Supporting Information

Ni and Fe Catalyzed Cascade Radical Reactions of Oxime Esters with Diselenides

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1. General Information

All reactions were carried out under argon atmosphere using standard Schlenk techniques. $FeCl_2$ was purchased from Bide Pharmatech. Other reagents were purchased from Adamas-beta® and Aladdin®. All commercial reagents were used as received. γ,δ-Unsaturated oxime esters, cyclic ketoxime esters and aryl diselenides were prepared according to literature procedures using commercial reagents (Sect. 2.1). ^1H NMR and ^{13}C NMR spectra were recorded at room temperature on a Bruker 400 or a 600 MHz spectrometer. Chemical shifts (δ) are reported in ppm with the following abbreviations used for the observed multiplicities: s (singlet), d (doublet), t (triplet), q (quartet), br (broad), m (multiplet for unresolved lines). ^1H NMR chemical shifts were referenced to the residual solvent signal for $CDCl_3$ (7.26 ppm), and ^{13}C NMR chemical shifts were referenced to the solvent signal of $CDCl_3$ (77.16 ppm). Analytical TLC was performed on pre-coated silica gel plates. After elution, the plates were visualized by UV illumination at 254-360 nm and by staining with 5% $PdCl_2$ solvent (in 10% hydrochloric acid solvent). The crude products were purified by column chromatography using silica gel (200-300 mesh). GC analyses were performed on a Shimadzu Nexis GC-2030 equipped with an ZB-5MS plus column (30 mm × 0.32 mm × 0.50 μm). HRMS analysis was performed on Shimadzu LCMS-ITTOF and Waters Vion IMS QTof. Isomeric ratios of the products were determined by ^1H NMR analysis of crude reaction mixtures.

2. Experimental Procedures

2.1 Preparation of Oxime Ester Derivatives

The oxime esters **1a-1u**, **4a-4l** (listed in Table S1) and aryl diselenides **2r-2w** (listed in Table S2) were synthesized according to literature procedures. Spectral data can be found in the following references: **1a-1b**[1, 2], **1e**[3], **1i-1j**[3], **1l-1m**[3, 4], **1o-1q**[5, 6], **4a-4g**[7-9], **4i-4k**[10-12], **2r-2w**[13, 14]

Table S1: Oxime ester derivatives used in this study

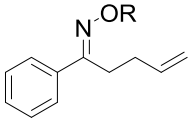
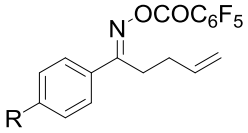
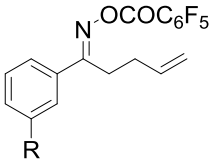
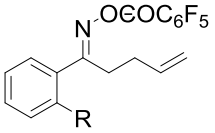
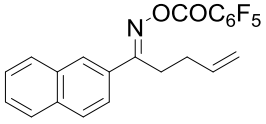
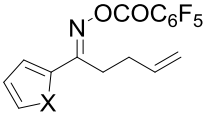
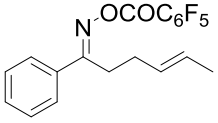
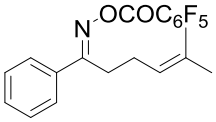
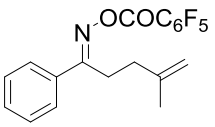
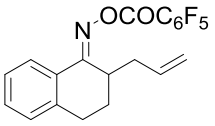
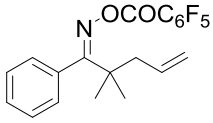
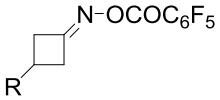
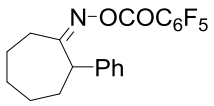
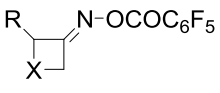
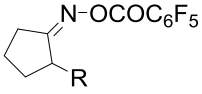
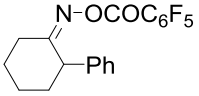
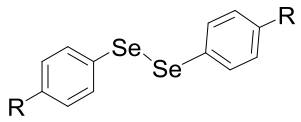
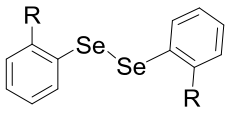
 **1a**, R = COC_6F_5 **1aa**, R = Ac	 **1b**, R = Cl **1c**, R = F **1d**, R = Me **1e**, R = OMe	 **1f**, R = Cl **1g**, R = OMe	 **1h**, R = Cl **1i**, R = OMe
 **1j**	 **1k**, X = S **1l**, X = O	 **1m**	 **1n**
 **1o**	 **1p**	 **1q**	
 **4a**, R = H **4b**, R = Ph **4c**, R = OBn **4d**, R = COOEt  **4l**	 **4e**, R = Me, X = CH_2 **4f**, R = Bn, X = CH_2 **4g**, R = H, X = O **4h**, R = H, X = S	 **4i**, R = Me **4j**, R = H	 **4k**

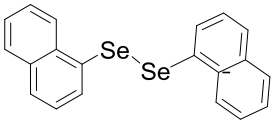
Table S2: Aryl diselenides used in this study



2r, R = Me
2s, R = OMe
2t, R = F

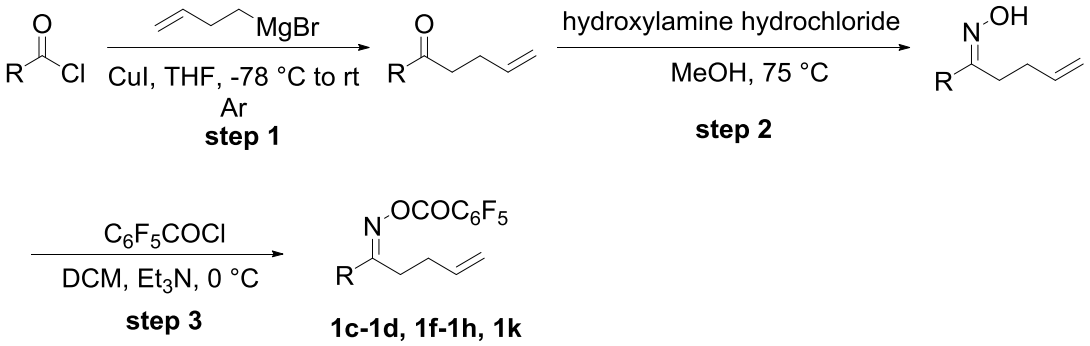


2u, R = Me
2v, R = OMe



2w

Synthesis of oxime esters **1c-1d**, **1f-1h**, **1k**



1c-1d, 1f-1h, 1k

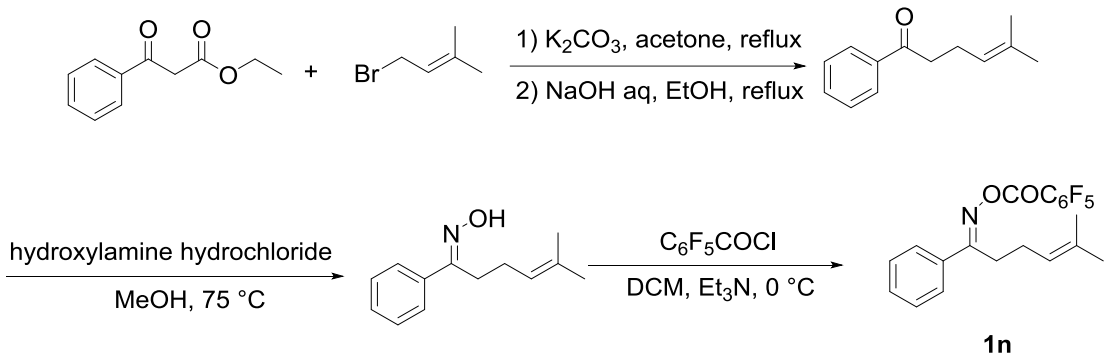
Step 1: A dry two-neck flask equipped with a stirring bar was charged with CuI (5mol%) and then the flask was refilled with argon. The acid chloride (1.1 equiv.) and THF (0.7 M) were subsequently added. The mixture was stirred at room temperature for a few minutes and then cooled down to -78 ℃. 3-Butenylmagnesium bromide (10 mmol, 0.5 M in THF) was added dropwise to the mixture. After completion, the reaction mixture was allowed to warm up to room temperature overnight. Then the reaction was quenched by adding saturated aqueous NH_4Cl solution and diluted with EA. The layers were separated, and the aqueous layer was extracted with EA. The combined organic layers were dried over $MgSO_4$, concentrated in *vacuo*. Purification of the crude product by column chromatography on silica gel provided the ketone.

Step 2: To hydroxylamine hydrochloride (1.2 equiv.) and sodium acetate (1.2 equiv.) was added the solution of alkenyl ketone in MeOH (0.3 M). The reaction mixture was heated to 75 ℃ and stirred overnight. The resulting mixture was cooled

down to room temperature, diluted with brine and extracted with EA. The organic layer was dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by column chromatography on silica gel provided the oxime.

Step 3: To a solution of *γ,δ*-unsaturated oxime (1.0 equiv) in DCM (1.0 M) were added triethylamine (2.0 equiv) and C_6F_5COCl (1.2 equiv) at 0 ºC. After stirring for 6 h, water was added and the mixture was diluted with EA. The organic layer was washed with water and dried over anhydrous Na_2SO_4. The solvent was removed in *vacuo* and the residue was subjected to column chromatography with the indicated eluent system to obtain *γ,δ*-unsaturated oxime esters **1c**-**1d**, **1f**- **1h**, **1k**.

Synthesis of compound **1n**



1n

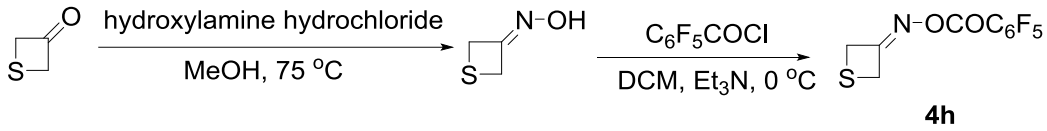
To a flask charged with potassium carbonate (1.2 equiv) was added a solution of ethyl benzoacetate (10 mmol) and 1-bromo-3-methylbut-2-ene (1.08 equiv) in acetone (0.4 M). The reaction mixture was heated at reflux overnight. After cooling, solids were removed by filtration and washed with acetone. The combined filtrates were concentrated in *vacuo*. Then the residue was dissolved in ethanol. 10 % Aqueous sodium hydroxide solution was added to the solution. The reaction mixture was heated at reflux for 3 h. After cooling, the mixture was extracted with EA. The combined organic layers were dried over $MgSO_4$ and concentrated in *vacuo*. Purification of the crude product by column chromatography on silica gel provided 5-methyl-1-phenylhex-4-en-1-one.

To a flask charged with hydroxylamine hydrochloride (1.2 equiv.) and sodium acetate (1.2 equiv.) was added the solution of 5-methyl-1-phenylhex-4-en-1-one in

MeOH (0.3 M). The reaction mixture was heated to 75 ℃ and stirred overnight. The resulting mixture was cooled down to room temperature, diluted with brine and extracted with EA. The organic layer was dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by column chromatography on silica gel provided the 5-methyl-1-phenylhex-4-en-1-one oxime.

To a solution of 5-methyl-1-phenylhex-4-en-1-one oxime (1.0 equiv) in DCM (1.0 M) were added triethylamine (2.0 equiv) and C_6F_5COCl (1.2 equiv) at 0 ℃. After stirring for 6 h, water was added and the mixture was diluted with EA. The organic layer was washed with water and dried over anhydrous Na_2SO_4. The solvent was removed in *vacuo* and the residue was subjected to column chromatography with the indicated eluent system to obtain 1-phenylhex-4-en-1-one *o*-perfluorobenzoyl oxime **1n**.

Synthesis of oxime ester **4h**



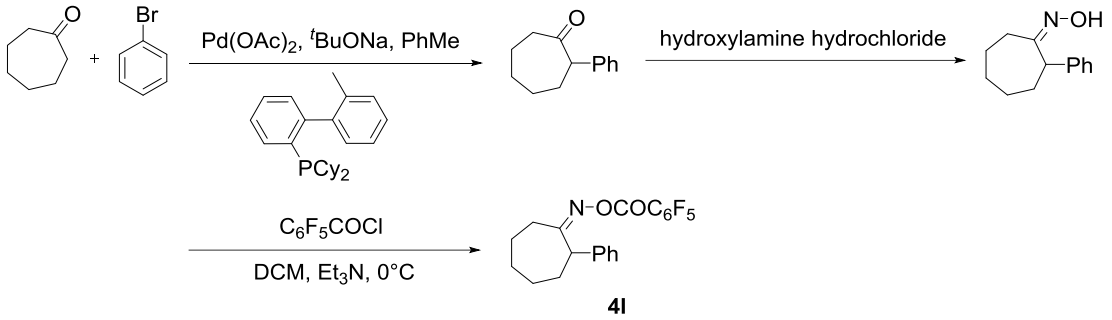
4h

To a flask charged with hydroxylamine hydrochloride (1.2 equiv.) and sodium acetate (1.2 equiv.) was added a solution of thietan-3-one (10 mmol) in MeOH (0.3 M). The reaction mixture was heated to 75 ℃ and stirred overnight. The resulting mixture was cooled down to room temperature, diluted with brine and extracted with EA. The organic layer was dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by column chromatography on silica gel provided the thietan-3-one oxime.

To a solution of thietan-3-one oxime (1.0 equiv) in DCM (1.0 M) were added triethylamine (2.0 equiv) and C_6F_5COCl (1.2 equiv) at 0 ℃. After stirring for 6 h, water was added and the mixture was diluted with EA. The organic layer was washed with water and dried over anhydrous Na_2SO_4. The solvent was removed in *vacuo* and the residue was subjected to column chromatography with the indicated eluent system

to obtain thietan-3-one *o*-perfluorobenzoyl oxime **4h**.

Synthesis of oxime ester **4l**



4l

A 100 mL flame-dried schlenk tube was filled with argon, $Pd(OAc)_2$ (2.5 mol%), ligand (5 mol%), ^{t}BuONa (1.3 equiv.), ketone (10.0 mmol), aryl bromides (1.3 equiv) and toluene (1.0 M). The tube was then sealed and heated at 45 ℃ under stirring for 12 hours, and finally quenched with H_2O. The resulting mixture was extracted with EA. The organic layer was dried over Na_2SO_4 and concentrated in vacuo. Purification of the crude product by column chromatography on silica gel provided the 2-phenylcycloheptan-1-one.

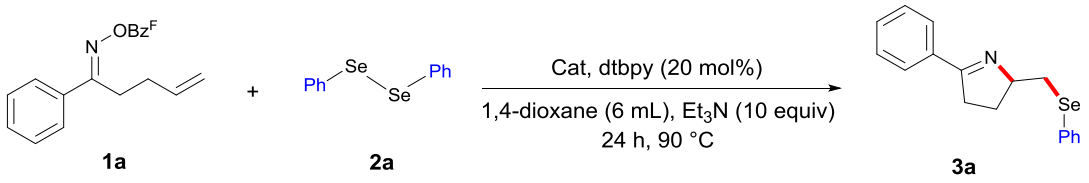
To a flask charged with hydroxylamine hydrochloride (1.2 equiv.) and sodium acetate (1.2 equiv.) was added a solution of 2-phenylcycloheptan-1-one (1.0 equiv) in MeOH (0.3 M). The reaction mixture was heated to 75 ℃ and stirred overnight. The resulting mixture was cooled down to room temperature, diluted with brine and extracted with EA. The organic layer was dried over Na_2SO_4 and concentrated in *vacuo*. Purification of the crude product by column chromatography on silica gel provided the 2-phenylcycloheptan-1-one oxime.

To a solution of 2-phenylcycloheptan-1-one oxime (1.0 equiv) in DCM (1.0 M) were added triethylamine (2.0 equiv) and C_6F_5COCl (1.2 equiv) at 0 ℃. After stirring for 6 h, water was added and the mixture was diluted with EA. The organic layer was washed with water and dried over anhydrous Na_2SO_4. The solvent was removed in *vacuo* and the residue was subjected to column chromatography with the indicated eluent system to obtain thietan-3-one *o*-perfluorobenzoyl oxime **4l**.

2.2 Screening of reaction conditions

To a screw-cap vial equipped with a magnetic stirring bar were added catalyst (0.02 mmol), ligand (0.02 mmol) and solvent under argon. The mixture was stirred at 80 ℃ for 30 min. The *γ,δ*-unsaturated *O*-acyloxime (0.20 mmol), aryl diselenide (0.15 mmol) and base (2.00 mmol) were added sequentially under argon. After the addition, the mixture was stirred at the specified temperature for the indicated reaction time. Then, the mixture was allowed to cool down to room temperature and the solvent was removed in *vacuo*. Then, the residue was re-dissolved and flushed through a glass pipette filled with silica gel inside (approx. 4.0 cm in length) using ethyl acetate (approx. 5.0 mL) as an eluent to remove the metal salt. Yields were determined by GC using *n*-dodecane as the internal calibration standard. The results are summarized in Tables S3-S8.

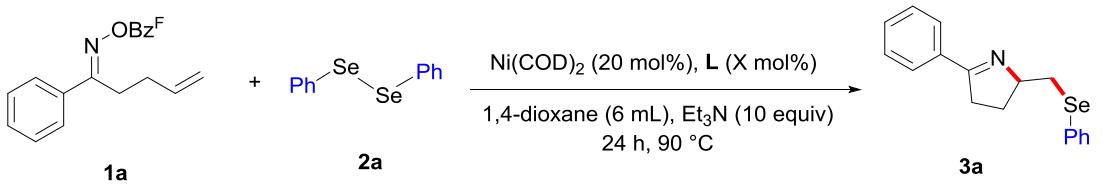
Table S3 Screening of metal salts.



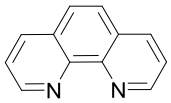
1a **2a** **3a**

Entry	Cat (X mol%)	L	GC Yield (%)
1	No Cat	--	N.P
2	No Cat	dtbpy	N.P
3	$NiBr_2$ (20)	--	17
4	$NiBr_2$ (20)	dtbpy	58
5	$AgSbF_6$ (20)	dtbpy	40
6	CuOAc (20)	dtbpy	74
7	$FeCl_2$ (20)	dtbpy	77
8	$FeCl_3$ (20)	dtbpy	73
9	$Pd(OAc)_2$ (20)	dtbpy	57
10	$Ni(OTf)_2$ (20)	dtbpy	54
11	$NiCl_2$•glyme (20)	dtbpy	21
12	$Ni(COD)_2$ (20)	dtbpy	85
13	$Ni(COD)_2$ (20)	--	17
14	$Ni(COD)_2$ (5)	dtbpy	38
15	$Ni(COD)_2$ (10)	dtbpy	58
16	$Ni(COD)_2$ (20)	dtbpy	64

Table S4 Screening of Ligands

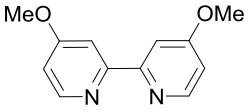


1a **2a** **3a**



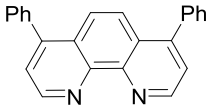
L1

L1 (5 mol%): 78%
L1 (10 mol%): 82%
L1 (20 mol%): 90%
L1 (30 mol%): 75%



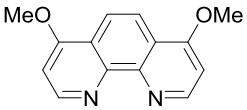
L5 (20 mol%)

50%



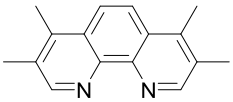
L6 (20 mol%)

44%



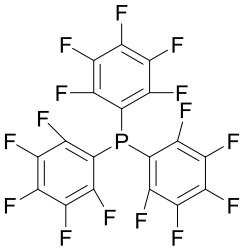
L7 (20 mol%)

13%



L8 (20 mol%)

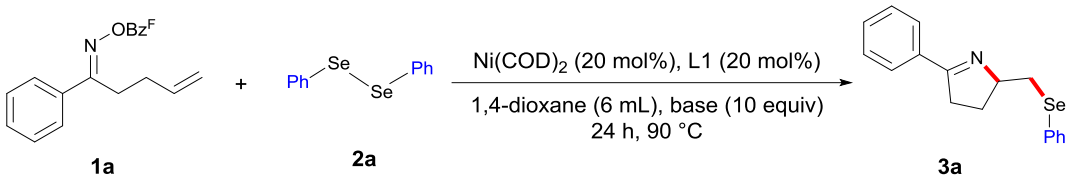
34%



L9 (20 mol%)

29%

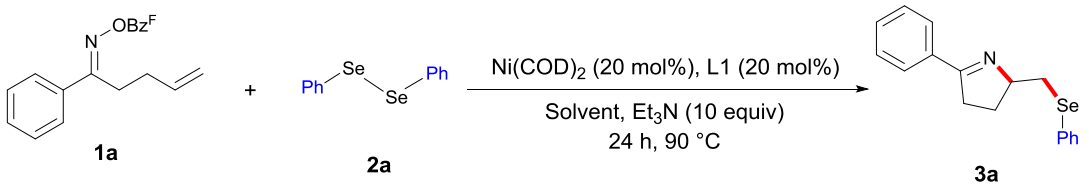
Table S5 Screening of bases.



1a **2a** **3a**

Entry	Base	GC Yield (%)
1	None	/
2	Et_3N	90
3	DBU	7
4	EDA	/
5	$^{i}Pr_2NEt$	81
6	TMEDA	83
7	K_2CO_3	25
8	Na_2CO_3	26
9	Cs_2CO_3	46
10	$^{t}BuOK$	/
11	NaOH	38
12	Et_3N (5 equiv)	74

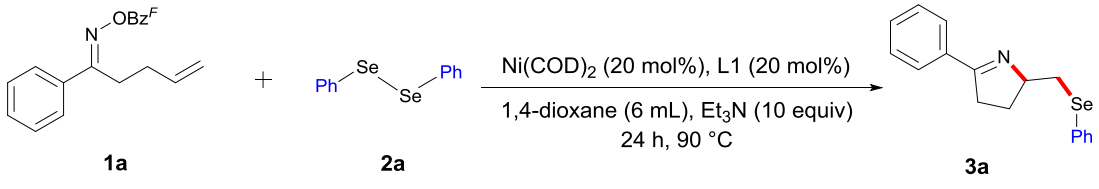
Table S6 Screening of solvents.



1a 2a 3a

Entry	Solvent (X mL)	GC Yield (%)
1	1,4-dioxane (6 mL)	90
2	DMF (6 mL)	7
3	CH_3CN (6 mL)	12
4	toluene (6 mL)	45
5	THF (6 mL)	24
6	DMSO (6 mL)	17
7	DCM (6 mL)	18
8	EA (6 mL)	13
9	NMP (6 mL)	19
10	EtOH (6 mL)	trace
11	MeOH (6 mL)	trace
12	cyclohexane (6 mL)	6
13	1,4-dioxane (2 mL)	61
14	1,4-dioxane (4 mL)	79
15	1,4-dioxane (8 mL)	41

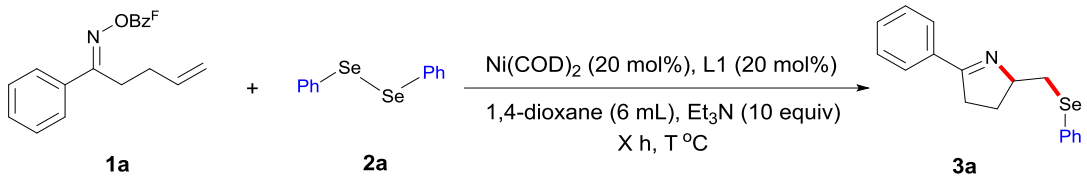
Table S7 Screening equivalences of the diselenide 2a



1a 2a 3a

Entry	2a (X equiv)	GC Yield (%)[b]
1	0.5	58
2	1.0	80
3	1.5	90
4	2.0	92

Table S8 Screening of reaction time and temperature



1a **2a** **3a**

Entry	Temperature (°C)	Time (h)	Conv (%)	GC Yield (%)
1	r.t	24	1<	/
2	40	24	17	10
3	60	24	21	14
4	80	24	>99	85
5	90	24	>99	90
6	90	6	89	86
7	90	12	>99	90
8	90	36	>99	89

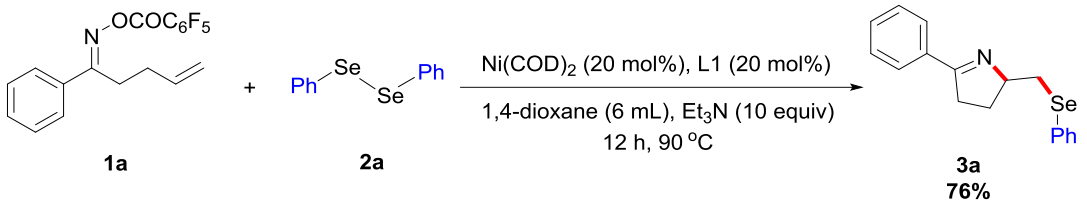
2.3 General procedure for the selenylation of oxime esters

Method A: To a screw-cap vial equipped with a magnetic stirring bar were added $Ni(COD)_2$ (0.04 mmol, 11.0 mg, 0.2 equiv), **L1** (0.04 mmol, 7.2 mg, 0.2 equiv) and 1,4-dioxane (6 mL) under argon. The mixture was stirred at 80 ℃ for 30 min. Then, oxime esters (0.20 mmol, 1.0 equiv), aryl diselenides (0.30 mmol, 1.5 equiv) and Et_3N (2.00 mmol, 202.4 mg, 10.0 equiv) were added sequentially under argon. After the addition, the reaction mixture was stirred at 90 ℃ for 12 h. Then, the mixture was allowed to cool down to room temperature and the solvent was removed *in vacuo*. The crude products were purified by column chromatography.

Method B: To a screw-cap vial equipped with a magnetic stirring bar were added $FeCl_2$ (0.04 mmol, 5.1 mg, 0.2 equiv), **L1** (0.04 mmol, 7.2 mg, 0.2 equiv) and 1,4-dioxane (6 mL) under argon. The mixture was stirred at 80 ℃ for 30 min. Then, the oxime esters (0.20 mmol, 1.0 equiv), aryl diselenides (0.30 mmol, 1.5 equiv) and Et_3N (2.00 mmol, 202.4 mg, 10.0 equiv) were added sequentially under argon. After

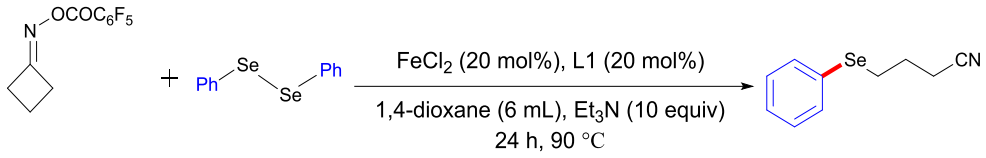
the addition, the reaction mixture was stirred at 90 ℃ for 24 h. Then, the mixture was allowed to cool down to room temperature and the solvent was removed *in vacuo*. The crude products were purified by column chromatography.

2.4 Procedure for reactions performed at a larger scale



1a **2a** **3a** **76%**

To a screw-cap vial equipped with a magnetic stirring bar were added $Ni(COD)_2$ (0.16 mmol, 44.0 mg, 0.2 equiv), **L1** (0.16 mmol, 28.8 mg, 0.2 equiv) and 1,4-dioxane (24 mL) under argon. The mixture was stirred at 80 ℃ for 30 min. Then, *γ,δ*-unsaturated oxime ester **1a** (0.80 mmol, 1.0 equiv), aryl diselenide **2a** (1.20 mmol, 1.5 equiv) and Et_3N (8.00 mmol, 809.5 mg, 10.0 equiv) were added sequentially under argon. After the addition, the reaction mixture was stirred at 90 ℃ for 12 h. Then, the mixture was allowed to cool down to room temperature and the solvent was removed *in vacuo*. The crude products were purified by column chromatography. The isolated yield of **3a** is 76%.

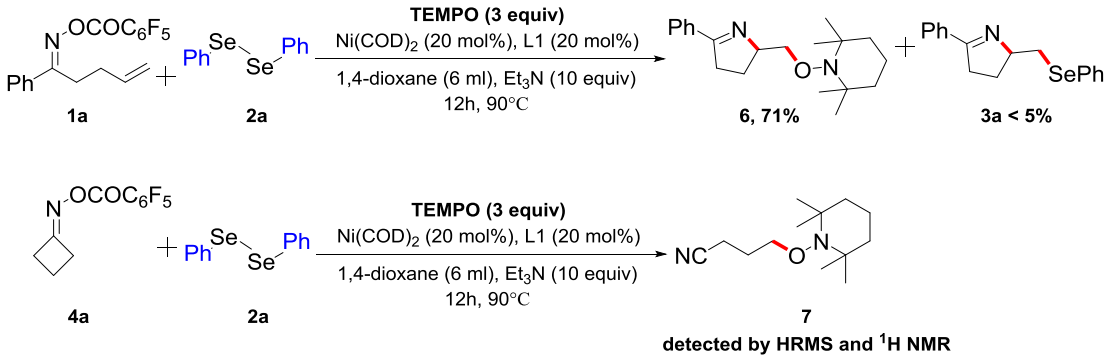


4a **2a** **5a** **79%**

To a screw-cap vial equipped with a magnetic stirring bar were added $FeCl_2$ (0.2 mmol, 25.4 mg, 0.2 equiv), **L1** (0.2 mmol, 36.0 mg, 0.2 equiv) and 1,4-dioxane (30 mL) under argon. The mixture was stirred at 80 ℃ for 30 min. Then, the cycloketoxime ester **4a** (1.0 mmol, 1.0 equiv), aryl diselenide **2a** (1.5 mmol, 1.5 equiv) and Et_3N (10.0 mmol, 1.01 g, 10.0 equiv) were added sequentially under argon. After the addition, the reaction mixture was stirred at 90 ℃ for 24 h. Then, the mixture was allowed to cool down to room temperature and the solvent was removed *in vacuo*.

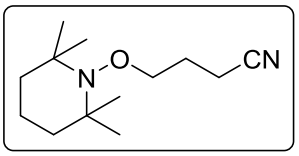
The crude products were purified by column chromatography. The isolated yield of **5a** is 79%.

3. Trapping the radical intermediate with TEMPO



To a screw-cap vial equipped with a magnetic stirring bar were added $Ni(COD)_2$ (0.04 mmol, 20.0 mol%), **L1** (0.04 mmol, 20.0 mol%) and 1,4-dioxane (6.0 mL) under argon. The mixture was stirred at 80 ℃ for 30 min. Then, oxime ester (0.20 mmol,1.0 equiv), **2a** (0.30 mmol, 1.5 equiv), Et_3N (2.00 mmol, 10.0 equiv) and TEMPO (0.60 mmol, 3 equiv) were added sequentially under argon. After the addition, the reaction mixture was stirred at 90 ℃ for 12 h. Then, the mixture was allowed to cool down to room temperature and the solvent was removed in *vacuo*. The crude products were purified by column chromatography to yield **6** in 71% yield. However, compound **7** decomposed during purification. The mass of compound 7 was detected by HRMS.

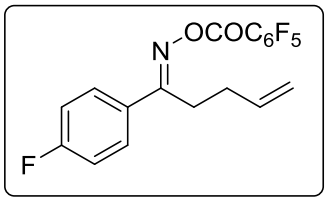
4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)butanenitrile (7)



HRMS (ESI): *m/z* calcd for $C_{14}H_{13}NSe$ $[M+H]^+$ 225.1966, found 225.1958.

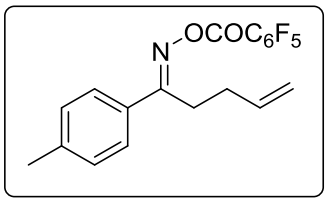
4. Spectral Data

1-(4-Fluorophenyl)pent-4-en-1-one O-perfluorobenzoyl oxime (1c)



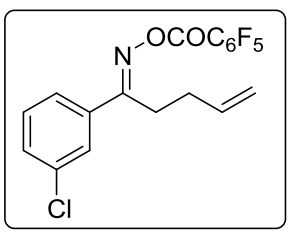
1c: (59%, 2.2 g, white solid; m.p. 68-70 ℃); **^{1}H NMR** (400 MHz, Chloroform-d) *δ* 7.76 (dd, *J* = 8.9, 5.3 Hz, 2H), 7.13 (t, *J* = 8.6 Hz, 2H), 5.78 (td, *J* = 16.9, 6.6 Hz, 1H), 5.08 – 4.98 (m, 2H), 3.02 – 2.93 (m, 2H), 2.38 – 2.27 (m, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 167.1, 164.6 (d, *J* = 251.9 Hz), 156.4, 146.5 (d, *J* = 27.0 Hz), 144.6(d, *J* = 78.5 Hz), 142.8 (d, *J* = 36.1 Hz), 138.7 (d, *J* = 34.4 Hz), 137.0 (d, *J* = 34.6 Hz), 136.0, 129.7 (d, *J* = 8.7 Hz), 129.2 (d, *J* = 3.3 Hz), 116.2, 116.0 (d, *J* = 21.9 Hz), 107.0 (d, *J* = 35.9 Hz), 30.7, 28.1; **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -108.65, -136.51 – -137.94 (m), -147.29 (tt, *J* = 21.0, 5.0 Hz), -159.59 – -159.76 (m); **HRMS** (ESI): *m/z* calcd for $C_{18}H_{11}F_6NO_2$ $[M+Na]^+$ 410.0591, found 410.0590.

1-(P-tolyl)pent-4-en-1-one O-perfluorobenzoyl oxime (1d)



1d: (78%, 3.0 g, white solid; m.p. 43-45 ℃); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.68 – 7.62 (m, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 5.80 (td, *J* = 16.9, 6.6 Hz, 1H), 5.09 – 4.97 (m, 2H), 3.01 – 2.93 (m, 2H), 2.40 (s, 3H), 2.35 – 2.28 (m, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 167.9, 156.5, 146.4 (d, *J* = 14.7 Hz), 144.5 (d, *J* = 72.7 Hz), 142.6 (d, *J* = 26.7 Hz), 141.6, 138.7 (d, *J* = 34.3 Hz), 137.0 (d, *J* = 34.3 Hz), 136.2, 130.1, 129.5, 127.4, 115.9, 107.2 (d, *J* = 32.6 Hz), 30.8, 28.0, 21.3; **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -136.90 – -137.58 (m), -147.65 (tt, *J* = 21.0, 4.9 Hz), -159.43 – -160.29 (m); **HRMS** (ESI): *m/z* calcd for $C_{19}H_{14}F_5NO_2$ $[M+Na]^+$ 406.0842, found 406.0848.

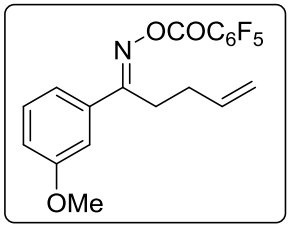
1-(3-Chlorophenyl)pent-4-en-1-one O-perfluorobenzoyl oxime (1f)



1f: (86%, 3.4 g, white solid; m.p. 86-88 ℃); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.74 (s, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 8.1 Hz, 1H), 7.43 – 7.34 (m, 1H), 5.78 (dtt, *J* = 16.7, 9.2, 4.5 Hz, 1H), 5.10 – 4.97 (m, 2H), 3.02 – 2.91 (m, 2H), 2.33 (q, *J* = 7.6 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 167.2, 156.4, 146.6 (d, *J* = 26.9 Hz), 144.7 (d, *J* = 67.7 Hz), 142.9 (d, *J* = 26.6 Hz), 138.8 (d, *J* = 34.7 Hz), 137.1 (d, *J* = 34.3 Hz), 135.9, 135.1, 135.0, 131.2, 130.2, 127.6, 125.7, 116.5, 106.9 (d, *J* = 31.4 Hz), 30.7, 28.2; **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -136.53 – -137.08 (m), -147.05 (tt, *J* =

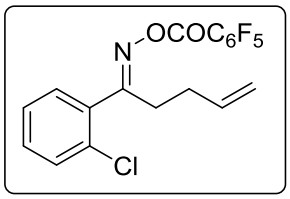
20.9, 5.0 Hz), -159.16 – -159.76 (m); **HRMS** (ESI): *m/z* calcd for $C_{18}H_{11}ClF_5NO_2$ $[M+Na]^+$ 426.0296, found 426.0296.

1-(3-Methoxyphenyl)pent-4-en-1-one O-perfluorobenzoyl oxime (1g)



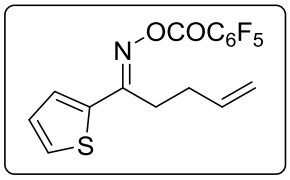
1g: (77%, 3.0 g, white solid; m.p. 43-44 ℃) **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.34 (t, *J* = 9.1 Hz, 1H), 7.30 (s, 2H), 7.02 (d, *J* = 8.1 Hz, 1H), 5.86 – 5.72 (m, 1H), 5.02 (t, *J* = 13.0 Hz, 2H), 3.84 (s, 3H), 2.97 (t, *J* = 9.3 Hz, 2H), 2.33 (q, *J* = 7.6 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 168.2, 159.9, 156.6, 146.5 (d, *J* = 17.8 Hz), 144.7 (d, *J* = 71.1 Hz), 142.8 (d, *J* = 26.6 Hz), 138.8 (d, *J* = 34.3 Hz), 137.1 (d, *J* = 34.4 Hz), 136.2, 134.5, 130.0, 120.0, 117.1, 116.3, 112.8, 107.2 (d, *J* = 31.3 Hz), 55.5, 30.9, 28.5; **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -136.89 – -137.21 (m), -147.50 (tt, *J* = 21.0, 4.9 Hz), -159.34 – -159.90 (m); **HRMS** (ESI): *m/z* calcd for $C_{19}H_{14}F_5NO_3$ $[M+Na]^+$ 422.0792, found 422.0794.

1-(2-Chlorophenyl)pent-4-en-1-one O-perfluorobenzoyl oxime (1h)



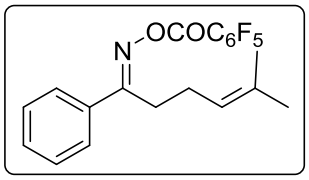
1h: (67%, 2.7 g, white solid; m.p.42-43 ℃); **^{1}H NMR** (600 MHz, Chloroform-d) δ 7.39 – 7.23 (m, 4H), 5.72 – 5.62 (m, 1H), 4.98 – 4.91 (m, 2H), 2.99 – 2.93 (m, 2H), 2.17 (q, J = 8.6, 7.8 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 169.8, 156.5, 146.6 (d, *J* = 27.1 Hz), 144.7 (d, *J* = 44.0 Hz), 142.8 (d, *J* = 26.4 Hz), 138.8 (d, *J* = 29.0 Hz), 137.1 (d, *J* = 30.3 Hz), 136.1, 133.2, 132.7, 131.2, 131.0, 130.1, 127.0, 116.2, 106.9 (d, *J* = 29.4 Hz), 30.6, 29.7; **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -136.83 (dp, *J* = 17.5, 5.8 Hz), -147.11 – -147.37 (m), -159.47 – -160.09 (m); **HRMS** (ESI): *m/z* calcd for $C_{18}H_{11}ClF_5NO_2$ $[M+Na]^+$ 426.0296, found 426.0288.

1-(Thiophen-2-yl)pent-4-en-1-one O-perfluorobenzoyl oxime (1k)



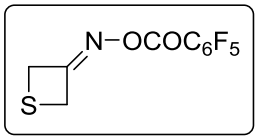
1k: (66%, 2.4 g, yellow solid; m.p. 103-104 ℃); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.53 – 7.46 (m, 2H), 7.11 (dd, *J* = 5.1, 3.7 Hz, 1H), 5.83 (td, *J* = 16.9, 6.7 Hz, 1H), 5.16 – 4.98 (m, 2H), 3.01 – 2.92 (m, 2H), 2.48 – 2.39 (m, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 163.3, 156.4, 146.5 (d, *J* = 11.3 Hz), 144.9 – 144.3 (m), 138.7 (d, *J* = 12.9 Hz), 137.1 (d, *J* = 28.2 Hz), 136.2 (d, *J* = 10.8 Hz), 130.4, 130.2, 127.7, 116.4, 107.0 (d, *J* = 33.8 Hz), 31.4, 29.0; **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -136.49 – -137.65 (m), -147.37 (tt, *J* = 21.2, 5.1 Hz), -159.07 – -160.23 (m); **HRMS** (ESI): *m/z* calcd for $C_{16}H_{10}F_5NO_2S$ $[M+Na]^+$ 398.0250, found 398.0255.

5-Methyl-1-phenylhex-4-en-1-one O-perfluorobenzoyl oxime (1n)



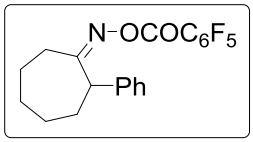
1n: (76%, 30 g, white solid; m.p. 65-66 ℃); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.77 – 7.72 (m, 2H), 7.52 – 7.39 (m, 3H), 5.10 (tt, *J* = 7.4, 1.6 Hz, 1H), 2.94 – 2.89 (m, 2H), 2.27 (q, *J* = 7.7 Hz, 2H), 1.64 (s, 3H), 1.50 (s, 3H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 168.7, 156.7, 146.5 (d, *J* = 18.8 Hz), 144.6 (d, *J* = 65.9 Hz), 142.7 (d, *J* = 17.5 Hz), 138.8 (d, *J* = 28.9 Hz), 137.1 (d, *J* = 34.2 Hz), 133.8, 133.3, 131.1, 128.9, 127.6, 122.1, 107.3 (d, *J* = 32.5 Hz), 29.1, 25.7, 25.4, 17.6; **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -136.67 – -137.76 (m), -147.65 (tt, *J* = 21.0, 4.8 Hz), -159.24 – -160.33 (m); **HRMS** (ESI): *m/z* calcd for $C_{20}H_{16}F_5NO_2$ $[M+Na]^+$ 420.0999, found 420.0997.

Thietan-3-one O-perfluorobenzoyl oxime (4h)



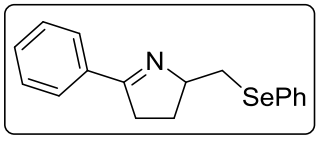
4h: (60%, 1.7 g, yellow solid; m.p. 88-90 ℃)**; ^{1}H NMR** (600 MHz, Chloroform-*d*) δ 4.24 – 4.18 (m, 4H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 163.9, 156.0, 146.6 (d, *J* = 16.4 Hz), 144.8 (d, *J* = 25.9 Hz), 143.0 (d, *J* = 26.6 Hz), 138.8 (d, *J* = 28.6 Hz), 137.1 (d, *J* = 33.3 Hz), 106.3 (d, *J* = 31.4 Hz), 35.4 (d, *J* = 13.5 Hz); **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -136.33 – -137.22 (m), -146.72 (tt, *J* = 20.9, 5.2 Hz), -159.23 – -159.73 (m); **HRMS** (ESI): *m/z* calcd for $C_{10}H_4F_5NO_2S$ $[M+Na]^+$ 319.9781, found 319.9783.

2-phenylcycloheptan-1-one O-perfluorobenzoyl oxime (4l)



4l: (33%,1.3g, transparent oil); **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.43 – 7.30 (m, 5H), 4.12 (dd, J = 10.9, 6.5 Hz, 1H), 3.10 (dd, J = 12.6, 4.5 Hz, 1H), 2.49 – 2.39 (m, 1H), 2.24 – 2.13 (m, 1H), 2.10 – 1.92 (m, 4H), 1.62 (d, J = 9.6 Hz, 1H), 1.49 (t, J = 11.3 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 174.71, 156.95, 146.27 (d, *J* = 17.8 Hz), 144.41 (d, *J* = 49.9 Hz), 142.53 (d, *J* = 9.9 Hz), 140.54, 138.63 (d, *J* = 24.1 Hz), 136.95 (d, *J* = 22.0 Hz), 128.70, 127.35, 127.12, 107.40 (d, *J* = 32.5 Hz), 48.58, 31.25, 30.73, 27.96, 26.35, 25.75.; **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -136.92 – -137.48 (m), -148.08 (t, *J* = 20.9 Hz), -159.98 (dd, *J* = 20.8, 14.3 Hz); **HRMS** (ESI): *m/z* calcd for $C_{20}H_{16}F_5NO_2$ $[M+Na]^+$ 420.0998, found 420.0996.

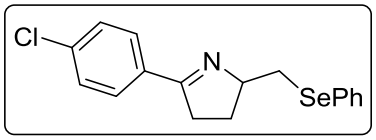
5-Phenyl-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3a)



3a: (**Method A:** 90%, 56.9 mg**, Method B:**80%, 50.4 mg; yellow oil; NMR data are in accordance with literature values[15]); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.84 –

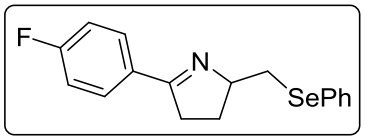
7.75 (m, 2H), 7.60 – 7.52 (m, 2H), 7.41 (dd, *J* = 9.1, 6.9 Hz, 3H), 7.24 (d, *J* = 6.8 Hz, 3H), 4.58 – 4.47 (m, 1H), 3.47 (dd, *J* = 12.0, 4.7 Hz, 1H), 3.09 (dd, *J* = 12.1, 7.9 Hz, 2H), 2.99 – 2.85 (m, 1H), 2.29 (dt, *J* = 8.0, 2.5 Hz, 1H), 1.88 – 1.74 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 134.4, 132.7, 130.7, 129.13, 128.5, 127.9, 126.9, 125.9, 73.0, 35.5, 34.5, 28.7.

5-(4-Chlorophenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3b)



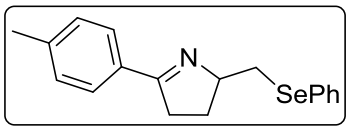
3b: (**Method A:** 66%, 46.1 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 8.6 Hz, 2H), 7.54 (dd, *J* = 7.6, 1.9 Hz, 2H), 7.36 (d, *J* = 8.6 Hz, 2H), 7.25 – 7.18 (m, 3H), 4.52 (s, 1H), 3.43 (dd, *J* = 12.1, 4.8 Hz, 1H), 3.17 – 2.98 (m, 2H), 2.96 – 2.82 (m, 1H), 2.36 – 2.22 (m, 1H), 1.88 – 1.74 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 172.4, 136.8, 132.9, 132.7, 130.6, 129.2, 129.2, 128.8, 126.9, 73.1, 35.5, 34.5, 28.8; **HRMS** (ESI): *m/z* calcd for $C_{17}H_{16}ClNSe$ $[M+H]^+$ 350.0214, found 350.0214.

5-(4-Fluorophenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3c)



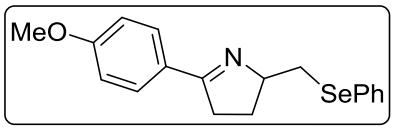
3c: (**Method A:** 78%, 51.9 mg, yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.79 (dd, *J* = 8.7, 5.6 Hz, 2H), 7.58 – 7.54 (m, 2H), 7.24 (d, *J* = 7.3 Hz, 3H), 7.08 (t, *J* = 8.6 Hz, 2H), 4.52 (d, *J* = 6.4 Hz, 1H), 3.45 (dd, *J* = 12.1, 4.8 Hz, 1H), 3.16 – 3.01 (m, 2H), 2.94 – 2.85 (m, 1H), 2.34 – 2.25 (m, 1H), 1.86 – 1.77 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 172.2, 164.3 (d, *J* = 250.7 Hz), 132.7, 130.7 (d, *J* = 3.2 Hz), 130.7, 129.9 (d, *J* = 8.7 Hz), 129.1, 126.9, 115.5 (d, *J* = 21.7 Hz), 73.0, 35.5, 34.5, 28.8; **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -109.68 – -109.86 (m); **HRMS** (ESI): *m/z* calcd for $C_{17}H_{16}FNSe$ $[M+H]^+$ 334.0510, found 334.0516.

2-((Phenylselanyl)methyl)-5-(p-tolyl)-3,4-dihydro-2*H*-pyrrole (3d)



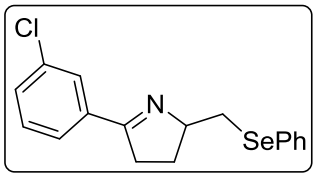
3d: (**Method A:** 85%, 55.9 mg, yellow solid; m.p. 44-45 ℃); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.71 (s, 2H), 7.58 – 7.53 (m, 2H), 7.27 – 7.18 (m, 5H), 4.54 – 4.46 (m, 1H), 3.48 (dd, *J* = 12.1, 4.7 Hz, 1H), 3.11 – 3.01 (m, 2H), 2.90 (dt, *J* = 17.1, 8.7 Hz, 1H), 2.38 (s, 3H), 2.32 – 2.23 (m, 1H), 1.84 – 1.74 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 173.3, 140.9, 132.7, 131.7, 130.7, 129.2, 129.1, 127.9, 126.8, 72.8, 35.4, 34.5, 28.7, 21.6; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NSe$ $[M+H]^+$ 330.0761, found 330.0768.

5-(4-Methoxyphenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3e)



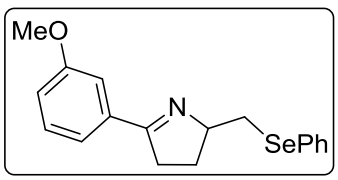
3e: (**Method A:** 81%, 56.0 mg, yellow solid; m.p. 68-69 ℃); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 8.8 Hz, 2H), 7.55 (d, *J* = 7.9 Hz, 2H), 7.23 (d, *J* = 7.1 Hz, 3H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.54 – 4.43 (m, 1H), 3.84 (s, 3H), 3.46 (dd, *J* = 12.0, 4.7 Hz, 1H), 3.13 – 2.98 (m, 2H), 2.95 – 2.81 (m, 1H), 2.32 – 2.22 (m, 1H), 1.77 (d, *J* = 23.5 Hz, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 172.9, 161.7, 132.7, 130.7, 129.6, 129.1, 127.1, 126.8, 113.8, 72.7, 55.5, 35.4, 34.6, 28.8; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NOSe$ $[M+H]^+$ 346.0710, found 346.0711.

5-(3-Chlorophenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3f)



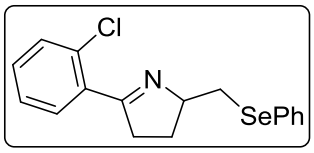
3f: (**Method A:** 78%, 54.4 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.79 (s, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 7.8 Hz, 2H), 7.42 – 7.18 (m, 5H), 4.53 (t, *J* = 6.8 Hz, 1H), 3.43 (dd, *J* = 12.2, 4.9 Hz, 1H), 3.18 – 2.96 (m, 2H), 2.93 – 2.80 (m, 1H), 2.34 – 2.21 (m, 1H), 1.88 – 1.76 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.3, 136.2, 134.7, 132.8, 130.6, 130.6, 129.8, 129.2, 128.0, 127.0, 126.1, 73.2, 35.5, 34.4, 28.7; **HRMS** (ESI): *m/z* calcd for $C_{17}H_{16}ClNSe$ $[M+H]^+$ 350.0214, found 350.0224.

5-(3-Methoxyphenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3g)



3g: (**Method A:** 80%, 55.2 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.59 – 7.50 (m, 2H), 7.40 (s, 1H), 7.36 – 7.27 (m, 2H), 7.23 (d, *J* = 6.6 Hz, 3H), 6.98 (d, *J* = 7.8 Hz, 1H), 4.58 – 4.47 (m, 1H), 3.84 (s, 3H), 3.47 (dd, *J* = 12.1, 4.6 Hz, 1H), 3.06 (t, *J* = 12.3 Hz, 2H), 2.98 – 2.84 (m, 1H), 2.35 – 2.22 (m, 1H), 1.83 (d, *J* = 16.9 Hz, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 159.8, 135.8, 132.8, 130.7, 129.5, 129.1, 126.9, 120.7, 117.2, 112.3, 73.0, 55.5, 35.6, 34.5, 28.7; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NOSe$ $[M+H]^+$ 346.0710, found 346.0713.

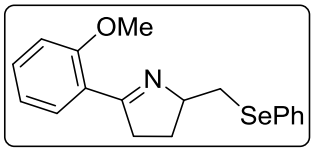
5-(2-Chlorophenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3h)



3h: (**Method A:** 74%, 51.6 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 6.8 Hz, 2H), 7.53 (d, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.31 (dd, *J* = 16.0, 8.0 Hz, 5H), 4.47 (d, *J* = 13.8 Hz, 1H), 3.49 (dd, *J* = 12.1, 4.8 Hz, 1H), 3.20 – 2.98 (m, 3H), 2.38 – 2.25 (m, 1H), 1.90 – 1.76 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.6, 135.3, 132.6, 132.5, 130.6, 130.6, 130.5, 130.2,

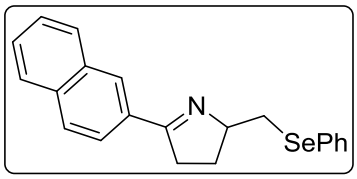
129.2, 126.9, 126.9, 72.6, 39.0, 34.0, 29.5; **HRMS** (ESI): *m/z* calcd for $C_{17}H_{16}ClNSe$ $[M+H]^+$ 350.0214, found 350.0214.

5-(2-Methoxyphenyl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3i)



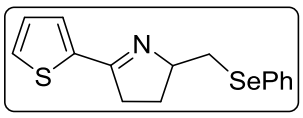
3i: (**Method A:** 68%, 47.0 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.72 – 7.68 (m, 1H), 7.56 (d, *J* = 9.6 Hz, 2H), 7.40 – 7.33 (m, 1H), 7.22 (d, *J* = 6.0 Hz, 2H), 6.99 – 6.88 (m, 2H), 4.46 – 4.34 (m, 1H), 3.84 (s, 3H), 3.48 (dd, *J* = 12.0, 4.6 Hz, 1H), 3.18 – 2.93 (m, 3H), 2.31 – 2.17 (m, 1H), 1.82 – 1.68 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.5, 158.3, 132.6, 131.5, 130.8, 130.3, 129.14, 126.8, 124.7, 120.8, 111.4, 71.7, 55.6, 38.9, 34.3, 29.3; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NOSe$ $[M+H]^+$ 346.0710, found 346.0713.

5-(Naphthalen-2-yl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3j)



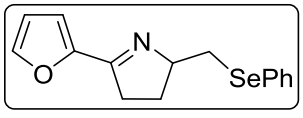
3j: (**Method A:** 66%, 48.2 mg, yellow solid; m.p. 76-77 ℃); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.18 (s, 1H), 8.06 (d, *J* = 8.6 Hz, 1H), 7.90 (dd, *J* = 17.1, 8.0 Hz, 3H), 7.63 – 7.52 (m, 4H), 7.31 – 7.26 (m, 3H), 4.60 (d, *J* = 13.9 Hz, 1H), 3.55 (dd, *J* = 12.1, 4.8 Hz, 1H), 3.32 – 3.01 (m, 3H), 2.38 (q, *J* = 13.3 Hz, 1H), 1.97 – 1.83 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 134.6, 133.1, 132.8, 132.0, 130.7, 129.2, 128.9, 128.5, 128.2, 127.9, 127.3, 126.9, 126.5, 124.8, 73.1, 35.5, 34.6, 28.8; **HRMS** (ESI): *m/z* calcd for $C_{21}H_{19}NSe$ $[M+H]^+$ 366.0761, found 366.0770.

2-((Phenylselanyl)methyl)-5-(thiophen-2-yl)-3,4-dihydro-2*H*-pyrrole (3k)



3k: (**Method A:** 54%, 34.7 mg, **Method B:**68%, 43.7 mg; white oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.53 – 7.46 (m, 2H), 7.11 (dd, *J* = 5.1, 3.7 Hz, 1H), 5.83 (td, *J* = 16.9, 6.7 Hz, 1H), 5.16 – 4.98 (m, 2H), 3.01 – 2.92 (m, 2H), 2.48 – 2.37 (m, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 163.3, 156.4, 146.5 (d, *J* = 11.3 Hz), 144.7 (d, *J* = 44.2 Hz), 142.8, 138.7 (d, *J* = 12.9 Hz), 137.1 (d, *J* = 28.2 Hz), 136.2, 136.1, 130.4, 130.2, 127.70, 116.4, 107.0 (d, *J* = 33.8 Hz), 31.4, 29.0; **HRMS** (ESI): *m/z* calcd for $C_{15}H_{15}NSSe$ $[M+H]^+$ 322.0169, found 322.0172.

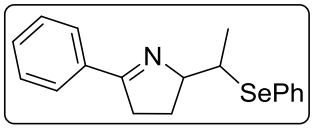
5-(Furan-2-yl)-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3l)



3l: (**Method A:** 44%, 26.8 mg, **Method B:**60%, 36.6 mg; yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.50 – 7.46 (m, 3H), 7.17 (dd, *J* = 9.5, 7.0 Hz, 3H), 6.74 (d, *J* = 3.4 Hz, 1H), 6.41 (dd, *J* = 3.5, 1.7 Hz, 1H), 4.45 – 4.37 (m, 1H), 3.47 (dd, *J* = 12.2, 4.3

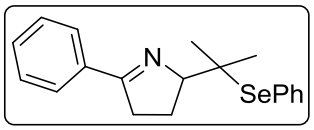
Hz, 1H), 2.94 (dd, *J* = 12.1, 8.7 Hz, 2H), 2.76 (dt, *J* = 19.0, 9.7 Hz, 1H), 2.25 – 2.17 (m, 1H), 1.67 (d, *J* = 7.2 Hz, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 164.1, 149.9, 144.9, 132.6, 130.5, 129.2, 126.9, 113.7, 111.7, 73.1, 35.3, 34.0, 28.5; **HRMS** (ESI): *m/z* calcd for $C_{15}H_{15}NOSe$ $[M+H]^+$ 306.0397, found 306.0399.

5-Phenyl-2-(1-(phenylselanyl)ethyl)-3,4-dihydro-2*H*-pyrrole (3m)



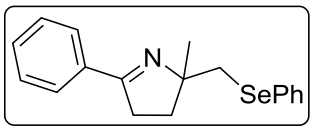
3m: (**Method A:** Reaction conditions: heating and stirring at 90 ℃ for 24 h, 53%, 34.8 mg, yellow oil, obtained in a diastereomer ratio greater than 20:1); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.83 – 7.77 (m, 2H), 7.60 – 7.56 (m, 2H), 7.38 (d, *J* = 7.7 Hz, 3H), 7.23 (dd, *J* = 4.1, 2.3 Hz, 3H), 4.50 – 4.40 (m, 1H), 3.85 – 3.74 (m, 1H), 3.11 – 2.83 (m, 2H), 2.22 – 2.09 (m, 1H), 1.95 (d, *J* = 10.3 Hz, 1H), 1.38 (d, *J* = 7.0 Hz, 3H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.6, 134.8, 134.5, 130.7, 129.9, 129.1, 128.5, 127.9, 127.4, 77.3, 44.4, 35.8, 25.2, 17.5; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NSe$ $[M+H]^+$ 330.0761, found 330.0765.

5-Phenyl-2-(2-(phenylselanyl)propan-2-yl)-3,4-dihydro-2*H*-pyrrole (3n)



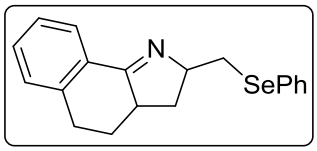
3n: (**Method A:** Reaction conditions: heating and stirring at 90 ℃ for 24 h, 70%, 48.0 mg, yellow oil; NMR data are in accordance with literature values[15]); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.69 (dd, *J* = 7.7, 1.9 Hz, 2H), 7.56 – 7.48 (m, 2H), 7.35 – 7.16 (m, 4H), 7.15 – 7.02 (m, 2H), 4.18 – 4.08 (m, 1H), 2.95 – 2.71 (m, 2H), 2.11 – 1.86 (m, 2H), 1.36 (s, 3H), 1.26 (s, 3H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.1, 138.6, 134.7, 130.5, 128.7 – 128.4 (m), 127.9 (d, *J* = 13.6 Hz), 82.5, 51.9, 35.6, 28.3, 26.4, 25.6; **HRMS** (ESI): *m/z* calcd for $C_{19}H_{21}NSe$ $[M+H]^+$ 344.0918, found 344.0921.

2-Methyl-5-phenyl-2-((phenylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3o)



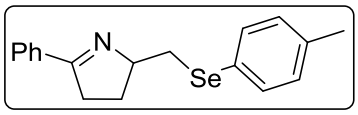
3o: (**Method A:** Reaction conditions: heating and stirring at 90 ℃ for 24 h, 53%, 34.9 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.85 – 7.77 (m, 2H), 7.55 (dd, *J* = 7.6, 2.0 Hz, 2H), 7.42 (dd, *J* = 9.3, 7.1 Hz, 3H), 7.23 (q, *J* = 5.1 Hz, 3H), 3.35 (d, *J* = 10.4 Hz, 2H), 3.09 (t, *J* = 8.0 Hz, 2H), 2.16 (dt, *J* = 12.9, 8.1 Hz, 1H), 1.97 – 1.84 (m, 1H), 1.49 (s, 3H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 171.4, 134.5, 132.4, 131.8, 130.5, 129.0, 128.4, 127.9, 126.6, 76.9, 41.6, 36.0, 34.1, 27.8; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NSe$ $[M+H]^+$ 330.0761, found 330.0767.

2-((Phenylselanyl)methyl)-3,3a,4,5-tetrahydro-2*H*-benzo(g)indole (3p)



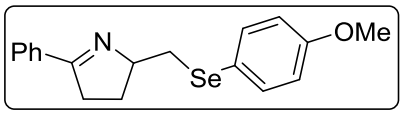
3p: (**Method A:** Reaction conditions: heating and stirring at 90 ℃ for 24 h, 51%, 34.8 mg, white oil, obtained in a 2.2:1 ratio of diastereomers, data are given for the mixture); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.08 (dd, *J* = 7.7, 1.4 Hz, 2H(major isomer)), 8.03 (dd, *J* = 7.9, 1.4 Hz, 1H(minor isomer)), 7.65 – 7.49 (m, 6H(major isomer)), 7.34 (td, *J* = 7.5, 1.6 Hz, 3H(minor isomer)), 7.30 – 7.03 (m, 8H(major isomer)+4H(minor isomer)), 4.64 (td, *J* = 8.6, 4.2 Hz, 1H(minor isomer)), 4.28 – 4.10 (m, 2H(major isomer)), 3.63 (dd, *J* = 11.9, 4.9 Hz, 2H(major isomer)), 3.29 (dd, *J* = 12.2, 4.5 Hz, 1H(minor isomer)), 3.19 – 2.76 (m, 8H(major isomer)+4H(minor isomer)), 2.54 (ddd, *J* = 12.3, 7.9, 6.2 Hz, 2H(minor isomer)), 2.33 – 2.19 (m, 4H(major isomer)), 1.79 (dt, *J* = 13.1, 9.2 Hz, 1H(minor isomer)), 1.69 (td, *J* = 12.9, 4.8 Hz, 2H(major isomer)), 1.65 – 1.53 (m, 1H(minor isomer)), 1.40 – 1.23 (m, 2H(major isomer)); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.6, 174.1, 141.2, 132.6, 132.6, 131.6, 131.0, 130.9, 130.7, 130.6, 130.0, 123.0, 129.3, 129.1, 129.1, 129.0, 128.9, 126.9, 126.8, 126.5, 126.5, 126.2, 126.1, 71.1, 71.0, 47.9, 46.0, 37.8, 34.9, 34.63, 32.7, 30.1, 30.1, 30.0, 29.7; **HRMS** (ESI): *m/z* calcd for $C_{19}H_{19}NSe$ $[M+H]^+$ 342.0761, found 342.0768.

5-Phenyl-2-((p-tolylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3r)



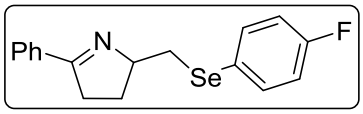
3r: (**Method A:** 57%, 37.5 mg**, Method B:** 66%, 43.2 mg; yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 6.7 Hz, 2H), 7.48 – 7.36 (m, 5H), 7.06 (d, *J* = 7.8 Hz, 2H), 4.53 – 4.45 (m, 1H), 3.44 (dd, *J* = 12.1, 4.6 Hz, 1H), 3.12 – 2.99 (m, 2H), 2.96 – 2.87 (m, 1H), 2.31 (s, 4H), 1.85 – 1.76 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 173.4, 136.9, 134.4, 133.2, 130.7, 130.0, 128.5, 127.9, 126.7, 73.0, 35.4, 34.8, 28.7, 21.2; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NSe$ $[M+H]^+$ 330.0761, found 330.0772.

2-(((4-Methoxyphenyl)selanyl)methyl)-5-phenyl-3,4-dihydro-2*H*-pyrrole (3s)



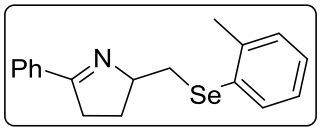
3s: (**Method A:** 14%, 9.7 mg**, Method B:** 55%, 38.2 mg; yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.88 – 7.76 (m, 2H), 7.52 (d, *J* = 8.8 Hz, 2H), 7.41 (t, *J* = 7.7 Hz, 4H), 6.79 (d, *J* = 8.7 Hz, 2H), 4.52 – 4.40 (m, 1H), 3.78 (s, 3H), 3.39 (dd, *J* = 12.1, 4.6 Hz, 1H), 3.14 – 3.02 (m, 1H), 3.02 – 2.84 (m, 2H), 2.33 – 2.21 (m, 1H), 1.85 – 1.74 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.3, 159.3, 135.6, 134.5, 130.6, 128.5, 127.9, 120.3, 114.8, 73.1, 55.4, 35.5, 35.1, 28.7; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NOSe$ $[M+H]^+$ 346.0710, found 346.0718.

2-(((4-Fluorophenyl)selanyl)methyl)-5-phenyl-3,4-dihydro-2*H*-pyrrole (3t)



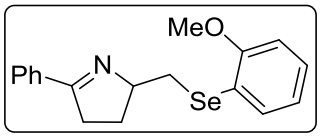
3t: (**Method A:** 18%, 10.0 mg**, Method B:** 58%, 38.9 mg; yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.84 – 7.73 (m, 2H), 7.54 (dd, *J* = 8.6, 5.5 Hz, 2H), 7.46 – 7.36 (m, 3H), 6.94 (t, *J* = 8.8 Hz, 2H), 4.55 – 4.43 (m, 1H), 3.40 (dd, *J* = 12.1, 4.8 Hz, 1H), 3.16 – 3.00 (m, 2H), 2.99 – 2.86 (m, 1H), 2.35 – 2.18 (m, 1H), 1.86 – 1.72 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 162.4 (d, *J* = 246.4 Hz), 135.4 (d, *J* = 7.7 Hz), 130.7, 128.5, 127.9, 125.0 (d, *J* = 3.5 Hz), 116.3 (d, *J* = 21.5 Hz), 73.0, 35.5 (d, *J* = 1.6 Hz), 28.7; **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -115.10; **HRMS** (ESI): *m/z* calcd for $C_{17}H_{16}FNSe$ $[M+H]^+$ 334.0510, found 334.0515.

5-Phenyl-2-((o-tolylselanyl)methyl)-3,4-dihydro-2*H*-pyrrole (3u)



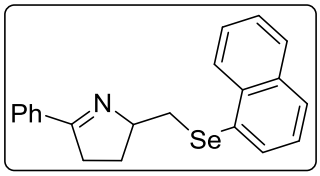
3u: (**Method A:** 58%, 38.2 mg, **Method B:** 52%, 34.1 mg; yellow solid, m.p. 54-55 ℃); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 7.4 Hz, 1H), 7.47 – 7.38 (m, 3H), 7.19 – 7.08 (m, 3H), 4.56 – 4.48 (m, 1H), 3.46 (dd, *J* = 11.9, 4.7 Hz, 1H), 3.09 (d, *J* = 11.7 Hz, 1H), 3.04 (d, *J* = 8.4 Hz, 1H), 2.97 – 2.88 (m, 1H), 2.43 (s, 3H), 2.37 – 2.26 (m, 1H), 1.89 – 1.80 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 173.4, 139.3, 134.4, 131.7, 131.7, 130.7, 130.0, 128.5, 127.9, 126.7 (d, *J* = 18.8 Hz), 72.8, 35.4, 33.3, 28.8, 22.5; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NSe$ $[M+H]^+$ 330.0761, found 330.0766.

2-(((2-Methoxyphenyl)selanyl)methyl)-5-phenyl-3,4-dihydro-2*H*-pyrrole (3v)



3v: (**Method A:** 54%, 37.3 mg**, Method B:** 43%, 29.5 mg; yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.0 Hz, 2H), 7.45 – 7.36 (m, 3H), 7.19 – 7.09 (m, 3H), 6.76 (d, *J* = 7.8 Hz, 1H), 4.57 – 4.49 (m, 1H), 3.77 (s, 3H), 3.47 (dd, *J* = 12.1, 4.8 Hz, 1H), 3.14 – 3.03 (m, 2H), 2.96 – 2.87 (m, 1H), 2.33 – 2.24 (m, 1H), 1.85 – 1.76 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 173.4, 159.8, 134.4, 131.7, 130.7, 129.8, 128.5, 127.9, 124.7, 117.9, 112.6, 72.9, 55.3, 35.5, 34.4, 28.7; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NOSe$ $[M+H]^+$ 346.0710, found 346.0716.

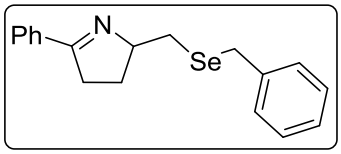
2-((Naphthalen-1-ylselanyl)methyl)-5-phenyl-3,4-dihydro-2*H*-pyrrole (3w)



3w: (**Method A:** 53%, 38.7 mg， **Method B:** 69%, 50.5 mg； yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.43 (d, *J* = 8.3 Hz, 1H), 7.90 – 7.76 (m, 5H), 7.63 – 7.46 (m, 2H), 7.44 – 7.33 (m, 4H), 4.55 – 4.42 (m, 1H), 3.51 (dd, *J* = 11.9, 4.7 Hz, 1H), 3.09 (dd, *J* = 11.9, 8.2 Hz, 2H), 2.97 – 2.84 (m, 1H), 2.35 – 2.22 (m, 1H), 1.91 – 1.78 (m, 1H); **^{13}C NMR** (101 MHz,

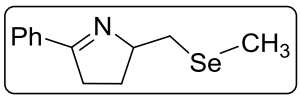
Chloroform-*d*) δ 173.5, 134.5, 134.4, 134.1, 132.6, 130.7, 129.9, 128.7, 128.5, 128.4, 127.9, 127.8, 126.7, 126.3, 125.9, 73.1, 35.5, 34.8, 28.8; **HRMS** (ESI): *m/z* calcd for $C_{21}H_{19}NSe$ $[M+H]^+$ 366.0761, found 366.0765.

2-((Benzylselanyl)methyl)-5-phenyl-3,4-dihydro-2*H*-pyrrole (3x)



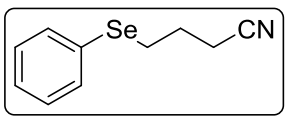
3x: (**Method A:** 58%, 38.2 mg**, Method B:** 73%, 48.0 mg; yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 7.8, 1.9 Hz, 2H), 7.42 (d, *J* = 2.1 Hz, 3H), 7.29 (dd, *J* = 9.2, 6.7 Hz, 4H), 7.22 – 7.16 (m, 1H), 4.57 – 4.46 (m, 1H), 3.87 (s, 2H), 3.12 – 3.01 (m, 1H), 2.98 – 2.88 (m, 2H), 2.83 (dd, *J* = 12.3, 6.9 Hz, 1H), 2.29 – 2.16 (m, 1H), 1.77 – 1.68 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.2, 139.8, 134.5, 130.7, 129.2, 129.1, 128.6, 127.9, 126.8, 73.5, 35.5, 30.7, 28.8, 28.1; **HRMS** (ESI): *m/z* calcd for $C_{18}H_{19}NSe$ $[M+H]^+$ 330.0761, found 330.0765.

2-((Methylselanyl)methyl)-5-phenyl-3,4-dihydro-2*H*-pyrrole (3y)



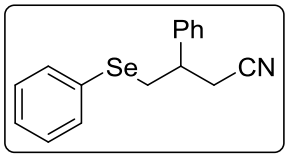
3y: (**Method A:** 47%, 23.8 mg**, Method B:** 54%, 27.1 mg; light yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 6.5 Hz, 2H), 7.49 – 7.37 (m, 3H), 4.57 – 4.49 (m, 1H), 3.13 – 3.05 (m, 1H), 3.02 (dd, *J* = 12.4, 4.8 Hz, 1H), 2.97 – 2.89 (m, 1H), 2.80 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.33 – 2.22 (m, 1H), 2.06 (s, 3H), 1.84 – 1.75 (m, 1H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 173.3, 134.5, 130.7, 128.6, 127.9, 73.4, 35.5, 32.3, 28.7, 5.4; **HRMS** (ESI): *m/z* calcd for $C_{12}H_{15}NSe$ $[M+H]^+$ 254.0448, found 254.0450.

4-(Phenylselanyl)butanenitrile (5a)



5a: (**Method A:** 86%, 38.7 mg; **Method B:**98%, 44.1 mg; light yellow oil; NMR data are in accordance with literature values[16]); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.55 – 7.46 (m, 2H), 7.34 – 7.24 (m, 3H), 2.99 (t, *J* = 7.0 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 2.07 – 1.93 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 133.4, 129.4, 128.8, 127.6, 119.2, 26.2, 25.8, 17.1.

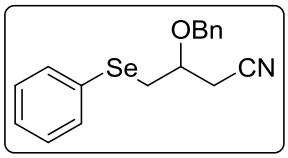
3-Phenyl-4-(phenylselanyl)butanenitrile (5b)



5b: (**Method A:** 76%, 45.8 mg, yellow oil; NMR data are in accordance with literature values[16]); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.51 – 7.42 (m, 2H), 7.39 – 7.21 (m, 6H), 7.18 (d, *J* = 7.9 Hz, 2H), 3.29 (dd, *J* = 12.3, 6.2 Hz, 1H), 3.26 – 3.07 (m, 2H), 2.86 (dd, *J* = 16.8, 5.3 Hz, 1H), 2.75 (dd, *J* = 16.8, 7.5 Hz, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 140.7, 133.2, 129.5, 129.1,

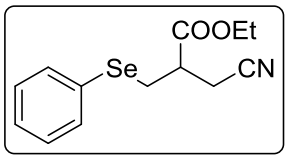
129.1, 128.0, 127.6, 127.2, 118.2, 42.3, 32.7, 24.2.

3-(Benzyloxy)-4-(phenylselanyl)butanenitrile (5c)



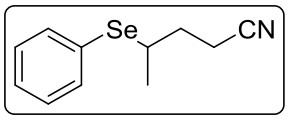
5c: (**Method A:**60%, 38.0 mg, yellow oil; NMR data are in accordance with literature values[16]); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.51 – 7.44 (m, 2H), 7.31 (dd, *J* = 16.7, 8.5 Hz, 8H), 4.57 (d, *J* = 3.4 Hz, 2H), 3.81 (p, *J* = 5.5 Hz, 1H), 3.21 (dd, *J* = 13.2, 4.9 Hz, 1H), 3.03 (dd, *J* = 13.2, 7.6 Hz, 1H), 2.79 – 2.64 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 137.1, 133.2, 129.5, 129.1, 128.7, 128.2, 128.1, 127.7, 117.3, 74.3, 72.2, 30.9, 23.1.

Ethyl 3-cyano-2-((phenylselanyl)methyl)propanoate (5d)



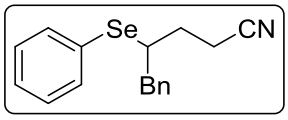
5d: (**Method A:** 50%, 29.7 mg, yellow oil; NMR data are in accordance with literature values[16]); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.57 – 7.51 (m, 2H), 7.33 – 7.26 (m, 3H), 4.11 (ddq, *J* = 41.3, 10.7, 7.1 Hz, 2H), 3.33 (dd, *J* = 13.1, 5.6 Hz, 1H), 3.13 (dd, *J* = 13.2, 7.4 Hz, 1H), 2.98 – 2.91 (m, 1H), 2.80 (dd, *J* = 6.5, 0.9 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 171.0, 133.8, 129.6, 128.4, 128.1, 117.5, 62.0, 42.2, 28.2, 19.4, 14.2.

4-(Phenylselanyl)pentanenitrile (5e)



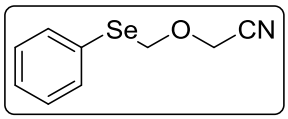
5e: (**Method A:** 74%, 35.4 mg, yellow oil);**^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.50 – 7.43 (m, 2H), 7.21 (p, *J* = 7.6, 7.2 Hz, 3H), 3.21 (h, *J* = 6.9 Hz, 1H), 2.45 (td, *J* = 7.3, 5.1 Hz, 2H), 1.82 (t, *J* = 7.2 Hz, 2H), 1.36 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 135.6, 129.2, 128.2, 127.6, 119.4, 37.9, 32.96, 22.0, 15.9; **HRMS** (ESI): *m/z* calcd for $C_{10}H_{11}NSe$ $[M+H]^+$ 240.0291, found 240.0287.

5-Phenyl-4-(phenylselanyl)pentanenitrile (5f)



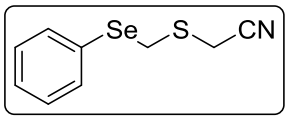
5f: (**Method A:**83%, 52.3 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.61 – 7.53 (m, 2H), 7.40 – 7.26 (m, 6H), 7.19 (d, *J* = 6.8 Hz, 2H), 3.48 – 3.37 (m, 1H), 3.18 (dd, *J* = 14.1, 6.3 Hz, 1H), 2.89 (dd, *J* = 14.1, 8.8 Hz, 1H), 2.67 – 2.54 (m, 2H), 2.11 – 1.95 (m, 1H), 1.86 – 1.72 (m, 1H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 138.8, 135.6, 129.4, 129.1, 128.7, 128.3, 127.7, 126.9, 119.3, 45.5, 42.5, 30.0, 16.0; **HRMS** (ESI): *m/z* calcd for $C_{17}H_{17}NSe$ $[M+H]^+$ 316.0604, found 316.0608.

2-((Phenylselanyl)methoxy)acetonitrile (5g)



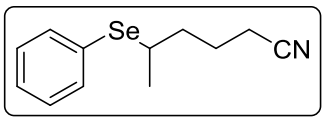
5g: (**Method A:**78%, 35.4 mg, yellow oil; NMR data are in accordance with literature values[16]); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.61 – 7.53 (m, 2H), 7.36 – 7.27 (m, 3H), 5.35 (s, 2H), 4.45 (s, 2H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 133.1, 129.5, 129.1, 128.0, 115.3, 71.8, 53.3.

2-(((Phenylselanyl)methyl)thio)acetonitrile (5h)



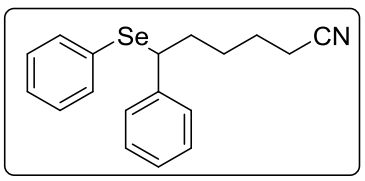
5h: (**Method A:** 62%, 30.1 mg, yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.62 – 7.55 (m, 2H), 7.39 – 7.27 (m, 3H), 4.13 (s, 2H), 3.49 (s, 2H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 134.1, 129.5, 128.5, 128.4, 116.7, 29.9, 17.7; **HRMS** (ESI): *m/z* calcd for C_9H_9NSSe $[M+H]^+$ 243.9699, found 243.9703.

5-(Phenylselanyl)hexanenitrile (5i)



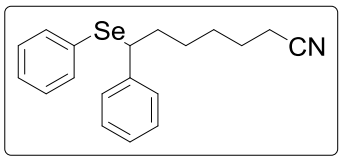
5i: (**Method A:**, 79%, 38.5 mg; light yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.59 – 7.52 (m, 2H), 7.30 (t, *J* = 5.3 Hz, 3H), 3.25 (h, *J* = 6.7 Hz, 1H), 2.33 (t, *J* = 6.9 Hz, 2H), 1.83 (p, *J* = 6.5, 6.0 Hz, 2H), 1.78 – 1.69 (m, 2H), 1.43 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 135.3, 129.1, 128.5, 127.8, 119.5, 38.6, 36.4, 23.8, 22.3, 17.0; **HRMS** (ESI): *m/z* calcd for $C_{12}H_{15}NSSe$ $[M+H]^+$ 254.0448, found 254.0446.

6-Phenyl-6-(phenylselanyl)hexanenitrile (5k)



5k: (**Method A:**, 77%, 50.7 mg; yellow oil); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 – 7.35 (m, 2H), 7.27 (d, *J* = 3.2 Hz, 1H), 7.26 – 7.22 (m, 3H), 7.20 (dt, *J* = 5.4, 2.4 Hz, 2H), 7.16 (dd, *J* = 5.3, 3.1 Hz, 2H), 4.23 – 4.17 (m, 1H), 2.29 – 2.21 (m, 2H), 2.14 – 1.97 (m, 2H), 1.69 – 1.56 (m, 2H), 1.53 – 1.36 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.8, 135.5, 129.3, 128.8, 128.4, 127.9, 127.6, 127.1, 119.4, 47.9, 35.2, 27.5, 25.0, 17.0; **HRMS** (ESI): m/z calcd for $C_{18}H_{19}NSe$ $[M+Na]^+$ 352.0576, found 352.0587.

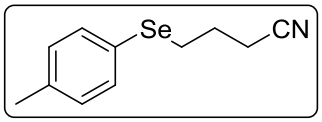
7-phenyl-7-(phenylselanyl)heptanenitrile (5l)



5l: (**Method A:**, 78%, 53.2 mg; yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.43 – 7.37 (m, 2H), 7.30 – 7.16 (m, 8H), 4.23 (dd, *J* = 9.0, 6.4 Hz, 1H), 2.27 (t, *J* =

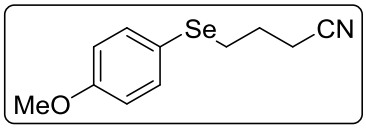
7.1 Hz, 2H), 2.13 – 1.99 (m, 2H), 1.62 – 1.55 (m, 2H), 1.43 (dd, *J* = 14.1, 6.4 Hz, 2H), 1.37 – 1.27 (m, 2H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 142.26, 135.56, 129.54, 128.90, 128.45, 127.92, 127.76, 127.06, 119.74, 48.37, 35.77, 28.27, 27.59, 25.21, 17.11; **HRMS** (ESI): *m/z* calcd for $C_{11}H_{13}NSe$ $[M+Na]^+$ 366.0736, found 366.0733.

4-(P-tolylselanyl)butanenitrile (5m)



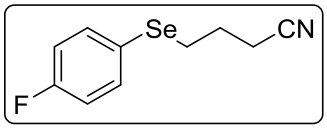
5m: (**Method A:** 30%, 14.3 mg**, Method B:** 84%, 40.2 mg; yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.41 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 7.8 Hz, 2H), 2.94 (t, *J* = 7.0 Hz, 2H), 2.49 (t, *J* = 7.1 Hz, 2H), 2.33 (s, 3H), 1.97 (p, *J* = 7.0 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 137.9, 133.9, 130.3, 124.9, 119.2, 26.5, 25.8, 21.3, 17.1; **HRMS** (ESI): *m/z* calcd for $C_{11}H_{13}NSe$ $[M+H]^+$ 240.0291, found 240.0292.

4-((4-Methoxyphenyl)selanyl)butanenitrile (5n)



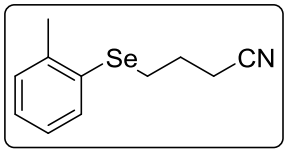
5n: (**Method A:** 94%, 47.9 mg**, Method B:** 54%, 27.6 mg; yellow oil; NMR data are in accordance with literature values[17]); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.51 – 7.43 (m, 2H), 6.85 – 6.78 (m, 2H), 3.79 (s, 3H), 2.88 (t, *J* = 7.0 Hz, 2H), 2.48 (t, *J* = 7.1 Hz, 2H), 1.93 (p, *J* = 7.1 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 159.8, 136.2, 119.2, 118.5, 115.1, 55.4, 27.1, 25.7, 17.0.

4-((4-Fluorophenyl)selanyl)butanenitrile (5o)



5o: (**Method A:**45%, 21.9 mg**, Method B:** 54%, 26.0 mg; yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.56 – 7.46 (m, 2H), 7.04 – 6.94 (m, 2H), 2.94 (t, *J* = 7.0 Hz, 2H), 2.50 (t, *J* = 7.0 Hz, 2H), 1.97 (p, *J* = 7.0 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 162.8 (d, *J* = 247.9 Hz), 136.1 (d, *J* = 7.9 Hz), 123.2 (d, *J* = 3.3 Hz), 119.1, 116.7 (d, *J* = 21.5 Hz), 27.0, 25.8, 17.1; **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -113.70; **HRMS** (ESI): *m/z* calcd for $C_{10}H_{10}FNSe$ $[M+H]^+$ 244.0040, found 244.0042.

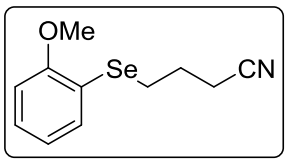
4-(O-tolylselanyl)butanenitrile (5p)



5p: (**Method A:** 64%, 30.6 mg**, Method B:** 54%, 25.8 mg; yellow oil; NMR data are in accordance with literature values[17]); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.43 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.24 – 7.16 (m, 2H), 7.12 (t, *J* = 7.5 Hz, 1H), 2.97 (t, *J* = 7.0 Hz, 2H), 2.50 (t, *J* = 7.0 Hz, 2H), 2.43 (s, 3H), 2.00 (p, *J* = 7.0 Hz,

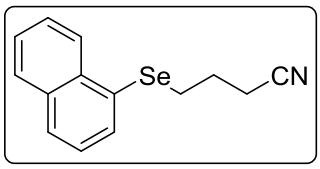
2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 140.0, 132.3, 130.4, 129.9, 127.5, 126.8, 119.1, 25.7, 24.9, 22.5, 17.3.

4-((2-Methoxyphenyl)selanyl)butanenitrile (5q)



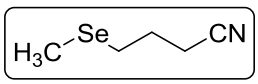
5q: (**Method A:** 20%, 10.2 mg**, Method B:** 34%, 17.3 mg; yellow oil); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.22 – 7.15 (m, 1H), 7.09 – 7.02 (m, 2H), 6.81 (ddd, *J* = 8.2, 2.6, 0.9 Hz, 1H), 3.80 (s, 3H), 2.99 (t, *J* = 7.0 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 2.05 – 1.93 (m, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 160.0, 130.2, 129.8, 125.2, 119.2, 118.6, 113.2, 55.4, 26.0, 25.8, 17.1; **HRMS** (ESI): *m/z* calcd for $C_{11}H_{13}NOSe$ $[M+H]^+$ 256.0240, found 256.0244.

4-(Naphthalen-1-ylselanyl)butanenitrile (5r)



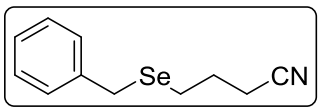
5r: (**Method A:** 55%, 30.3 mg**, Method B:** 74%, 40.6 mg; yellow oil); **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.40 (d, *J* = 8.3 Hz, 1H), 7.89 – 7.76 (m, 3H), 7.61 – 7.49 (m, 2H), 7.39 (t, *J* = 7.7 Hz, 1H), 3.02 (t, *J* = 7.0 Hz, 2H), 2.48 (t, *J* = 7.1 Hz, 2H), 1.92 (q, *J* = 7.0 Hz, 2H); **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 134.5, 134.2, 133.5, 129.2, 128.9, 128.0, 127.6, 127.1, 126.5, 125.9, 119.1, 26.3, 25.8, 17.2; **HRMS** (ESI): *m/z* calcd for $C_{14}H_{13}NSe$ $[M+Na]^+$ 298.0110, found 298.0110.

4-(Methylselanyl)butanenitrile (5s)



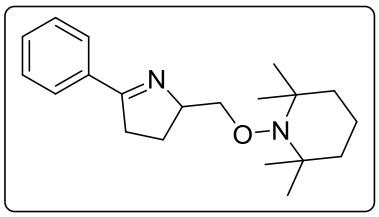
5s: (**Method A:** 41%, 13.4 mg**, Method B:** 56%, 18.1 mg; light yellow oil; NMR data are in accordance with literature values[18]); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 2.66 (t, *J* = 7.0 Hz, 2H), 2.52 (t, *J* = 7.0 Hz, 2H), 2.07 – 1.96 (m, 5H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 119.3, 25.7, 23.6, 17.2, 4.4.

4-(Benzylselanyl)butanenitrile (5t)



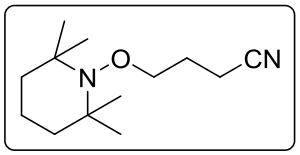
5t: (**Method A:** 58%, 27.7 mg**, Method B:** 64%, 30.4 mg; yellow oil; NMR data are in accordance with literature values[19]); **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.28 (dt, *J* = 10.6, 7.2 Hz, 4H), 7.21 (t, *J* = 6.9 Hz, 1H), 3.77 (s, 2H), 2.57 (t, *J* = 7.0 Hz, 2H), 2.39 (d, *J* = 14.1 Hz, 2H), 1.87 (p, *J* = 7.1 Hz, 2H); **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 138.9, 128.9, 128.8, 127.1, 119.2, 27.4, 25.9, 22.1, 17.2.

2,2,6,6-Tetramethyl-1-((5-phenyl-3,4-dihydro-2*H*-pyrrol-2-yl)methoxy)piperidine (6)



6: (71%, 44.6 mg, yellow oil; NMR data are in accordance with literature values[3]**); ^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.88 – 7.81 (m, 2H), 7.46 – 7.33 (m, 3H), 4.44 (d, *J* = 13.9 Hz, 1H), 4.08 (d, *J* = 12.9 Hz, 1H), 3.96 (d, *J* = 13.9 Hz, 1H), 3.12 – 2.84 (m, 2H), 2.18 – 1.99 (m, 2H), 1.59 – 1.32 (m, 6H), 1.21 (s, 3H), 1.15 (s, 3H), 1.10 (s, 3H), 0.94 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 134.9, 130.4, 128.5, 127.8, 79.2, 72.5, 60.0, 39.7, 35.5, 33.3, 33.1, 26.0, 20.4, 20.1, 17.2.

4-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)butanenitrile (7)



7: (61%; yield was determined by ^{1}H NMR analysis of the crude reaction mixture using 1,3,5-trimethoxybenzene as an internal standard) **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 3.82 (t, *J* = 5.8 Hz, 2H), 2.47 (t, *J* = 7.2 Hz, 2H), 1.87 (p, *J* = 6.6 Hz, 2H), 1.42 (d, *J* = 4.8 Hz, 4H), 1.36 (s, 2H), 1.10 (d, *J* = 23.8 Hz, 12H); **HRMS** (ESI): *m/z* calcd for $C_{14}H_{13}NSe$ $[M+H]^+$ 225.1966, found 225.1958.

5. References

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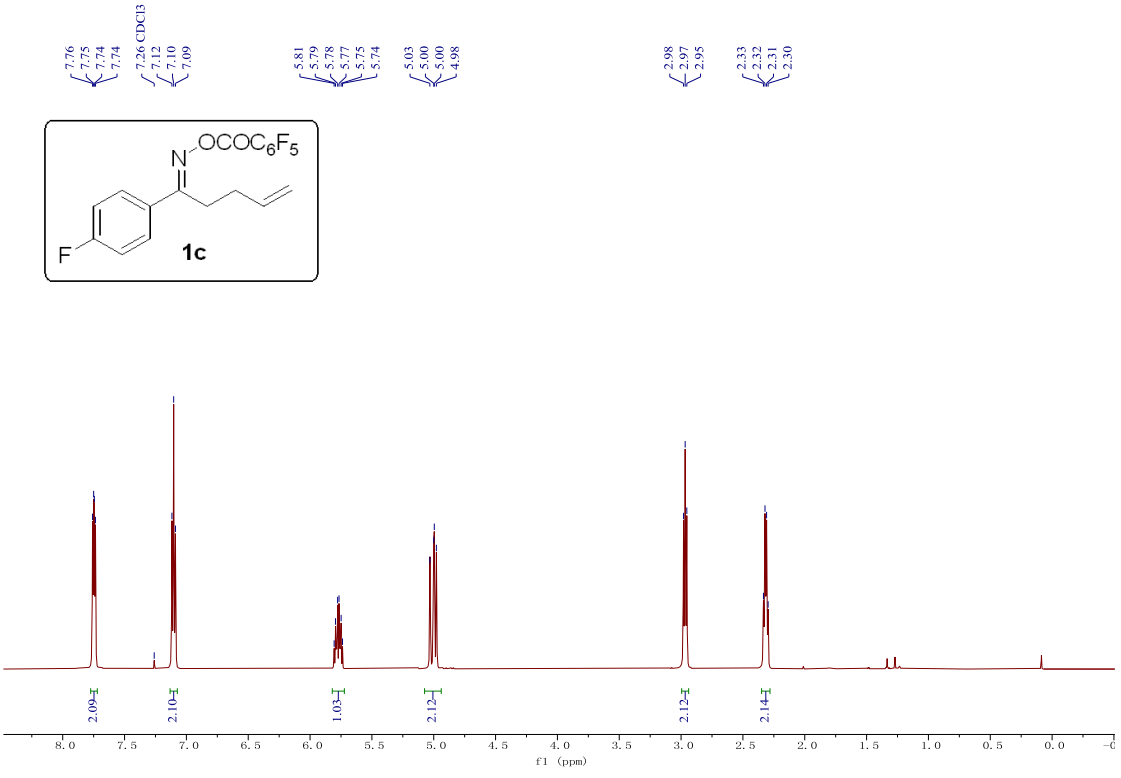
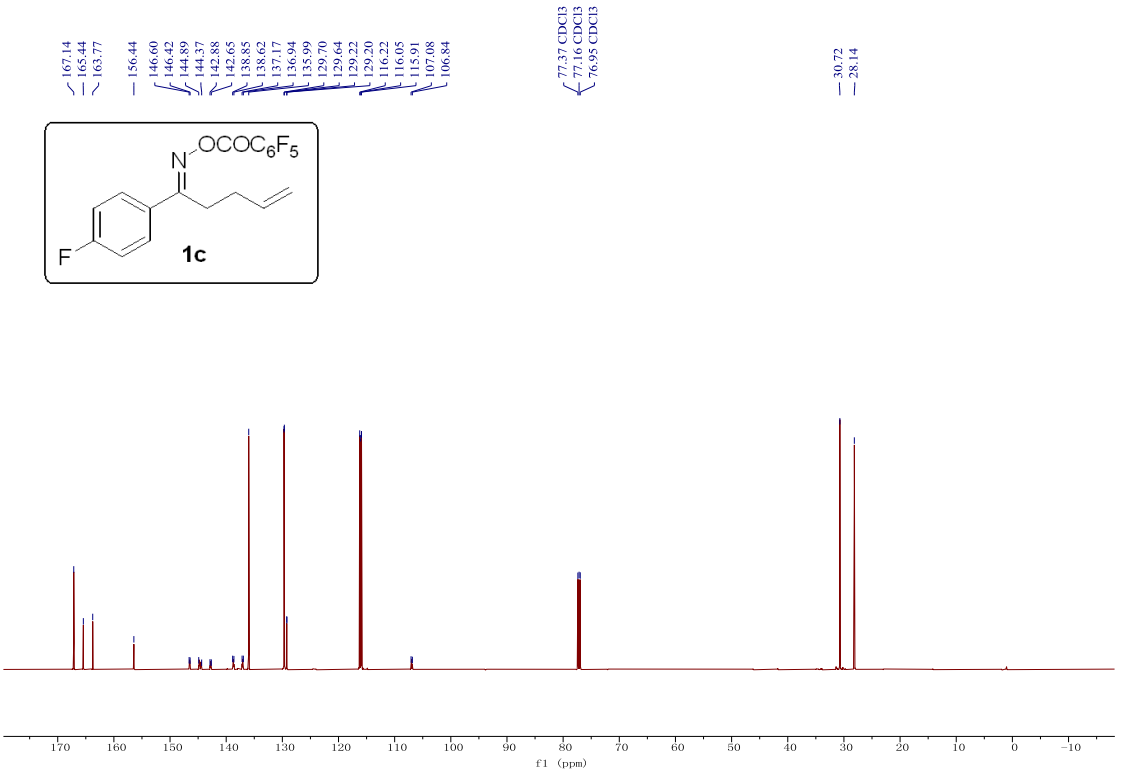
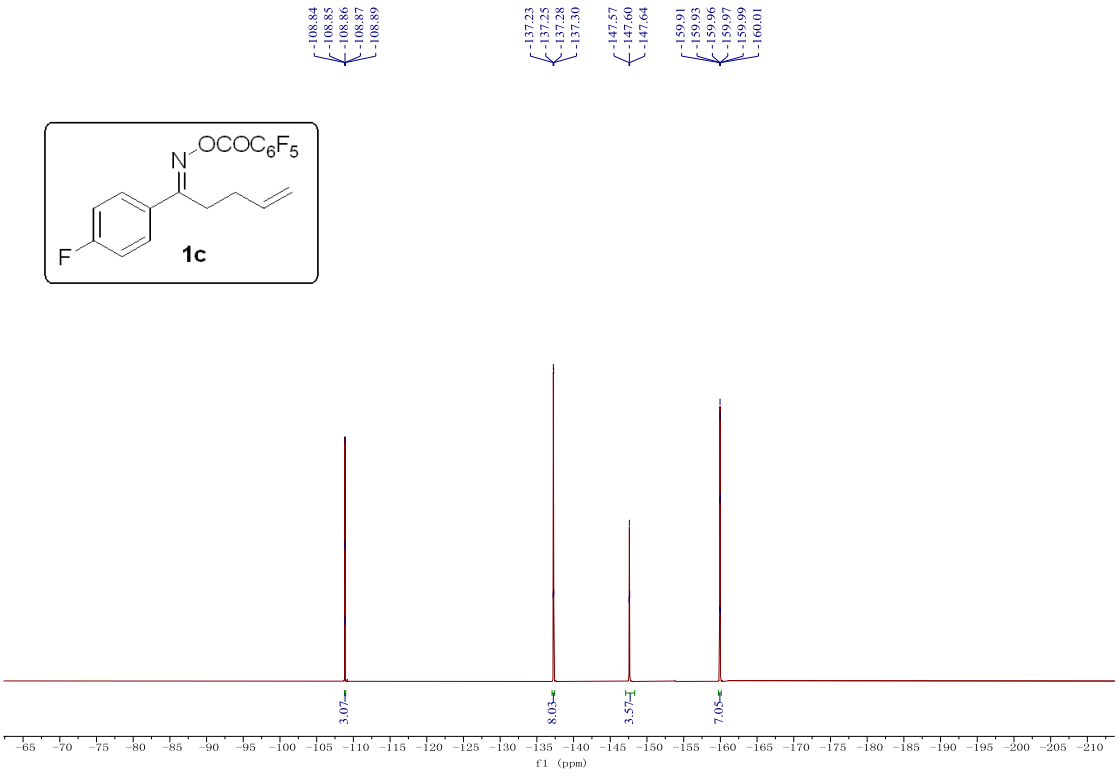
6. ^{1}H, ^{13}C, and ^{19}F NMR Spectra



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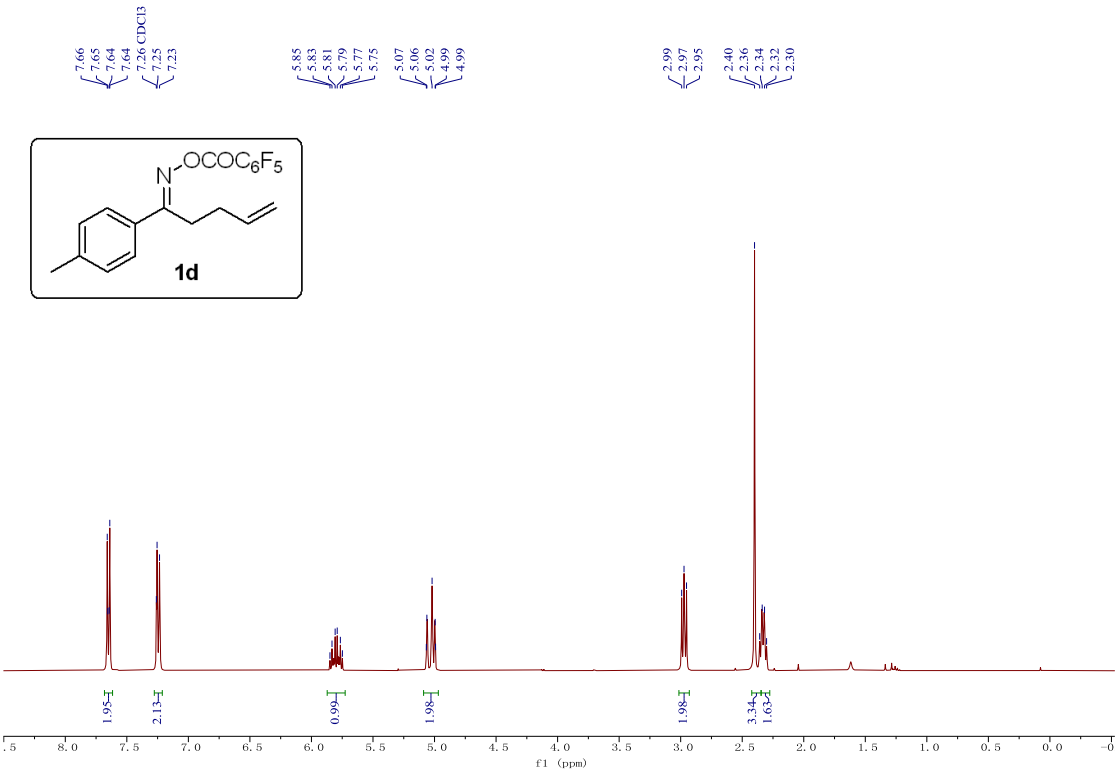
7.05
f1 (ppm)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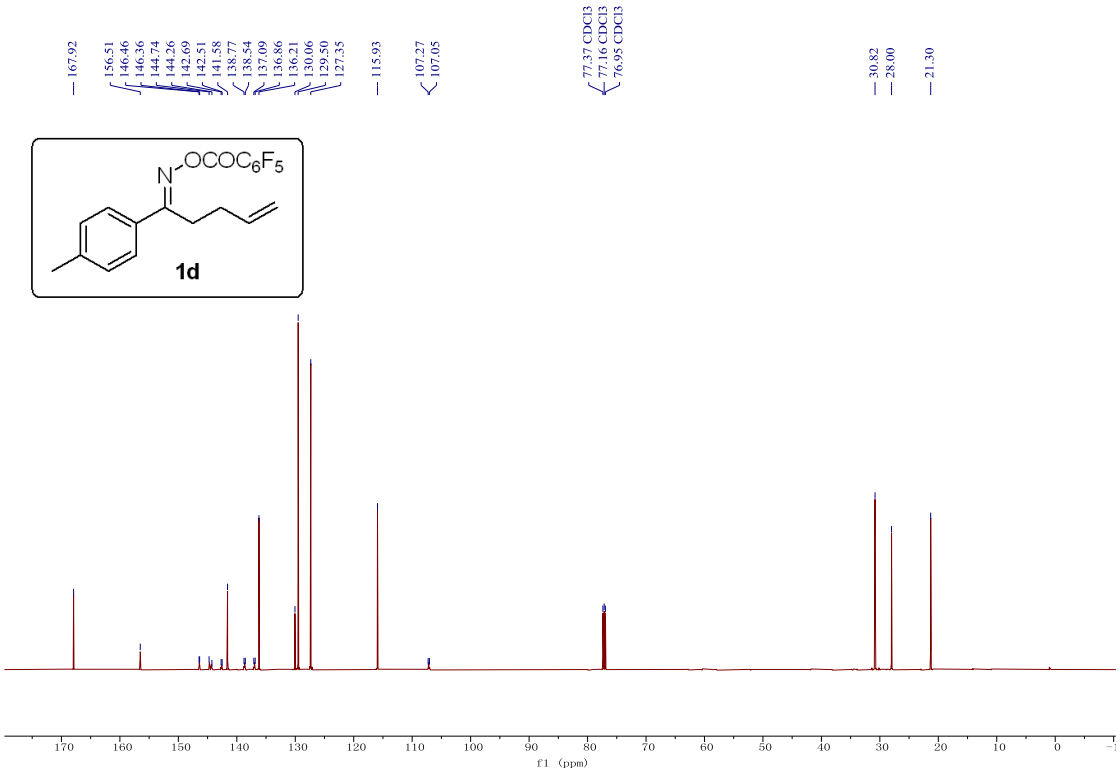
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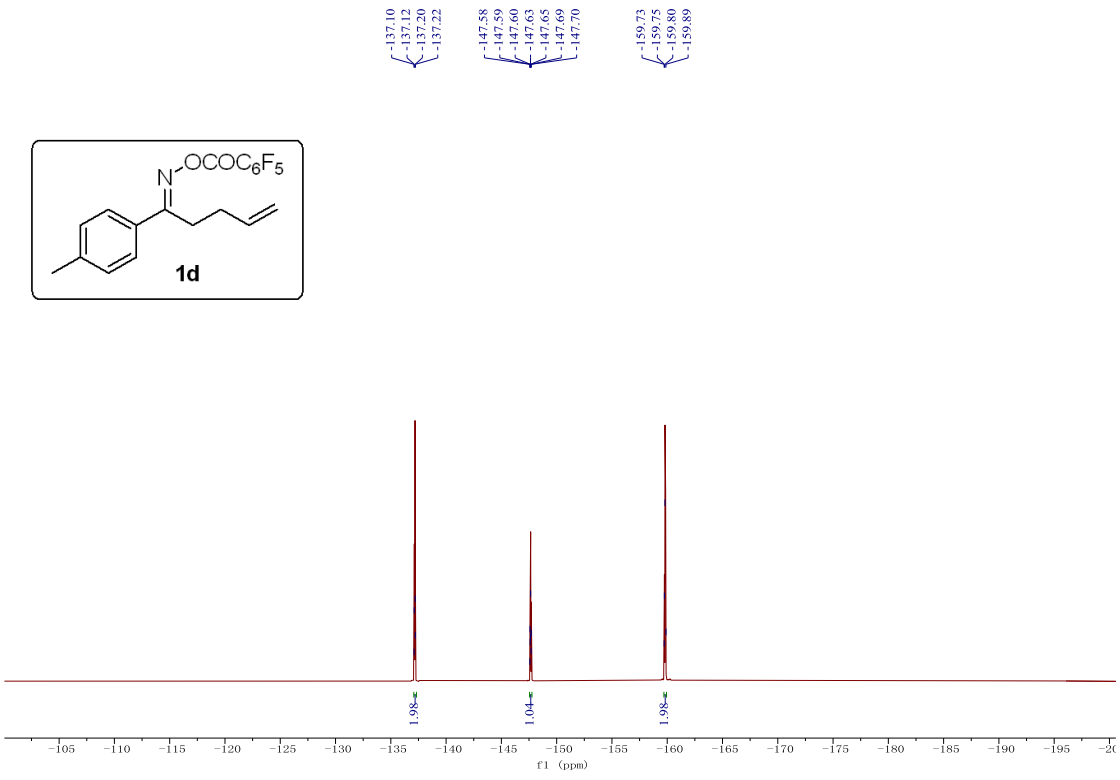
f1 (ppm)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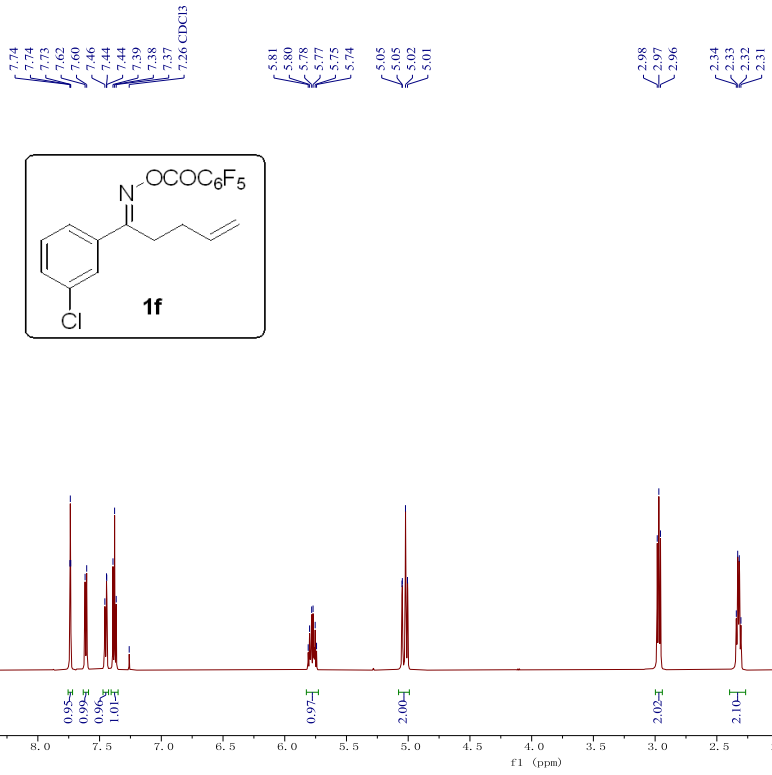
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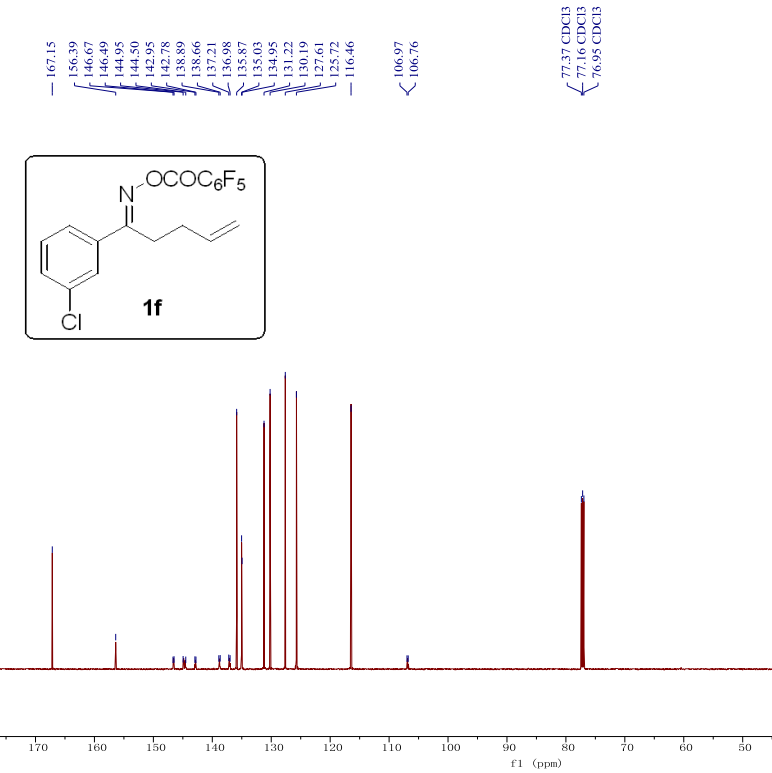
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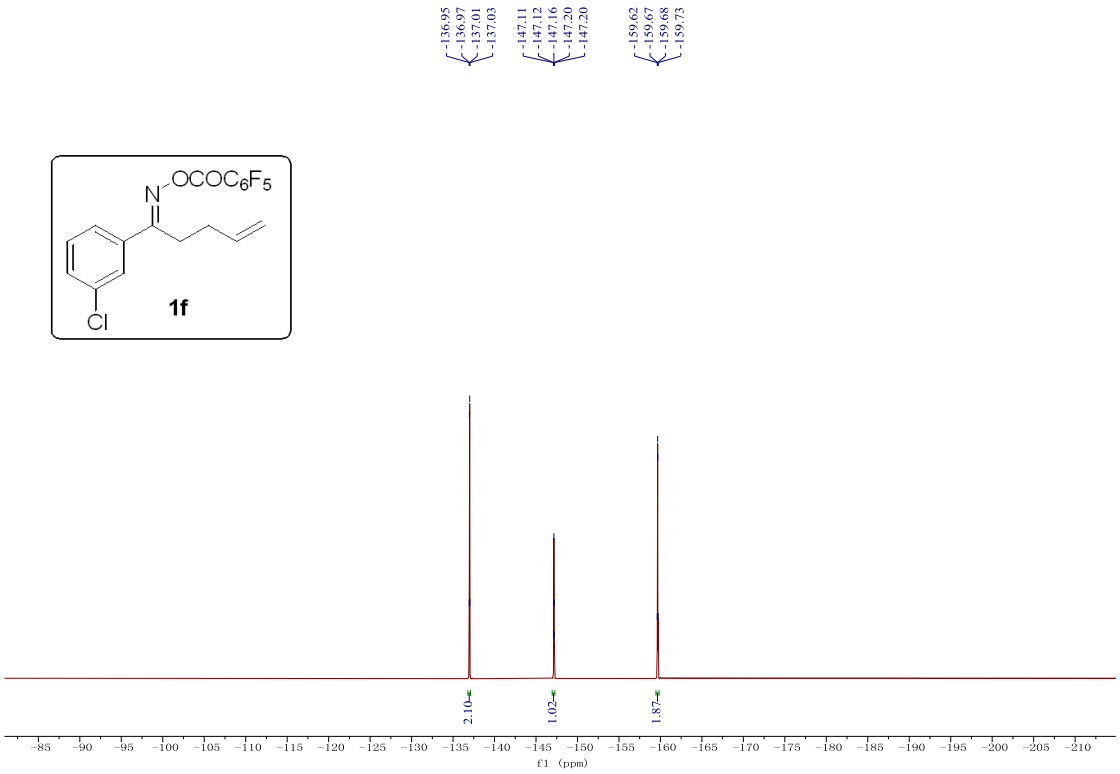
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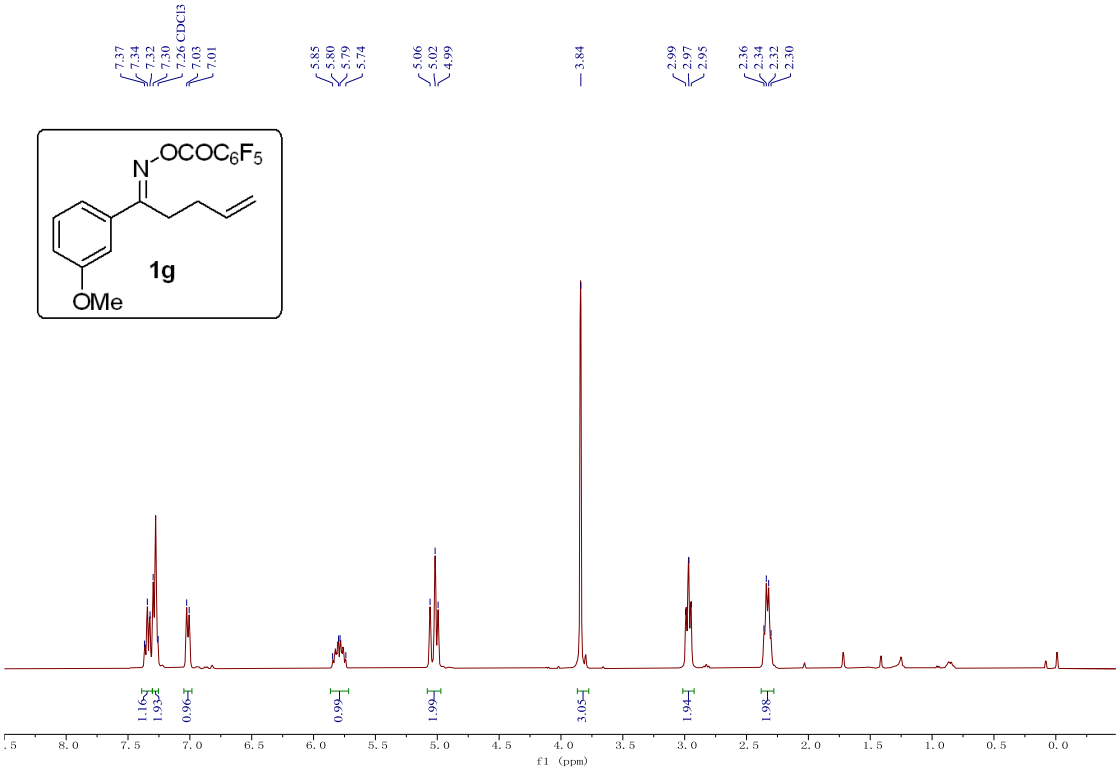
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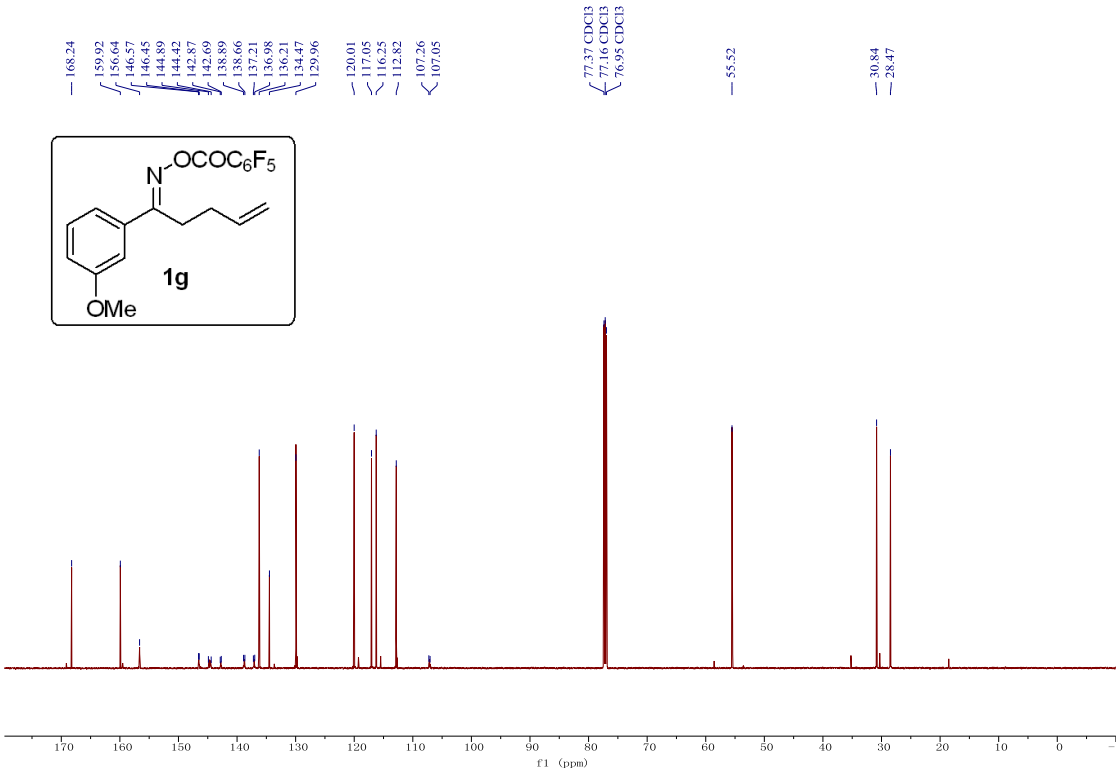
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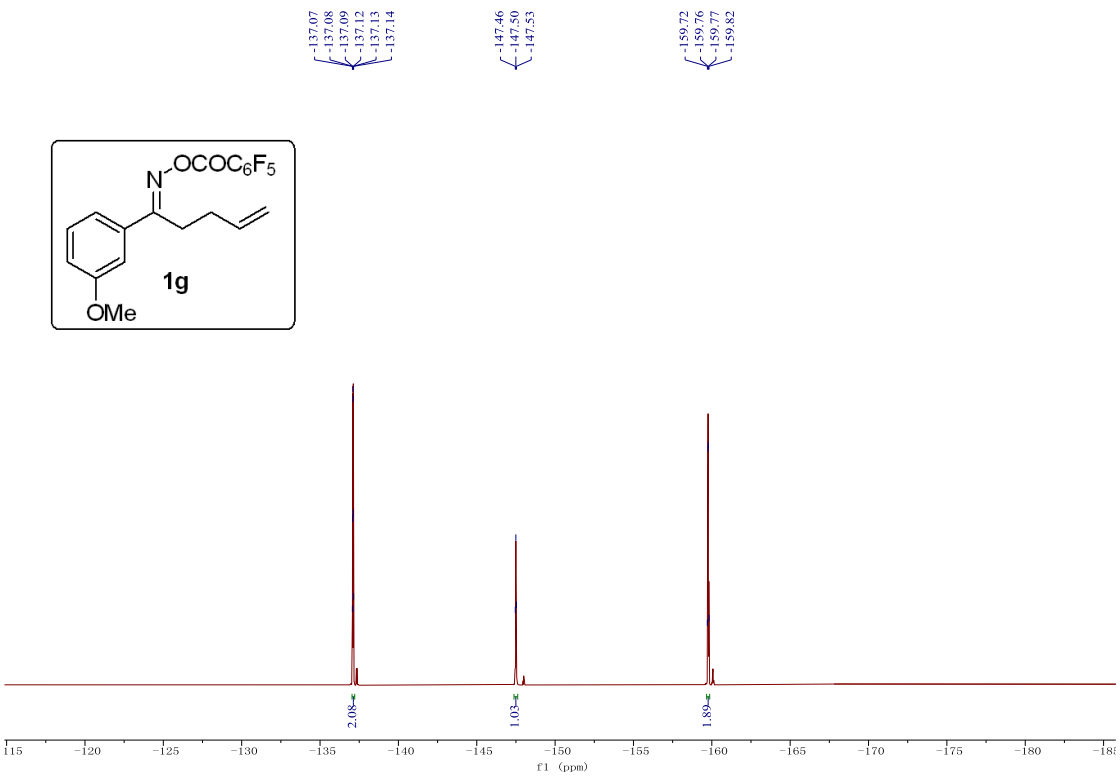
136.21
134.47
129.96
120.01
117.05
116.25
112.82
107.26
107.05
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
55.52
30.84
28.47
OCOC6F5
N
1g
OMe
f1 (ppm)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137.08
137.09
137.12
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137.14
147.46
147.50
147.53
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159.77
159.82
OCOC6F5
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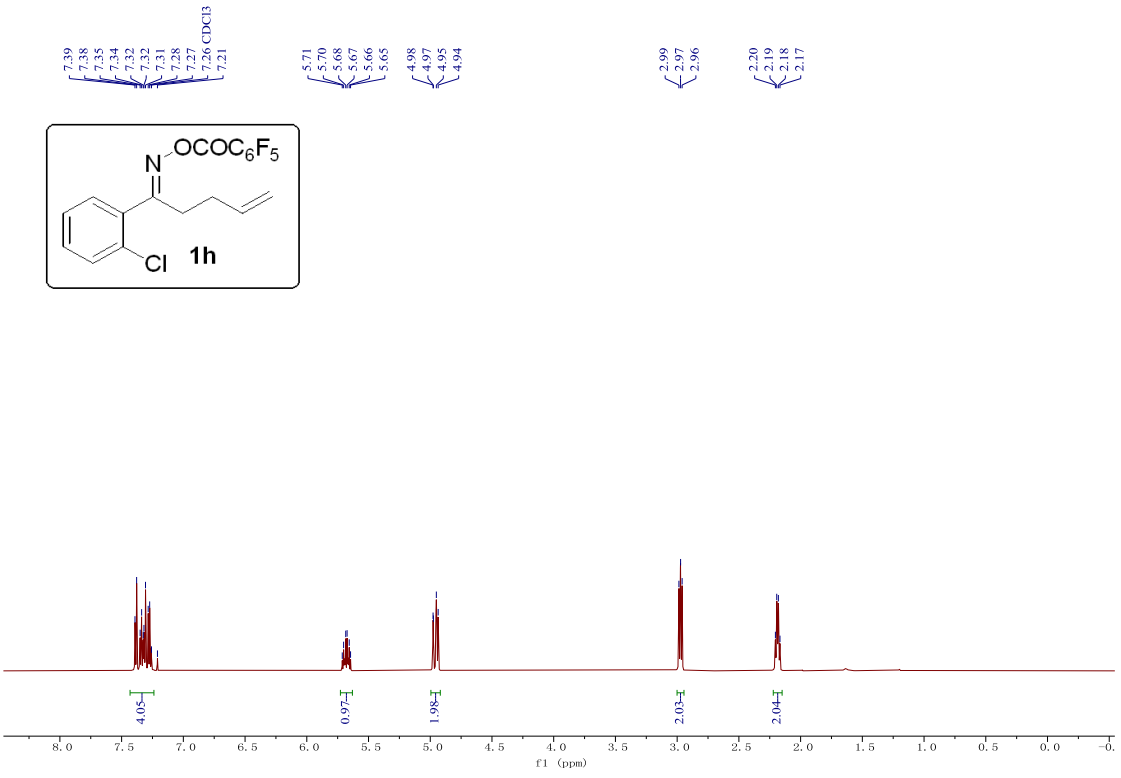
1g
OMe
2.08
1.03
1.89
f1 (ppm)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7.38
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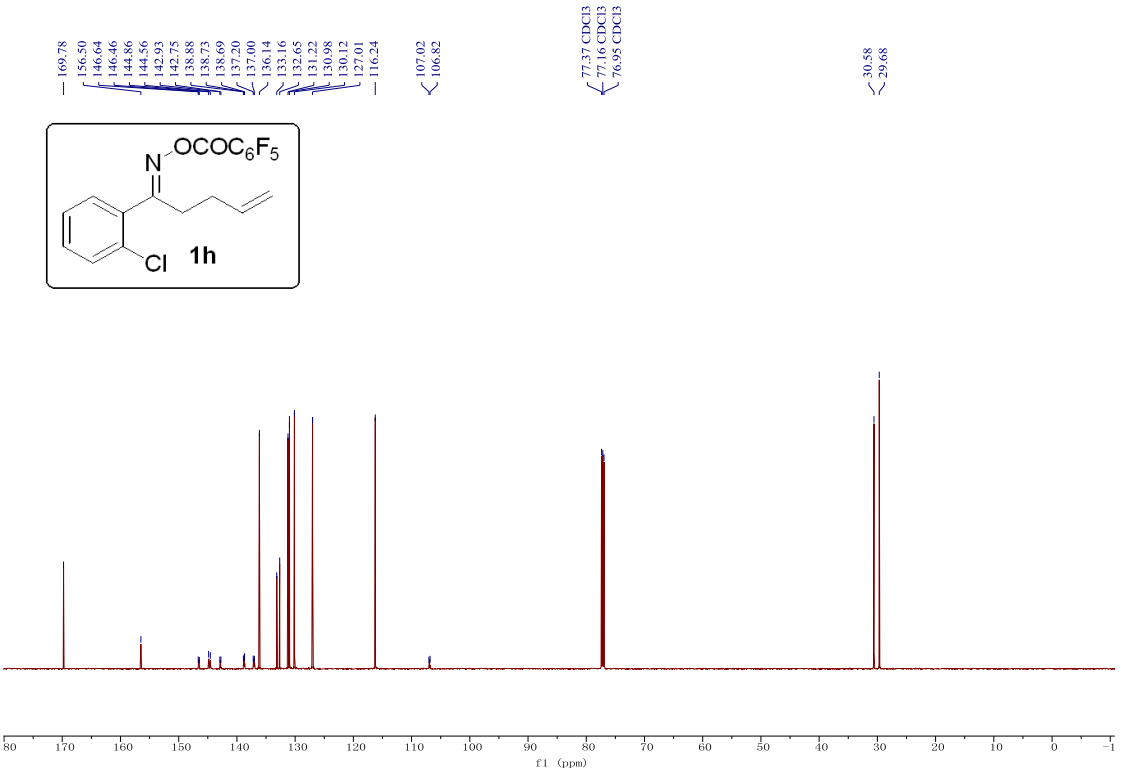
7.32
7.31
7.28
7.27
7.26 CDCl3
7.21
5.71
5.70
5.68
5.67
5.66
5.65
4.98
4.97
4.95
4.94
2.99
2.97
2.96
2.20
2.19
2.18
2.17
OCOC6F5
N
Cl
1h
4.05
0.97
1.98
2.03
2.04
f1 (ppm)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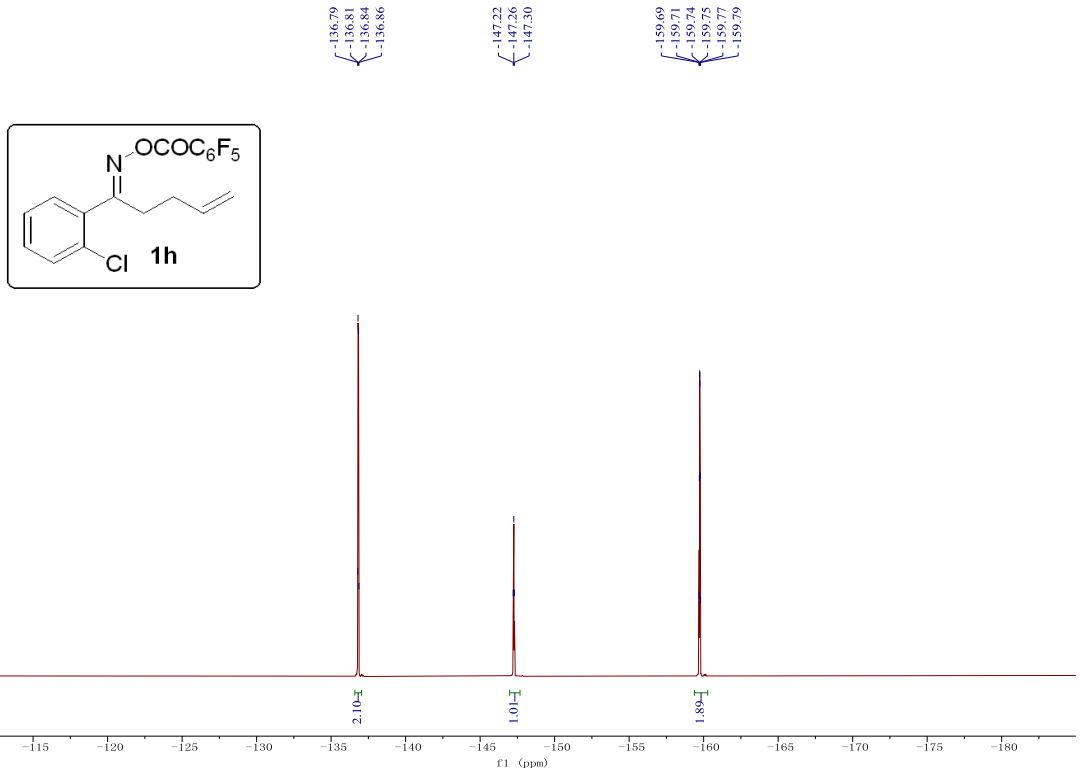
137.00
136.14
133.16
132.65
131.22
130.98
130.12
127.01
116.24
107.02
106.82
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
30.58
29.68
OCOC6F5
N
Cl
1h
f1 (ppm)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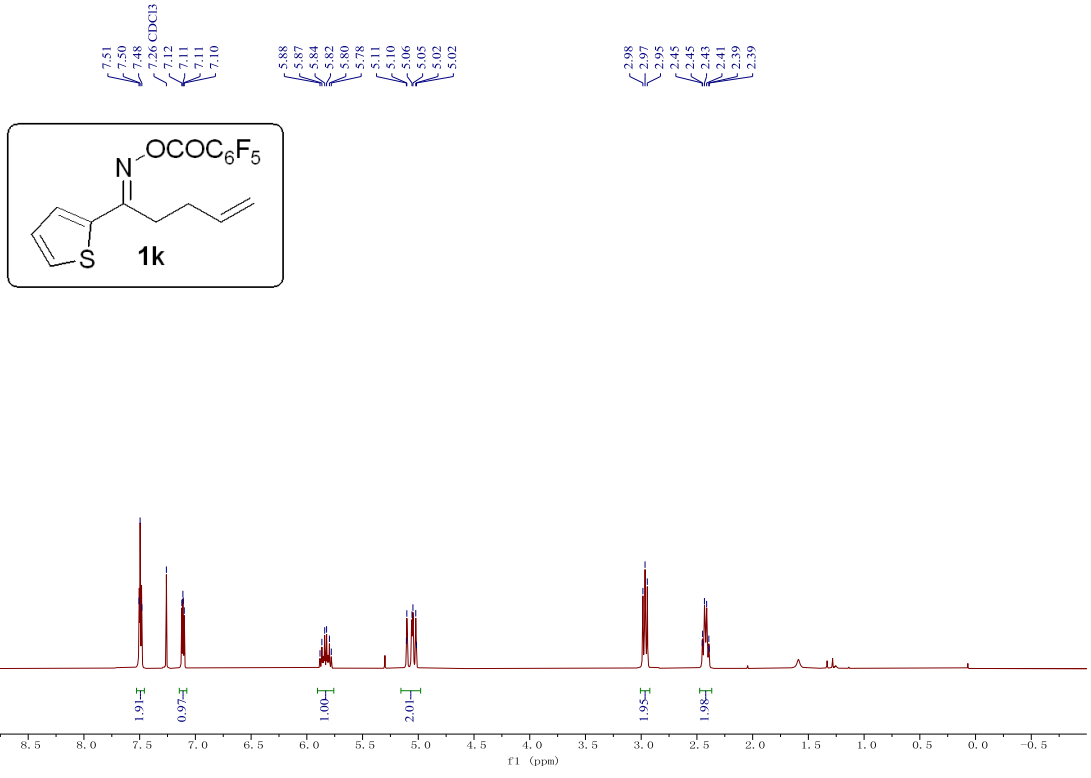
-159.75
-159.77
-159.79
N
OCOC6F5
Cl
1h
2.10
1.01
1.89
−115
−120
−125
−130
−135
−140
−145
−150
−155
−160
−165
−170
−175
−180
f1 (ppm)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7.48
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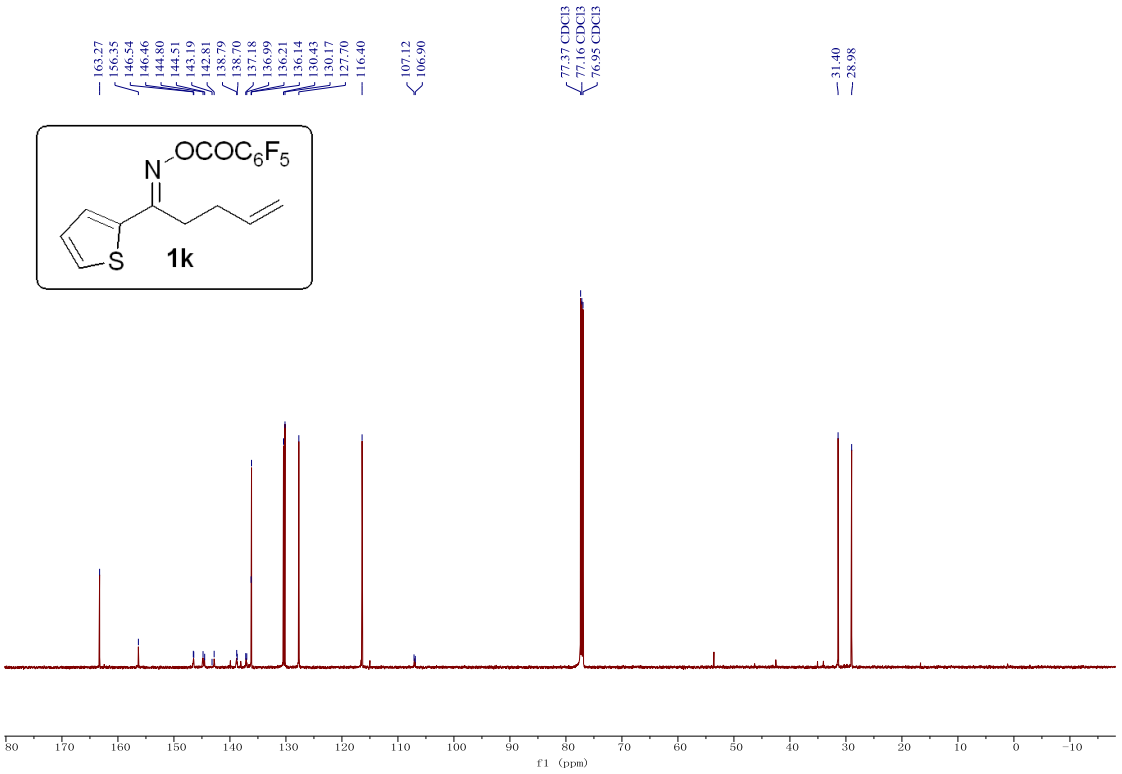
7.12
7.11
7.11
7.10
5.88
5.87
5.84
5.82
5.80
5.78
5.11
5.10
5.06
5.05
5.02
5.02
2.98
2.97
2.95
2.45
2.45
2.43
2.41
2.39
2.39
N
OCOC6F5
S
1k
1.91
0.97
1.00
2.01
1.95
1.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)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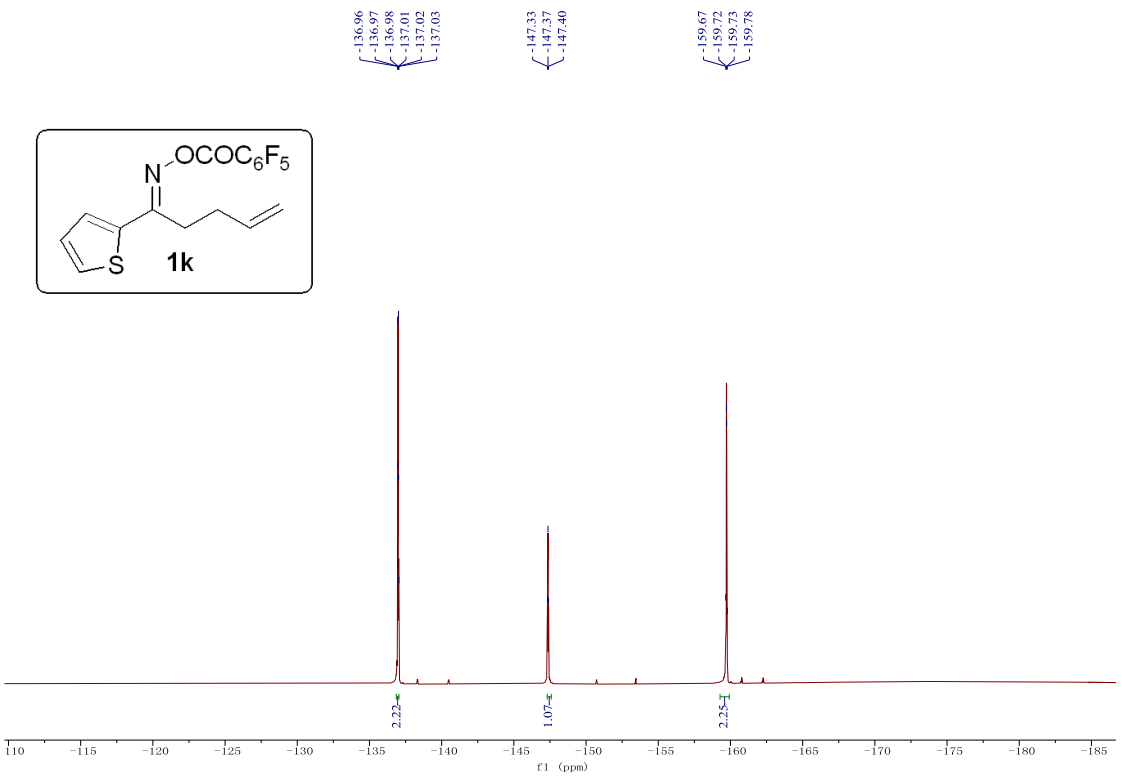
138.70
137.18
136.99
136.21
136.14
130.43
130.17
127.70
116.40
107.12
106.90
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
31.40
28.98
N
OCOC6F5
S
1k
f1 (ppm)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136.98
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137.03
147.33
147.37
147.40
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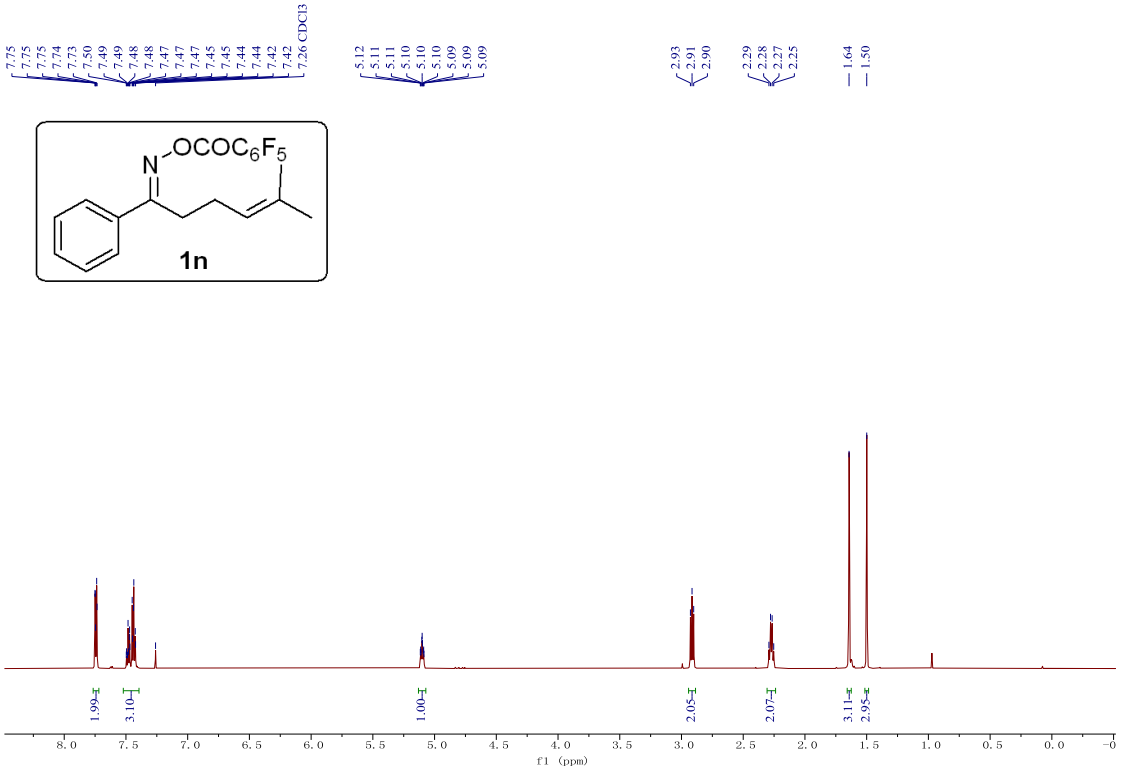
159.73
159.78
N
OCOC6F5
S
1k
2.22
1.07
2.25
f1 (ppm)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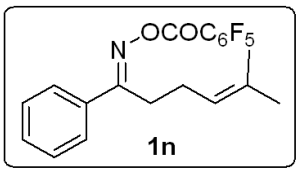
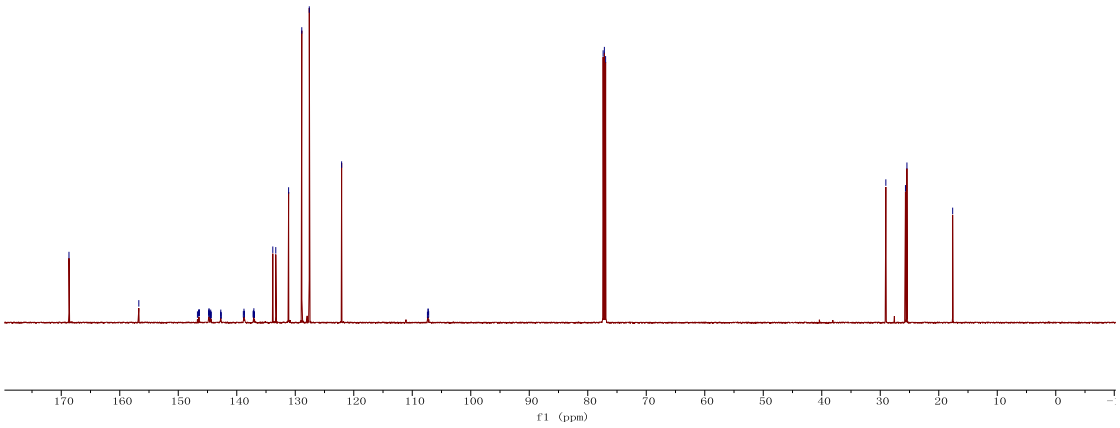
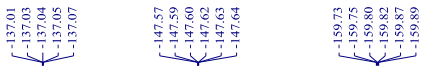
7.75
7.74
7.73
7.50
7.49
7.49
7.48
7.48
7.47
7.47
7.47
7.45
7.45
7.44
7.44
7.42
7.42
7.26 CDCl3
5.12
5.11
5.11
5.10
5.10
5.10
5.09
5.09
5.09
2.93
2.91
2.90
2.29
2.28
2.27
2.25
1.64
1.50
N
OCOC6F5
1n
1.99
3.10
1.00
2.05
2.07
3.11
2.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


168.68
156.74
146.72
146.52
146.48
146.44
146.39
144.80
144.76
144.72
144.68
144.54
144.49
144.45
144.42
144.37
142.76
142.73
142.64
138.88
138.77
138.69
137.20
137.16
137.07
137.01
136.97
133.84
133.33
131.14
128.88
127.60
122.07
107.41
107.30
107.27
107.19
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
29.05
25.69
25.44
17.62


N
OCOC6F5
1n


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-137.01
-137.03
-137.04
-137.05
-137.07
-147.57
-147.59
-147.60
-147.62
-147.63
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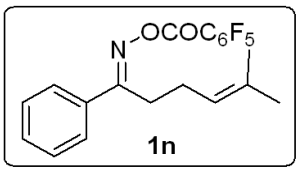
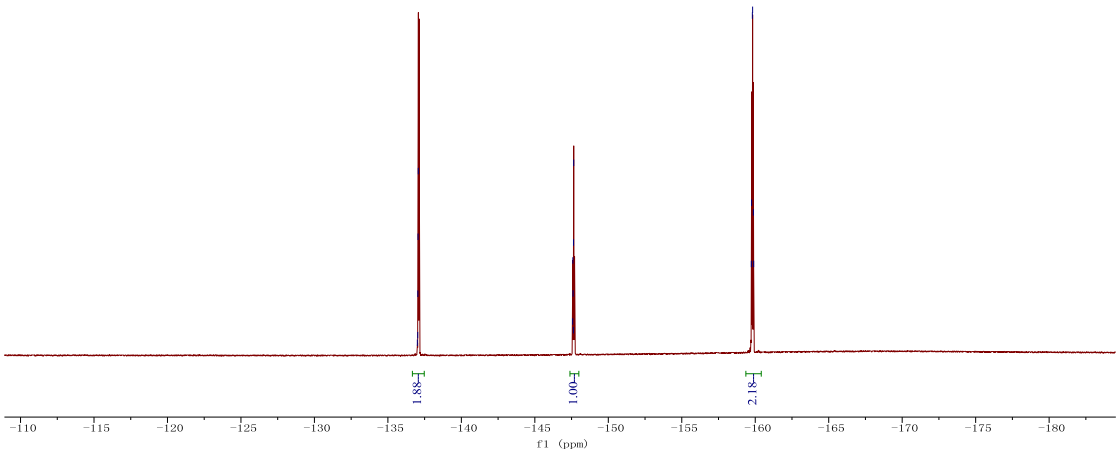
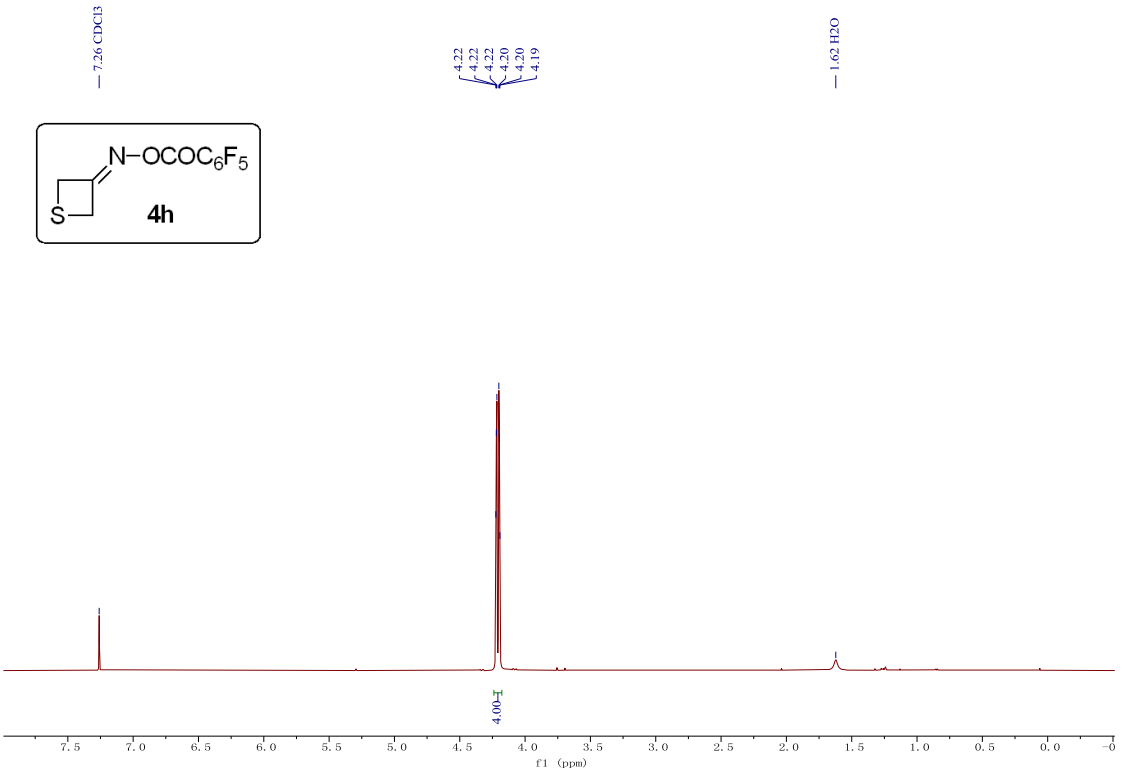
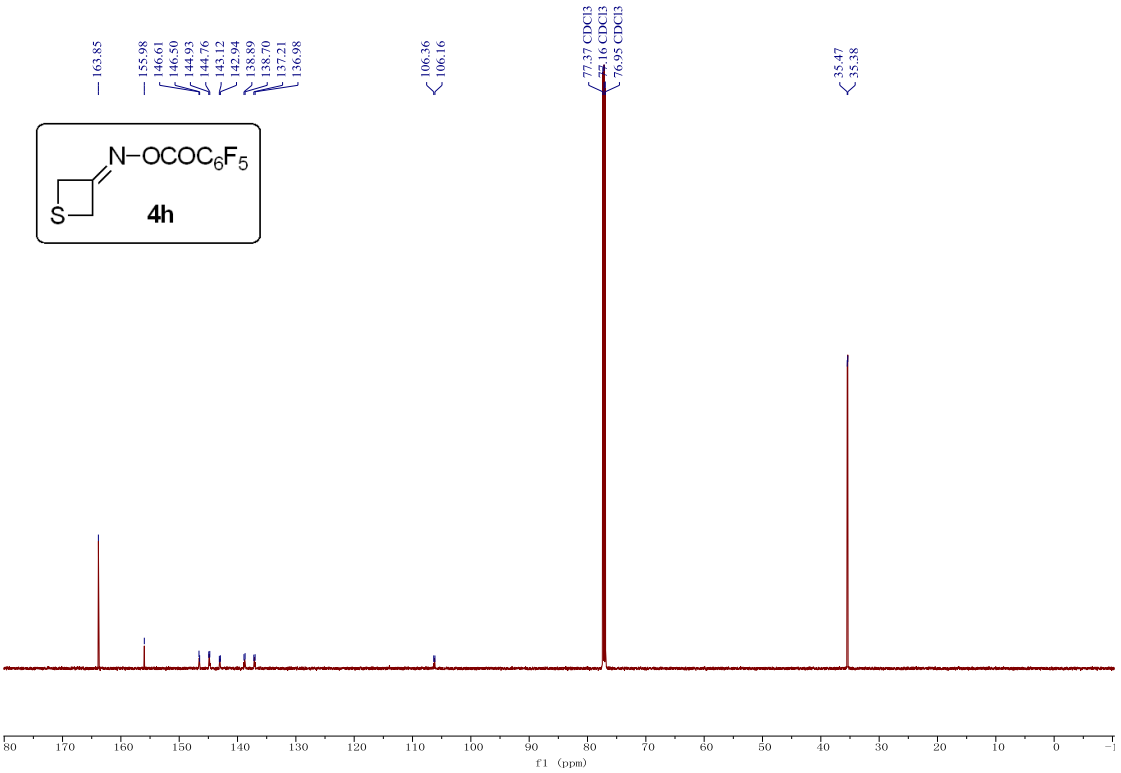
-159.75
-159.80
-159.82
-159.87
-159.89


N
OCOC6F5
1n


1.88
1.00
2.18
-110
-115
-120
-125
-130
-135
-140
-145
-150
-155
-160
-165
-170
-175
-180
f1 (ppm)


7.26 CDCl3
4.22
4.22
4.22
4.20
4.20
4.19
1.62 H2O
N–OCOC6F5
S
4h
4.00
f1 (ppm)


163.85
155.98
146.61
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144.93
144.76
143.12
142.94
138.89
138.70
137.21
136.98
106.36
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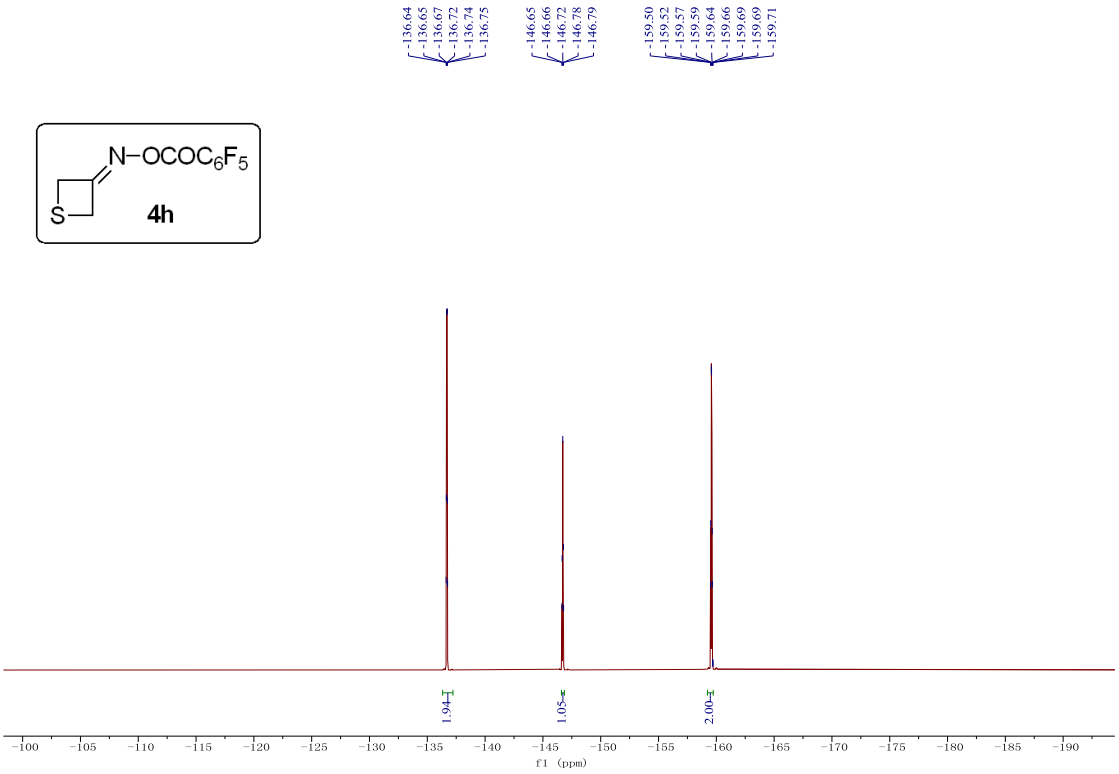
77.16 CDCl3
76.95 CDCl3
35.47
35.38
N–OCOC6F5
S
4h
f1 (ppm)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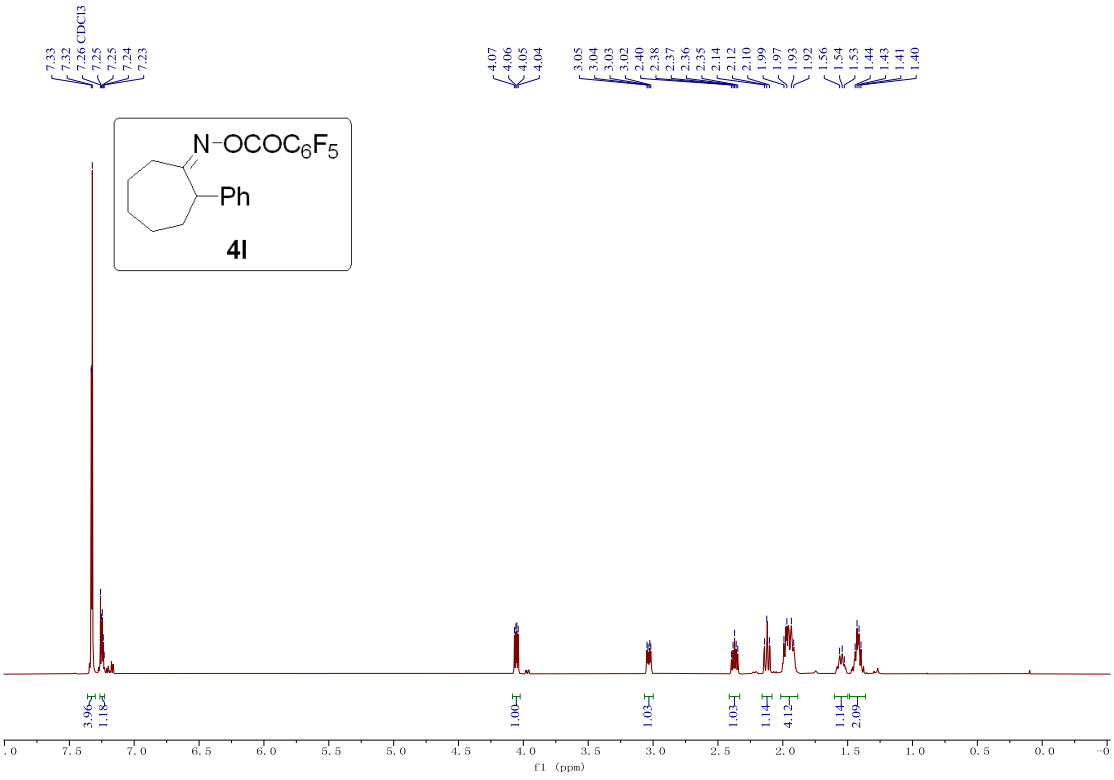
-146.65
-146.66
-146.72
-146.78
-146.79
-159.50
-159.52
-159.57
-159.59
-159.64
-159.66
-159.69
-159.69
-159.71
N–OCOC6F5
S
4h
1.94
1.05
2.00
f1 (ppm)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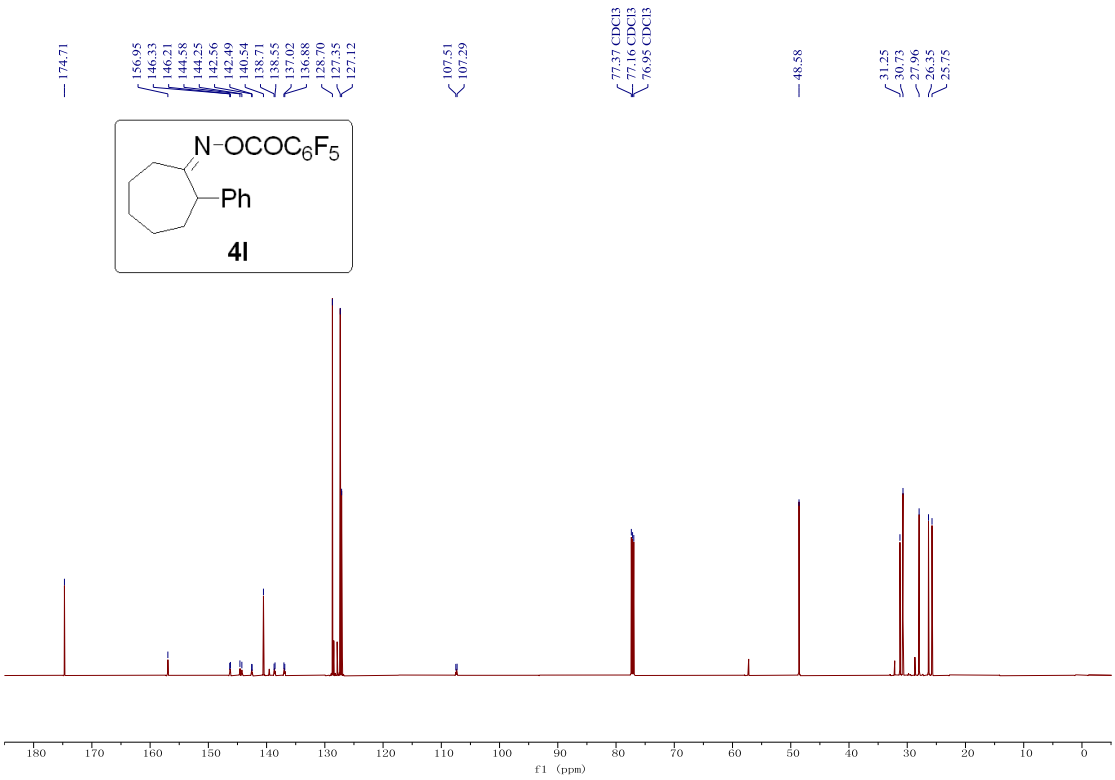
7.25
7.24
7.23
4.07
4.06
4.05
4.04
3.05
3.04
3.03
3.02
2.40
2.38
2.37
2.36
2.35
2.14
2.12
2.10
1.99
1.97
1.93
1.92
1.56
1.54
1.53
1.44
1.43
1.41
1.40
N–OCOC6F5
Ph
4l
3.96
1.18
1.00
1.03
1.03
1.14
4.12
1.14
2.09
f1 (ppm)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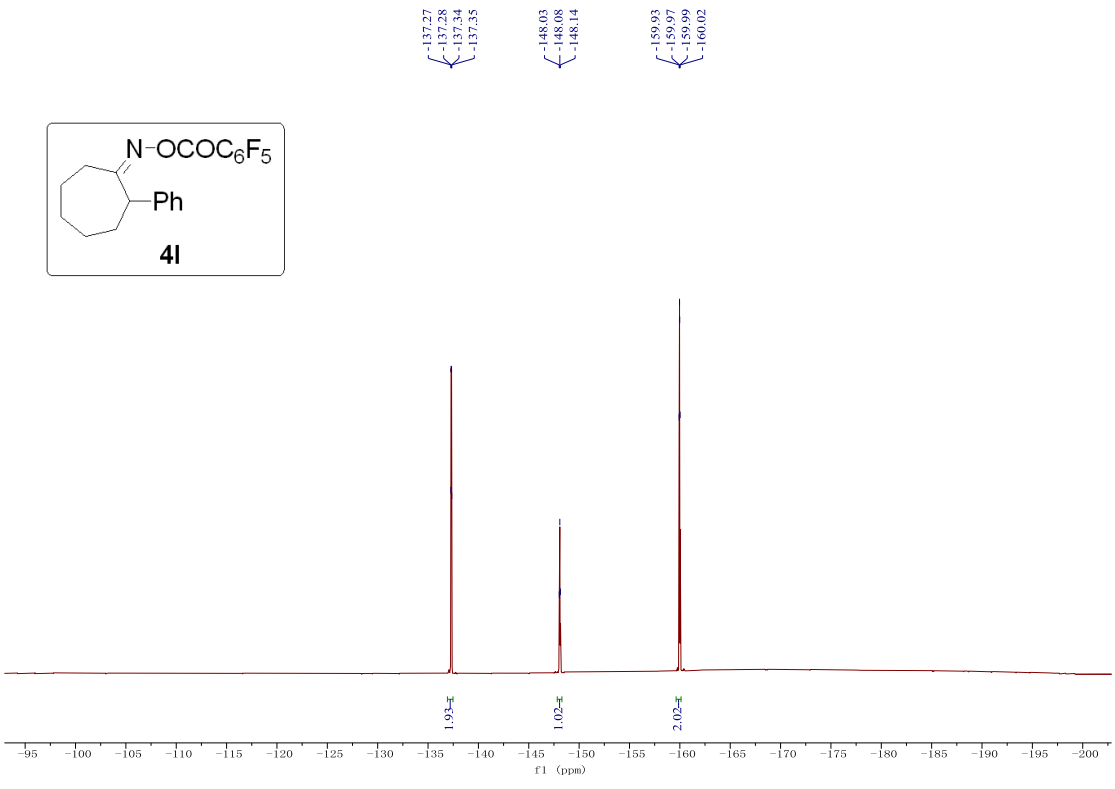
144.25
142.56
142.49
140.54
138.71
138.55
137.02
136.88
128.70
127.35
127.12
107.51
107.29
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
48.58
31.25
30.73
27.96
26.35
25.75
N-OCOC6F5
Ph
4l
f1 (ppm)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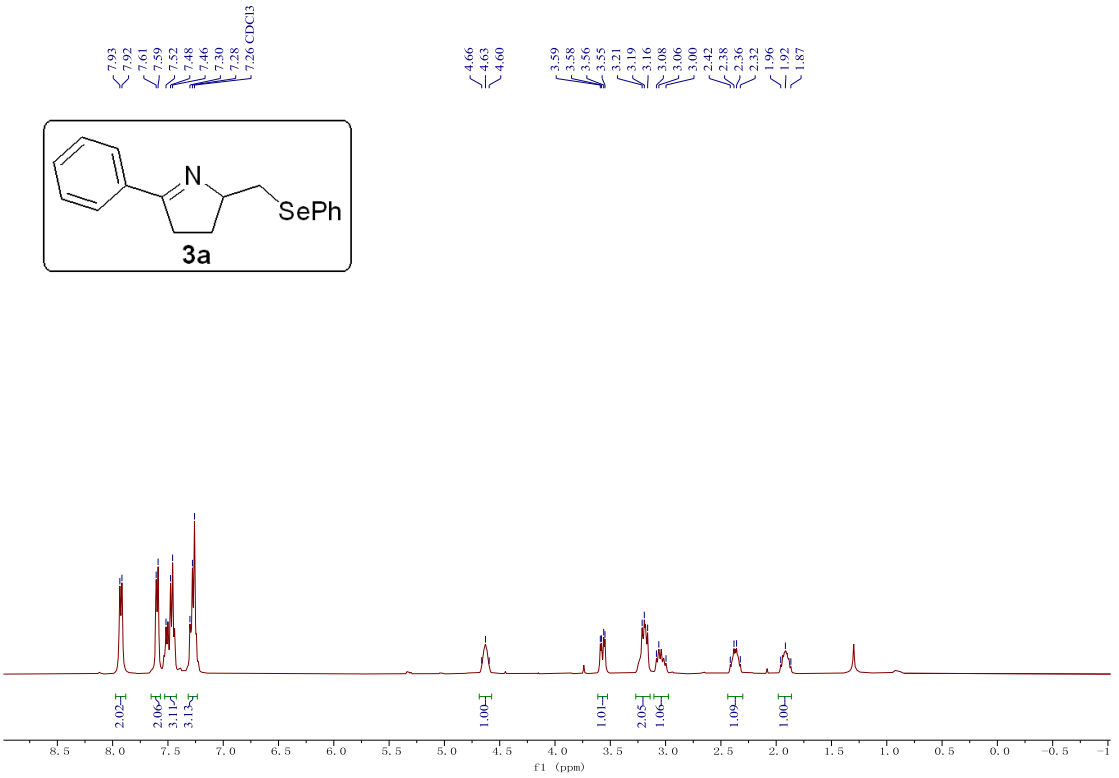
-137.28
-137.34
-137.35
-148.03
-148.08
-148.14
-159.93
-159.97
-159.99
-160.02
N-OCOC6F5
Ph
4l
1.93
1.02
2.02
f1 (ppm)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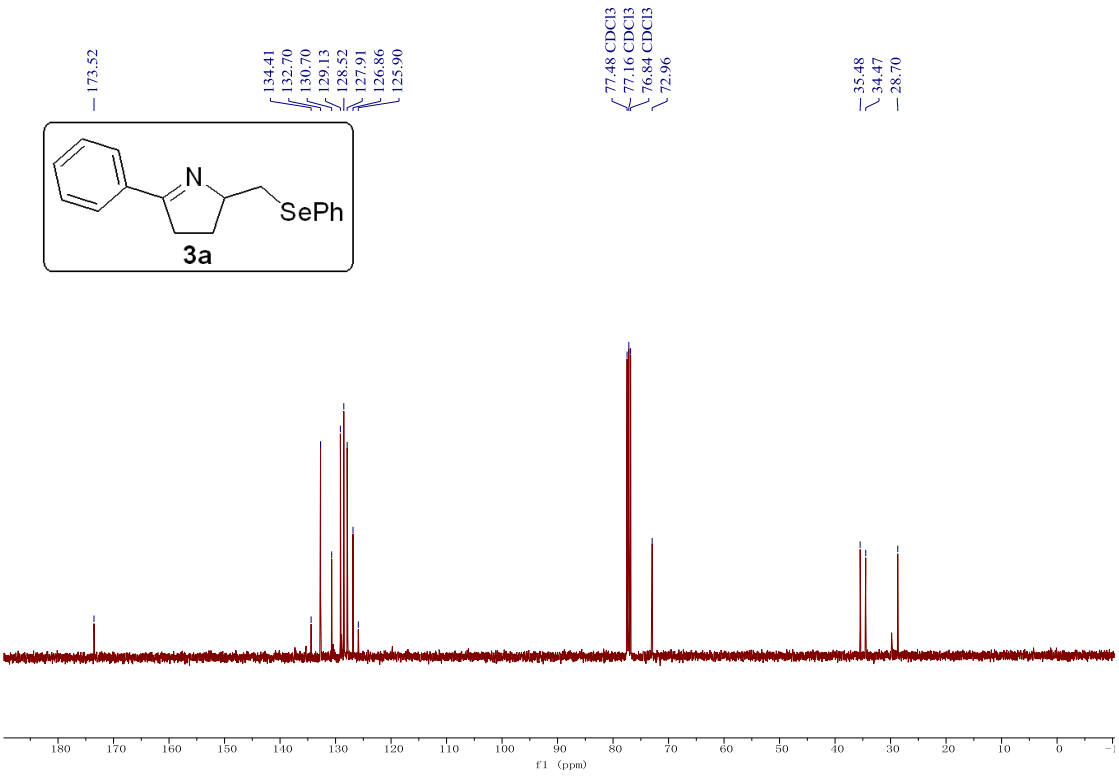
7.92
7.61
7.59
7.52
7.48
7.46
7.30
7.28
7.26 CDCl3
4.66
4.63
4.60
3.59
3.58
3.56
3.55
3.21
3.19
3.16
3.08
3.06
3.00
2.42
2.38
2.36
2.32
1.96
1.92
1.87
N
SePh
3a
2.02
2.06
3.11
3.13
1.00
1.01
2.05
1.06
1.09
1.00
f1 (ppm)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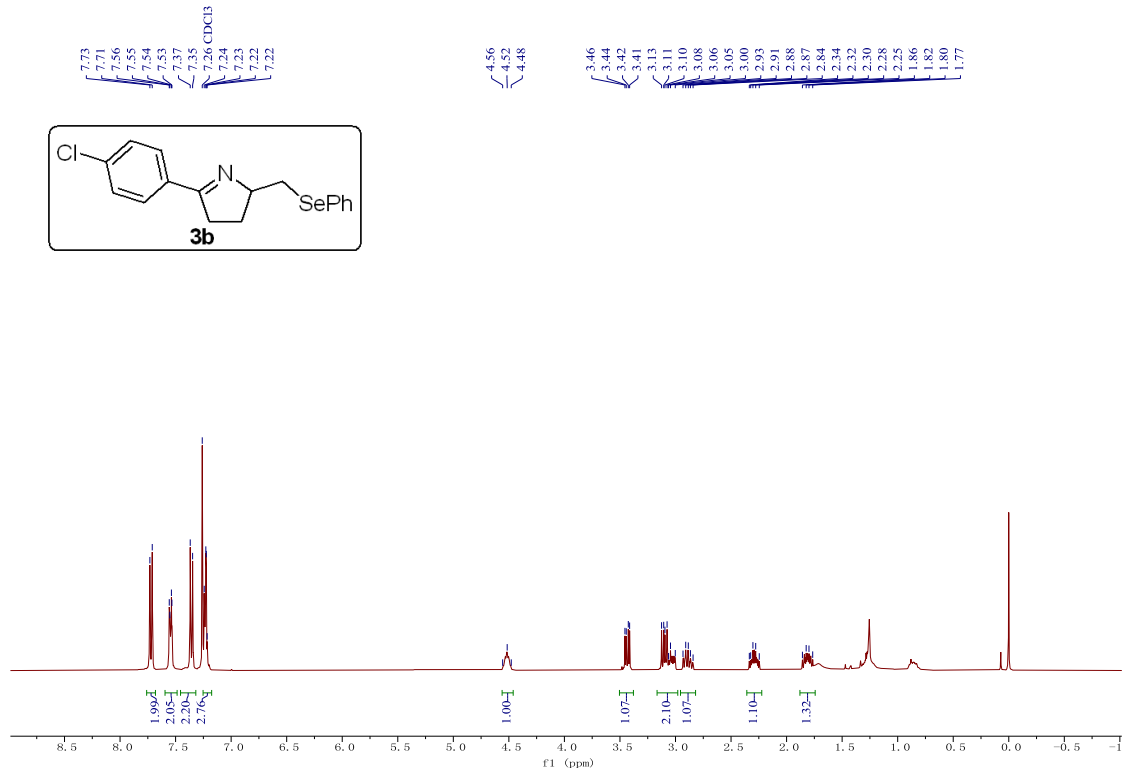
134.41
132.70
130.70
129.13
128.52
127.91
126.86
125.90
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
72.96
35.48
34.47
28.70
N
SePh
3a
f1 (ppm)


7.73
7.71
7.56
7.55
7.54
7.53
7.37
7.35
7.26 CDCl3
7.24
7.23
7.22
7.22
4.56
4.52
4.48
3.46
3.44
3.42
3.41
3.13
3.11
3.10
3.08
3.06
3.05
3.00
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2.91
2.88
2.87
2.84
2.34
2.32
2.30
2.28
2.25
1.86
1.82
1.80
1.77
Cl
N
SePh
3b
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2.20
2.76
1.00
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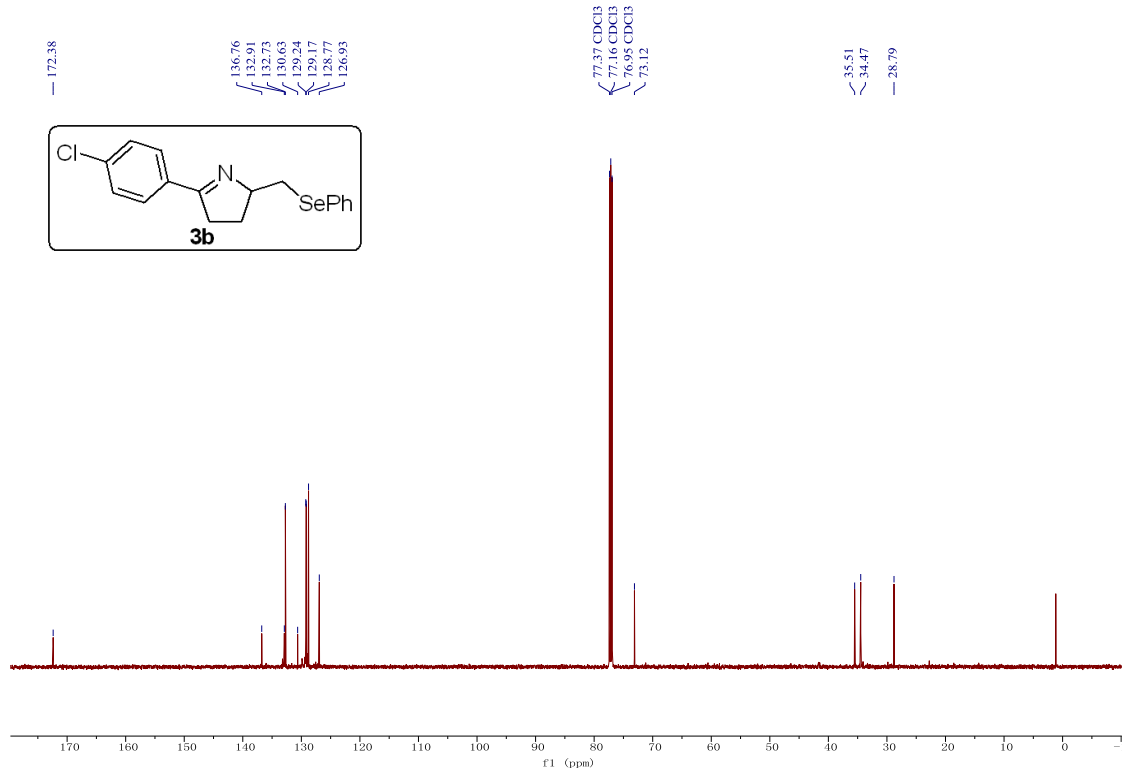
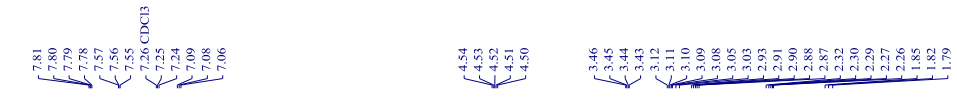
2.10
1.07
1.10
1.32
f1 (ppm)


172.38
136.76
132.91
132.73
130.63
129.24
129.17
128.77
126.93
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
73.12
35.51
34.47
28.79
Cl
N
SePh
3b
f1 (ppm)


7.81
7.80
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7.78
7.57
7.56
7.55
7.26 CDCl3
7.25
7.24
7.09
7.08
7.06
4.54
4.53
4.52
4.51
4.50
3.46
3.45
3.44
3.43
3.12
3.11
3.10
3.09
3.08
3.05
3.03
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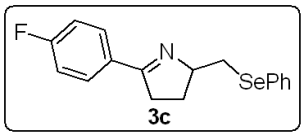
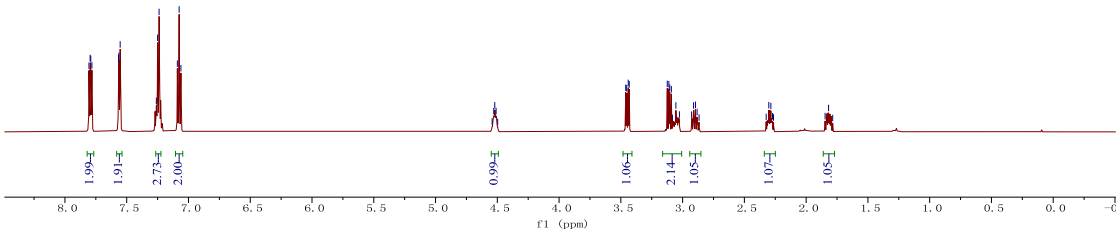
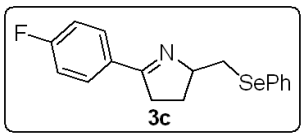
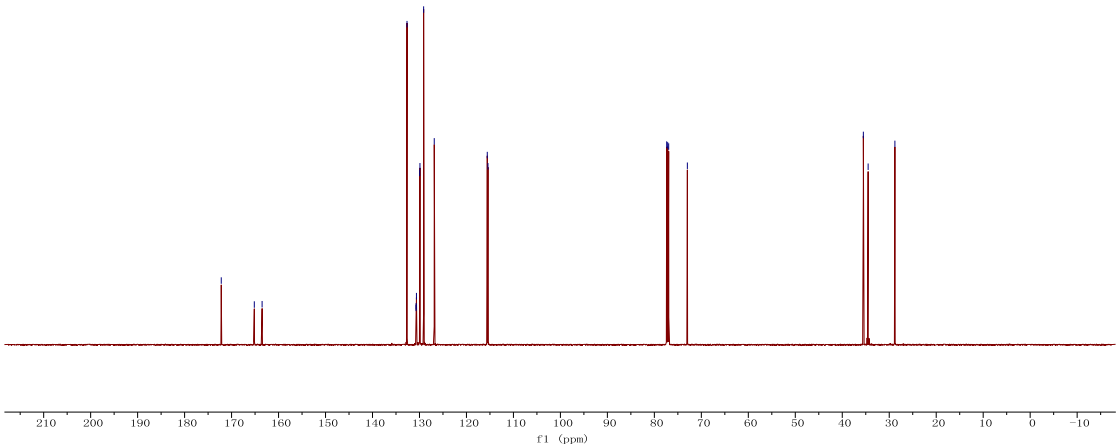
2.27
2.26
1.85
1.82
1.79

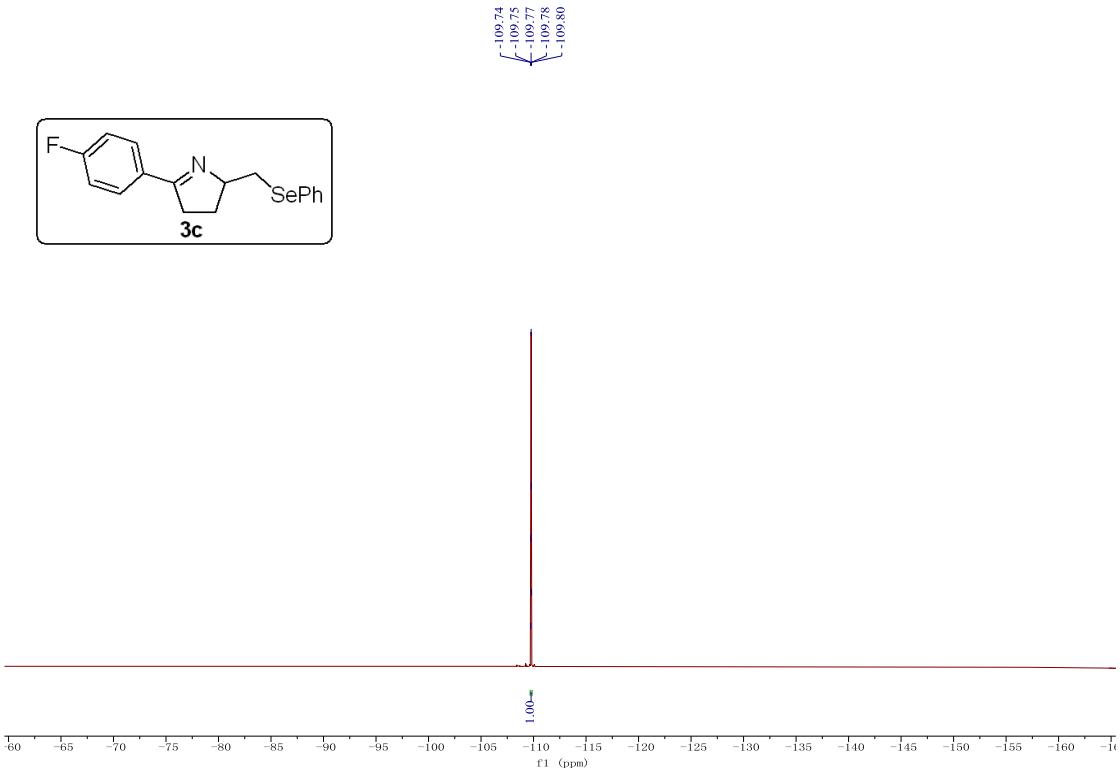

F
N
SePh
3c


1.99
1.91
2.73
2.00
0.99
1.06
2.14
1.05
1.07
1.05
f1 (ppm)


172.20
165.16
163.50
132.65
130.73
130.71
130.65
129.96
129.90
129.12
126.86
115.57
115.42
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
72.97
35.51
34.49
28.80


F
N
SePh
3c


f1 (ppm)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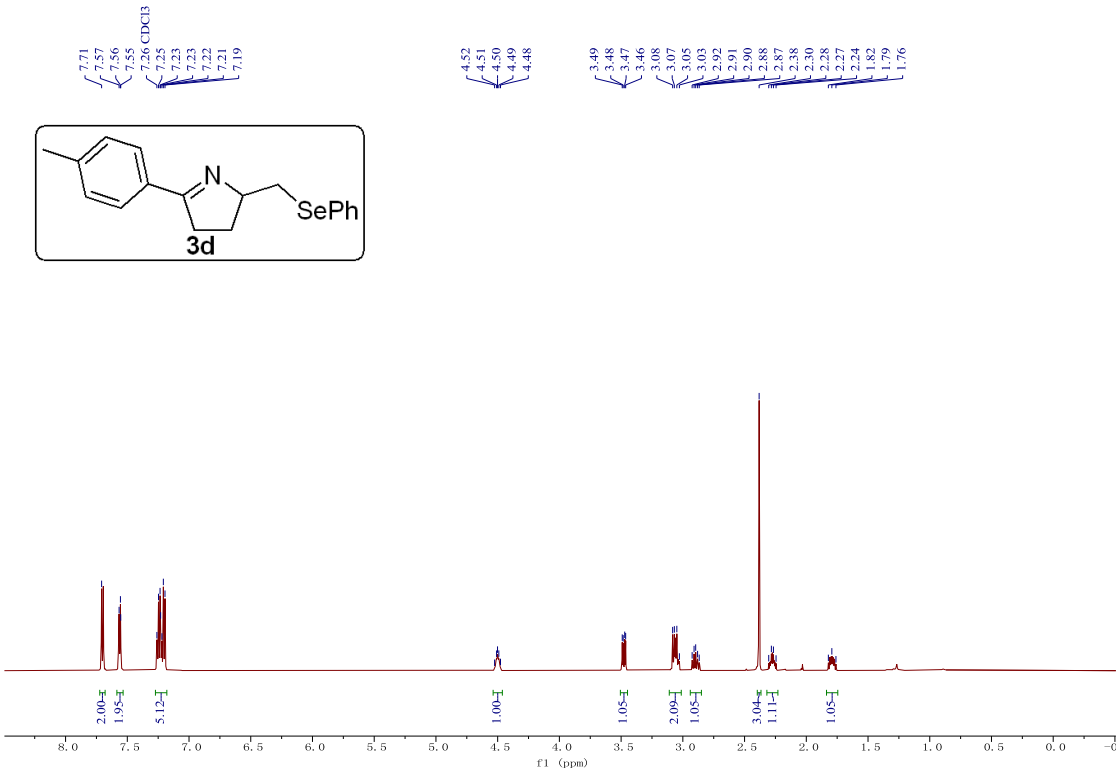
-109.78
-109.80
F
N
SePh
3c
1.00
f1 (ppm)


7.71
7.57
7.56
7.55
7.26 CDCl3
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4.50
4.49
4.48
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3.48
3.47
3.46
3.08
3.07
3.05
3.03
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2.87
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2.30
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2.27
2.24
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1.79
1.76
N
SePh
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1.05
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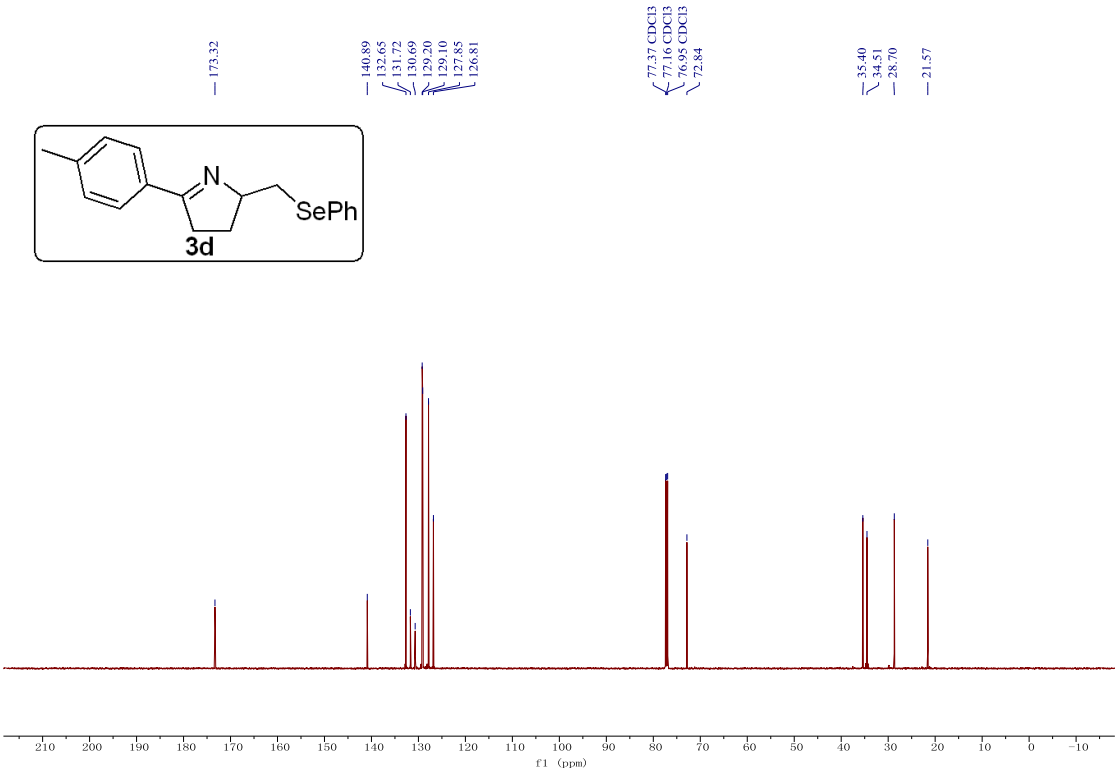
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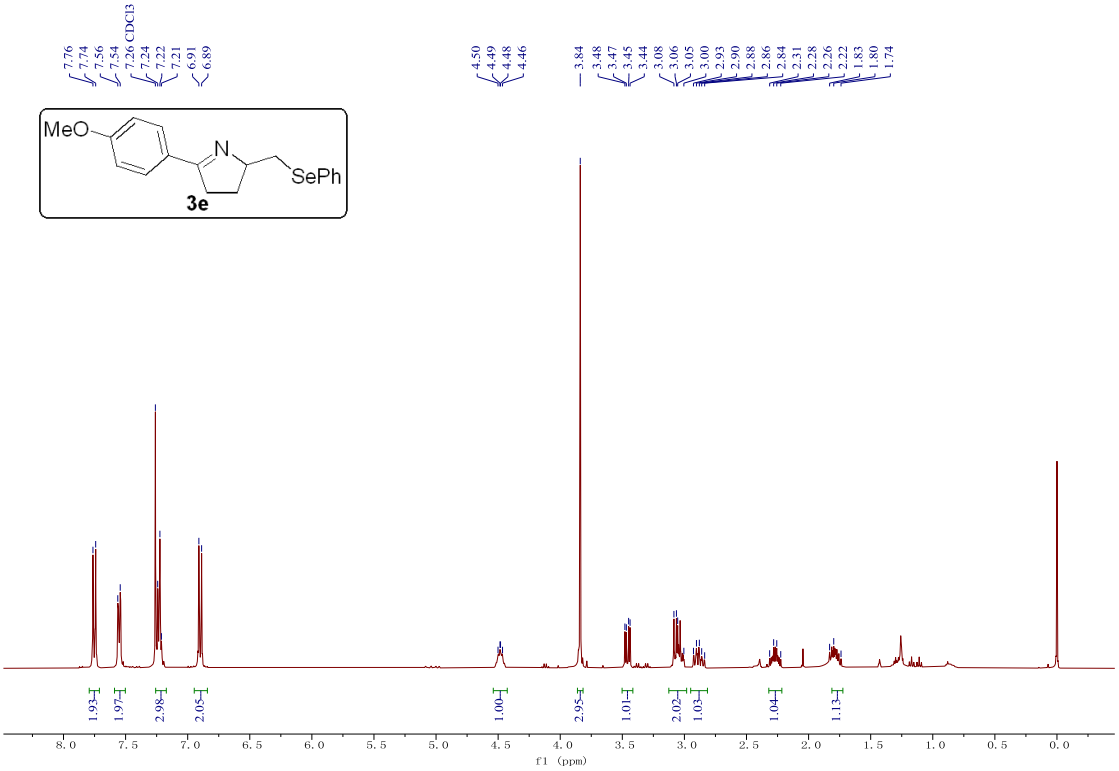
f1 (ppm)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2.31
2.28
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2.22
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1.80
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MeO
N
SePh
3e
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1.01
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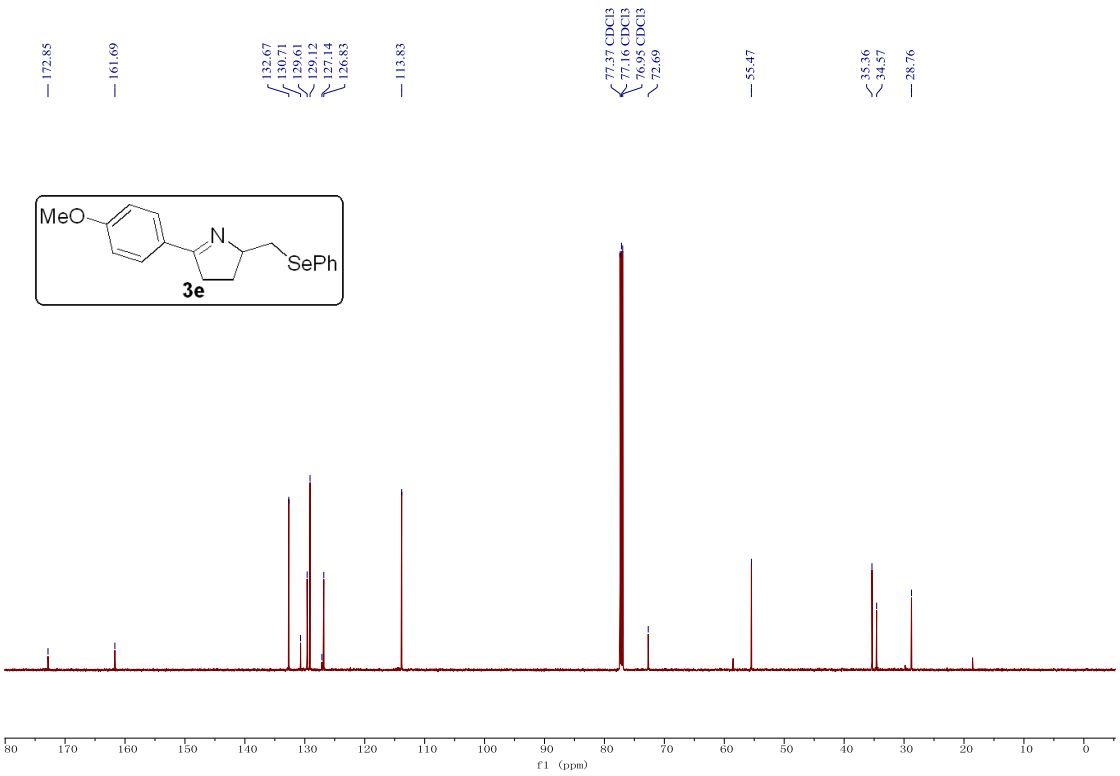
f1 (ppm)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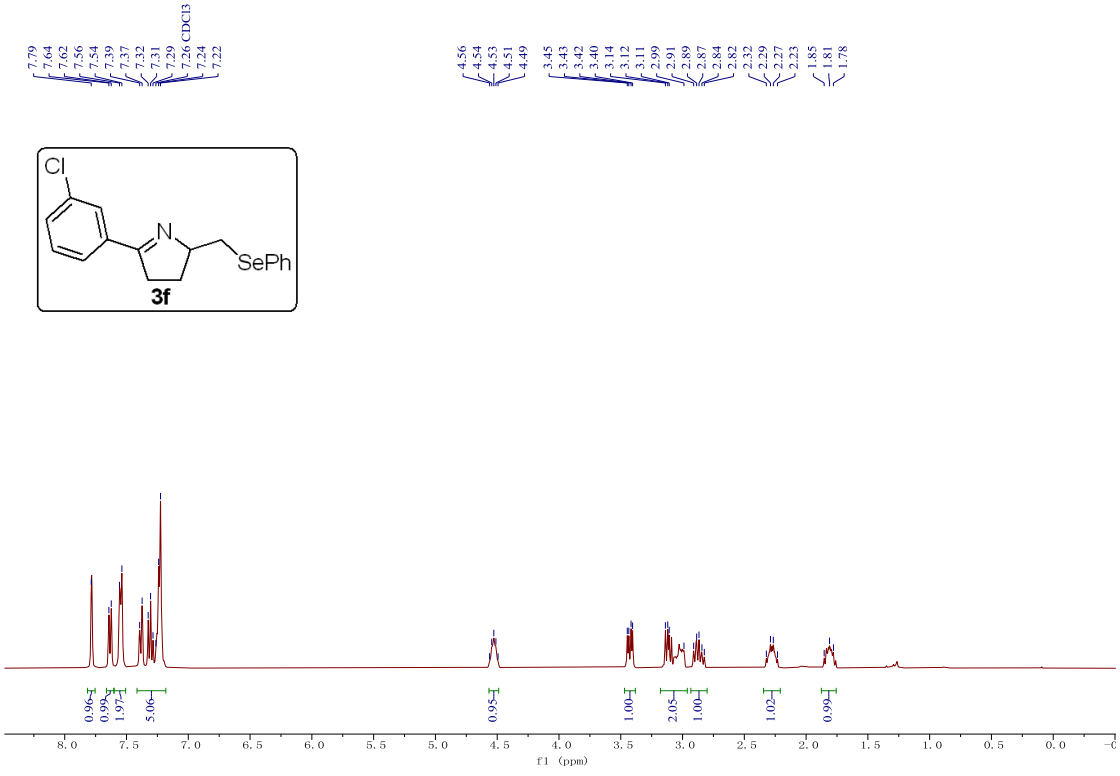
MeO
N
SePh
3e
f1 (ppm)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4.54
4.53
4.51
4.49
3.45
3.43
3.42
3.40
3.14
3.12
3.11
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2.89
2.87
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2.82
2.32
2.29
2.27
2.23
1.85
1.81
1.78
Cl
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SePh
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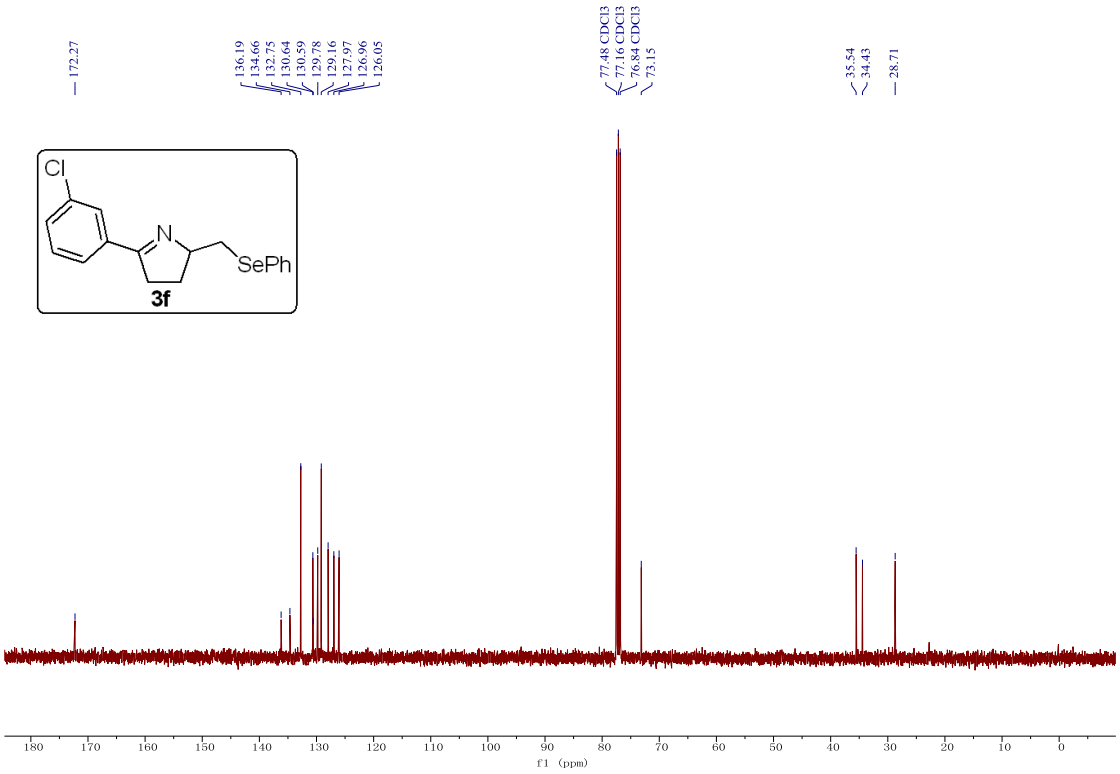
f1 (ppm)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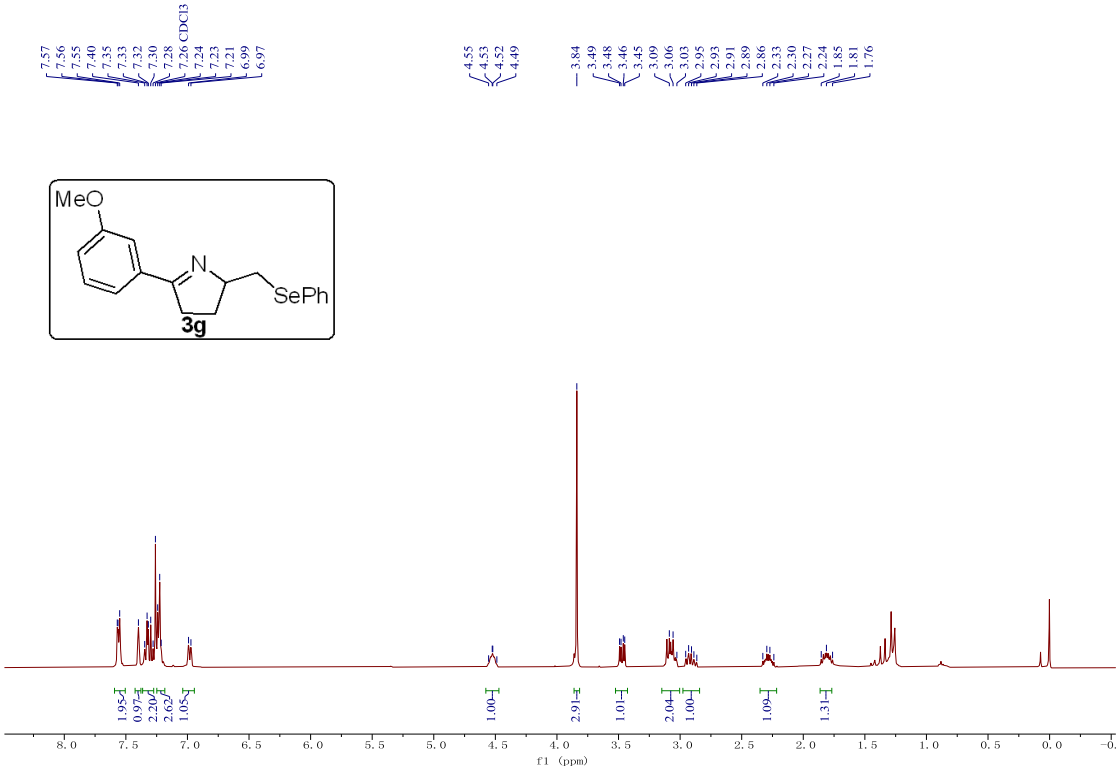
SePh
3f
f1 (ppm)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7.26 CDCl3
7.24
7.23
7.21
6.99
6.97
4.55
4.53
4.52
4.49
3.84
3.49
3.48
3.46
3.45
3.09
3.06
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2.27
2.24
1.85
1.81
1.76
MeO
N
SePh
3g
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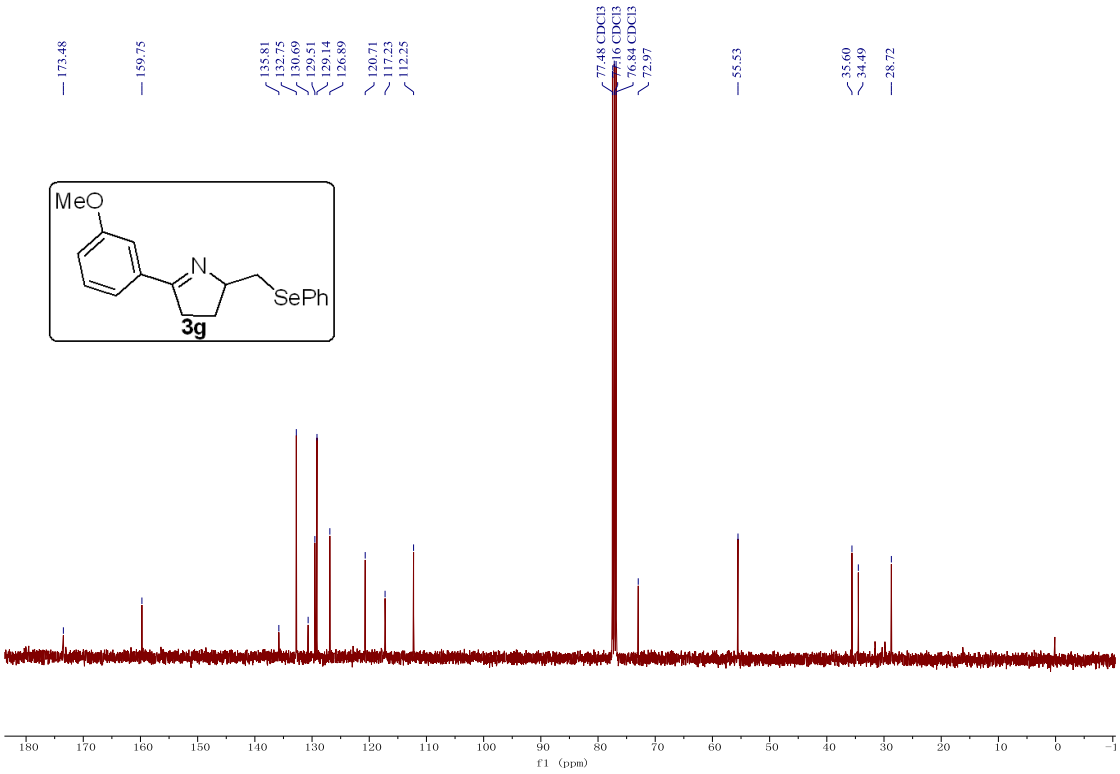
f1 (ppm)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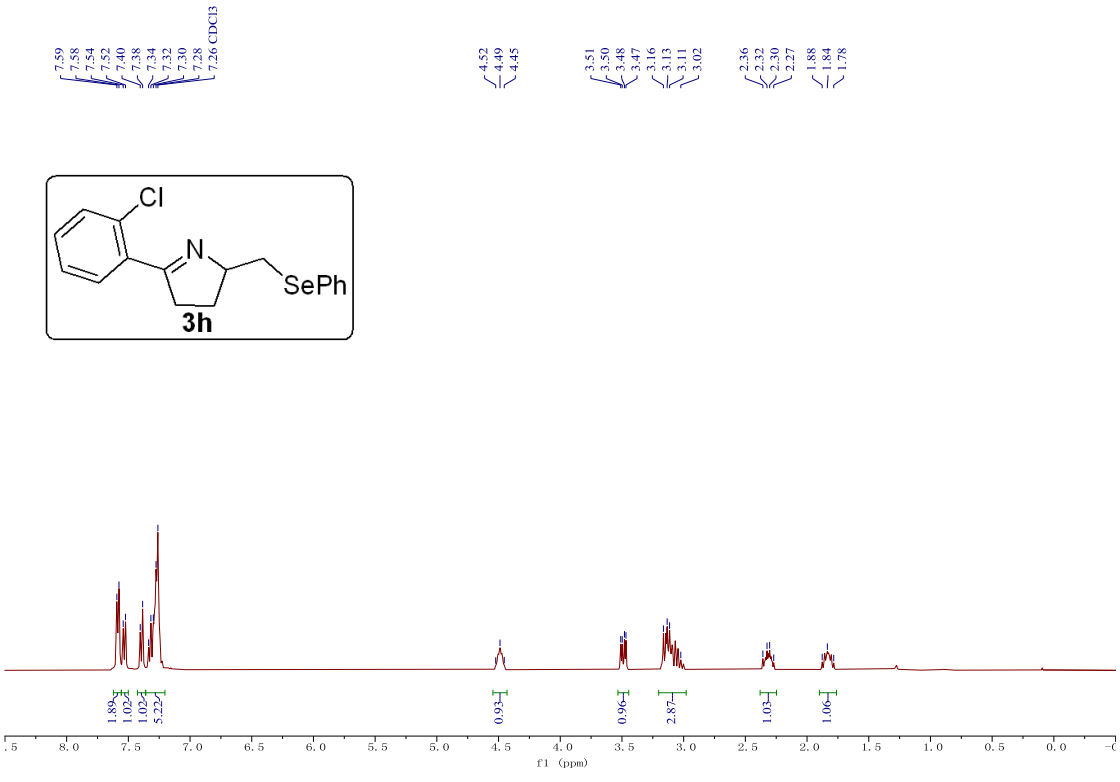
N
SePh
3g
f1 (ppm)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7.28
7.26 CDCl3
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3.48
3.47
3.16
3.13
3.11
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2.30
2.27
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1.84
1.78
Cl
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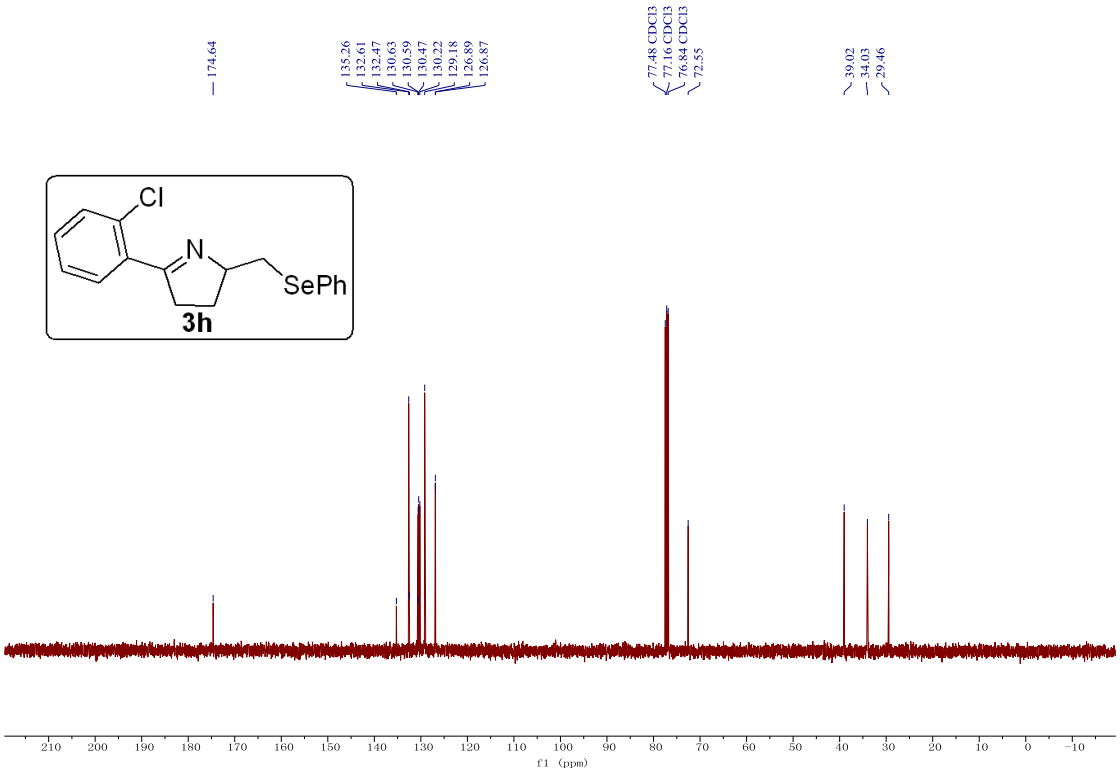
1.06
f1 (ppm)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77.48 CDCl3
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76.84 CDCl3
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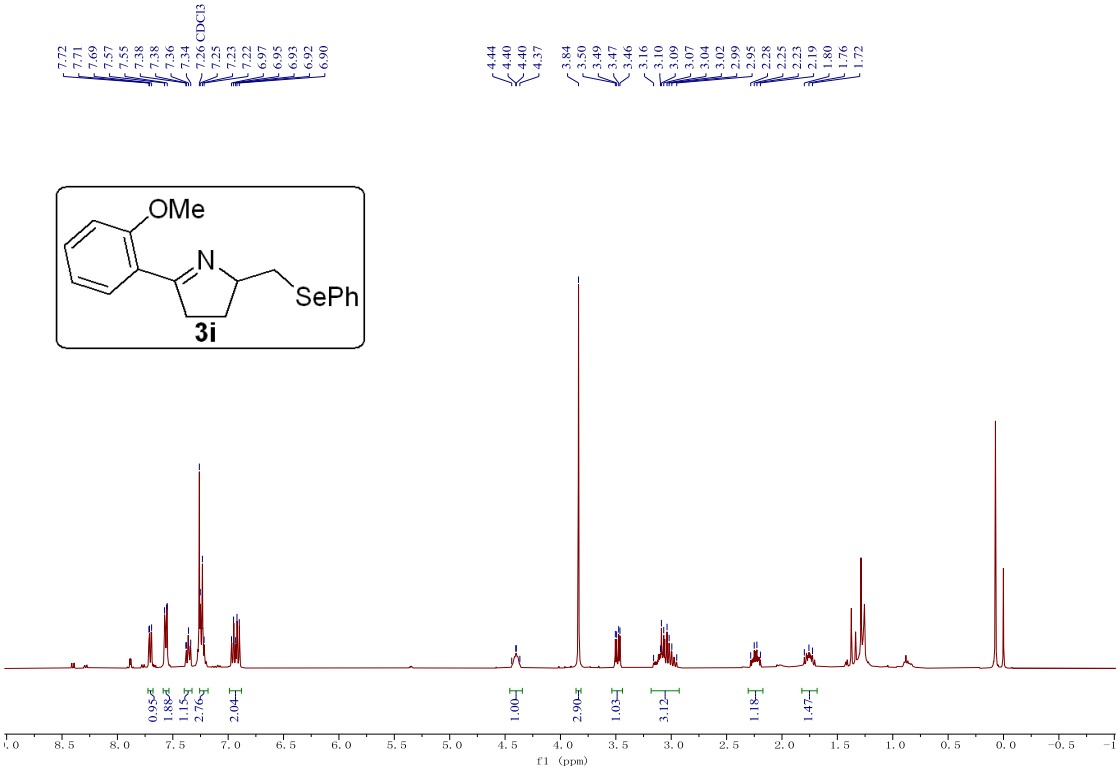
39.02
34.03
29.46
Cl
N
SePh
3h
f1 (ppm)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7.26 CDCl3
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6.97
6.95
6.93
6.92
6.90
4.44
4.40
4.40
4.37
3.84
3.50
3.49
3.47
3.46
3.16
3.10
3.09
3.07
3.04
3.02
2.99
2.95
2.28
2.25
2.23
2.19
1.80
1.76
1.72
OMe
N
SePh
3i
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2.76
2.04
1.00
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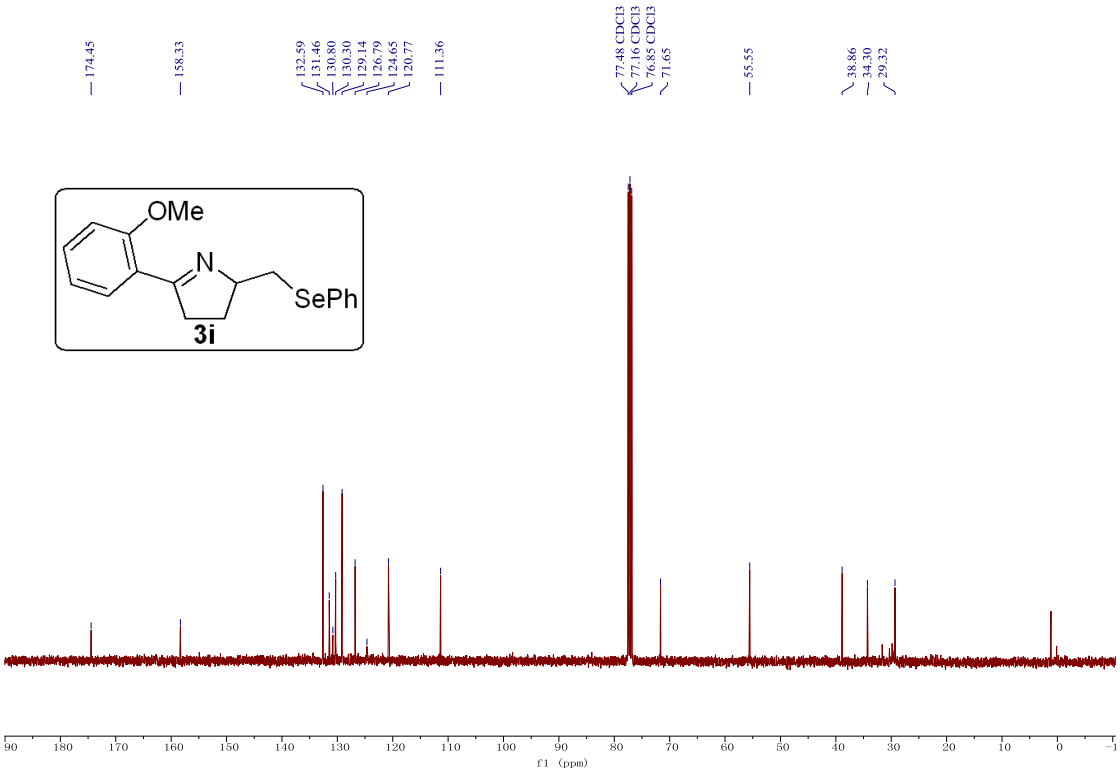
f1 (ppm)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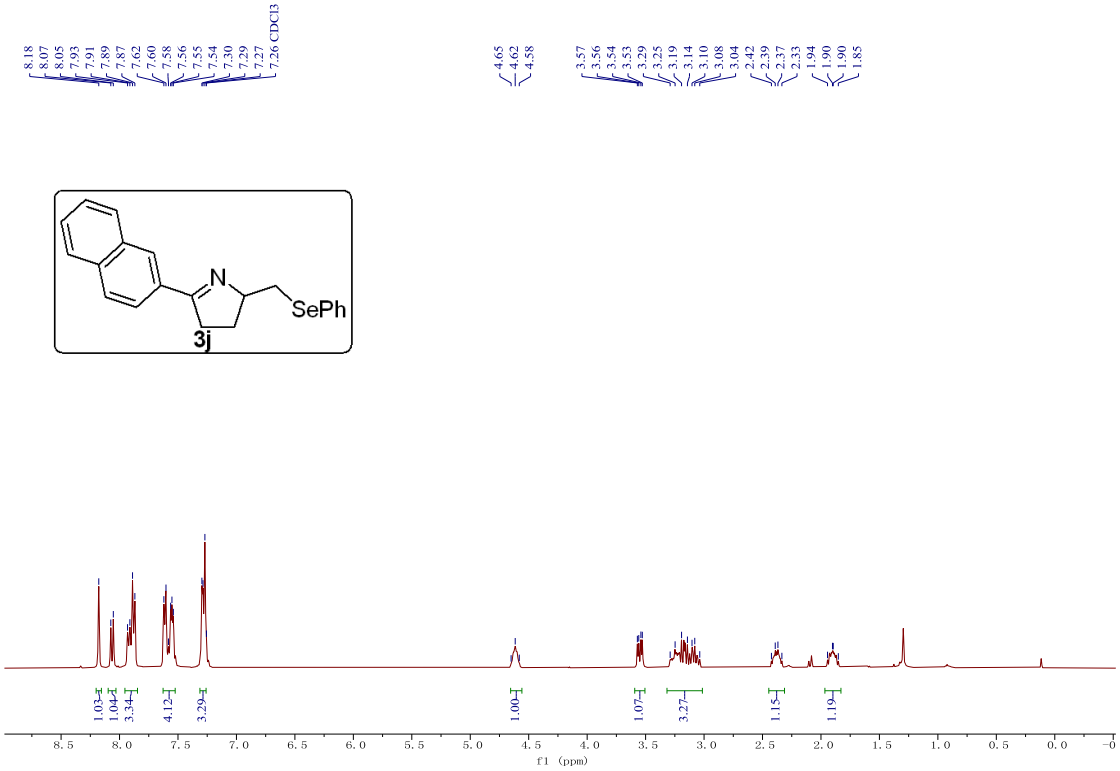
29.32
OMe
N
SePh
3i
f1 (ppm)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7.30
7.29
7.27
7.26 CDCl3
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4.62
4.58
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3.56
3.54
3.53
3.29
3.25
3.19
3.14
3.10
3.08
3.04
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2.39
2.37
2.33
1.94
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N
SePh
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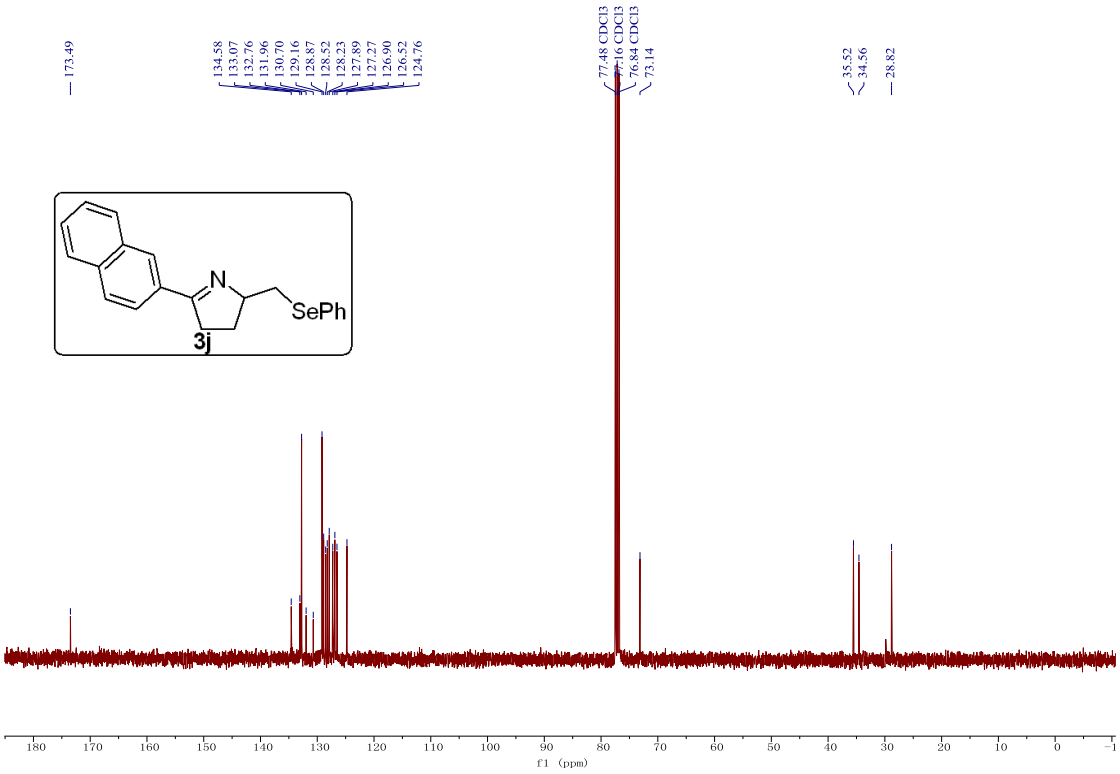
1.19
f1 (ppm)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126.52
124.76
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76.84 CDCl3
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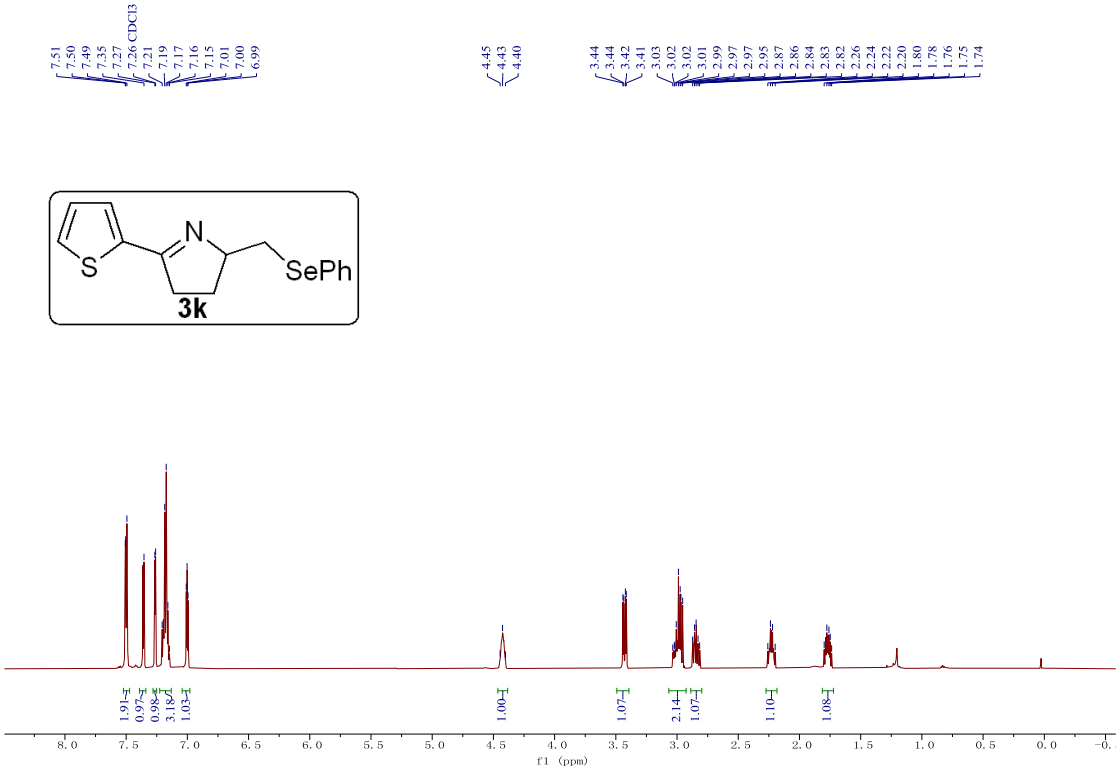
28.82
N
SePh
3j
f1 (ppm)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3.03
3.02
3.02
3.01
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2.97
2.95
2.87
2.86
2.84
2.83
2.82
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2.22
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1.78
1.76
1.75
1.74
S
N
SePh
3k
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1.07
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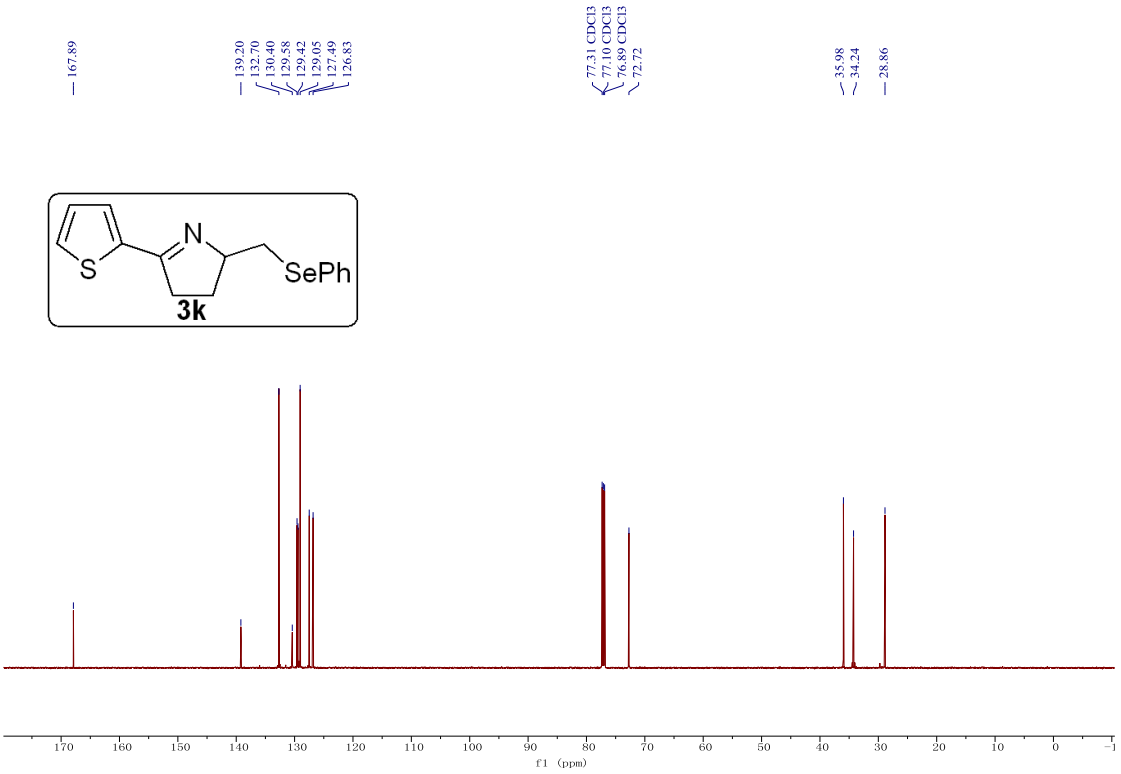
1.08
f1 (ppm)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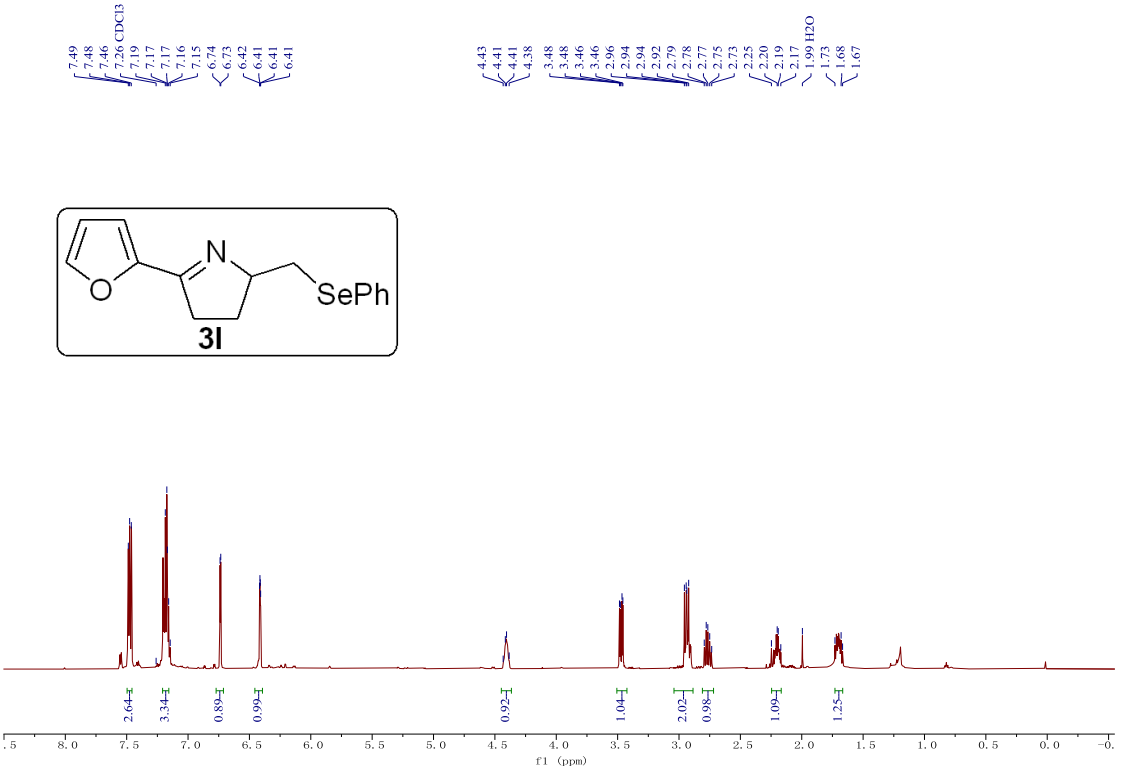
3k
f1 (ppm)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6.41
6.41
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4.41
4.41
4.38
3.48
3.48
3.46
3.46
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2.94
2.94
2.92
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2.78
2.77
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2.73
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2.20
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N
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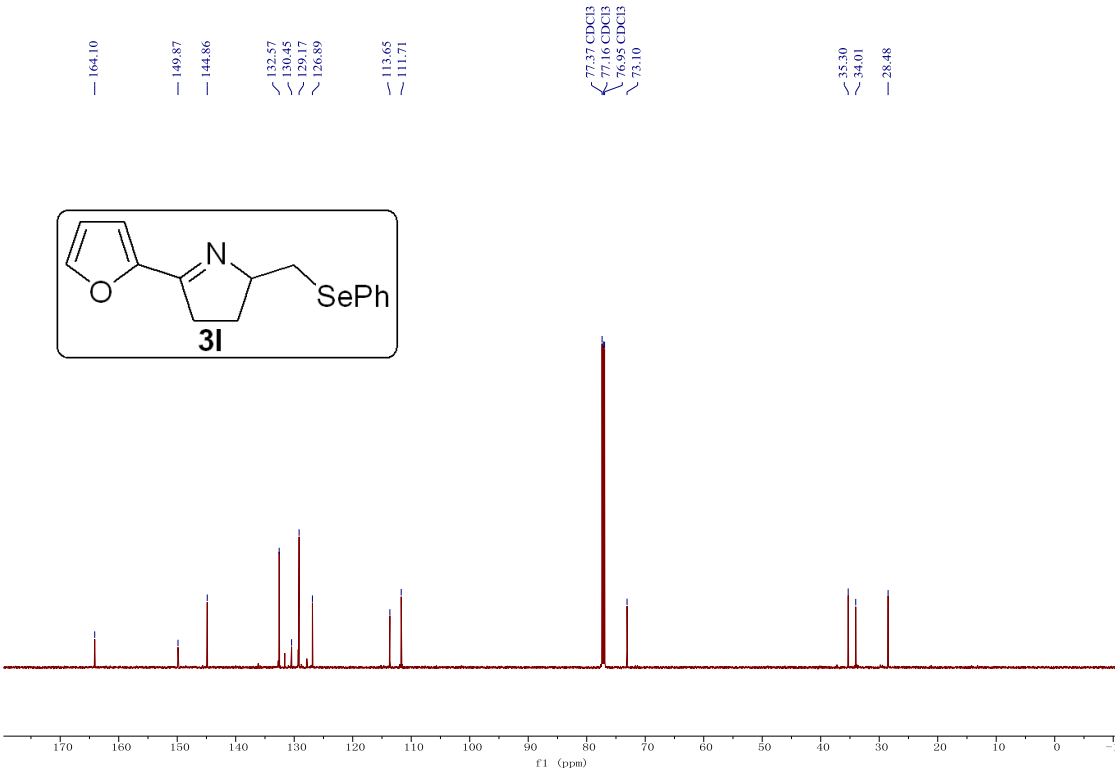
f1 (ppm)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113.65
111.71
77.37 CDCl3
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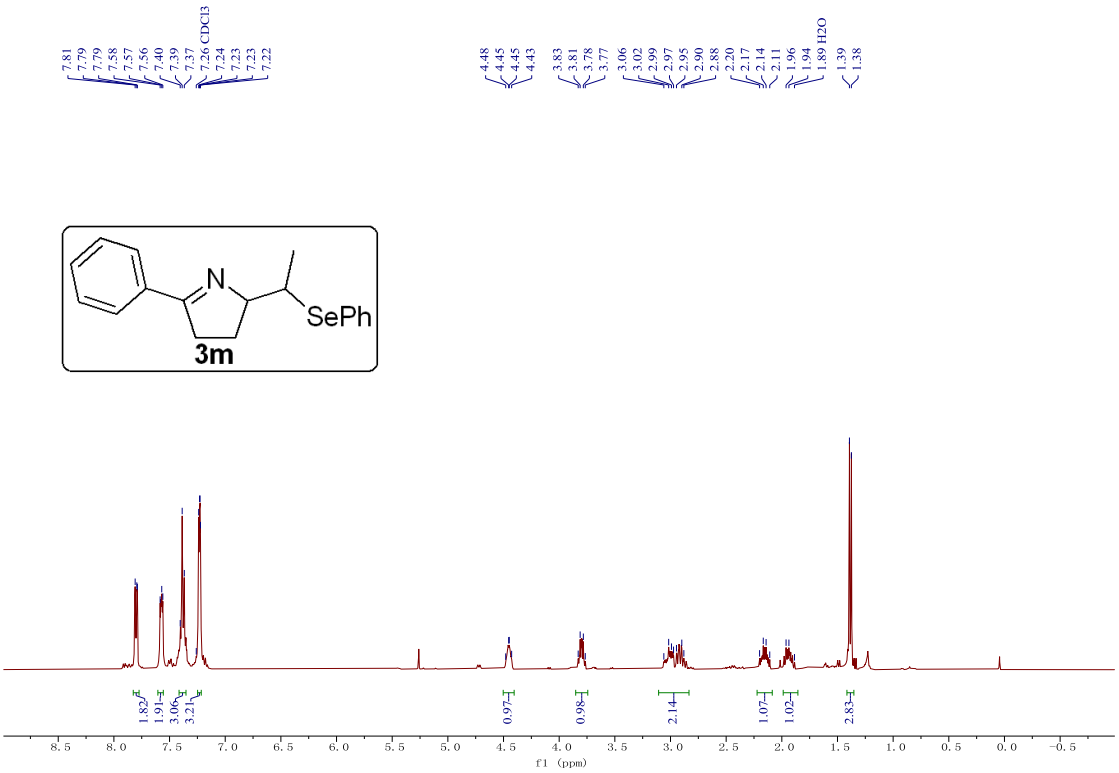
76.95 CDCl3
73.10
35.30
34.01
28.48
O
N
SePh
3l
f1 (ppm)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4.45
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4.43
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3.02
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2.17
2.14
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1.96
1.94
1.89 H2O
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1.38
N
SePh
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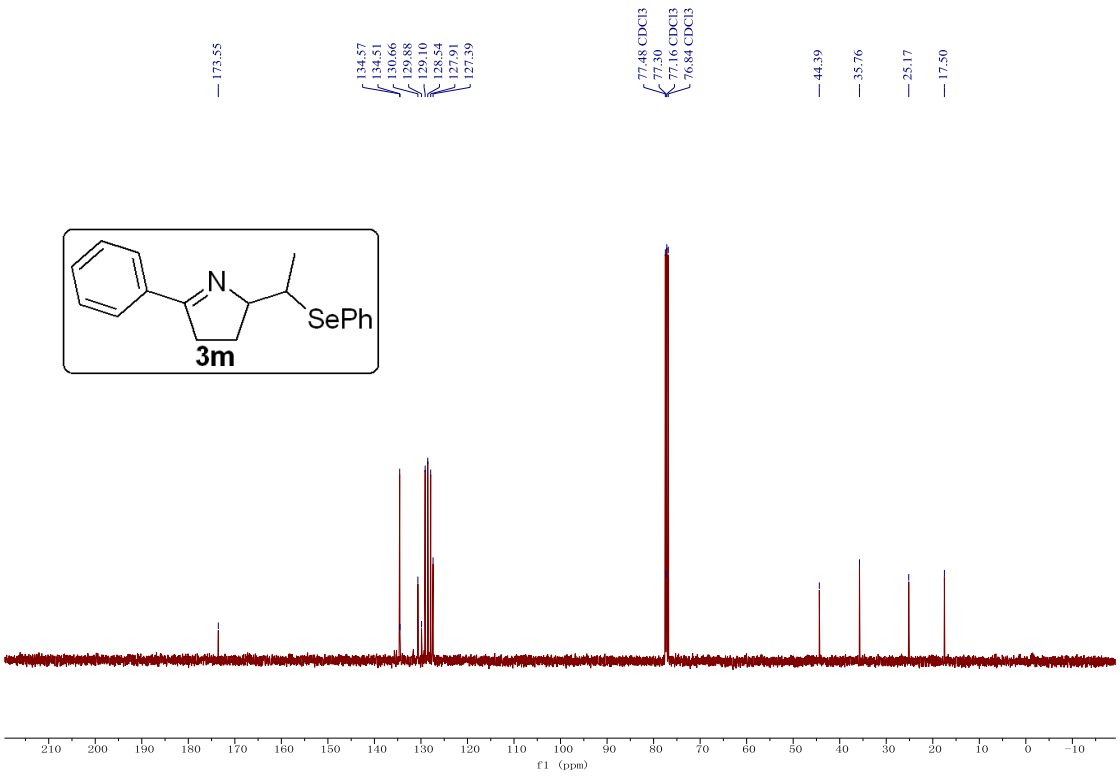
f1 (ppm)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76.84 CDCl3
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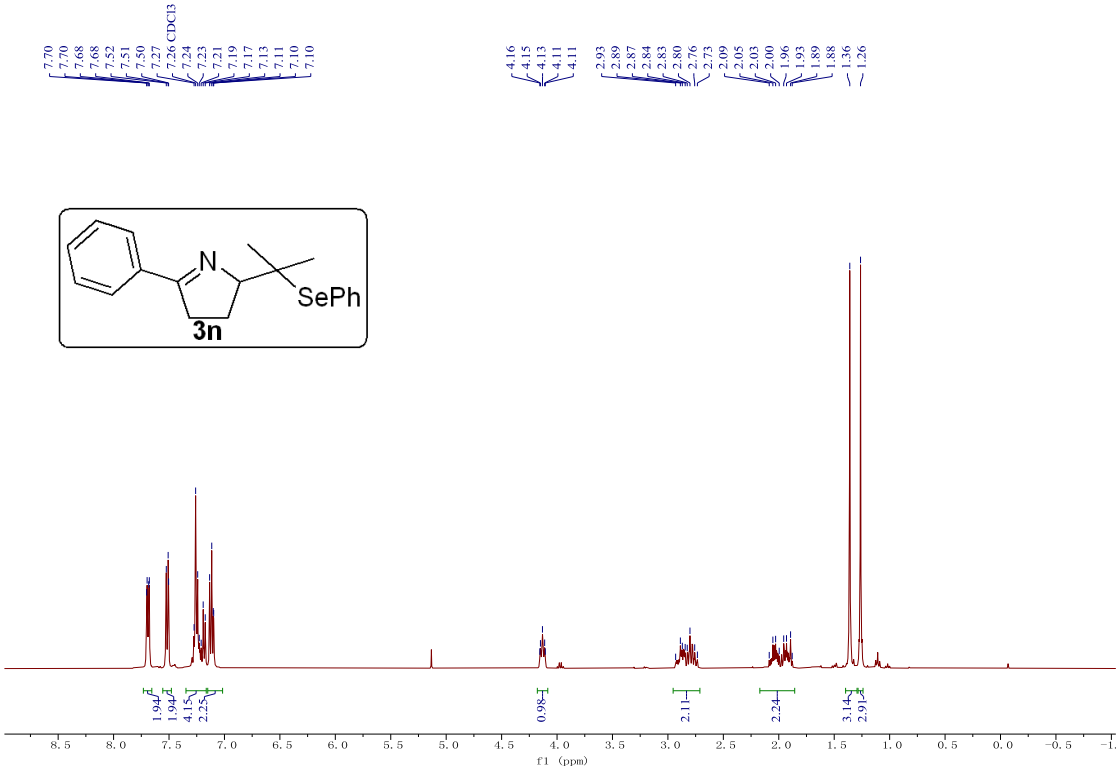
SePh
3m
f1 (ppm)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7.19
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7.13
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7.10
7.10
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4.11
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2.87
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2.83
2.80
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2.73
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1.88
1.36
1.26
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4.15
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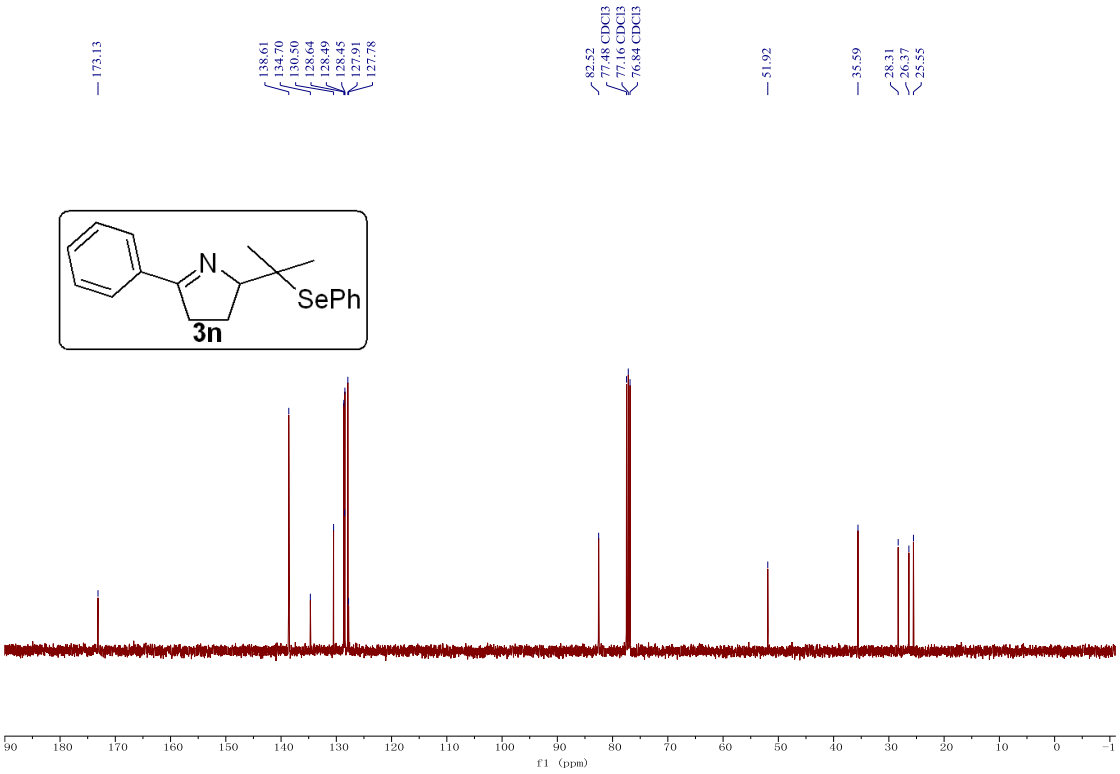
2.24
3.14
2.91
f1 (ppm)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25.55
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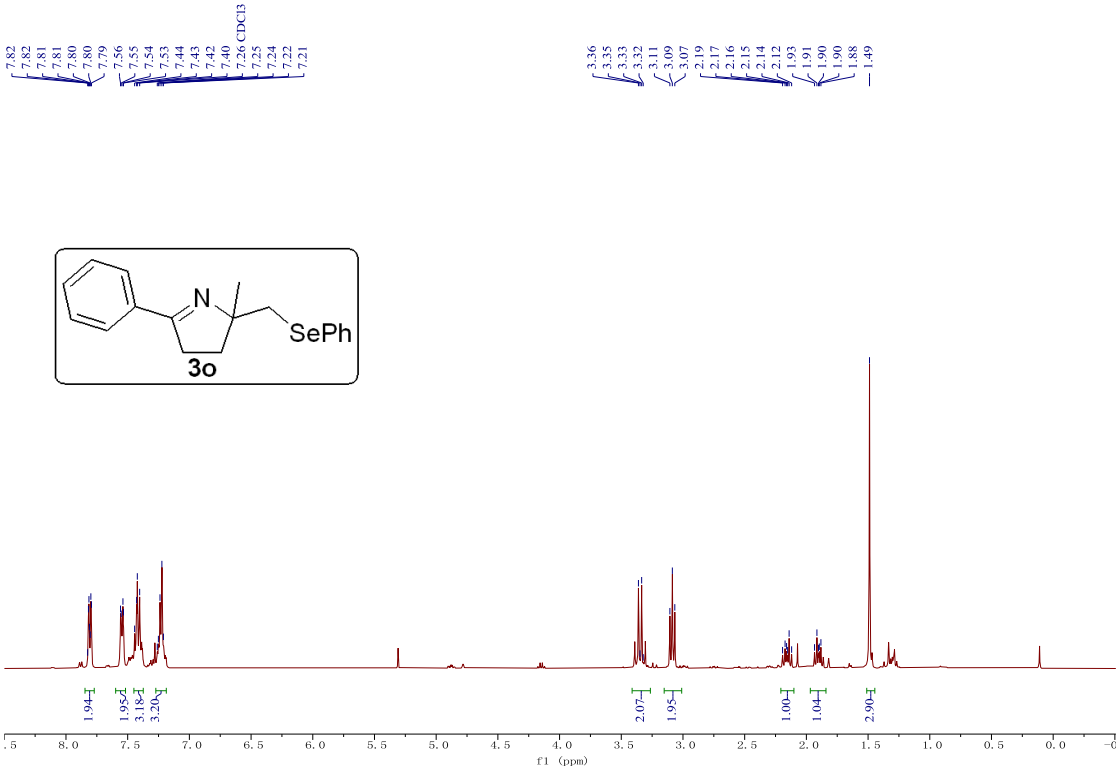
SePh
3n
f1 (ppm)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7.26 CDCl3
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3.32
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2.17
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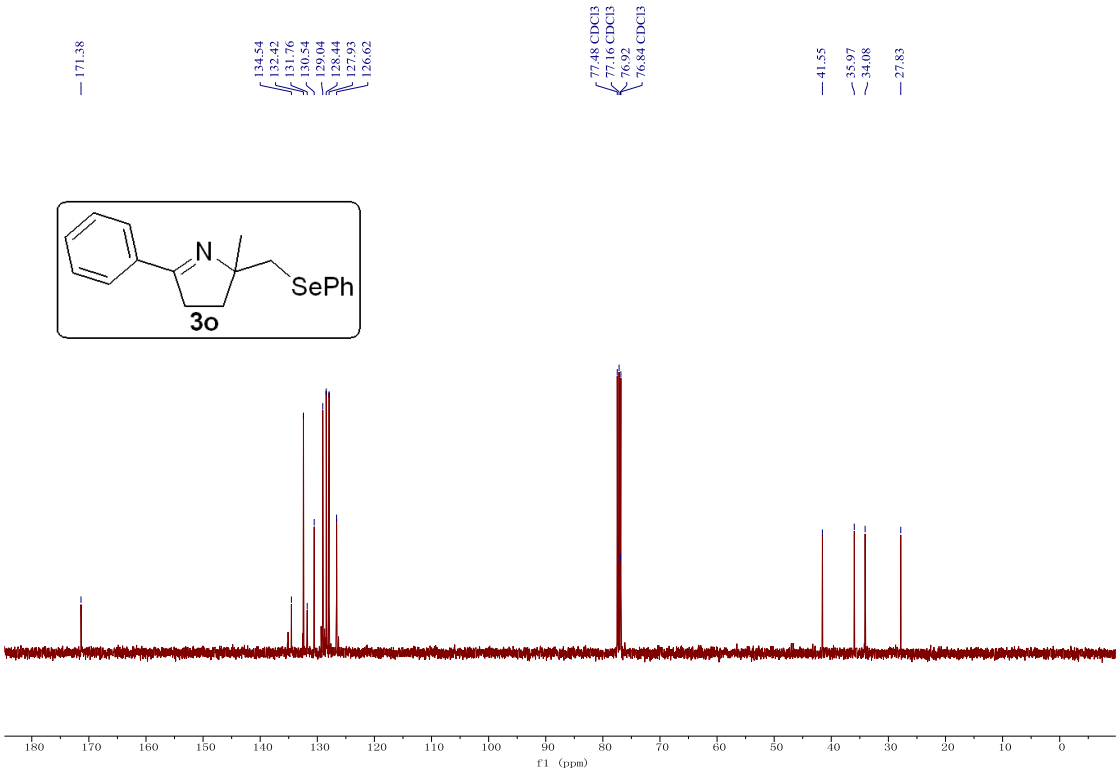
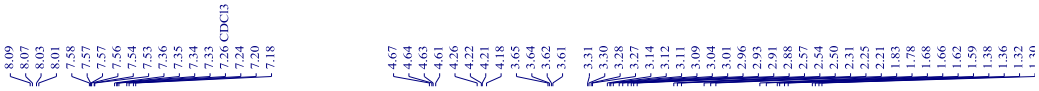
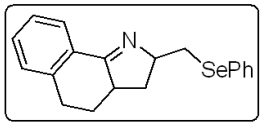
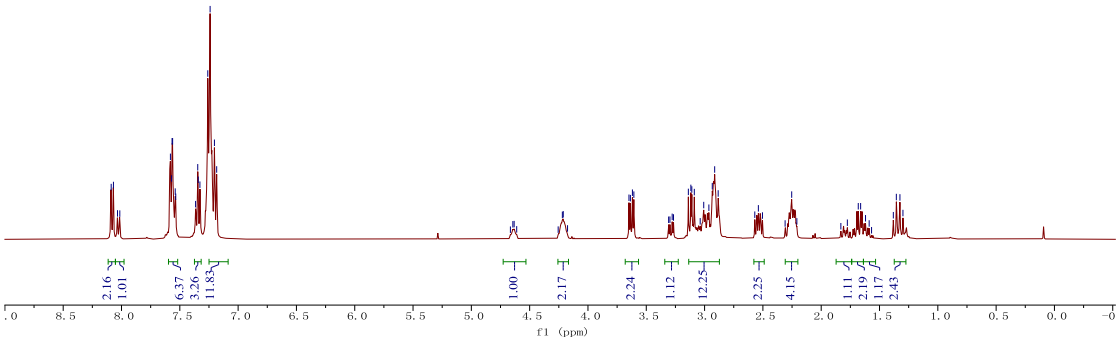
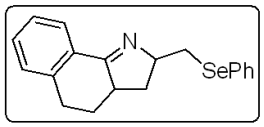




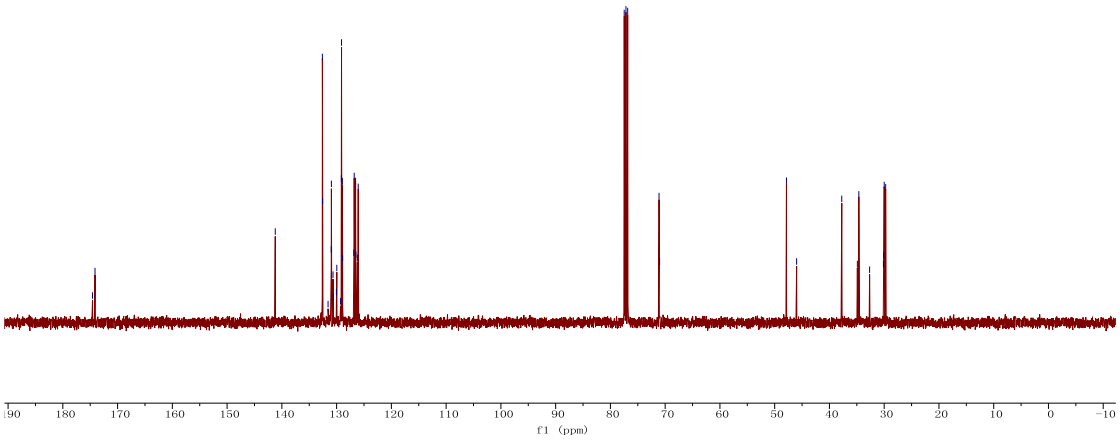


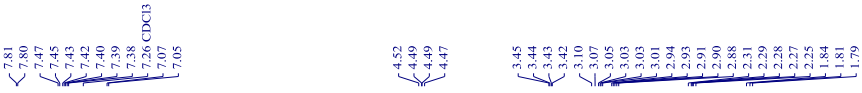
3p ^{1}HNMR ($CDCl_3$, 600MHz, 2.2:1 ratio of diastereoisomers)

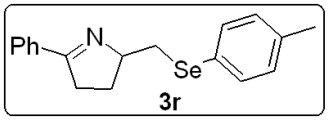


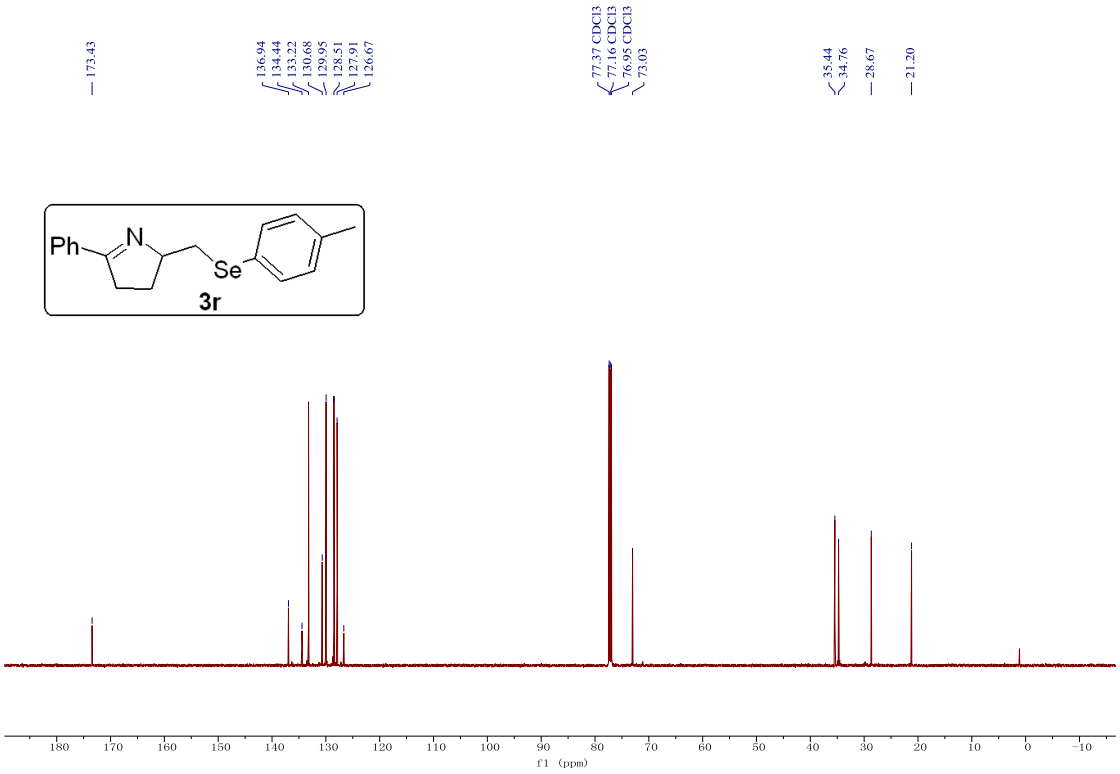


3p ^{1}HNMR ($CDCl_3$, 600MHz, 2.2:1 ratio of diastereoisomers)





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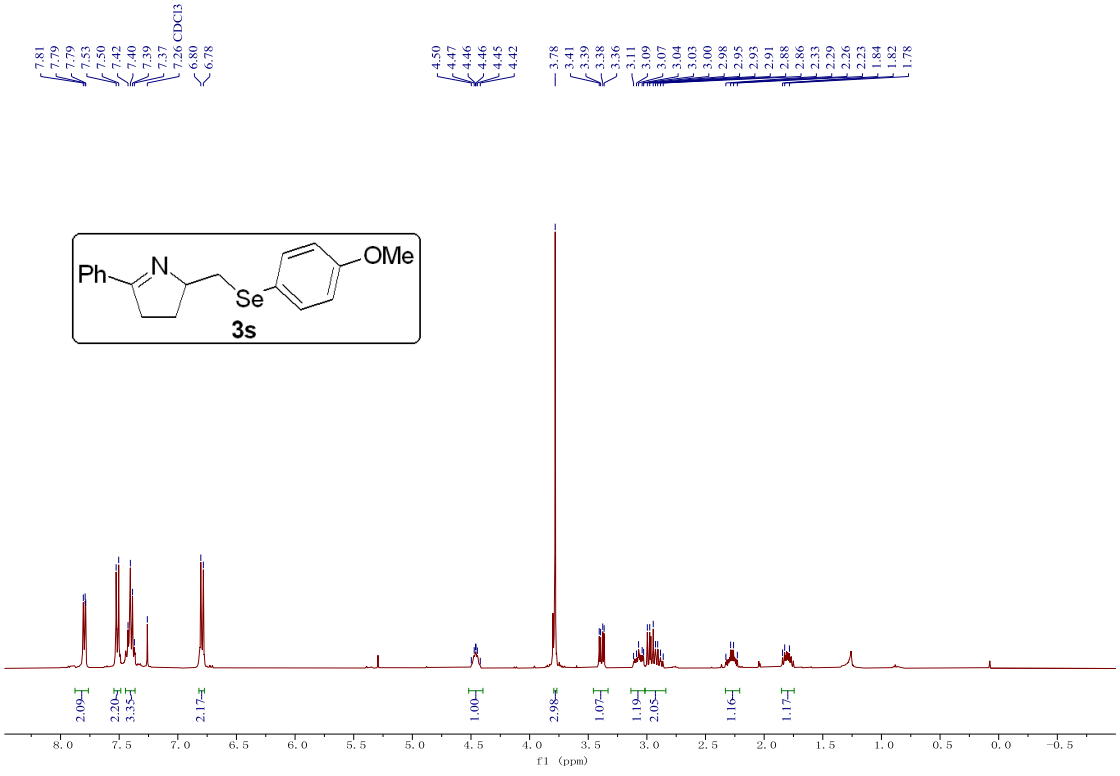
136.94
134.44
133.22
130.68
129.95
128.51
127.91
126.67
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
73.03
35.44
34.76
28.67
21.20
Ph
N
Se
3r
f1 (ppm)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7.79
7.79
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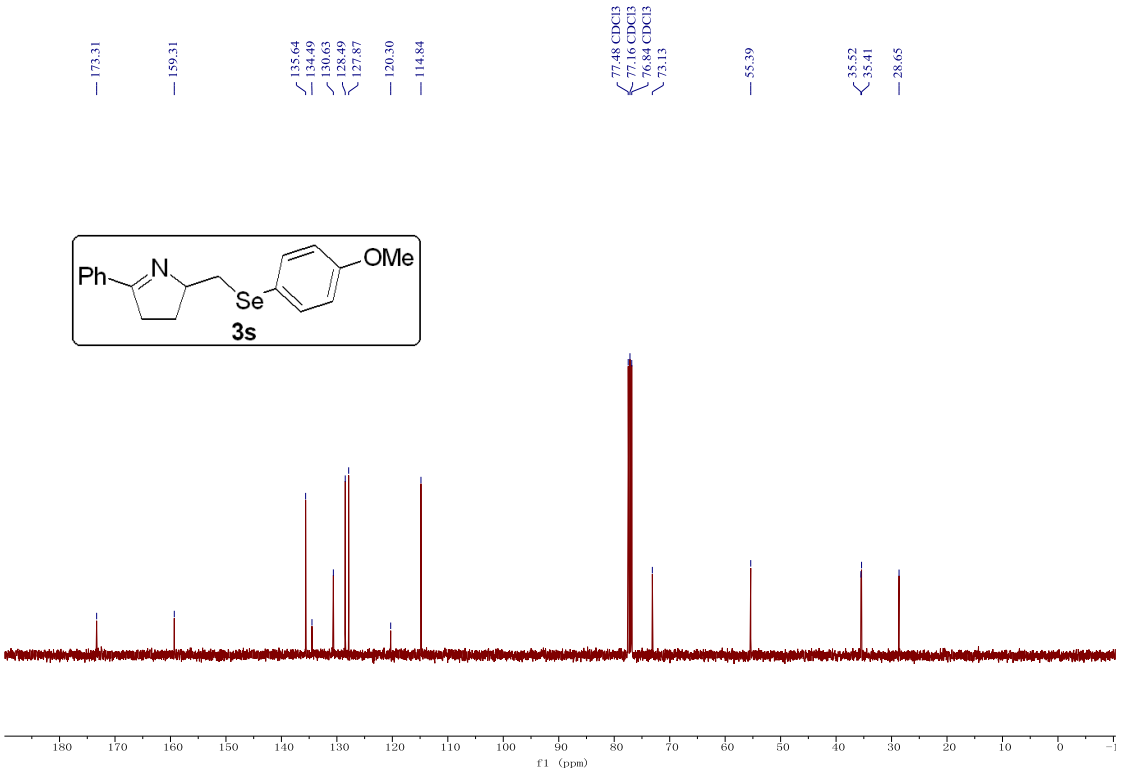
7.42
7.40
7.39
7.37
7.26 CDCl3
6.80
6.78
4.50
4.47
4.46
4.46
4.45
4.42
3.78
3.41
3.39
3.38
3.36
3.11
3.09
3.07
3.04
3.03
3.00
2.98
2.95
2.93
2.91
2.88
2.86
2.33
2.29
2.26
2.23
1.84
1.82
1.78
Ph
N
Se
OMe
3s
2.09
2.20
3.35
2.17
1.00
2.98
1.07
1.19
2.05
1.16
1.17
f1 (ppm)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159.31
135.64
134.49
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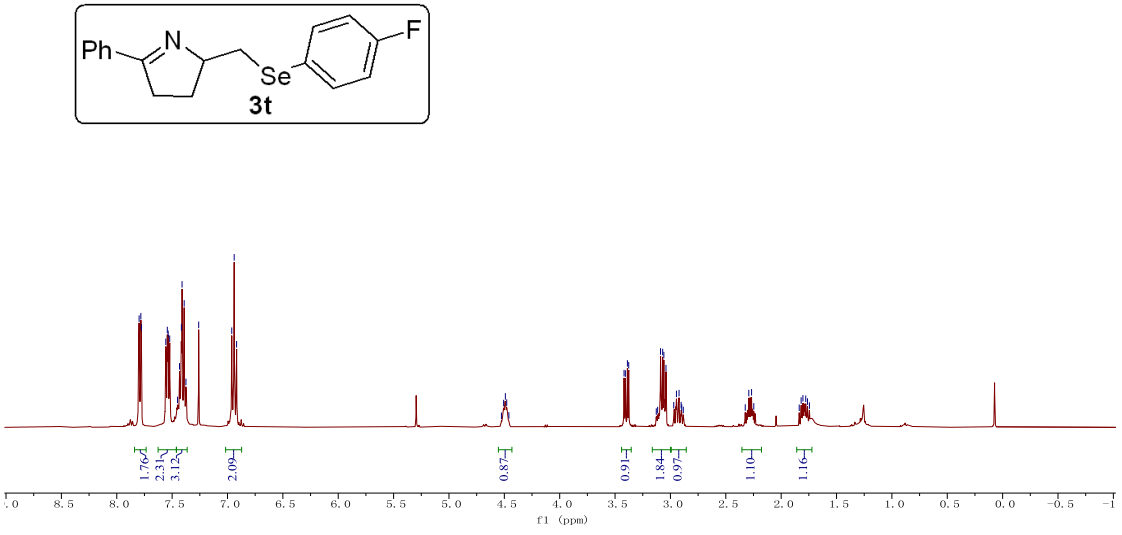
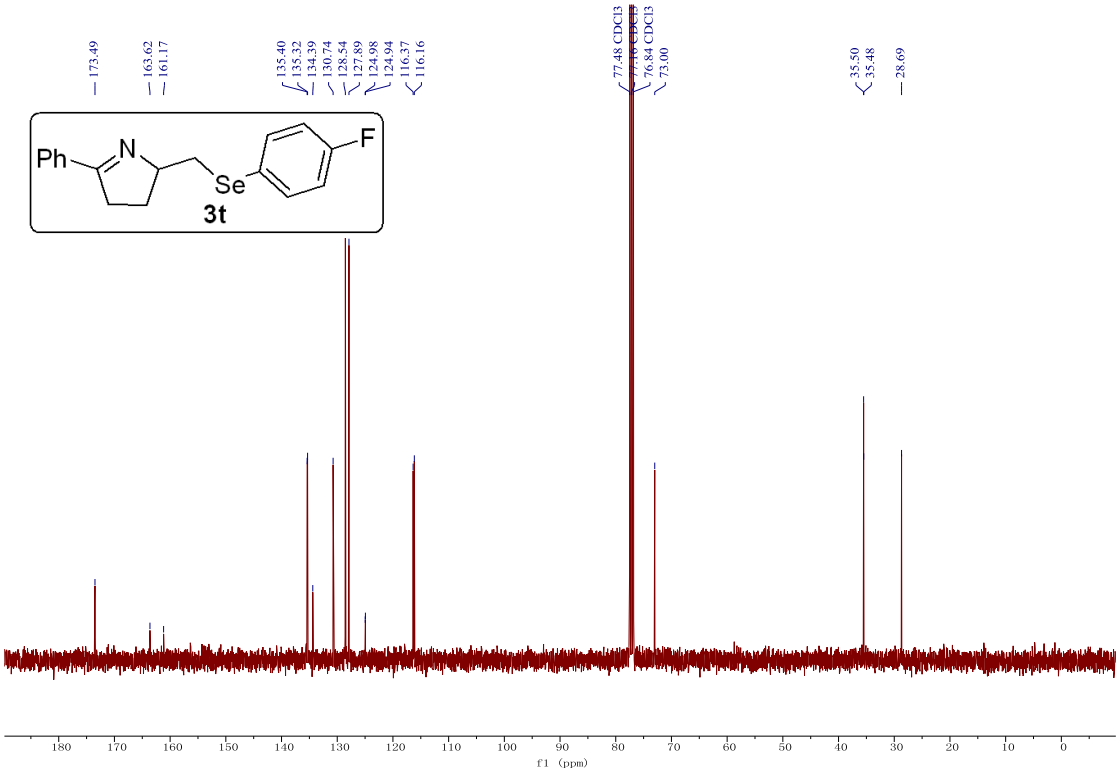
128.49
127.87
120.30
114.84
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
73.13
55.39
35.52
35.41
28.65
Ph
N
Se
OMe
3s
f1 (ppm)


7.80
7.78
7.78
7.56
7.54
7.54
7.52
7.43
7.42
7.41
7.39
7.38
7.26 CDCl3
6.94
6.92
4.53
4.51
4.49
4.48
4.46
3.42
3.41
3.39
3.38
3.13
3.12
3.09
3.07
3.06
3.04
2.97
2.95
2.92
2.90
2.90
2.88
2.32
2.29
2.27
2.25
1.84
1.82
1.80
1.78
1.76
1.74


Ph
N
Se
F
3t
1.76
2.31
3.12
2.09
0.87
0.91
1.84
0.97
1.10
1.16
f1 (ppm)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163.62
161.17
135.40
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134.39
130.74
128.54
127.89
124.98
124.94
116.37
116.16
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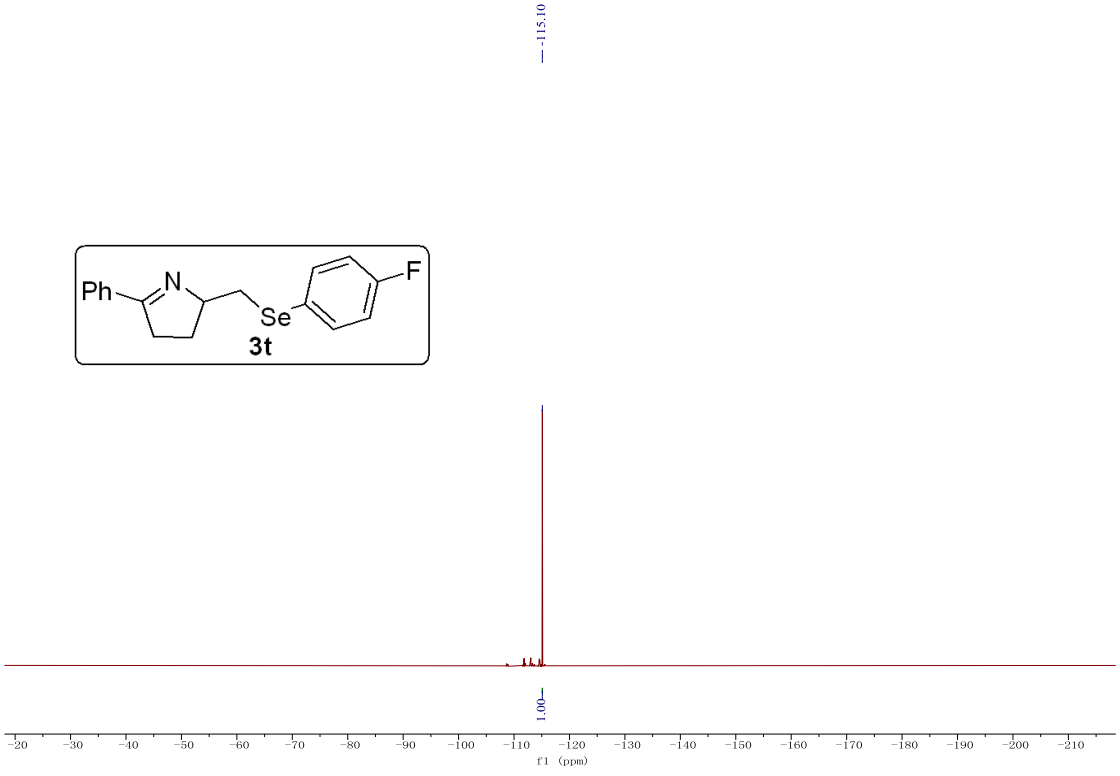
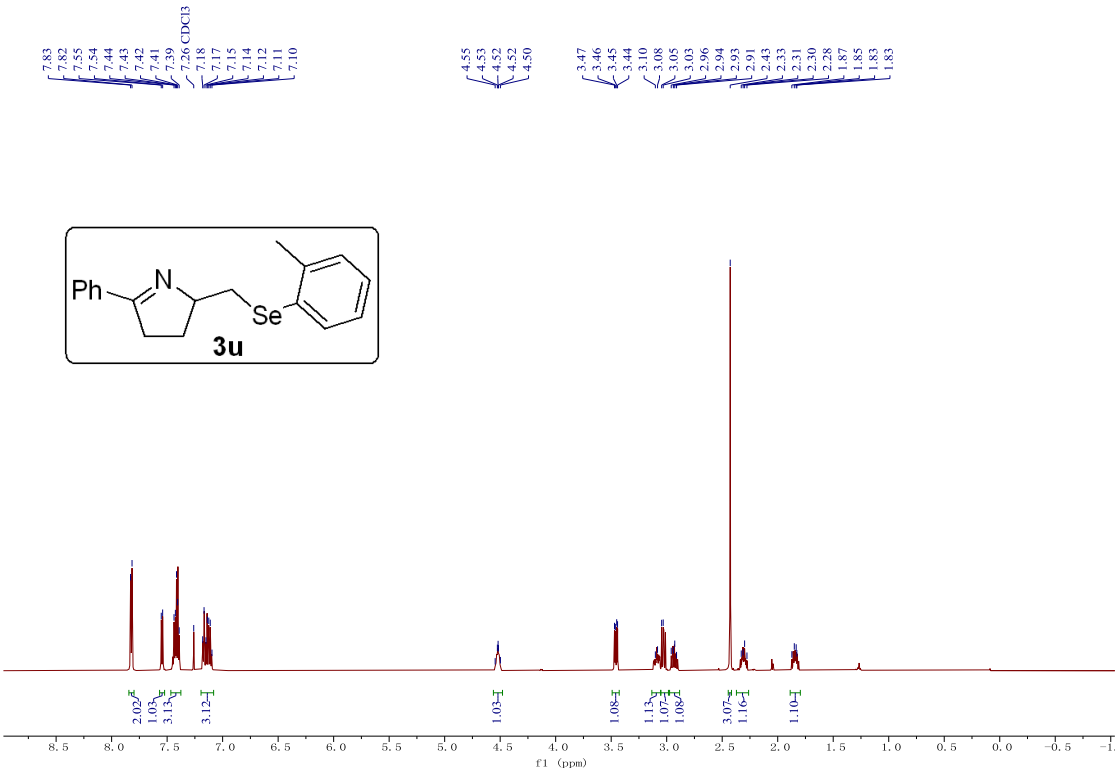
76.84 CDCl3
73.00
35.50
35.48
28.69
Ph
N
Se
F
3t
f1 (ppm)


-115.10
Ph
N
Se
F
3t
1.00
f1 (ppm)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7.82
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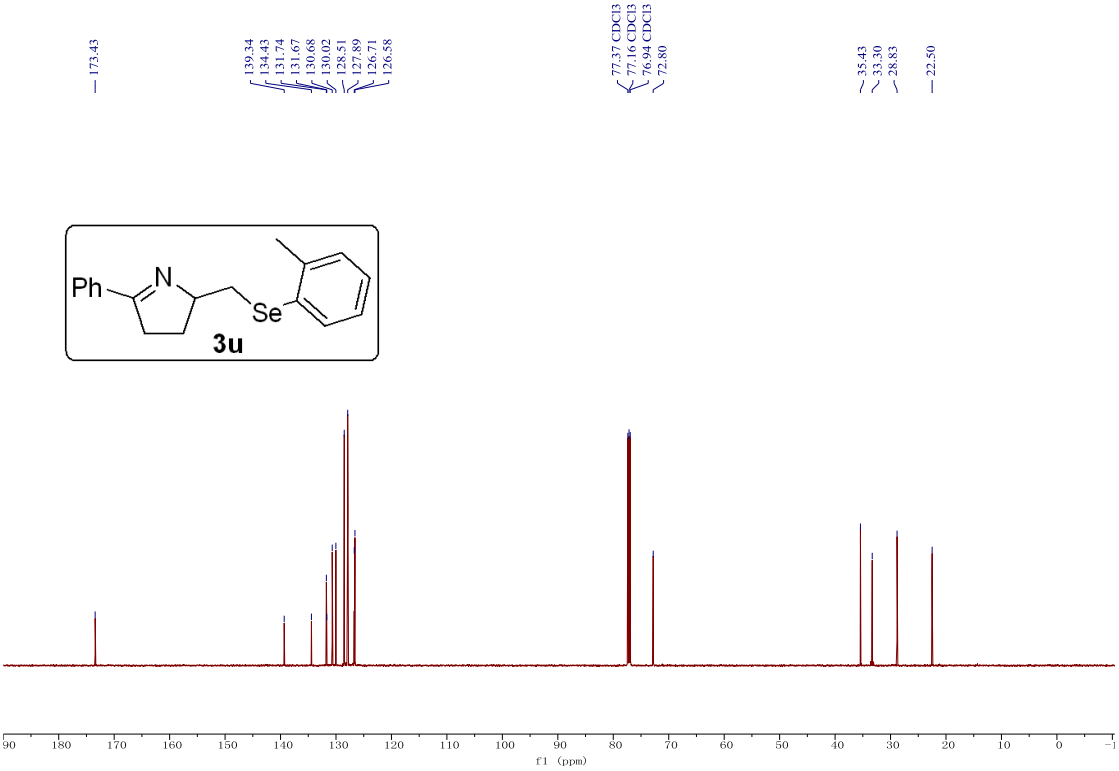
7.54
7.44
7.43
7.42
7.41
7.39
7.26 CDCl3
7.18
7.17
7.15
7.14
7.12
7.11
7.10
4.55
4.53
4.52
4.52
4.50
3.47
3.46
3.45
3.44
3.10
3.08
3.05
3.03
2.96
2.94
2.93
2.91
2.43
2.33
2.31
2.30
2.28
1.87
1.85
1.83
1.83
Ph
N
Se
3u
2.02
1.03
3.13
3.12
1.03
1.08
1.13
1.07
1.08
3.07
1.16
1.10
f1 (ppm)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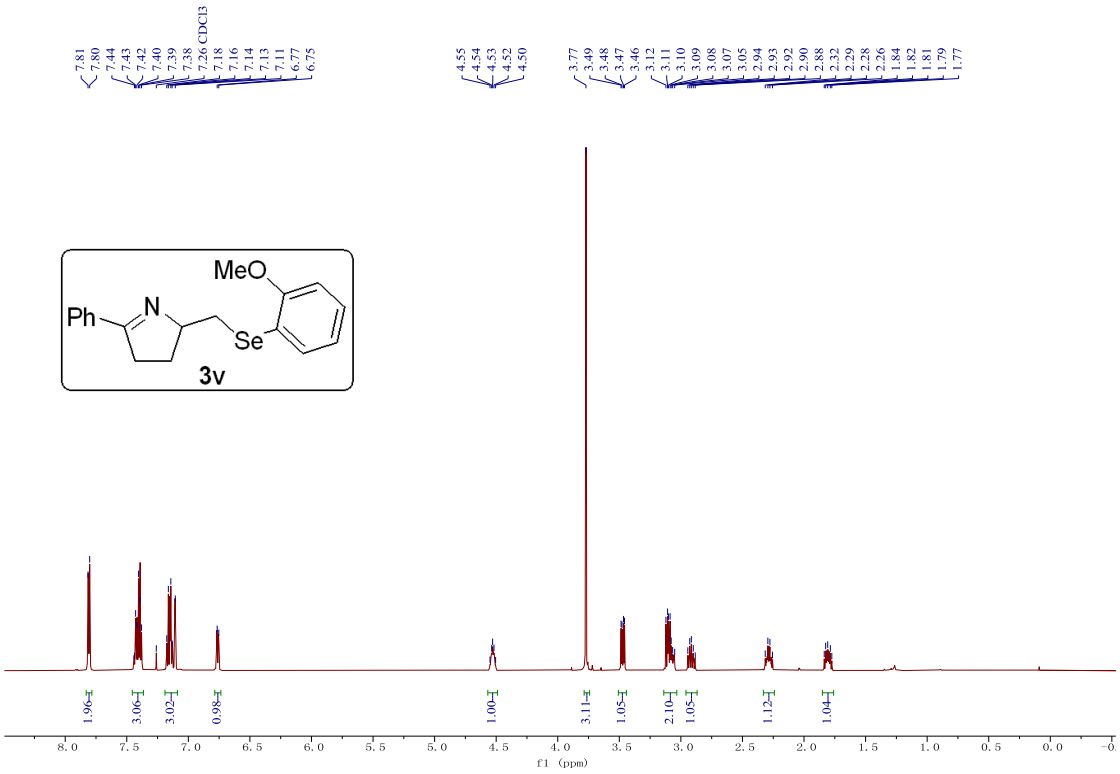
139.34
134.43
131.74
131.67
130.68
130.02
128.51
127.89
126.71
126.58
77.37 CDCl3
77.16 CDCl3
76.94 CDCl3
72.80
35.43
33.30
28.83
22.50
Ph
N
Se
3u
f1 (ppm)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7.80
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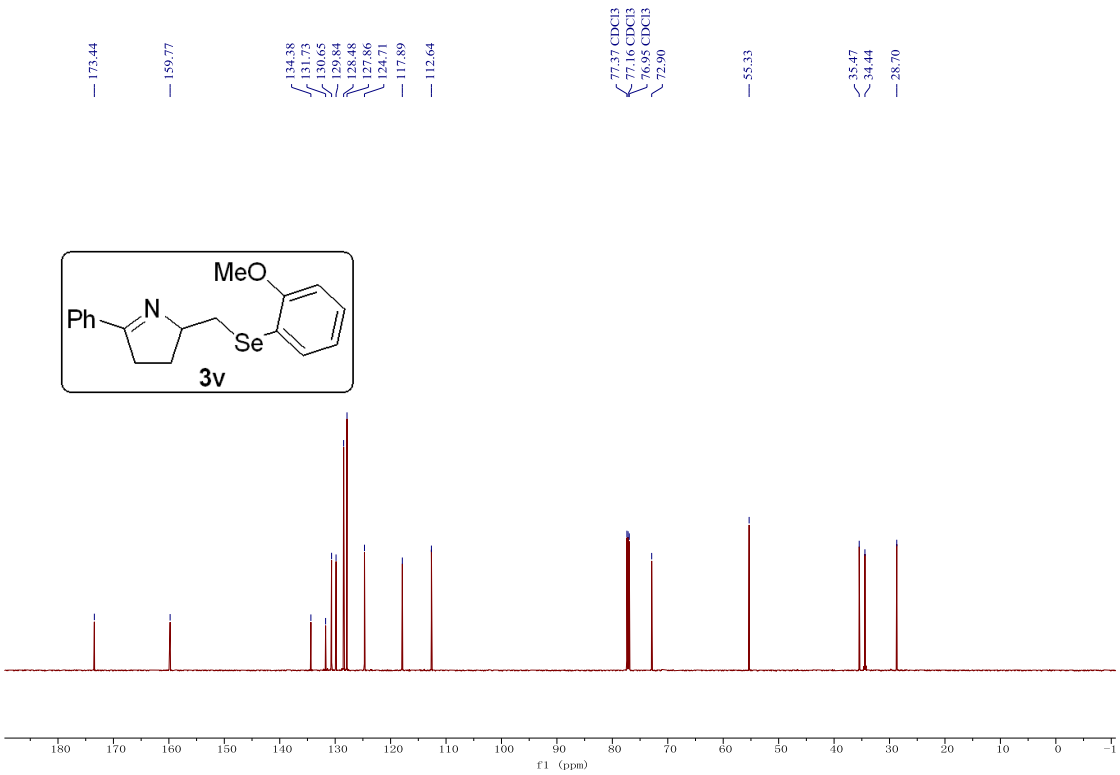
7.43
7.42
7.40
7.39
7.38
7.26 CDCl3
7.18
7.16
7.14
7.13
7.11
6.77
6.75
4.55
4.54
4.53
4.52
4.50
3.77
3.49
3.48
3.47
3.46
3.12
3.11
3.10
3.09
3.08
3.07
3.05
2.94
2.93
2.92
2.90
2.88
2.32
2.29
2.28
2.26
1.84
1.82
1.81
1.79
1.77
MeO
Ph
N
Se
3v
1.96
3.06
3.02
0.98
1.00
3.11
1.05
2.10
1.05
1.12
1.04
f1 (ppm)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127.86
124.71
117.89
112.64
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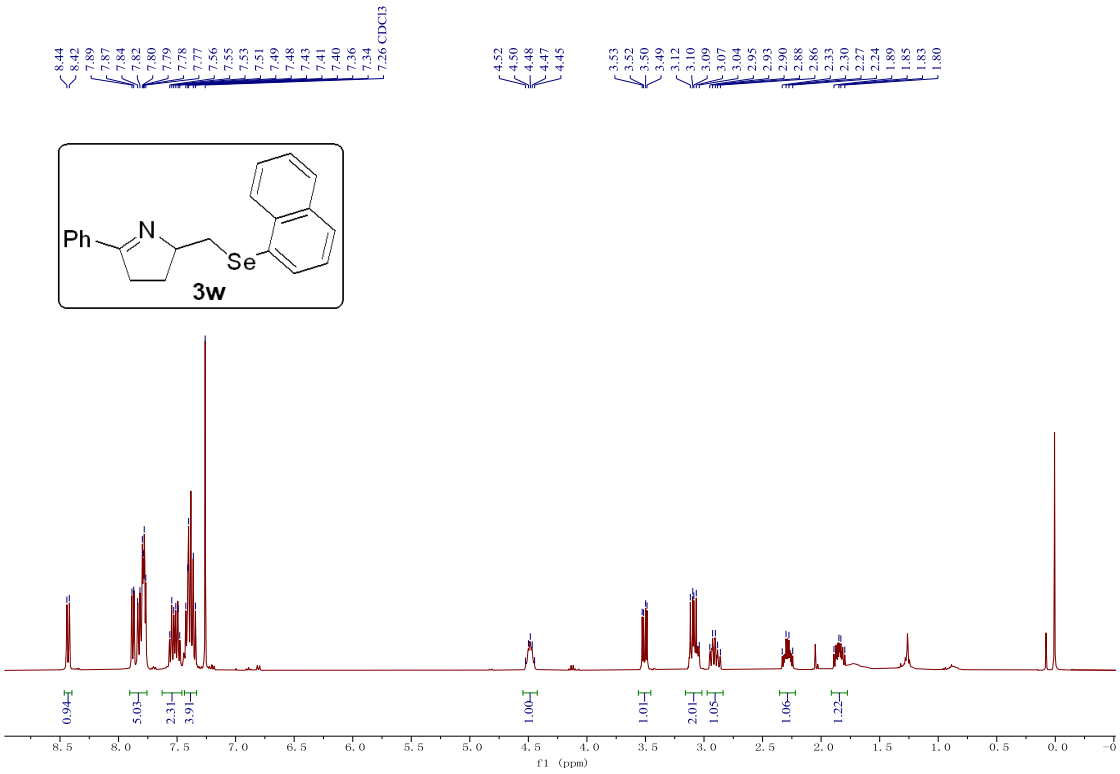
77.16 CDCl3
76.95 CDCl3
72.90
55.33
35.47
34.44
28.70
MeO
Ph
N
Se
3v
f1 (ppm)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7.82
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7.78
7.77
7.56
7.55
7.53
7.51
7.49
7.48
7.43
7.41
7.40
7.36
7.34
7.26 CDCl3
4.52
4.50
4.48
4.47
4.45
3.53
3.52
3.50
3.49
3.12
3.10
3.09
3.07
3.04
2.95
2.93
2.90
2.88
2.86
2.33
2.30
2.27
2.24
1.89
1.85
1.83
1.80
Ph
N
Se
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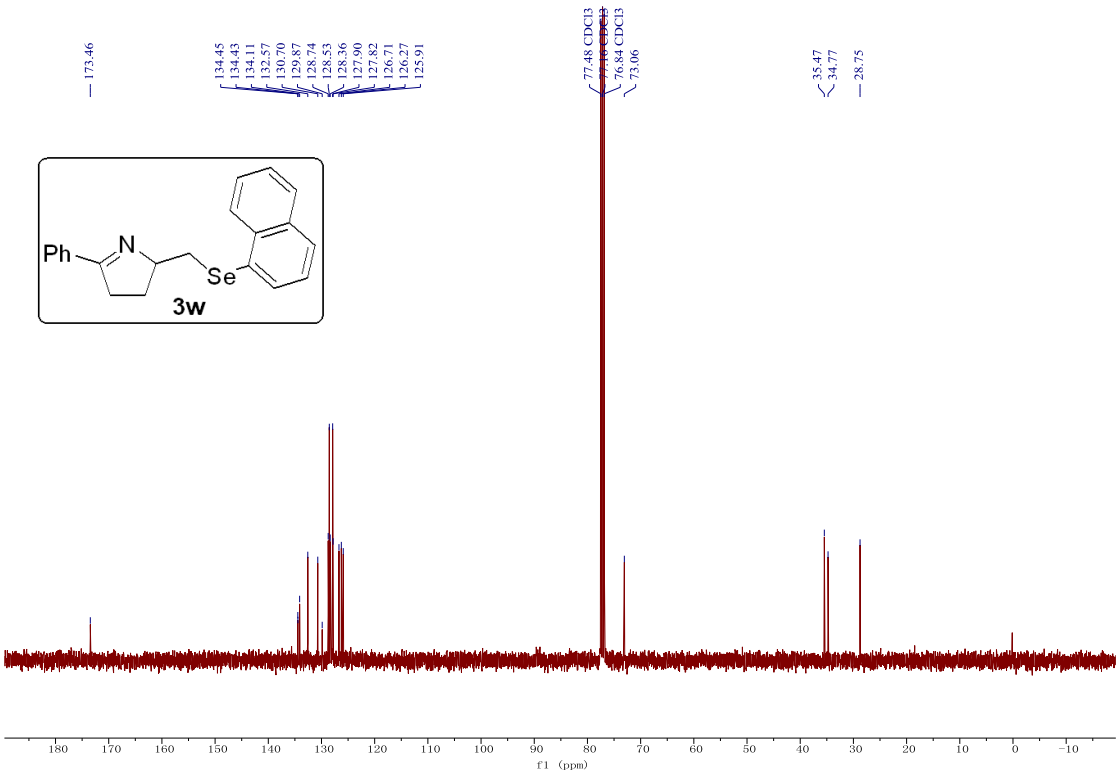
2.31
3.91
1.00
1.01
2.01
1.05
1.06
1.22
f1 (ppm)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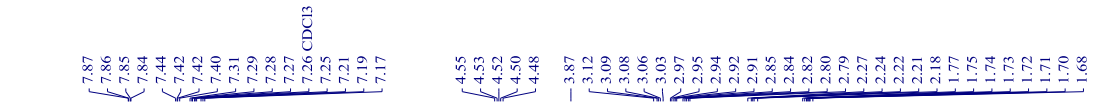
134.43
134.11
132.57
130.70
129.87
128.74
128.53
128.36
127.90
127.82
126.71
126.27
125.91
77.48 CDCl3
76.84 CDCl3
73.06
35.47
34.77
28.75
Ph
N
Se
3w
f1 (ppm)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7.86
7.85
7.84
7.44
7.42
7.42
7.40
7.31
7.29
7.28
7.27
7.26 CDCl3
7.25
7.21
7.19
7.17
4.55
4.53
4.52
4.50
4.48
3.87
3.12
3.09
3.08
3.06
3.03
2.97
2.95
2.94
2.92
2.91
2.85
2.84
2.82
2.80
2.79
2.27
2.24
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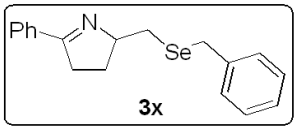
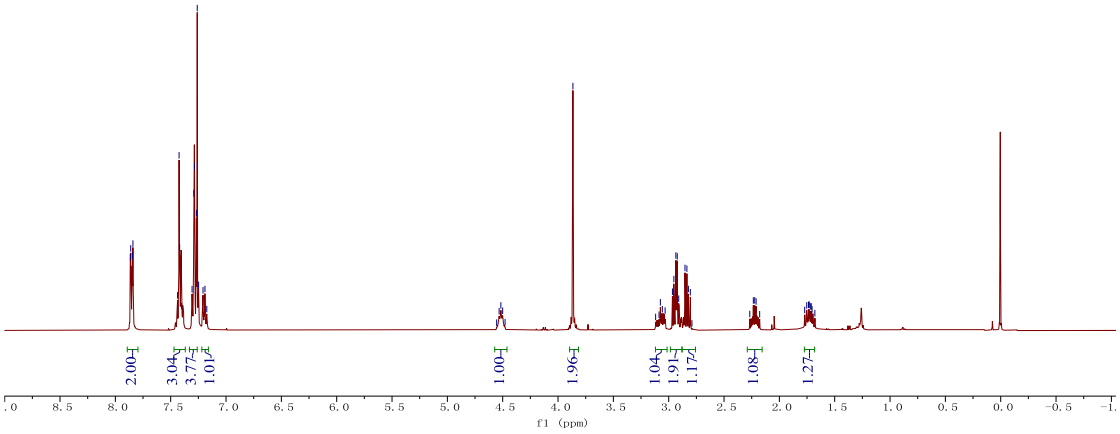
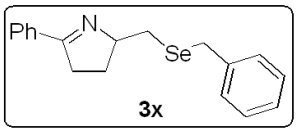
1.75
1.74
1.73
1.72
1.71
1.70
1.68


Ph
N
Se

3x

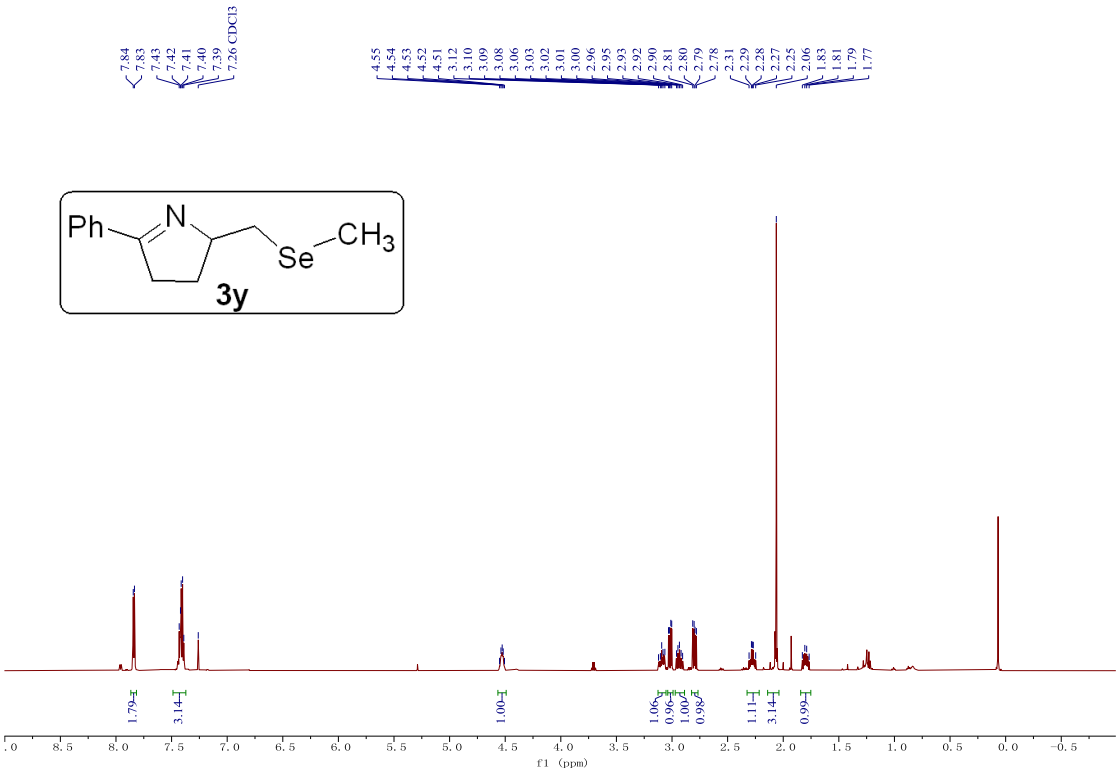

2.00
3.04
3.77
1.01
1.00
1.96
1.04
1.91
1.17
1.08
1.27
f1 (ppm)


173.22
139.76
134.53
130.66
129.15
129.06
128.59
127.89
126.75
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
73.54
35.53
30.73
28.80
28.12


Ph
N
Se

3x

f1 (ppm)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7.39
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4.52
4.51
3.12
3.10
3.09
3.08
3.06
3.03
3.02
3.01
3.00
2.96
2.95
2.93
2.92
2.90
2.81
2.80
2.79
2.78
2.31
2.29
2.28
2.27
2.25
2.06
1.83
1.81
1.79
1.77
Ph
N
Se
CH3
3y
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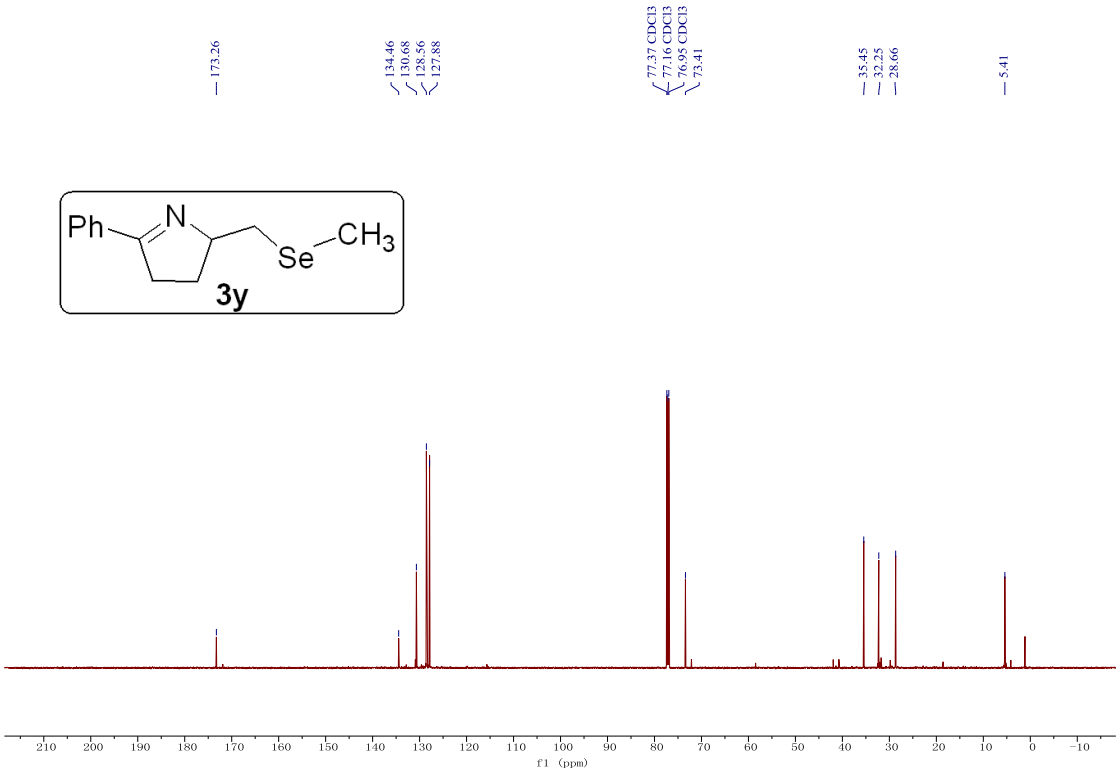
1.00
0.98
1.11
3.14
0.99
f1 (ppm)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128.56
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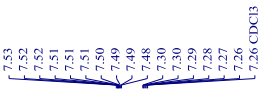
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
73.41
35.45
32.25
28.66
5.41
Ph
N
Se
CH3
3y
f1 (ppm)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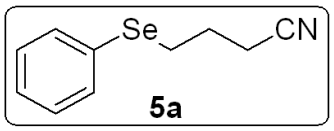
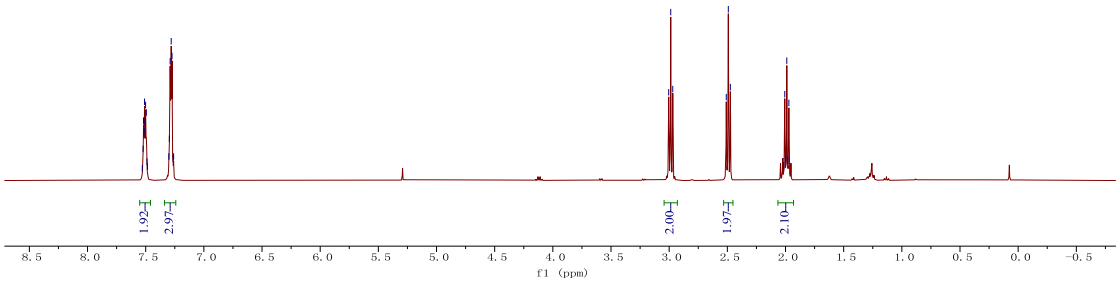
7.52
7.51
7.51
7.51
7.50
7.49
7.49
7.48
7.30
7.30
7.29
7.28
7.27
7.26
7.26 CDCl3


3.00
2.99
2.97
2.51
2.49
2.47
2.01
2.01
2.00
1.99
1.98
1.97


Se
CN
5a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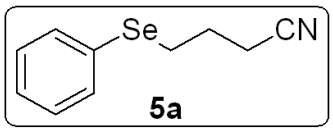
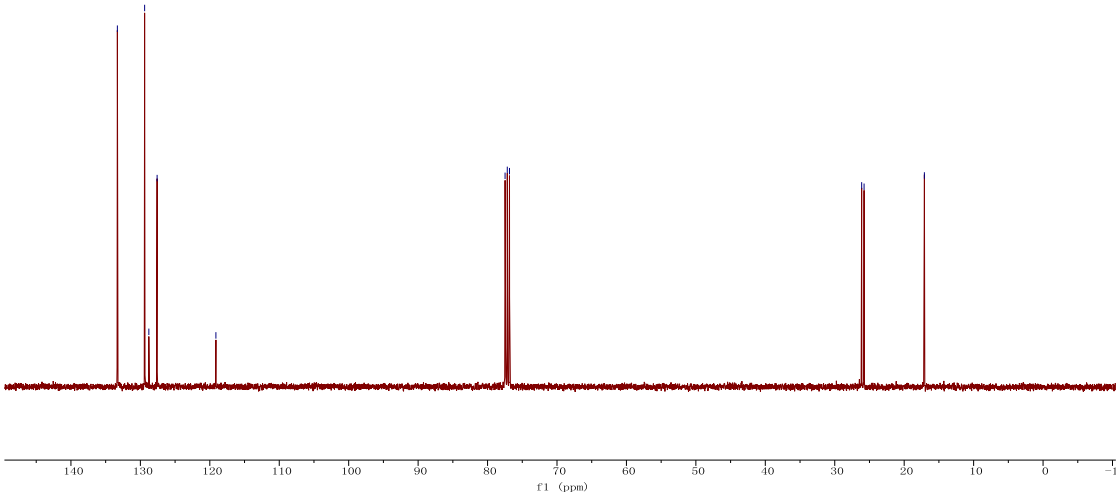
2.97
2.00
1.97
2.10
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

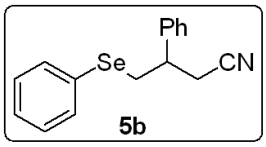

133.31
129.39
128.78
127.59
119.12
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
26.12
25.78
17.09

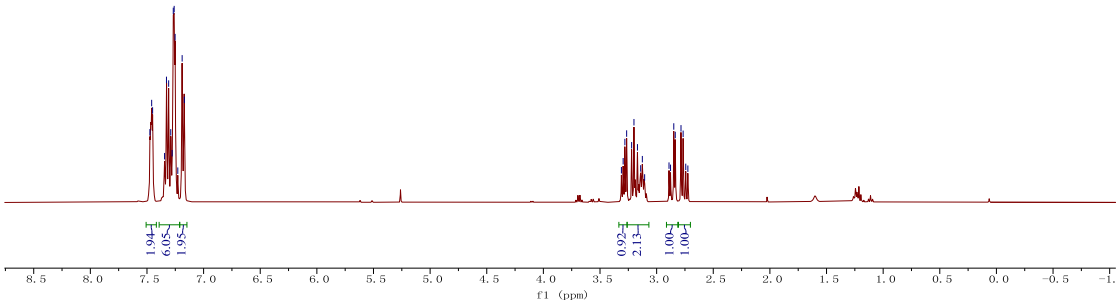

Se
CN
5a


140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−1
f1 (ppm)


7.47
7.46
7.45
7.34
7.33
7.31
7.29
7.28
7.27
7.26 CDCl3
7.25
7.23
7.19
7.17


3.31
3.30
3.28
3.26
3.22
3.20
3.17
3.14
3.13
3.11
2.89
2.88
2.85
2.84
2.78
2.77
2.74
2.72

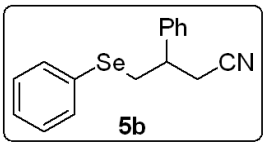

Ph
Se
CN
5b

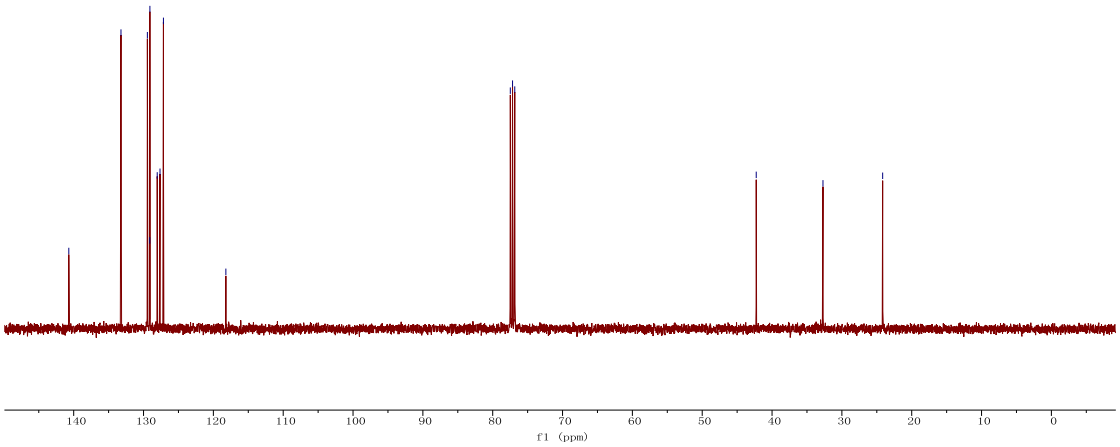

1.94
6.05
1.95
0.92
2.13
1.00
1.00
f1 (ppm)

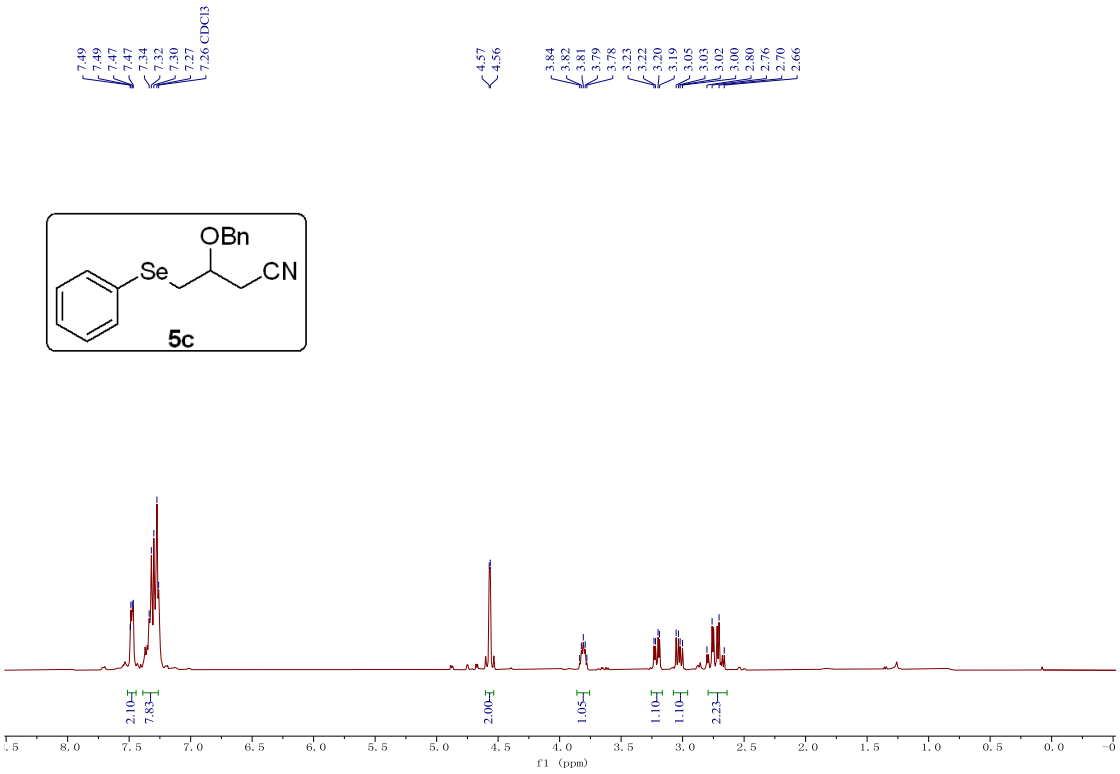

140.70
133.23
129.45
129.11
129.09
128.04
127.64
127.15
118.21


77.48 CDCl3
77.16 CDCl3
76.84 CDCl3


42.27
32.71
24.16


Ph
Se
CN
5b


f1 (ppm)


7.49
7.49
7.47
7.47
7.34
7.32
7.30
7.27
7.26 CDCl3
4.57
4.56
3.84
3.82
3.81
3.79
3.78
3.23
3.22
3.20
3.19
3.05
3.03
3.02
3.00
2.80
2.76
2.70
2.66
OBn
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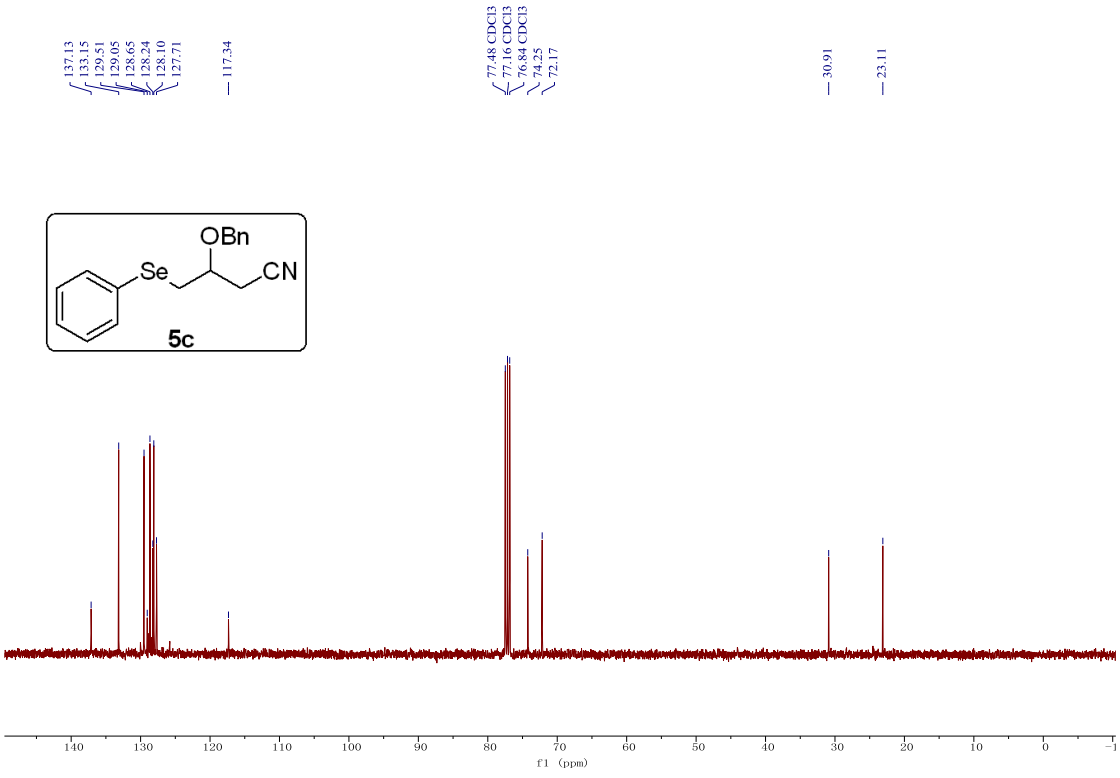
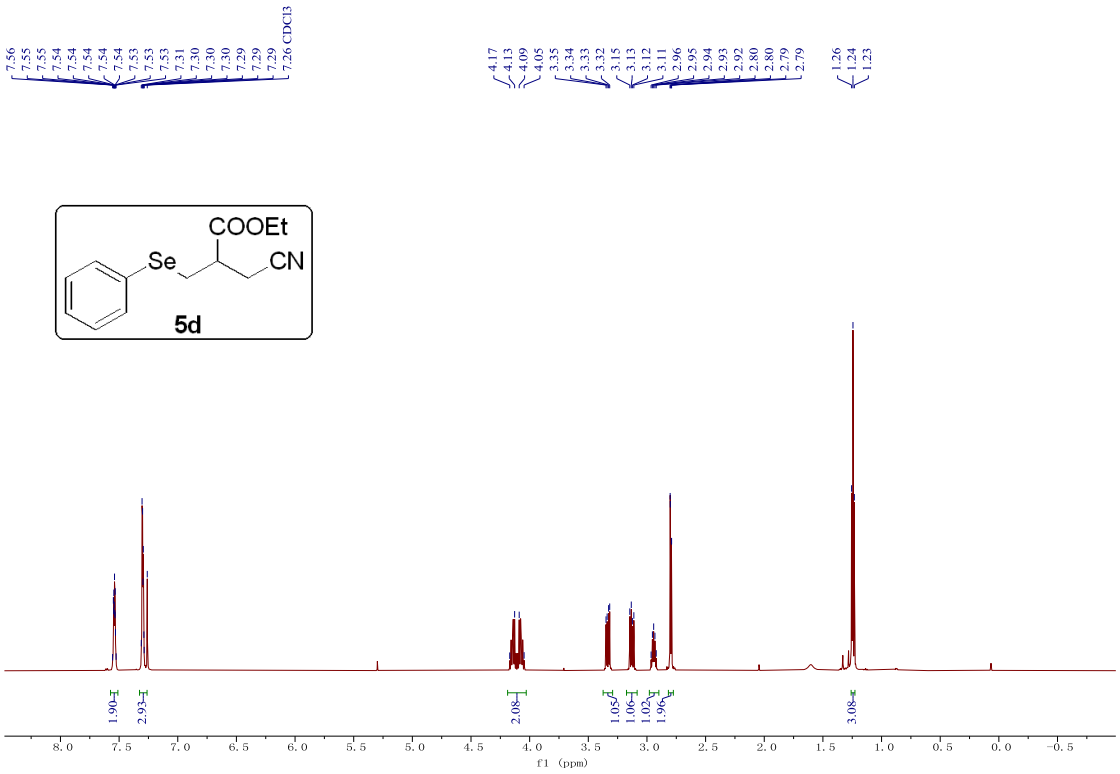
CN
5c
2.10
7.83
2.00
1.05
1.10
1.10
2.23
f1 (ppm)


137.13
133.15
129.51
129.05
128.65
128.24
128.10
127.71
117.34
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
74.25
72.17
30.91
23.11
OBn
Se
CN
5c
f1 (ppm)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7.55
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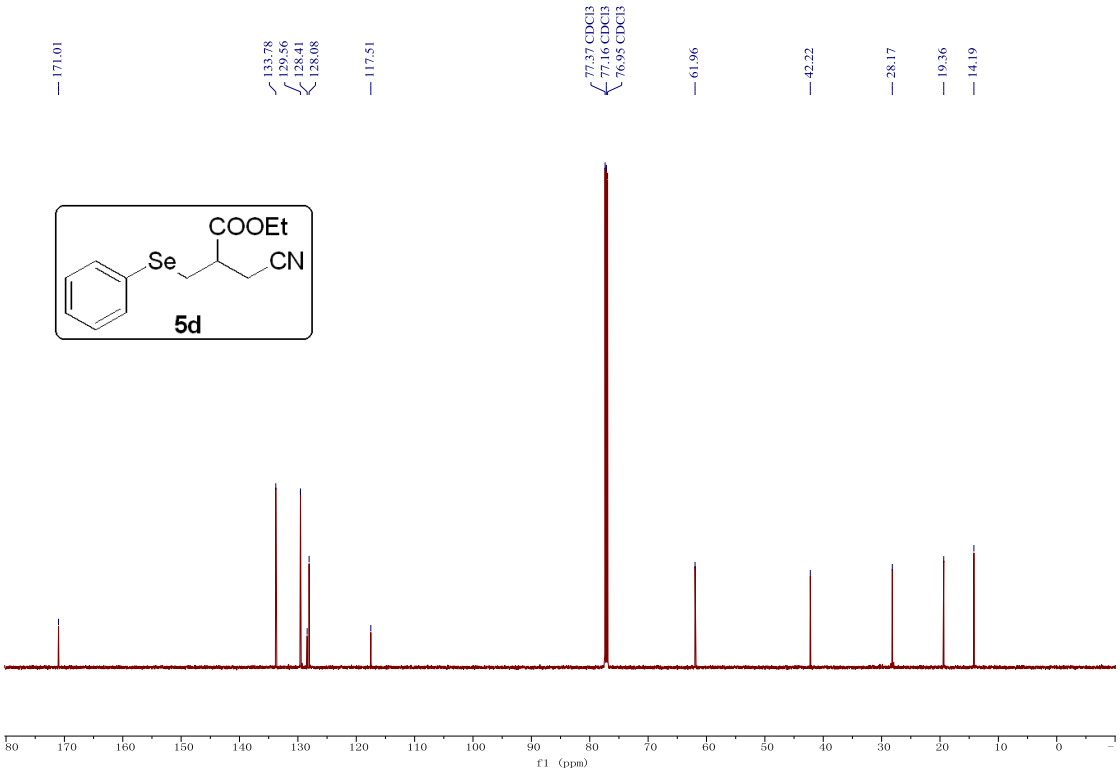
7.54
7.53
7.53
7.53
7.31
7.30
7.30
7.30
7.29
7.29
7.29
7.26 CDCl3
4.17
4.13
4.09
4.05
3.35
3.34
3.33
3.32
3.15
3.13
3.12
3.11
2.96
2.95
2.94
2.93
2.92
2.80
2.80
2.79
2.79
1.26
1.24
1.23
COOEt
Se
CN
5d
1.90
2.93
2.08
1.05
1.06
1.02
1.96
3.08
f1 (ppm)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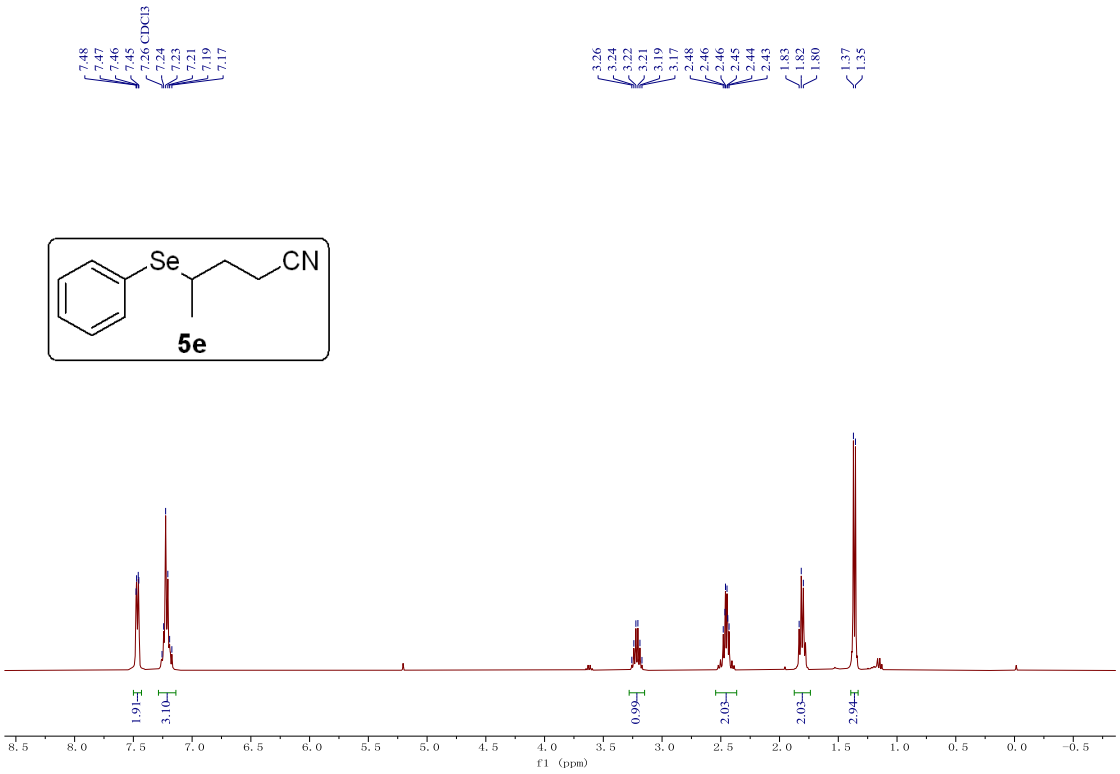
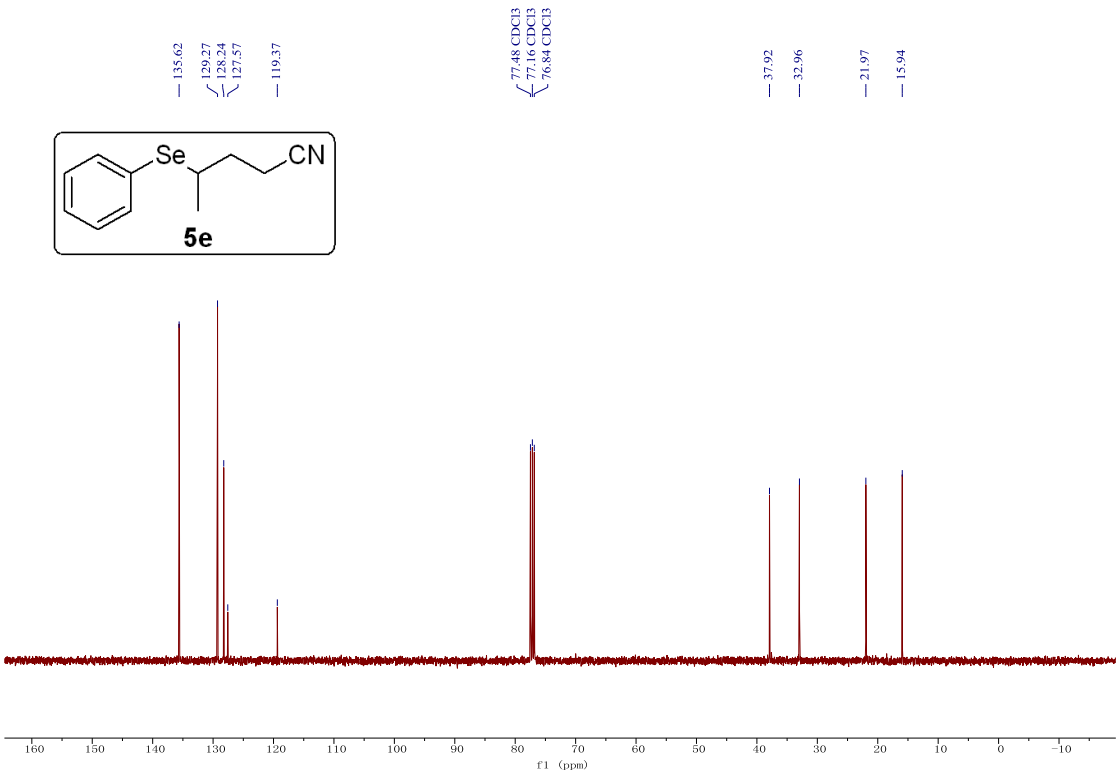
133.78
129.56
128.41
128.08
117.51
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
61.96
42.22
28.17
19.36
14.19
COOEt
Se
CN
5d
f1 (ppm)


7.48
7.47
7.46
7.45
7.26 CDCl3
7.24
7.23
7.21
7.19
7.17
3.26
3.24
3.22
3.21
3.19
3.17
2.48
2.46
2.46
2.45
2.44
2.43
1.83
1.82
1.80
1.37
1.35
Se
CN
5e
1.91
3.10
0.99
2.03
2.03
2.94
f1 (ppm)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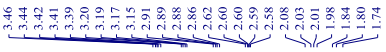
129.27
128.24
127.57
119.37
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
37.92
32.96
21.97
15.94
Se
CN
5e
f1 (ppm)


7.57
7.56
7.55
7.37
7.36
7.35
7.34
7.32
7.29
7.28
7.28
7.26 CDCl3
7.19
7.18


3.46
3.44
3.42
3.41
3.39
3.20
3.19
3.17
3.15
2.91
2.89
2.88
2.86
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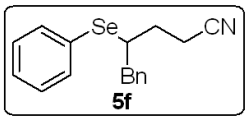
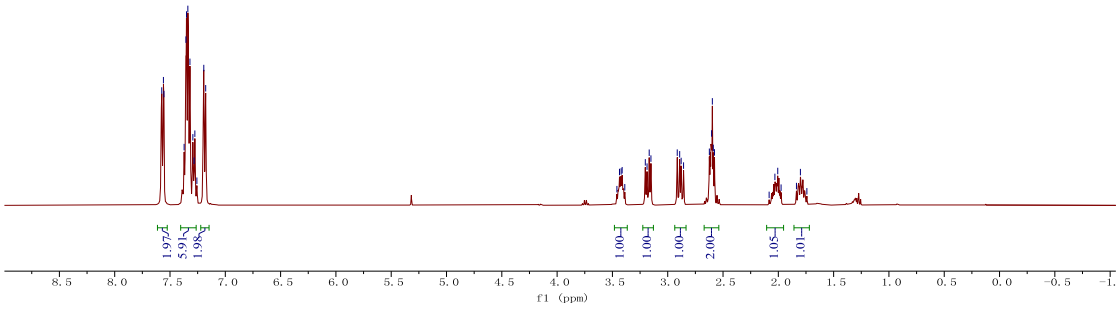
2.03
2.01
1.98
1.84
1.80
1.74

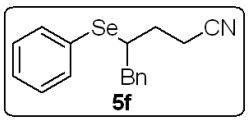

Se
CN
Bn
5f

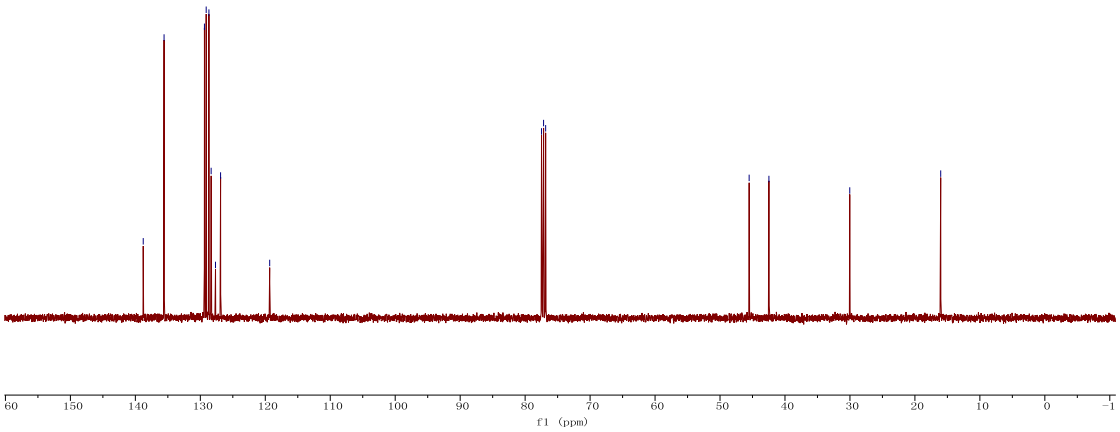

1.97
5.91
1.98
1.00
1.00
1.00
2.00
1.05
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.
f1 (ppm)

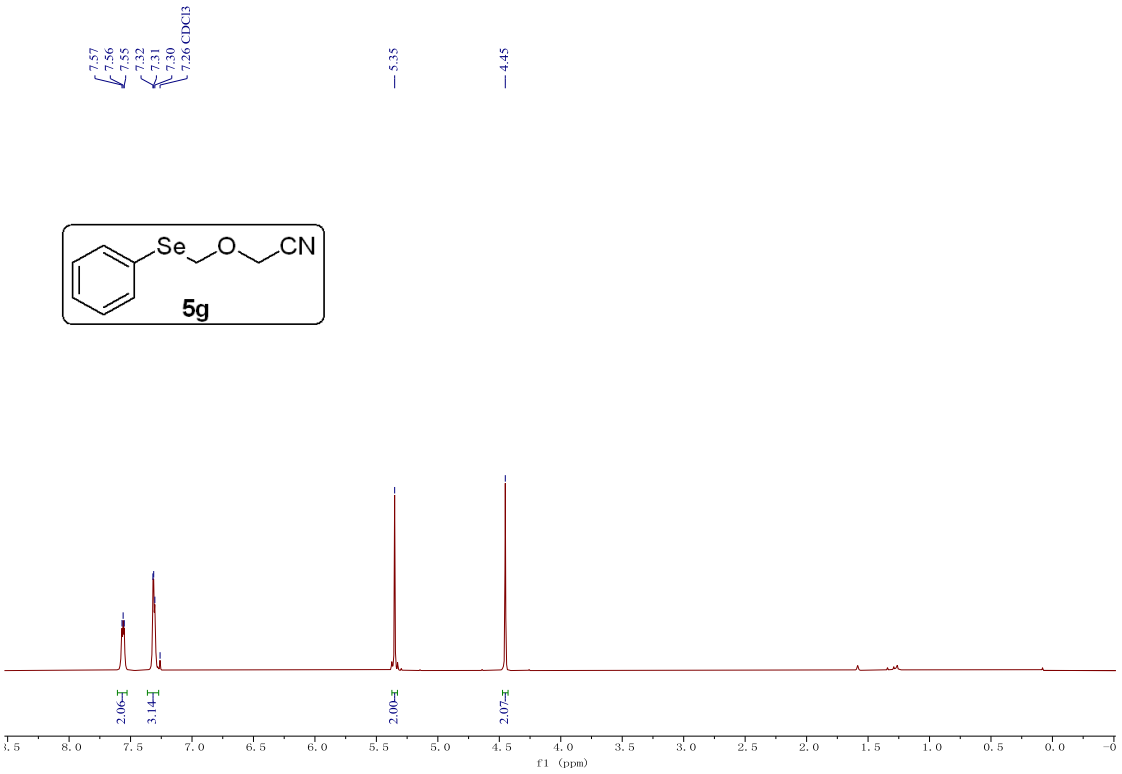

138.79
135.58
129.37
129.10
128.68
128.33
127.67
126.88
119.32

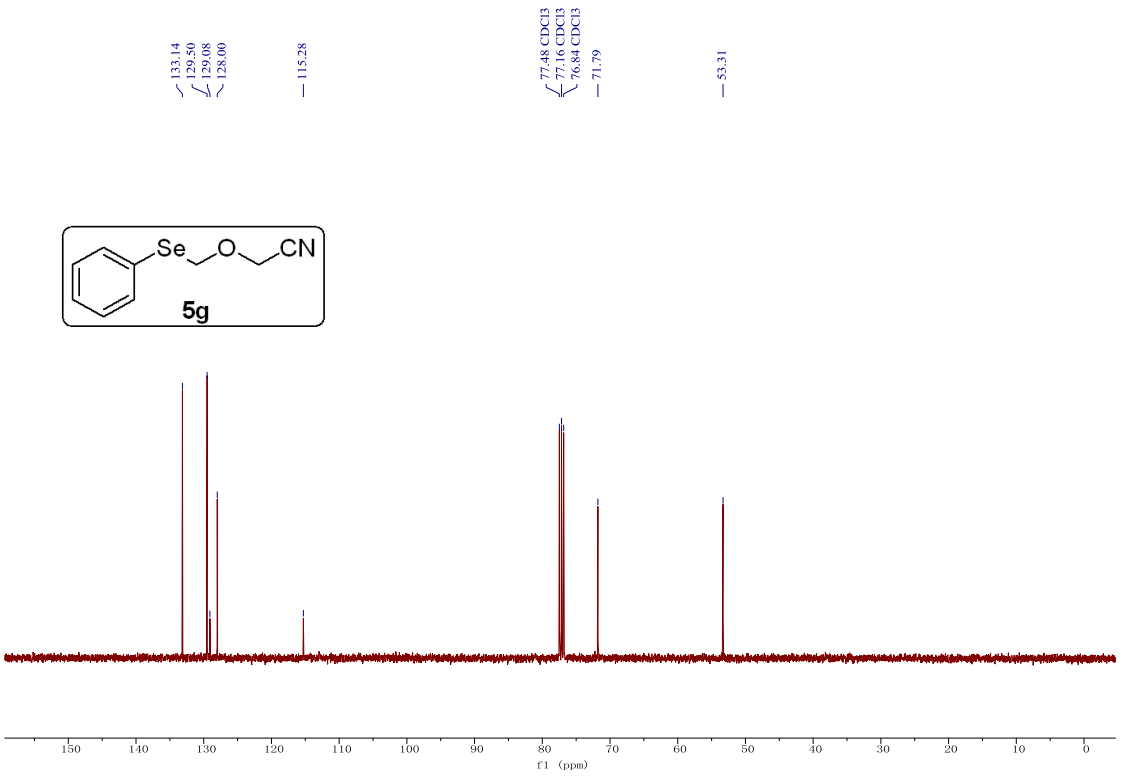
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3

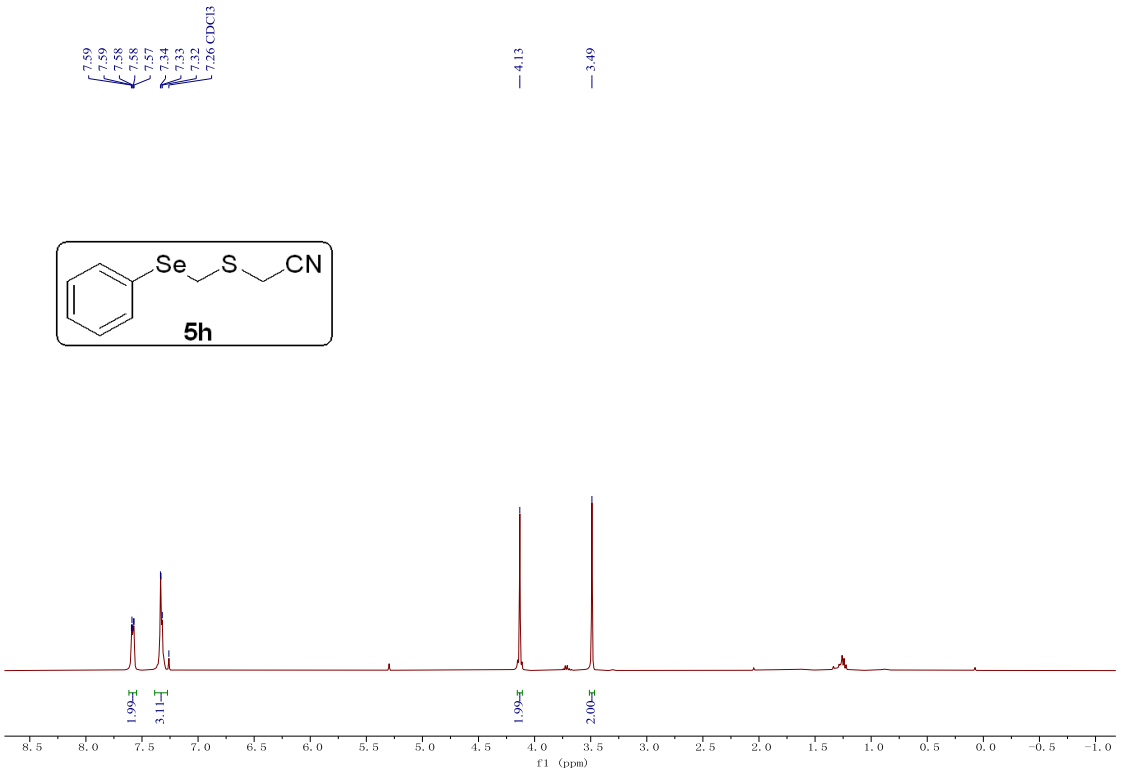

45.52
42.47
30.03
16.03

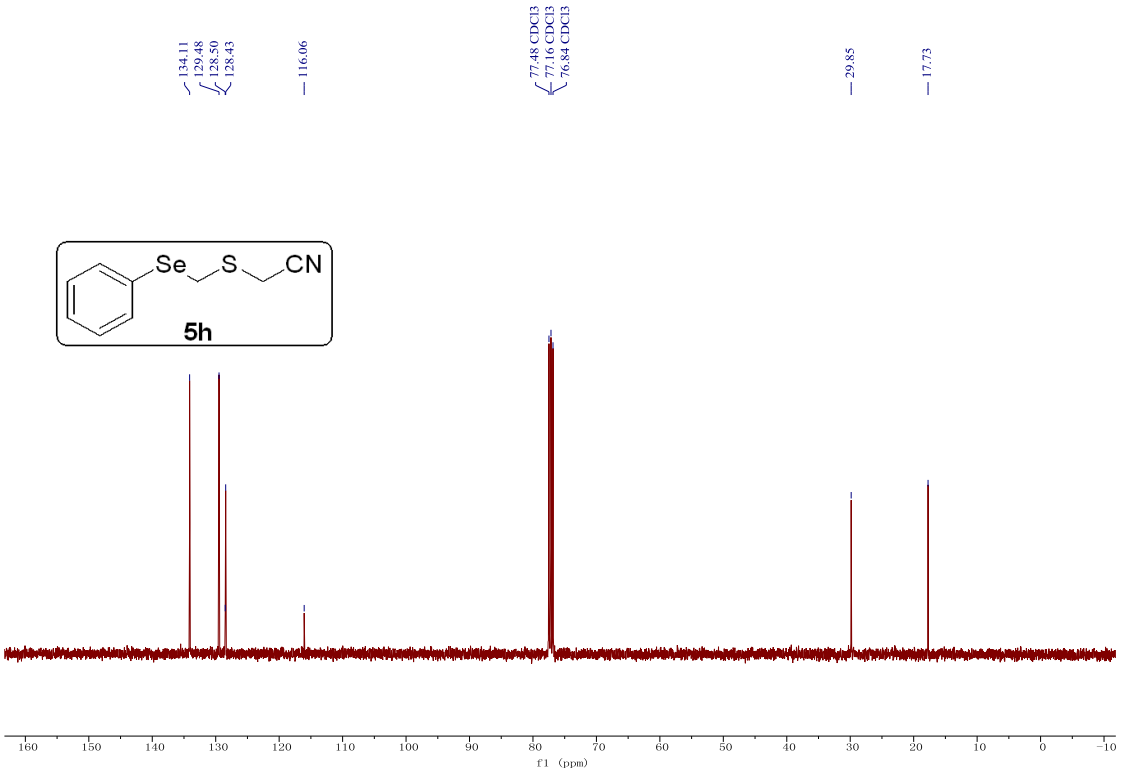

Se
CN
Bn
5f

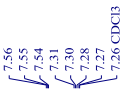

60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−1
f1 (ppm)


7.57
7.56
7.55
7.32
7.31
7.30
7.26 CDCl3
5.35
4.45
Se
O
CN
5g
2.06
3.14
2.00
2.07
f1 (ppm)

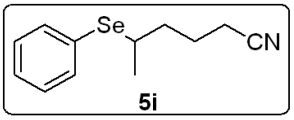

133.14
129.50
129.08
128.00
115.28
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
71.79
53.31
Se
O
CN
5g
f1 (ppm)

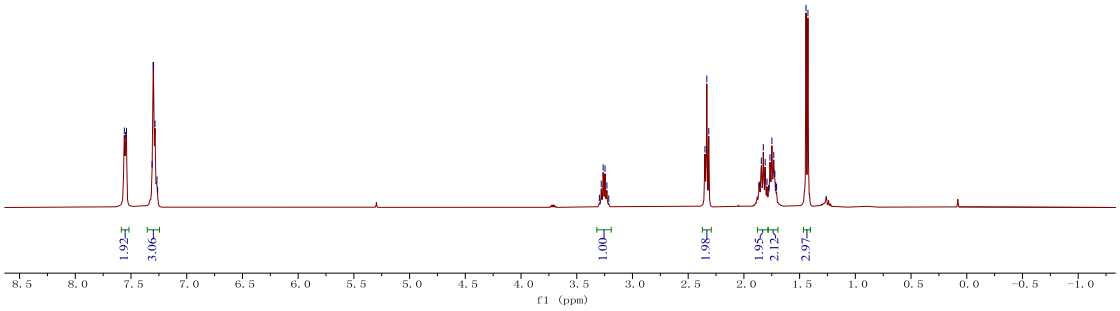

7.59
7.59
7.58
7.58
7.57
7.34
7.33
7.32
7.26 CDCl3
4.13
3.49
Se
S
CN
5h
1.99
3.11
1.99
2.00
f1 (ppm)


134.11
129.48
128.50
128.43
116.06
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
29.85
17.73
Se
S
CN
5h
f1 (ppm)


7.56
7.55
7.54
7.31
7.30
7.28
7.27
7.26 CDCl3


3.30
3.28
3.26
3.25
3.23
3.21
2.35
2.33
2.32
1.86
1.84
1.83
1.81
1.79
1.78
1.77
1.75
1.73
1.72
1.71
1.71
1.44
1.43


Se
CN
5i

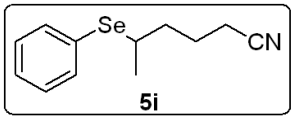

1.92
3.06
1.00
1.98
1.95
2.12
2.97
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

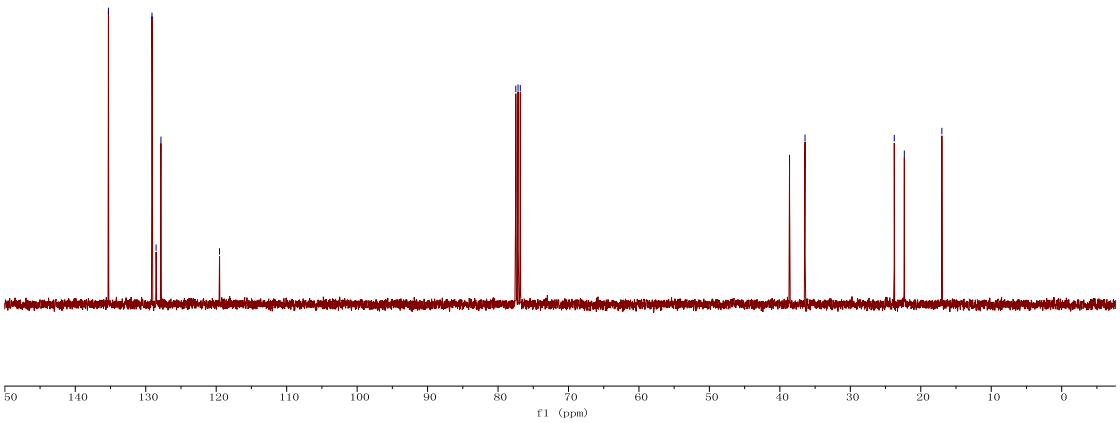

135.30
129.11
128.53
127.84
119.53

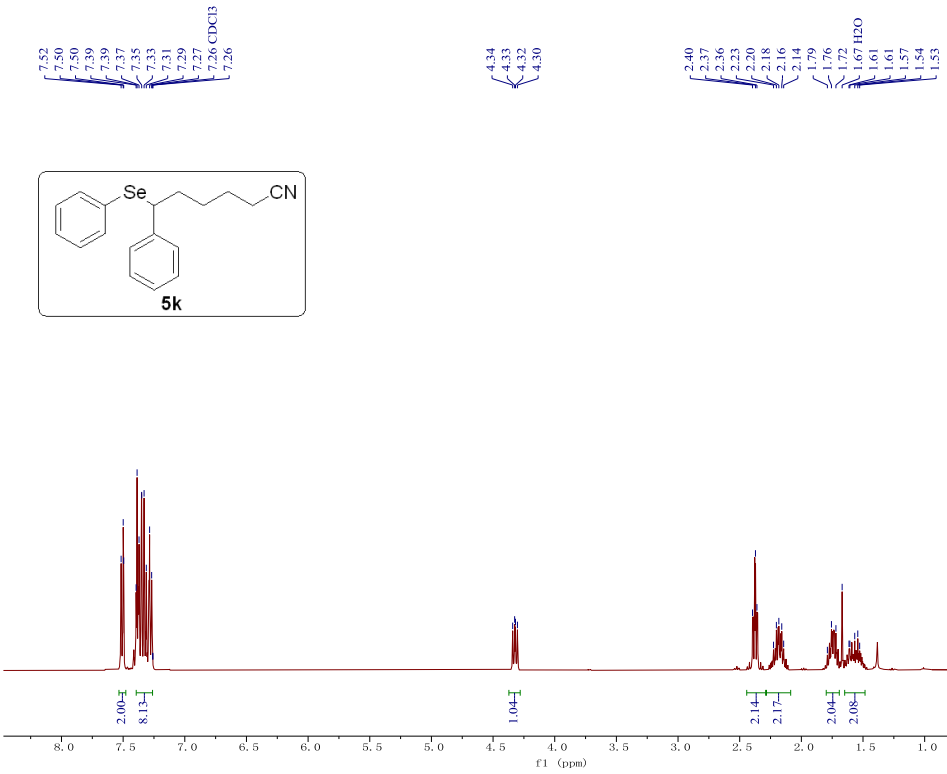
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3

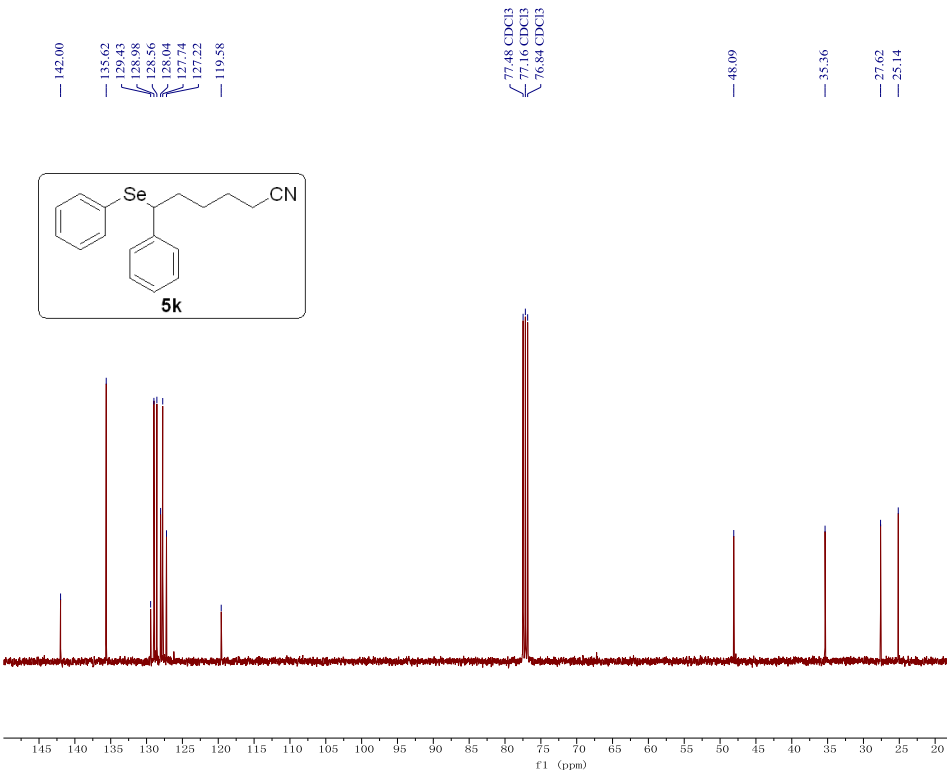

38.63
36.43

23.76
22.34
17.01


Se
CN
5i


50
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.52
7.50
7.50
7.39
7.39
7.37
7.35
7.33
7.31
7.29
7.27
7.26 CDCl3
7.26
4.34
4.33
4.32
4.30
2.40
2.37
2.36
2.23
2.20
2.18
2.16
2.14
1.79
1.76
1.72
1.67 H2O
1.61
1.61
1.57
1.54
1.53
Se
CN
5k
2.00
8.13
1.04
2.14
2.17
2.04
2.08
f1 (ppm)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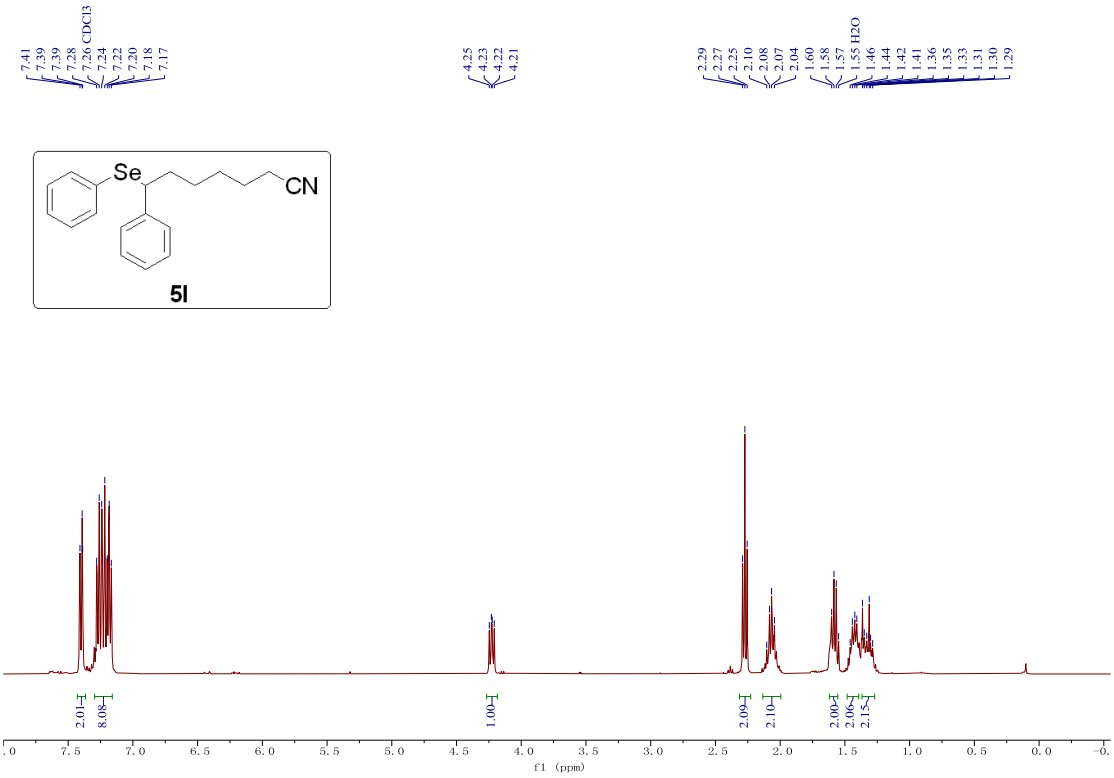
135.62
129.43
128.98
128.56
128.04
127.74
127.22
119.58
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
48.09
35.36
27.62
25.14
17.11
Se
CN
5k
f1 (ppm)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7.39
7.39
7.28
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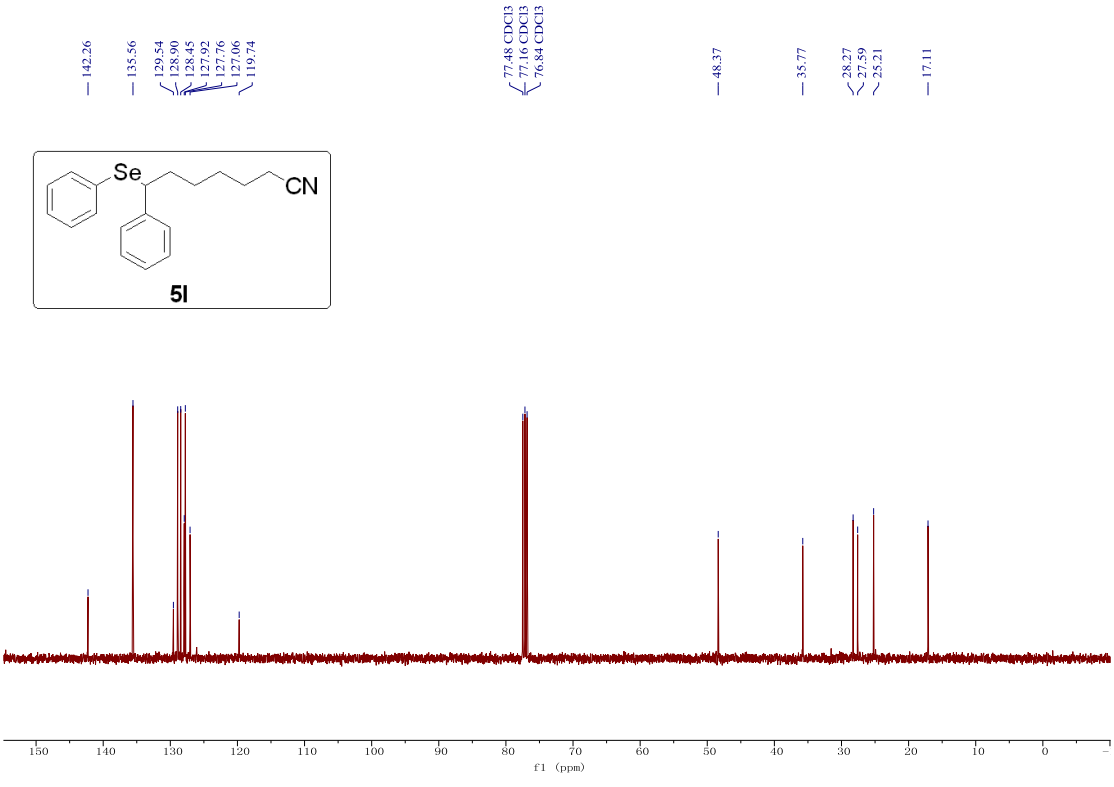
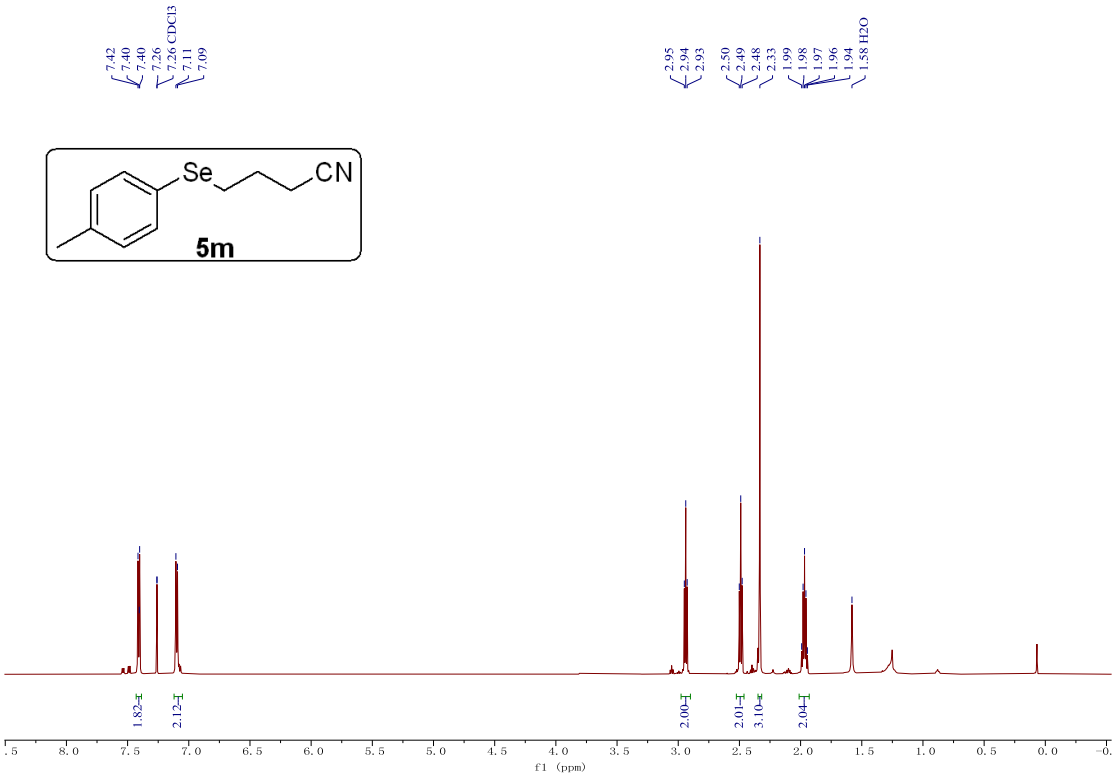
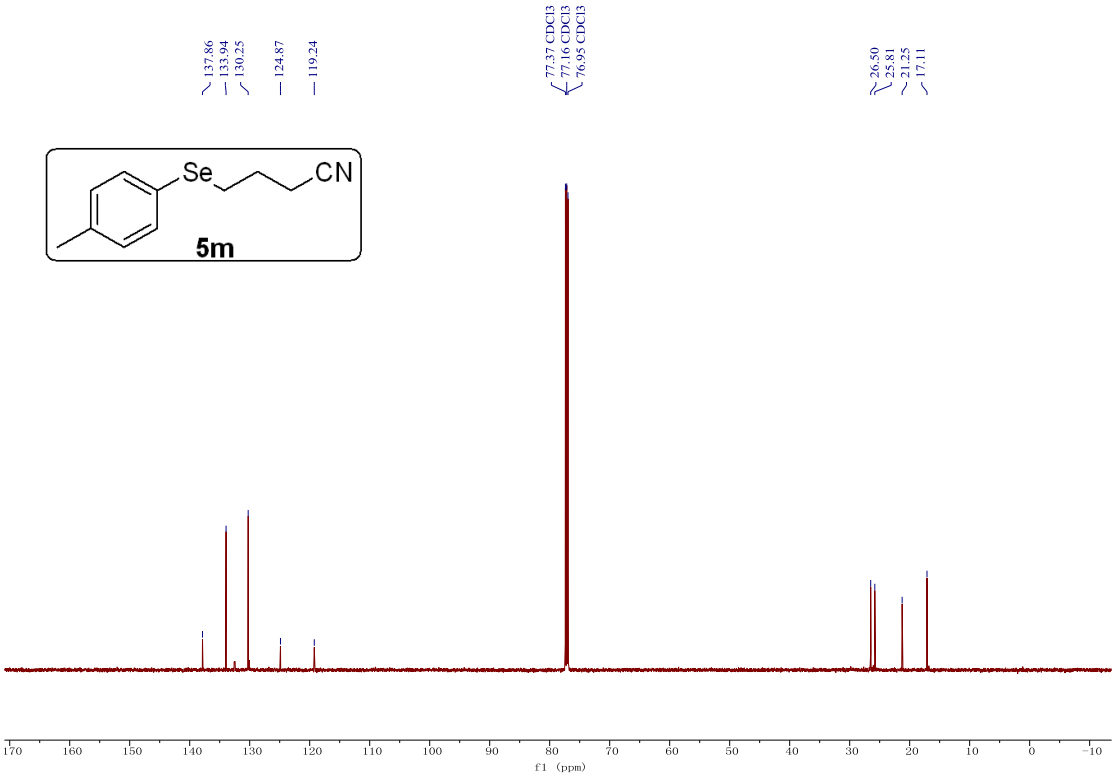
7.22
7.20
7.18
7.17
4.25
4.23
4.22
4.21
2.29
2.27
2.25
2.10
2.08
2.07
2.04
1.60
1.58
1.57
1.55 H2O
1.46
1.44
1.42
1.41
1.36
1.35
1.33
1.31
1.30
1.29
Se
CN
5l
2.01
8.08
1.00
2.09
2.10
2.00
2.06
2.15
f1 (ppm)


142.26
135.56
129.54
128.90
128.45
127.92
127.76
127.06
119.74
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
48.37
35.77
28.27
27.59
25.21
17.11
Se
CN
5l
f1 (ppm)


7.42
7.40
7.40
7.26
7.26 CDCl3
7.11
7.09
2.95
2.94
2.93
2.50
2.49
2.48
2.33
1.99
1.98
1.97
1.96
1.94
1.58 H2O
Se
CN
5m
1.82
2.12
2.00
2.01
3.10
2.04
f1 (ppm)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133.94
130.25
124.87
119.24
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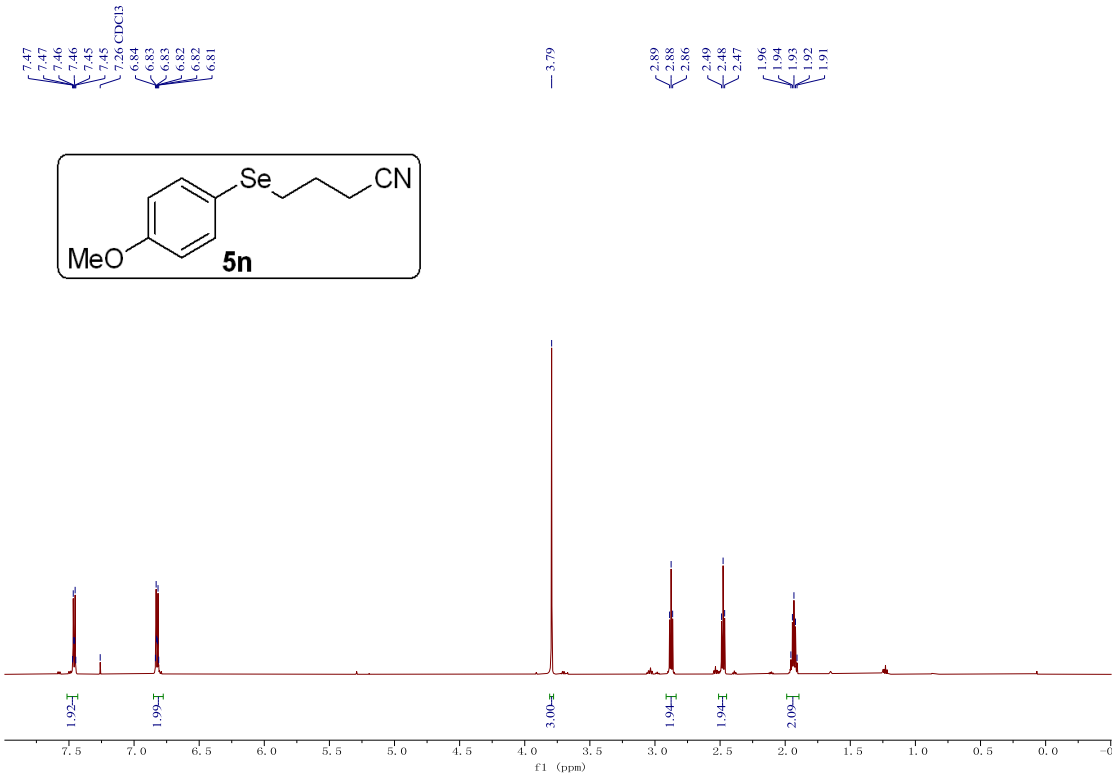
77.16 CDCl3
76.95 CDCl3
26.50
25.81
21.25
17.11
Se
CN
5m
f1 (ppm)


7.47
7.47
7.46
7.46
7.45
7.45
7.26 CDCl3
6.84
6.83
6.83
6.82
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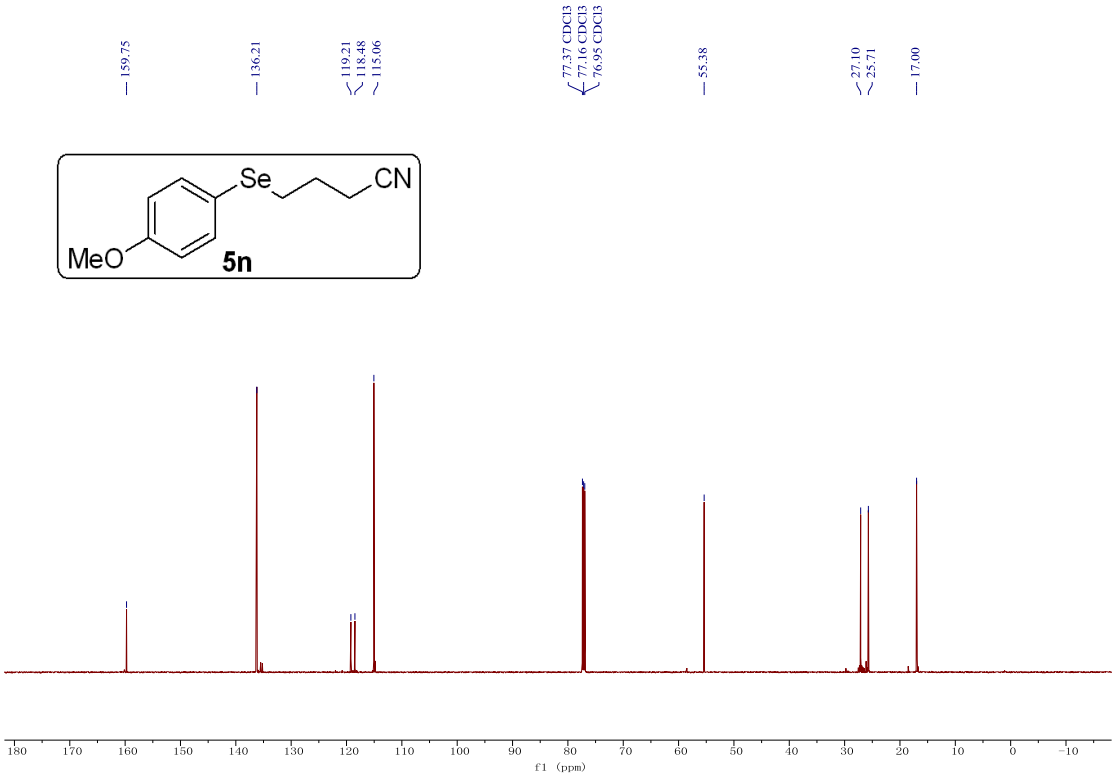
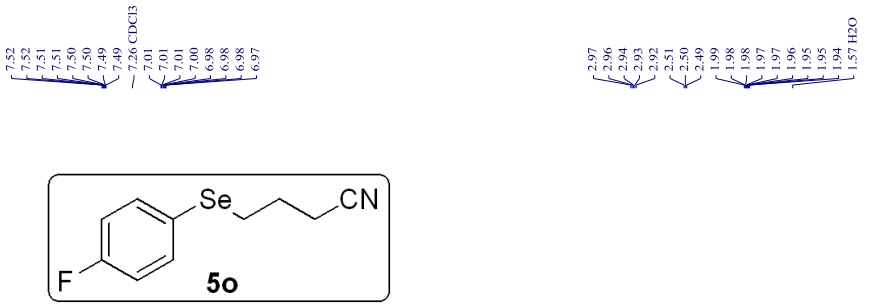
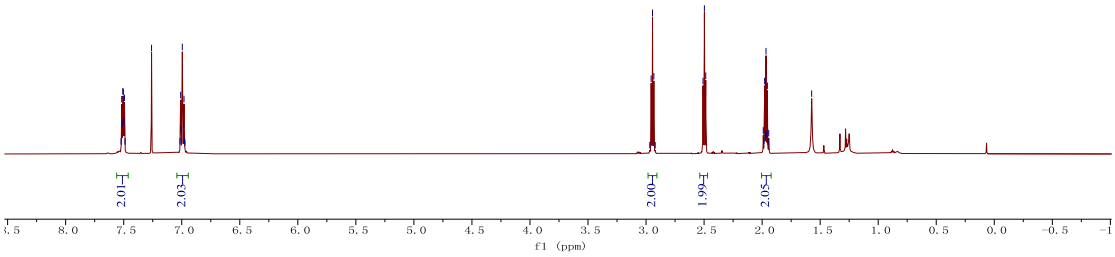
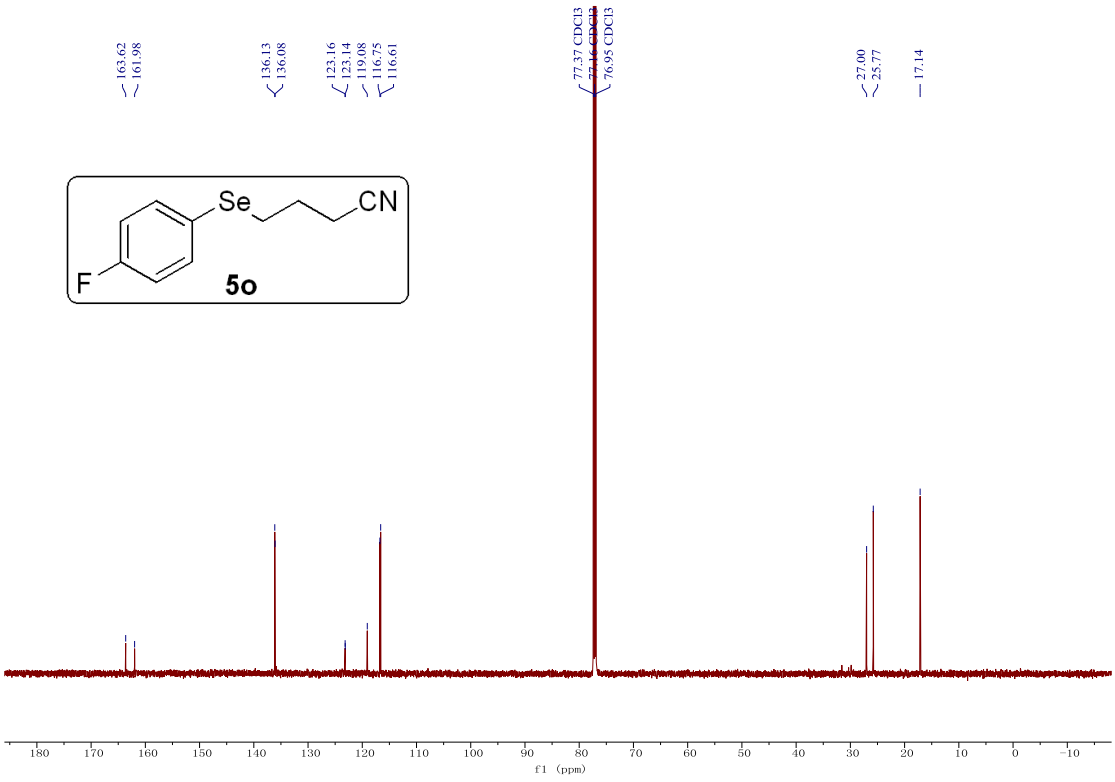
6.81
3.79
2.89
2.88
2.86
2.49
2.48
2.47
1.96
1.94
1.93
1.92
1.91
Se
CN
MeO
5n
1.92
1.99
3.00
1.94
1.94
2.09
f1 (ppm)


159.75
136.21
119.21
118.48
115.06
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
55.38
27.10
25.71
17.00
Se
CN
MeO
5n
f1 (ppm)

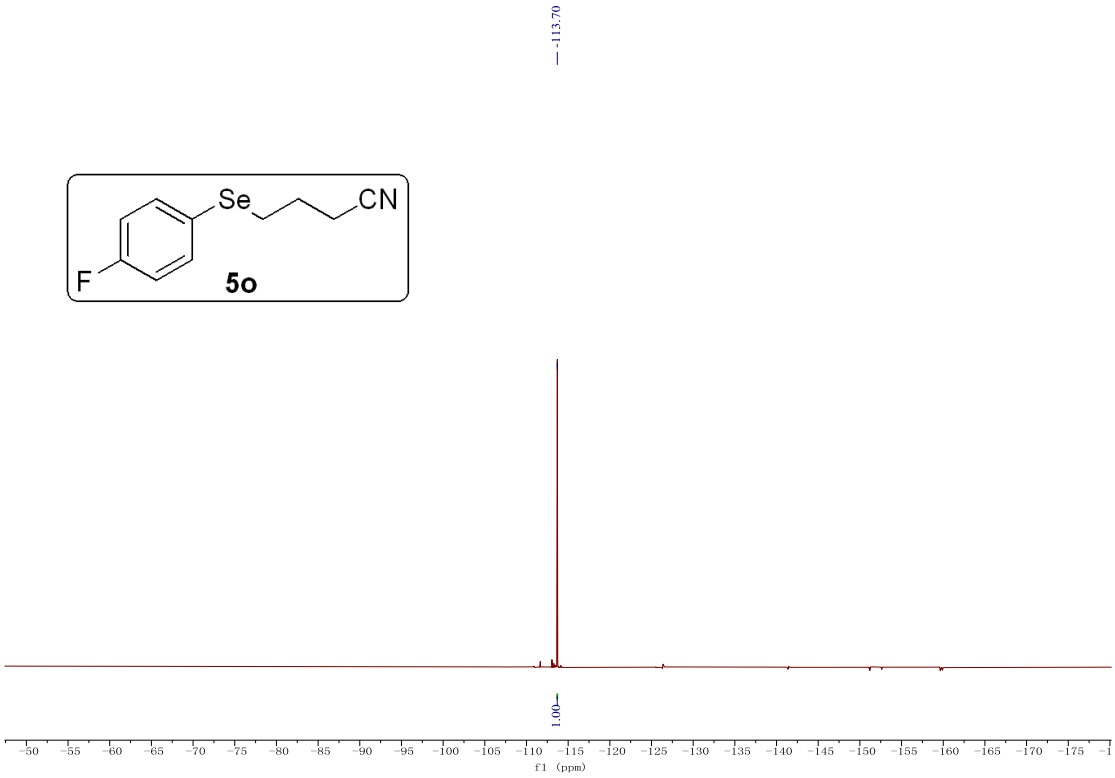

7.52
7.52
7.51
7.51
7.50
7.50
7.49
7.49
7.26 CDCl3
7.01
7.01
7.01
7.00
6.98
6.98
6.98
6.97
2.97
2.96
2.94
2.93
2.92
2.51
2.50
2.49
1.99
1.98
1.98
1.97
1.97
1.96
1.95
1.95
1.94
1.57 H2O

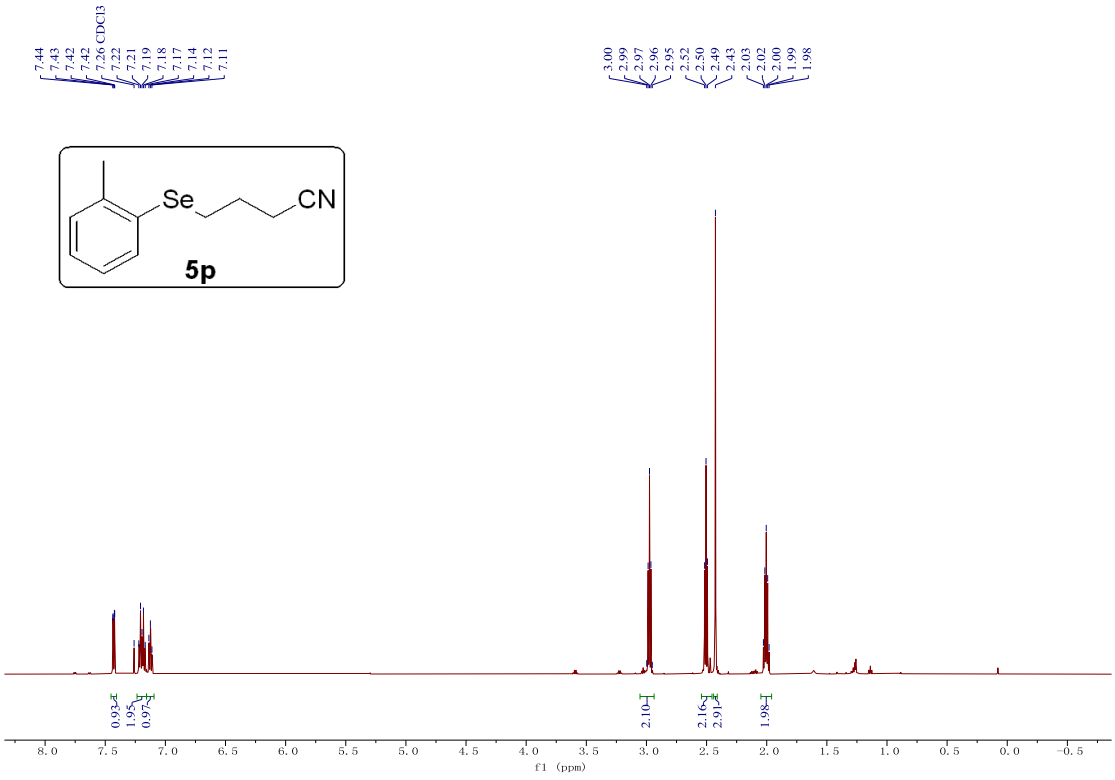
Se
CN
F
5o


2.01
2.03
2.00
1.99
2.05
f1 (ppm)


163.62
161.98
136.13
136.08
123.16
123.14
119.08
116.75
116.61
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
27.00
25.77
17.14
f1 (ppm)

Se
CN
F
5o


-113.70
Se
CN
F
5o
1.00
f1 (ppm)


7.44
7.43
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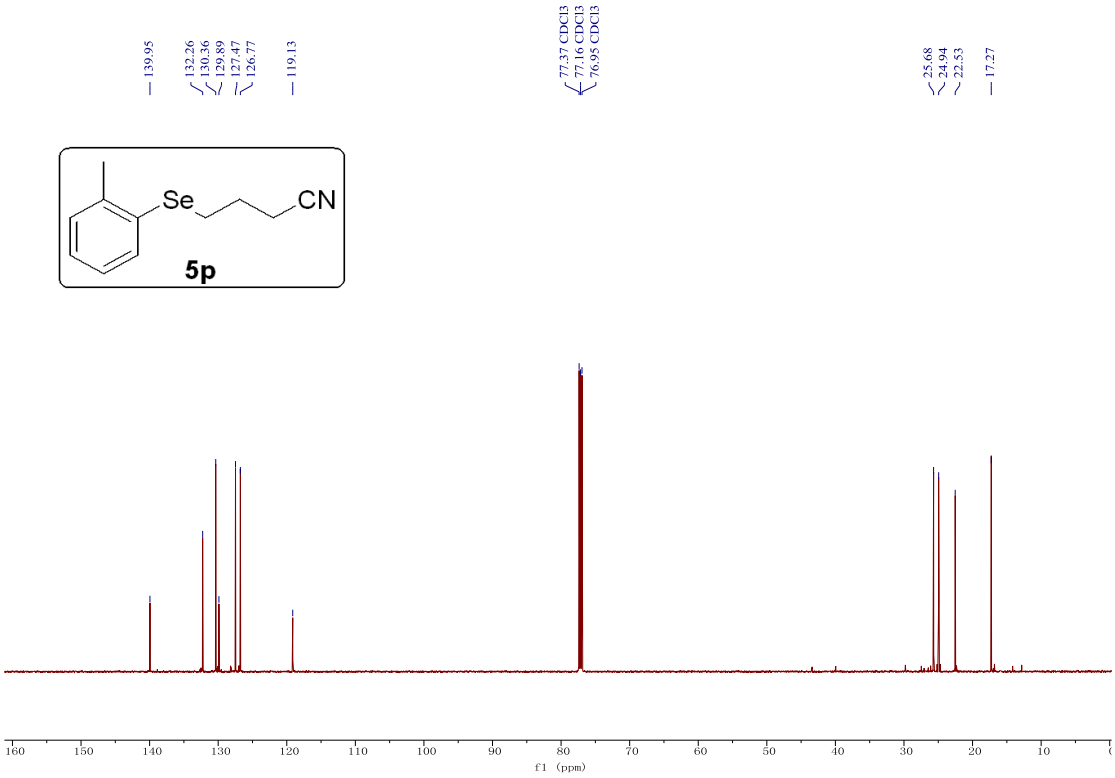
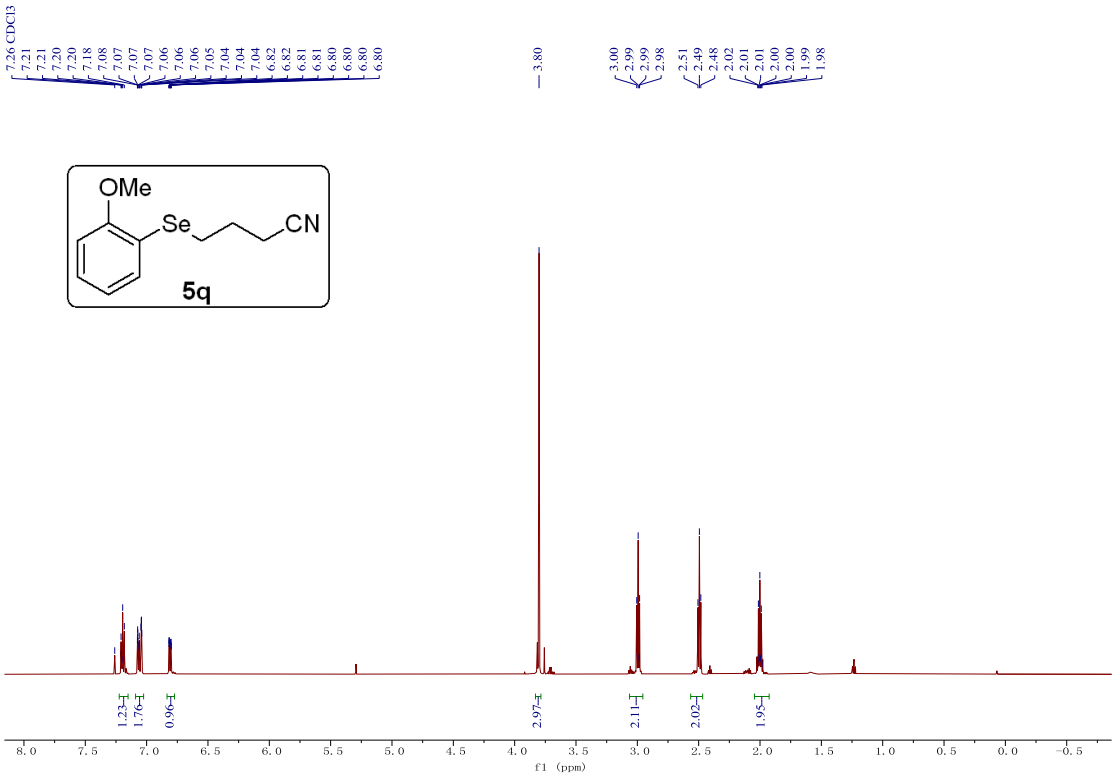
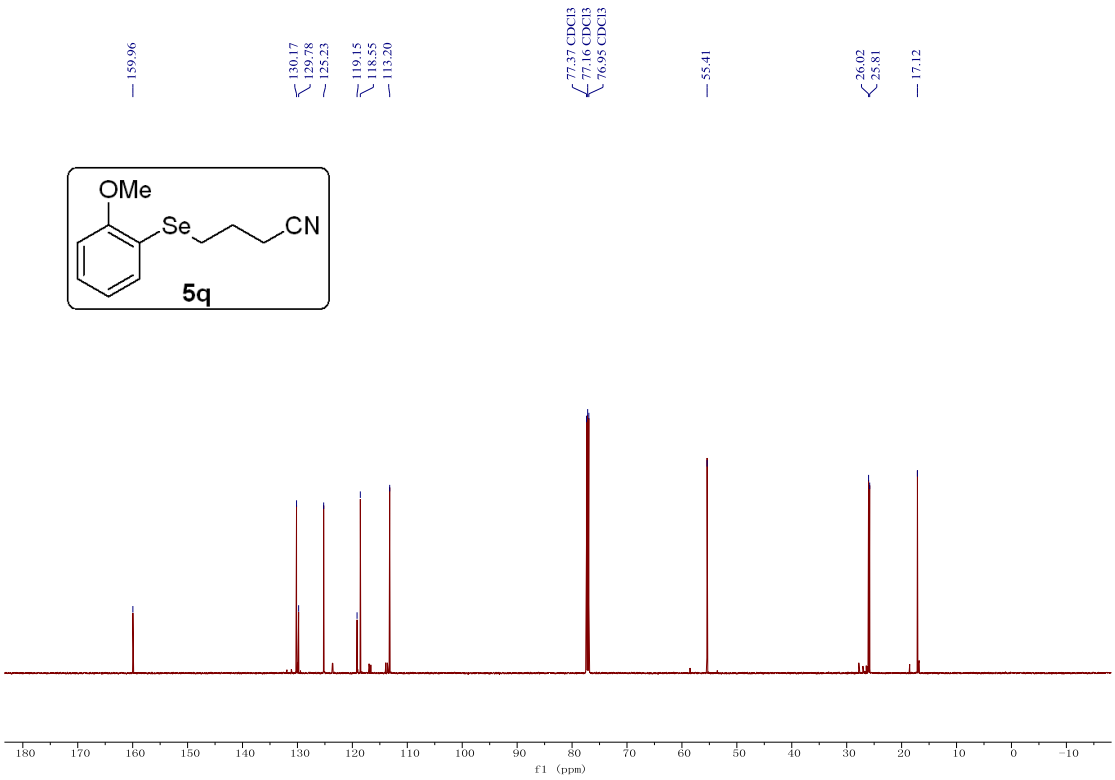
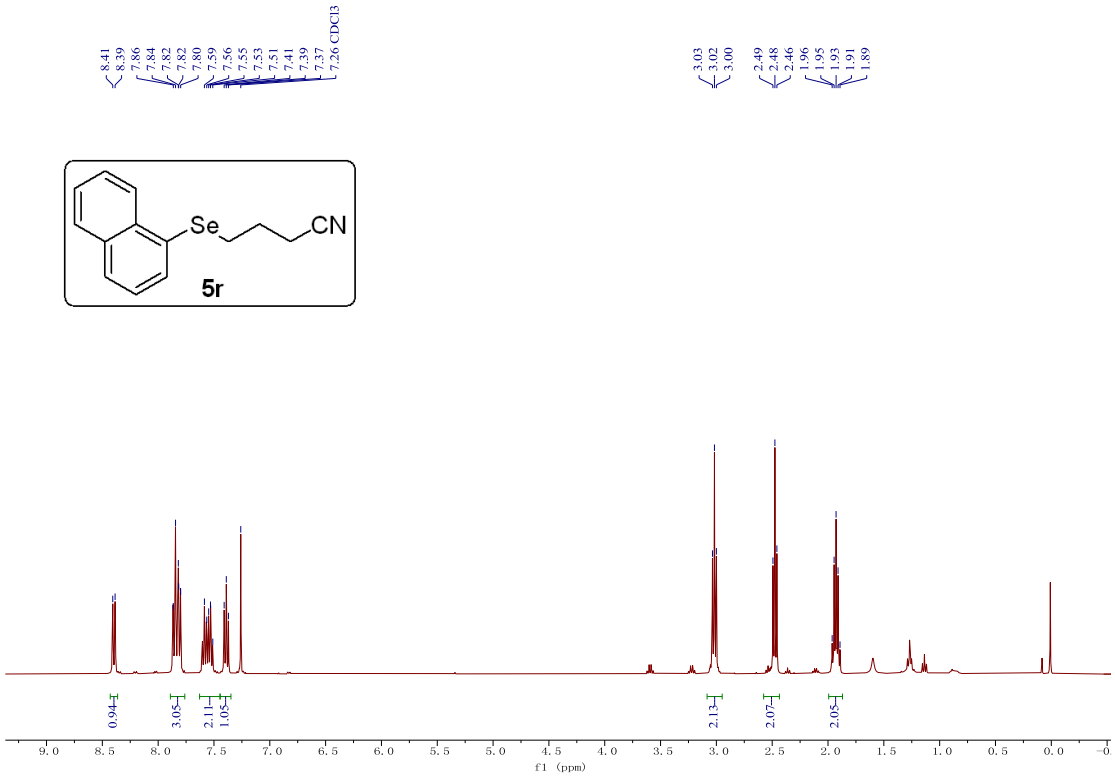
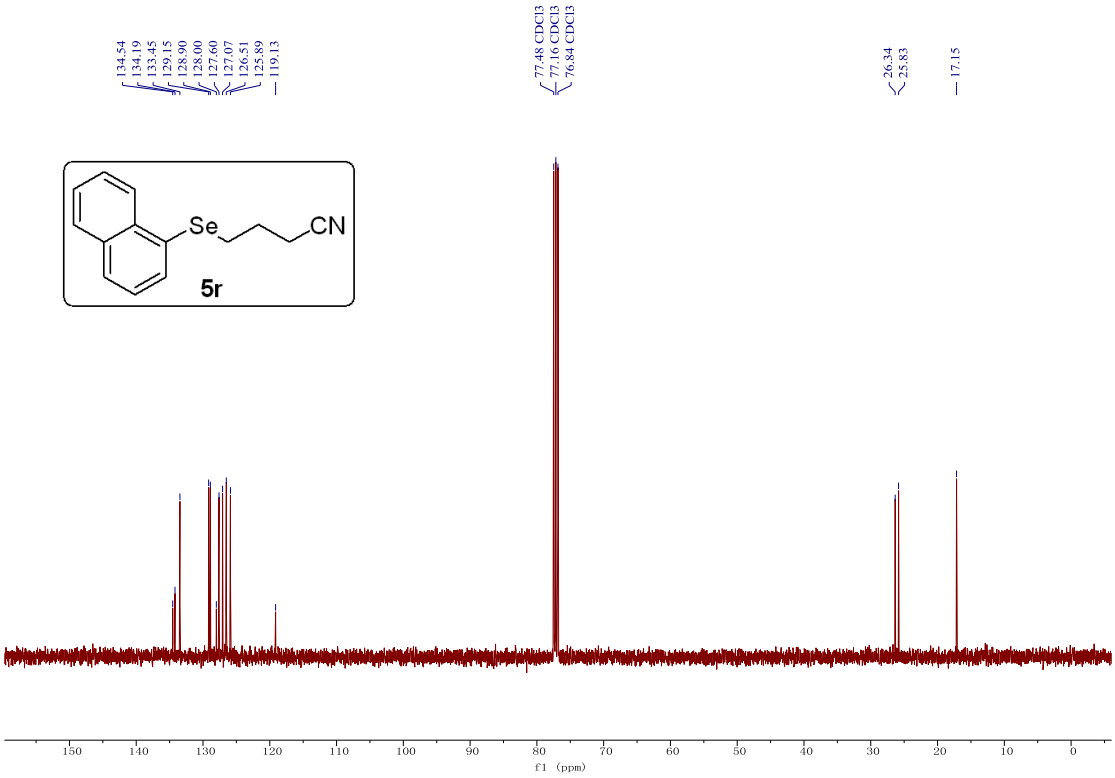
7.42
7.26 CDCl3
7.22
7.21
7.19
7.18
7.17
7.14
7.12
7.11
3.00
2.99
2.97
2.96
2.95
2.52
2.50
2.49
2.43
2.03
2.02
2.00
1.99
1.98
Se
CN
5p
0.93
1.95
0.97
2.10
2.16
2.91
1.98
f1 (ppm)

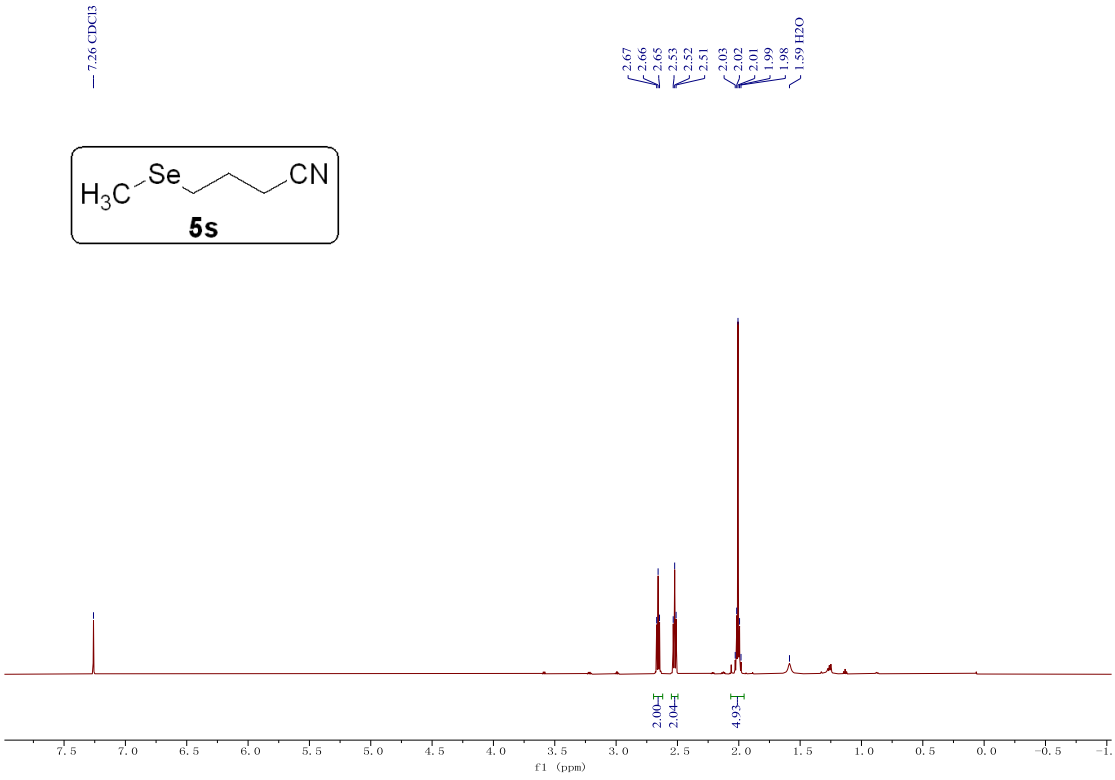

139.95
132.26
130.36
129.89
127.47
126.77
119.13
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
25.68
24.94
22.53
17.27
Se
CN
5p
f1 (ppm)

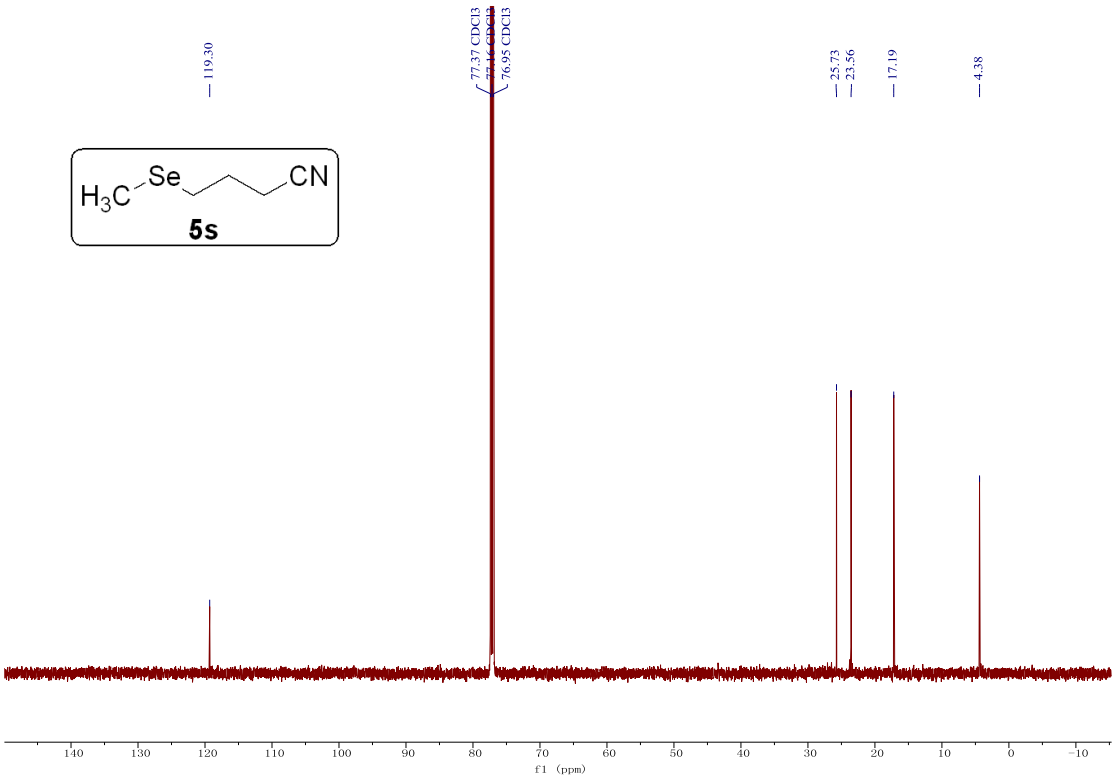

7.26 CDCl3
7.21
7.21
7.20
7.20
7.18
7.08
7.07
7.07
7.07
7.06
7.06
7.06
7.05
7.04
7.04
7.04
6.82
6.82
6.81
6.81
6.80
6.80
6.80
6.80
6.80
3.80
3.00
2.99
2.99
2.98
2.51
2.49
2.48
2.02
2.01
2.01
2.00
2.00
1.99
1.98
OMe
Se
CN
5q
1.23
1.76
0.96
2.97
2.11
2.02
1.95
f1 (ppm)

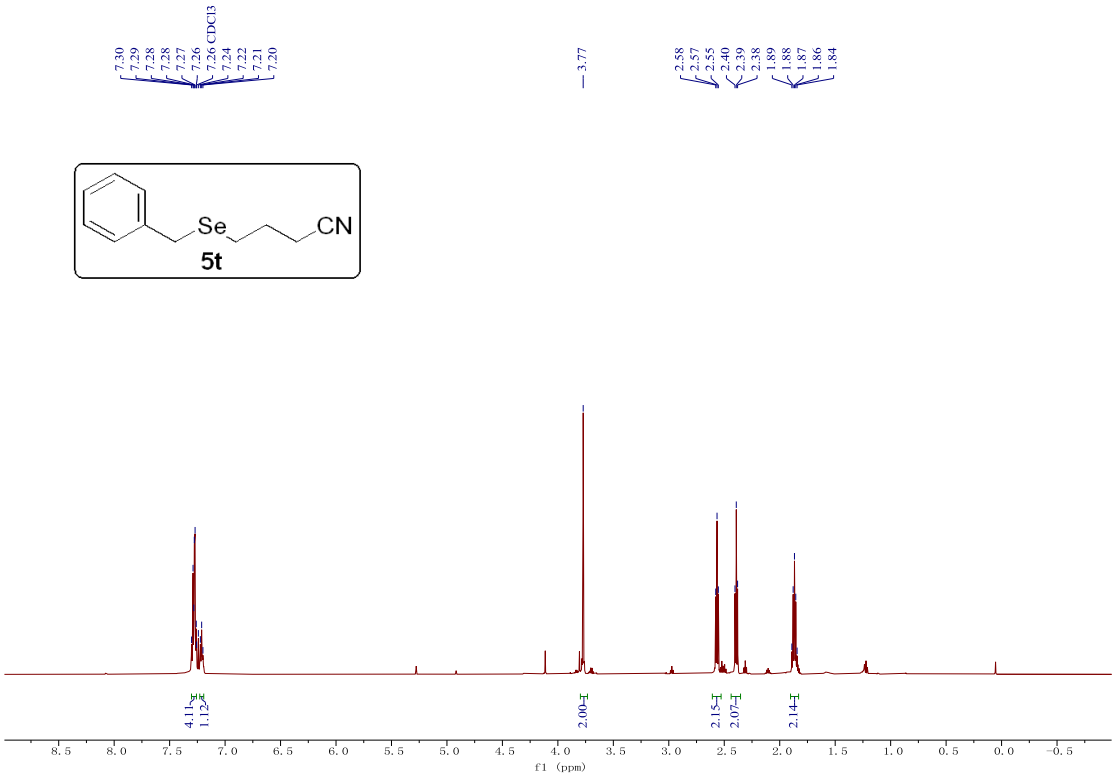

159.96
130.17
129.78
125.23
119.15
118.55
113.20
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
55.41
26.02
25.81
17.12
OMe
Se
CN
5q
f1 (ppm)

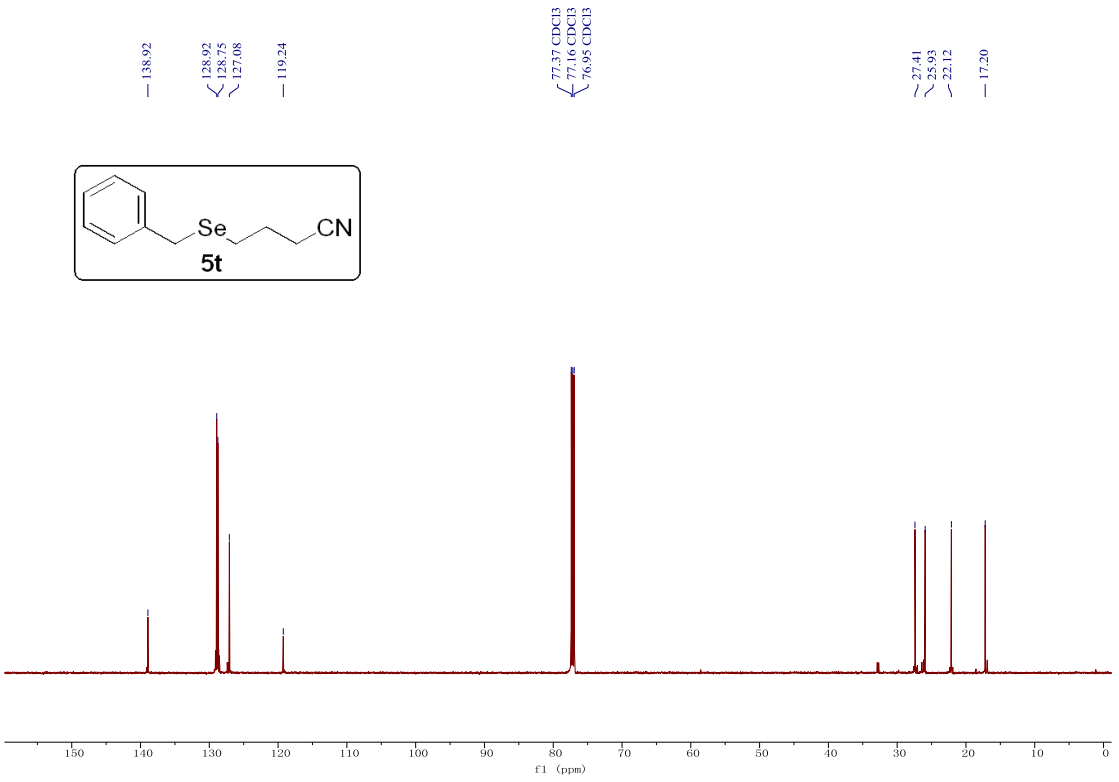

8.41
8.39
7.86
7.84
7.82
7.82
7.80
7.59
7.56
7.55
7.53
7.51
7.41
7.39
7.37
7.26 CDCl3
3.03
3.02
3.00
2.49
2.48
2.46
1.96
1.95
1.93
1.91
1.89
Se
CN
5r
0.94
3.05
2.11
1.05
2.13
2.07
2.05
f1 (ppm)

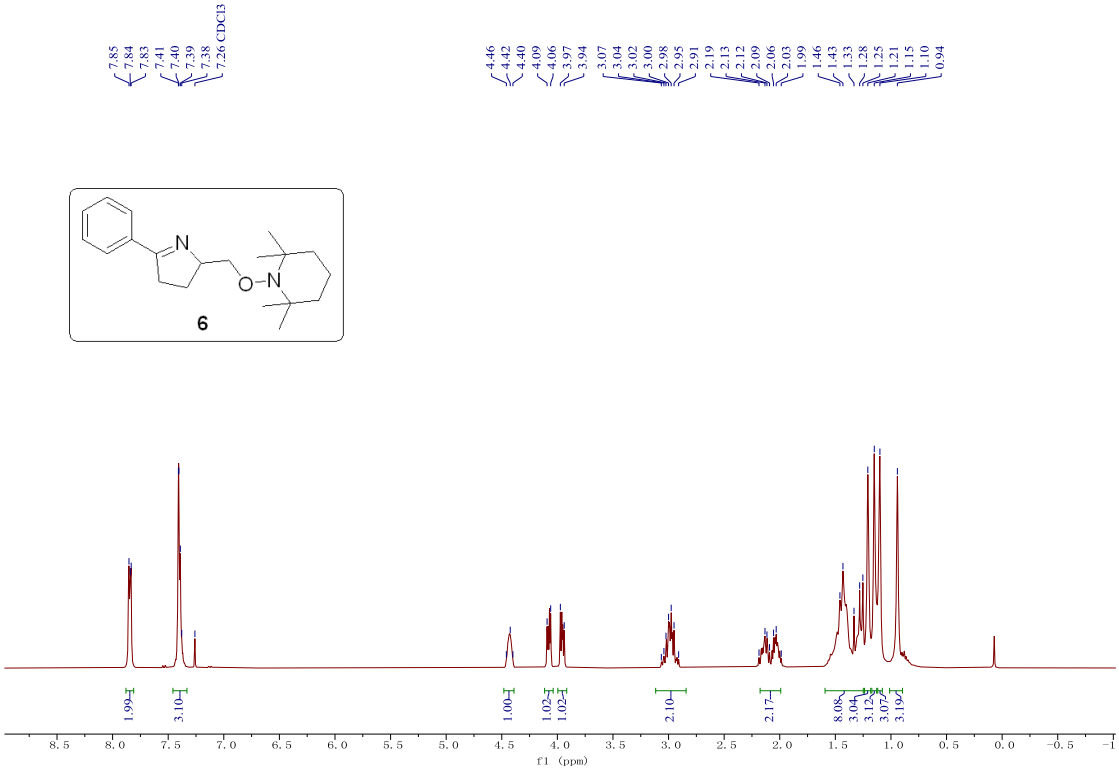

134.54
134.19
133.45
129.15
128.90
128.00
127.60
127.07
126.51
125.89
119.13
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
26.34
25.83
17.15
Se
CN
5r
f1 (ppm)

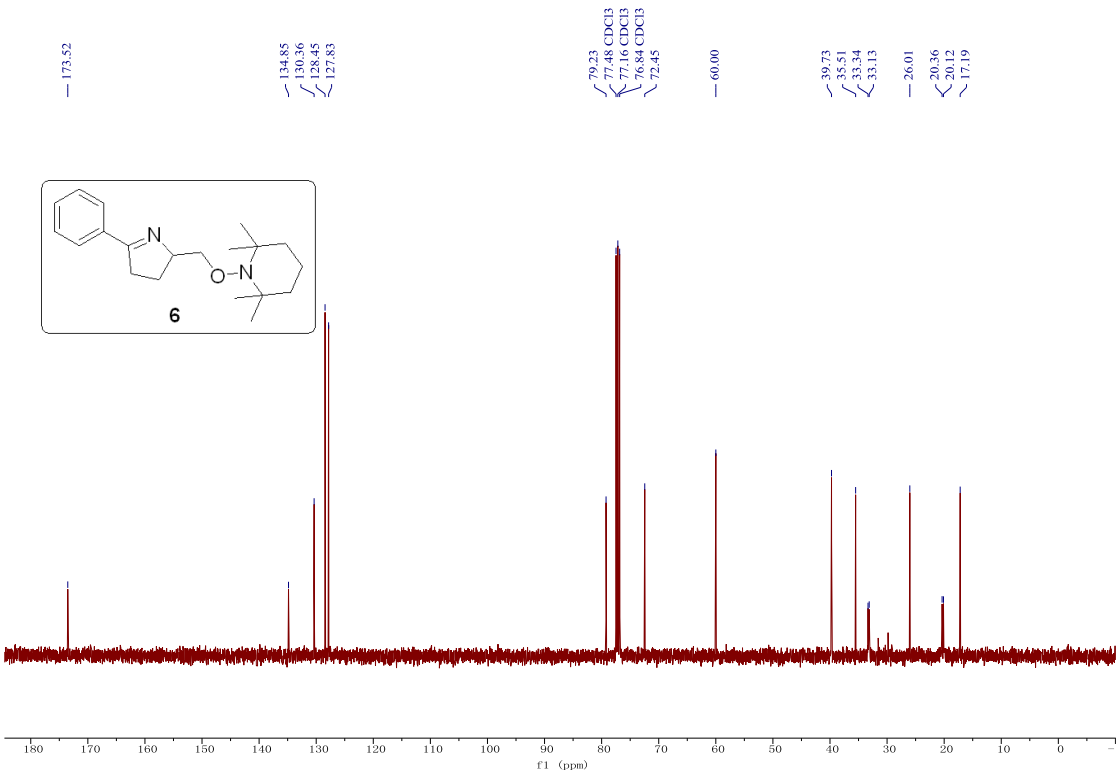

7.26 CDCl3
2.67
2.66
2.65
2.53
2.52
2.51
2.03
2.02
2.01
1.99
1.98
1.59 H2O
H3C
Se
CN
5s
2.00
2.04
4.93
f1 (ppm)

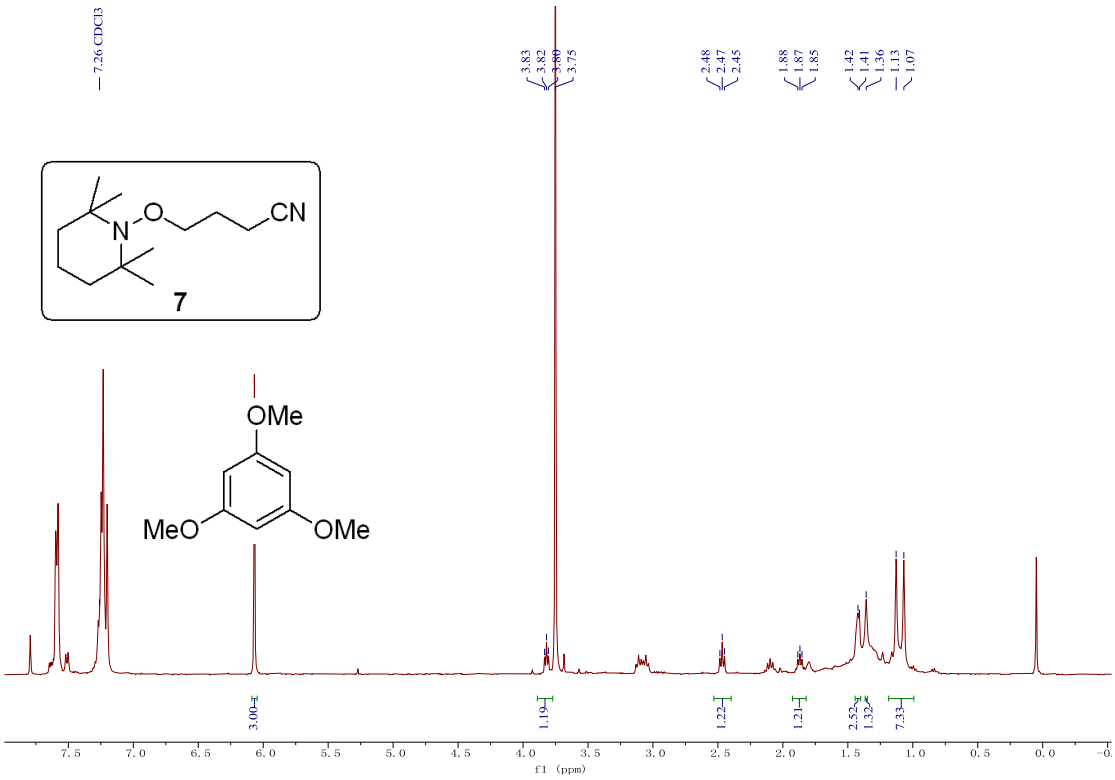

119.30
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
25.73
23.56
17.19
4.38
H3C
Se
CN
5s
f1 (ppm)

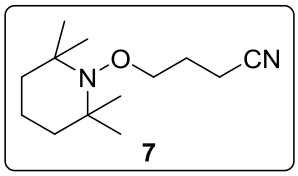

7.30
7.29
7.28
7.28
7.27
7.26
7.26 CDCl3
7.24
7.22
7.21
7.20
3.77
2.58
2.57
2.55
2.40
2.39
2.38
1.89
1.88
1.87
1.86
1.84
Se
CN
5t
4.11
1.12
2.00
2.15
2.07
2.14
f1 (ppm)


138.92
128.92
128.75
127.08
119.24
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
27.41
25.93
22.12
17.20
Se
CN
5t
f1 (ppm)


7.85
7.84
7.83
7.41
7.40
7.39
7.38
7.26 CDCl3
4.46
4.42
4.40
4.09
4.06
3.97
3.94
3.07
3.04
3.02
3.00
2.98
2.95
2.91
2.19
2.13
2.12
2.09
2.06
2.03
1.99
1.46
1.43
1.33
1.28
1.25
1.21
1.15
1.10
0.94
N
O-N
6
1.99
3.10
1.00
1.02
1.02
2.10
2.17
8.08
3.04
3.12
3.07
3.19
f1 (ppm)


173.52
134.85
130.36
128.45
127.83
79.23
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
72.45
60.00
39.73
35.51
33.34
33.13
26.01
20.36
20.12
17.19
N
O-N
6
f1 (ppm)


7.26 CDCl3
3.83
3.82
3.80
3.75
2.48
2.47
2.45
1.88
1.87
1.85
1.42
1.41
1.36
1.13
1.07
CN
N
O
7
OMe
MeO
OMe
3.00
1.19
1.22
1.21
2.53
1.32
7.33
f1 (ppm)



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Created by: waters, waters

Created on: May 05, 2022

Item name: MS-20220505

Created time: 14:47:08 China Standard Time

Analysis Information

Item name:	MS-20220505	Analysis Method Item name:	MS-NOPDA-0929
Version:	2	Analysis Method Version:	1
Modified date:	May 05, 2022 14:44:46 China Standard Time	Sample Set Created date:	May 05, 2022 14:27:02 China Standard Time
Modified by:	waters, waters	Sample Set Instrument system name:	Vion UPLC
Folder:	Company/Sample/2022		

MS Instrument Type: Waters Vion® IMS QTof

Experiment Settings:

Experiment type:	ESI+	Scan Mode:	MS
Capillary voltage:	3.0kv	Low mass:	50m/z
Soure temperature:	120°C	High mass:	2000m/z
Desolvation temperature:	450°C	Scan time:	0.200s
Cone gas:	50L/h		
Desolvation gas:	800L/h		

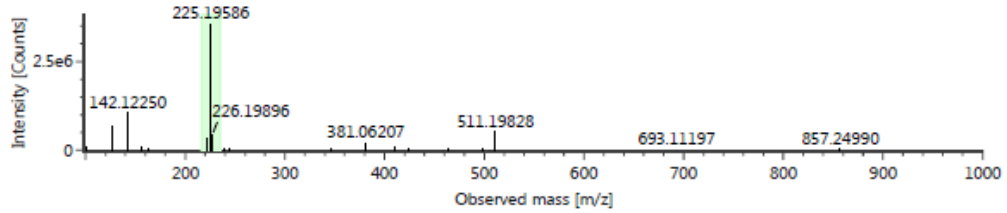
Item name: 20220505-CXZ-2, Sample position: 1:E,8, Replicate number: 1

.	Formula	Neutral mass (Da)	Observed m/z	Mass error (mDa)	Mass error (ppm)	Response	Adducts	Identification status
1	C13H24N2O	224.18886	225.19586	-0.3	-1.3	657026	+H	Identified

Item name: 20220505-CXZ-2

Item description:

Channel name: Time 0.8248 +/- 0.0500 minutes



Item name: 20220505-CXZ-2

Item description:

Channel name: Time 0.8248 +/- 0.0500 minutes