Synthesis of 2-Acyl-Benzo[1,3-*d*]selenazoles *via* domino oxidative cyclization of Methyl Ketones with Bis(2-Aminophenyl) Diselenide

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SUPPLEMENTARY MATERIAL

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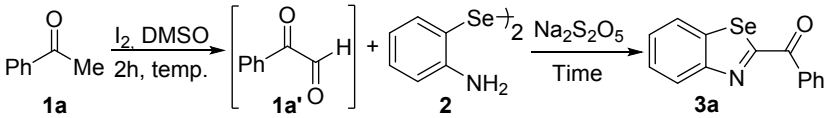
General Information

The reactions were monitored by TLC carried out on Merck silica gel (60 F254) by using UV light as visualizant agent and 5% vanillin in 10% H_2SO_4 and heat as developing agents. Baker silica gel (particle size 0.040-0.063 mm) was used for flash chromatography. Proton nuclear magnetic resonance spectra (^{1}H NMR) were obtained at 300 MHz on Bruker DPX 300 spectrometer or at 400 MHz on Bruker Avance 400 III. Spectra were recorded in DMSO-d_6 or $CDCl_3$ solutions. Chemical shifts are reported in ppm, referenced to tetramethylsilane (TMS) as the external reference. Coupling constants (*J*) are reported in Hertz. Abbreviations to denote the multiplicity of a particular signal are s (singlet), d (doublet), dd (doublet of doublet), t (triplet), td (triplet of doublet) and m (multiplet). Carbon-13 nuclear magnetic resonance spectra (^{13}C NMR) were obtained at 75 MHz on Bruker DPX 300 spectrometer or at 100 MHz on Bruker Avance 400 III. Chemical shifts are reported in ppm, referenced to the solvent peak of DMSO-d_6 or $CDCl_3$. Low-resolution mass spectra were obtained with a Shimadzu GC-MS-QP2010 mass spectrometer. High resolution mass spectra (HRMS) were recorded on a Bruker Micro TOF-QII spectrometer 10416. The reactions under were conducted using a CEM Discover, mode operating systems working at 2.45 GHz, with a Power programmable from 1 to 300 W.

Optimization of the Reaction Conditions

In order to obtain the best reaction condition, several reactions were performed by varying stoichiometry, time, temperature, the use of inert and non-inert atmosphere, and the presence or absence of $Na_2S_2O_5$ as reducing agent. These results are presented in Table S1 for conventional heating and in Table S2 for reaction performed under microwave irradiation.

Table S1. Optimization of the reaction conditions under conventional heating.[a]

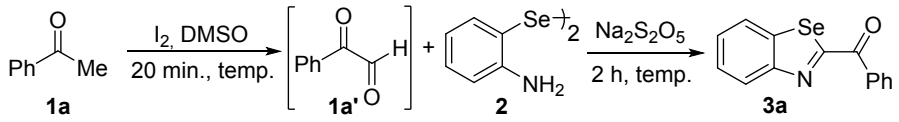


Entry	**1a** (mmol)	I_2 (mmol)	Time (h)	Temp. (°C)	Yield of **3a** (%)
1	0.50	0.55	24	120	44
2[b]	0.50	0.55	24	120	42
3	0.50	0.55	48	120	74
4[c]	0.50	0.55	48	120	-
5	0.70	0.70	48	120	43
6	0.70	0.70	48	100	66
7	0.50	0.55	48	100	55
8[d]	0.70	0.70	48	100	80
9[d]	0.50	0.55	48	100	58

10[d]	0.70	0.70	48	80	35
11[d]	0.70	0.70	36	100	45

[a] Reaction was performed using **1a** and molecular iodine (I_2), in DMSO (1.5 mL) for 2 h under N_2 atmosphere, followed by addition of **2** (0.25 mmol) and $Na_2S_2O_5$ (0.5 mmol). [b] Reaction using molecular sieve (4Å). [c] Multicomponent reaction. [d] Reaction was performed in air.

Table S2. Optimization of the reaction conditions under microwave irradiation.[a]



Entry	**1a** (mmol)	I_2 (mmol)	$Na_2S_2O_5$ (mmol)	Temp. (°C)	Yield of **3a** (%)
1	0.50	0.55	0.50	120	60
2	0.70	0.70	0.50	120	74
3	1.00	1.00	0.50	120	44
4	0.50	0.55	0.00	120	62
5	0.70	0.70	0.00	120	60
6	0.70	0.70	0.50	150	36
7[b]	0.70	0.70	0.50	120	70
8[b]	1.00	1.00	0.50	120	74
9[b]	0.50	0.55	0.50	120	63
10[b]	0.50	0.55	0.00	120	51
11[b]	0.50	0.55	0.25	120	58
12[b]	0.50	0.55	0.75	120	32
13[b]	0.70	0.70	0.50	100	86
14[b]	0.70	0.70	0.50	80	75

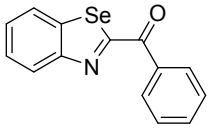
[a] Reaction was performed using **1a** and molecular iodine (I_2), in DMSO (1.5 mL) for 20 min. under N_2 atmosphere under focused MW irradiation (200 W), followed by addition of **2** (0.25 mmol) and $Na_2S_2O_5$ (0.5 mmol). [b] Reaction was performed in air.

General Procedure for the Synthesis of 2-acyl-benzo[1,3-*d*]selenazole 3a-l under Conventional Heating

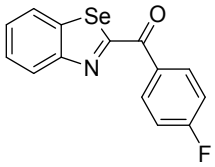
In a round-bottom flask 25 mL equipped with a magnetic stir bar, the aryl methyl ketone **1a-l** (0.70 mmol) was dissolved in DMSO (1.5 mL) and molecular iodine (0.7 mmol) was added. The reaction mixture was left to stir at 100 °C for about 2 hours (to *in situ* formation of 2-arylethan-1,2-dione **1'a-l**).[1] After this, bis(2-aminophenyl) disselenide **2** (0.25 mmol) and sodium metabisulfite (0.50 mmol) were added, and the reaction was maintained for 48 hours at 100 °C. After this time, the reaction mixture was cooled to room temperature, quenched with saturated solution of $Na_2S_2O_3$ (20 mL) and the reaction was extracted with ethyl acetate (3x 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was purified by chromatography on silica gel using hexane as eluent to provide **3a-l**.

General Procedure for the Synthesis of 2-acyl-benzo[1,3-*d*]selenazole 3a-l under Microwave Irradiation

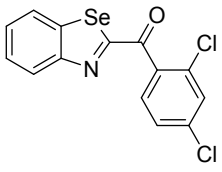
In a 10 mL glass vial equipped with a magnetic stir bar, the aryl methyl ketone **1a-l** (0.70 mmol) was dissolved in DMSO (1.5 mL) and molecular iodine (0.7 mmol) was added. The reaction mixture was left to stir at 100 °C (measured with an IR sensor on the outer surface of the reaction vial) for about 20 minutes (to *in situ* formation of 2-arylethan-1,2-dione **1'a-l**) under microwave irradiation (irradiation power of 200 W and the ramp temperature rate was 3 min). After this, bis(2-aminophenyl) disselenide **2** (0.25 mmol) and sodium metabisulfite (0.50 mmol) were added, and the reaction was maintained for 2 hours at 100 °C. After this time, the reaction mixture was cooled to room temperature, quenched with saturated solution of $Na_2S_2O_3$ (20 mL) and the reaction was extracted with ethyl acetate (3x 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was purified by chromatography on silica gel using hexane as eluent to provide **3a-l**.



2-(phenylmethanone)benzo[1,3-*d*]selenazole (3a): Yield: 0.115 g (80% - Conventional heating); 0.123 g (86% - Microwave Irradiation); yellow solid; mp 94-96 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30 (d, J = 7.9 Hz, 2H), 7.02 (d, J = 7.9 Hz, 1H), 6.76 (d, J = 7.9 Hz, 1H), 6.41-6.37 (m, 1H), 6.31-6.27 (m, 3H), 6.20-6.16 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 186.1, 173.2, 155.9, 140.8, 134.4, 133.8, 131.3, 128.4, 127.4, 127.3, 126.7, 125.3. MS *m/z* (relative intensity): 287 ($M^{+\cdot}$) (14), 259 (12), 105 (100), 77 (53), 51 (13). HRMS calcd. for $C_{14}H_{10}NOSe$: $[M+H]^+$ 287.9922. Found: 287.9934.

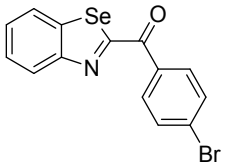


2-(4-fluorophenylmethanone)benzo[1,3-*d*]selenazole (3b): Yield: 0.106 g (70% - Conventional heating); 0.121 g (80% - Microwave Irradiation); yellow solid; mp 123-125 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.65 (dd, J = 8.8, 5.6 Hz, 2H), 8.27 (d, J = 8.0 Hz, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.56 (td, J = 8.0, 0.9 Hz, 1H), 7.45 (td, J = 8.0, 0.9 Hz, 1H), 7.25-7.19 (t, J = 8.8 Hz, 2H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 184.3, 173.2, 166.3 (d, J = 256.7 Hz), 155.8, 140.8, 134.2 (d, J = 9.4 Hz), 130.6 (d, J = 2.9 Hz), 127.4, 127.4, 126.8, 125.3, 115.7 (d, J = 21.8 Hz). MS *m/z* (relative intensity): 305 (M^+) (6), 124 (8), 123 (100), 95 (57), 75 (21), 40 (7). HRMS calcd. for $C_{14}H_{10}FNOSe$: $[M+H]^+$ 306.9906. Found: 306.9871.

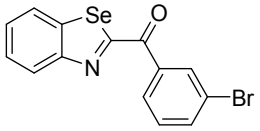


2-(2,4-dichlorophenylmethanone)benzo[1,3-*d*]selenazole (3c): Yield: 0.146 g (83% - Conventional heating); 0.152 g (86% - Microwave Irradiation); yellow solid; mp 141-143 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.20 (d, J = 8.0 Hz, 1H),

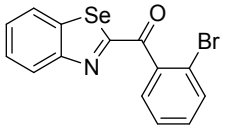
8.04 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 8.3 Hz, 1H), 7.56 -7.52 (m, 2H), 7.46 (td, *J* = 8.0, 1.0 Hz, 1H), 7.41 (dd, *J* = 8.0, 1.9 Hz, 1H). ^{13}C NMR (75.5 MHz, $CDCl_3$) *δ*: 187.4, 171.5, 155.6, 141.2, 138.0, 133.8, 133.8, 132.0, 130.5, 127.8, 127.7, 127.0, 126.8, 125.5. MS *m/z* (relative intensity): 320 (19), 175 (65), 173 (100), 145 (29), 109 (21). HRMS calcd. for $C_{14}H_{10}Cl_2NOSe$: $[M+H]^+$ 355.9148. Found: 355.9146.



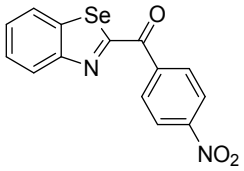
2-(4-bromophenylmethanone)benzo[1,3-*d*]selenazole (3d): Yield: 0.160 g (88% - Conventional heating); 0.161 g (89% - Microwave Irradiation); orange solid; mp 94-96 °C. ^{1}H NMR (300 MHz, $CDCl_3$) *δ*: 8.46 (d, *J* = 8.6 Hz, 2H), 8.28 (d, *J* = 8.1 Hz, 1H), 8.04 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 8.6 Hz, 2H), 7.57 (td, *J* = 8.1, 1.0 Hz, 1H), 7.46 (td, *J* = 8.1, 1.0 Hz, 1H). ^{13}C NMR (75.5 MHz, $CDCl_3$) *δ*: 185.0, 172.9, 155.8, 140.9, 133.1, 132.8, 131.8, 129.4, 127.6, 127.5, 126.9, 125.3. MS *m/z* (relative intensity): 365 (M^+) (17), 185 (91), 183 (100), 155 (43), 76 (46), 75(44). HRMS calcd. for $C_{14}H_{10}BrNOSe$: $[M+H]^+$ 366.9105. Found: 366.9083.



2-(3-bromophenylmethanone)benzo[1,3-*d*]selenazole (3e): Yield: 0.131 g (72% - Conventional heating); 0.151 g (83% - Microwave Irradiation); yellow solid; mp 98-100 °C. ^{1}H NMR (300 MHz, $CDCl_3$) *δ*: 8.68 (t, *J* = 1.6 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.29 (d, *J* = 8.0 Hz, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.78-7.76 (m, 1H), 7.56 (td, *J* = 8.0, 1.0 Hz, 1H), 7.48-7.40 (m, 2H). ^{13}C NMR (75.5 MHz, $CDCl_3$) *δ*: 184.6, 172.4, 155.8, 140.9, 136.6, 136.1, 134.0, 130.0, 129.9, 127.6 (2C), 126.9, 125.3, 122.5. MS *m/z* (relative intensity): 365 (M^+) (18), 185 (95), 183 (100), 155 (52), 76 (61), 75 (56). HRMS calcd. for $C_{14}H_9BrNOSe$: $[M]^+$ 365.9032. Found: 365.9024.

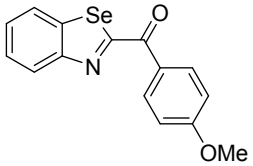


2-(2-bromophenylmethanone)benzo[1,3-*d*]selenazole (3f): Yield: 0.171 g (94% - Conventional heating); 0.171 g (94% - Microwave Irradiation); yellow solid; mp 96-98 °C. ^{1}H NMR (300 MHz, $CDCl_3$) *δ*: 8.19 (d, *J* = 8.0 Hz, 1H), 8.04 (d, *J* = 8.0 Hz, 1H), 7.73-7.69 (m, 2H), 7.55-7.39 (m, 4H). ^{13}C NMR (75.5 MHz, $CDCl_3$) *δ*: 189.3, 171.5, 155.6, 141.2, 137.6, 133.6, 132.3, 130.7, 127.7, 127.6, 126.9 (2C), 125.4, 120.6. MS *m/z* (relative intensity): 286 (51), 185 (94), 183 (100), 76 (48), 75 (50). HRMS calcd. for $C_{14}H_9BrNOSe$: $[M]^+$ 365.9032. Found: 365.9023.

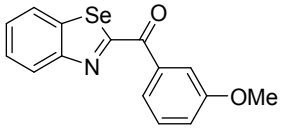


2-(4-nitrophenylmethanone)benzo[1,3-*d*]selenazole (3g): Yield: 0.091 g (55% - Conventional heating); 0.112 g (68% - Microwave Irradiation); yellow solid;

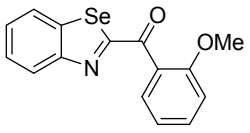
mp 174-176 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.73 (dt, J = 9.0, 2.0 Hz, 2H), 8.40 (dt, J = 9.0 Hz, J = 2.0 Hz, 2H), 8.33-8.30 (m, 1H), 8.09-8.06 (m, 1H), 7.64-7.58 (m, 1H), 7.54-7.48 (m, 1H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 184.7, 171.8, 155.7, 150.5, 141.0, 139.2, 132.3, 127.9, 127.7, 127.2, 125.4, 123.4. MS *m/z* (relative intensity): 332 (M^+) (13), 150 (100), 104 (59), 92 (34), 76 (56), 50 (25). HRMS calcd. for $C_{14}H_9N_2O_3Se$: $[M]^+$ 332.9778. Found: 332.9772.



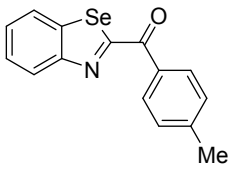
2-(4-methoxyphenylmethanone)benzo[1,3-*d*]selenazole (3h): Yield: 0.104 g (75% - Conventional heating); 0.118 g (75% - Microwave Irradiation); yellow solid; mp 108-110 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.64 (dt, J = 9.0, 2.1 Hz, 1H), 8.29-8.26 (m, 1H), 8.04-8.01 (m, 1H), 7.58-7.52 (m, 1H), 7.46-7.41 (m, 1H), 7.03 (dt, J = 9.0, 2.1 Hz, 1H), 3.91 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 184.1, 174.2, 164.3, 155.9, 140.6, 133.9, 127.2, 127.1, 127.0, 126.7, 125.3, 113.9, 55.6. MS *m/z* (relative intensity): 317 (M^+) (6), 135 (100), 107 (10), 92 (17), 77 (26), 64 (10). HRMS calcd. for $C_{15}H_{13}NO_2Se$: $[M+H]^+$ 318,0027. Found: 318.0034.



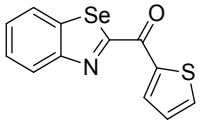
2-(3-methoxyphenylmethanone)benzo[1,3-*d*]selenazole (3i): Yield: 0.052 g (33% - Conventional heating); 0.104 g (66% - Microwave Irradiation); yellow solid; mp 88-90 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.30-8.27 (m, 1H), 8.23-8.20 (m, 1H), 8.05-8.02 (m, 2H), 7.59-7.53 (m, 1H), 7.49-7.43 (m, 2H), 7.23-7.19 (m, 1H), 3.90 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 185.7, 173.1, 159.4, 155.8, 140.7, 135.4, 129.4, 127.4, 127.3, 126.7, 125.2, 124.1, 120.4, 115.2, 55.4. MS *m/z* (relative intensity): 317 (M^+) (13), 135 (100), 107 (38), 92 (26), 77 (37), 64 (17). HRMS calcd. for $C_{15}H_{12}NO_2Se$: $[M]^+$ 318.0027. Found: 318.0042.



2-(2-methoxyphenylmethanone)benzo[1,3-*d*]selenazole (3j): Yield: 0.080 g (51% - Conventional heating); 0.110 g (70% - Microwave Irradiation); orange solid; mp 117-119 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.19 (d, J = 8.1 Hz, 1H), 8.02 (d, J = 8.1 Hz, 1H), 7.78 (dd, J = 7.6, 1.7 Hz, 1H), 7.56-7.49 (m, 2H), 7.44-7.40 (m, 1H), 7.10-7.04 (m, 2H), 3.80 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 188.9, 173.3, 158.8, 155.6, 140.9, 133.5, 131.2, 127.3, 127.2, 126.6, 125.7, 125.4, 120.2, 112.1, 55.9. MS *m/z* (relative intensity): 317 (M^+) (5), 136 (9), 135 (100), 99 (22), 77 (40), 64(10). HRMS calcd. for $C_{15}H_{12}NO_2Se$: $[M]^+$ 318.0027. Found: 318.0038.



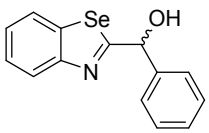
2-(tolylmethanone)benzo[1,3-*d*]selenazole (3k): Yield: 0.105 g (70% - Conventional heating); 0.120 g (80% - Microwave Irradiation); yellow solid; mp 88-90 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.47 (d, J =8.2 Hz, 2H), 8.27 (d, J = 8.1 Hz, 1H), 8.02 (d, J = 8.1 Hz, 1H), 7.56-7.52 (m, 1H), 7.45-7.41 (m, 1H), 7.34 (d, J = 8.2 Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 185.6, 173.6, 155.9, 144.9, 140.7, 131.8, 131.5, 129.2, 127.4, 127.2, 126.7, 125.3, 21.8. MS *m/z* (relative intensity): 301 (M^+) (8), 120 (9), 119 (100), 91 (54), 65 (29). HRMS calcd. for $C_{15}H_{12}NOSe$: $[M+H]^+$ 302.0078. Found: 302.0084.



2-[(thiophen-2-yl)methanone]benzo[1,3-*d*]selenazole (3l): Yield: 0.069 g (47% - Conventional heating); 0.106 g (73% - Microwave Irradiation); yellow solid; mp 81-83 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.68 (dd, J =3.8, 0.8 Hz, 1H), 8.22 (d, J = 8.1 Hz, 1H), 7.95 (d, J = 8.1 Hz, 1H), 7.77 (dd, J = 4.9, 0.8 Hz, 1H), 7.52-7.47 (m, 1H), 7.40-7.34 (m, 1H), 7.19 (dd, J = 4.9, 3.8 Hz, 1H). ^{13}C NMR (75.5 MHz, $CDCl_3$) δ: 177.7, 172.4, 155.4, 140.5, 138.7, 137.2, 136.6, 128.3, 127.2, 127.1, 126.7, 125.2. MS *m/z* (relative intensity): 293 (M^+) (9), 265 (6), 111 (100), 83 (12), 65 (29). HRMS calcd. for $C_{12}H_8NOSSe$: $[M+H]^+$ 293.9483. Found: 293.9491.

General Procedure for the Synthesis of 2-(phenyl)methanol benzo[*d*][1,3]selenazole (4)

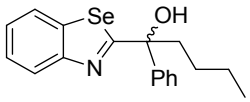
In a 10 mL Schlenk vial equipped with a magnetic stir bar under nitrogen atmosphere, the 2-(phenylmethanone)benzo[1,3-*d*]selenazole **3a** (0.50 mmol) was dissolved in MeOH (5.0 mL) and the system was cooled to -10 °C. After that, $NaBH_4$ (0.75 mmol) was added in one portion and the reaction was kept at -10 °C for 15 min., followed by additional 12 h at room temperature. After this time, the reaction was quenched with aqueous NH_4Cl (20 mL) and extracted with ethyl acetate (3x 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was purified by chromatography on silica gel (hexane/ethyl acetate = 80:20) to provide **4**.



2-(phenyl)methanol benzo[1,3-*d*]selenazole (4): Yield: 0.143 g (99%); yellow solid; mp 105-107 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.00 (d, J = 8.1 Hz, 1H), 7.88 (d, J = 8.1 Hz, 1H), 7.55-7.52 (m, 2H), 7.47-7.28 (m, 5H), 6.02 (d, J = 3.1 Hz, 1H), 3.76 (d, J = 3.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.6, 154.2, 140.9, 138.0, 128.7, 128.4, 126.6, 126.0, 124.9, 124.8, 124.4, 76.0. MS *m/z* (relative intensity): 289 (M^+) (6), 106 (8), 105 (100), 77 (83), 51 (31). HRMS calcd. for $C_{14}H_{11}NOSe$: $[M+H]^+$ 290.0084. Found: 290.0092.

General Procedure for the Synthesis of 1-(benzo[1,3-*d*]selenazol-2-yl)-1-phenylpentan-1-ol (5)

In a 10 mL Schlenk vial equipped with a magnetic stir bar under nitrogen atmosphere, the 2-(phenylmethanone)benzo[1,3-*d*]selenazole **3a** (0.50 mmol) was dissolved in THF (1.0 mL) and the system was cooled to -10 °C. After that, the previously prepared butylmagnesium bromide (1.0 mL of a 0.5 M solution in THF, 0.5 mmol), was added dropwise. The reaction was kept at -10 °C for 15 min, followed by additional 1 h at room temperature. After this time, the reaction was quenched with aqueous NH_4Cl (20 mL) and extracted with ethyl acetate (3x 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was purified by chromatography on silica gel (hexane/ethyl acetate = 80:20) to provide **5**.



1-(benzo[1,3-*d*]selenazol-2-yl)-1-phenylpentan-1-ol (5): Yield: 0.120 g (70%); yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (dd, J = 8.1, 0.7 Hz, 1H), 7.81 (dd, J = 8.1, 0.7 Hz, 1H), 7.66-7.63 (m, 2H), 7.42-7.36 (m, 1H), 7.33-7.28 (m, 2H), 7.24-7.19 (m, 2H), 3.22 (br, 1H), 2.41 (t, J = 7.0 Hz, 2H), 1.36-1.30 (m, 4H), 0.83 (t, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 184.8, 154.4, 143.6, 138.1, 128.2, 127.4, 125.8, 125.5, 124.8, 124.7, 124.4, 80.5, 42.0, 25.5, 22.7, 13.9. MS *m/z* (relative intensity): 345 ($M^{+\cdot}$) (4), 288 (100), 83 (61), 105 (94), 77 (98). HRMS calcd. for $C_{18}H_{19}NOSe$: $[M+H]^+$ 346.0710. Found: 346.0710.

References

1. Y.-P. Zhu, M. Lian, F.-C. Jia, M.-C. Liu, J.-J. Yuan, Q.-H. Gao, A.-X. Wu, *Chem. Commun.* **2012**, *48*, 9086-9088.

SELECTED SPECTRA

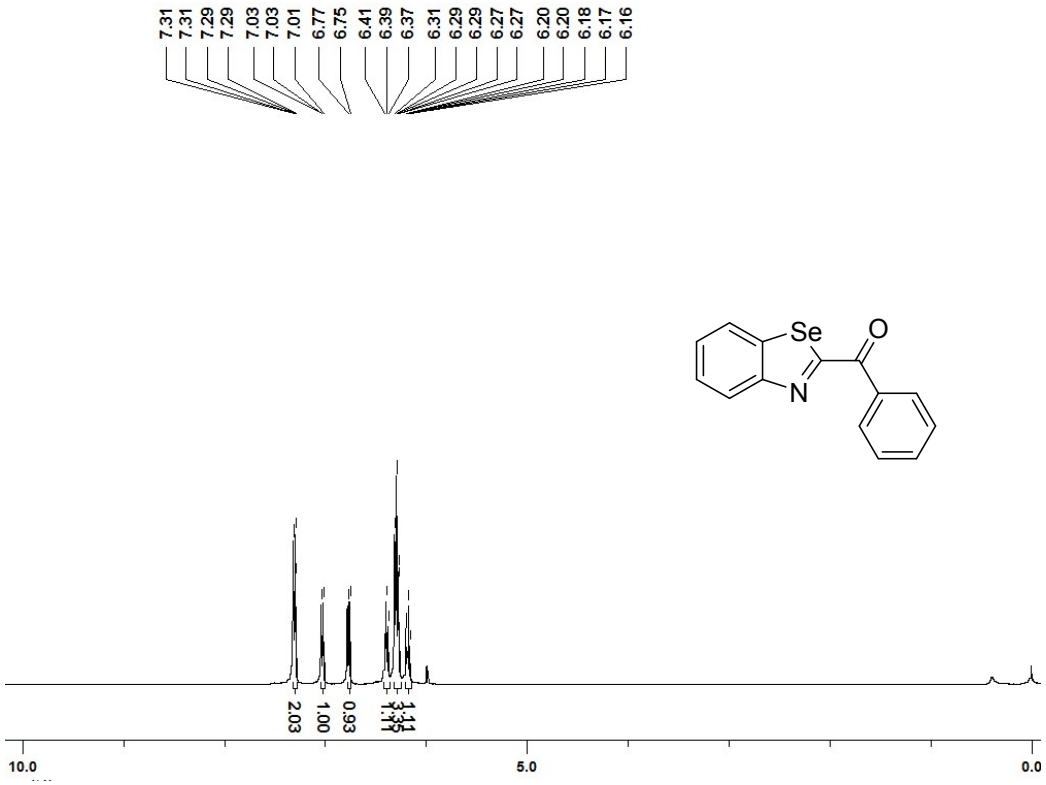


Figure 1. ^{1}H NMR (400 MHz) spectrum for compound **3a** in $CDCl_3$.

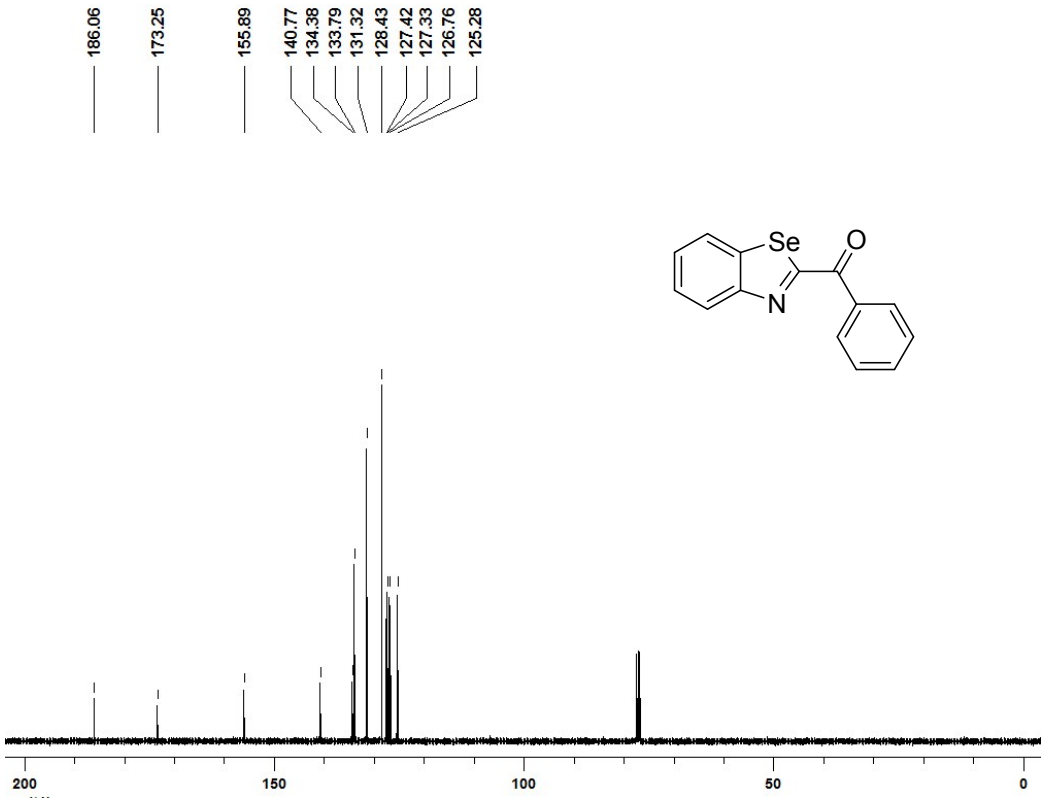
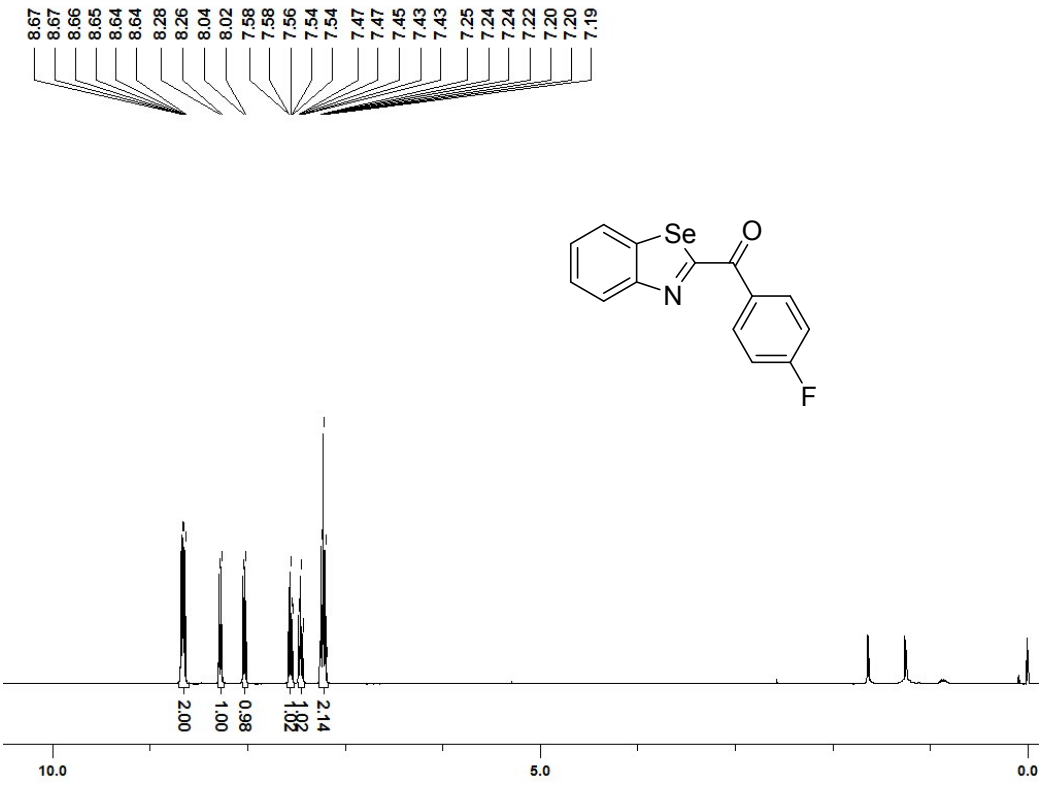


Figure 2. ^{13}C NMR (100 MHz) spectrum for compound **3a** in $CDCl_3$.



. **Figure 3.** ^{1}H NMR (300 MHz) spectrum for compound **3b** in $CDCl_3$.

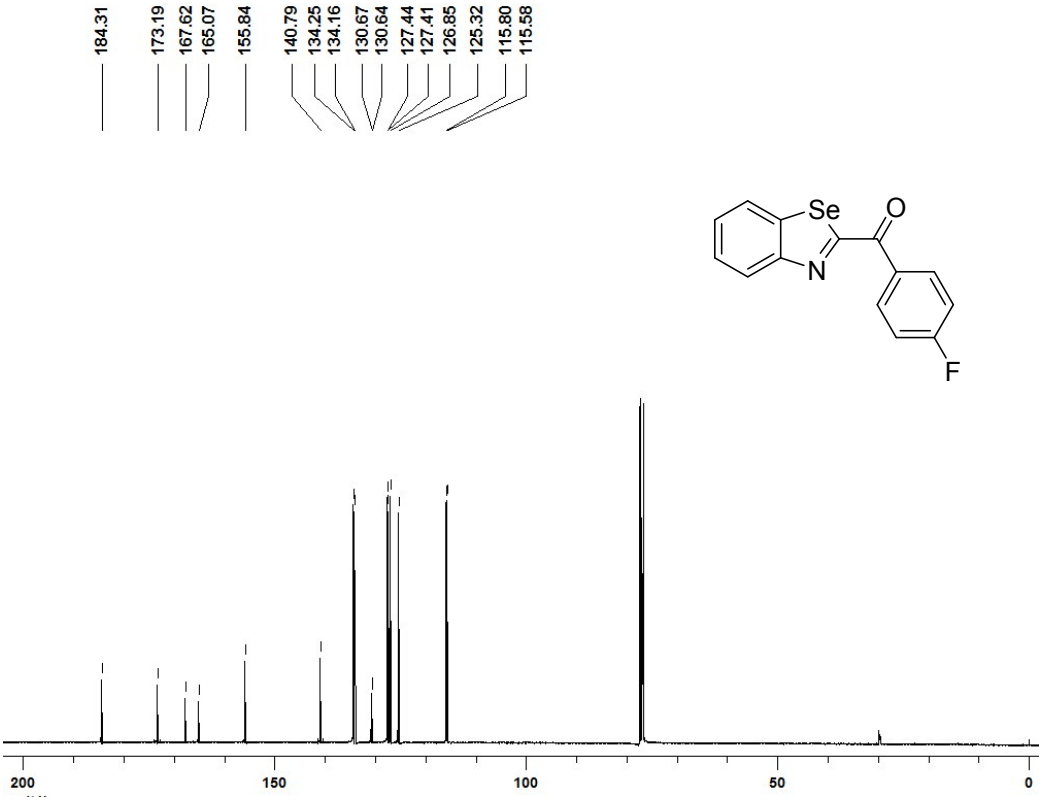
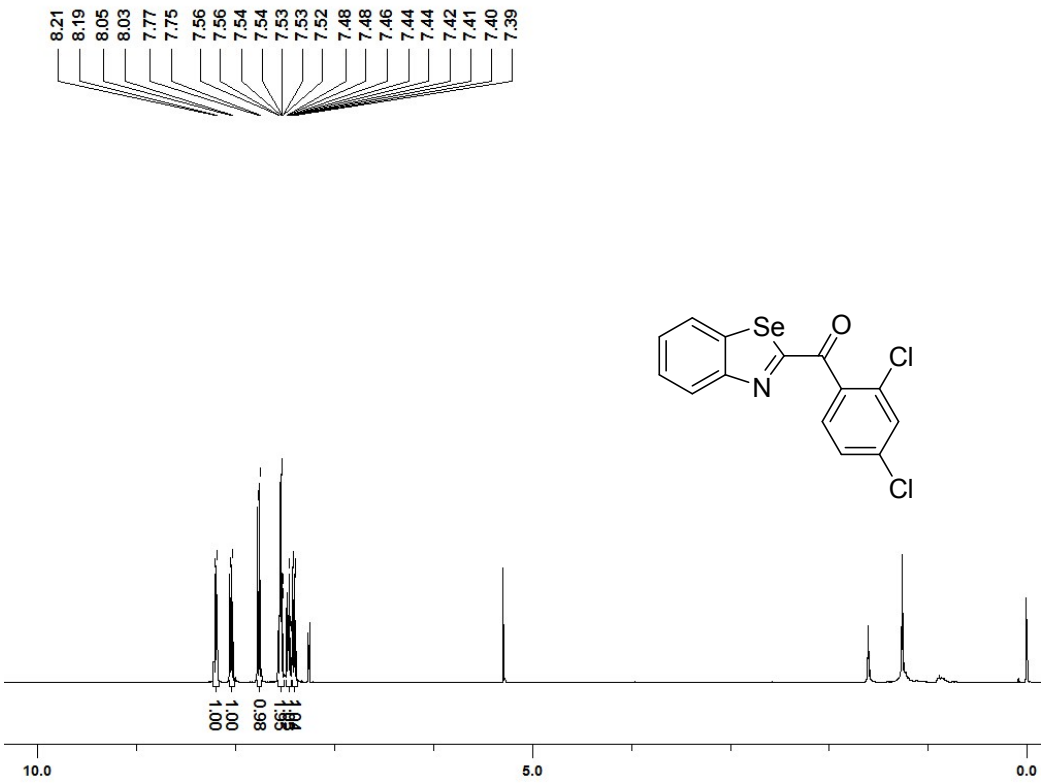


Figure 4. ^{13}C NMR (75.5 MHz) spectrum for compound **3b** in $CDCl_3$.



. **Figure 5.** ^{1}H NMR (300 MHz) spectrum for compound **3c** in $CDCl_3$.

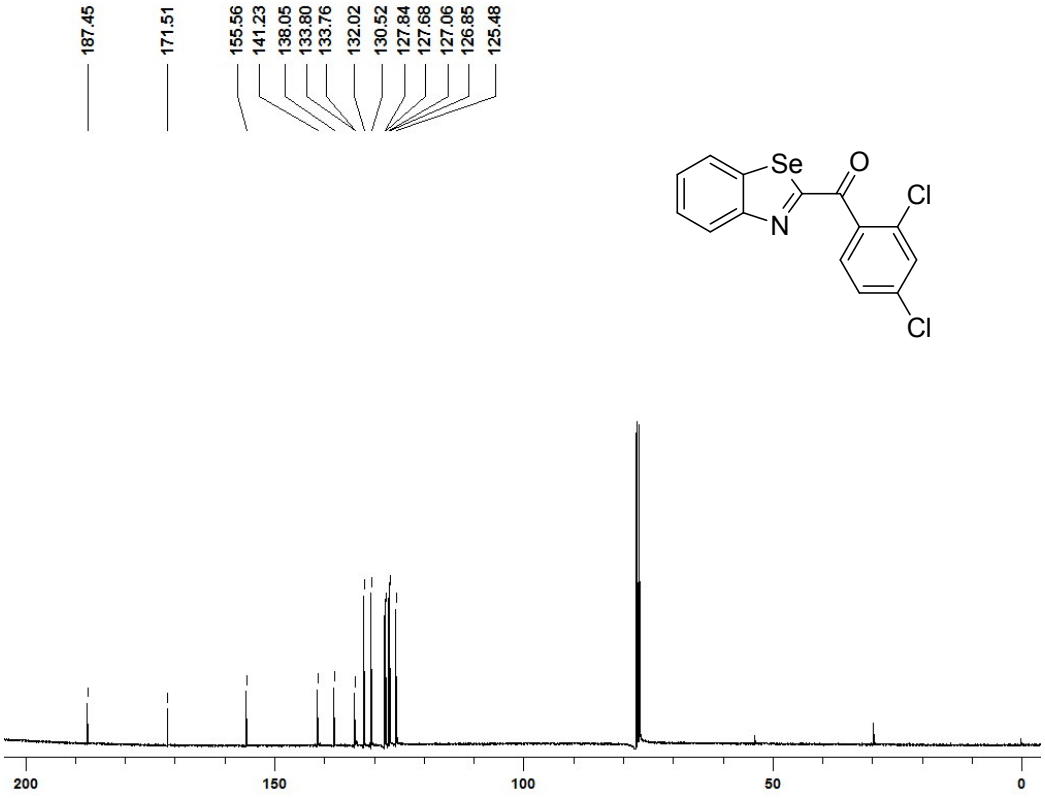


Figure 6. ^{13}C NMR (75.5 MHz) spectrum for compound **3c** in $CDCl_3$.

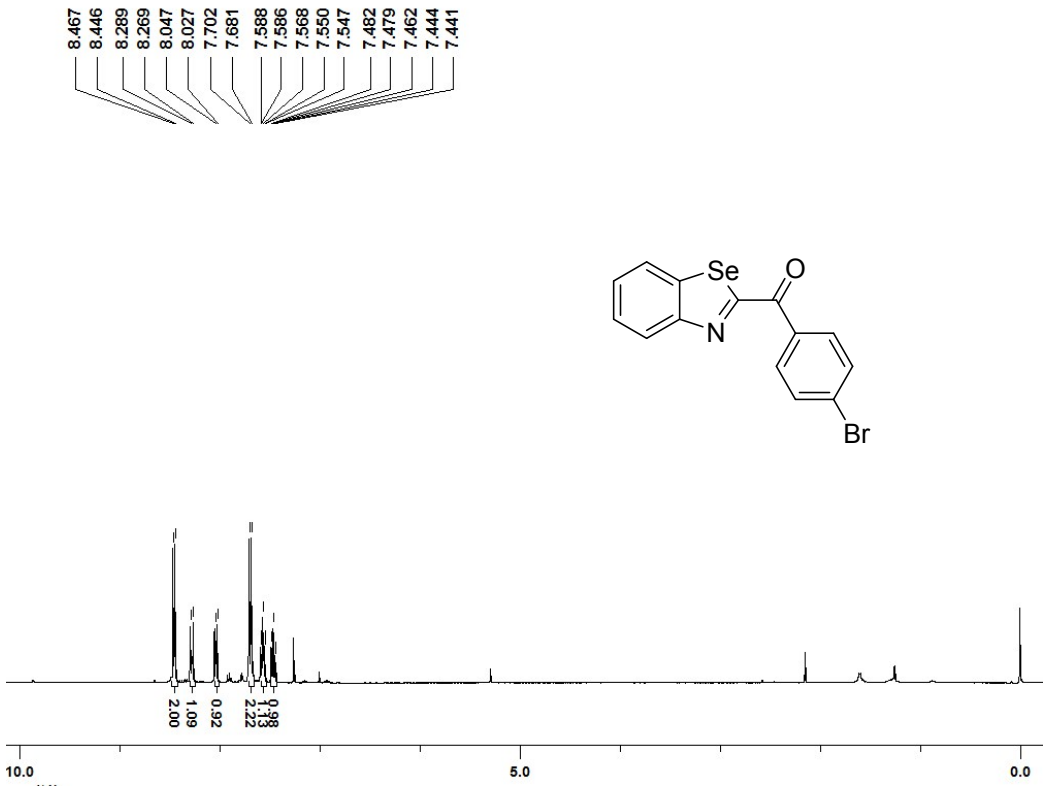


Figure 7. ^{1}H NMR (300 MHz) spectrum for compound **3d** in $CDCl_3$.

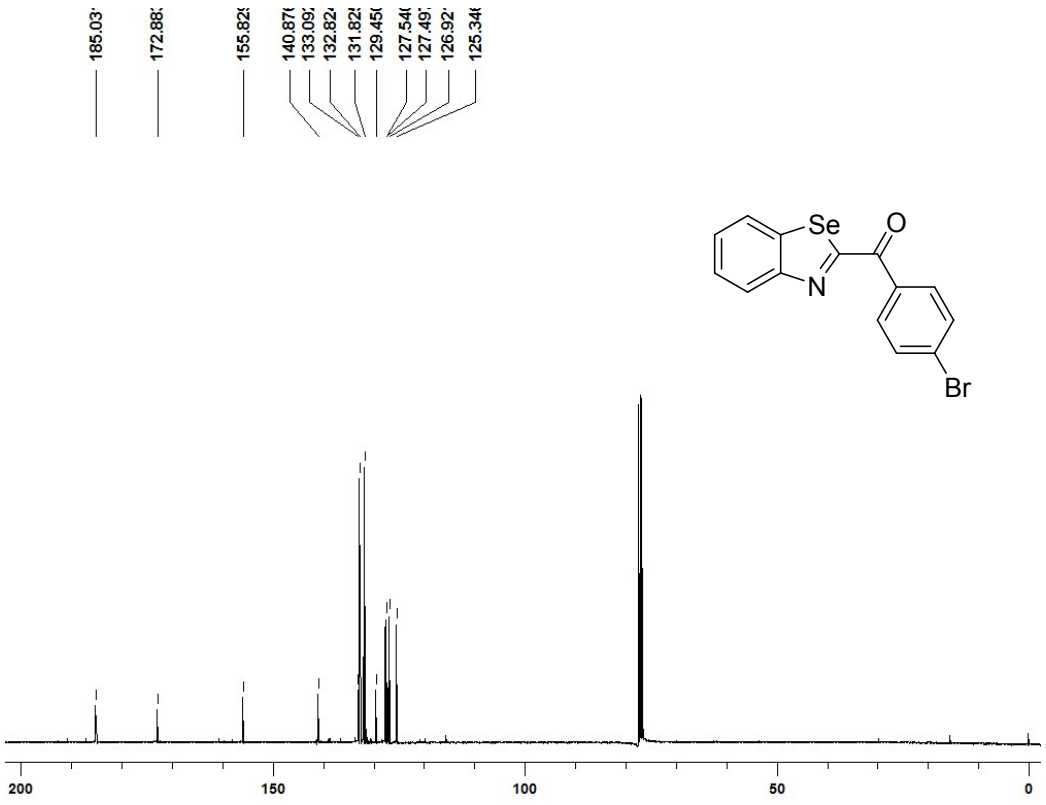


Figure 8. ^{13}C NMR (75.5 MHz) spectrum for compound **3d** in $CDCl_3$.

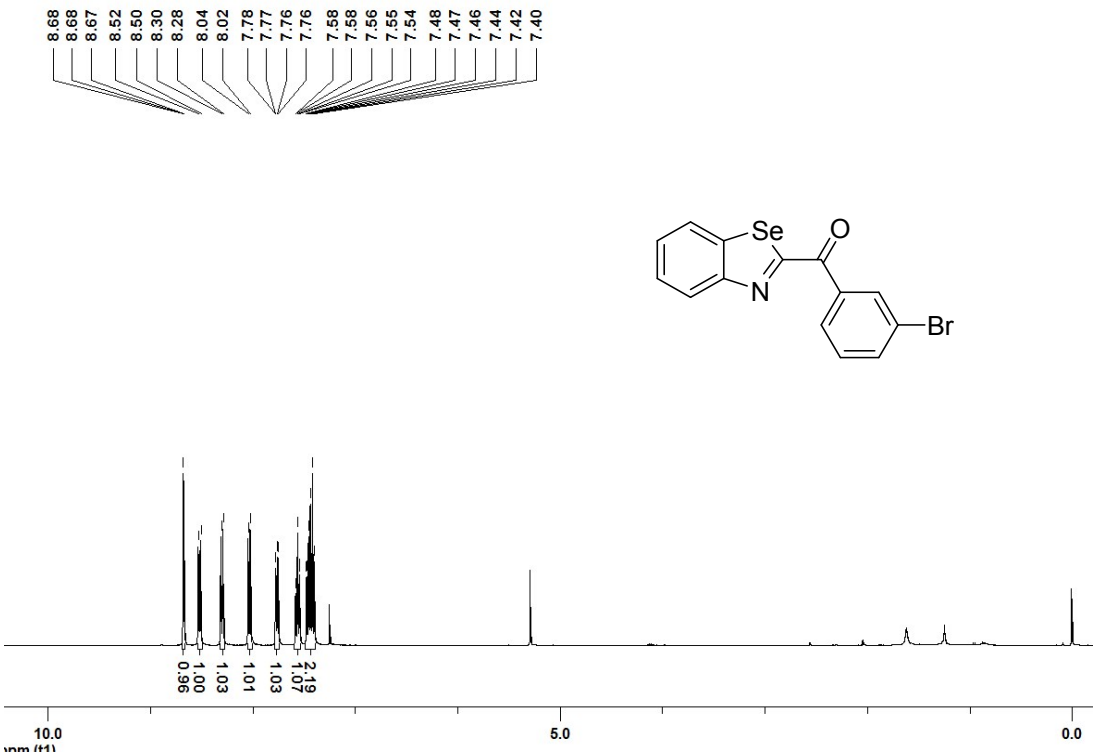


Figure 9. ^{1}H NMR (300 MHz) spectrum for compound **3e** in $CDCl_3$.

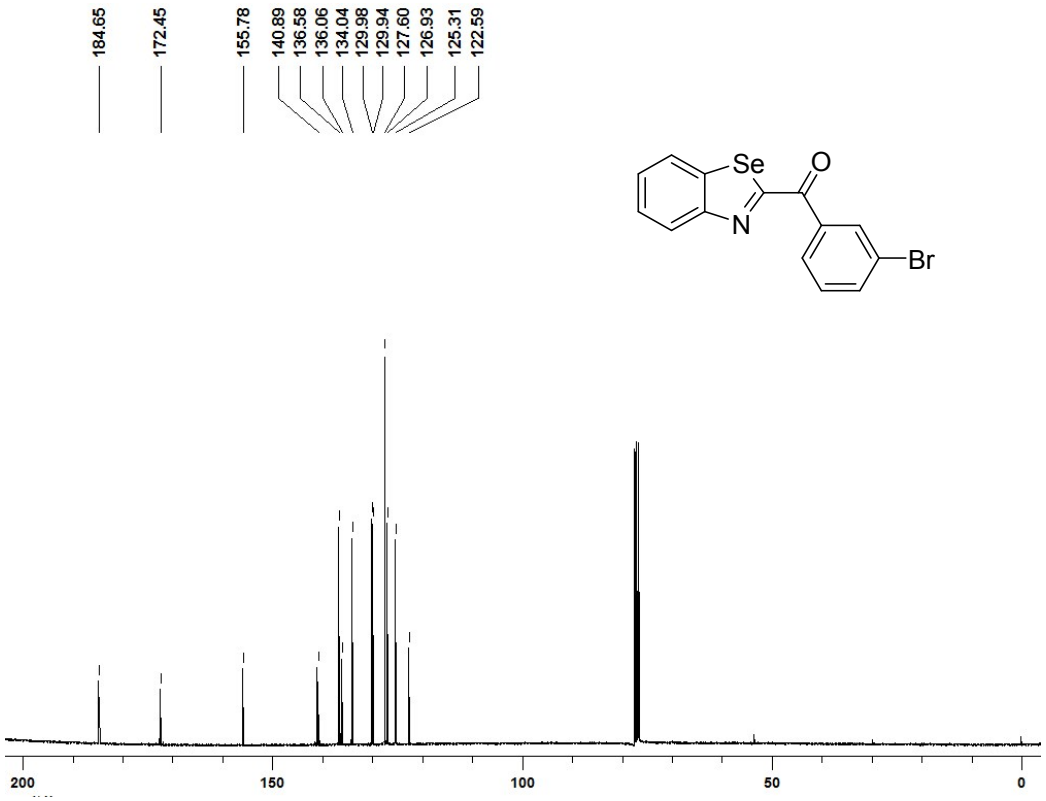
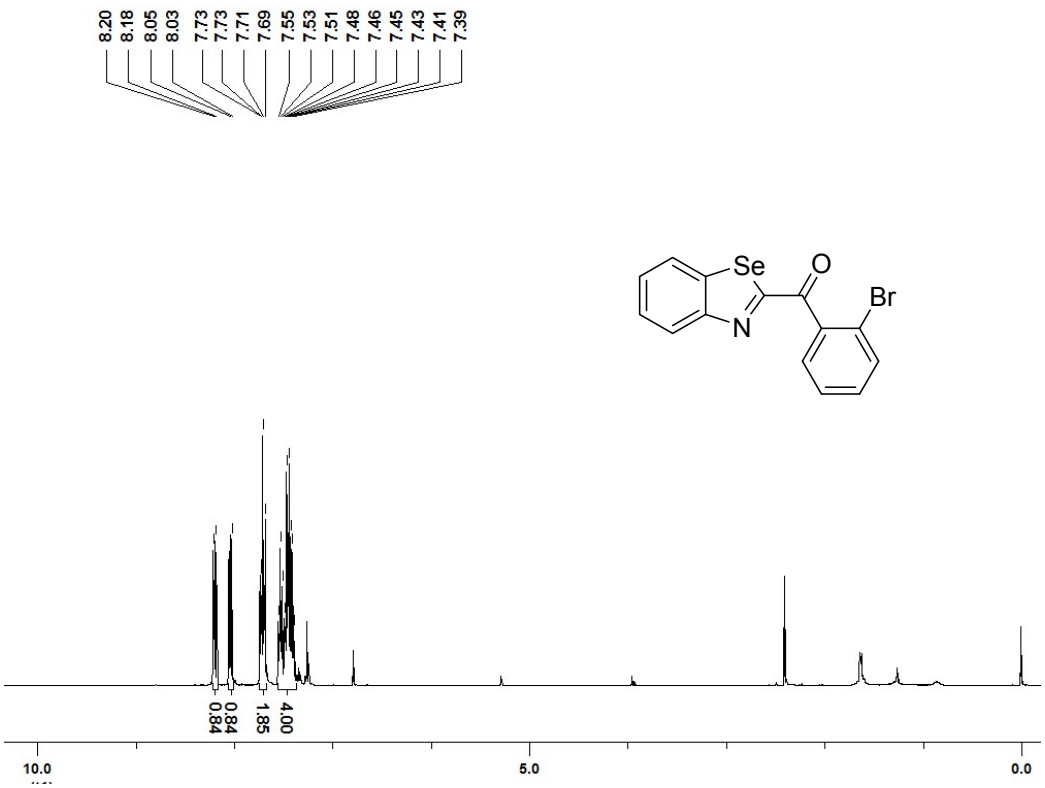


Figure 10. ^{13}C NMR (75.5 MHz) spectrum for compound **3e** in $CDCl_3$.



. **Figure 11.** ^{1}H NMR (300 MHz) spectrum for compound **3f** in $CDCl_3$.

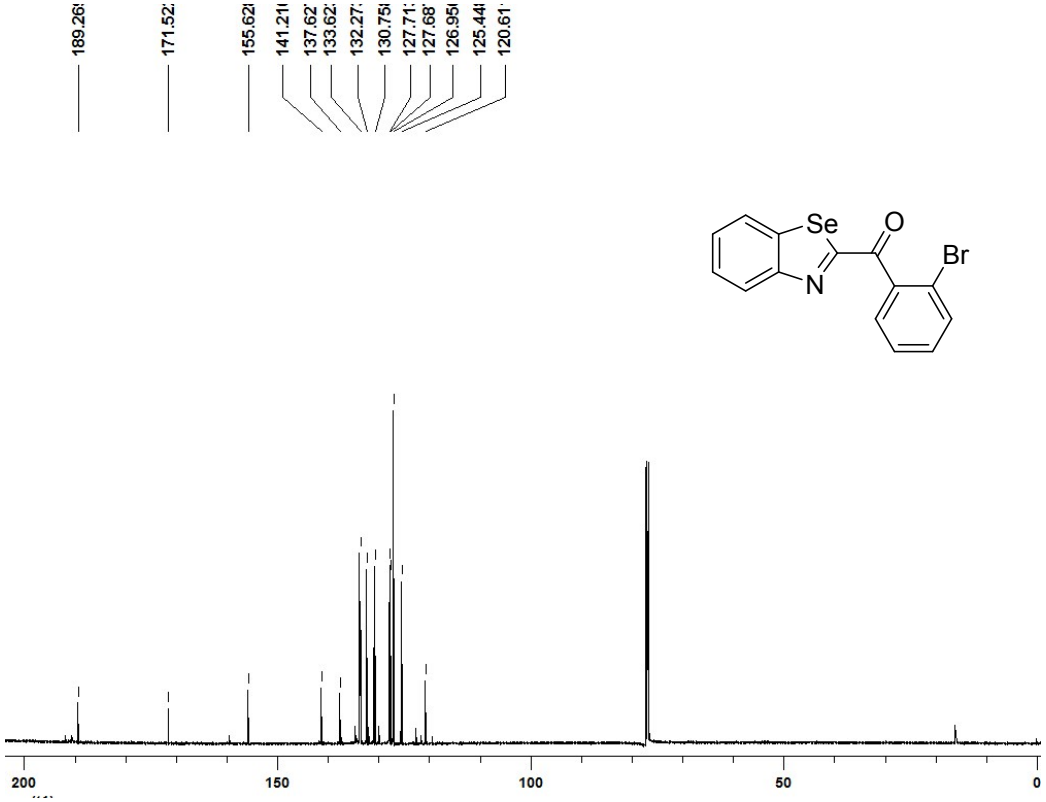


Figure 12. ^{13}C NMR (75.5 MHz) spectrum for compound **3f** in $CDCl_3$.

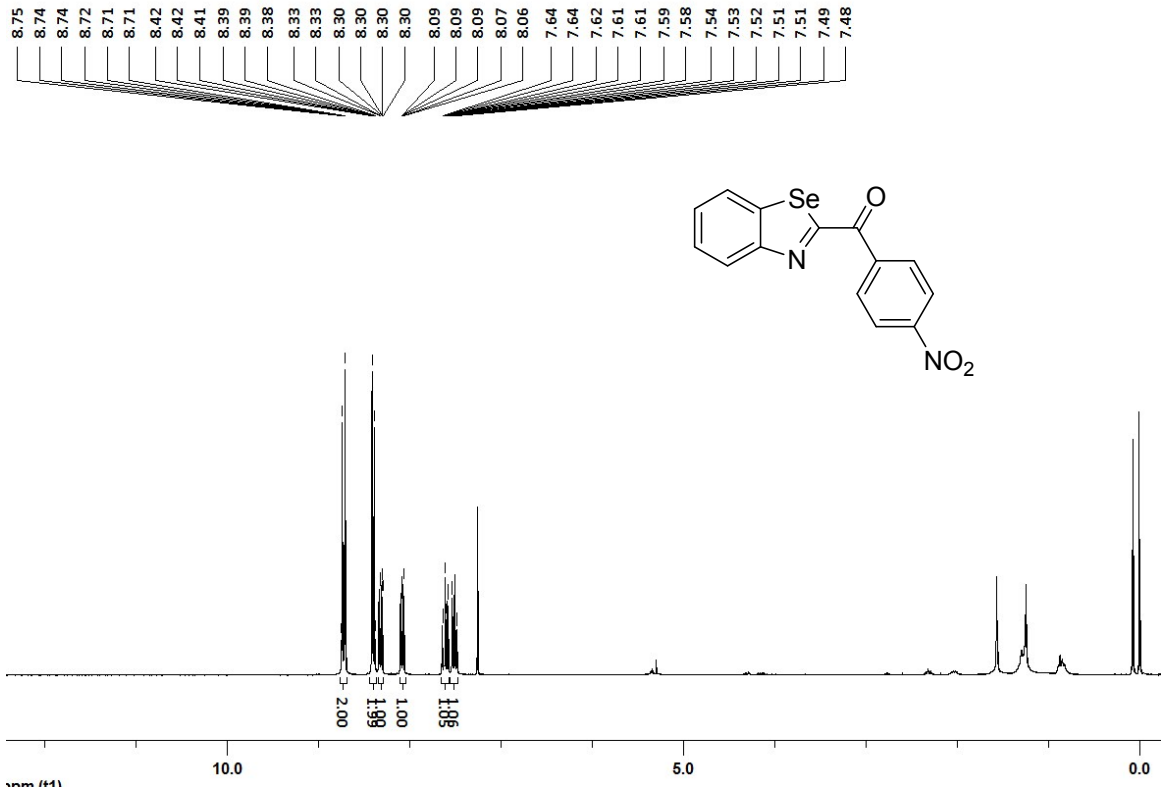


Figure 13. ^{1}H NMR (300 MHz) spectrum for compound **3g** in $CDCl_3$.

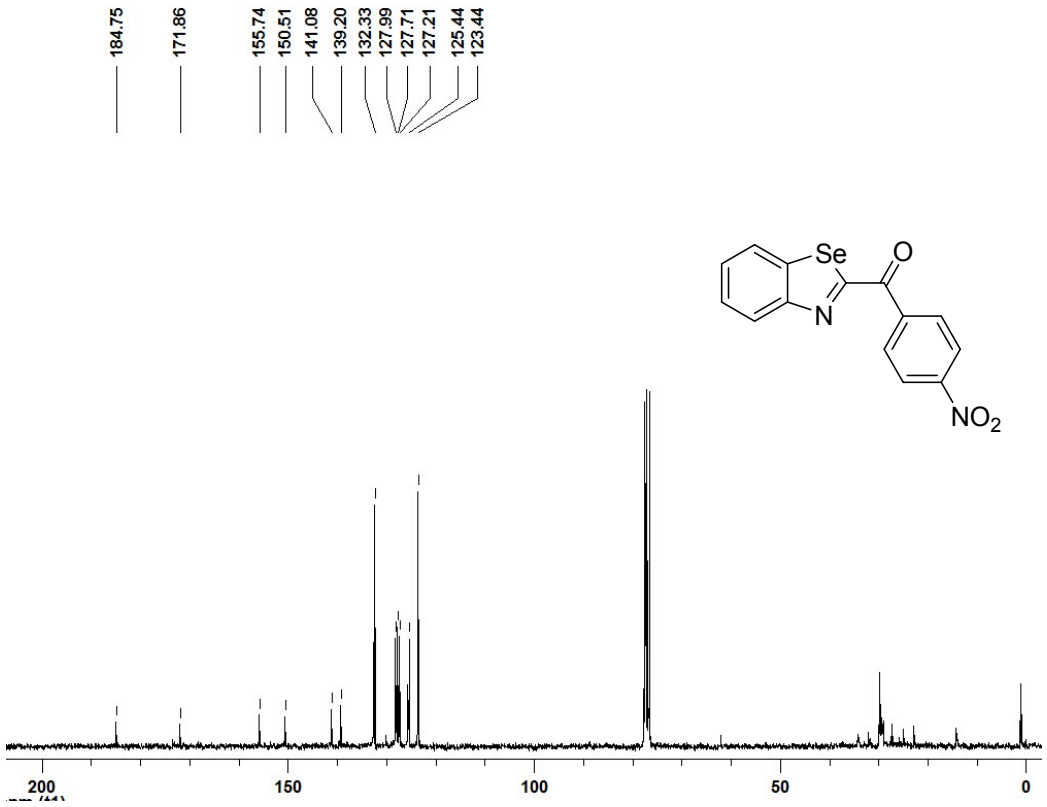


Figure 14. ^{13}C NMR (75.5 MHz) spectrum for compound **3g** in $CDCl_3$.

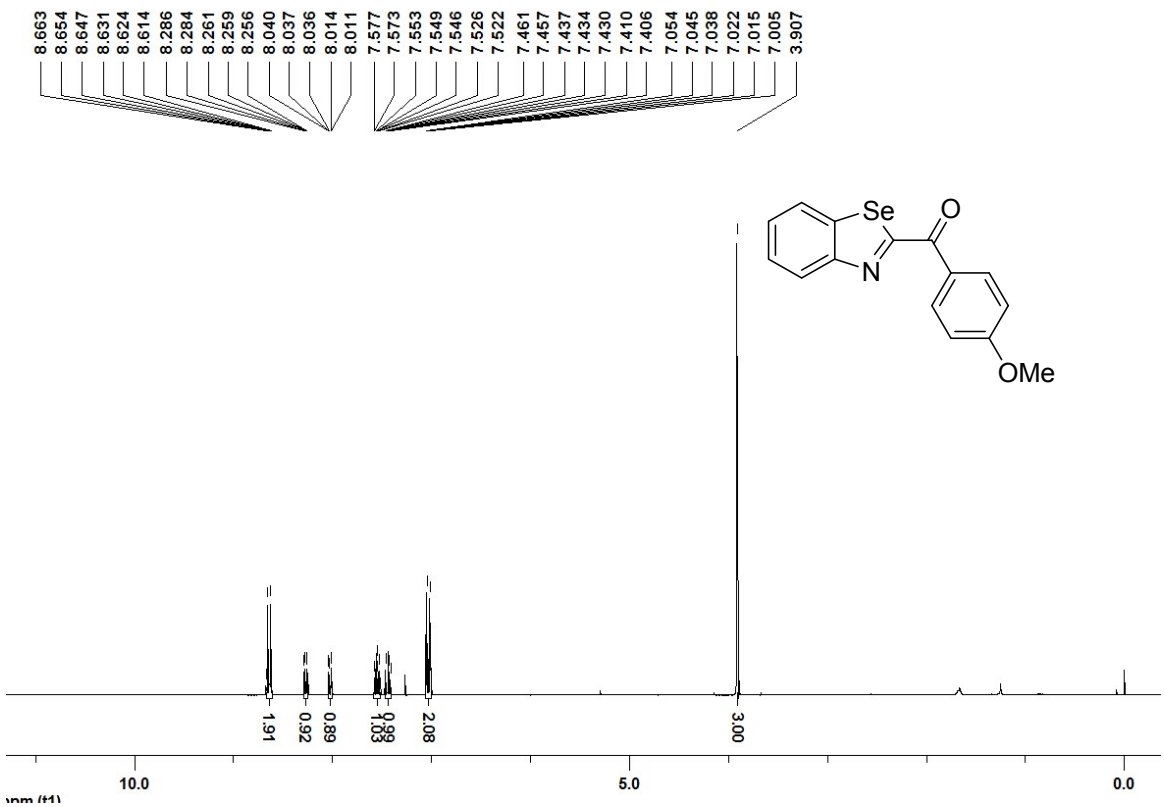


Figure 15. ^{1}H NMR (300 MHz) spectrum for compound **3h** in $CDCl_3$.

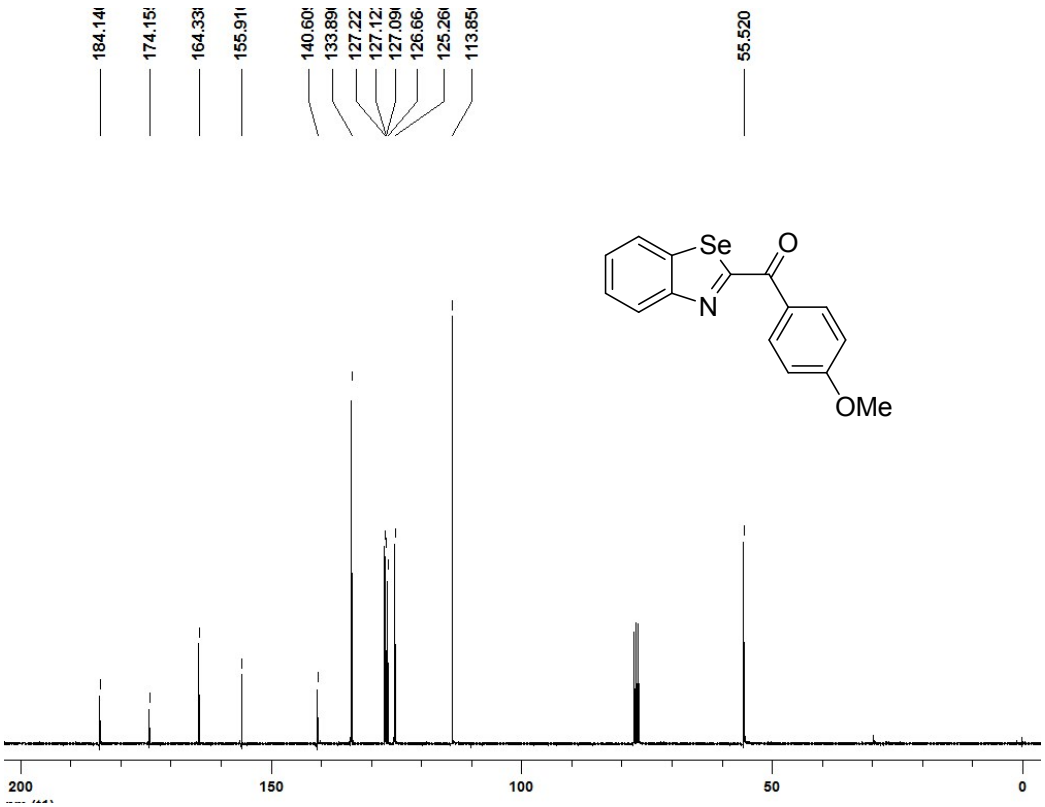


Figure 16. ^{13}C NMR (75.5 MHz) spectrum for compound **3h** in $CDCl_3$.

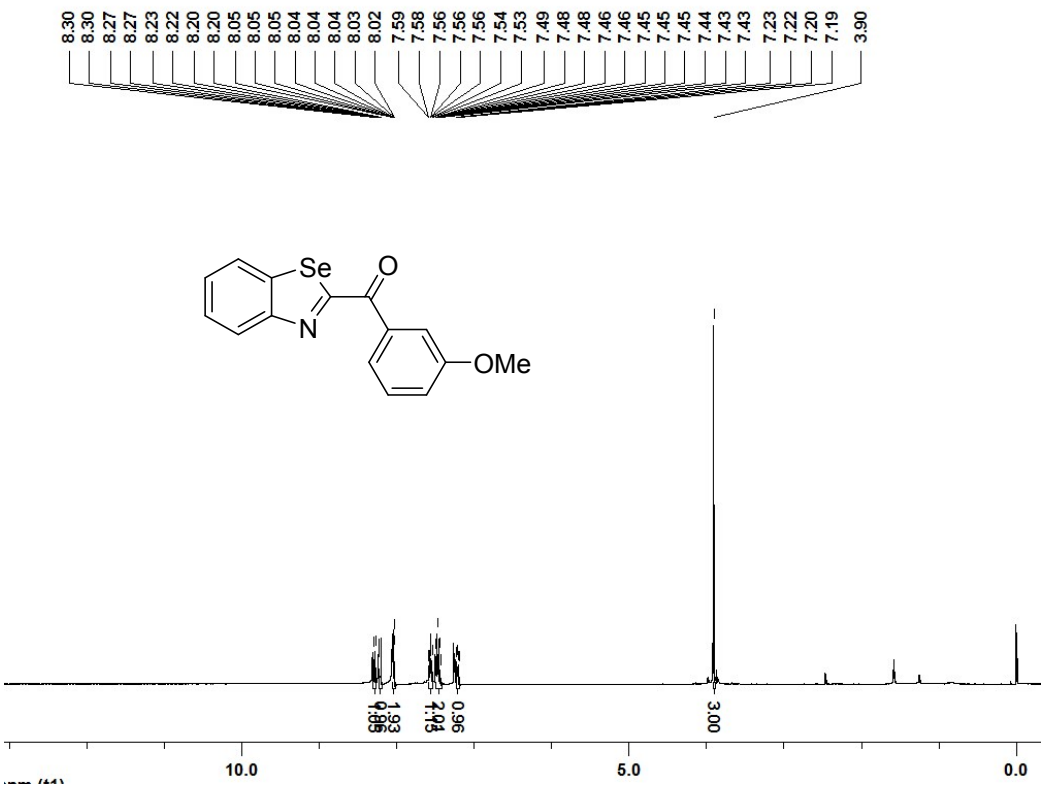


Figure 17. ^{1}H NMR (300 MHz) spectrum for compound **3i** in $CDCl_3$.

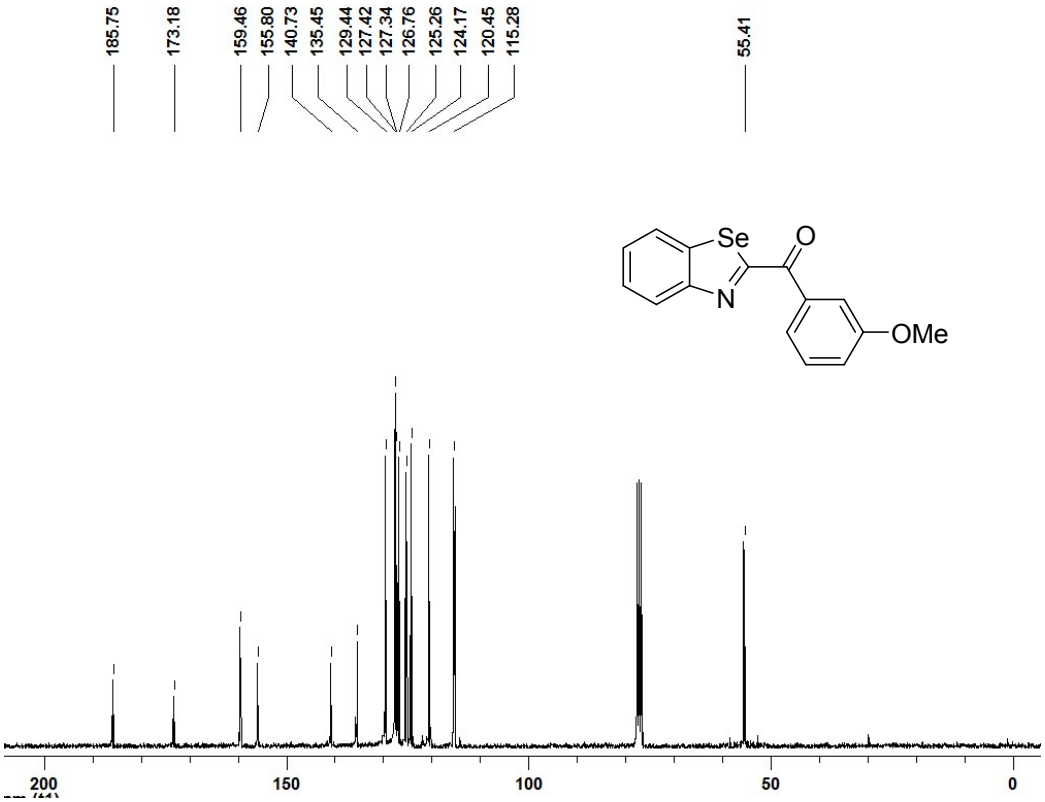


Figure 18. ^{13}C NMR (75.5 MHz) spectrum for compound **3i** in $CDCl_3$.

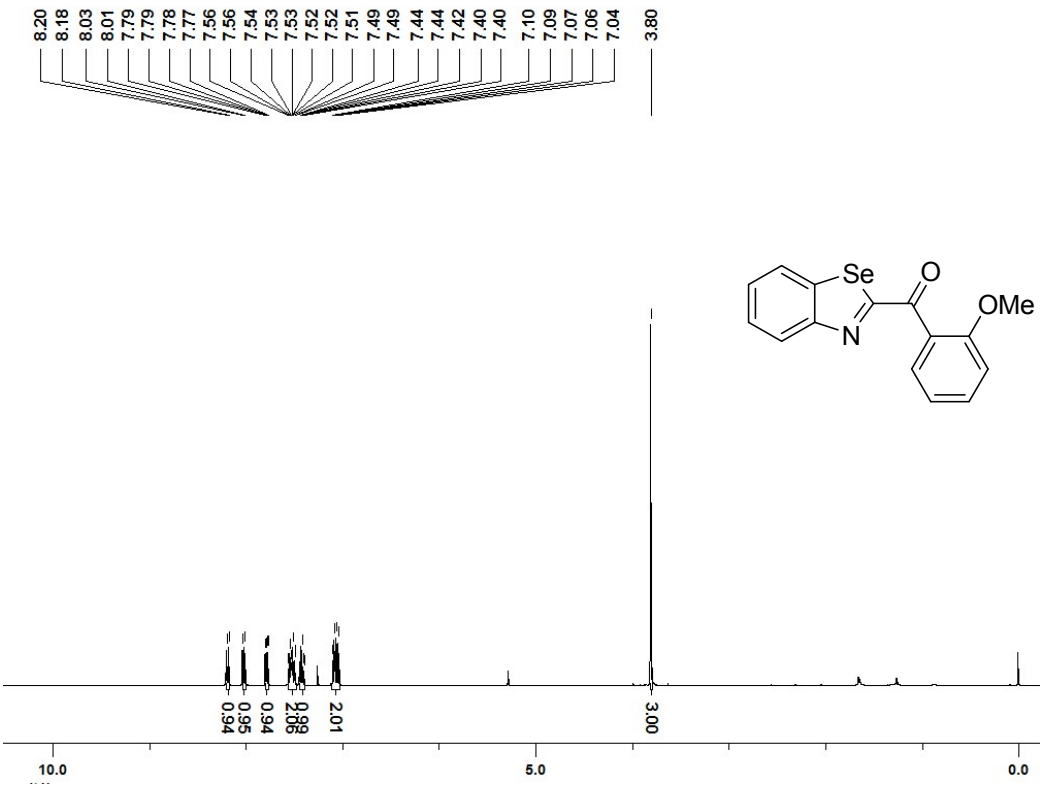


Figure 19. ^{1}H NMR (300 MHz) spectrum for compound **3j** in $CDCl_3$.

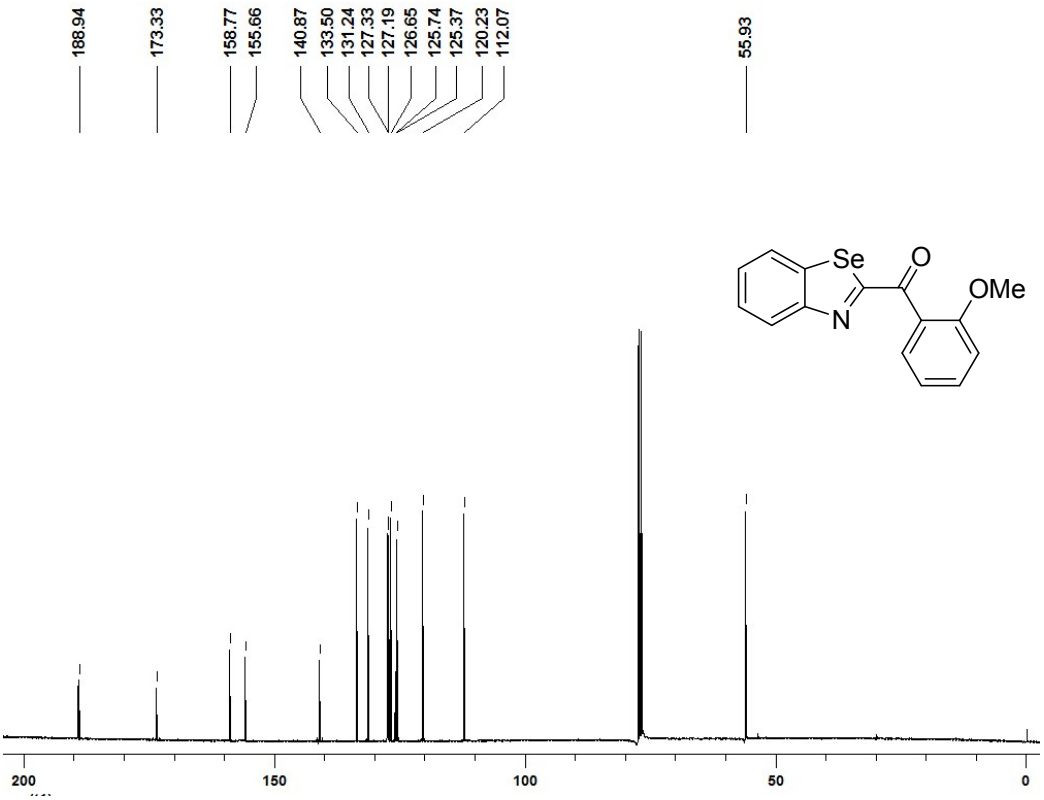


Figure 20. ^{13}C NMR (75.5 MHz) spectrum for compound **3j** in $CDCl_3$.

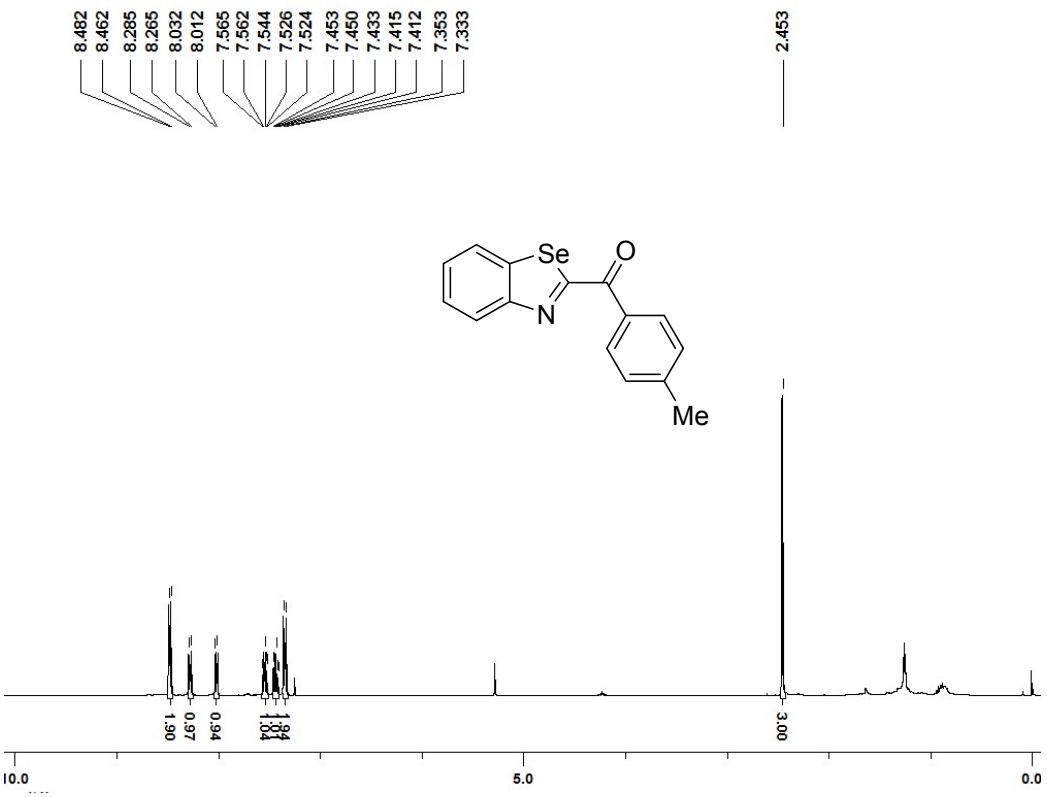


Figure 21. ^{1}H NMR (300 MHz) spectrum for compound **3k** in $CDCl_3$.

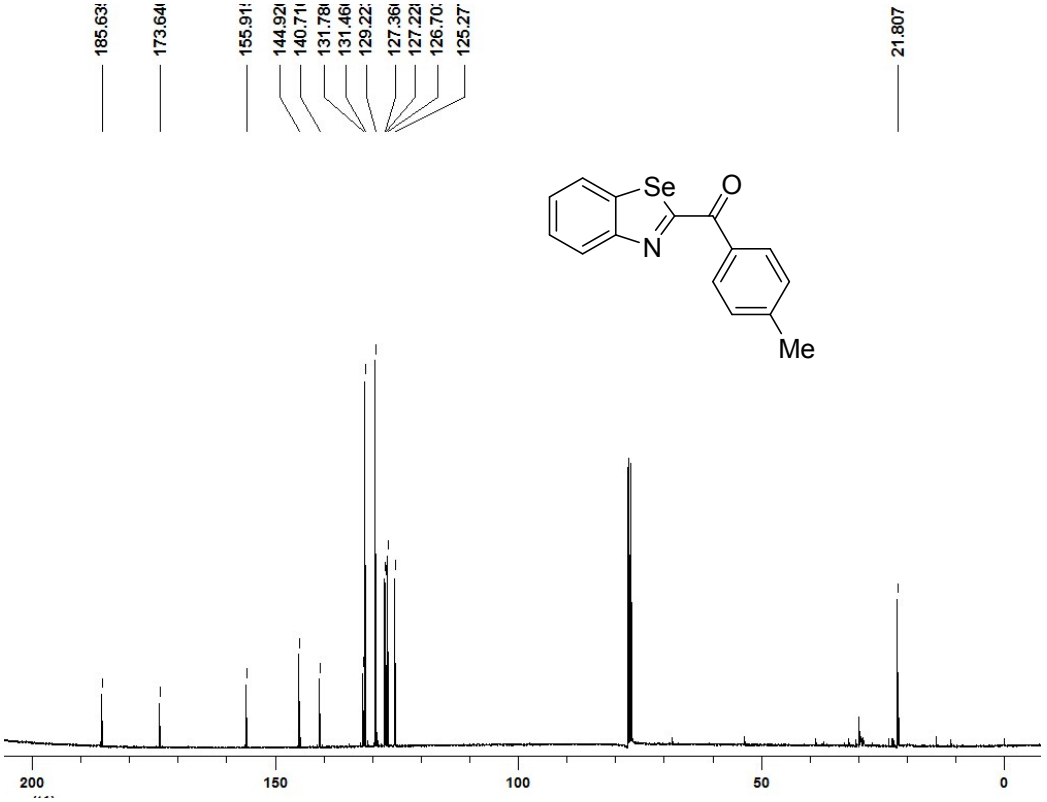


Figure 22. ^{13}C NMR (75.5 MHz) spectrum for compound **3k** in $CDCl_3$.

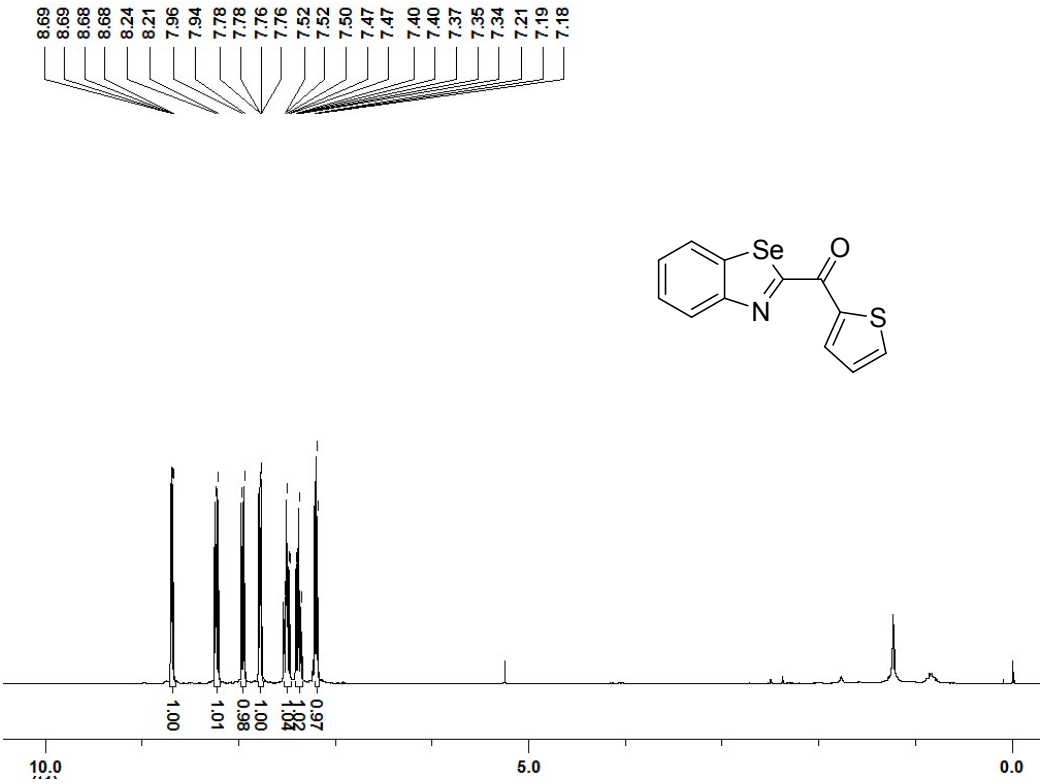


Figure 23. ^{1}H NMR (300 MHz) spectrum for compound **3l** in $CDCl_3$.

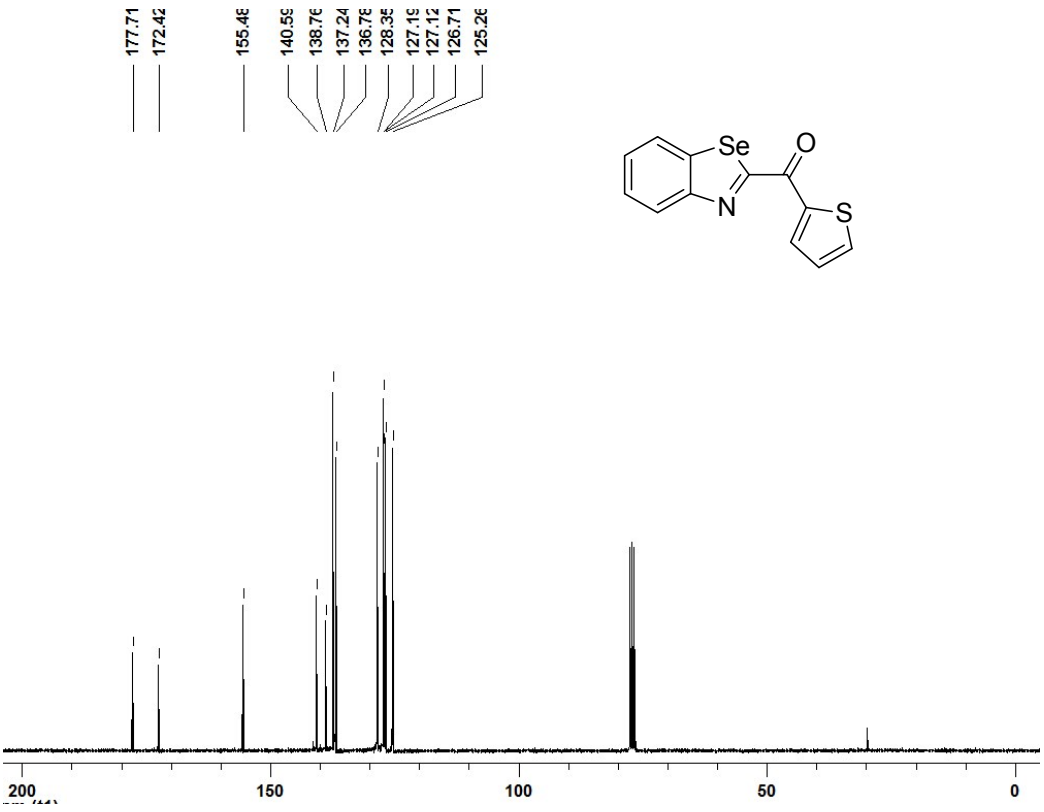


Figure 24. ^{13}C NMR (75.5 MHz) spectrum for compound **3l** in $CDCl_3$.

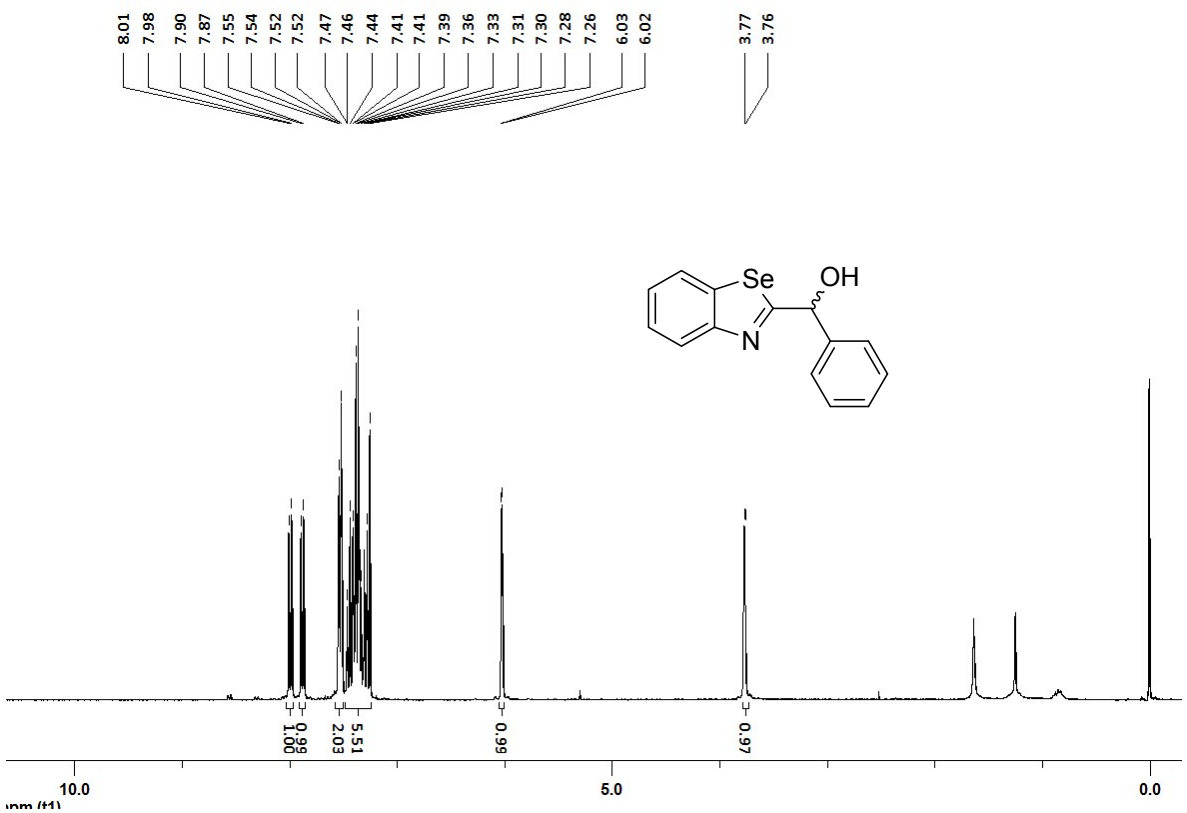


Figure 25. ^{1}H NMR (300 MHz) spectrum for compound **4** in $CDCl_3$.

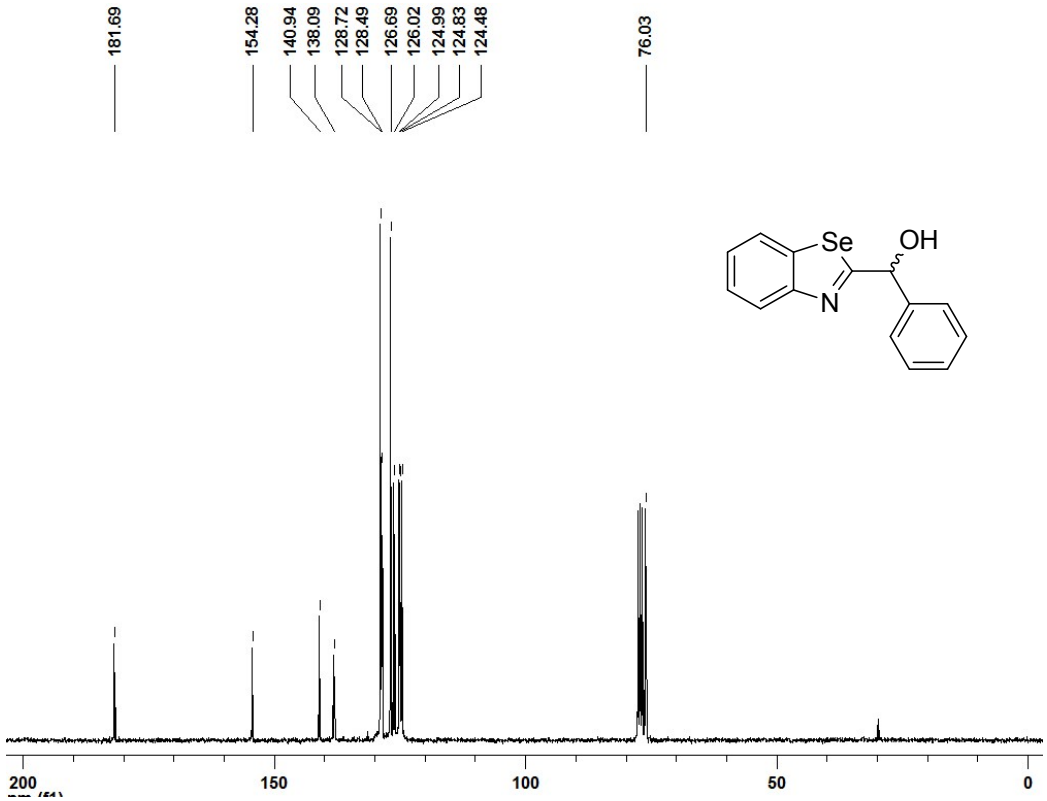


Figure 26. ^{13}C NMR (75.5 MHz) spectrum for compound **4** in $CDCl_3$.

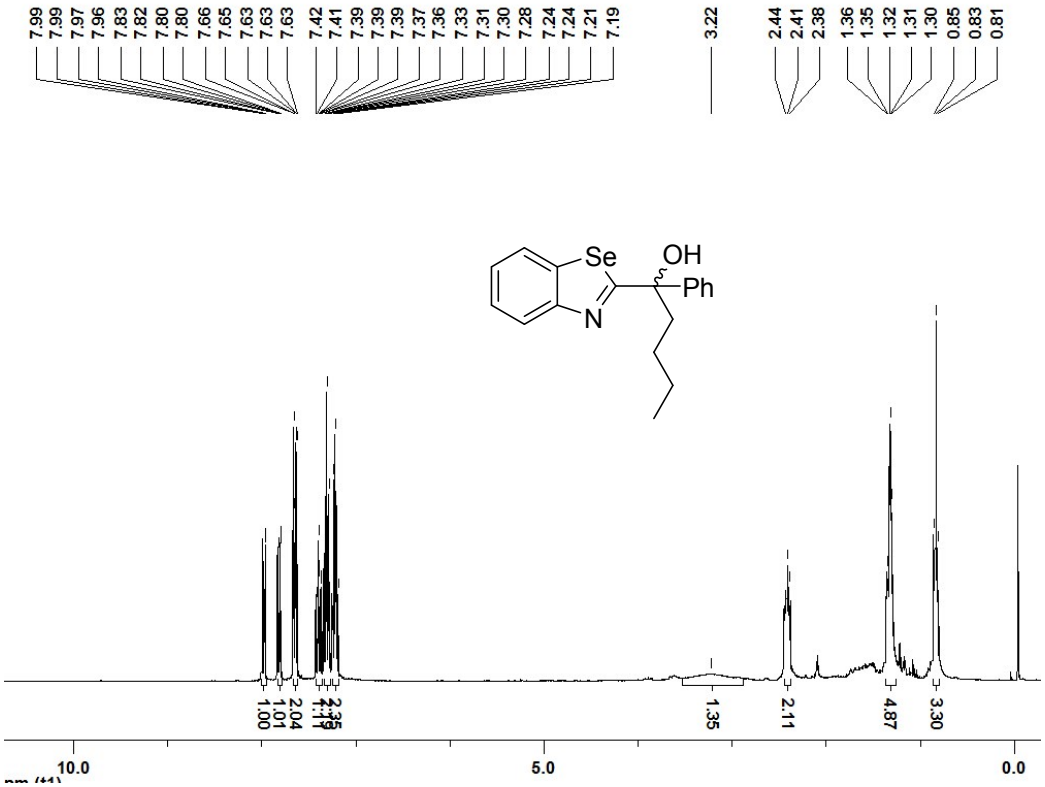


Figure 27. ^{1}H NMR (300 MHz) spectrum for compound **5** in $CDCl_3$.

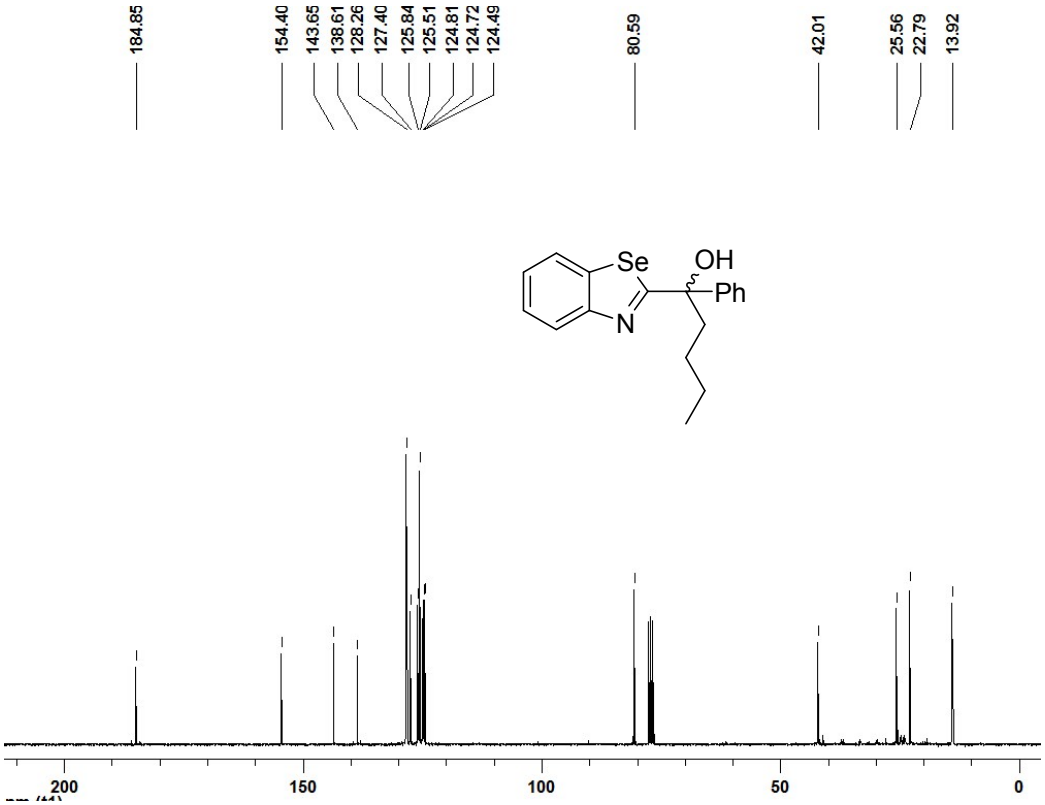


Figure 28. ^{13}C NMR (75.5 MHz) spectrum for compound **5** in $CDCl_3$.