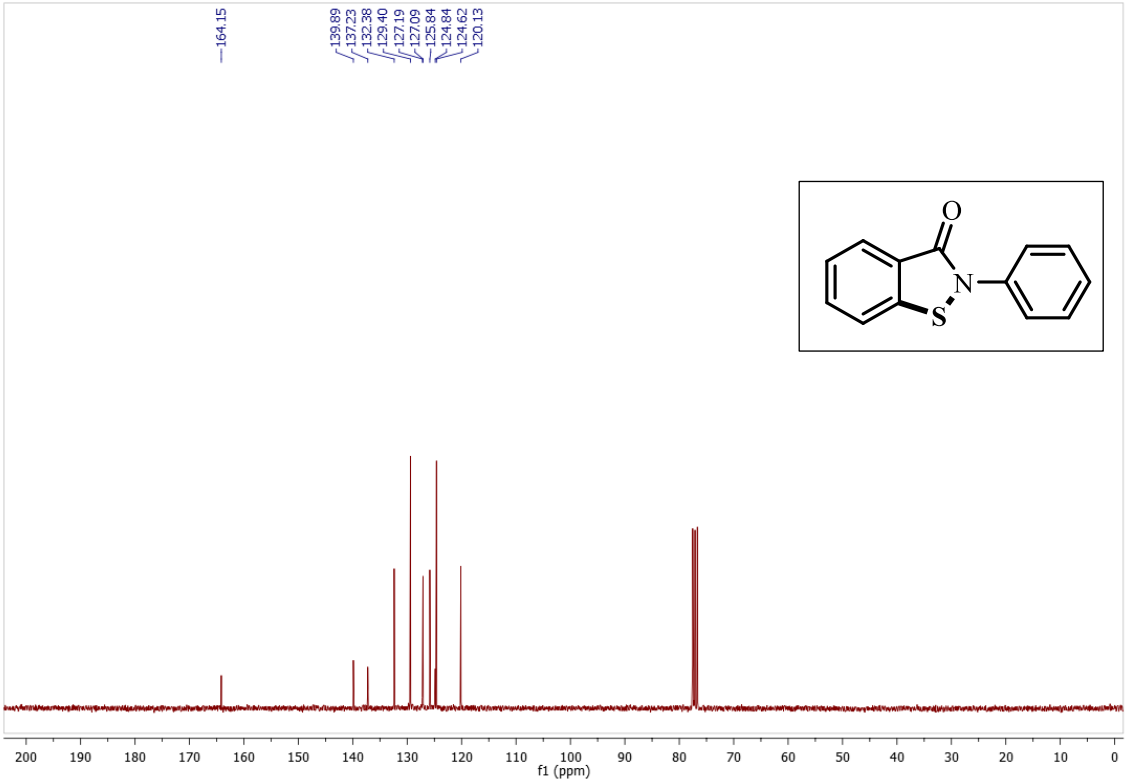
^{13}C-NMR of Compound 2a



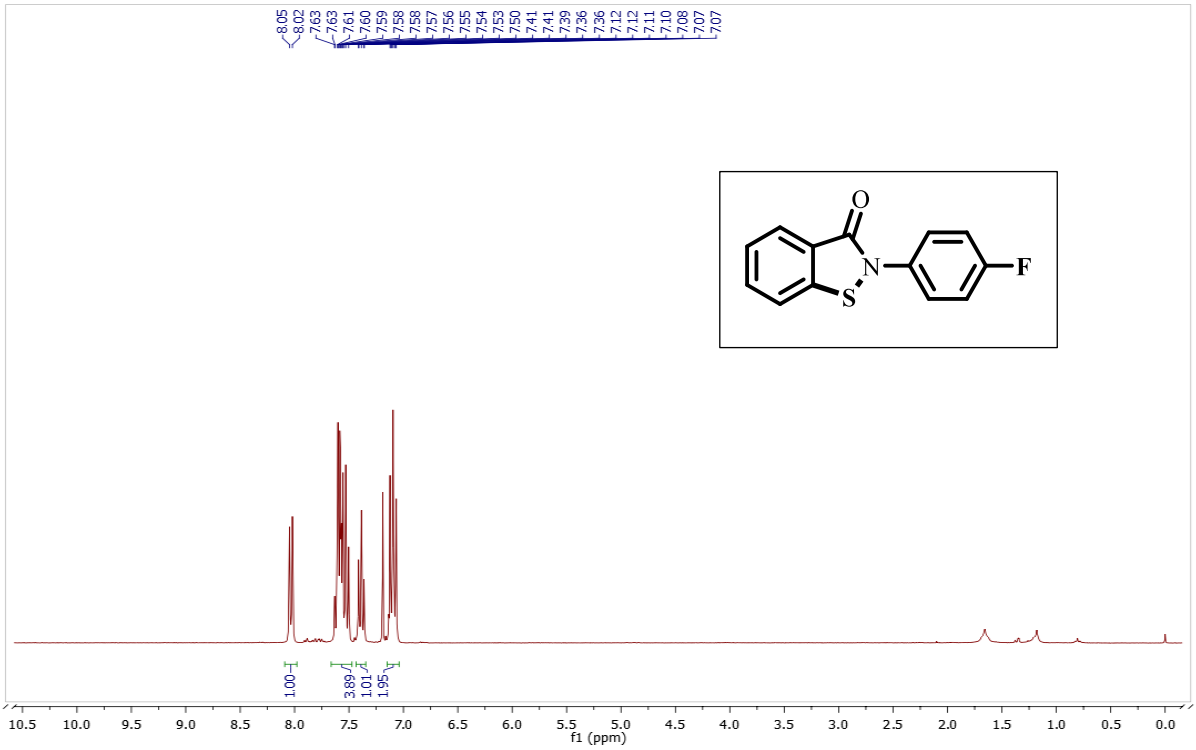
^{1}H-NMR of Compound 2b



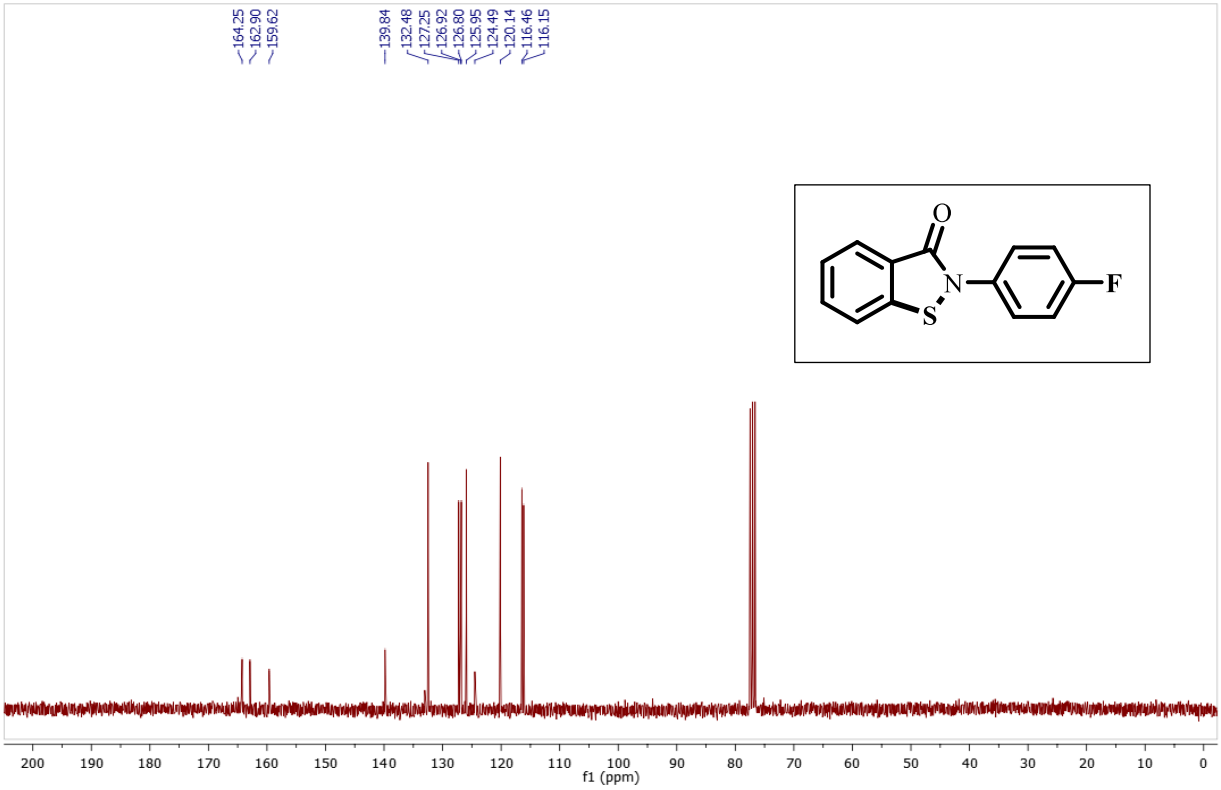
^{13}C-NMR of Compound 2b



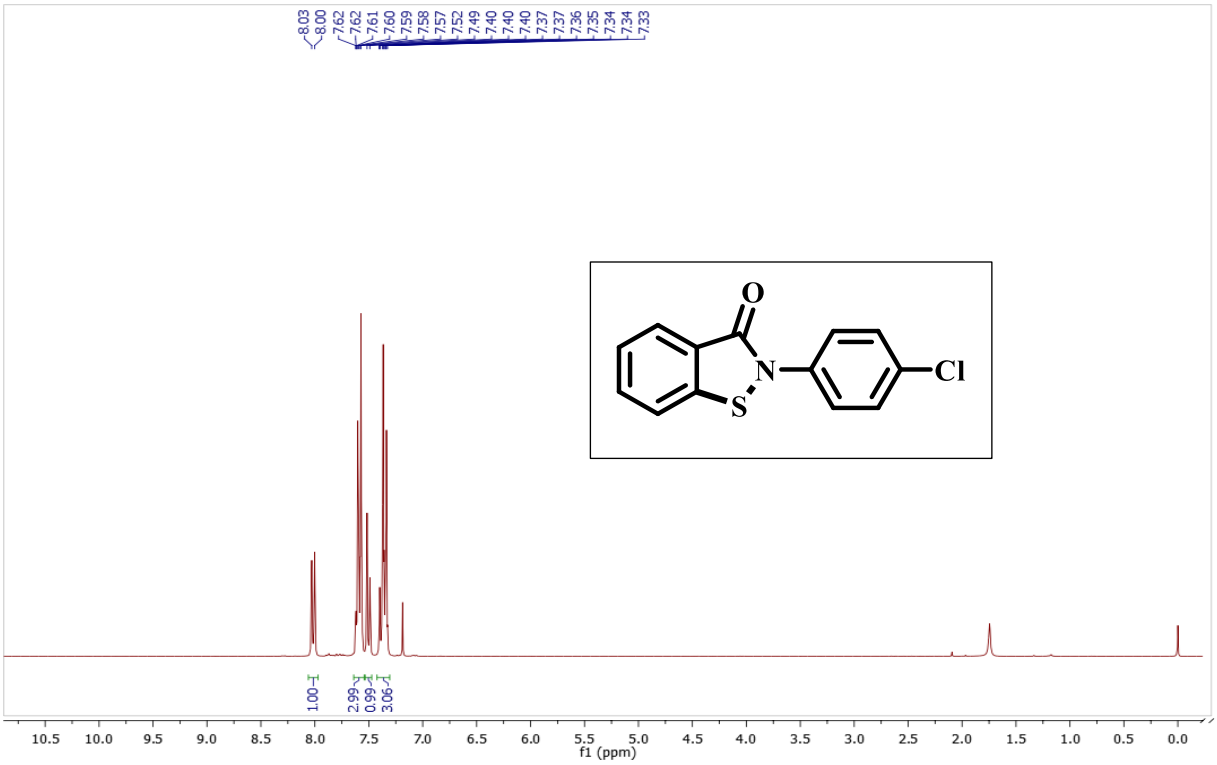
^{1}H-NMR of Compound 2c



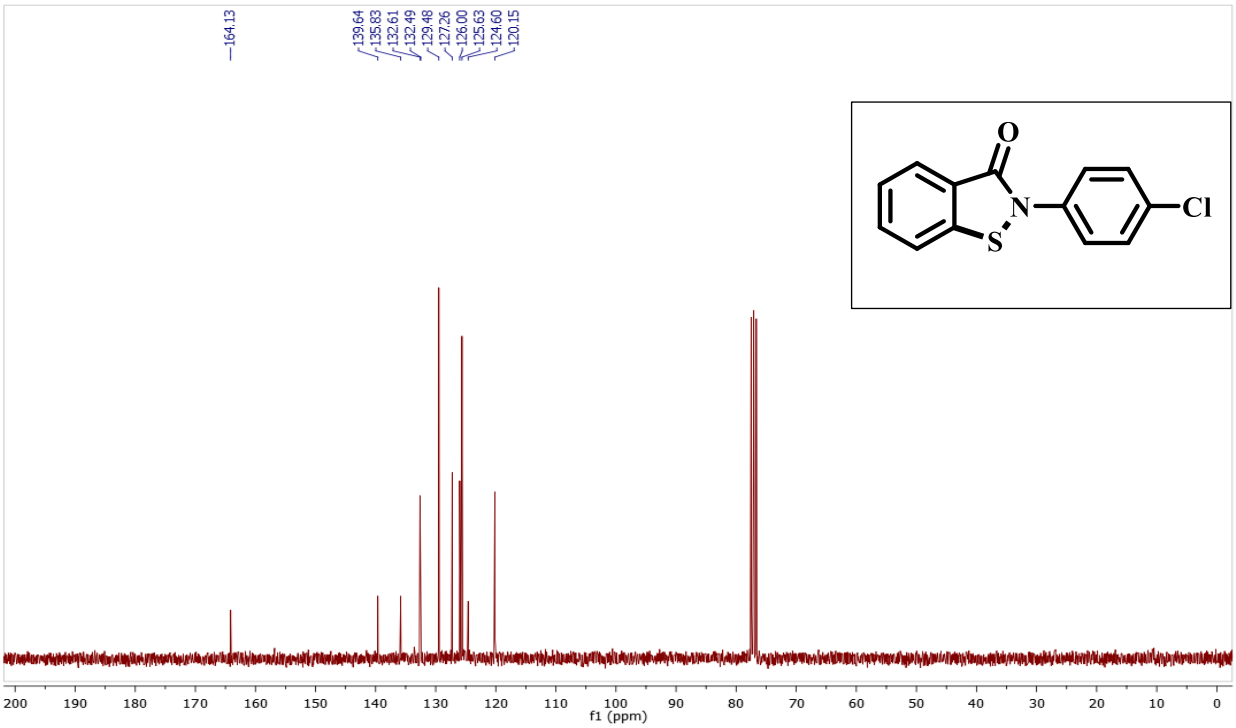
^{13}C-NMR of Compound 2c



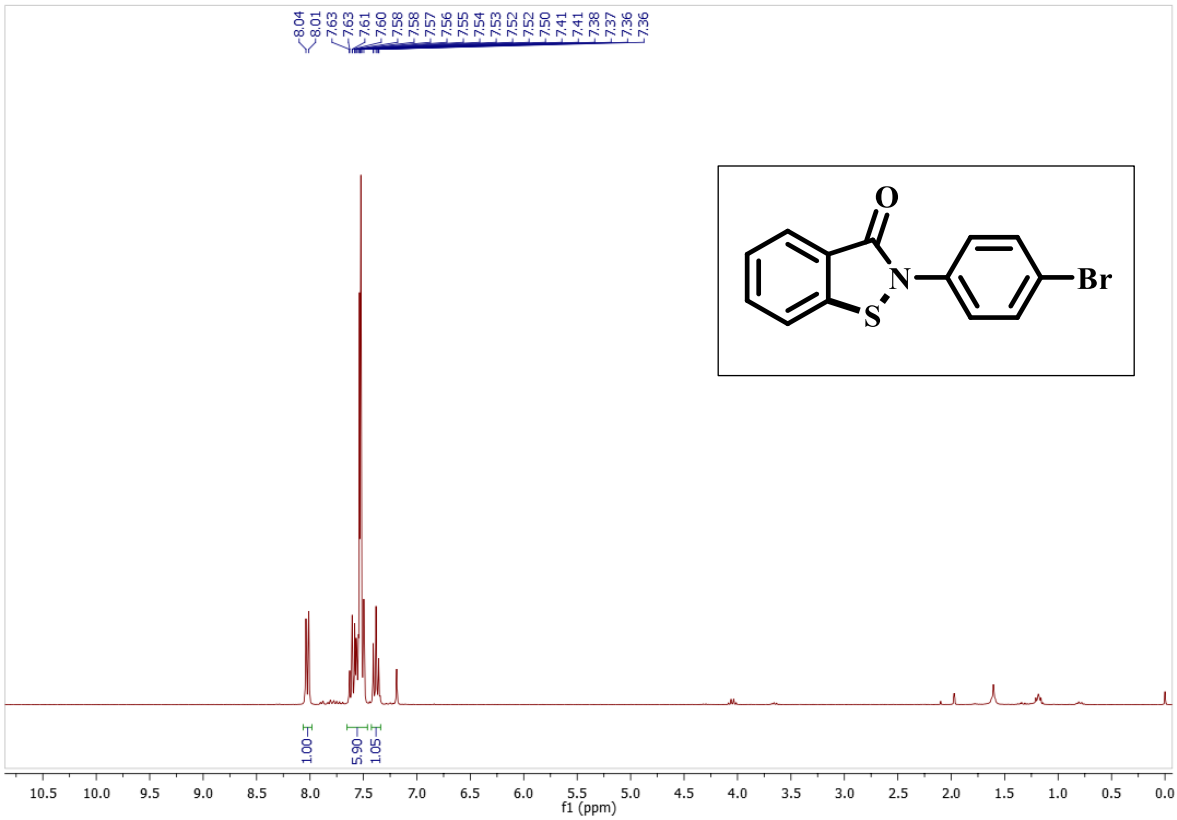
[1]H-NMR of Compound 2d



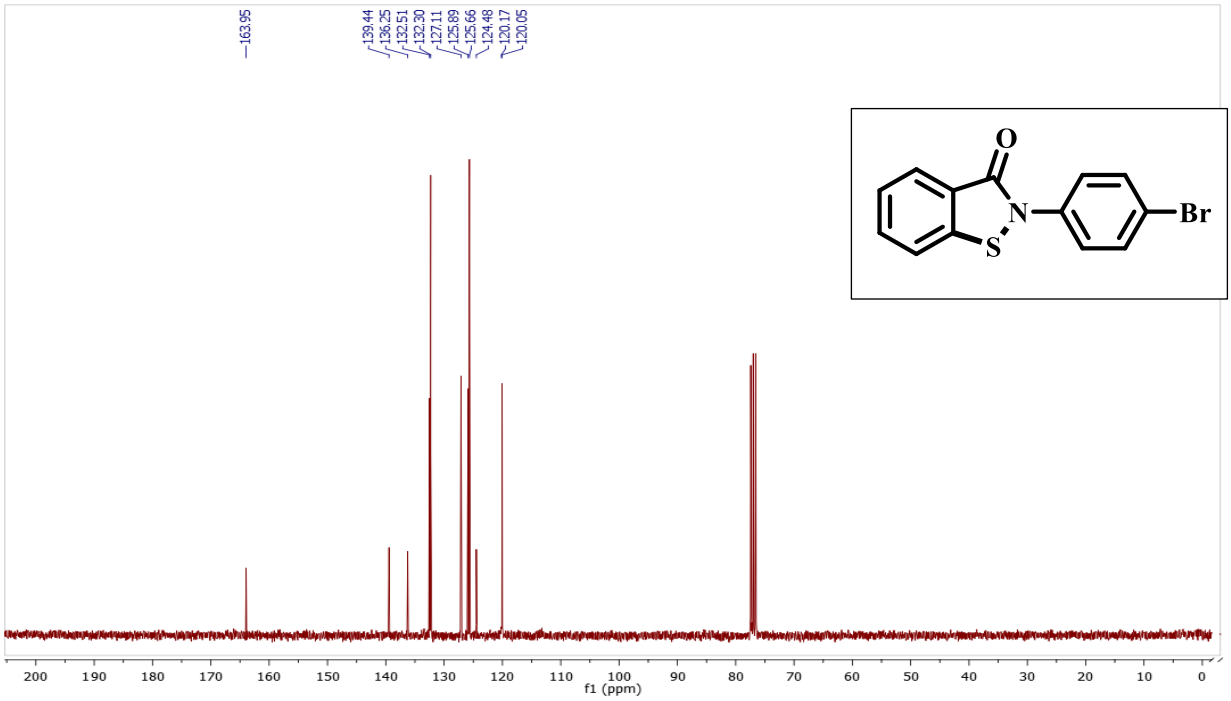
[13]C-NMR of Compound 2d



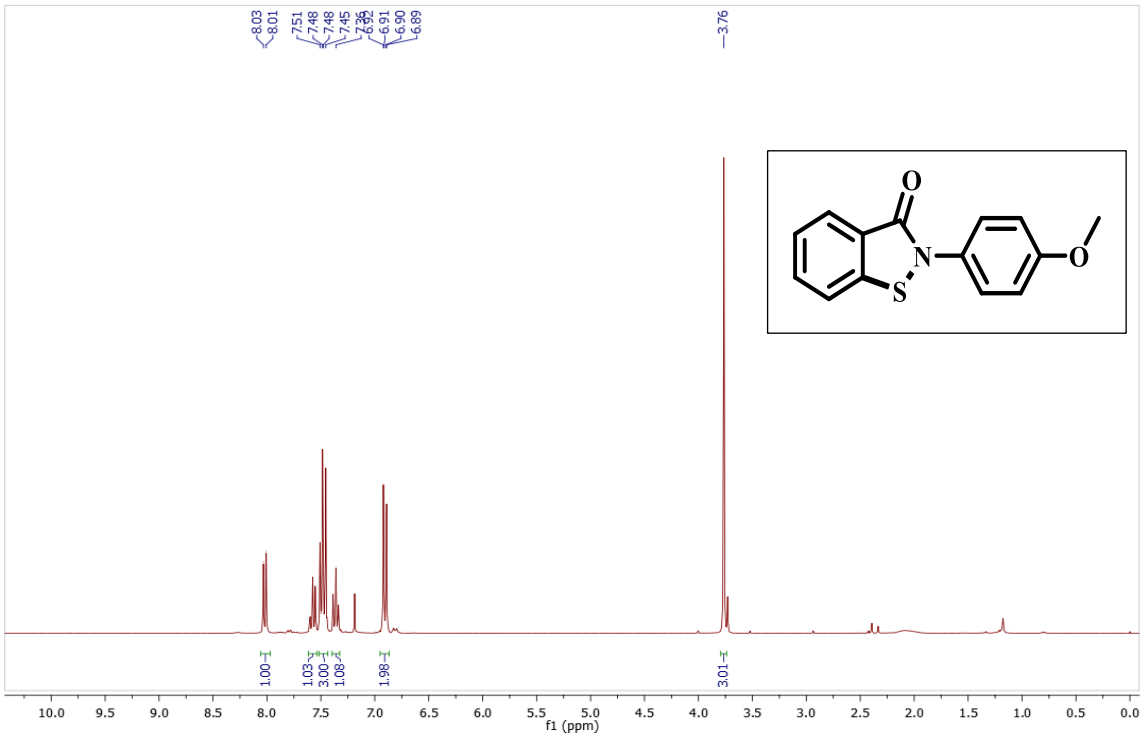
^{1}H-NMR of Compound 2e



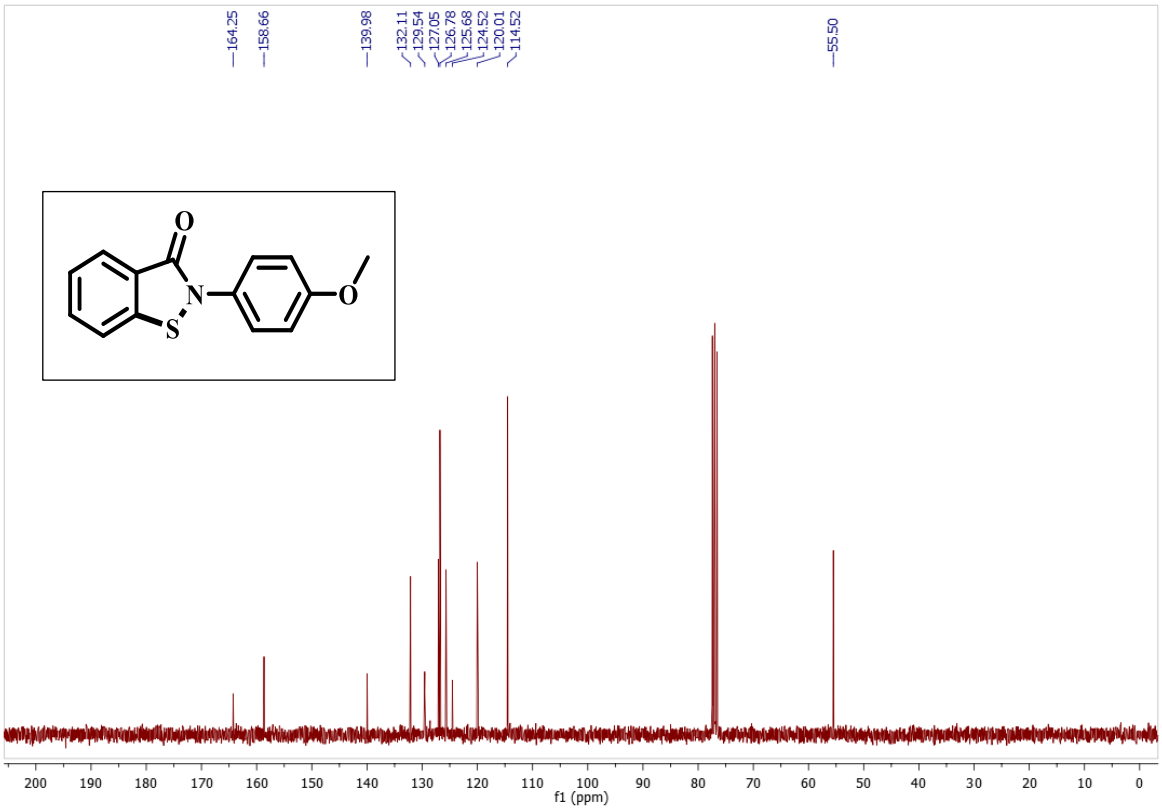
^{13}C-NMR of Compound 2e



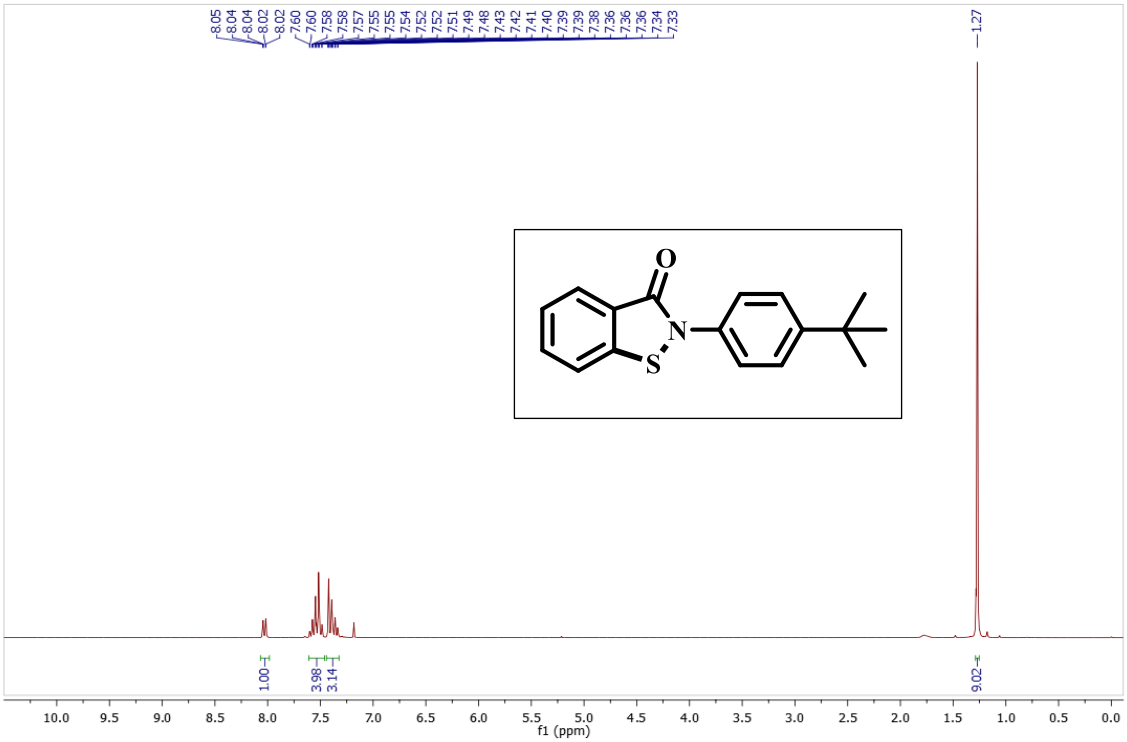
^{1}H-NMR of Compound 2f



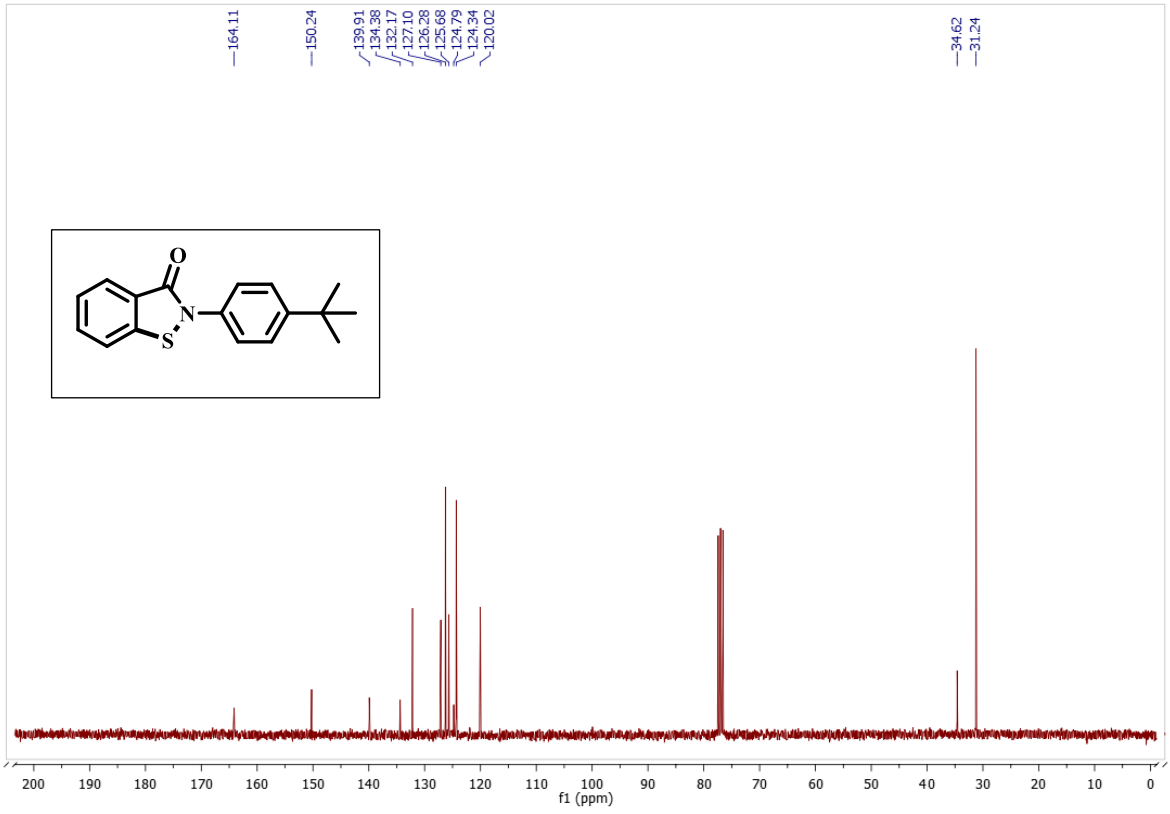
^{13}C-NMR of Compound 2f



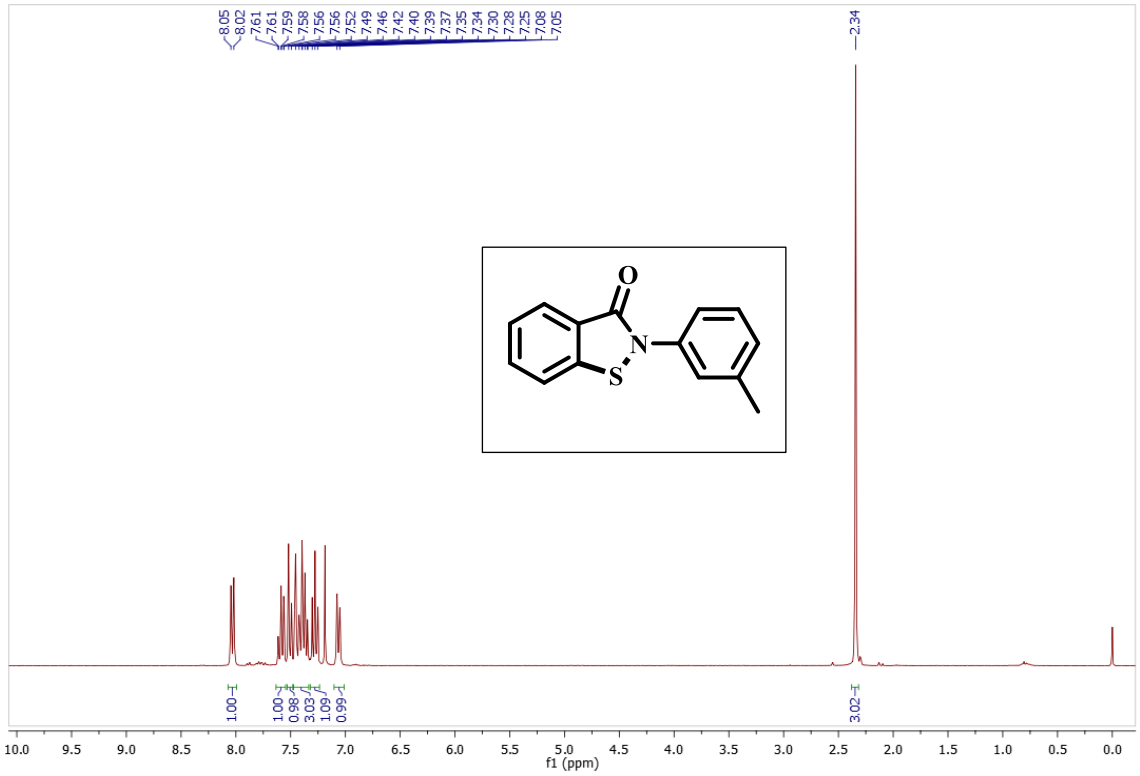
^{1}H-NMR of Compound 2g



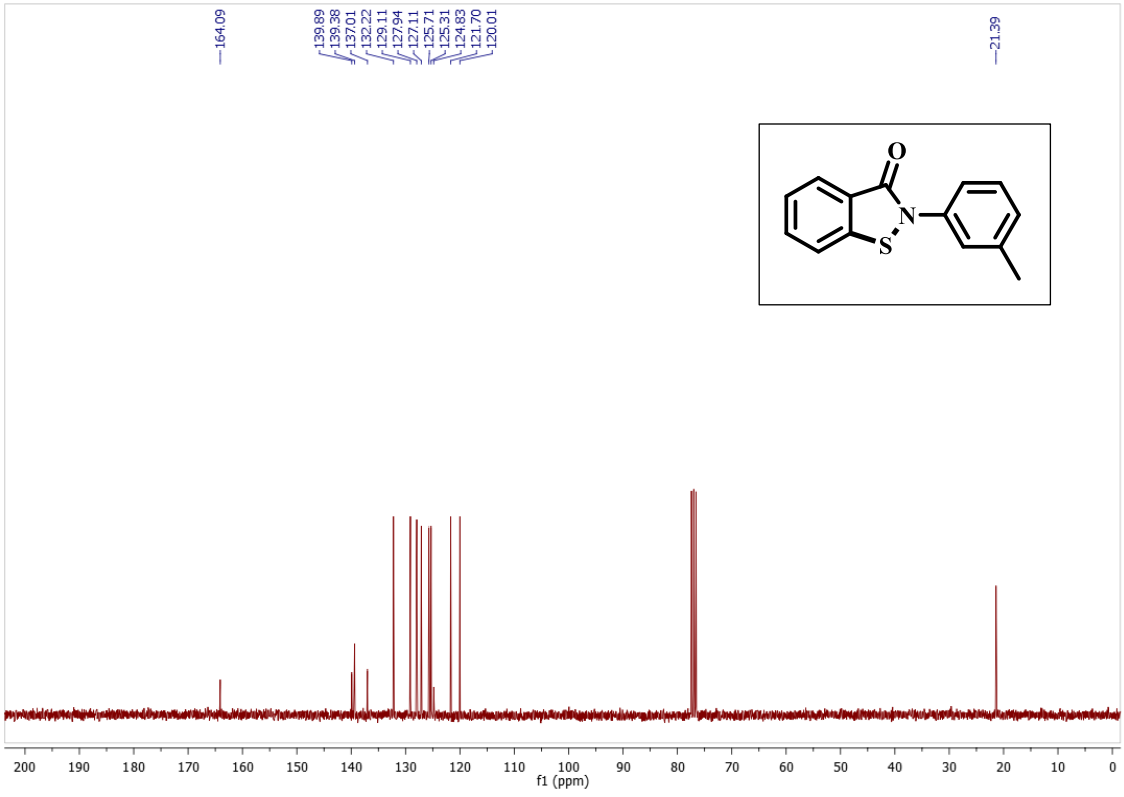
^{13}C-NMR of Compound 2g



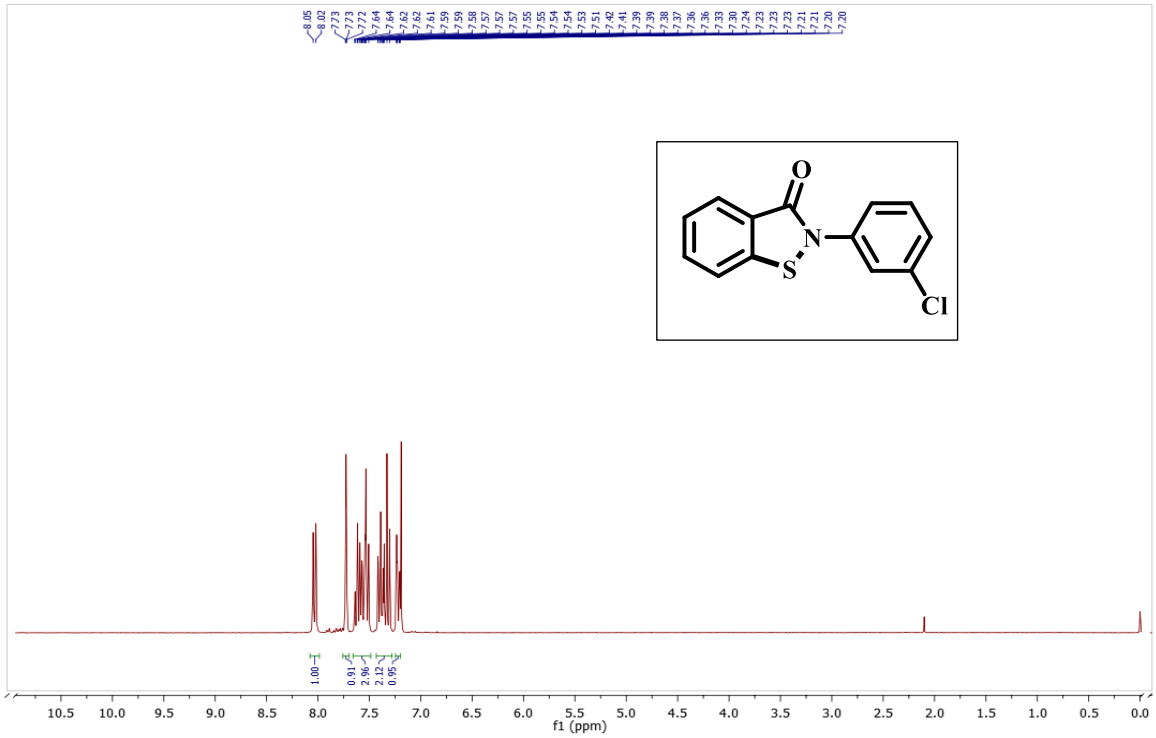
^{1}H-NMR of Compound 2h



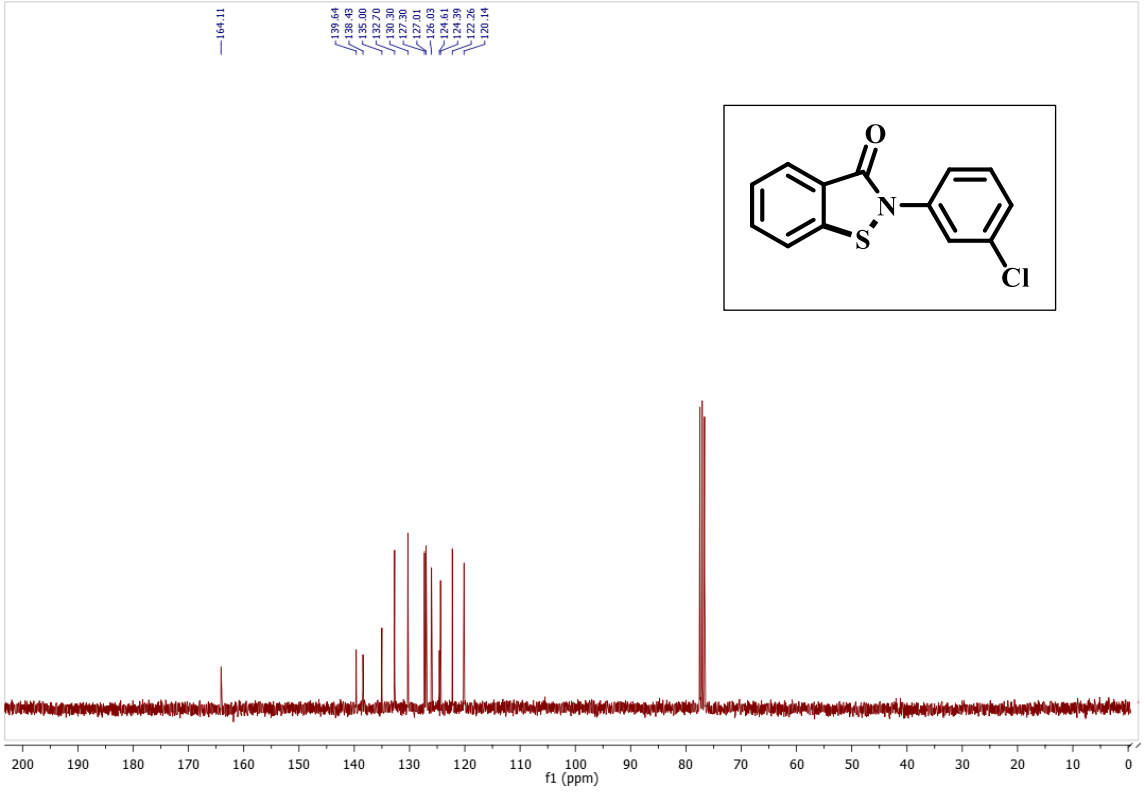
^{13}C-NMR of Compound 2h



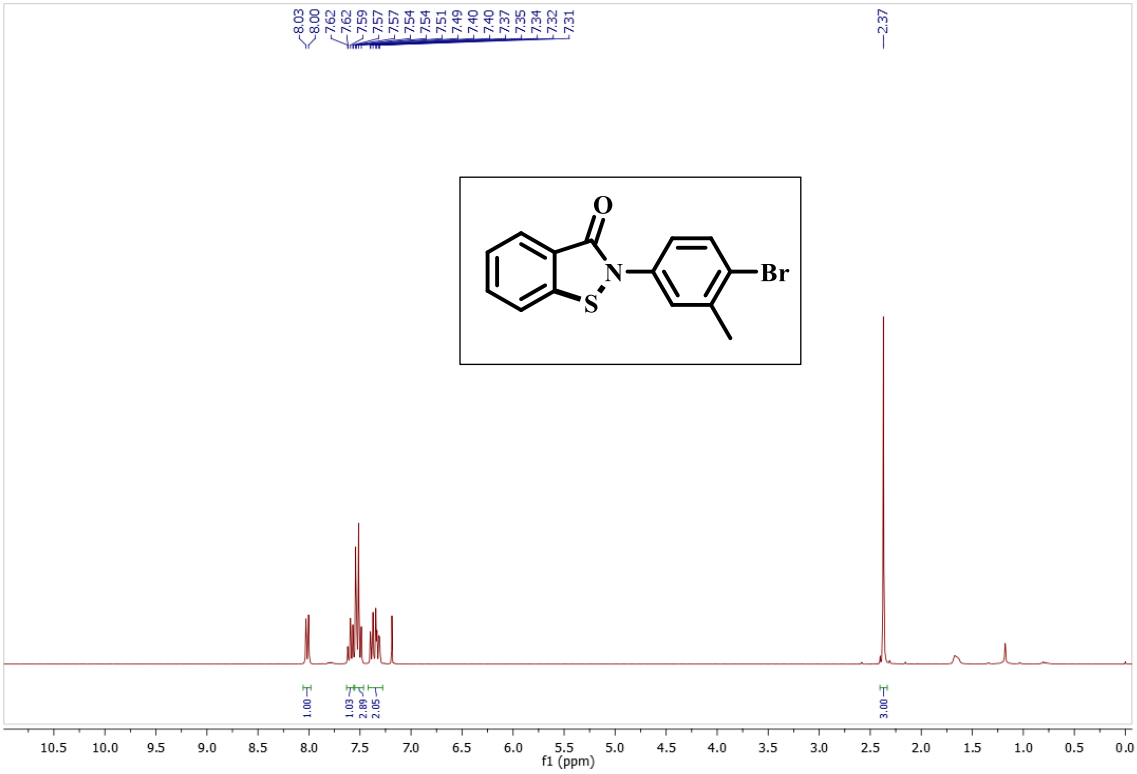
^{1}H-NMR of Compound 2i



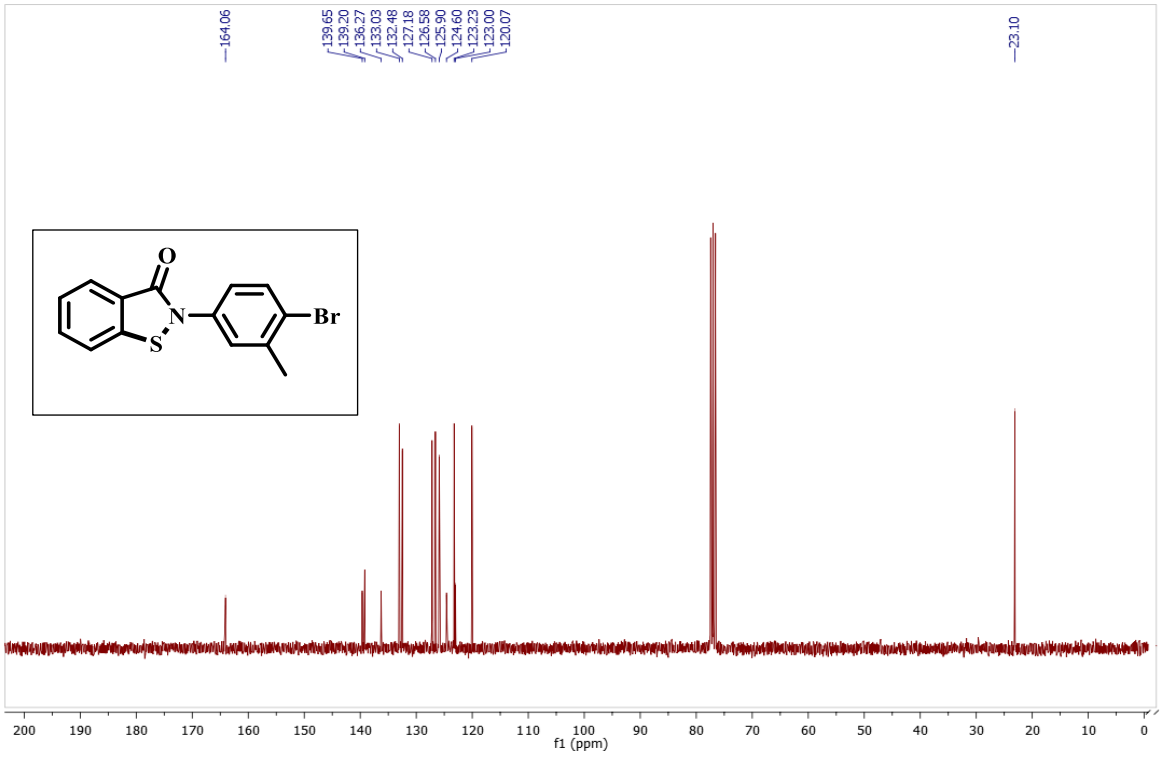
^{13}C-NMR of Compound 2i



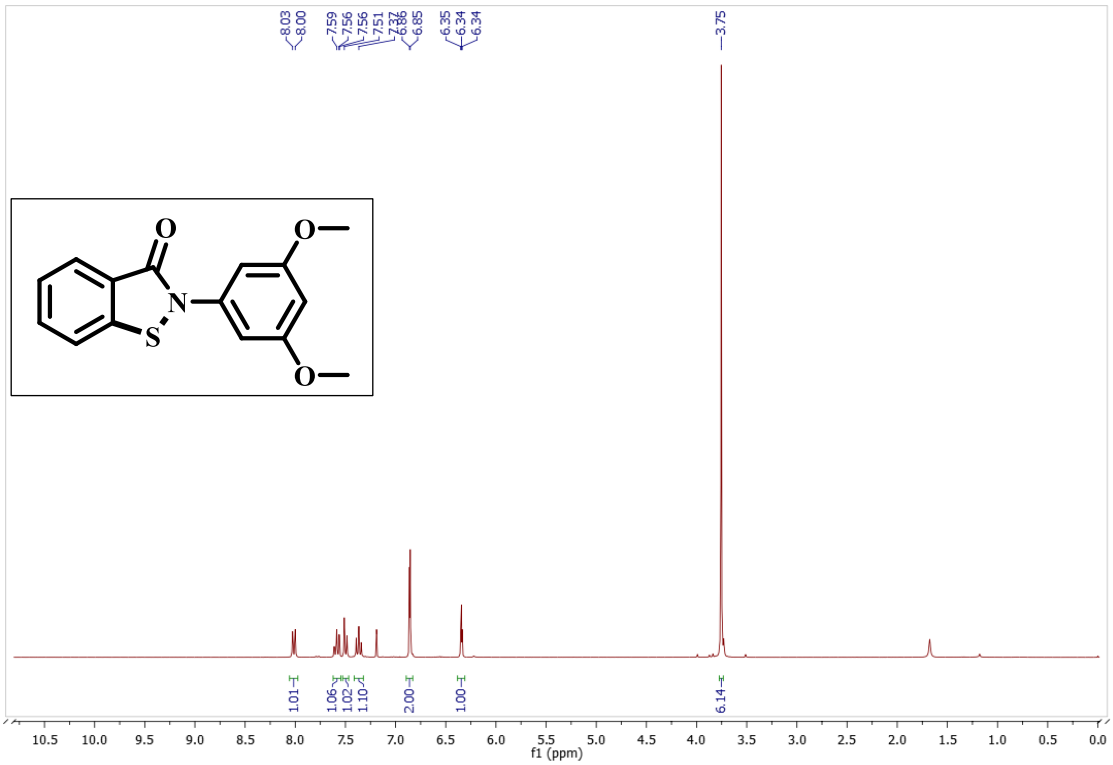
^{1}H-NMR of Compound 2j



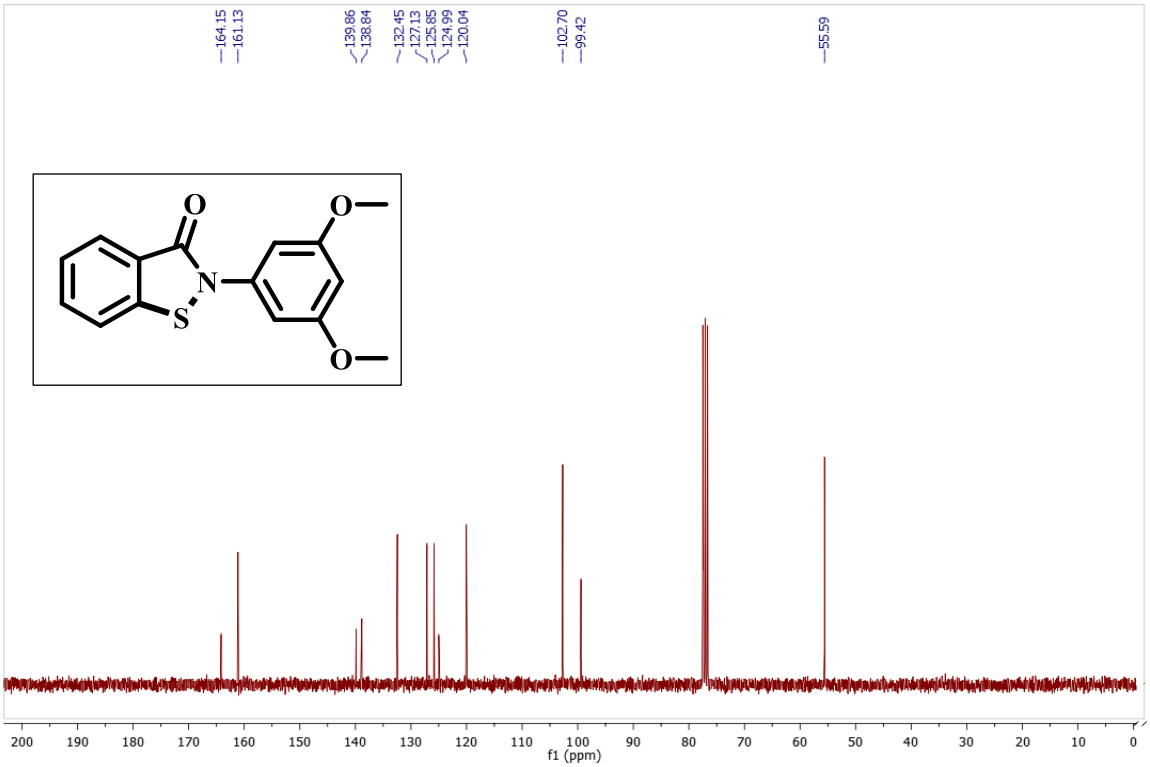
^{13}C-NMR of Compound 2j



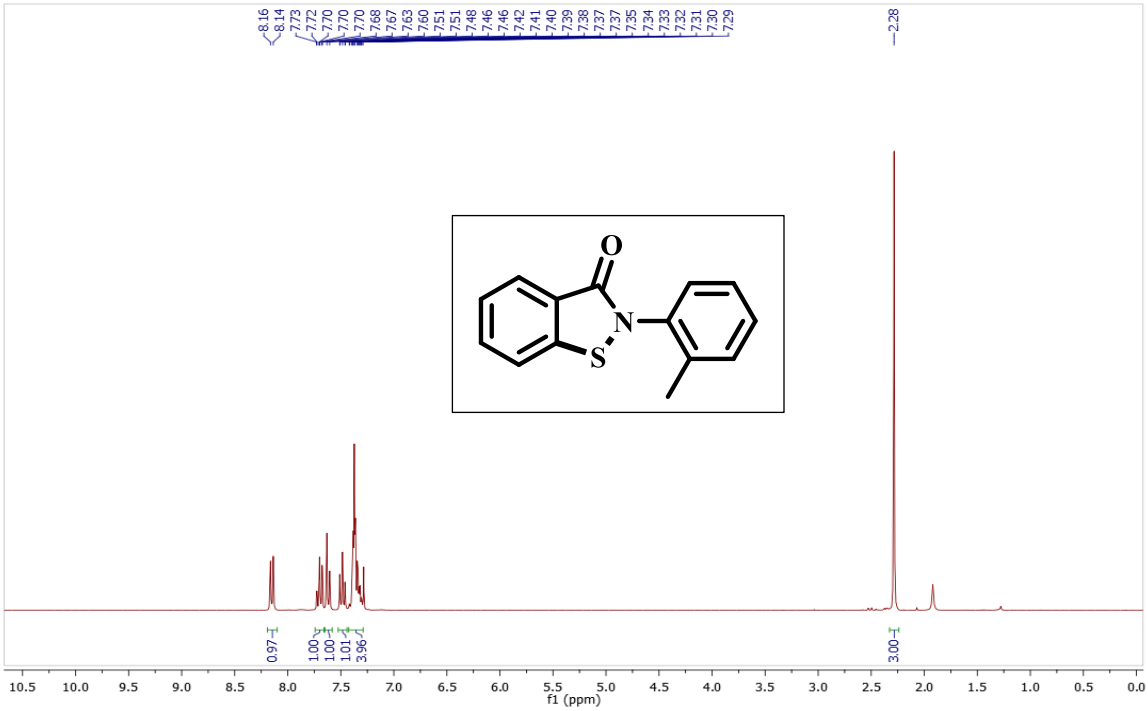
^{1}H-NMR of Compound 2k



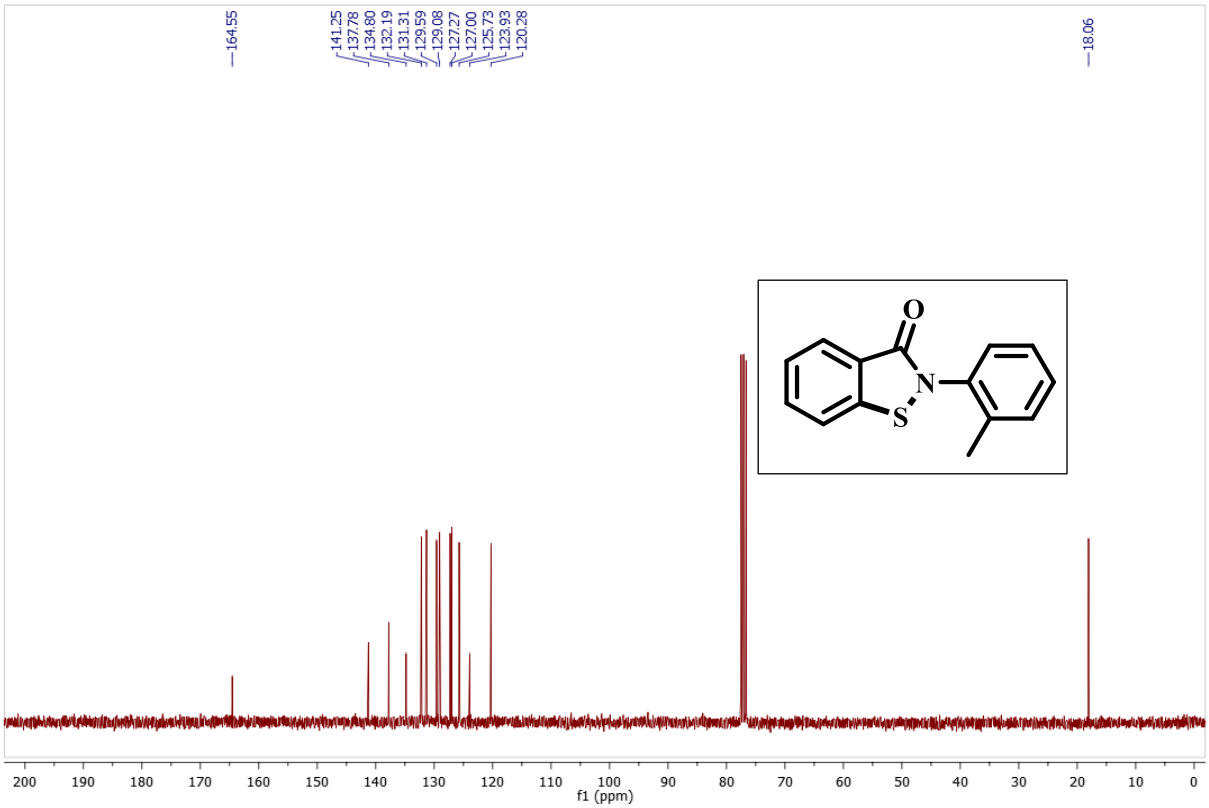
^{13}C-NMR of Compound 2k



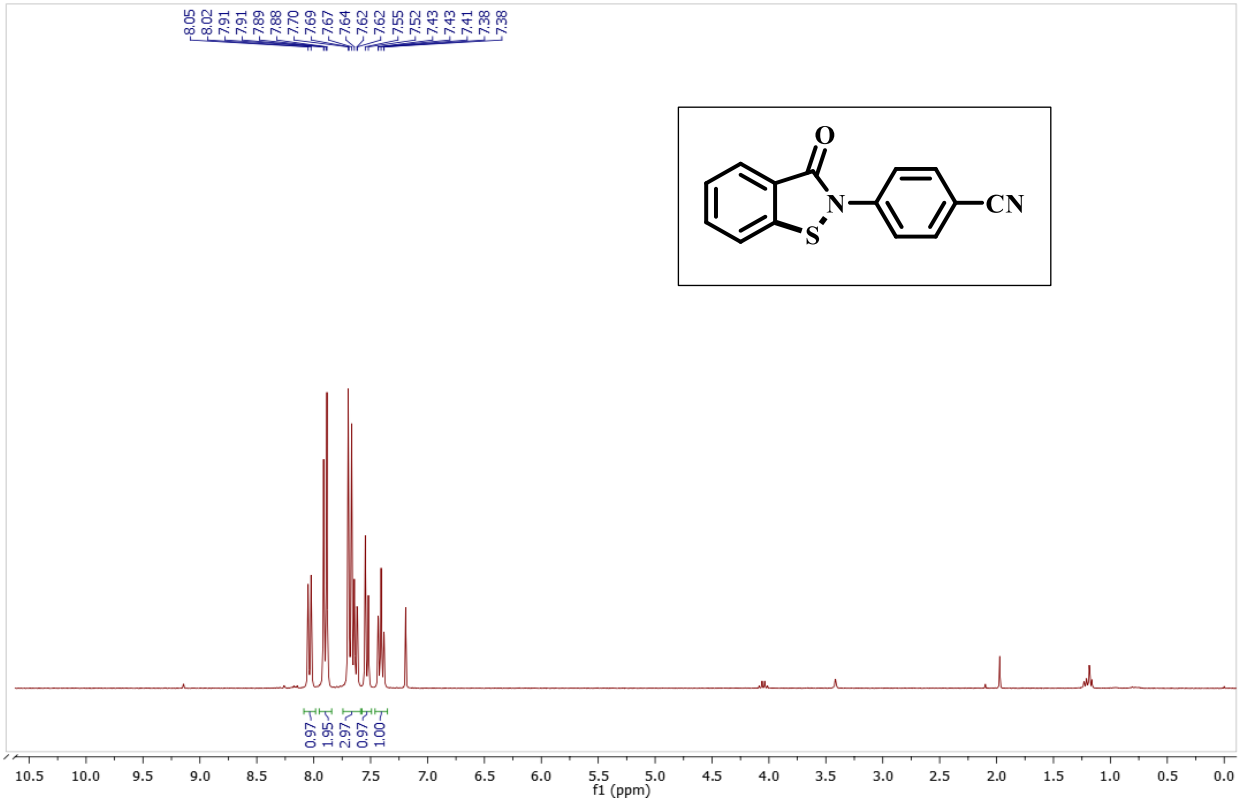
^{1}H-NMR of Compound 2l



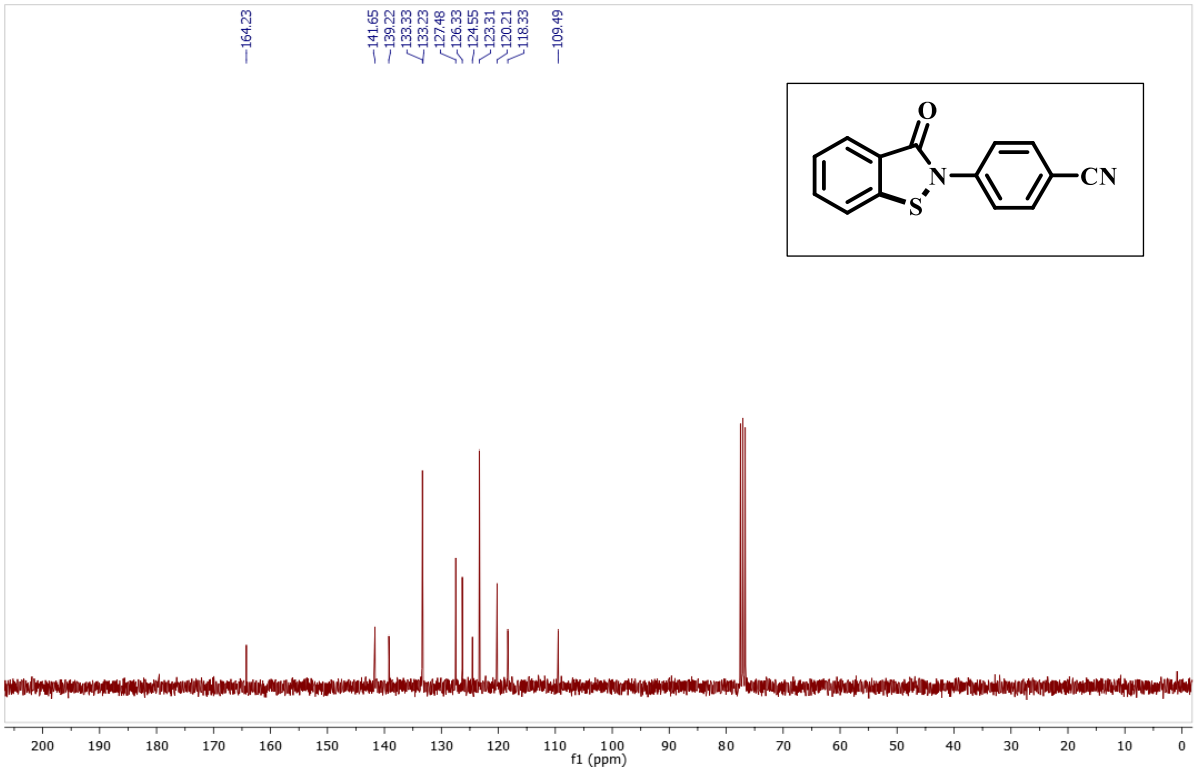
^{13}C-NMR of Compound 2l



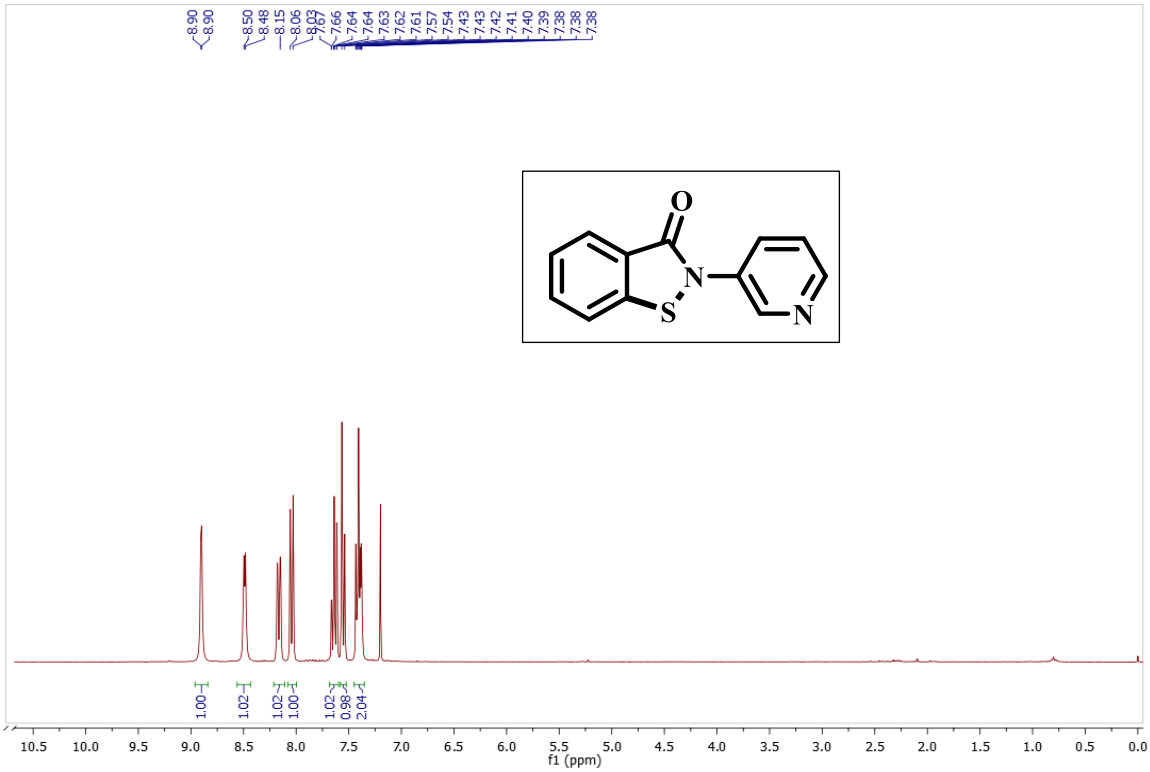
^{1}H-NMR of Compound 2m



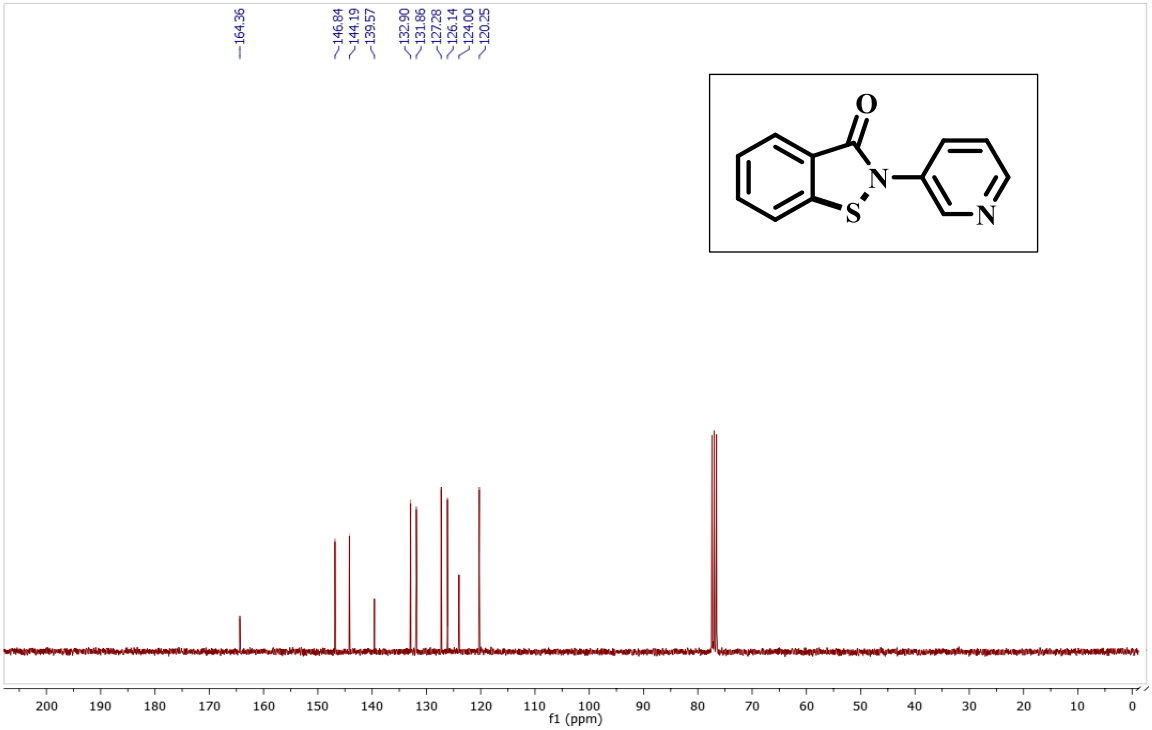
^{13}C-NMR of Compound 2m



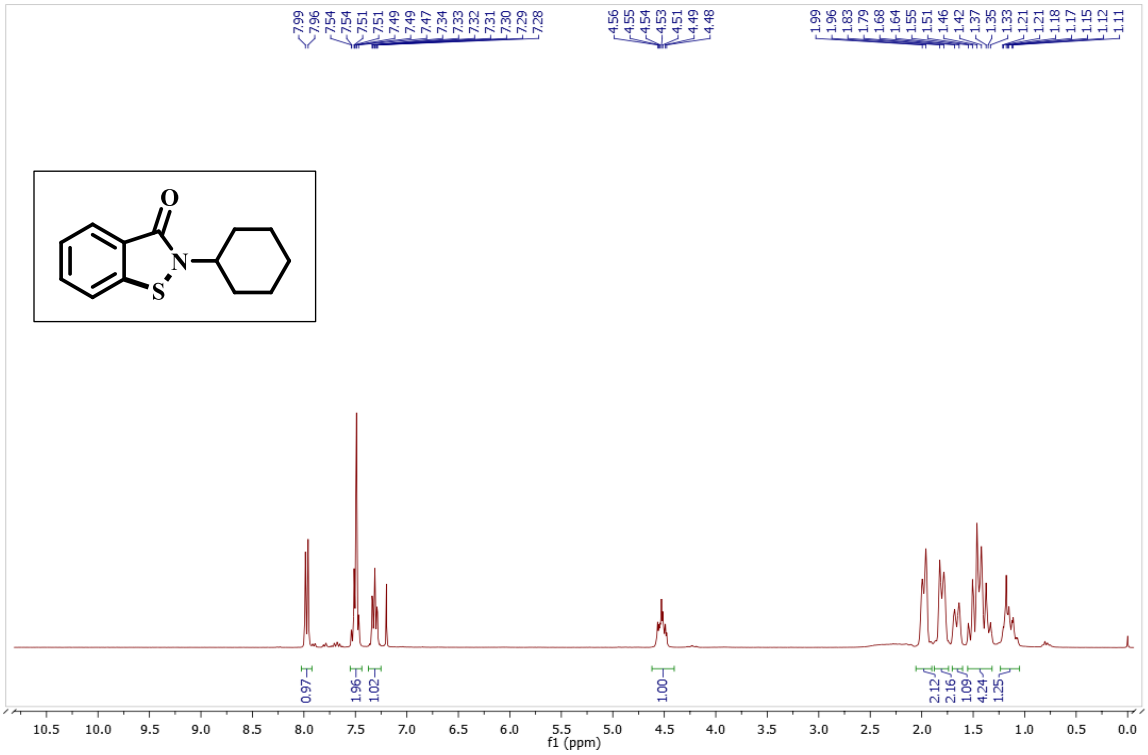
^{1}H-NMR of Compound 2n



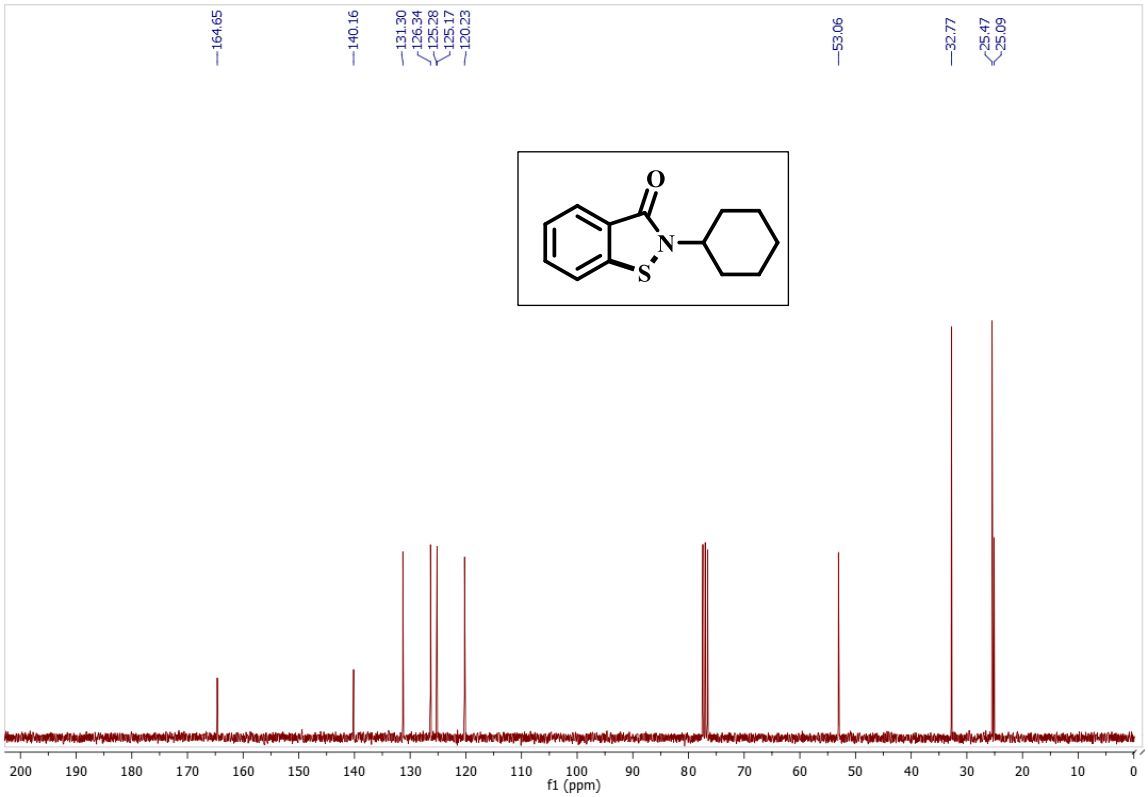
^{13}C-NMR of Compound 2n



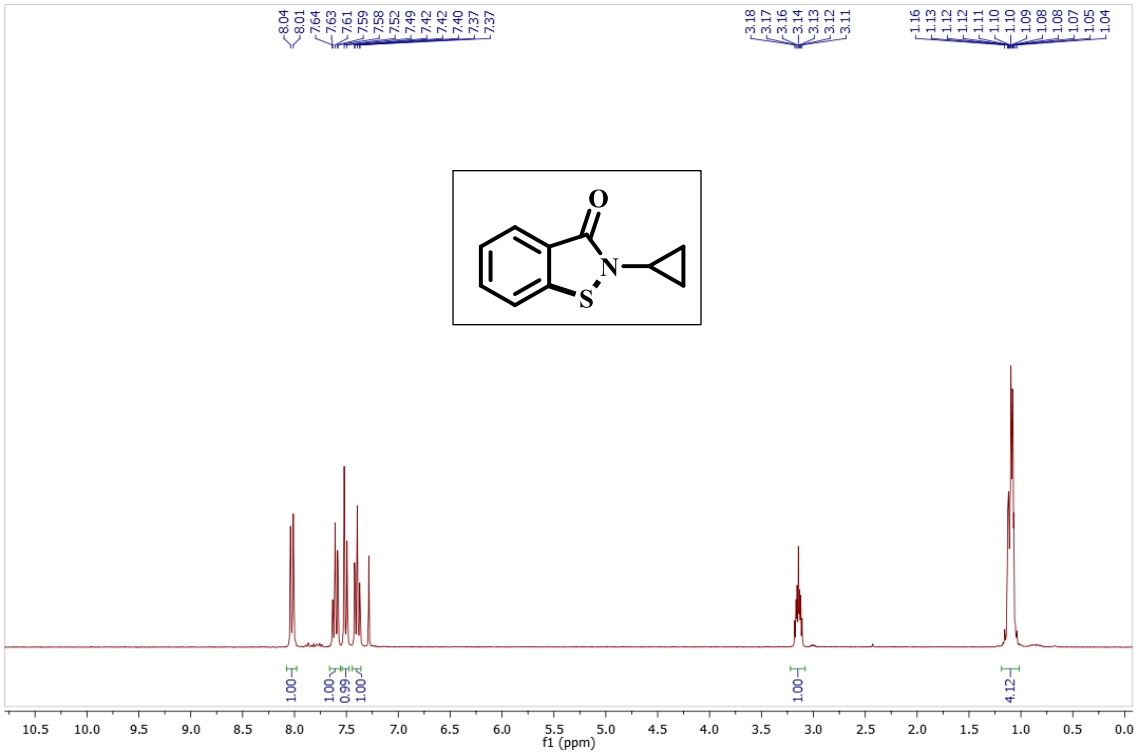
^{1}H-NMR of Compound 2o



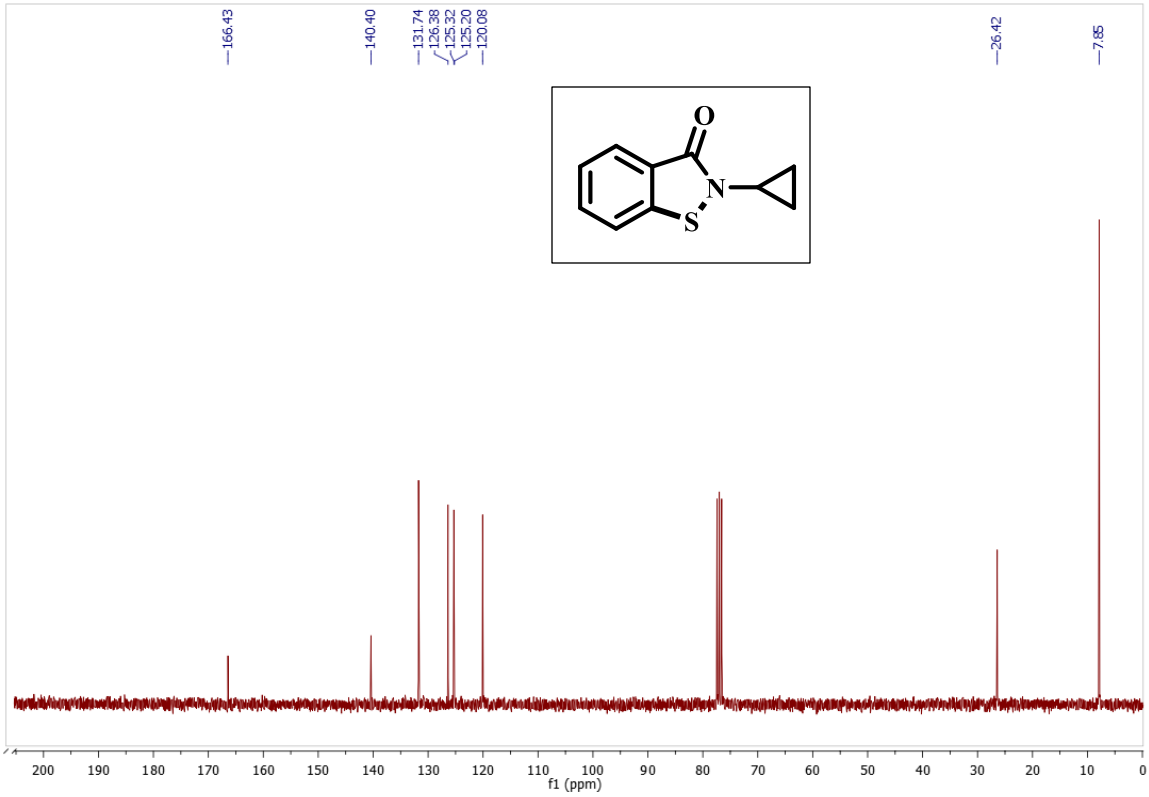
^{13}C-NMR of Compound 2o



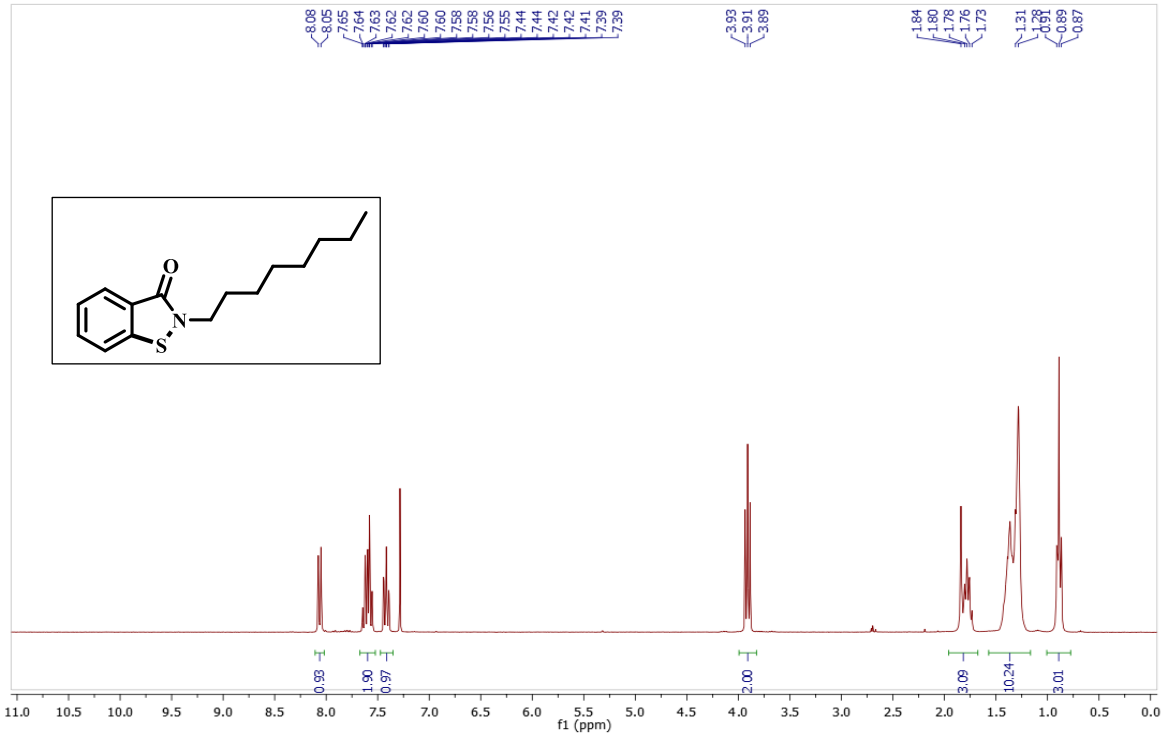
^{1}H-NMR of Compound 2p



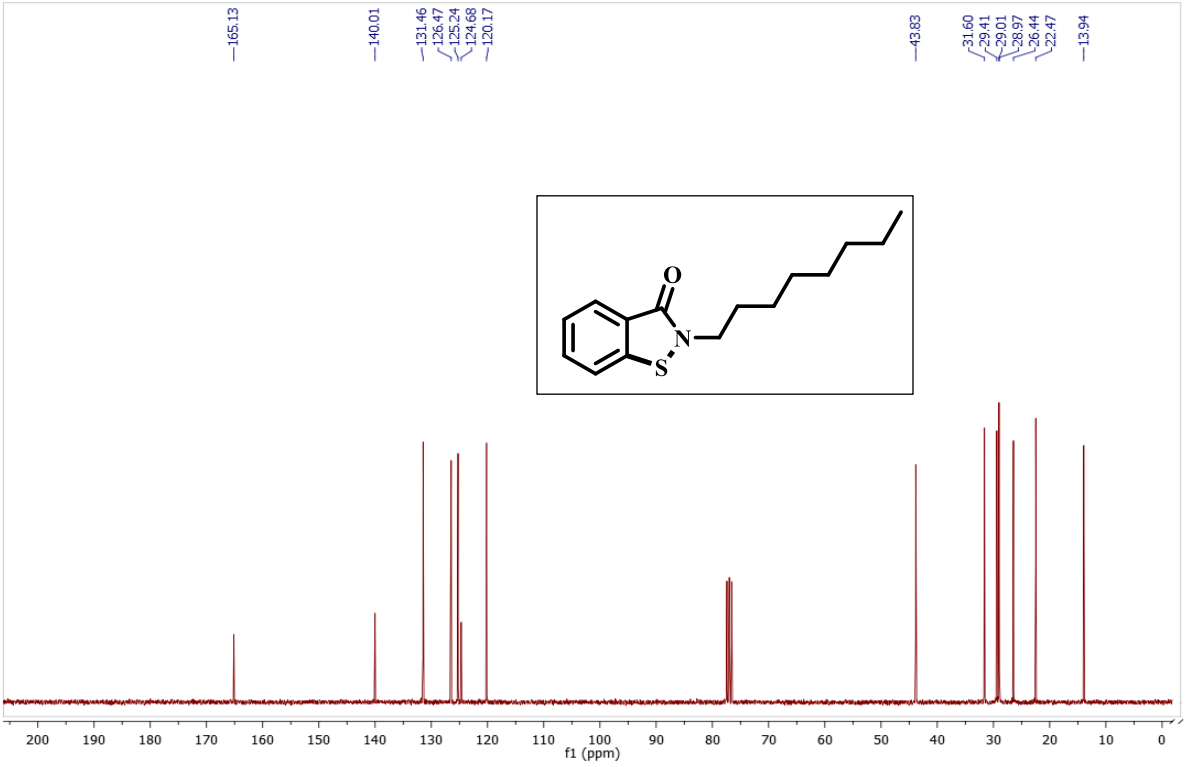
^{13}C-NMR of Compound 2p



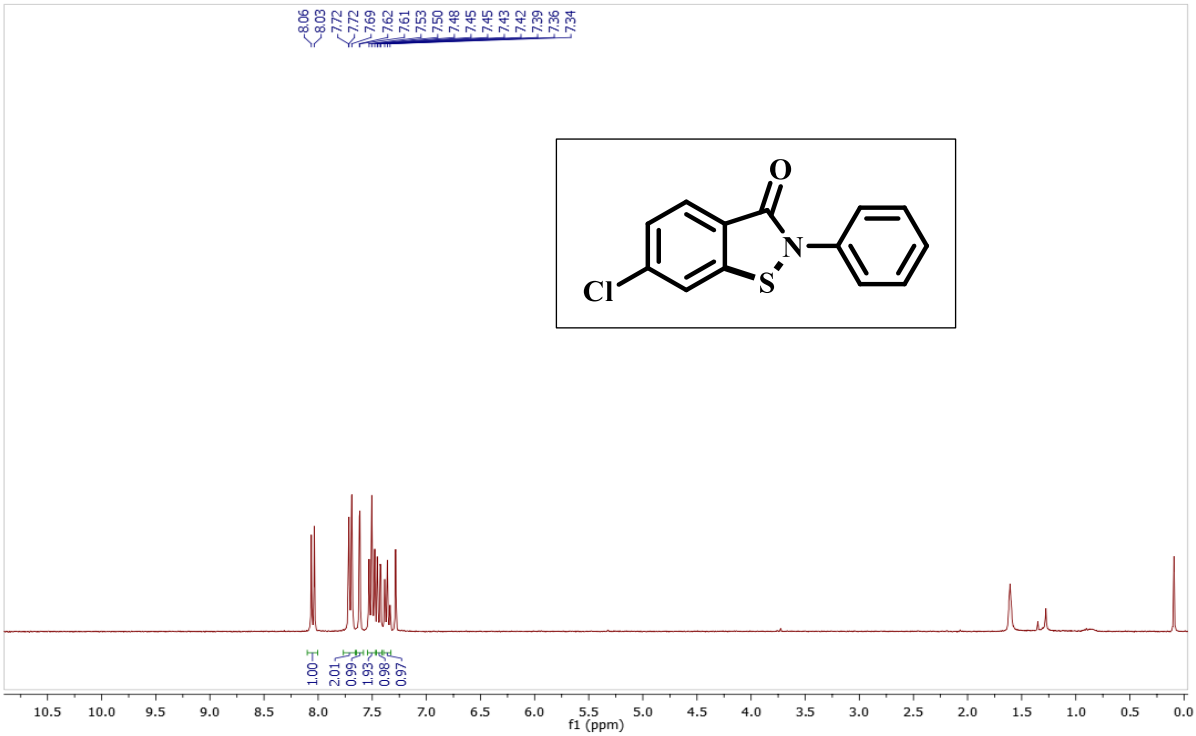
^{1}H-NMR of Compound 2q



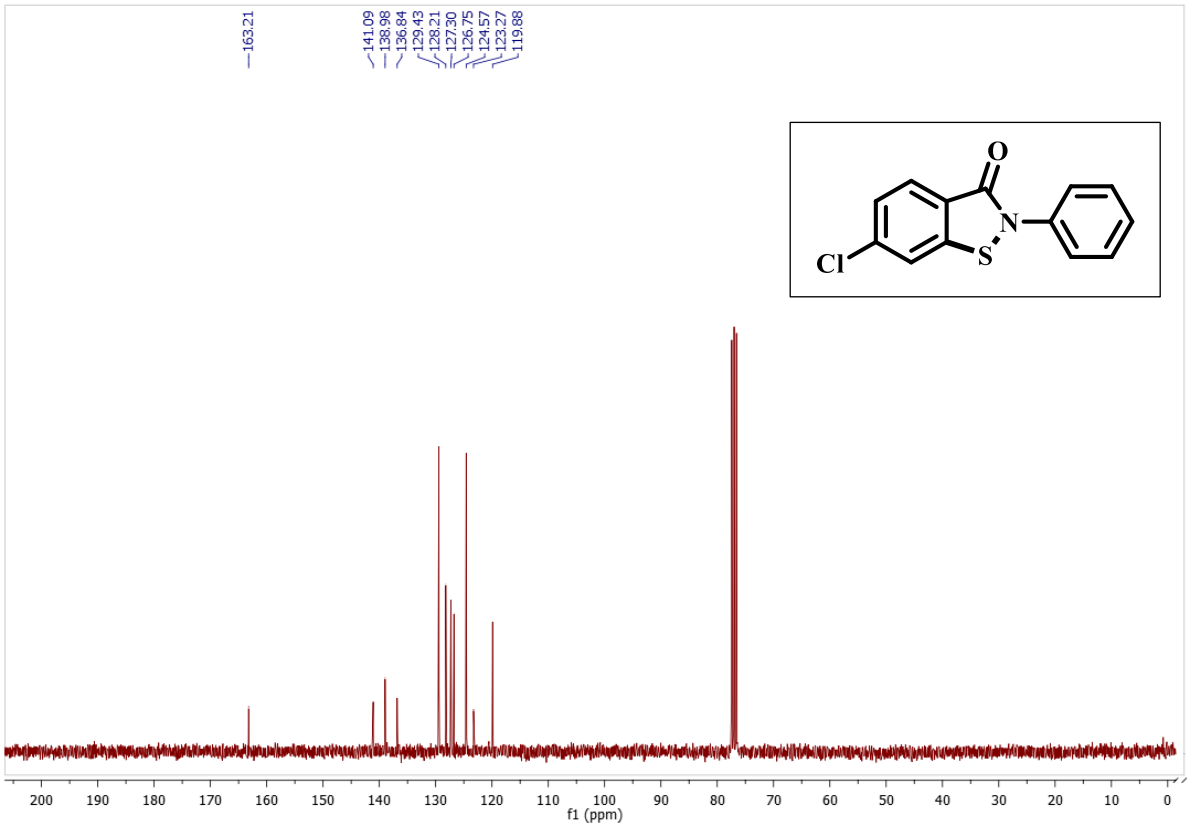
^{13}C-NMR of Compound 2q



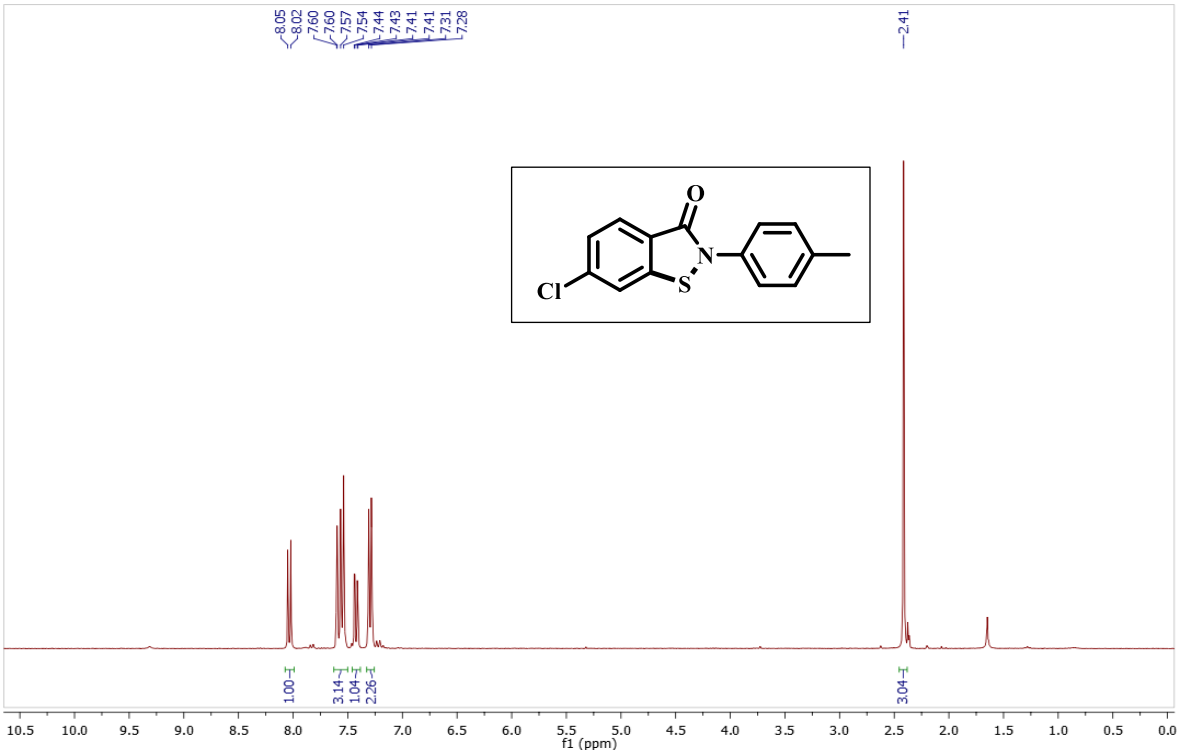
^{1}H-NMR of Compound 2r



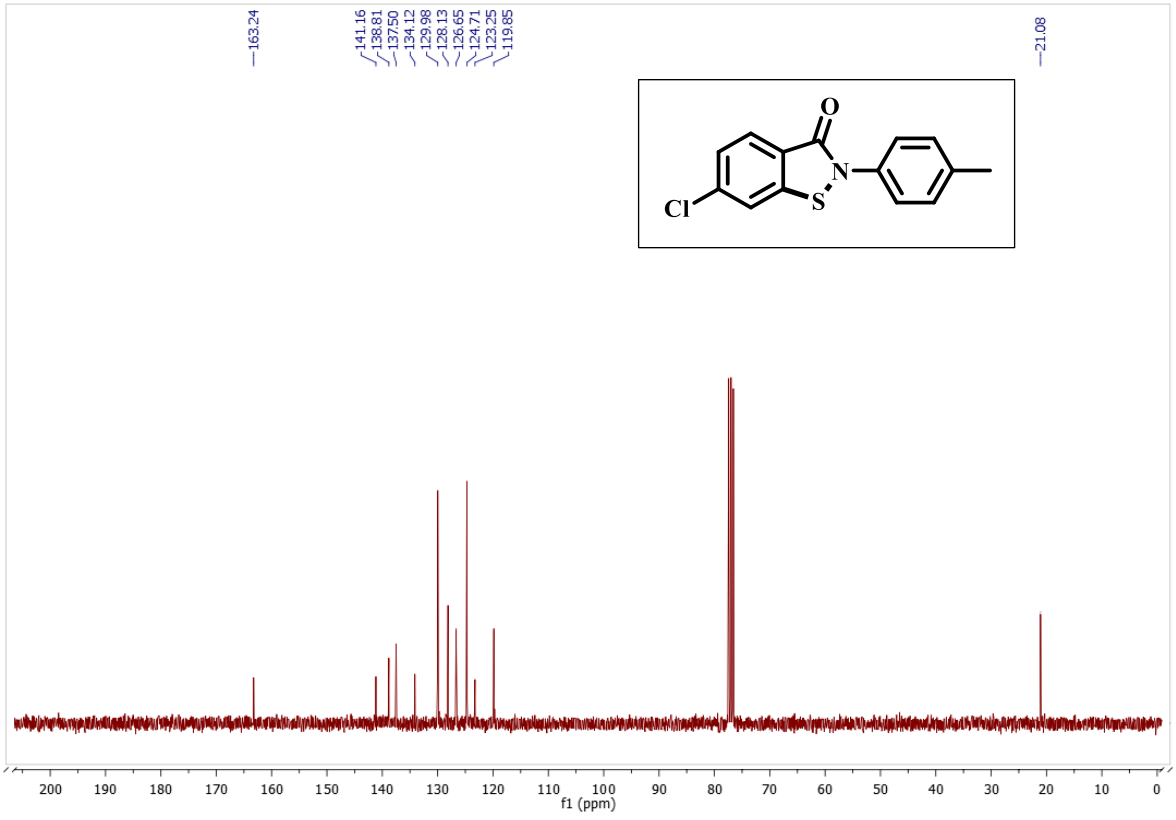
^{13}C-NMR of Compound 2r



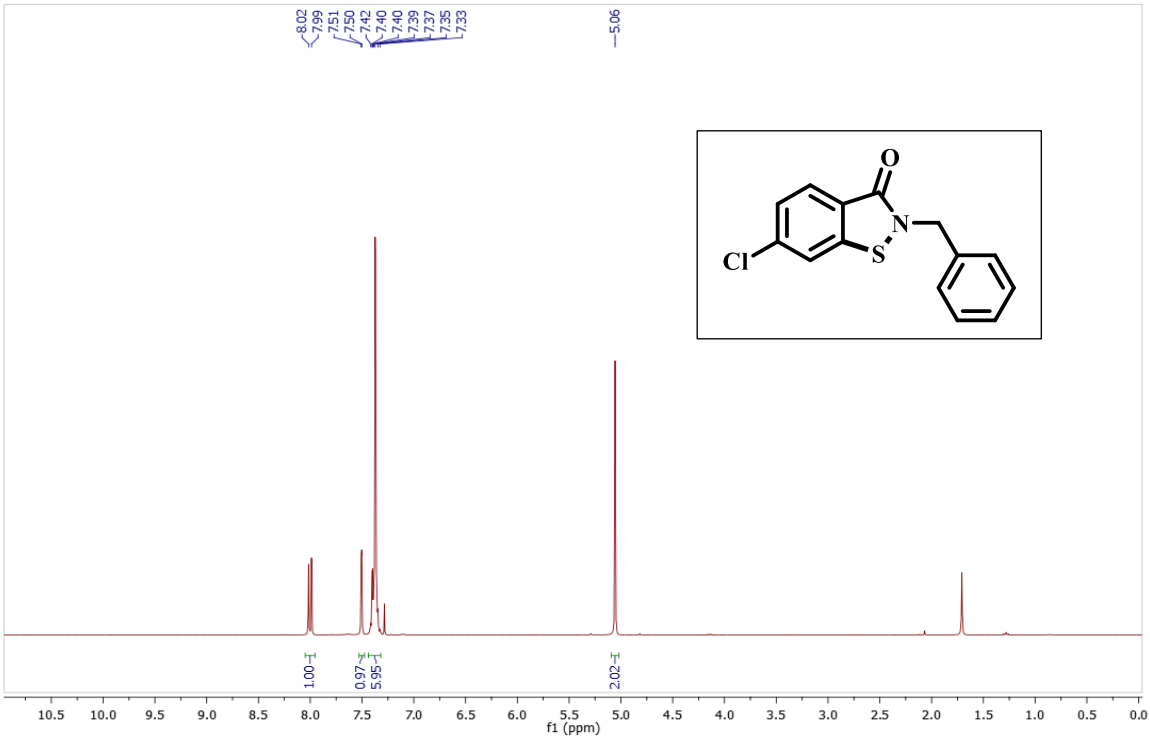
^{1}H-NMR of Compound 2s



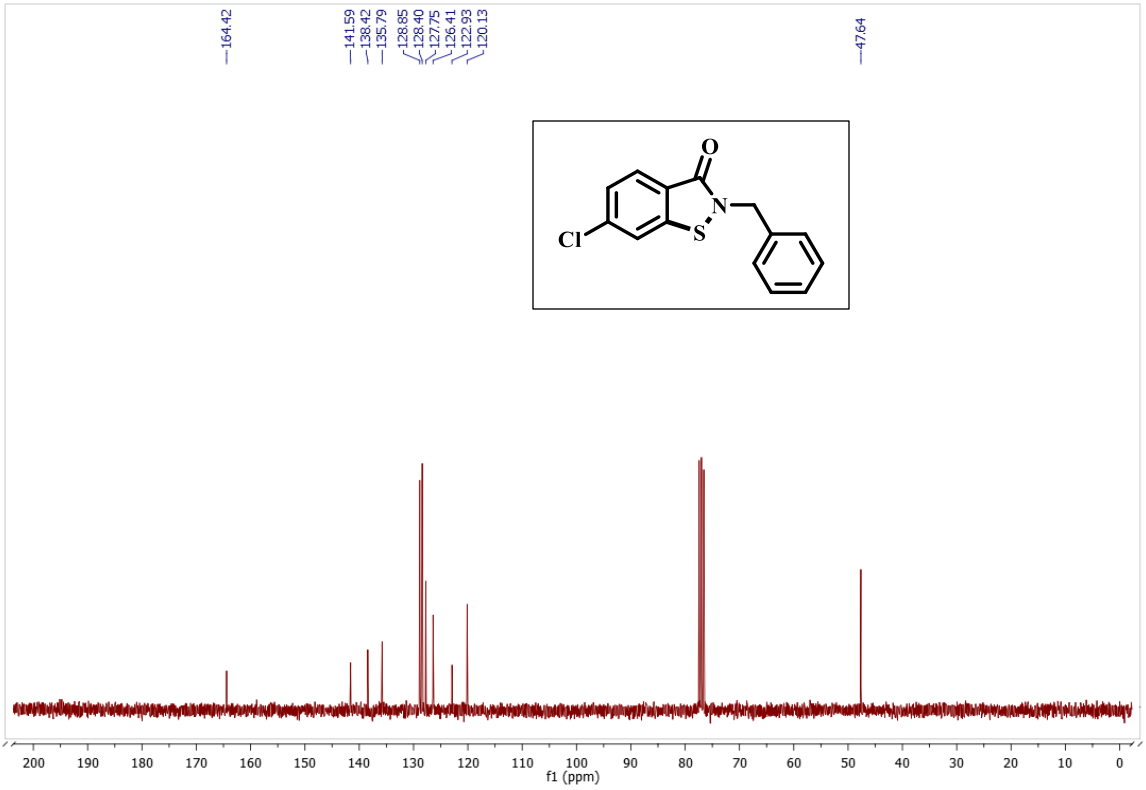
^{13}C-NMR of Compound 2s



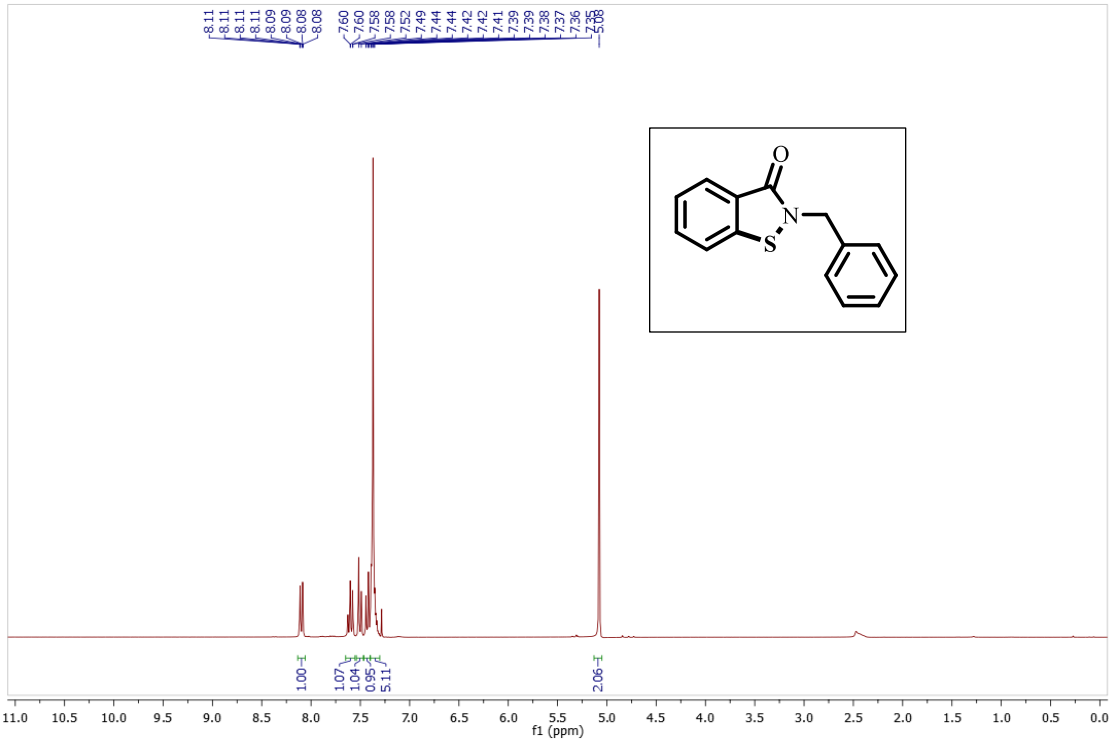
^{1}H-NMR of Compound 2t



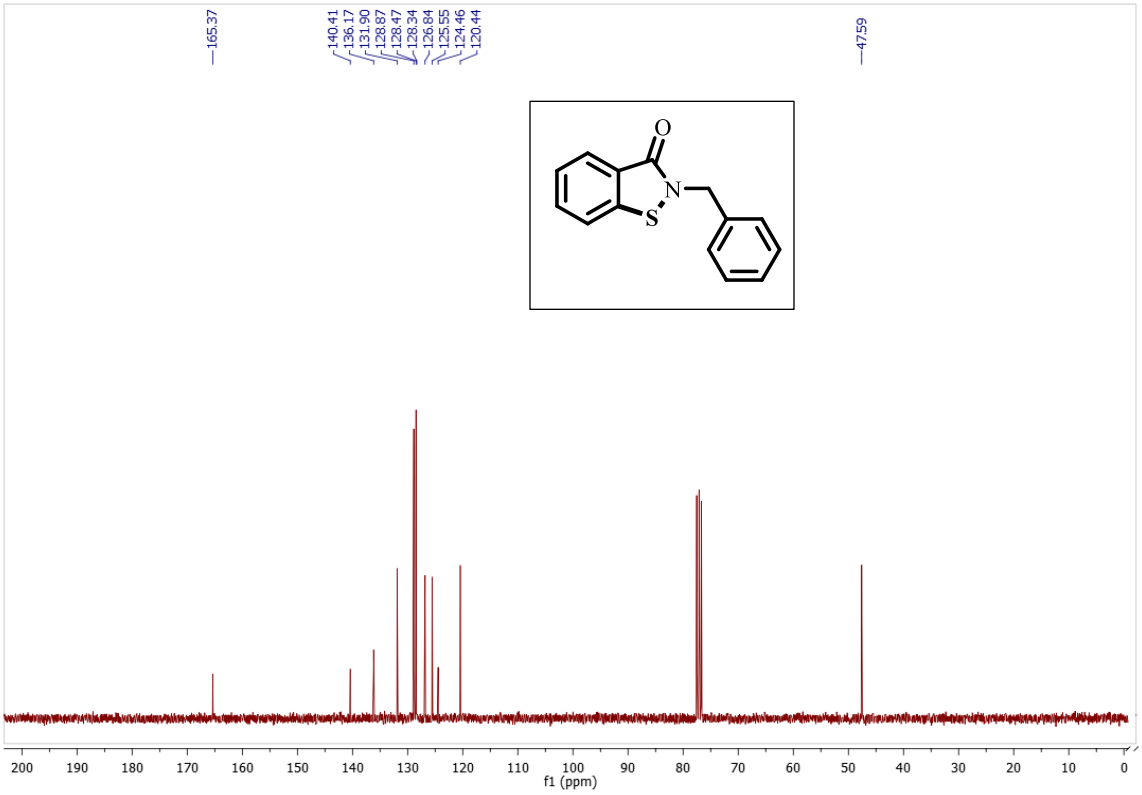
^{13}C-NMR of Compound 2t



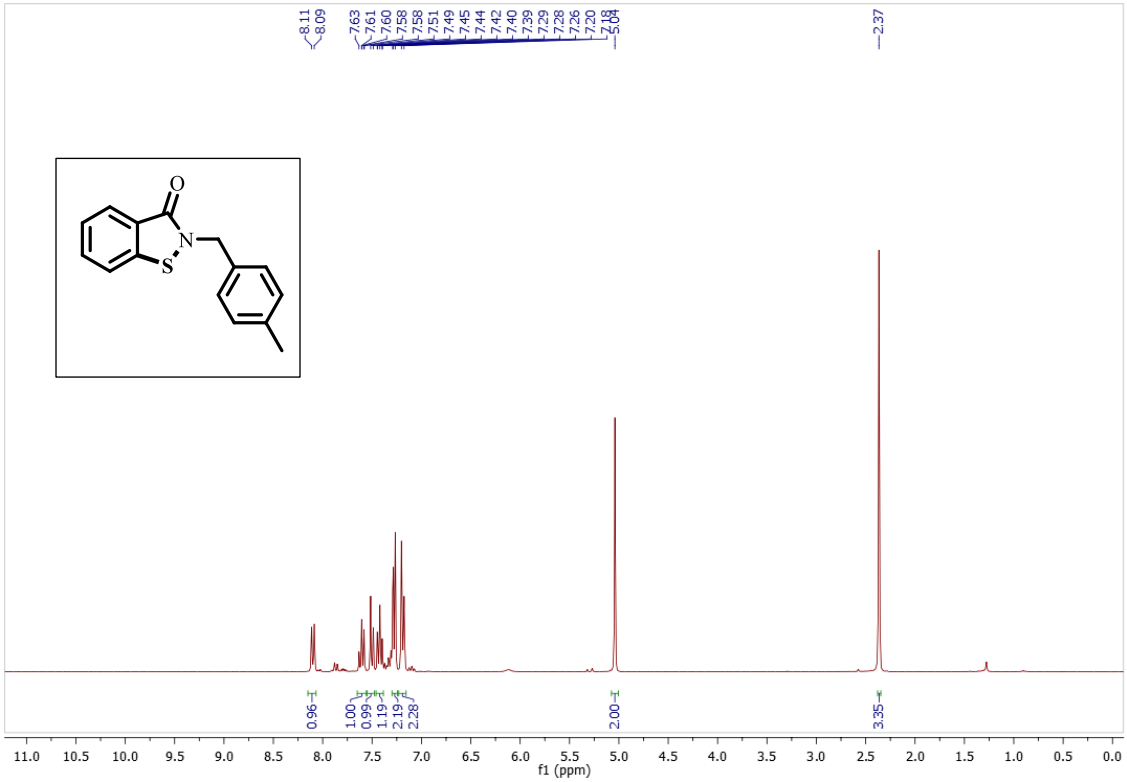
^{1}H-NMR of Compound 2u



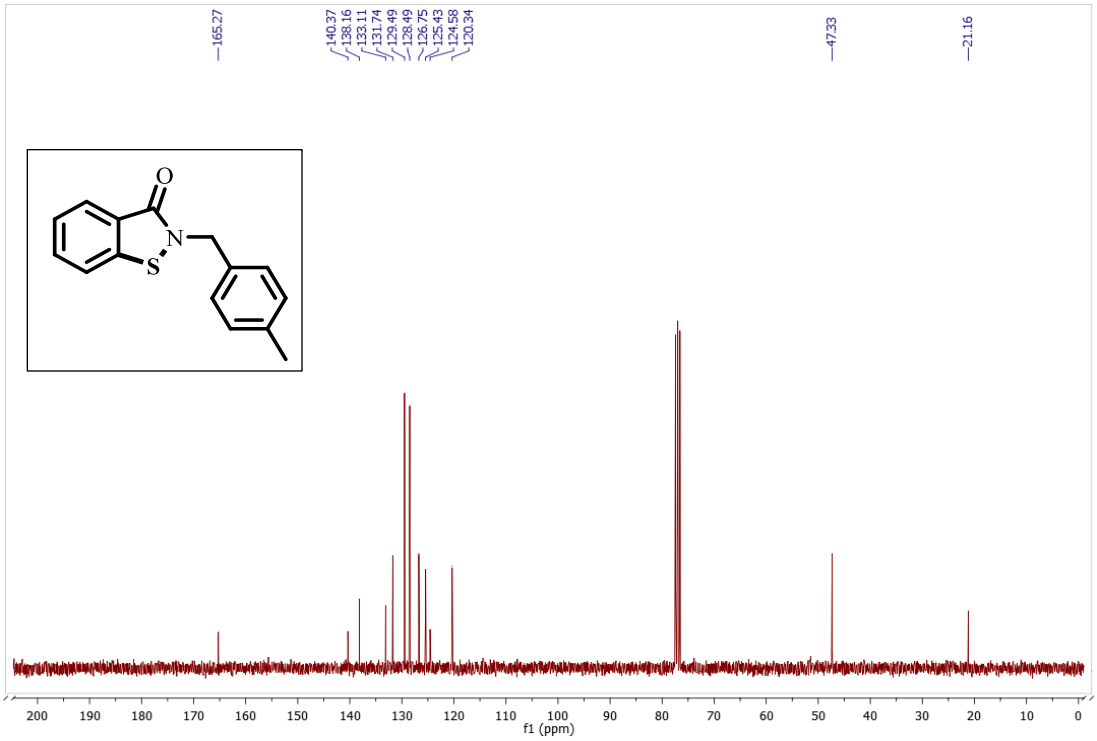
^{13}C-NMR of Compound 2u



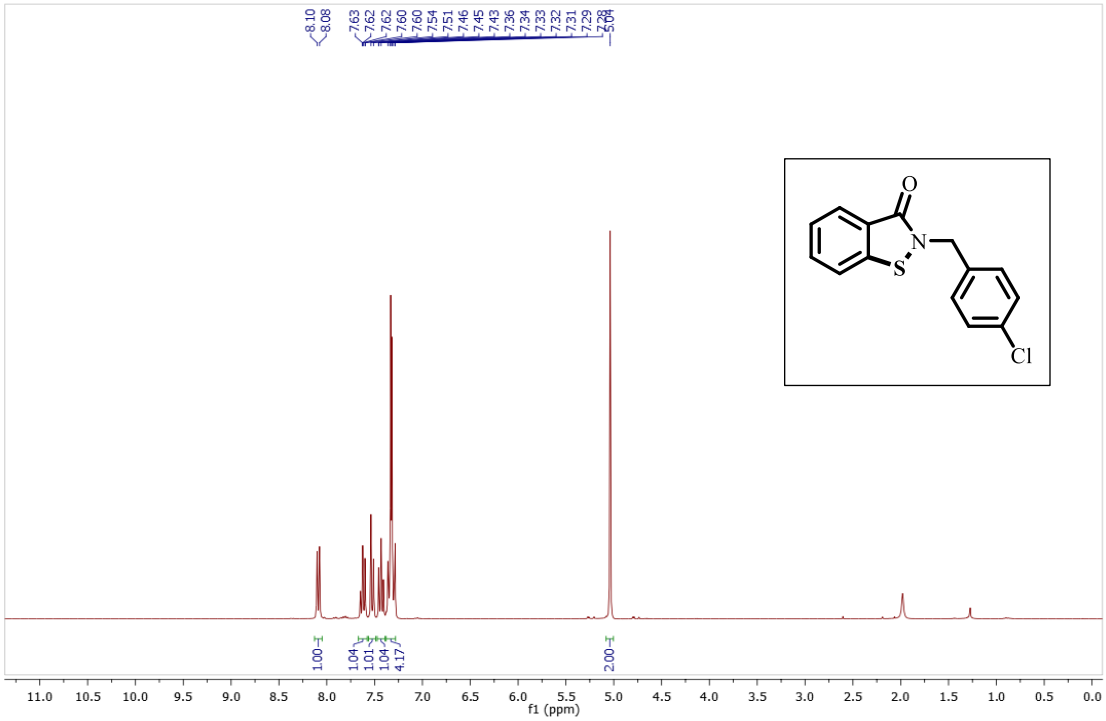
^{1}H-NMR of Compound 2v



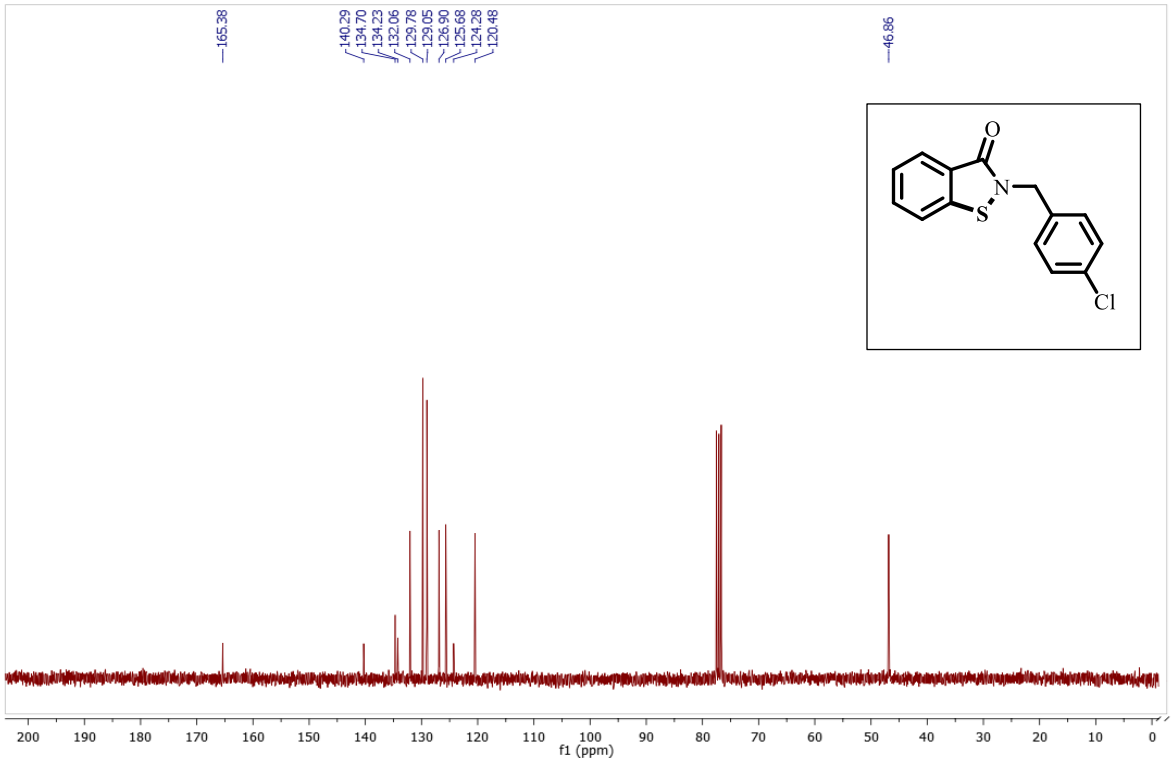
^{13}C-NMR of Compound 2v



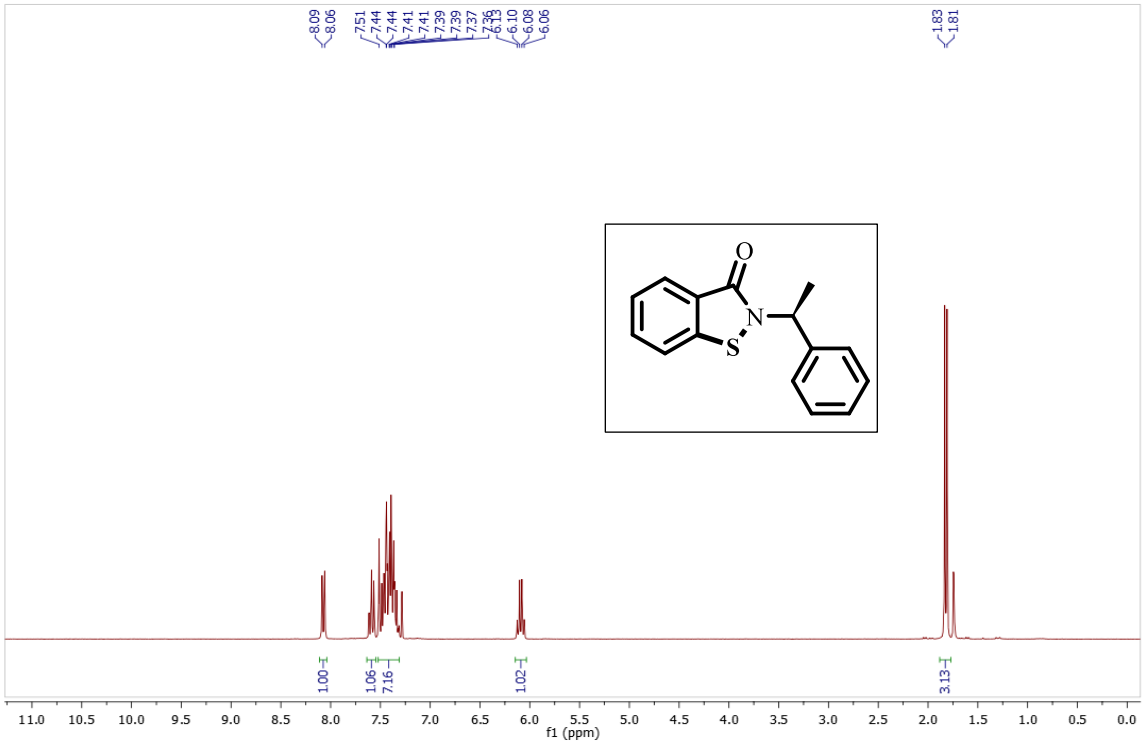
^{1}H-NMR of Compound 2w



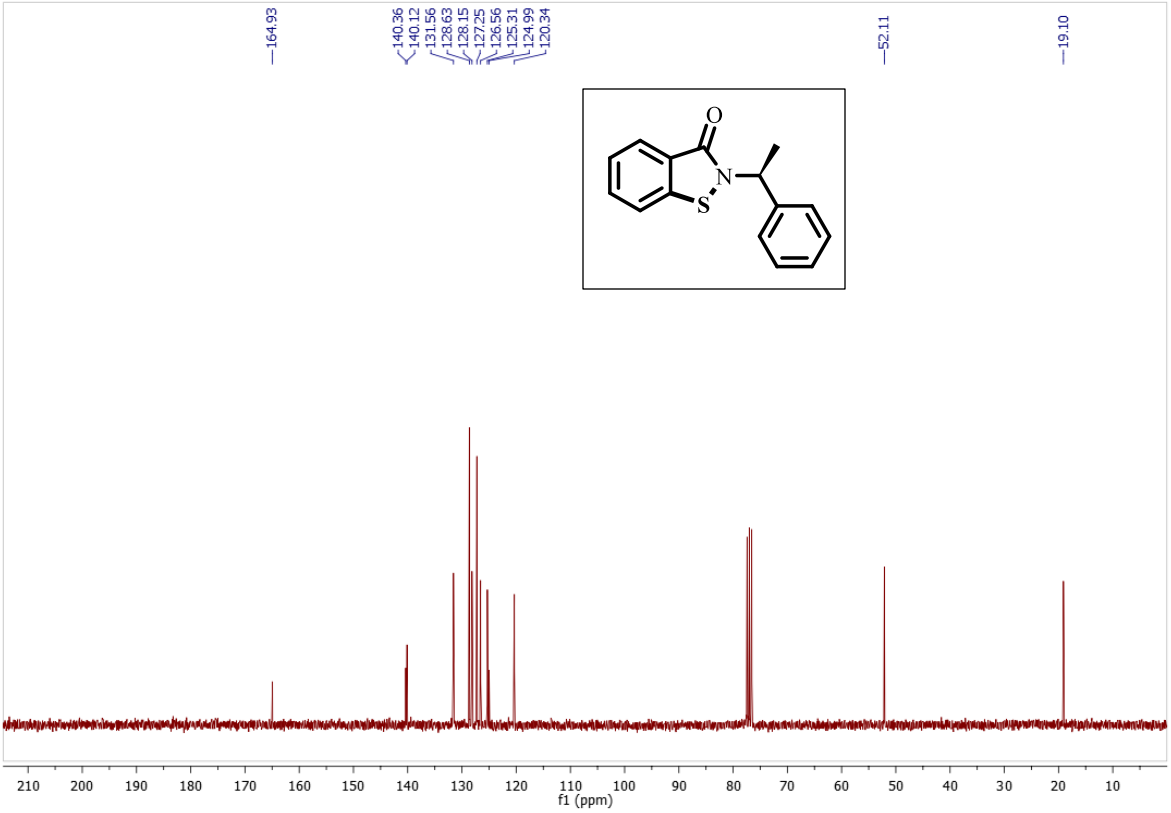
^{13}C-NMR of Compound 2w



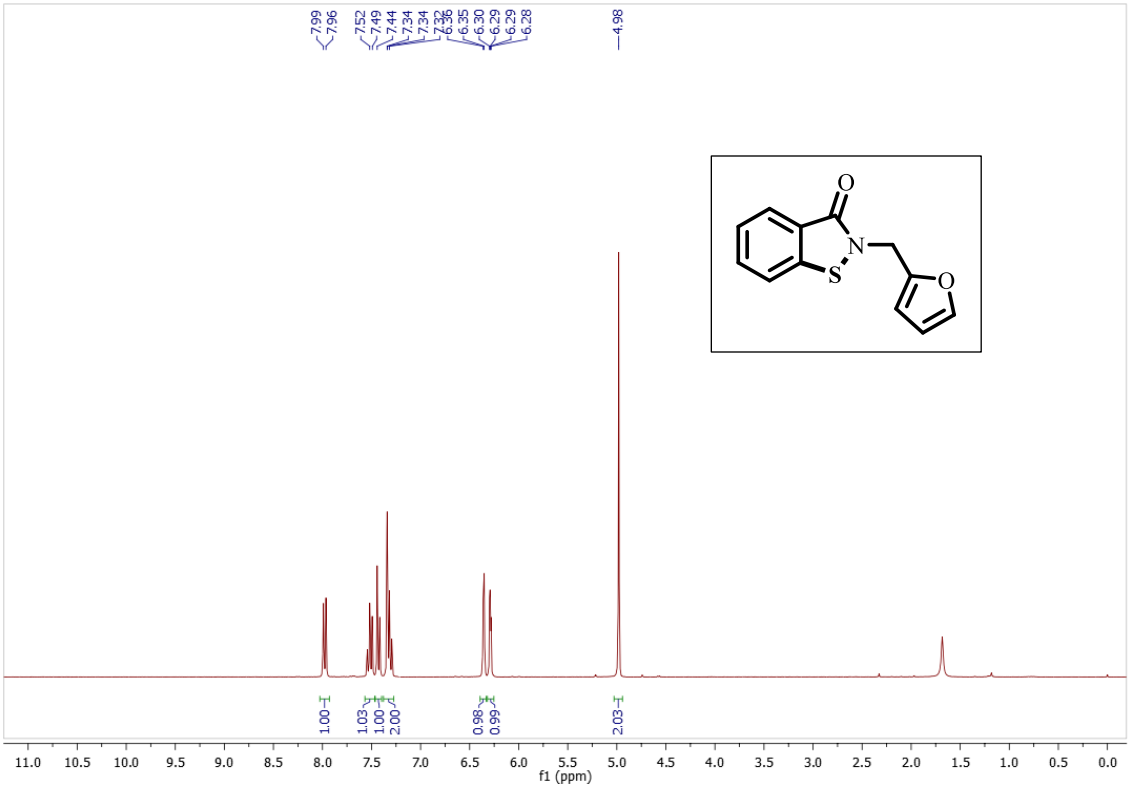
^{1}H-NMR of Compound 2x



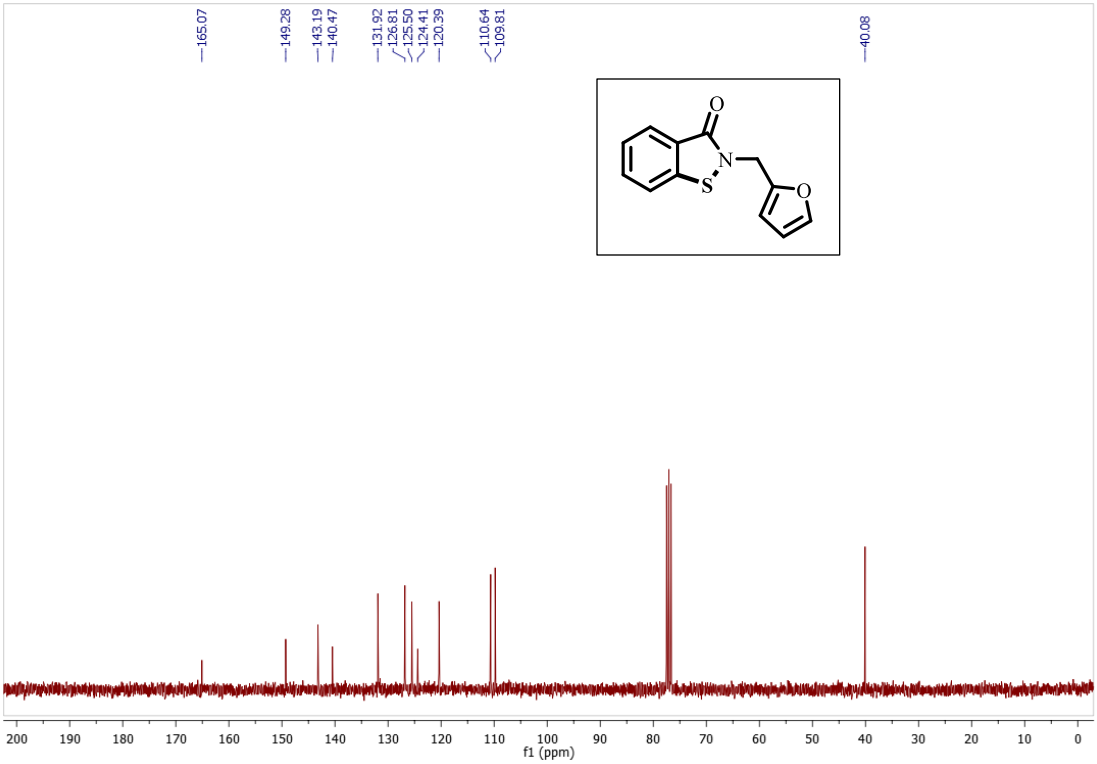
^{13}C-NMR of Compound 2x



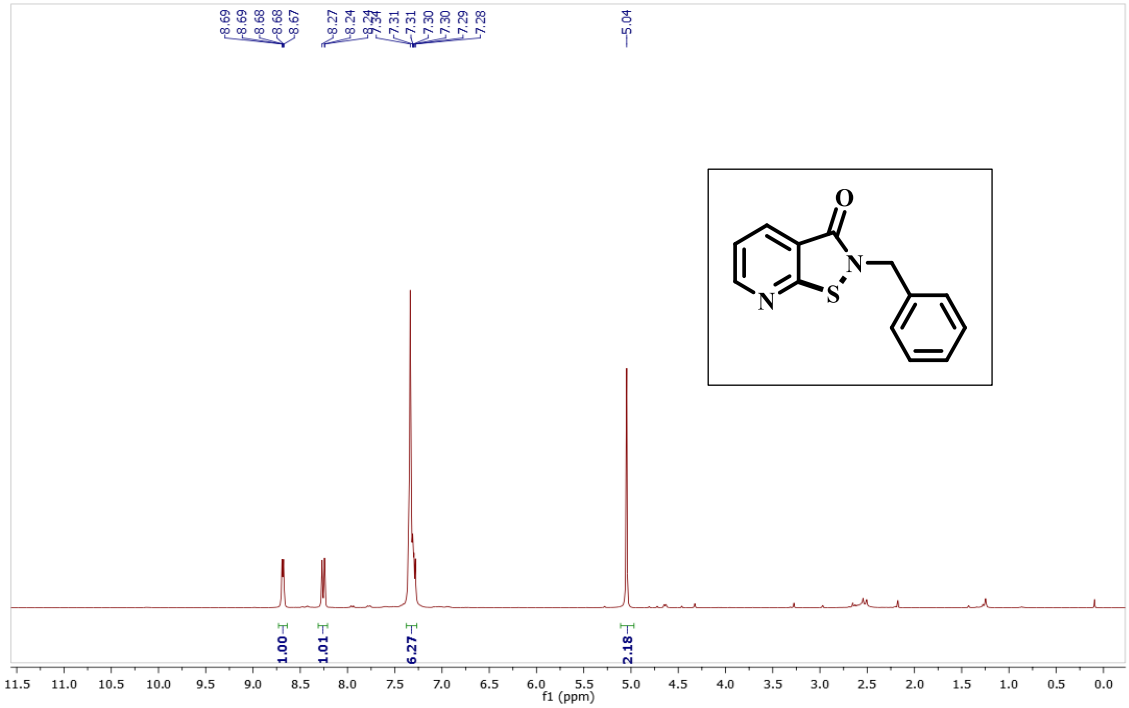
^{1}H-NMR of Compound 2y



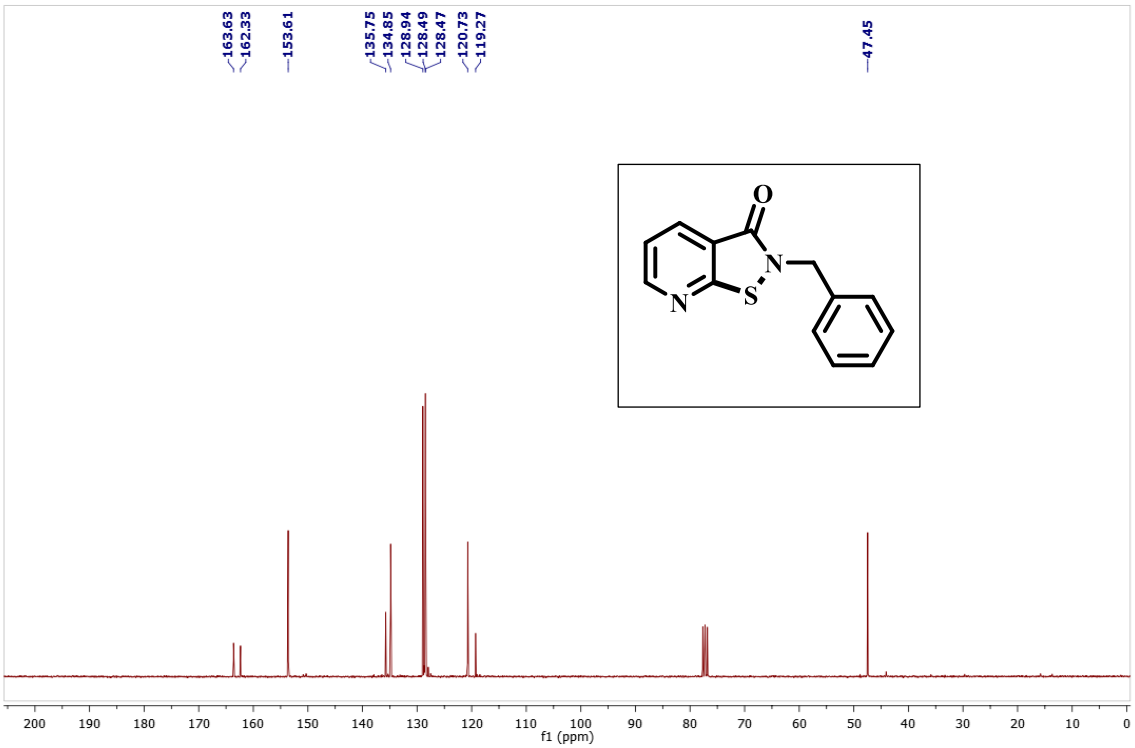
^{13}C-NMR of Compound 2y



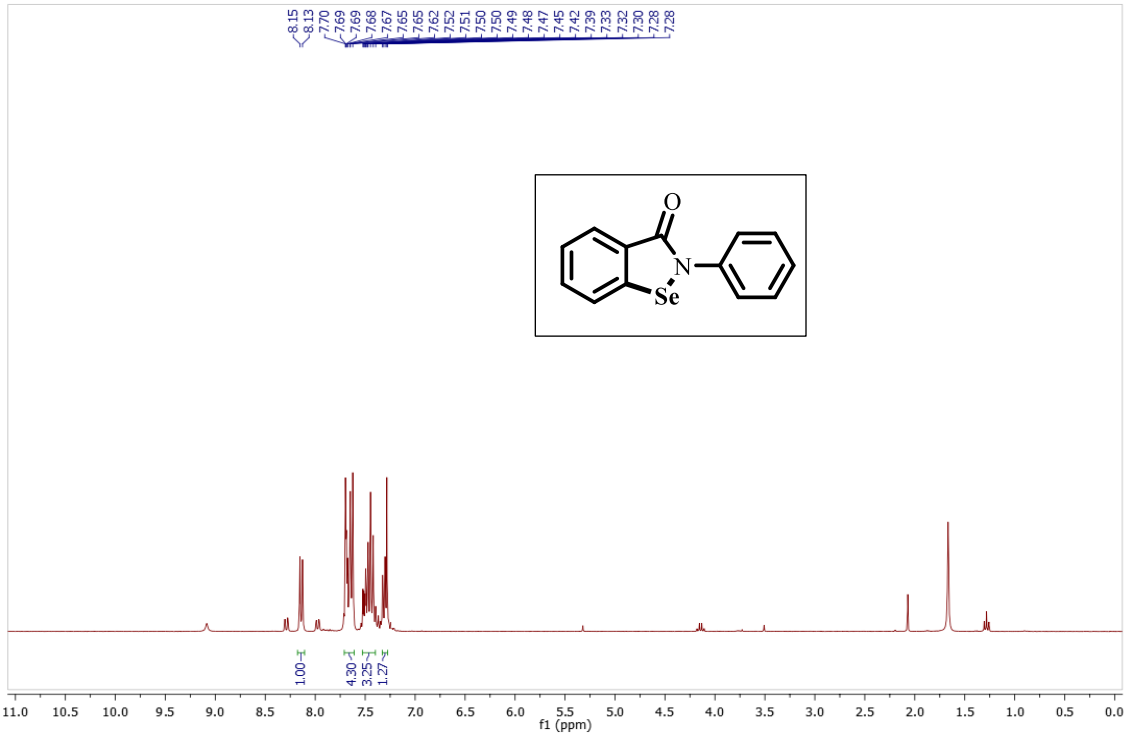
^{1}H-NMR of Compound 2z



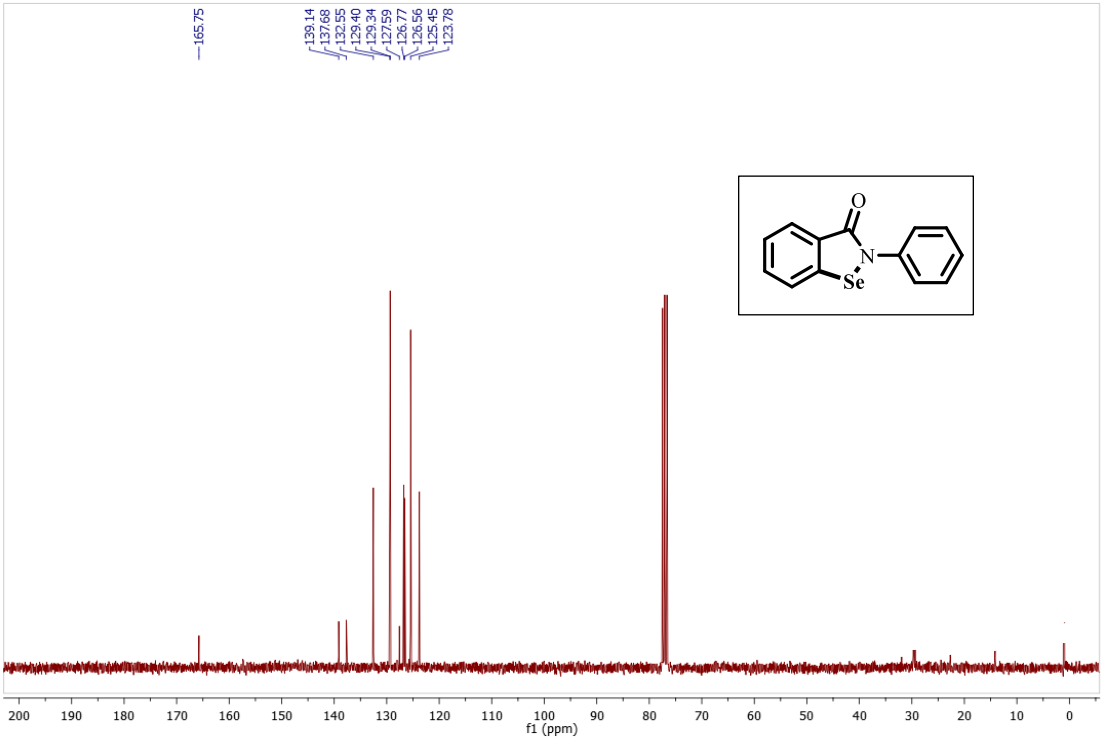
^{13}C-NMR of Compound 2z



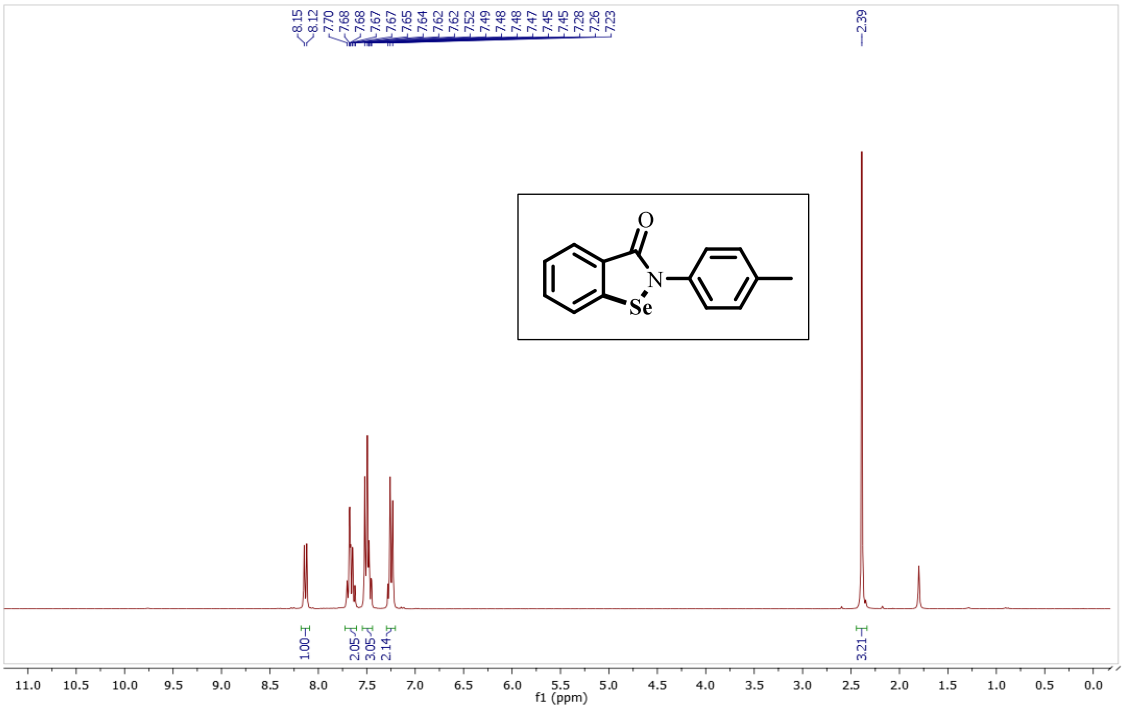
^{1}H-NMR of Compound 3a



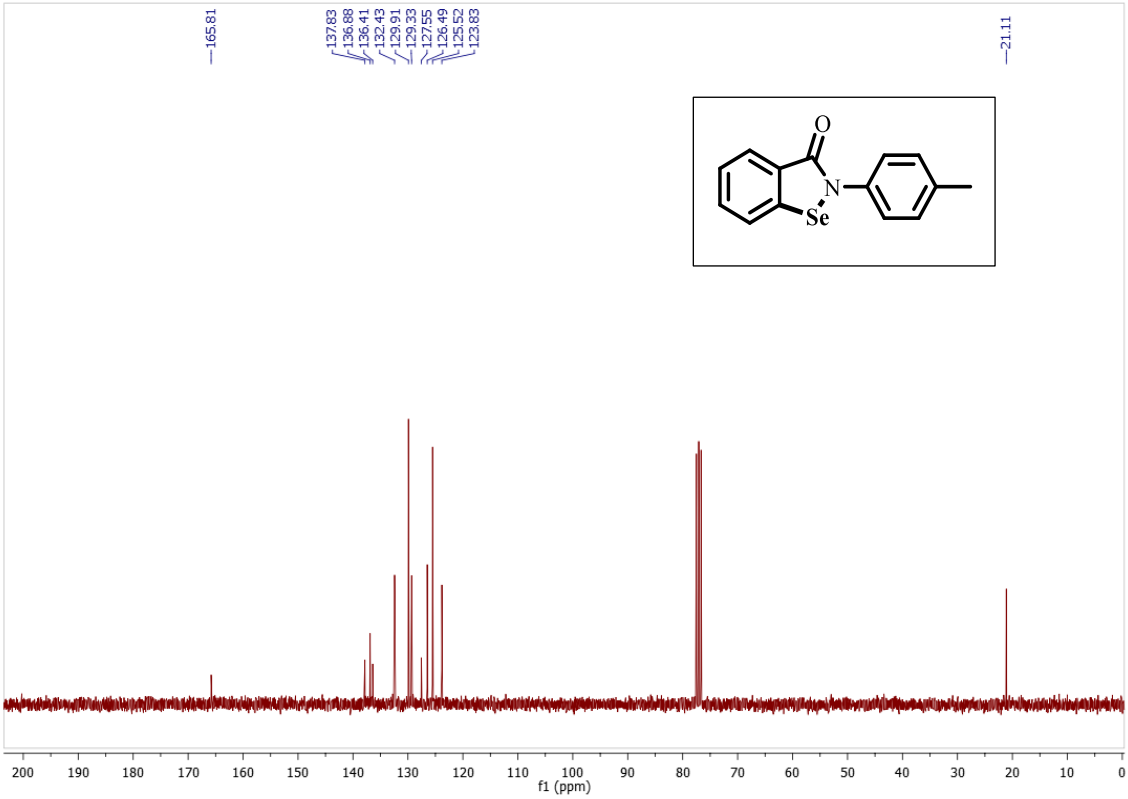
^{13}C-NMR of Compound 3a



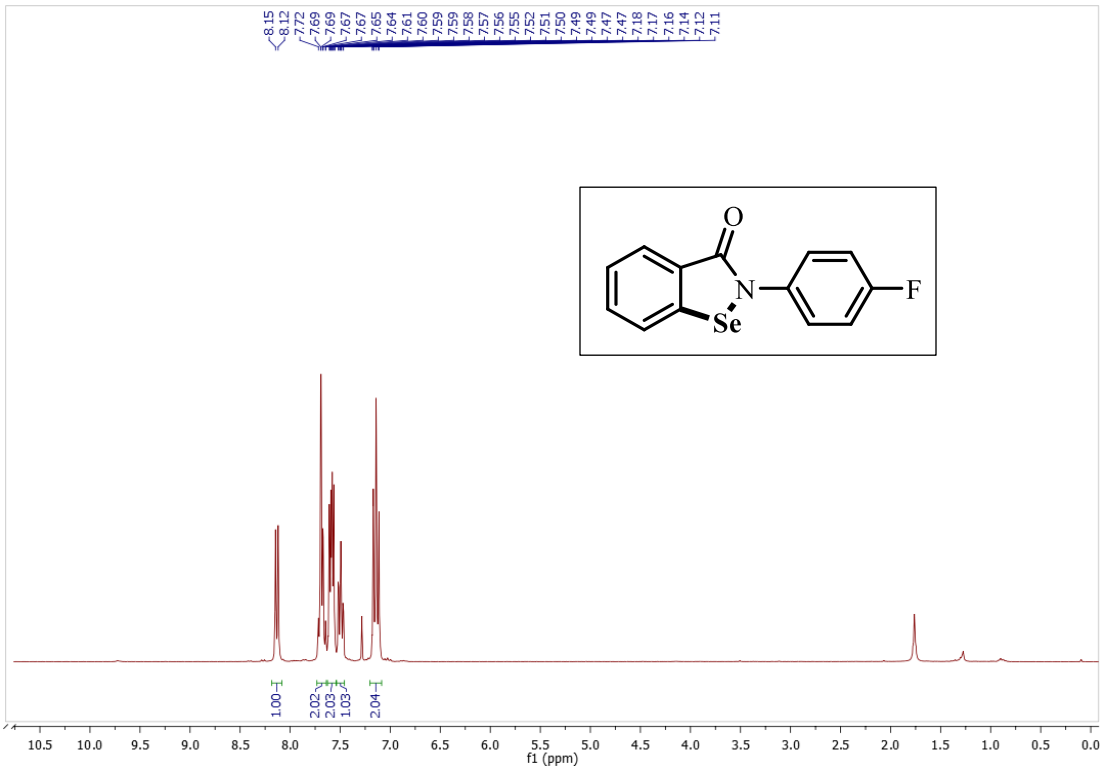
^{1}H-NMR of Compound 3b



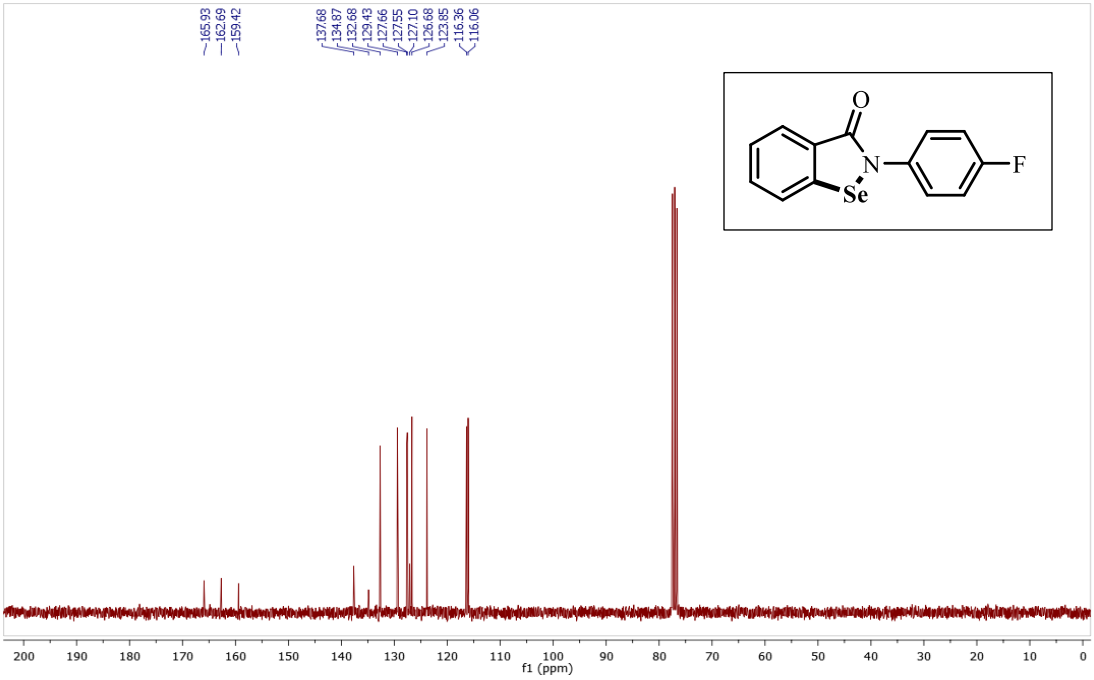
^{13}C-NMR of Compound 3b



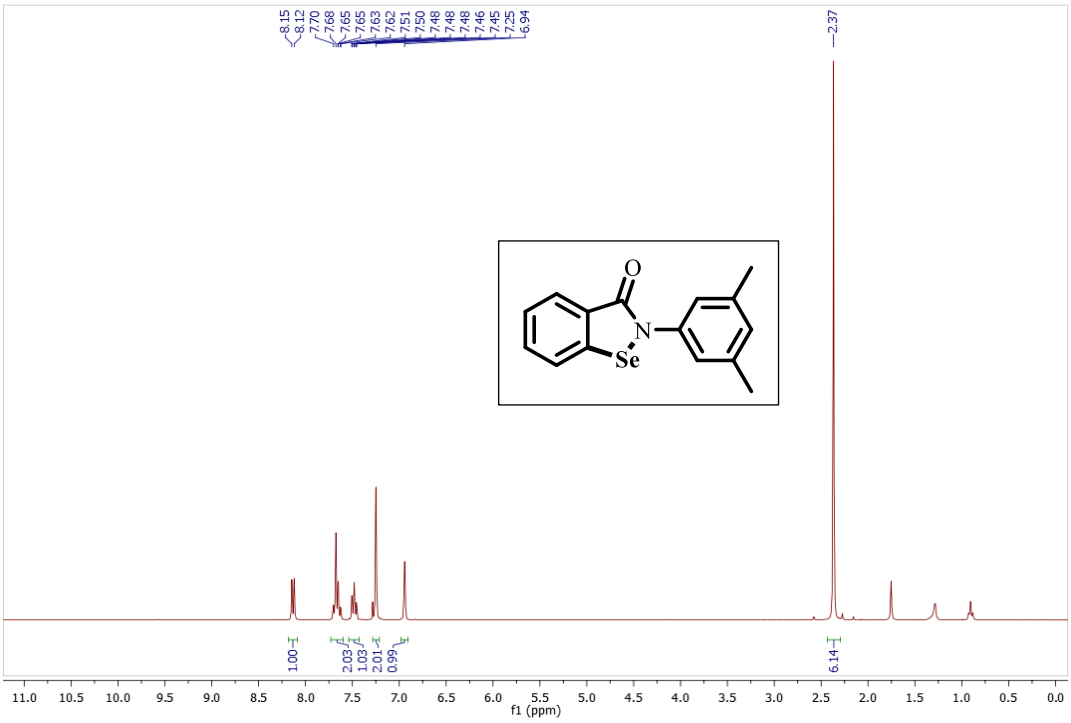
^{1}H-NMR of Compound 3c



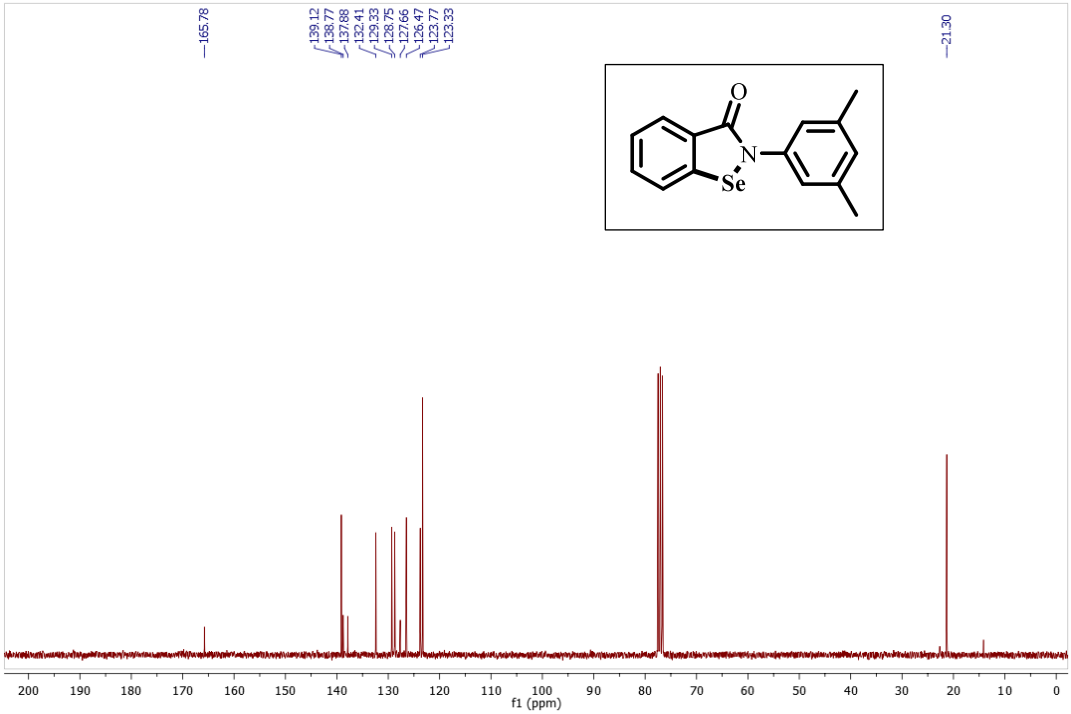
^{13}C-NMR of Compound 3c



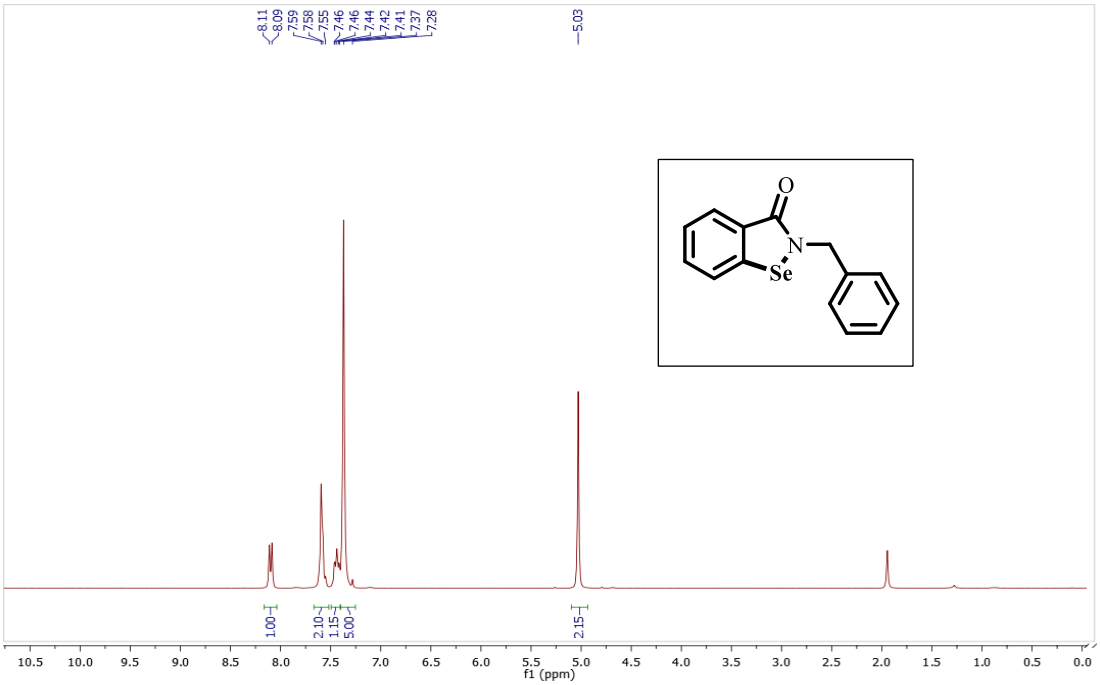
^{1}H-NMR of Compound 3d



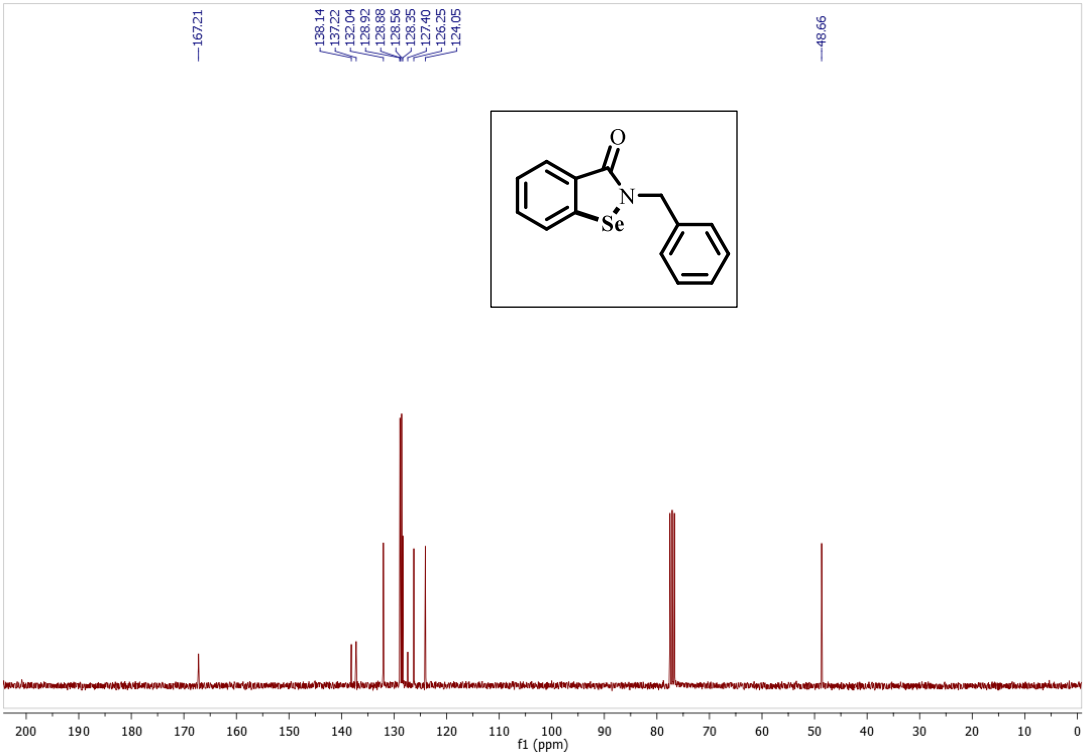
^{13}C-NMR of Compound 3d



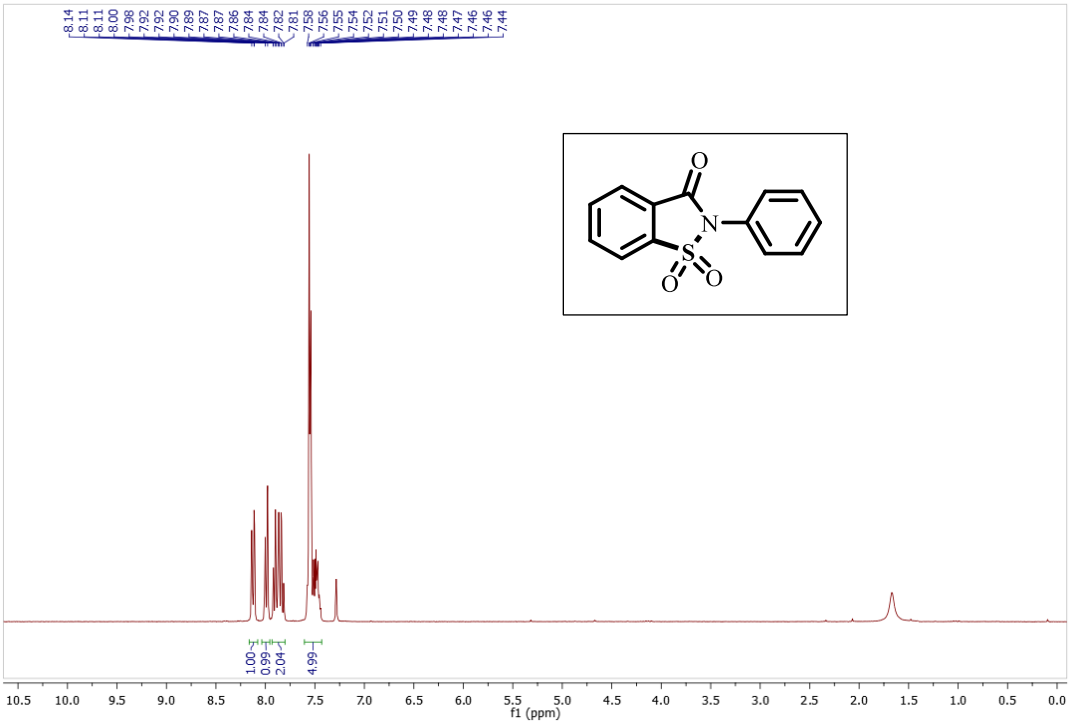
^{1}H-NMR of Compound 3e



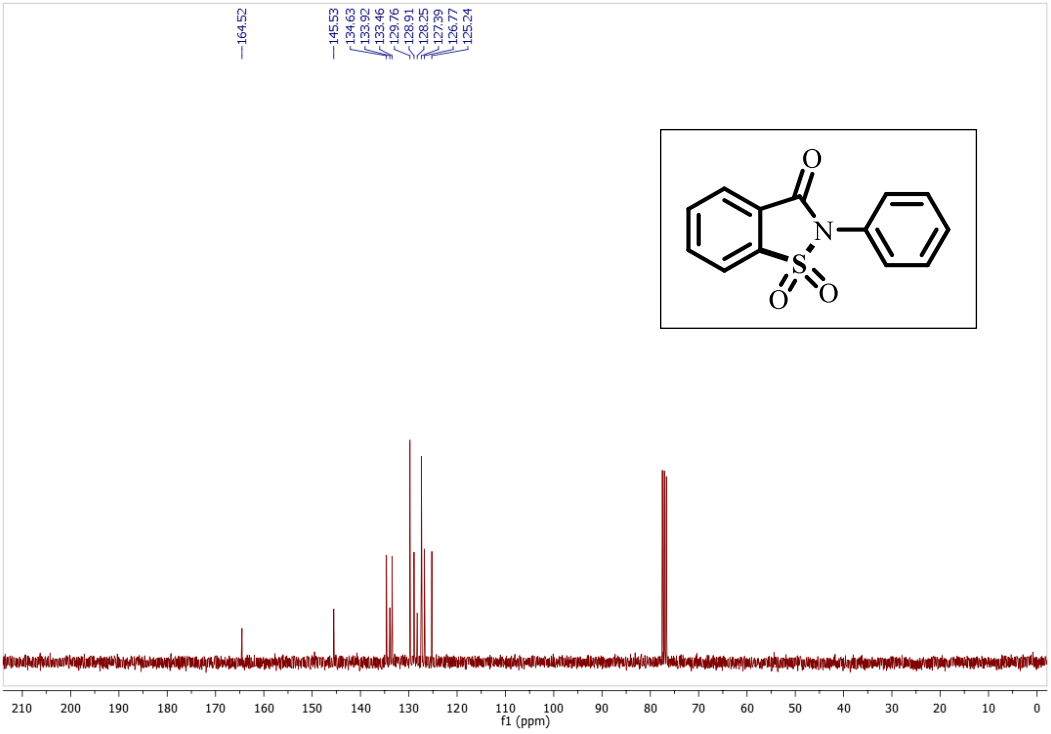
^{13}C-NMR of Compound 3e



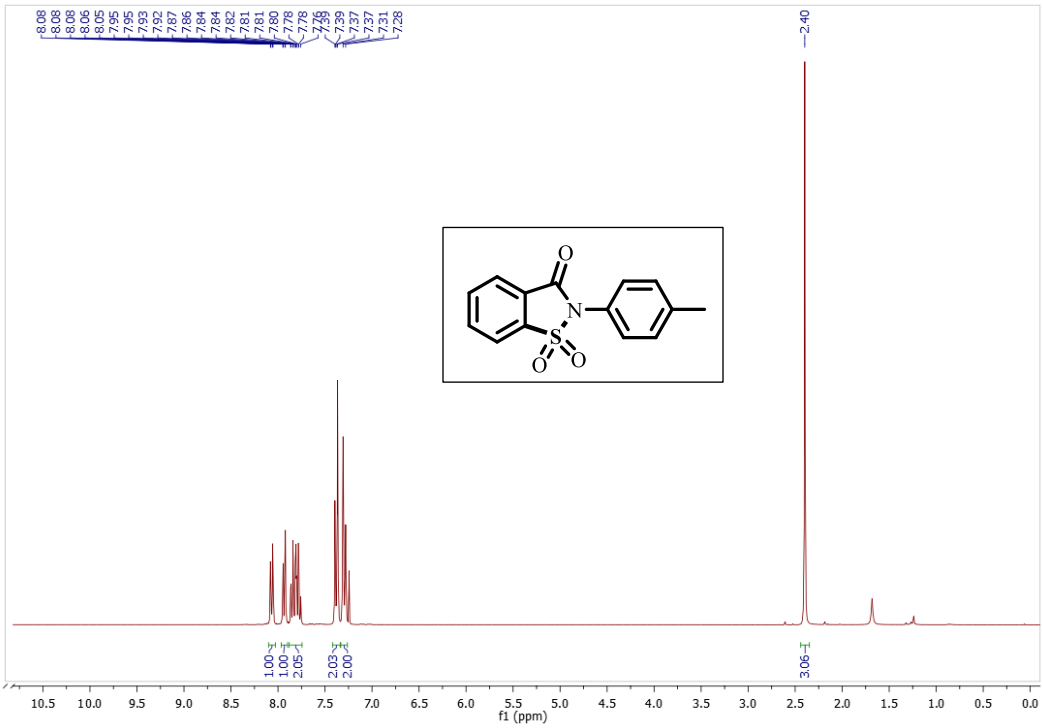
^{1}H-NMR of Compound 4a



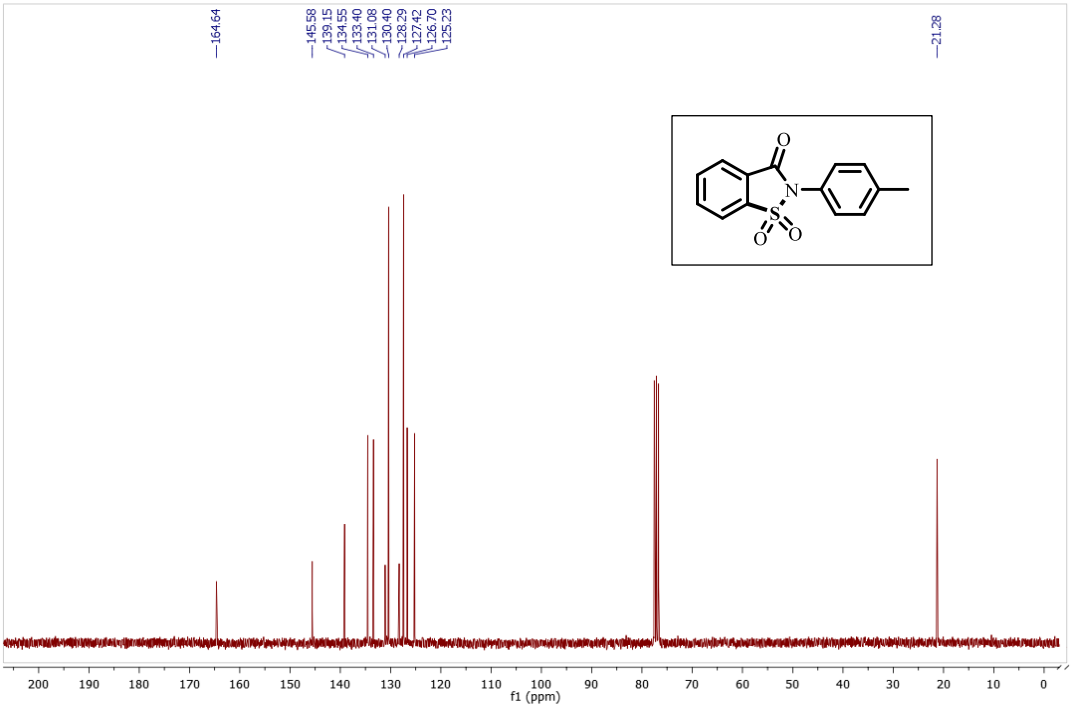
^{13}C-NMR of Compound 4a



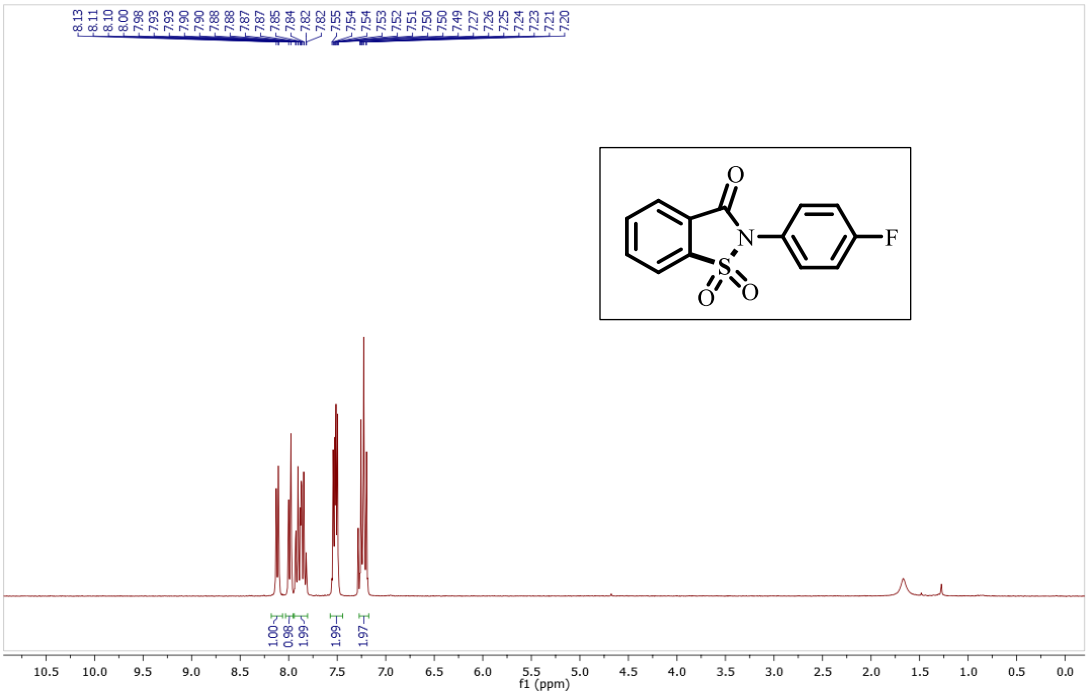
^{1}H-NMR of Compound 4b



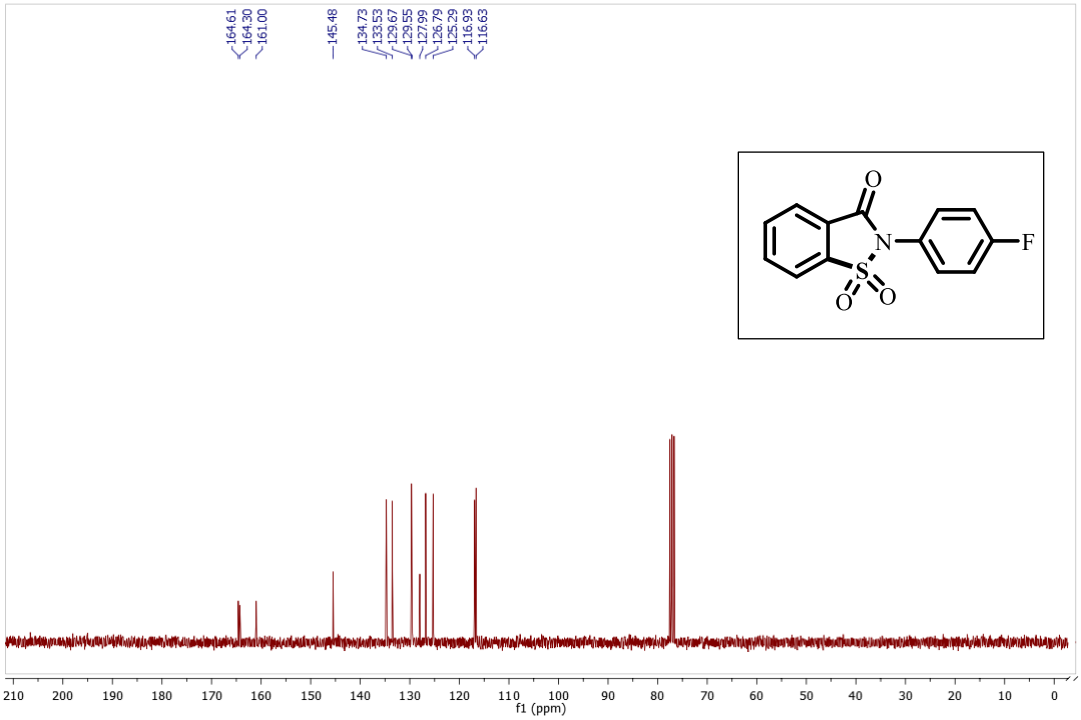
^{13}C-NMR of Compound 4b



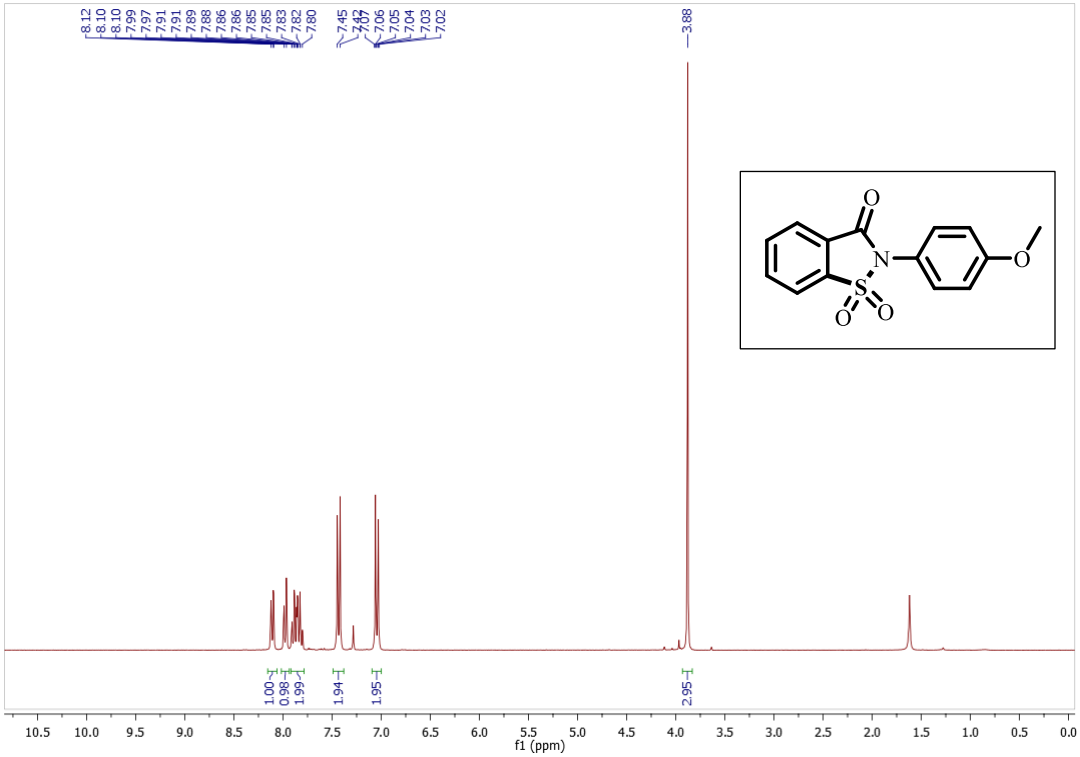
^{1}H-NMR of Compound 4c



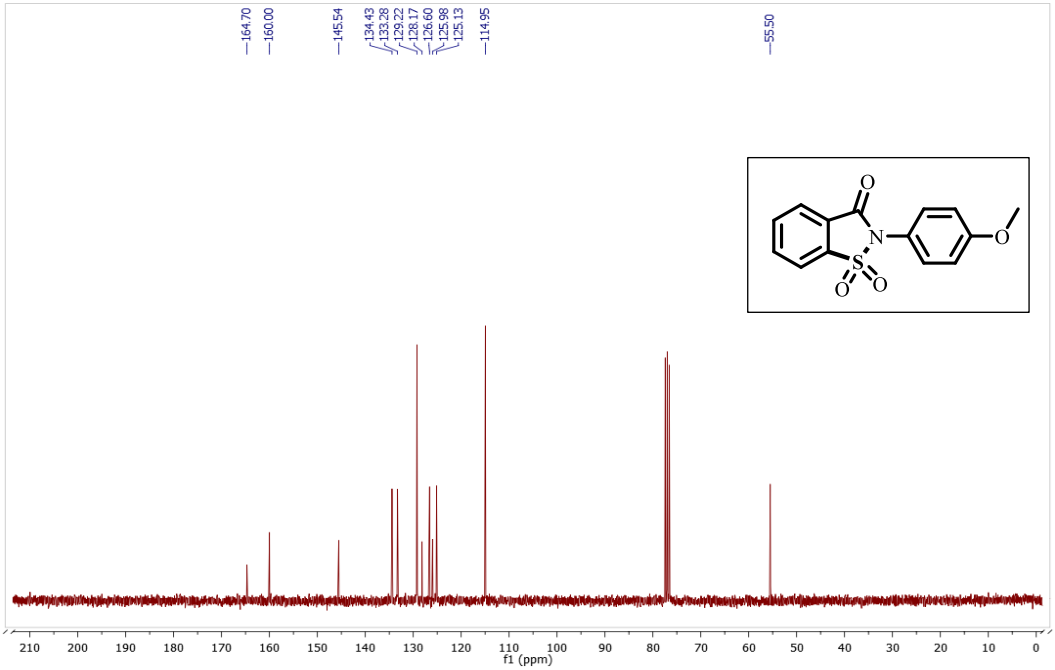
^{13}C-NMR of Compound 4c



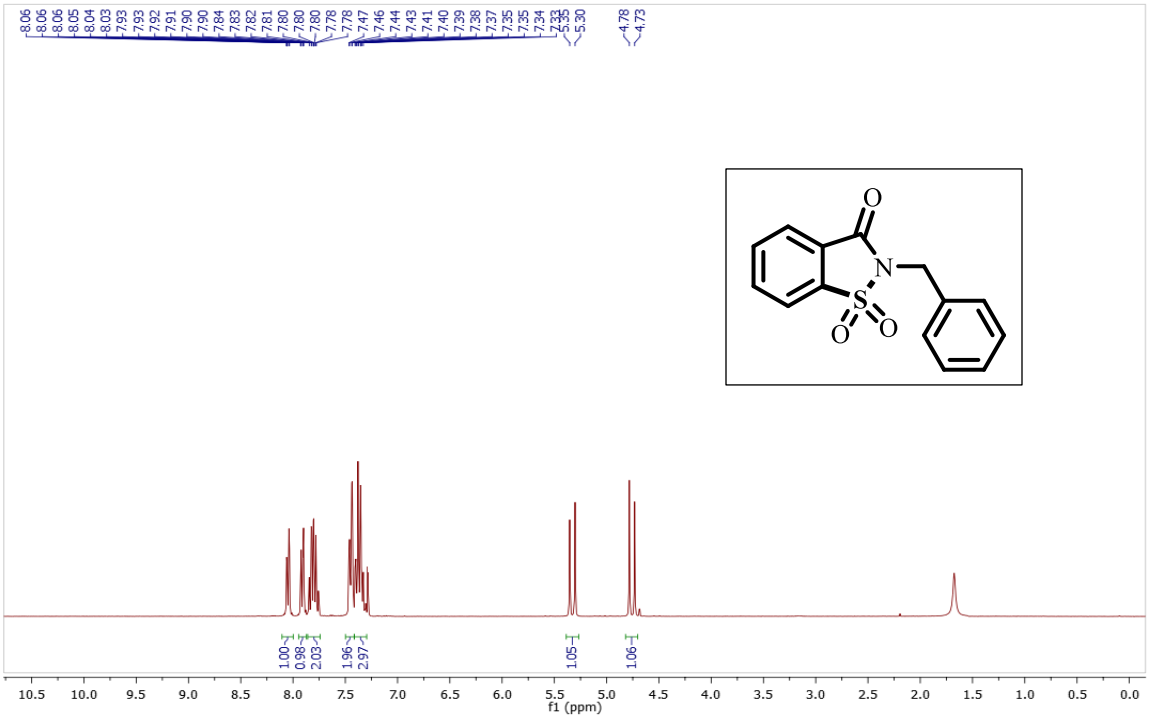
^{1}H-NMR of Compound 4d



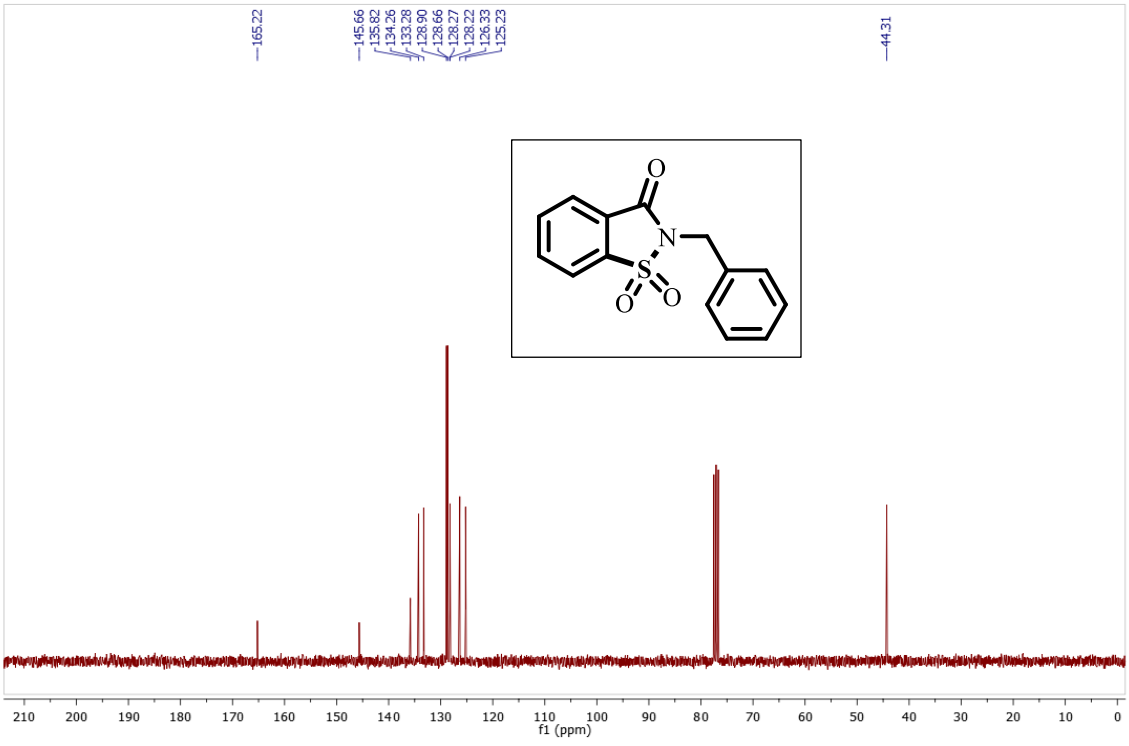
^{13}C-NMR of Compound 4d



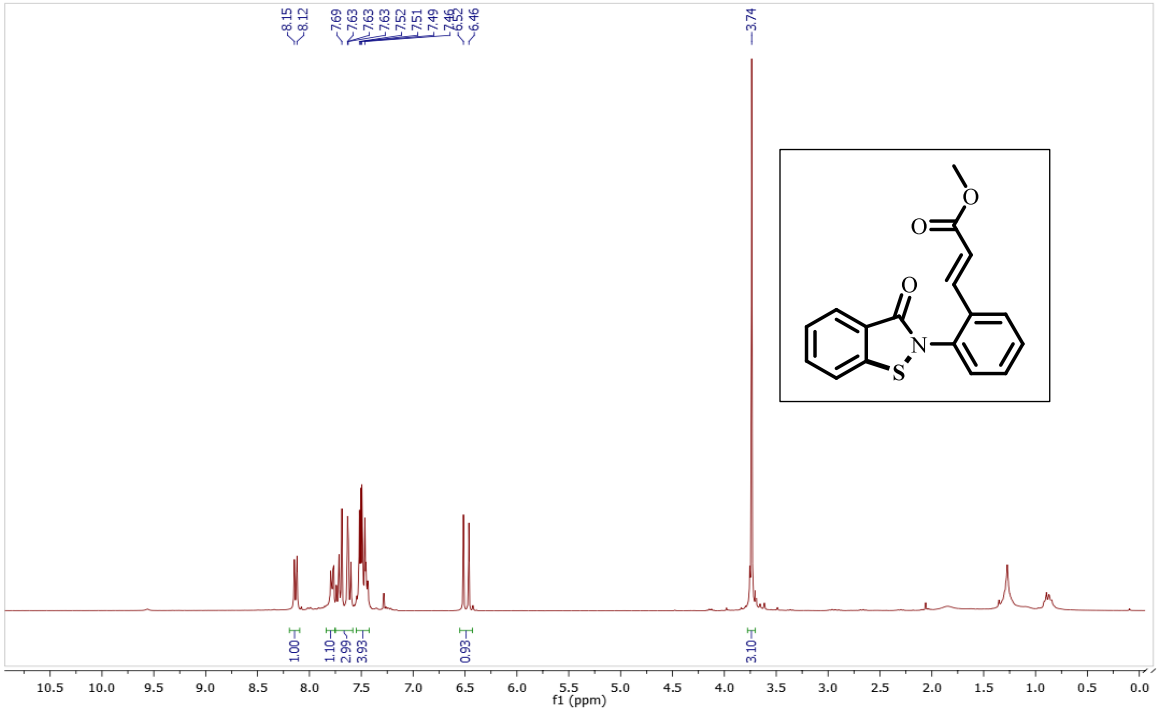
^{1}H-NMR of Compound 4e



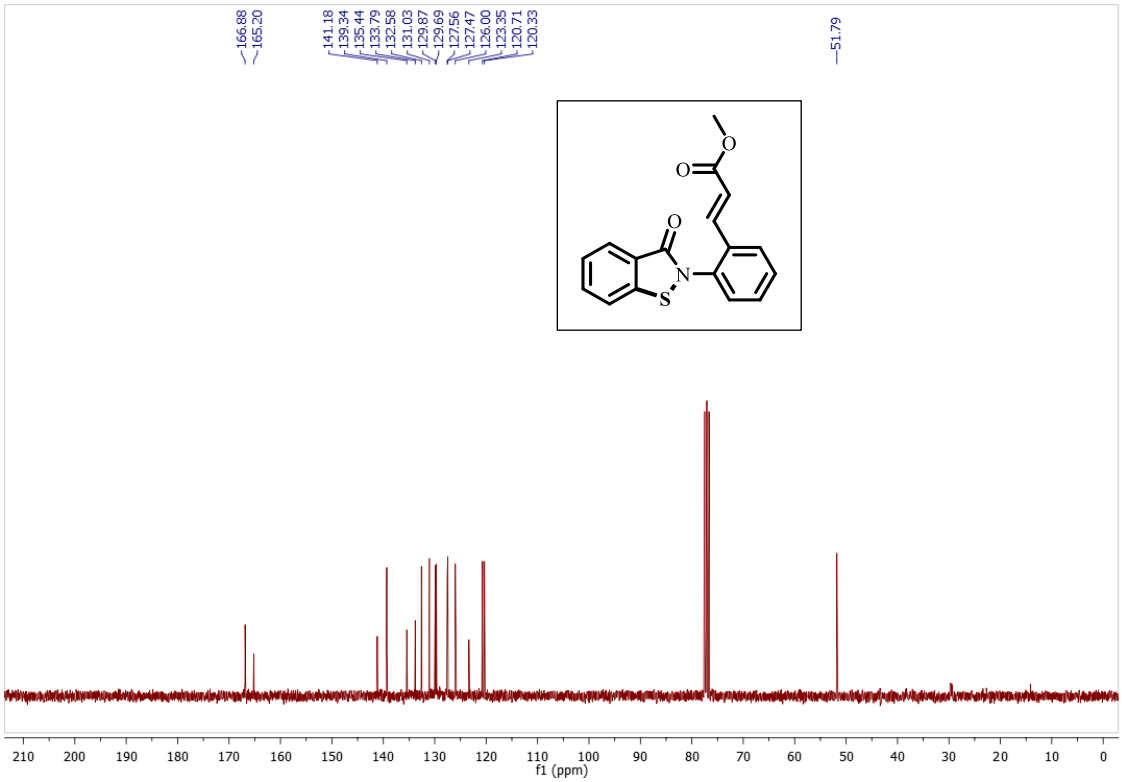
^{13}C-NMR of Compound 4e



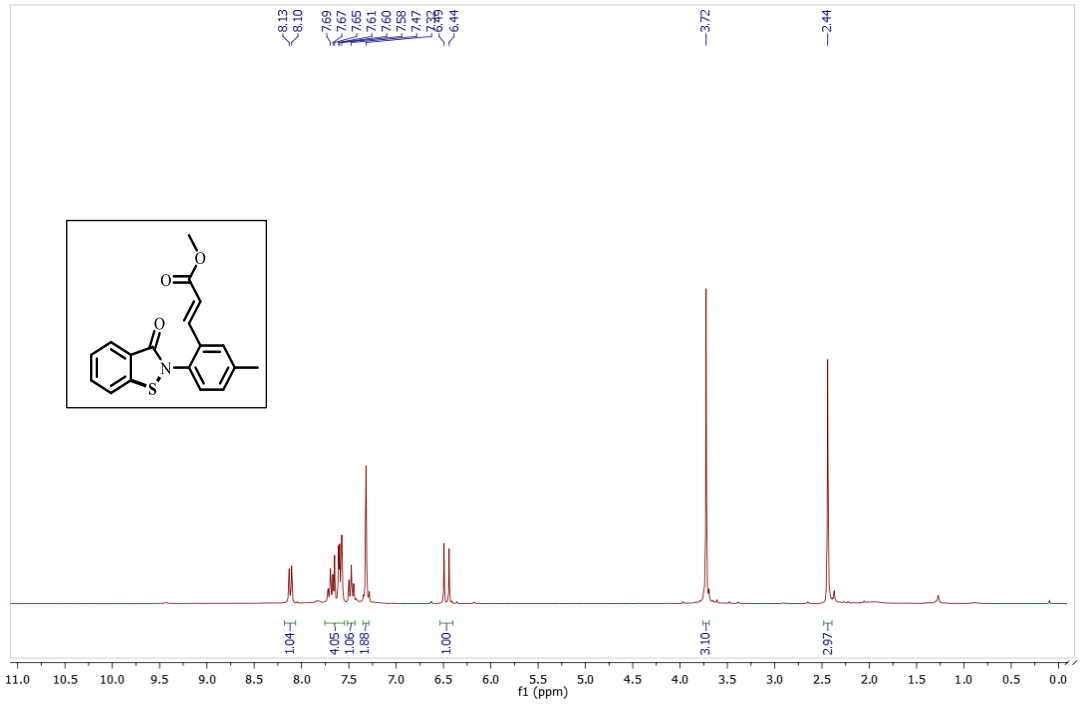
^{1}H-NMR of Compound 5a



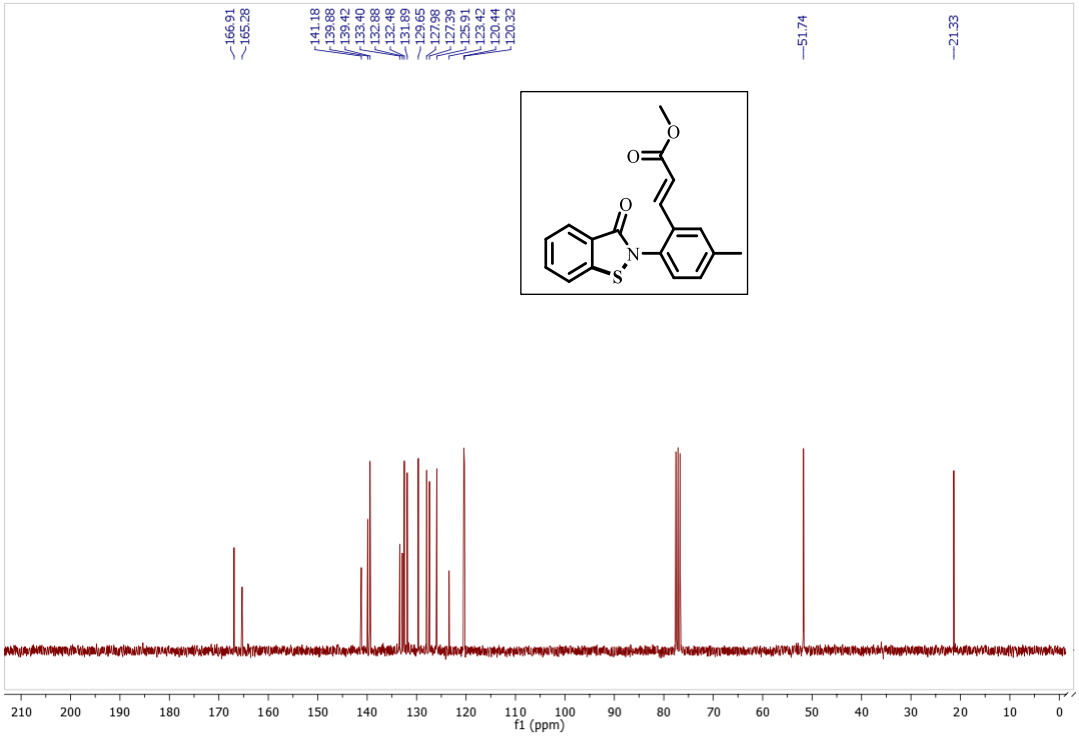
^{13}C-NMR of Compound 5a



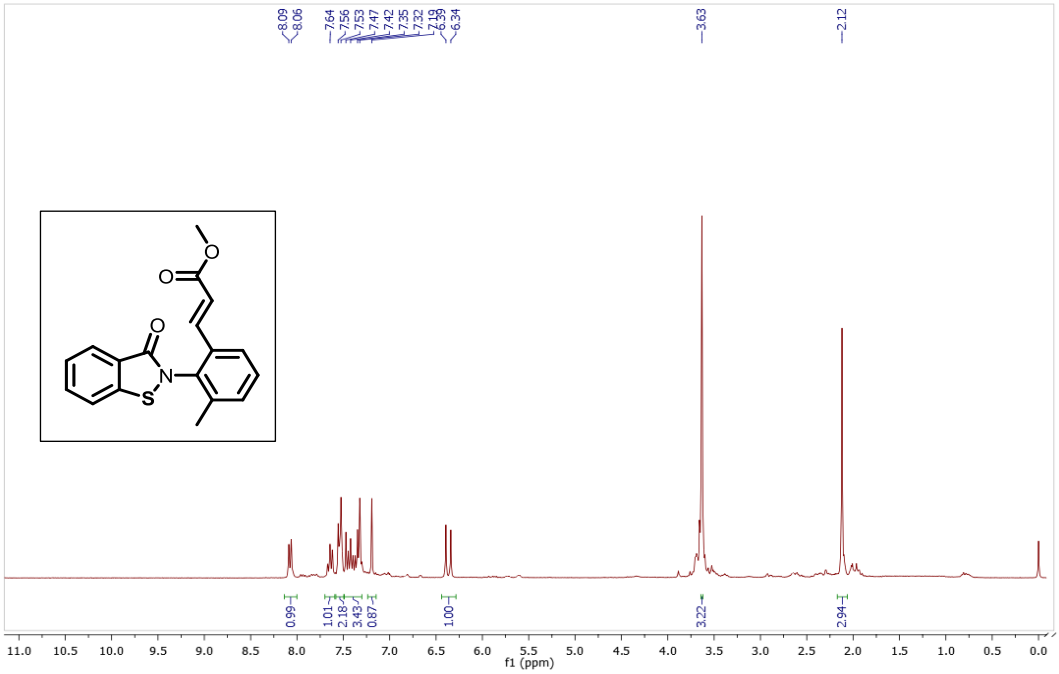
^{1}H-NMR of Compound 5b



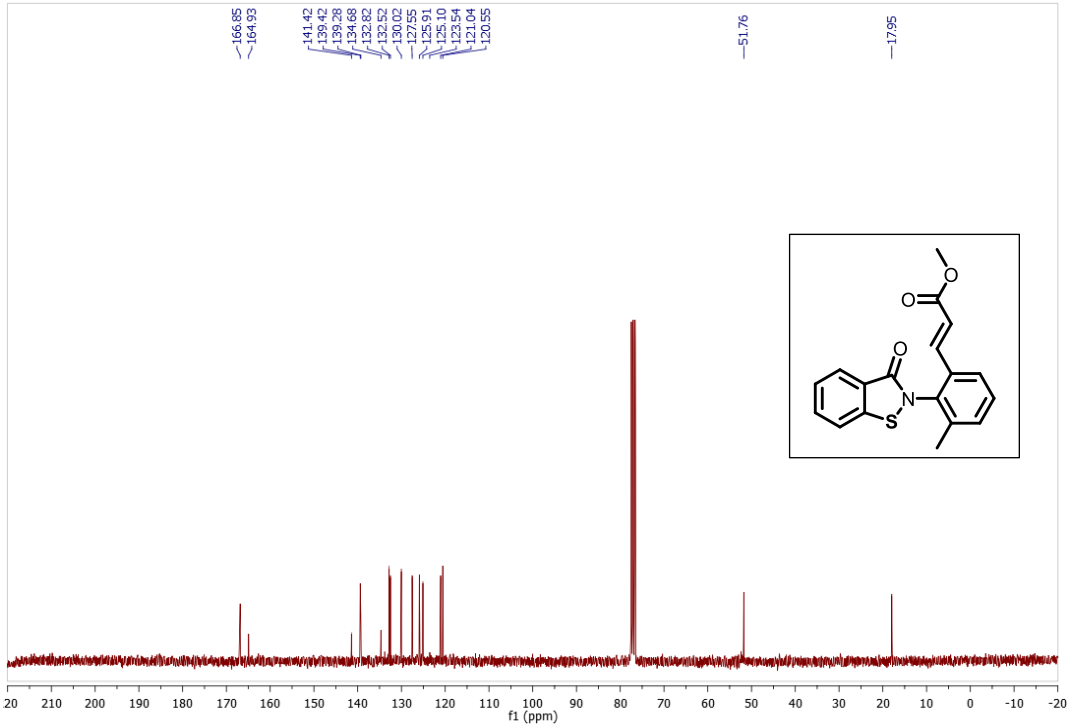
^{13}C-NMR of Compound 5b



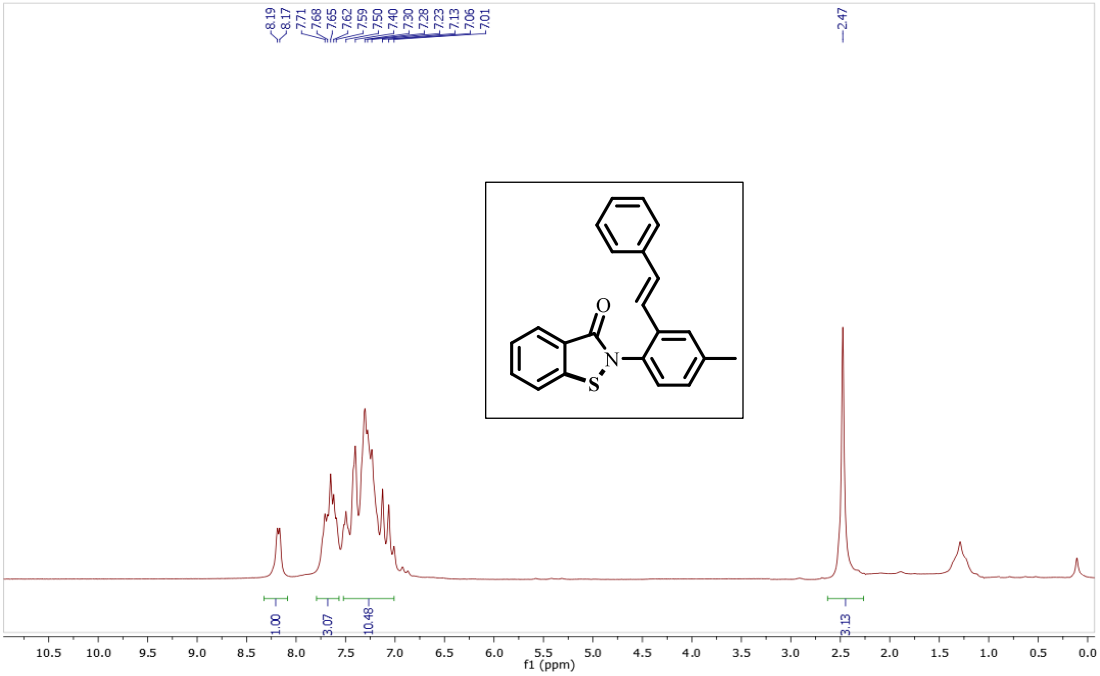
^{1}H-NMR of Compound 5c



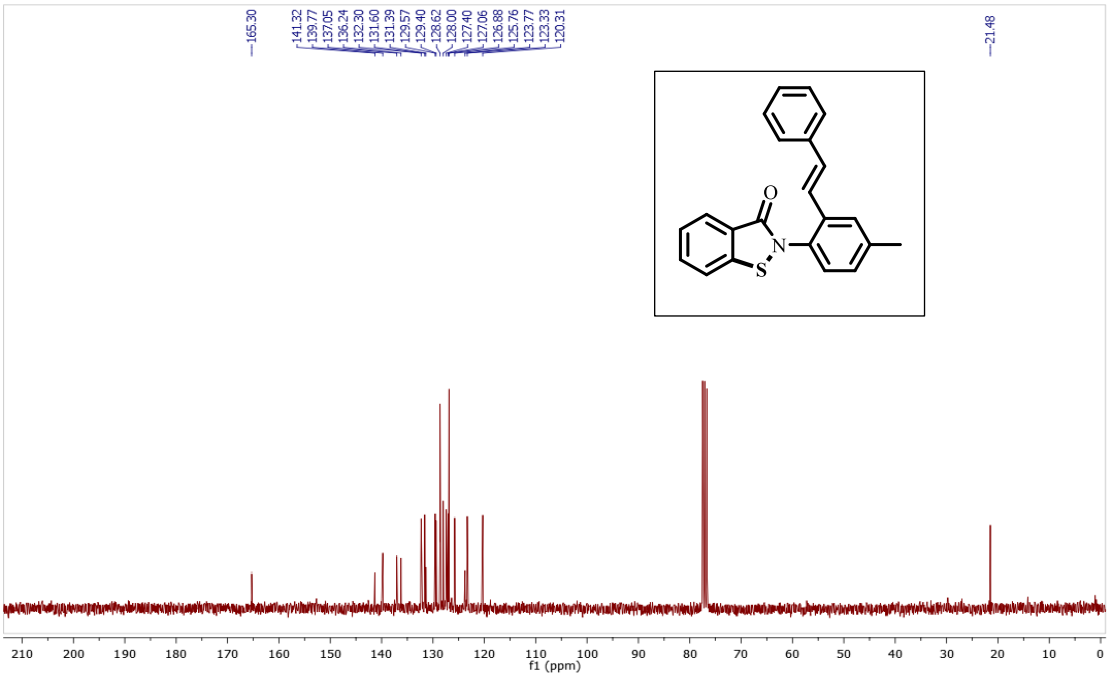
^{13}C-NMR of Compound 5c



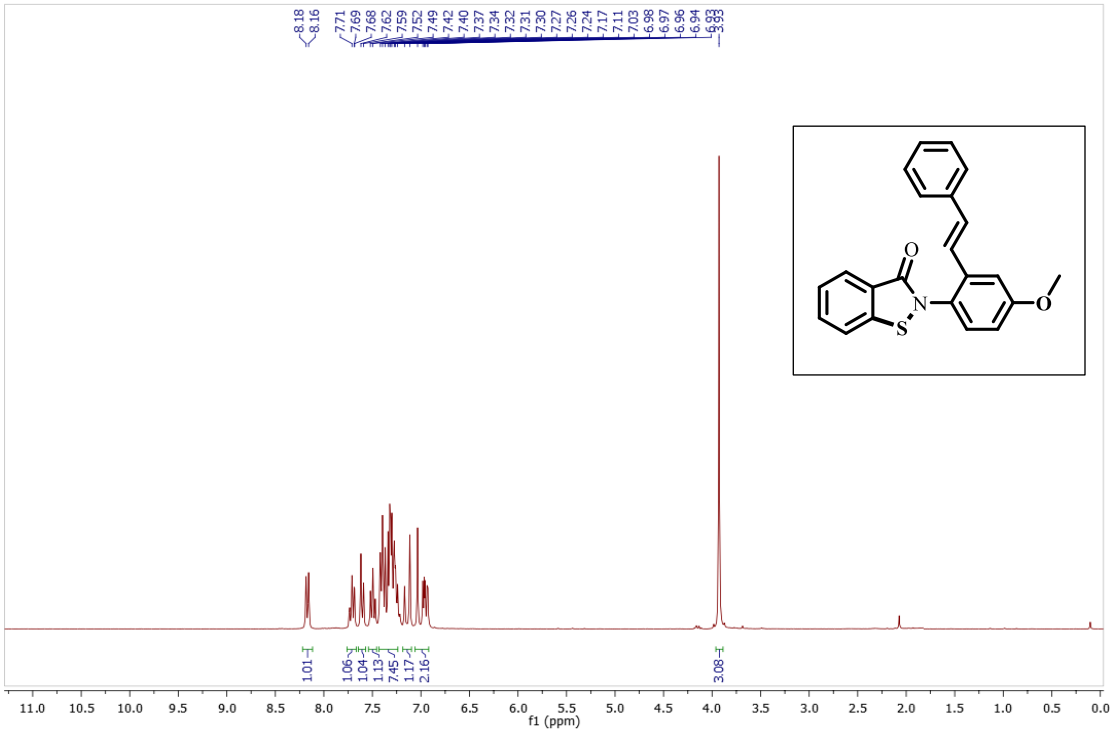
^{1}H-NMR of Compound 5d



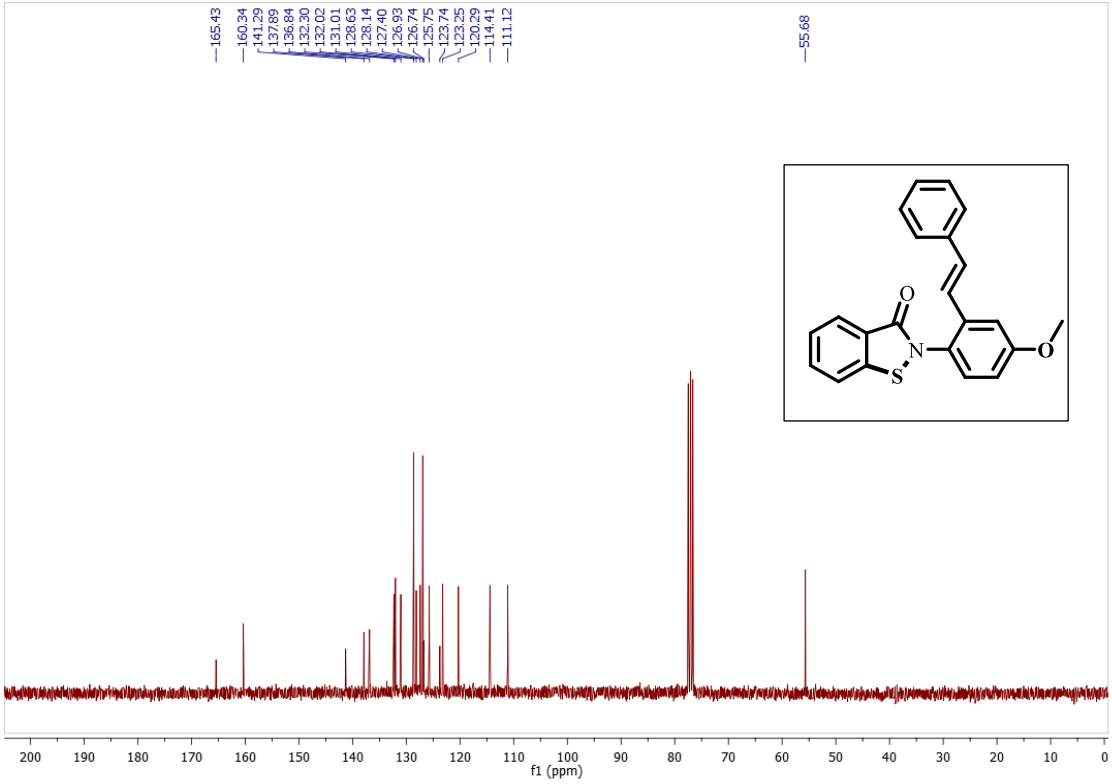
^{13}C-NMR of Compound 5d



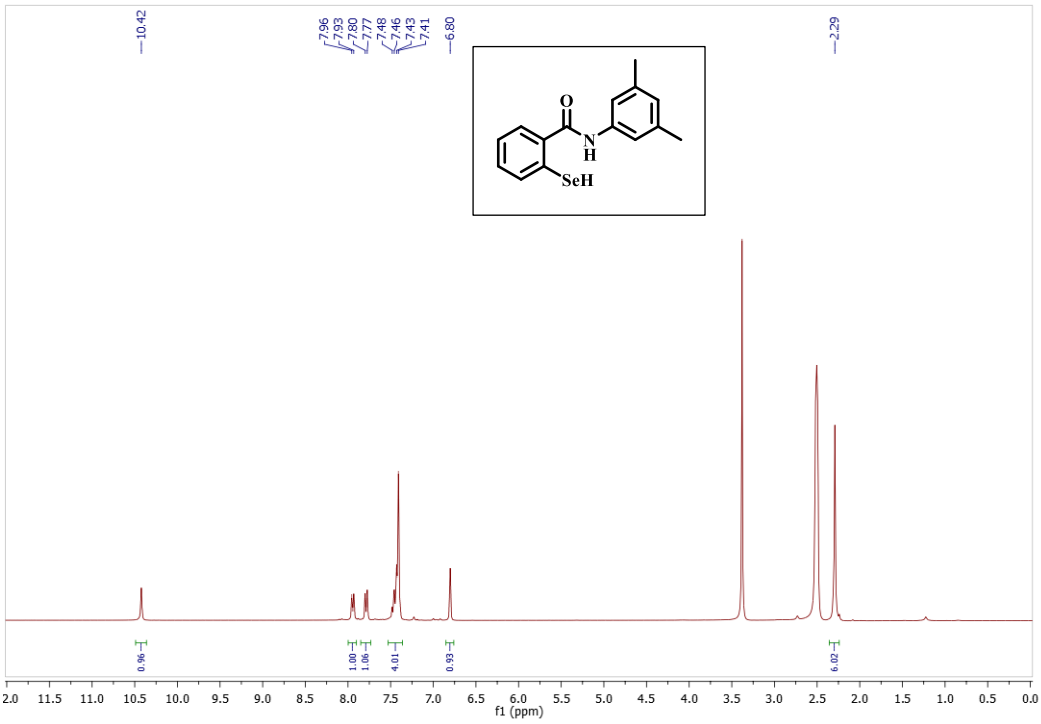
^{1}H-NMR of Compound 5e



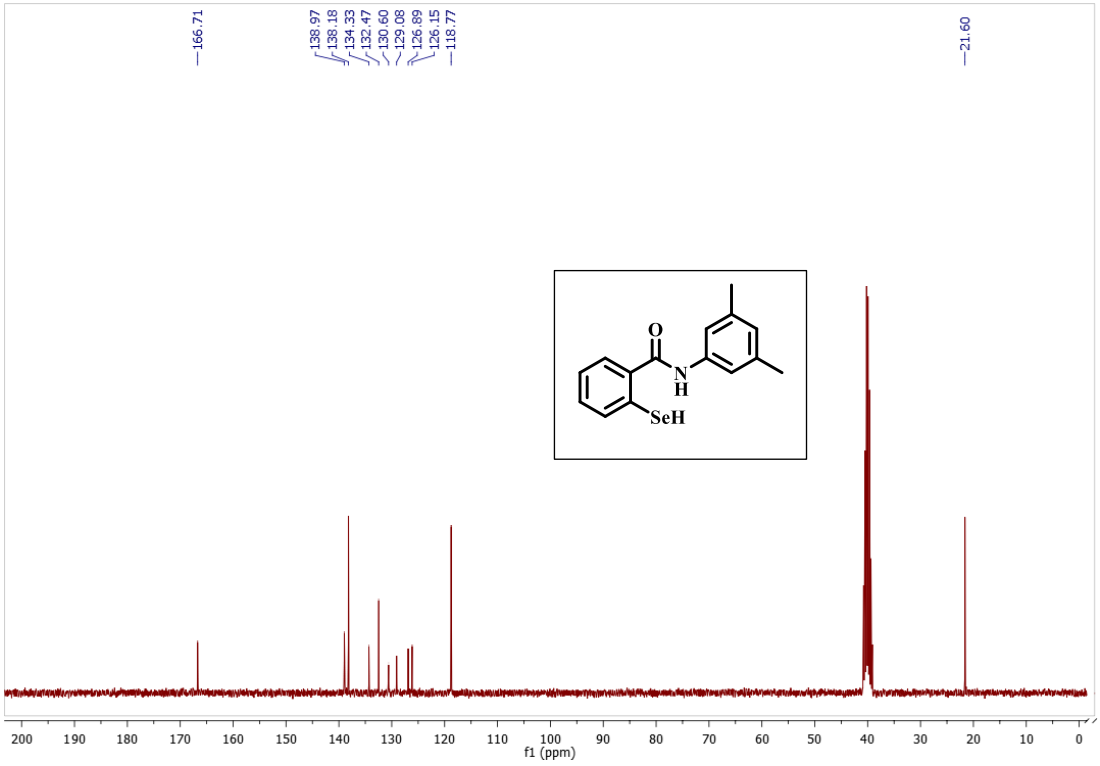
^{13}C-NMR of Compound 5e



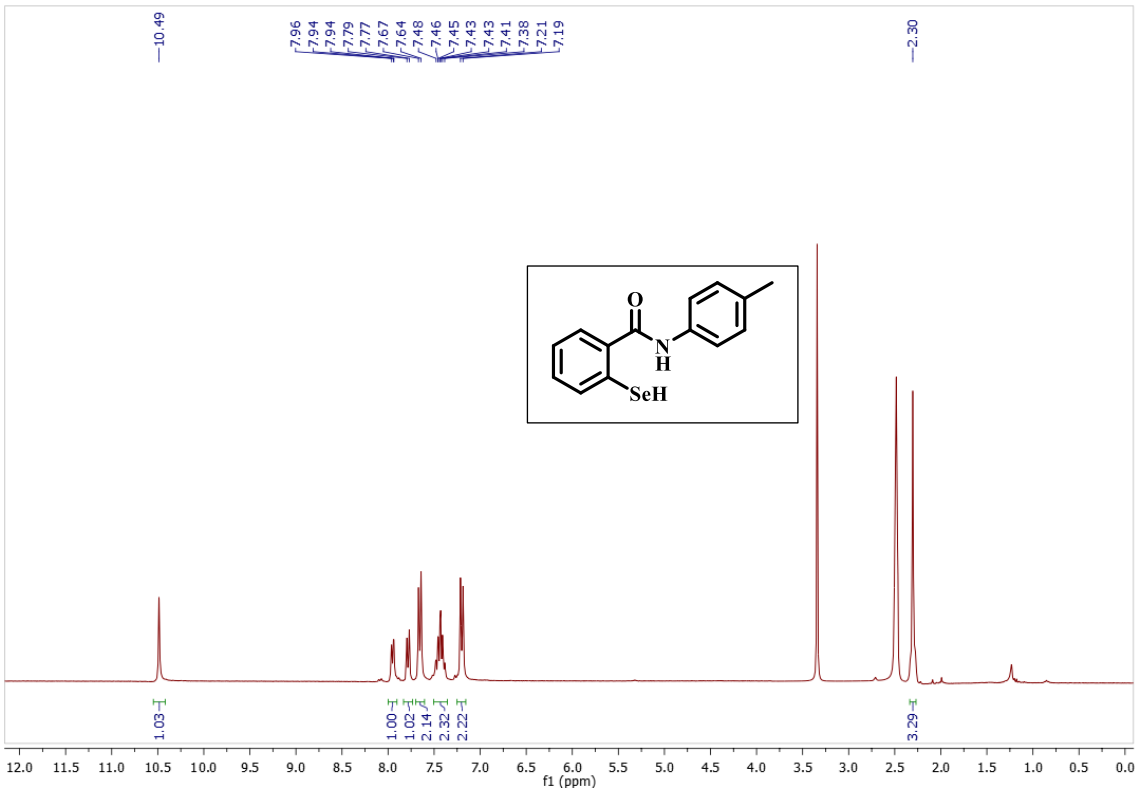
[1]H-NMR of Compound 6a



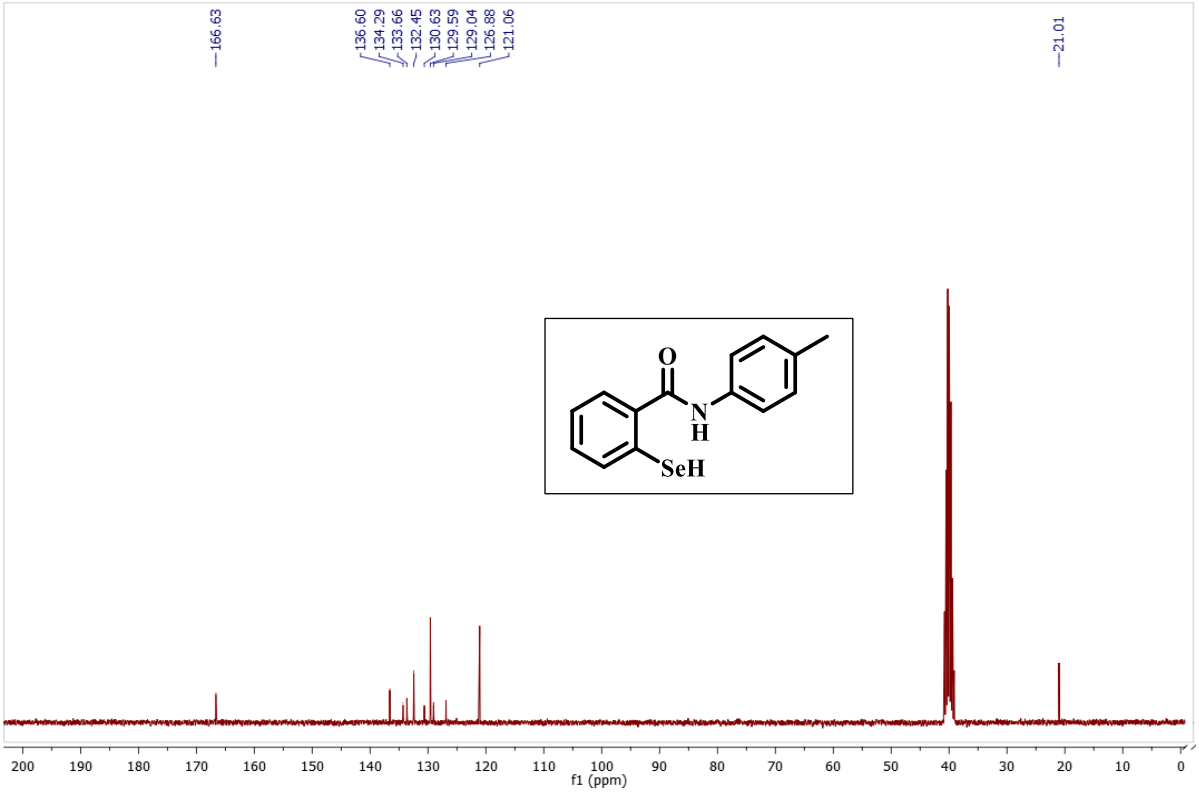
[13]C-NMR of Compound 6a



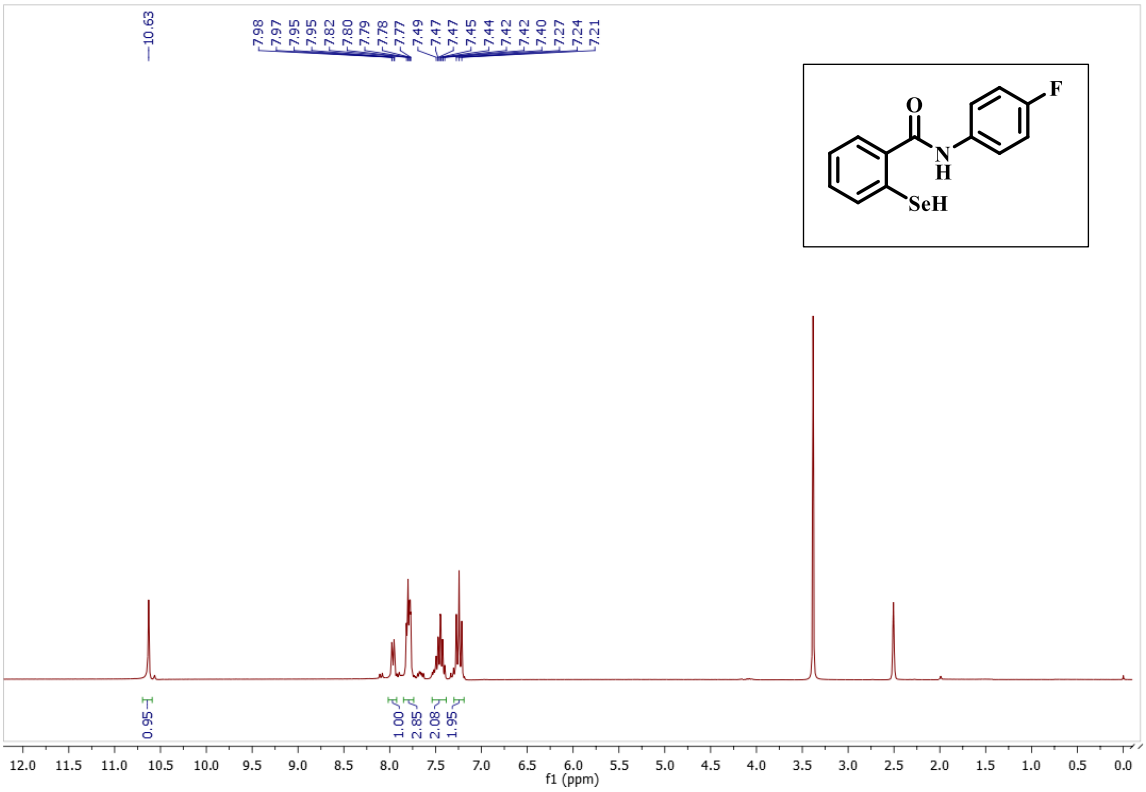
[1]H-NMR of Compound 6b



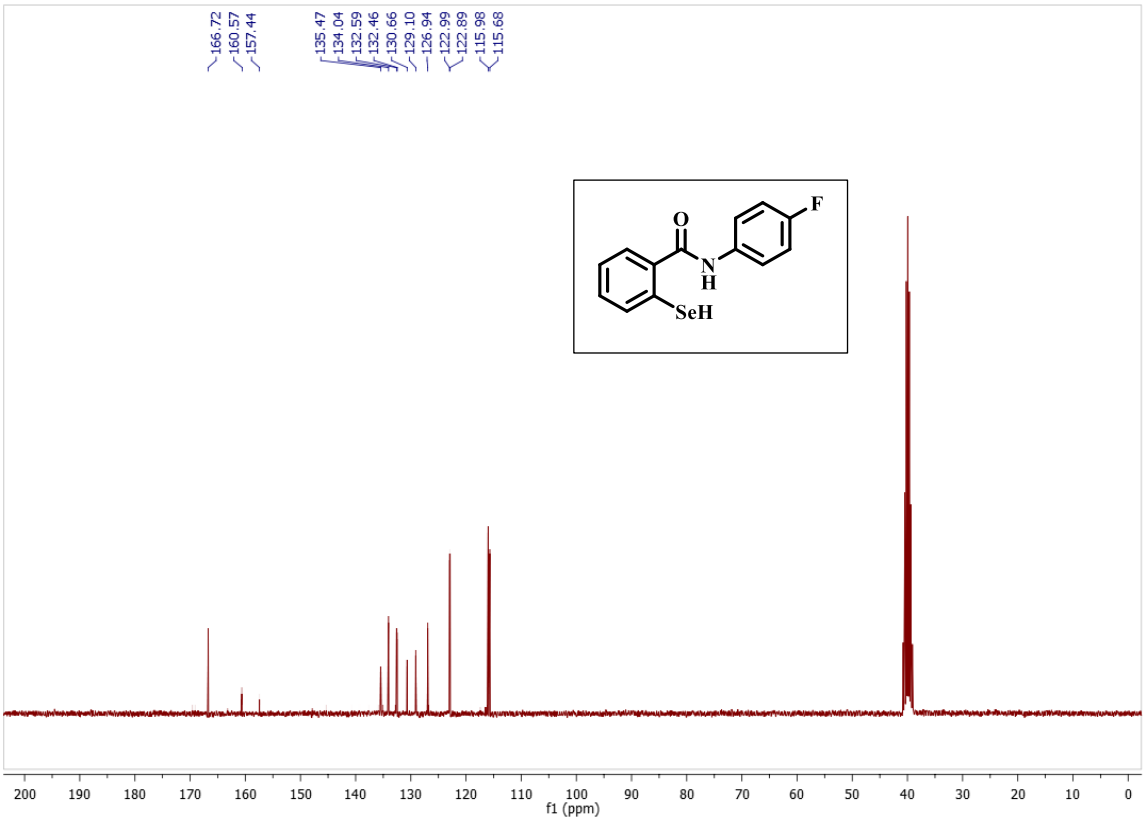
[13]C-NMR of Compound 6b



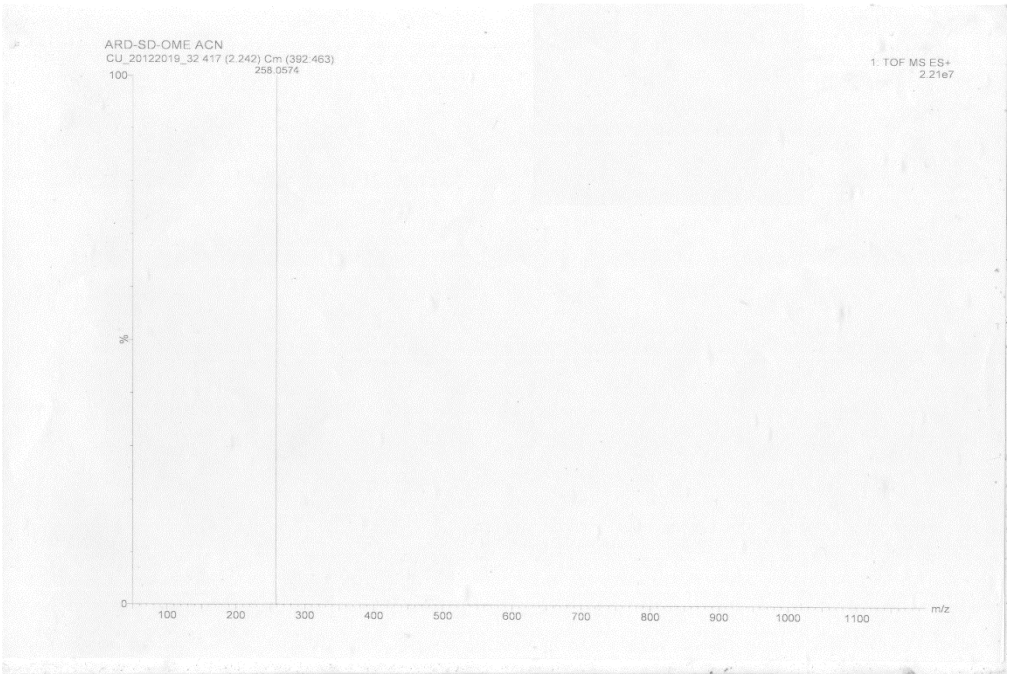
[1]H-NMR of Compound 6c



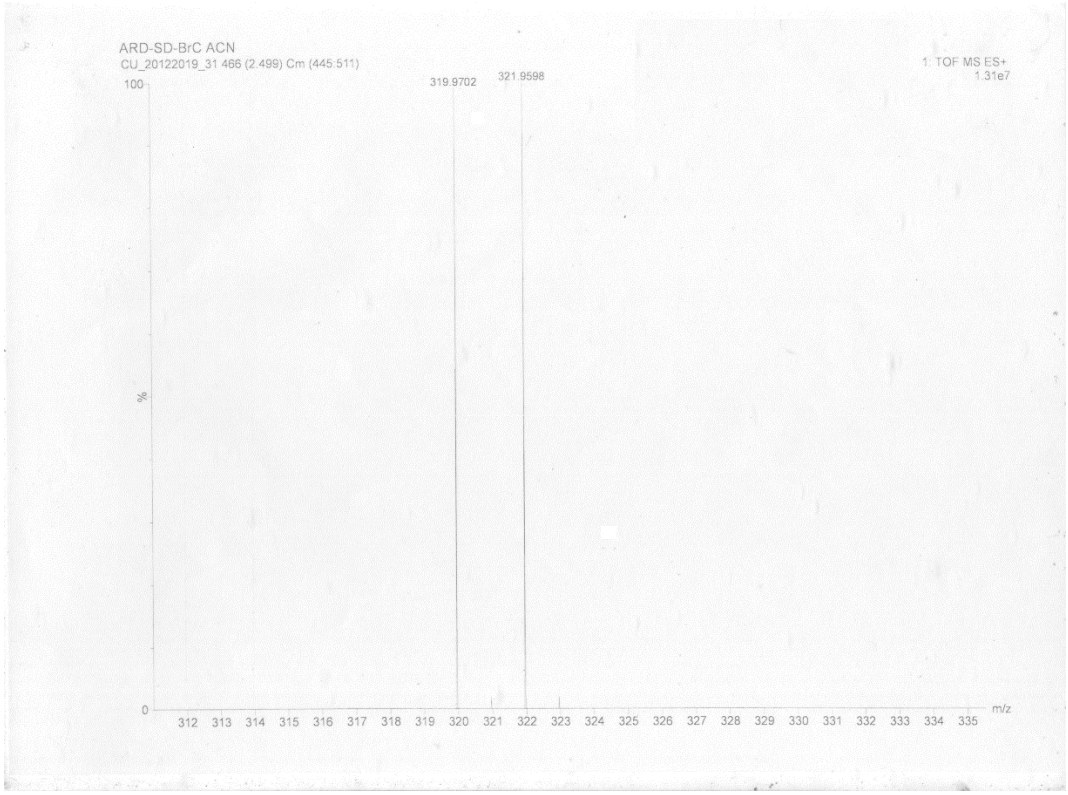
[13]C-NMR of Compound 6c



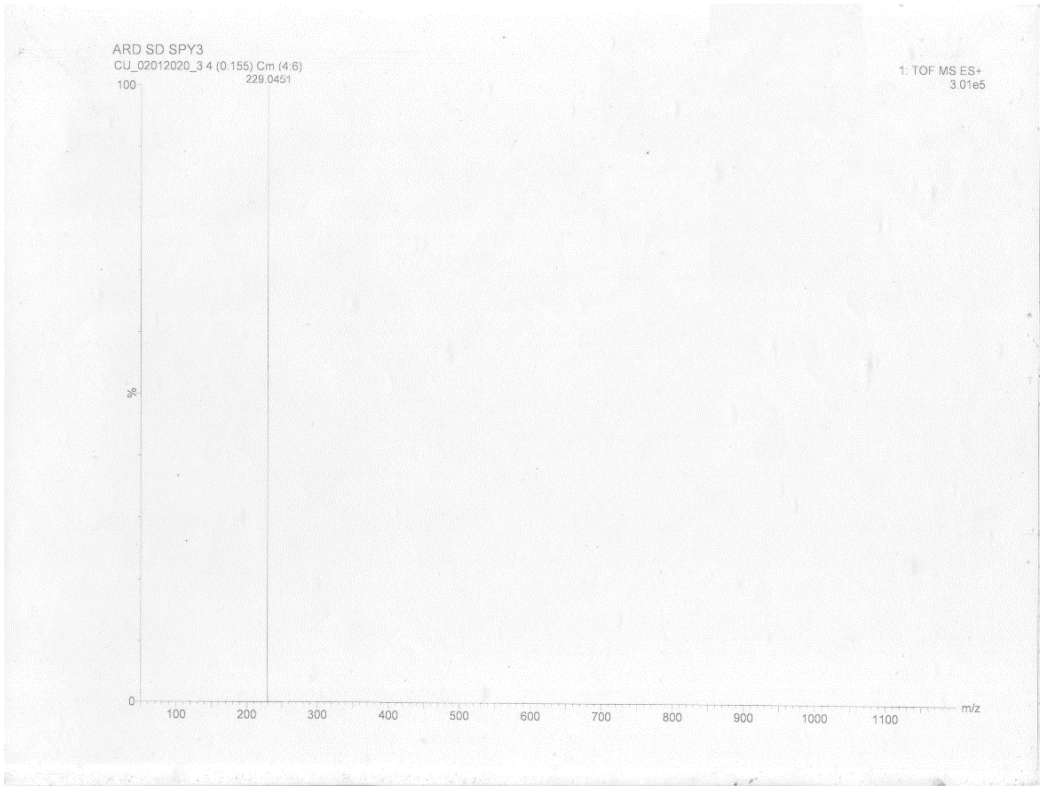
HRMS Spectra 2-(4-methoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one (2f) [$C_{14}H_{11}N_2OS$ $+H^+$]:



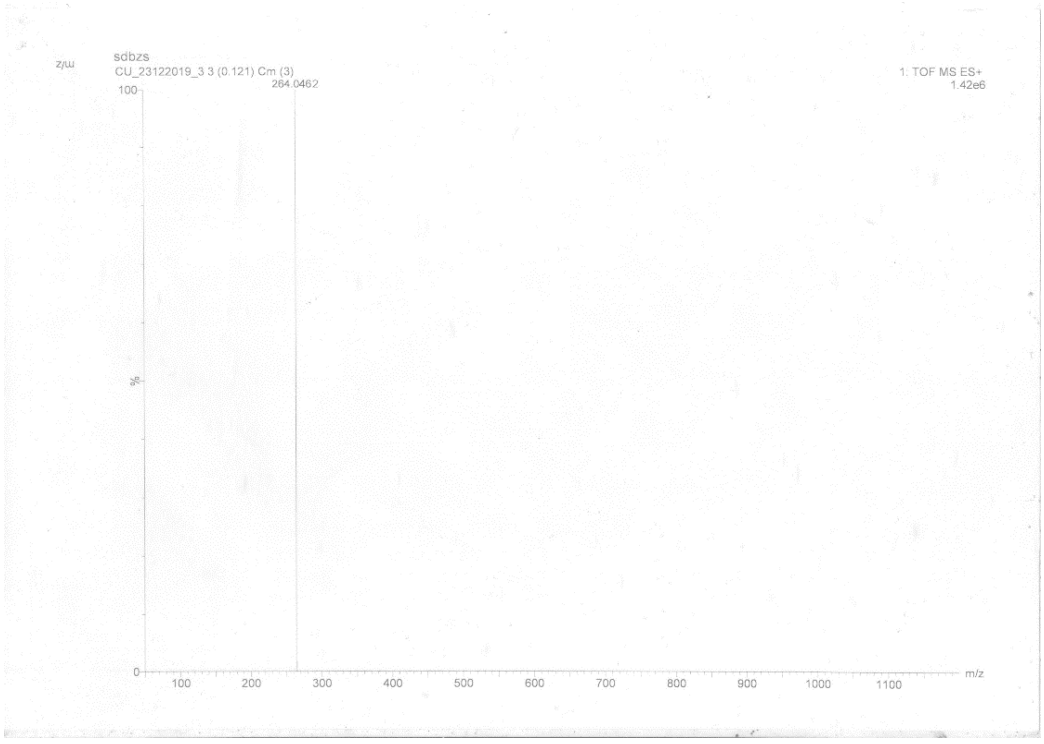
HRMS Spectra 2-(4-bromo-3-methylphenyl)benzo[*d*]isothiazol-3(2*H*)-one (2j) [$C_{14}H_{10}BrNOS$ $+H^+$]:



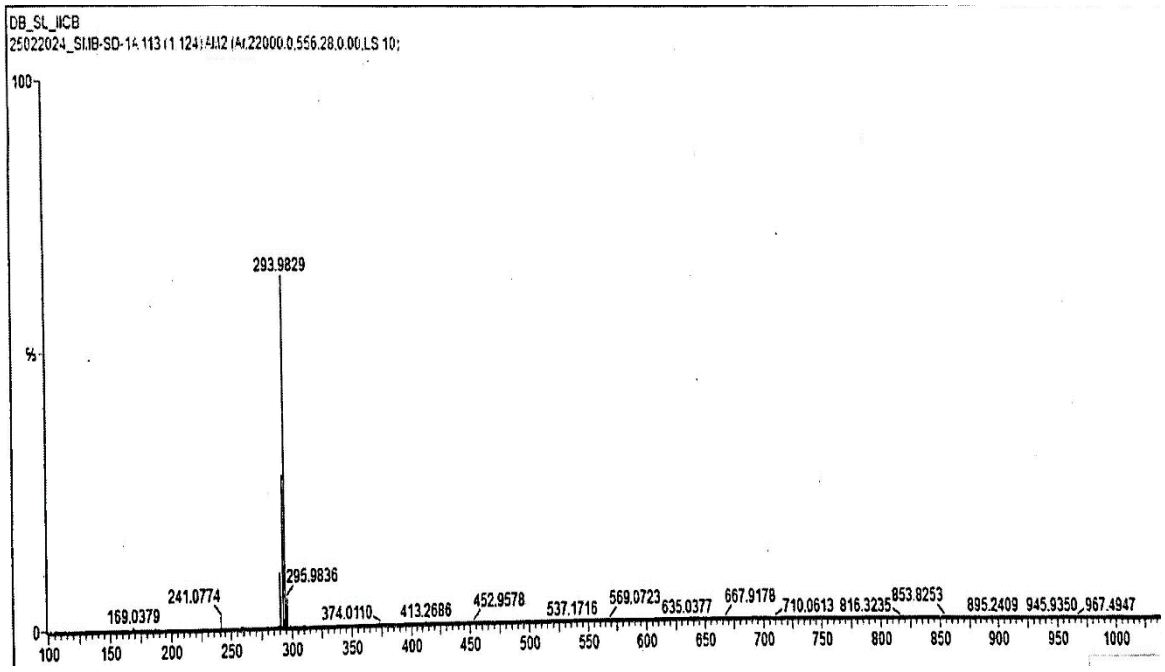
HRMS Spectra 2-(pyridin-3-yl)benzo[*d*]isothiazol-3(2*H*)-one (2n) [$C_{12}H_8N_2OS + H^+$]:



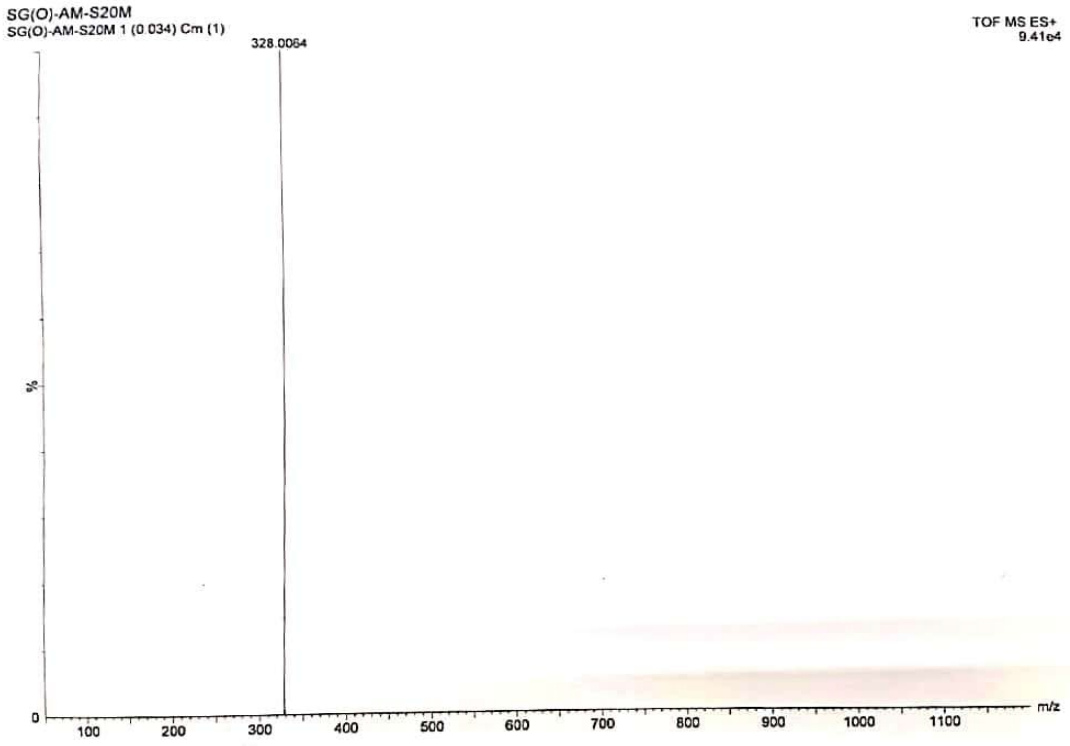
HRMS Spectra of 2-benzylbenzo[*d*]isothiazol-3(2*H*)-one (2u) [$C_{14}H_{11}NOS + Na^+$]:



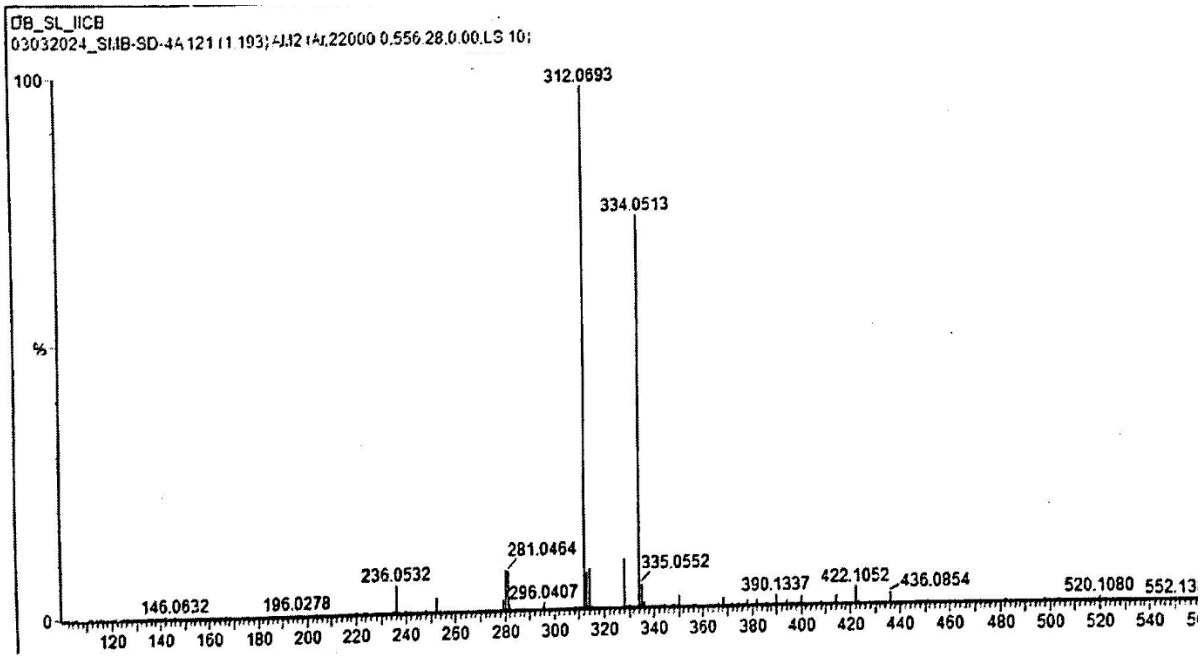
HRMS Spectra of 2-(4-fluorophenyl)benzo[*d*][1,2]selenazol-3(2*H*)-one (3c) [$C_{13}H_9FNOSe + H^+$]:



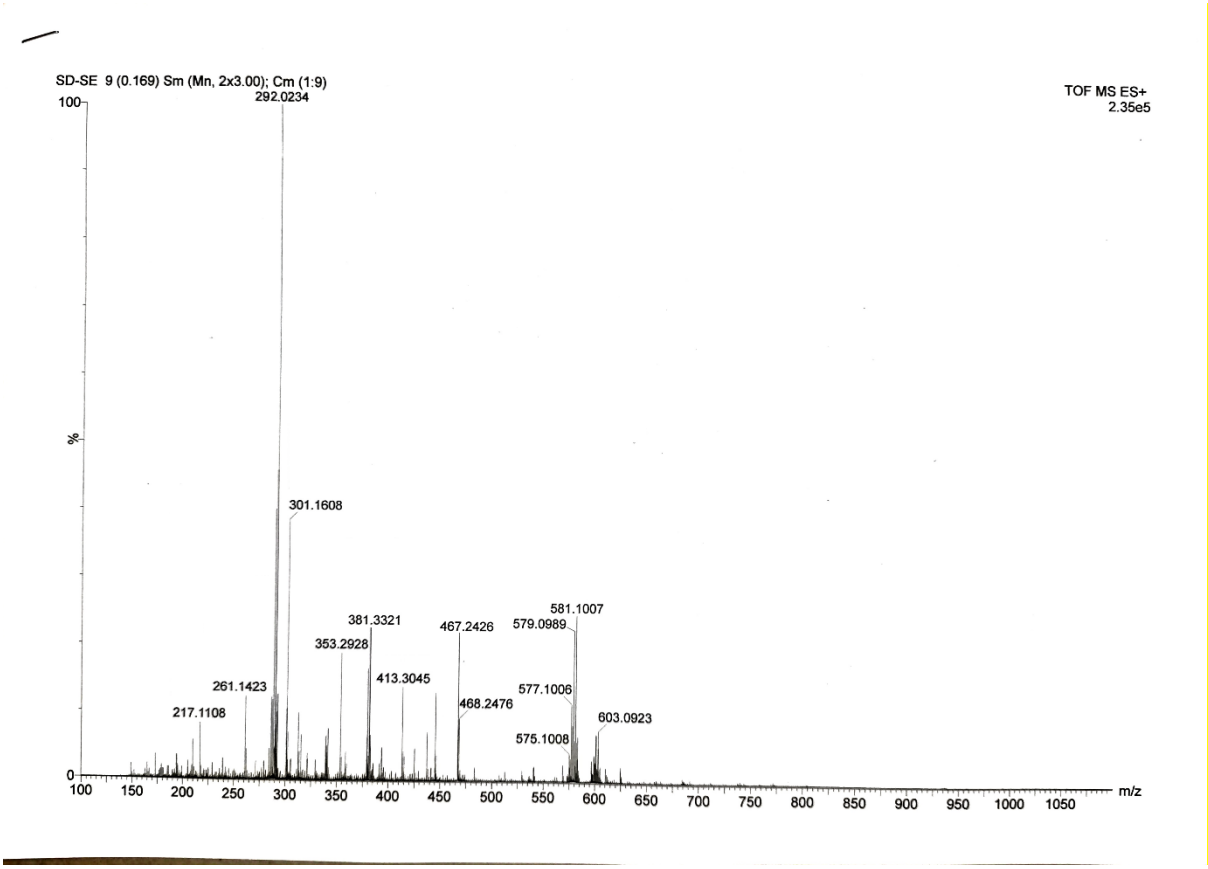
HRMS Spectra of 2-(4-methoxyphenyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4d) [$C_{14}H_{11}NO_4S + K^+$]:

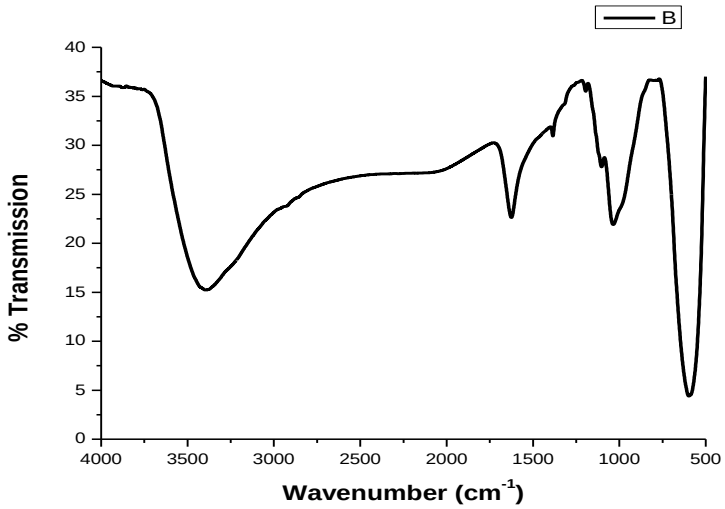


HRMS Spectra of Methyl (*E*)-3-(2-(3-oxobenzo[*d*]isothiazol-2(3*H*)-yl)phenyl)acrylate (5a) [$C_{17}H_{13}NO_3S+H^+$]:

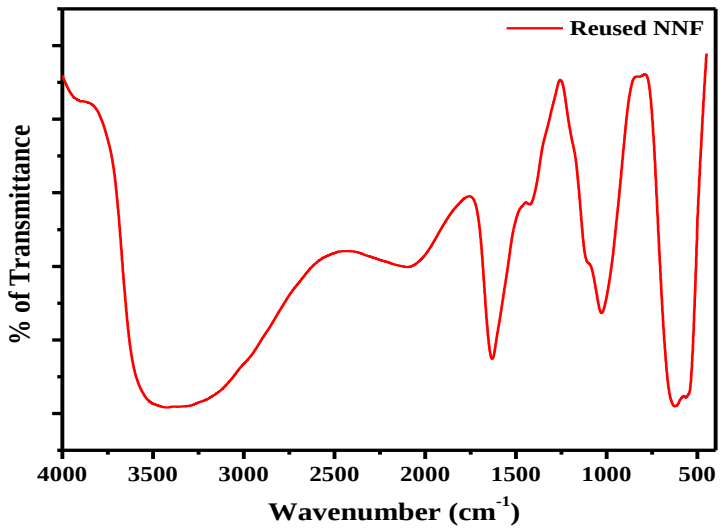


HRMS Spectra of 2-hydroseleno-*N*-(*p*-tolyl)benzamide (6b) [$C_{14}H_{13}NOSe+H^+$]:

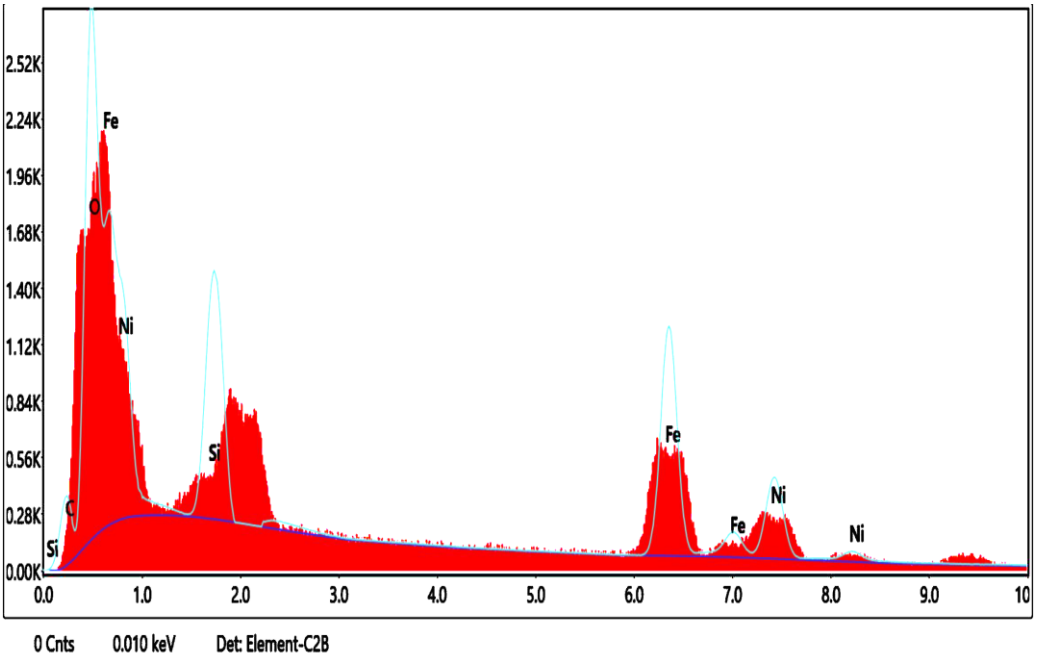




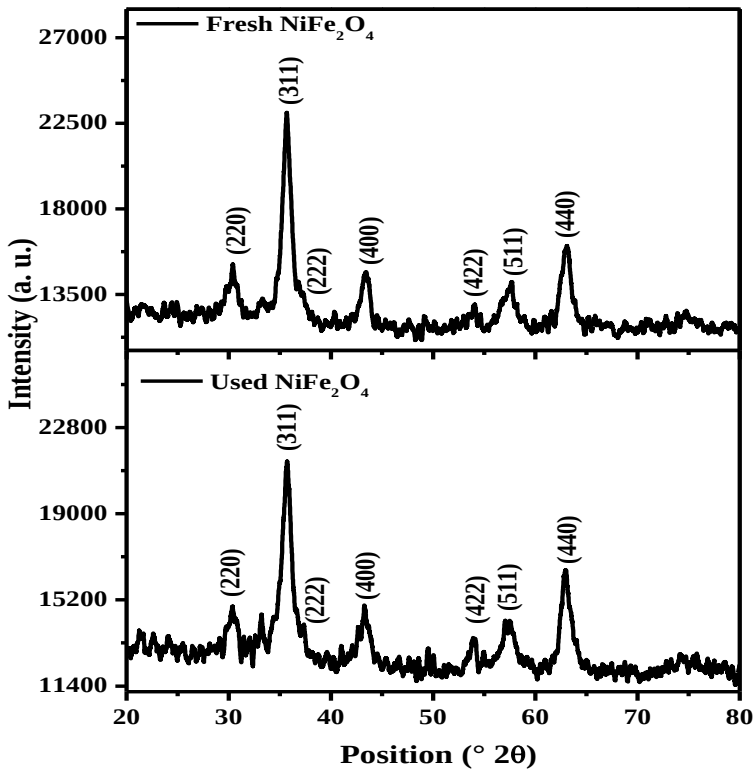
FT-IR Spectra of freshly prepared Nano nickel ferrite (Figure S1)



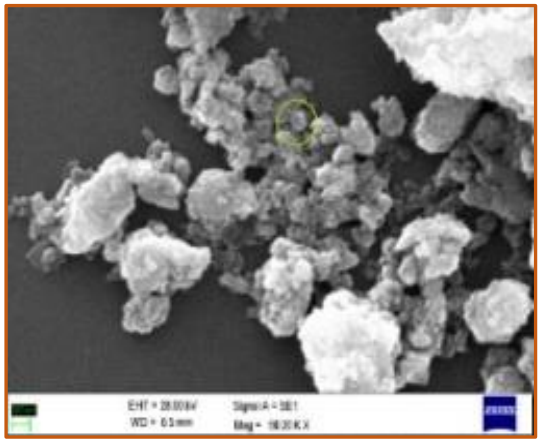
FT-IR spectra of Nano nickel ferrite after 5th run (Figure S2)



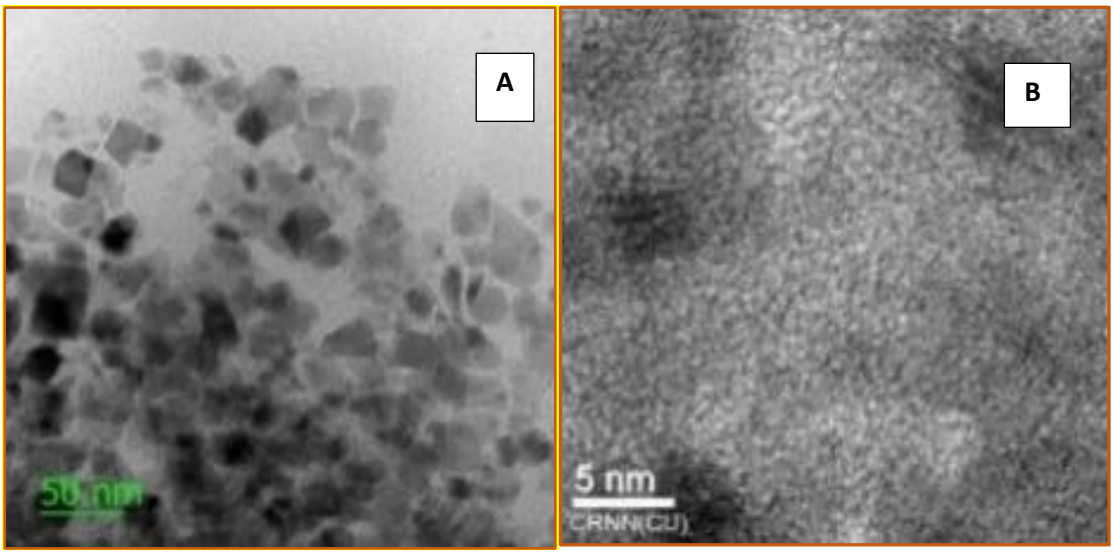
EDAX spectra of the Nano nickel ferrite (Figure S3)



Comparative X-ray Diffraction pattern of freshly prepared Nano nickel ferrite and after 5th use. (Figure S4)

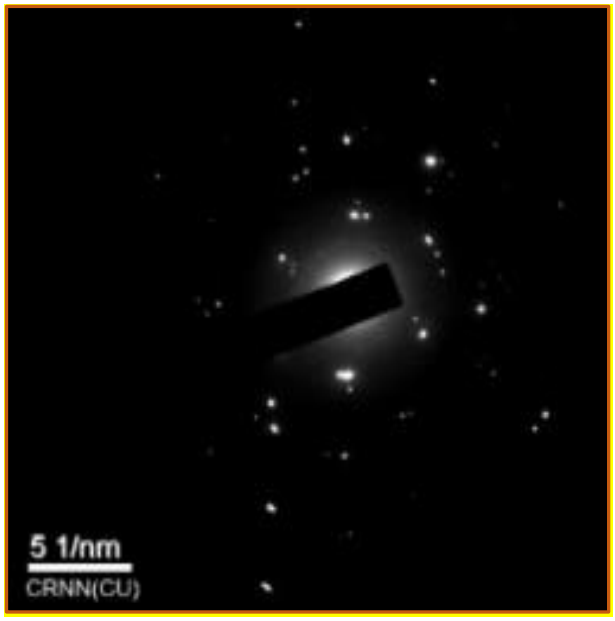


SEM image of the freshly prepared Nano nickel ferrite (Figure S5)

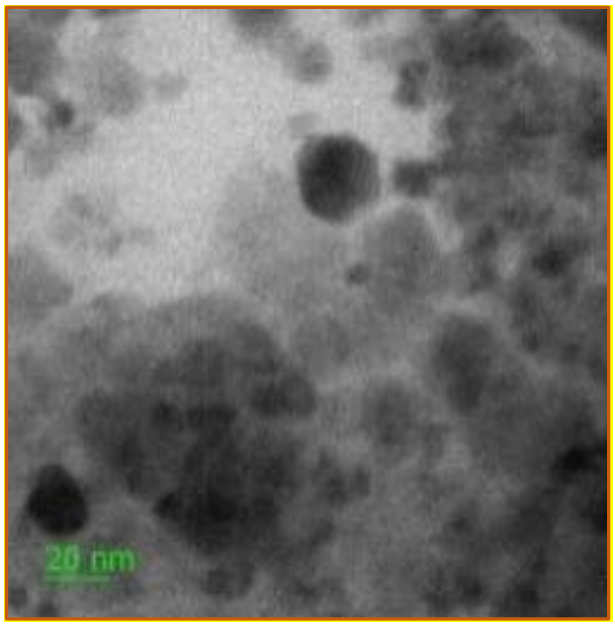


TEM (A) and HRTEM (B) image of the freshly prepared Nano nickel ferrite

(Figure S6)



SAED micro graph of freshly prepared Nano nickel ferrite (Figure S7)



HRTEM image of the Nano nickel ferrite after 5th run (Figure S8)

The ICP-AES analysis data:

Instrument: ARCOS 130MV

Metal ion	Composition (%)
Ni	17.167
Fe	35.505
Cu	0.000
Pd	0.000

Table S1: The ICP-AES analysis of reaction mixture after half the reaction