

Supporting Information for

Versatile electrooxidative amino- and oxyselenation of alkenes

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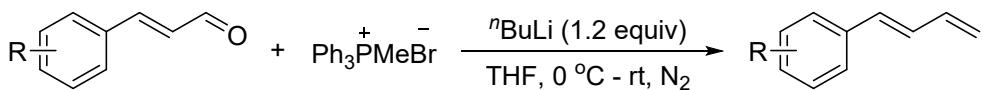
1. General considerations

¹H, ¹³C and ¹⁹F NMR spectra were recorded on Varian Inova-400 or 600 MHz spectrometers. ¹H and ¹³C NMR chemical shifts were determined relative to internal standard TMS at δ 0.0 ppm or CDCl₃ (δ (¹H), 7.26 ppm; δ (¹³C), 77.16 ppm) or DMSO-D₆ (δ (¹H), 2.50 ppm; δ (¹³C), 39.52 ppm). Chemical shifts (δ) are reported in ppm, and coupling constants (J) are reported in Hertz (Hz). The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. The HRMS analysis was obtained on a Agilent 6540 UHD Q-TOF mass spectrometer. The melting point was recorded on BÜCHI (M-560) and uncorrected. Analytical thin layer chromatography (TLC) was performed on 0.25 mm silica gel 60 F254 plates and viewed by UV light (254 nm). Column chromatographic purification was performed using 200-300 mesh silica gel. Electrochemical reactions were performed on IKA ElectraSyn 2.0 pro. X-ray single crystal diffraction data were collected on a Bruker D8 VENTURE. H₂ detection experiment was conducted on a ES20B-H₂ gas detector (Shenzhen Eyesky Technology Co., Ltd). Cyclic voltammetry (CV) was carried out on a CHI660E electrochemical workstation (CH Instruments, Ins).

All the chemical reagents were purchased from commercial sources and used as received unless otherwise indicated. Aryl 1,3-butadienes^[1] and diselenides^[2] were prepared by the reported procedures.

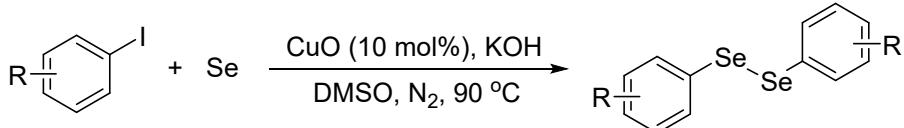
2. Experimental procedures

2.1 General procedure for the synthesis of aryl 1,3-butadienes^[1]



Under nitrogen atmosphere, methyltriphenylphosphonium bromide (3.0 mmol) in THF (20 mL) was added ⁿBuLi (1.2 mL, 2.5 M in THF, 3.0 mmol) slowly at 0 °C in a flame-dried round-bottom flask. After stirring for 20 min, the cinnamaldehyde (2.5 mmol) was added. The reaction mixture was then warmed to room temperature and stirred for another 5-10 hours. After the starting material was consumed completely which was detected by TLC, the reaction mixture was quenched with sat. NH₄Cl aq. (15 mL) and extracted with ethyl acetate (20 mL × 3). The combined organic layers were dried over Na₂SO₄, concentrated in vacuo and purified by flash chromatography on silica gel with petroleum ether (60-90 °C) to afford the desired 1,3-butadiene products.

2.2 General procedure for the synthesis of diselenides^[2]

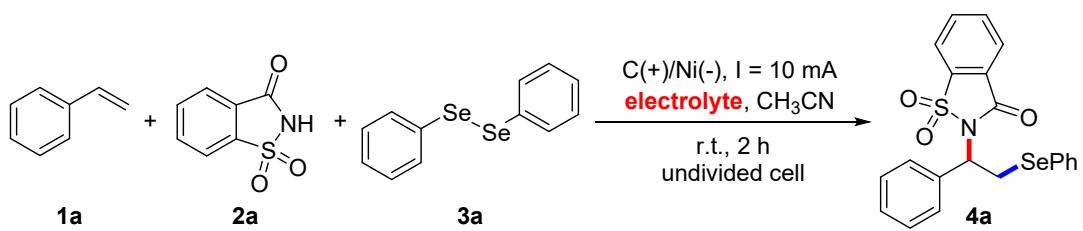


Under nitrogen atmosphere, to a stirred solution of Se (6.0 mmol) powder and aryl iodides (3.0 mmol) in dry DMSO (6.0 mL) was added CuO nanoparticles (10 mol%) followed by KOH (2.0 equiv) at 90 °C. The progress of the reaction was monitored by TLC. After the reaction was complete, the reaction mixture was allowed to cool, which was subjected to column chromatographic separation to give pure diselenides.

2.3 Optimization of reaction conditions

2.3.1 Optimization of aminoselenation of styrene

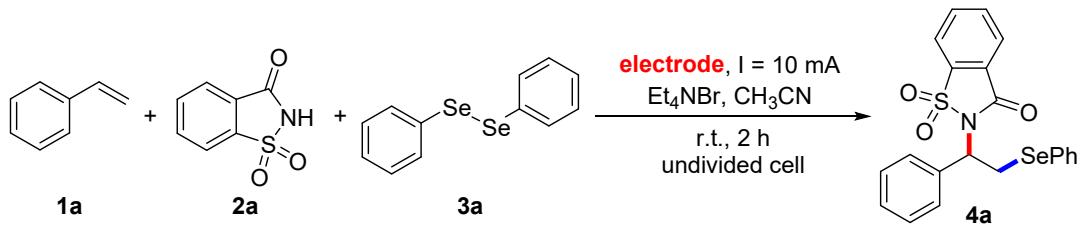
Table S1 Screening of electrolytes^a



Entry	Electrolyte	Yield (%) ^b
1	"Bu ₄ NBr	83
2	Me ₄ NCl	66
3	Me ₄ NBr	92
4	Et₄NBr	92
5	Me ₄ NBF ₄	24
6	"Pr ₄ NBr	86
7	"Bu ₄ NI	n.r.
8	"Bu ₄ NClO ₄	trace
9	"Bu ₄ NHSO ₄	trace
10	"Bu ₄ NBF ₄	trace
11	"Bu ₄ NPF ₆	trace

^a Reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), electrolyte (0.2 mmol), CH₃CN (4.0 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields. n.r. = no reaction.

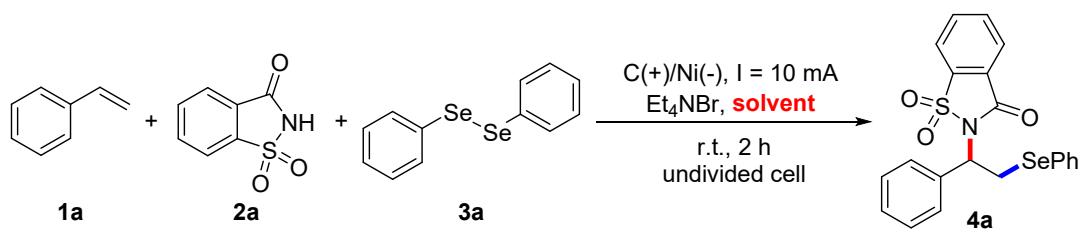
Table S2 Screening of electrodes^a



Entry	Electrode	Yield (%) ^b
1	C-Ni	92
2	C-C	86
3	C-Cu	84
4	C-Zn	62

^a Reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), Et₄NBr (0.2 mmol), CH₃CN (4.0 mL), anode (immersed surface area 8 × 5 mm²), cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields.

Table S3 Screening of solvents^a

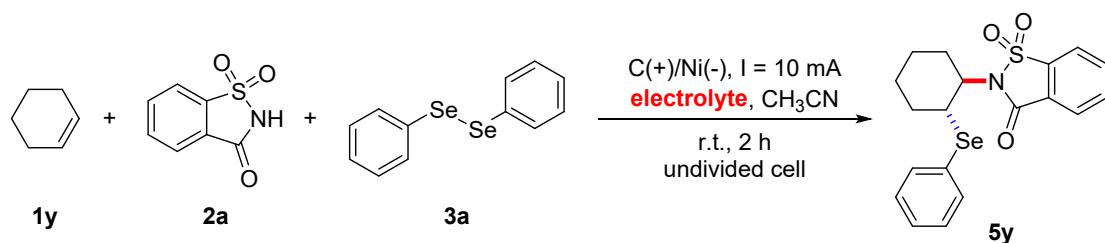


Entry	Solvent	Yield (%) ^b
1	CH_3CN	92
2	MeOH	n.r.
3	DMF	46
4	DMSO	n.r.
5	DMA	trace

^a Reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), Et_4NBr (0.2 mmol), solvent (4.0 mL), C anode (immersed surface area $8 \times 5 \text{ mm}^2$), Ni cathode (immersed surface area $8 \times 5 \text{ mm}^2$), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields. DMF = *N,N*-dimethylformamide. DMSO = dimethyl sulfoxide. DMA = *N,N*-dimethylacetamide.

2.3.2 Optimization of aminoselenation of cyclohexene

Table S4 Screening of electrolytes^a



Entry	Electrolyte	Yield (%) ^b
1	Et_4NBr	66
2	Bu_4NBr	68
3	Cetyltrimethylammonium bromide	59
4	Bu_4NPf_6	44
5	Bu_4NI	n.r.
6	TEABF_4	35
7	KBr	54
8	Bu_4NCl	23
9	NaBr	53
10	TBAClO_4	28
11	Bu_4NOAc	45
12	Bu_4NBr_3	43
13	$[\text{BzM}_3\text{N}] \text{Br}$	trace
14	HMMImBr	71

15	[P ₁₄]Br	68
16	[PMIm]Br	77
17	[P ₄₄₄₄]Br	59
18	VBIImBr	78
19	BzMIImBr	63

The figure shows seven chemical structures of imidazolium and phosphonium salts. From left to right: 1. [BzM₃N]Br, a benzyltrimethylammonium cation with a bromide anion. 2. HMMIImBr, 1-hexyl-2,3-dimethylimidazolium bromide. 3. [P₁₄]Br, 1-butyl-1-methylpyrrolidinium bromide. 4. [PMIm]Br, 1-methyl-3-propylimidazolium bromide. 5. [P₄₄₄₄]Br, tetrabutylphosphonium bromide. 6. VBIImBr, 1-butyl-3-vinylimidazolium bromide. 7. BzMIImBr, 1-benzyl-3-methylimidazolium bromide.

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), electrolyte (0.2 mmol), CH₃CN (4 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields. [BzM₃N]Br = benzyltrimethylammonium bromide. HMMIImBr = 1-hexyl-2,3-dimethylimidazolium bromide. [P₁₄]Br = 1-butyl-1-methylpyrrolidinium bromide. [PMIm]Br = 1-methyl-3-propylimidazolium bromide. [P₄₄₄₄]Br = tetrabutylphosphonium bromide. VBIImBr = 1-butyl-3-vinylimidazolium bromide. BzMIImBr = 1-benzyl-3-methylimidazolium bromide.

Table S5 Screening of quantity of 1y^a

1y	2a	3a	C(+)/Ni(-), I = 10 mA VBIImBr, CH ₃ CN r.t., 2 h undivided cell		5y
Entry	1y (mmol)	Yield (%) ^b			
1	0.3	78			
2	0.2	71			
3	0.4	53			

^a Reaction conditions: **1y** (x mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBIImBr (0.2 mmol), CH₃CN (4 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields.

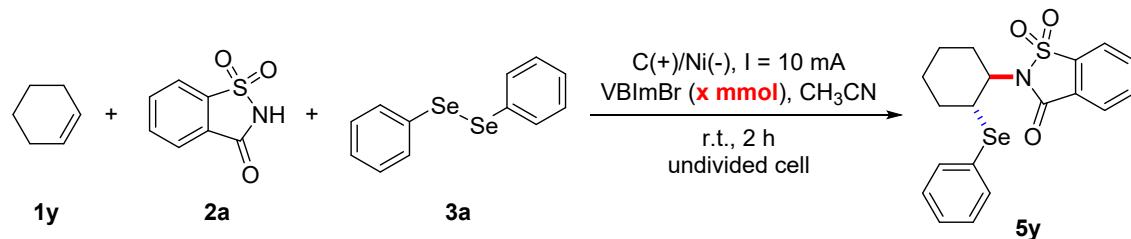
Table S6 Screening of quantity of 3a^a

1y	2a	3a	C(+)/Ni(-), I = 10 mA VBIImBr, CH ₃ CN r.t., 2 h undivided cell		5y
Entry	3a (mmol)	Yield (%) ^b			
1	0.15	78			
2	0.1	71			
3	0.2	53			

Entry	3a (mmol)	Yield (%) ^b
1	0.15	78
2	0.1	49
3	0.2	70

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (x mmol), VBImBr (0.2 mmol), CH₃CN (4 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields.

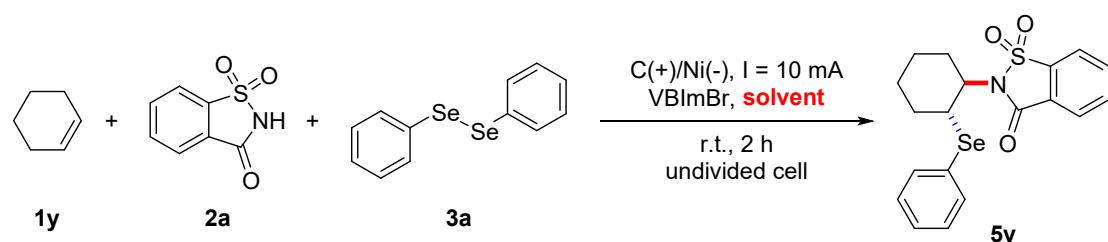
Table S7 Screening of quantity of electrolyte^a



Entry	VBImBr (mmol)	Yield (%) ^b
1	0.2	78
2	0.1	66
3	0.3	53

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBImBr (x mmol), CH₃CN (4 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields.

Table S8 Screening of solvents^a

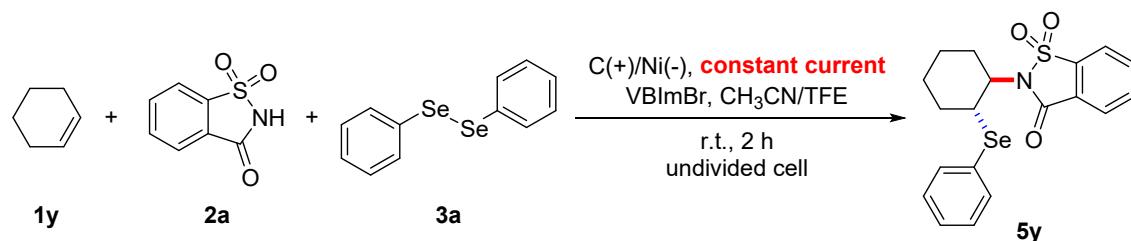


Entry	Solvent	Yield (%) ^b
1	CH ₃ CN	78
2	H ₂ O	n.r.
3	CH ₃ CH ₂ OH	61
4	CH ₃ OH	trace
5	Isopropyl alcohol	78
6	CH ₃ CH ₃ CH ₃ CH ₂ OH	trace
7	HOCH ₂ CH ₂ OH	n.r.
8	DMSO	27
9	NMP	n.r.
10	DMF	trace

11	HFIP	87
12	TFE	49
13	CH ₃ CN/CH ₂ Cl ₂ (3:1)	69
14	CH ₃ CN/HFIP (3:1)	92
15	CH ₃ CN/CH ₃ CH ₂ OH (1:3)	59
16	CH ₃ CH ₂ OH/HFIP (3:1)	Trace
17	CH ₃ CN/TFE (3:1)	94
18	CH₃CN/TFE (1:3)	95
19	CH ₃ CN/TFE (0.5:3.5)	76
20	CH ₃ CN/TFE (0.2:3.8)	66

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), solvent (4 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields. NMP = 1-methyl-2-pyrrolidinone. HFIP = 1,1,1,3,3,3-Hexafluoro-2-propanol. TFE = 2,2,2-trifluoroethanol.

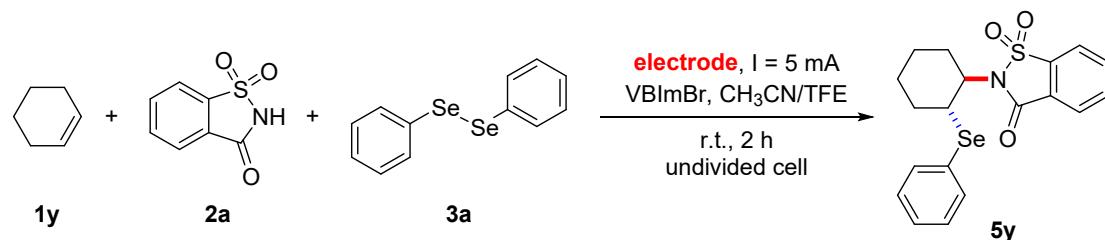
Table S9 Screening of constant current^a



Entry	Constant current (mA)	Yield (%) ^b
1	10	95
2	without current	n.r.
3	5	97
4	3	80
5	8	92
6	15	64

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 1:3), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), x mA, 2 h, r.t., undivided cell. ^b Isolated yields.

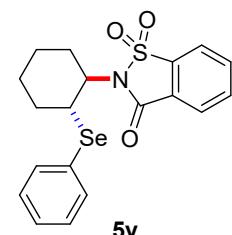
Table S10 Screening of electrodes^a



Entry	Electrode	Yield (%) ^b
1	C-Ni	97
2	C-Cu	91
3	C-Fe	96
4	C-Zn	85
5	C-C	99

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 1:3), anode (immersed surface area 8 × 5 mm²), cathode (immersed surface area 8 × 5 mm²), 5 mA, 2 h, r.t., undivided cell. ^b Isolated yields.

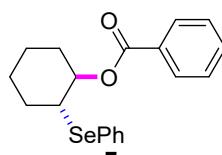
Table S11 Screening of reaction time^a

1y	2a	3a	C(+) / C(-), I = 5 mA VBImBr, CH₃CN/TFE r.t., time undivided cell		5y
Entry		Time (h)		Yield (%)^b	
1		2		99	
2		1.5		95	
3		2.5		98	
4		3		92	

^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 1:3), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), 5 mA, x h, r.t., undivided cell. ^b Isolated yields.

2.3.3 Optimization of oxyselenation of cyclohexene

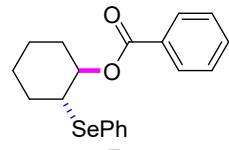
Table S12 Screening of solvents^a

1y	6a	3a	C(+) / C(-), I = 5 mA VBImBr, solvent r.t., 2.5 h undivided cell		7a
Entry		Solvent		Yield (%)^b	
1		CH ₃ CN		21	
2		DMF		trace	
3		DMA		n.r.	
4		DMSO		n.d.	
5		DCM		23	
6		DCE		trace	
7		CH ₃ CN/TFE(3:1)		53	

8	CH ₃ CN/TFE(3.5:0.5)	57
9	CH₃CN/TFE(2:2)	90
10	CH ₃ CN/TFE(1:3)	73

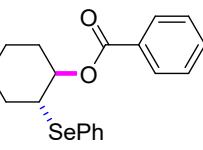
^a Reaction conditions: **1y** (0.2 mmol), **6a** (0.4 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), solvent (4 mL), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), 5 mA, 2.5 h, r.t., undivided cell. ^b Isolated yields. DCM = dichloromethane. DCE = dichloroethane.

Table S13 Screening of electrolytes^a

1y	6a	3a	C(+)/C(-), I = 5 mA electrolyte, CH₃CN/TFE r.t., 2.5 h undivided cell		7a
Entry				Electrolyte	Yield (%)^b
1				VBImBr	90
2				NaBr	86
3				Et ₄ NBr	75
4				DMMI	trace
5				Et ₄ NBF ₄	trace

^a Reaction conditions: **1y** (0.2 mmol), **6a** (0.4 mmol), **3a** (0.15 mmol), electrolyte (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 2:2), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), 5 mA, 2.5 h, r.t., undivided cell. ^b Isolated yields.

Table S14 Screening of constant current^a

1y	6a	3a	C(+)/C(-), constant current VBImBr, CH₃CN/TFE r.t., 2.5 h undivided cell		7a
Entry				Constant current (mA)	Yield (%)^b
1				5	90
2				without current	n.r.
3				3	82
4				7	88

^a Reaction conditions: **1y** (0.2 mmol), **6a** (0.4 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 2:2), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), x mA, 2.5 h, r.t., undivided cell. ^b Isolated yields.

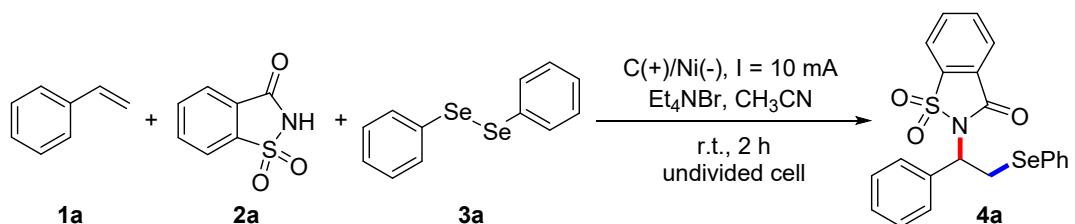
Table S15 Screening of reaction time^a

1y	6a	3a		7a
Entry	Time (h)	Yield (%) ^b		
1	2.5	90		
2	2	75		
3	1.5	79		

^a Reaction conditions: **1y** (0.2 mmol), **6a** (0.4 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 2:2), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), 5 mA, x h, r.t., undivided cell. ^b Isolated yields.

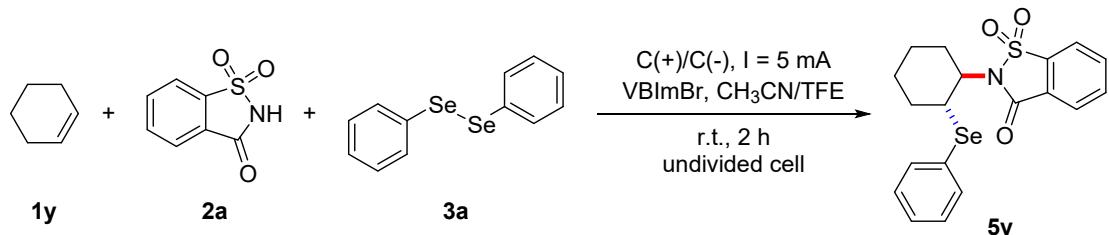
2.4 General procedure for amino- and oxyselemination of alkenes

2.4.1 General procedure for aminoselenation of activated alkenes (taking **4a** as an example)



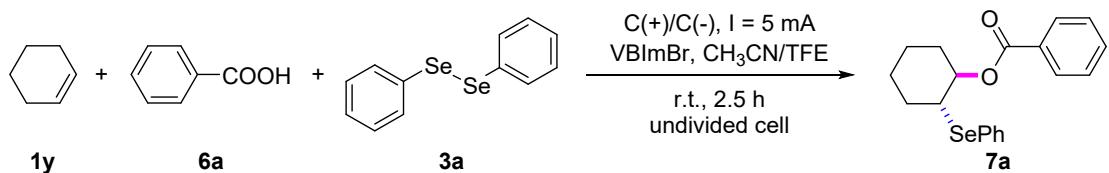
Under air, a mixture of styrene **1a** (35 µL, 0.3 mmol), saccharin **2a** (37.0 mg, 0.2 mmol), diphenyl diselenide **3a** (46.8 mg, 0.15 mmol), Et₄NBr (42.0 mg, 0.2 mmol) and CH₃CN (4.0 mL) were added in an oven dried undivided bottle (10 mL). The bottle was equipped with graphite rod as the anode and nickel plate as the cathode. The resulting mixture was stirred and electrolyzed at a constant current mode with a constant current 10 mA at ambient temperature for 2 h. When the reaction was finished, the resulting mixture and all the volatiles were evaporated under reduced pressure. The resultant residue was purified by silica gel column chromatography (eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v) to afford the desired product **4a** as a white solid (81.5 mg, 92% yield).

2.4.2 General procedure for aminoselenation of unactivated alkenes (taking **5y** as an example)



Under air, a mixture of cyclohexene **1y** (30 μL , 0.3 mmol), saccharin **2a** (37.0 mg, 0.2 mmol), diphenyl diselenide **3a** (46.8 mg, 0.15 mmol), VBImBr (46.2 mg, 0.2 mmol) and $\text{CH}_3\text{CN}/\text{TFE}$ (4 mL, v:v = 1:3) were added in an oven dried undivided bottle (10 mL). The bottle was equipped with graphite rod as the anode and the cathode. The resulting mixture was stirred and electrolyzed at a constant current mode with a constant current 5 mA at ambient temperature for 2 h. When the reaction was finished, the resulting mixture and all the volatiles were evaporated under reduced pressure. The resultant residue was purified by silica gel column chromatography (eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v) to afford the desired product **5y** as a yellow oil (83.8 mg, 99% yield).

2.4.3 General procedure for oxyselenation of unactivated alkenes (taking **7a** as an example)

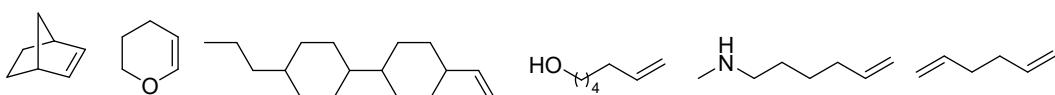


Under air, a mixture of cyclohexene **1y** (20 μL , 0.2 mmol), benzoic acid **6a** (48.9 mg, 0.4 mmol), diphenyl diselenide **3a** (46.8 mg, 0.15 mmol), VBImBr (46.2 mg, 0.2 mmol) and $\text{CH}_3\text{CN}/\text{TFE}$ (4 mL, v:v = 2:2) were added in an oven dried undivided bottle (10 mL). The bottle was equipped with graphite rod as the anode and the cathode. The resulting mixture was stirred and electrolyzed at a constant current mode with a constant current 5 mA at ambient temperature for 2.5 h. When the reaction was finished, the resulting mixture and all the volatiles were evaporated under reduced pressure. The resultant residue was purified by silica gel column chromatography (eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v) to afford the desired product **7a** as a yellow oil (64.7 mg, 90% yield).

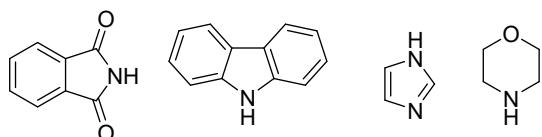
2.5 Unreacted substrates

2.5.1 Unreacted substrates for aminoselenation

Unreacted olefins:

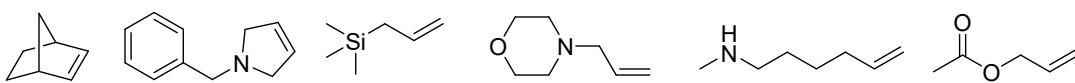


Unreacted nucleophilic reagents:

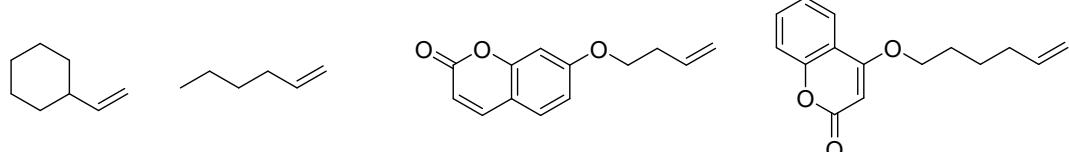


2.5.2 Unreacted substrates for oxyselemination

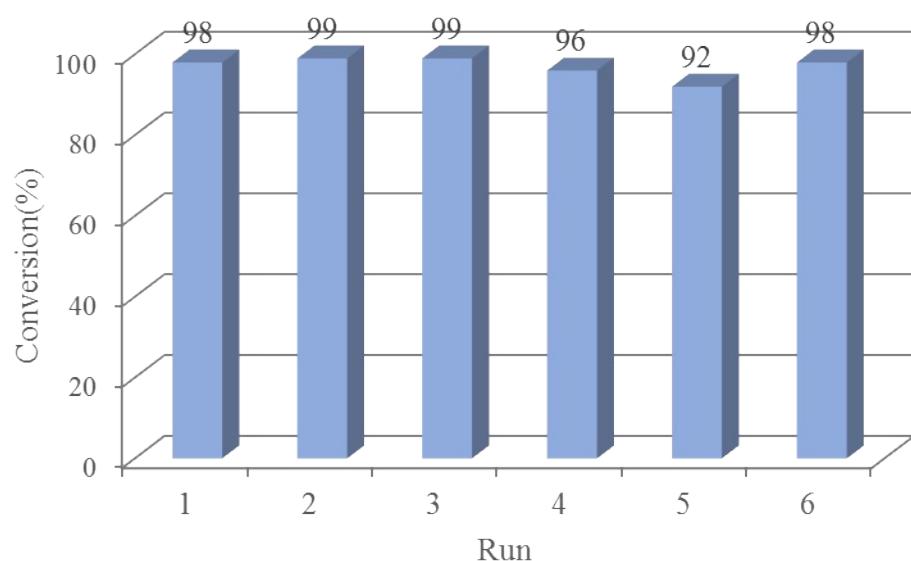
Unreacted olefins:



Regio selectivity of olefins:



2.6 Recycling experiment of VBImBr

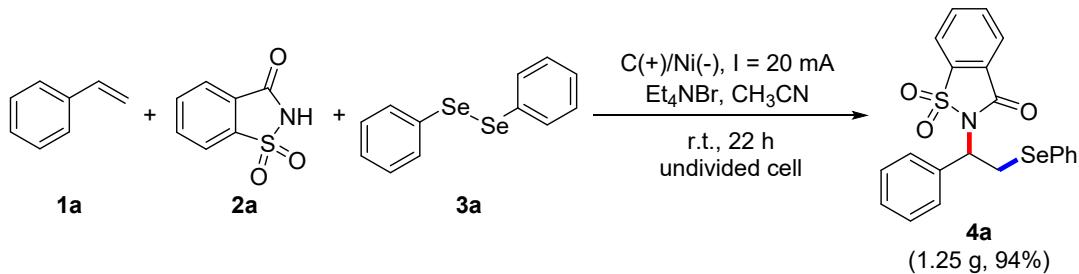


It is significant to note that the ionic liquid VBImBr could be easily recycled.

After the reaction was complete, water and ethyl acetate were added, and then the VBImBr was separated in the water layer and reused after drying in vacuo. The VBImBr was utilized repeatedly six times without any loss of activity.

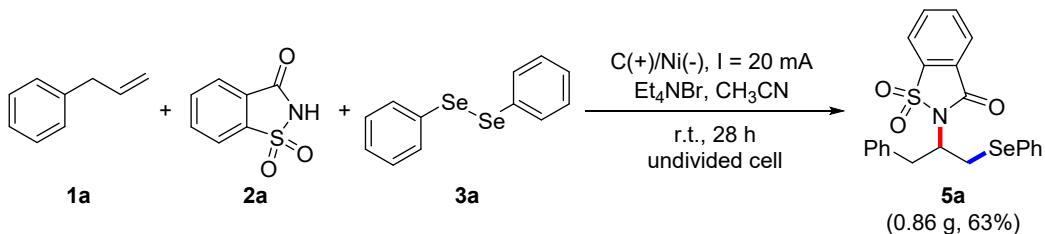
2.7 Gram-scale synthesis

2.7.1 Gram-scale synthesis of **4a** at 3 mmol scale



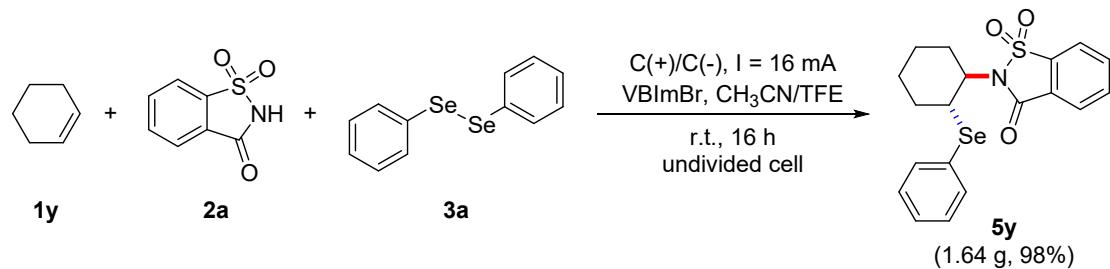
^a Reaction conditions: **1a** (4.5 mmol), **2a** (3 mmol), **3a** (2.25 mmol), Et₄NBr (3 mmol), CH₃CN (20 mL), C anode (27 mm × 15 mm × 1 mm), Ni cathode (27 mm × 15 mm × 1 mm), 20 mA, 22 h, r.t., undivided cell.

2.7.2 Gram-scale synthesis of **5a** at 3 mmol scale



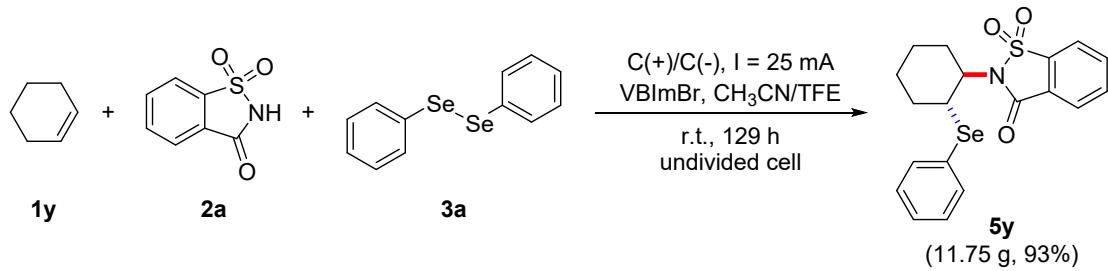
^a Reaction conditions: **allylbenzene** (4.5 mmol), **2a** (3 mmol), **3a** (2.25 mmol), Et₄NBr (3 mmol), CH₃CN (20 mL), C anode (27 mm × 15 mm × 1 mm), Ni cathode (27 mm × 15 mm × 1 mm), 20 mA, 28 h, r.t., undivided cell.

2.7.3 Gram-scale synthesis of **5y** at 4 mmol scale



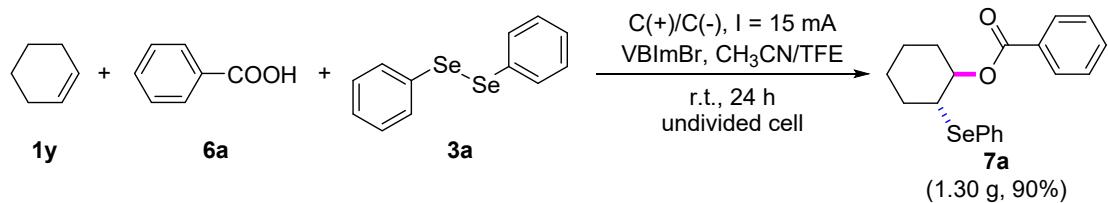
^a Reaction conditions: **1y** (6 mmol), **2a** (4 mmol), **3a** (3 mmol), VBImBr (4 mmol), CH₃CN/TFE (40 mL, v:v = 1:3), C anode (25 mm × 15 mm × 1 mm), C cathode (25 mm × 15 mm × 1 mm), 16 mA, 16 h, r.t., undivided cell.

2.7.4 Gram-scale synthesis of **5y** at 30 mmol scale



^a Reaction conditions: **1y** (135 mmol), **2a** (30 mmol), **3a** (15 mmol), VBImBr (30 mmol), CH₃CN/TFE (260 mL, v:v = 7:6), C anode (45 mm × 25 mm × 1 mm), C cathode (45 mm × 25 mm × 1 mm), 25 mA, 129 h, r.t., undivided cell.

2.7.5 Gram-scale synthesis of **7a** at 4 mmol scale

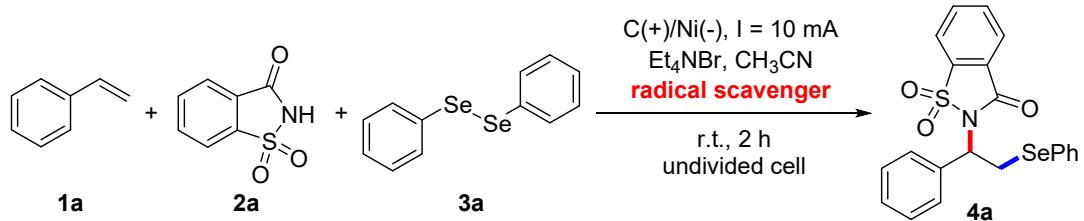


^a Reaction conditions: **1y** (4 mmol), **6a** (8 mmol), **3a** (3 mmol), VBImBr (4 mmol), CH₃CN/TFE (40 mL, v:v = 1:1), C anode (25 mm × 15 mm × 1 mm), C cathode (25 mm × 15 mm × 1 mm), 15 mA, 24 h, r.t., undivided cell.

3. Mechanistic studies

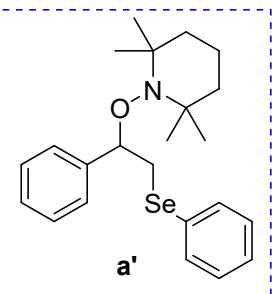
3.1 Radical trapping experiments

3.1.1 Table S16 of radical trapping experiments of aminoselenation of styrene^a

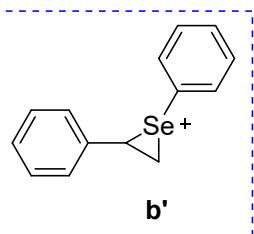


Entry	Radical scavenger (mmol)	Yield of 4a (%) ^b
1	none	92
2	BHT (0.4)	98
3	activated carbon (0.8)	92
4	TEMPO (0.4)	79

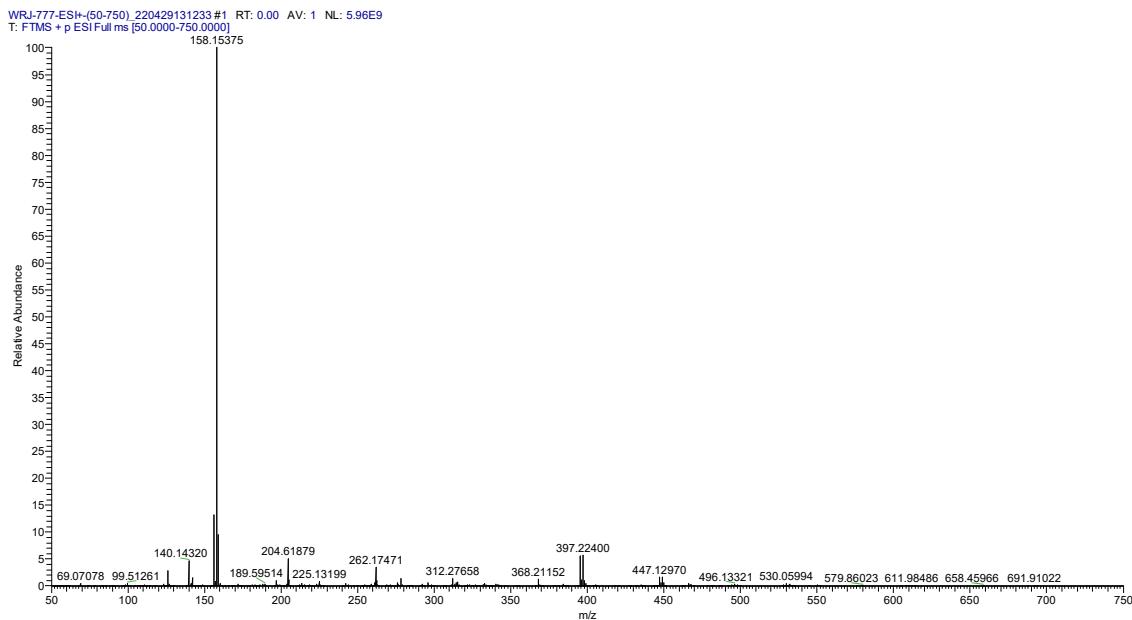
^a Reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), Et₄NBr (0.2 mmol), radical scavenger, CH₃CN (4.0 mL), C anode (immersed surface area 8 × 5 mm²), Ni cathode (immersed surface area 8 × 5 mm²), 10 mA, 2 h, r.t., undivided cell. ^b Isolated yields.



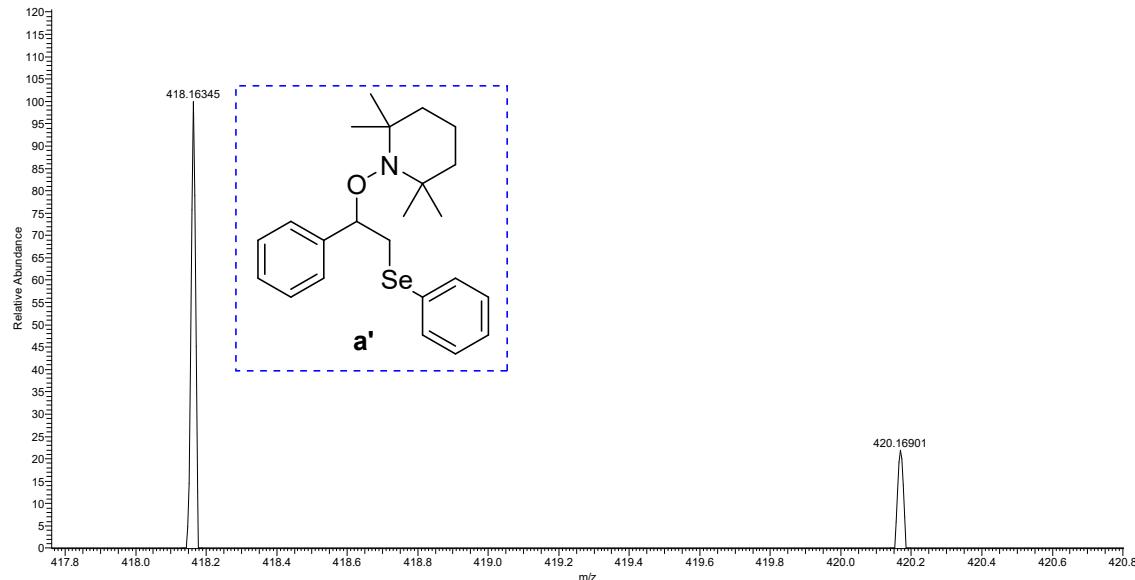
Detected by HRMS of **a'** calcd for $C_{23}H_{32}NOSe^+$
 $[M+H]^+$: 418.1644; Found: 418.1635.

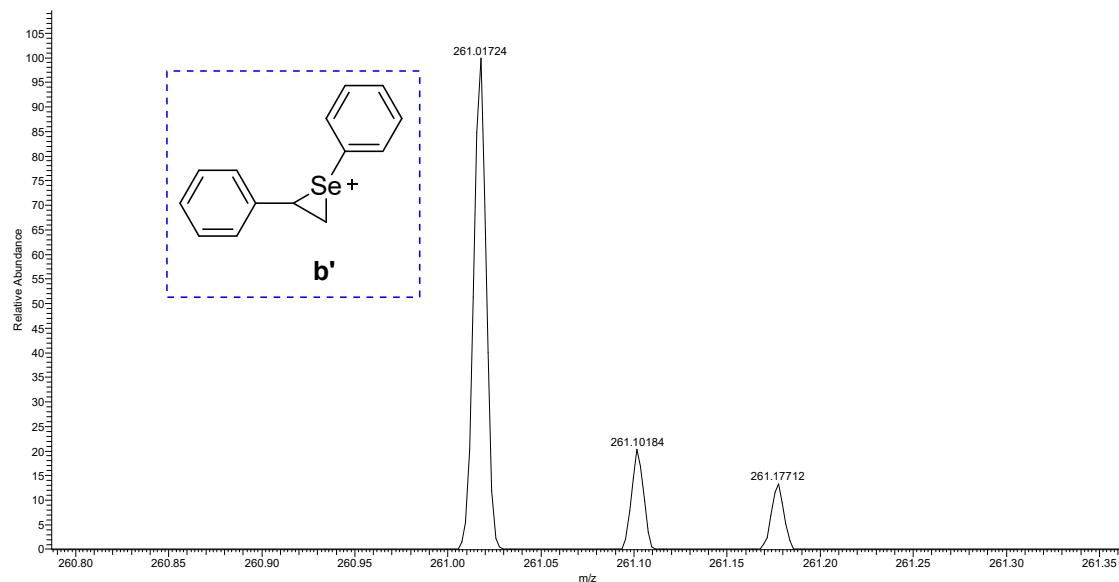


Detected by HRMS of **b'** calcd for $C_{14}H_{13}Se^+$
 $[M]^+$: 261.0177; Found: 261.0172.

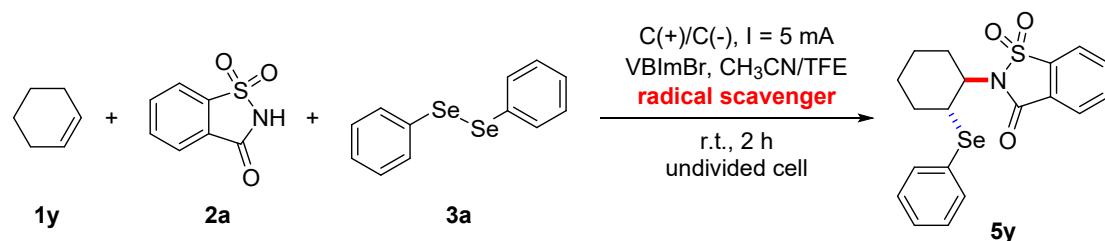


WRJ-777-ESI+-{50-750}_220429131233#1 RT: 0.00 AV: 1 NL: 6.33E6
T: FTMS + p ESI Full ms [50.000-750.000]



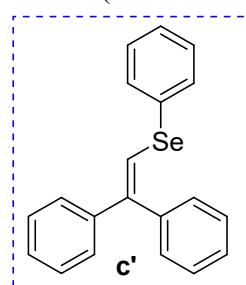


3.1.2 Table S17 of radical trapping experiments of aminoselenation of cyclohexene^a

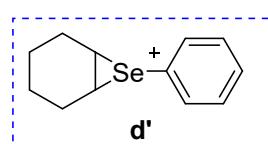


Entry	Radical scavenger (mmol)	Yield of 5y (%) ^b
1	none	99
2	BHT (0.4)	91
3	activated carbon (0.8)	83
5	1,1-diphenylethylene (0.4)	87
7	TEMPO (0.4)	20
8	TEMPO (0.8)	trace

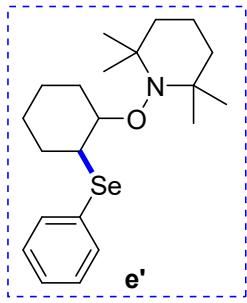
^a Reaction conditions: **1y** (0.3 mmol), **2a** (0.2 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), radical scavenger, CH₃CN/TFE (4 mL, v:v = 1:3), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), 5 mA, 2 h, r.t., undivided cell. ^b Isolated yields.



Detected by HRMS of **c'** calcd for C₂₀H₁₇Se
[M+H]⁺: 337.0490; Found: 337.0485.

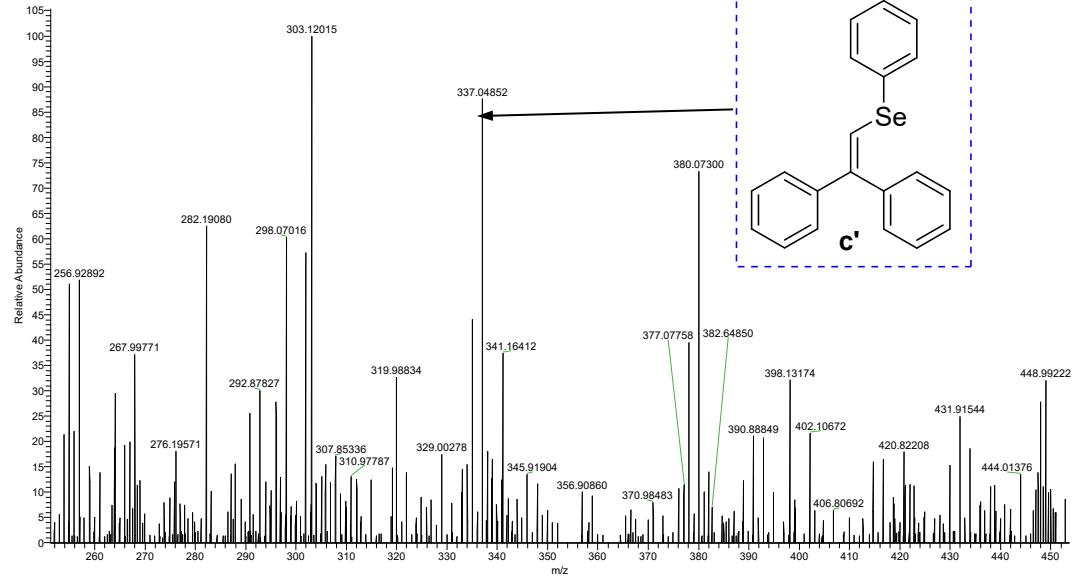


Detected by HRMS of **d'** calcd for C₁₂H₁₅Se⁺
[M]⁺: 239.0334; Found: 239.0329.

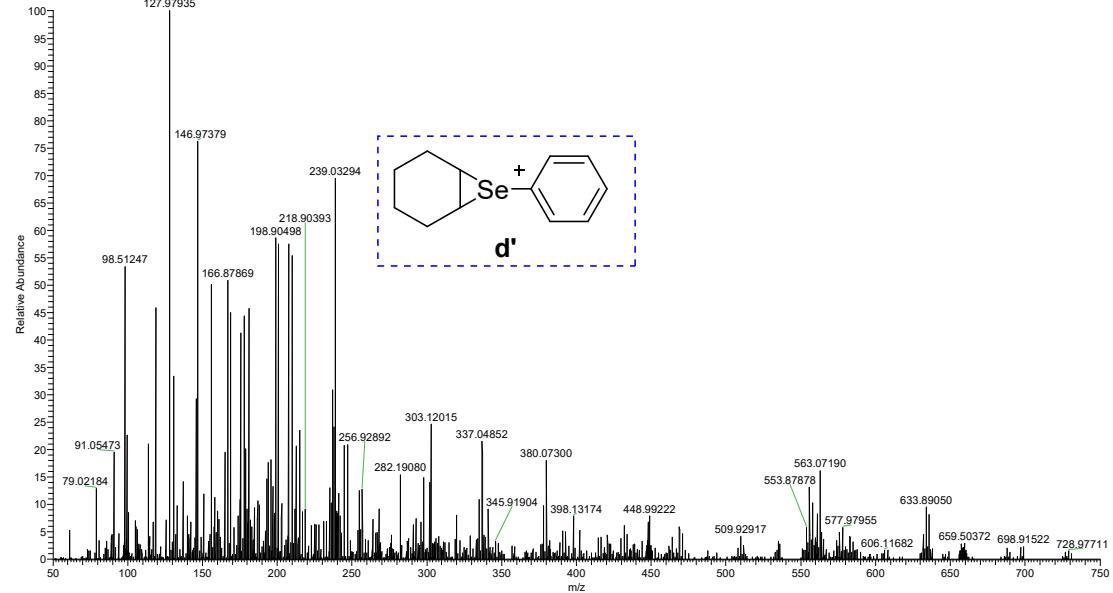


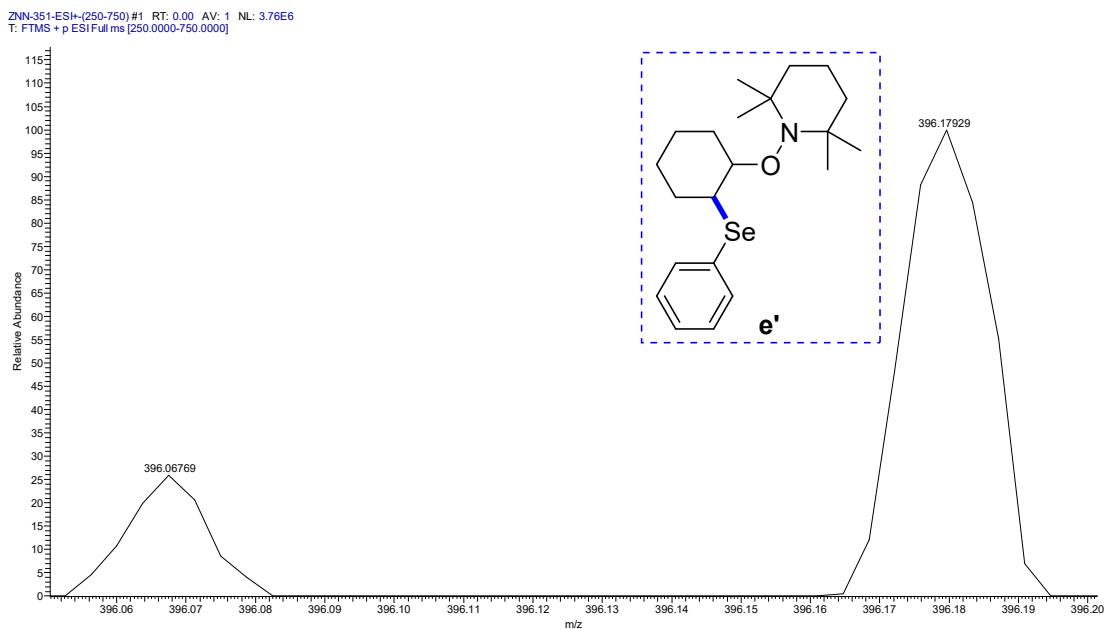
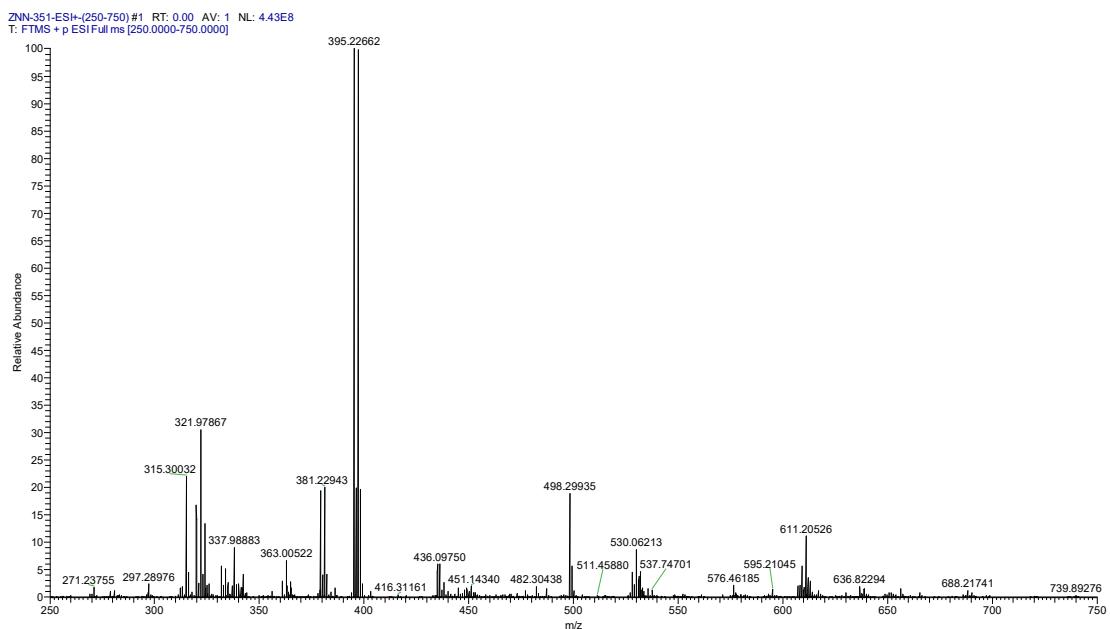
Detected by HRMS of **e'** calcd for C₂₁H₃₃NOSe
[M+H]⁺: 396.1800; Found: 396.1793.

ZNN-374-ESI+(50-750) #1 RT: 0.00 AV: 1 NL: 2.77E7
T: FTMS + p ESI Full ms [50.0000-750.0000]

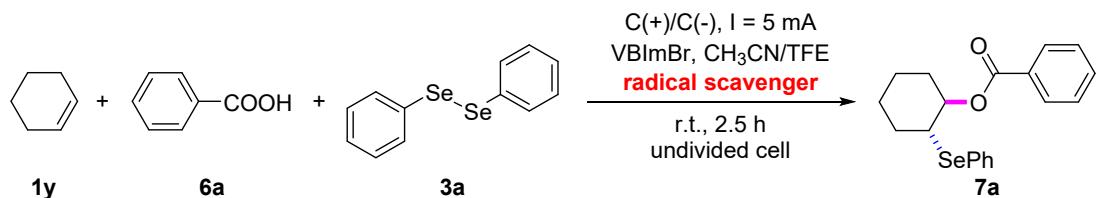


ZNN-374-ESI+(50-750) #1 RT: 0.00 AV: 1 NL: 1.12E8
T: FTMS + p ESI Full ms [50.0000-750.0000]





3.1.3 Table S18 of radical trapping experiments of oxyselenation of cyclohexene^a



Entry	Radical scavenger (mmol)	Yield of 7a (%) ^b
1	none	90
2	BHT (0.4)	92
3	activated carbon (0.8)	75
4	1,1-diphenylethylene (0.4)	84
5	TEMPO (0.4)	trace

^a Reaction conditions: **1y** (0.2 mmol), **6a** (0.4 mmol), **7a** (0.15 mmol), VBImBr (0.2 mmol), radical scavenger, CH₃CN/TFE (4 mL, v:v = 2:2), C anode (immersed surface area 8 × 5 mm²), C cathode (immersed surface area 8 × 5 mm²), 5 mA, 2.5 h, r.t., undivided cell. ^b Isolated yields.

3.2 Cyclic voltammetry experiments

3.2.1 Cyclic voltammetry experiments for aminoselenation of styrene

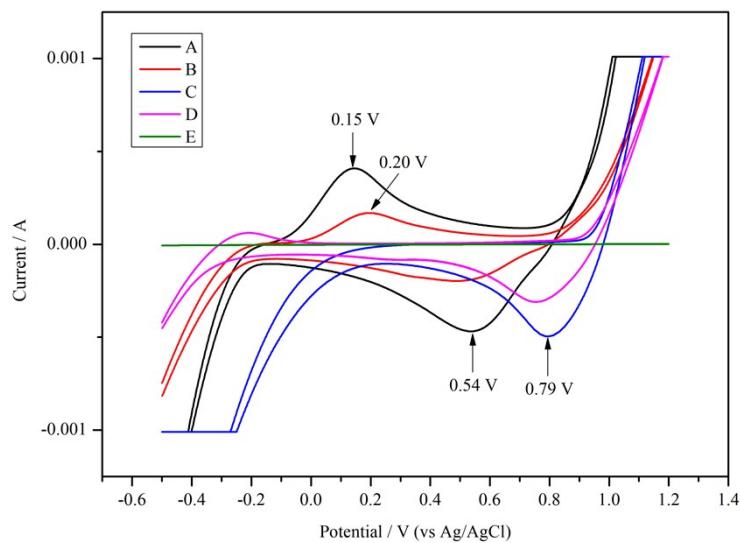


Figure S1 Cyclic voltammograms using Pt disk as work electrode, a glassy carbon disk and Ag/AgCl as counter and reference electrode, respectively, at 100 mV/s scan rate: **A:** **3a** (0.15 mmol), Et₄NBr (0.2 mmol), CH₃CN (4.0 mL); **B:** **1a** (0.3 mmol), **3a** (0.15 mmol), Et₄NBr (0.2 mmol), CH₃CN (4.0 mL); **C:** **2a** (0.2 mmol), Et₄NBr (0.2 mmol), CH₃CN (4.0 mL); **D:** Et₄NBr (0.2 mmol), CH₃CN (4.0 mL). **E:** CH₃CN (4.0 mL).

3.2.2 Cyclic voltammetry experiments for aminoselenation of cyclohexene

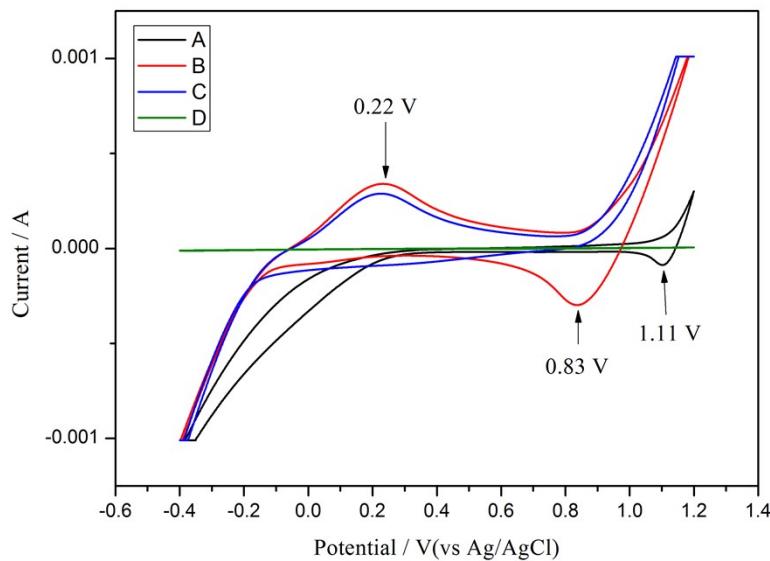


Figure S2 Cyclic voltammograms using Pt disk as work electrode, a glassy carbon disk and Ag/AgCl as counter and reference electrode, respectively, at 100 mV/s scan rate: **A:** **2a** (0.2 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 1:3). **B:** **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 1:3). **C:** **1y** (0.3 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 1:3). **D:** CH₃CN/TFE (4 mL, v:v = 1:3).

3.2.3 Cyclic voltammetry experiments for oxyselenation of cyclohexene

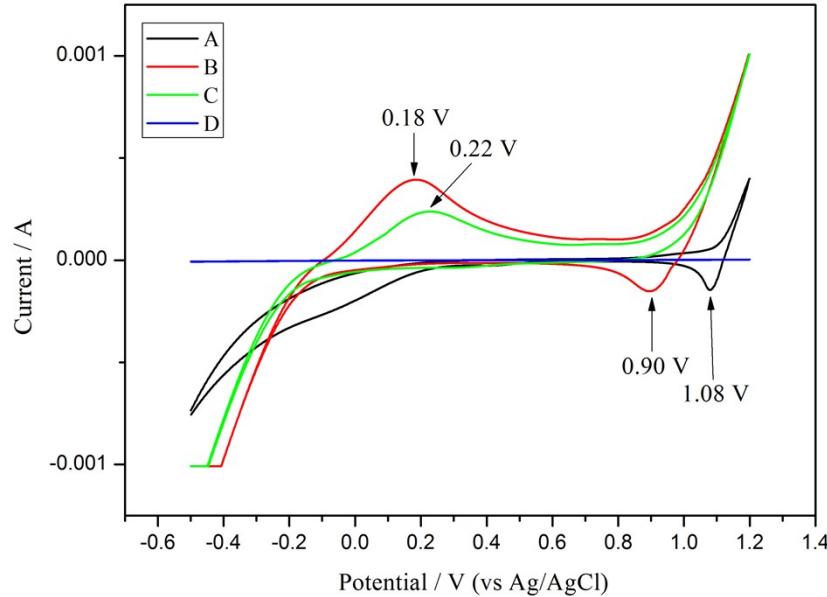
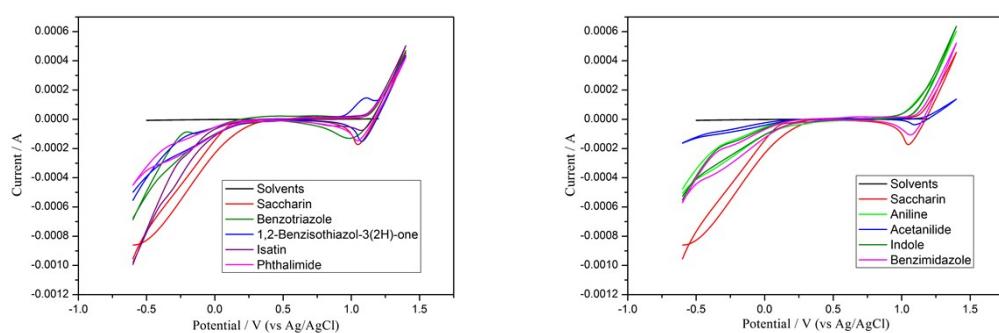


Figure S3 Cyclic voltammograms using Pt disk as work electrode, a glassy carbon disk and Ag/AgCl as counter and reference electrode, respectively, at 100 mV/s scan rate: **A:** **6a** (0.4 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 2:2). **B:** **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 2:2). **C:** **1y** (0.2 mmol), **3a** (0.15 mmol), VBImBr (0.2 mmol), CH₃CN/TFE (4 mL, v:v = 2:2). **D:** CH₃CN/TFE (4 mL, v:v = 2:2).

3.2.4 Cyclic voltammetry for various N-centered nucleophiles



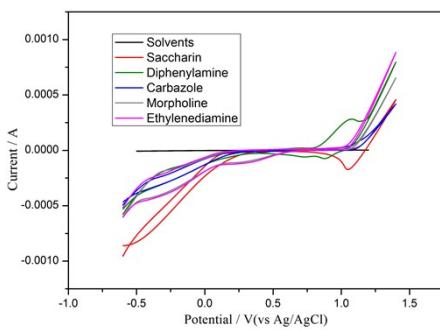


Figure S4 Cyclic voltammograms using Pt disk as work electrode, a glassy carbon disk and Ag/AgCl as counter and reference electrode, respectively, at 100 mV/s scan rate.

3.3 H₂ detection experiments

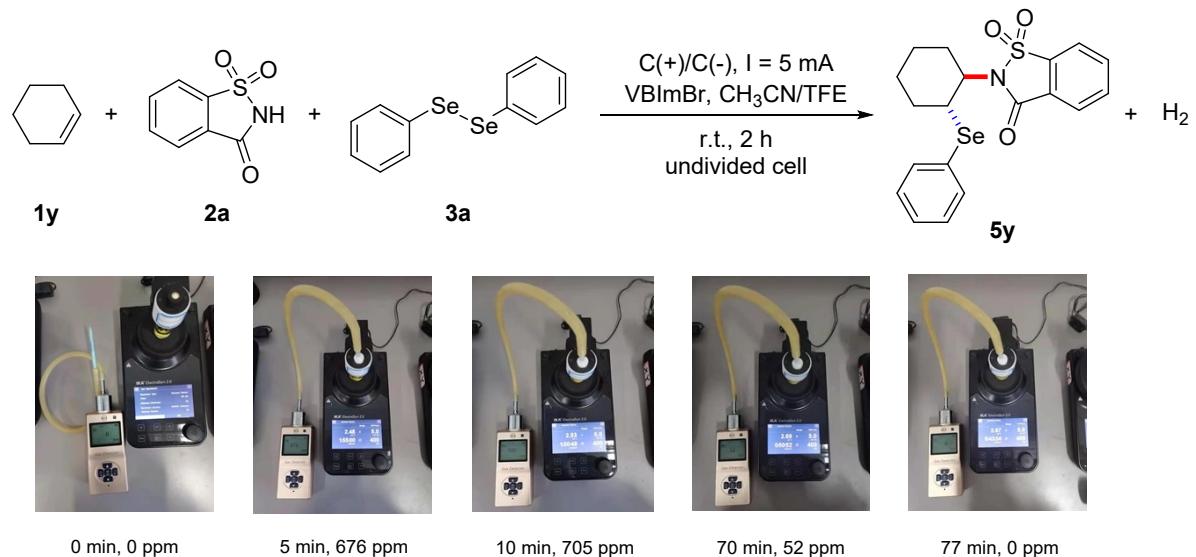
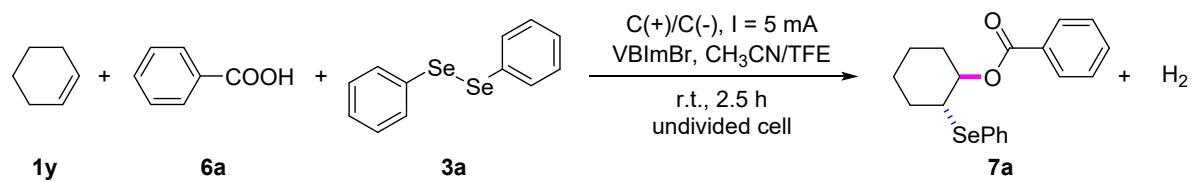


Figure S5 H₂ detection experiment by a H₂ detector at different reaction time

In order to demonstrate the release of H₂ during electrochemical aminoselenation of cyclohexene, the model reaction of cyclohexene (**1y**), saccharin (**2a**) and diphenyl diselenide (**3a**) was monitored by a H₂ detector under standard conditions. Just as shown in Figure S5, as the reaction proceeded, the H₂ was observed clearly and the concentration increased gradually.



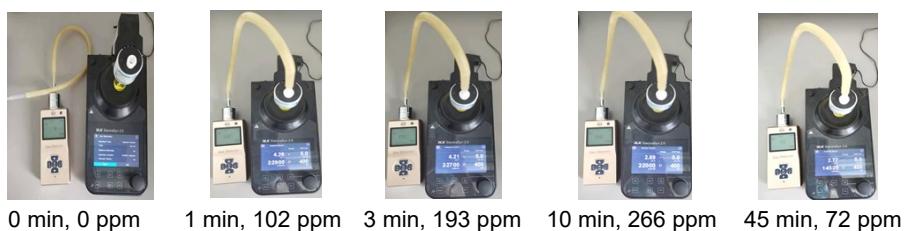
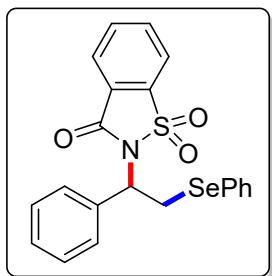


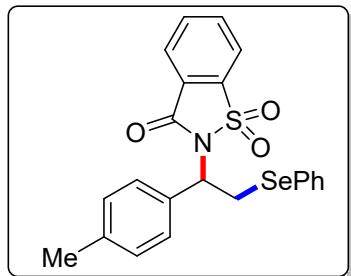
Figure S6 H₂ detection experiment by a H₂ detector at different reaction time

In order to demonstrate the release of H₂ during electrochemical oxyselenation of cyclohexene, the model reaction of cyclohexene (**1y**), benzoic acid (**6a**) and diphenyl diselenide (**3a**) was monitored by a H₂ detector under standard conditions. Just as shown in Figure S6, as the reaction proceeded, the H₂ was observed clearly and the concentration increased gradually.

4. Analytical data

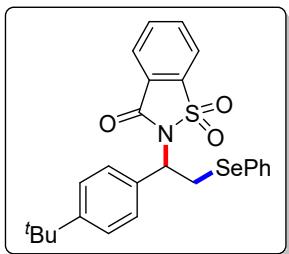


2-(1-phenyl-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4a): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 81.8 mg, 92% yield. White solid. m.p.: 95.8-97.3 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98 (d, *J* = 7.3 Hz, 1H), 7.88-7.76 (m, 3H), 7.60-7.56 (m, 4H), 7.38-7.33 (m, 3H), 7.27-7.26 (m, 3H), 5.42 (t, *J* = 8.1 Hz, 1H), 4.11 (dd, *J* = 12.9, 8.4 Hz, 1H), 3.84 (dd, *J* = 12.9, 7.7 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 137.4, 136.7, 134.8, 134.4, 133.8, 129.3, 129.1, 128.9, 128.7, 128.6, 127.7, 127.2, 125.2, 120.8, 57.9, 29.1.

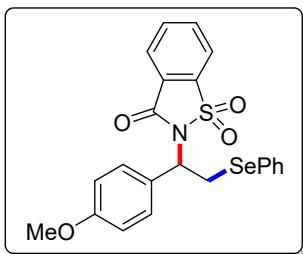


2-(2-(phenylselanyl)-1-(p-tolyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4b): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1,

v/v). 73.5 mg, 76% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.97-7.95 (m, 1H), 7.85-7.74 (m, 3H), 7.57-7.55 (m, 2H), 7.47 (d, *J* = 8.1 Hz, 2H), 7.26-7.24 (m, 3H), 7.16 (d, *J* = 8.1 Hz, 2H), 5.38 (t, *J* = 8.1 Hz, 1H), 4.06 (dd, *J* = 12.8, 8.3 Hz, 1H), 3.83 (dd, *J* = 12.8, 7.9 Hz, 1H), 2.33 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 138.8, 137.5, 134.8, 134.3, 133.8, 133.6, 129.4, 129.3, 129.2, 128.6, 127.7, 127.2, 125.2, 120.8, 57.6, 29.1, 21.3.

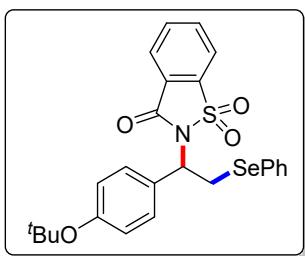


2-(1-(4-(*tert*-butyl)phenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2*H*)-one 1,1-dioxide (4c): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 88 mg, 88% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.97-7.94 (m, 1H), 7.86-7.72 (m, 3H), 7.59-7.56 (m, 2H), 7.54-7.52 (m, 2H), 7.41-7.36 (m, 2H), 7.27-7.24 (m, 3H), 5.41 (t, *J* = 7.5 Hz, 1H), 4.14 (dd, *J* = 13.0, 8.8 Hz, 1H), 3.83 (dd, *J* = 12.9, 7.4 Hz, 1H), 1.31 (s, 9H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 151.7, 137.4, 134.8, 134.3, 133.9, 133.8, 129.2, 129.2, 128.3, 127.7, 127.3, 125.6, 125.2, 120.9, 57.8, 34.7, 31.4, 29.3.

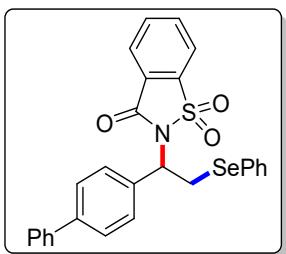


2-(1-(4-methoxyphenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2*H*)-one 1,1-dioxide (4d): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 62.1 mg, 66% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.95 (d, *J* = 7.5 Hz, 1H), 7.84-7.73 (m, 3H), 7.56-7.49 (m, 4H), 7.25-7.23 (m, 3H), 6.86 (d, *J* = 8.7 Hz, 2H), 5.36 (t, *J* = 8.1 Hz, 1H), 4.03 (dd, *J* = 12.8, 8.1 Hz, 1H), 3.82 (dd, *J* = 12.8, 8.1 Hz, 1H), 3.78 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.9, 158.8, 137.5,

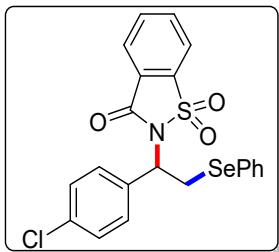
134.8, 134.4, 133.8, 130.1, 129.3, 129.2, 128.6, 127.7, 127.3, 125.2, 120.8, 114.0, 57.5, 55.4, 29.3.



2-(1-(4-(*tert*-butoxy)phenyl)-2-(phenylselanyl)ethyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4e): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 85.3 mg, 83% yield. White solid. m.p.: 136.6-138.6 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.96 (m, 1H), 7.87-7.76 (m, 3H), 7.56-7.54 (m, 2H), 7.46-7.46 (m, 2H), 7.26-7.23 (m, 3H), 6.95-6.92 (m, 2H), 5.36 (t, *J* = 8.0 Hz, 1H), 4.08 (dd, *J* = 12.8, 8.7 Hz, 1H), 3.77 (dd, *J* = 12.8, 7.5 Hz, 1H), 1.33 (s, 9H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 156.0, 137.5, 134.8, 134.4, 133.9, 131.3, 129.4, 129.3, 129.2, 127.7, 127.3, 125.3, 123.8, 120.9, 78.8, 57.6, 29.4, 29.0. **HRMS** (ESI-Orbitrap) m/z calcd for C₂₅H₂₅NO₄SSeNa [M+Na]⁺: 538.0562; found: 538.0562.

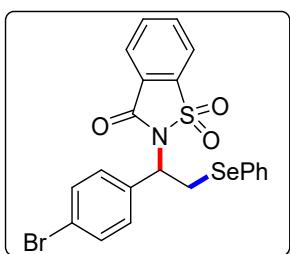


2-(1-([1,1'-biphenyl]-4-yl)-2-(phenylselanyl)ethyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4f): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 96.1 mg, 93% yield. White solid. m.p.: 126.2-128.1 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.99-7.97 (m, 1H), 7.88-7.75 (m, 3H), 7.67-7.65 (m, 2H), 7.60-7.57 (m, 6H), 7.45-7.41 (m, 2H), 7.37-7.33 (m, 1H), 7.27-7.26 (m, 3H), 5.45 (t, *J* = 8.1 Hz, 1H), 4.13 (dd, *J* = 12.9, 8.3 Hz, 1H), 3.89 (dd, *J* = 12.9, 7.9 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 141.7, 140.6, 137.5, 135.7, 134.9, 134.4, 133.9, 129.3, 129.1, 129.1, 128.9, 127.8, 127.6, 127.4, 127.2, 125.3, 120.9, 57.7, 29.1.



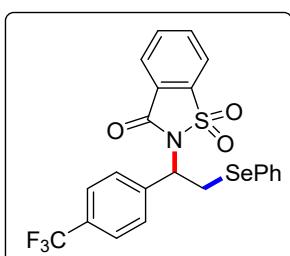
2-(1-(4-chlorophenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one

1,1-dioxide (4g): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 82.7 mg, 87% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.97-7.95 (m, 1H), 7.87-7.75 (m, 3H), 7.55-7.49 (m, 4H), 7.32-7.24 (m, 5H), 5.35 (t, *J* = 8.1 Hz, 1H), 4.03 (dd, *J* = 12.9, 7.9 Hz, 1H), 3.81 (dd, *J* = 12.9, 8.2 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 137.4, 135.1, 135.0, 134.8, 134.5, 133.9, 130.2, 129.4, 128.8, 128.8, 127.9, 127.1, 125.3, 120.9, 57.2, 28.8.



2-(1-(4-bromophenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one

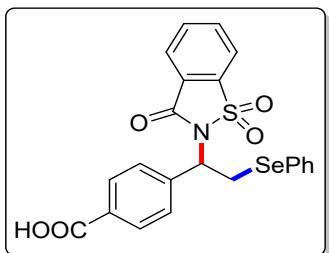
1,1-dioxide (4h): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 84.5 mg, 81% yield. White solid. m.p.: 99.3-105.5 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.96 (m, 1H), 7.87-7.76 (m, 3H), 7.57-7.54 (m, 2H), 7.35-7.24 (m, 6H), 7.03-6.98 (m, 1H), 5.35 (t, *J* = 8.1 Hz, 1H), 4.05 (dd, *J* = 13.0, 8.2 Hz, 1H), 3.79 (dd, *J* = 13.0, 7.9 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.7, 137.3, 135.6, 134.9, 134.4, 133.9, 131.8, 130.5, 129.3, 128.7, 127.8, 127.0, 125.3, 123.0, 120.9, 57.2, 28.7.



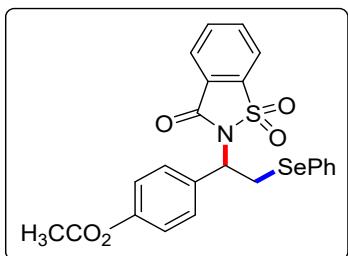
2-(2-(phenylselanyl)-1-(4-(trifluoromethyl)phenyl)ethyl)benzo[d]isothiazol-

3(2H)-one 1,1-dioxide (4i): Known compound^[3]. (Eluent: petroleum ether (60-90

$^{\circ}\text{C}$)/EtOAc = 6:1, v/v). 54.2 mg, 53% yield. Colorless oil. $^{\text{1H}}$ NMR (CDCl_3 , 400 MHz) δ 7.98 (d, J = 7.4 Hz, 1H), 7.89-7.77 (m, 3H), 7.68 (d, J = 8.2 Hz, 2H), 7.58 (d, J = 8.4 Hz, 2H), 7.54-7.51 (m, 2H), 7.29-7.22 (m, 3H), 5.40 (t, J = 8.1 Hz, 1H), 4.05 (dd, J = 13.0, 7.8 Hz, 1H), 3.84 (dd, J = 13.0, 8.3 Hz, 1H); $^{\text{13C}}$ NMR (CDCl_3 , 100 MHz) δ 158.8, 140.6, 137.4, 135.1, 134.5, 134.0, 130.9 (q, J_{CF} = 32.4 Hz), 129.4, 129.2, 128.6, 128.0, 127.0, 125.6 (q, J_{CF} = 3.7 Hz), 125.4, 124.0 (q, J_{CF} = 270.7 Hz), 121.0, 57.4, 28.6; $^{\text{19F}}$ NMR (CDCl_3 , 376 MHz) δ -62.7.

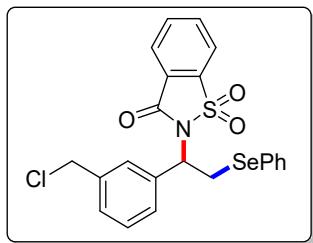


4-(1-(1,1-dioxido-3-oxobenzo[d]isothiazol-2(3H)-yl)-2-(phenylselanyl)ethyl)benzoic acid (4j): Known compound^[3]. (Eluent: dichloromethane/methyl alcohol = 20:1, v/v). 59.4 mg, 61% yield. White solid. m.p.: 200.3-201.0 $^{\circ}\text{C}$. $^{\text{1H}}$ NMR ($\text{DMSO-}d_6$, 400 MHz) δ 13.00 (s, 1H), 8.27 (d, J = 7.6 Hz, 1H), 8.07-7.92 (m, 5H), 7.66 (d, J = 8.4 Hz, 2H), 7.52-7.49 (m, 2H), 7.26-7.24 (m, 3H), 5.46 (t, J = 8.0 Hz, 1H), 4.06 (dd, J = 12.8, 9.0 Hz, 1H), 3.92 (dd, J = 12.8, 7.2 Hz, 1H); $^{\text{13C}}$ NMR ($\text{DMSO-}d_6$, 100 MHz) δ 167.0, 158.5, 141.7, 136.4, 136.1, 135.4, 132.3, 130.8, 129.5, 129.3, 128.9, 128.3, 127.2, 125.9, 125.3, 121.5, 56.0, 27.9.

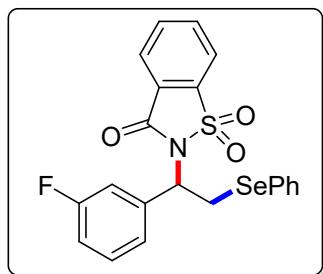


4-(1-(1,1-dioxido-3-oxobenzo[d]isothiazol-2(3H)-yl)-2-(phenylselanyl)ethyl)phenyl acetate (4k): Known compound^[3]. (Eluent: petroleum ether (60-90 $^{\circ}\text{C}$)/EtOAc = 4:1, v/v). 81.9 mg, 82% yield. Colorless oil. $^{\text{1H}}$ NMR (CDCl_3 , 400 MHz) δ 7.95-7.93 (m, 1H), 7.85-7.73 (m, 3H), 7.61-7.54 (m, 4H), 7.27-7.23 (m, 3H), 7.09-7.05 (m, 2H), 5.36 (t, J = 8.0 Hz, 1H), 4.10 (dd, J = 13.0, 8.6 Hz,

1H), 3.78 (dd, $J = 13.0, 7.6$ Hz, 1H), 2.27 (s, 3H); **^{13}C NMR** (CDCl_3 , 100 MHz) δ 169.3, 158.8, 150.9, 137.4, 134.9, 134.4, 134.4, 134.0, 130.0, 129.3, 128.9, 127.8, 127.2, 125.3, 121.8, 120.9, 57.4, 29.1, 21.3.

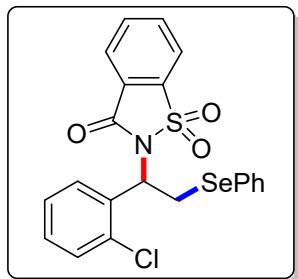


2-(1-(3-(chloromethyl)phenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4l): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 74.1 mg, 75% yield. Colorless oil. **^1H NMR** (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 1H), 7.87-7.75 (m, 3H), 7.57-7.53 (m, 4H), 7.37-7.35 (m, 2H), 7.27-7.24 (m, 3H), 5.38 (t, $J = 8.1$ Hz, 1H), 4.56 (s, 2H), 4.06 (dd, $J = 12.9, 8.24$ Hz, 1H), 3.81 (dd, $J = 12.9, 7.9$ Hz, 1H); **^{13}C NMR** (CDCl_3 , 100 MHz) δ 158.8, 138.0, 137.4, 136.9, 134.9, 134.4, 133.9, 129.4, 129.3, 129.2, 129.1, 128.9, 128.9, 127.8, 127.1, 125.3, 120.9, 57.5, 45.8, 29.0; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{22}\text{H}_{18}\text{ClNO}_3\text{SSeNa} [\text{M}+\text{Na}]^+$: 513.9753; found: 513.9750.



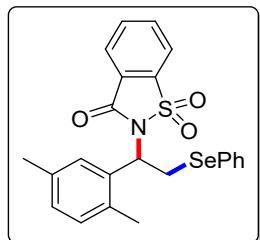
2-(1-(3-fluorophenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4m): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 70.6 mg, 77% yield. White solid. m.p.: 112-113 °C. **^1H NMR** (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 1H), 7.87-7.75 (m, 3H), 7.55-7.52 (m, 2H), 7.45 (s, 4H), 7.26-7.22 (m, 2H), 5.33 (t, $J = 8.1$ Hz, 1H), 4.02 (dd, $J = 12.9, 7.9$ Hz, 1H), 3.81 (dd, $J = 12.9, 8.3$ Hz, 1H); **^{13}C NMR** (CDCl_3 , 100 MHz) δ 164.0, 160.2 (d, $J_{CF} = 271.0$ Hz), 139.2 (d, $J_{CF} = 7.2$ Hz), 137.4, 135.0, 134.5, 134.0, 130.2 (d, $J_{CF} = 8.1$ Hz), 129.4, 128.8, 127.9, 127.1, 125.4, 124.4 (d, $J_{CF} = 2.9$ Hz), 120.9, 116.0 (d, $J_{CF} = 8.0$ Hz), 115.8 (d, $J_{CF} =$

9.0 Hz), 57.3, 28.9; **¹⁹F NMR** (CDCl₃, 376 MHz) δ -112.0; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₁H₁₇FNO₃SSe [M+H]⁺: 462.0073; found: 462.0077.

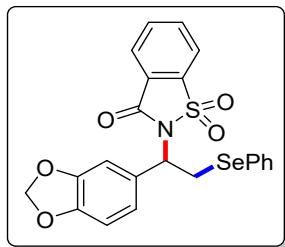


2-(1-(2-chlorophenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one

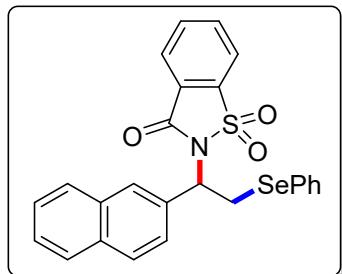
1,1-dioxide (4n): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 70.3 mg, 74% yield. White solid. m.p.: 167.6-170.5 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 8.06-8.03 (m, 1H), 7.83-7.75 (m, 4H), 7.62-7.60 (m, 2H), 7.40-7.38 (m, 1H), 7.32-7.25 (m, 5H), 6.01 (t, J = 8.1 Hz, 1H), 4.01 (dd, J = 13.0, 8.4 Hz, 1H), 3.75 (dd, J = 13.0, 7.8 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.1, 137.7, 135.0, 134.4, 134.4, 134.2, 133.9, 130.2, 130.0, 129.4, 129.3, 128.9, 127.9, 127.3, 127.0, 125.4, 120.8, 54.2, 29.2.



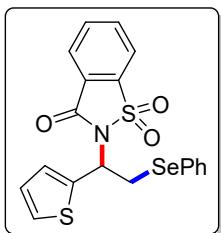
2-(1-(2,5-dimethylphenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4o): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 61.4 mg, 65 % yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.02 (m, 1H), 7.83-7.77 (m, 3H), 7.57-7.55 (m, 2H), 7.52 (s, 1H), 7.27-7.23 (m, 3H), 7.04 (s, 2H), 5.71 (t, J = 8.0 Hz, 1H), 4.04 (dd, J = 12.9, 8.2 Hz, 1H), 3.72 (dd, J = 12.9, 7.8 Hz, 1H), 2.31 (s, 3H), 2.22 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.2, 137.8, 135.9, 134.9, 134.3, 134.2, 134.0, 133.9, 130.7, 129.7, 129.4, 129.2, 128.6, 127.8, 127.1, 125.4, 120.7, 54.3, 30.1, 21.4, 19.0. **HRMS** (ESI-Orbitrap) m/z calcd for C₂₃H₂₁NO₃SSeNa [M+Na]⁺: 494.0300; found: 494.0303.



2-(1-(benzo[d][1,3]dioxol-5-yl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4p): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 81.7 mg, 84% yield. White solid. m.p. 127.4-128.6 °C; **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.96 (m, 1H), 7.87-7.76 (m, 3H), 7.56-7.53 (m, 2H), 7.27-7.24 (m, 3H), 7.09 (d, *J* = 1.8 Hz, 1H), 7.03 (dd, *J* = 8.1, 1.8 Hz, 1H), 6.76 (d, *J* = 8.1 Hz, 1H), 5.94-5.93 (m, 2H), 5.31 (t, *J* = 8.1 Hz, 1H), 3.99 (dd, *J* = 12.8, 8.0 Hz, 1H), 3.79 (dd, *J* = 12.9, 8.2 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 148.0, 147.9, 137.4, 134.9, 134.4, 133.8, 130.4, 129.3, 129.0, 127.8, 127.2, 125.3, 122.7, 120.9, 109.1, 108.2, 101.4, 57.9, 29.3; **HRMS** (ESI-Orbitrap) *m/z* calcd for C₂₂H₁₇NO₅SSeNa [M+Na]⁺: 509.9885; found: 509.9887.

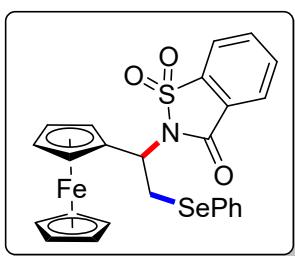


2-(1-(naphthalen-2-yl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4q): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 92.2 mg, 94% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.03 (d, 1H), 7.97-7.94 (m, 1H), 7.85-7.69 (m, 7H), 7.59-7.57 (m, 2H), 7.49-7.47 (m, 2H), 7.26-7.24 (m, 3H), 5.59 (t, *J* = 8.1 Hz, 1H), 4.19 (dd, *J* = 12.9, 8.2 Hz, 1H), 3.97 (dd, *J* = 12.9, 8.0 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 137.4, 134.8, 134.4, 134.1, 133.9, 133.4, 133.1, 129.3, 129.1, 128.5, 128.4, 128.2, 127.8, 127.7, 127.2, 126.7, 126.4, 126.1, 125.3, 120.9, 58.0, 29.1.

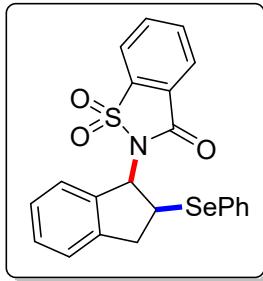


2-(2-(phenylselanyl)-1-(thiophen-2-yl)ethyl)benzo[d]isothiazol-3(2H)-one

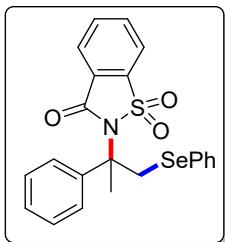
1,1-dioxide (4r): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 75.2 mg, 84% yield. White solid. m.p.: 89.2-90.4 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.99-7.97 (m, 1H), 7.88-7.77 (m, 3H), 7.59-7.56 (m, 2H), 7.28 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.27-7.23 (m, 4H), 6.97 (dd, *J* = 5.1, 3.6 Hz, 1H), 5.62 (t, *J* = 7.9 Hz, 1H), 4.07 (dd, *J* = 13.0, 8.6 Hz, 1H), 3.81 (dd, *J* = 13.0, 7.4 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.6, 139.3, 137.5, 134.9, 134.4, 134.0, 129.3, 128.9, 128.2, 127.9, 127.1, 126.8, 126.5, 125.3, 120.9, 52.8, 30.5.



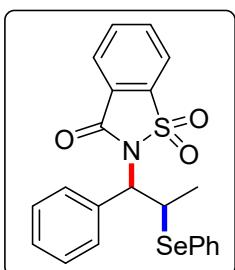
2-(1-ferrocenyl-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4s): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 50.7 mg, 45% yield. Yellow solid. m.p.: 152.0-152.2 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.95-7.93 (s, 1H), 7.85-7.74 (m, 5H), 7.31-7.29 (m, 3H), 5.34 (dd, *J* = 11.9, 8.8 Hz, 1H), 4.4 (d, *J* = 1.0 Hz, 1H), 4.4 (d, *J* = 1.1 Hz, 1H), 4.19-4.13 (m, 3H), 4.0 (s, 5H), 3.8 (dd, *J* = 13.3, 10.1 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.7, 137.7, 135.2, 134.7, 134.2, 129.4, 129.3, 128.2, 127.3, 125.2, 120.8, 84.8, 69.1, 69.0, 68.7, 68.2, 67.7, 54.6, 29.8. **HRMS** (ESI-Orbitrap) *m/z* calcd for C₂₅H₂₂FeNO₃SSe [M+H]⁺: 551.9830; found: 551.9810.



2-(2-(phenylselanyl)-2,3-dihydro-1*H*-inden-1-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4t): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 4:1, v/v). 71.1 mg, 78% yield. White solid. m.p.: 133.9-134.7 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.96 (m, 1H), 7.90-7.77 (m, 3H), 7.71-7.68 (m, 2H), 7.33-7.18 (m, 7H), 5.75 (d, *J* = 7.0 Hz, 1H), 4.67 (q, *J* = 8.0 Hz, 1H), 3.71 (dd, *J* = 16.3, 8.0 Hz, 1H), 3.10 (dd, *J* = 16.4, 7.3 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.6, 142.1, 137.7, 137.2, 136.1, 134.9, 134.4, 129.2, 129.1, 128.4, 127.3, 127.2, 125.3, 124.7, 124.6, 121.0, 63.6, 42.5, 38.9.

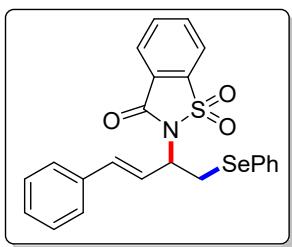


2-(2-phenyl-1-(phenylselanyl)propan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (4u): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 63.7 mg, 70% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.89-7.87 (m, 3H), 7.83-7.79 (m, 1H), 7.70-7.69 (m, 2H), 7.54-7.52 (m, 2H), 7.50-7.47 (m, 2H), 7.35-7.24 (m, 3H), 7.11-7.05 (m, 3H), 4.55 (d, *J* = 12.1 Hz, 1H), 3.89 (d, *J* = 12.1 Hz, 1H), 2.23 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 143.8, 137.9, 134.8, 134.0, 134.0, 129.6, 128.8, 128.6, 127.6, 127.4, 126.4, 125.5, 125.0, 120.3, 68.0, 37.6, 25.7.



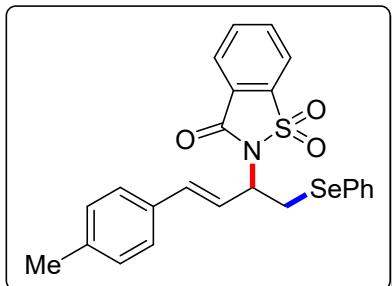
2-(1-phenyl-2-(phenylselanyl)propyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4v)

Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 83.5 mg, 87% yield. White solid. m.p. 143.8-144.5 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.96 (m, 1H), 7.84-7.74 (m, 3H), 7.65-7.62 (m, 2H), 7.43-7.41 (m, 2H), 7.34-7.28 (m, 4H), 7.26-7.21 (m, 2H), 5.05 (d, *J* = 11.7 Hz, 1H), 4.69-4.61 (m, 1H), 1.56 (d, *J* = 6.8 Hz, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.7, 137.2, 136.5, 136.5, 136.5, 134.8, 134.4, 129.7, 129.0, 128.9, 128.4, 127.5, 127.1, 125.2, 120.9, 63.4, 37.7, 20.7.



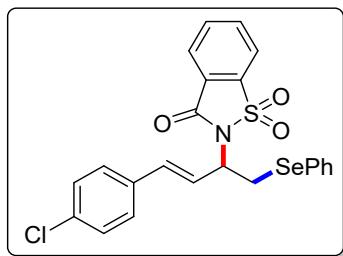
(E)-2-(4-phenyl-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4w)

New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 78.7 mg, 84% yield, Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.01-7.99 (m, 1H), 7.87-7.79 (m, 3H), 7.61-7.57 (m, 2H), 7.38-7.36 (m, 2H), 7.33-7.23 (m, 6H), 6.72 (d, *J* = 15.8 Hz, 1H), 6.57 (dd, *J* = 15.7, 8.4 Hz, 1H), 5.03 (q, *J* = 8.2 Hz, 1H), 3.75 (dd, *J* = 12.8, 7.4 Hz, 1H), 3.58 (dd, *J* = 12.8, 8.4 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.6, 137.7, 135.8, 135.5, 134.9, 134.4, 133.7, 129.3, 129.0, 128.6, 128.4, 127.6, 127.3, 127.0, 125.3, 124.1, 120.9, 56.9, 29.9. **HRMS** (ESI-Orbitrap) m/z calcd for C₂₃H₁₉NO₃SSeNa [M+Na]⁺: 492.0143; found: 492.0143.

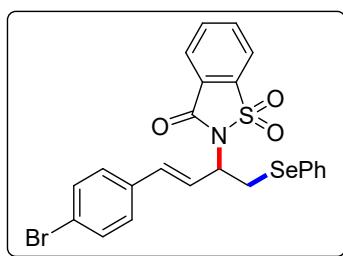


(E)-2-(1-(phenylselanyl)-4-(*p*-tolyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4x): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 76.7 mg, 79% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.02-7.99

(m, 1H), 7.89-7.79 (m, 3H), 7.60-7.58 (m, 2H), 7.28-7.26 (m, 5H), 7.11 (d, J = 8.0 Hz, 2H), 6.68 (d, J = 15.8 Hz, 1H), 6.51 (dd, J = 15.8, 8.5 Hz, 1H), 5.02 (q, J = 8.2 Hz, 1H), 3.75 (dd, J = 12.8, 7.5 Hz, 1H), 3.57 (dd, J = 12.7, 8.2 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.6, 138.3, 137.7, 135.4, 134.8, 134.3, 133.6, 133.0, 129.3, 129.3, 129.1, 127.6, 127.3, 126.9, 125.2, 123.0, 120.8, 57.0, 30.0, 21.3; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{24}\text{H}_{22}\text{NO}_3\text{SSe}$ [M+H] $^+$: 484.0480; found: 484.0480.

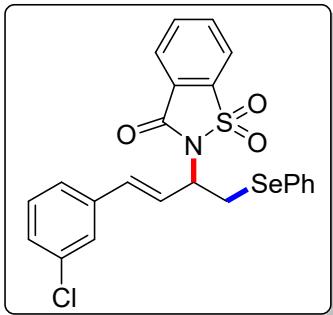


(E)-2-(4-(4-chlorophenyl)-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4y): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 91.1 mg, 91% yield. Colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.01-7.99 (m, 1H), 7.89-7.78 (m, 3H), 7.58-7.56 (m, 2H), 7.26-7.25 (m, 7H), 8.65 (d, J = 15.8 Hz, 1H), 6.51 (dd, J = 15.8, 8.3 Hz, 1H), 5.00 (q, J = 7.6 Hz, 1H), 3.71 (dd, J = 12.8, 7.2 Hz, 1H), 3.57 (dd, J = 12.8, 8.5 Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.6, 137.7, 134.9, 134.4, 134.3, 134.2, 134.1, 133.7, 129.3, 128.9, 128.8, 128.2, 127.7, 127.2, 125.3, 124.8, 120.9, 56.7, 29.7; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{19}\text{ClNO}_3\text{SSe}$ [M+H] $^+$: 503.9934; found: 503.9935.

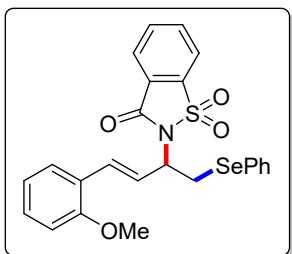


(E)-2-(4-(4-bromophenyl)-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4z): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 100.9 mg, 92% yield. Colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.02-8.00 (m, 1H), 7.90-7.79 (m, 3H), 7.58-7.56 (m, 2H), 7.42-7.40 (m, 2H), 7.27-7.25 (m, 3H), 7.21-7.19 (m, 2H), 6.63 (d, J = 15.8 Hz, 1H), 6.53 (dd, J = 15.8,

8.2 Hz, 1H), 4.99 (q, J = 8.2 Hz, 1H), 3.71 (dd, J = 12.8, 7.2 Hz, 1H), 3.57 (dd, J = 12.8, 8.5 Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.6, 137.6, 134.9, 134.7, 134.4, 134.2, 133.6, 131.7, 129.3, 128.9, 128.5, 127.7, 127.2, 125.3, 124.9, 122.3, 120.9, 56.7, 29.7; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{18}\text{BrNO}_3\text{SSeNa}$ [M+Na] $^+$: 569.9248; found: 569.9251.

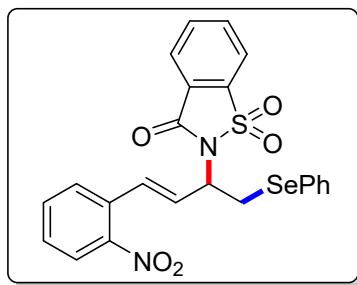


(E)-2-(4-(3-chlorophenyl)-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4a'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 97.0 mg, 96% yield. white solid. m.p.: 150.9-153.5 °C. ^1H NMR (CDCl_3 , 400 MHz) δ 8.00-7.98 (m, 1H), 7.88-7.77 (m, 3H), 7.57-7.55 (m, 2H), 7.30 (s, 1H), 7.26-7.24 (m, 3H), 7.20-7.19 (m, 3H), 6.62 (d, J = 15.8 Hz, 1H), 6.52 (dd, J = 15.8, 8.1 Hz, 1H), 4.98 (q, J = 7.5 Hz, 1H), 3.70 (dd, J = 12.8, 7.1 Hz, 1H), 3.56 (dd, J = 12.8, 8.6 Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.6, 137.7, 134.9, 134.6, 134.5, 134.1, 133.7, 129.9, 129.4, 128.9, 128.3, 127.7, 127.2, 126.8, 125.7, 125.3, 125.3, 120.9, 56.6, 29.6; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{19}\text{ClNO}_3\text{SSe}$ [M+H] $^+$: 503.9934; found: 503.9935.

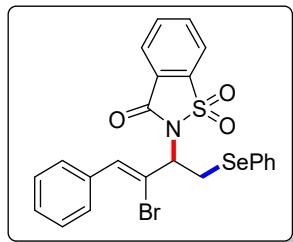


(E)-2-(4-(2-methoxyphenyl)-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4b'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 86.6 mg, 87% yield. white solid. m.p.: 130.8-131.6 °C. ^1H NMR (CDCl_3 , 400 MHz) δ 8.01-7.99 (m, 1H), 7.88-7.77 (m, 3H), 7.60-7.57 (m, 2H), 7.39 (dd, J = 7.7, 1.7 Hz, 1H), 7.26-7.20 (m, 4H), 7.04 (d, J = 16.0 Hz, 1H), 6.90-6.83 (m, 2H), 6.59 (dd, J = 15.9, 8.6 Hz, 1H), 5.05 (q, J = 7.9 Hz, 1H),

3.82 (s, 3H), 3.75 (dd, $J = 12.8, 7.8$ Hz, 1H), 3.55 (dd, $J = 12.8, 8.0$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.6, 157.1, 137.8, 134.8, 134.3, 133.7, 130.4, 129.5, 129.3, 129.2, 127.6, 127.5, 127.3, 125.2, 124.8, 124.4, 120.8, 120.7, 111.0, 57.5, 55.6, 30.2; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{24}\text{H}_{21}\text{NO}_4\text{SSeNa} [\text{M}+\text{Na}]^+$: 522.0249; found: 522.0248.

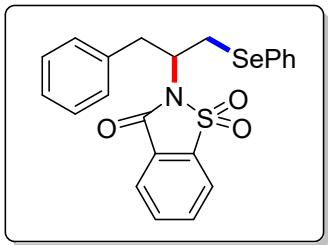


(E)-2-(4-(2-nitrophenyl)-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4c'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 66.6 mg, 65% yield. Yellow oil. ^1H NMR (CDCl_3 , 400 MHz) δ 7.94-7.92 (m, 1H), 7.89-7.86 (m, 1H), 7.81-7.71 (m, 3H), 7.53-7.49 (m, 2H), 7.48-7.41 (m, 2H), 7.34-7.30 (m, 1H), 7.20-7.12 (m, 4H), 6.42 (dd, $J = 15.6, 8.2$ Hz, 1H), 4.98 (q, $J = 8.2$ Hz, 1H), 3.62 (dd, $J = 12.8, 7.4$ Hz, 1H), 3.51 (dd, $J = 12.8, 8.7$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.6, 147.7, 137.7, 135.0, 134.5, 133.8, 133.4, 132.0, 131.1, 129.4, 129.3, 128.9, 128.6, 127.8, 127.2, 125.4, 124.7, 120.9, 56.1, 29.4; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{18}\text{N}_2\text{O}_5\text{SSeNa} [\text{M}+\text{Na}]^+$: 536.9994; found: 537.0002.

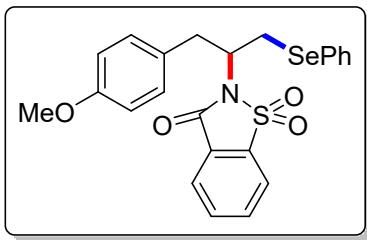


(Z)-2-(3-bromo-4-phenyl-1-(phenylselanyl)but-3-en-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (4d'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 66.2 mg, 60% yield. White solid. m.p.: 153.4-154.6 °C. ^1H NMR (CDCl_3 , 400 MHz) δ 8.07-8.05 (m, 1H), 7.90-7.80 (m, 3H), 7.64-7.62 (m, 2H), 7.59-7.57 (m, 2H), 7.38-7.28 (m, 6H), 7.25-7.24 (m, 1H), 5.34 (t, $J = 7.7$ Hz, 1H),

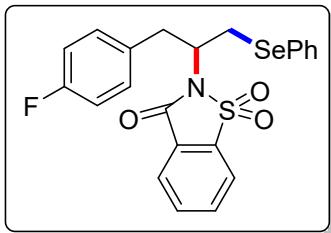
3.93 (dd, $J = 13.0, 8.6$ Hz, 1H), 3.78 (dd, $J = 13.0, 7.2$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 158.9, 137.7, 135.1, 134.9, 134.5, 134.3, 133.0, 129.4, 129.3, 128.8, 128.7, 128.3, 128.1, 126.8, 125.6, 121.0, 120.8, 62.2, 28.9; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{18}\text{BrNO}_3\text{SSeNa} [\text{M}+\text{Na}]^+$: 569.9248; found: 569.9251.



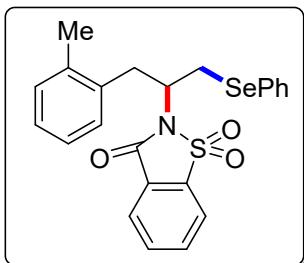
2-(1-phenyl-3-(phenylselanyl)propan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5a): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 71.8 mg, 79% yield. Colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.56-7.53 (m, 2H), 7.31-7.20 (m, 8H), 4.10-3.98 (m, 3H), 3.19 (dd, $J = 14.8, 5.2$ Hz, 1H), 2.89 (dd, $J = 14.7, 8.8$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 159.3, 138.6, 137.6, 135.1, 135.0, 134.5, 129.3, 129.2, 128.5, 128.1, 127.7, 127.2, 126.8, 125.4, 121.1, 44.3, 43.2, 38.9; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{22}\text{H}_{19}\text{NO}_3\text{SSeNa} [\text{M}+\text{Na}]^+$: 480.0143; found: 480.0146.



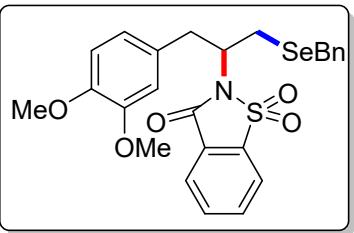
2-(1-(4-methoxyphenyl)-3-(phenylselanyl)propan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5b): New compound. (Eluent: petroleum ether (60-90 °C) / EtOAc = 6:1, v/v). 66.5 mg, 68% yield. Colorless oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.04-8.02 (m, 1H), 7.92-7.79 (m, 3H), 7.56-7.54 (m, 2H), 7.27-7.25 (m, 3H), 7.17-7.15 (m, 2H), 6.84-6.81 (m, 2H), 4.11-3.95 (m, 3H), 3.78 (s, 3H), 3.12 (dd, $J = 14.7, 5.2$ Hz, 1H), 2.84 (dd, $J = 14.6, 8.4$ Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 159.2, 158.4, 137.6, 135.0, 134.5, 130.5, 130.2, 129.2, 128.1, 128.0, 127.8, 127.2, 125.4, 121.0, 113.9, 55.3, 44.2, 43.5, 38.0; HRMS (ESI) m/z Calcd for $\text{C}_{23}\text{H}_{22}\text{NO}_4\text{SSe} [\text{M}+\text{H}]^+$: 488.0429; Found: 488.0417.



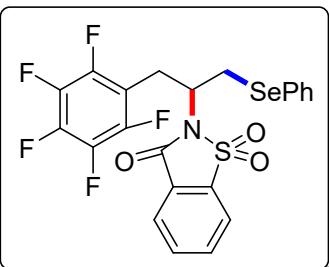
2-(1-(4-fluorophenyl)-3-(phenylselanyl)propan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5c): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 53.7 mg, 56% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.93-7.81 (m, 3H), 7.54-7.51 (m, 2H), 7.29-7.24 (m, 3H), 7.21-7.17 (m, 2H), 7.00-6.93 (m, 2H), 4.12-4.02 (m, 2H), 3.98-3.91 (m, 1H), 3.16 (dd, J = 14.7, 5.2 Hz, 1H), 2.84 (dd, J = 14.7, 9.2 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 161.8 (d, J = 243.0 Hz), 159.3, 137.6, 135.1, 134.6, 134.2 (d, J = 3.4 Hz), 130.7 (d, J = 7.9 Hz), 129.3, 128.2, 127.4 (d, J = 38.7 Hz), 125.5, 121.1, 115.4, 115.2, 44.3, 43.4, 38.0; **¹⁹F NMR** (CDCl₃, 376 MHz) δ -116.3; **HRMS** (ESI) m/z Calcd for C₂₂H₁₈FNO₃SSeNa [M+Na]⁺: 498.0049; Found: 498.0050.



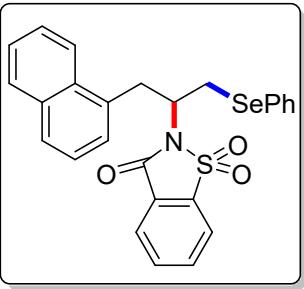
2-(1-(phenylselanyl)-3-(o-tolyl)propan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5d): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 50.9 mg, 54% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.06-8.04 (m, 1H), 7.92-7.80 (m, 3H), 7.55-7.52 (m, 2H), 7.27-7.21 (m, 4H), 7.15-7.12 (m, 3H), 4.11-4.01 (m, 3H), 3.18 (dd, J = 14.8, 5.0 Hz, 1H), 2.97-2.91 (m, 1H), 2.24 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 137.6, 136.8, 136.4, 135.0, 134.5, 133.0, 130.6, 129.8, 129.2, 128.0, 127.9, 127.2, 126.9, 126.0, 125.4, 121.1, 44.5, 42.4, 36.7, 19.6; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₃H₂₁NO₃SSeNa [M+Na]⁺: 494.0300; found: 494.0298.



2-(1-(benzylselanyl)-3-(3,4-dimethoxyphenyl)propan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5e): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 64.4 mg, 61% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.07-8.05 (m, 1H), 7.94-7.81 (m, 3H), 7.26-7.19 (m, 5H), 6.75-6.71 (m, 3H), 4.12-3.97 (m, 2H), 3.83 (s, 6H), 3.78-3.76 (m, 2H), 3.52 (m, 1H), 3.06 (dd, *J* = 14.3, 5.1 Hz, 1H), 2.79 (dd, *J* = 14.2, 8.7 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 148.8, 147.7, 138.8, 137.7, 135.0, 134.5, 131.1, 129.1, 128.6, 127.3, 126.9, 125.5, 121.4, 121.0, 112.4, 111.0, 55.9, 55.9, 44.1, 40.3, 38.9, 27.7; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₅H₂₅NO₅SSeNa [M+Na]⁺: 554.0511; found: 554.0502.

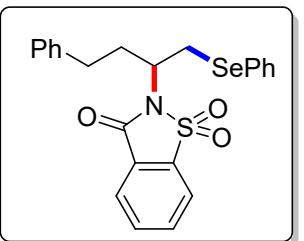


2-(1-(perfluorophenyl)-3-(phenylselanyl)propan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5f): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 48.2 mg, 44% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.08-8.06 (m, 1H), 7.95-7.83 (m, 3H), 7.52-7.49 (m, 2H), 7.28-7.24 (m, 3H), 4.17 (dd, *J* = 18.8, 10.2 Hz, 1H), 4.03-4.94 (m, 2H), 3.30-3.27 (m, 1H), 3.05 (dd, *J* = 14.4, 9.3 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 146.3, 144.3, 137.6, 136.5, 135.2, 134.7, 134.5, 129.6, 129.4, 128.3, 127.3, 127.1, 125.6, 121.3, 44.2, 41.0, 26.9; **¹⁹F NMR** (CDCl₃, 376 MHz) δ -64.3, -78.5, -84.7; **HRMS** (ESI) m/z Calcd for C₂₂H₁₄F₅NO₃SSeNa [M+Na]⁺: 569.9672; Found: 569.9673.

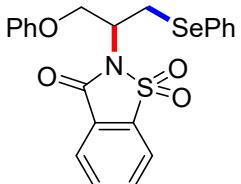


2-(1-(naphthalen-1-yl)-3-(phenylselanyl)propan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5g):

New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 4:1, v/v). 52.4 mg, 48% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.10-8.07 (m, 1H), 7.96-7.93 (m, 1H), 7.90-7.82 (m, 4H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.55-7.41 (m, 6H), 7.30-7.22 (m, 3H), 4.34-4.26 (m, 1H), 4.17-4.15 (m, 2H), 3.70 (dd, *J* = 14.8, 6.0 Hz, 1H), 3.40 (dd, *J* = 14.8, 9.2 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.4, 137.6, 135.1, 135.0, 134.6, 134.5, 134.0, 131.9, 129.2, 129.0, 128.0, 127.8, 127.5, 127.2, 126.2, 125.7, 125.5, 125.4, 123.5, 121.1, 44.5, 42.5, 36.7; **HRMS** (ESI) m/z Calcd for C₂₆H₂₁NO₃SSeNa [M+Na]⁺: 530.0300; Found: 530.0300.

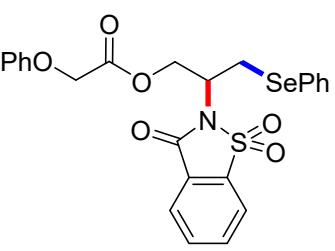


2-(4-phenyl-1-(phenylselanyl)butan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5h): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 45.5 mg, 48% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.04-8.02 (m, 1H), 7.91-7.80 (m, 3H), 7.67-7.64 (m, 2H), 7.33-7.30 (m, 3H), 7.26-7.22 (m, 2H), 7.18-7.14 (m, 3H), 4.11-4.00 (m, 2H), 3.76-3.69 (m, 1H), 3.10-3.03 (m, 1H), 2.79-2.71 (m, 1H), 2.18-2.09 (m, 1H), 1.92-1.83 (m, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.2, 141.3, 137.6, 135.1, 135.0, 134.5, 129.4, 128.6, 128.5, 128.2, 127.5, 127.2, 126.1, 125.4, 121.1, 44.4, 41.9, 33.8, 33.7; **HRMS** (ESI) m/z Calcd for C₂₃H₂₁NO₃SSeNa [M+Na]⁺: 494.0300; Found: 494.0303.

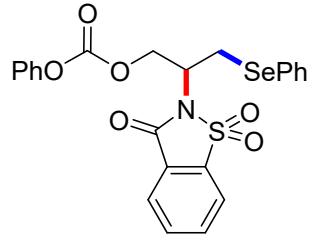


2-(1-phenoxy-3-(phenylselanyl)propan-2-yl)benzo[*d*]isothiazol-3(*2H*)-one

1,1-dioxide (5i): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 55.7 mg, 59% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.70-7.68 (m, 2H), 7.33-7.24 (m, 5H), 6.95 (t, *J* = 7.4 Hz, 1H), 6.87-6.85 (m, 2H), 4.40-4.24 (m, 3H), 4.16 (dd, *J* = 14.8, 6.7 Hz, 1H), 4.11-4.04 (m, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 158.4, 137.6, 135.0, 134.9, 134.5, 129.5, 129.4, 128.3, 127.5, 127.2, 125.4, 121.3, 121.1, 114.8, 68.7, 41.3, 40.9; **HRMS** (ESI) m/z Calcd for C₂₂H₁₉NO₄SSeNa [M+Na]⁺: 496.0092; Found: 496.0091.

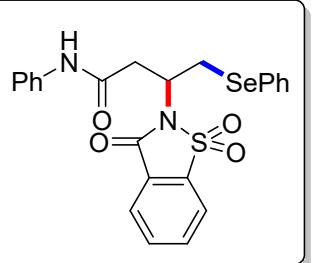


2-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(*3H*)-yl)-3-(phenylselanyl)propyl 2-phenoxyacetate (5j): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 4:1, v/v). 48.9 mg, 46% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.01-8.00 (m, 1H), 7.89-7.76 (m, 3H), 7.63-7.61 (m, 2H), 7.31-7.23 (m, 5H), 6.96 (t, *J* = 7.4 Hz, 1H), 6.89-6.87 (m, 2H), 4.63-4.52 (m, 3H), 4.39 (dd, *J* = 11.8, 5.8 Hz, 1H), 4.13-4.00 (m, 2H), 3.92-3.85 (m, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 168.7, 159.2, 157.8, 137.5, 135.1, 134.6, 129.7, 129.6, 128.6, 127.1, 126.8, 125.5, 121.8, 121.2, 114.8, 65.5, 65.0, 40.8, 39.5; **HRMS** (ESI) m/z Calcd for C₂₄H₂₁NO₆SSeNa [M+Na]⁺: 554.0147; Found: 554.0146.



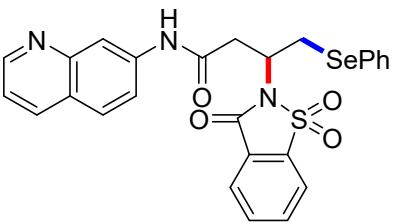
2-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(*3H*)-yl)-3-(phenylselanyl)propyl phenyl carbonate (5k**):**

New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 35.8 mg, 35% yield. purple oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.07-8.05 (m, 1H), 7.94-7.80 (m, 3H), 7.70-7.67 (m, 2H), 7.39-7.30 (m, 5H), 7.25-7.21 (m, 1H), 7.18-7.15 (m, 2H), 4.61 (dd, *J* = 11.6, 4.4 Hz, 1H), 4.46 (dd, *J* = 11.6, 6.5 Hz, 1H), 4.21 (dd, *J* = 15.0, 9.4 Hz, 1H), 4.61 (dd, *J* = 14.9, 6.3 Hz, 1H), 4.03-4.36 (m, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 153.4, 151.2, 137.6, 135.2, 135.2, 135.1, 134.6, 129.6, 129.6, 128.7, 127.2, 126.7, 126.2, 125.5, 121.2, 68.8, 40.9, 39.4; **HRMS** (ESI) m/z Calcd for C₂₃H₁₉NO₆SSeNa [M+Na]⁺: 539.9991; Found: 539.9990.

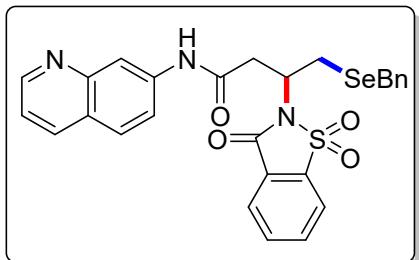


3-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(*3H*)-yl)-*N*-phenyl-4-(phenylselanyl)butanamide (5l**):**

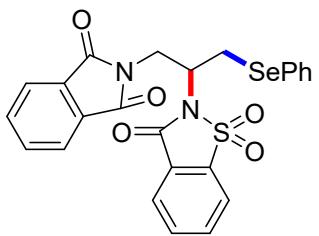
New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 3:1, v/v). 52.7 mg, 53% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.01-7.99 (m, 1H), 7.88-7.67 (m, 6H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.32-7.25 (m, 5H), 7.06 (t, *J* = 7.3 Hz, 1H), 4.10 (s, 3H), 2.93-2.89 (m, 1H), 2.73 (dd, *J* = 15.1, 5.6 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 168.0, 159.6, 137.8, 137.4, 135.2, 135.0, 134.7, 129.5, 129.0, 128.4, 127.8, 127.0, 125.5, 124.4, 121.2, 119.1, 43.9, 40.5, 36.8; **HRMS** (ESI) m/z Calcd for C₂₃H₂₀N₂O₄SSeNa [M+Na]⁺: 523.0201; Found: 523.0203.



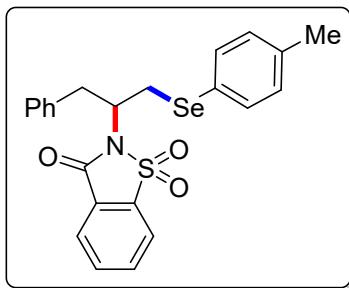
3-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(*3H*)-yl)-4-(phenylselanyl)-*N*-(quinolin-8-yl)butanamide (5m**):** New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 4:1, v/v). 79.4 mg, 72% yield. White solid. m.p.: 145.5-148.6 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 9.85 (s, 1H), 8.80 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.75 (dd, *J* = 5.6, 3.4 Hz, 1H), 8.15 (dd, *J* = 8.3, 1.7 Hz, 1H), 8.01-7.99 (m, 1H), 7.88-7.72 (m, 5H), 7.50-7.43 (m, 3H), 7.32-7.29 (m, 3H), 4.26-4.15 (m, 3H), 3.16 (dd, *J* = 15.9, 4.6 Hz, 1H), 2.91 (dd, *J* = 16.0, 7.7 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 168.5, 159.4, 148.2, 138.4, 137.6, 136.5, 135.5, 135.0, 134.5, 134.3, 129.5, 128.4, 128.0, 127.5, 127.4, 127.2, 125.4, 121.7, 121.1, 116.9, 44.1, 41.0, 37.0; **HRMS** (ESI) m/z Calcd for C₂₆H₂₂N₃O₄SSe [M+H]⁺: 552.0491; Found: 552.0490.



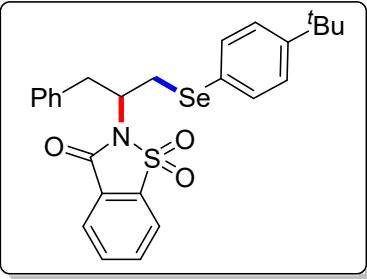
4-(benzylselanyl)-3-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(*3H*)-yl)-*N*-(quinolin-8-yl)butanamide (5n**):** New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 48.8 mg, 43% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 9.79 (s, 1H), 8.77-8.74 (m, 2H), 8.13-8.11 (m, 1H), 8.00-7.99 (m, 1H), 7.86-7.77 (m, 3H), 7.47-7.36 (m, 5H), 7.25 (m, 2H), 7.17-7.16 (m, 1H), 4.18-4.02 (m, 4H), 3.86-3.85 (m, 1H), 3.12-3.08 (m, 1H), 2.92-2.87 (m, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 168.6, 159.3, 148.2, 139.0, 138.4, 137.6, 136.4, 134.9, 134.4, 134.4, 129.1, 128.7, 128.0, 127.4, 127.2, 127.0, 125.4, 121.7, 121.6, 121.1, 116.7, 44.0, 41.8, 34.5, 28.9; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₇H₂₄N₃O₄SSe [M+H]⁺: 566.0647; found: 566.0639.



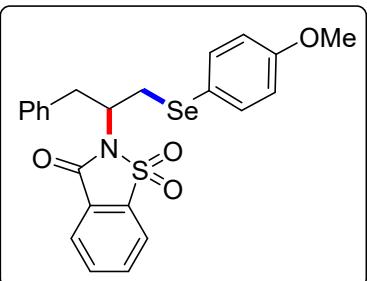
2-(2-(1,1-dioxido-3-oxobenzo[*d*]isothiazol-2(*3H*)-yl)-3-(phenylselanyl)propyl)isoindoline-1,3-dione (5o): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 2:1, v/v). 59.2 mg, 56% yield. White solid. m.p. 153.4-158.4 °C. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.08-8.05 (m, 1H), 7.93-7.83 (m, 3H), 7.82-7.77 (m, 2H), 7.74-7.69 (m, 2H), 7.63-7.60 (m, 2H), 7.24-7.19 (m, 3H), 4.25-4.94 (m, 5H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 168.2, 159.3, 137.6, 135.1, 134.9, 134.6, 134.1, 131.9, 129.4, 128.2, 127.2, 126.8, 125.5, 123.5, 121.2, 42.2, 40.9, 40.4; **HRMS** (ESI) m/z Calcd for $\text{C}_{24}\text{H}_{18}\text{N}_2\text{O}_5\text{SSeNa}$ [$\text{M}+\text{Na}]^+$: 548.9994; Found: 548.9996.



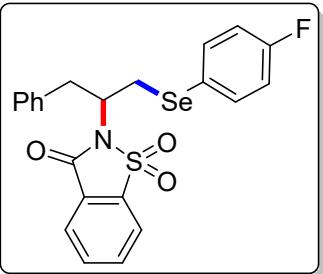
2-(1-phenyl-3-(*p*-tolylselanyl)propan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5p): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 53.6 mg, 57% yield. Colorless oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.45-7.43 (m, 2H), 7.31-7.20 (m, 5H), 7.09-7.07 (m, 2H), 4.06-4.04 (m, 2H), 3.99-3.92 (m, 1H), 3.15 (dd, $J = 14.6, 5.4$ Hz, 1H), 2.87 (dd, $J = 14.6, 8.9$ Hz, 1H), 3.32 (s, 3H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.3, 138.7, 138.3, 137.7, 135.6, 135.0, 134.5, 130.1, 129.2, 128.5, 127.3, 126.8, 125.4, 123.7, 121.1, 44.4, 43.0, 38.9, 21.3; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{21}\text{NO}_3\text{SSeNa}$ [$\text{M}+\text{Na}]^+$: 494.0300; found: 492.0304.



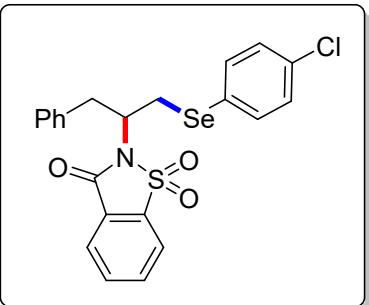
4-(1-((4-(*tert*-butyl)phenyl)selanyl)-3-phenylpropan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5q**):** New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 77 mg, 75% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.49-7.45 (m, 2H), 7.31-7.19 (m, 7H), 4.09-4.07 (m, 2H), 4.03-3.95 (m, 1H), 3.17 (dd, *J* = 14.7, 5.4 Hz, 1H), 2.90 (dd, *J* = 14.7, 8.9 Hz, 1H), 1.30 (s, 9H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 151.3, 138.7, 137.6, 135.0, 135.0, 134.5, 129.2, 128.5, 127.3, 126.7, 126.4, 125.4, 124.0, 121.1, 44.5, 42.8, 39.0, 34.7, 31.4; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₆H₂₇NO₃SSeNa [M+Na]⁺: 536.0769; found: 536.0765.



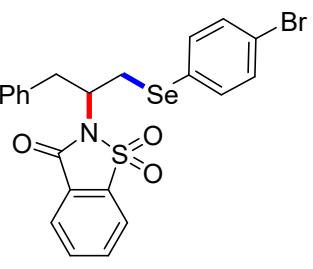
2-(1-((4-methoxyphenyl)selanyl)-3-phenylpropan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5r**):** New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 55.6 mg, 57% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.02 (m, 1H), 7.92-7.80 (m, 3H), 7.50-7.47 (m, 2H), 7.31-7.20 (m, 5H), 6.83-6.79 (m, 2H), 4.08-4.98 (m, 2H), 3.92-3.84 (m, 1H), 3.79 (s, 3H), 3.11 (dd, *J* = 14.6, 5.6 Hz, 1H), 2.86 (dd, *J* = 14.6, 9.0 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 160.1, 159.3, 138.8, 137.9, 137.6, 135.0, 134.5, 129.2, 128.5, 127.3, 126.7, 125.4, 121.1, 117.2, 114.9, 55.4, 44.4, 43.2, 38.9; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₃H₂₁NO₄SSeNa [M+Na]⁺: 510.0249; found: 510.0248.



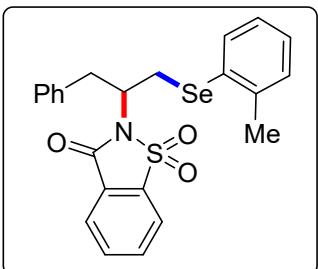
2-(1-((4-fluorophenyl)selanyl)-3-phenylpropan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5s**):** New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 38 mg, 35% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.93-7.81 (m, 3H), 7.51-7.48 (m, 2H), 7.31-7.21 (m, 5H), 6.96-6.92 (m, 2H), 4.05-4.03 (m, 2H), 3.97-3.89 (m, 1H), 3.15 (dd, *J* = 14.7, 5.5 Hz, 1H), 2.88 (dd, *J* = 14.6, 9.0 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 163.0 (d, *J*_{CF} = 246.6 Hz), 159.3, 138.5, 137.7 (d, *J*_{CF} = 8.0 Hz), 137.6, 134.8 (d, *J*_{CF} = 48.8 Hz), 129.2, 128.6, 127.2, 126.9, 125.5, 122.2, 121.1, 116.6, 116.3, 44.3, 43.9, 39.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ -113.2; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₂H₁₈FNO₃SSeNa [M+Na]⁺: 498.0049; found: 498.0050.



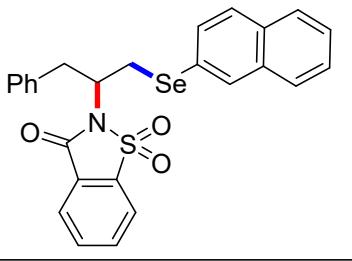
2-(1-((4-chlorophenyl)selanyl)-3-phenylpropan-2-yl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5t**):** New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 49.2 mg, 50% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.93-7.81 (m, 3H), 7.45-7.41 (m, 2H), 7.31-7.18 (m, 7H), 4.10-4.95 (m, 3H), 3.18 (dd, *J* = 14.7, 5.3 Hz, 1H), 2.88 (dd, *J* = 14.8, 8.5 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 138.3, 137.6, 136.4, 135.1, 134.6, 134.4, 129.4, 129.2, 128.6, 127.2, 126.9, 126.0, 125.5, 121.1, 44.3, 43.8, 39.1; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₂H₁₈ClNO₃SSeNa [M+Na]⁺: 513.9753; found: 513.9746.



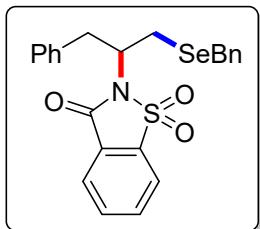
2-(1-((4-bromophenyl)selanyl)-3-phenylpropan-2-yl)benzo[d]isothiazol-3(2*H*)-one 1,1-dioxide (5u): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 51 mg, 51% yield. Colorless oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.04 (d, J = 7.0 Hz, 1H), 7.93-7.82 (m, 3H), 7.38-7.27 (m, 6H), 7.24-7.21 (m, 3H), 4.07-3.96 (m, 3H), 3.18 (dd, J = 14.7, 5.1 Hz, 1H), 2.88 (dd, J = 14.9, 8.9 Hz, 1H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.3, 138.3, 137.6, 136.6, 135.1, 134.6, 132.3, 129.2, 128.6, 127.2, 126.9, 126.8, 125.5, 122.5, 121.1, 44.3, 43.8, 39.1; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{22}\text{H}_{19}\text{BrNO}_3\text{SSe}$ [M+H]⁺: 535.9429; found: 535.9418.



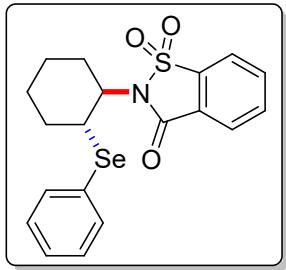
2-(1-phenyl-3-(*o*-tolylselanyl)propan-2-yl)benzo[d]isothiazol-3(2*H*)-one 1,1-dioxide (5v): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 47 mg, 50% yield. Colorless oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.56 (d, J = 7.4 Hz, 1H), 7.30-7.14 (m, 7H), 7.11-7.07 (m, 1H), 4.16-4.97 (m, 3H), 3.27 (dd, J = 14.5, 4.6 Hz, 1H), 2.88 (dd, J = 14.6, 8.8 Hz, 1H), 2.36 (s, 3H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.3, 141.0, 138.6, 137.7, 135.0, 134.5, 134.5, 130.3, 129.5, 129.2, 128.5, 128.0, 127.3, 126.8, 126.7, 125.5, 121.1, 44.4, 42.5, 38.9, 22.8. **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{21}\text{NO}_3\text{SSeNa}$ [M+Na]⁺: 494.0300; found: 492.0311.



2-(1-(naphthalen-2-ylselanyl)-3-phenylpropan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5w): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 40.0 mg, 39% yield. Colorless oil. **1H NMR** (CDCl_3 , 400 MHz) δ 8.02 (s, 1H), 7.98 (d, J = 7.3 Hz, 1H), 7.89 (d, J = 7.4 Hz, 1H), 7.84-7.74 (m, 4H), 7.71 (d, J = 8.6 Hz, 1H), 7.57 (dd, J = 8.5, 1.7 Hz, 1H), 7.49-7.44 (m, 2H), 7.32-7.21 (m, 5H), 4.18-4.11 (m, 3H), 3.23 (dd, J = 14.1, 4.6 Hz, 1H), 3.96 (dd, J = 14.4, 8.1 Hz, 1H); **13C NMR** (CDCl_3 , 100 MHz) δ 159.3, 138.5, 137.6, 135.0, 134.5, 134.0, 133.9, 132.6, 131.7, 129.4, 129.2, 128.8, 128.6, 127.8, 127.6, 127.2, 126.9, 126.5, 126.4, 125.4, 121.0, 44.5, 43.1, 39.0; **HRMS** (ESI) m/z Calcd for $\text{C}_{26}\text{H}_{21}\text{NO}_3\text{SSeNa}$ [M+Na]⁺: 530.0300; Found: 530.0306.

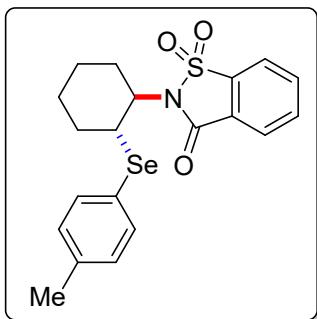


2-(1-(benzylselanyl)-3-phenylpropan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5x): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 63.3 mg, 67% yield. Colorless oil. **1H NMR** (CDCl_3 , 400 MHz) δ 8.06-8.04 (m, 1H), 7.93-7.80 (m, 3H), 7.24-7.14 (m, 10H), 4.09-3.98 (m, 2H), 3.71 (s, 2H), 3.54-3.52 (m, 1H), 3.12-3.14 (m, 1H), 2.83-2.77 (m, 1H); **13C NMR** (CDCl_3 , 100 MHz) δ 159.2, 138.7, 137.7, 135.0, 134.5, 129.3, 129.1, 128.6, 128.4, 127.3, 126.9, 126.6, 125.5, 121.1, 44.3, 40.5, 39.5, 27.8; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{22}\text{NO}_3\text{SSe}$ [M+H]⁺: 472.0480; found: 472.0474.



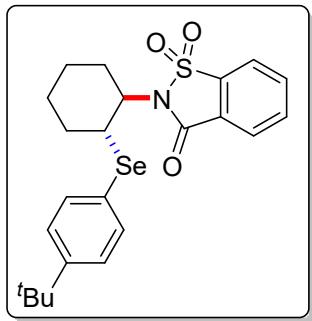
2-(2-(phenylselanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5y):

Known compound^[4]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 83.8 mg, 99% yield. Yellow oil. ¹H NMR (CDCl₃, 400 MHz) δ 7.96-7.95 (m, 1H), 7.88-7.76 (m, 3H), 7.58-7.56 (m, 2H), 7.19-7.13 (m, 3H), 4.11 (s, 2H), 2.30-2.16 (m, 3H), 1.90-1.87 (m, 1H), 1.71-1.66 (m, 1H), 1.52-1.32 (m, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 158.9, 137.3, 136.1, 134.7, 134.2, 128.8, 127.9, 127.7, 127.3, 125.3, 120.7, 58.7, 34.9, 31.9, 29.7, 25.8.



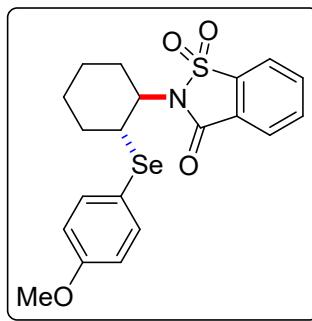
2-(2-(p-tolylselanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5z):

New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 46.6 mg, 54% yield. Yellow oil. ¹H NMR (CDCl₃, 400 MHz) δ 7.96-7.94 (m, 1H), 7.88-7.76 (m, 3H), 7.47-7.44 (m, 2H), 6.95 (d, *J* = 7.8 Hz, 2H), 4.06 (s, 2H), 2.25 (s, 6H), 1.88-1.86 (m, 1H), 1.71-1.68 (m, 1H), 1.46-1.33 (m, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 158.9, 137.9, 137.4, 136.4, 134.6, 134.1, 129.6, 127.4, 125.2, 123.9, 120.7, 58.9, 34.8, 31.9, 26.7, 25.8, 21.2; HRMS (ESI-Orbitrap) m/z calcd for C₂₀H₂₁NO₃SSeNa [M+Na]⁺: 458.0300; found: 458.0297.

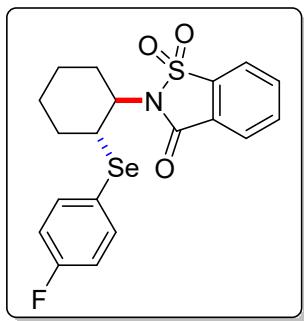


2-(2-((4-(*tert*-butyl)phenyl)selanyl)cyclohexyl)benzo[*d*]isothiazol-3(2*H*)-one

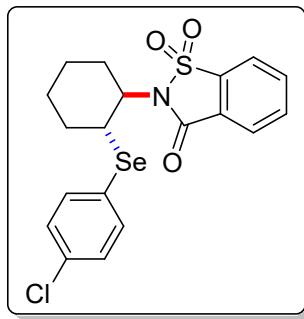
1,1-dioxide (5a'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 15:1, v/v). 87.9 mg, 92% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.96-7.95 (m, 1H), 7.89-7.76 (m, 3H), 7.50-7.48 (m, 2H), 7.19-7.16 (m, 2H), 4.07 (s, 2H), 2.29-2.15 (m, 3H), 1.89-1.87 (m, 1H), 1.72-1.70 (m, 1H), 1.49-1.33 (m, 3H), 1.26 (s, 9H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 151.0, 137.4, 135.9, 134.6, 134.2, 127.4, 125.9, 125.2, 124.1, 120.8, 58.9, 35.0, 34.6, 31.9, 31.4, 26.7, 25.8; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₃H₂₇NO₃SSeNa [M+Na]⁺:500.0769; found: 500.0764.



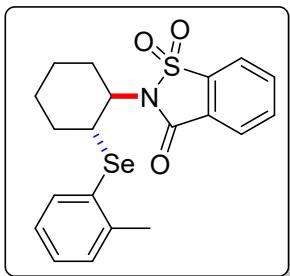
2-(2-((4-methoxyphenyl)selanyl)cyclohexyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5b'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 57.4 mg, 64% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.97-7.95 (m, 1H), 7.89-7.76 (m, 3H), 7.52-7.48 (m, 2H), 6.69 (d, *J* = 8.0 Hz, 2H), 4.14-3.99 (m, 2H), 3.73 (s, 3H), 2.24-2.14 (m, 3H), 1.86-1.85 (m, 1H), 1.70-1.65 (m, 1H), 1.41-1.31 (m, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.8, 158.9, 138.3, 137.4, 134.6, 134.2, 127.4, 125.2, 120.7, 117.6, 114.4, 58.9, 55.2, 43.8, 34.6, 31.9, 26.6, 25.8; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₀H₂₁NO₄SSeNa [M+Na]⁺:474.0249; found: 474.0252.



2-(2-((4-fluorophenyl)selanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5c'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 50.0 mg, 61% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.97-7.95 (m, 1H), 7.89-7.78 (m, 3H), 7.56-7.53 (m, 2H), 6.82 (t, *J* = 8.4 Hz, 2H), 4.05 (s, 2H), 2.25-2.14 (m, 3H), 1.89-1.87 (m, 1H), 1.73-1.70 (m, 1H), 1.47-1.33 (m, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 162.9 (d, *J*_{CF} = 264 Hz), 158.9, 138.4 (d, *J*_{CF} = 8 Hz), 137.4, 134.7, 134.3, 127.3, 125.2, 122.2, 120.8, 115.9 (d, *J*_{CF} = 21 Hz), 58.9, 34.7, 31.9, 26.7, 25.8; **¹⁹F NMR** (CDCl₃, 376 MHz) δ -36.1; **HRMS** (ESI-Orbitrap) m/z calcd for C₁₉H₁₈NO₃FSSeNa [M+Na]⁺:462.0049; found: 462.0066.

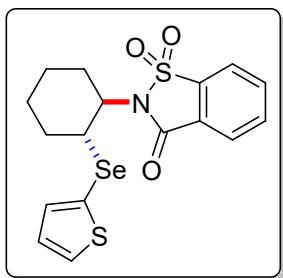


2-(2-((4-chlorophenyl)selanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5d'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 15:1, v/v). 47.8 mg, 53% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.93-7.91 (m, 1H), 7.88-7.77 (m, 3H), 7.49-7.47 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 4.11 (s, 2H), 2.27-2.14 (m, 3H), 1.90-1.88 (m, 1H), 1.74-1.72 (m, 1H), 1.48-1.35 (m, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 137.5, 137.3, 134.7, 134.3, 134.2, 128.9, 127.2, 126.0, 125.2, 120.8, 59.0, 34.7, 31.9, 26.7, 25.7; **HRMS** (ESI-Orbitrap) m/z calcd for C₁₉H₁₉ClNO₃SSeNa [M+H]⁺:455.9895; found: 455.9903.

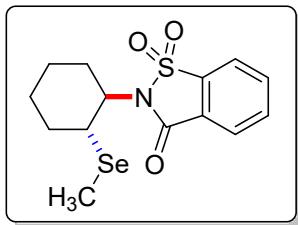


2-(2-(*o*-tolylselanyl)cyclohexyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5e'):

New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 53.2 mg, 61% yield. Yellow oil. **$^1\text{H NMR}$** (CDCl_3 , 400 MHz) δ 7.96-7.94 (m, 1H), 7.89-7.76 (m, 3H), 7.56 (dd, J = 7.7, 6.6 Hz, 1H), 7.14-7.05 (m, 2H), 6.91 (t, J = 7.3 Hz, 1H), 4.18 (s, 2H), 2.40 (s, 3H), 2.29-2.16 (m, 3H), 1.93-1.88 (m, 1H), 1.73-1.69 (m, 1H), 1.62-1.52 (m, 1H), 1.45-1.25 (m, 2H); **$^{13}\text{C NMR}$** (CDCl_3 , 100 MHz) δ 158.9, 141.9, 137.4, 136.0, 134.7, 134.2, 129.9, 129.3, 128.0, 127.3, 126.2, 125.2, 120.8, 58.6, 34.9, 31.9, 26.7, 25.9, 23.2; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{SSeNa}$ $[\text{M}+\text{Na}]^+$: 458.0300; found: 458.0307.

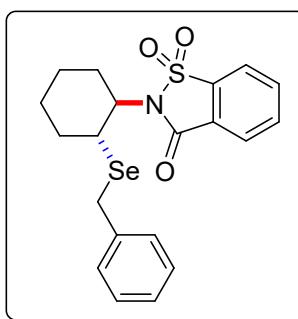


2-(2-(thiophen-2-ylselanyl)cyclohexyl)benzo[*d*]isothiazol-3(2*H*)-one 1,1-dioxide (5f'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 15:1, v/v). 44.6 mg, 52% yield. Yellow oil. **$^1\text{H NMR}$** (CDCl_3 , 400 MHz) δ 8.06-8.04 (m, 1H), 7.92-7.81 (m, 3H), 7.37 (d, J = 5.2 Hz, 1H), 7.27-7.26 (m, 1H), 6.95-6.93 (m, 1H), 4.16 (s, 1H), 3.91 (s, 1H), 2.28-2.16 (m, 3H), 1.87-1.84 (m, 1H), 1.72-1.60 (m, 1H), 1.40-1.25 (m, 3H); **$^{13}\text{C NMR}$** (CDCl_3 , 100 MHz) δ 158.9, 138.1, 137.5, 134.8, 134.4, 131.9, 128.2, 127.4, 125.4, 120.8, 120.6, 58.3, 34.5, 31.9, 26.6, 25.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_3\text{S}_2\text{SeNa}$ $[\text{M}+\text{Na}]^+$: 449.9707; found: 449.9708.



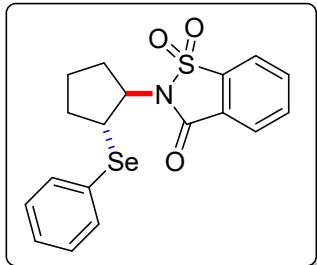
2-(2-(methylselanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide

(5g'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 69.8 mg, 97% yield. White solid. m.p.: 112.5-113.8 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.89-7.78 (m, 3H), 4.08 (s, 1H), 3.66 (s, 1H), 2.36-2.21 (m, 3H), 1.92 (s, 4H), 1.79-1.78 (m, 1H), 1.69-1.59 (m, 1H), 1.48-1.40 (m, 2H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 137.5, 134.7, 134.2, 127.3, 125.3, 120.8, 57.7, 39.9, 34.7, 31.7, 26.7, 26.0, 1.57; **HRMS** (ESI-Orbitrap) m/z calcd for C₁₄H₁₇NO₃SSeNa [M+Na]⁺:381.9987; found: 381.9986.



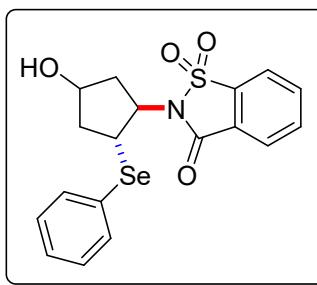
2-(2-(benzylselanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide

(5h'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 81.8 mg, 94% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.89-7.78 (m, 3H), 7.28-7.15 (m, 5H), 4.14 (s, 1H), 3.89-3.77 (m, 2H), 3.73 (s, 1H), 2.27-2.22 (m, 3H), 1.93-1.90 (m, 1H), 1.75-1.62 (m, 2H), 1.43-1.34 (m, 2H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.9, 138.8, 137.4, 134.7, 134.3, 129.1, 128.4, 127.3, 126.7, 125.2, 120.8, 58.0, 35.8, 31.8, 26.8, 26.1, 25.9; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₀H₂₁NO₃SSeNa [M+Na]⁺:458.0300; found: 458.0306.



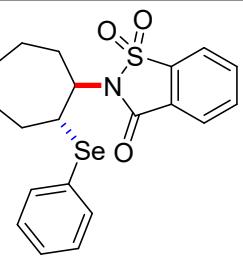
2-(2-(phenylselanyl)cyclopentyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5i')

(**5i'**): Known compound^[4]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 81.0 mg, 99% yield. Yellow oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 1H), 7.86-7.78 (m, 3H), 7.62-7.59 (m, 2H), 7.18-7.16 (m, 3H), 4.53-4.46 (m, 1H), 4.34-4.27 (m, 1H), 2.34-2.27 (m, 2H), 2.16-2.12 (m, 1H), 1.93-1.90 (m, 1H), 1.77-1.69 (m, 2H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 158.8, 137.5, 135.8, 134.7, 134.2, 128.9, 127.9, 127.6, 127.2, 125.1, 120.7, 60.0, 42.0, 32.3, 28.8, 22.7.



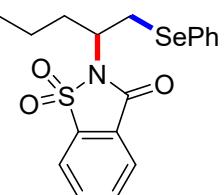
2-(4-hydroxy-2-(phenylselanyl)cyclopentyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5j')

(**5j'**): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 3:1, v/v). d.r. = 8:1, 60.8 mg, 72% yield. White solid. m.p.: 108.6-109.9 °C. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 1H), 7.87-7.77 (m, 3H), 7.62-7.60 (m, 2H), 7.24-7.17 (m, 3H), 4.81 (dd, J = 18.1, 9.2 Hz, 1H), 4.53-4.48 (m, 1H), 4.27 (dd, J = 17.8, 8.8 Hz, 1H), 2.76-2.69 (m, 1H), 2.60-2.53 (m, 1H), 2.15-2.09 (m, 1H), 1.85 (s, 1H), 1.82-1.75 (m, 1H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.5, 158.9, 137.5, 137.2, 136.7, 136.0, 135.1, 134.8, 134.6, 134.4, 129.1, 129.1, 128.5, 128.2, 127.4, 127.2, 126.3, 125.3, 125.2, 121.1, 120.8, 71.1, 70.4, 58.0, 57.1, 42.9, 42.1, 40.0, 39.9, 39.7, 38.8; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{18}\text{H}_{17}\text{NO}_4\text{SSeNa}$ [$\text{M}+\text{Na}]^+$: 445.9936; found: 445.9929.



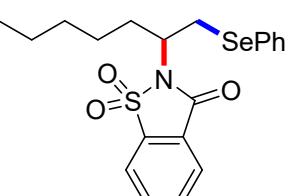
2-(2-(phenylselanyl)cycloheptyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5k')

(**5k'**): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 77.0 mg, 89% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.00-7.98 (m, 1H), δ 7.89-7.76 (m, 3H), δ 7.57-7.55 (m, 2H), δ 7.20-7.17 (m, 3H), δ 4.40-4.29 (m, 2H), δ 2.41-2.33 (m, 1H), δ 2.24-2.18 (m, 1H), δ 2.14-2.07 (m, 1H), δ 1.94-1.89 (m, 1H), δ 1.84-1.68 (m, 2H), δ 1.65-1.53 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 137.4, 135.4, 134.7, 134.2, 128.9, 127.7, 127.3, 125.2, 120.7, 61.2, 46.8, 33.1, 32.9, 27.6, 25.5, 25.4; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₀H₂₂NO₃SSe [M+H]⁺:436.0480; found:436.0466.



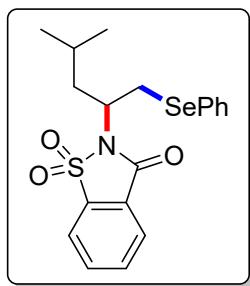
2-(1-(phenylselanyl)heptan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5l')

(**5l'**): Known compound^[4]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 57.6 mg, 68% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.04-8.02 (m, 1H), 7.91-7.80 (m, 3H), 7.65-7.63 (m, 2H), 7.30-7.27 (m, 3H), 4.05-3.95 (m, 2H), 3.75-3.67 (m, 1H), 1.84-1.76 (m, 1H), 1.71-1.52 (m, 2H), 1.49-1.24 (m, 3H), 0.88 (t, J = 7.3 Hz, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 137.7, 135.0, 134.5, 129.3, 128.0, 127.8, 127.3, 125.4, 121.1, 44.6, 42.4, 31.8, 29.7, 22.5, 14.1.

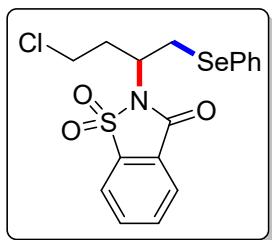


2-(1-(phenylselanyl)nonan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide

(5m'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 64.9 mg, 72% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.02 (m, 1H), 7.91-7.80 (m, 3H), 7.65-7.63 (m, 2H), 7.30-7.28 (m, 3H), 4.05-3.95 (m, 2H), 3.75-3.68 (m, 1H), 1.81-1.75 (m, 1H), 1.70-1.40 (m, 3H), 1.33-1.23 (m, 6H), 0.87-0.84 (m, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.3, 137.7, 135.0, 135.0, 134.5, 129.3, 128.0, 127.9, 127.3, 125.4, 121.1, 44.6, 42.5, 32.2, 31.8, 29.1, 27.6, 22.7, 14.2; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₁H₂₅NO₃SSeNa [M+Na]⁺: 474.0613; found: 474.0613.

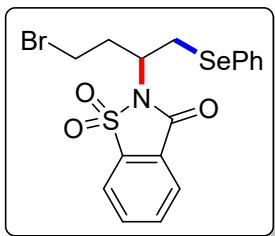


2-(4-methyl-1-(phenylselanyl)pentan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5n'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 53.3 mg, 63% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.04-8.02 (m, 1H), 7.91-7.80 (m, 3H), 7.66-7.63 (m, 2H), 7.31-7.28 (m, 3H), 4.06-3.92 (m, 2H), 3.78-3.70 (m, 1H), 2.05-1.96 (m, 1H), 1.59-1.46 (m, 2H), 0.90 (dd, J = 15.3, 6.7 Hz, 6H); **¹³C NMR** (CDCl₃, 100 MHz) δ 159.2, 137.6, 135.2, 134.9, 134.5, 129.3, 128.1, 127.5, 127.3, 125.4, 121.0, 45.1, 41.2, 40.4, 26.3, 23.4, 21.3; **HRMS** (ESI-Orbitrap) m/z calcd for C₁₉H₂₁NO₃SSeNa [M+Na]⁺: 446.0300; found: 446.0299.

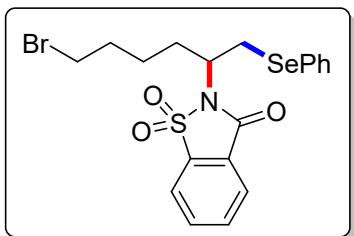


2-(4-chloro-1-(phenylselanyl)butan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5o'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 73.6 mg, 86% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.81 (m, 3H), 7.66-7.64 (m, 2H), 7.33-7.29 (m, 3H), 4.12 (dd, J = 14.6, 5.6 Hz, 1H), 3.95 (dd, J = 14.6, 9.9 Hz, 1H), 3.87-3.78 (m, 3H), 2.29-2.20 (m, 1H), 1.99-

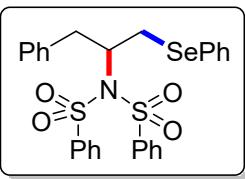
1.90 (m, 1H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.3, 137.6, 135.5, 135.1, 134.6, 129.5, 128.6, 127.2, 126.7, 125.5, 121.2, 44.4, 42.8, 39.5, 34.9; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{16}\text{ClNO}_3\text{SSeK}$ [M+K]⁺: 467.9336; found: 467.9332.



2-(4-bromo-1-(phenylselanyl)butan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5p'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 81.1 mg, 86% yield. Yellow oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.67-7.65 (m, 2H), 7.34-7.31 (m, 3H), 4.11 (dd, J = 14.6, 5.6 Hz, 1H), 3.95 (dd, J = 14.6, 9.9 Hz, 1H), 3.86-3.78 (m, 1H), 3.70-3.60 (m, 2H), 2.35-2.26 (m, 1H), 2.07-1.98 (m, 1H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.3, 137.5, 135.5, 135.1, 134.6, 129.5, 128.5, 127.1, 126.5, 125.5, 121.2, 44.3, 40.7, 34.9, 31.1; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{16}\text{BrNO}_3\text{SSeNa}$ [M+Na]⁺: 495.9092; found: 495.9089.

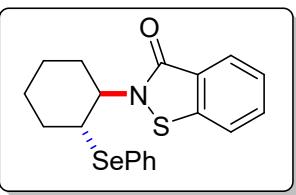


2-(6-bromo-1-(phenylselanyl)hexan-2-yl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (5q'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 80.5 mg, 80% yield. Yellow oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.05-8.03 (m, 1H), 7.92-7.80 (m, 3H), 7.66-7.63 (m, 2H), 7.32-7.30 (m, 3H), 4.07-3.94 (m, 2H), 3.71-3.65 (m, 1H), 3.40-3.35 (m, 2H), 1.87-1.79 (m, 4H), 1.63-1.55 (m, 2H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 159.3, 137.6, 135.1, 135.0, 134.5, 129.4, 128.2, 127.4, 127.2, 125.4, 121.1, 44.5, 42.0, 33.5, 32.4, 31.1, 26.2; HRMS (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{BrNO}_3\text{SSe}$ [M+H]⁺: 501.9585; found: 501.9590.

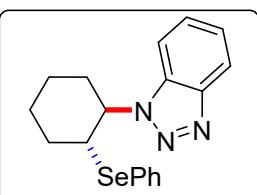


***N*-(1-phenyl-3-(phenylselanyl)propan-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (5r')**

(phenylsulfonyl)benzenesulfonamide (5r'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 45.1 mg, 39% yield. Colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.87-7.85 (m, 4H), 7.65-7.61 (m, 2H), 7.50-7.46 (m, 6H), 7.35-7.22 (m, 6H), 7.03-7.00 (m, 2H), 7.07-7.96 (m, 2H), 3.83-3.76 (m, 1H), 3.13 (dd, *J* = 14.7, 5.0 Hz, 1H), 2.68 (dd, *J* = 14.8, 10.5 Hz, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 139.3, 139.1, 135.2, 134.1, 129.3, 129.2, 129.1, 128.6, 128.5, 128.1, 126.7, 53.6, 45.0, 38.5; **HRMS** (ESI) m/z Calcd for C₂₇H₂₅NO₄S₂SeNa [M+Na]⁺: 594.0282; Found: 594.0274.

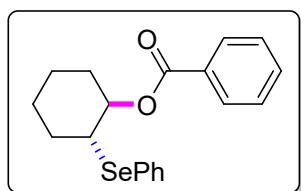


2-(2-(phenylselanyl)cyclohexyl)benzo[d]isothiazol-3(2H)-one (5s'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 22.3 mg, 29% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.05 (d, *J* = 7.8 Hz, 1H), 7.59-7.49 (m, 4H), 7.40-7.37 (m, 1H), 7.21-7.13 (m, 3H), 4.67 (s, 1H), 3.33 (s, 1H), 2.27-2.24 (m, 1H), 2.12-2.09 (m, 1H), 1.86-1.82 (m, 1H), 1.78-1.71 (m, 2H), 1.60-1.48 (m, 1H), 1.45-1.25 (m, 2H); **¹³C NMR** (CDCl₃, 100 MHz) δ 165.3, 140.2, 136.3, 131.7, 128.8, 128.0, 127.2, 126.9, 125.4, 125.2, 120.5, 57.2, 47.7, 34.6, 34.1, 26.8, 25.4; **HRMS** (ESI-Orbitrap) m/z calcd for C₁₉H₁₉NOSSeNa [M+Na]⁺: 412.0245; found: 412.0240.

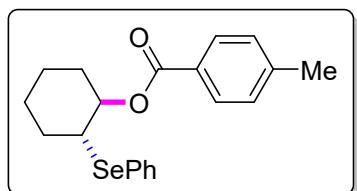


1-(2-(phenylselanyl)cyclohexyl)-1*H*-benzo[d][1,2,3]triazole (5t'): Known compound^[5]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 18.9 mg, 27%

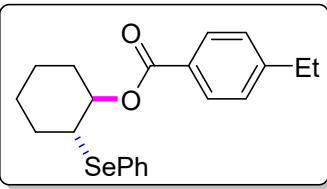
yield. White solid. m.p.: 106.3-107.9 °C. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.95 (m, 1H), 7.52-7.50 (m, 1H), 7.47-7.43 (m, 1H), 7.34-7.30 (m, 1H), 7.16-7.08 (m, 3H), 7.04-7.01 (m, 2H), 4.65 (td, *J* = 11.5, 4.4 Hz, 1H), 3.93 (ddd, *J* = 13.1, 10.3, 4.1 Hz, 1H), 2.47-2.44 (m, 1H), 2.34-2.24 (m, 1H), 2.18-2.15 (m, 1H), 1.99-1.98 (m, 1H), 1.88-1.86 (m, 1H), 1.76-1.65 (m, 1H), 1.59-1.46 (m, 1H); **¹³C NMR** (CDCl₃, 100 MHz) δ 145.9, 135.3, 133.0, 128.7, 127.9, 127.0, 123.7, 120.1, 109.8, 63.8, 47.3, 34.5, 34.1, 26.8, 25.4.



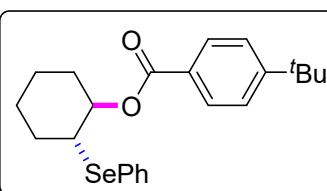
2-(phenylselanyl)cyclohexyl benzoate (7a): Known compound^[6]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 64.7 mg, 90 % yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.95 (m, 2H), 7.58-7.52 (m, 3H), 7.43-7.38 (m, 2H), 7.28-7.20 (m, 3H), 5.09 (td, *J* = 9.0, 4.0 Hz, 1H), 3.40 (ddd, *J* = 11.1, 8.2, 4.2 Hz, 1H), 2.27-2.21 (m, 2H), 1.81-1.67 (m, 2H), 1.64-1.35 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 166.0, 135.4, 133.0, 130.7, 129.9, 129.1, 128.7, 128.5, 127.8, 76.1, 46.4, 32.4, 31.7, 25.9, 23.7.



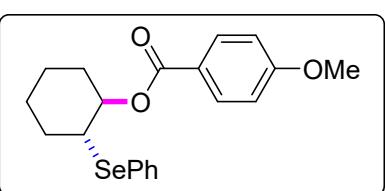
2-(phenylselanyl)cyclohexyl 4-methylbenzoate (7b): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 70.4 mg, 94 % yield. Orange-red oil liquid. **¹H NMR** (CDCl₃, 400 MHz) δ 7.87 (d, *J* = 8.2 Hz, 2H), 7.59-7.57 (m, 2H), 7.26-7.20 (m, 5H), 5.07 (td, *J* = 8.9, 3.9 Hz, 1H), 3.40 (td, *J* = 10.1, 4.2 Hz, 1H), 2.41 (s, 3H), 2.25-2.22 (m, 2H), 1.79-1.69 (m, 2H), 1.66-1.34 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 165.9, 143.5, 135.3, 129.8, 129.0, 129.0, 128.6, 127.9, 127.7, 75.6, 46.3, 32.3, 31.5, 25.7, 23.6, 21.7; **HRMS** (ESI-Orbitrap) *m/z* calcd for C₂₀H₂₂O₂SeNa [M+Na]⁺: 397.0683; Found: 397.0686.



2-(phenylselanyl)cyclohexyl 4-ethylbenzoate (7c): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 70.8 mg, 92 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.91-7.88 (m, 2H), 7.60-7.56 (m 2H), 7.26-7.21 (m, 5H), 5.08 (td, J = 8.9, 3.9 Hz, 1H), 3.40 (ddd, J = 11.2, 8.1, 4.2 Hz, 1H), 5.41 (q, J = 7.6 Hz, 2H), 2.26-2.21 (m, 2H), 1.80-1.66 (m, 2H), 1.64-1.35 (m, 4H), 1.26 (t, J = 7.6 Hz, 3H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.9, 149.7, 135.3, 129.9, 129.0, 128.6, 128.1, 127.9, 127.7, 75.6, 46.3, 32.3, 31.5, 29.1, 25.7, 23.6, 15.4; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{21}\text{H}_{24}\text{O}_2\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 411.0839; Found: 411.0844.

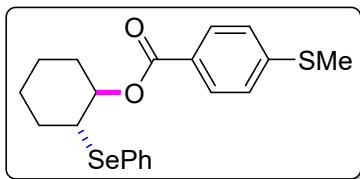


2-(phenylselanyl)cyclohexyl 4-(tert-butyl)benzoate (7d): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 75.8 mg, 91 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.94-7.91 (m, 2H), 7.59-7.57 (m, 2H), 7.45-7.42 (m, 2H), 7.27-7.21 (m, 3H), 5.08 (td, J = 8.9, 4.0 Hz, 1H), 3.40 (ddd, J = 11.2, 8.1, 4.1 Hz, 1H), 2.26-2.21 (m, 2H), 1.79-1.68 (m, 2H), 1.63-1.40 (m, 4H), 1.35 (s, 9H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.9, 156.5, 135.4, 129.7, 129.0, 128.5, 127.8, 127.7, 125.3, 75.5, 46.3, 35.1, 32.3, 31.5, 31.2, 25.7, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{28}\text{O}_2\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 439.1152; Found: 439.1149.

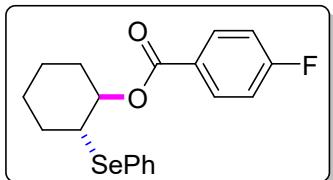


2-(phenylselanyl)cyclohexyl 4-methoxybenzoate (7e): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 30:1, v/v). 67.7 mg, 87 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.93-7.90 (m, 2H), 7.59-7.56 (m, 2H), 7.26-7.20 (m, 3H), 6.90-6.86 (m, 2H), 5.05 (td, J = 8.9, 4.0 Hz, 1H), 3.85 (s, 3H), 3.39

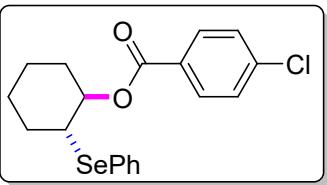
(ddd, $J = 11.2, 8.2, 4.2$ Hz, 1H), 2.24-2.20 (m, 2H), 1.78-1.68 (m, 2H), 1.65-1.34 (m, 4H); **^{13}C NMR** (CDCl_3 , 100 MHz) δ 165.5, 163.3, 135.3, 131.8, 129.0, 128.6, 127.6, 123.0, 113.6, 75.6, 55.5, 46.4, 32.3, 31.6, 25.7, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{20}\text{H}_{22}\text{O}_3\text{SeNa} [\text{M}+\text{Na}]^+$: 413.0632; Found: 413.0635.



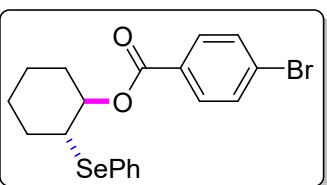
2-(phenylselanyl)cyclohexyl 4-(methylthio)benzoate (7f): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 66.0 mg, 81 % yield. Orange-red oil liquid. **^1H NMR** (CDCl_3 , 400 MHz) δ 7.86-7.83 (m, 2H), 7.57-7.55 (m, 2H), 7.26-7.20 (m, 5H), 5.06 (td, $J = 9.0, 3.9$ Hz, 1H), 3.38 (ddd, $J = 11.2, 8.3, 4.1$ Hz, 1H), 2.51 (s, 3H), 2.25-2.20 (m, 2H), 1.78-1.68 (m, 2H), 1.65-1.35 (m, 4H); **^{13}C NMR** (CDCl_3 , 100 MHz) δ 165.6, 145.3, 135.3, 130.1, 129.0, 128.6, 127.7, 126.7, 124.9, 75.8, 46.3, 32.3, 31.6, 25.7, 23.4, 15.0; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{20}\text{H}_{22}\text{O}_2\text{SSeNa} [\text{M}+\text{Na}]^+$: 429.0403; Found: 429.0399.



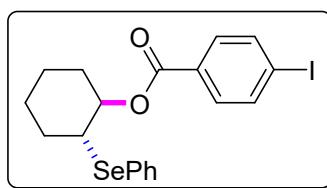
2-(phenylselanyl)cyclohexyl 4-fluorobenzoate (7g): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 73.0 mg, 97 % yield. Orange-red oil liquid. **^1H NMR** (CDCl_3 , 400 MHz) δ 7.96-7.91 (m, 2H), 7.56-7.54 (m, 2H), 7.27-7.19 (m, 3H), 7.08-7.03 (m, 2H), 5.07 (td, $J = 9.1, 4.0$ Hz, 1H), 3.38 (ddd, $J = 11.5, 8.4, 4.1$ Hz, 1H), 2.26-2.20 (m, 2H), 1.80-1.69 (m, 2H), 1.66-1.33 (m, 4H); **^{13}C NMR** (CDCl_3 , 100 MHz) δ 165.8 (d, $J_{CF} = 252$ Hz), 164.9, 135.2, 132.3 (d, $J_{CF} = 10$ Hz), 129.0, 128.6, 127.7, 126.8 (d, $J_{CF} = 3$ Hz), 115.4 (d, $J_{CF} = 22$ Hz), 76.4, 46.3, 32.4, 31.7, 25.8, 23.7; **^{19}F NMR** (CDCl_3 , 376 MHz) δ -31.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{FO}_2\text{SeNa} [\text{M}+\text{Na}]^+$: 401.0432; Found: 401.0430.



2-(phenylselanyl)cyclohexyl 4-chlorobenzoate (7h): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 66.4 mg, 84 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.87-7.83 (m, 2H), 7.57-7.54 (m, 2H), 7.26-7.20 (m, 5H), 7.38-7.34 (m, 2H), 7.28-7.20 (m, 3H), 5.07 (td, J = 9.1, 3.9 Hz, 1H), 3.38 (ddd, J = 11.3, 8.6, 4.1 Hz, 1H), 2.27-2.20 (m, 2H), 1.80-1.69 (m, 2H), 1.65-1.34 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.0, 139.3, 135.2, 131.2, 129.1, 129.0, 128.7, 128.6, 127.7, 76.6, 46.3, 32.4, 31.8, 25.9, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{ClO}_2\text{SeNa}$ [M+Na]⁺: 417.0136; Found: 417.0140.

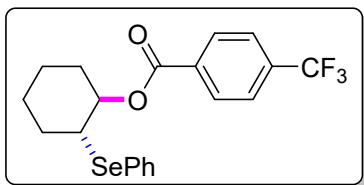


2-(phenylselanyl)cyclohexyl 4-bromobenzoate (7i): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 69.4 mg, 79 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.79-7.76 (m, 2H), 7.55-7.51 (m, 4H), 7.26-7.19 (m, 3H), 5.06 (td, J = 9.1, 4.0 Hz, 1H), 3.37 (ddd, J = 11.4, 8.6, 4.1 Hz, 1H), 2.26-2.19 (m, 2H), 1.79-1.69 (m, 2H), 1.94-1.33 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.1, 135.2, 131.6, 131.3, 129.5, 125.1, 128.6, 128.0, 127.7, 76.6, 46.2, 32.4, 31.7, 25.8, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{BrNaO}_2\text{SeNa}$ [M+Na]⁺: 460.9631; Found: 460.9638.

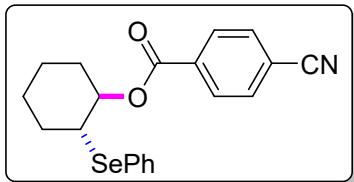


2-(phenylselanyl)cyclohexyl 4-iodobenzoate (7j): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 68.8 mg, 71 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.76-7.73 (m, 2H), 7.64-7.60 (m, 2H), 7.55-7.53 (m, 2H), 7.28-7.19 (m, 3H), 5.06 (td, J = 9.2, 4.0 Hz, 1H), 3.37 (ddd, J = 11.3,

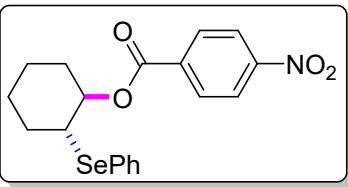
8.6, 4.1 Hz, 1H), 2.26-2.19 (m, 2H), 1.79-1.69 (m, 2H), 1.64-1.33 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.4, 137.7, 135.2, 131.2, 130.0, 129.1, 128.5, 127.7, 100.7, 76.5, 46.2, 32.4, 31.7, 25.8, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{IO}_2\text{SeNa} [\text{M}+\text{Na}]^+$: 508.9493; Found: 508.9495.



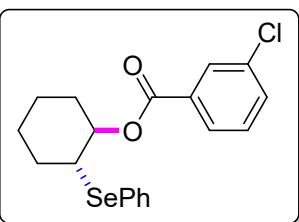
2-(phenylselanyl)cyclohexyl 4-(trifluoromethyl)benzoate (7k): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 61.3 mg, 72% yield. Yellow oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.03-8.01 (m, 2H), 7.66-7.64 (m, 2H), 7.55-7.53 (m, 2H), 7.24-7.18 (m, 3H), 5.10 (td, J = 9.3, 4.0 Hz, 1H), 3.40 (ddd, J = 11.4, 8.8, 4.1 Hz, 1H), 2.28-2.21 (m, 2H), 1.81-1.71 (m, 2H), 1.65-1.35 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 164.7, 135.2, 134.9, 134.3 (q, J_{CF} = 32 Hz), 133.8, 130.2, 129.1, 128.5, 127.8, 125.3 (q, J_{CF} = 3 Hz), 123.8 (q, J_{CF} = 271 Hz), 77.0, 46.2, 32.4, 31.8, 25.9, 23.7; **¹⁹F NMR** (CDCl_3 , 376 MHz) δ -63.1; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{20}\text{H}_{19}\text{F}_3\text{O}_2\text{SeNa} [\text{M}+\text{Na}]^+$: 451.0400; Found: 451.0397.



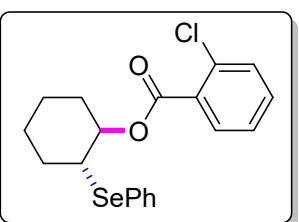
2-(phenylselanyl)cyclohexyl 4-cyanobenzoate (7l): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 30:1, v/v). 66.9 mg, 85% yield. White solid. m.p.: 54.9-55.7 °C. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.98-7.95 (m, 2H), 7.68-7.65 (m, 2H), 7.53-7.51 (m, 2H), 7.26-7.17 (m, 3H), 5.09 (td, J = 9.4, 4.0 Hz, 1H), 3.38 (ddd, J = 1.5, 8.9, 4.1 Hz, 1H), 2.27-2.19 (m, 2H), 1.81-1.70 (m, 2H), 1.63-1.36 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 164.2, 135.1, 134.3, 132.1, 130.2, 129.1, 128.5, 127.7, 118.2, 116.2, 77.5, 46.1, 32.3, 31.9, 25.9, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{20}\text{H}_{19}\text{NO}_2\text{SeNa} [\text{M}+\text{Na}]^+$: 408.0479; Found: 408.0480.



2-(phenylselanyl)cyclohexyl 4-nitrobenzoate (7m): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 30:1, v/v). 57.6 mg, 72 % yield. White solid. m.p.: 71.6-72.3 °C. **1H NMR** (CDCl_3 , 400 MHz) δ 8.22-8.20 (m, 2H), 8.04-8.02 (m, 2H), 7.54-7.51 (m, 2H), 7.24-7.18 (m, 3H), 5.10 (td, J = 9.3, 4.1 Hz, 1H), 3.39 (ddd, J = 11.5, 8.9, 4.1 Hz, 1H), 2.29-2.21 (m, 2H), 1.82-1.72 (m, 2H), 1.65-1.37 (m, 4H); **13C NMR** (CDCl_3 , 100 MHz) δ 164.0, 150.5, 135.9, 135.1, 130.8, 129.1, 128.5, 127.8, 123.4, 77.7, 46.1, 32.4, 31.9, 25.9, 23.8; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{NO}_4\text{SeNa} [\text{M}+\text{Na}]^+$: 428.0377; Found: 428.0380.

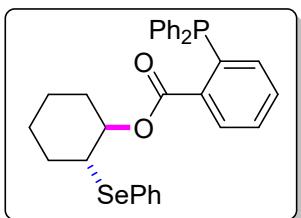


2-(phenylselanyl)cyclohexyl 3-chlorobenzoate (7n): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 64.5 mg, 82 % yield. Orange-red oil liquid. **1H NMR** (CDCl_3 , 400 MHz) δ 7.87-7.81 (m, 2H), 7.57-7.53 (m, 2H), 7.51-7.48 (m, 1H), 7.33 (t, J = 15.8 Hz, 1H), 7.26-7.18 (m, 3H), 5.09 (td, J = 9.2, 4.0 Hz, 1H), 3.38 (ddd, J = 11.3, 8.7, 4.1 Hz, 1H), 2.29-2.18 (m, 2H), 1.81-1.70 (m, 2H), 1.65-1.33 (m, 4H); **13C NMR** (CDCl_3 , 100 MHz) δ 164.1, 135.2, 134.4, 132.9, 132.3, 129.8, 129.6, 129.0, 128.5, 127.9, 127.8, 76.8, 46.2, 32.4, 31.8, 25.9, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{ClO}_2\text{SeNa} [\text{M}+\text{Na}]^+$: 417.0136; Found: 417.0140.

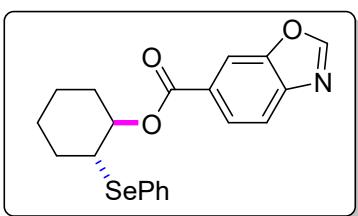


2-(phenylselanyl)cyclohexyl 2-chlorobenzoate (7o): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 74.0 mg, 94 % yield. Orange-red oil

liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.77 (dd, $J = 7.7, 1.6$ Hz, 1H), 7.61-7.57 (m, 2H), 7.45-7.37 (m, 2H), 7.29-7.21 (m, 4H), 5.11 (td, $J = 9.1, 4.1$ Hz, 1H), 3.37 (ddd, $J = 11.1, 8.2, 4.1$ Hz, 1H), 2.29-2.19 (m, 2H), 1.81-1.65 (m, 2H), 1.63-1.32 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 164.9, 135.4, 133.7, 132.4, 131.5, 131.0, 130.6, 129.0, 128.2, 127.8, 126.6, 76.4, 45.9, 32.3, 31.4, 25.6, 23.5; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{19}\text{ClO}_2\text{SeNa}$ [M+Na]⁺: 417.0136; Found: 417.0140.

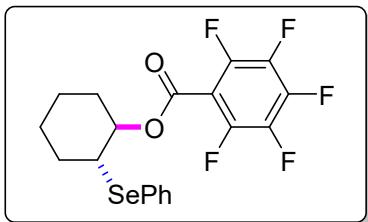


2-(phenylselanyl)cyclohexyl 2-(diphenylphosphoranylidene)benzoate (7p): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 2:1, v/v). 81.9 mg, 75% yield. Colourless oil. **¹H NMR** (CDCl_3 , 600 MHz) δ 7.96-7.95 (m, 1H), 7.72-7.63 (m, 5H), 7.61-7.43 (m, 10H), 7.28-7.21 (m, 4H), 4.76-4.73 (m, 1H), 2.98-2.94 (m, 1H), 2.04 (d, $J = 13.9$ Hz, 1H), 1.90-1.89 (m, 1H), 1.58 (s, 2H), 1.40-1.38 (m, 1H), 1.26-1.21 (m, 3H); **¹³C NMR** (CDCl_3 , 150 MHz) δ 165.9 (d, $J = 2.5$ Hz), 136.1 (d, $J = 5.0$ Hz), 135.5, 135.0 (d, $J = 7.5$ Hz), 133.6 (dd, $J = 90.0, 33.8$ Hz), 132.8, 132.2 (d, $J = 8.8$ Hz), 131.9 (d, $J = 8.8$ Hz), 131.5 (dd, $J = 12.5, 1.3$ Hz), 131.0 (d, $J = 8.8$ Hz), 130.8 (d, $J = 7.5$ Hz), 128.9, 128.3 (d, $J = 11.3$ Hz), 128.0, 127.7; **³¹P NMR** (CDCl_3 , 240 MHz) δ 34.1; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{31}\text{H}_{30}\text{PO}_2\text{Se}$ [M+H]⁺: 545.1149; Found: 545.1152.

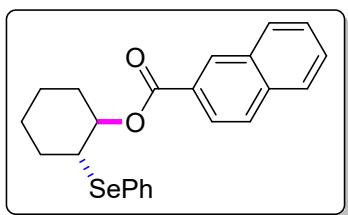


2-(phenylselanyl)cyclohexyl benzo[d]oxazole-6-carboxylate (7q): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 53.6 mg, 67% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.21 (s, 1H), 8.12 (dd, $J = 1.4, 0.5$ Hz, 1H), 8.0 (dd, $J = 8.4, 1.5$ Hz, 1H), 7.78 (d, $J = 8.4$ Hz, 1H), 7.56-7.53 (m, 3H), 7.22-7.17 (m, 3H), 5.11 (td, $J = 9.2, 4.0$ Hz, 1H), 3.41 (ddd, $J = 11.3, 8.7, 4.1$ Hz,

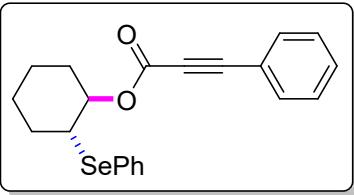
1H), 2.27-2.23 (m, 2H), 1.80-1.70 (m, 2H), 1.66-1.37 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 165.2, 154.9, 149.7, 143.8, 135.2, 129.0, 128.5, 128.3, 127.7, 126.5, 120.2, 112.9, 76.8, 46.3, 32.4, 31.8, 25.8, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₀H₁₉NO₃SeNa [M+Na]⁺: 424.0428; Found: 424.0430.



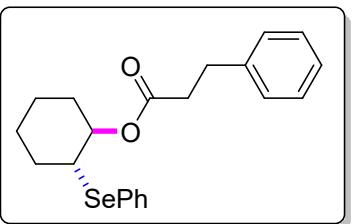
2-(phenylselanyl)cyclohexyl 2,3,4,5,6-pentafluorobenzoate (7r): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 48.0 mg, 53 % yield. Orange-red oil liquid. **¹H NMR** (CDCl₃, 400 MHz) δ 7.57-7.55 (m, 2H), 7.24-7.21 (m, 3H), 5.15 (td, *J* = 8.9, 4.0 Hz, 1H), 3.33 (ddd, *J* = 10.9, 7.9, 4.2 Hz, 1H), 2.24-2.19 (m, 2H), 1.79-1.67 (m, 2H), 1.65-1.53 (m, 2H), 1.47-1.36 (m, 2H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.3, 135.2, 131.6, 129.3, 129.0, 128.2, 127.8, 108.5, 78.3, 45.1, 31.7, 31.1, 25.4, 23.3; **¹⁹F NMR** (CDCl₃, 376 MHz) δ -138.0, 149.1, 160.7; **HRMS** (ESI-Orbitrap) m/z calcd for C₁₉H₁₅F₅O₂SeNa [M+Na]⁺: 473.0055; Found: 473.0058.



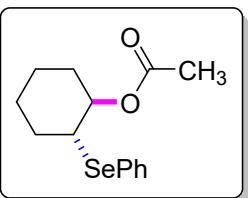
2-(phenylselanyl)cyclohexyl 2-naphthoate (7s): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 65.9 mg, 80% yield. Orange-red oil liquid. **¹H NMR** (CDCl₃, 400 MHz) δ 8.50 (s, 1H), 8.02 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.93-7.85 (m, 3H), 7.62-7.52 (m, 4H), 7.24-7.19 (m, 3H), 5.18 (td, *J* = 9.1, 4.0 Hz, 1H), 3.47 (ddd, *J* = 11.3, 8.4, 4.2 Hz, 1H), 2.33-2.27 (m, 2H), 1.83-1.70 (m, 2H), 1.68-1.37 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 166.0, 135.6, 135.3, 132.5, 131.2, 129.5, 129.0, 128.7, 128.2, 128.1, 127.8, 127.7, 126.6, 125.5, 76.2, 46.4, 32.4, 31.7, 25.8, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₃H₂₂O₂SeNa [M+Na]⁺: 433.0683; Found: 433.0679.



2-(phenylselanyl)cyclohexyl 3-phenylpropiolate (7t): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 54.3 mg, 71% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.66-7.62 (m, 2H), 7.60-7.57 (m, 2H), 7.47-7.43 (m, 1H), 7.40-7.35 (m, 2H), 7.29-7.26 (m, 3H), 5.01 (td, J = 9.0, 4.2 Hz, 1H), 3.27 (ddd, J = 11.3, 8.5, 4.2 Hz, 1H), 2.25-2.13 (m, 2H), 1.78-1.64 (m, 2H), 1.59-1.26 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 153.4, 135.6, 133.0, 130.6, 129.0, 128.6, 128.2, 127.9, 119.7, 86.4, 80.9, 77.2, 45.7, 32.3, 31.4, 25.6, 23.5; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{21}\text{H}_{20}\text{O}_2\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 407.0532; Found: 407.0526.

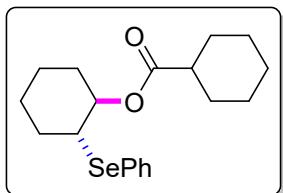


2-(phenylselanyl)cyclohexyl 3-phenylpropanoate (7u): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 64.1 mg, 83 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.60-7.55 (m, 2H), 7.32-7.26 (m, 5H), 7.22-7.18 (m, 3H), 4.88 (td, J = 4.2, 9.2 Hz, 1H), 3.21 (ddd, J = 11.4, 8.7, 4.1 Hz, 1H), 2.95 (t, J = 7.7 Hz, 2H), 2.57-2.53 (m, 2H), 2.18-2.03 (m, 2H), 1.72-1.64 (m, 2H), 1.56-1.26 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 172.2, 140.7, 135.1, 129.0, 128.7, 128.5, 128.4, 127.6, 126.3, 75.4, 46.1, 36.0, 32.3, 31.8, 31.0, 25.8, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{21}\text{H}_{24}\text{O}_2\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 411.0839; Found: 411.0844.

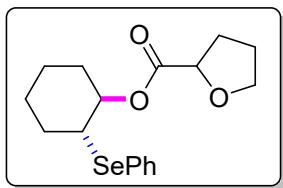


2-(phenylselanyl)cyclohexyl acetate (7v): Known compound^[7]. (Eluent:

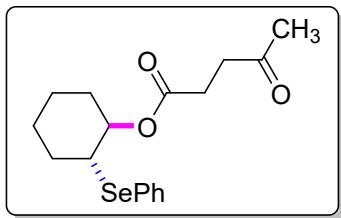
petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 54.4 mg, 92 % yield. Yellow oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.59-7.56 (m, 2H), 7.29-7.25 (m, 3H), 4.84 (td, J = 9.3, 4.2 Hz, 1H), 3.21 (ddd, J = 11.5, 8.9, 4.1 Hz, 1H), 2.17-2.05 (m, 2H), 1.95 (s, 3H), 1.74-1.62 (m, 2H), 1.55-1.24 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 170.5, 135.2, 129.0, 128.7, 127.7, 75.6, 46.2, 32.4, 31.9, 26.0, 23.7, 21.3.



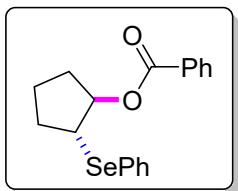
2-(phenylselanyl)cyclohexyl cyclohexanecarboxylate (7w): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 70:1, v/v). 54.4 mg, 74 % yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.59-7.55 (m, 2H), 7.28-7.25 (m, 3H), 4.82 (td, J = 9.0, 4.1 Hz, 1H), 3.21 (ddd, J = 11.4, 8.5, 4.1 Hz, 1H), 2.24-2.16 (m, 1H), 2.14-2.04 (m, 2H), 1.94-1.84 (m, 2H), 1.76-1.62 (m, 5H), 1.55-1.22 (m, 5H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 175.3, 135.2, 129.0, 128.7, 128.5, 128.4, 127.6, 74.5, 46.0, 43.4, 32.3, 31.7, 29.1, 25.9, 25.8, 25.6 (d, J = 1 Hz), 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{19}\text{H}_{26}\text{O}_2\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 389.0996; Found: 389.0996.



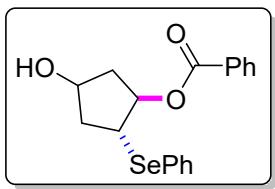
2-(phenylselanyl)cyclohexyl tetrahydrofuran-2-carboxylate (7x): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 58.5 mg, 83% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.57-7.55 (m, 2H), 7.29-7.24 (m, 3H), 4.89-4.82 (m, 1H), 4.44-4.33 (m, 1H), 4.08-3.99 (m, 1H), 3.93-3.88 (m, 1H), 3.24-3.13 (m, 1H), 2.25-1.83 (m, 6H), 1.72-1.61 (m, 2H), 1.50-1.25 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 172.8, 172.6, 135.4, 129.0, 128.1, 127.9, 127.8, 127.8, 76.8, 75.5, 75.1, 69.4, 46.1, 45.8, 32.7, 32.4, 31.8, 31.6, 25.9, 25.8, 25.2, 30.0, 23.7, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{22}\text{O}_3\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 377.0632; Found: 377.0627.



2-(phenylselanyl)cyclohexyl 4-oxopentanoate (7y): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 10:1, v/v). 55.5 mg, 79 % yield. Colorless oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.57-7.54 (m, 2H), 7.27-7.24 (m, 3H), 4.82 (td, J = 9.2, 4.2 Hz, 1H), 3.20 (ddd, J = 11.4, 8.8, 4.1 Hz, 1H), 2.78-2.61 (m, 2H), 2.55-2.38 (m, 2H), 2.16 (s, 3H), 2.14-2.03 (m, 2H), 1.72-1.62 (m, 2H), 1.53-1.25 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 206.7, 172.0, 135.1, 129.0, 128.7, 127.6, 75.7, 46.4, 38.1, 32.3, 31.7, 30.0, 28.3, 25.8, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{22}\text{O}_3\text{SeNa}$ [$\text{M}+\text{Na}]^+$: 377.0632; Found: 377.0638.

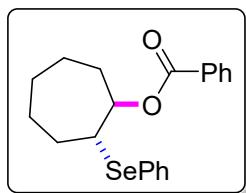


2-(phenylselanyl)cyclopentyl benzoate (7z): Known compound^[6]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 44.4 mg, 64% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.97-7.94 (m, 2H), 7.63-7.59 (m, 2H), 7.57-7.52 (m, 1H), 7.44-7.39 (m, 2H), 7.27-7.24 (m, 3H), 5.42-5.39 (m, 1H), 3.82-3.78 (m, 1H), 2.38-2.27 (m, 2H), 1.91-1.76 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.9, 134.3, 133.0, 130.5, 129.7, 129.3, 129.2, 128.4, 127.7, 82.2, 46.2, 31.4, 31.1, 22.8.

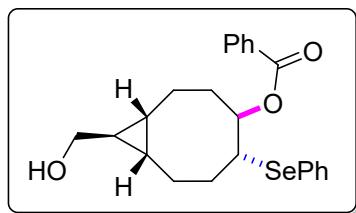


4-hydroxy-2-(phenylselanyl)cyclopentyl benzoate (7a'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 2:1, v/v). d.r. = 8:1, 69.5 mg, 96% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.93-7.90 (m, 2H), 7.63-7.60 (m, 2H), 7.56-7.52 (m, 1H), 7.42-7.38 (m, 2H), 7.26-7.23 (m, 3H), 5.53 (dt, J = 6.7, 4.0 Hz, 1H), 4.57-4.51 (m, 1H), 3.76-3.71 (m, 1H), 2.72-2.60 (m, 1H), 2.40-2.33 (m, 1H),

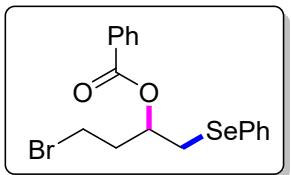
2.21-2.13 (m, 2H), 1.89-1.83 (m, 1H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 81.2, 81.0, 134.9, 134.6, 133.1, 130.1, 130.0, 129.7, 129.7, 129.3, 129.2, 129.1, 128.4, 127.9, 81.2, 80.9, 71.6, 71.6, 43.8, 43.7, 41.6, 41.3, 40.9; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{18}\text{H}_{19}\text{O}_3\text{Se}$ $[\text{M}+\text{H}]^+$: 361.0502; Found: 361.0507.



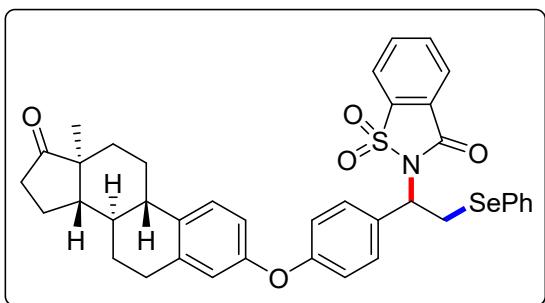
2-(phenylselanyl)cycloheptyl benzoate (7b'): Known compound^[6]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 63.4 mg, 85% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.01-7.98 (m, 2H), 7.61-7.52 (m, 3H), 7.44-7.40 (m, 2H), 7.25-7.21 (m, 3H), 5.37 (ddd, J = 8.3, 5.5, 3.6 Hz, 1H), 3.64 (ddd, J = 9.1, 6.7, 3.4 Hz, 1H), 2.27-2.20 (m, 1H), 2.06-1.92 (m, 2H), 1.87-1.51 (m, 7H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 165.7, 135.0, 132.9, 130.7, 129.7, 129.3, 129.1, 128.4, 127.7, 78.6, 48.8, 32.0, 31.7, 28.3, 26.6, 22.3.



(1R,8S,9S)-9-(hydroxymethyl)-5-(phenylselanyl)bicyclo[6.1.0]nonan-4-yl benzoate (7c'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 1:1, v/v). d.r. = 8:1, 85 mg, 99% yield. Colourless oil. **¹H NMR** (CDCl_3 , 400 MHz) δ 8.06-8.01 (m, 2H), 7.55-7.50 (m, 3H), 7.42 (q, J = 7.1 Hz, 2H), 7.27-7.25 (m, 3H), 5.21 (dt, J = 8.3, 3.0 Hz, 1H), 4.16-4.11 (m, 1H), 3.54-3.45 (m, 1H), 2.23-1.85 (m, 6H), 1.53 (s, 1H), 1.35-1.21 (m, 2H), 0.85-0.79 (m, 1H), 0.68-0.54 (m, 1H); **¹³C NMR** (CDCl_3 , 150 MHz) δ 165.9, 165.8, 134.2, 134.0, 133.1, 133.1, 130.8, 130.5, 130.3, 130.2, 129.9, 129.9, 129.7, 129.2, 128.5, 128.4, 127.4, 127.4, 76.3, 76.2, 66.6, 48.4, 47.4, 36.3, 35.3, 33.9, 33.8, 29.7, 29.4, 27.5, 26.8, 26.7, 25.2, 24.6, 22.6, 21.0, 20.7, 20.3; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{23}\text{H}_{26}\text{O}_3\text{SeNa}$ $[\text{M}+\text{Na}]^+$: 453.0939; Found: 453.0938.

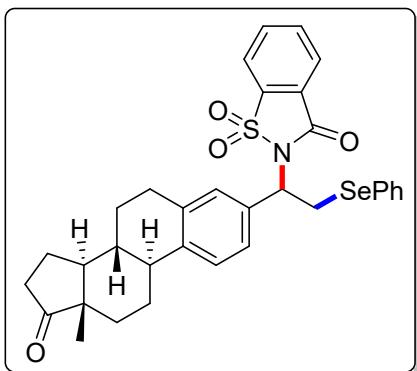


4-bromo-1-(phenylselanyl)butan-2-yl benzoate (7d'): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 61.2 mg, 74% yield. Colorless oil liquid. **1H NMR** (CDCl_3 , 400 MHz) δ 8.01-7.98 (m, 2H), 7.66-7.62 (m, 2H), 7.59-7.55 (m, 1H), 7.46-7.42 (m, 2H), 7.36-7.28 (m, 3H), 4.61 (dd, J = 11.5, 5.2 Hz, 1H), 4.43 (dd, J = 11.4, 7.8 Hz, 1H), 3.76-3.62 (m, 3H), 2.41-2.33 (m, 1H), 2.19-2.10 (m, 1H); **13C NMR** (CDCl_3 , 100 MHz) δ 166.2, 135.6, 133.3, 129.9, 129.8, 129.4, 128.5, 128.4, 127.2, 67.5, 41.8, 35.0, 31.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{17}\text{BrO}_2\text{SeNa} [\text{M}+\text{Na}]^+$: 434.9475; Found: 434.9479.

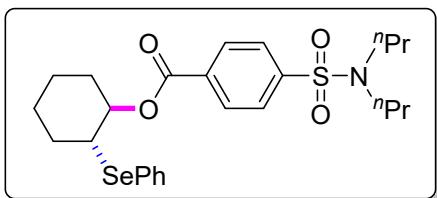


2-(1-((4-(((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)oxy)phenyl)-2-(phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (8a): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 53 mg, 75% yield. white solid. m.p.: 162.7-166.0 °C. **1H NMR** (CDCl_3 , 400 MHz) δ 7.98-7.96 (m, 1H), 7.88-7.77 (m, 3H), 7.56-7.54 (m, 2H), 7.53-7.50 (m, 2H), 7.25-7.22 (m, 4H), 6.95-6.92 (m, 2H), 6.81-6.75 (m, 2H), 5.36 (t, J = 8.0 Hz, 1H), 5.30 (s, 1H), 4.05 (dd, J = 12.9, 8.3 Hz, 1H), 3.80 (dd, J = 12.8, 7.9 Hz, 1H), 2.89-2.85 (m, 2H), 2.54-2.48 (m, 1H), 2.41-2.38 (m, 1H), 2.31-2.25 (m, 1H), 2.19-1.95 (m, 4H), 1.63-1.51 (m, 5H), 0.92 (s, 3H); **13C NMR** (CDCl_3 , 100 MHz) δ 158.8, 158.1, 154.4, 138.5, 137.5, 135.4, 134.9, 134.4, 133.9, 130.9, 130.2, 129.3, 129.1, 127.8, 127.3, 126.8, 125.3, 120.9, 119.7, 118.1, 117.0, 57.5, 50.5, 48.1, 44.2, 38.3, 36.0, 31.7, 29.6, 29.3, 26.5, 26.0, 21.7, 14.0; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{39}\text{H}_{38}\text{NO}_5\text{SSe} [\text{M}+\text{H}]^+$: 712.1630;

found: 712.1630.

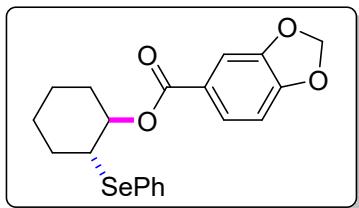


2-(1-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)-2-phenylselanyl)ethyl)benzo[d]isothiazol-3(2H)-one 1,1-dioxide (8b): Known compound^[3]. (Eluent: petroleum ether (60-90 °C)/EtOAc = 5:1, v/v). 112.6 mg, 91% yield. colorless oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.98-7.96 (m, 1H), 7.87-7.75 (m, 3H), 7.56-7.53 (m, 2H), 7.37-7.34 (m, 1H), 7.27-7.22 (m, 5H), 5.35 (td, *J* = 8.0, 2.6 Hz, 1H), 4.15-4.04 (m, 1H), 3.83-3.77 (m, 1H), 2.89-2.86 (m, 2H), 2.49 (dd, *J* = 19.3, 9.0 Hz, 1H), 2.42-2.37 (m, 1H), 2.29-2.24 (m, 1H), 2.18-1.94 (m, 4H), 1.65-1.57 (m, 2H), 1.54-1.42 (m, 4H), 0.89 (s, 3H); **¹³C NMR** (CDCl₃, 100 MHz) δ 158.8, 158.8, 140.4, 137.4, 136.8, 134.8, 134.3, 133.8, 133.8, 129.2, 127.6, 127.3, 126.0, 125.9, 125.6, 125.6, 125.2, 120.8, 57.7, 50.6, 48.0, 44.4, 38.0, 35.9, 31.6, 29.5, 29.2, 26.5, 25.6, 21.7, 13.9.

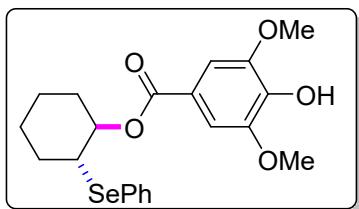


2-(phenylselanyl)cyclohexyl 4-(N,N-dipropylsulfamoyl)benzoate (8c): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 6:1, v/v). 89.4 mg, 86% yield. Colourless oil. **¹H NMR** (CDCl₃, 600 MHz) δ 8.02 (d, *J* = 7.9 Hz, 2H), 7.83 (d, *J* = 7.9 Hz, 2H), 7.55 (d, *J* = 7.1 Hz, 2H), 7.28-7.21 (m, 3H), 5.11 (t, *J* = 9.1 Hz, 1H), 3.43-3.39 (m, 1H), 3.11 (t, *J* = 7.2 Hz, 4H), 2.26 (t, *J* = 7.2 Hz, 4H), 1.82-1.73 (m, 2H), 1.66-1.53 (m, 6H), 1.49-1.38 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 6H); **¹³C NMR**

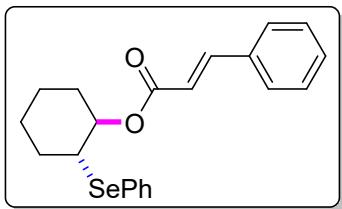
(CDCl₃, 100 MHz) δ 164.5, 144.1, 135.2, 133.9, 130.4, 129.1, 128.6, 127.8, 127.0, 50.1, 46.2, 32.4, 31.8, 25.9, 23.8, 22.1, 11.3; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₅H₃₄NO₄SSe [M+H]⁺: 524.1368; Found: 524.1368.



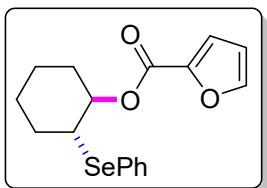
2-(phenylselanyl)cyclohexyl benzo[d][1,3]dioxole-5-carboxylate (8d): New compound. (Eluent: petroleum ether (60–90 °C)/EtOAc = 50:1, v/v). 57.6 mg, 71% yield. Orange-red oil liquid. **¹H NMR** (CDCl₃, 400 MHz) δ 7.58–7.54 (m, 3H), 7.37 (d, *J* = 1.7 Hz, 1H), 7.26–7.20 (m, 3H), 6.80 (d, *J* = 8.2 Hz, 1H), 6.03–6.02 (m, 2H), 5.05 (td, *J* = 9.0, 3.9 Hz, 1H), 3.37 (ddd, *J* = 11.1, 8.3, 4.1 Hz, 1H), 2.25–2.18 (m, 2H), 1.79–1.68 (m, 2H), 1.64–1.36 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 165.2, 151.6, 147.7, 135.3, 129.0, 128.6, 127.7, 125.5, 124.6, 109.7, 108.0, 101.8, 75.9, 46.3, 32.3, 31.6, 25.8, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₀H₂₀O₄SeNa [M+Na]⁺: 427.0425; Found: 427.0425.



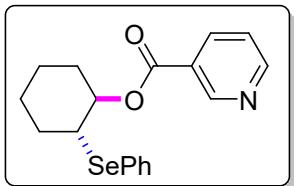
2-(phenylselanyl)cyclohexyl 4-hydroxy-3,5-dimethoxybenzoate (8e): New compound. (Eluent: petroleum ether (60–90 °C)/EtOAc = 2:1, v/v). 48.4 mg, 56% yield. Orange-red oil liquid. **¹H NMR** (CDCl₃, 400 MHz) δ 7.56–7.54 (m, 2H), 7.27 (s, 2H), 7.24–7.18 (m, 3H), 5.93 (s, 1H), 5.05 (td, *J* = 9.2, 4.0 Hz, 1H), 3.91 (s, 6H), 3.36 (ddd, *J* = 12.0, 8.6, 4.2 Hz, 1H), 2.26–2.19 (m, 2H), 1.78–1.69 (m, 2H), 1.65–1.33 (m, 4H); **¹³C NMR** (CDCl₃, 100 MHz) δ 165.7, 146.6, 139.3, 135.4, 129.0, 128.6, 127.7, 121.5, 106.9, 75.9, 55.5, 46.7, 32.7, 31.8, 25.9, 23.8; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₁H₂₄O₅SeNa [M+Na]⁺: 459.0687; Found: 459.0683.



2-(phenylselanyl)cyclohexyl cinnamate (8f): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 67.2 mg, 87% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.63-7.58 (m, 3H), 7.50-7.47 (m, 2H), 7.40-7.36 (m, 3H), 7.27-7.24 (m, 3H), 6.29 (d, J = 16.0 Hz, 1H), 4.98 (td, J = 9.1, 4.0 Hz, 1H), 3.32 (ddd, J = 11.4, 8.7, 4.1 Hz, 1H), 2.24-2.15 (m, 2H), 1.77-1.67 (m, 2H), 1.61-1.34 (m, 4H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 166.3, 144.8, 135.3, 134.6, 130.3, 129.0, 128.9, 128.8, 128.2, 127.6, 118.4, 75.8, 46.4, 32.4, 31.9, 25.9, 23.7; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{21}\text{H}_{22}\text{O}_2\text{SeNa}$ [M+Na]⁺: 409.0683; Found: 409.0686.

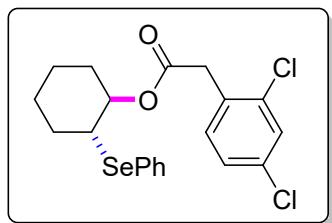


2-(phenylselanyl)cyclohexyl furan-2-carboxylate (8g): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 53.9 mg, 77% yield. Orange-red oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 7.59-7.54 (m, 3H), 7.27-7.20 (m, 3H), 7.03 (dd, J = 3.5, 0.8 Hz, 1H), 5.06 (td, J = 9.3, 4.1 Hz, 1H), 3.33 (ddd, J = 11.3, 8.5, 4.1 Hz, 1H), 2.24-2.15 (m, 2H), 1.78-1.66 (m, 2H), 1.60-1.49 (m, 2H), 1.45-1.32 (m, 2H); **¹³C NMR** (CDCl_3 , 100 MHz) δ 158.1, 146.3, 144.9, 135.3, 129.0, 128.4, 127.7, 118.0, 111.8, 76.1, 46.1, 32.4, 31.7, 25.8, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for $\text{C}_{17}\text{H}_{18}\text{O}_3\text{Na}$ [M+Na]⁺: 373.0319; Found: 373.0316.

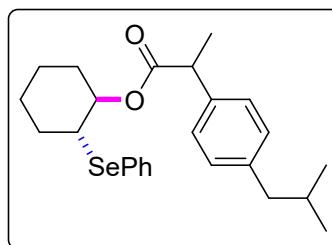


2-(phenylselanyl)cyclohexyl nicotinate (8h): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 8:1, v/v). 27.9 mg, 39% yield. Colorless oil liquid. **¹H NMR** (CDCl_3 , 400 MHz) δ 9.08 (d, J = 1.5 Hz, 1H), 8.74 (dd, J = 4.8, 1.7

Hz, 1H), 8.14 (dt, J = 8.0, 1.9 Hz, 1H), 7.54-7.52 (m, 2H), 7.34-7.31 (m, 1H), 7.24-7.17 (m, 3H), 5.12 (td, J = 9.2, 4.1 Hz, 1H), 3.38 (ddd, J = 11.4, 8.7, 4.2 Hz, 1H), 2.27-2.18 (m, 2H), 1.82-1.70 (m, 2H), 1.64-1.33 (m, 4H); ^{13}C NMR (CDCl₃, 100 MHz) δ 164.5, 153.3, 151.0, 137.2, 135.2, 129.1, 128.5, 127.8, 126.4, 123.3, 76.9, 46.2, 32.3, 31.8, 25.8, 23.7; HRMS (ESI-Orbitrap) m/z calcd for C₁₈H₁₉NO₂SeNa [M+Na]⁺: 384.0479; Found: 384.0471.

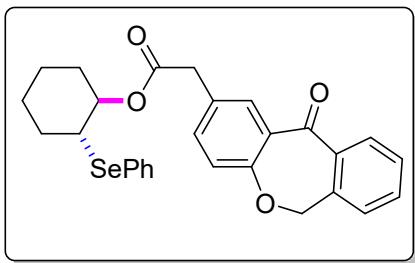


2-(phenylselanyl)cyclohexyl 2-(2,4-dichlorophenyl)acetate (8i): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 75.2 mg, 85% yield. Colourless oil. ^1H NMR (CDCl₃, 600 MHz) δ 7.54-7.54 (m, 2H), 7.38 (m, 1H), 7.26 (m, 3H), 7.19 (m, 2H), 4.89-4.86 (m, 1H), 3.63 (s, 2H), 3.19-3.16 (m, 1H), 2.12-2.06 (m, 2H), 1.68-1.61 (m, 2H), 1.51-1.27 (m, 4H); ^{13}C NMR (CDCl₃, 150 MHz) δ 169.4, 135.4, 135.1, 133.8, 132.4, 131.3, 129.3, 129.0, 128.6, 127.7, 127.2, 76.1, 45.9, 38.8, 32.3, 31.5, 25.7, 23.5; HRMS (ESI-Orbitrap) m/z calcd for C₂₀H₂₀Cl₂O₂SeNa [M+Na]⁺: 464.9898 ; Found: 464.9890.

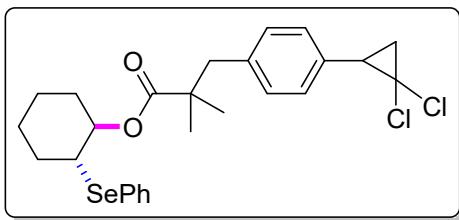


2-(phenylselanyl)cyclohexyl 2-(4-isobutylphenyl)propanoate (8j): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). d.r. = 1:1. 66.4 mg, 75% yield. Colourless oil. ^1H NMR (CDCl₃, 400 MHz) δ 7.60-7.59 (m, 1H), 7.49 (d, J = 7.1 Hz, 1H), 7.31-7.25 (m, 4H), 7.20 (d, J = 7.6 Hz, 1H), 7.12-7.10 (m, 1H), 4.87-4.83 (m, 1H), 3.73-3.54 (m, 1H), 3.25-3.20 (m, 1H), 2.48-2.46 (m, 2H), 2.14-2.09 (m, 1H), 2.00-1.96 (m, 1H), 1.89-1.83 (m, 1H), 1.66-1.61 (m, 2H), 1.5-1.50 (m, 4H), 1.47-1.30 (m, 3H), 0.92-0.89 (m, 6H); ^{13}C NMR (CDCl₃, 150 MHz) δ 174.0,

173.9, 140.5, 140.4, 138.1, 137.8, 135.2, 135.1, 129.3, 129.3, 19.0, 128.9, 128.9, 128.5, 127.6, 127.6, 127.5, 127.3, 75.4, 74.7, 46.0, 45.6, 45.6, 45.2, 45.1, 32.2, 31.9, 31.1, 30.9, 30.3, 25.7, 25.3, 23.4, 23.2, 22.5, 18.6, 18.4; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₅H₃₂O₂SeNa [M+Na]⁺: 465.1468; Found: 465.1468.



2-(phenylselanyl)cyclohexyl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-2-yl)acetate (8k): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 50:1, v/v). 76.2 mg, 75% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 8.10 (d, *J* = 2.2 Hz, 1H), 7.88 (d, *J* = 7.6 Hz, 1H), 7.55-7.52 (m, 3H), 7.47-7.40 (m, 2H), 7.34 (d, *J* = 7.4 Hz, 1H), 7.27-7.25 (m, 3H), 7.00 (d, *J* = 8.5 Hz, 1H), 5.15 (s, 2H), 4.87 (td, *J* = 9.1, 4.0, Hz, 1H), 3.51 (s, 1H), 3.22 (dt, *J* = 10.1, 4.1 Hz, 1H), 2.15-2.04 (m, 2H), 1.68-1.61 (m, 2H), 1.54-1.26 (m, 4H); **¹³C NMR** (CDCl₃, 150 MHz) δ 190.9, 170.7, 160.5, 140.6, 136.6, 135.6, 132.8, 132.6, 129.5, 129.3, 129.0, 128.7, 128.0, 127.9, 127.7, 125.1, 121.0, 76.0, 73.7, 46.0, 40.4, 32.3, 31.7, 25.8, 23.6; **HRMS** (ESI-Orbitrap) m/z calcd for C₂₈H₂₆O₄SeNa [M+Na]⁺: 529.0889; Found: 529.0886.



2-(phenylselanyl)cyclohexyl 3-(4-(2,2-dichlorocyclopropyl)phenyl)-2,2-dimethylpropanoate (8l): New compound. (Eluent: petroleum ether (60-90 °C)/EtOAc = 70:1, v/v). d.r. = 1:1, 78.3 mg, 75% yield. Yellow oil. **¹H NMR** (CDCl₃, 400 MHz) δ 7.56-7.54 (m, 2H), 7.29-7.24 (m, 3H), 7.09-7.07 (m, 2H), 6.89-6.85 (m, 2H), 4.93-4.86 (m, 1H), 3.15 (td, *J* = 10.0, 4.0 Hz, 1H), 2.81 (dd, *J* = 10.6, 8.5 Hz, 1H), 2.07-1.98 (m, 2H), 1.93-1.89 (m, 1H), 1.77-1.72 (m, 1H), 1.64 (s, 6H), 1.61-1.57 (m, 1H), 1.50-1.42 (m, 1H), 1.33-1.23 (m, 3H); **¹³C NMR** (CDCl₃, 150 MHz) δ 173.4

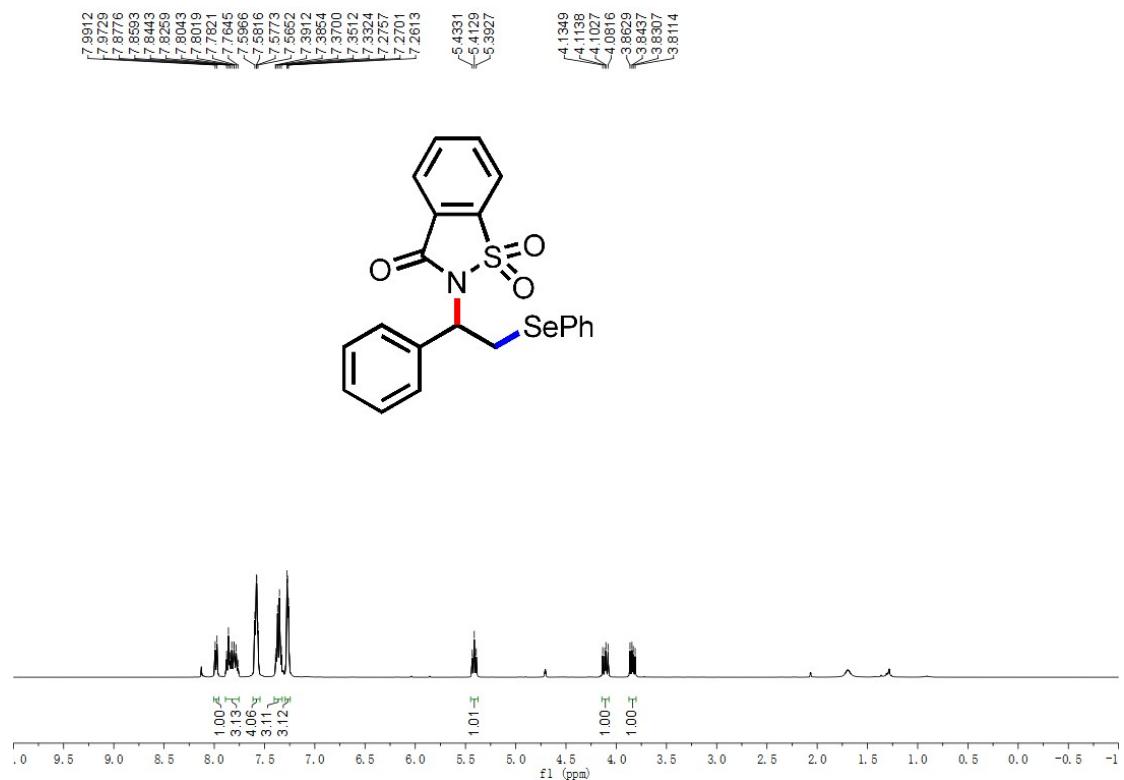
(d, $J = 2.5$ Hz), 155.2 (d, $J = 2.5$ Hz), 135.4, 129.7 (d, $J = 5.0$ Hz), 129.1, 128.0 (d, $J = 2.5$ Hz), 128.0, 127.9, 118.5 (d, $J = 3.8$ Hz), 79.3, 75.6, 61.0 (d, $J = 3.8$ Hz), 45.7, 34.9 (d, $J = 3.8$ Hz), 32.5, 31.2, 26.4 (d, $J = 8.8$ Hz), 25.9, 25.6, 25.0 (d, $J = 7.5$ Hz), 23.5; **HRMS** (ESI-Orbitrap) m/z calcd for $C_{26}H_{30}Cl_2O_2Se$ [M+Na] $^+$: 547.0686; Found: 547.0681.

5. References

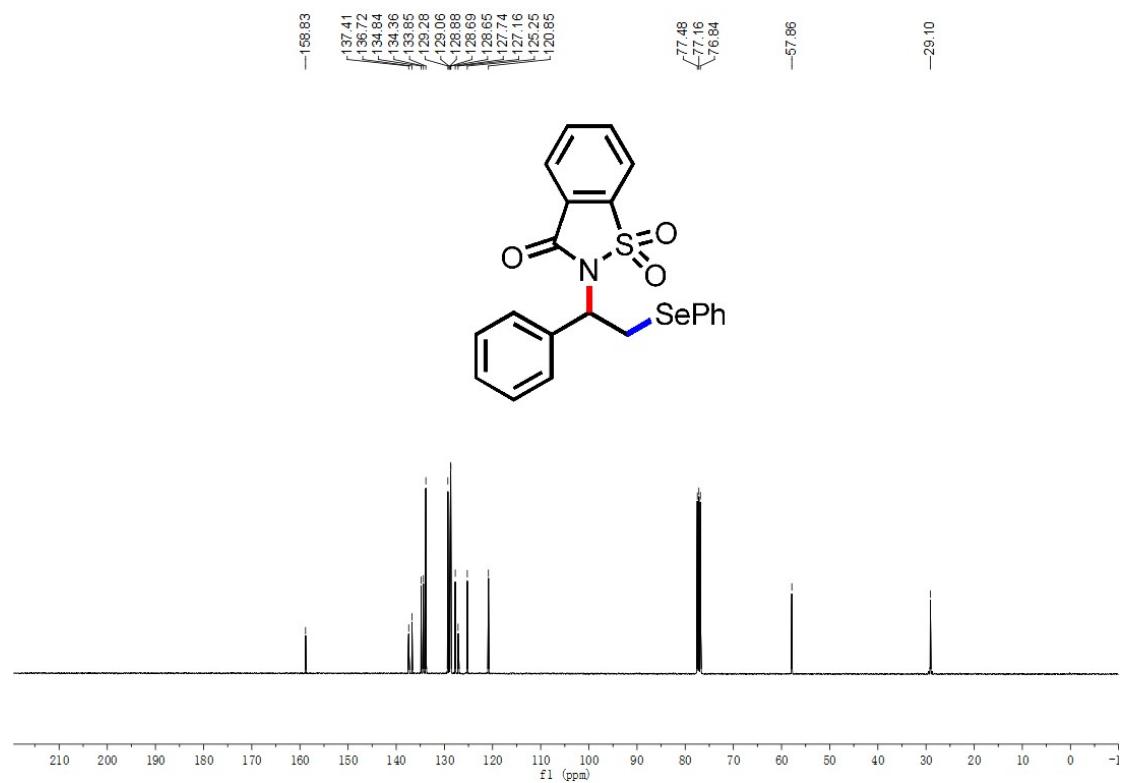
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6. Copies of NMR spectra

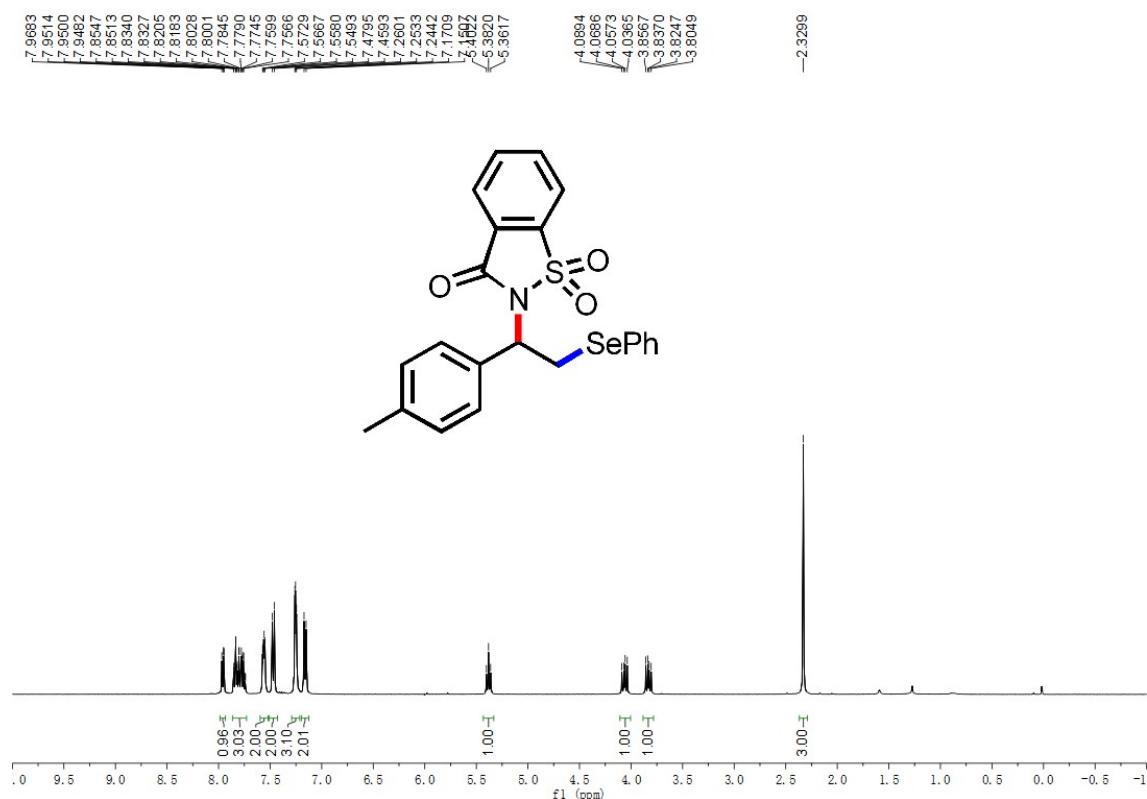
¹H NMR of product 4a in CDCl₃ (400 MHz)



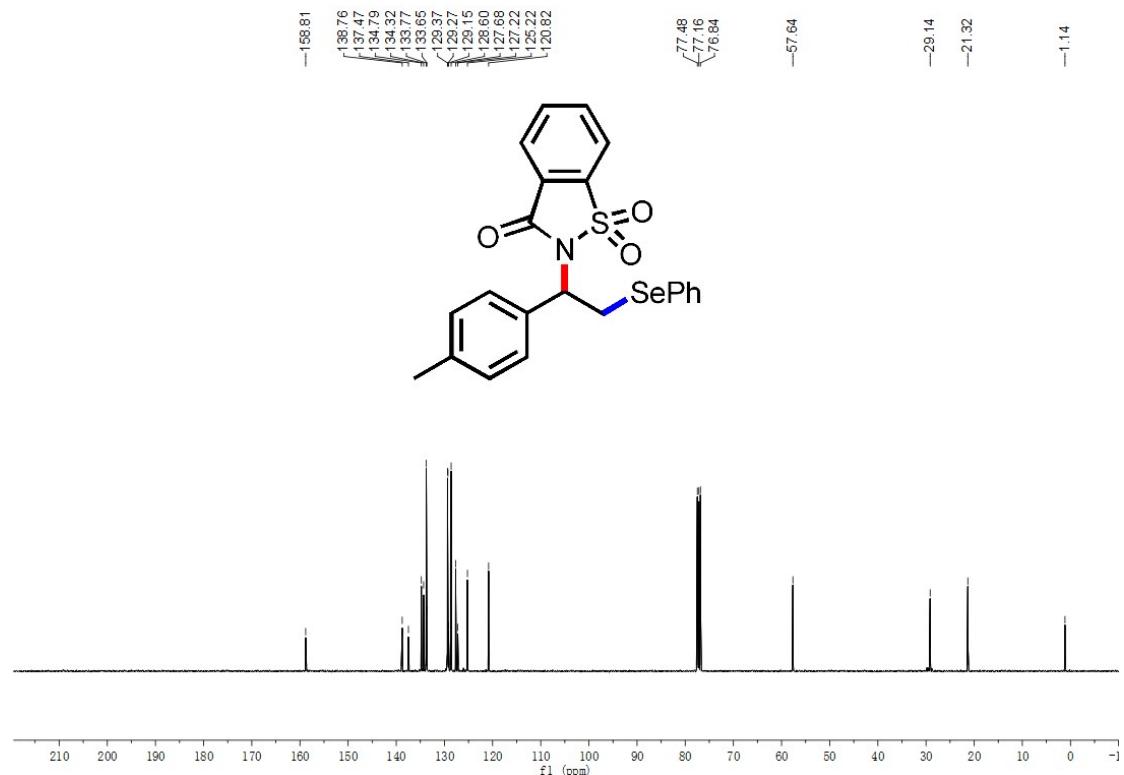
¹³C NMR of product 4a in CDCl₃ (100 MHz)



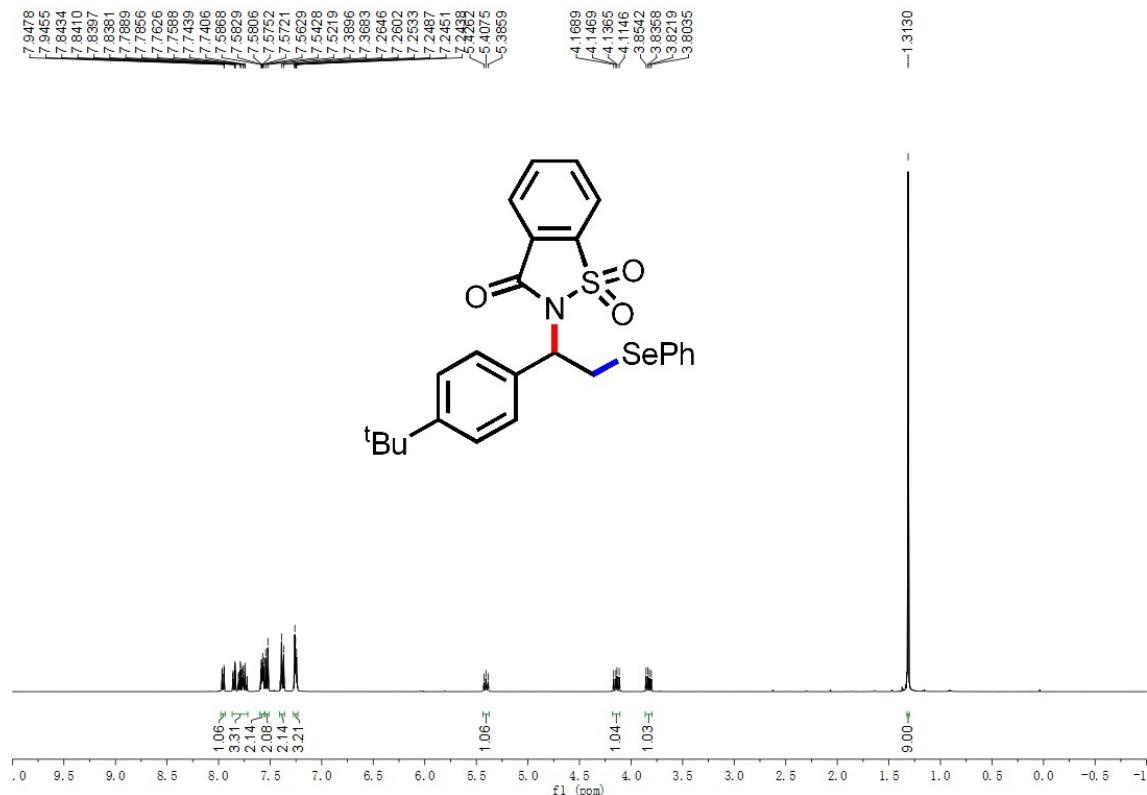
¹H NMR of product 4b in CDCl₃ (400 MHz)



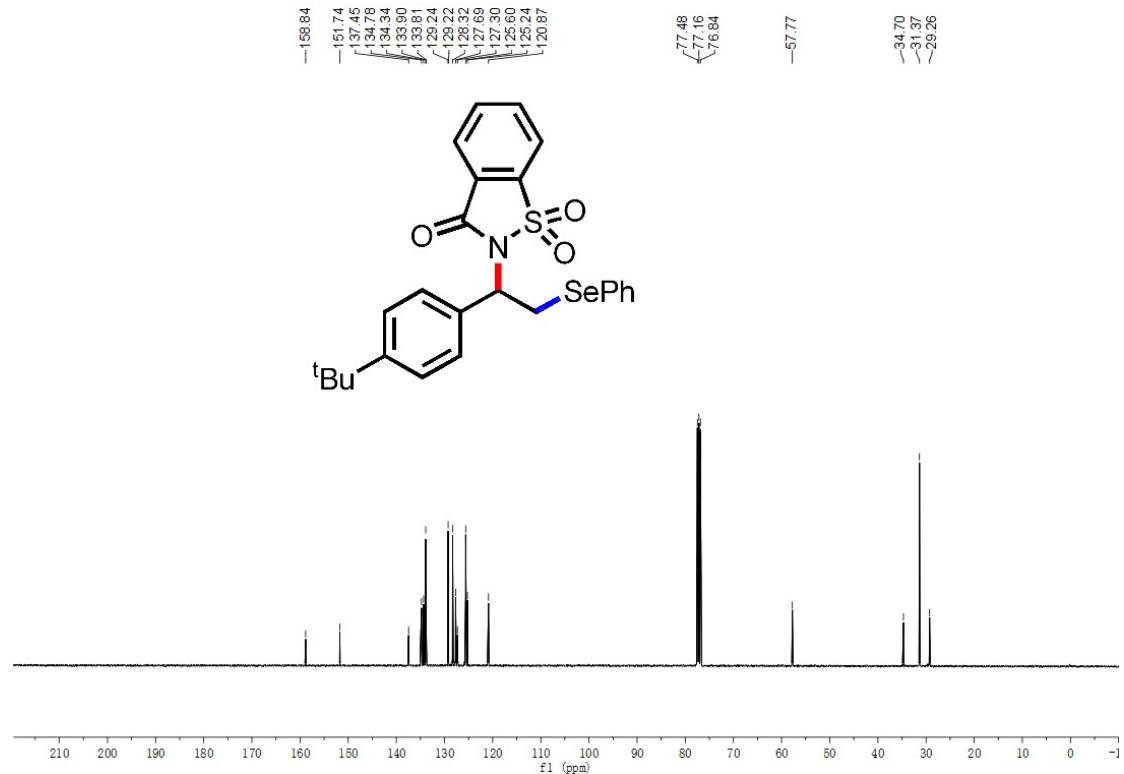
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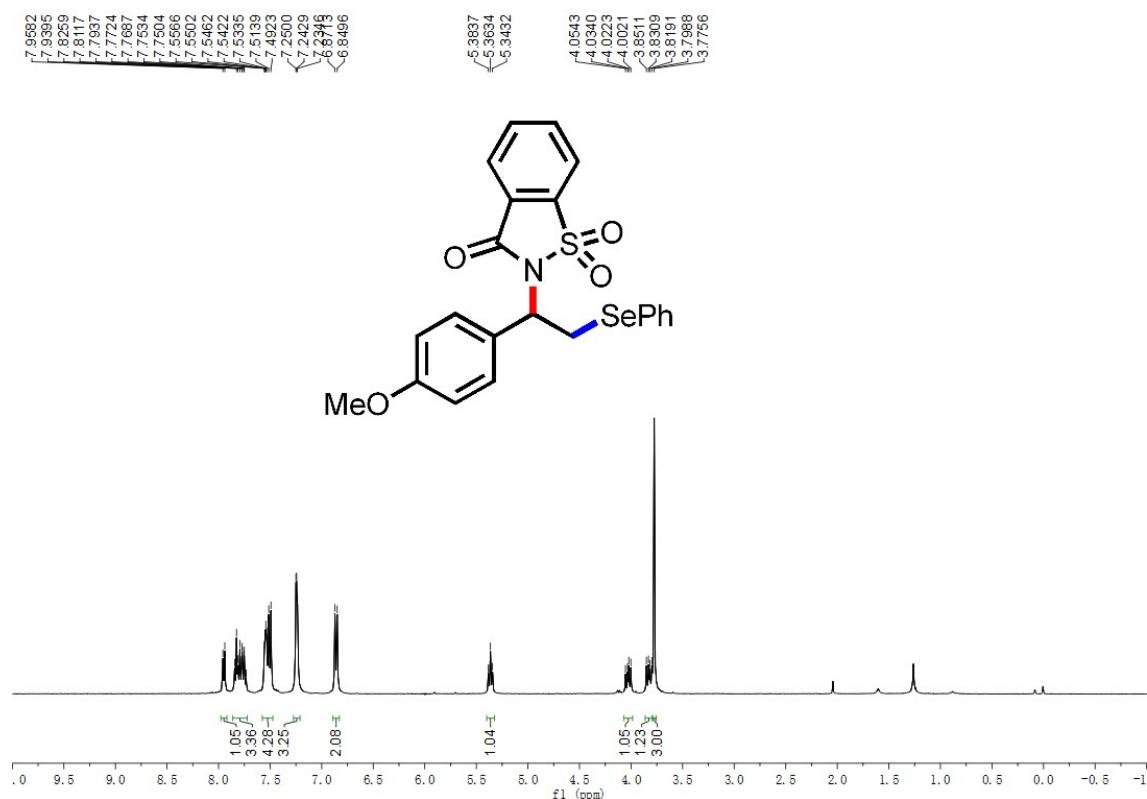
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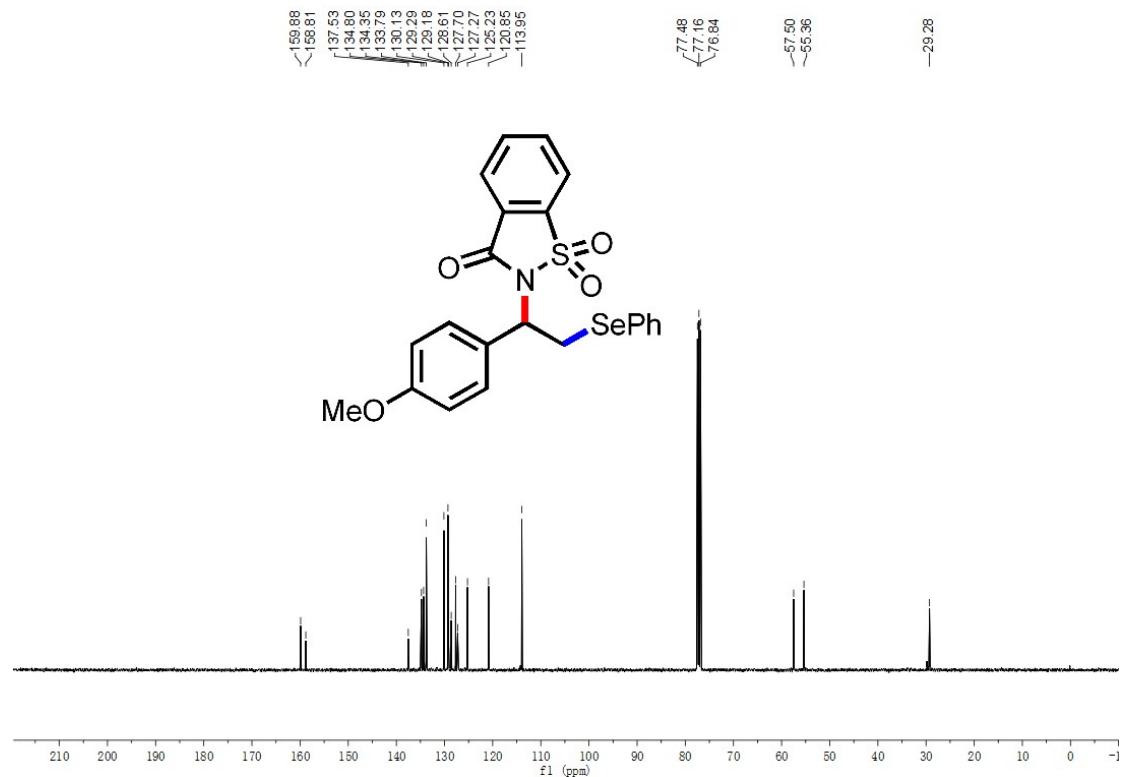
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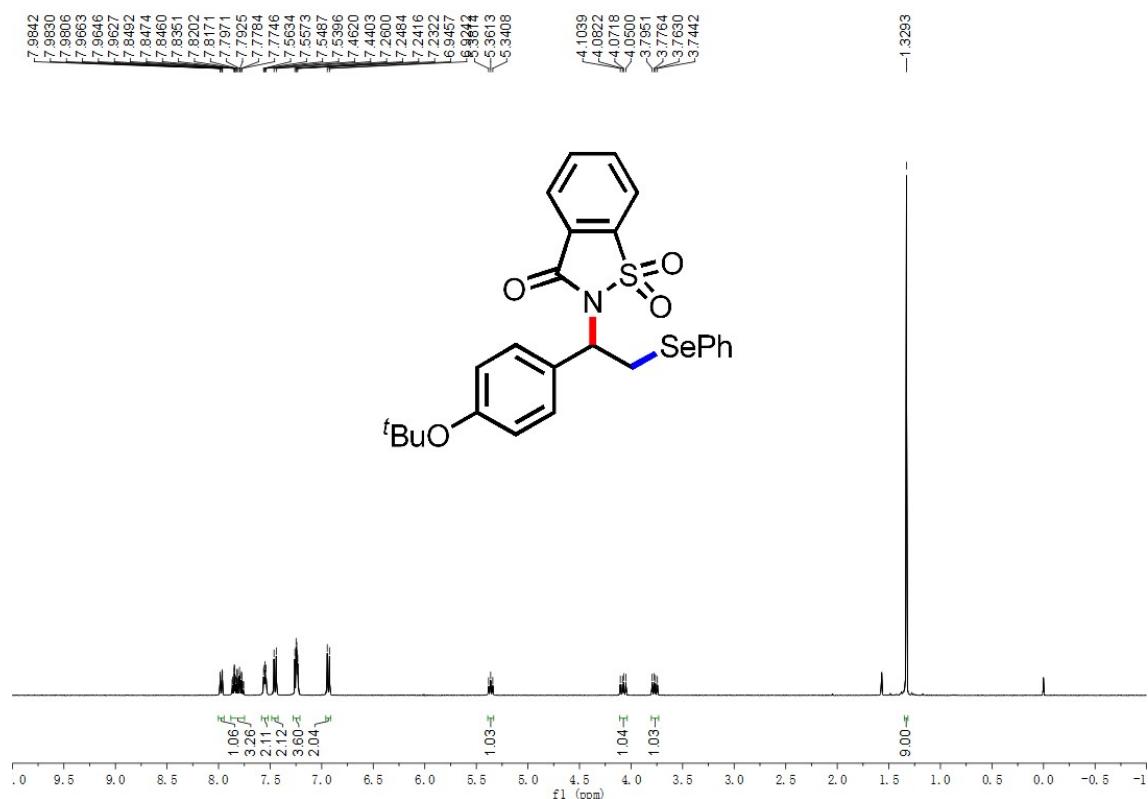
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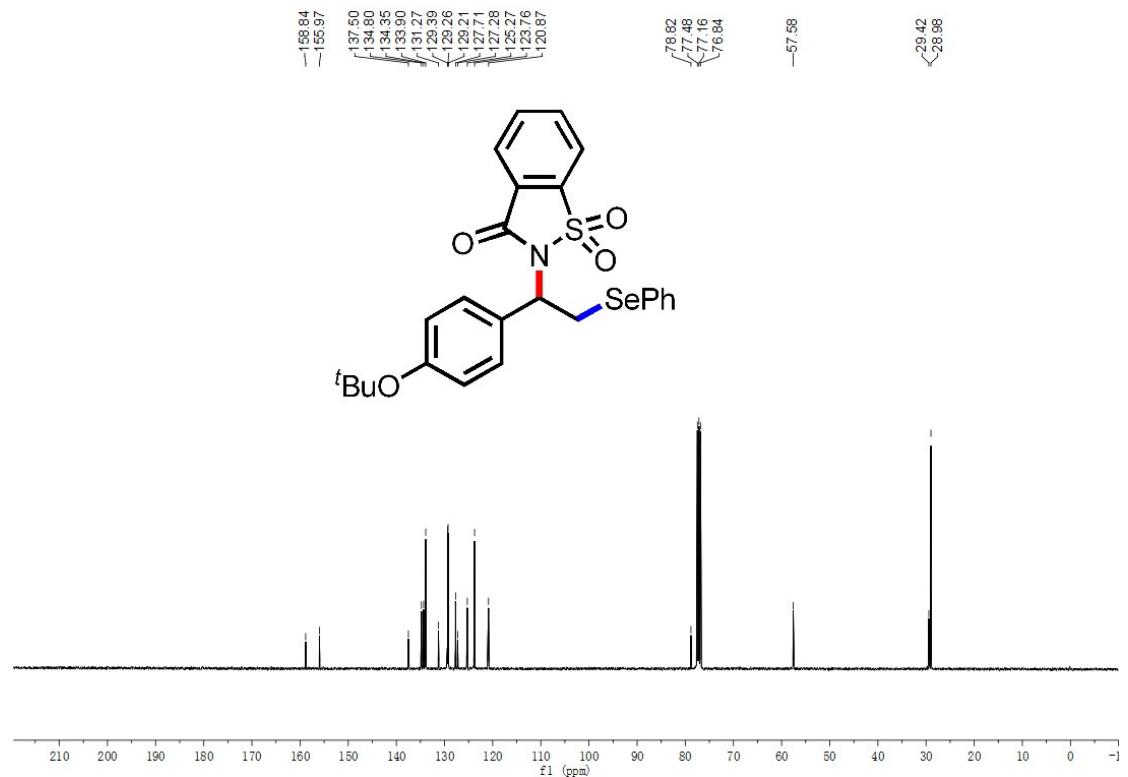
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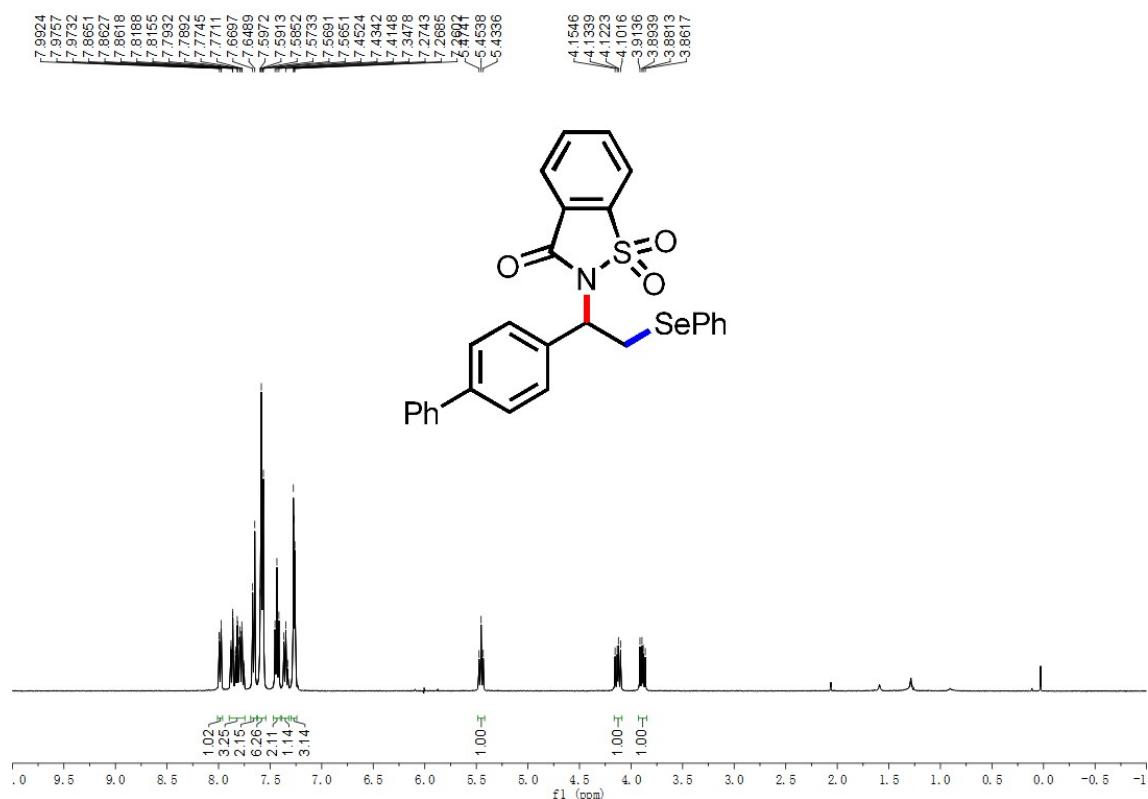
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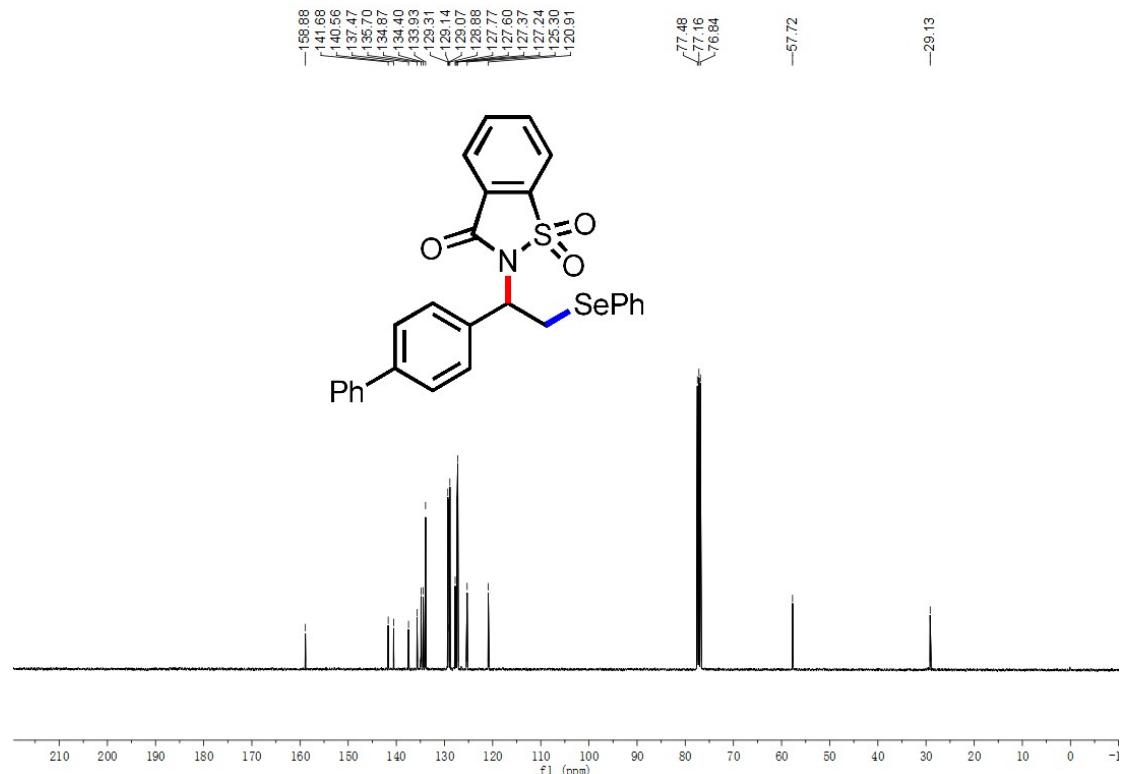
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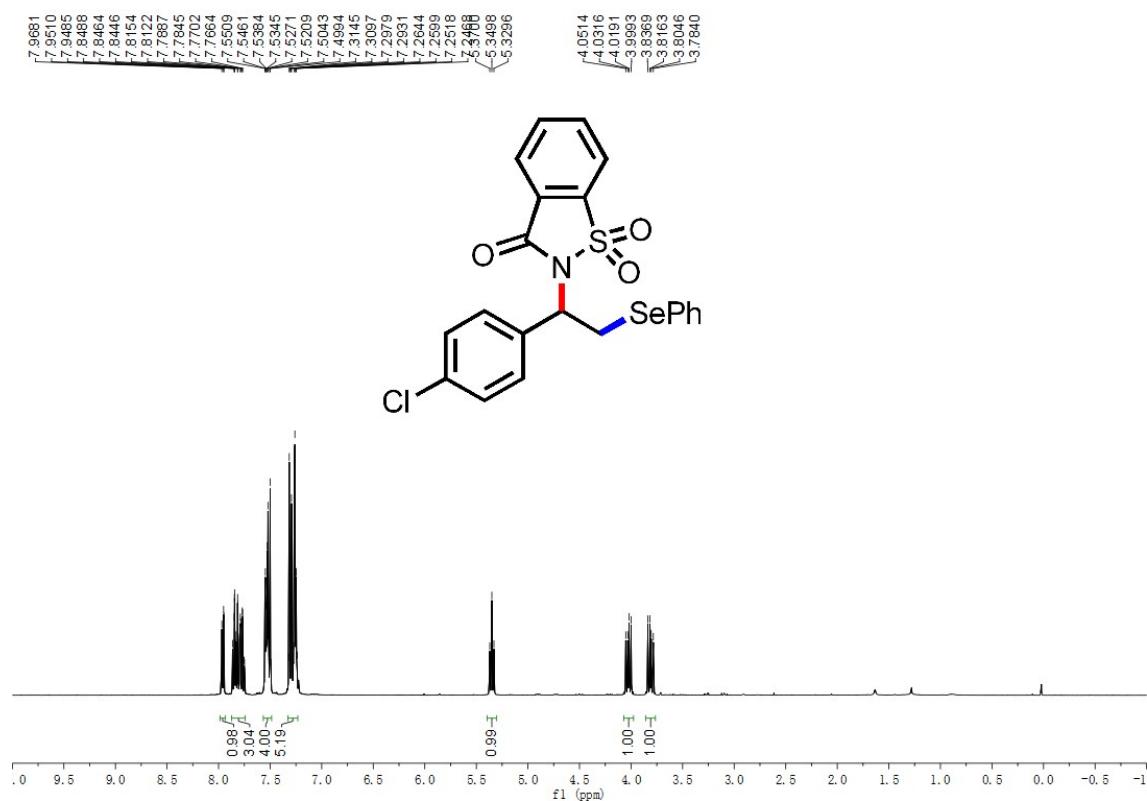
¹H NMR of product 4f in CDCl₃ (400 MHz)



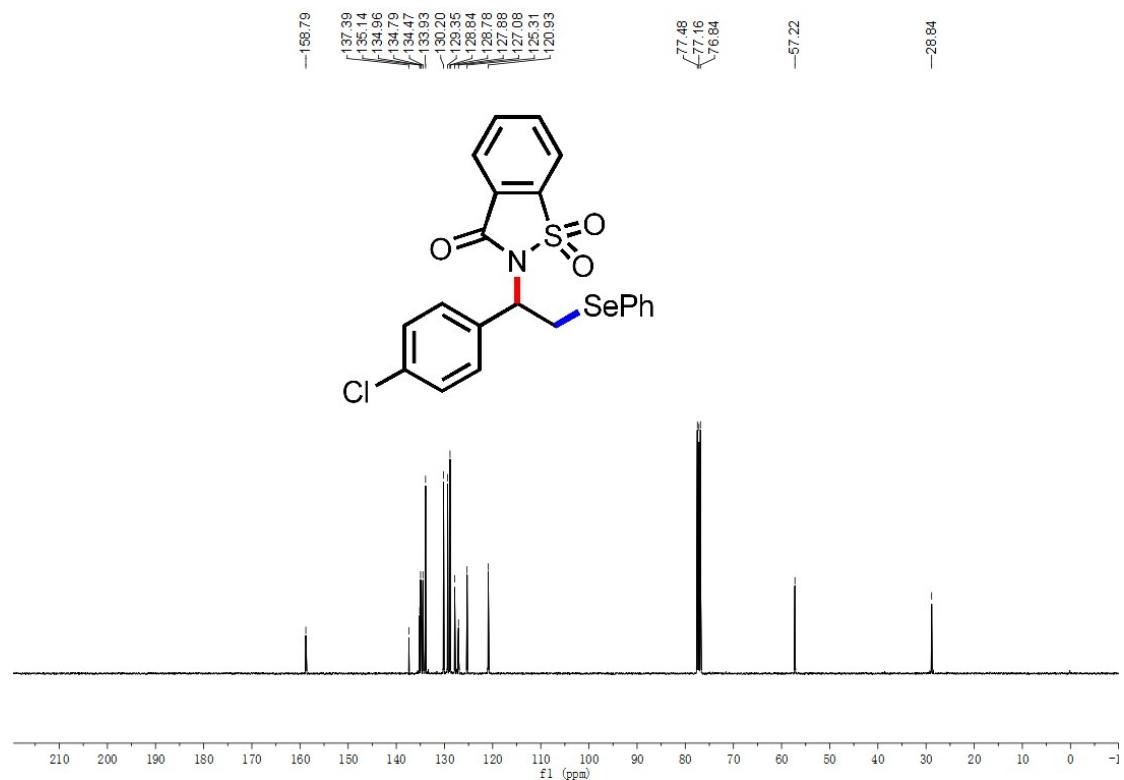
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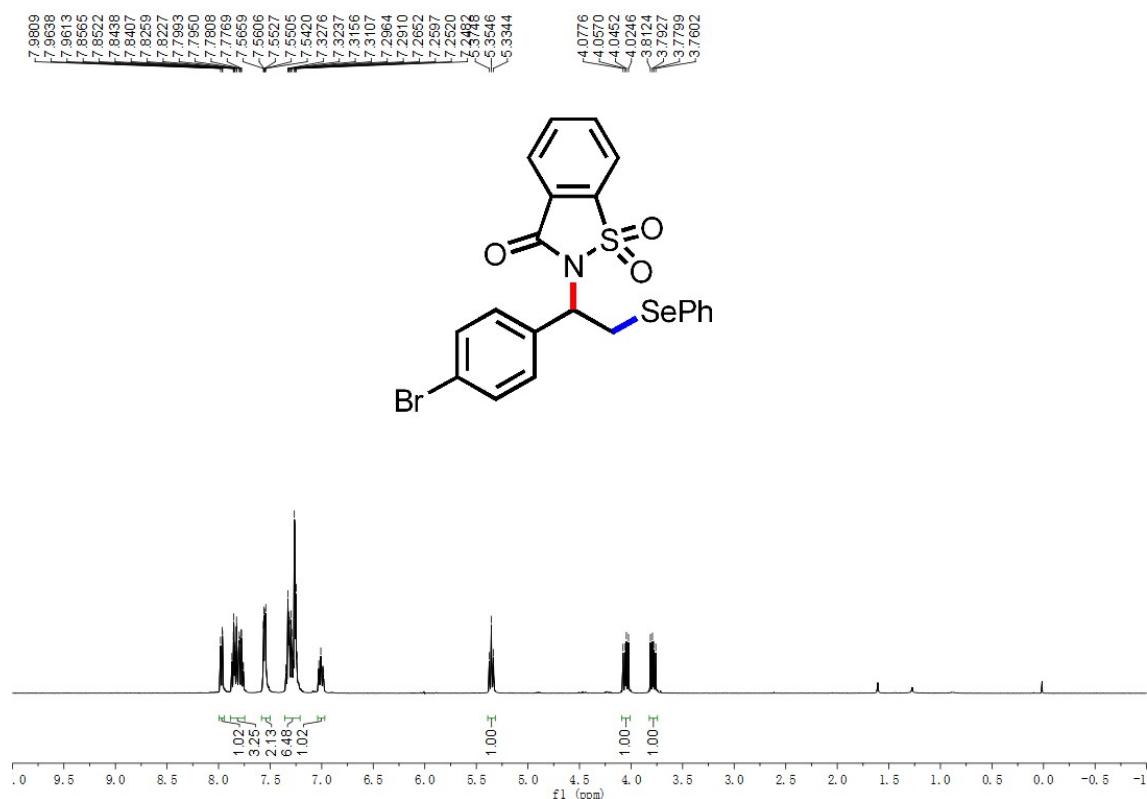
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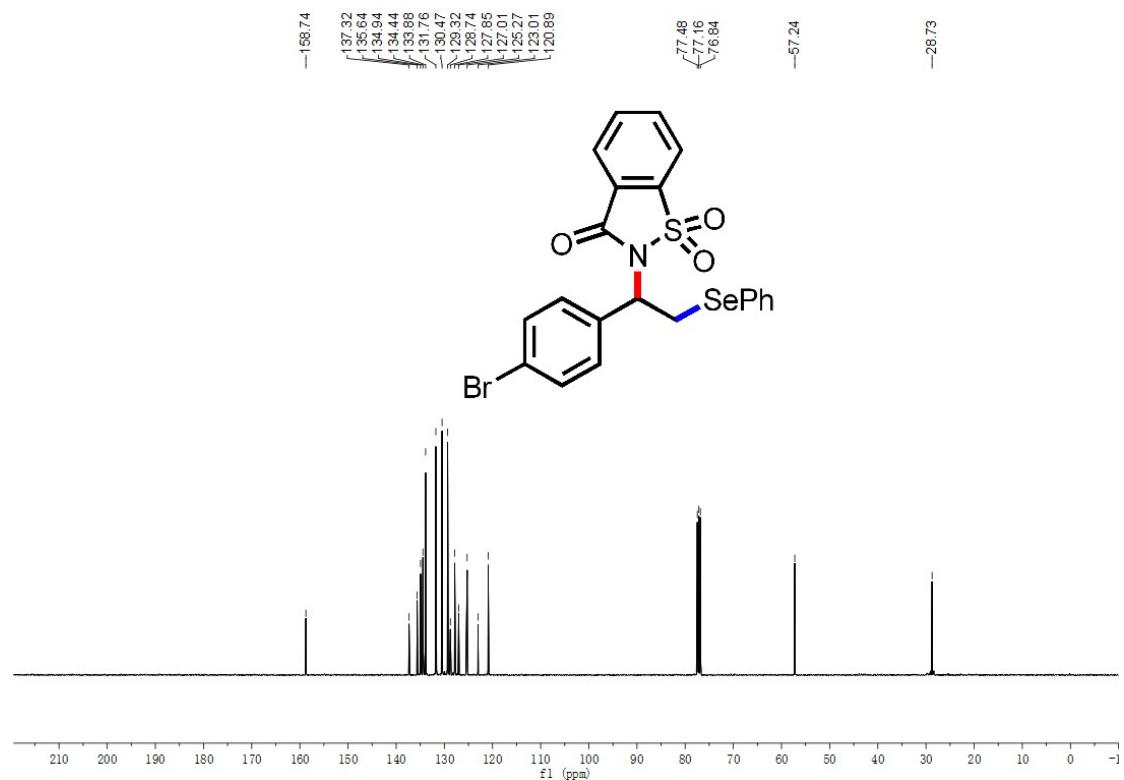
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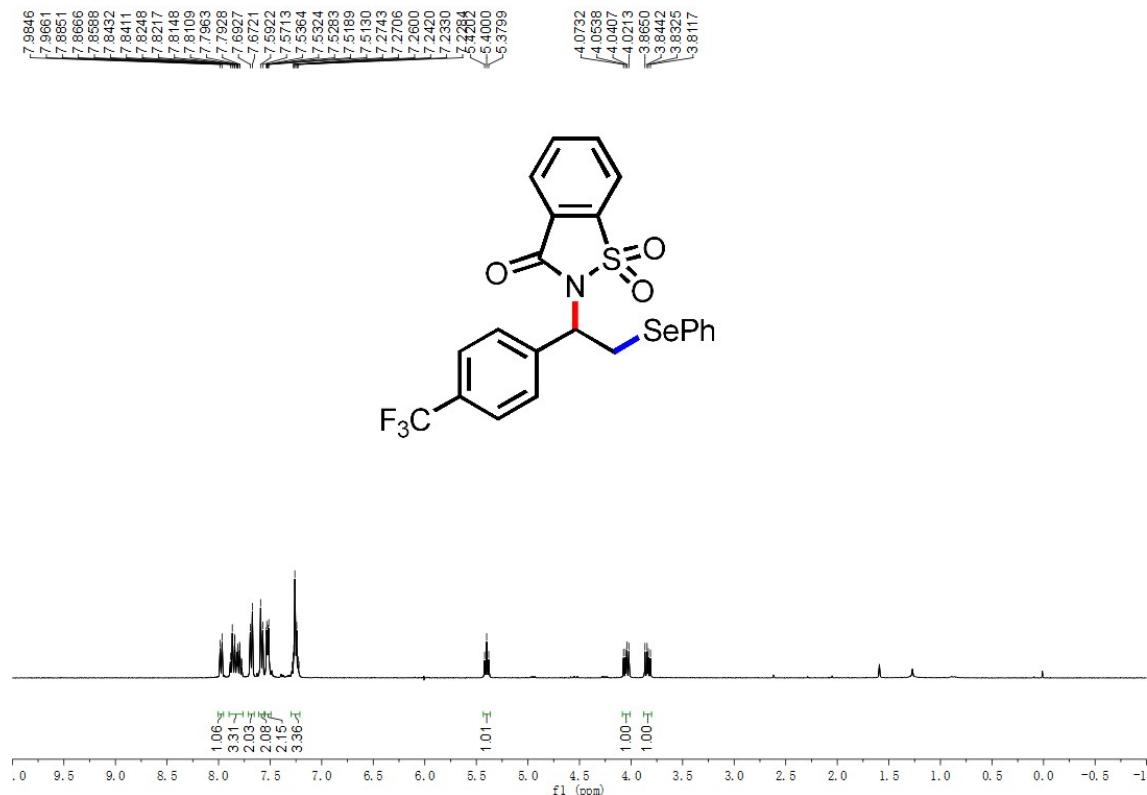
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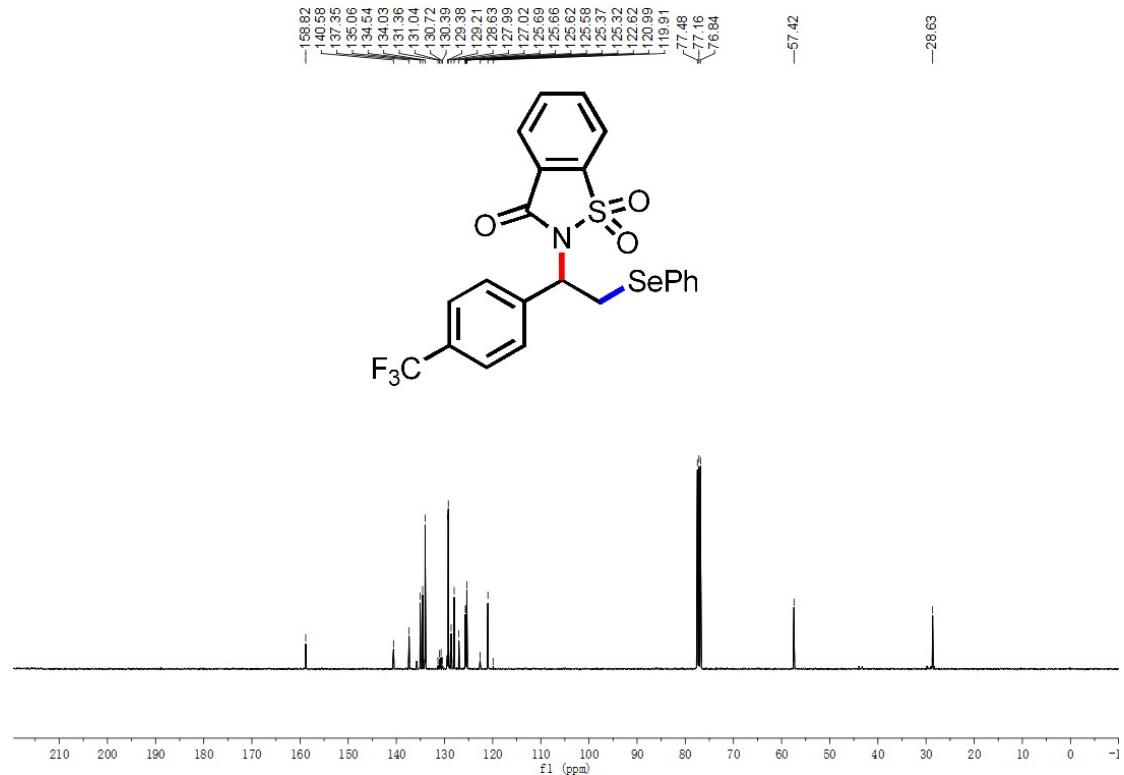
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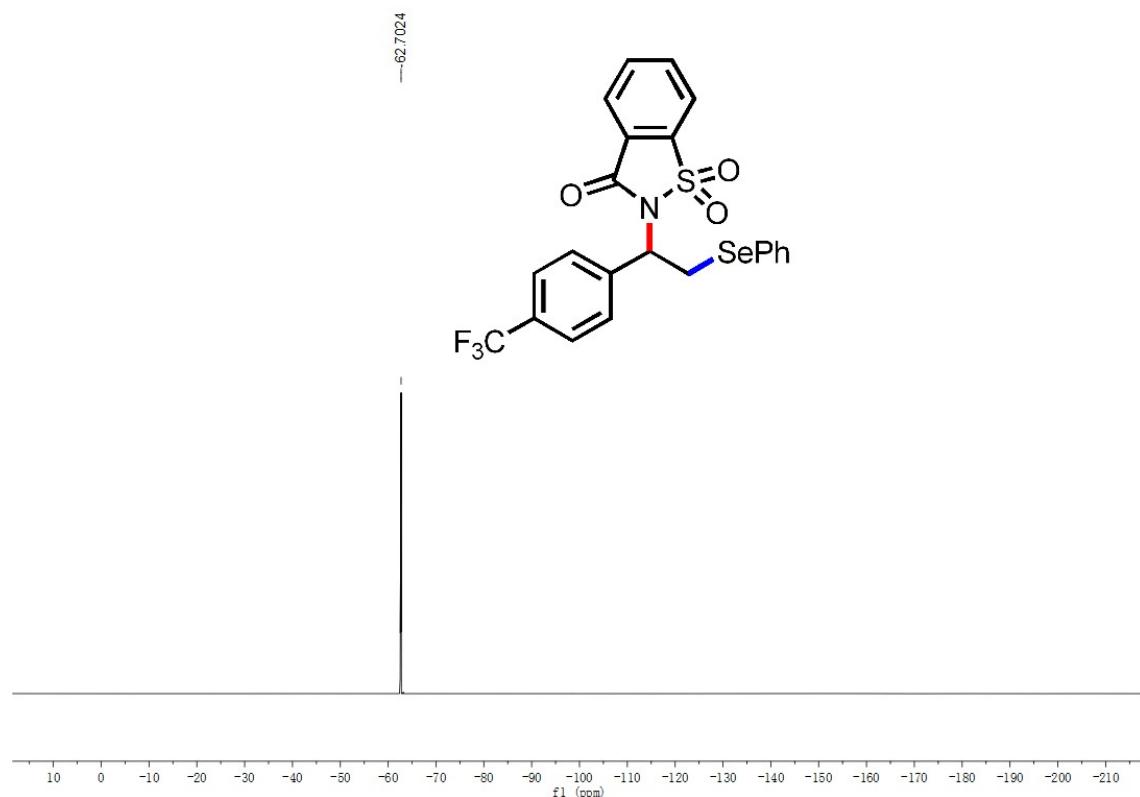
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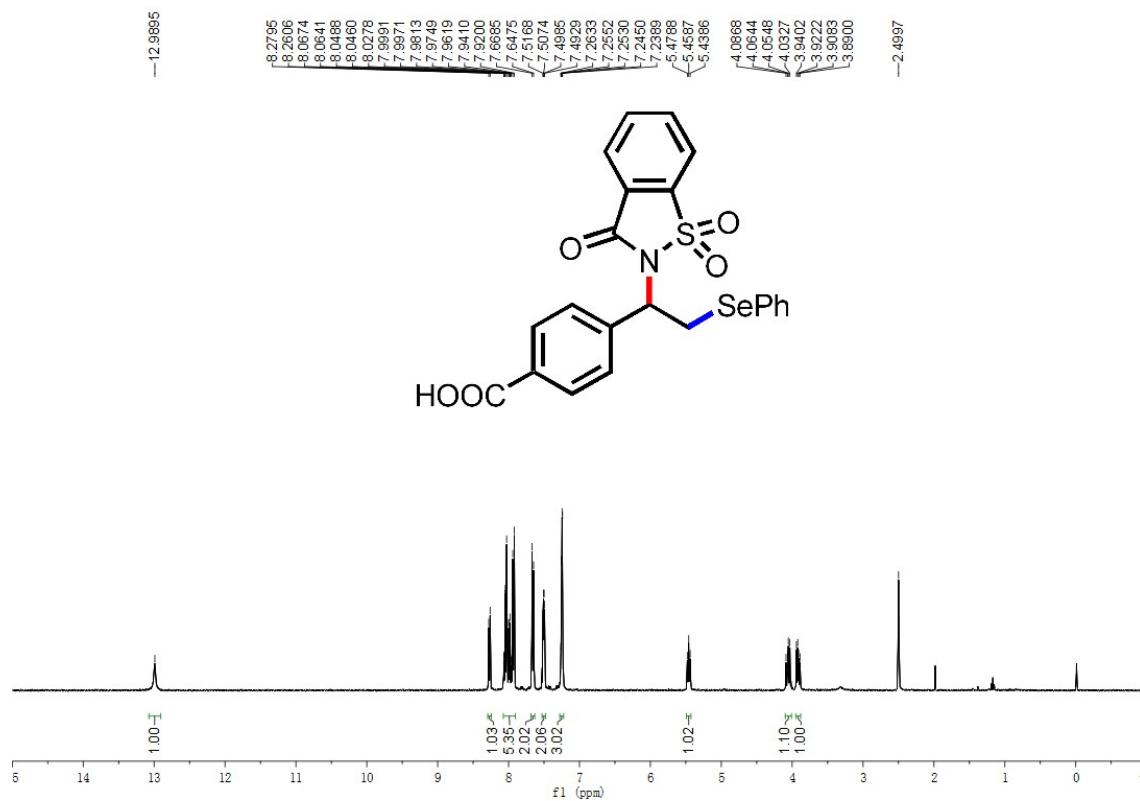
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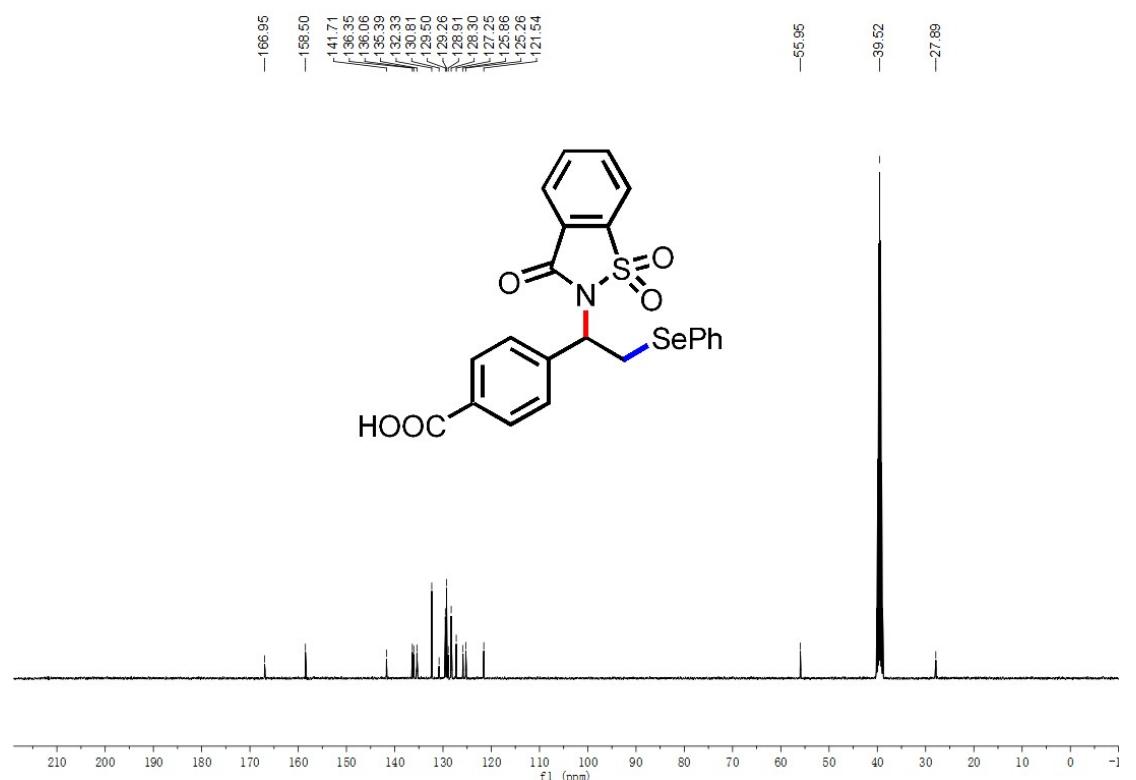
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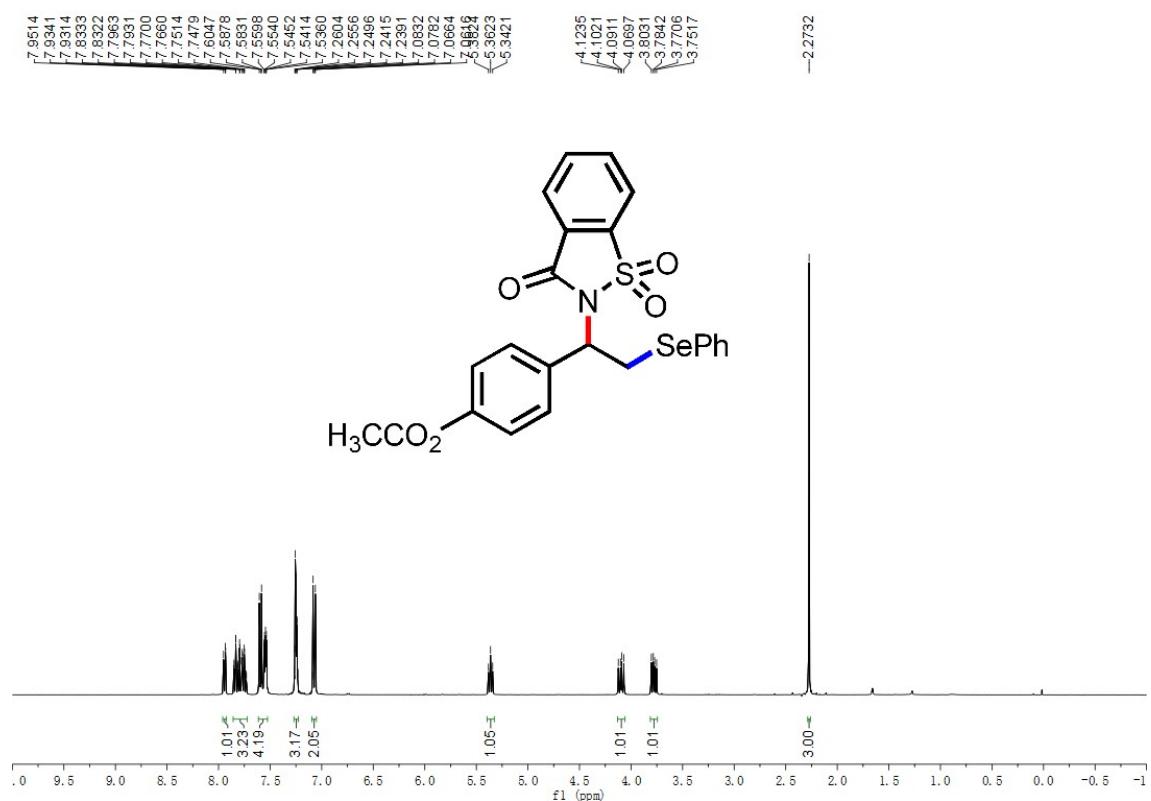
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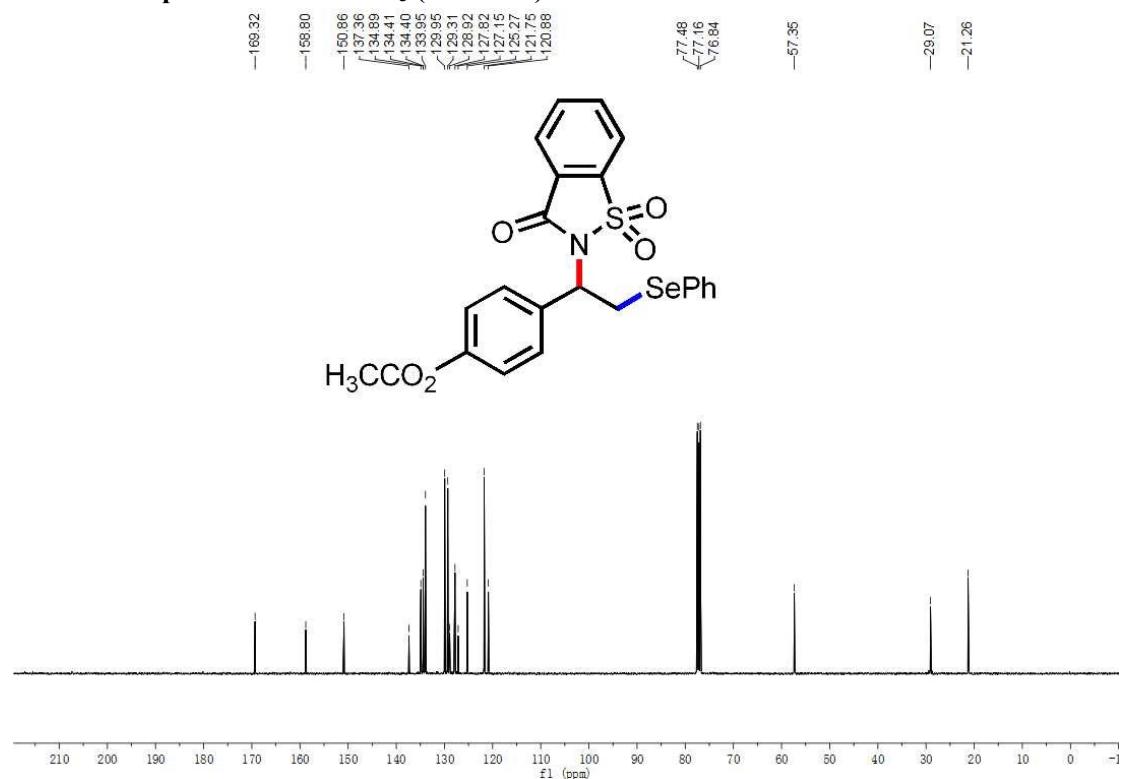
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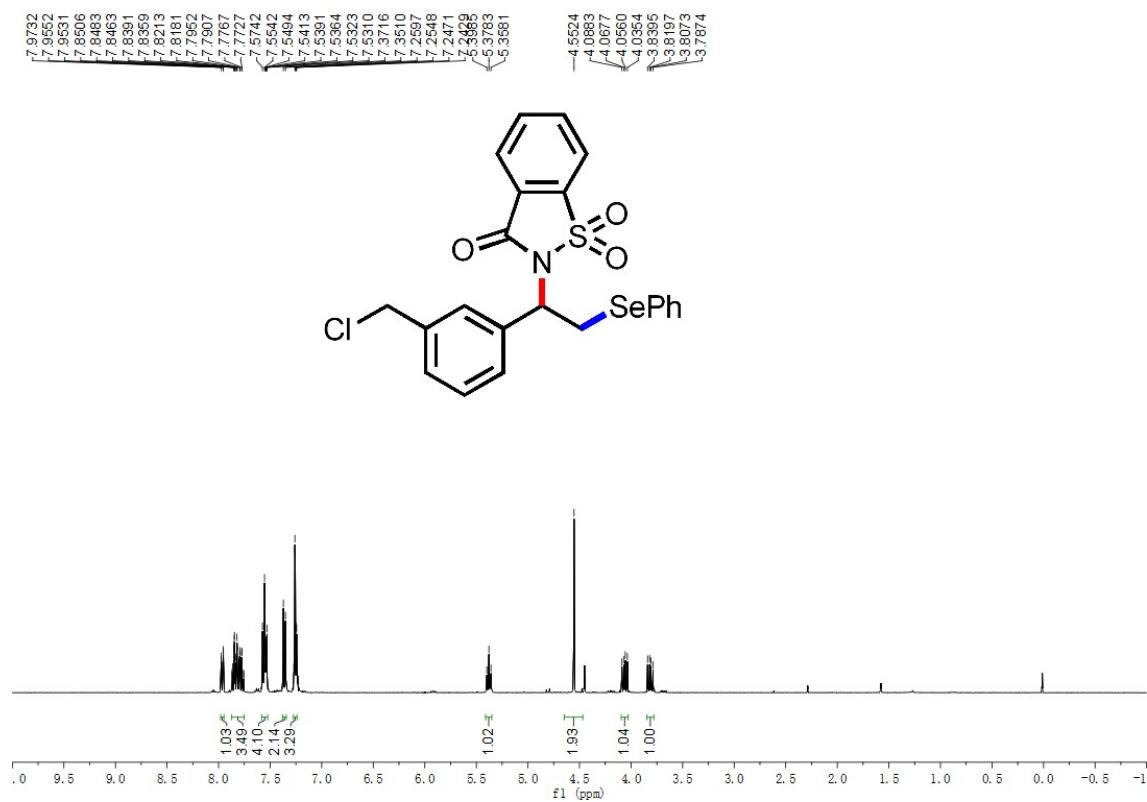
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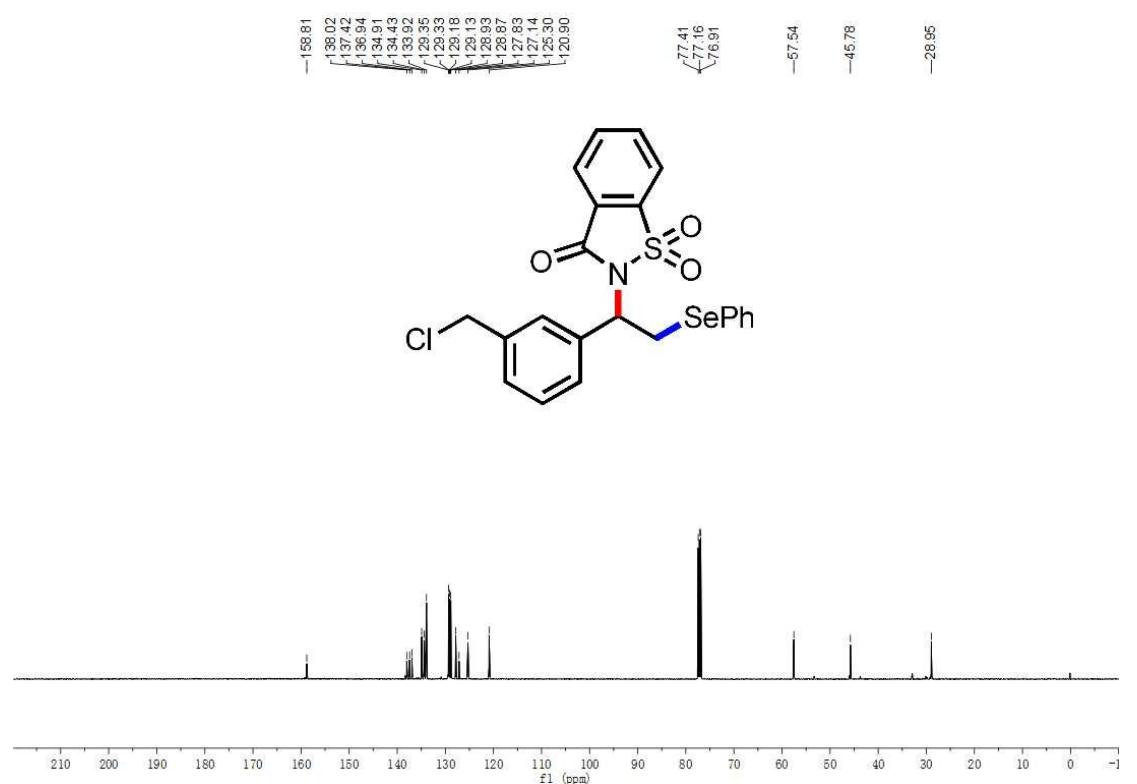
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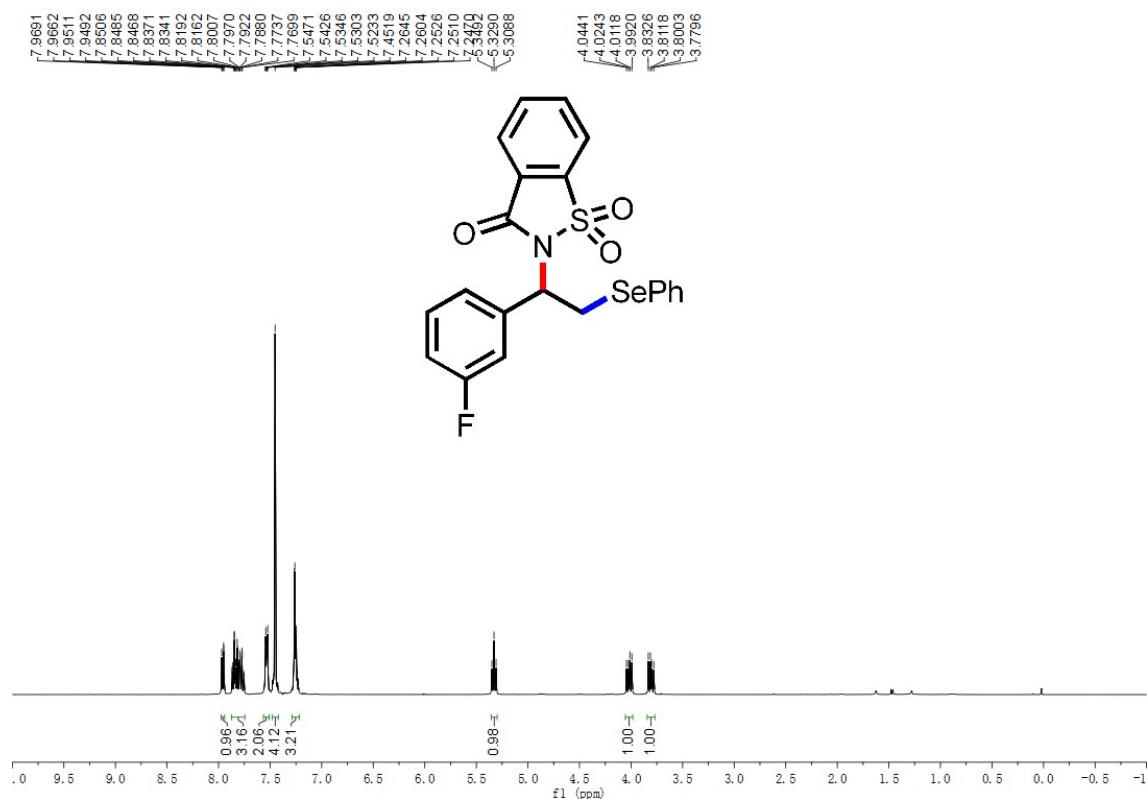
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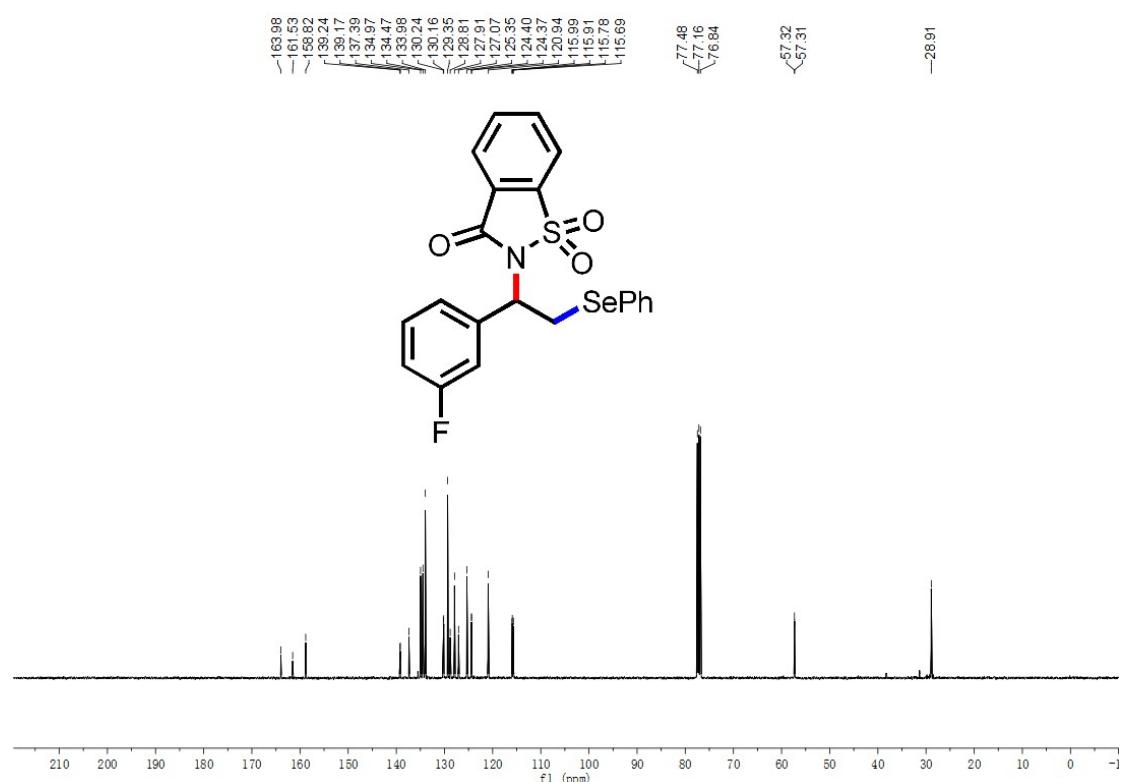
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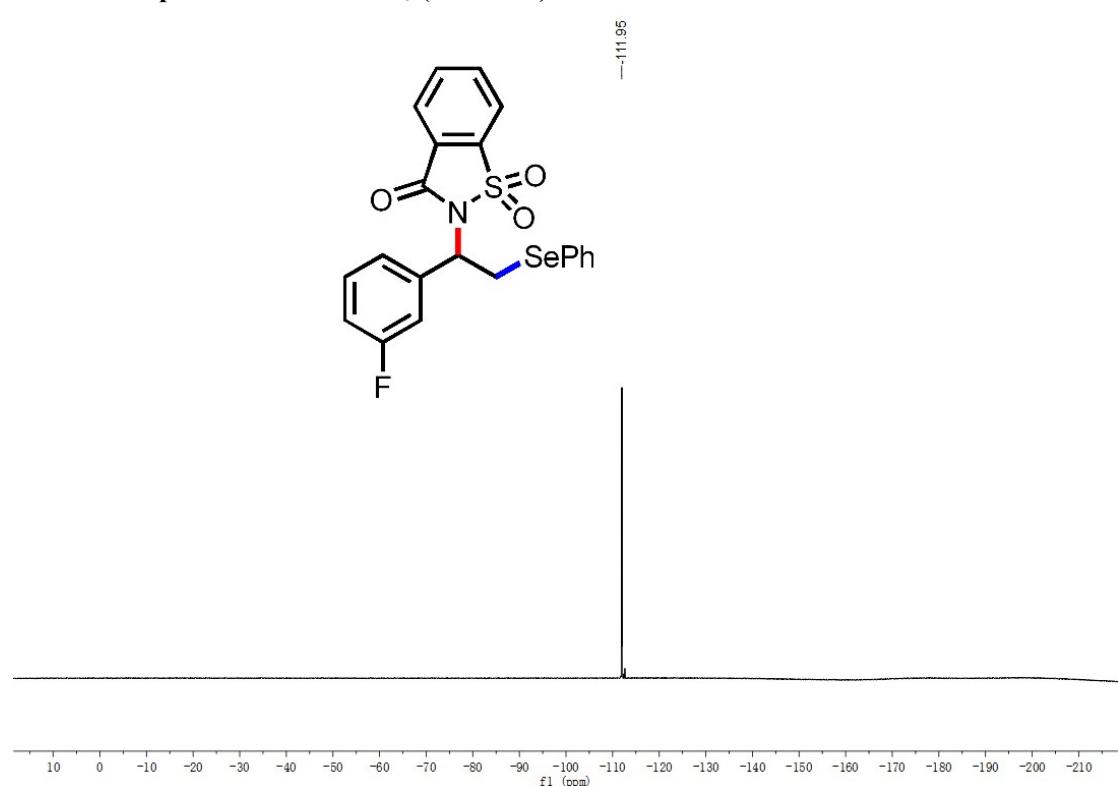
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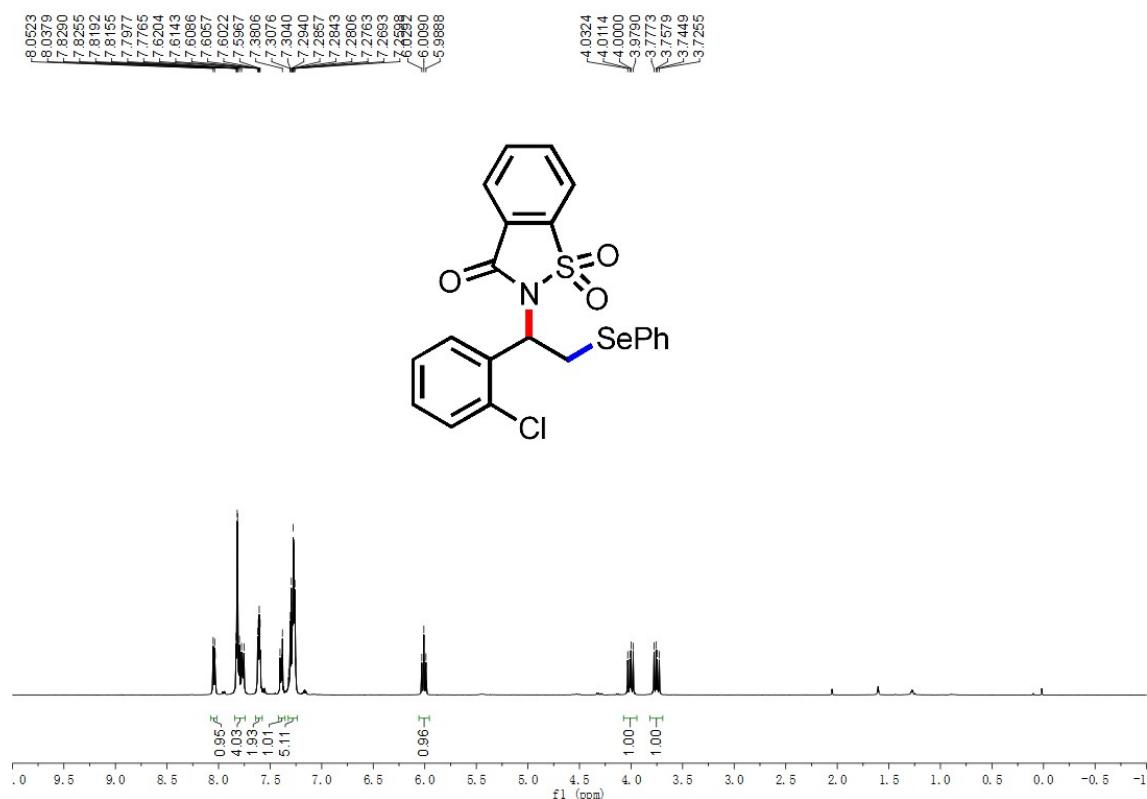
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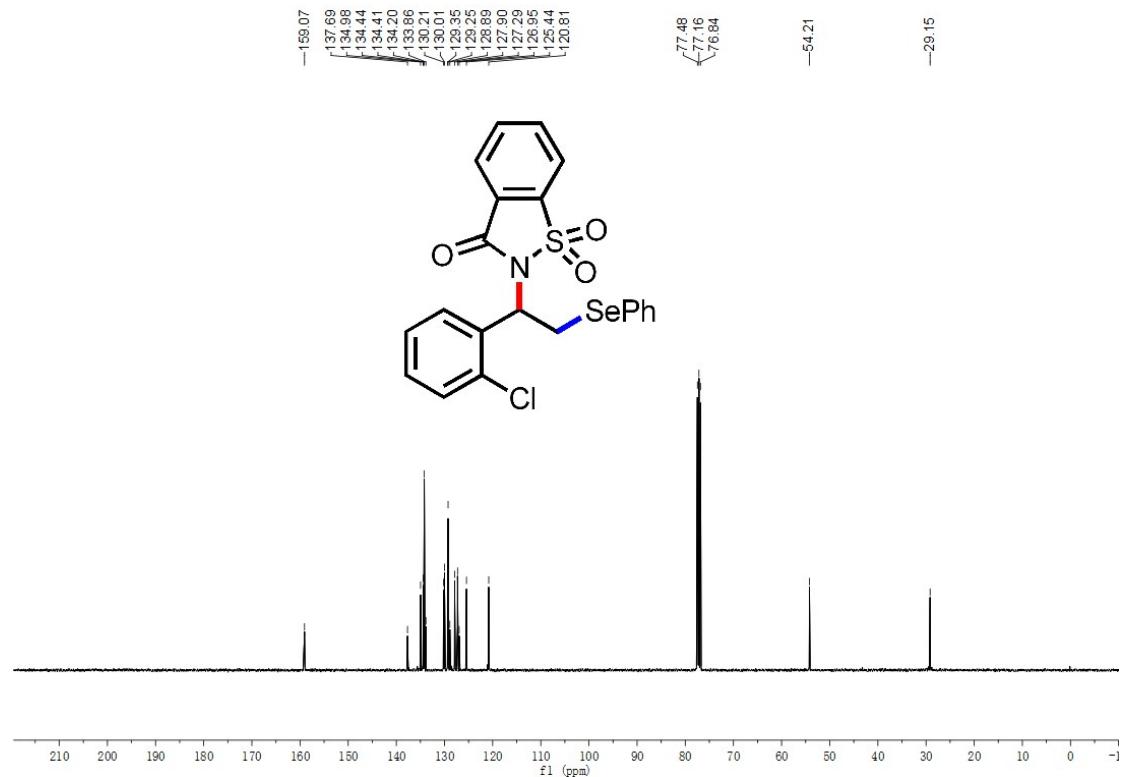
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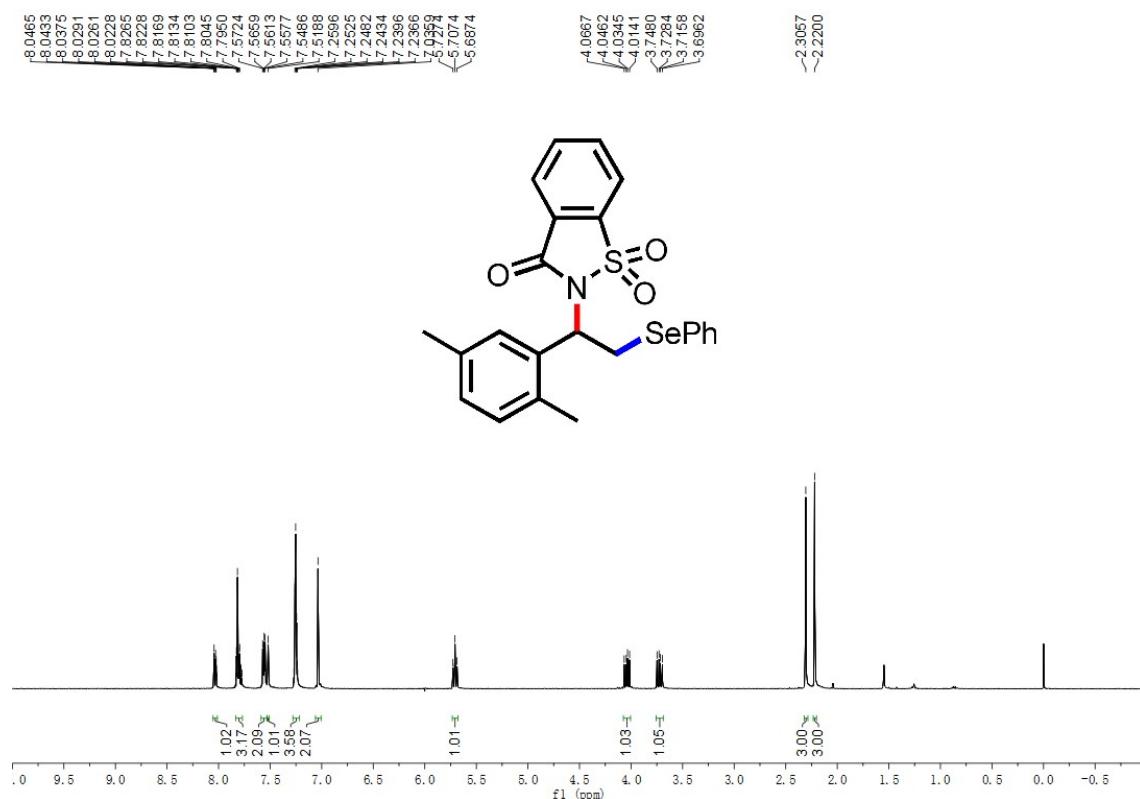
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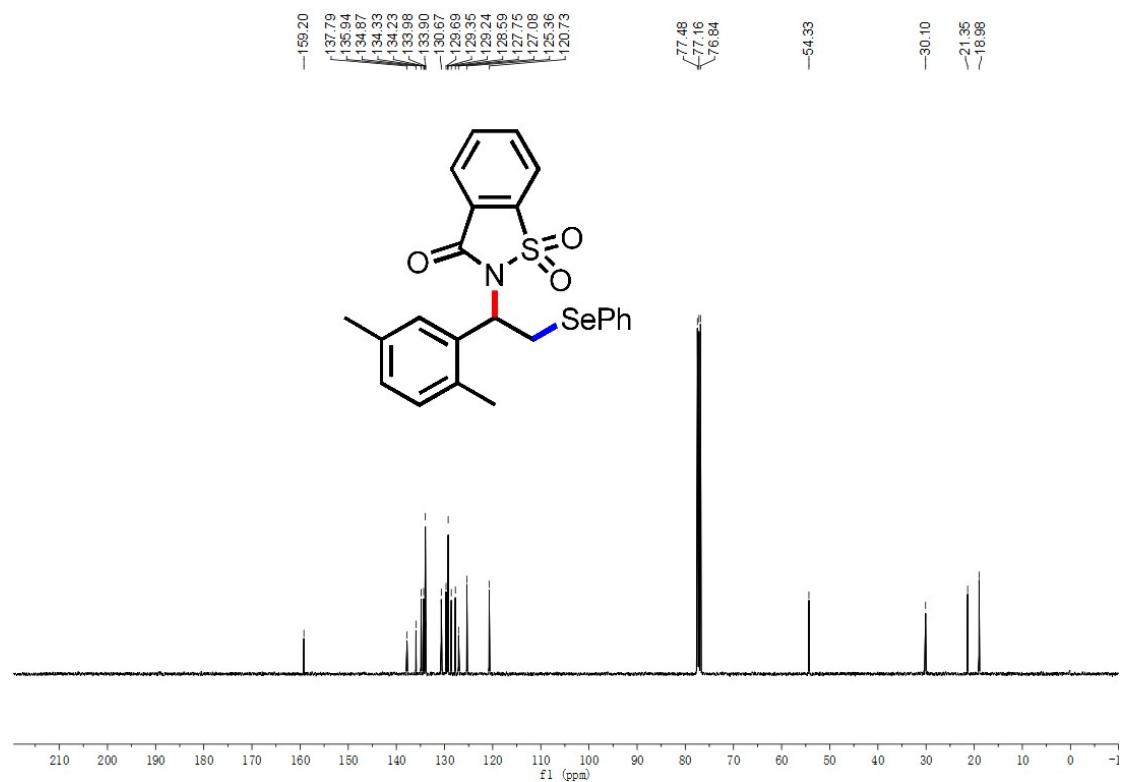
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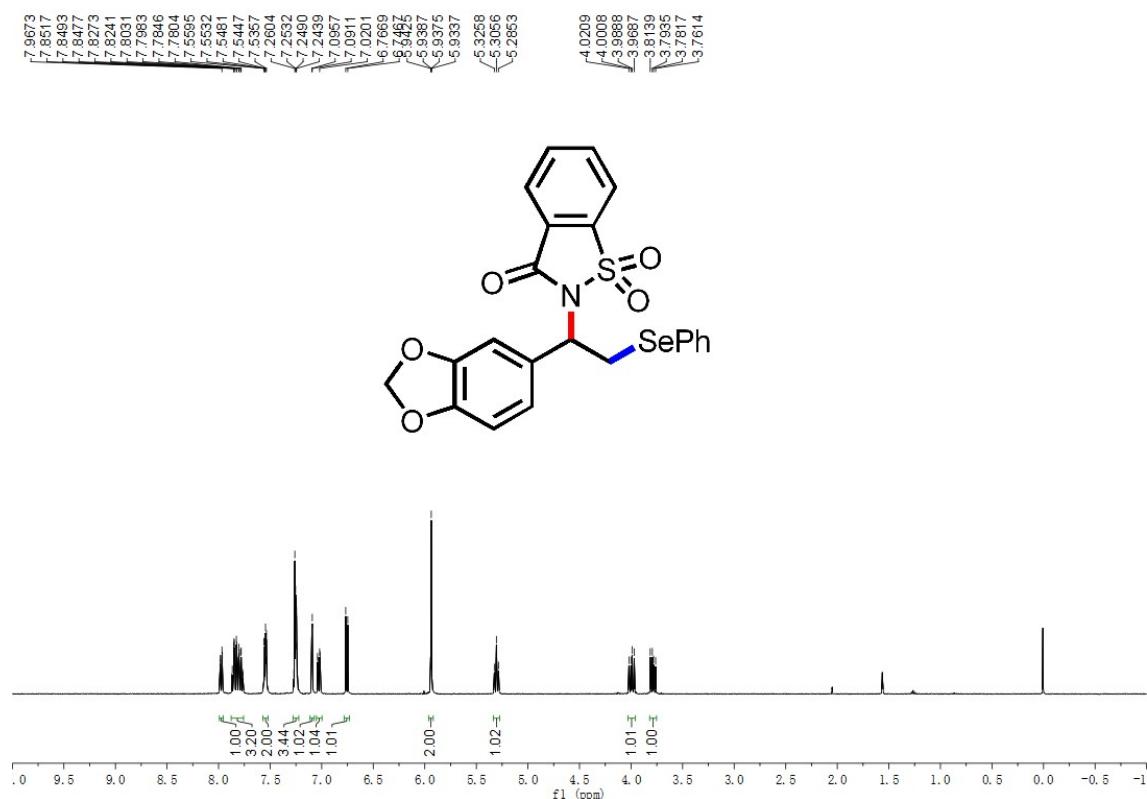
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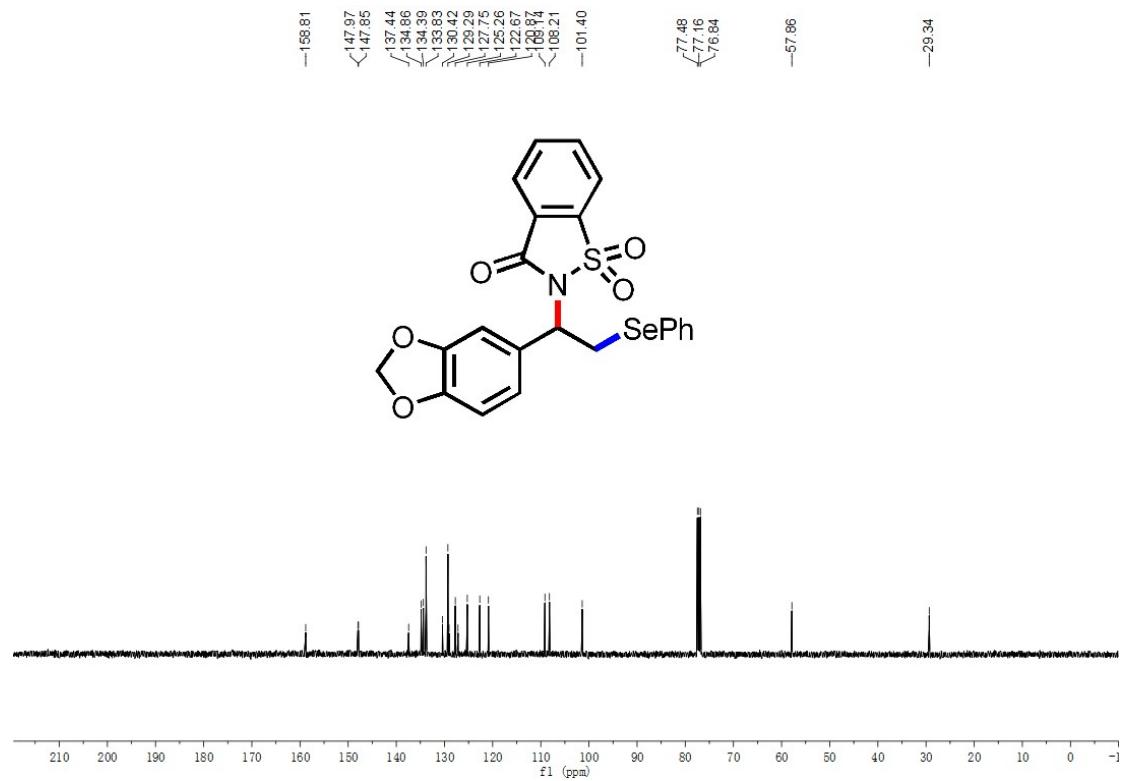
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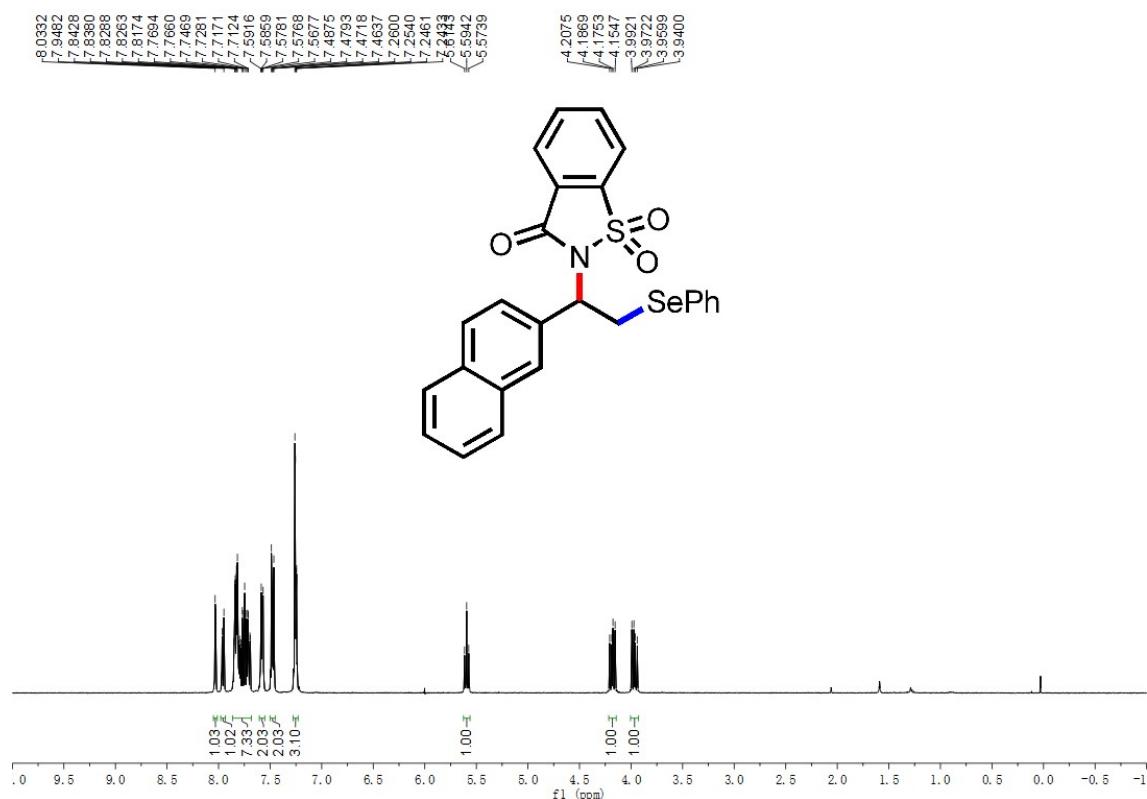
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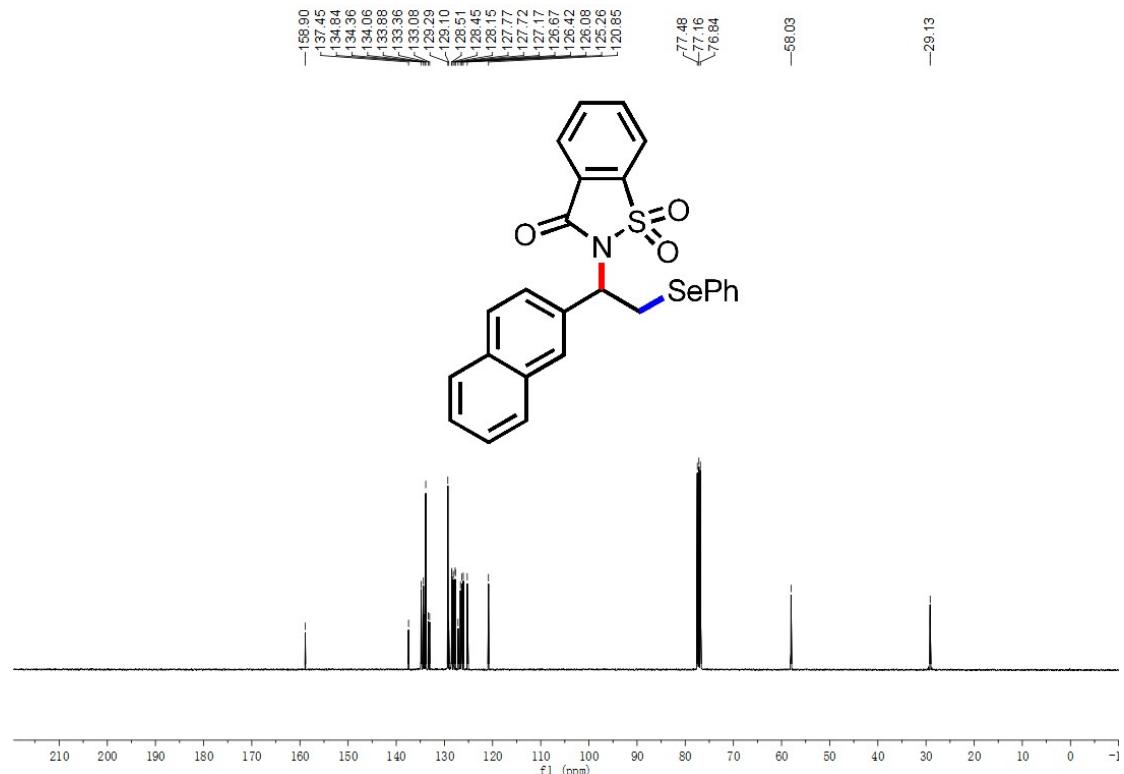
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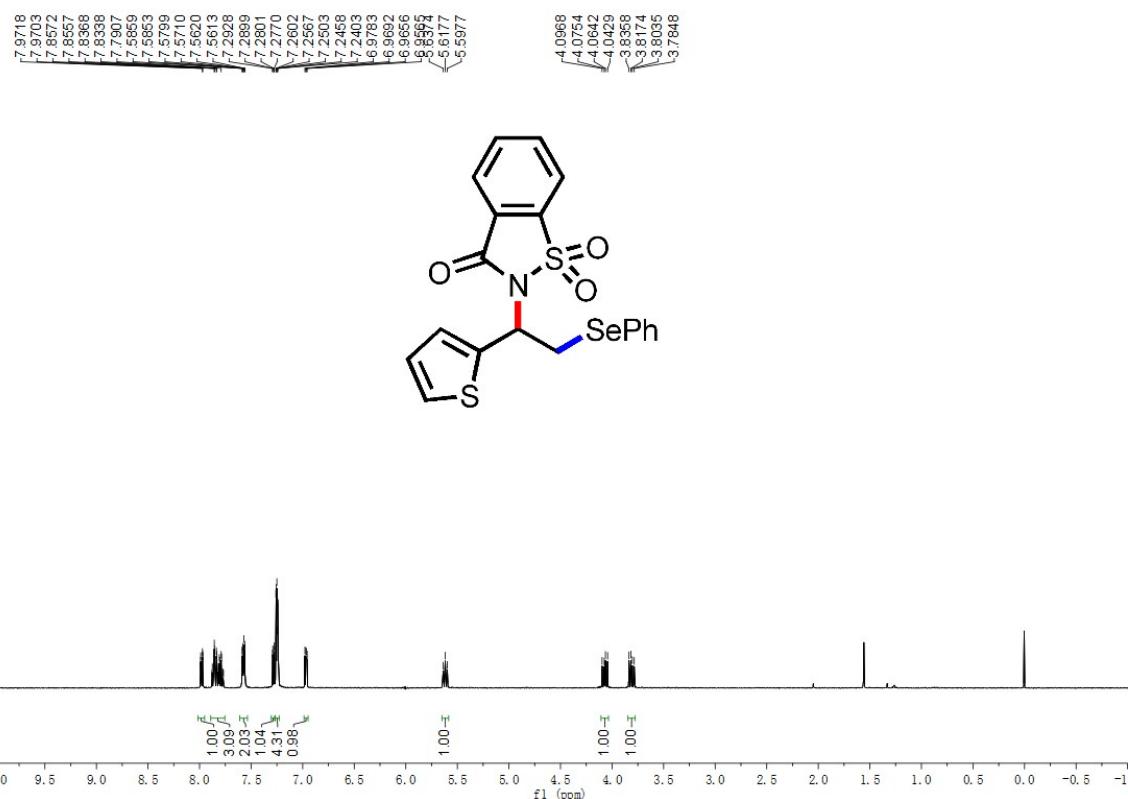
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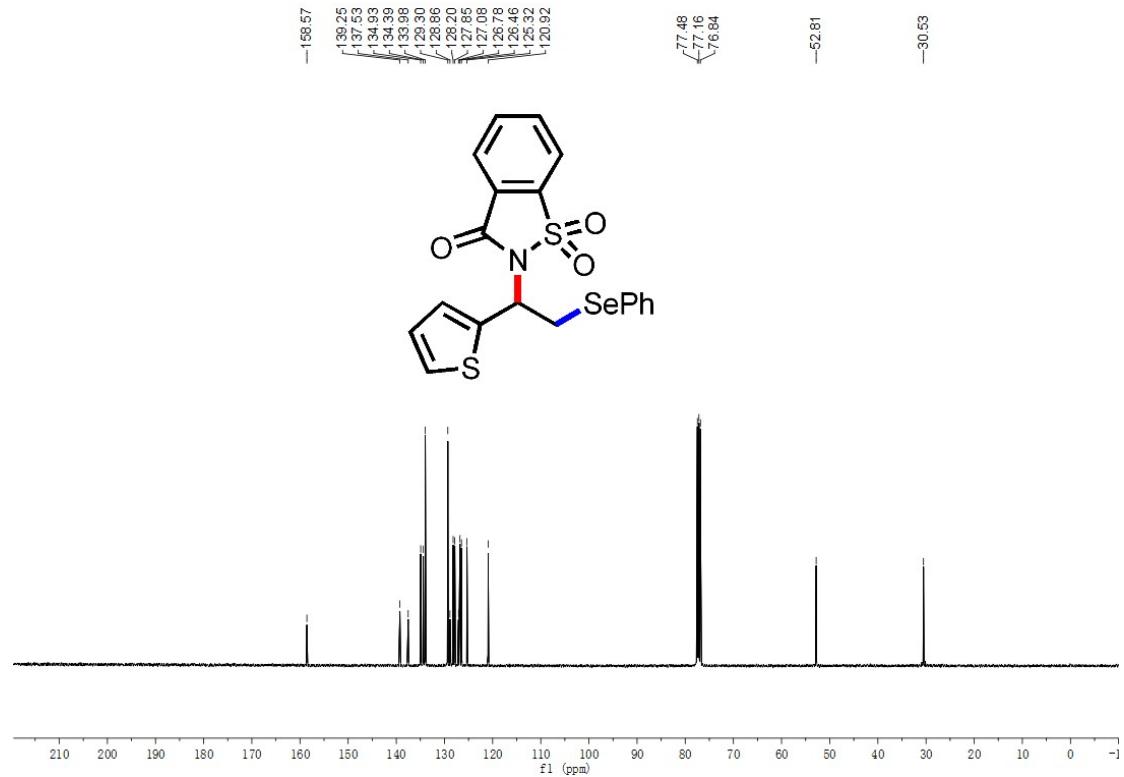
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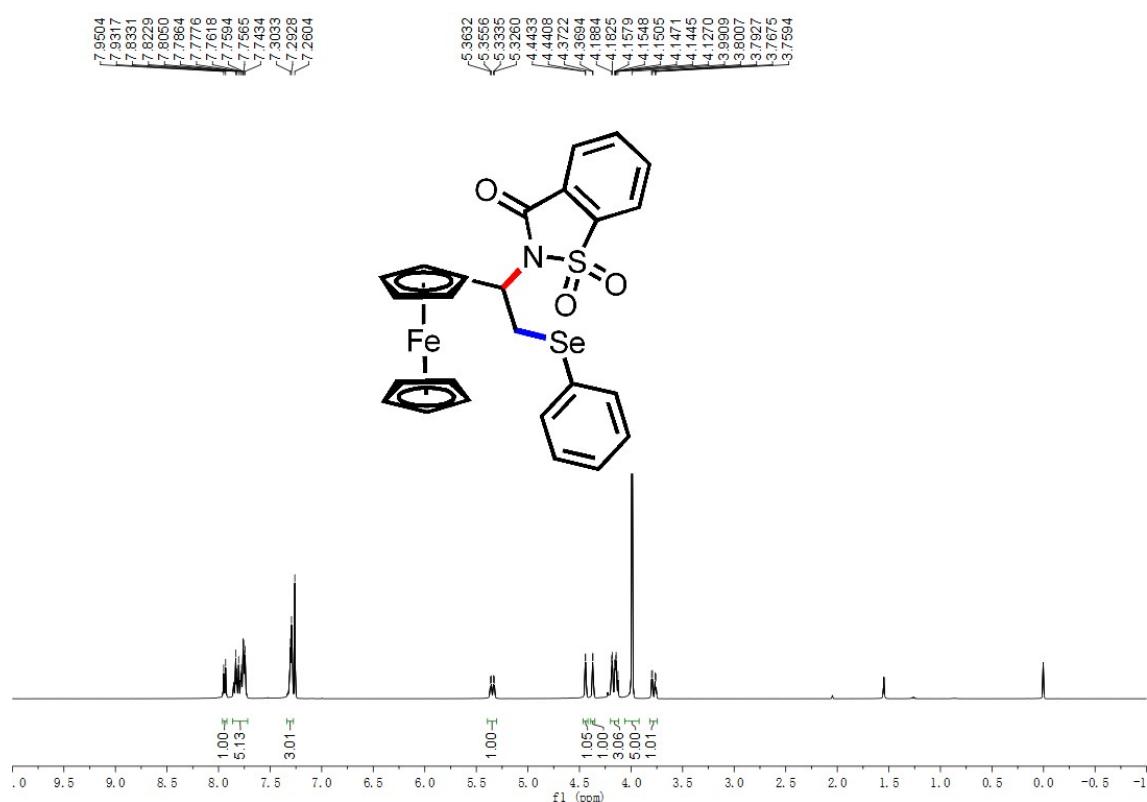
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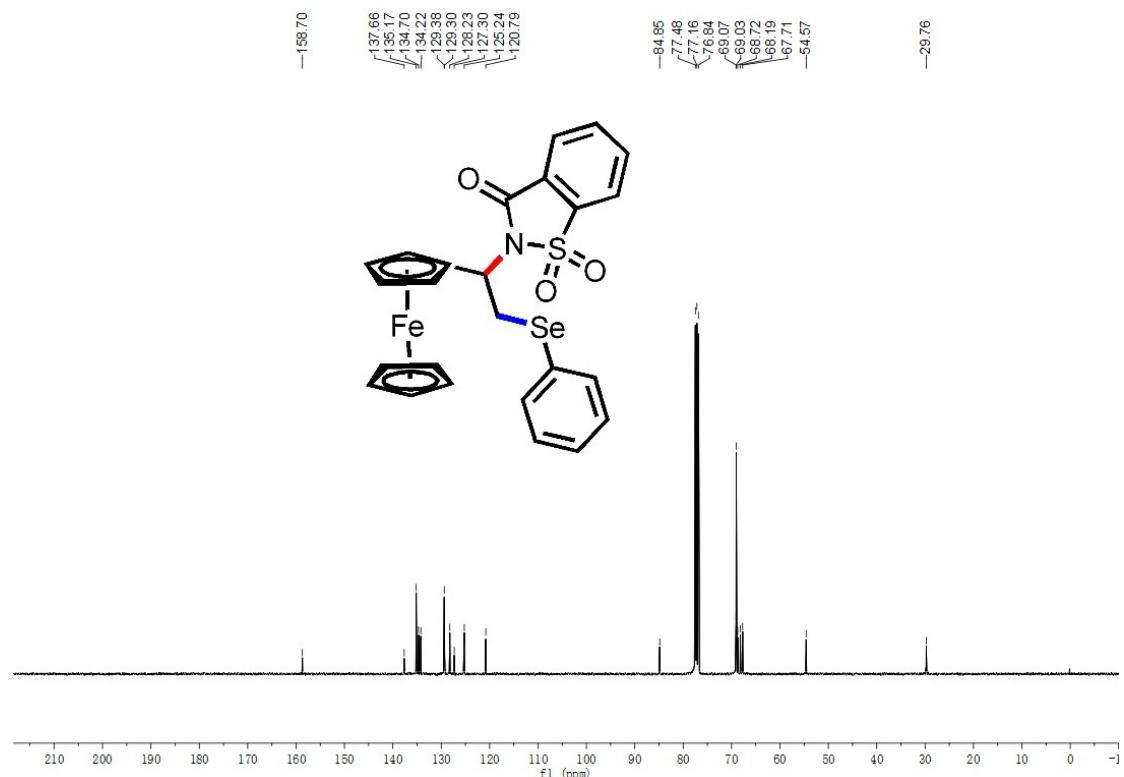
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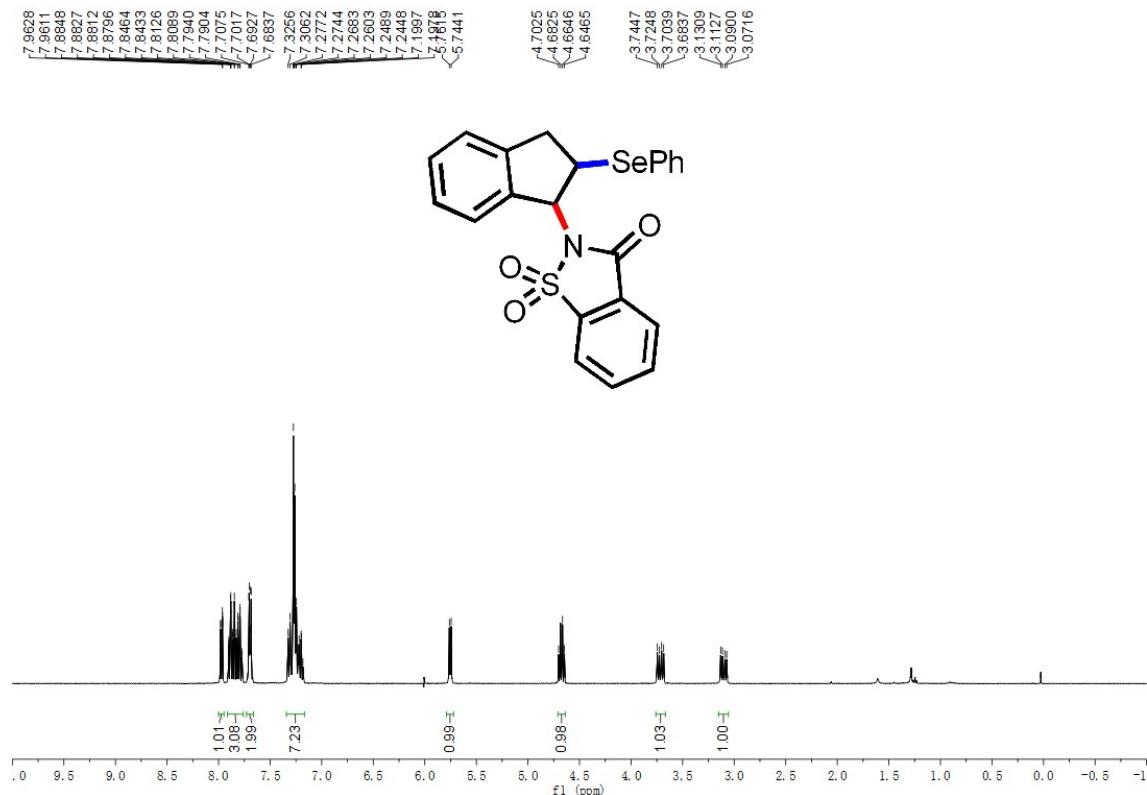
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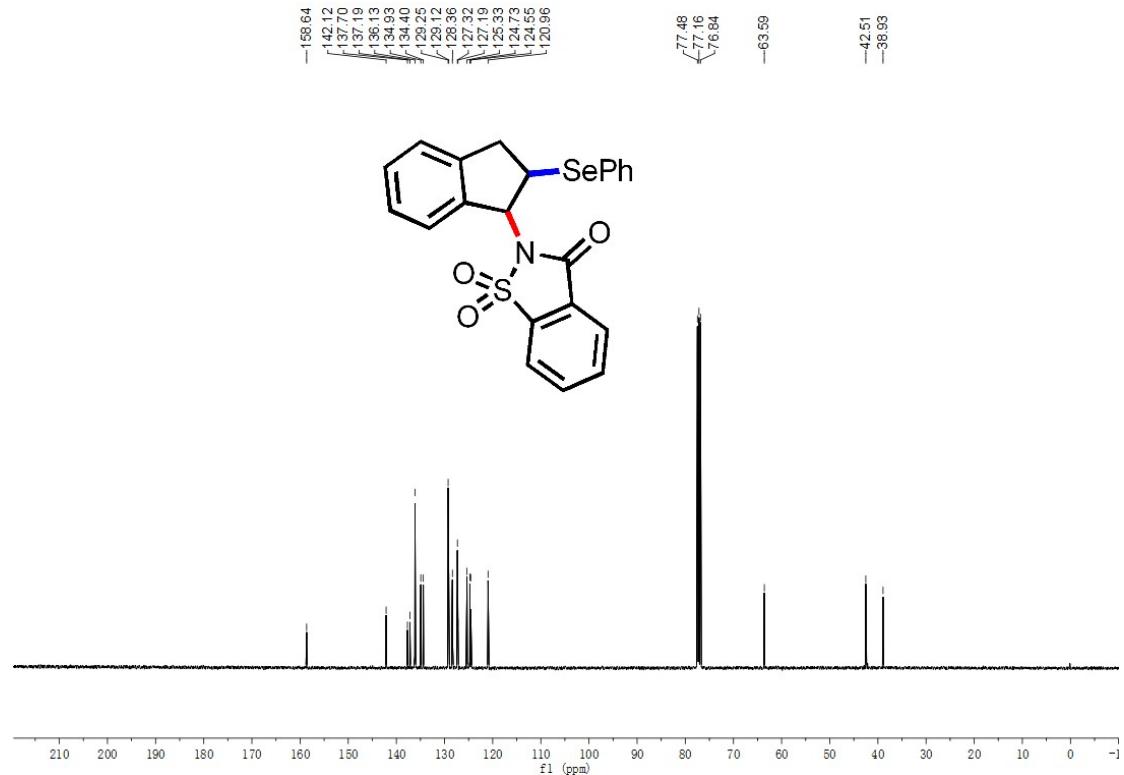
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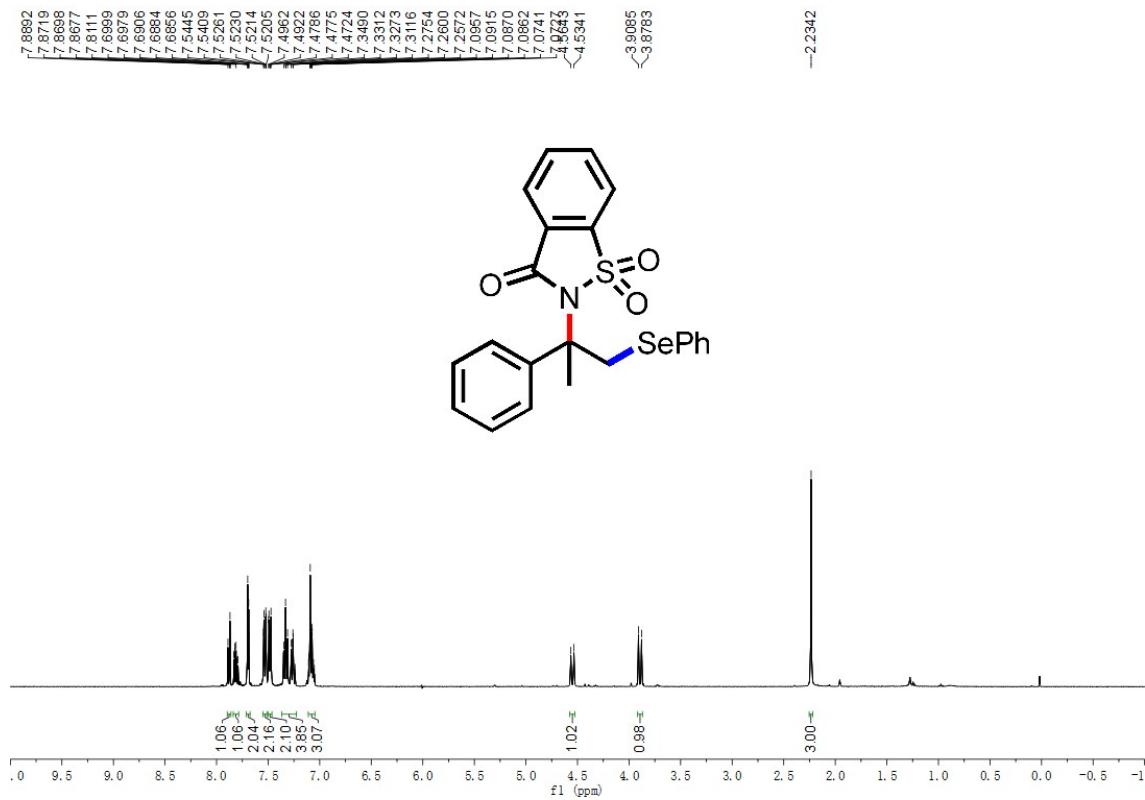
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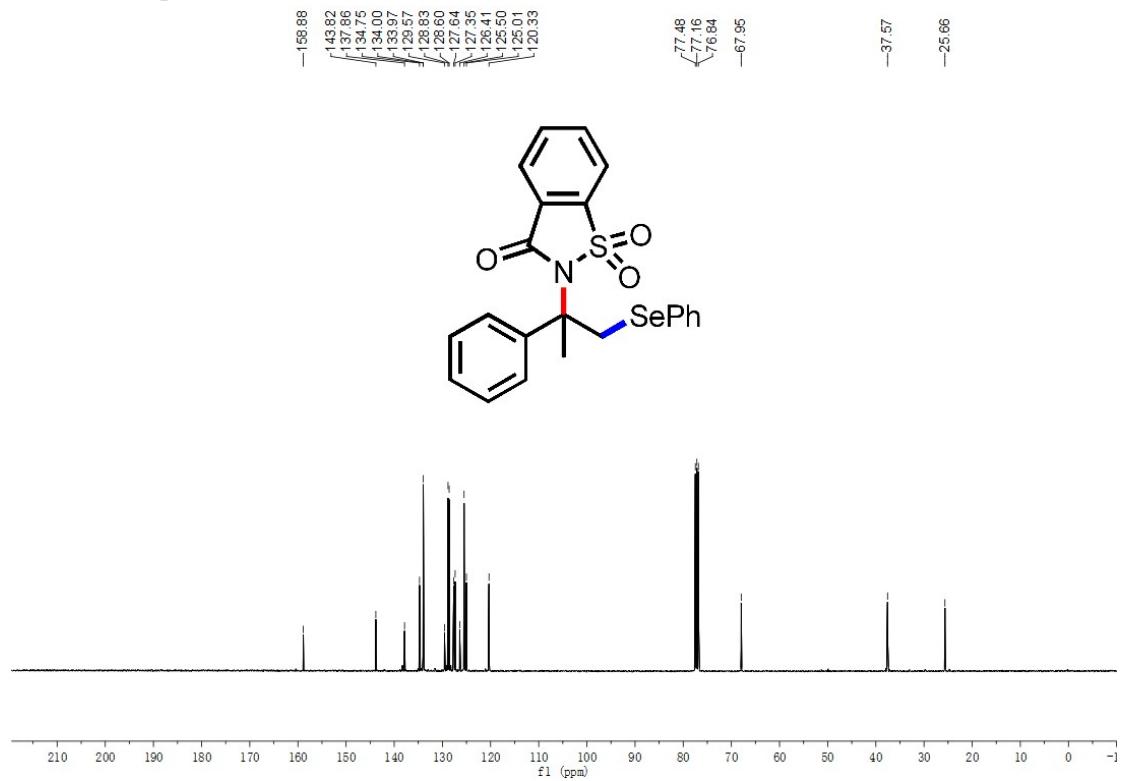
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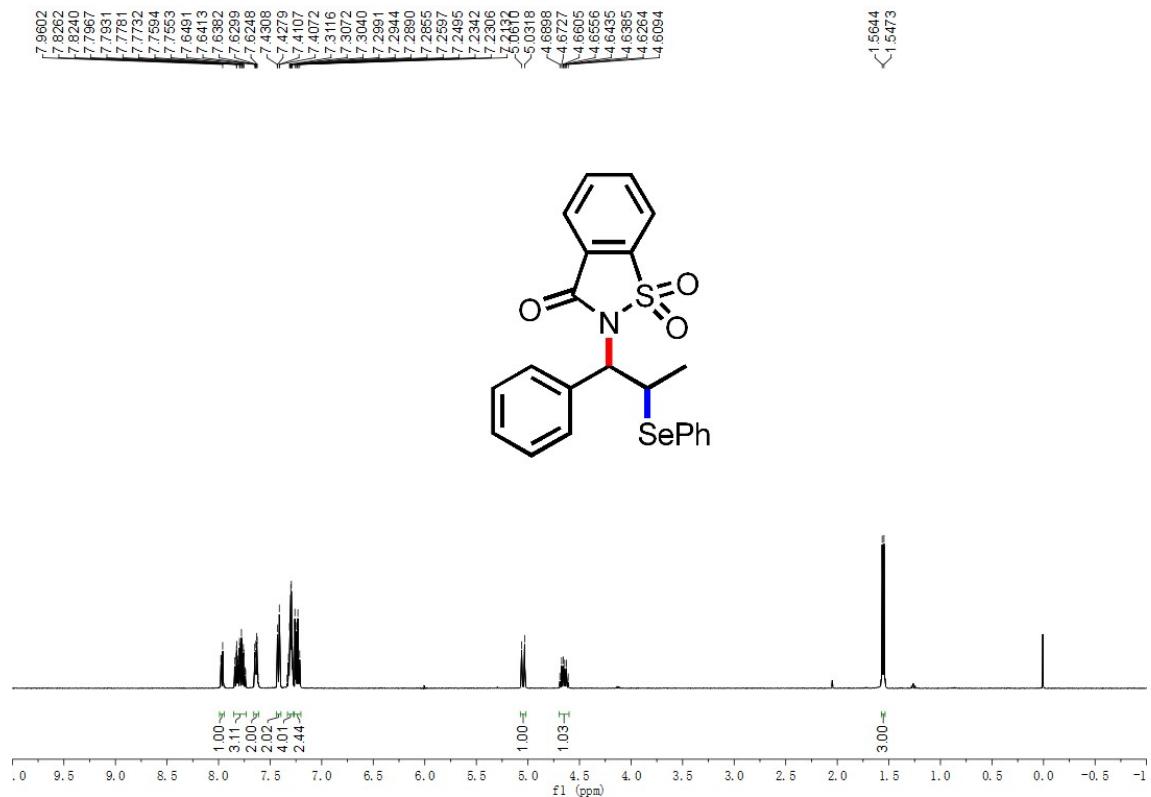
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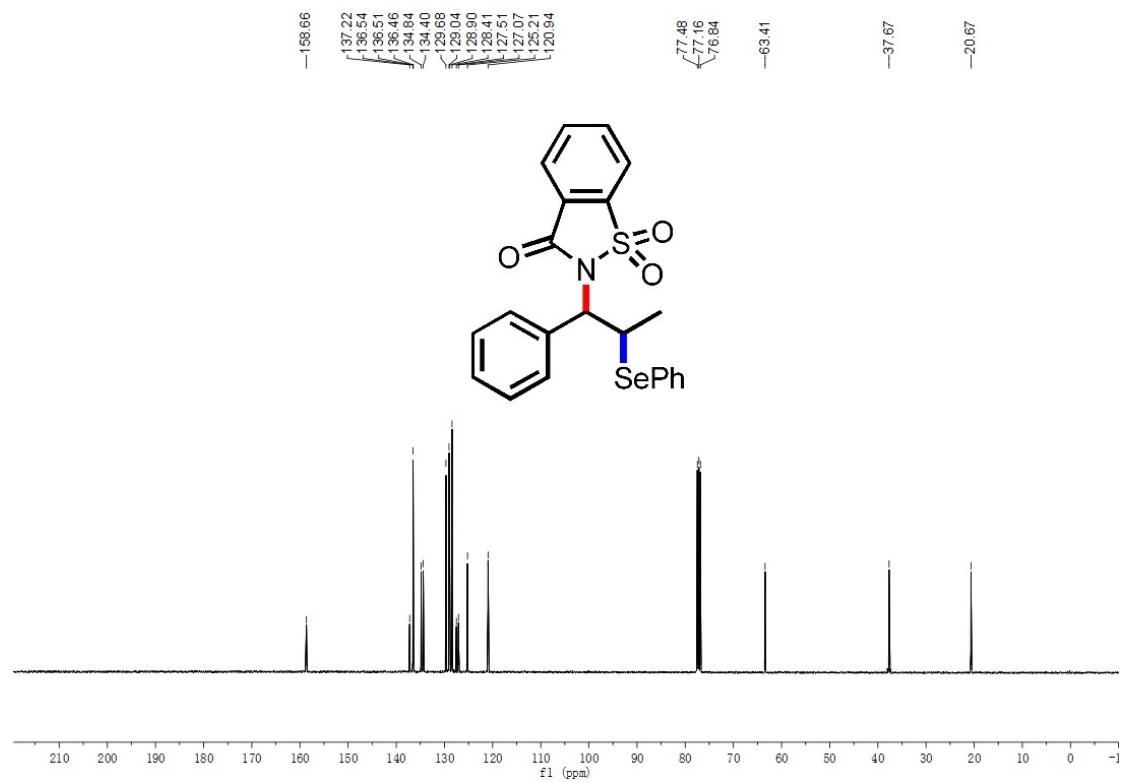
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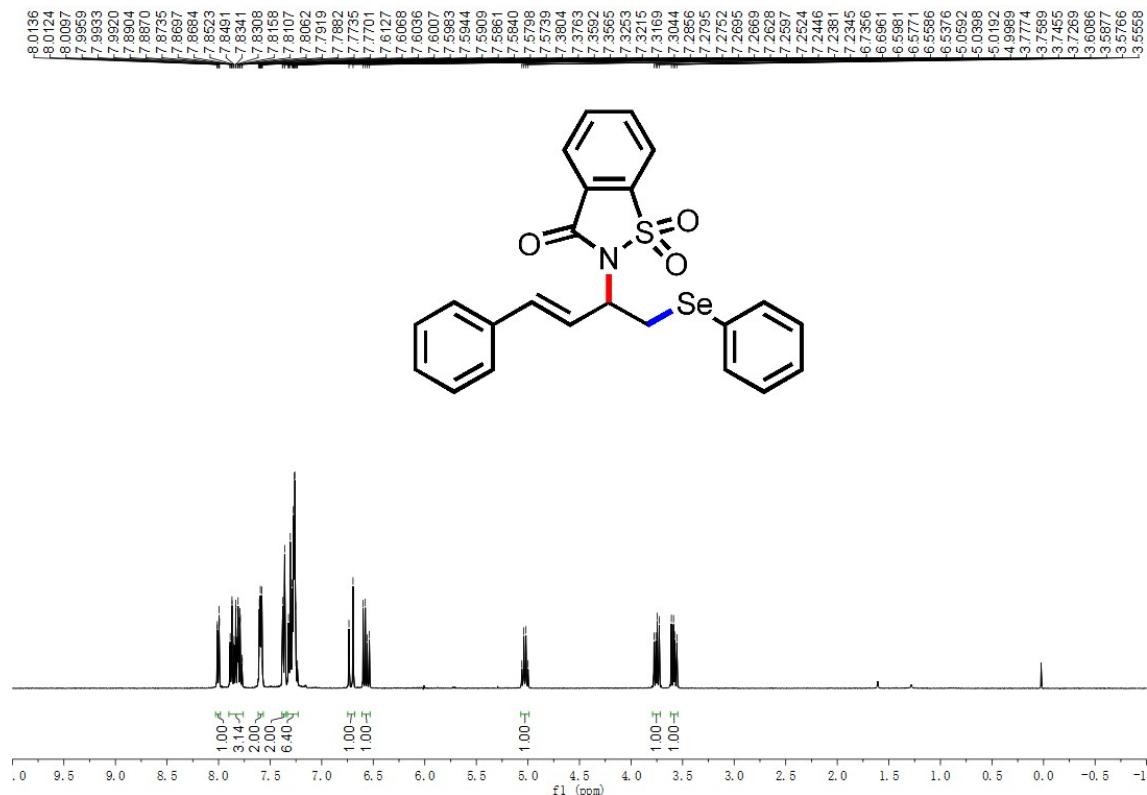
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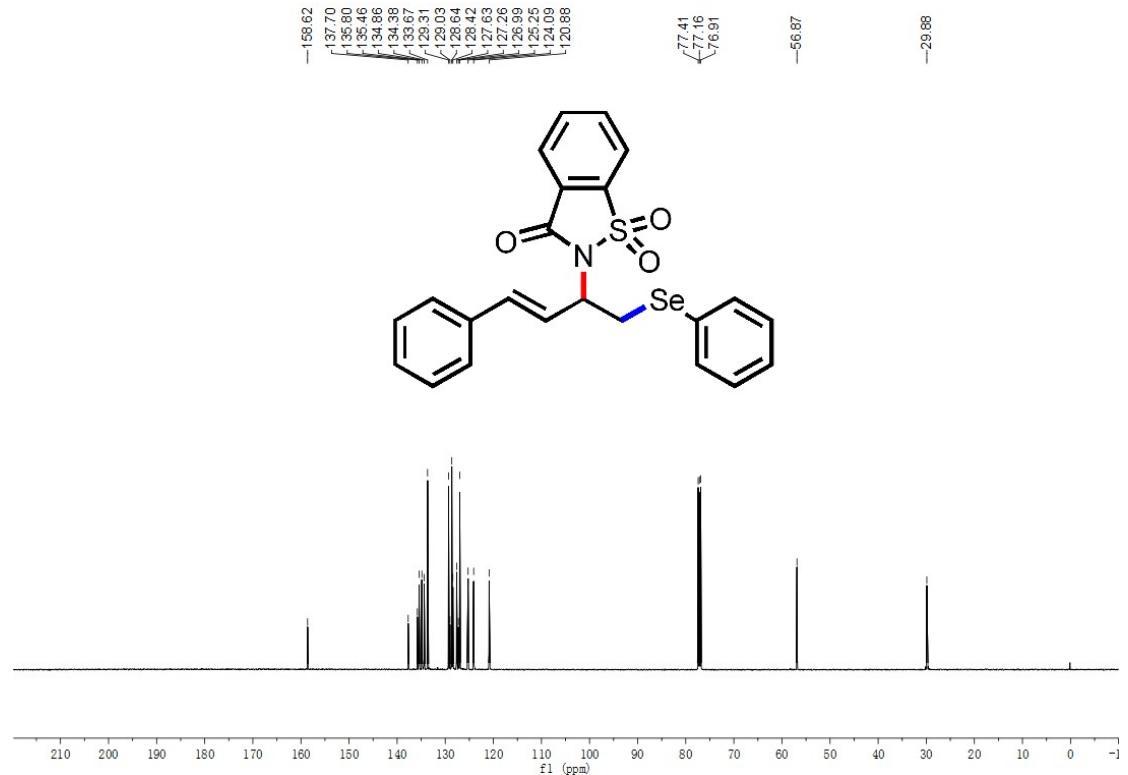
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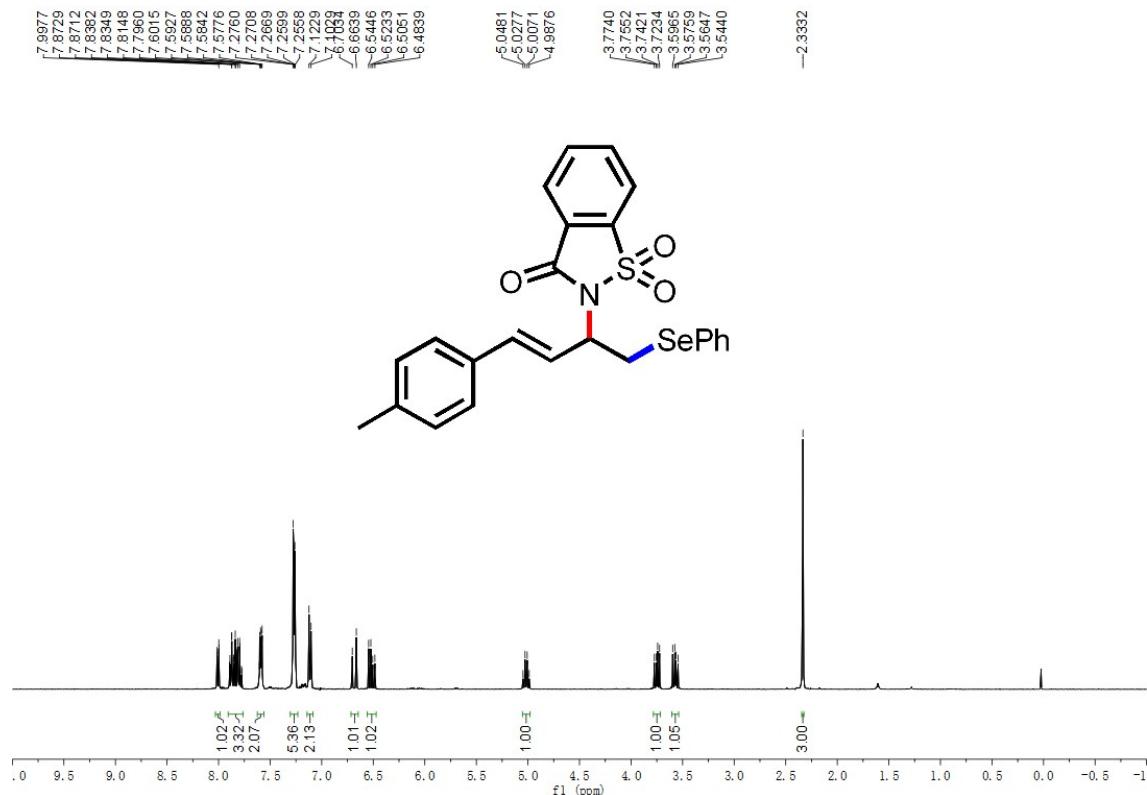
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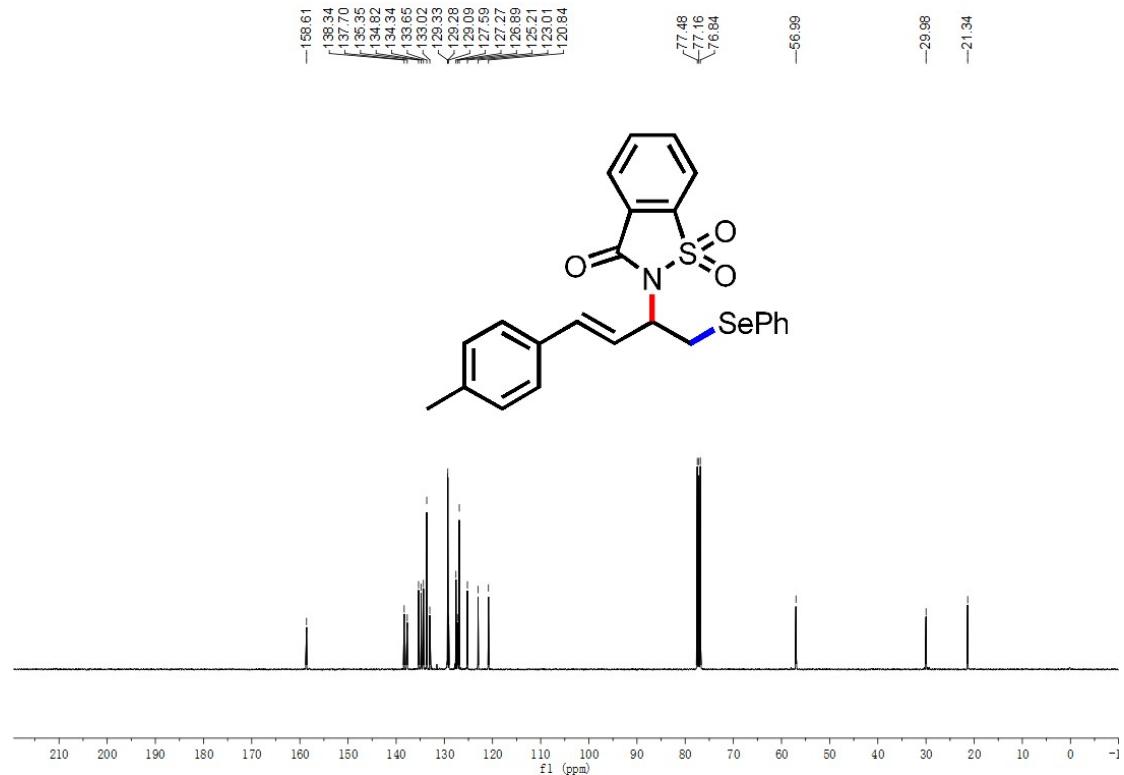
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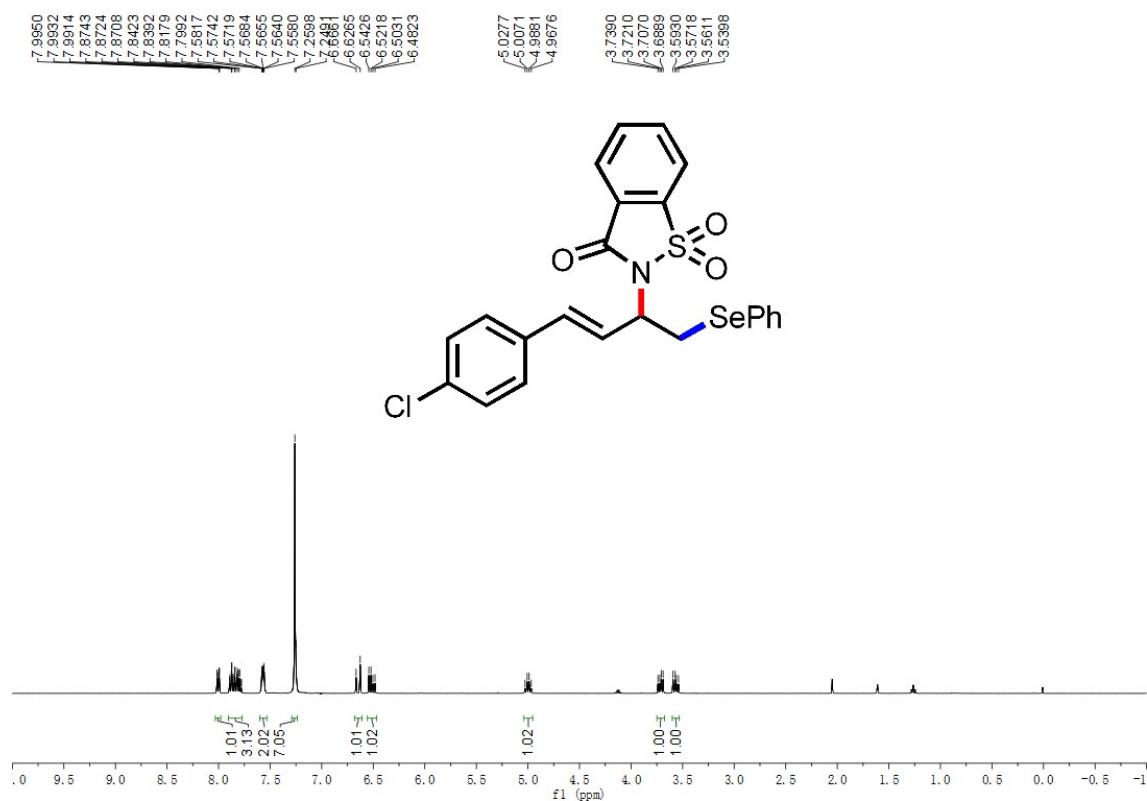
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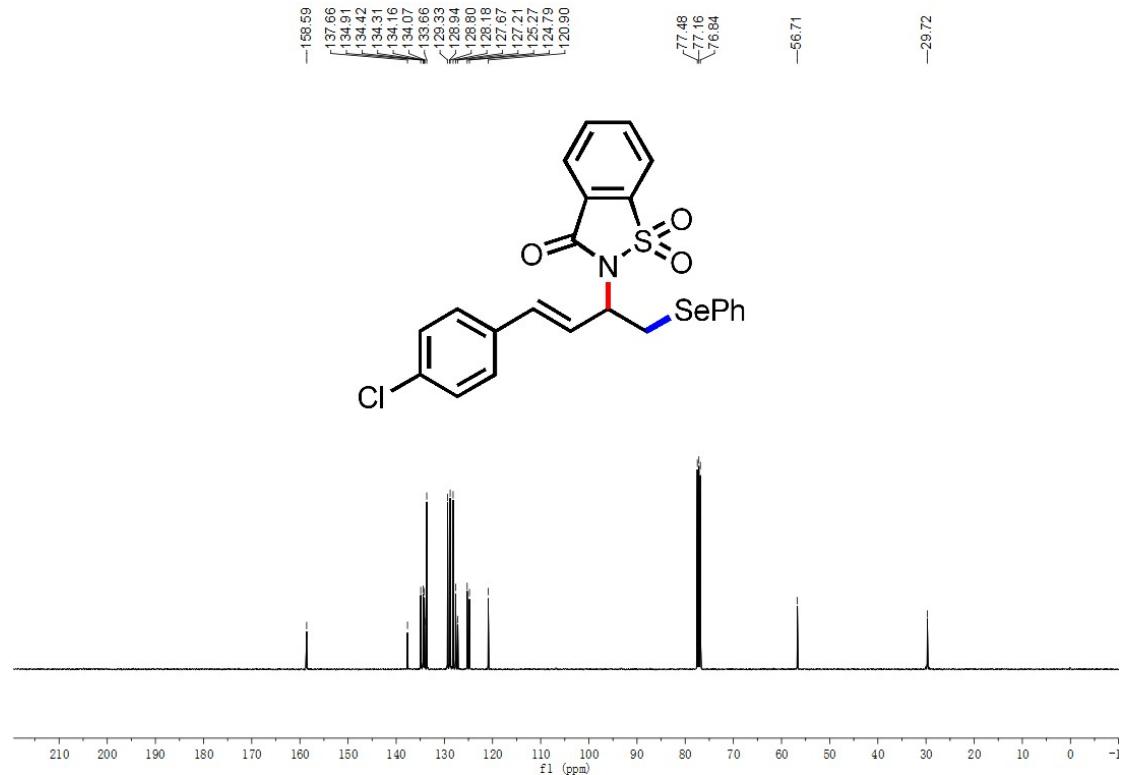
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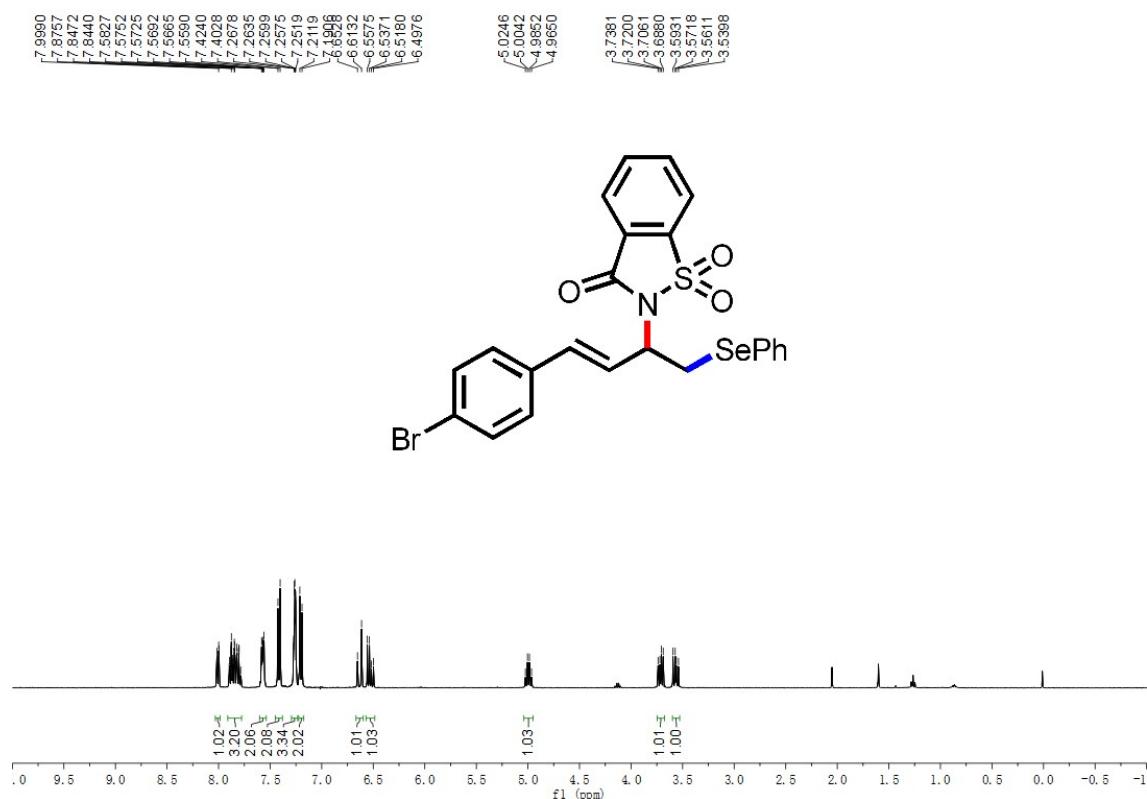
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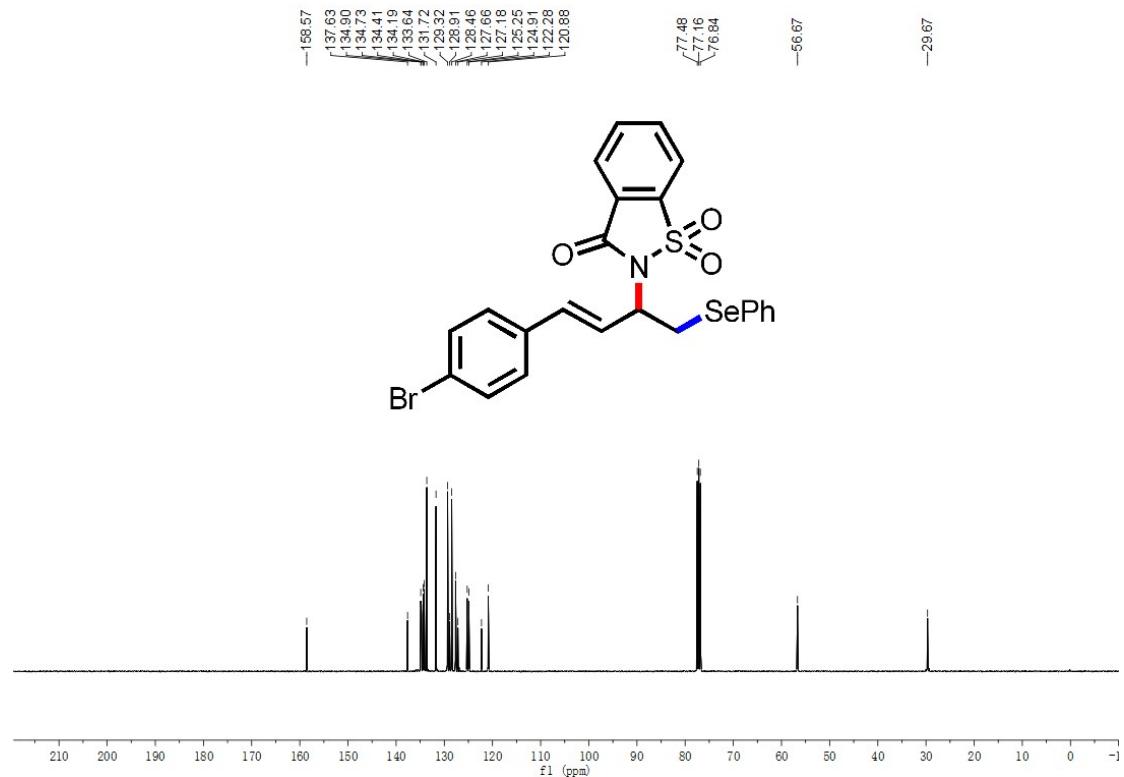
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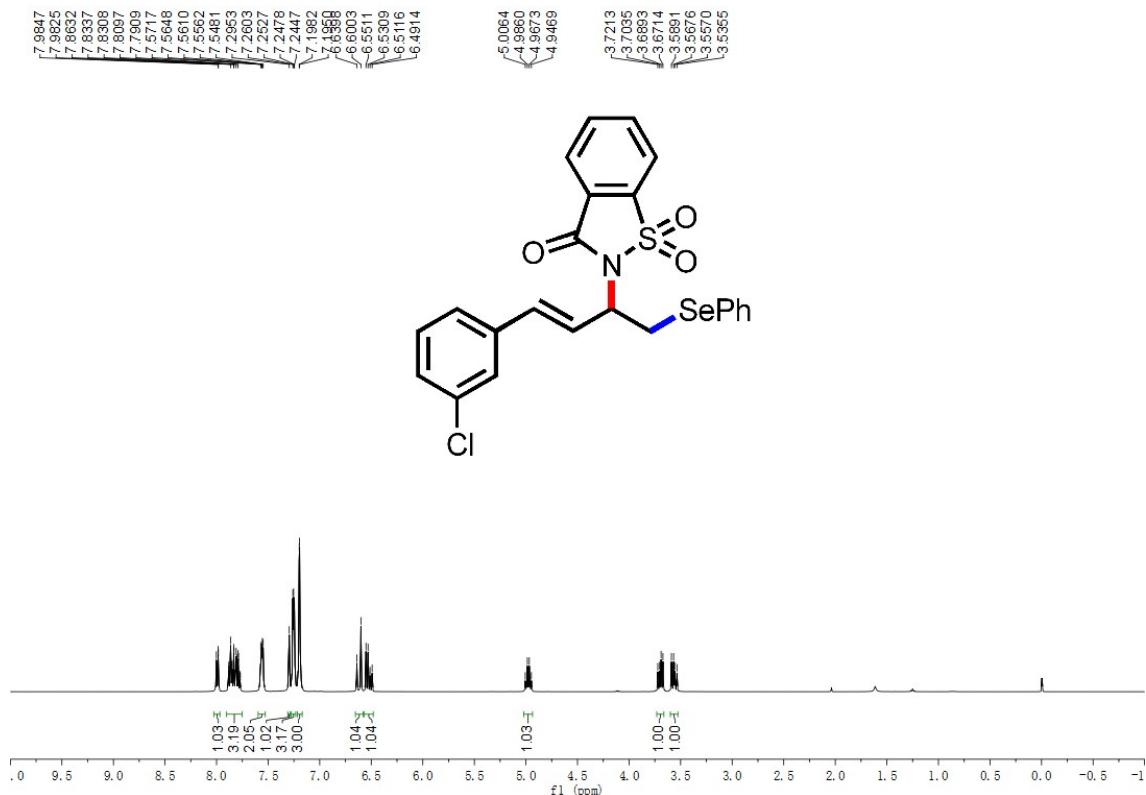
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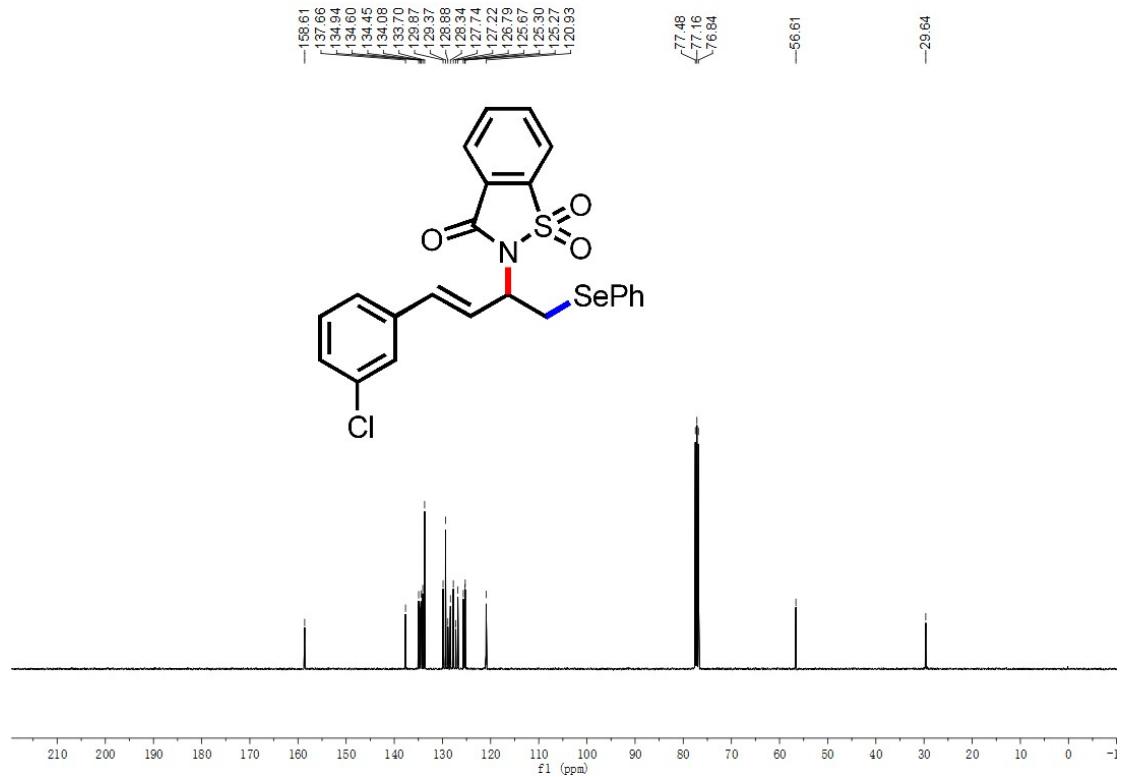
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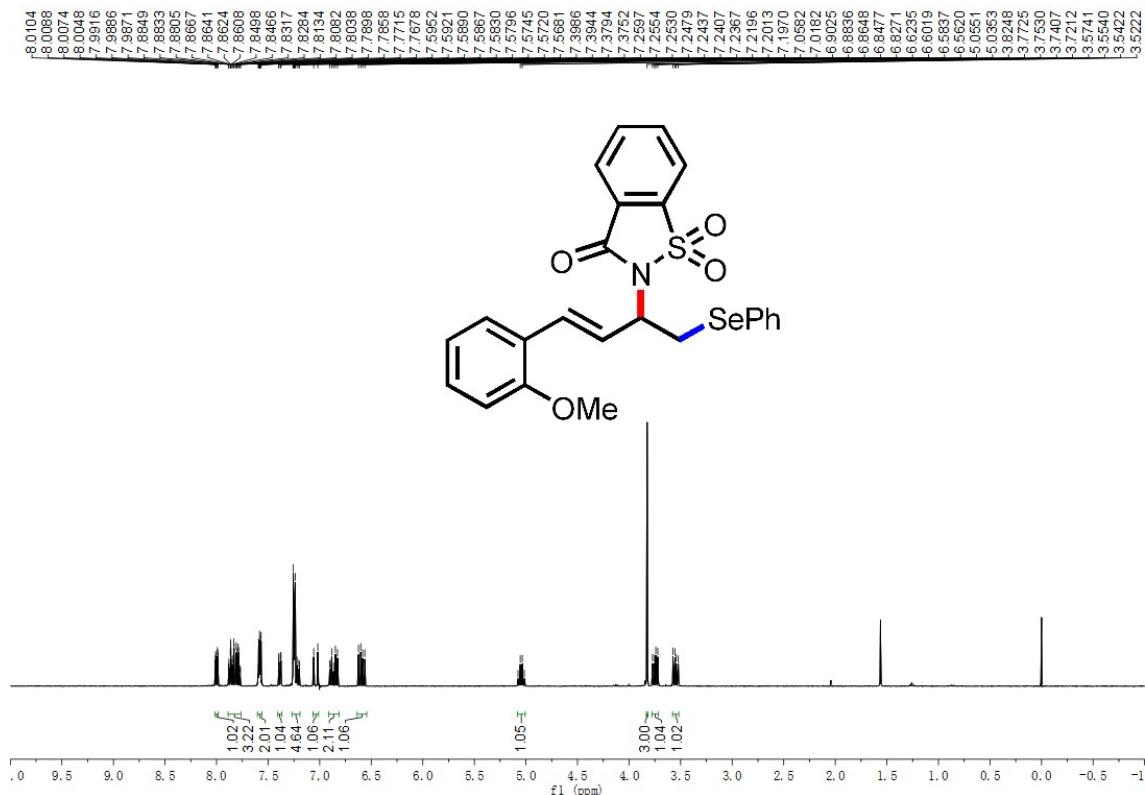
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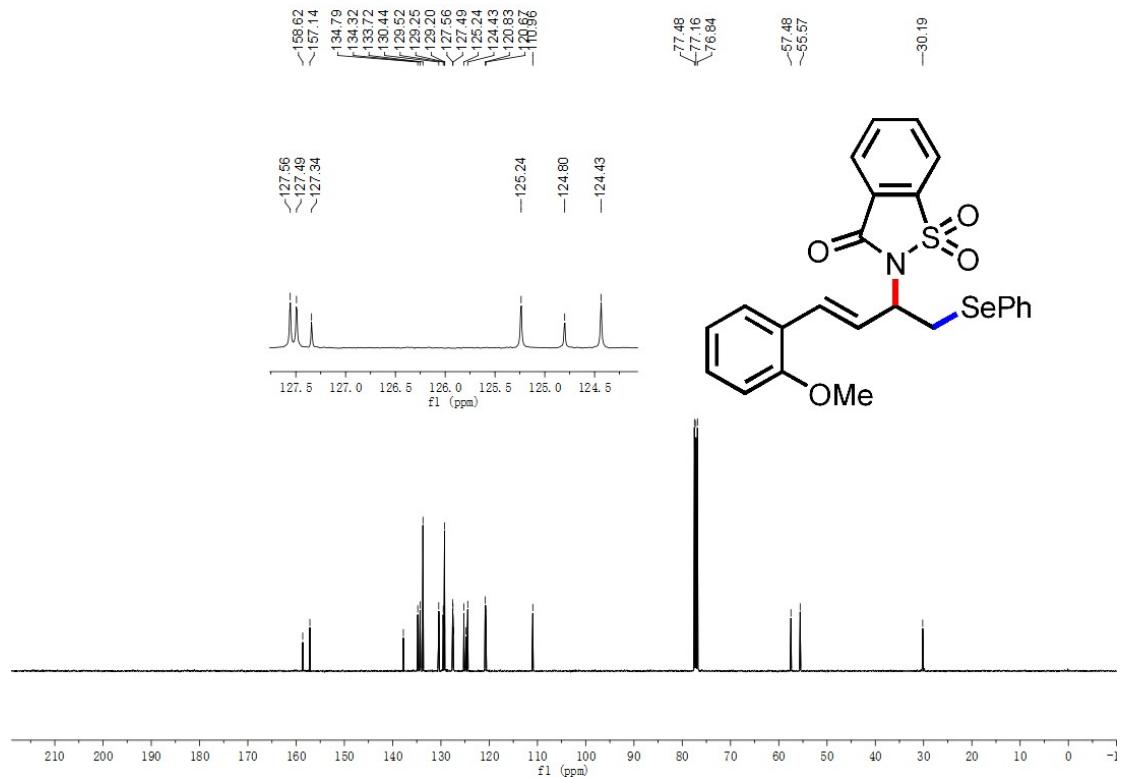
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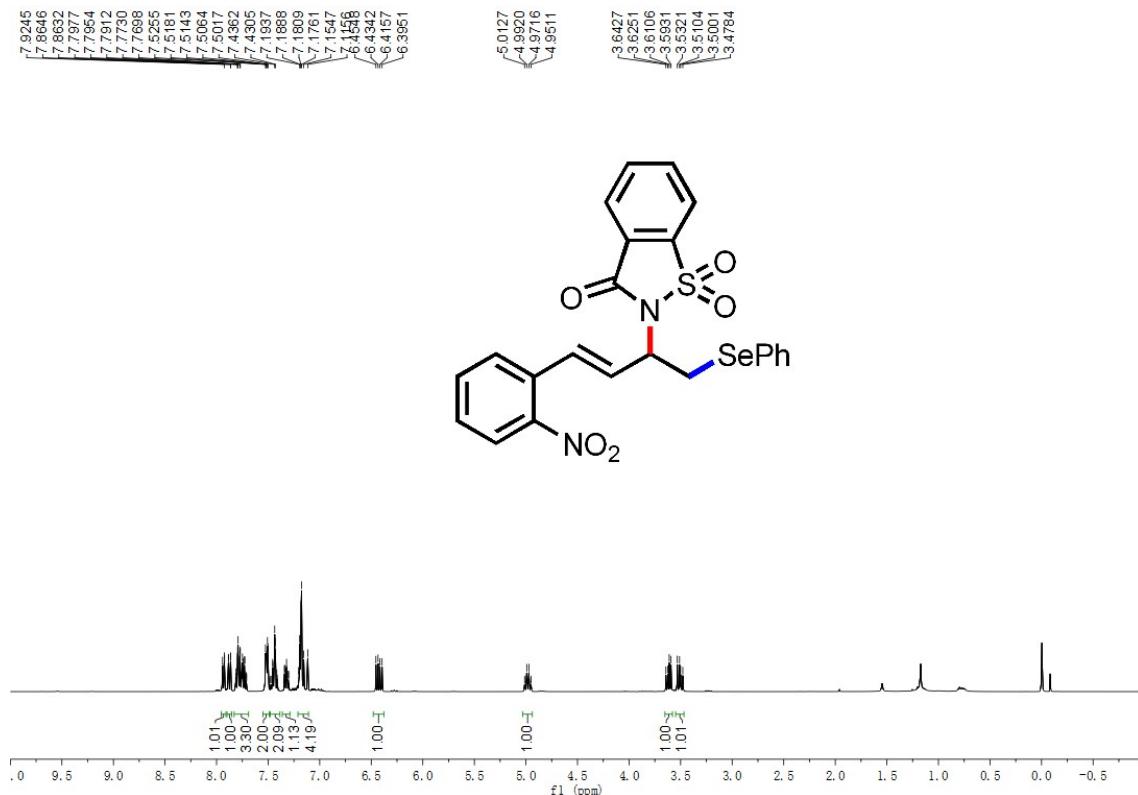
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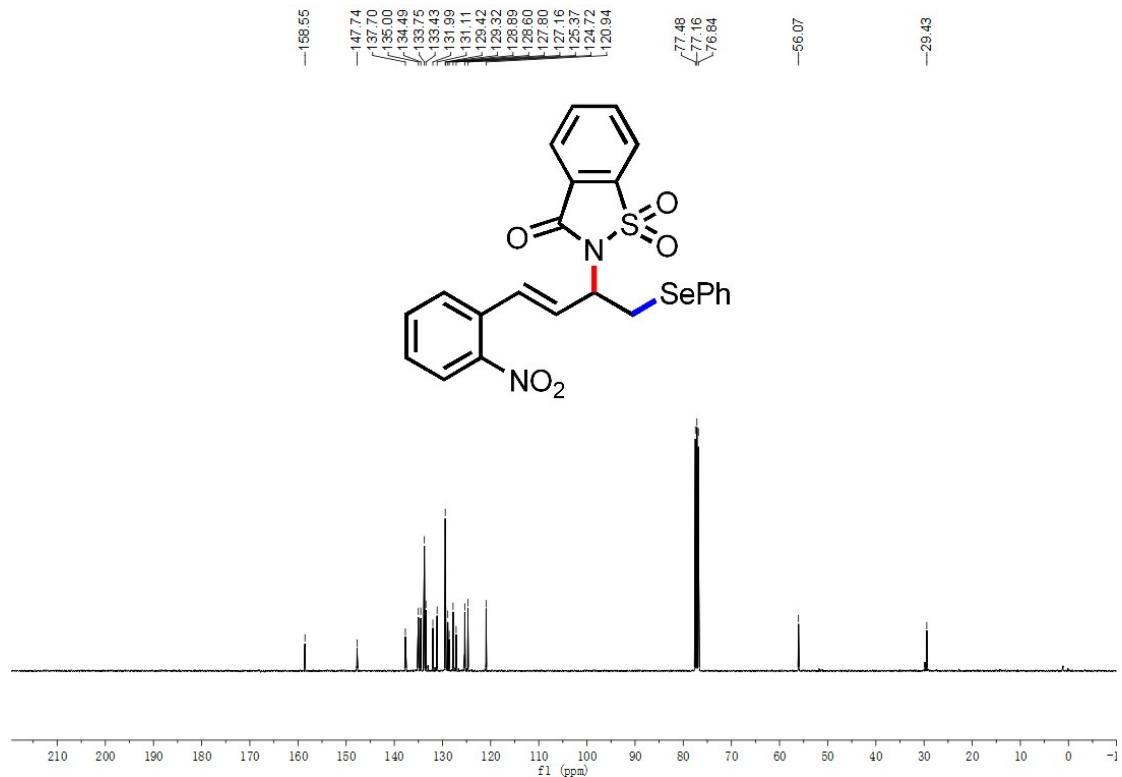
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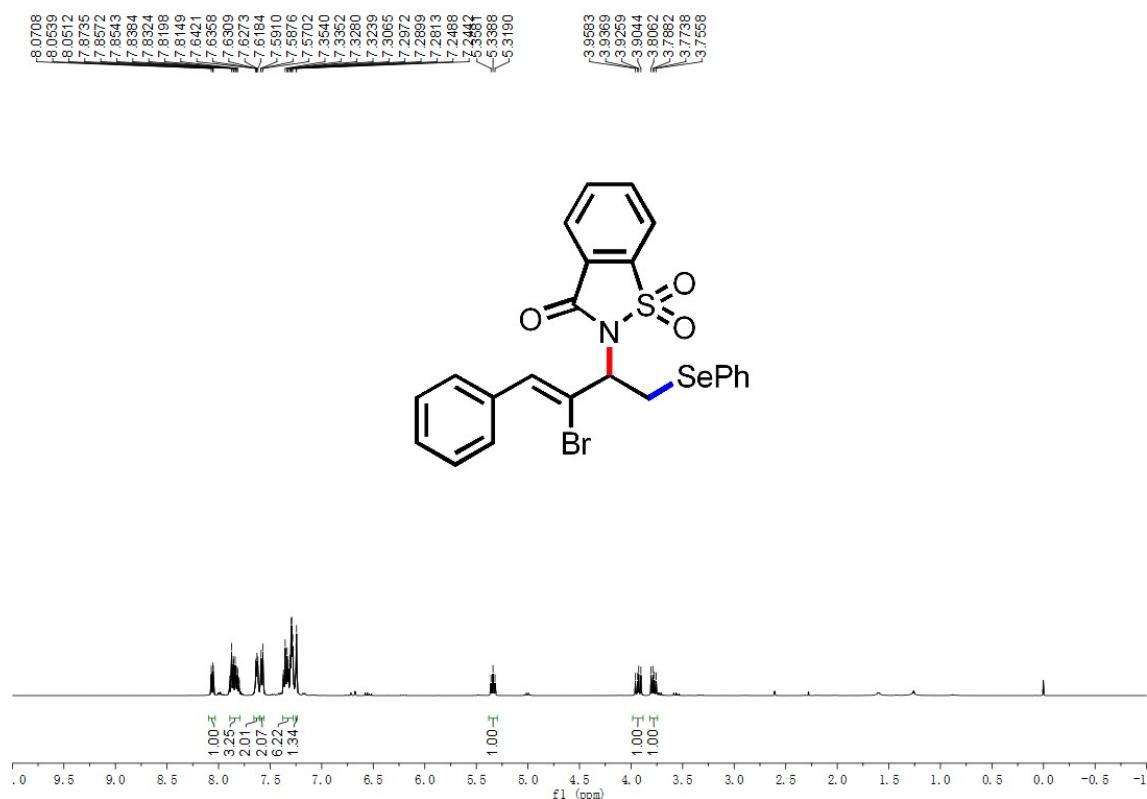
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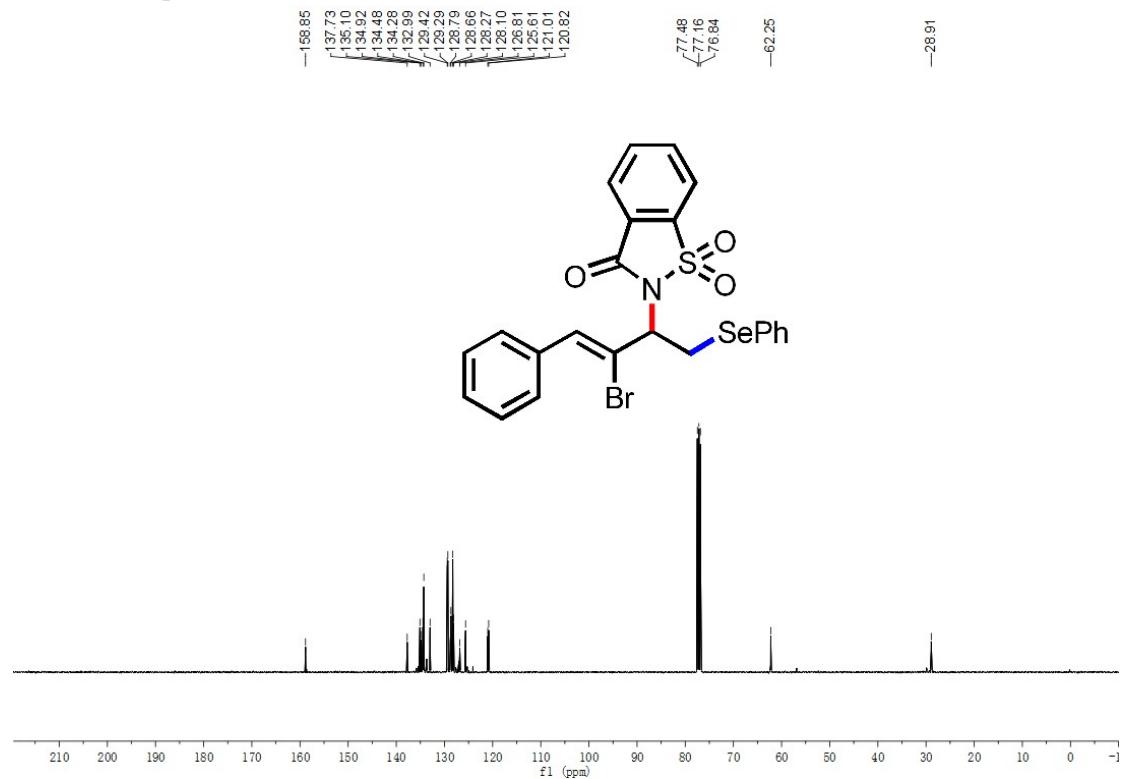
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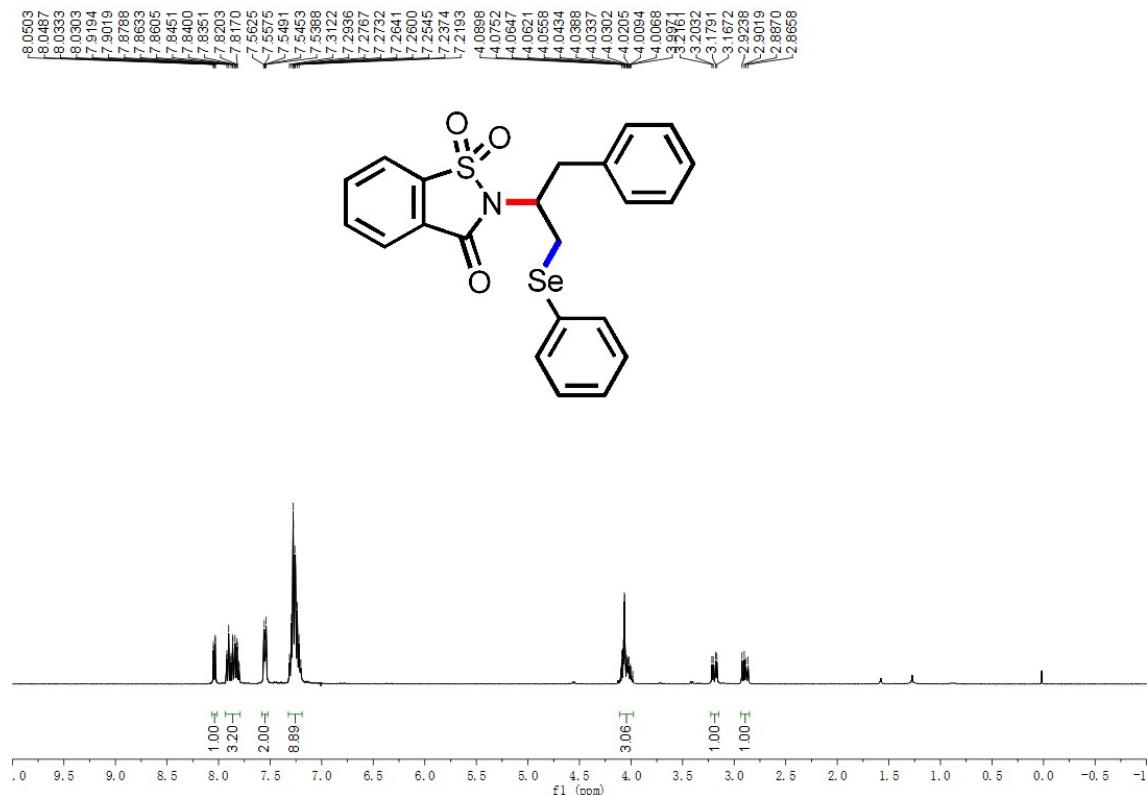
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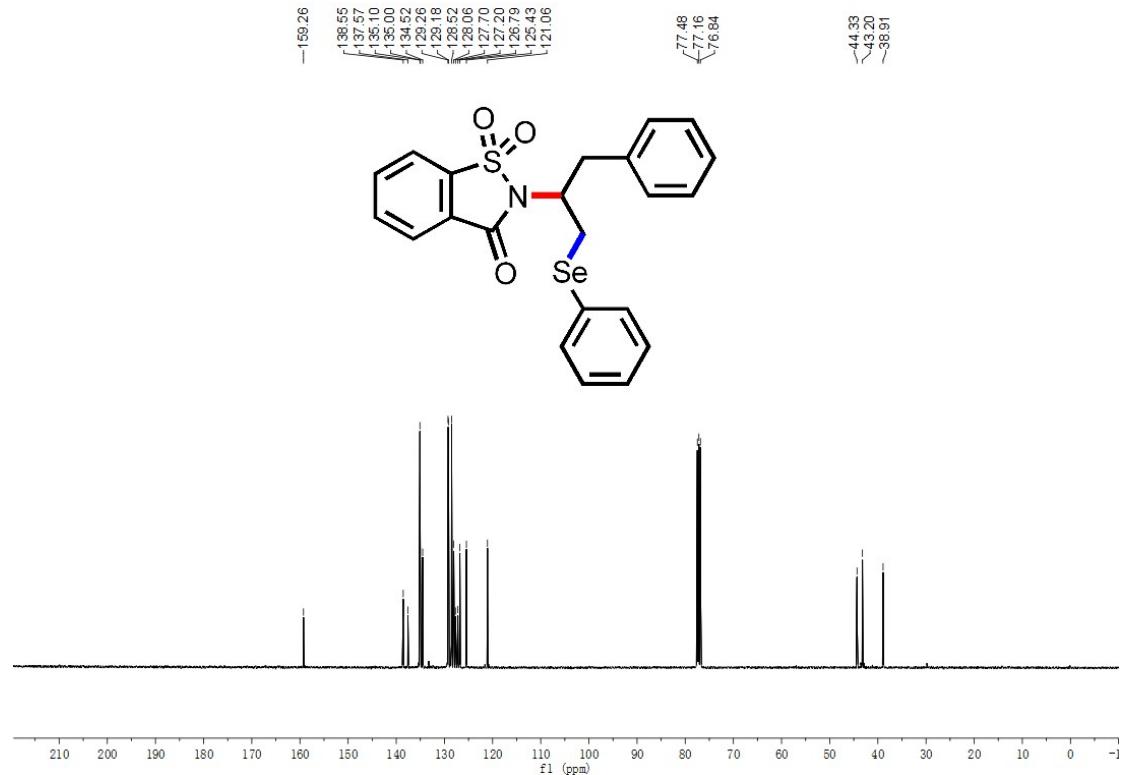
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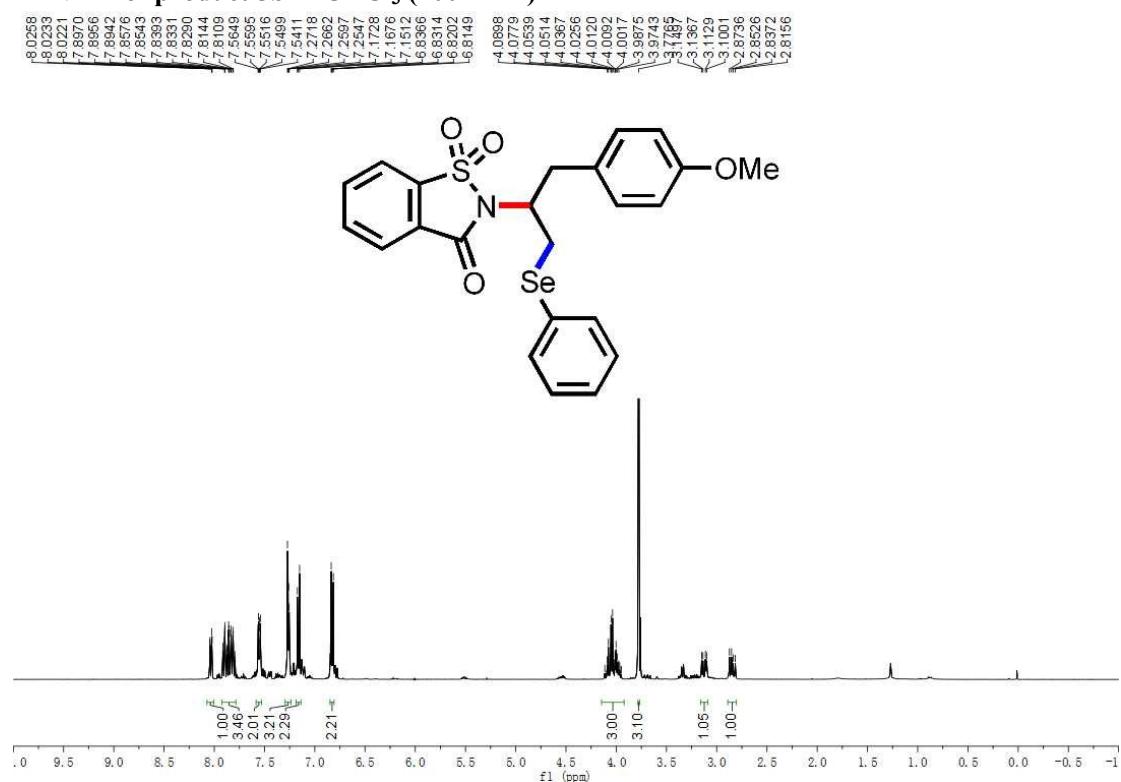
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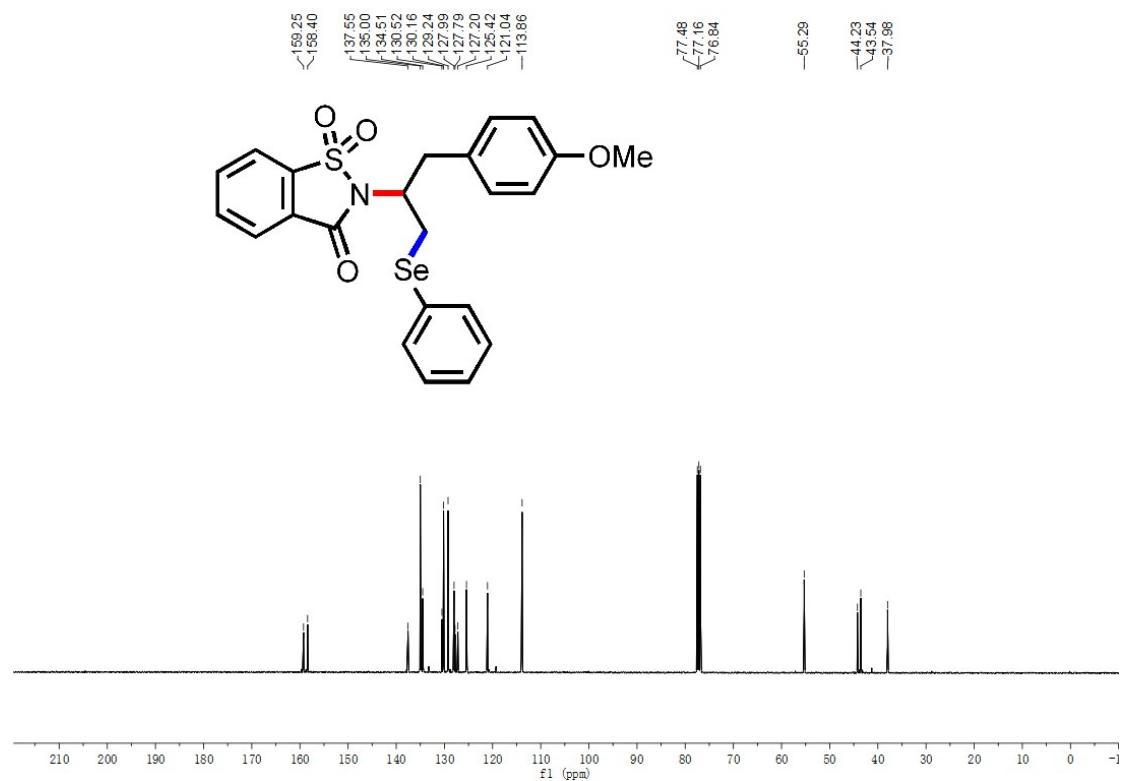
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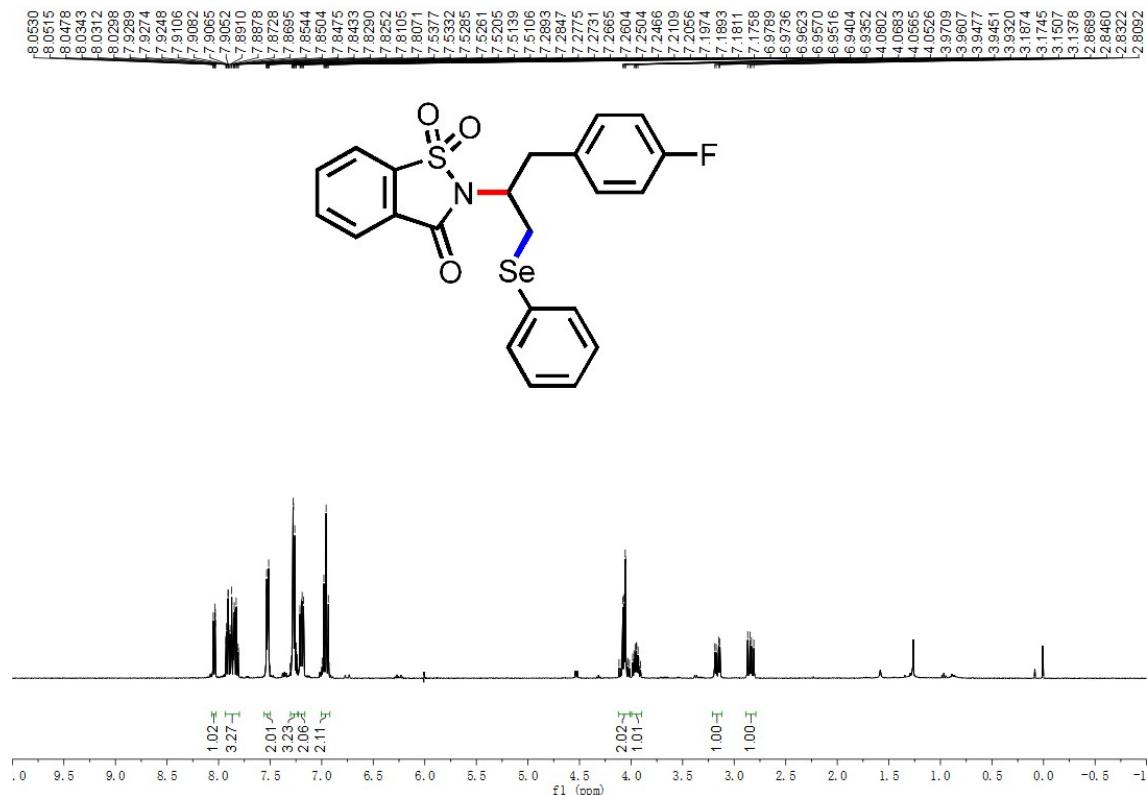
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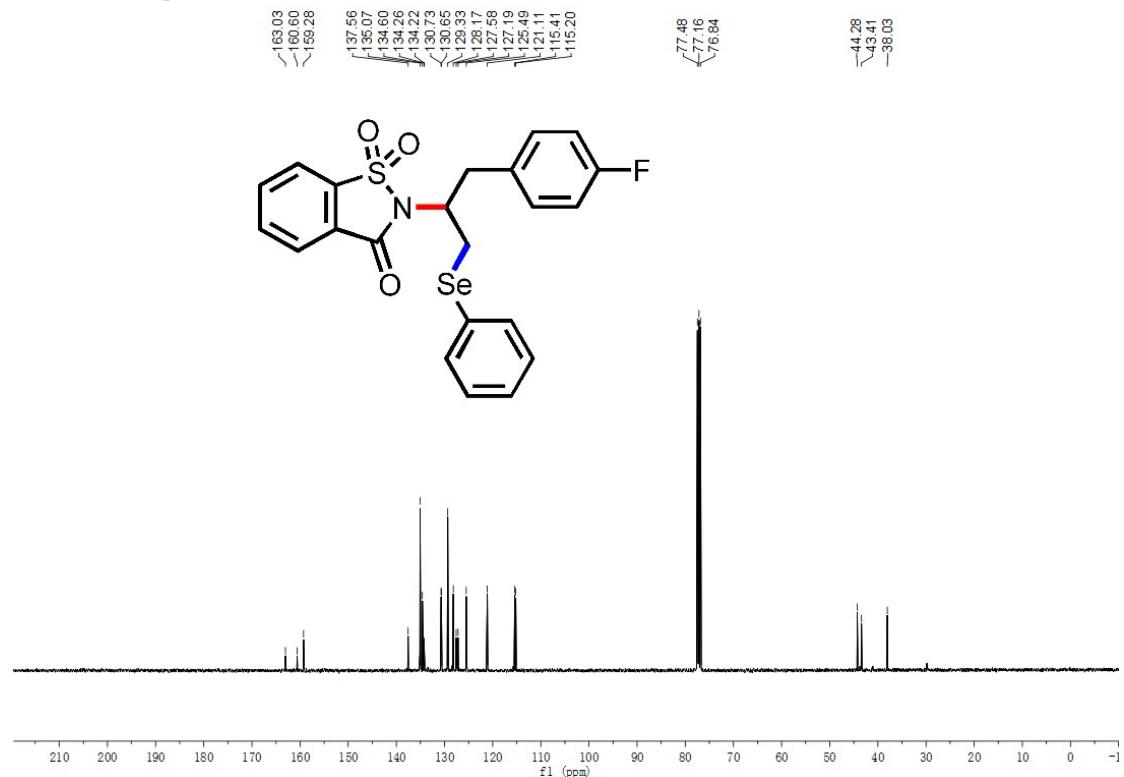
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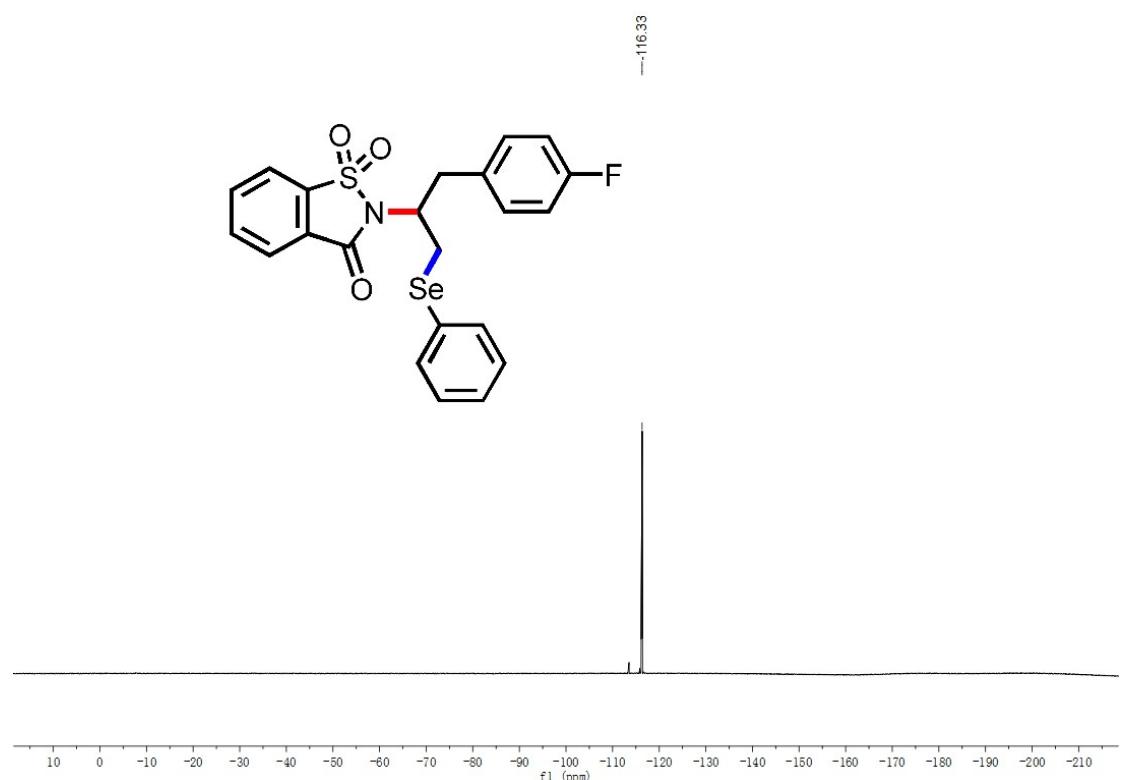
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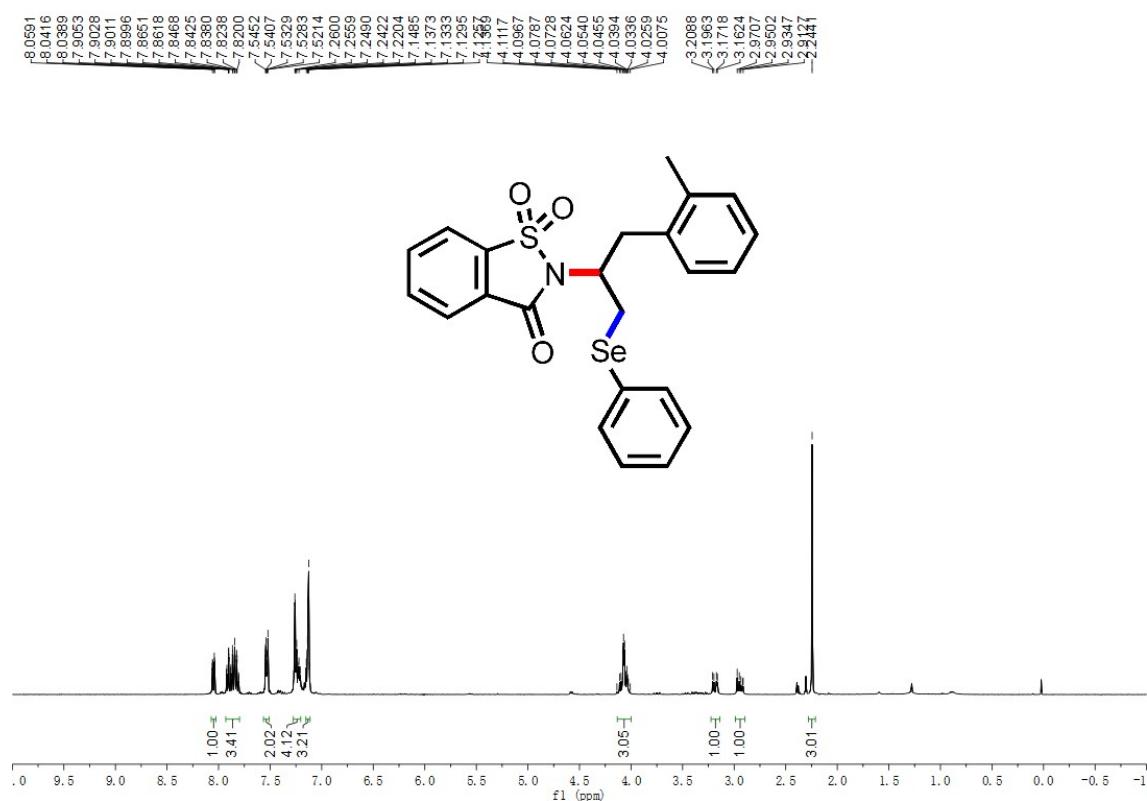
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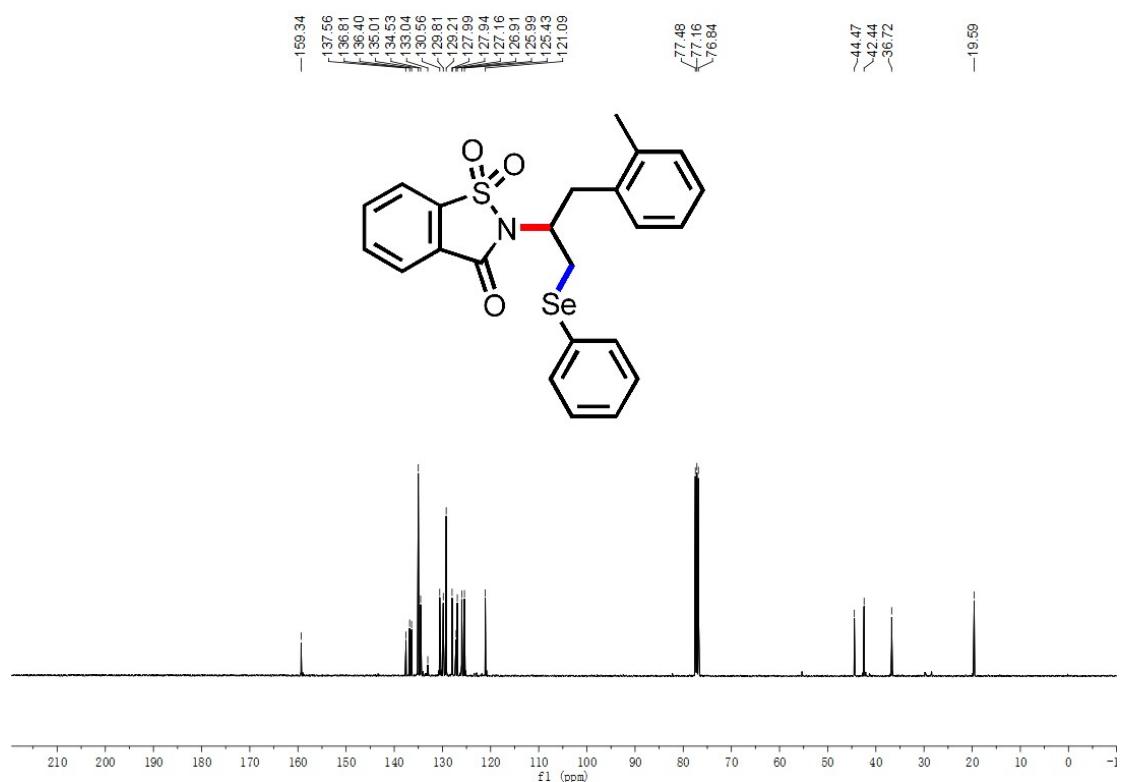
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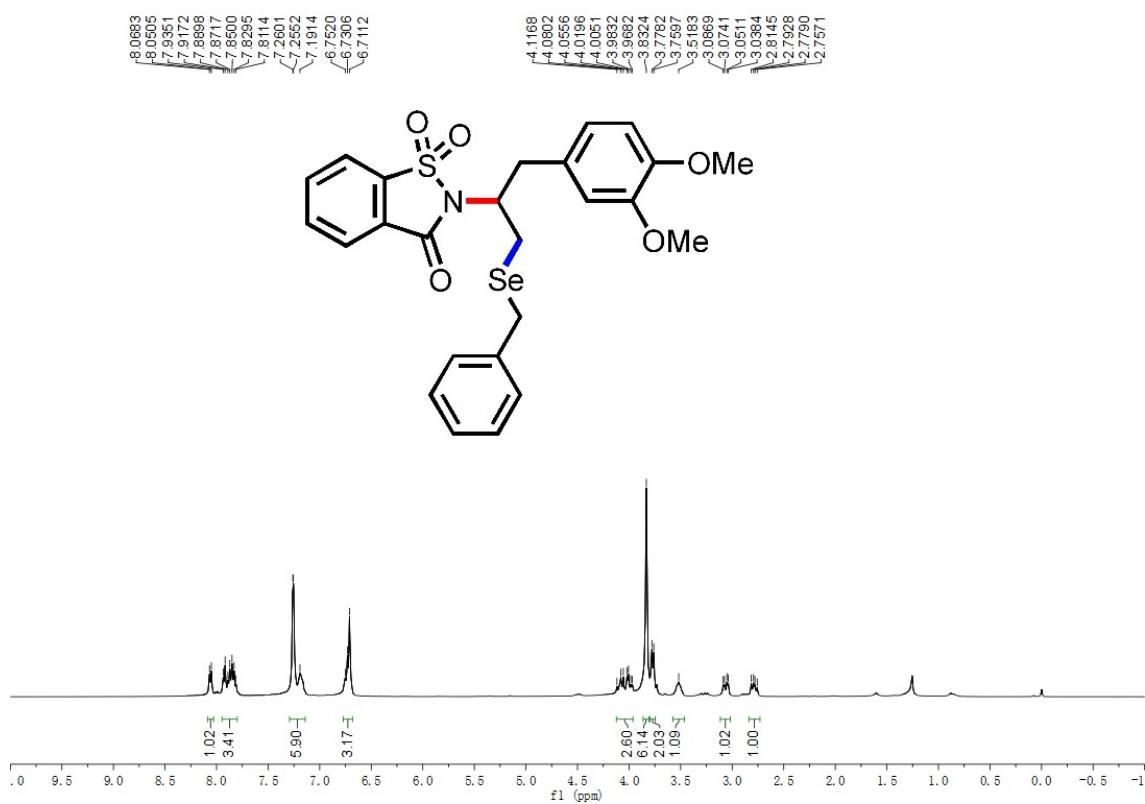
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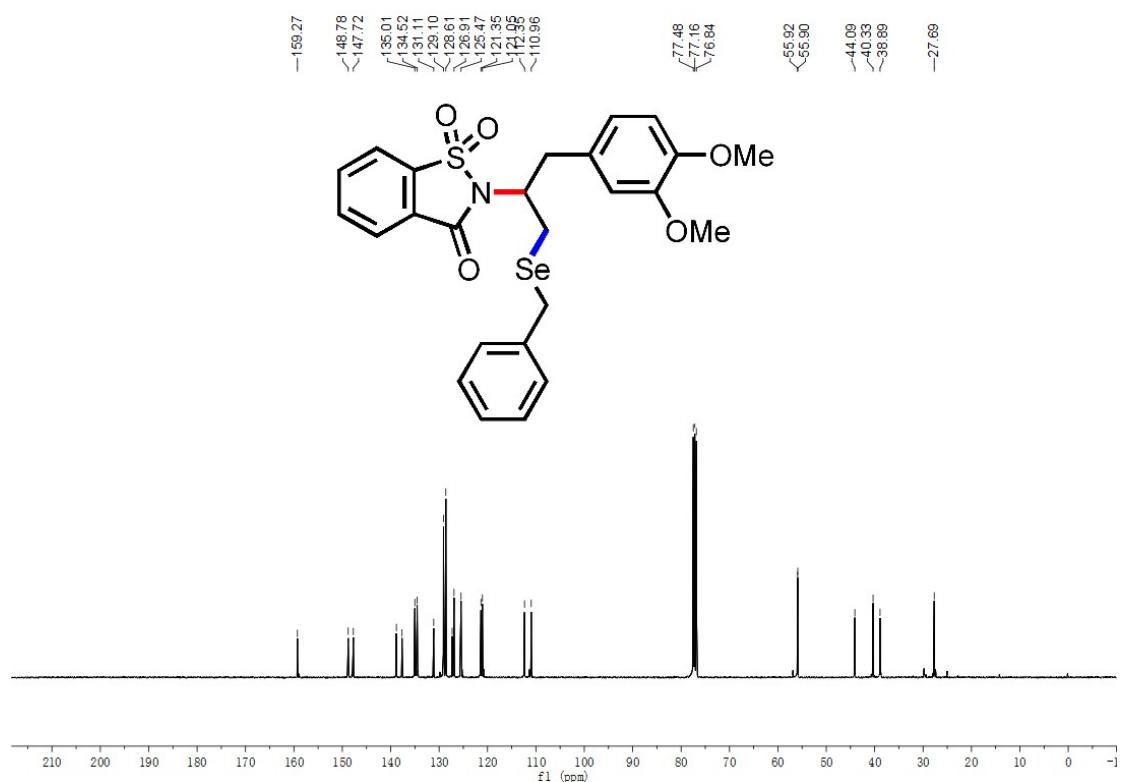
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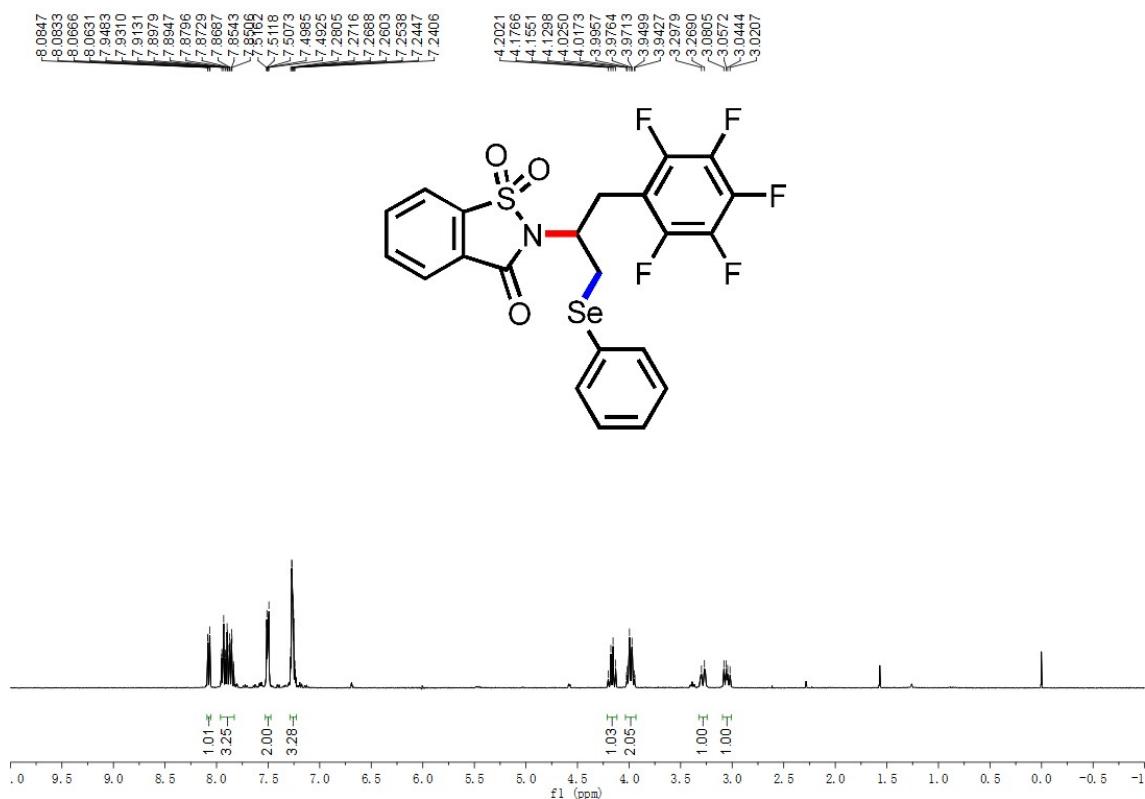
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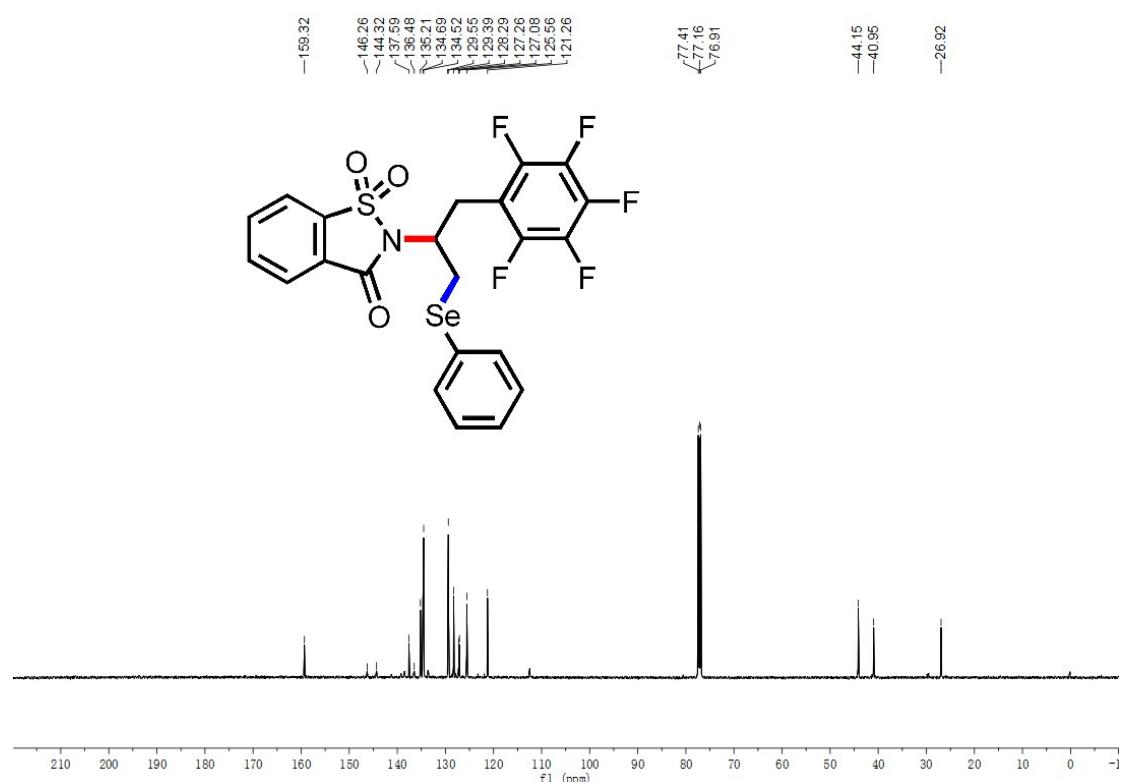
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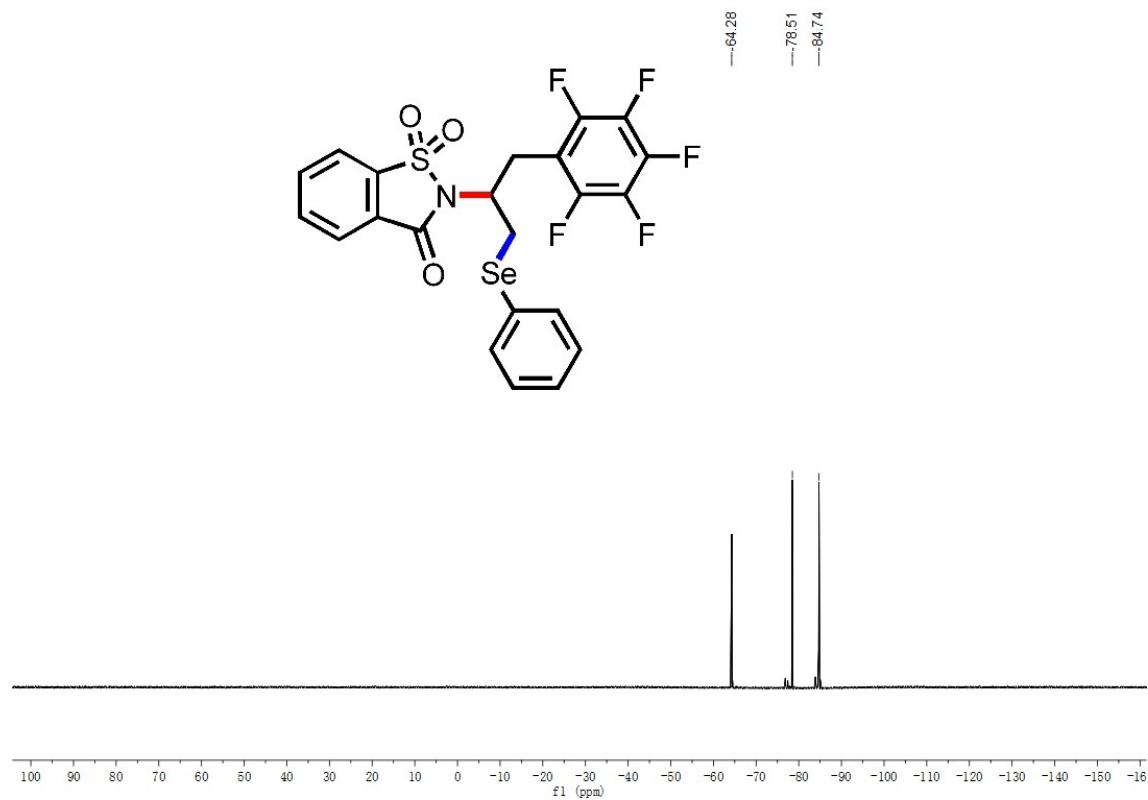
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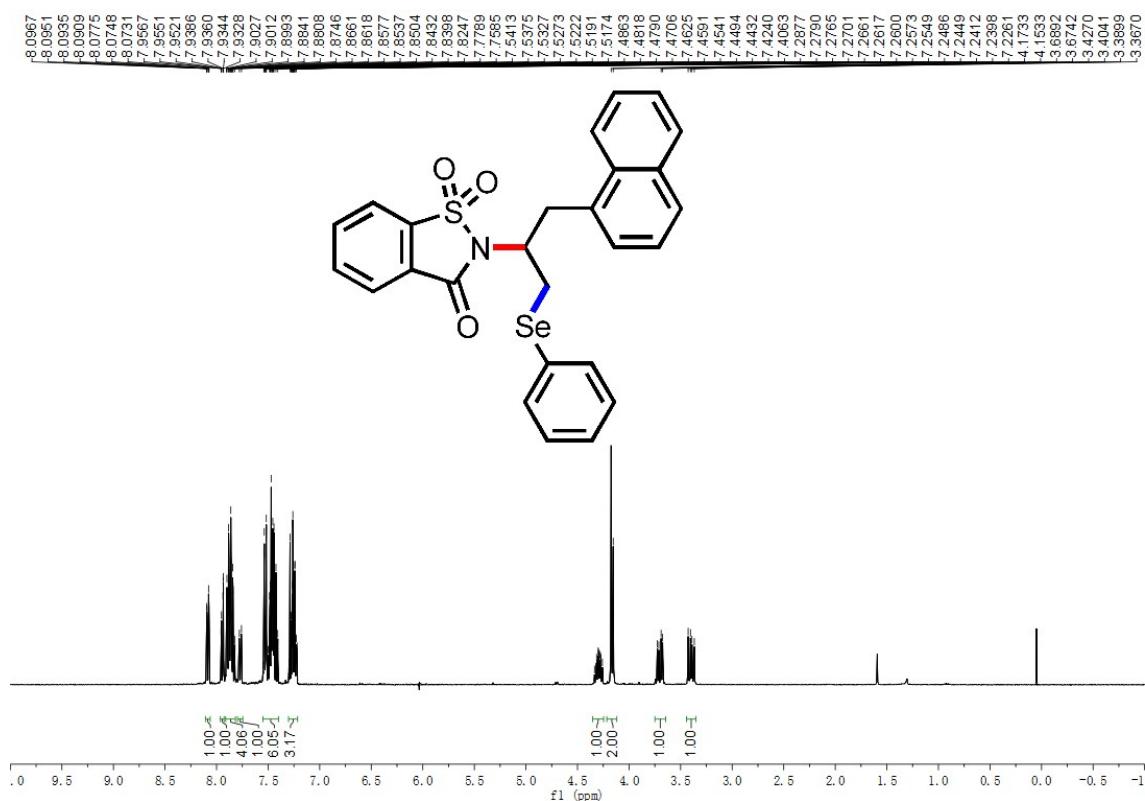
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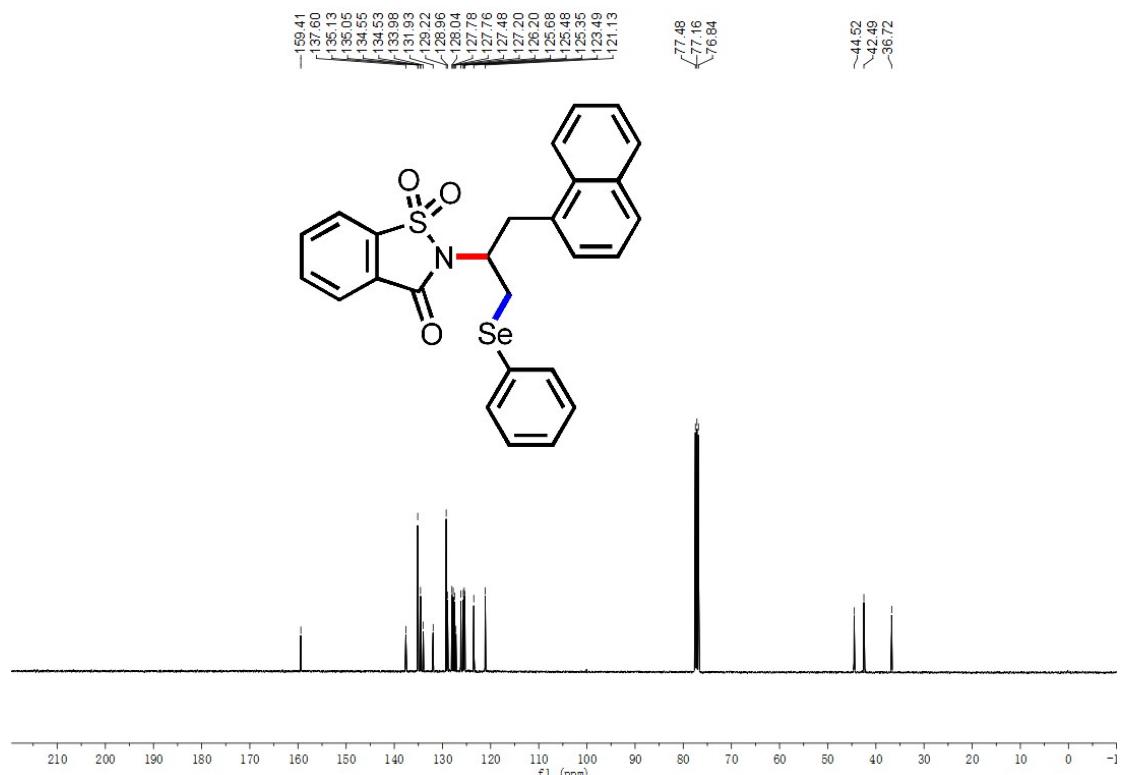
¹⁹F NMR of product 5f in CDCl₃ (376 MHz)



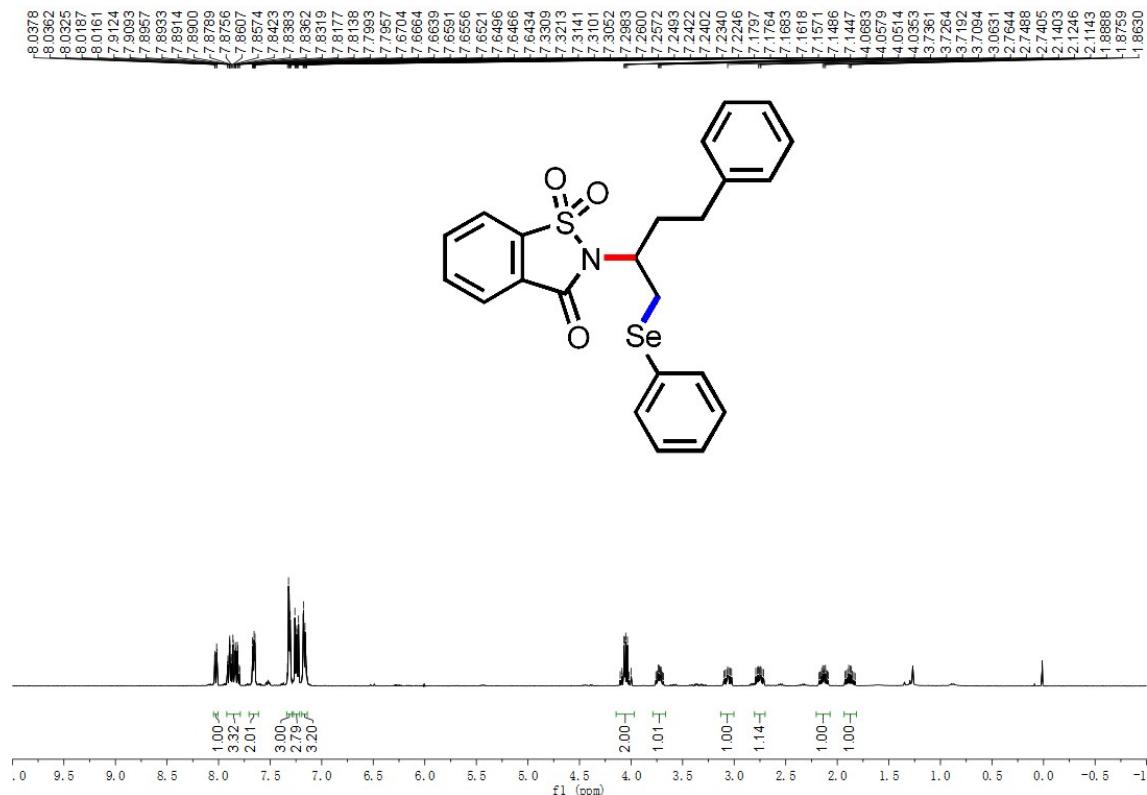
¹H NMR of product 5g in CDCl₃ (400 MHz)



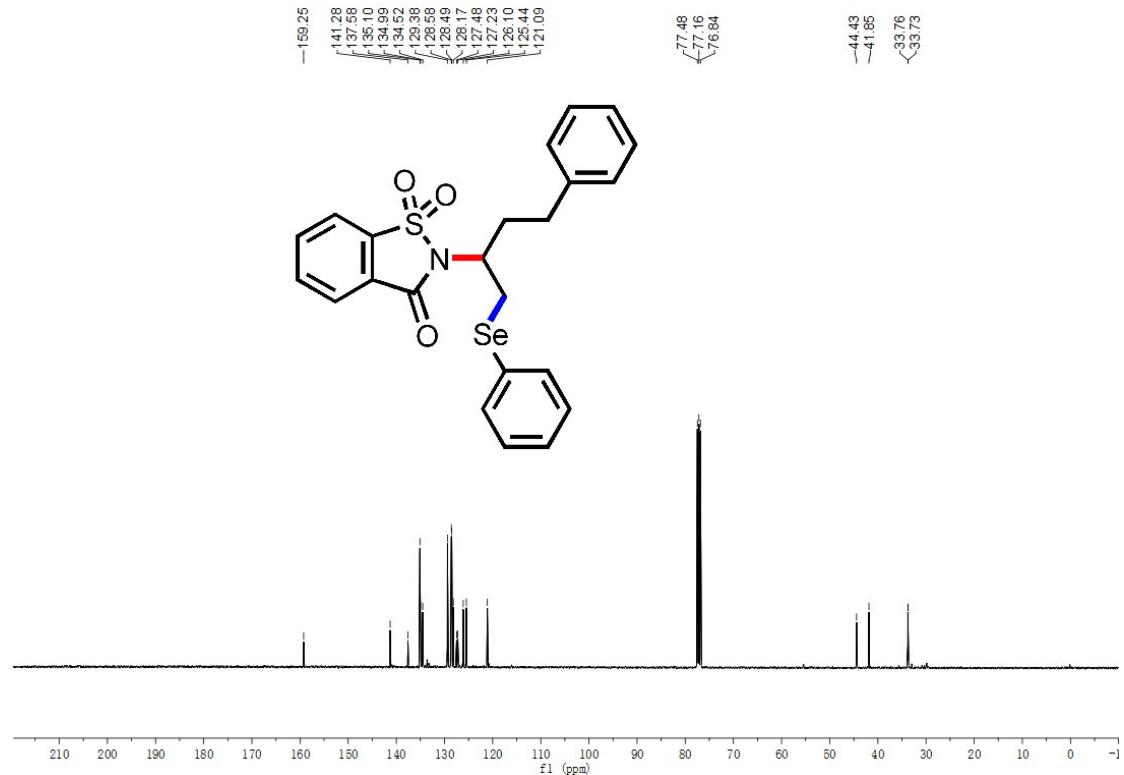
¹³C NMR of product 5g in CDCl₃ (100 MHz)



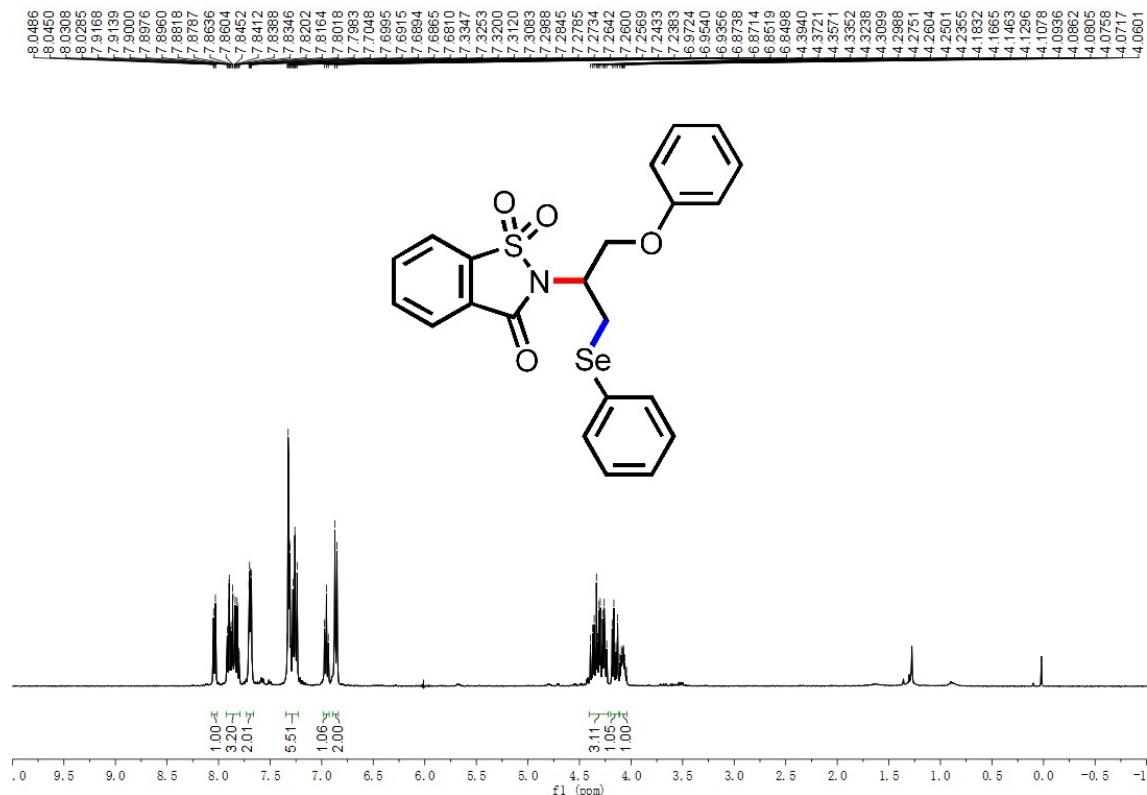
¹H NMR of product 5h in CDCl₃ (400 MHz)



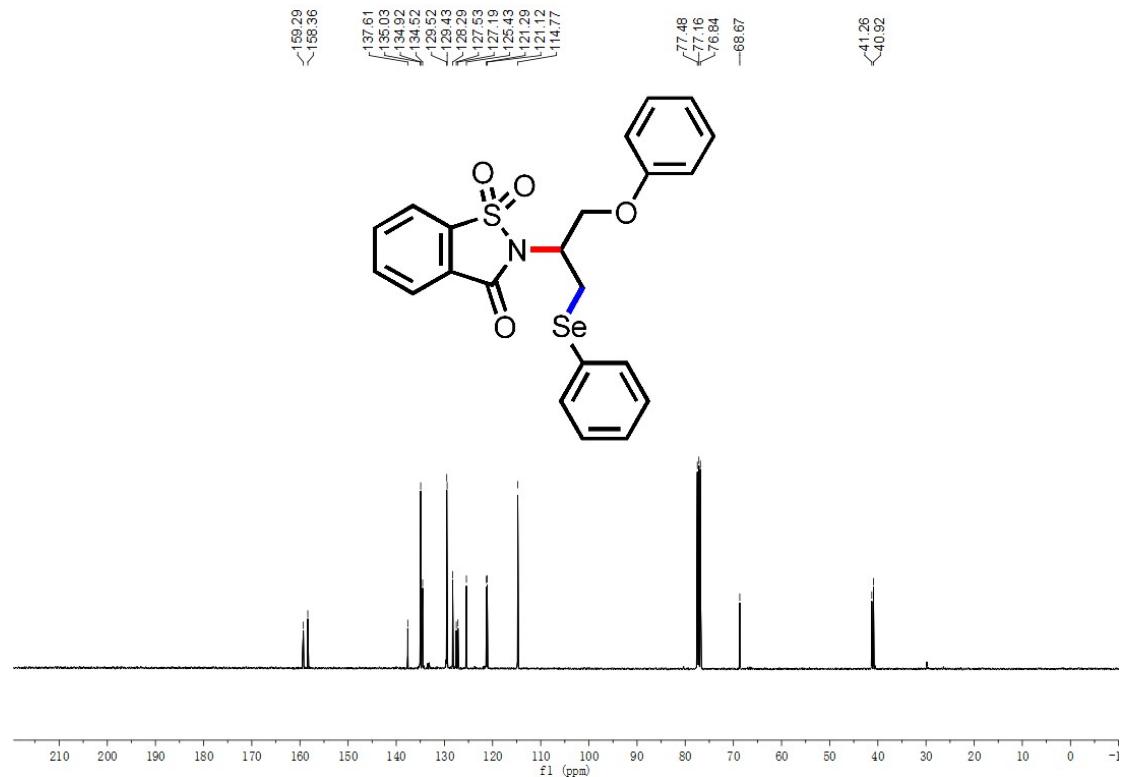
¹³C NMR of product 5h in CDCl₃ (100 MHz)



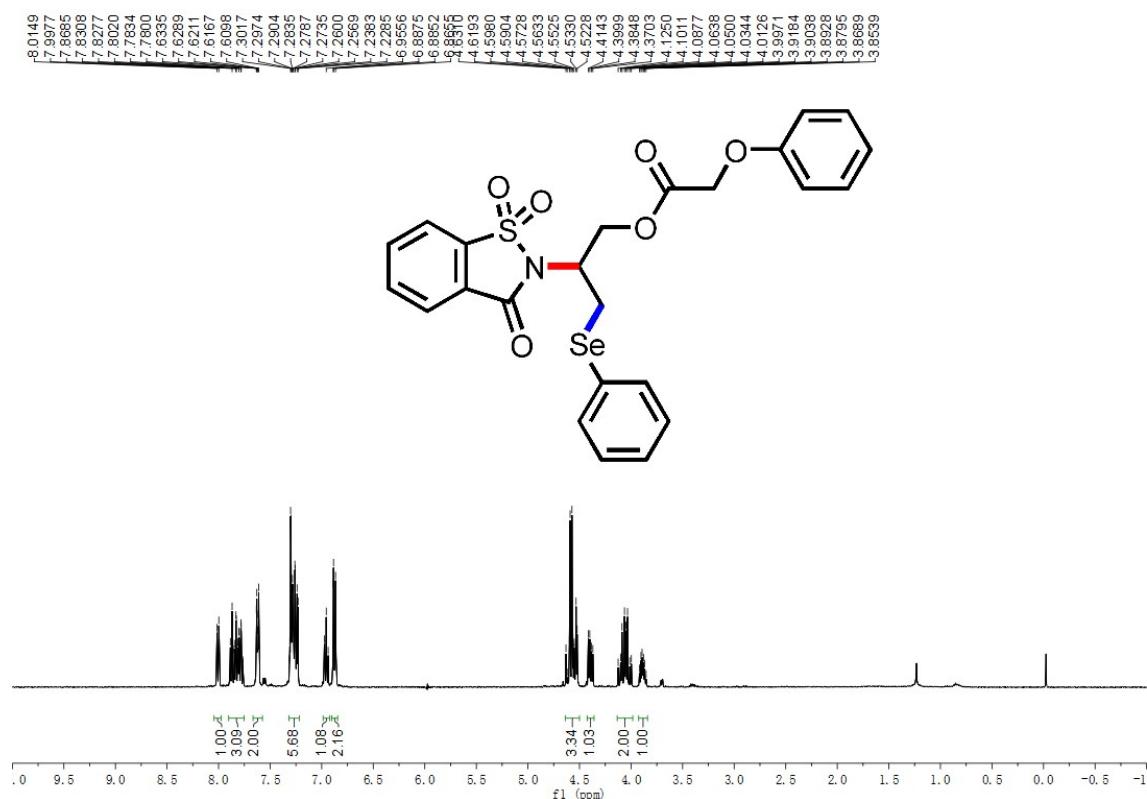
¹H NMR of product 5i in CDCl₃ (400 MHz)



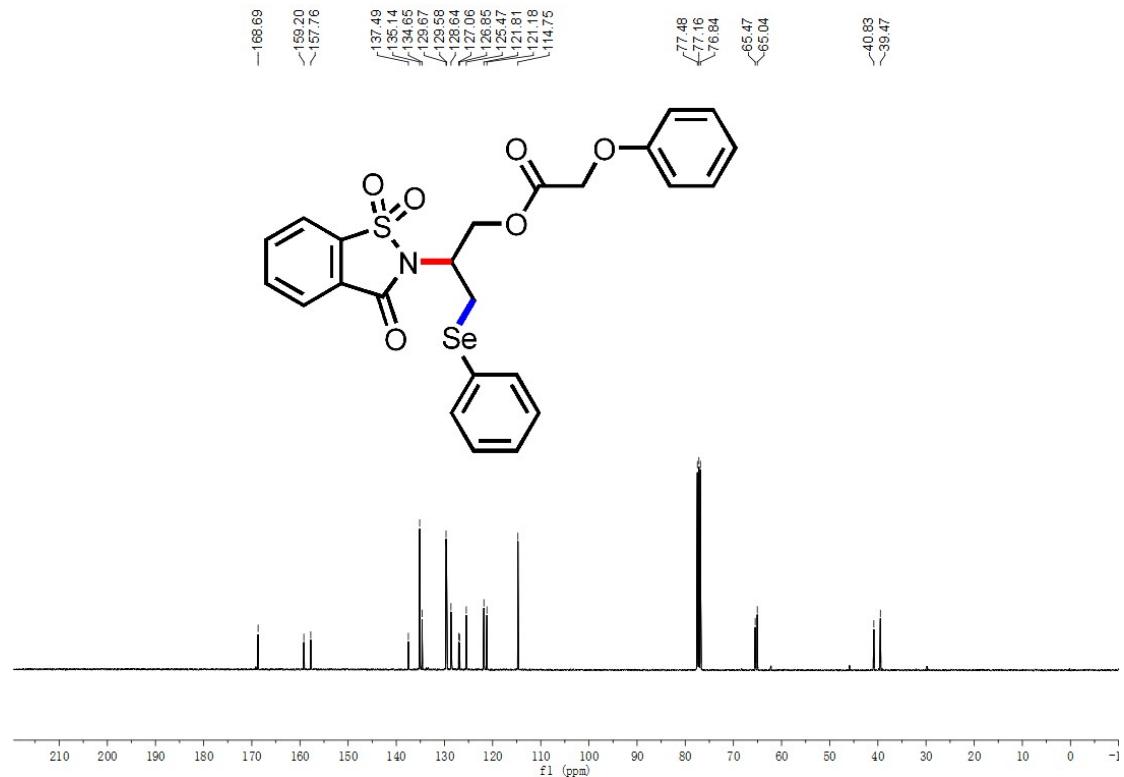
¹³C NMR of product 5i in CDCl₃ (100 MHz)



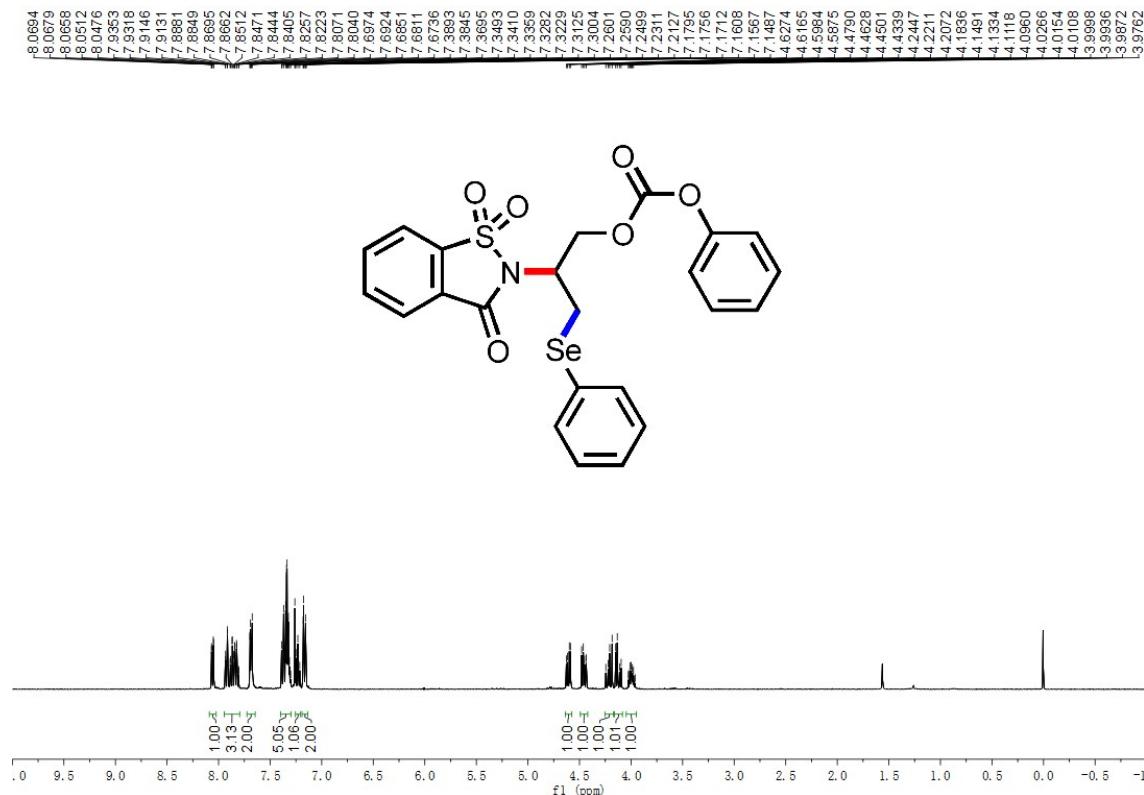
¹H NMR of product 5j in CDCl₃ (400 MHz)



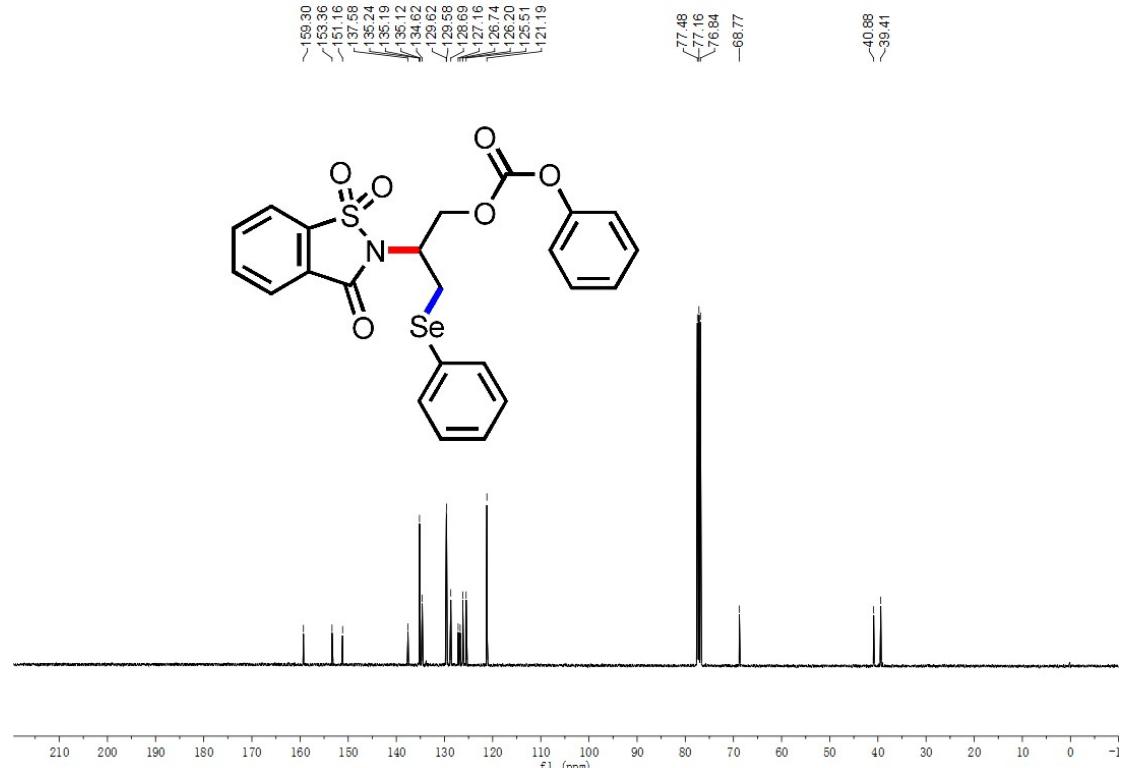
¹³C NMR of product 5j in CDCl₃ (100 MHz)



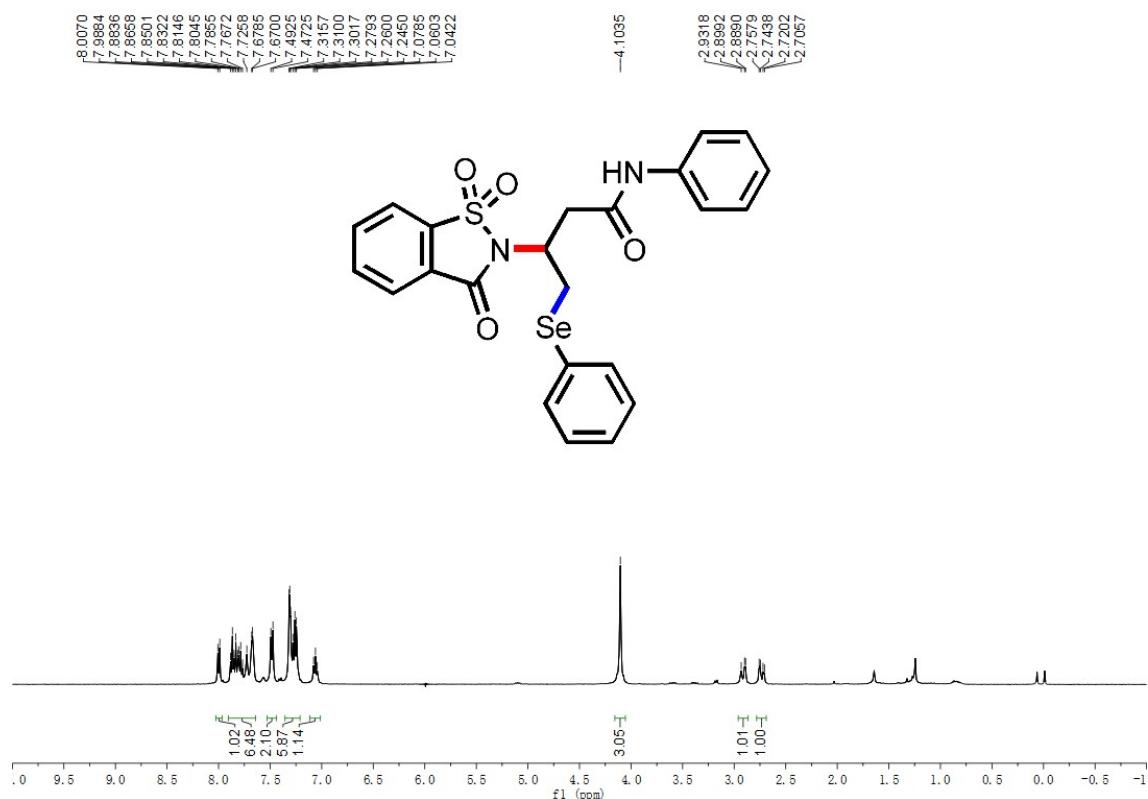
¹H NMR of product 5k in CDCl₃ (400 MHz)



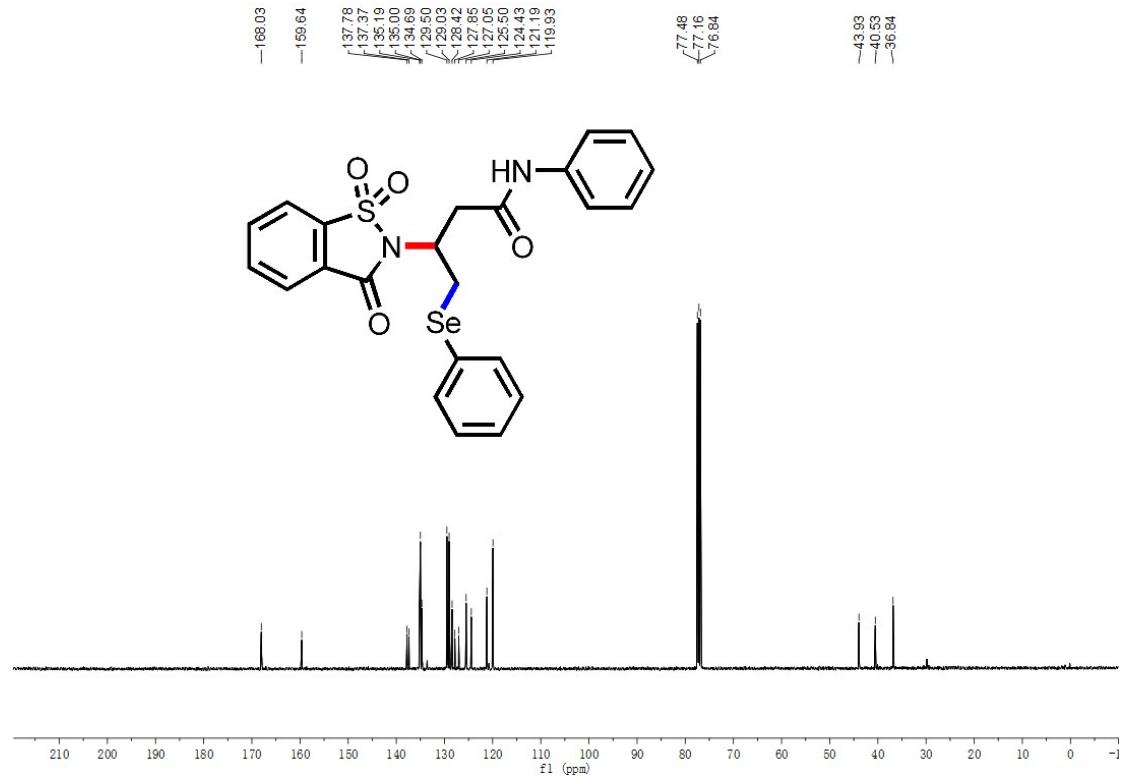
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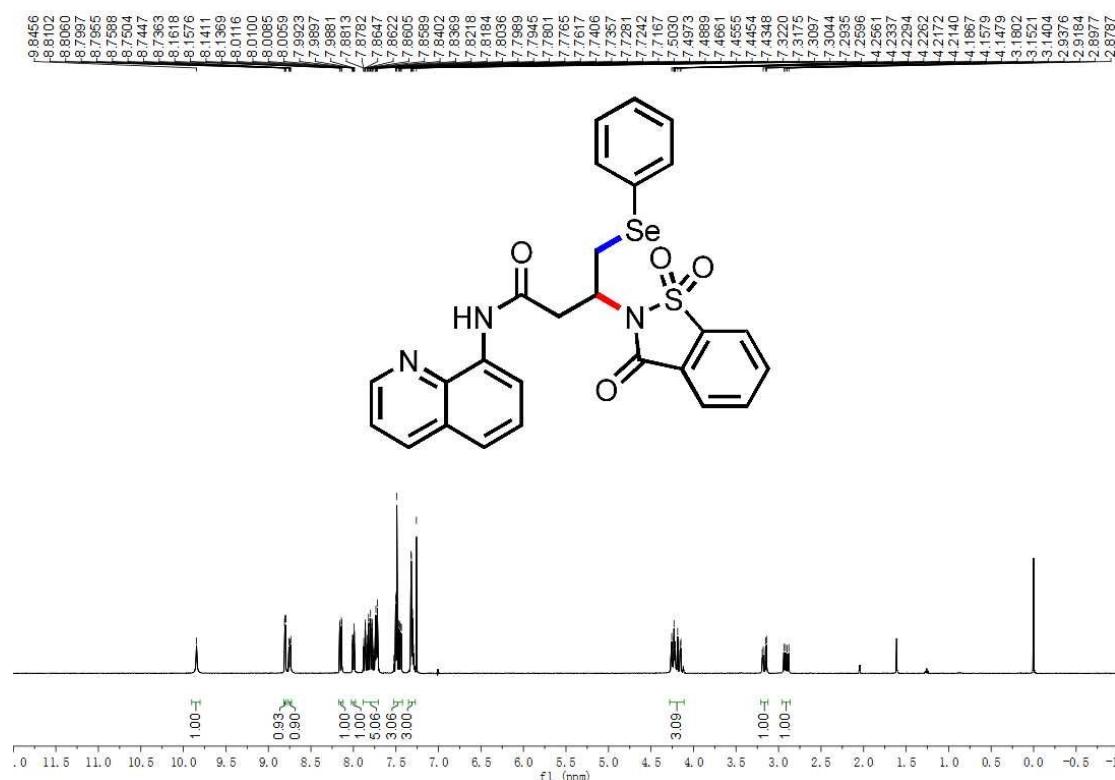
¹H NMR of product 5l in CDCl₃ (400 MHz)



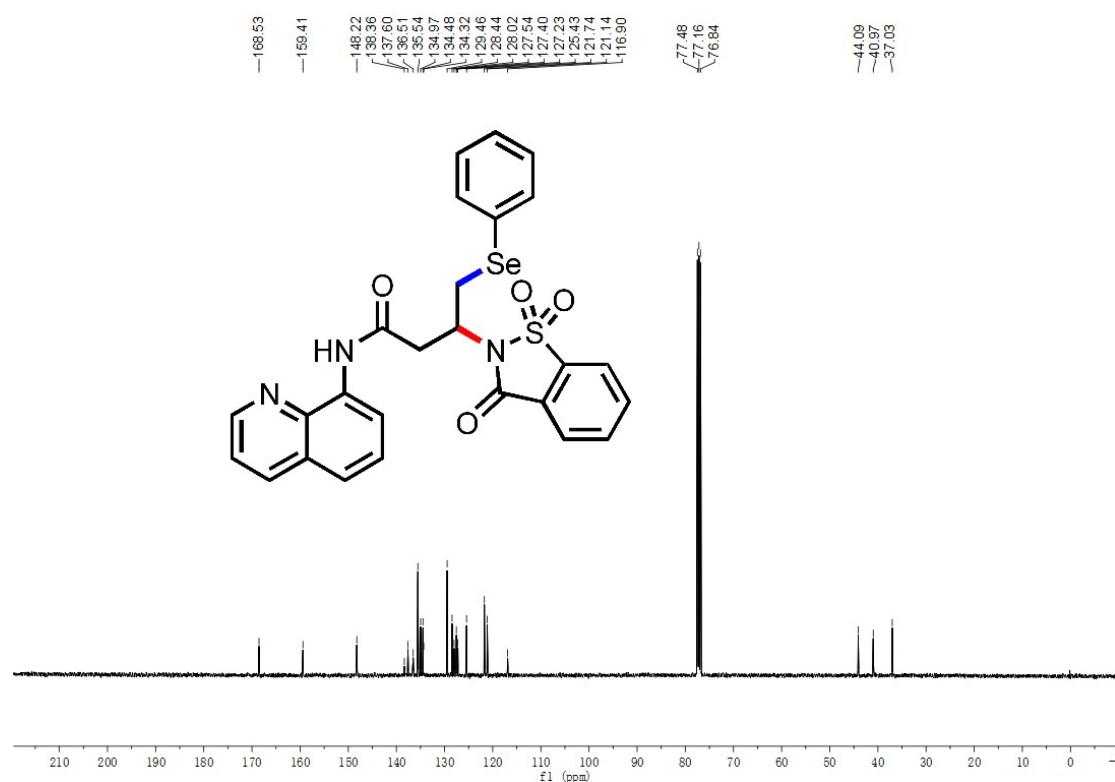
¹³C NMR of product 5l in CDCl₃ (100 MHz)



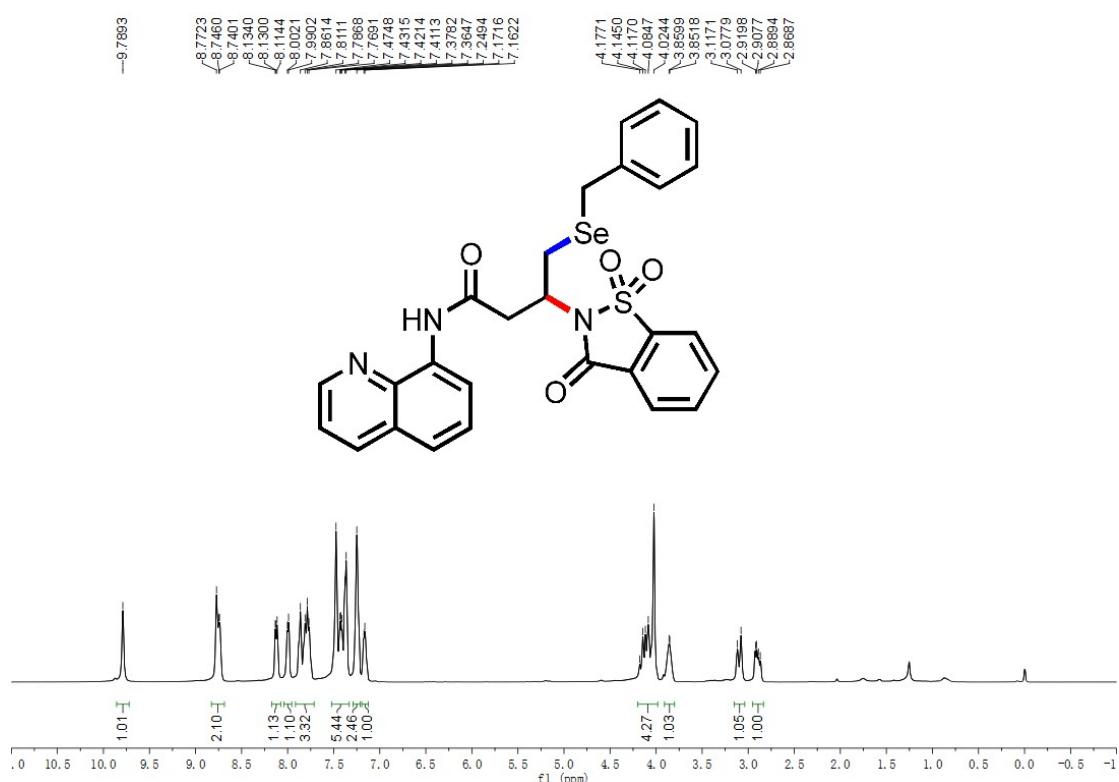
¹H NMR of product 5m in CDCl₃ (400 MHz)



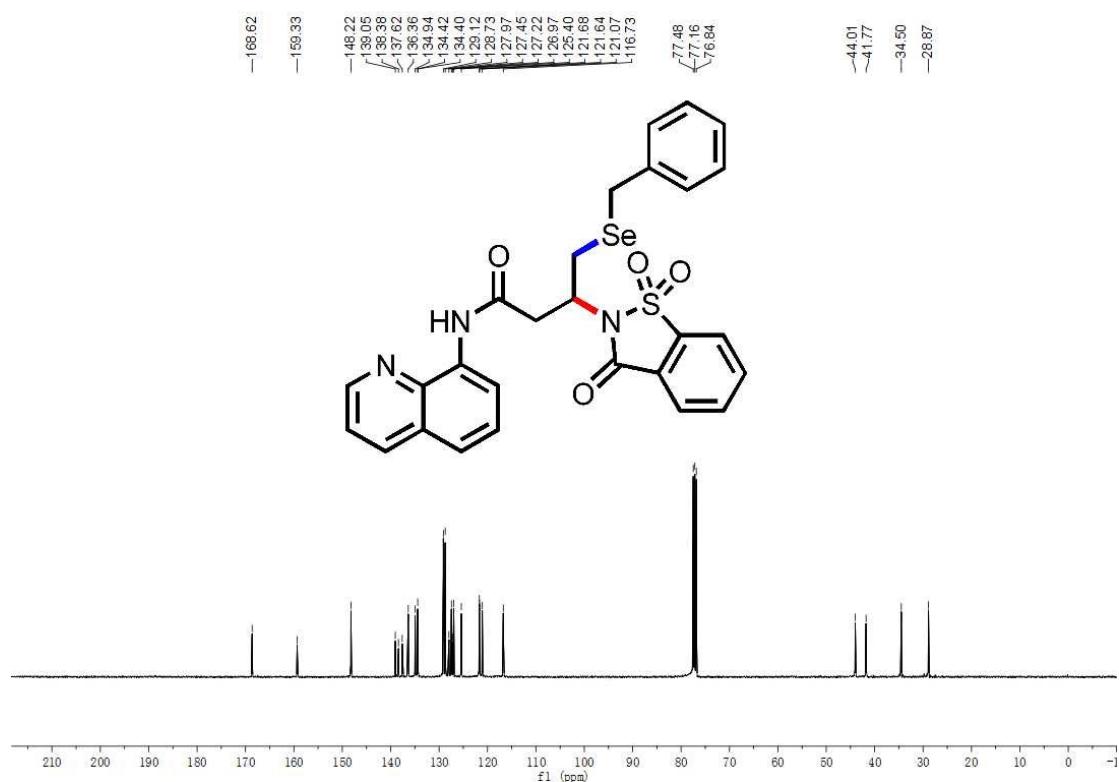
¹³C NMR of product 5m in CDCl₃ (100 MHz)



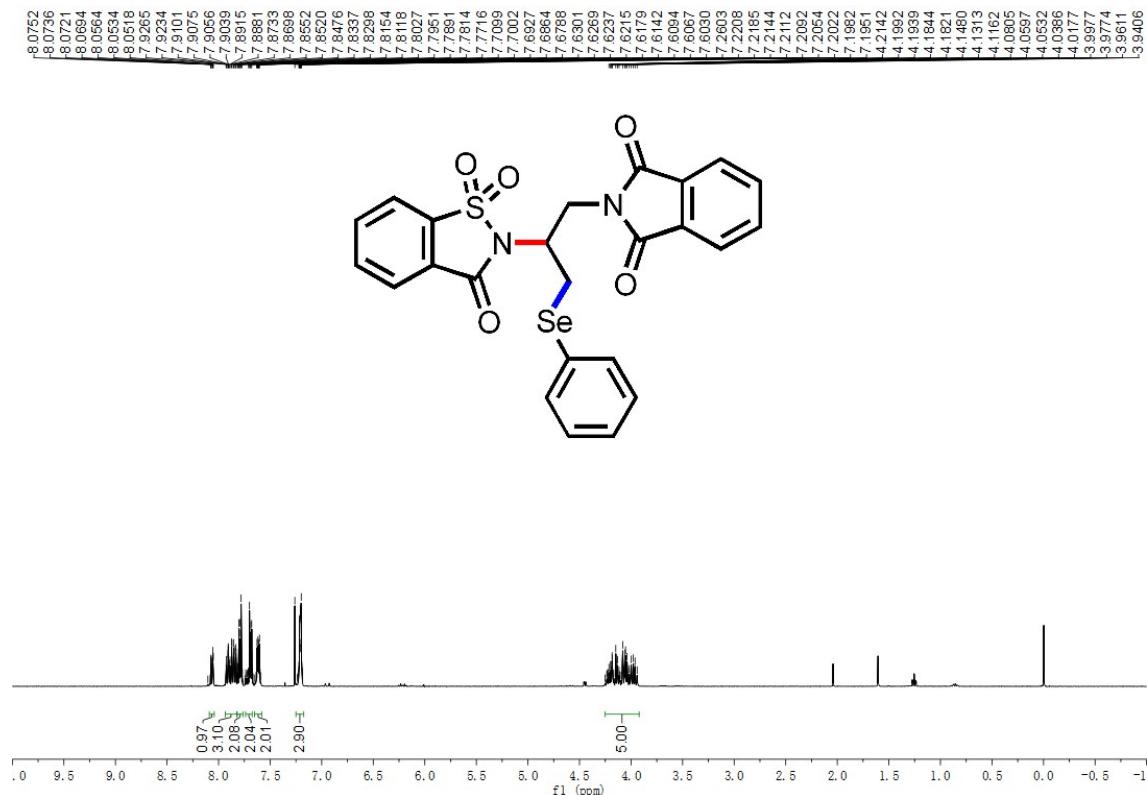
¹H NMR of product 5n in CDCl₃ (400 MHz)



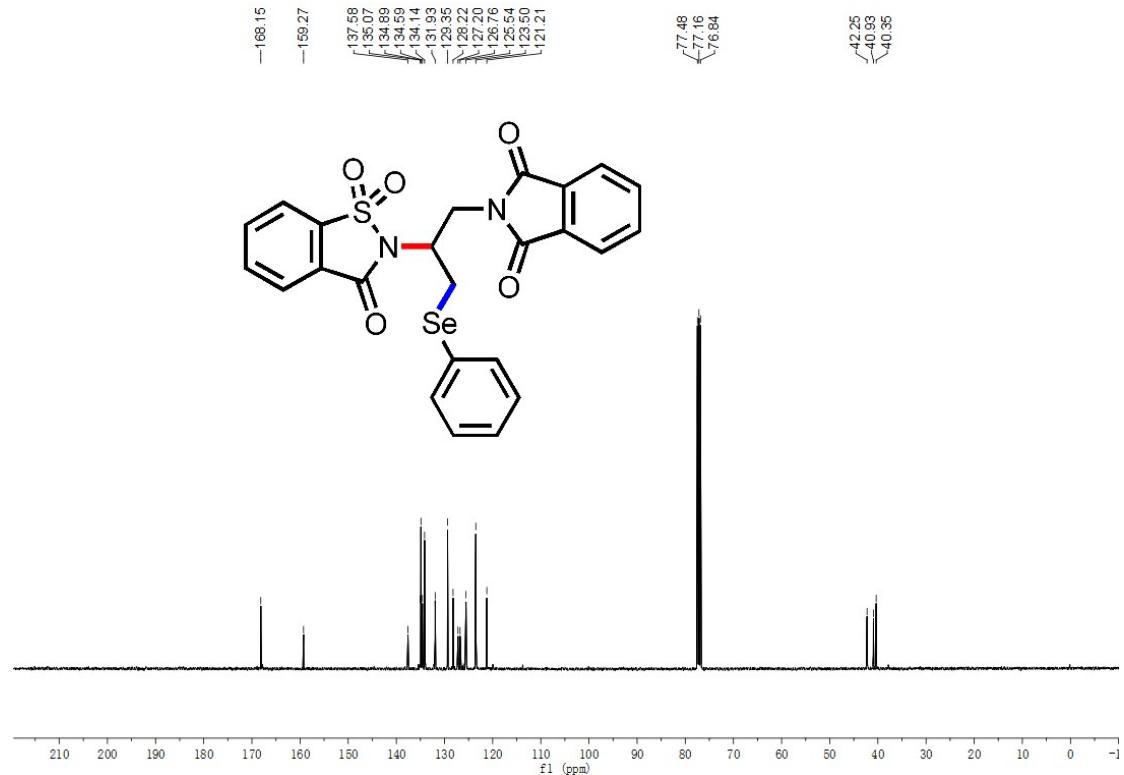
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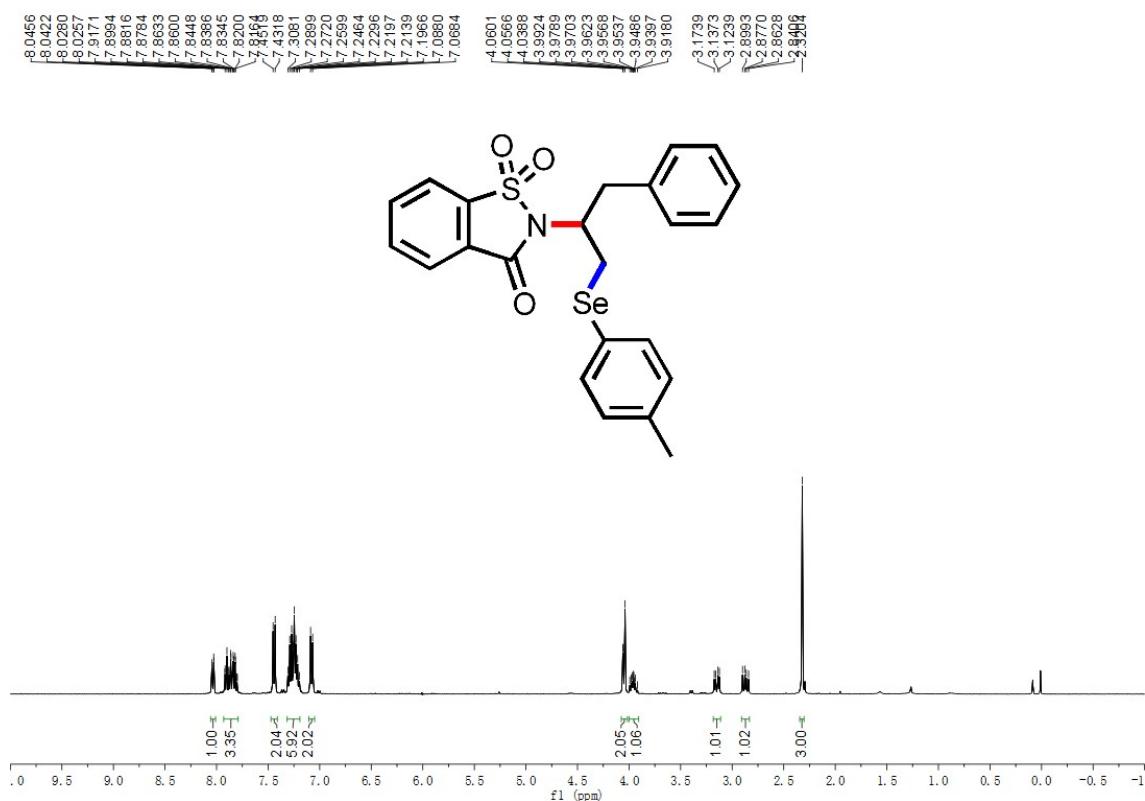
¹H NMR of product 5o in CDCl₃ (400 MHz)



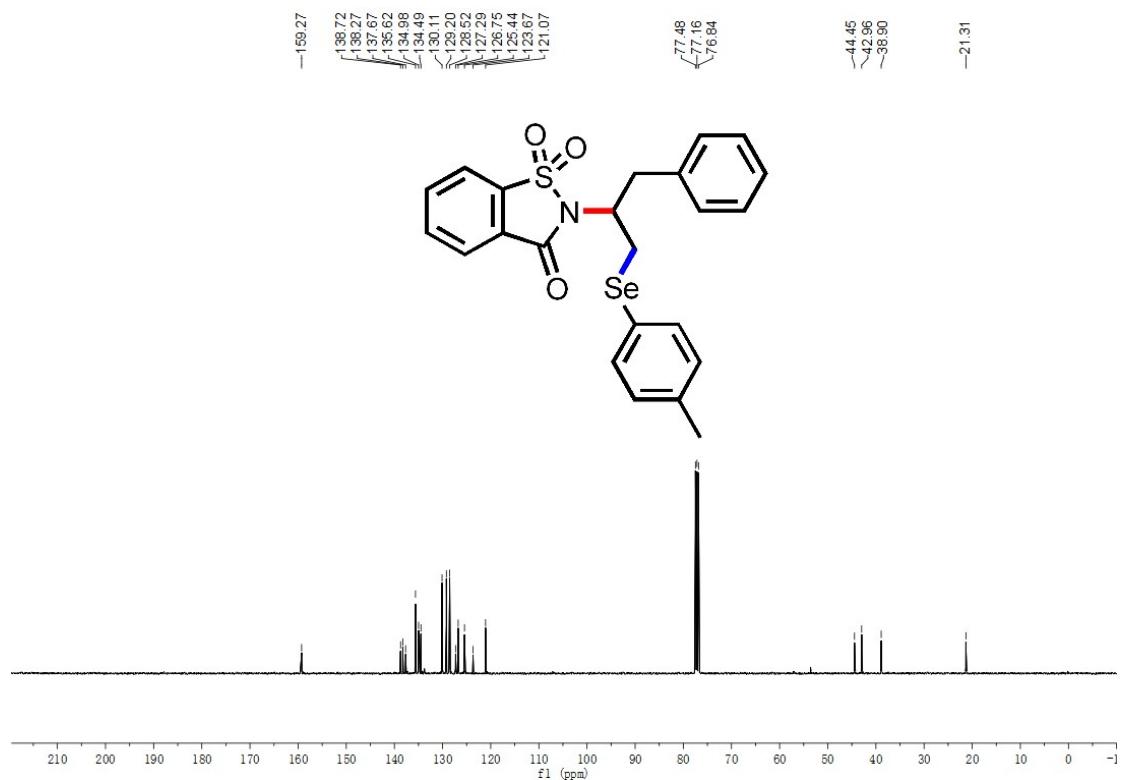
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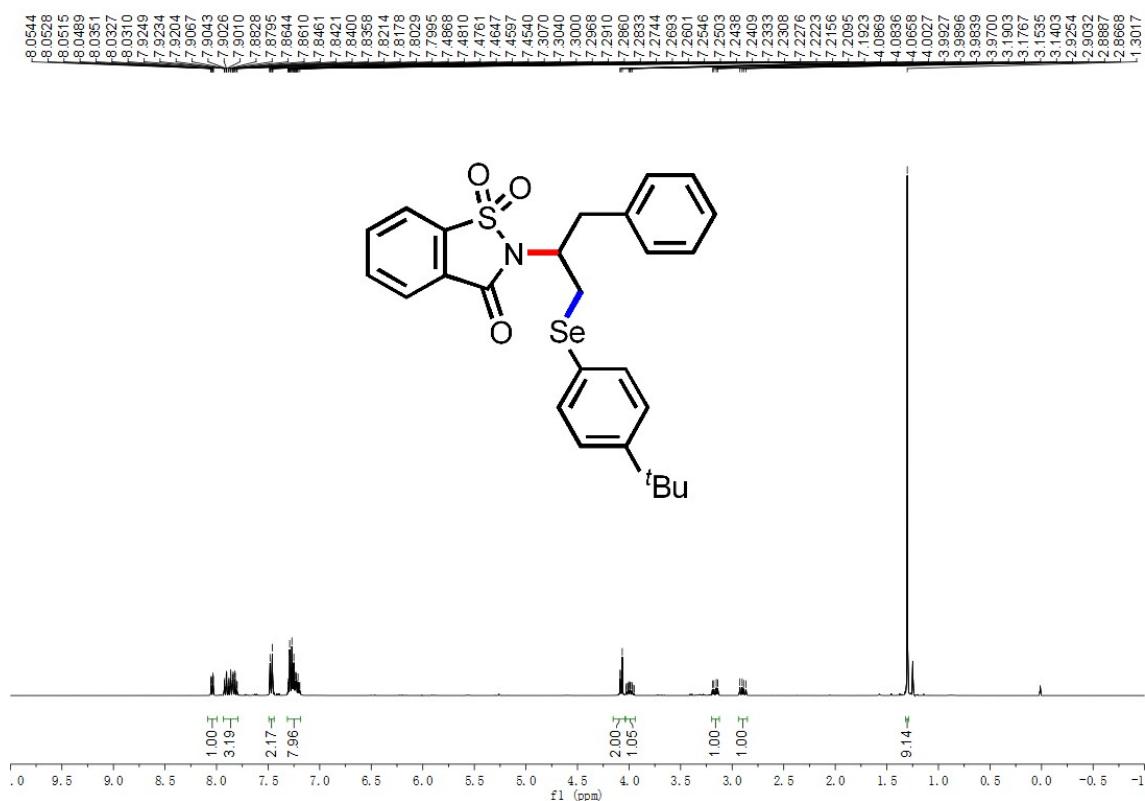
¹H NMR of product 5p in CDCl₃ (400 MHz)



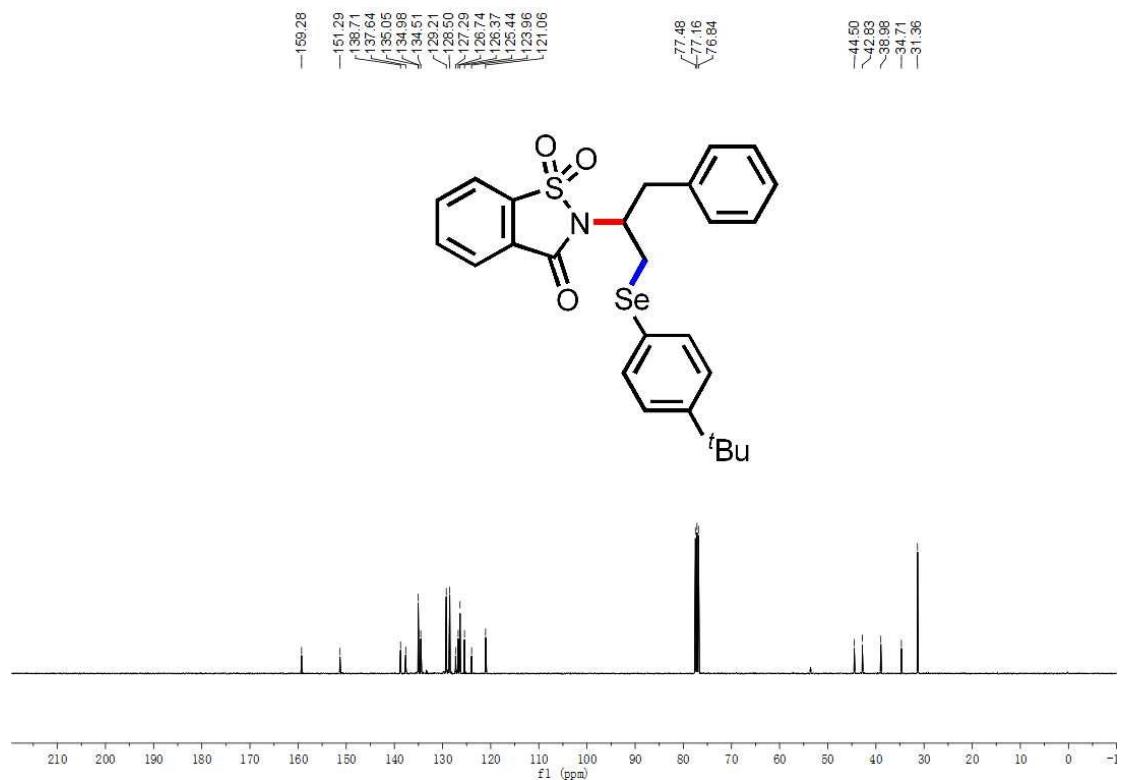
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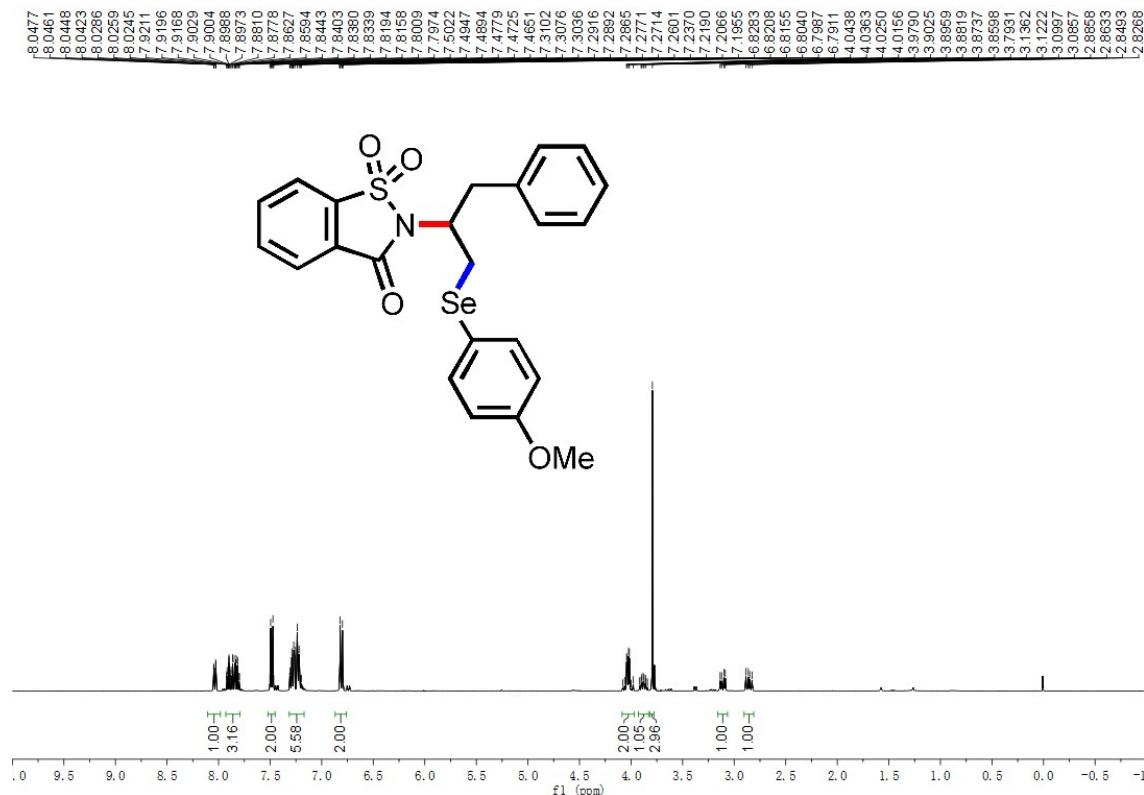
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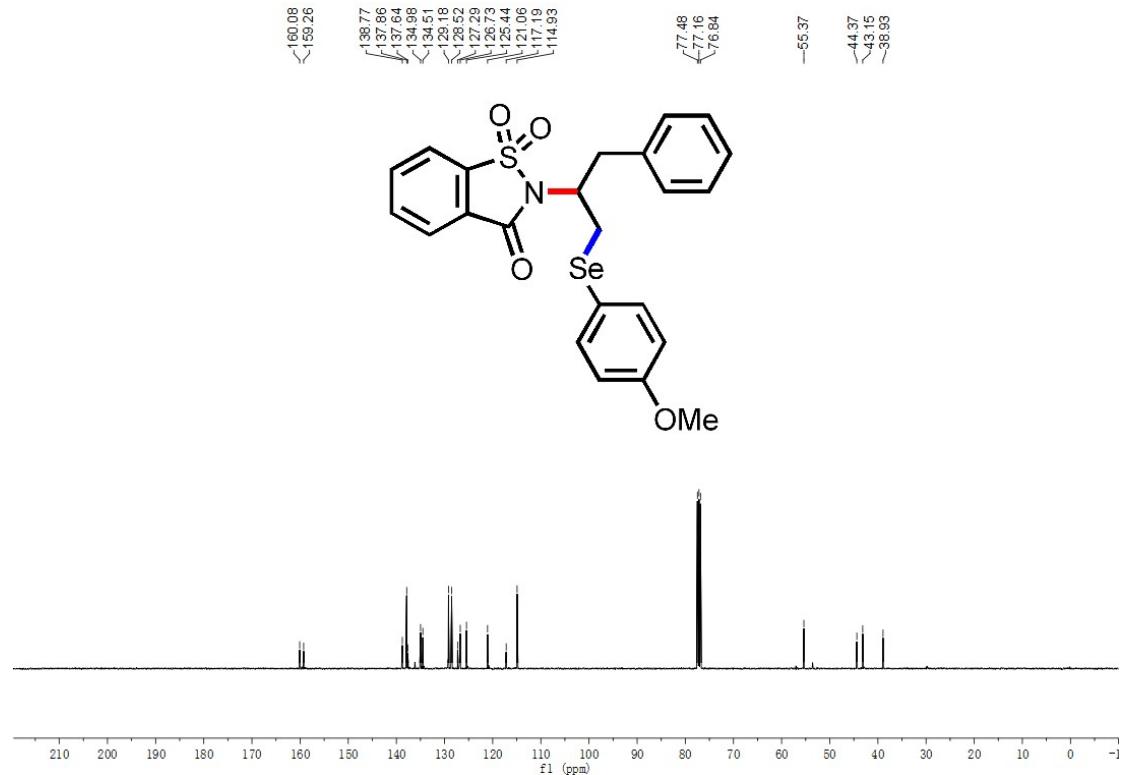
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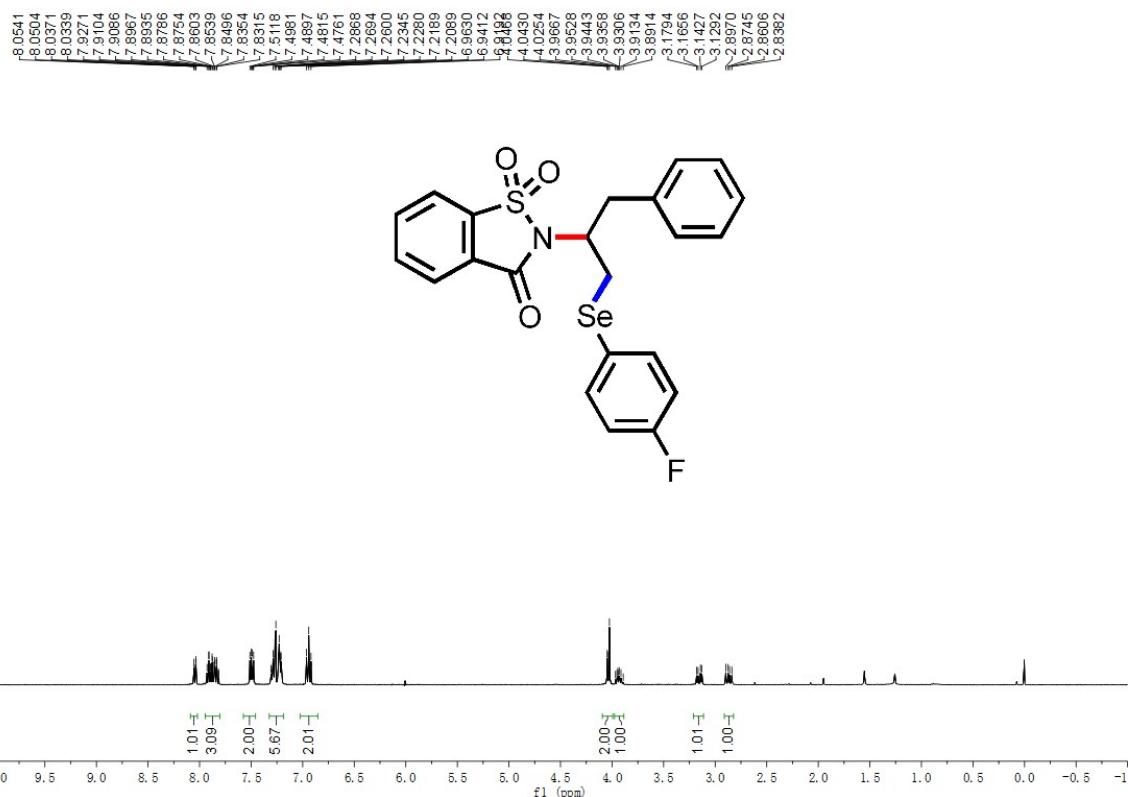
¹H NMR of product 5r in CDCl₃ (400 MHz)



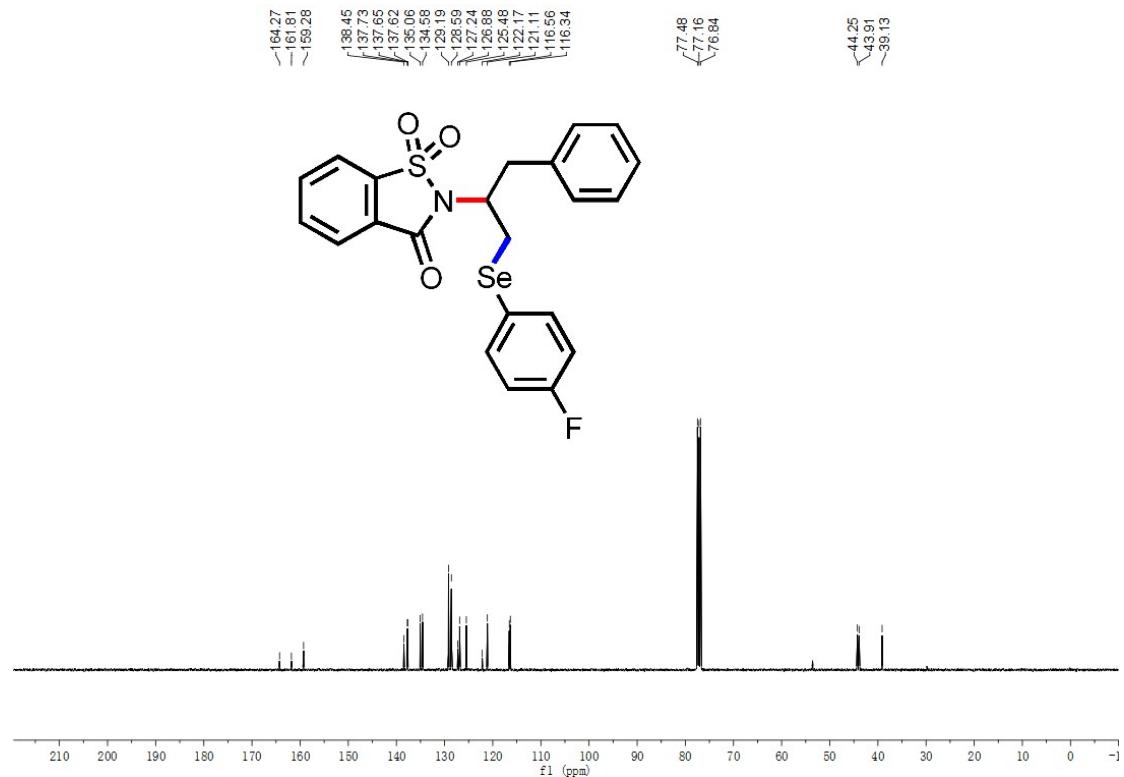
¹³C NMR of product 5r in CDCl₃ (100 MHz)



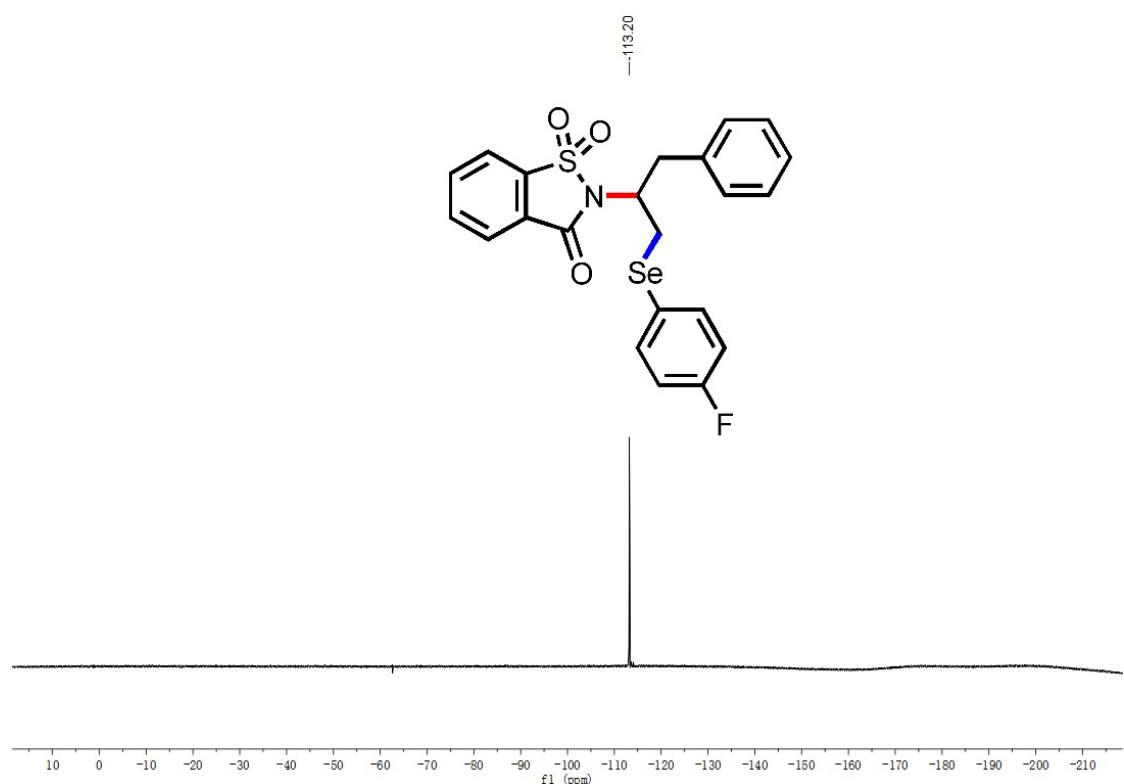
¹H NMR of product 5s in CDCl₃ (400 MHz)



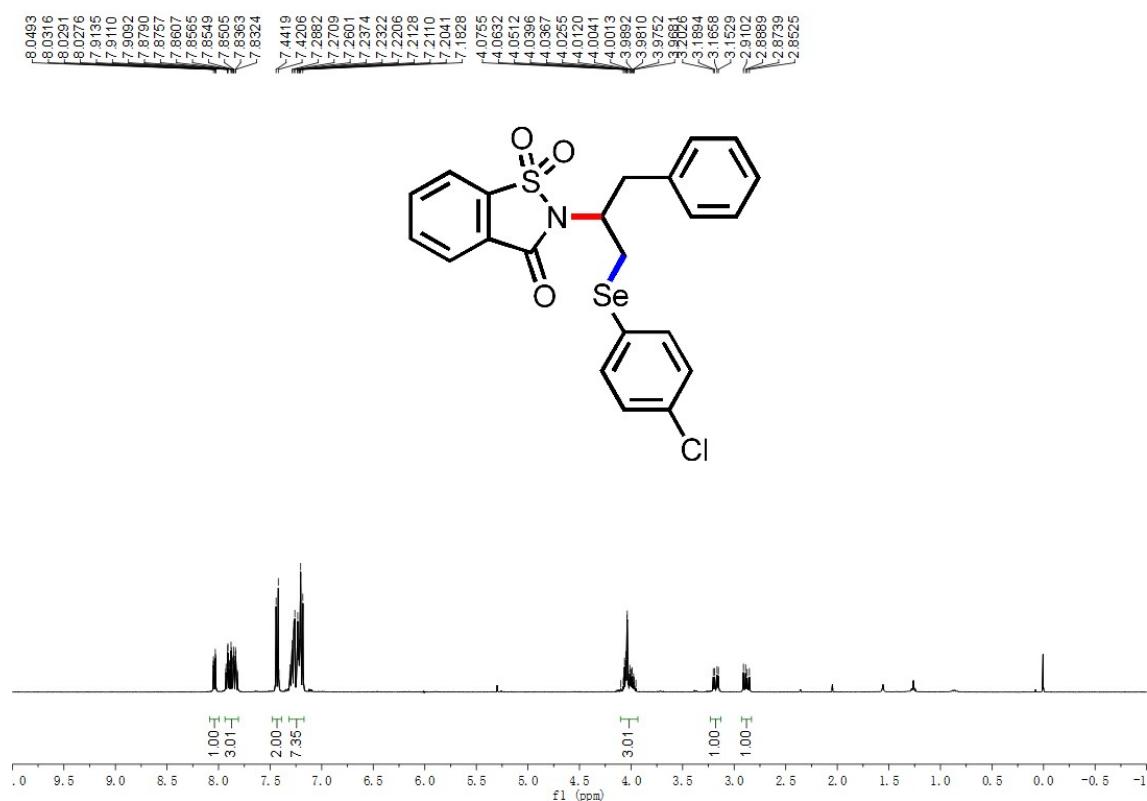
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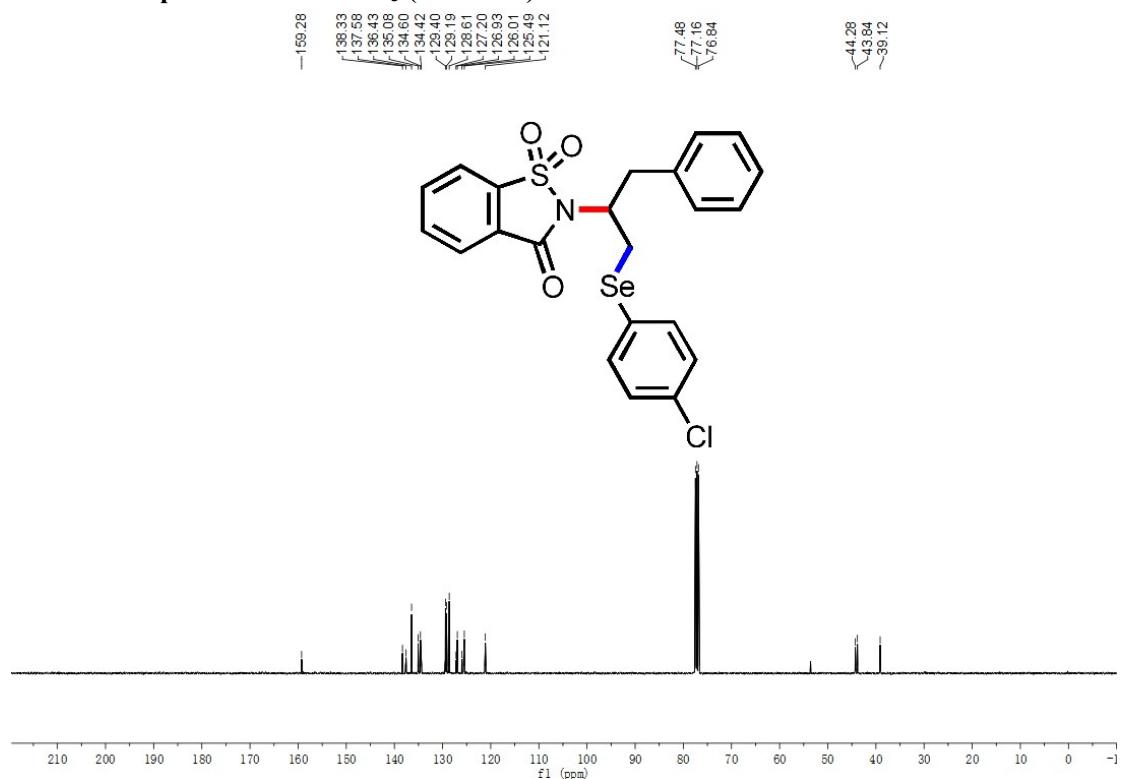
¹⁹F NMR of product 5s in CDCl₃ (376 MHz)



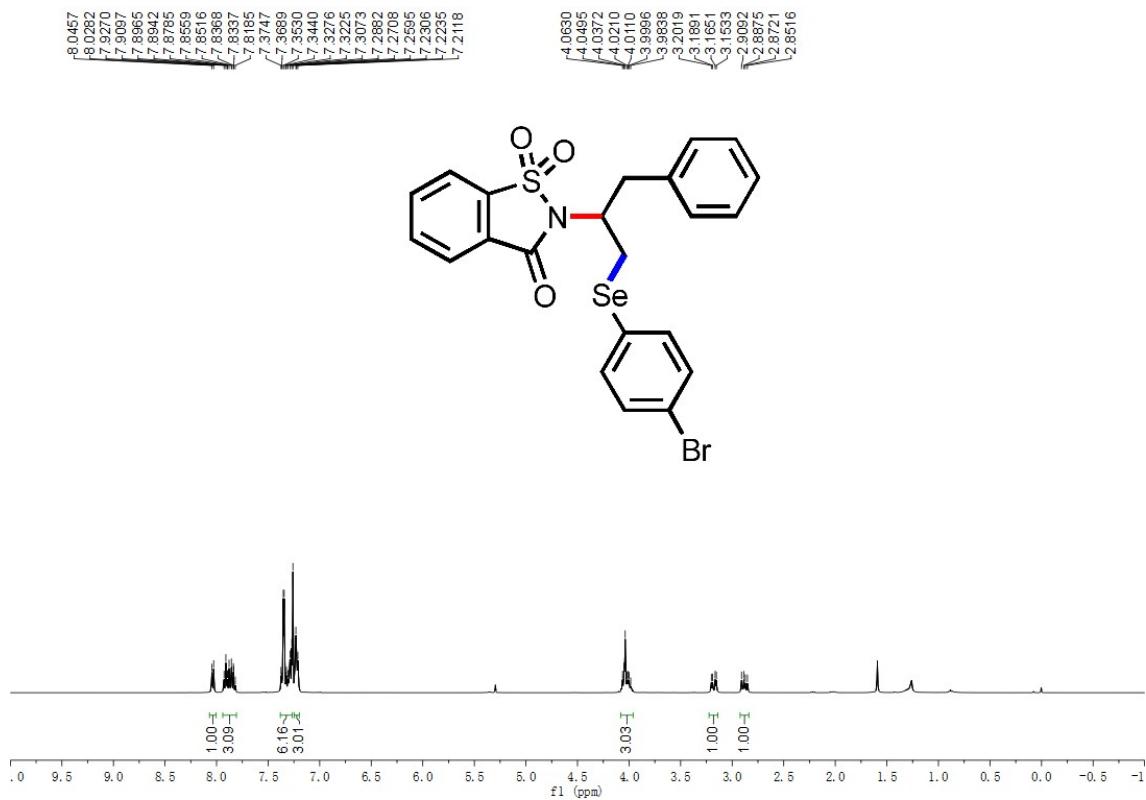
¹H NMR of product 5t in CDCl₃ (400 MHz)



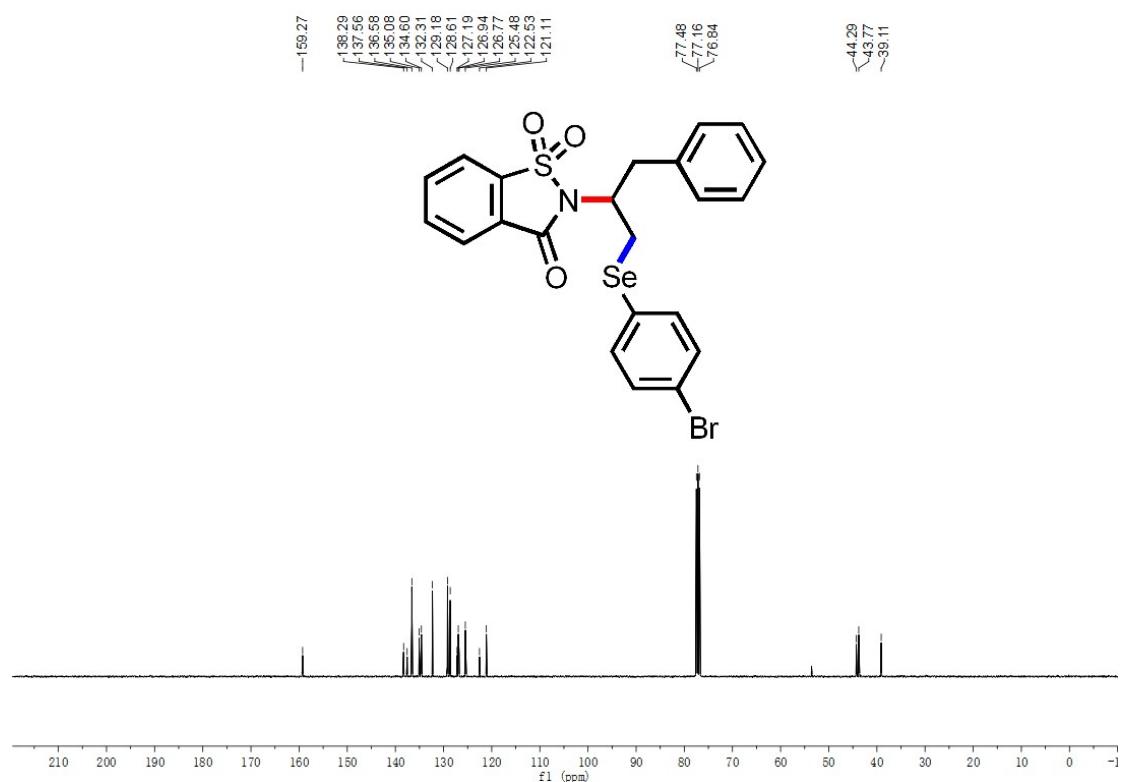
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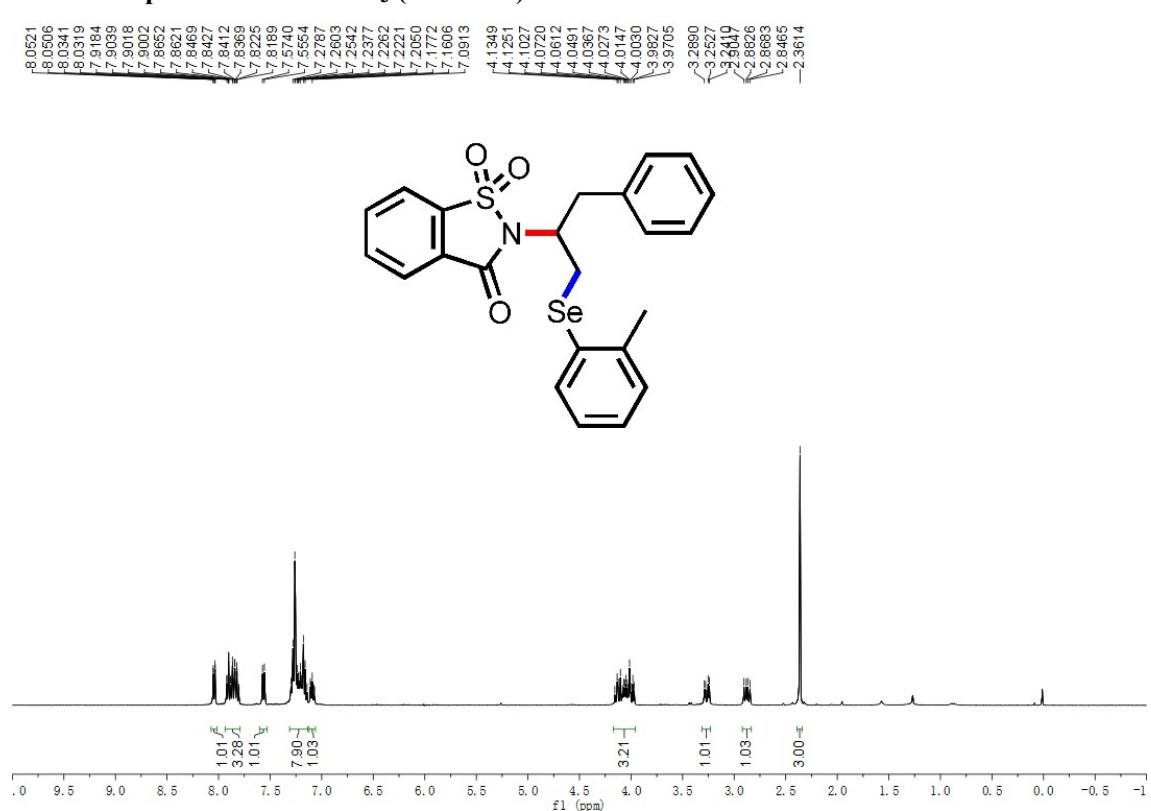
¹H NMR of product 5u in CDCl₃ (400 MHz)



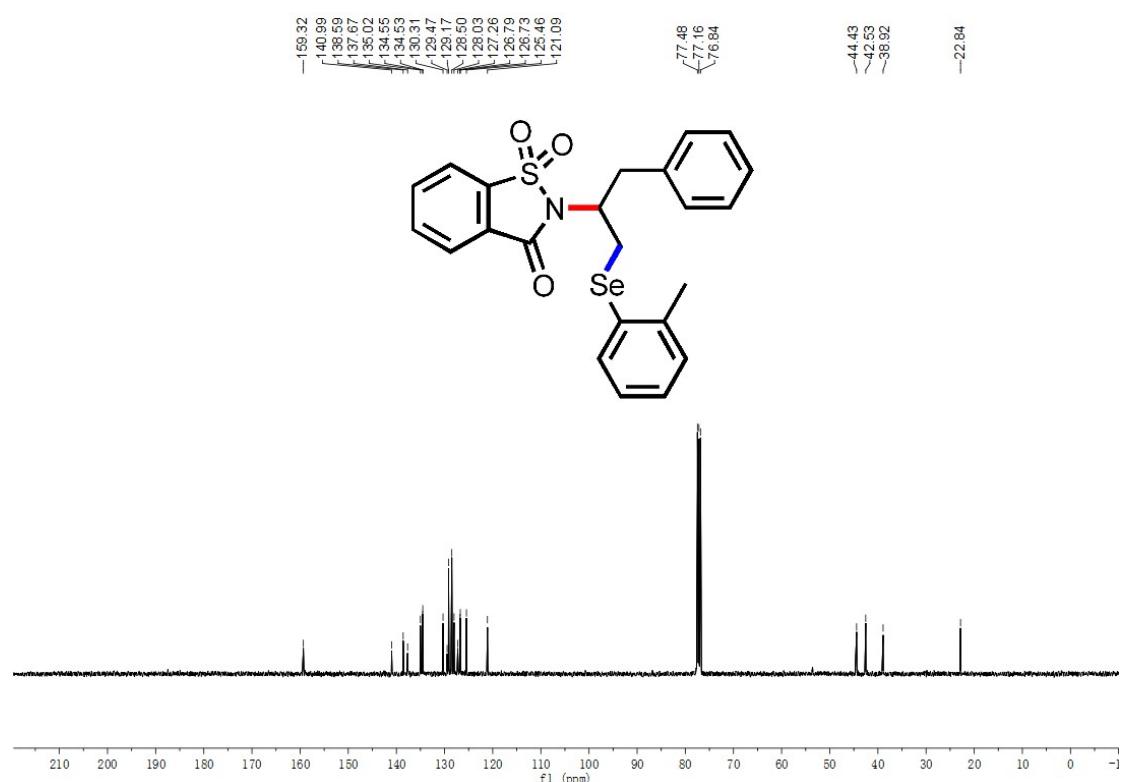
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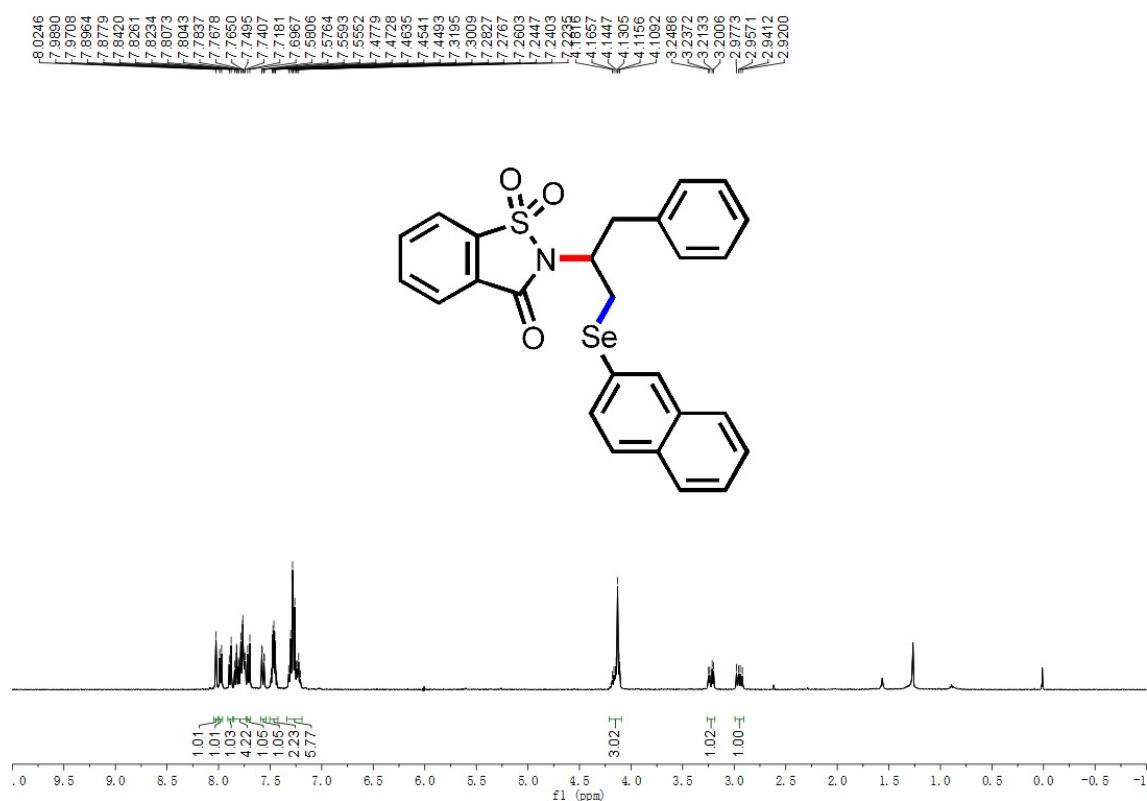
H NMR of product 5v in CDCl₃ (400 MHz)



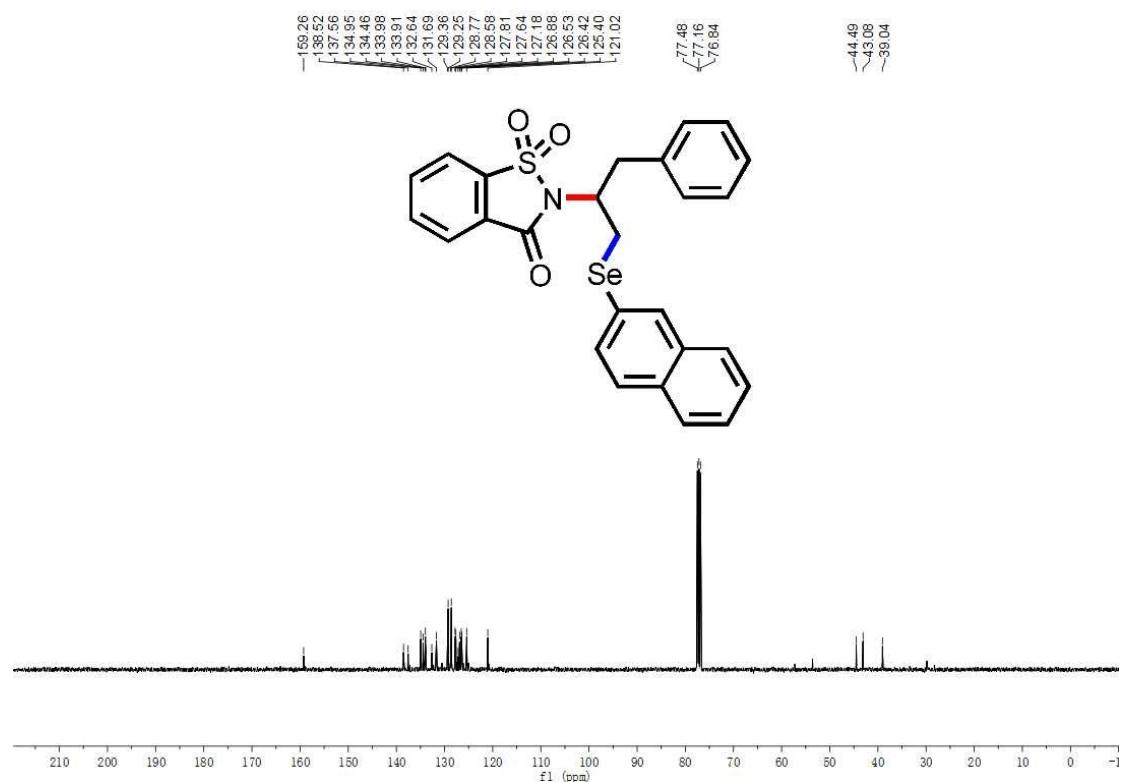
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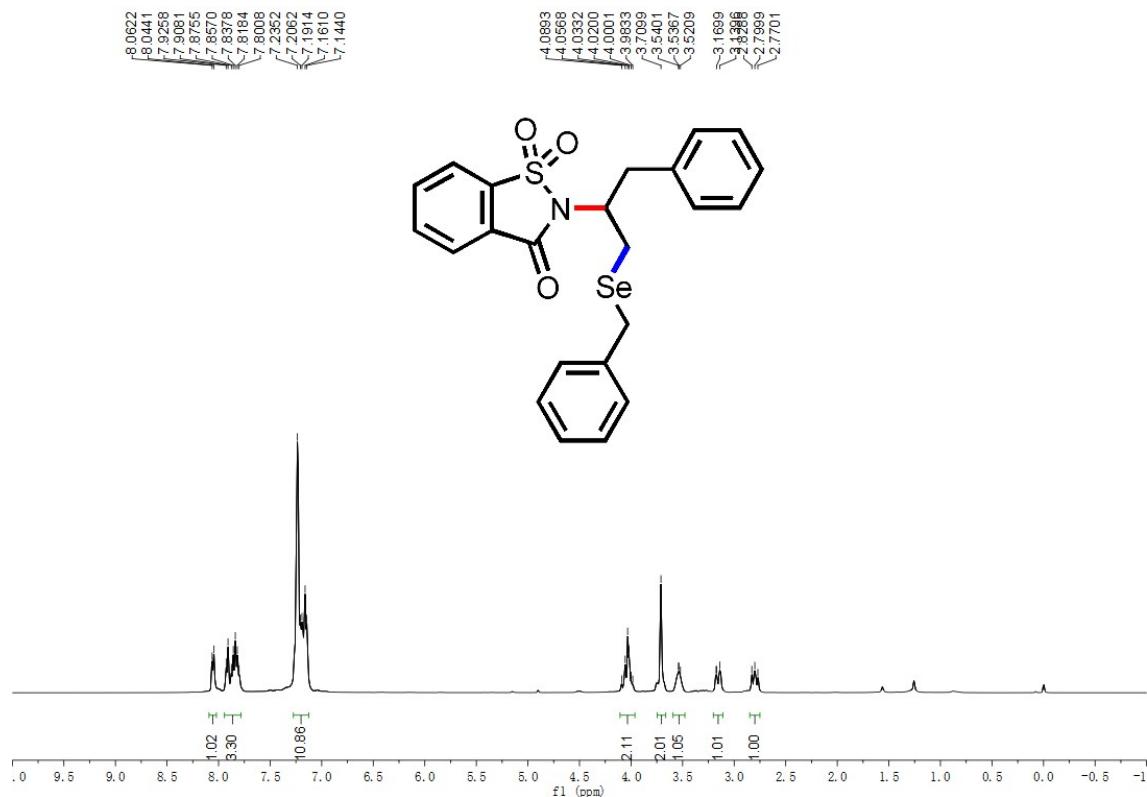
¹H NMR of product 5w in CDCl₃ (400 MHz)



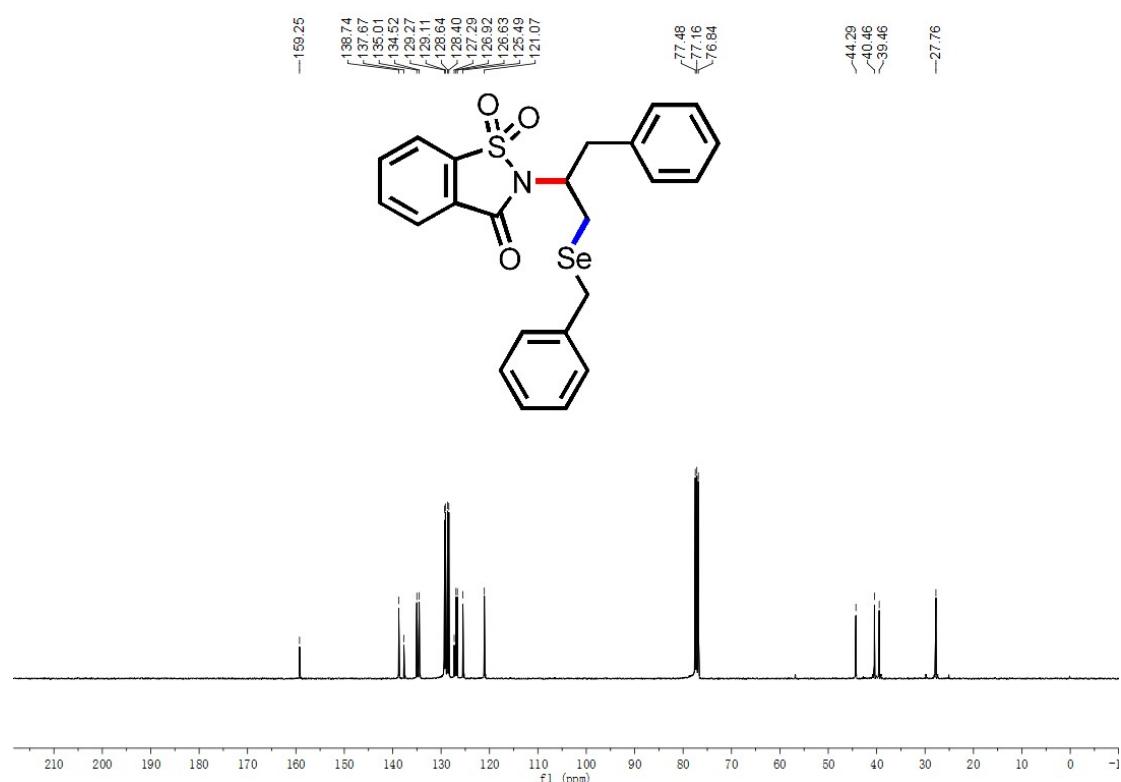
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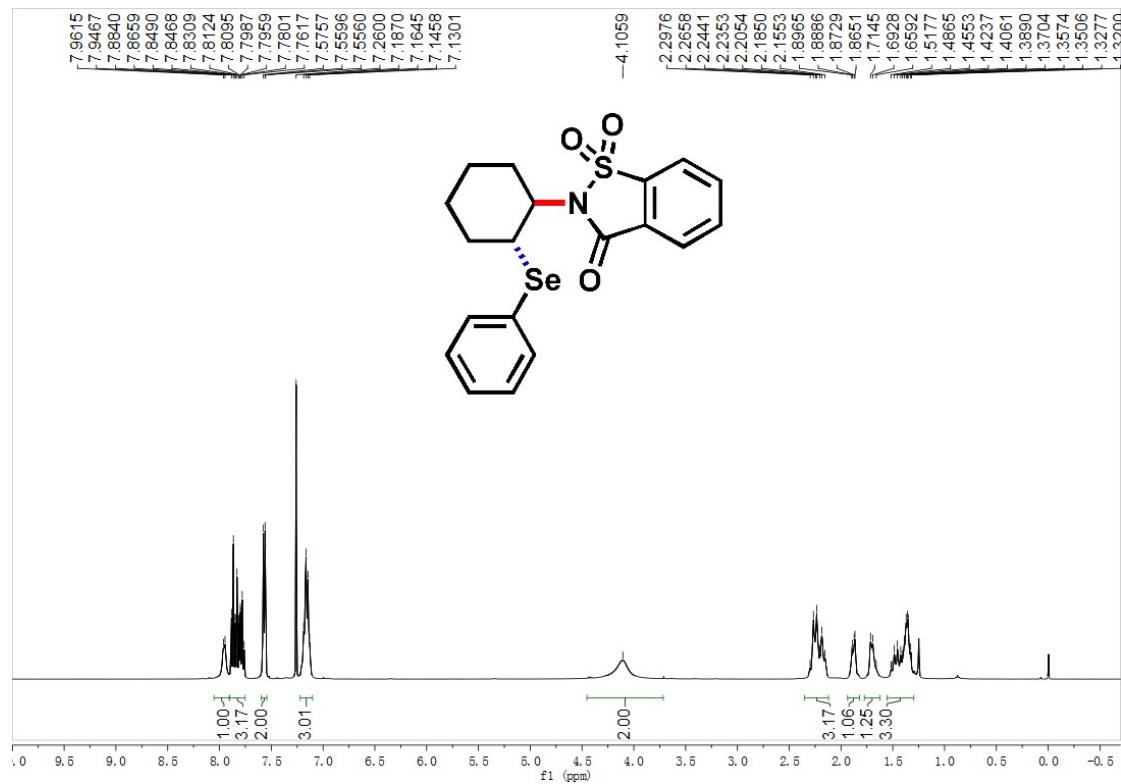
¹H NMR of product 5x in CDCl₃ (400 MHz)



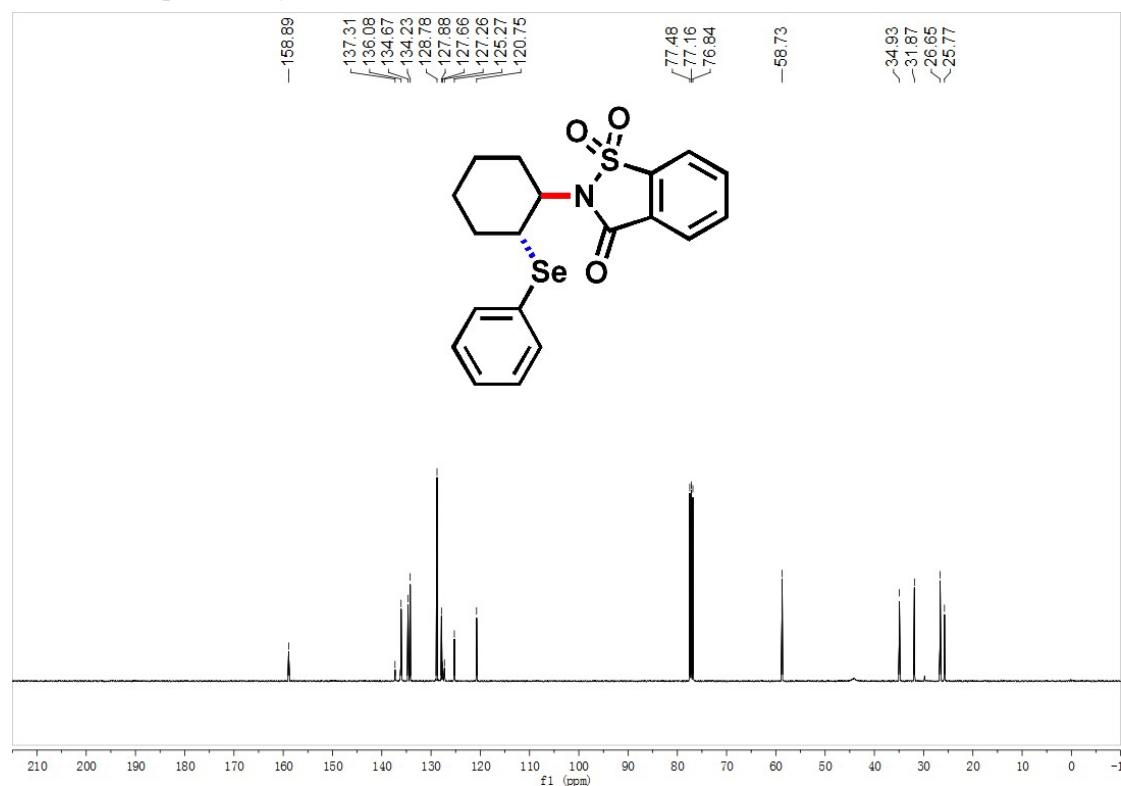
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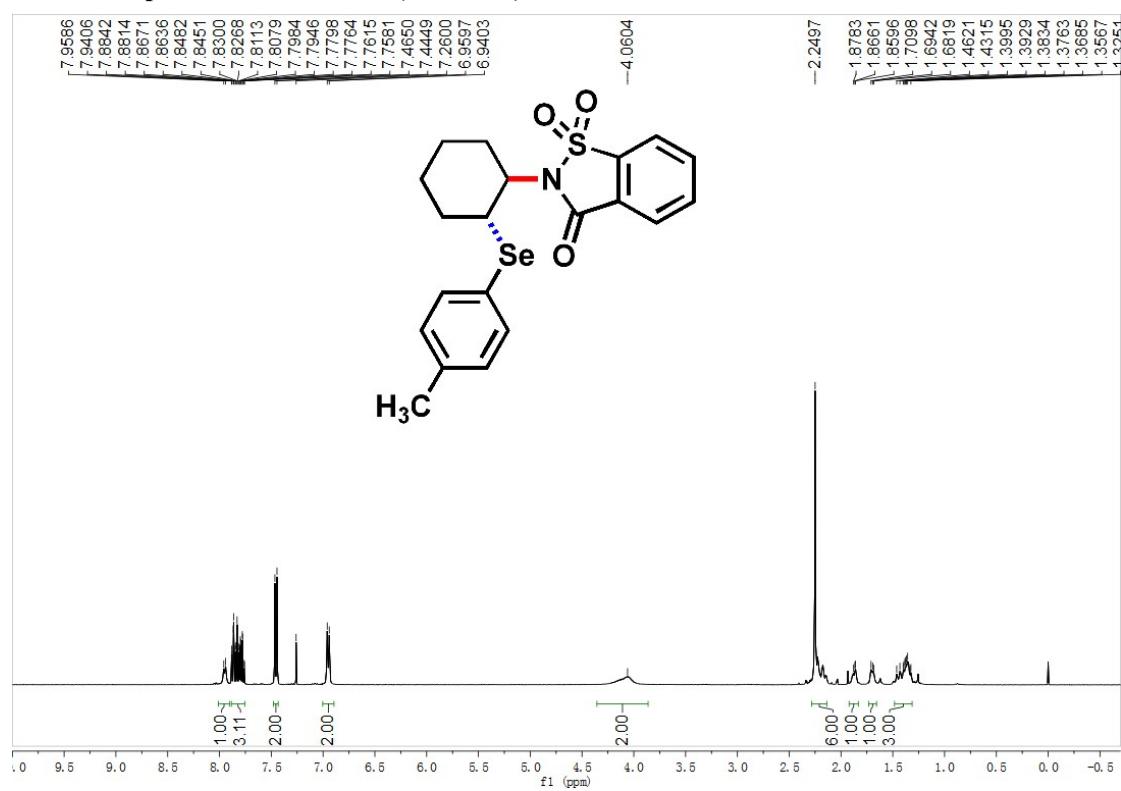
¹H NMR of product 5y in CDCl₃ (400 MHz)



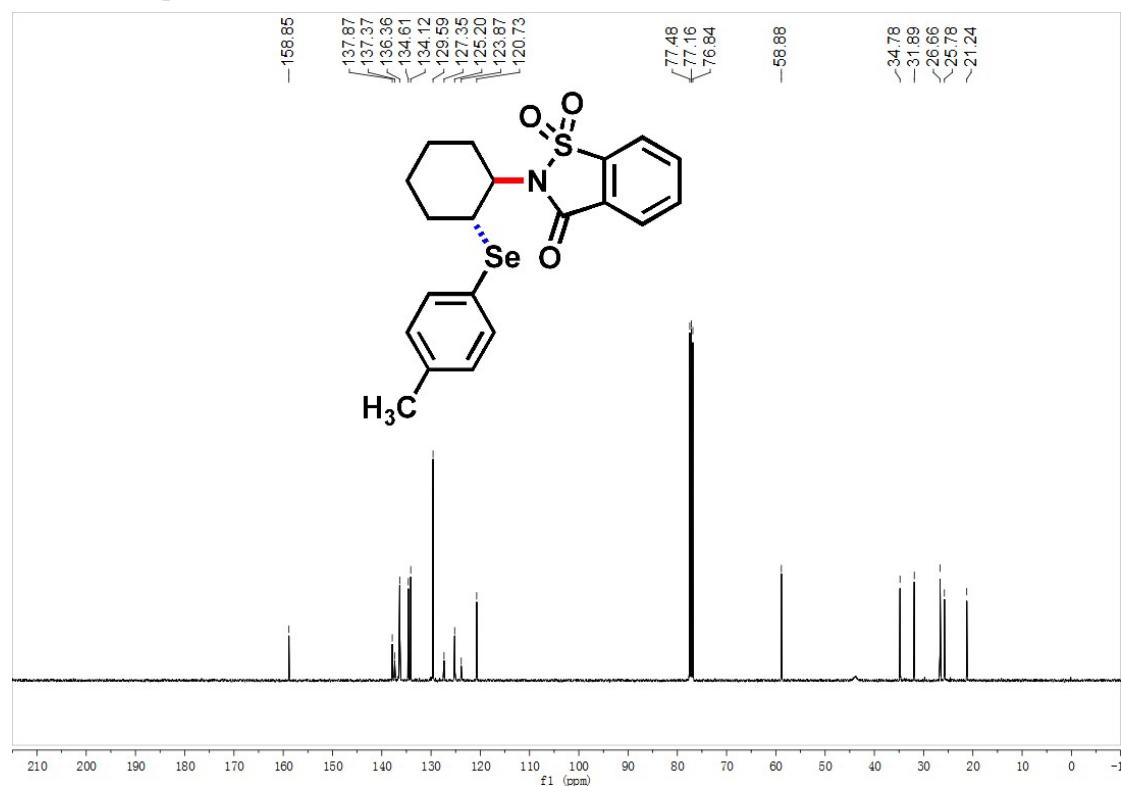
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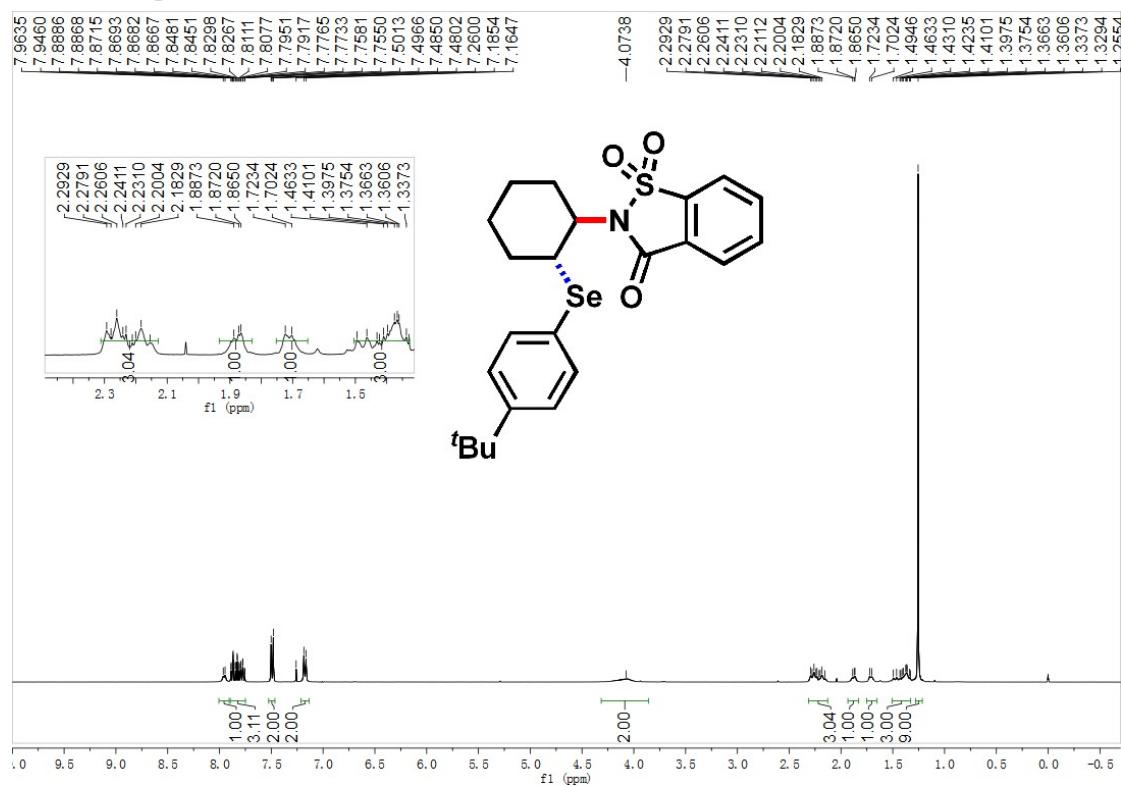
H NMR of product 5z in CDCl₃ (400 MHz)



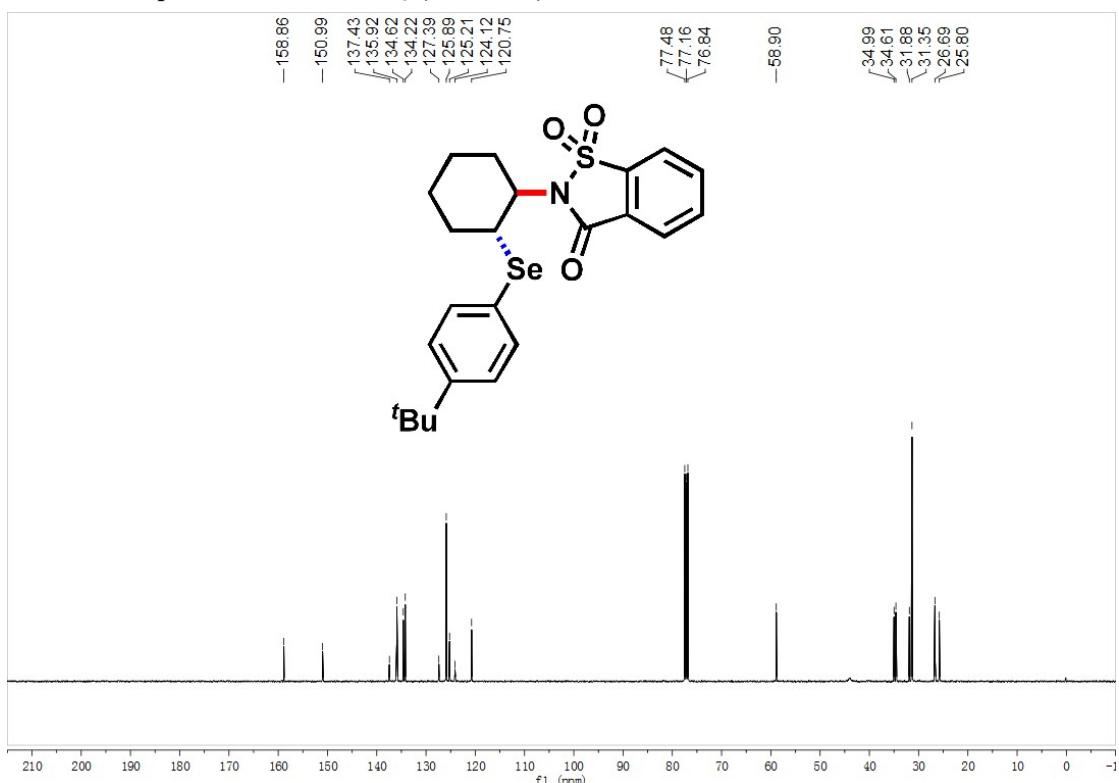
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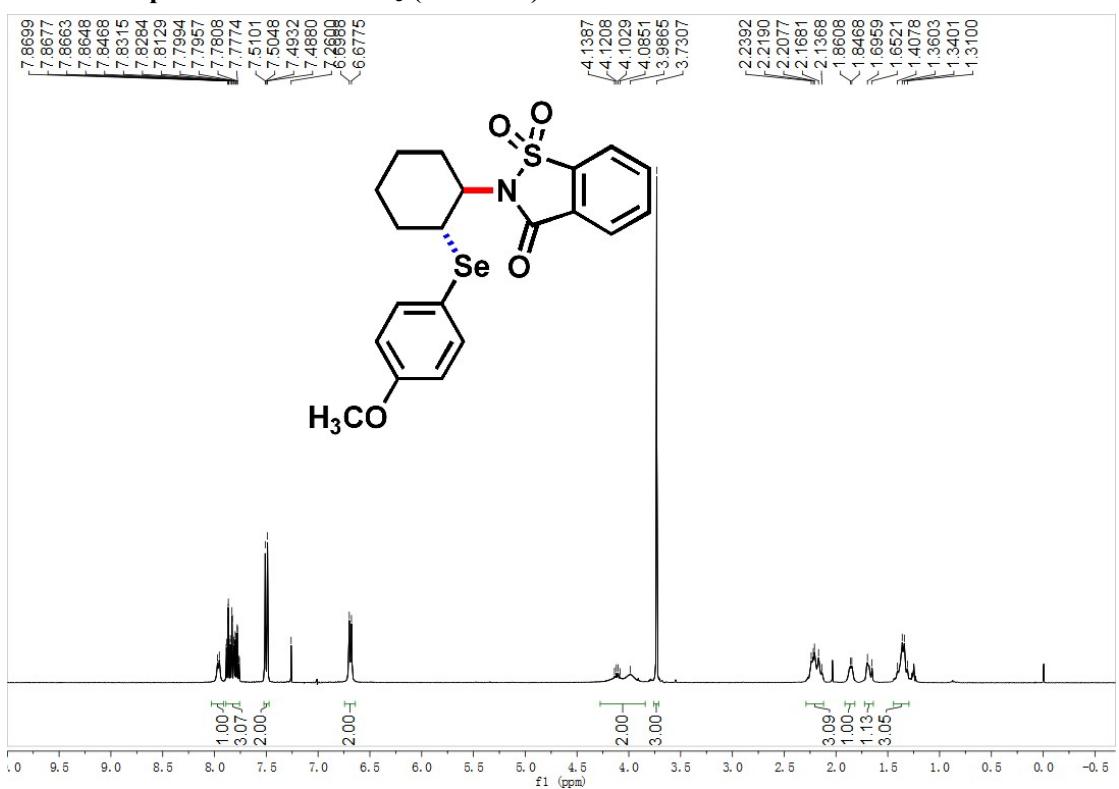
¹H NMR of product 5a' in CDCl₃ (400 MHz)



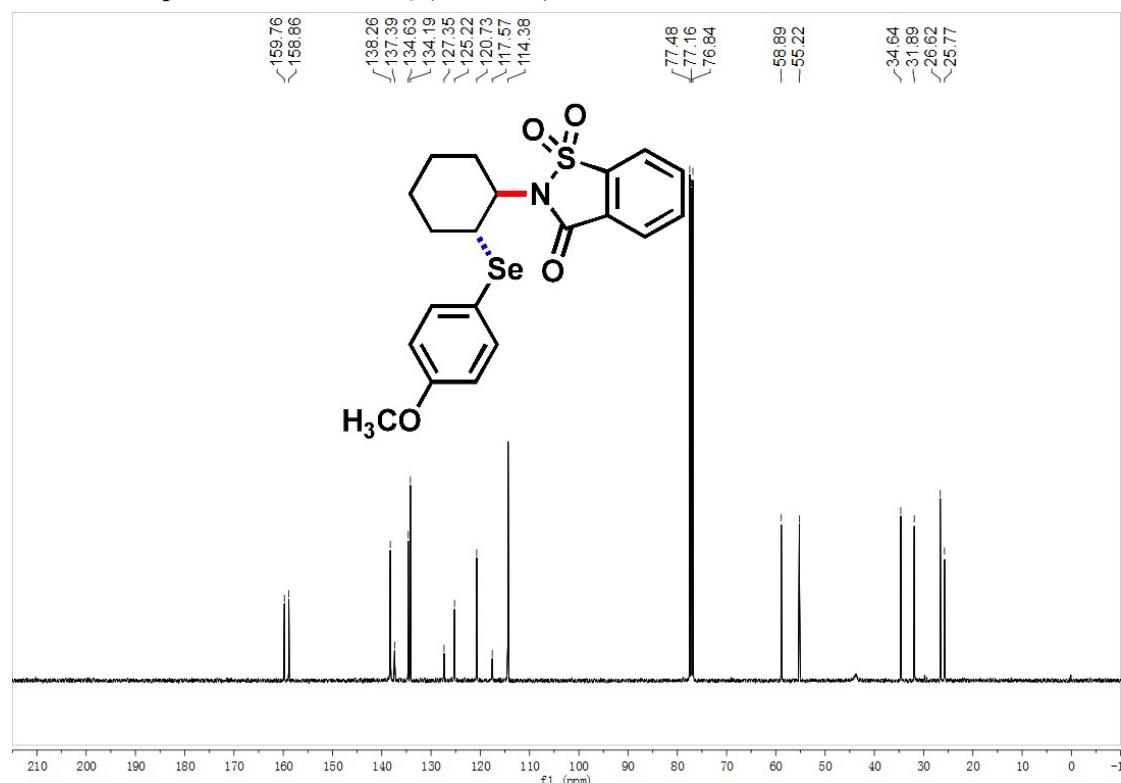
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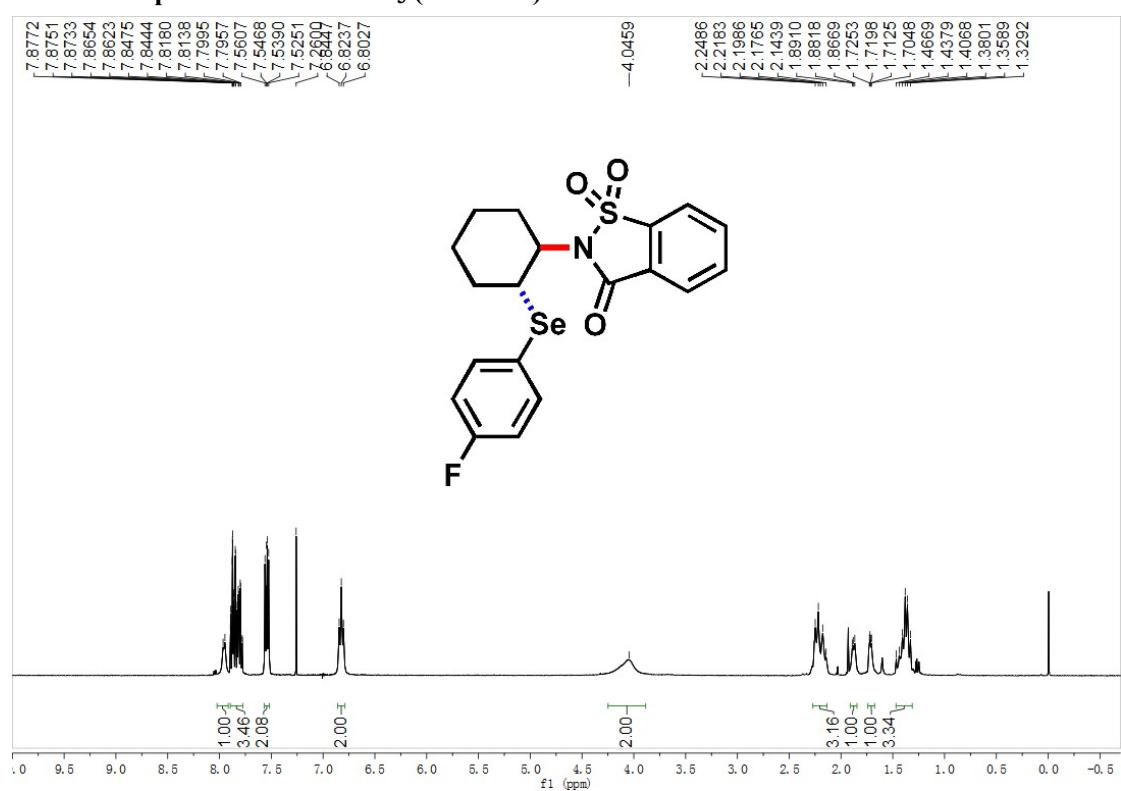
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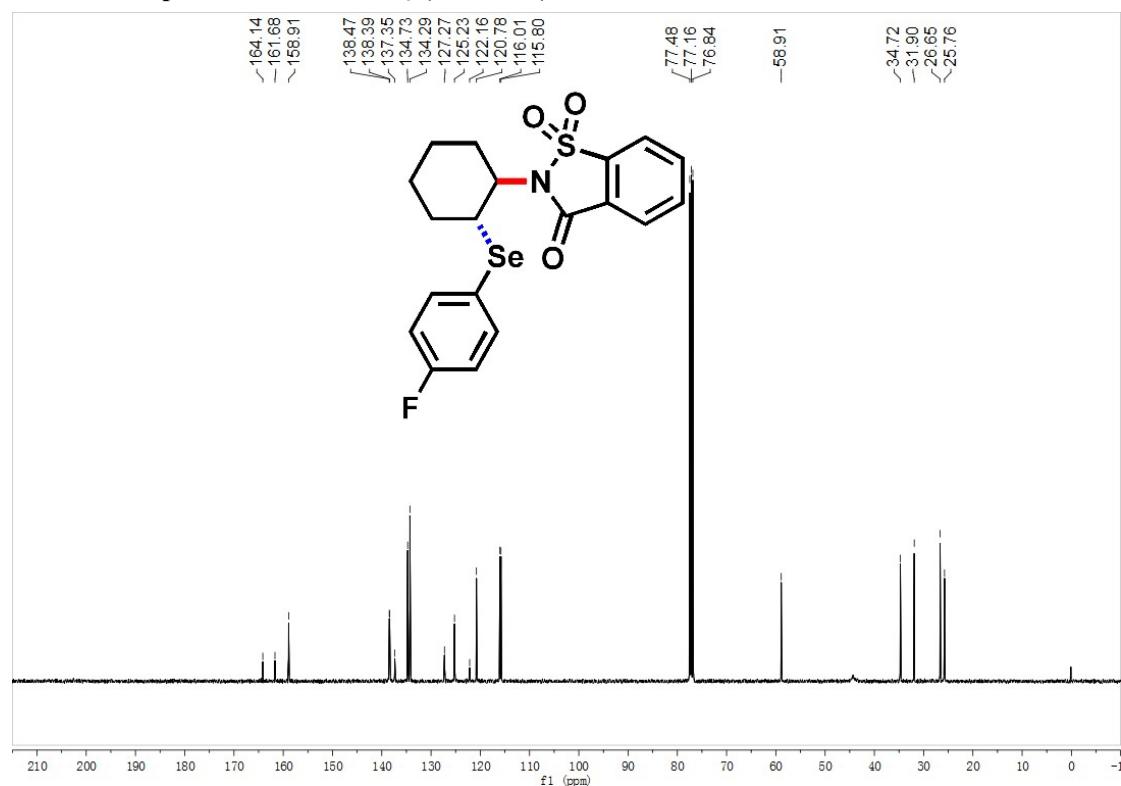
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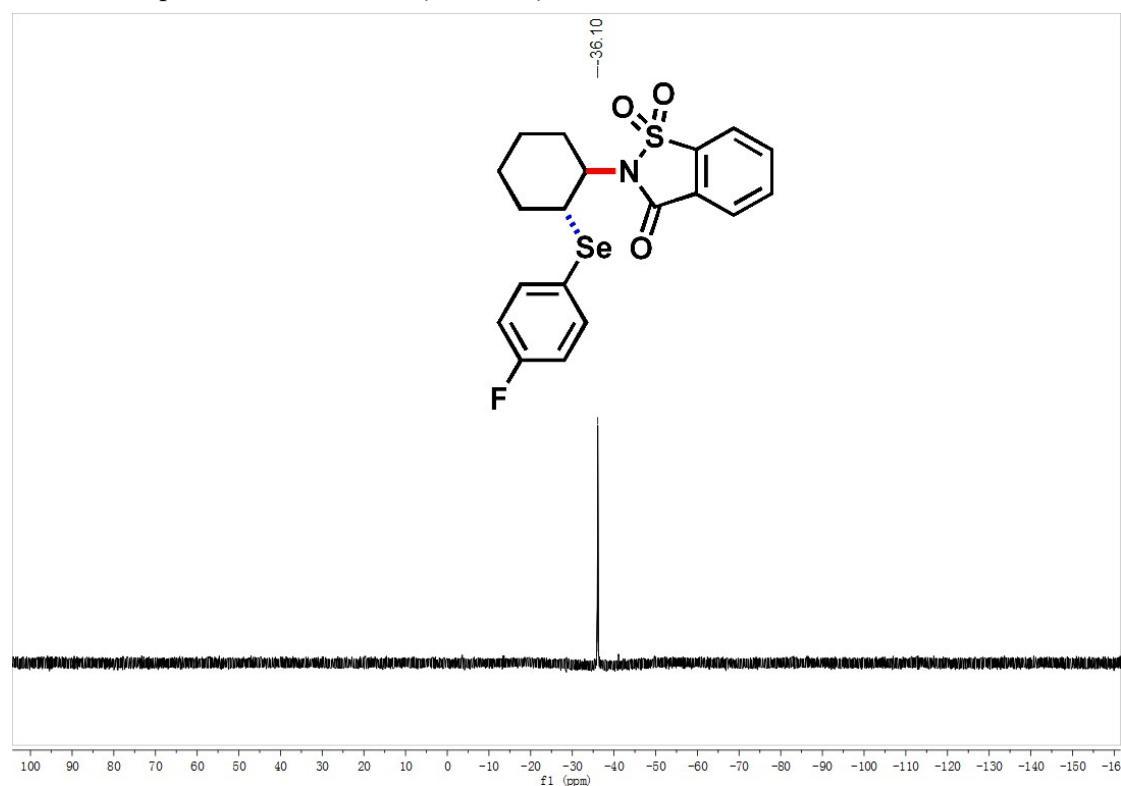
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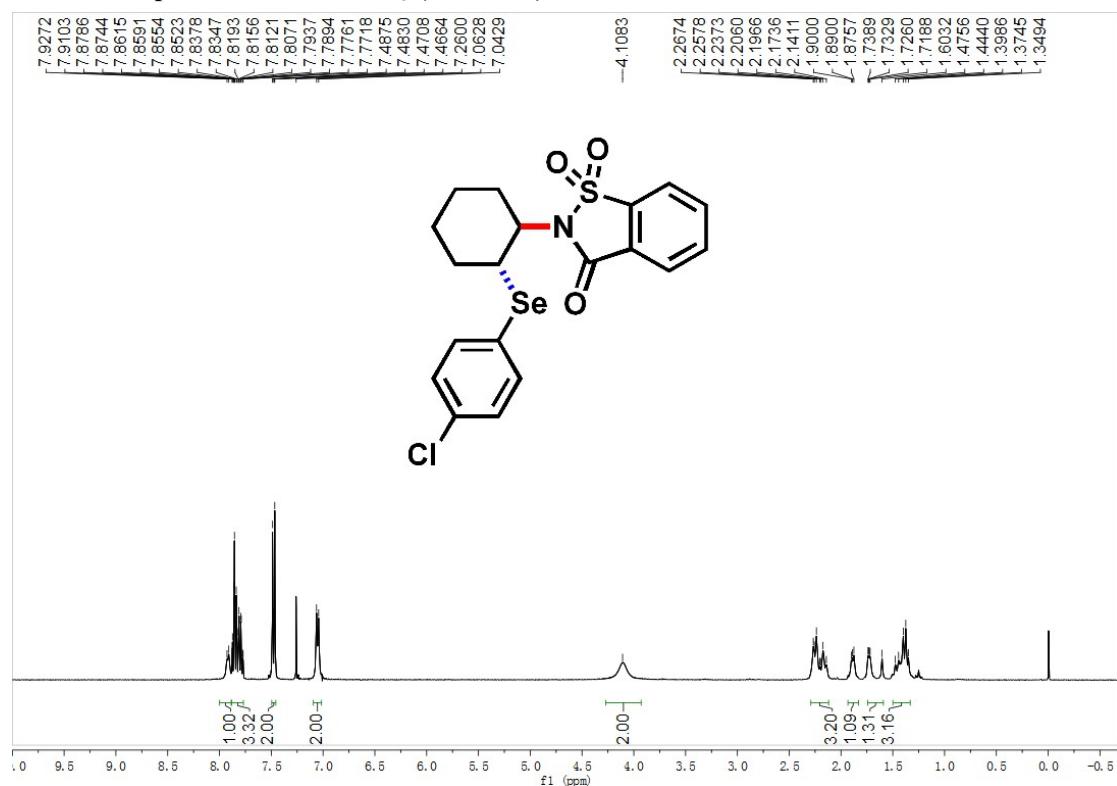
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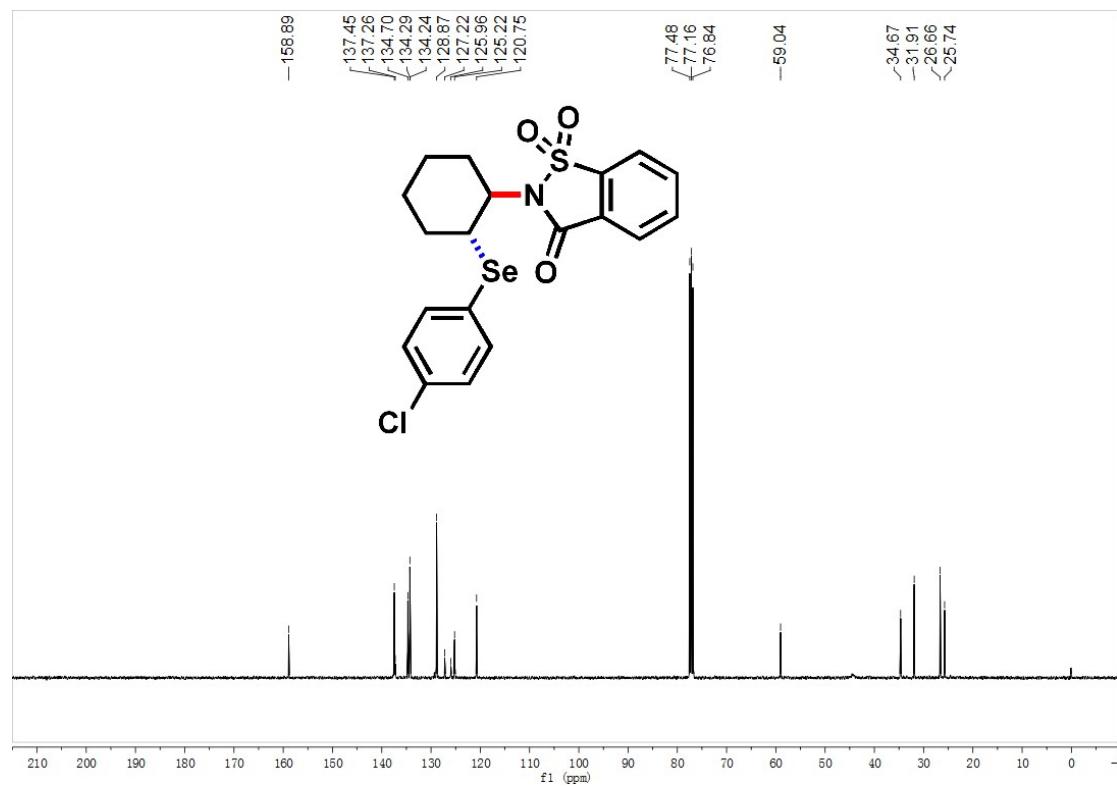
¹⁹F NMR of product 5c' in CDCl₃ (376 MHz)



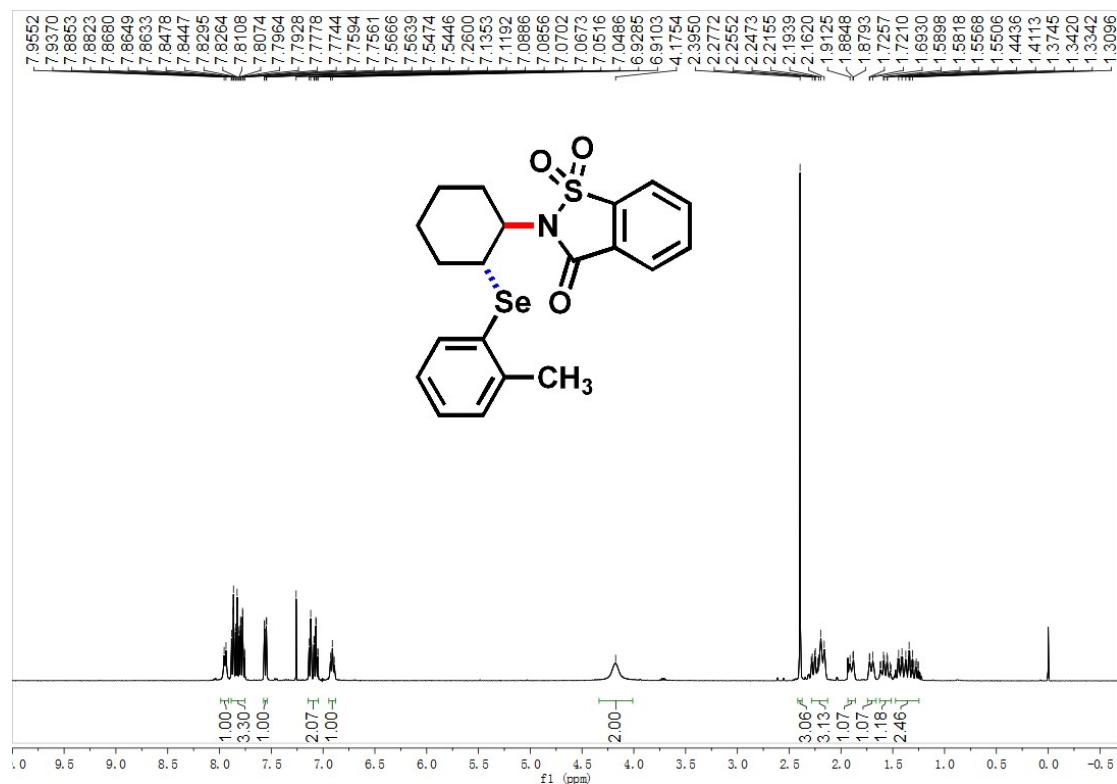
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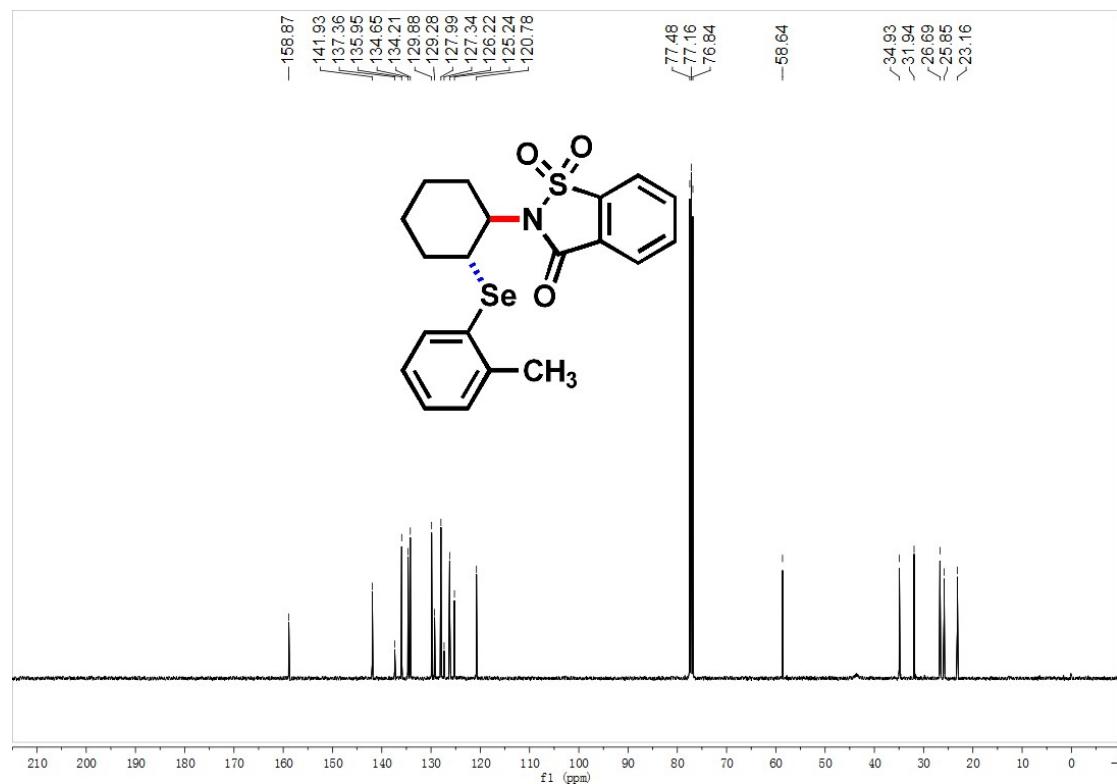
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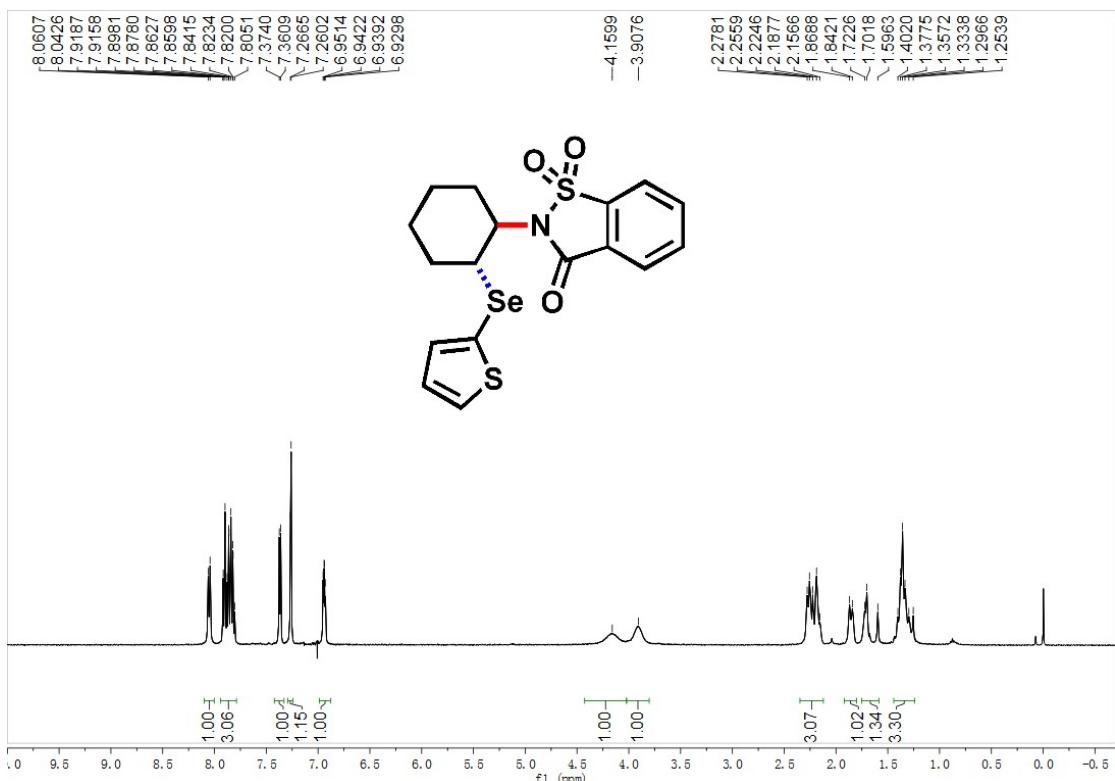
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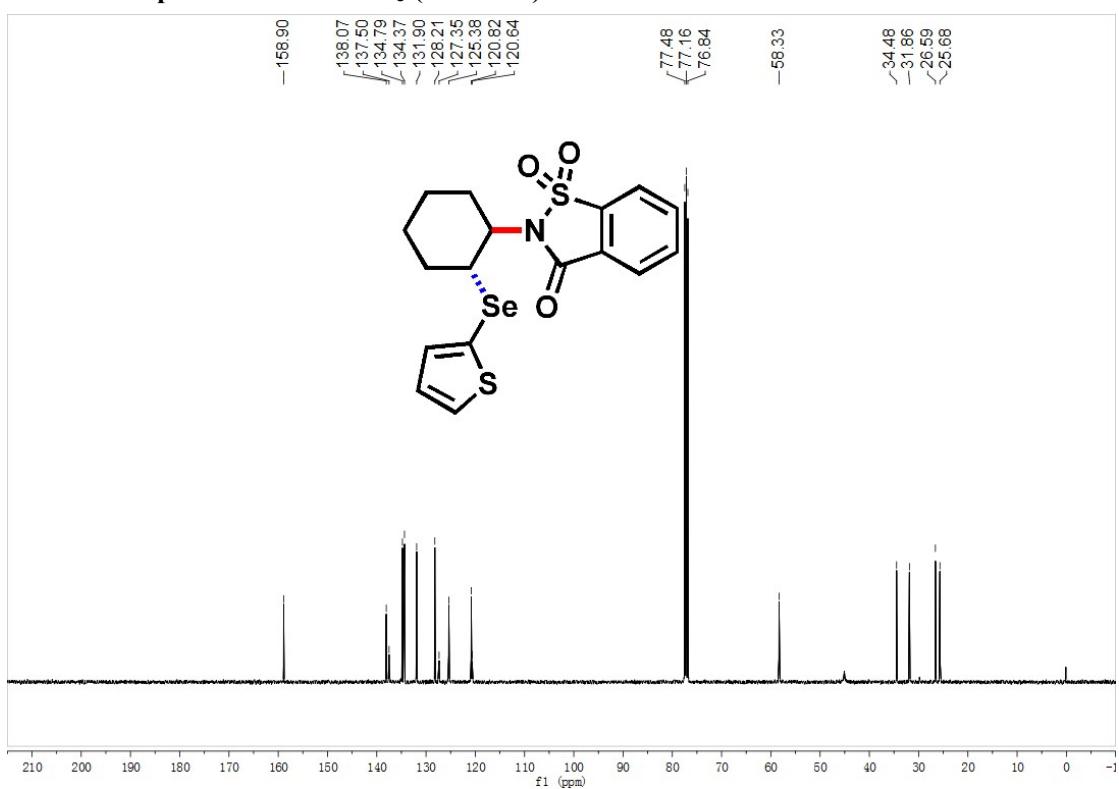
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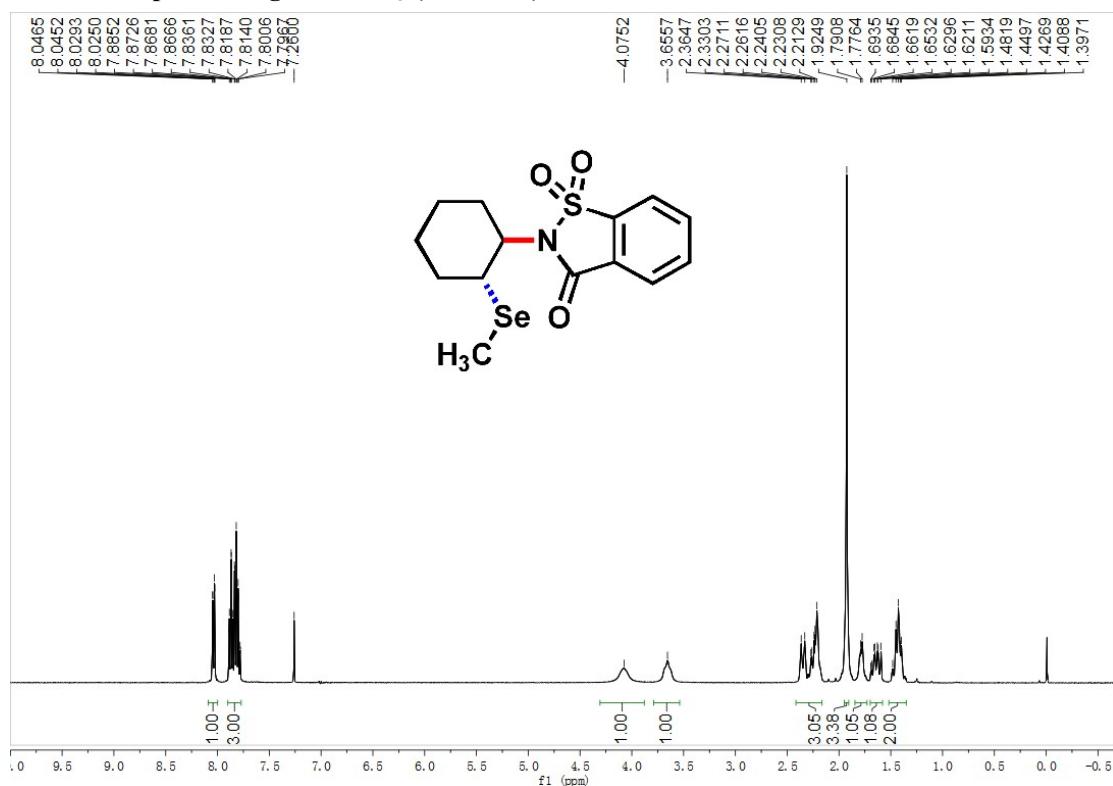
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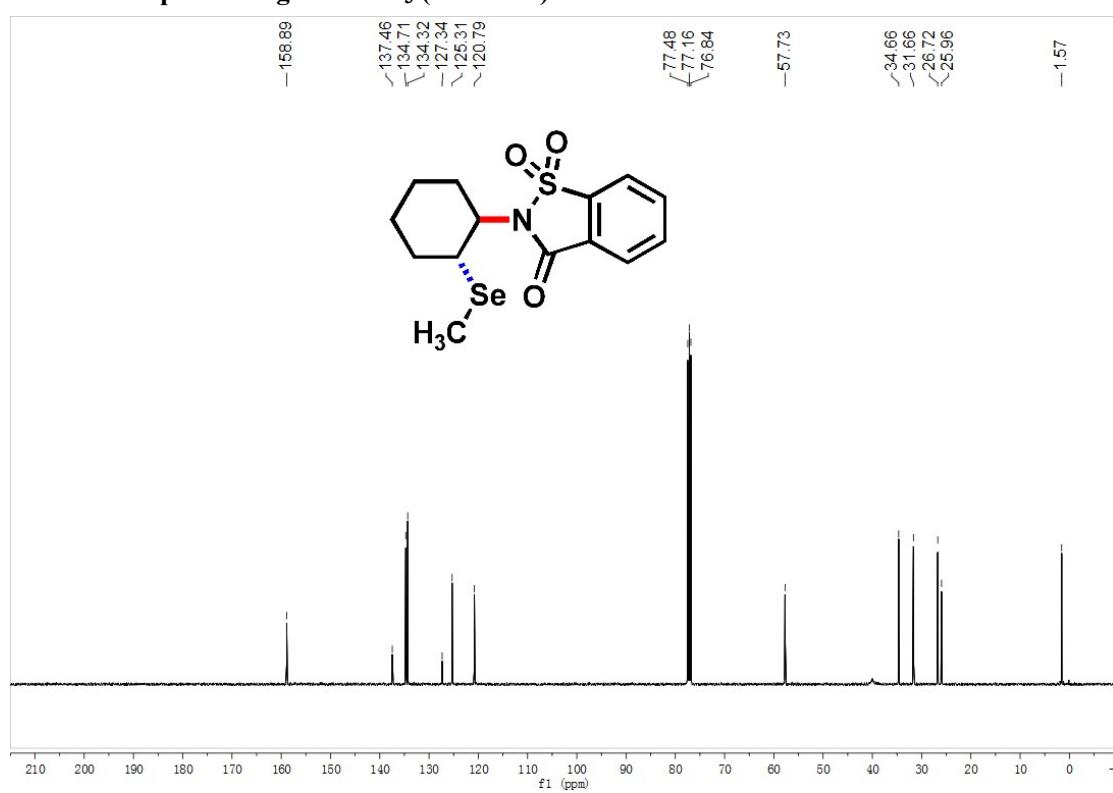
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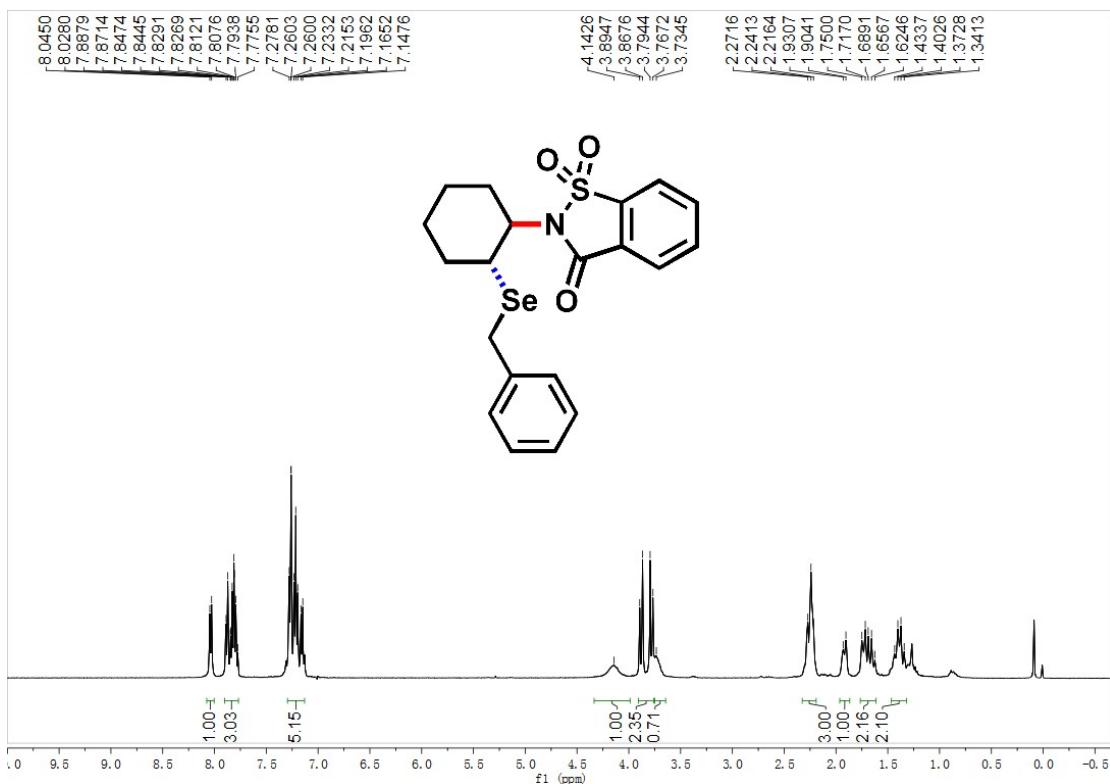
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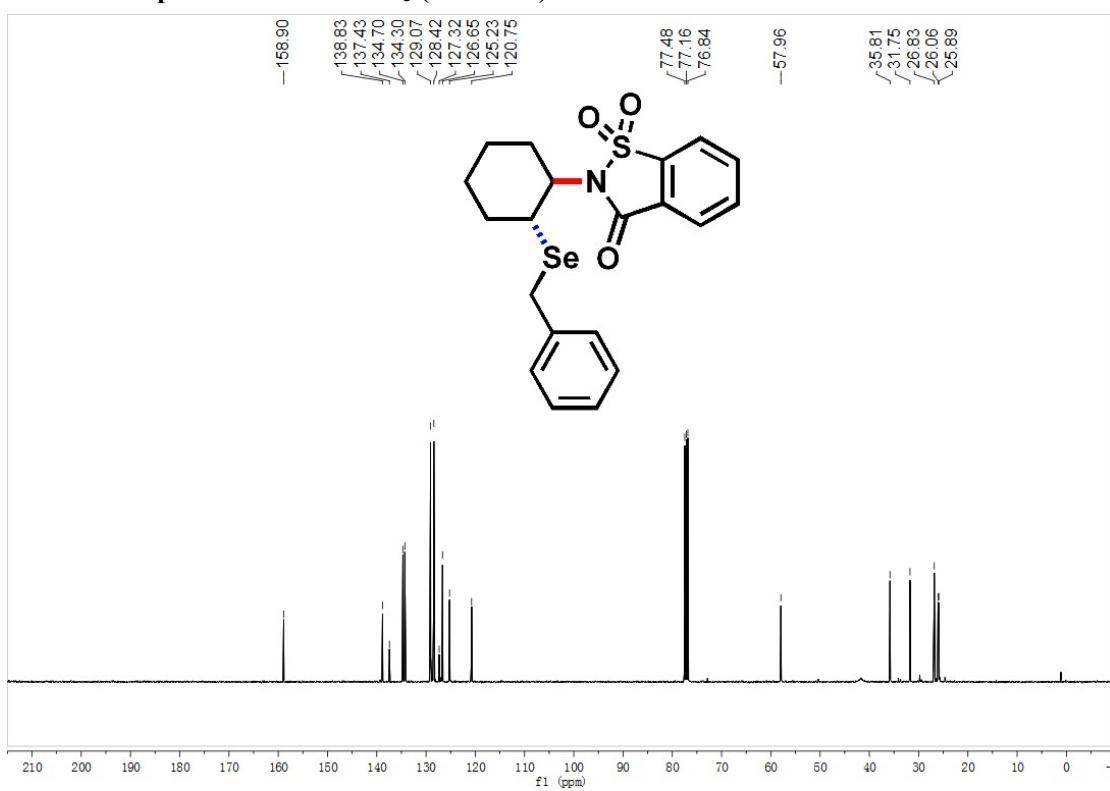
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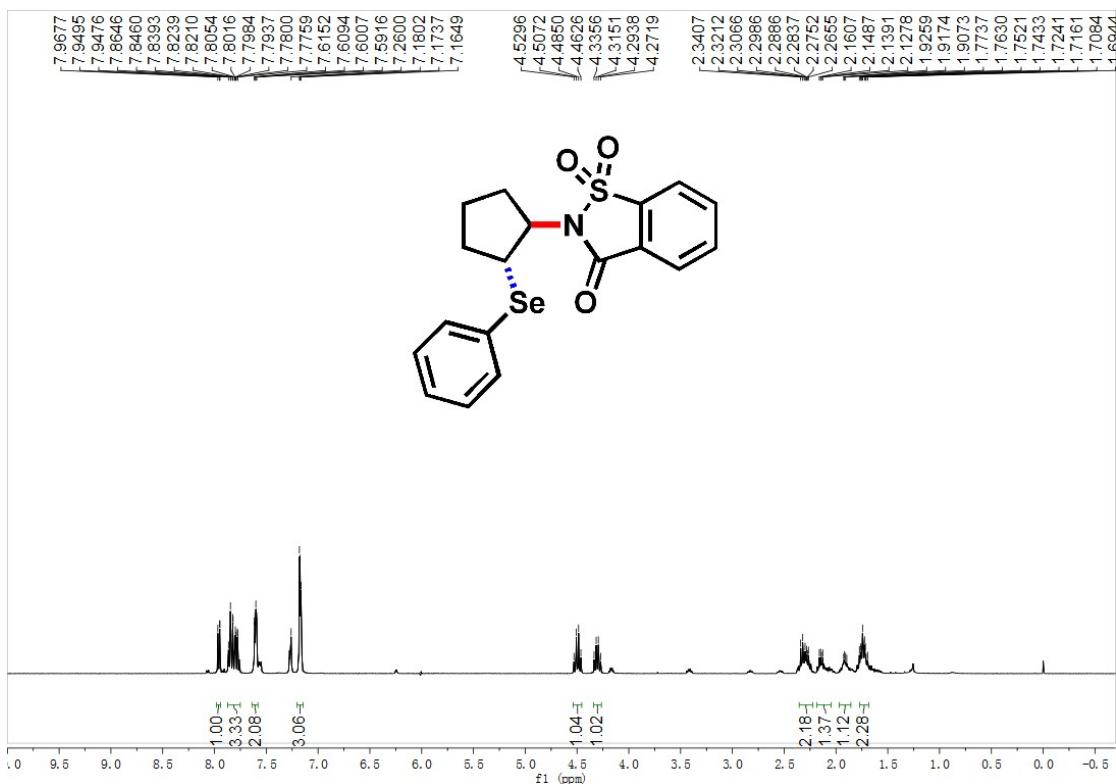
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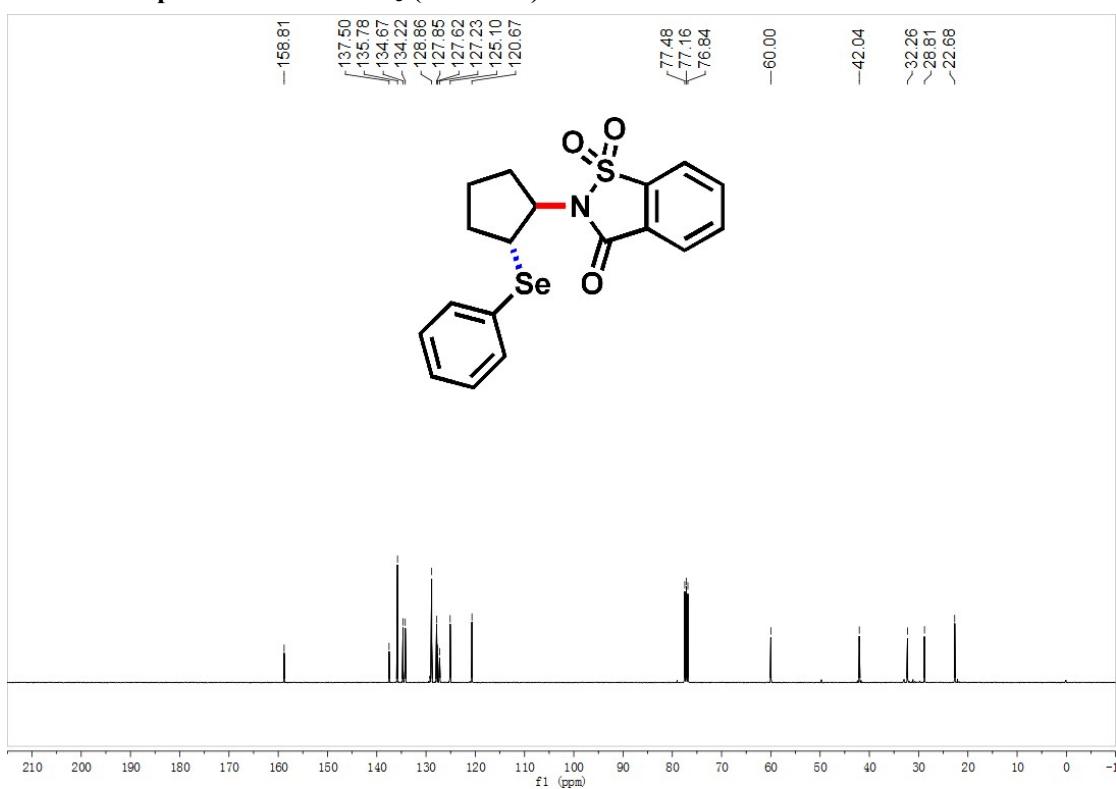
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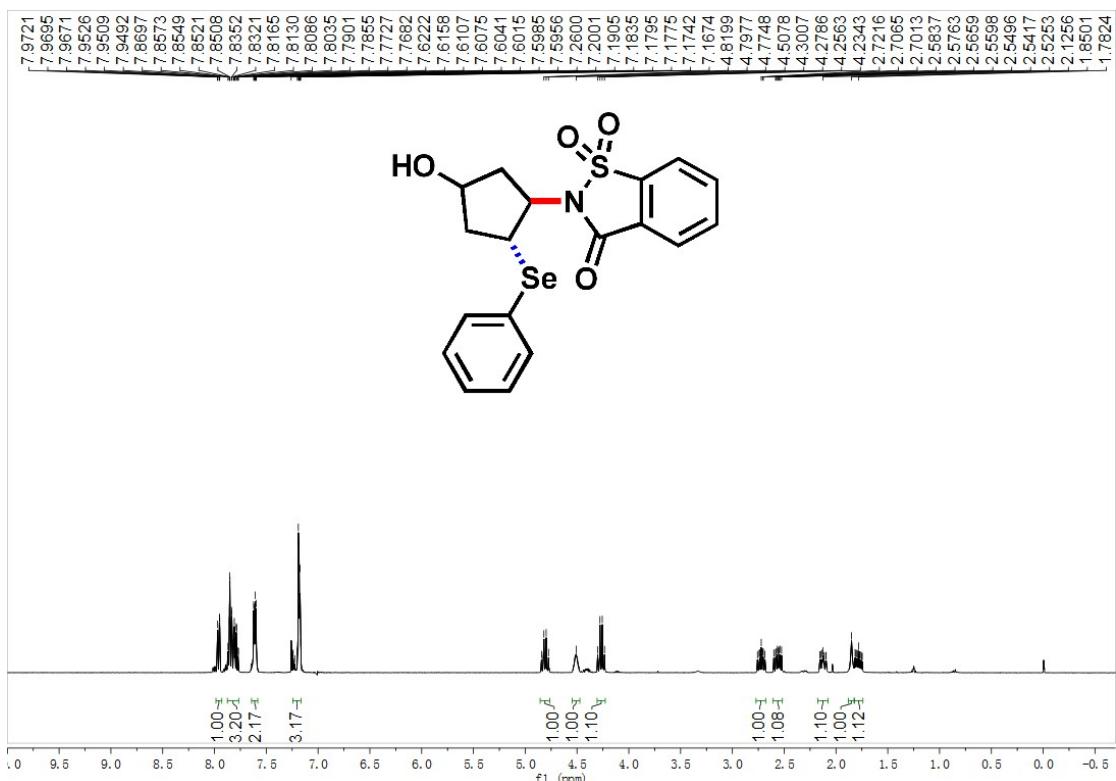
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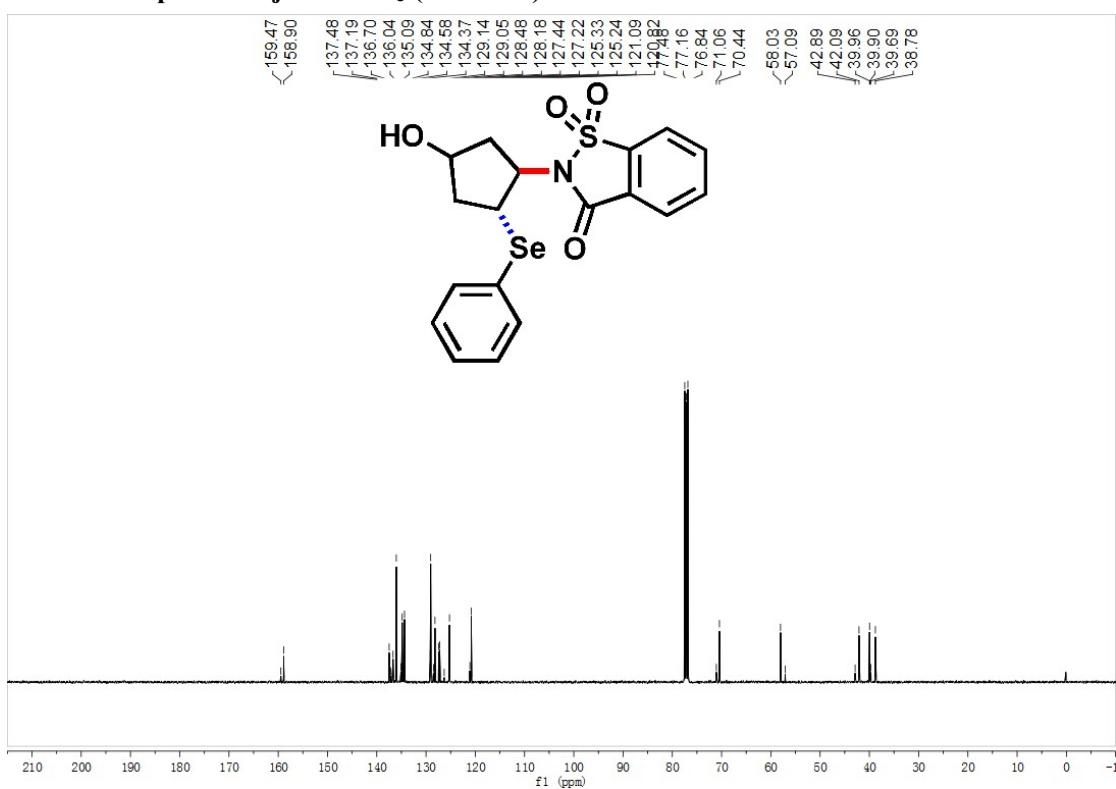
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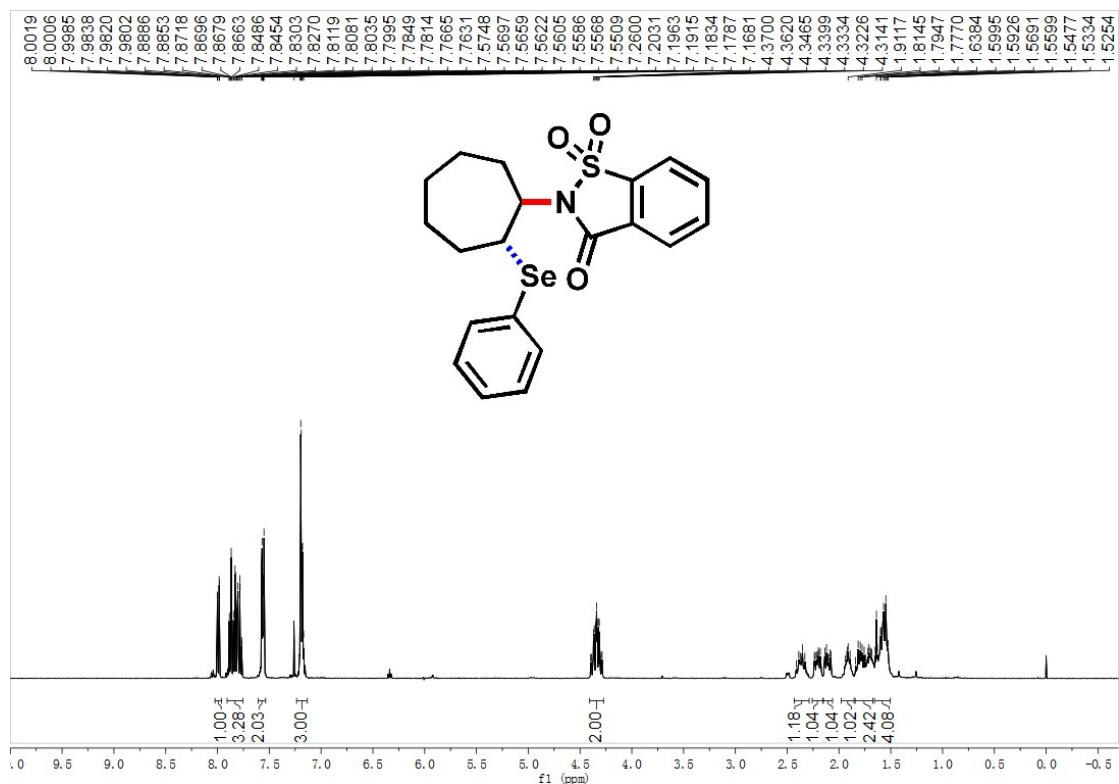
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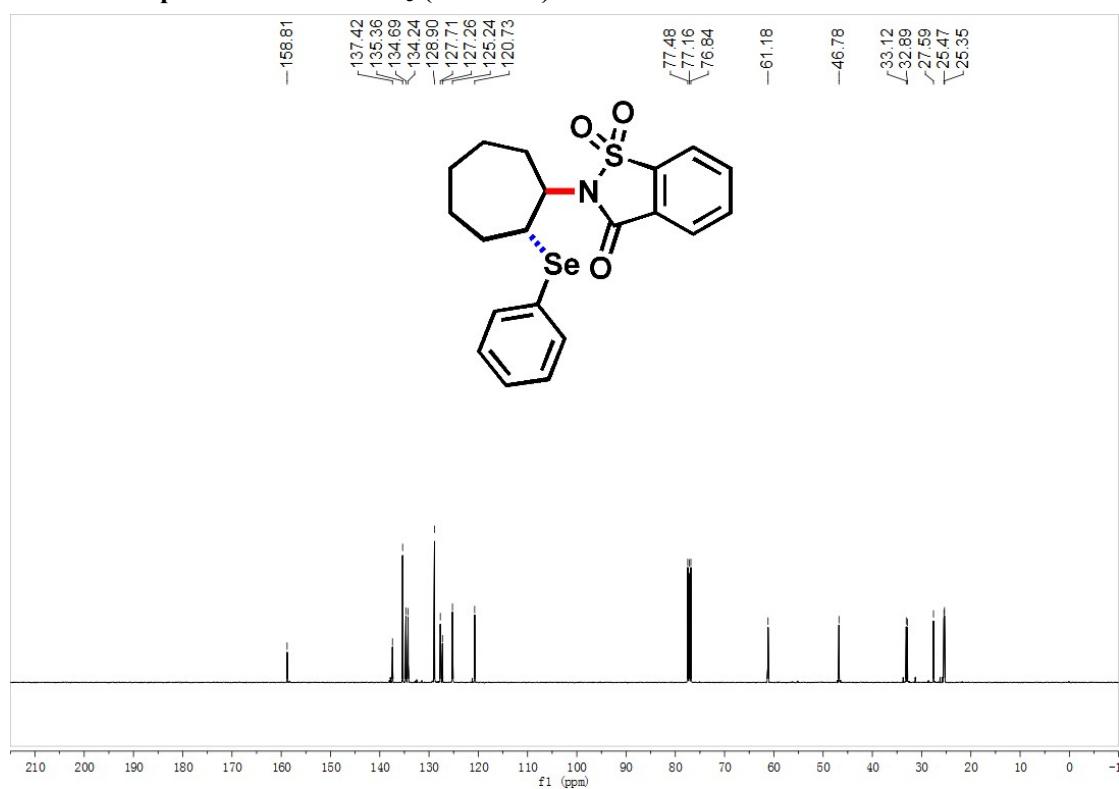
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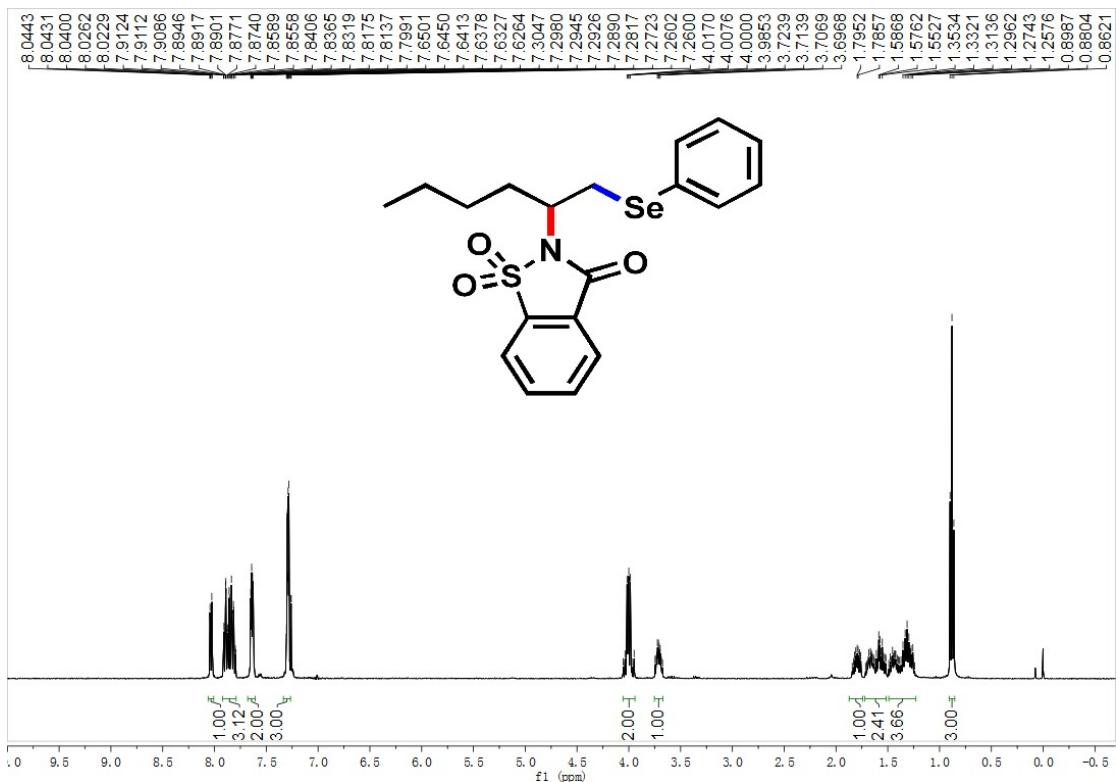
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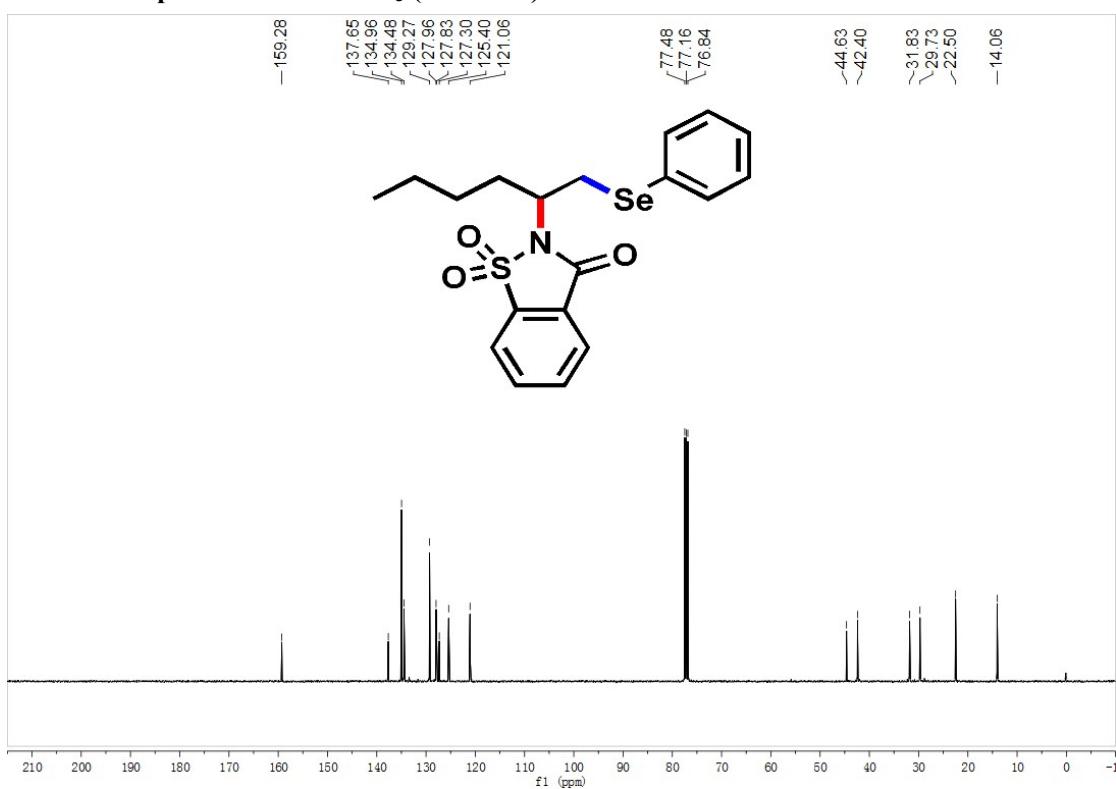
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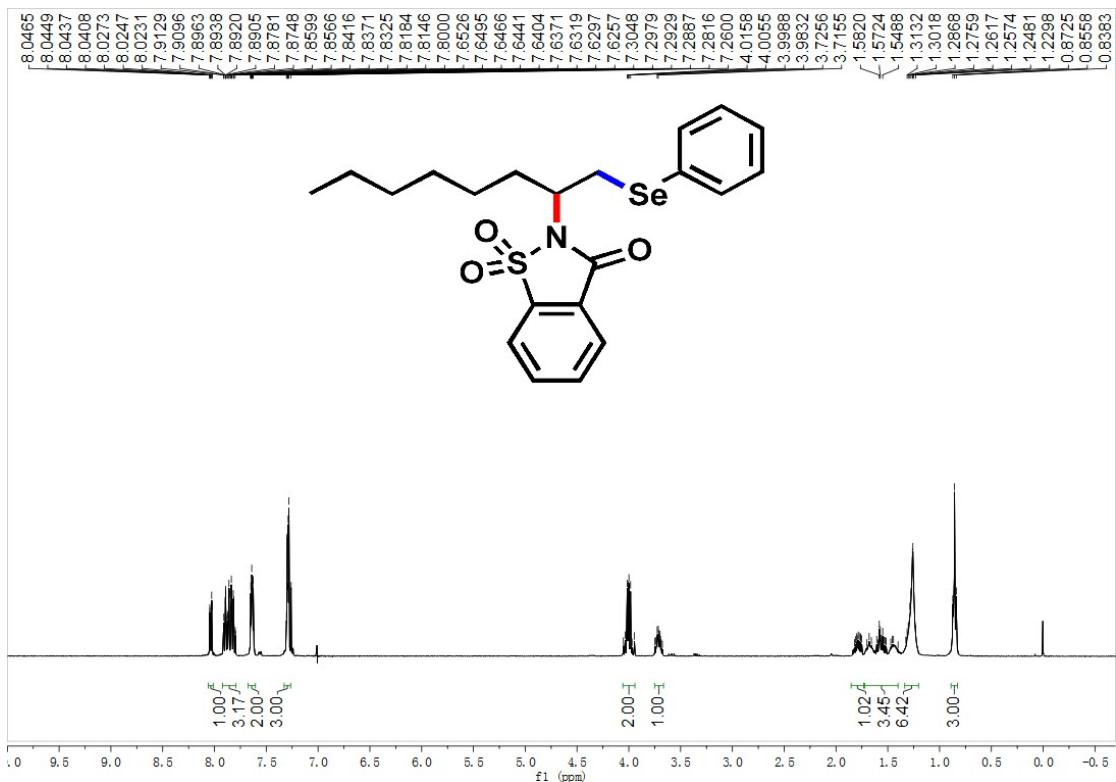
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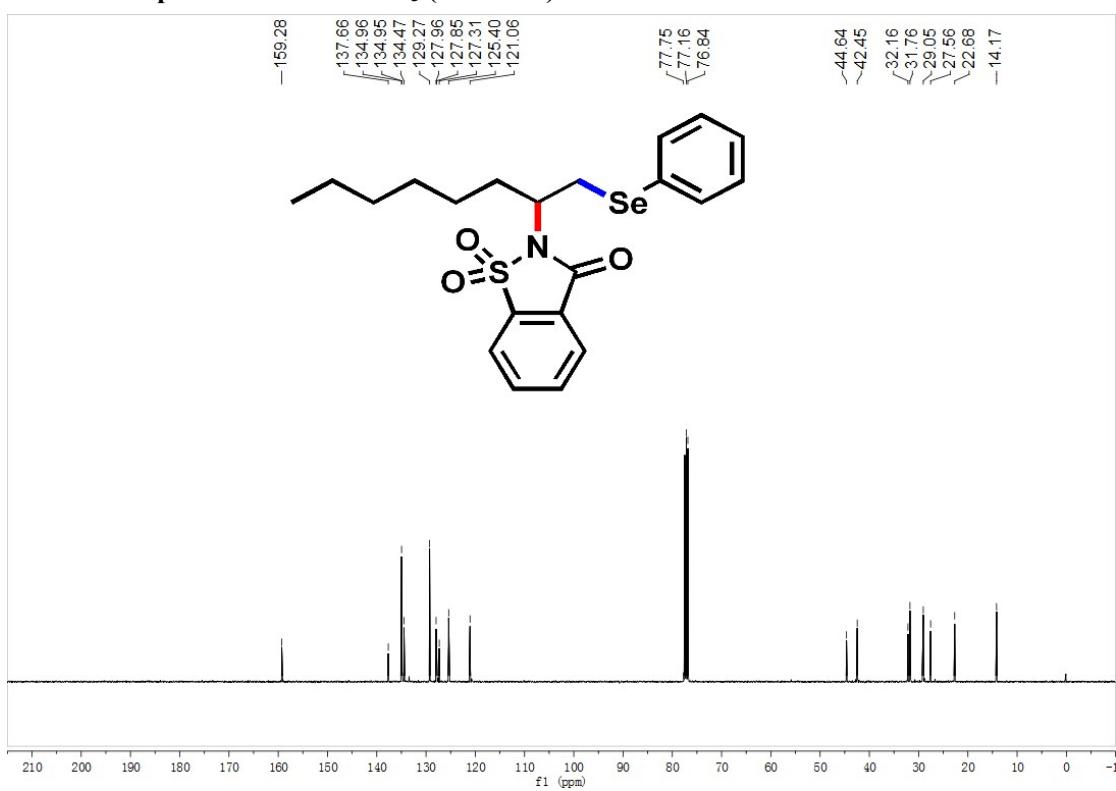
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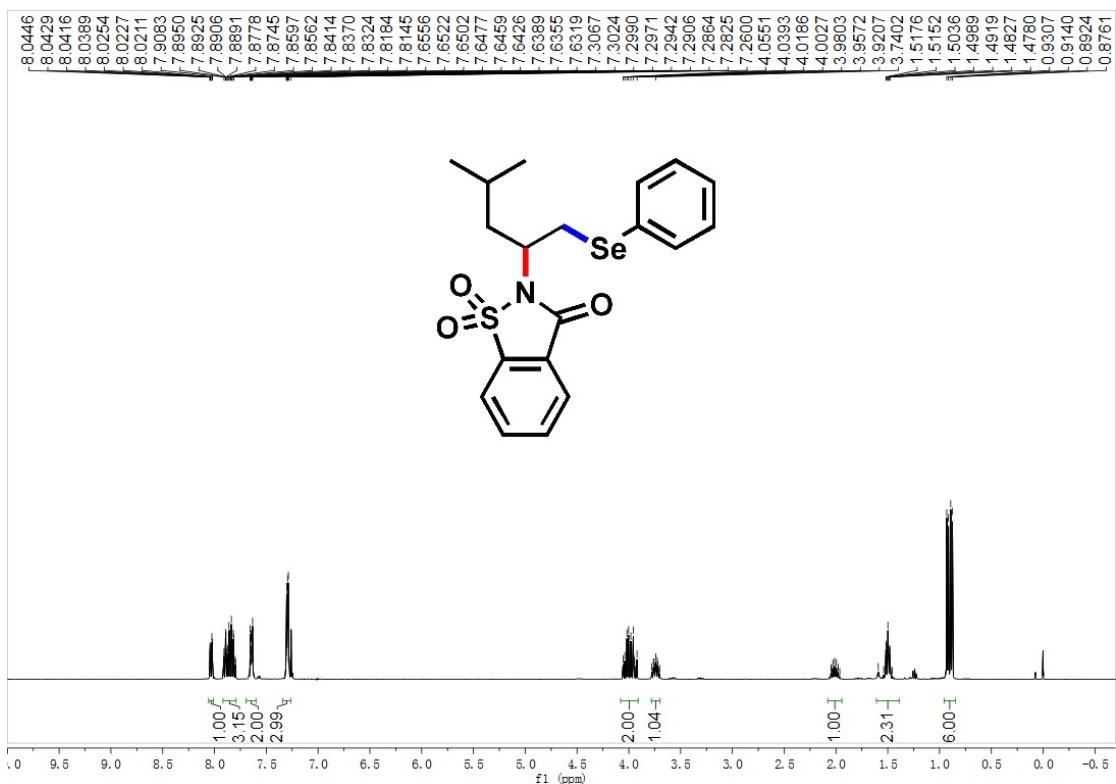
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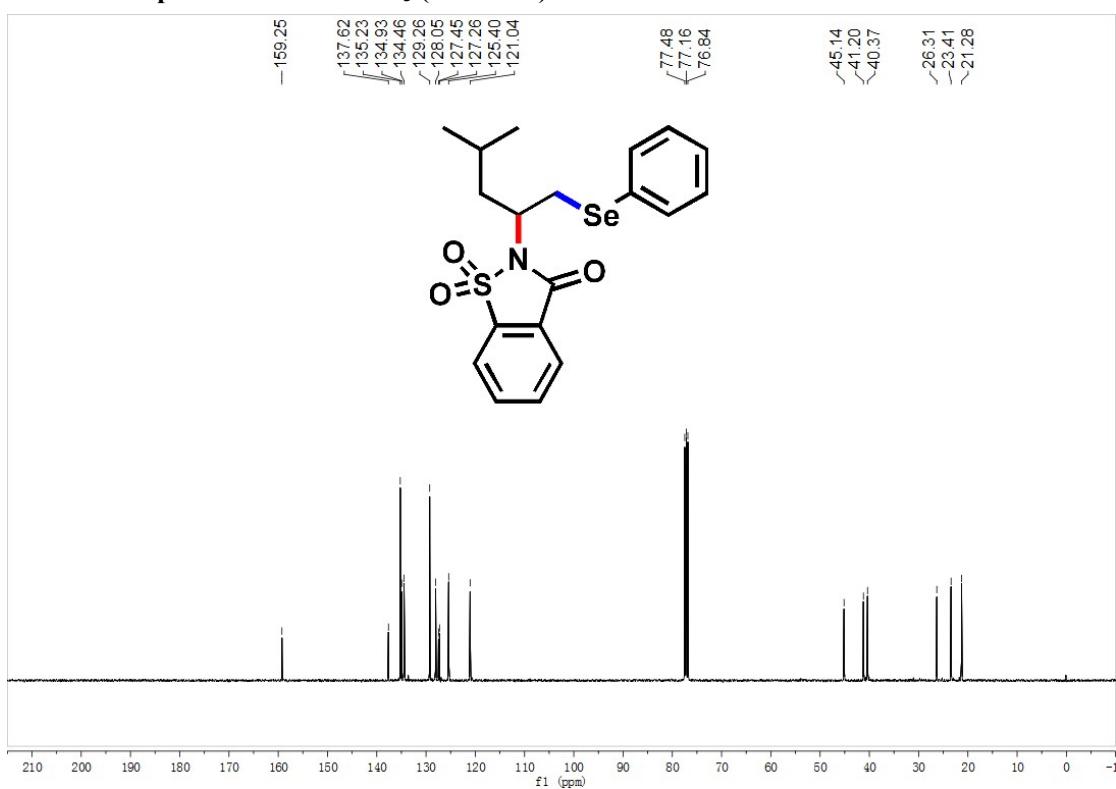
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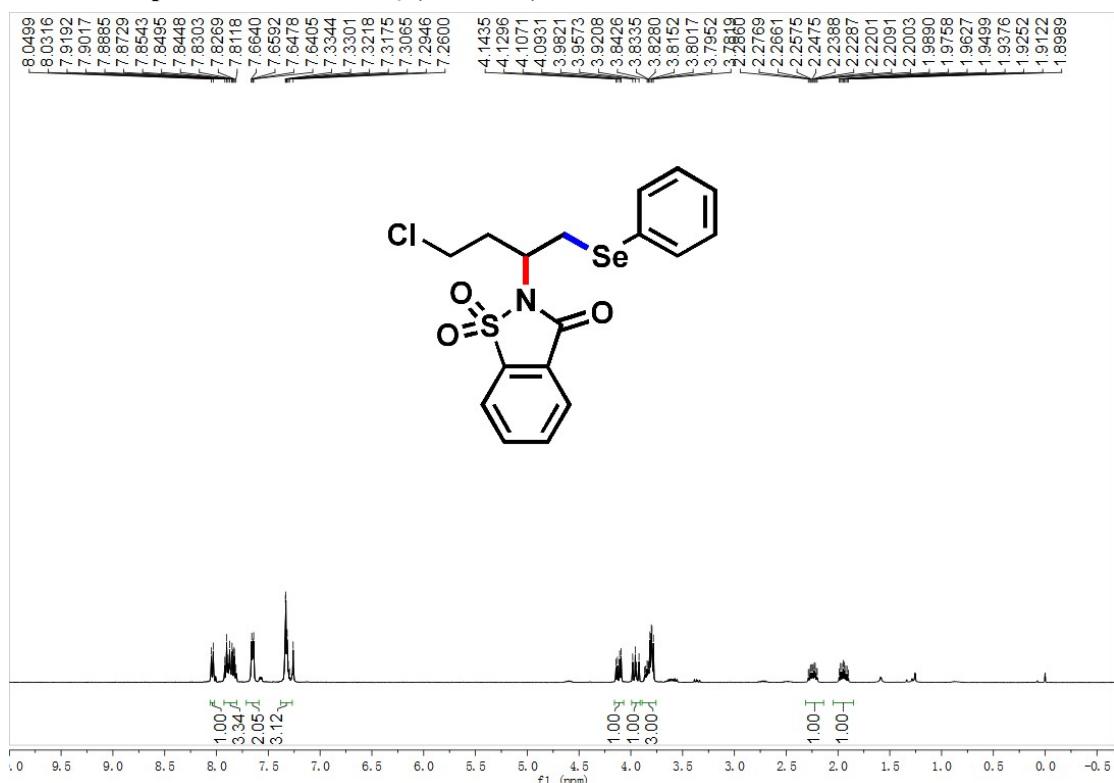
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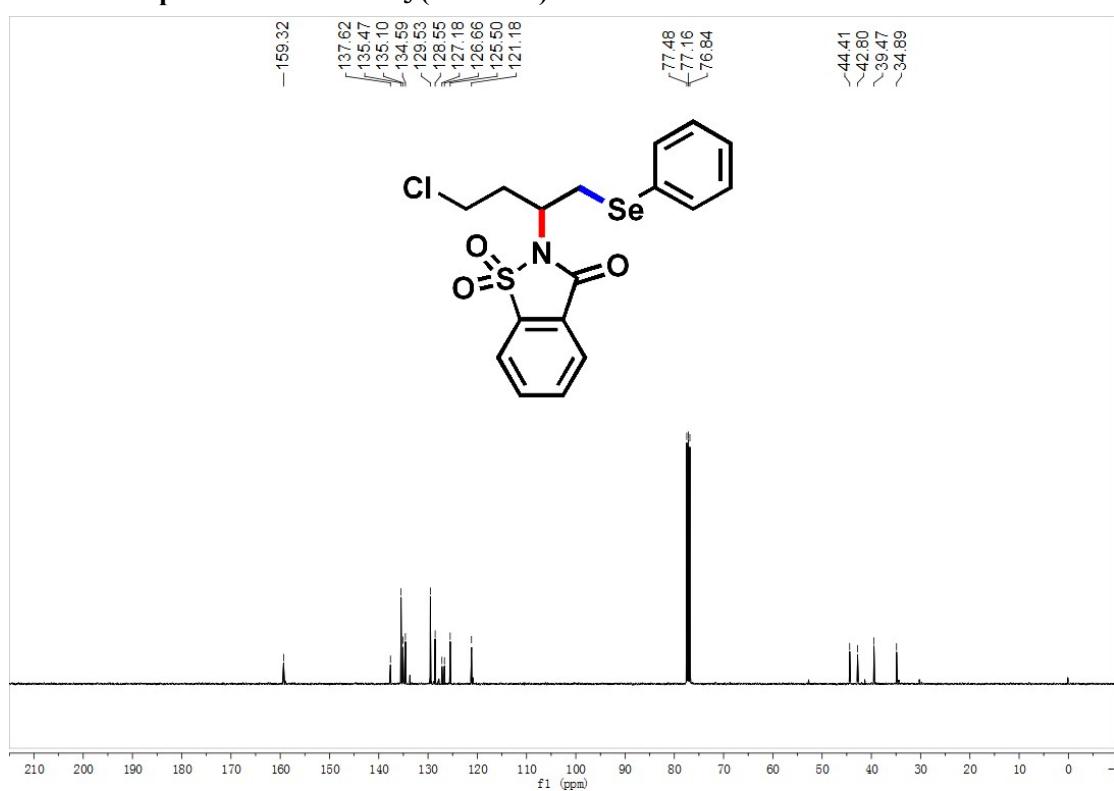
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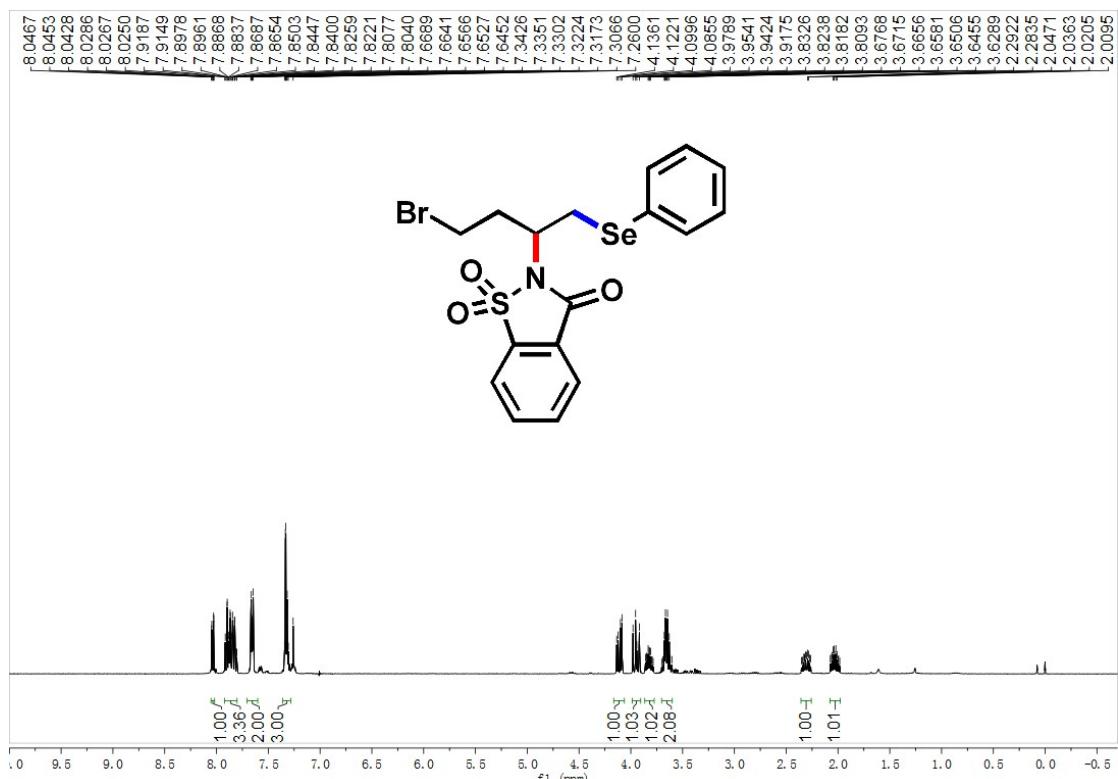
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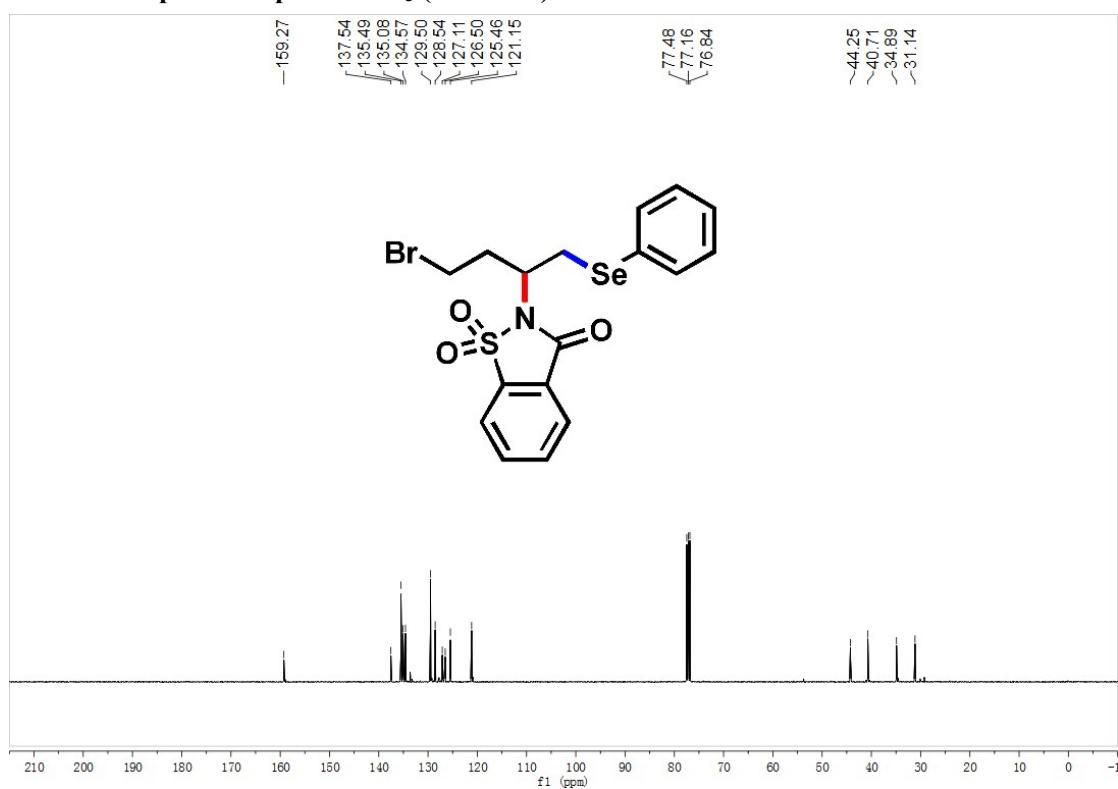
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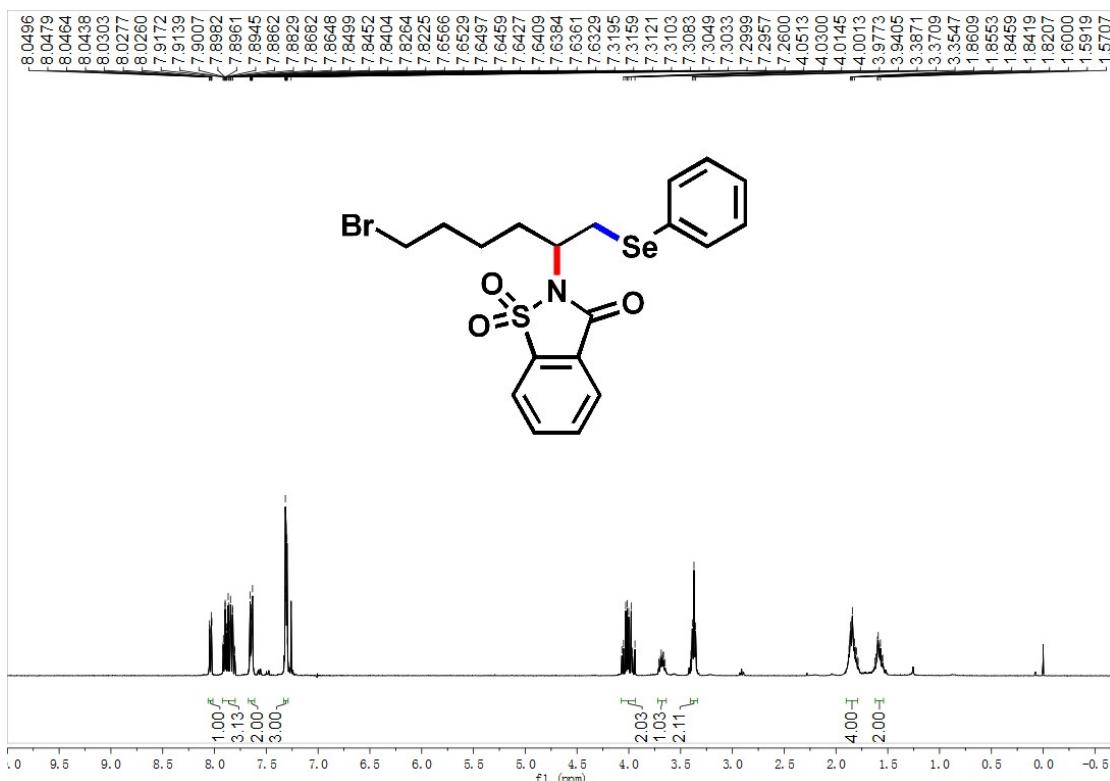
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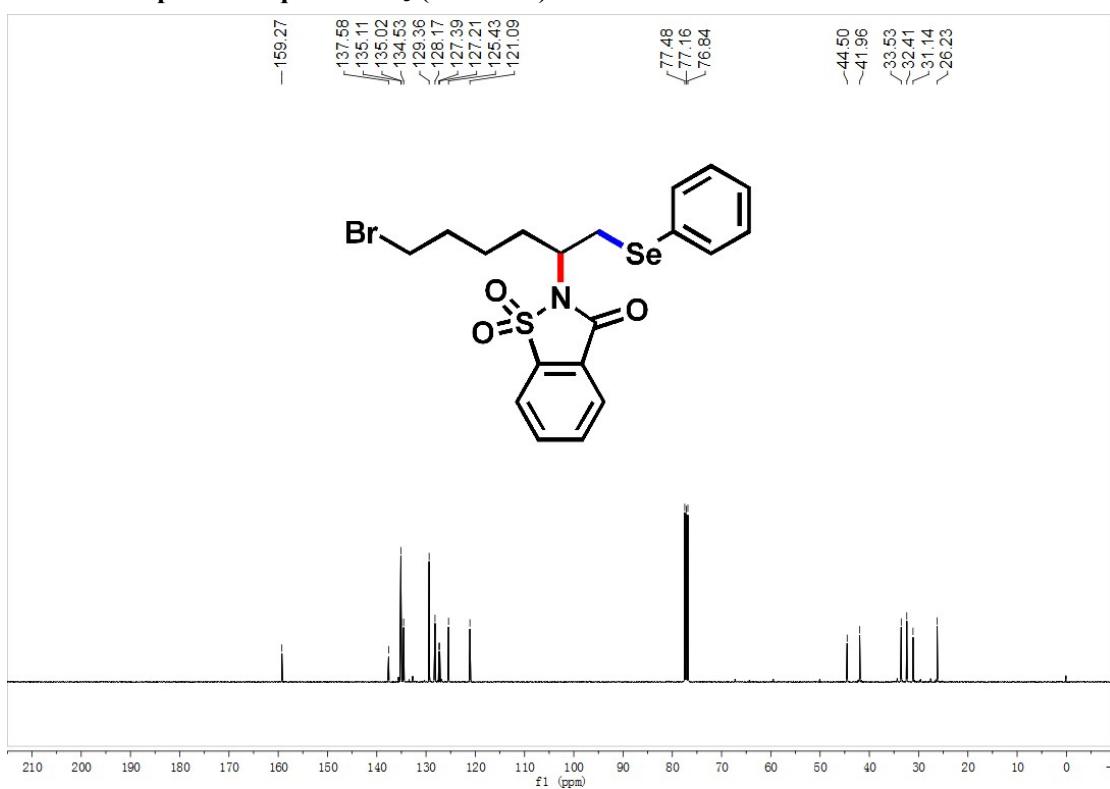
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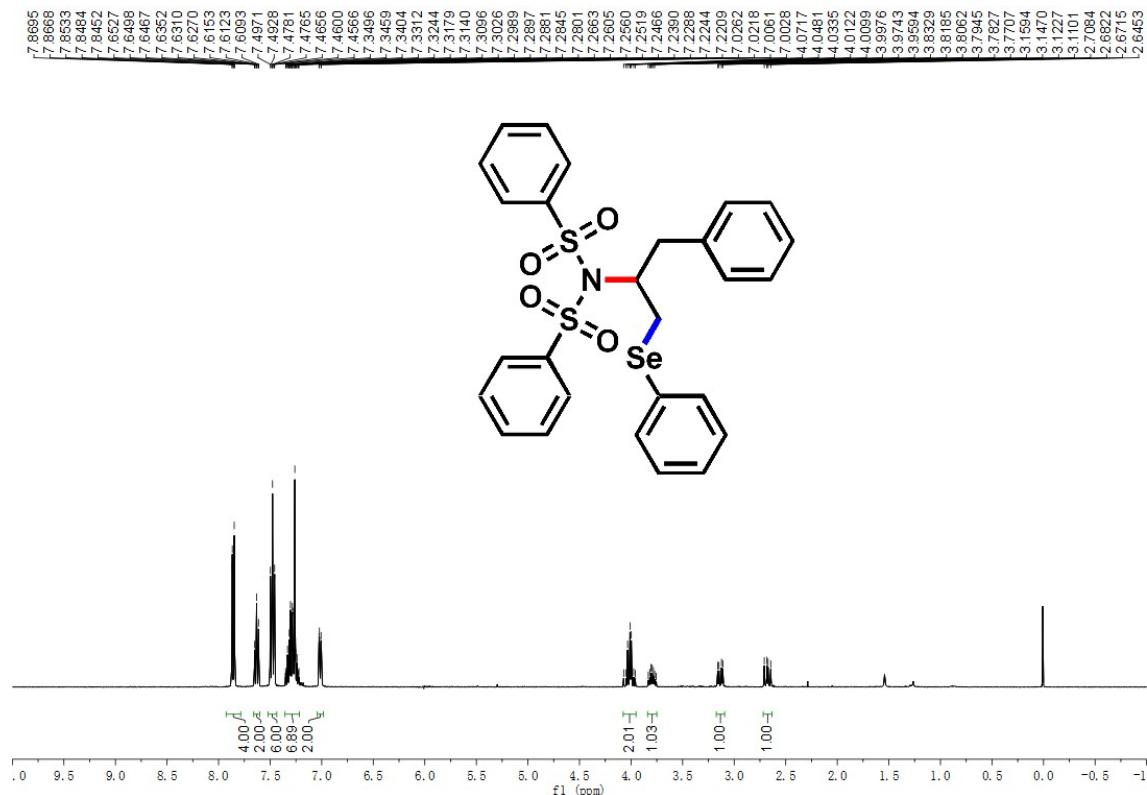
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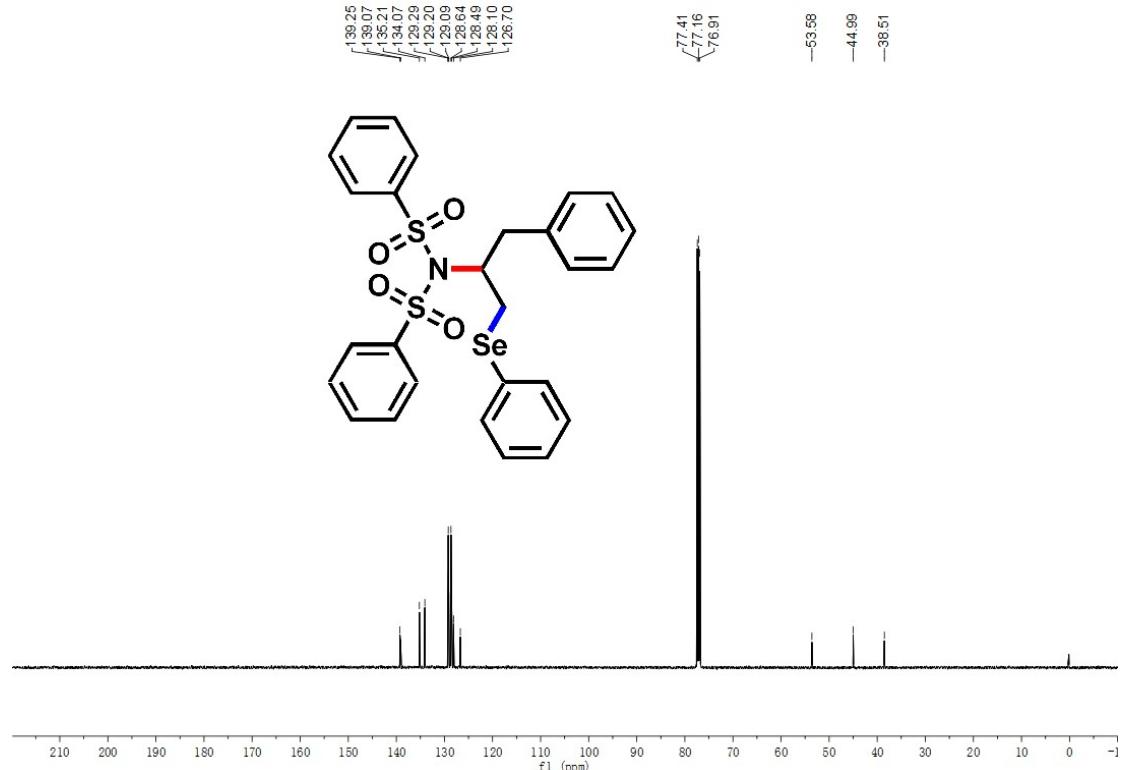
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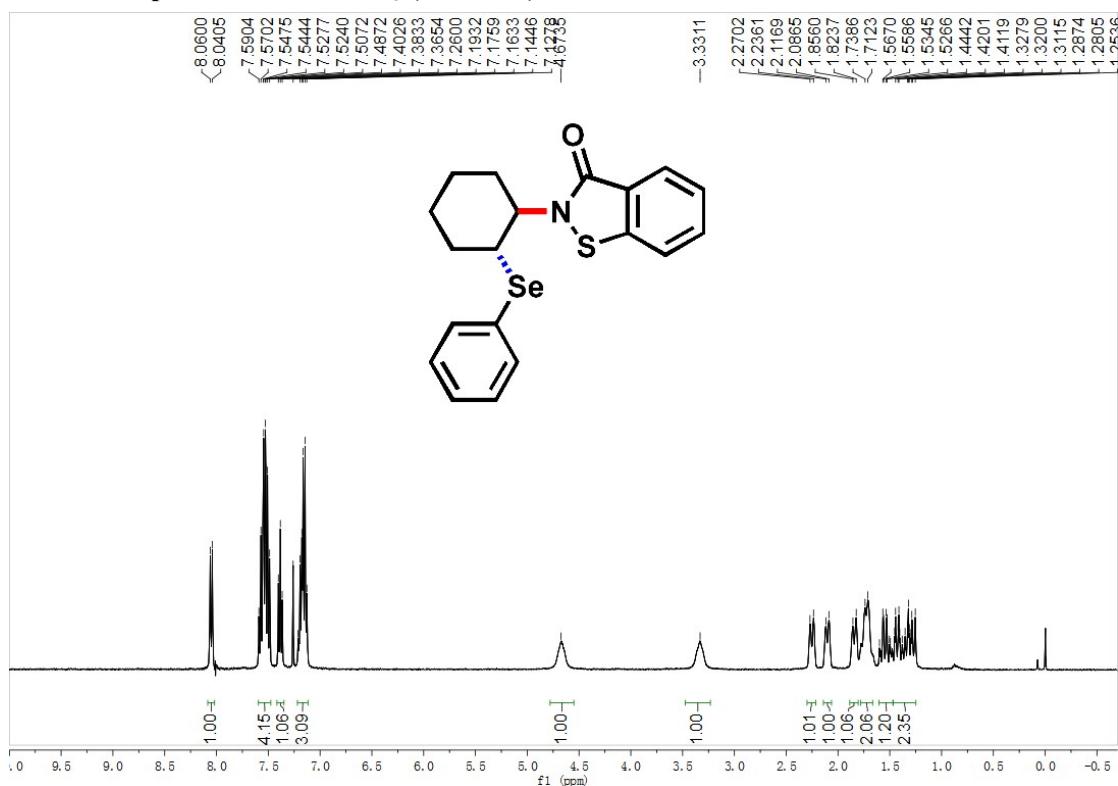
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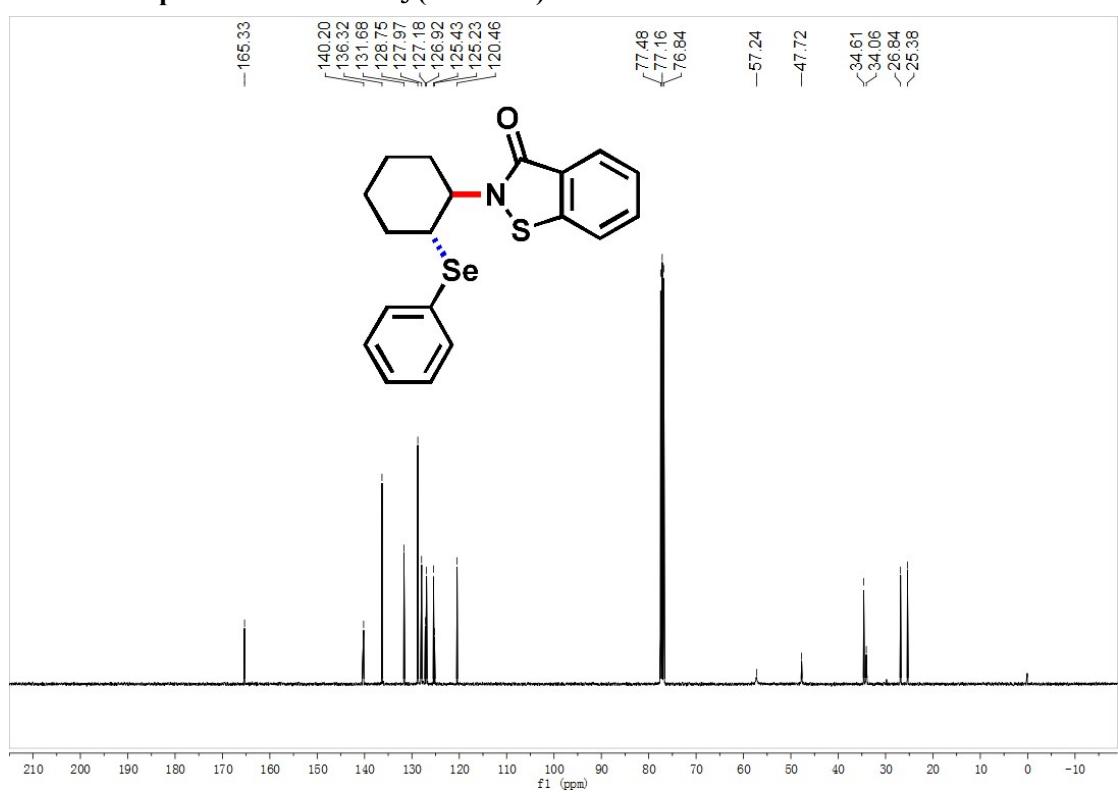
¹³C NMR of product 5r' in CDCl₃ (100 MHz)



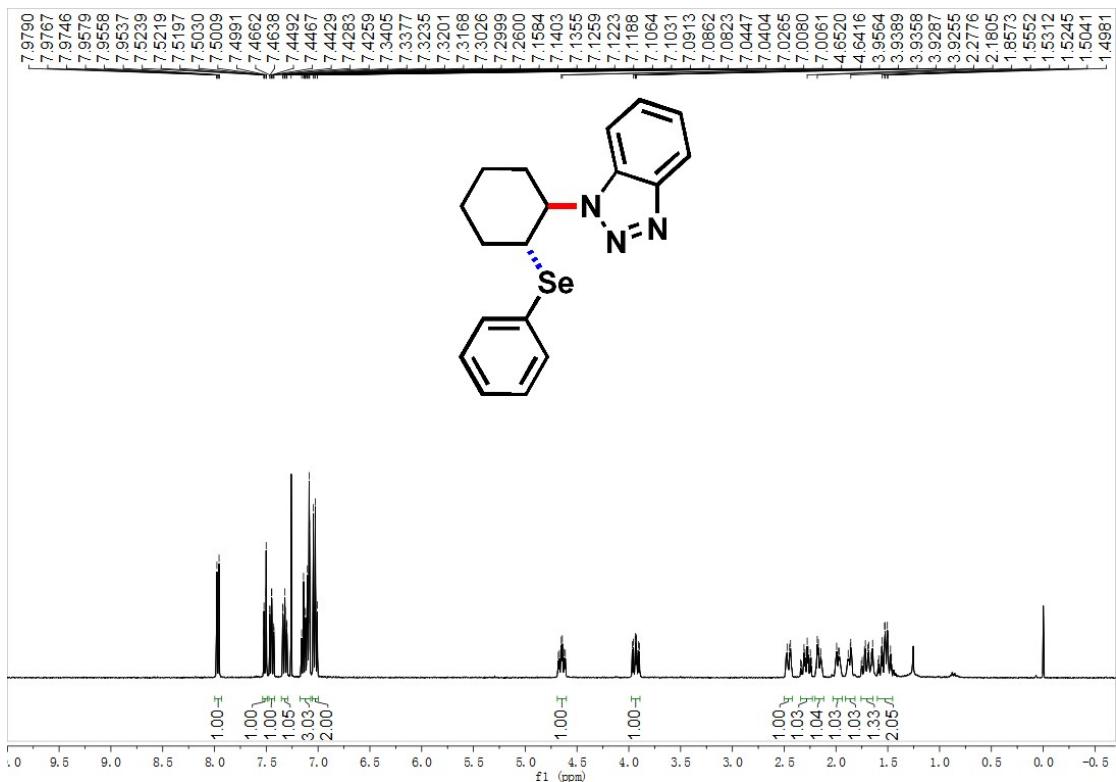
¹H NMR of product 5s' in CDCl₃ (400 MHz)



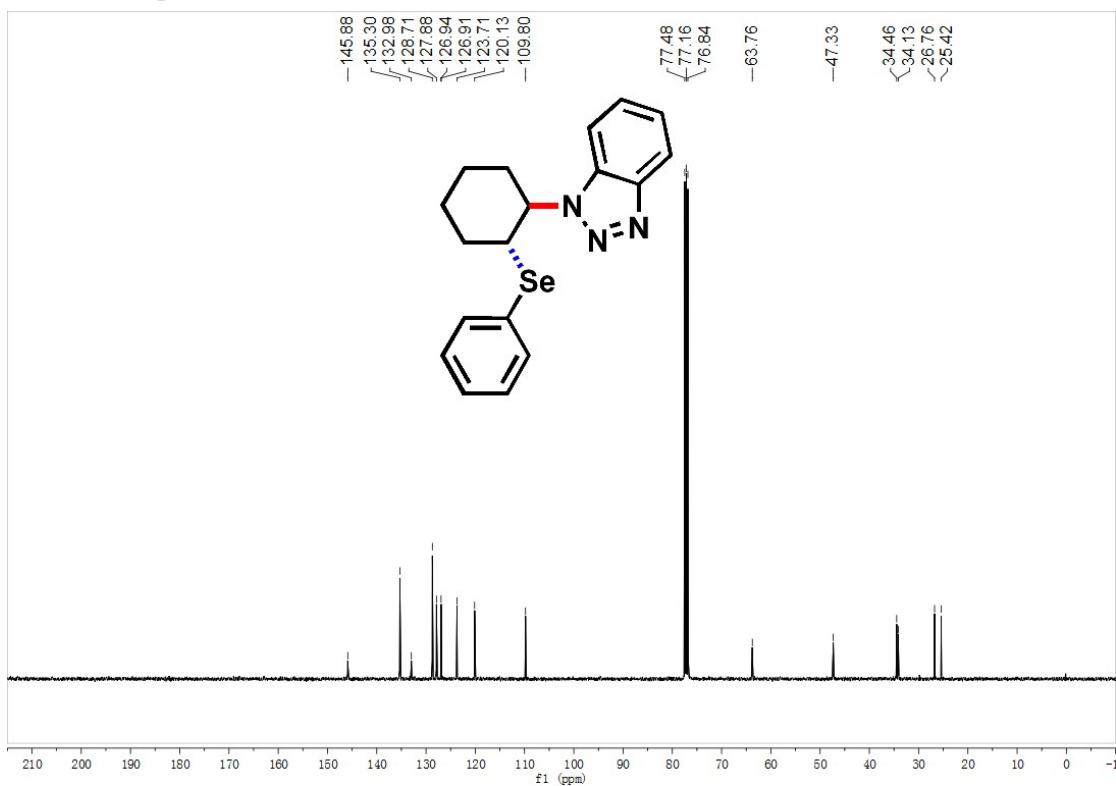
¹³C NMR of product 5s' in CDCl₃ (100 MHz)



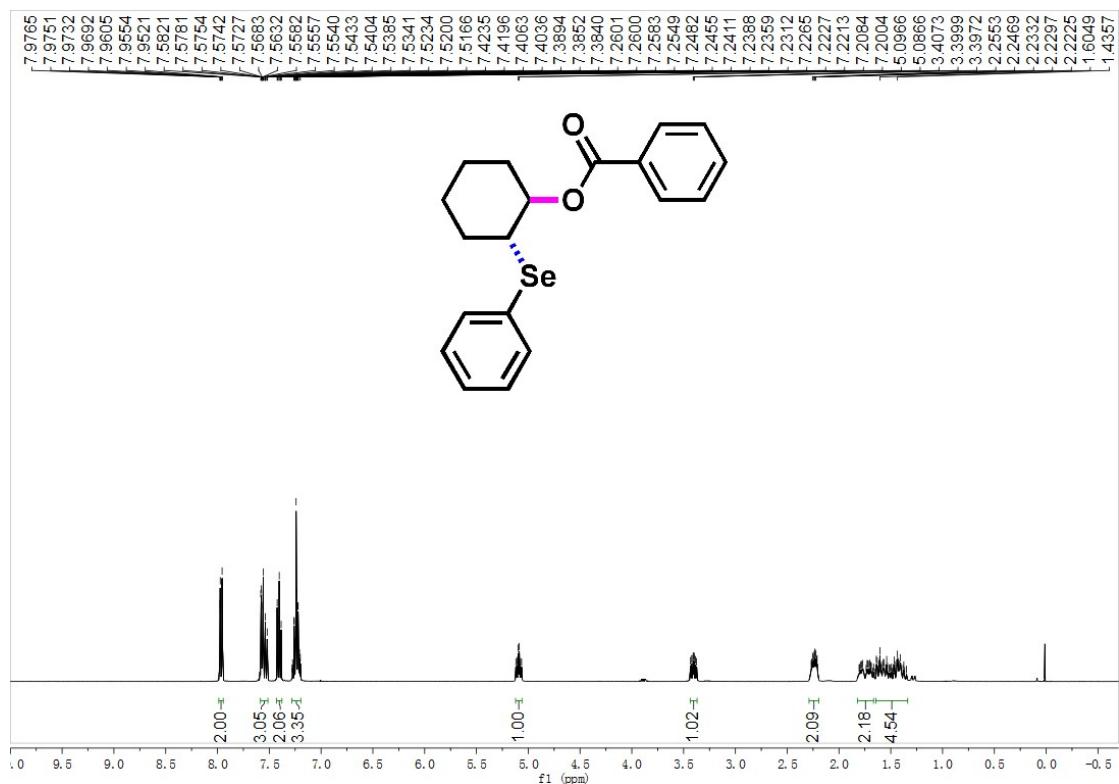
¹H NMR of product 5t' in CDCl₃ (400 MHz)



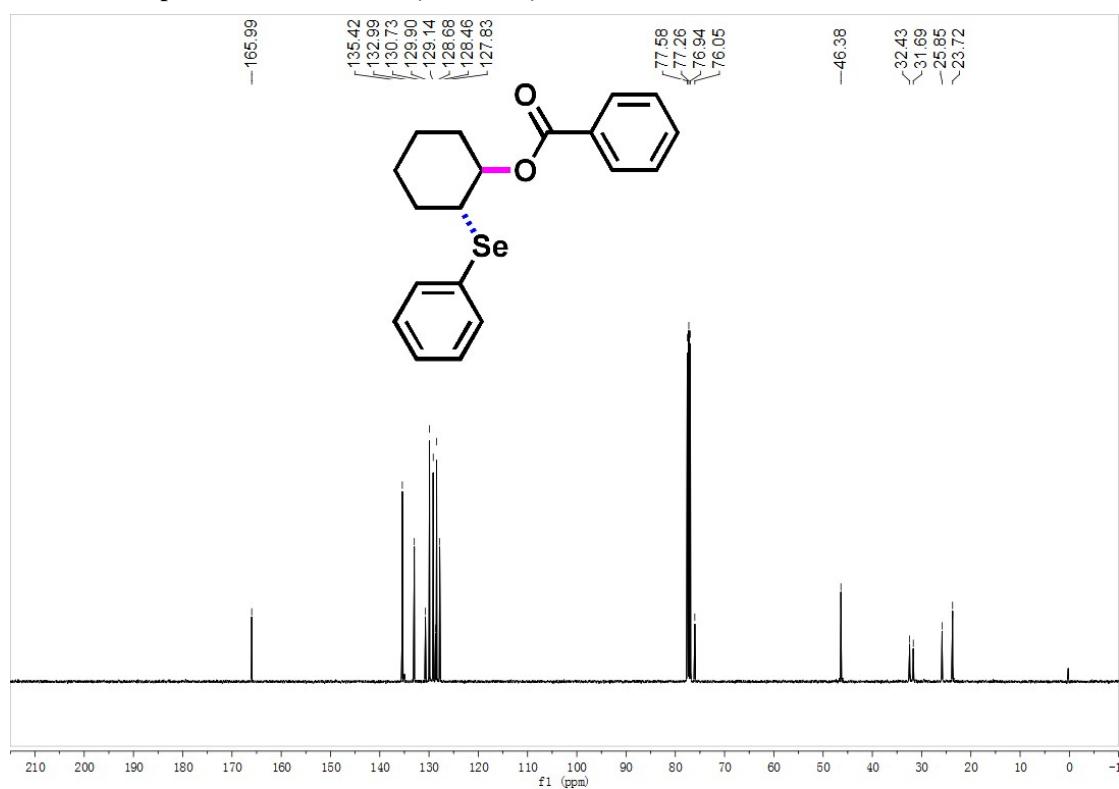
¹³C NMR of product 5t' in CDCl₃ (100 MHz)



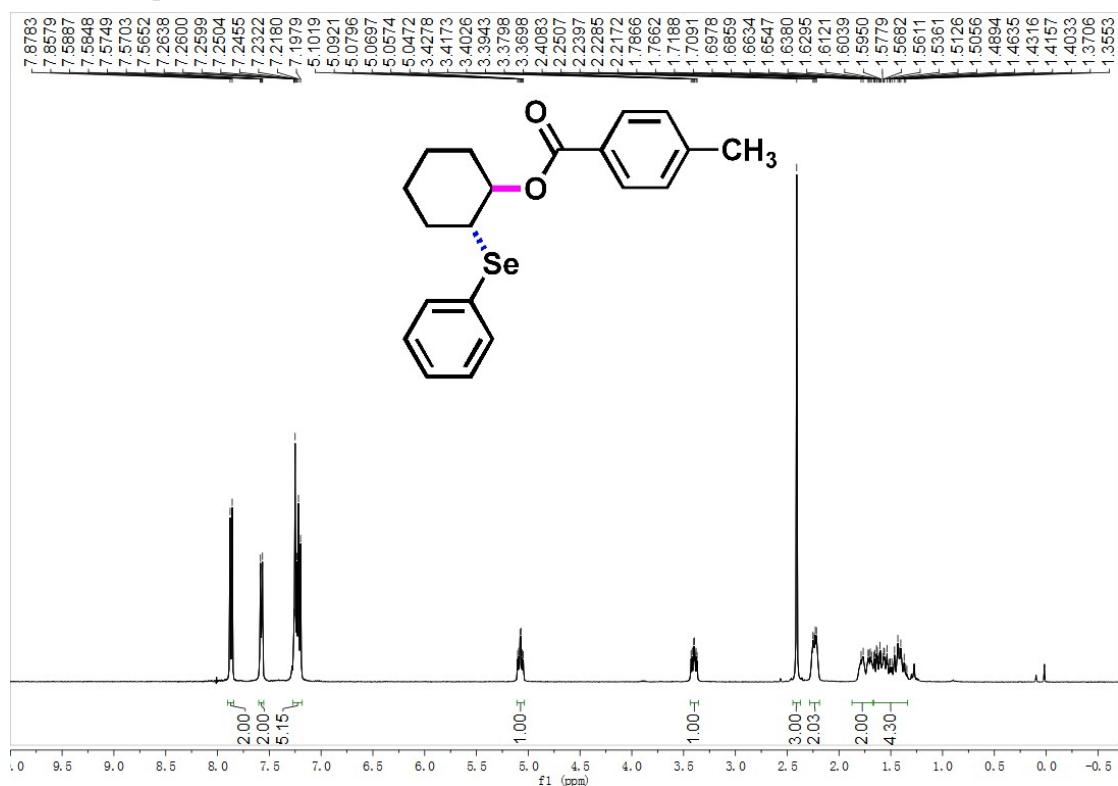
¹H NMR of product 7a in CDCl₃ (400 MHz)



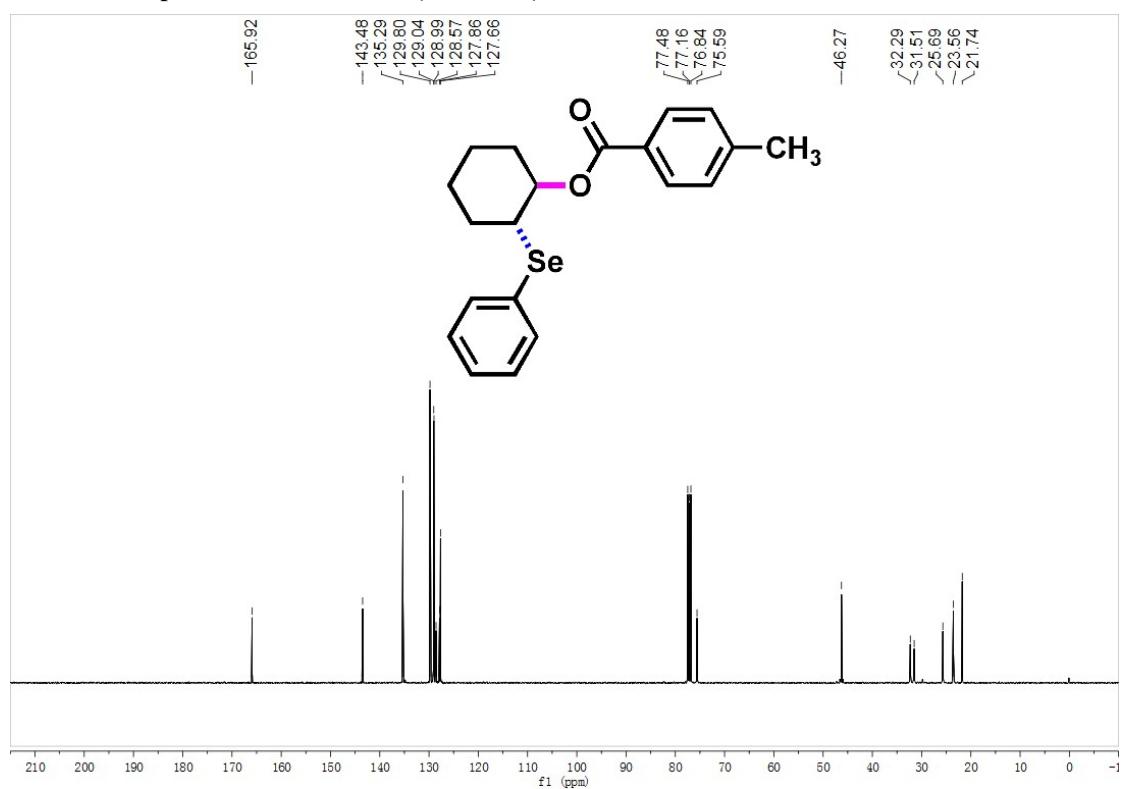
¹³C NMR of product 7a in CDCl₃ (100 MHz)



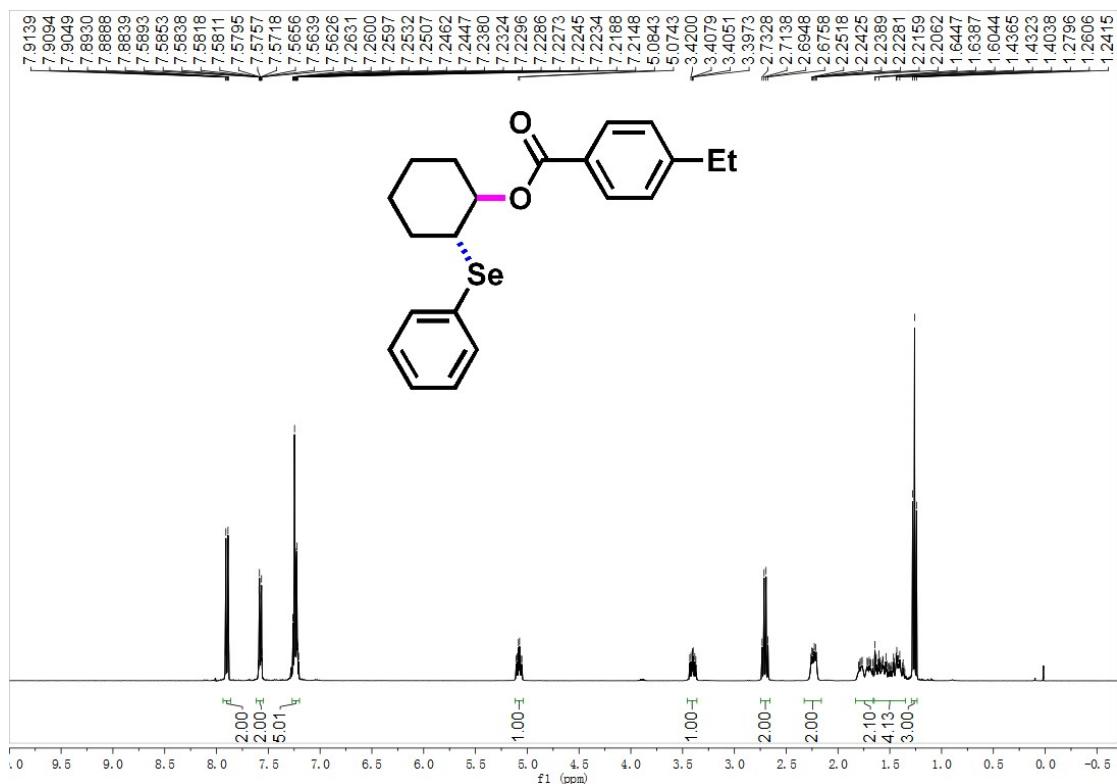
¹H NMR of product 7b in CDCl₃ (400 MHz)



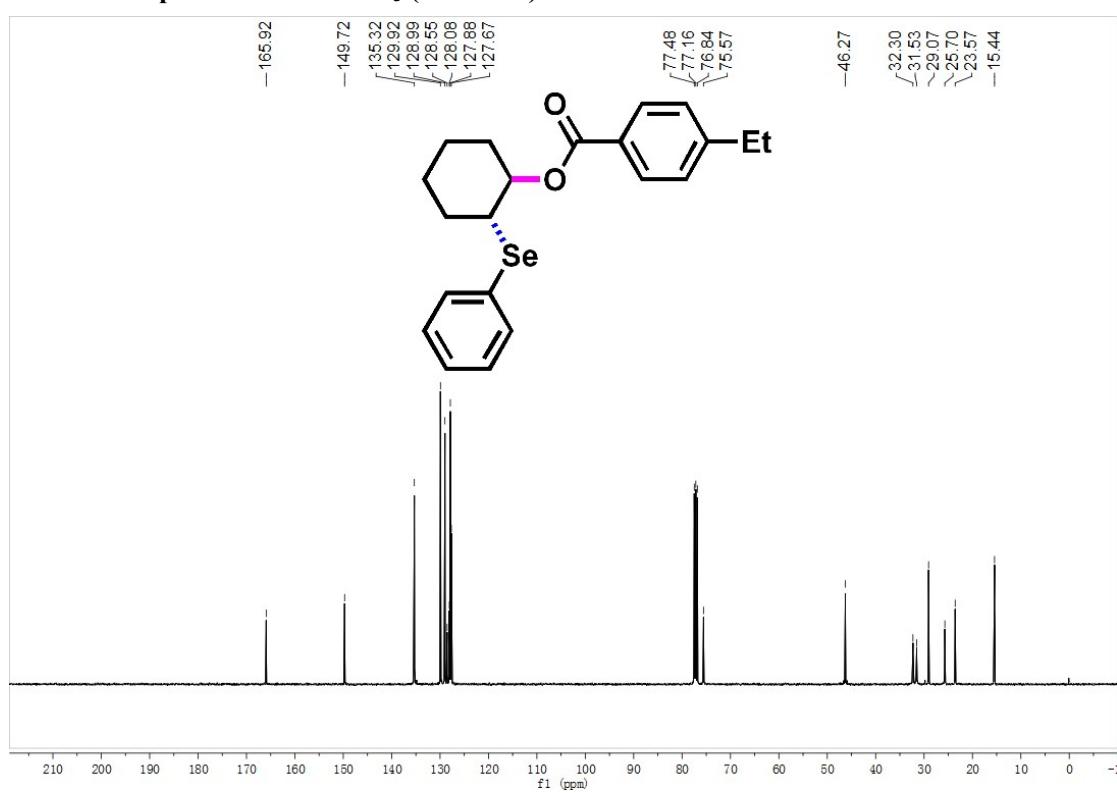
¹³C NMR of product 7b in CDCl₃ (100 MHz)



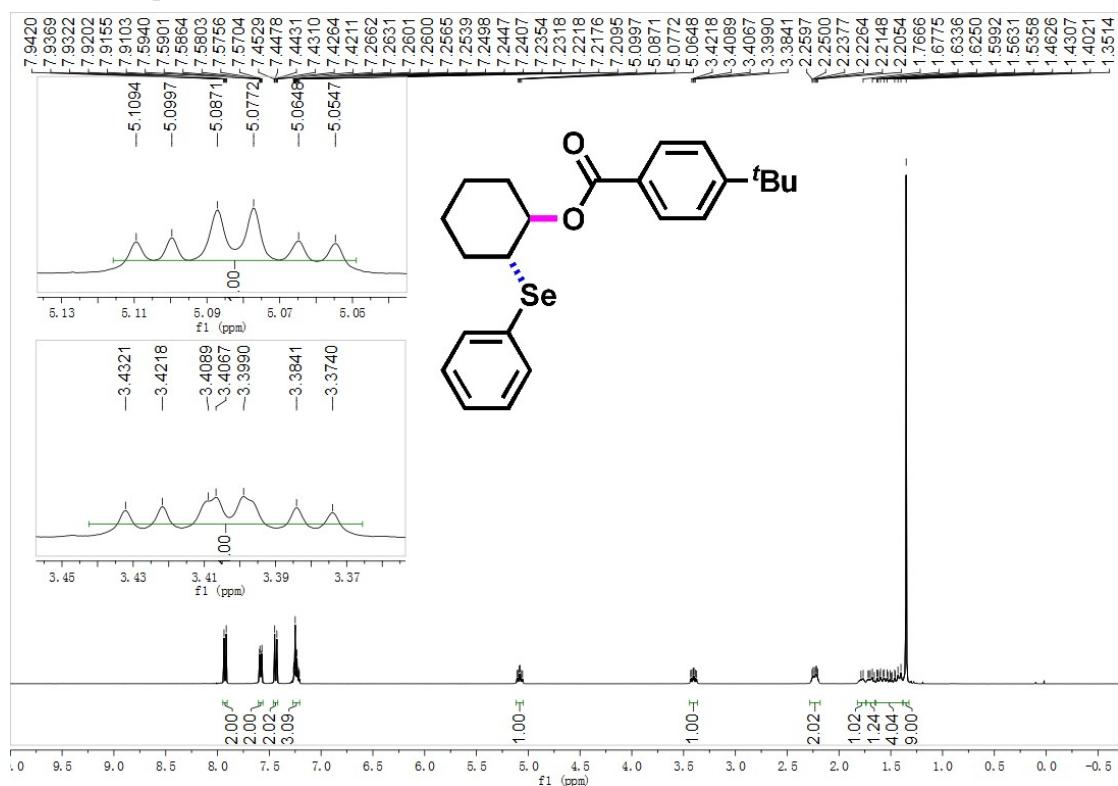
¹H NMR of product 7c in CDCl₃ (400 MHz)



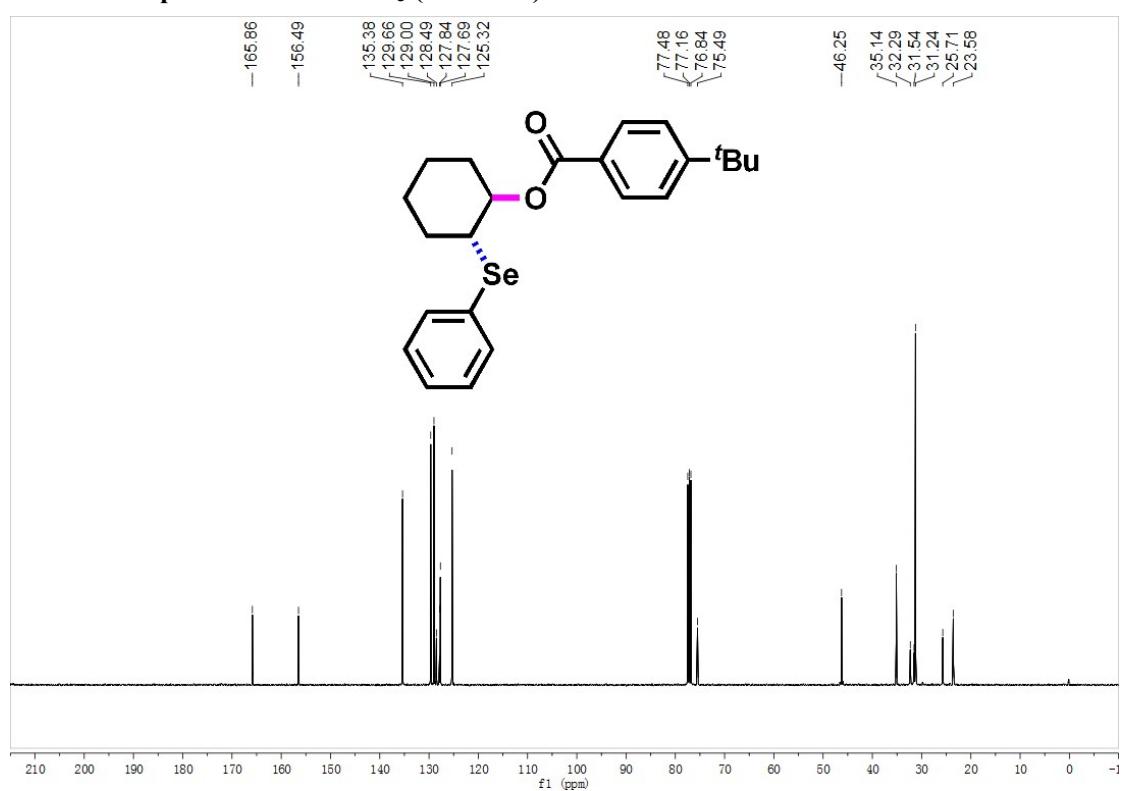
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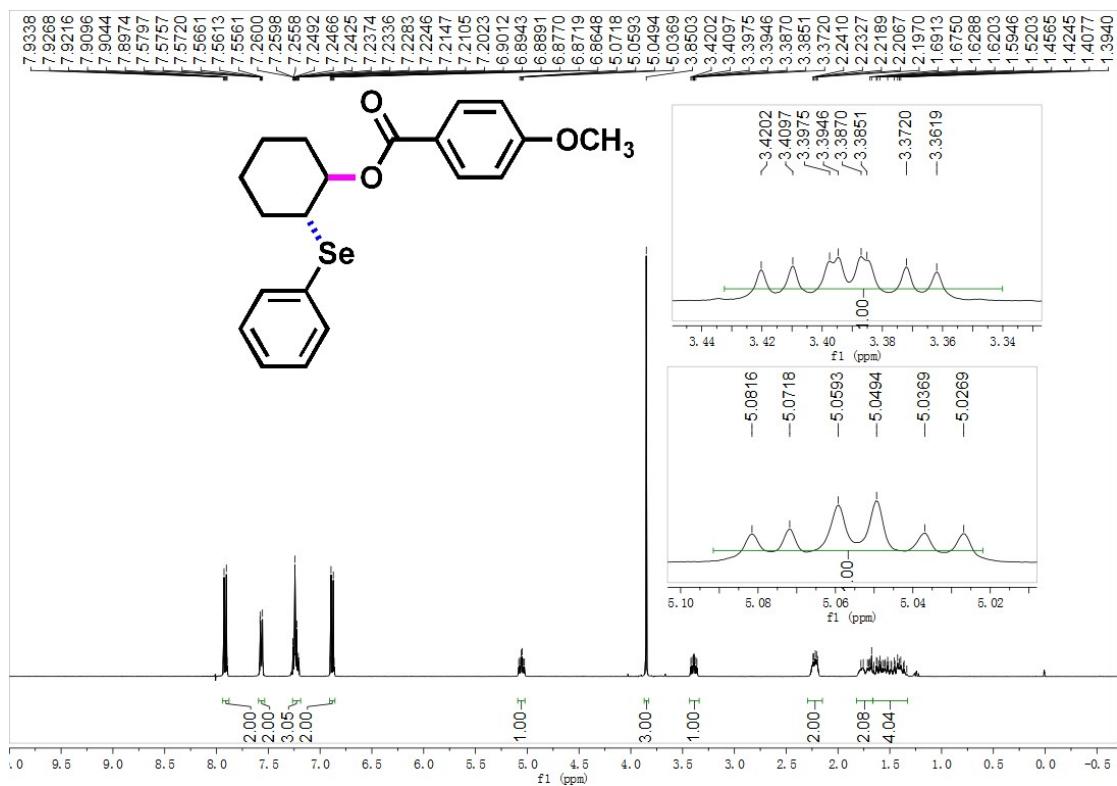
¹H NMR of product 7d in CDCl₃ (400 MHz)



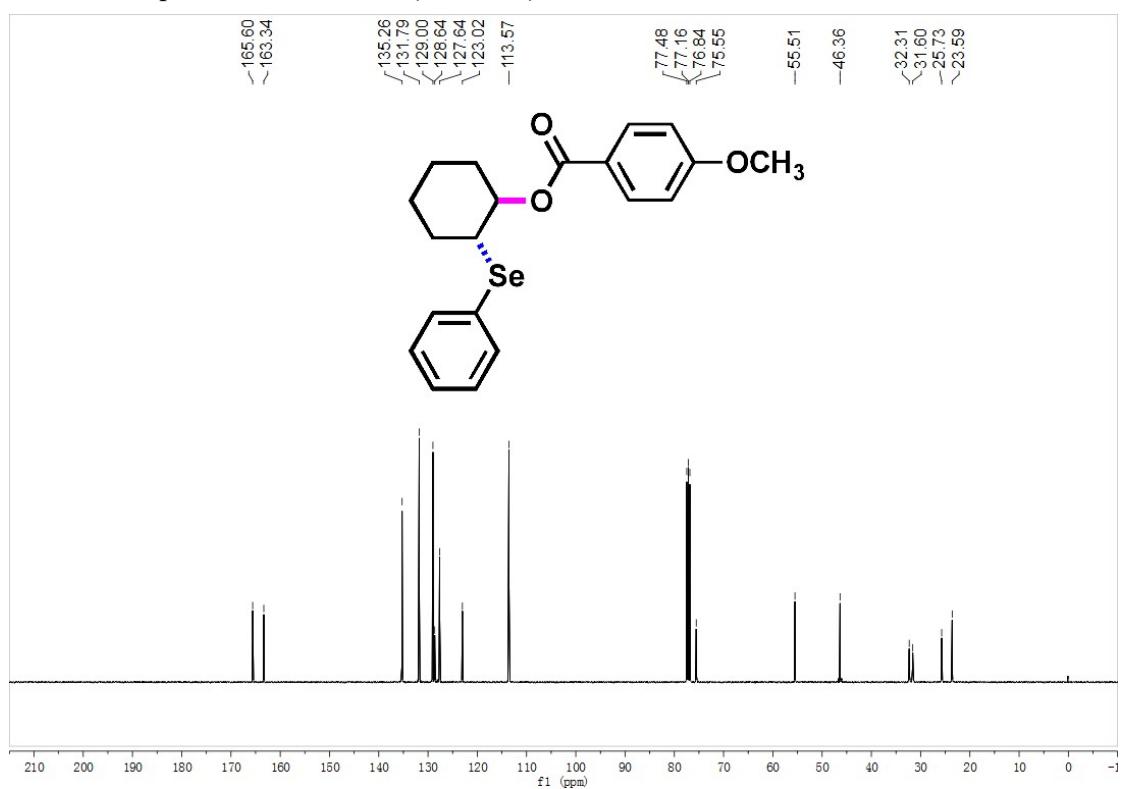
¹³C NMR of product 7d in CDCl₃ (100 MHz)



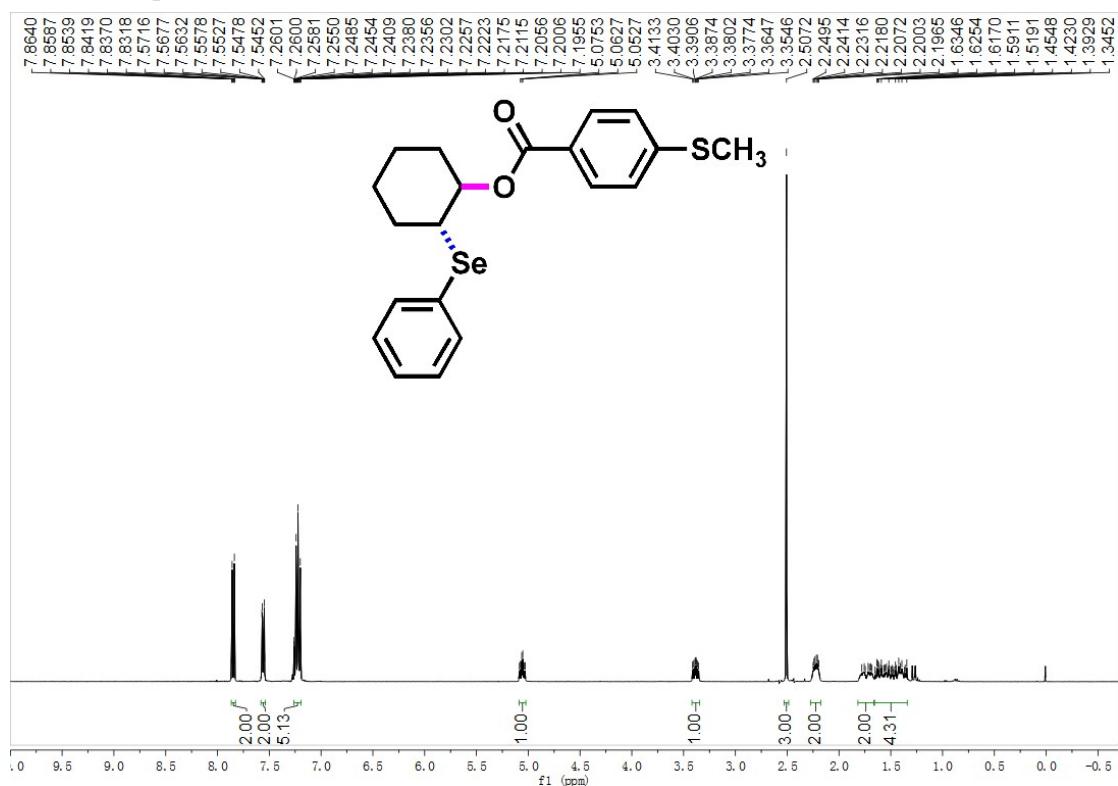
¹H NMR of product 7e in CDCl₃ (400 MHz)



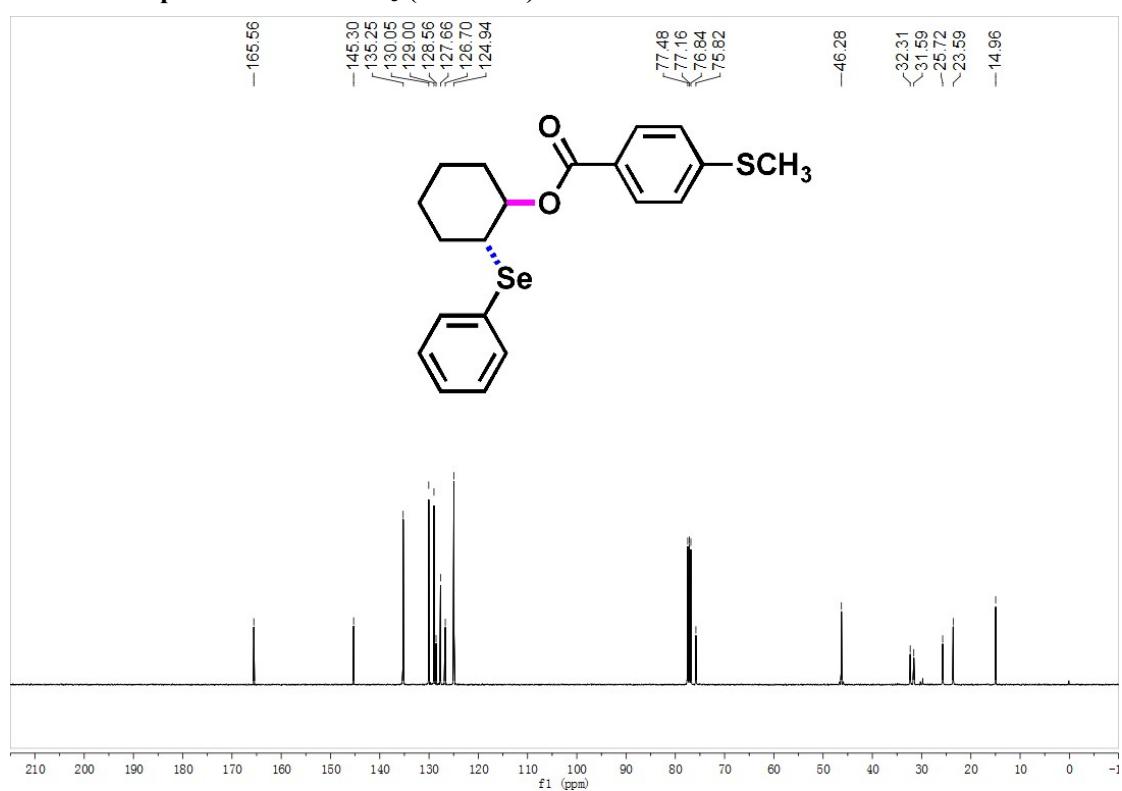
¹³C NMR of product 7e in CDCl₃ (100 MHz)



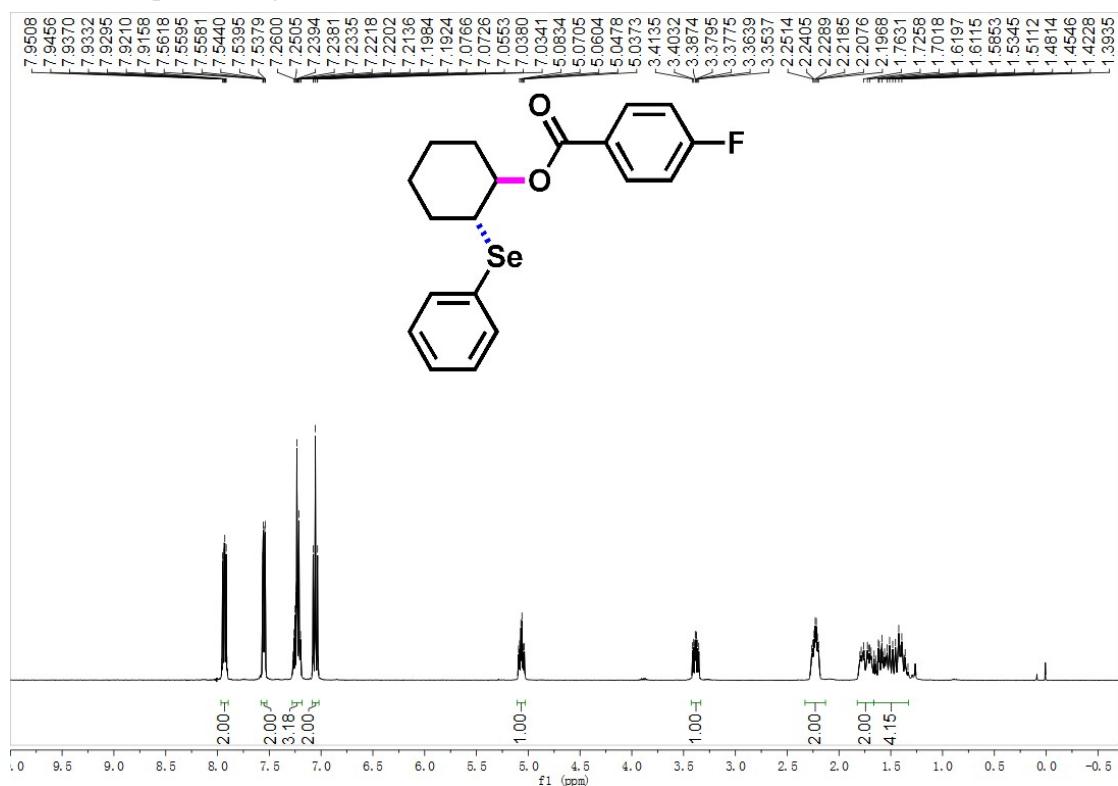
¹H NMR of product 7f in CDCl₃ (400 MHz)



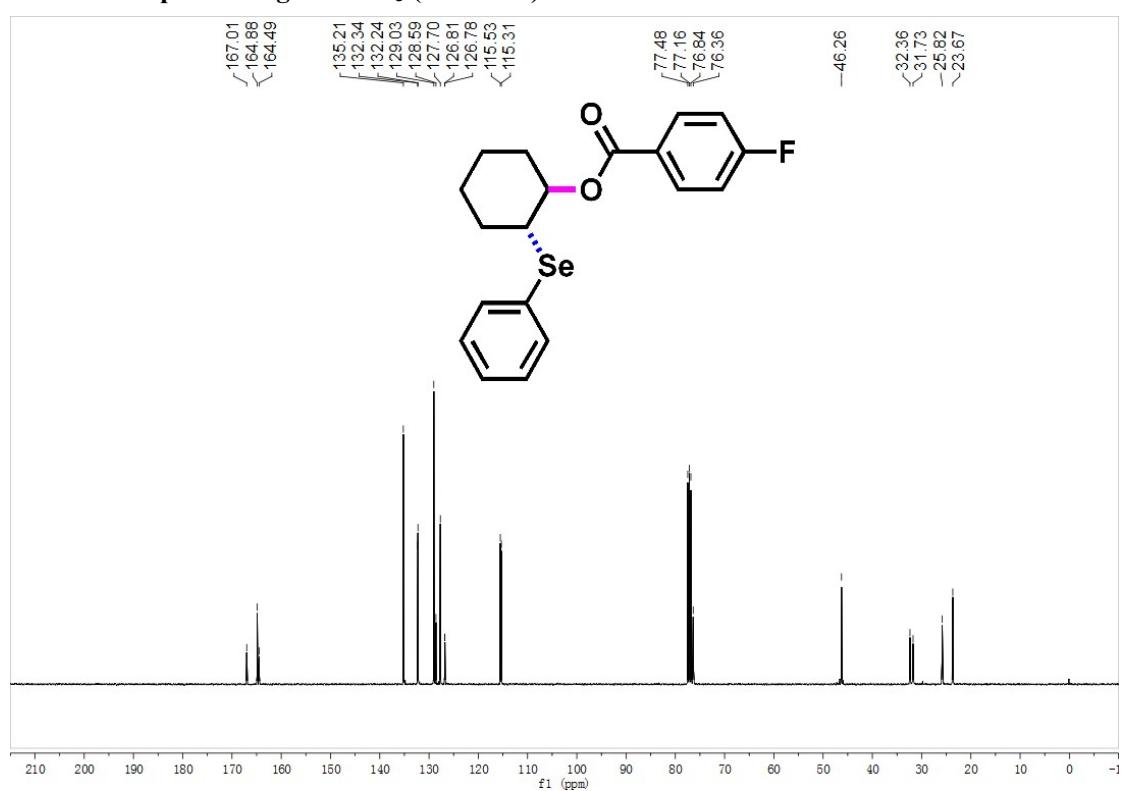
¹³C NMR of product 7f in CDCl₃ (100 MHz)



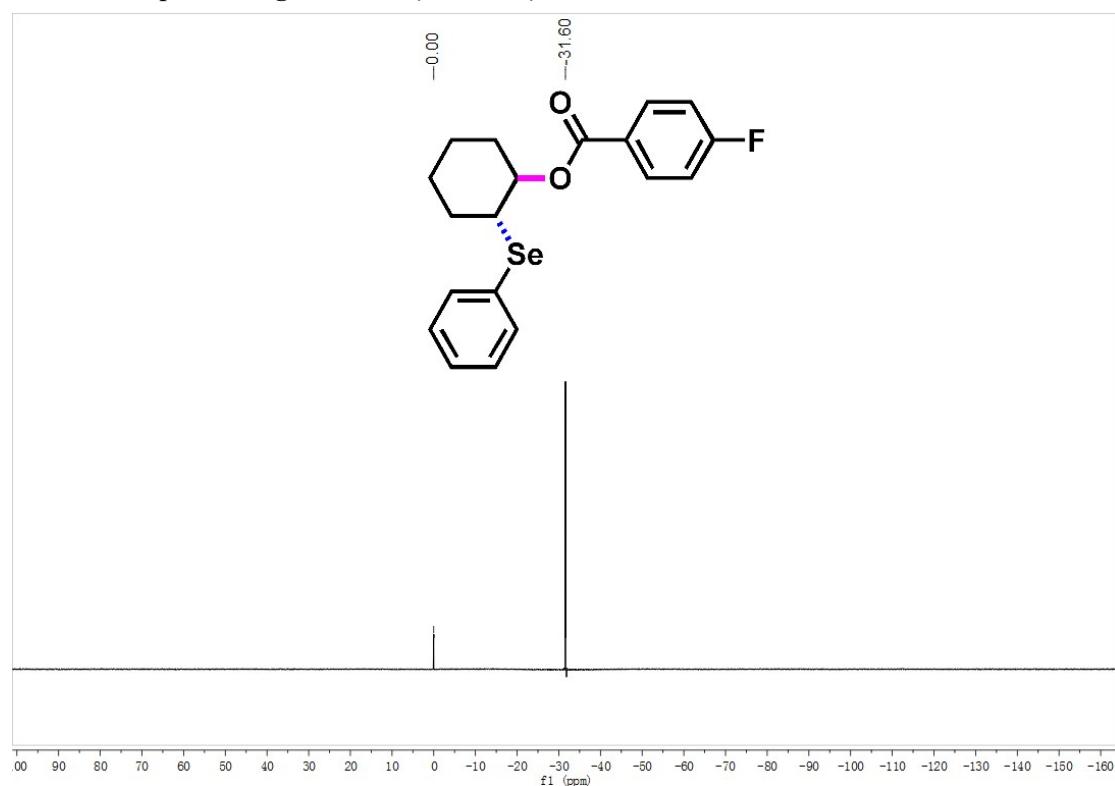
¹H NMR of product 7g in CDCl₃ (400 MHz)



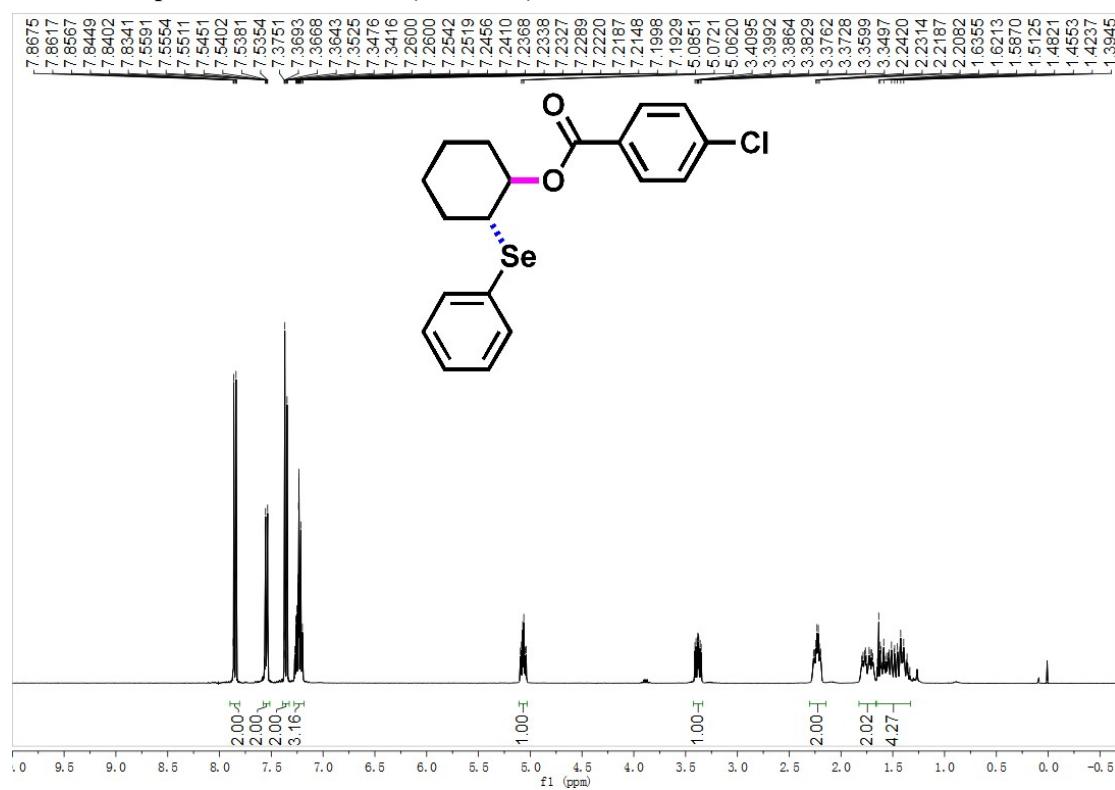
¹³C NMR of product 7g in CDCl₃ (100 MHz)



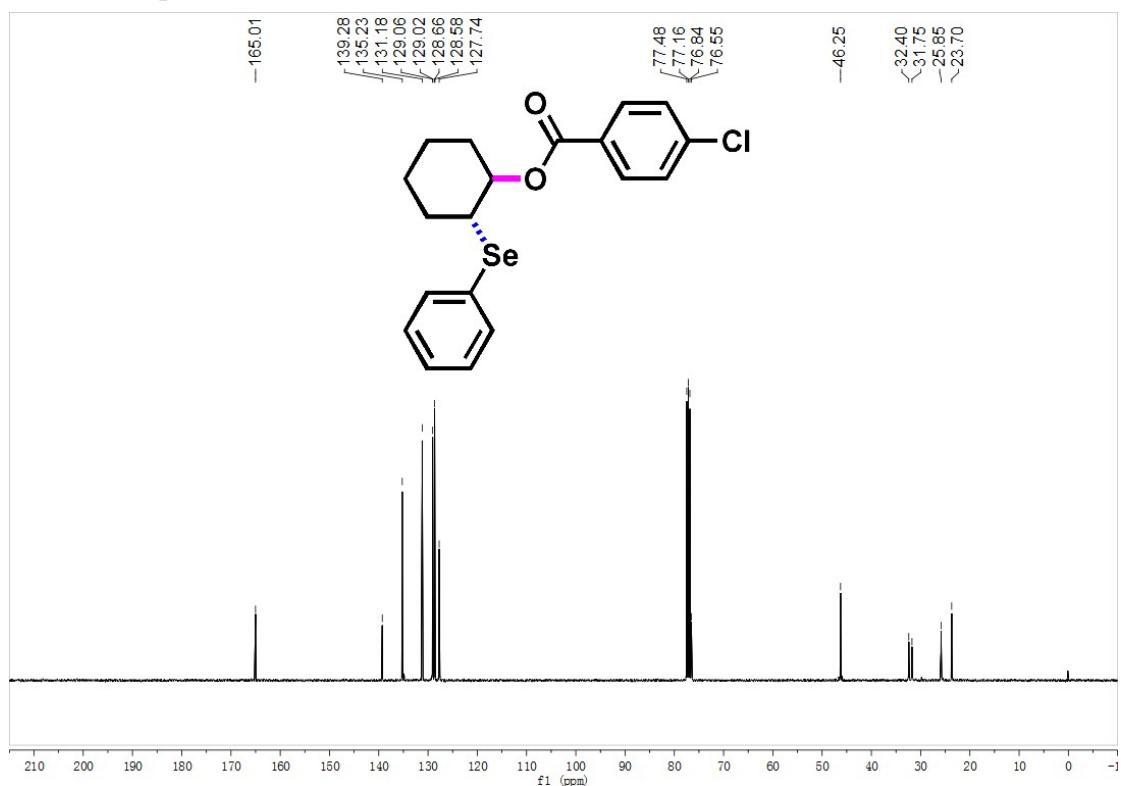
¹⁹F NMR of product 7g in CDCl₃ (376 MHz)



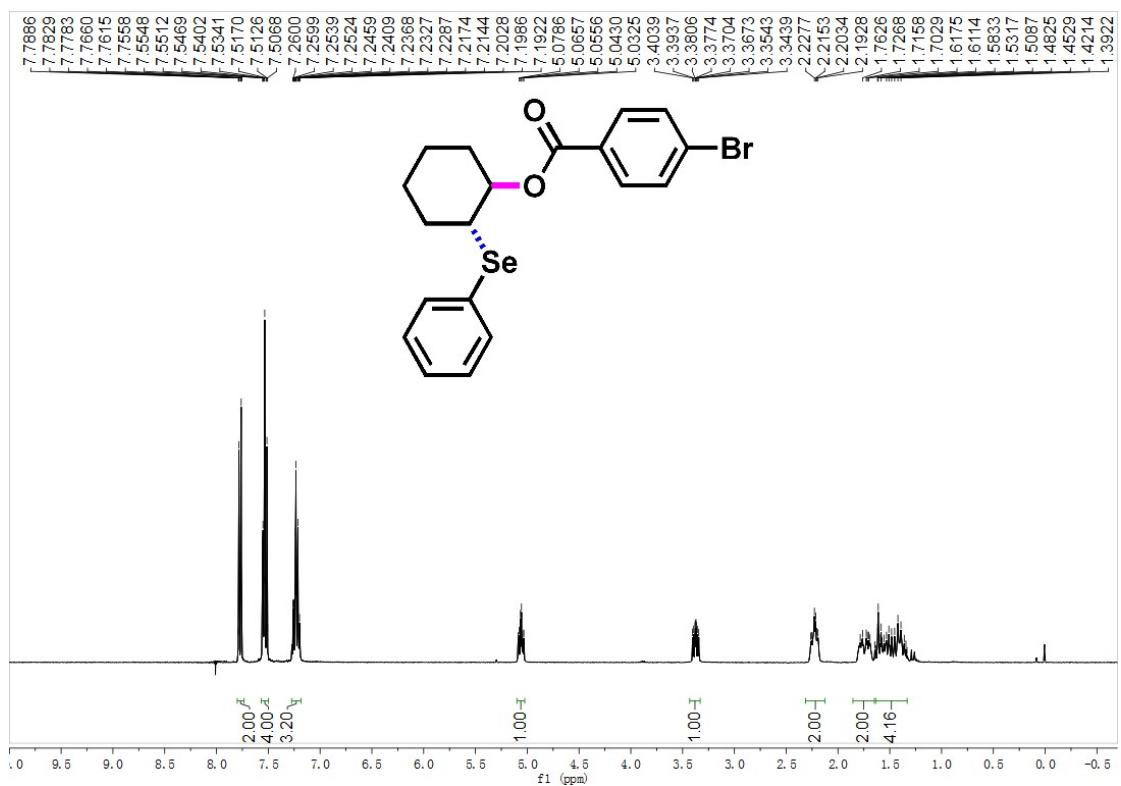
¹H NMR of product 7h in CDCl₃ (400 MHz)



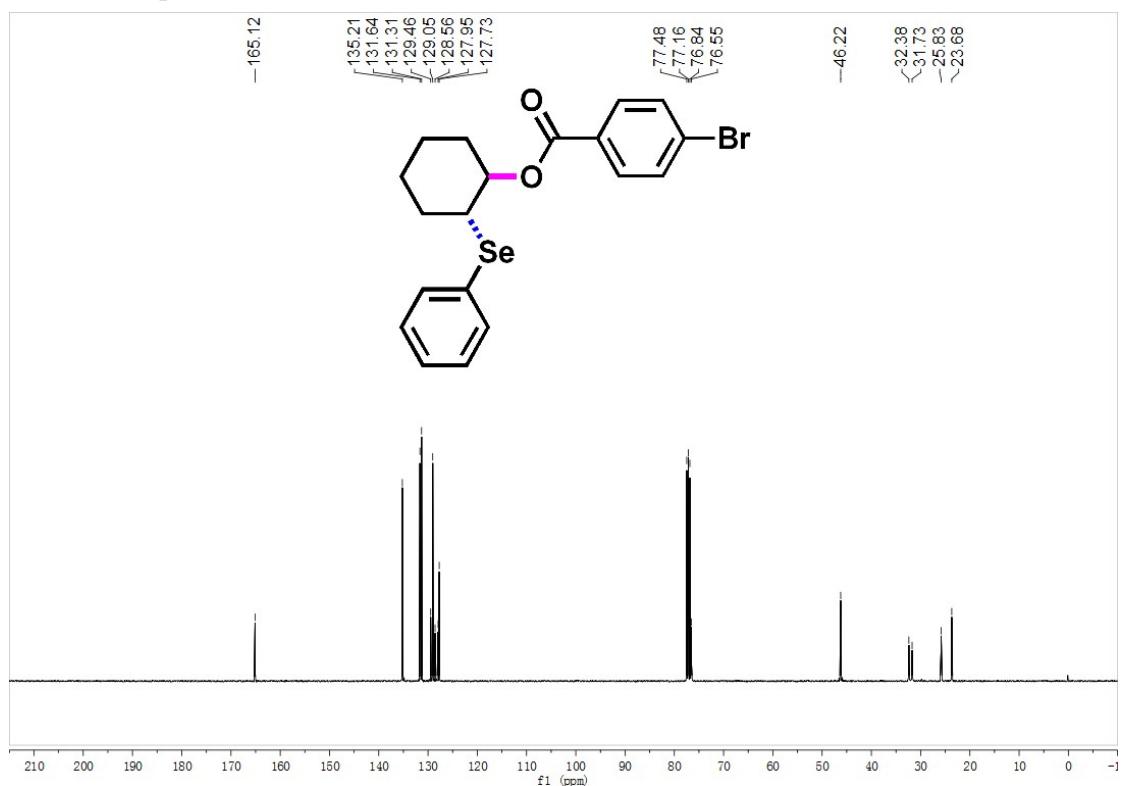
¹³C NMR of product 7h in CDCl₃ (100 MHz)



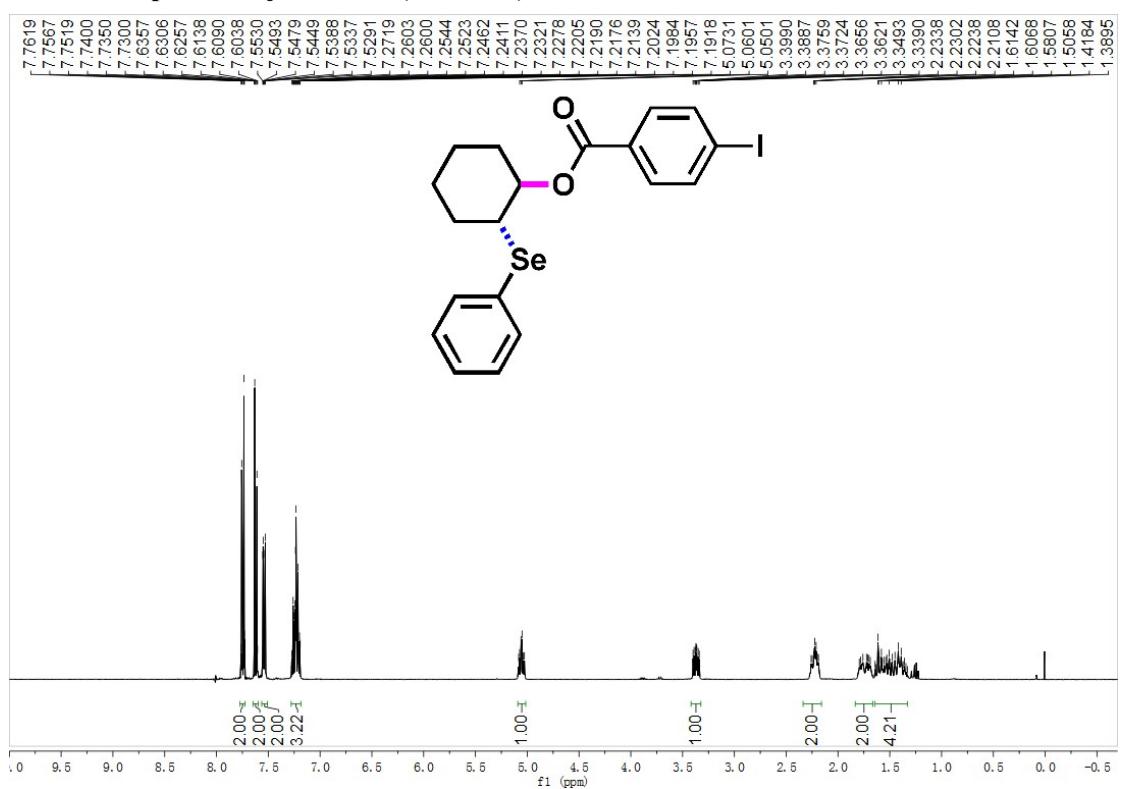
¹H NMR of product 7i in CDCl₃ (400 MHz)



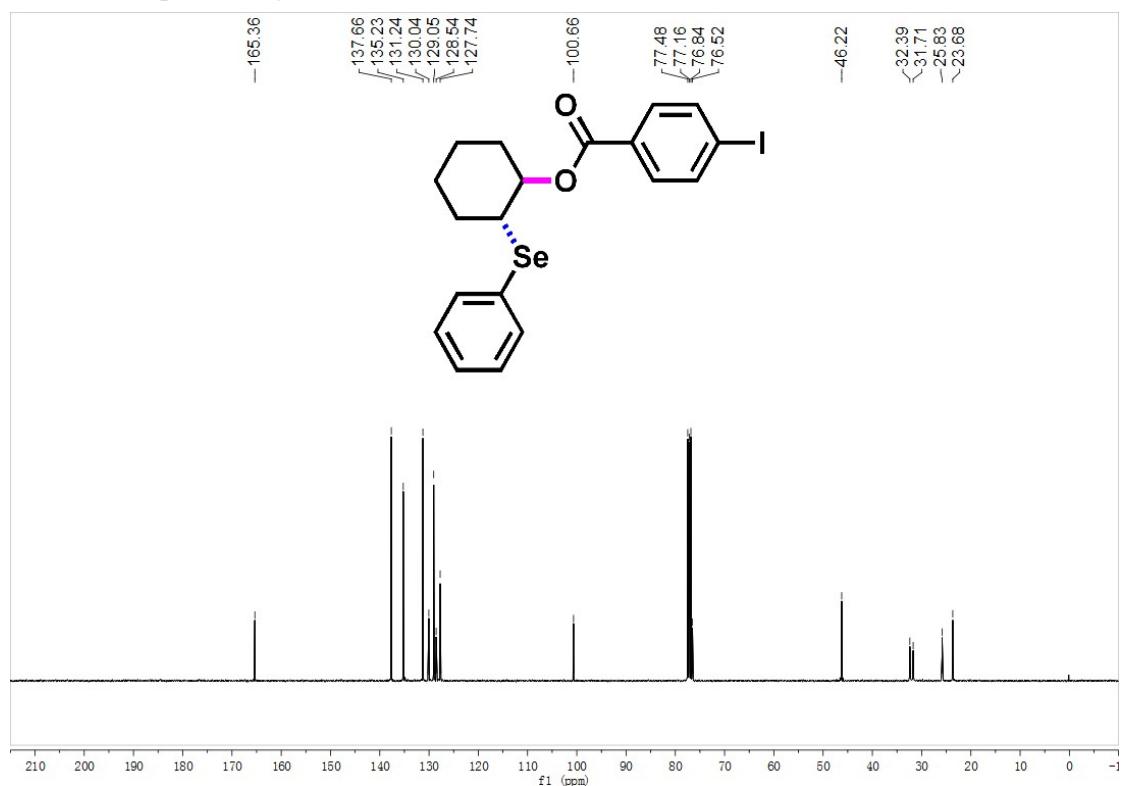
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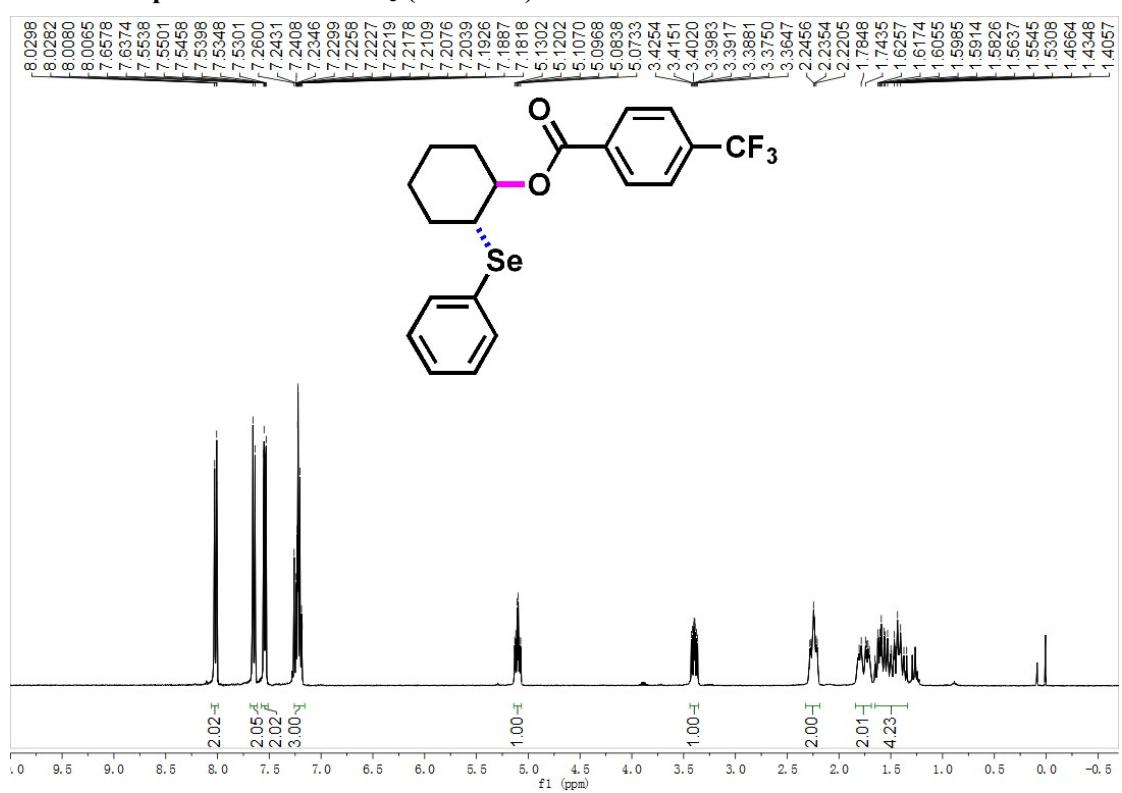
¹H NMR of product 7j in CDCl₃ (400 MHz)



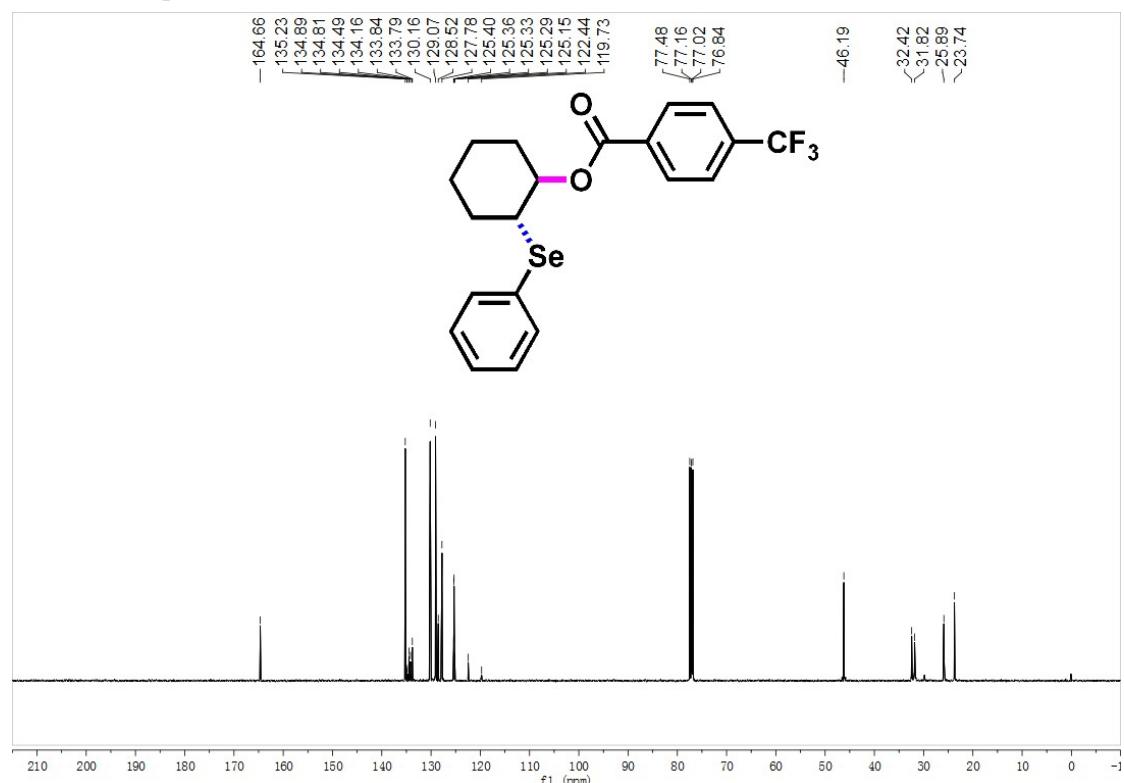
¹³C NMR of product 7j in CDCl₃ (100 MHz)



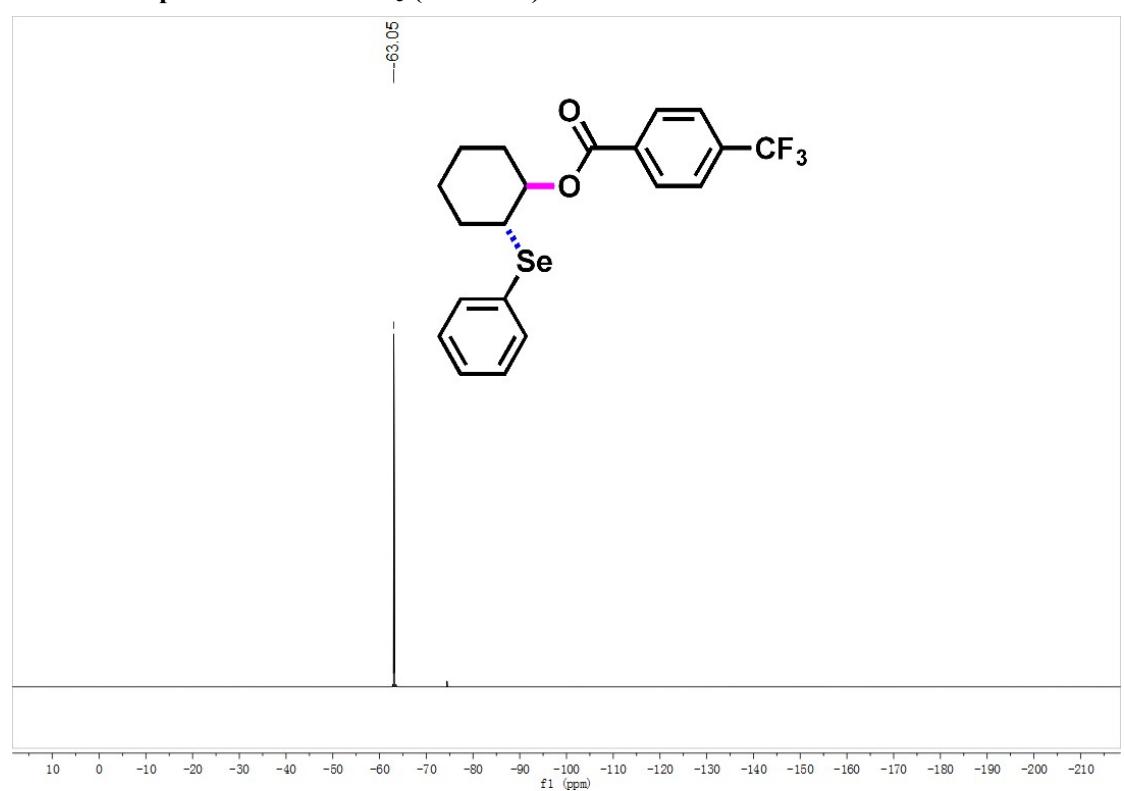
¹H NMR of product 7k in CDCl₃ (400 MHz)



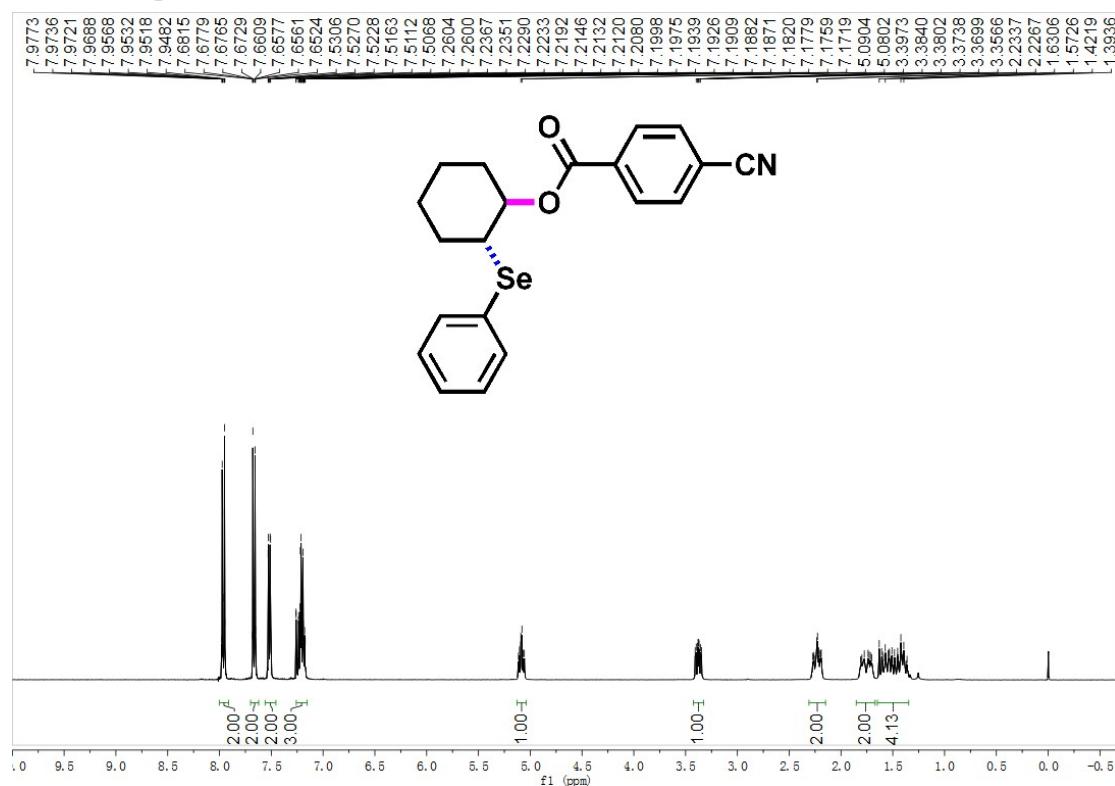
¹³C NMR of product 7k in CDCl₃ (100 MHz)



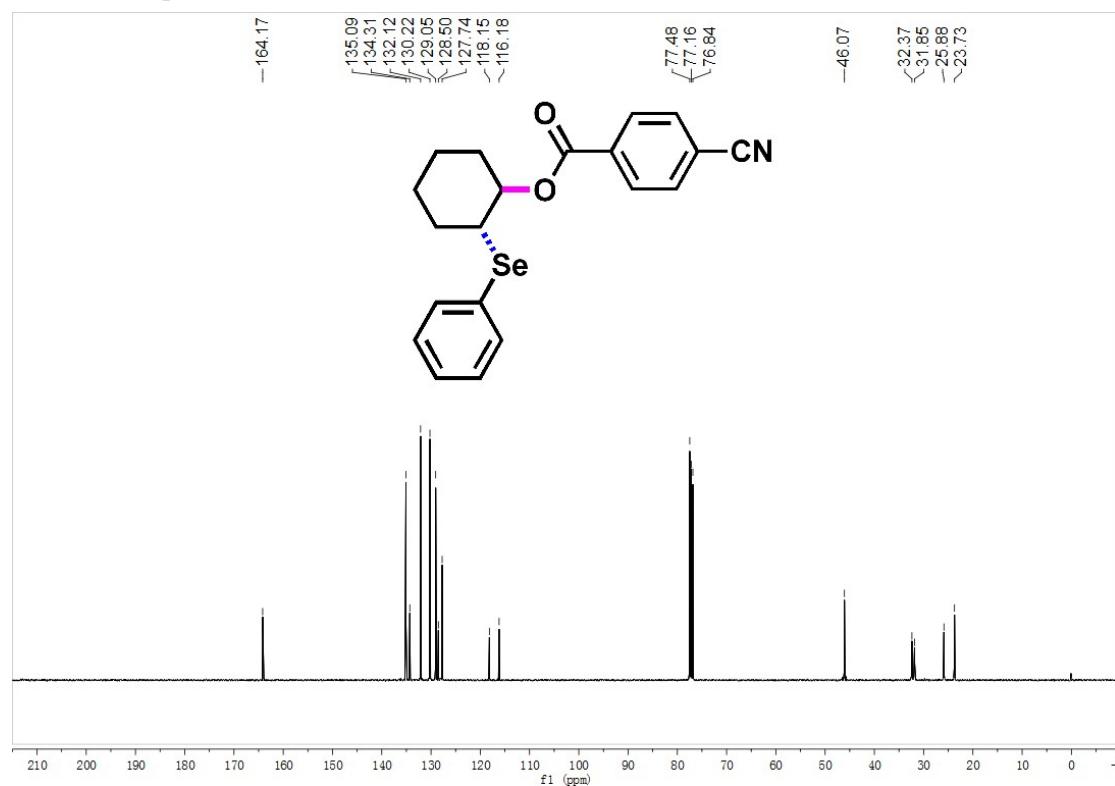
¹⁹F NMR of product 7k in CDCl₃ (376 MHz)



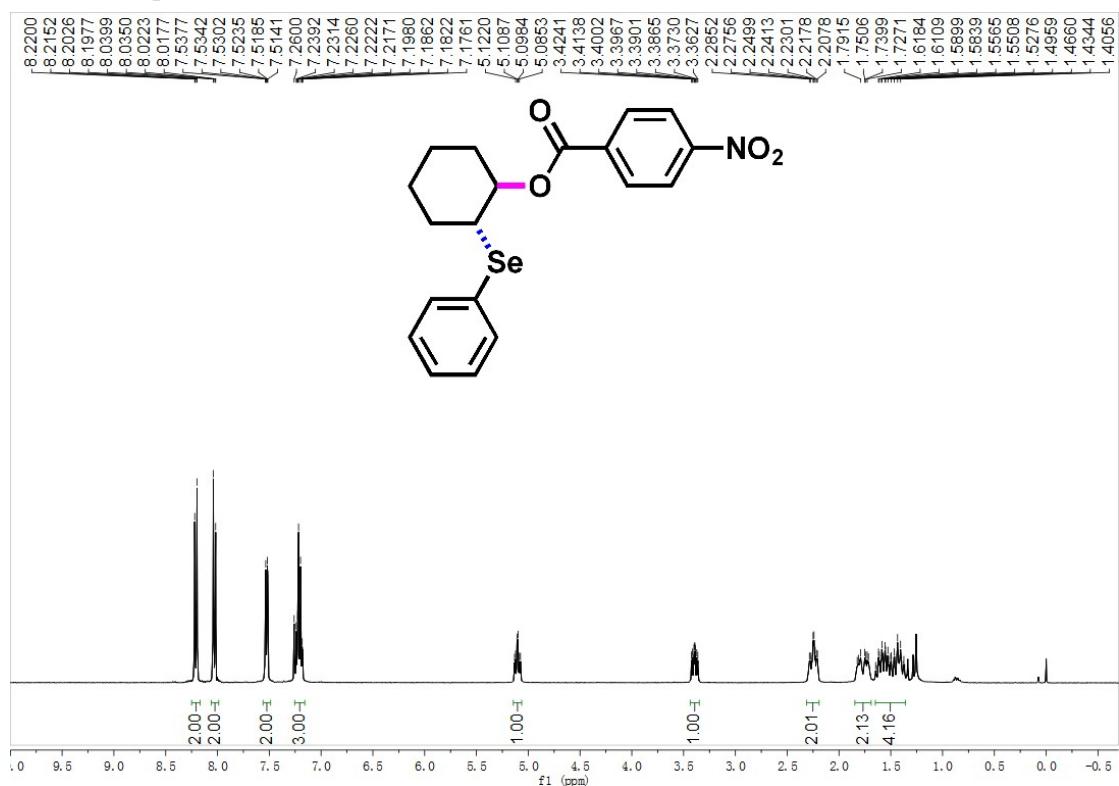
¹H NMR of product 7l in CDCl₃ (400 MHz)



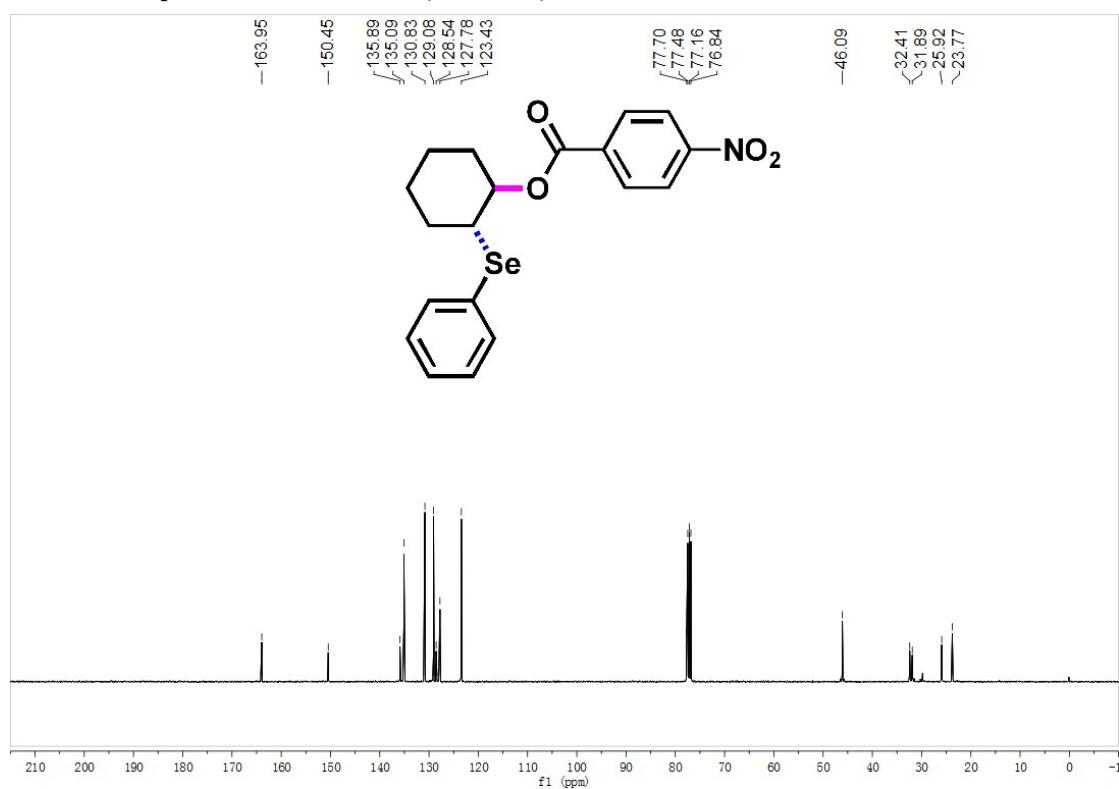
¹³C NMR of product 7l in CDCl₃ (100 MHz)



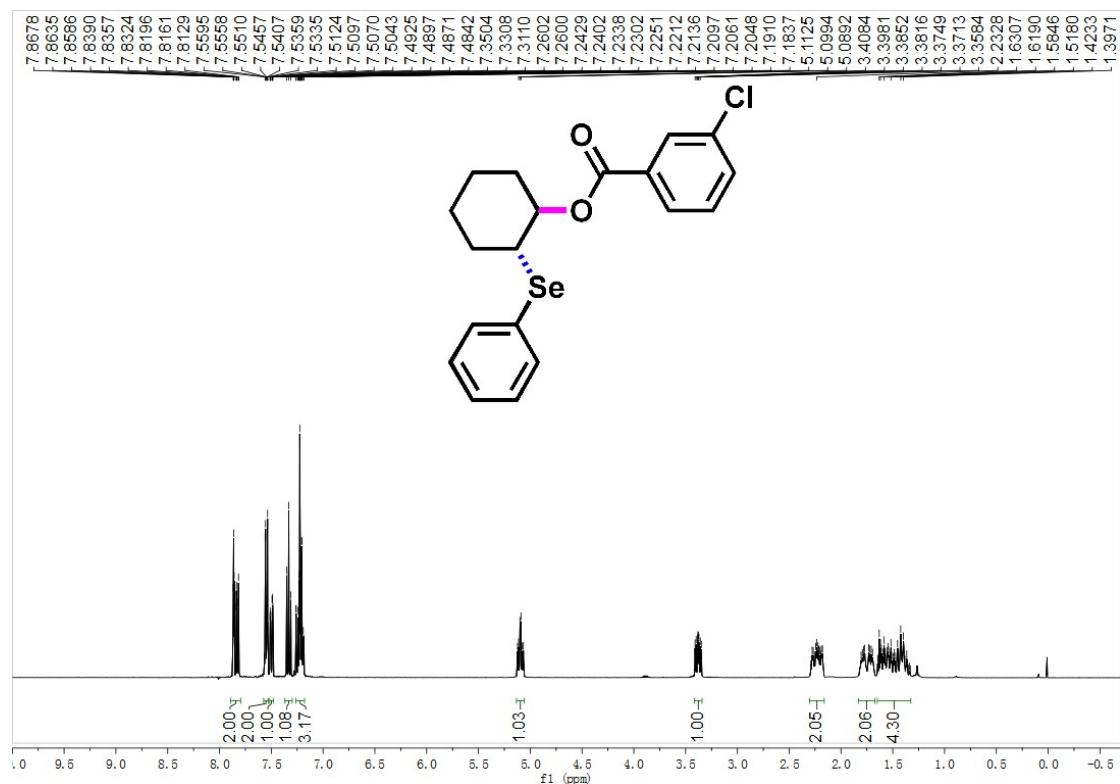
¹H NMR of product 7m in CDCl₃ (400 MHz)



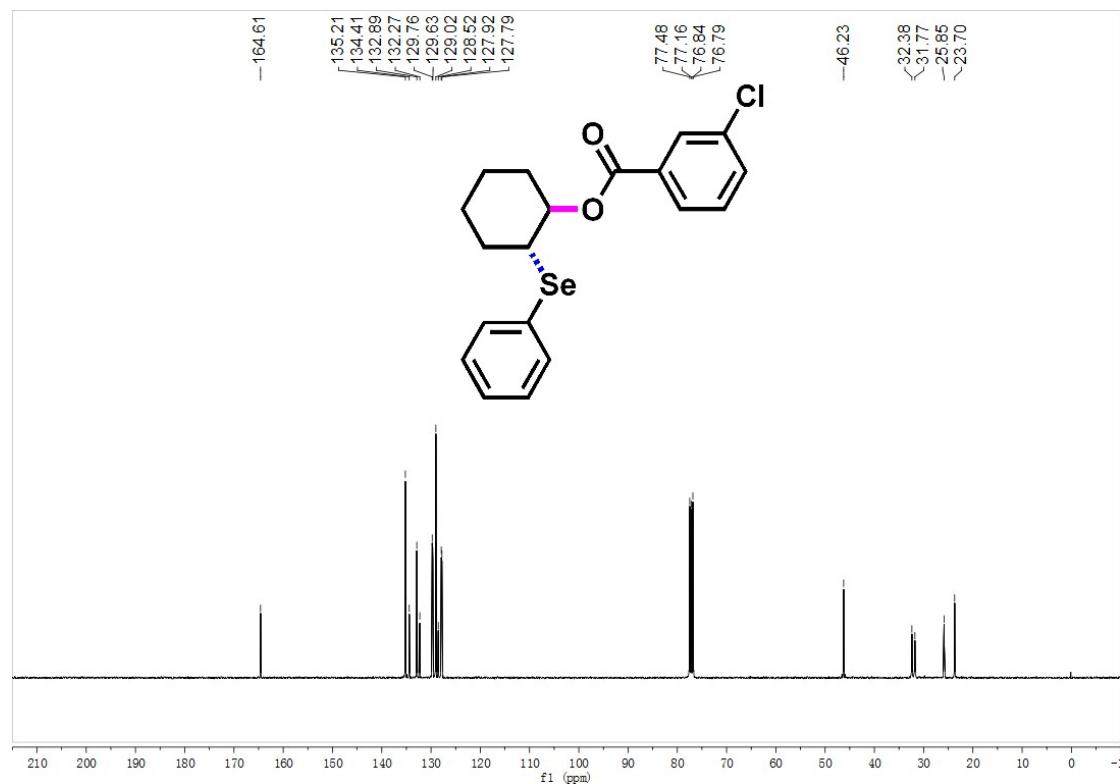
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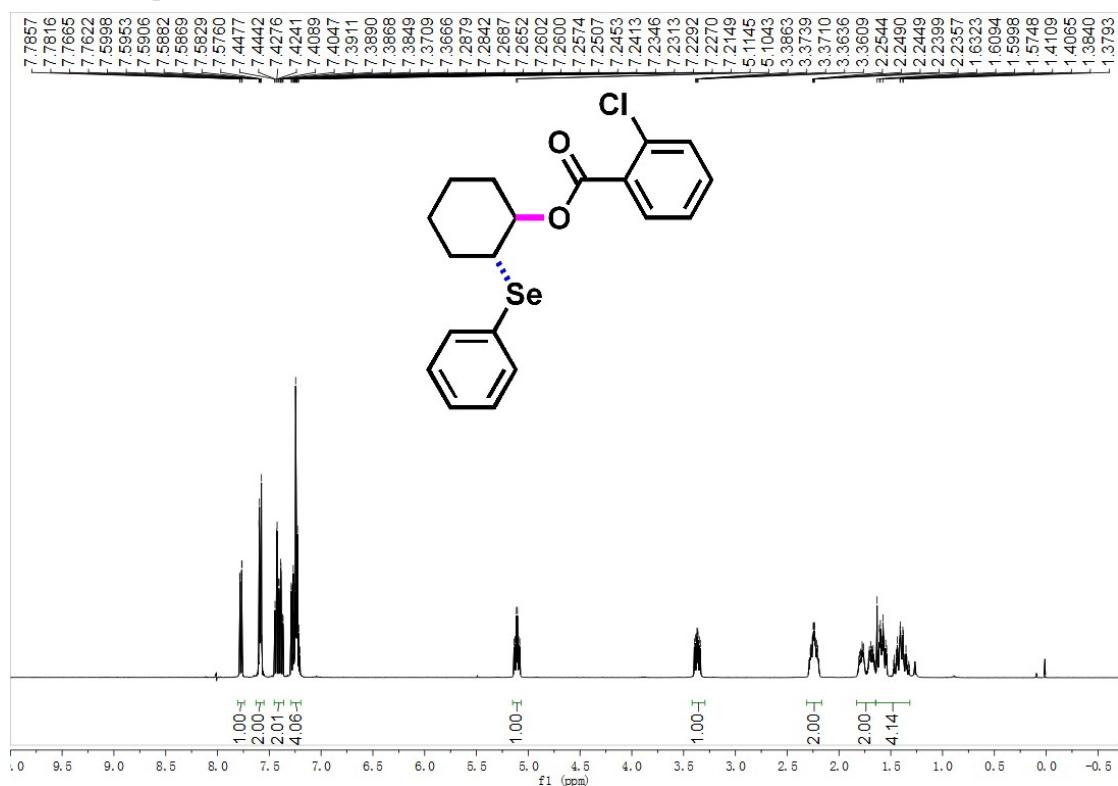
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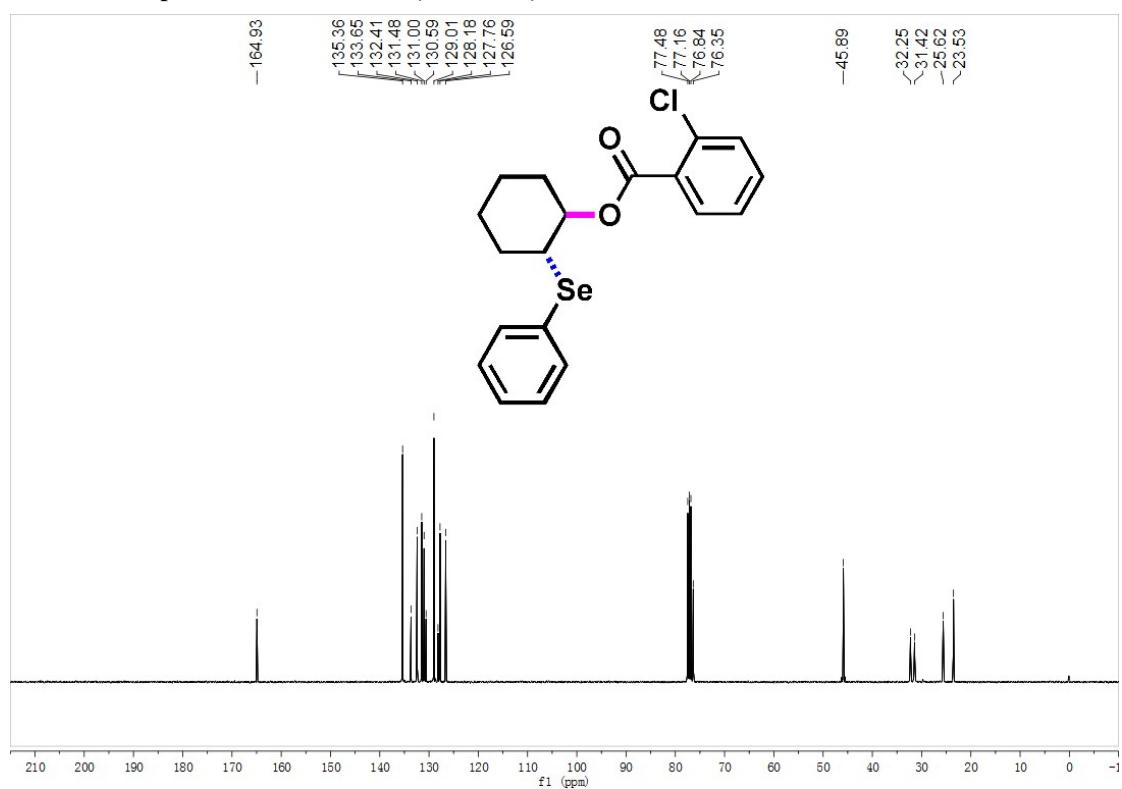
¹³C NMR of product 7n in CDCl₃ 100 MHz)



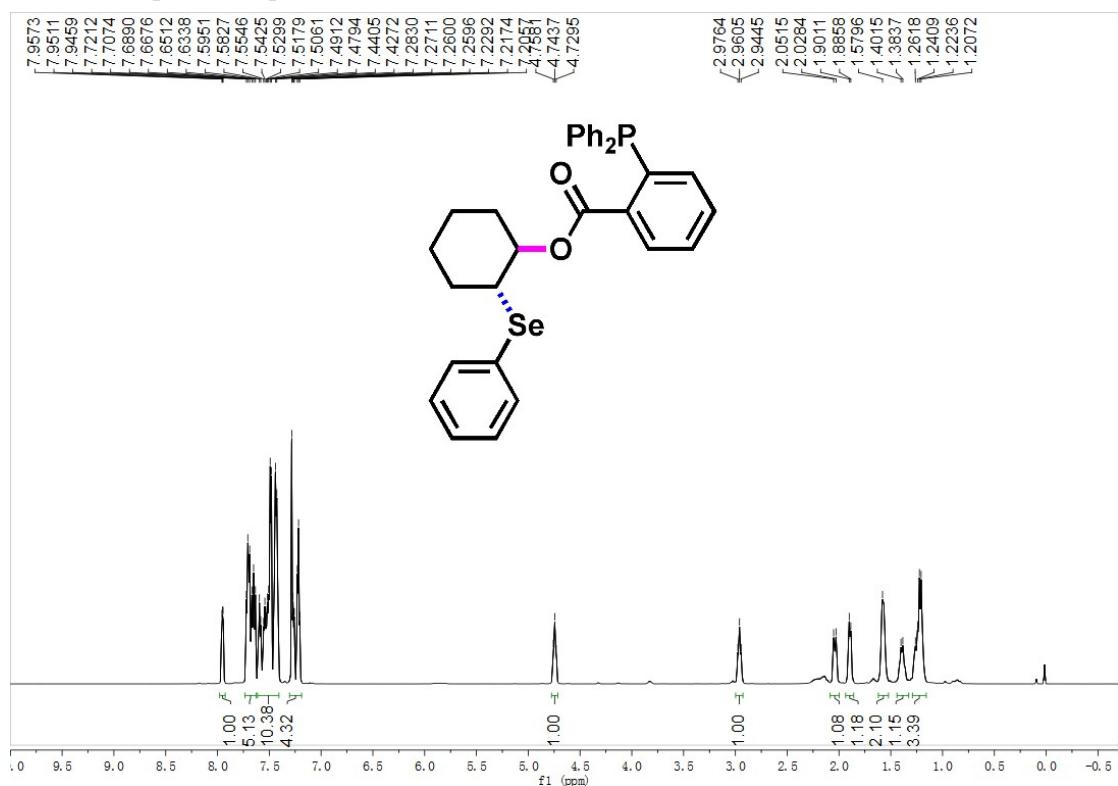
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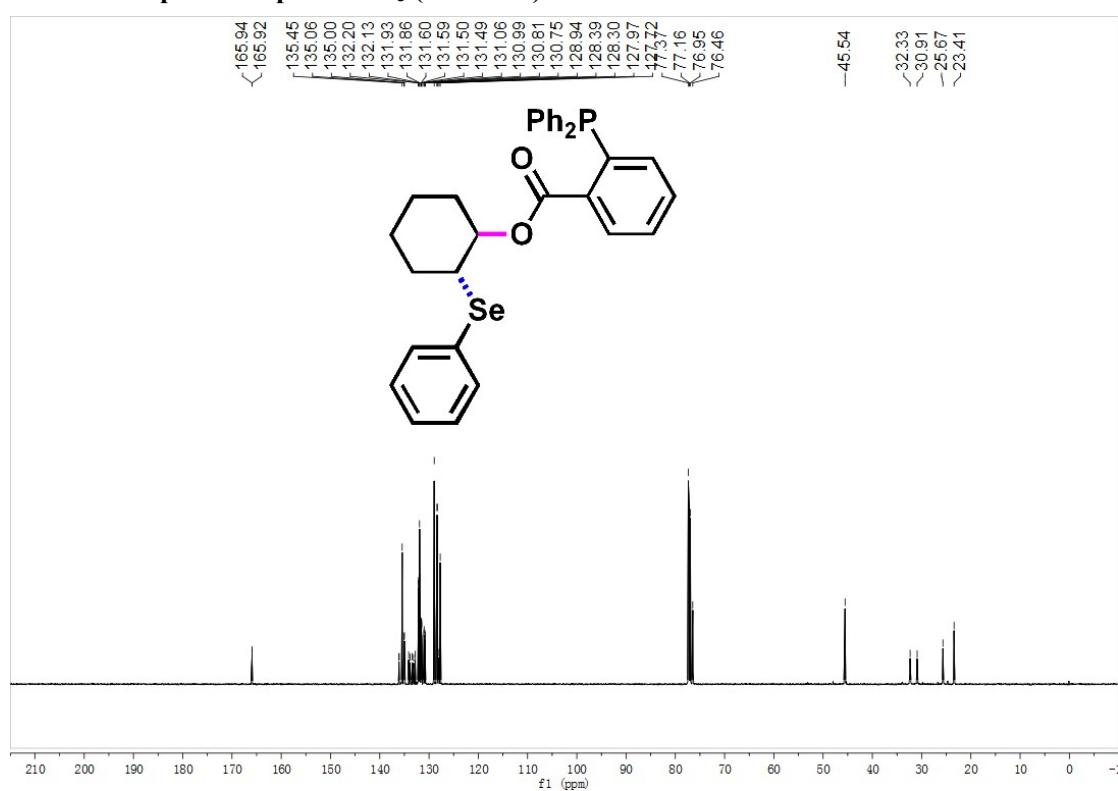
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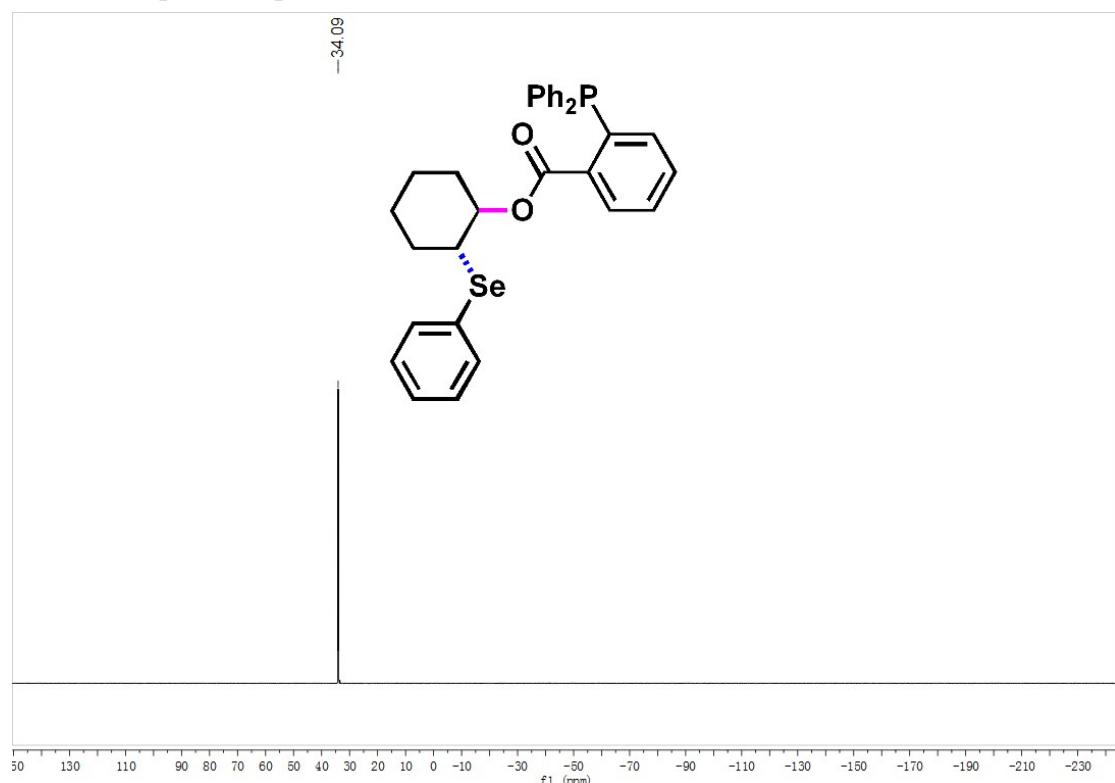
¹H NMR of product 7p in CDCl₃ (600 MHz)



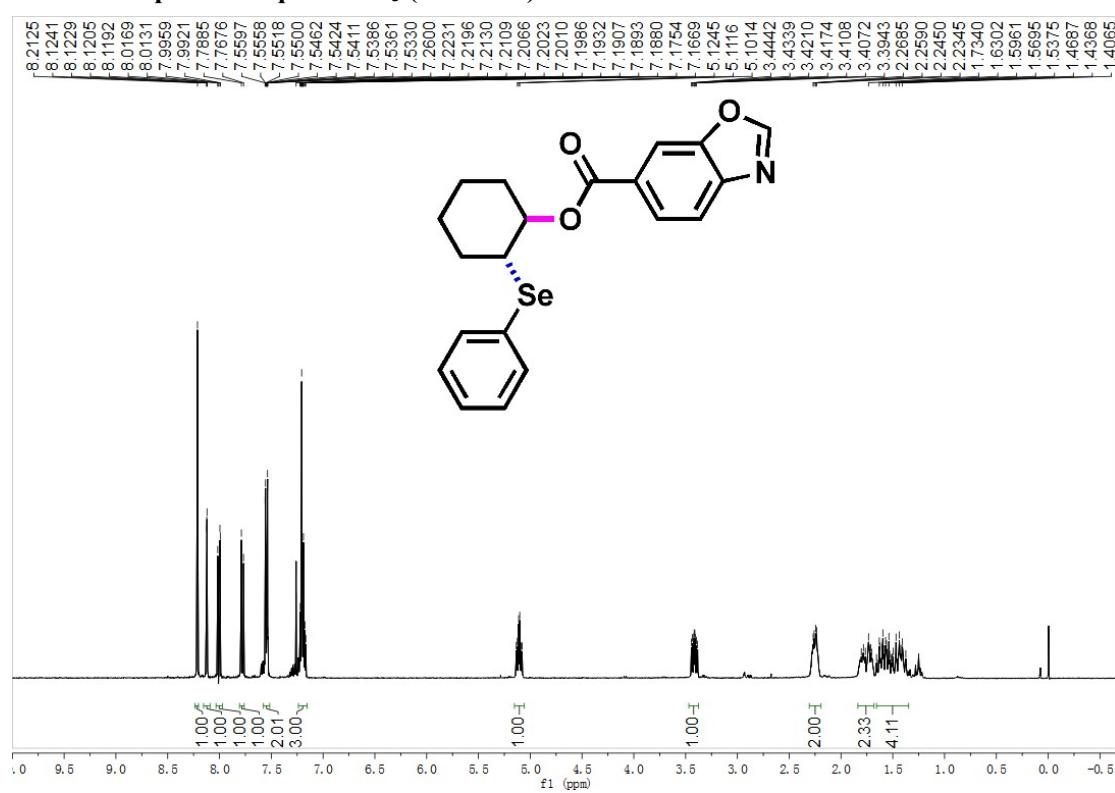
¹³C NMR of product 7p in CDCl₃ (150 MHz)



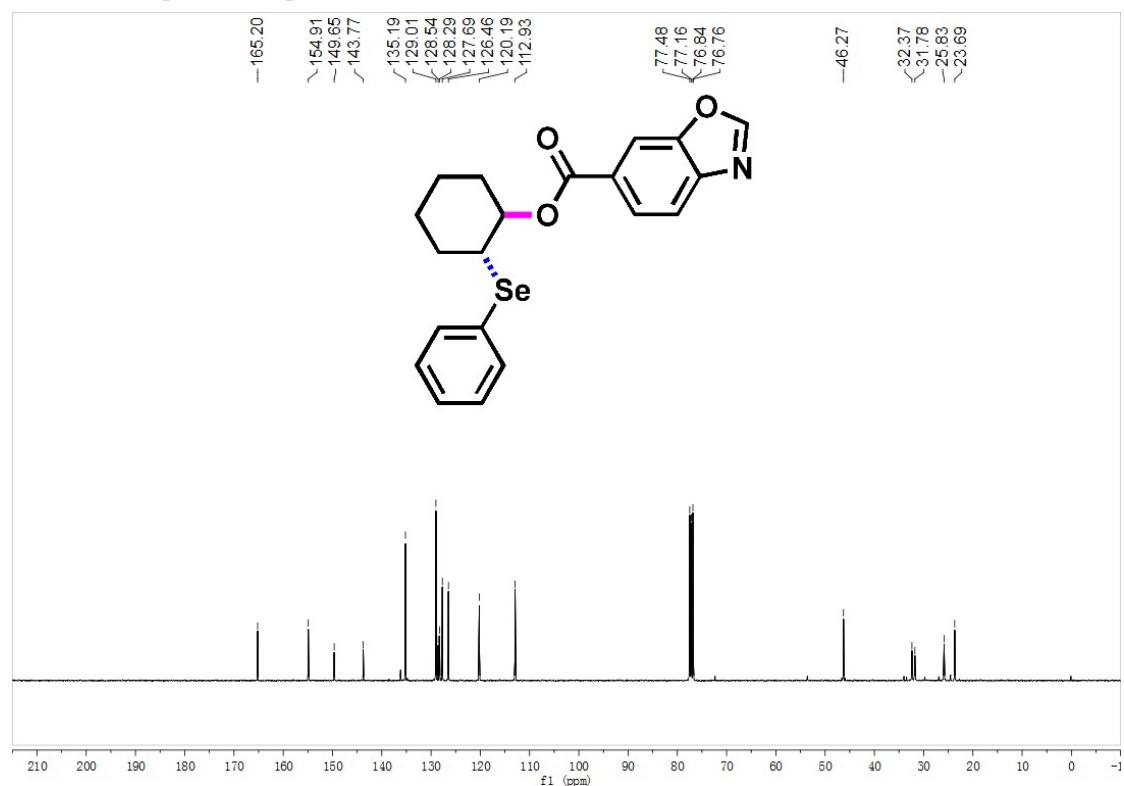
³¹P NMR of product 7p in CDCl₃ (240 MHz)



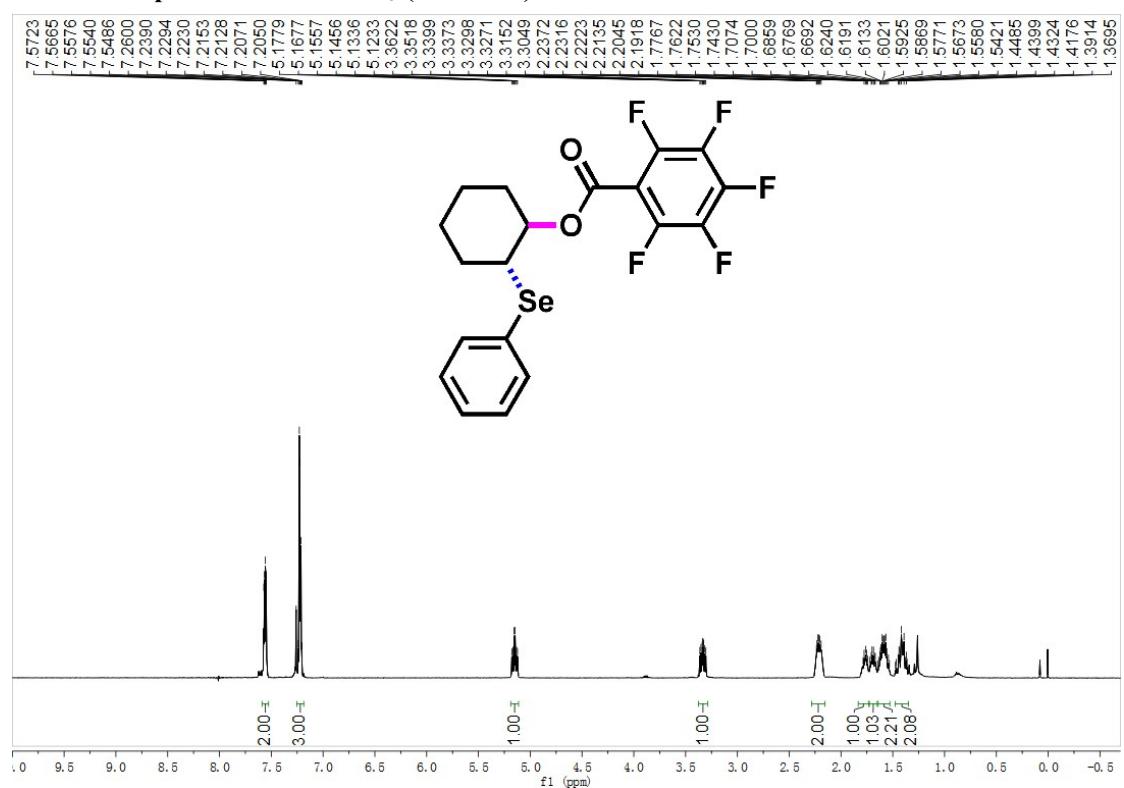
¹H NMR of product 7q in CDCl₃ (400 MHz)



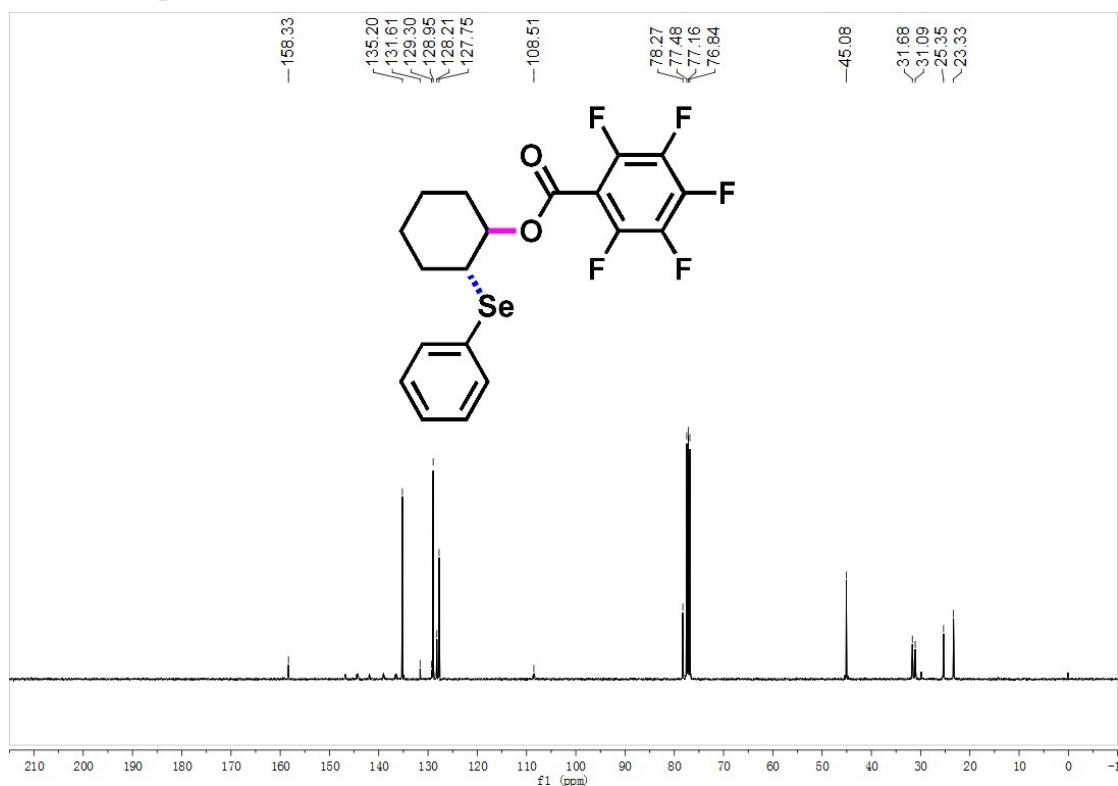
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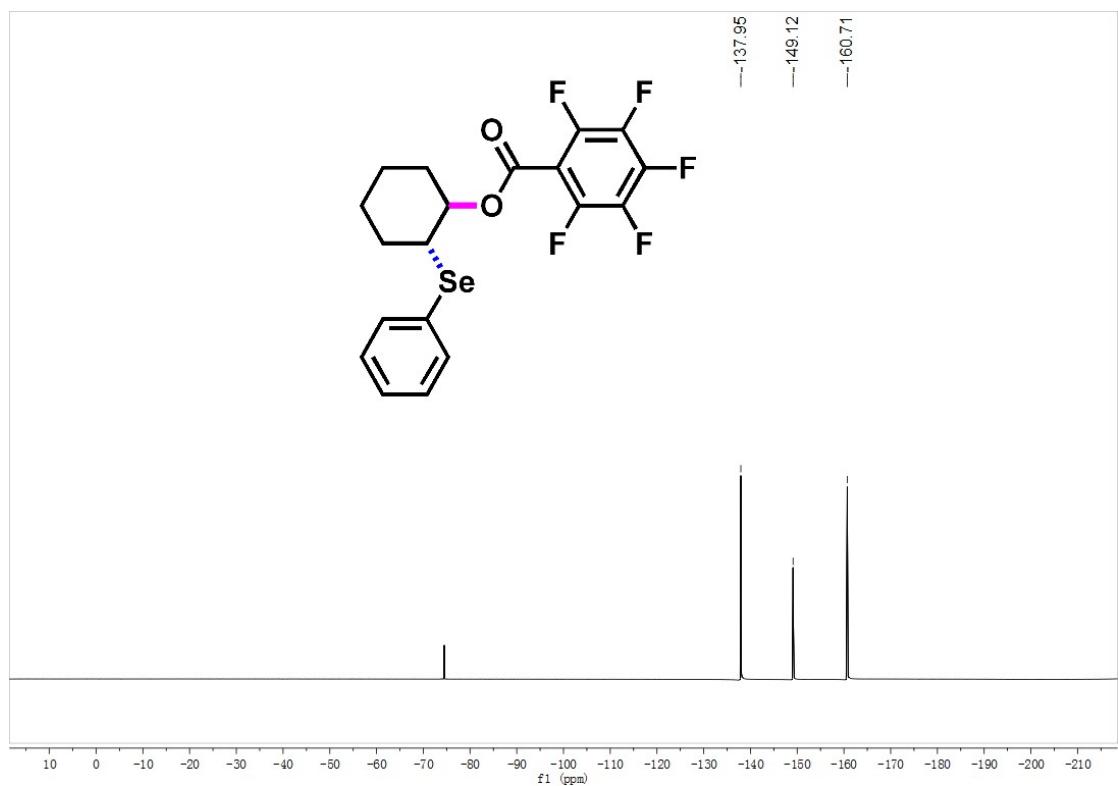
¹H NMR of product 7r in CDCl₃ (400 MHz)



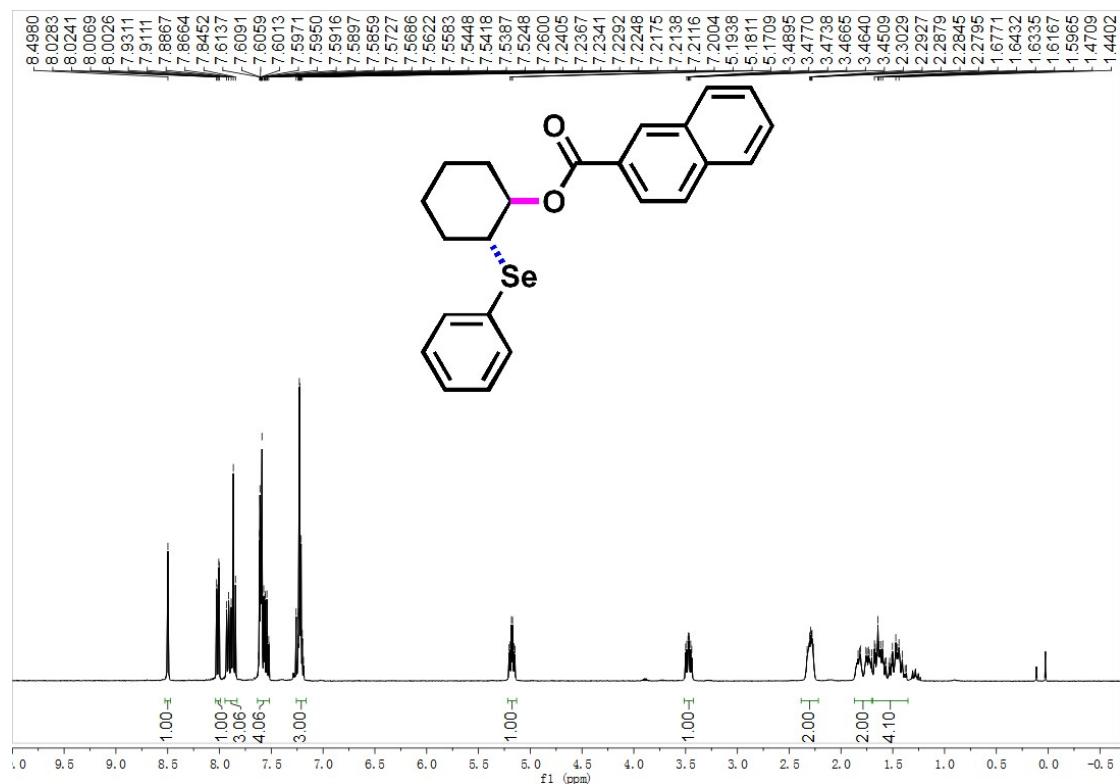
¹³C NMR of product 7r in CDCl₃ (100 MHz)



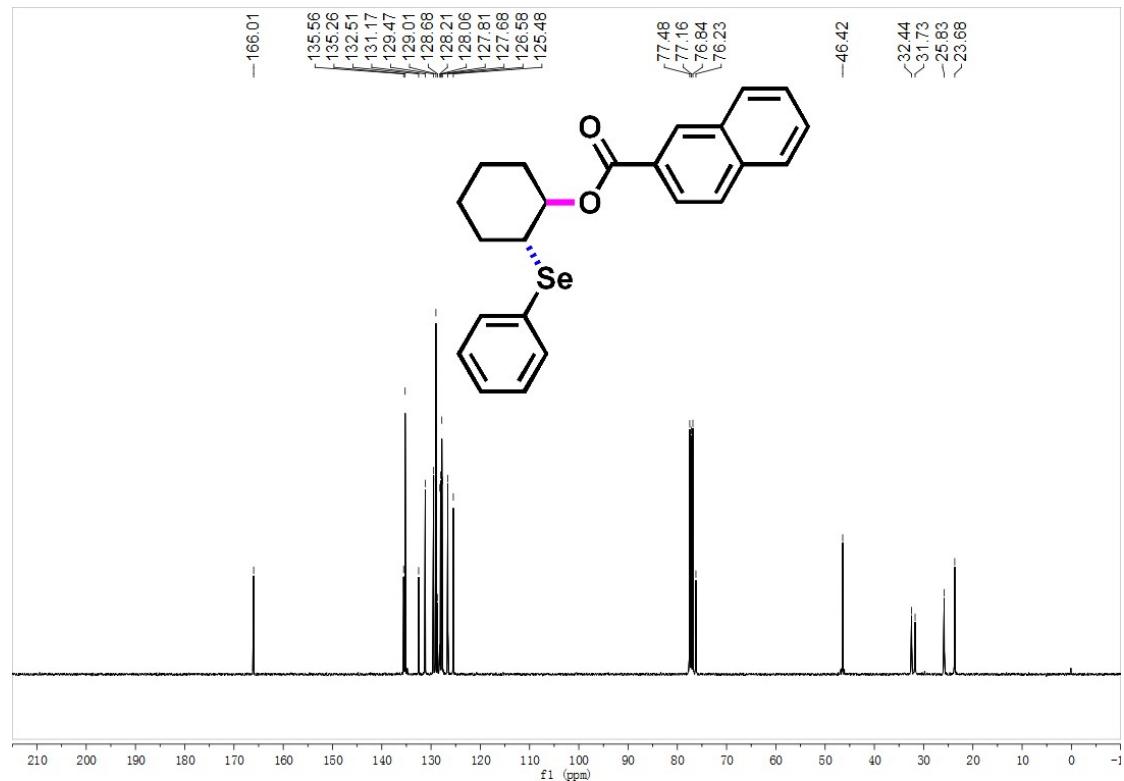
¹⁹F NMR of product 7r in CDCl₃ (376 MHz)



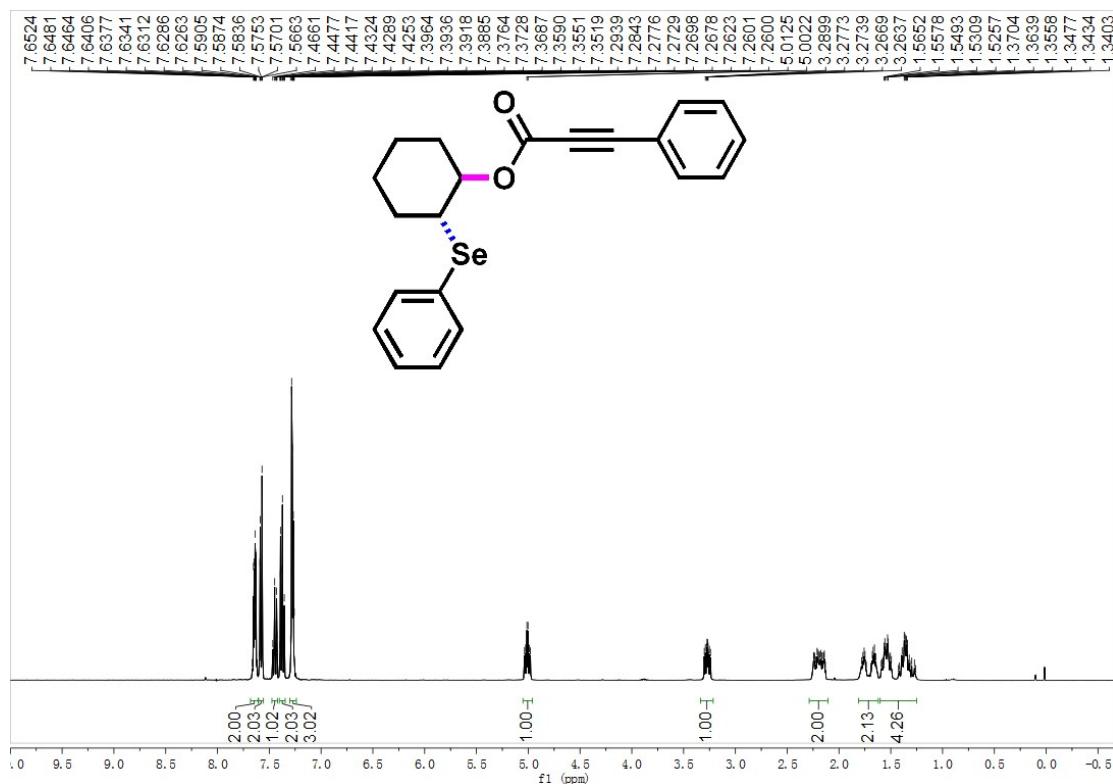
¹H NMR of product 7s in CDCl₃ (400 MHz)



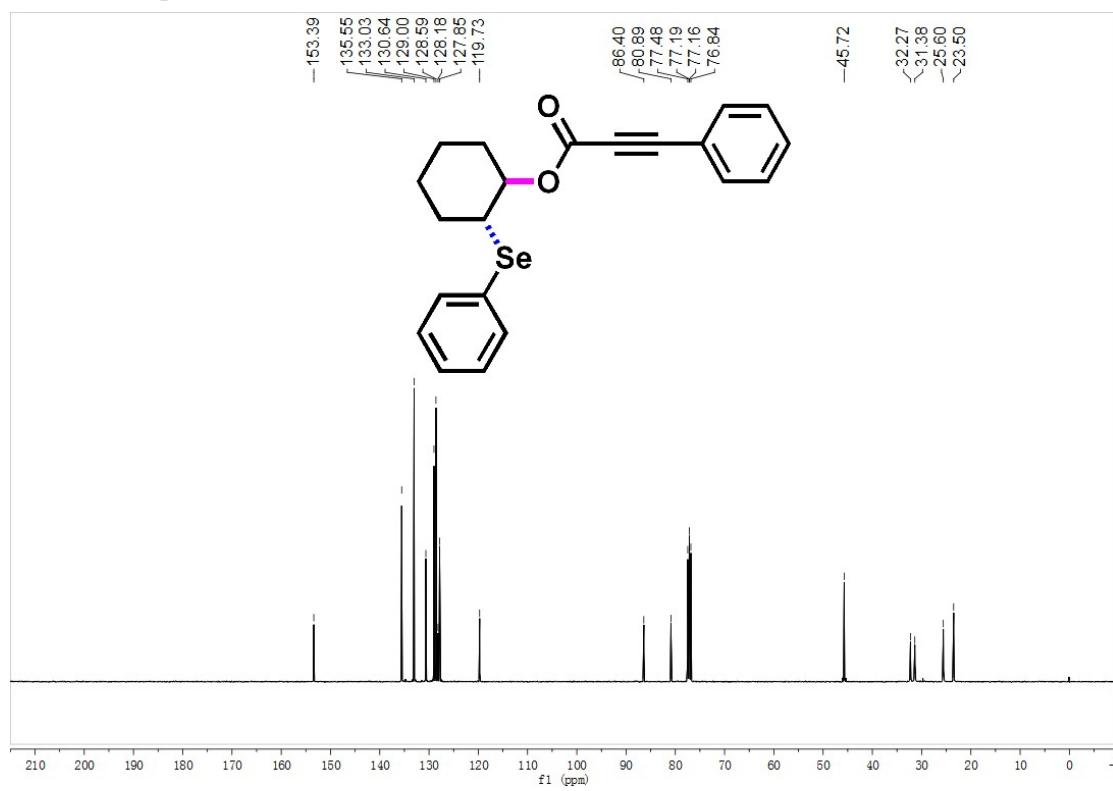
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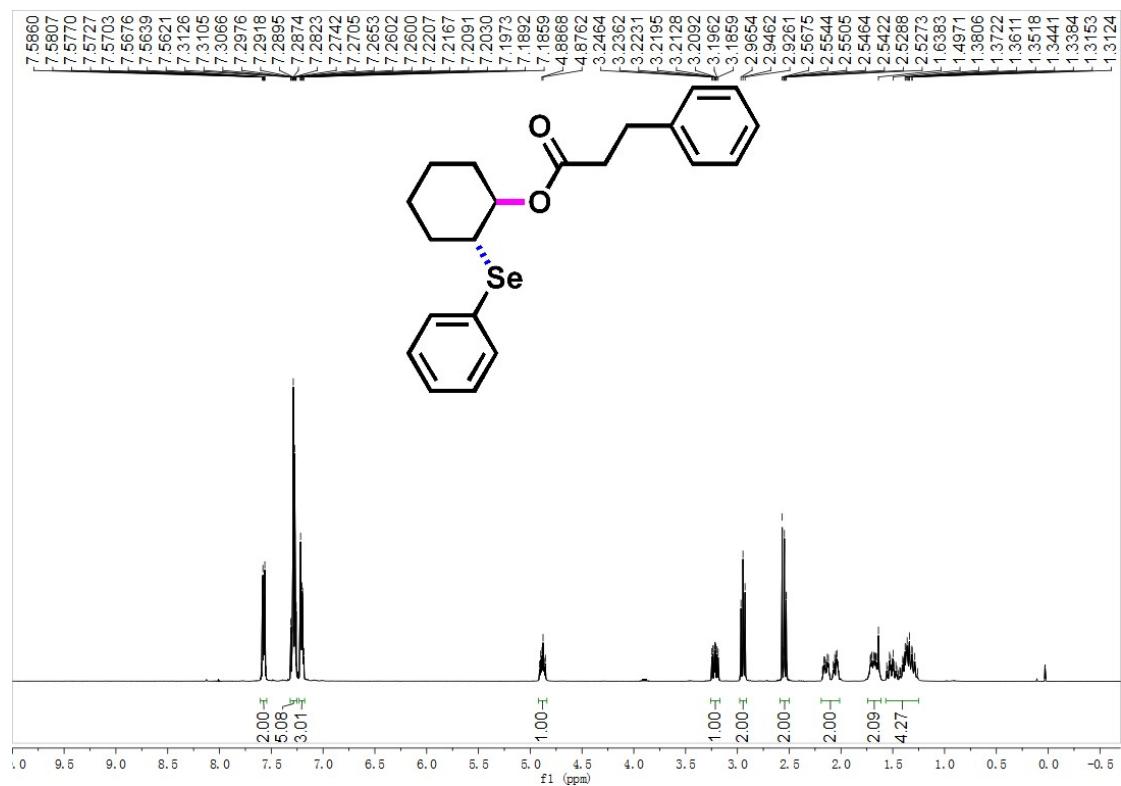
¹H NMR of product 7t in CDCl₃ (400 MHz)



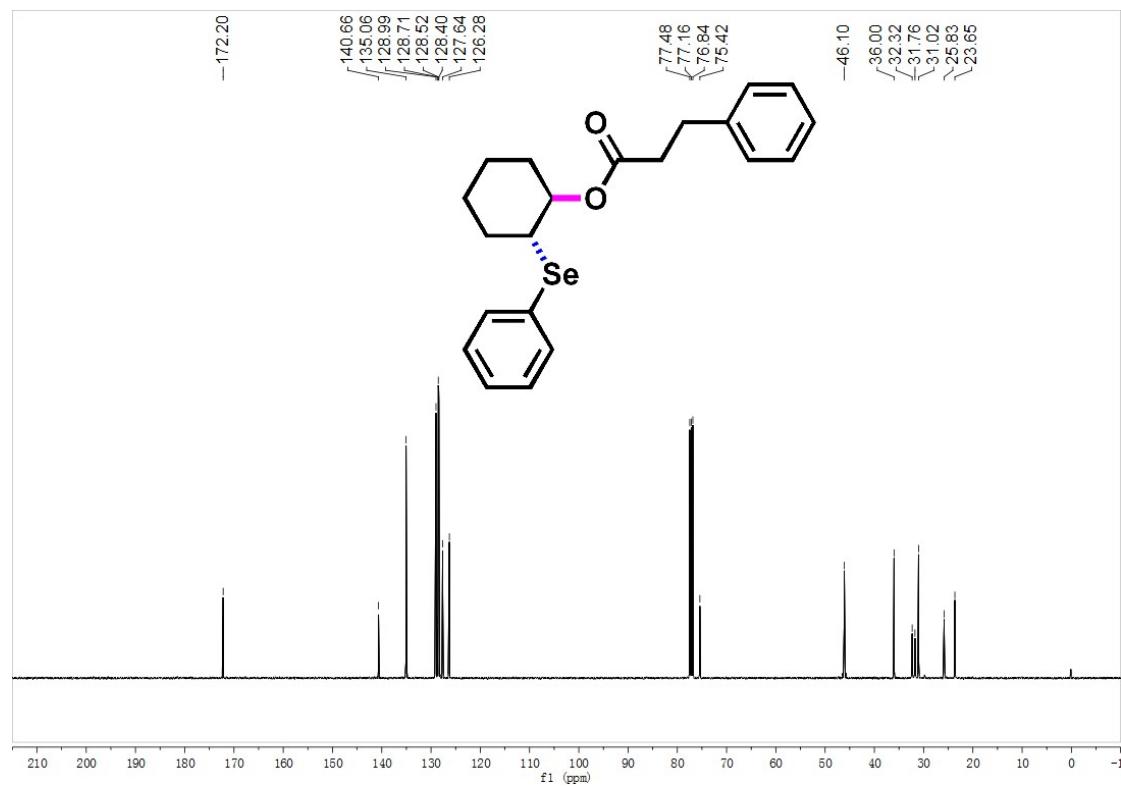
¹³C NMR of product 7t in CDCl₃ (100 MHz)



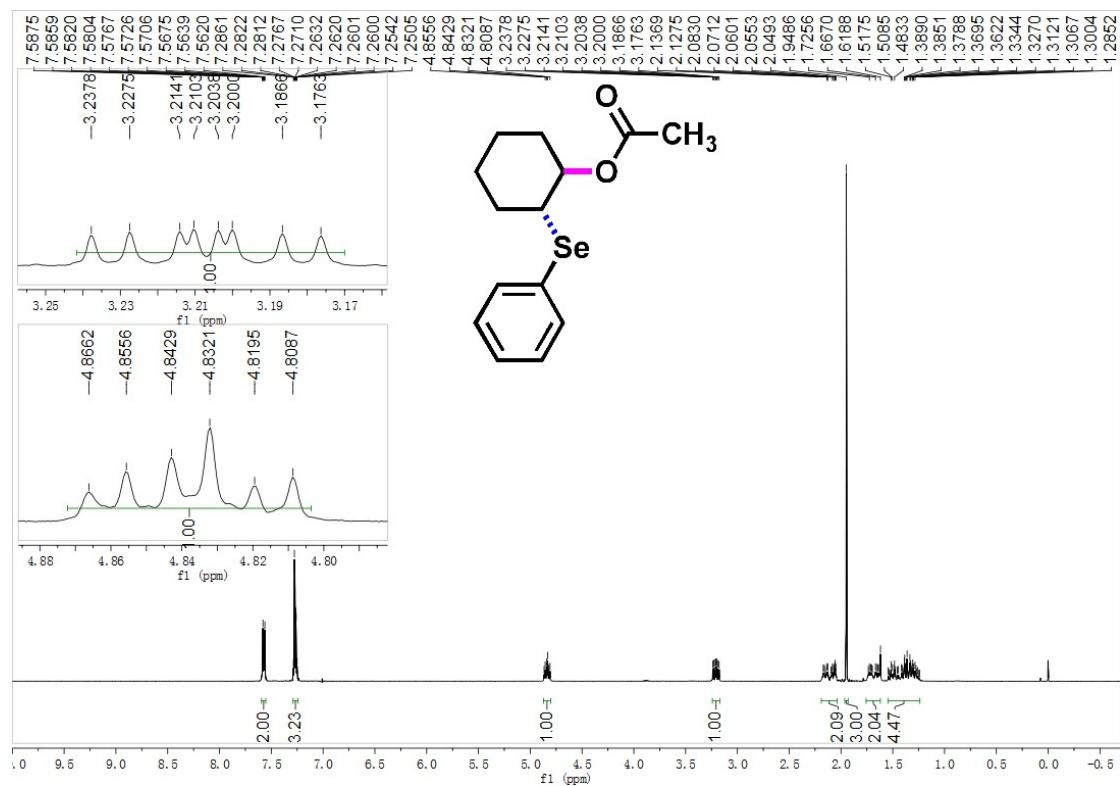
¹H NMR of product 7u in CDCl₃ (400 MHz)



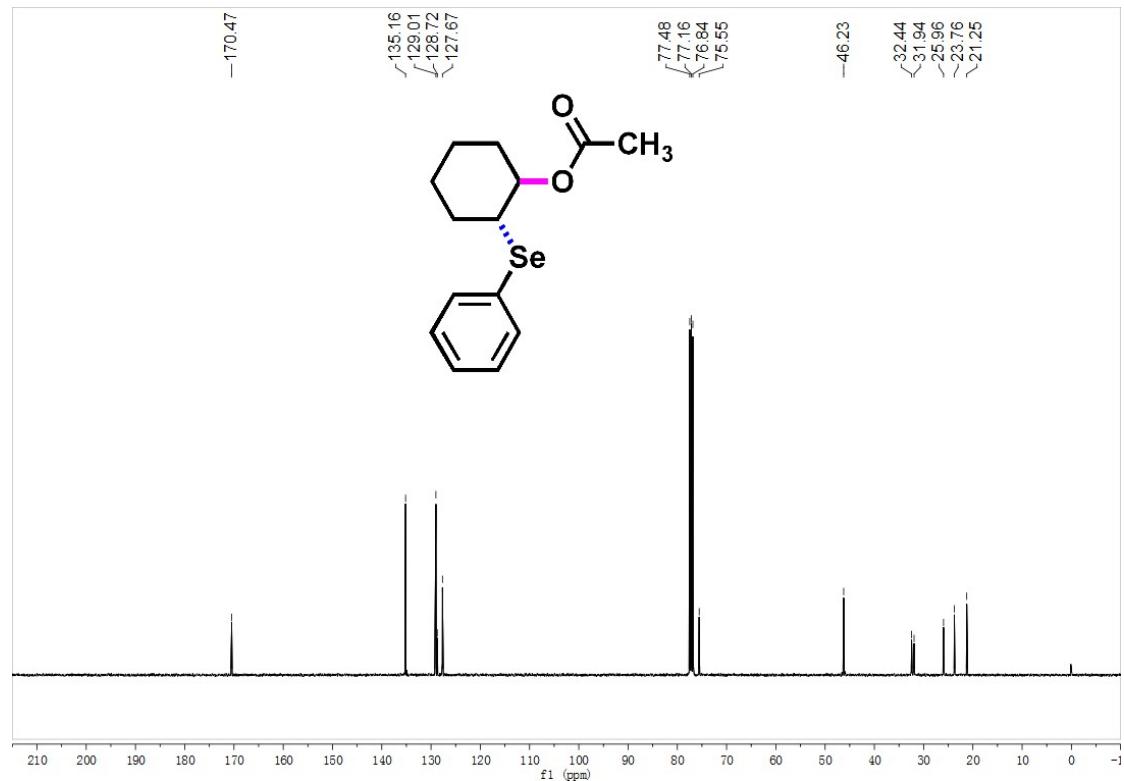
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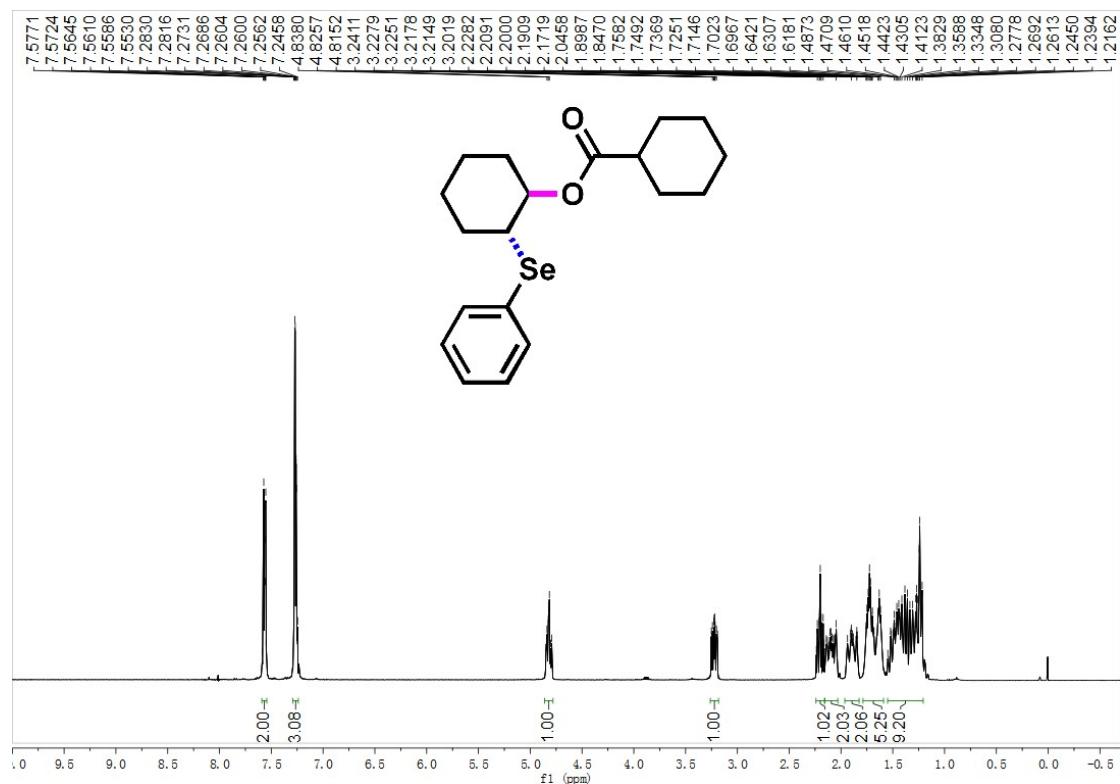
¹H NMR of product 7v in CDCl₃ (400 MHz)



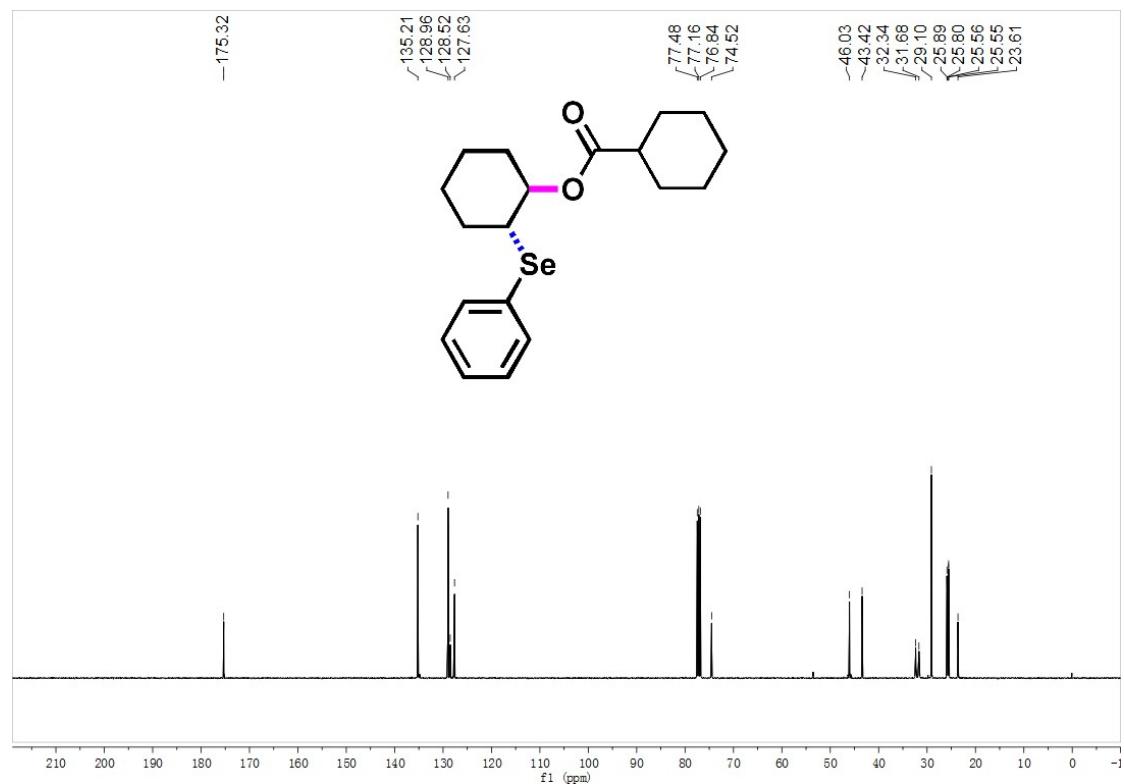
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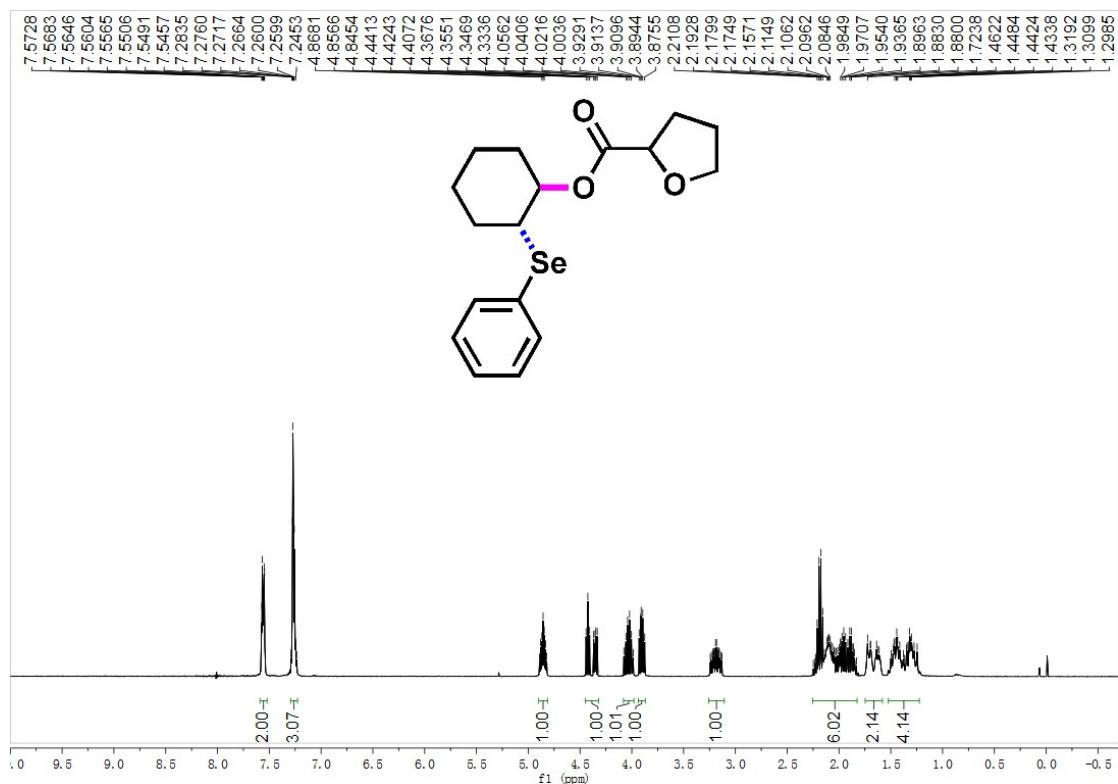
¹H NMR of product 7w in CDCl₃ (400 MHz)



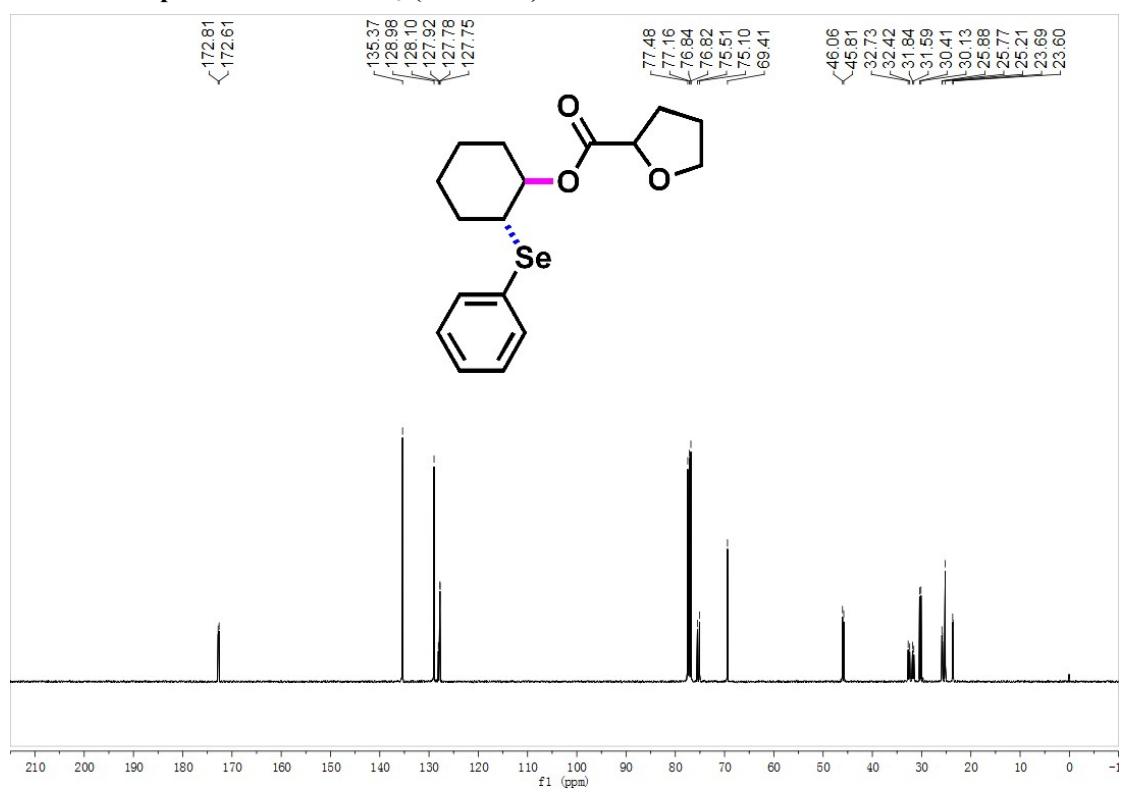
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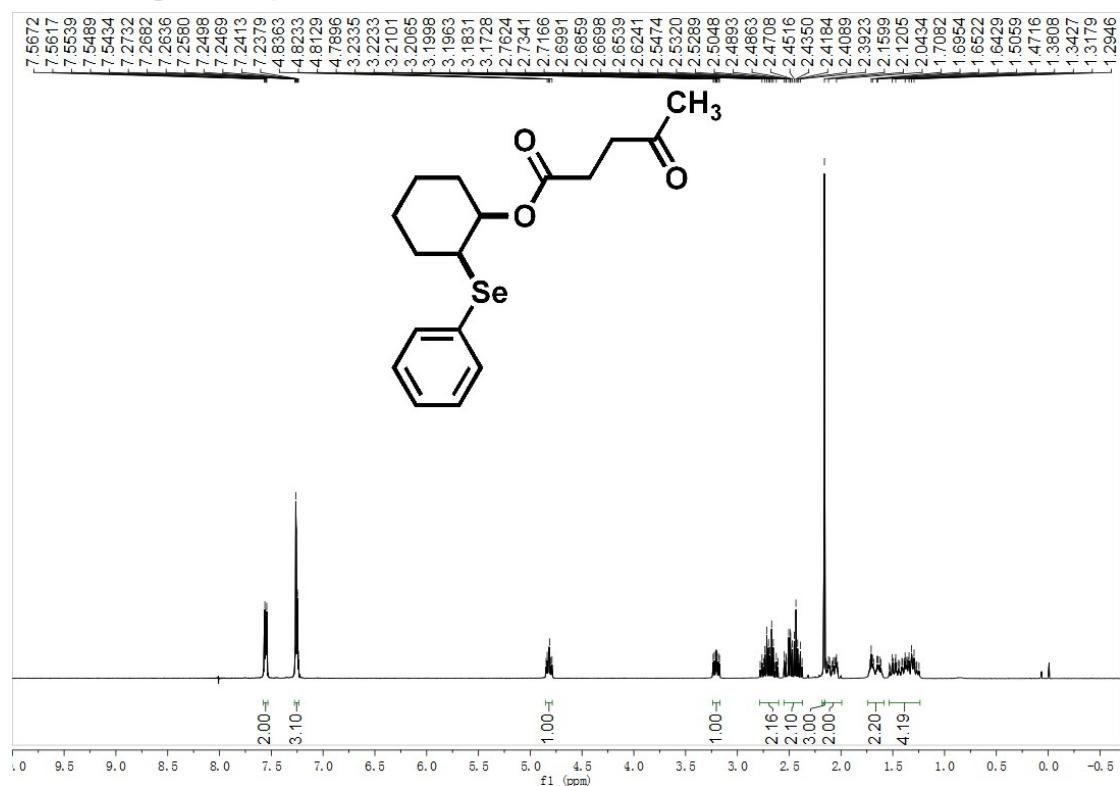
¹H NMR of product 7x in CDCl₃ (400 MHz)



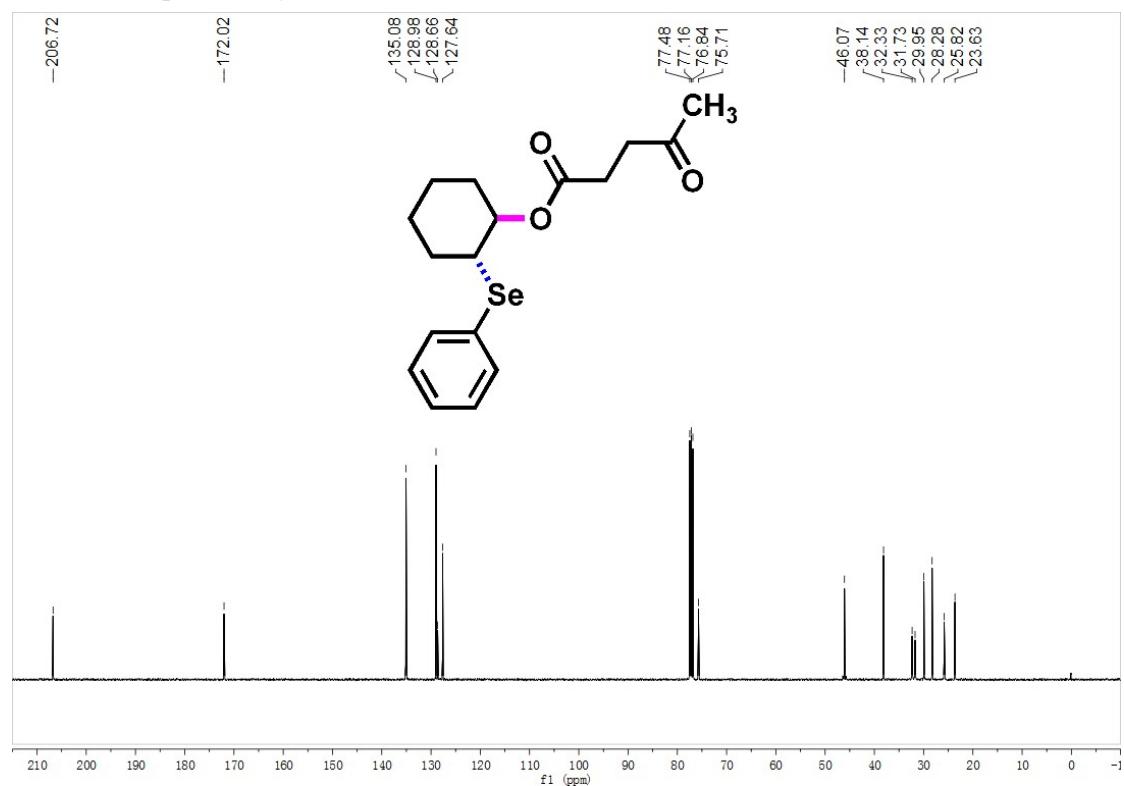
¹³C NMR of product 7x in CDCl₃ (100 MHz)



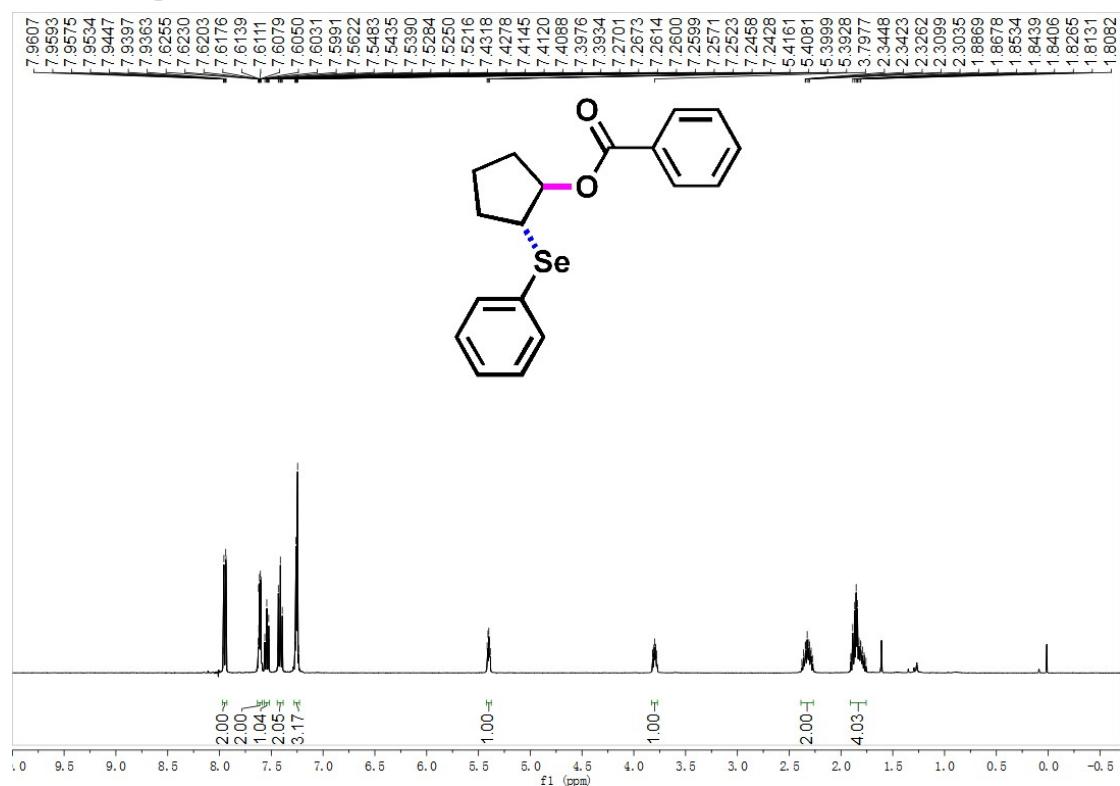
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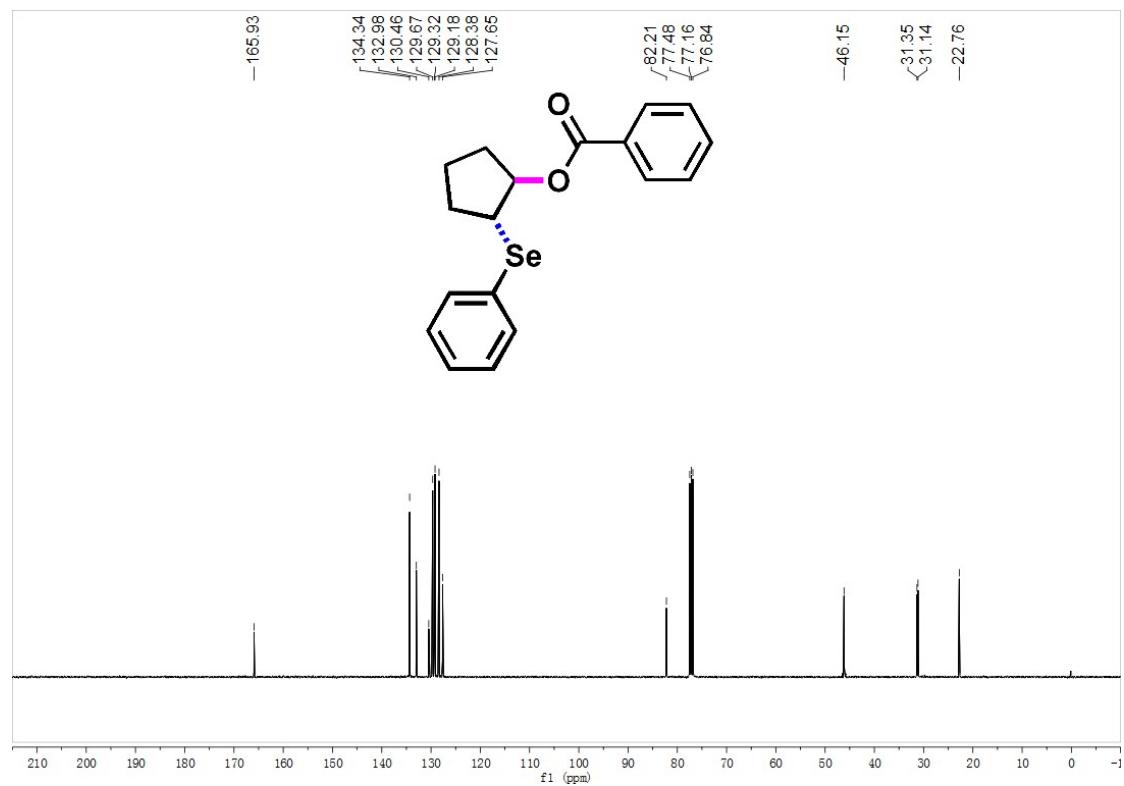
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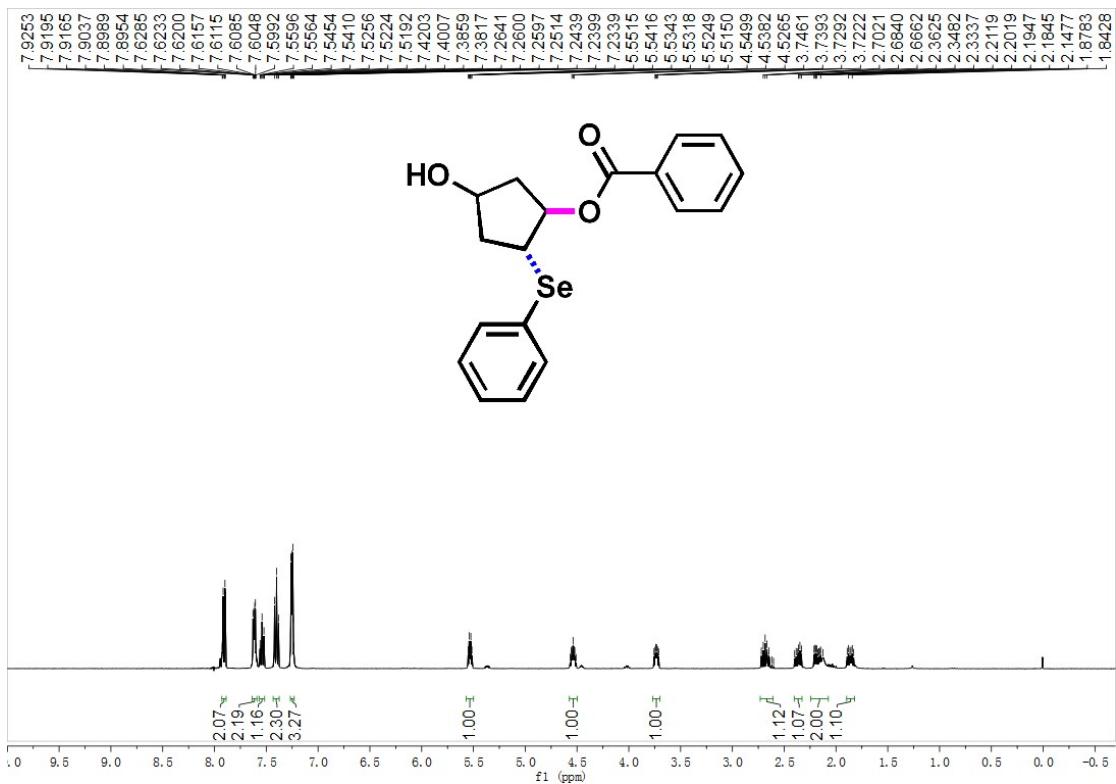
¹H NMR of product 7z in CDCl₃ (400 MHz)



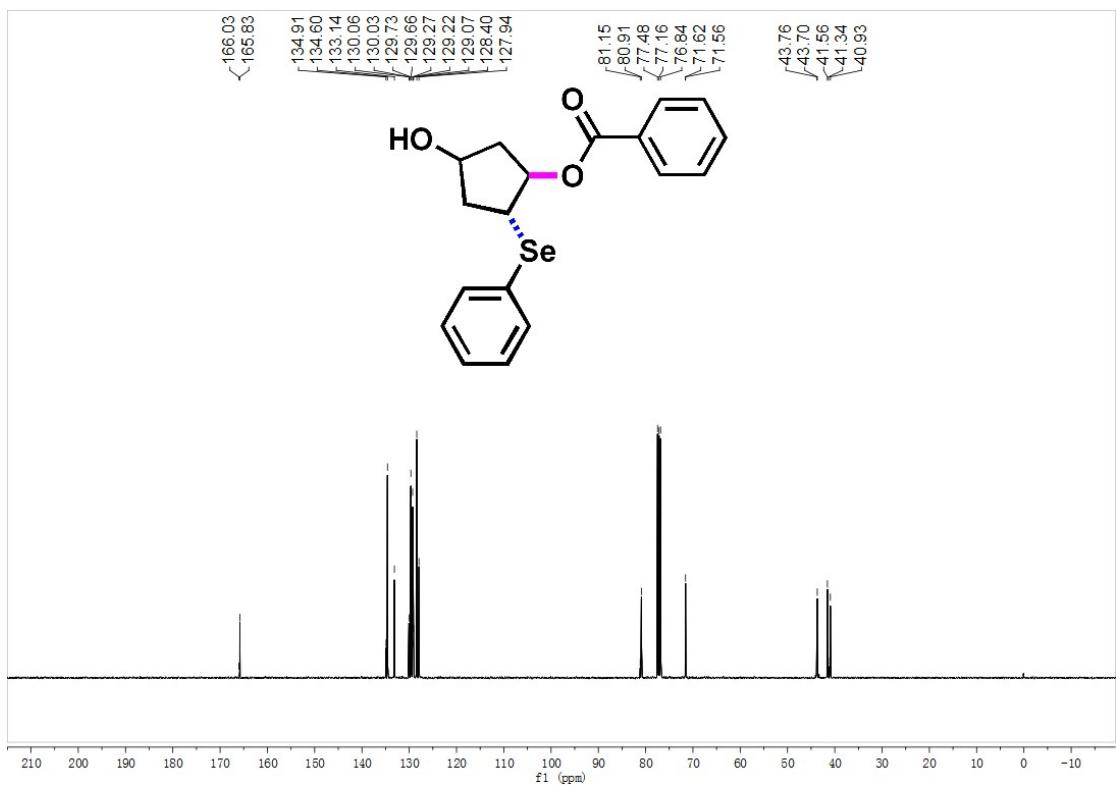
¹³C NMR of product 7z in CDCl₃ (100 MHz)



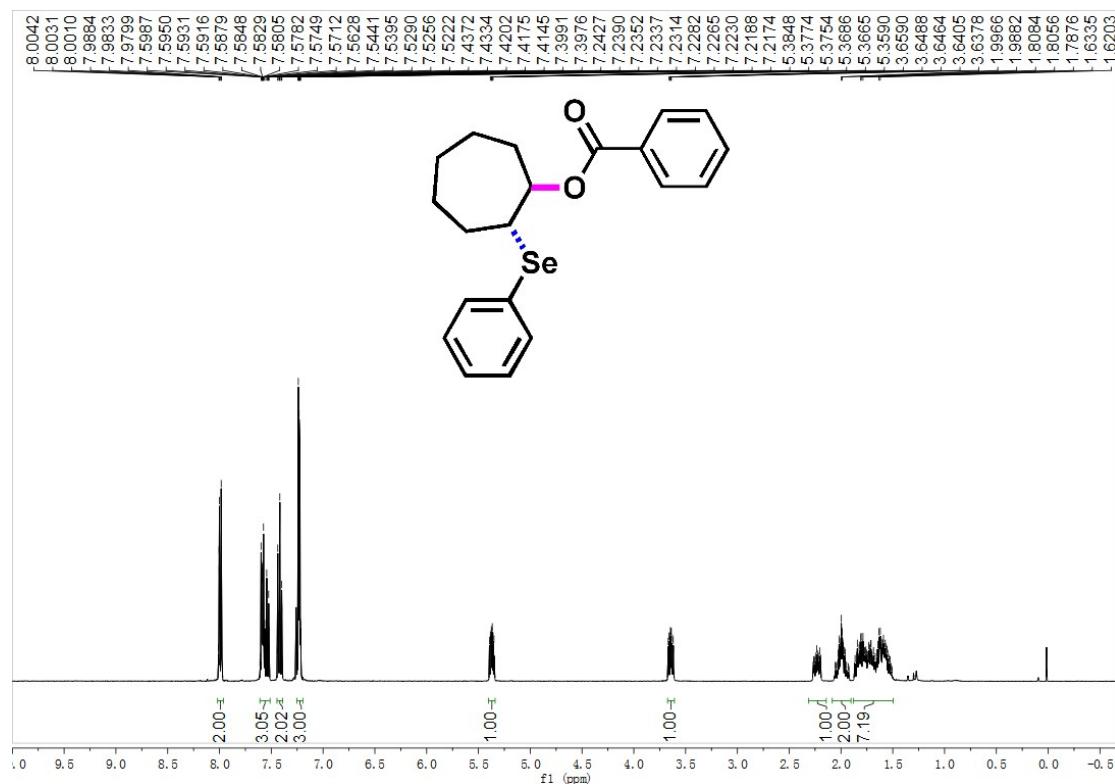
¹H NMR of product 7a' in CDCl₃ (400 MHz)



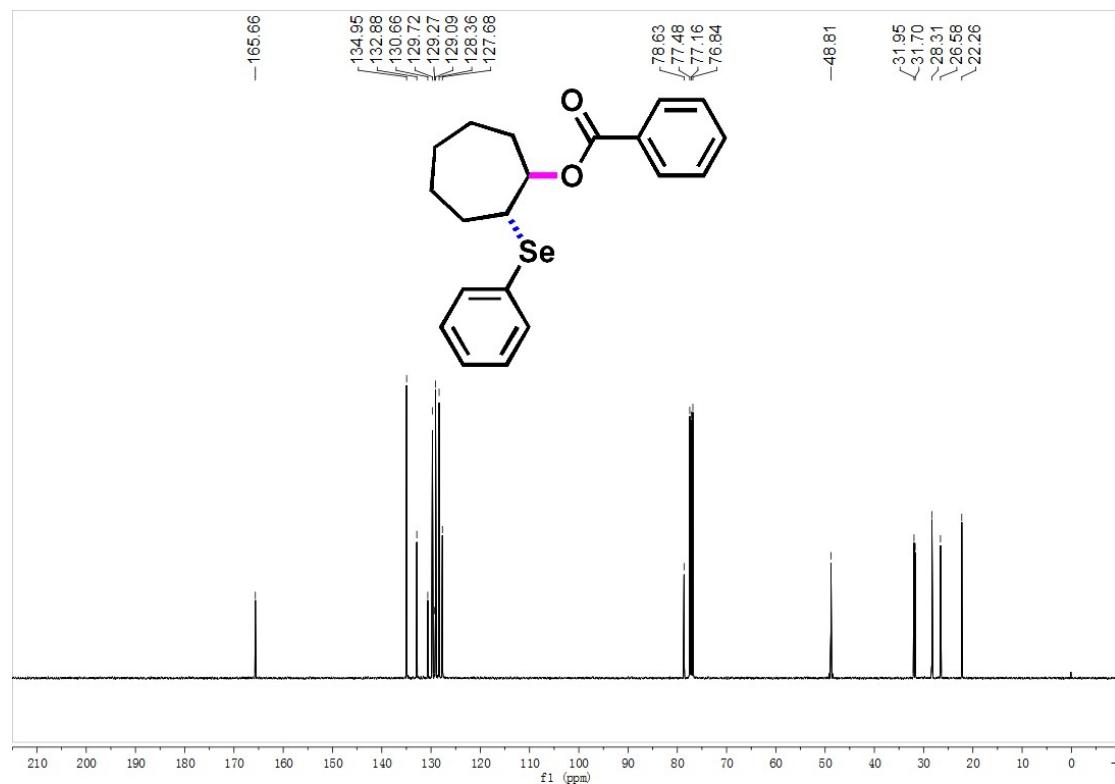
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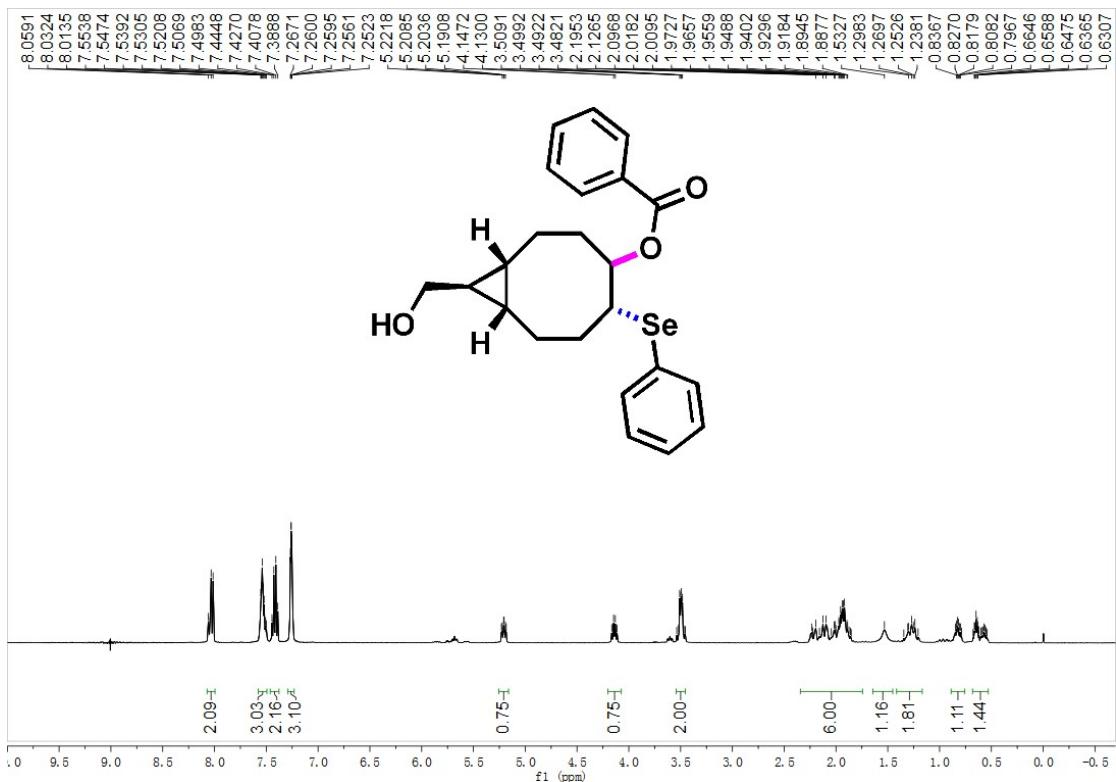
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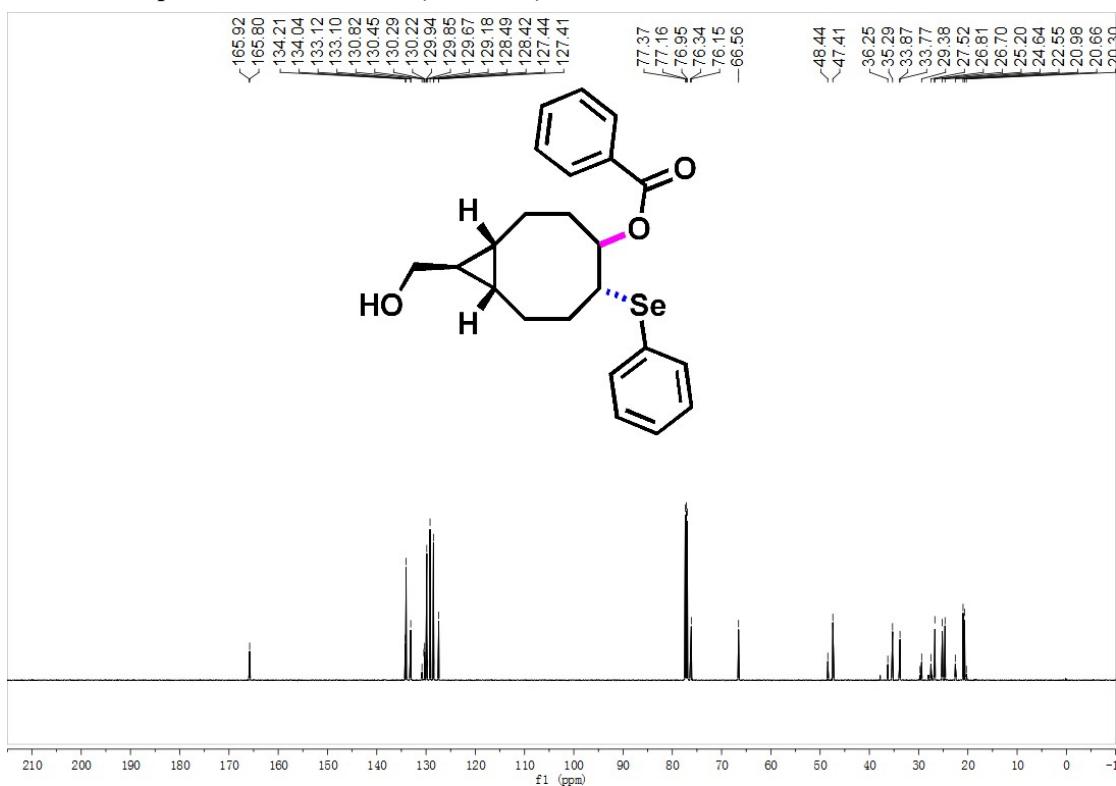
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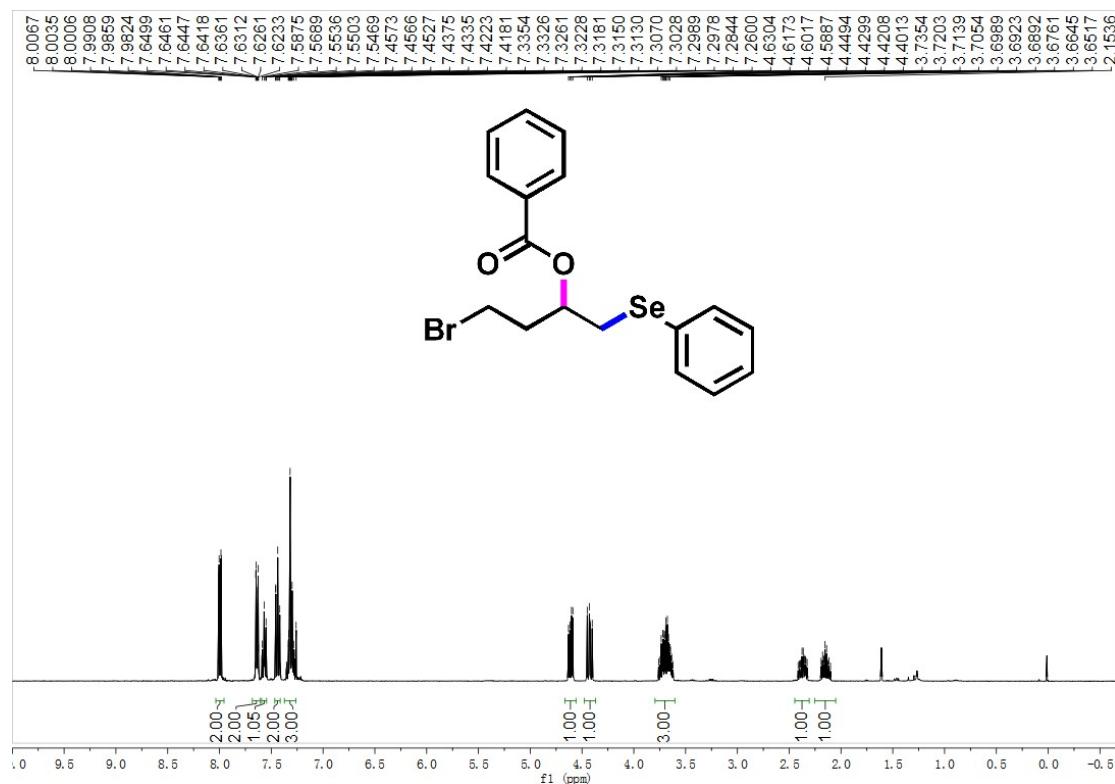
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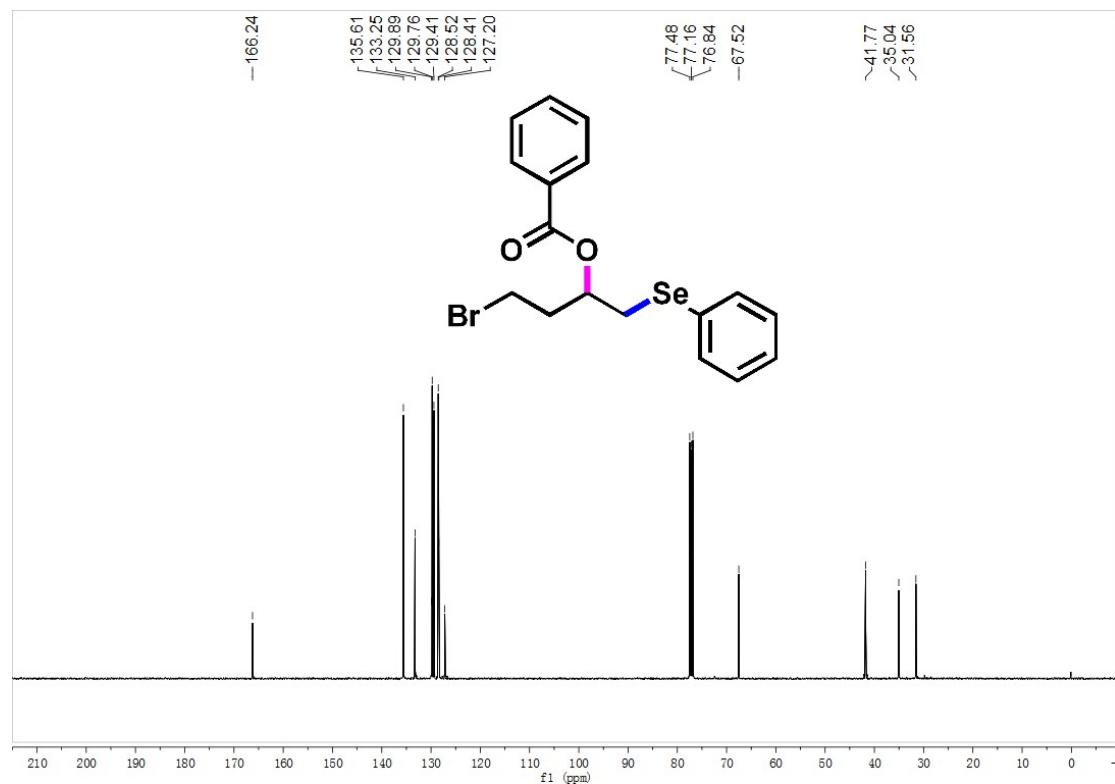
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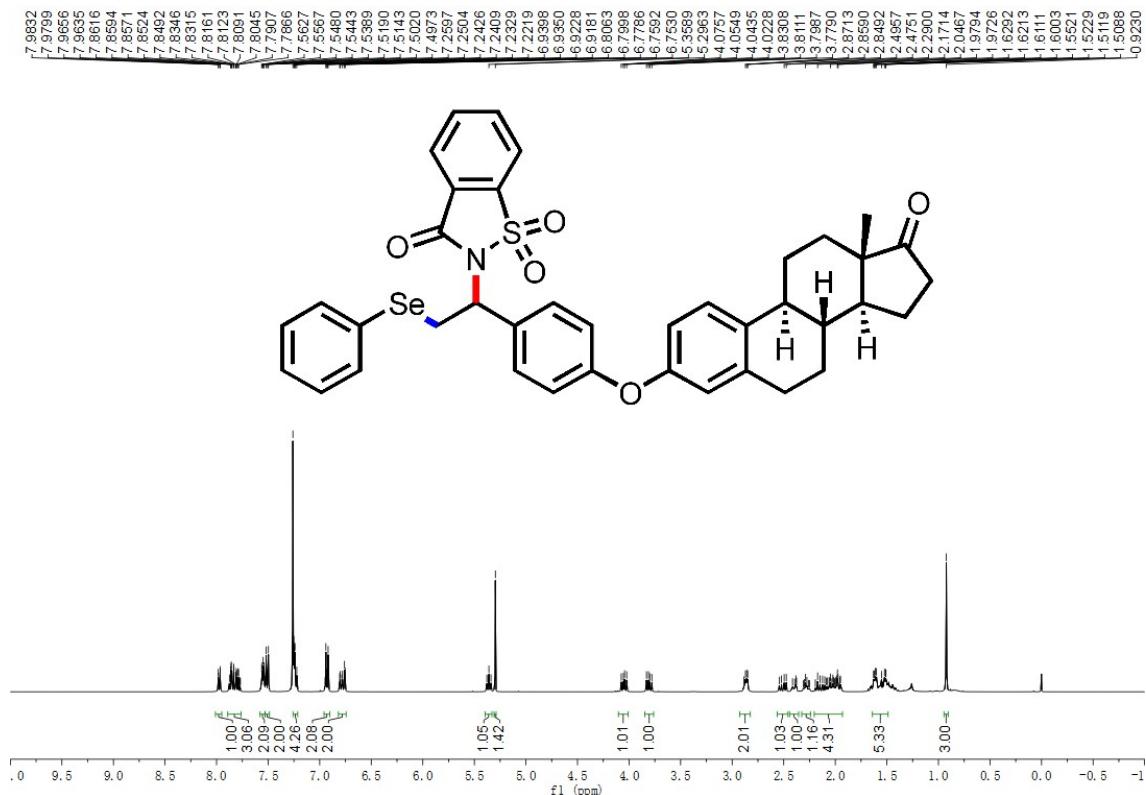
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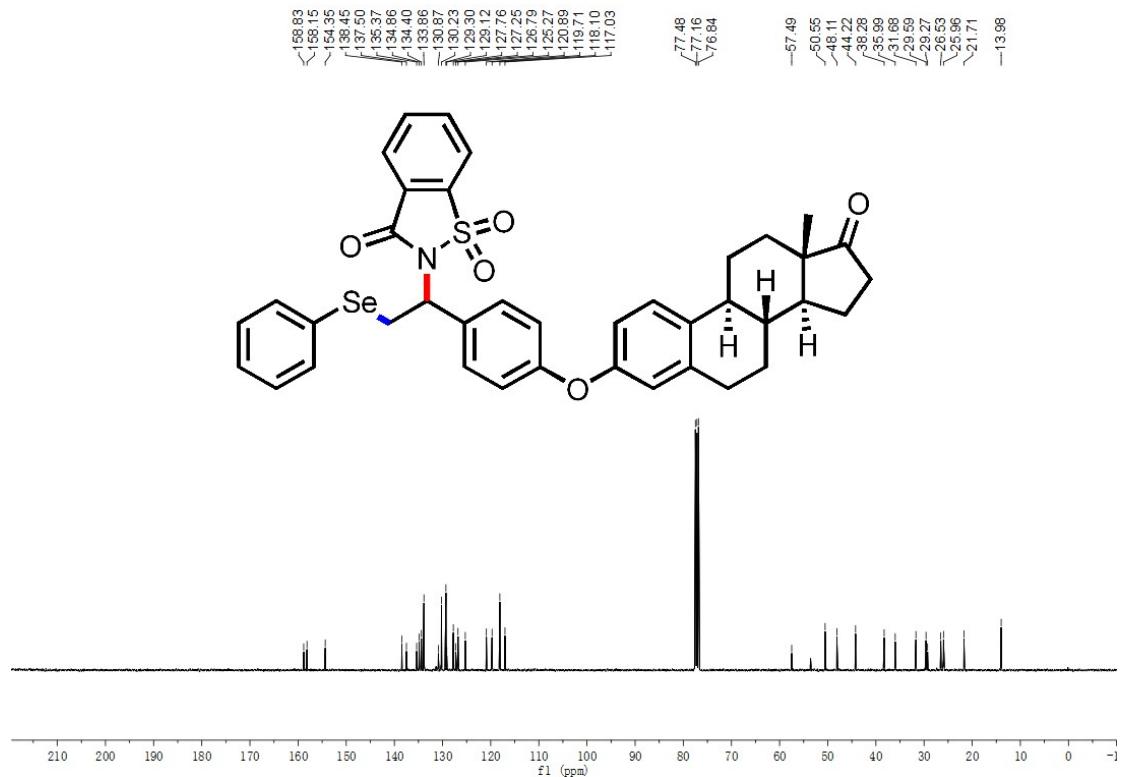
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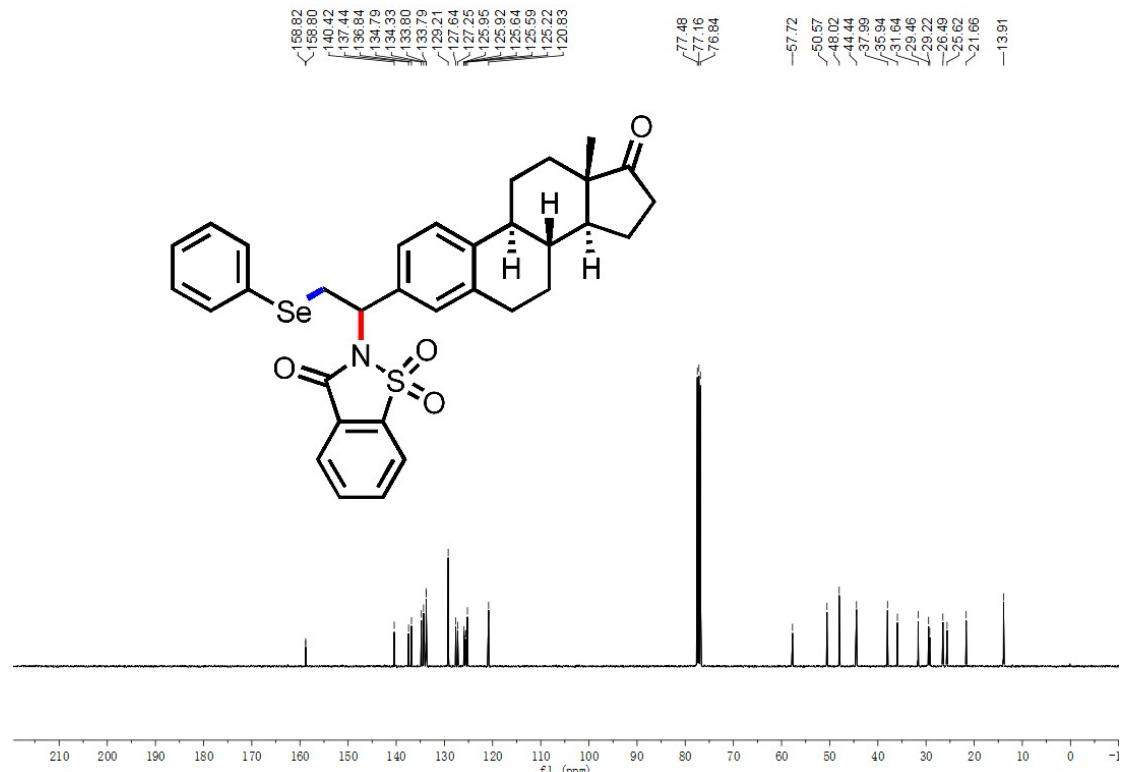
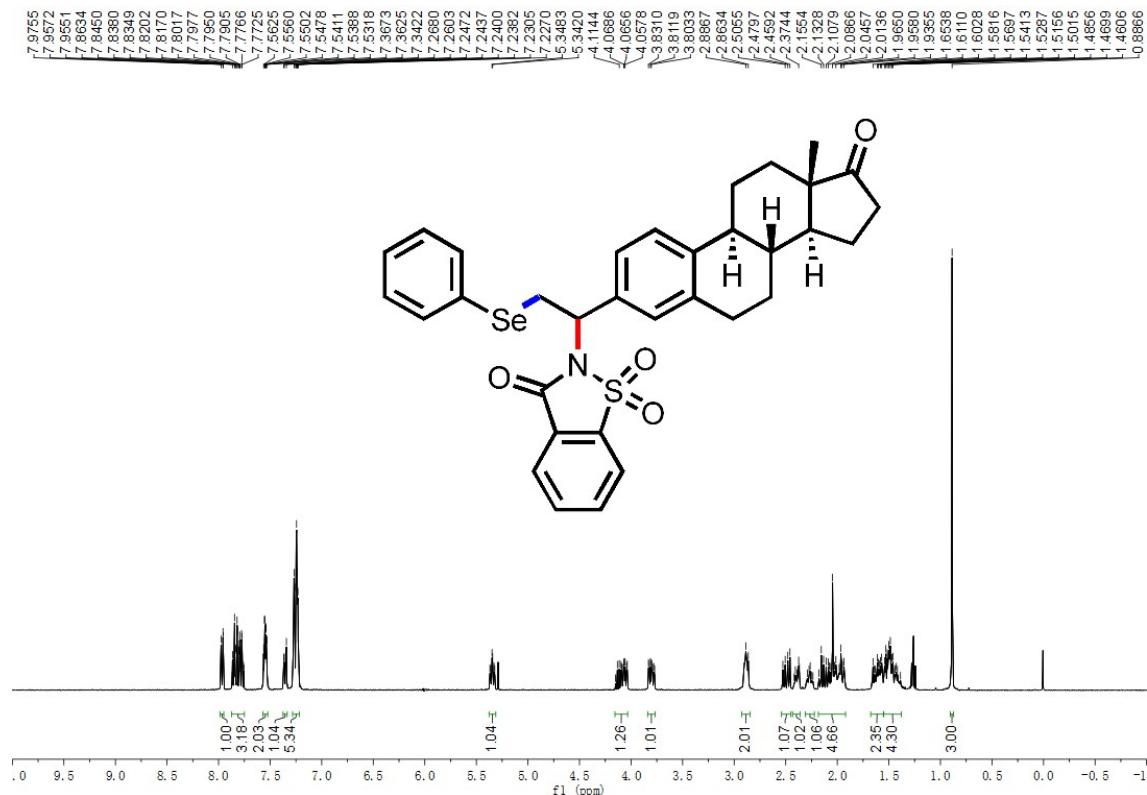
¹H NMR of product 8a in CDCl₃ (400 MHz)



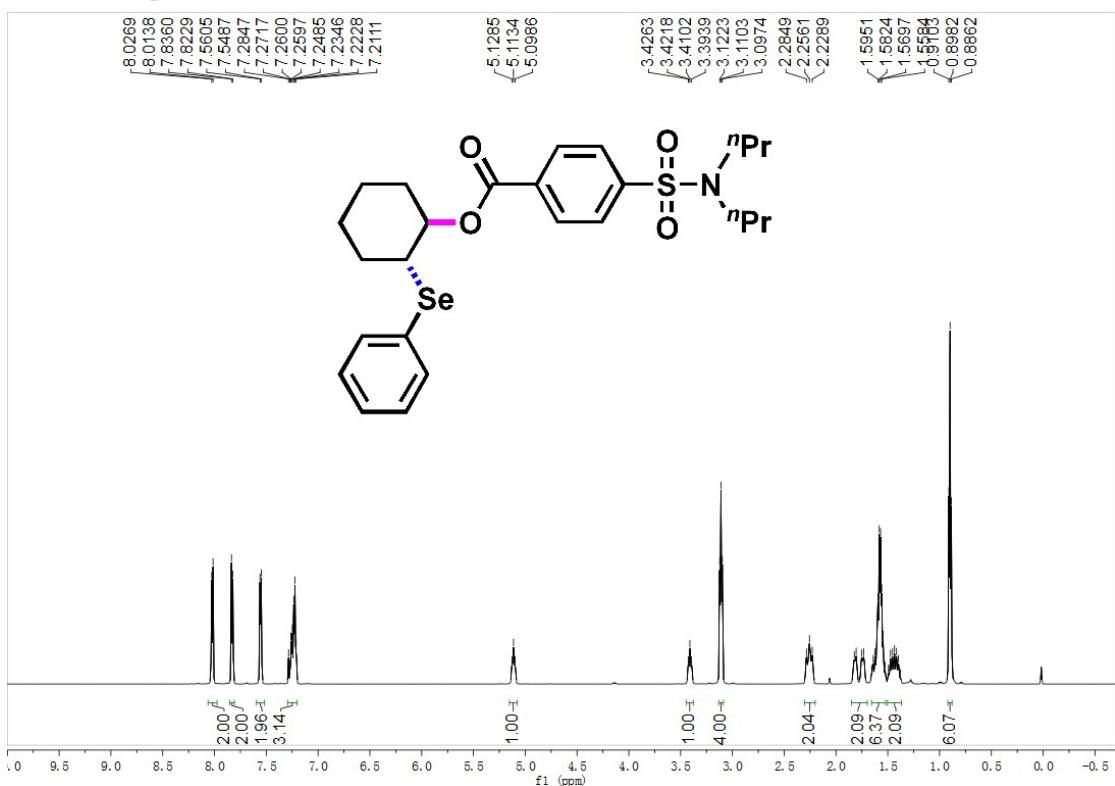
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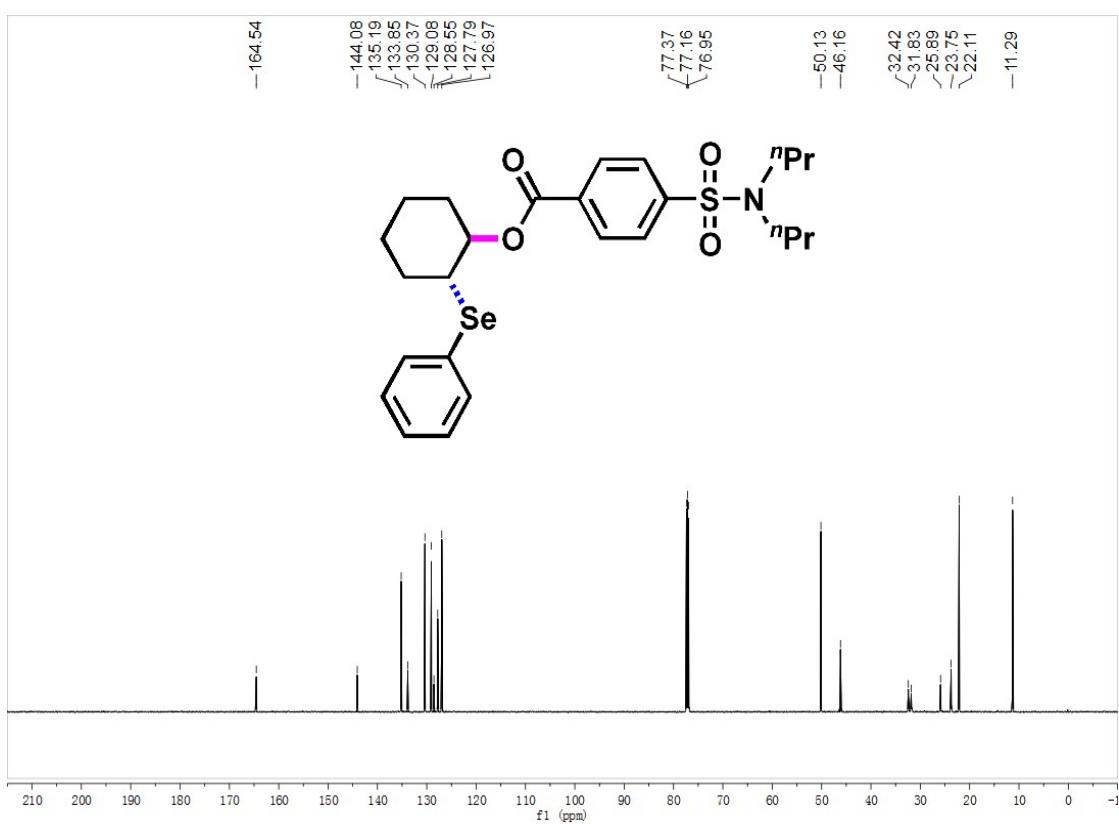
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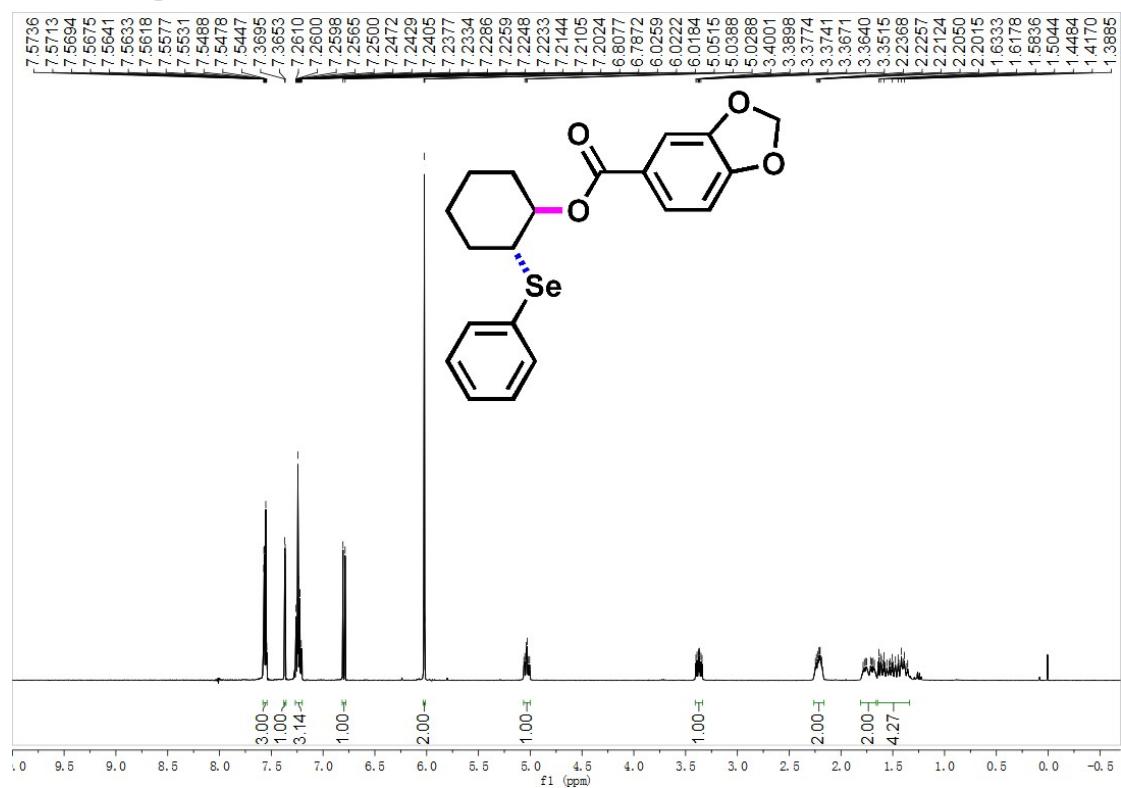
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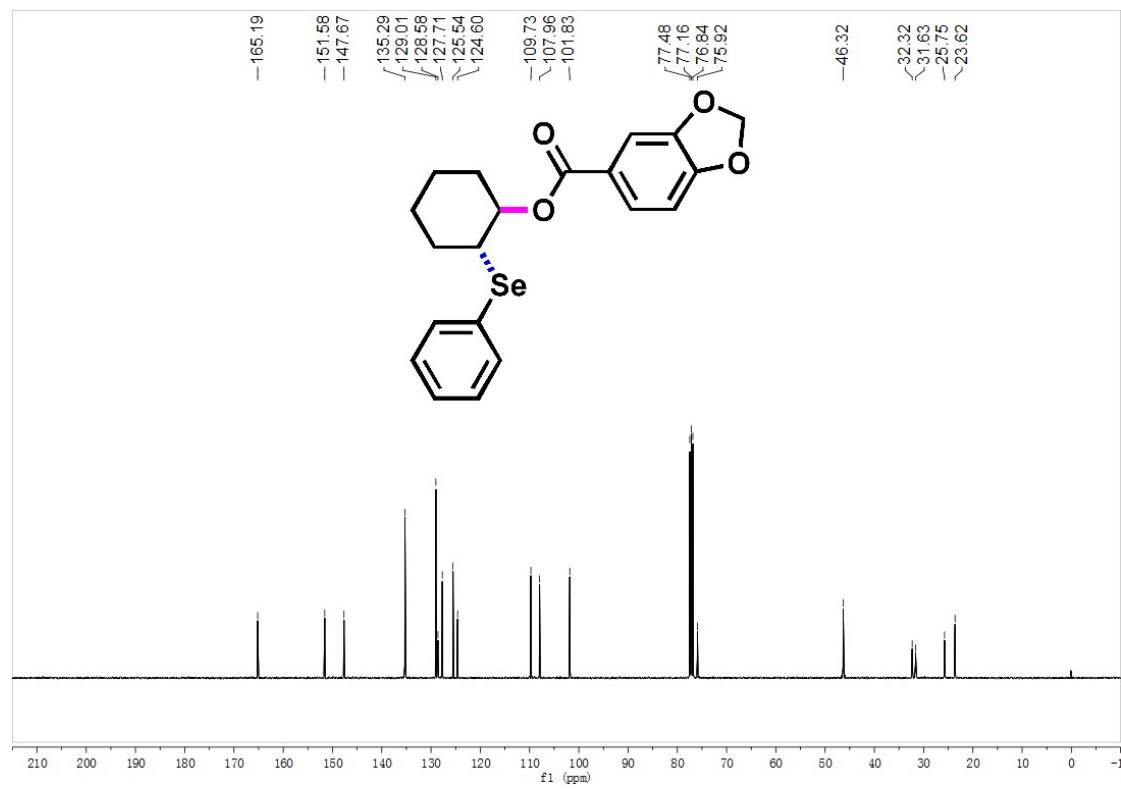
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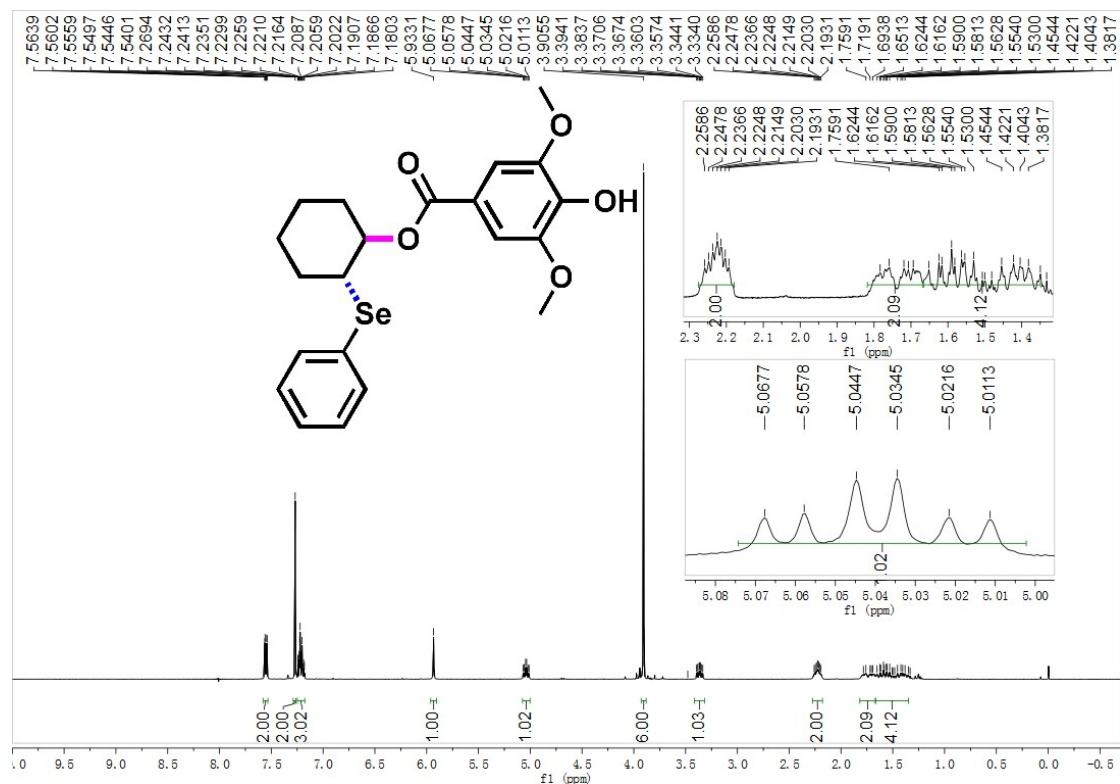
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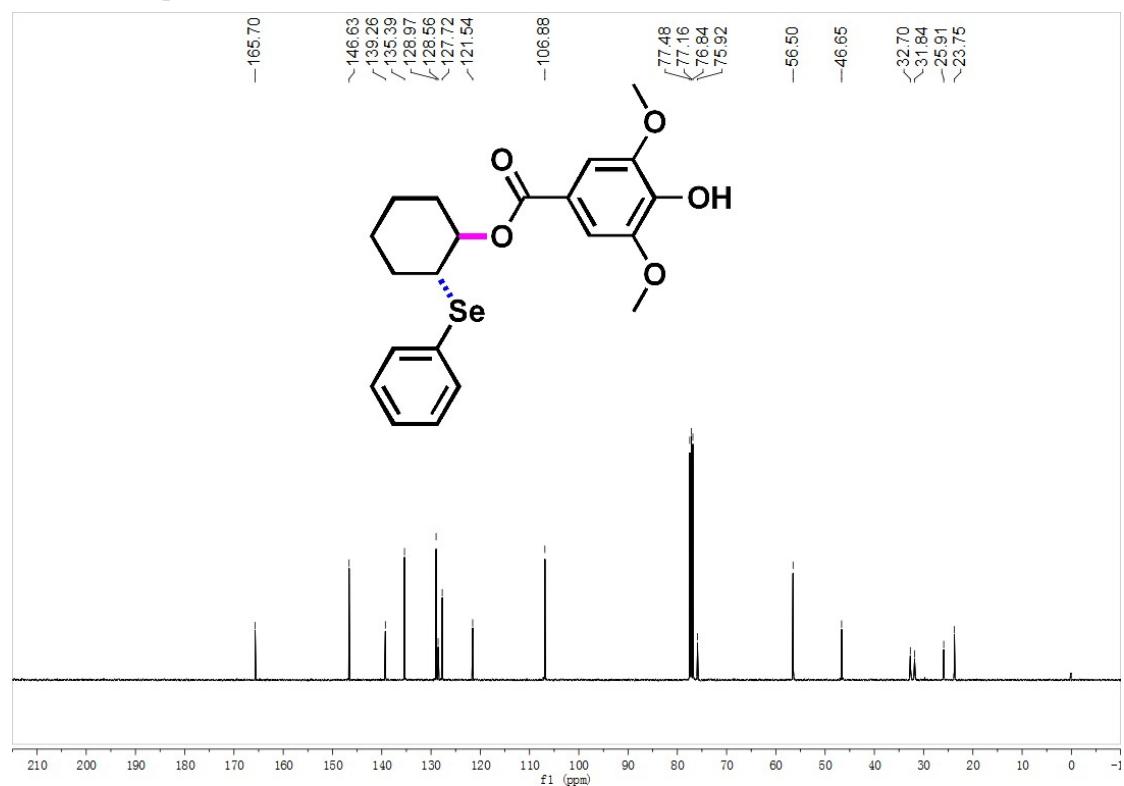
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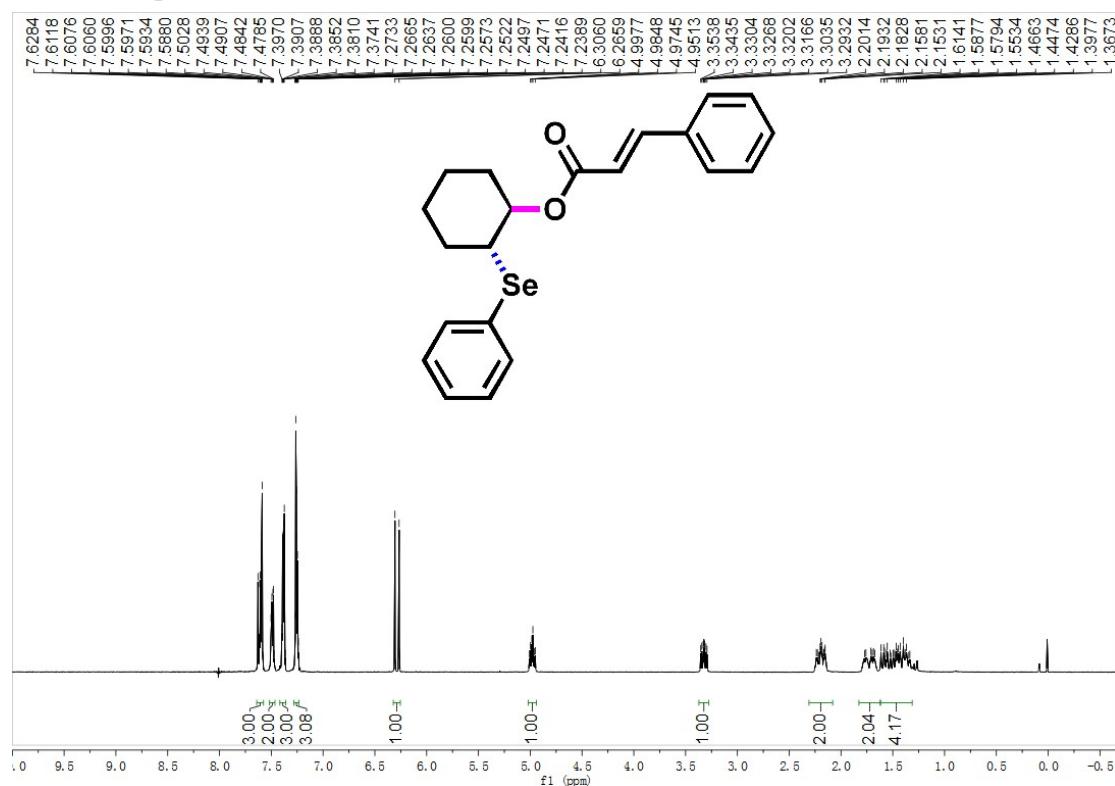
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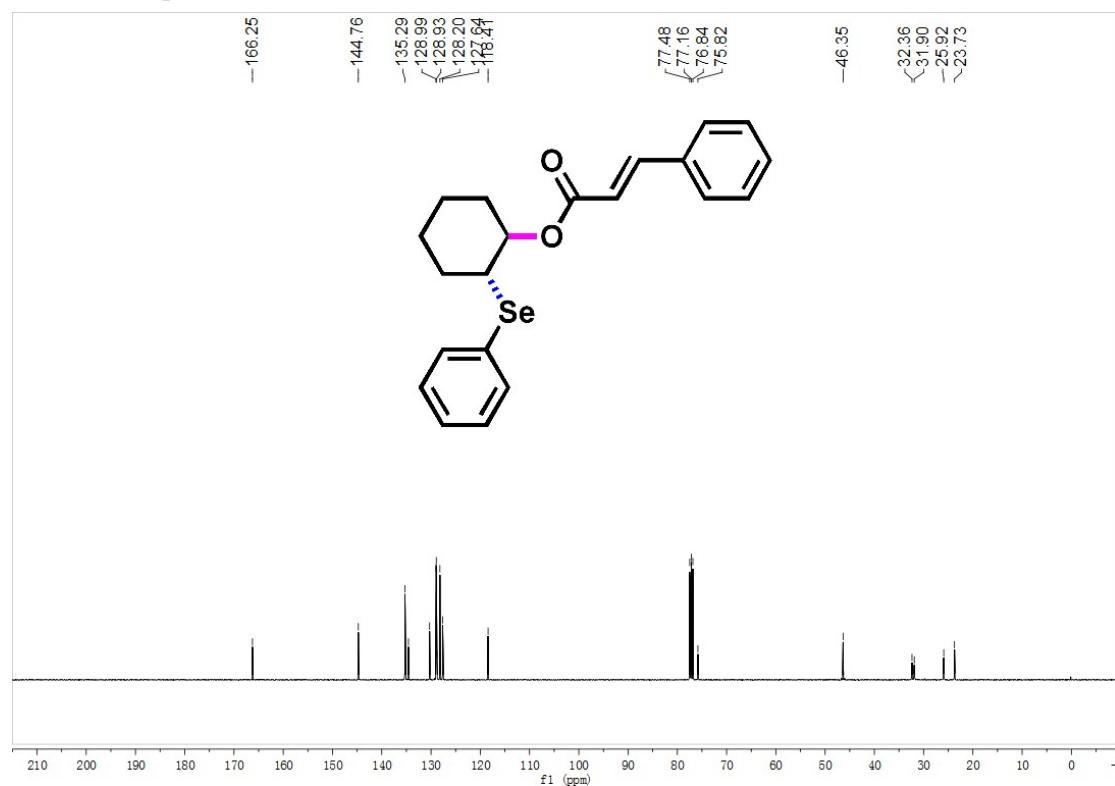
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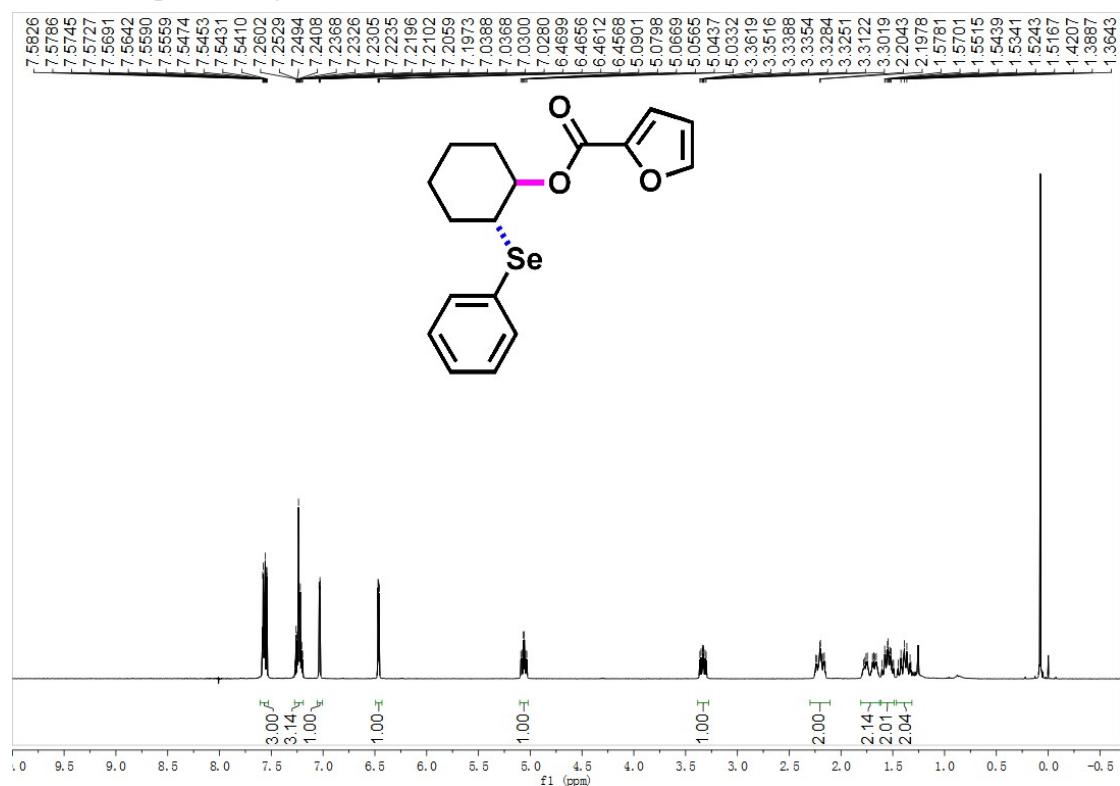
¹H NMR of product 8f in CDCl₃ (400 MHz)



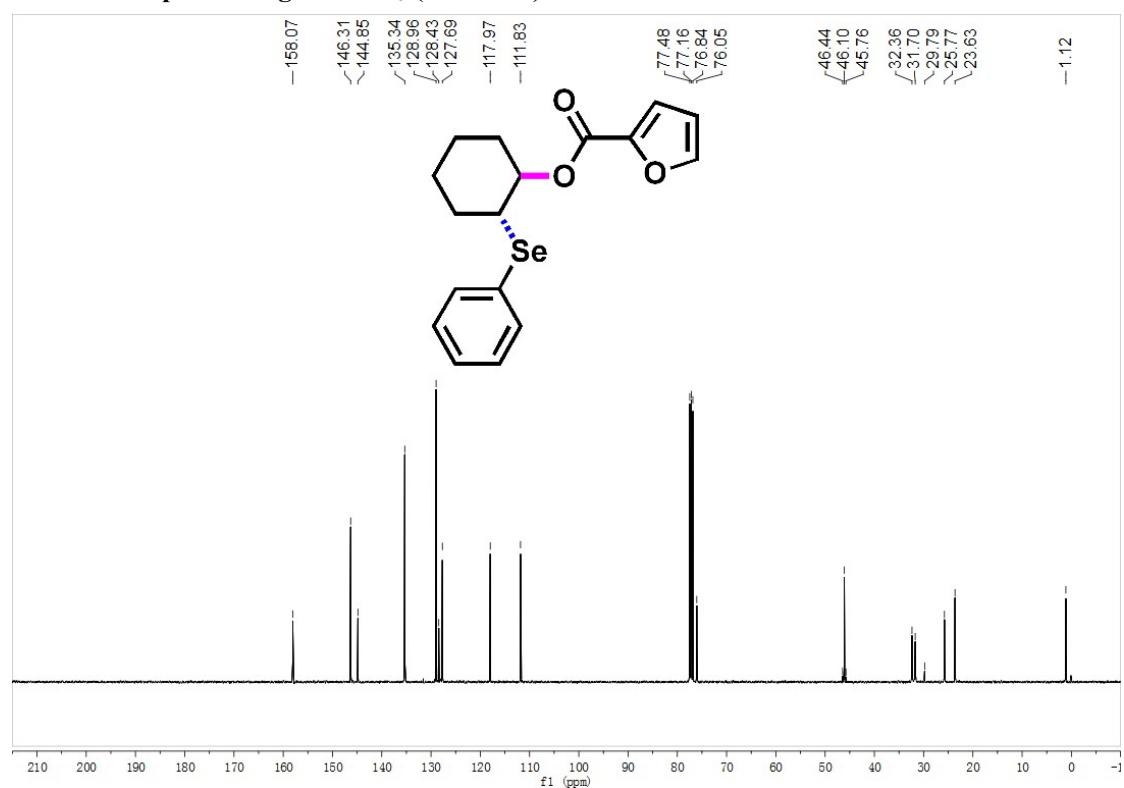
¹³C NMR of product 8f in CDCl₃ (100 MHz)



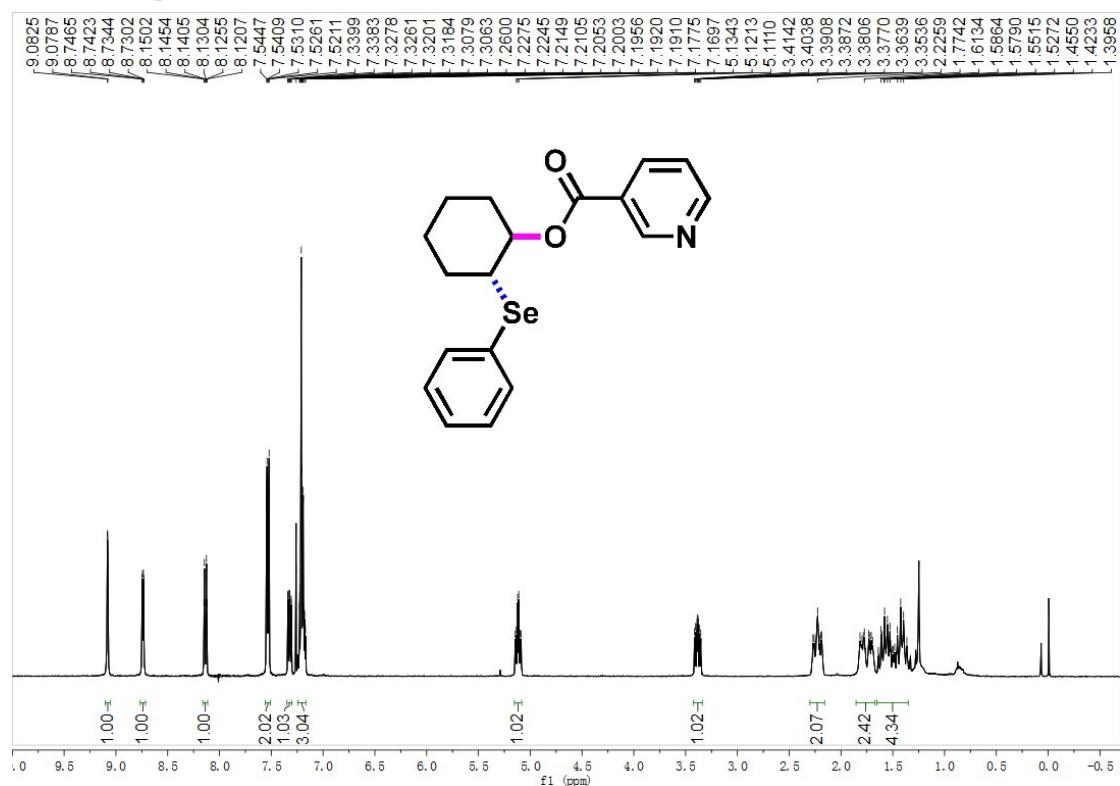
¹H NMR of product 8g in CDCl₃ (400 MHz)



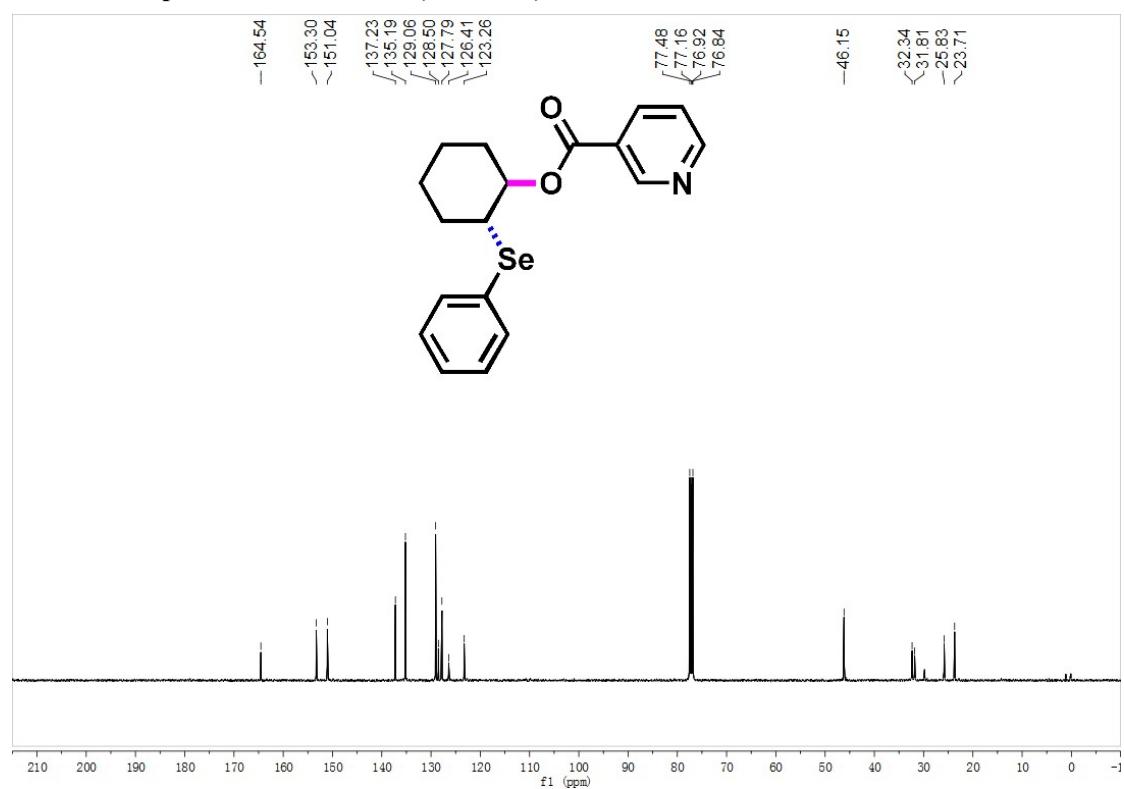
¹³C NMR of product 8g in CDCl₃ (100 MHz)



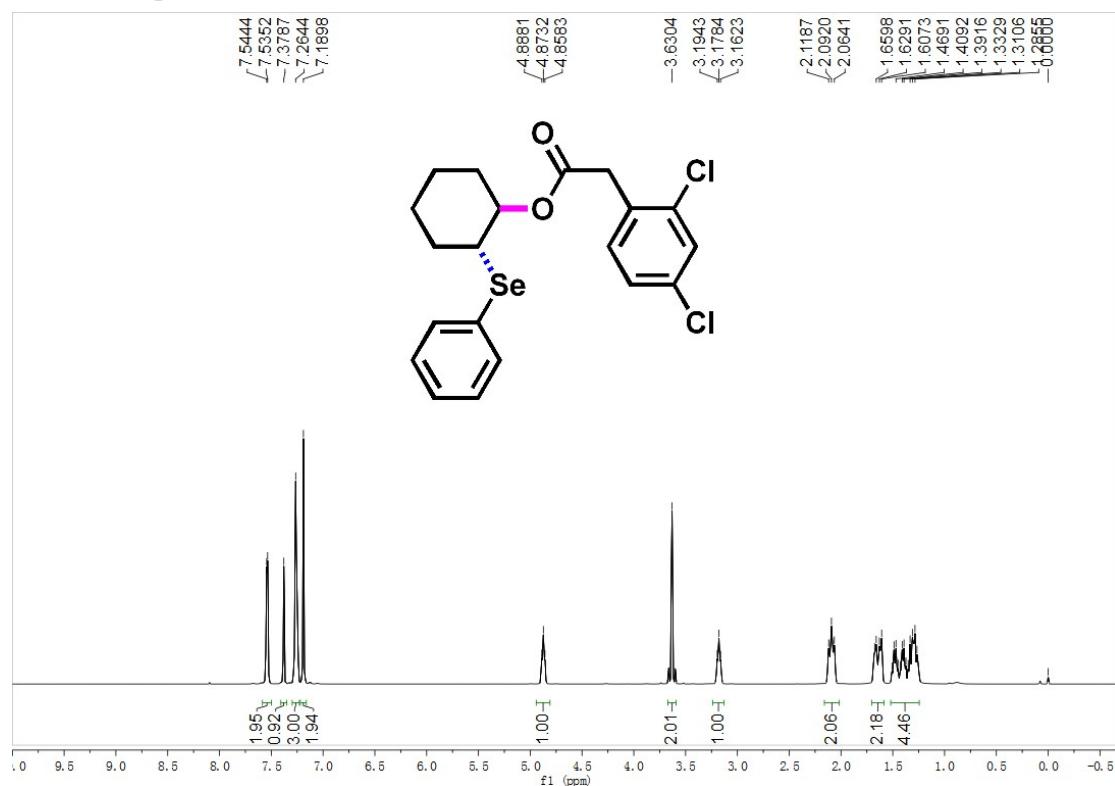
¹H NMR of product 8h in CDCl₃ (400 MHz)



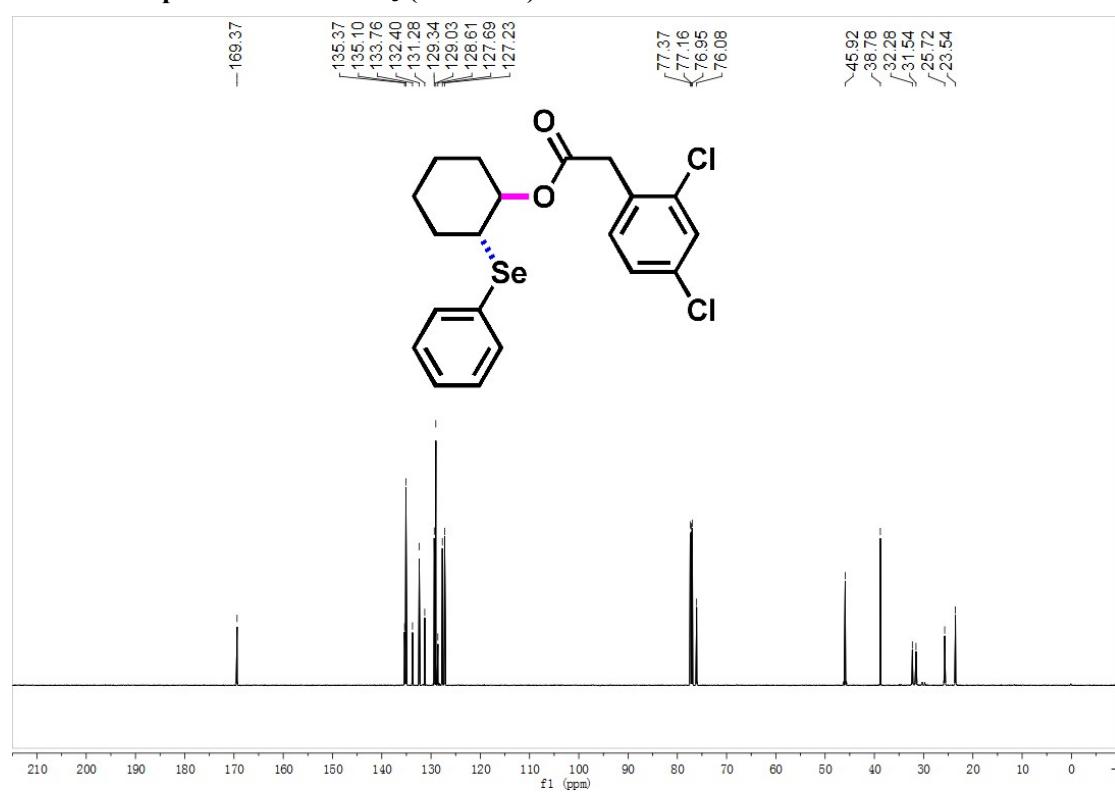
¹³C NMR of product 8h in CDCl₃ (100 MHz)



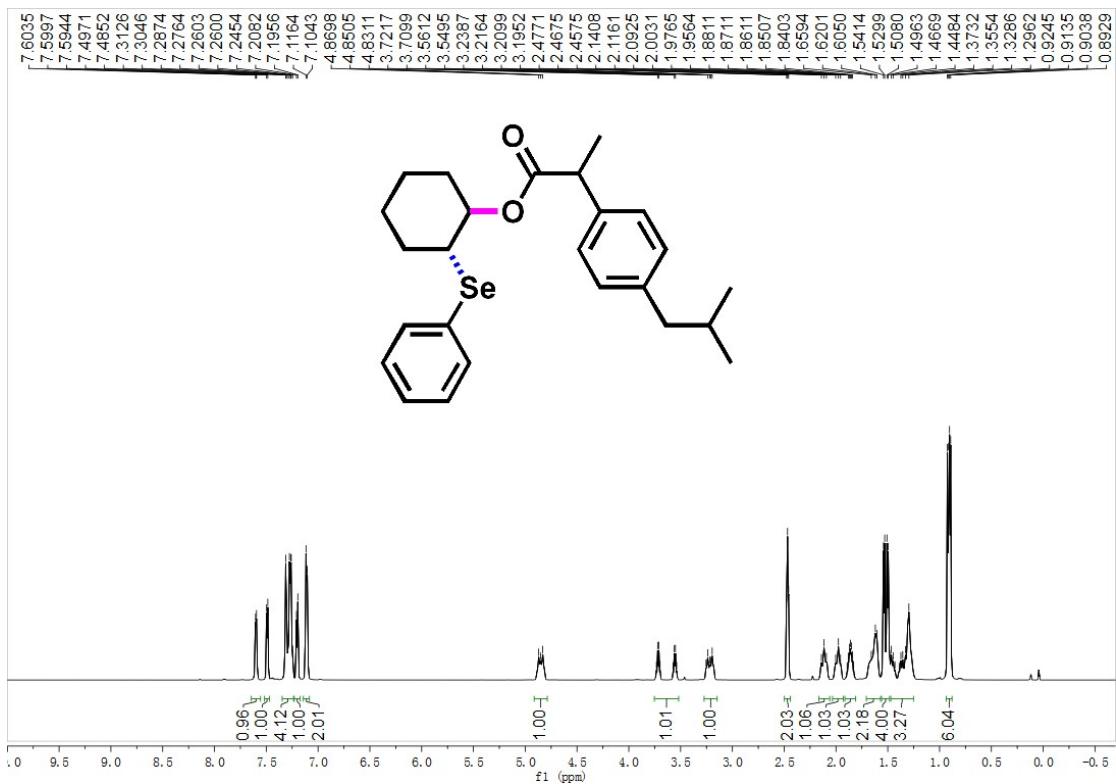
¹H NMR of product 8i in CDCl₃ (600 MHz)



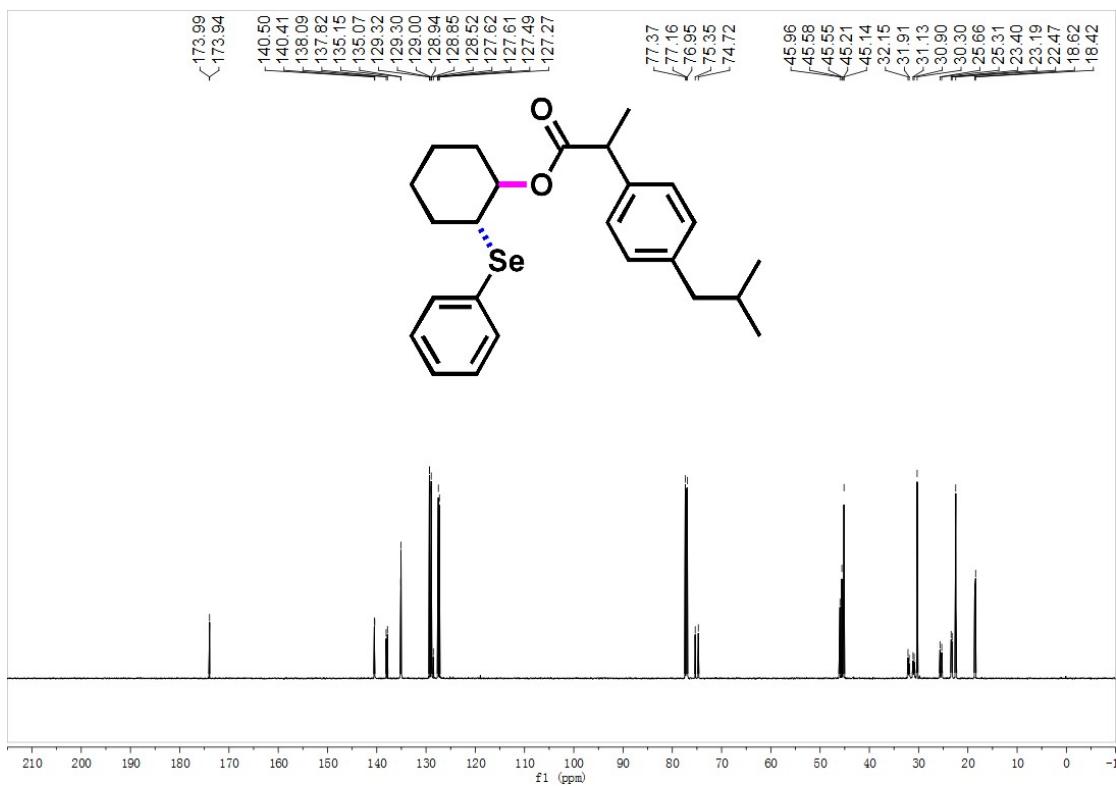
¹³C NMR of product 8i in CDCl₃ (150 MHz)



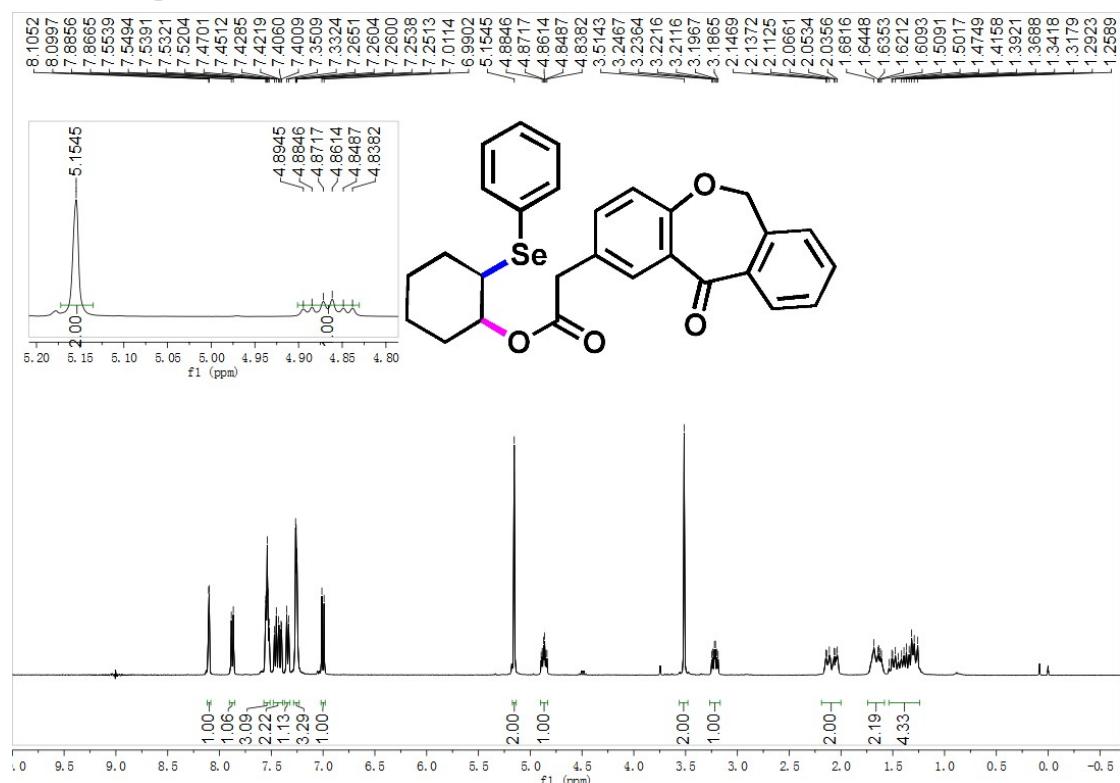
¹H NMR of product 8j in CDCl₃ (600 MHz)



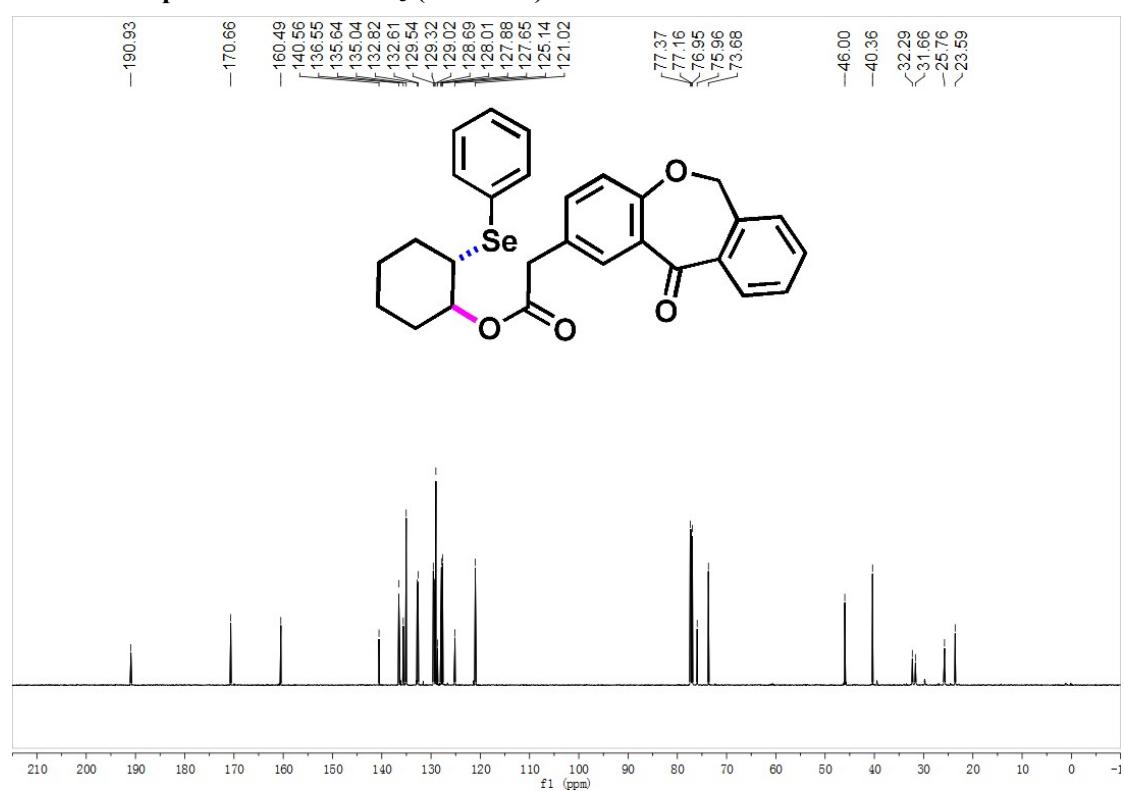
¹³C NMR of product 8j in CDCl₃ (150 MHz)



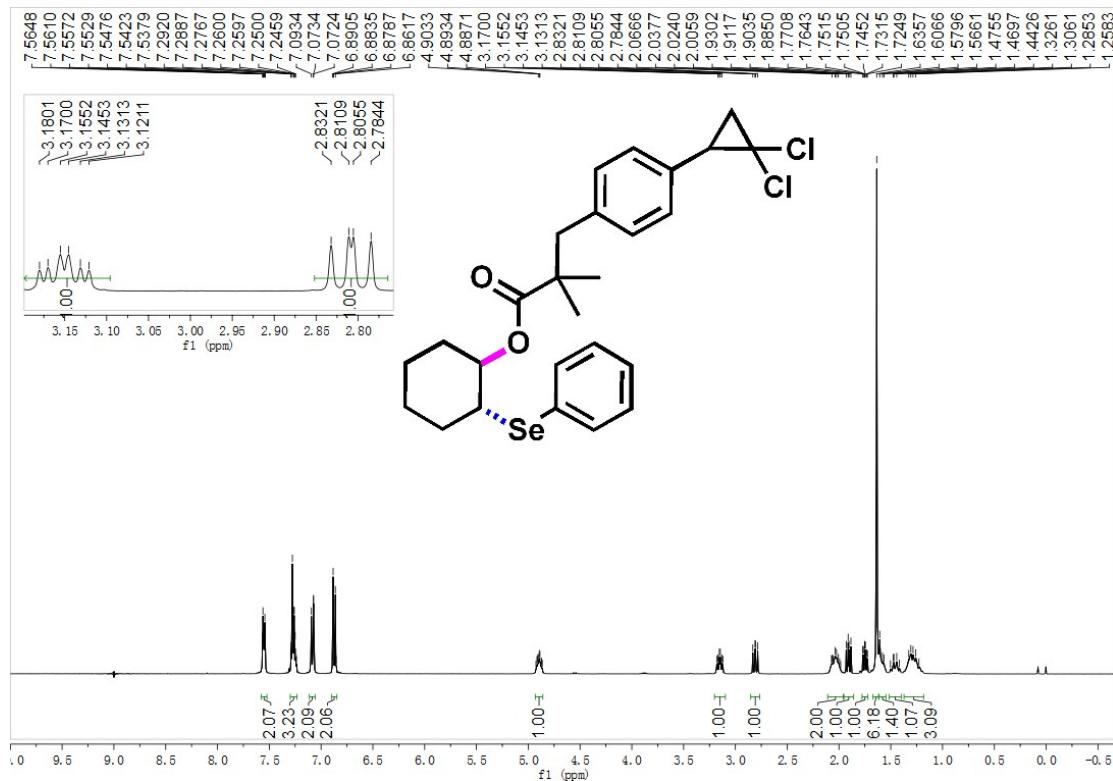
¹H NMR of product 8k in CDCl₃ (400 MHz)



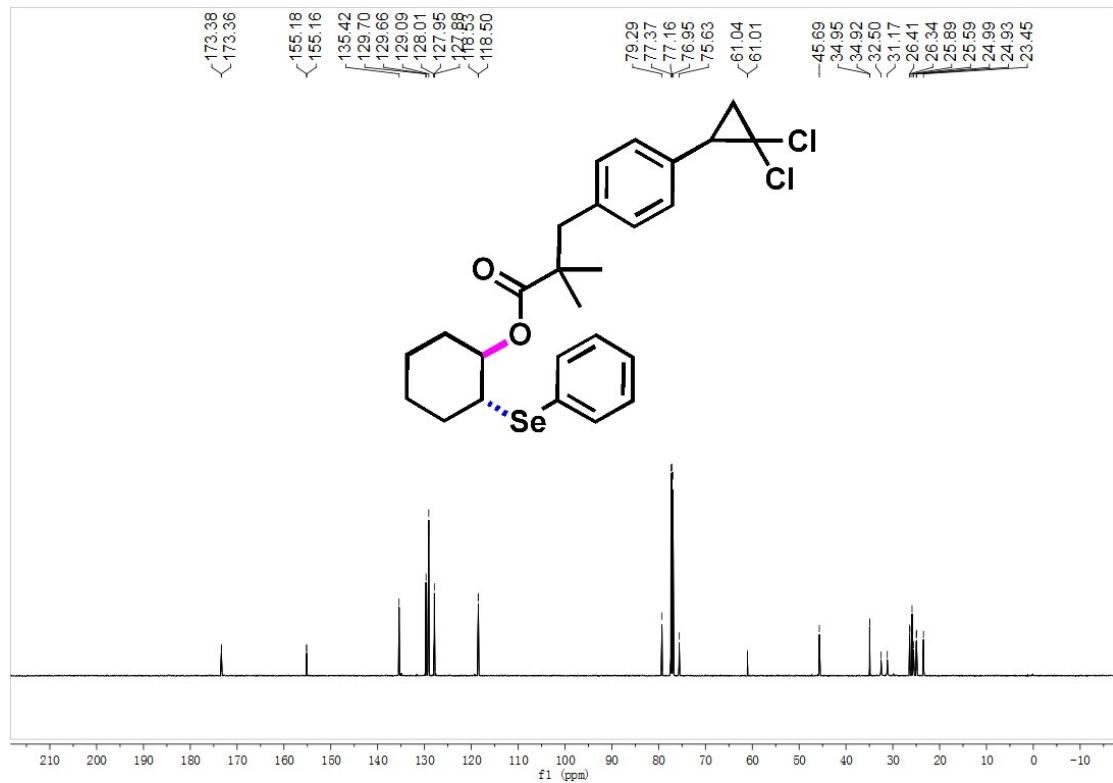
¹³C NMR of product 8k in CDCl₃ (150 MHz)



¹H NMR of product 8l in CDCl₃ (400 MHz)



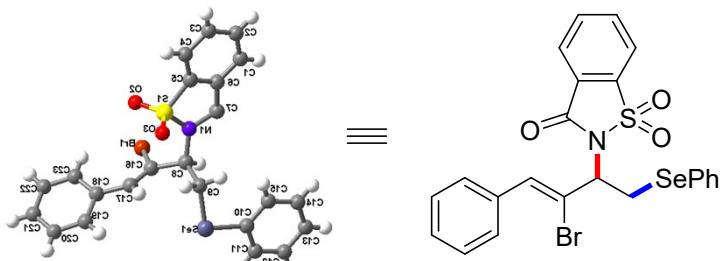
¹³C NMR of product 8l in CDCl₃ (150 MHz)



7. X-Ray crystallographic data

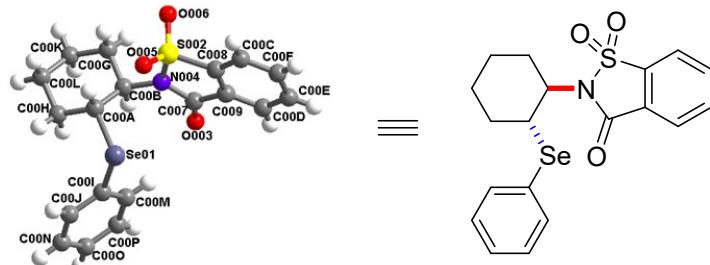
The suitable crystals were selected on a **XtaLAB Synergy, Dualflex, HyPix** diffractometer. The crystals were kept at 100.03(10) K during data collection. Using Olex2, the structures were solved with the ShelXT structure solution program using Intrinsic Phasing and refined with the ShelXL refinement package using Least Squares minimisation.

7.1 X-Ray crystallographic data for 4d' (CCDC 2233790)



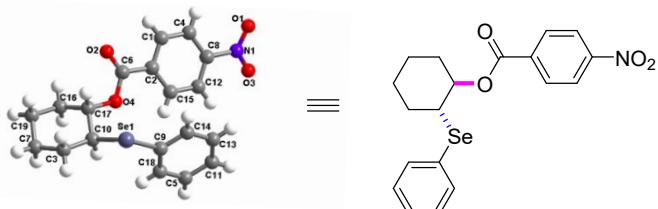
Identification code	CCDC 2233790
Empirical formula	C ₂₃ H ₁₈ BrNO ₃ SSe
Formula weight	547.31
Temperature/K	296.15
Crystal system	orthorhombic
Space group	Pbca
a/Å	9.9532(9)
b/Å	24.647(2)
c/Å	17.5152(15)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	4296.8(7)
Z	8
ρ _{calc} g/cm ³	1.692
μ/mm ⁻¹	3.730
F(000)	2176.0
Crystal size/mm ³	0.1 × 0.1 × 0.08
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.706 to 49.99
Index ranges	-11 ≤ h ≤ 11, -28 ≤ k ≤ 29, -13 ≤ l ≤ 20
Reflections collected	20591
Independent reflections	3771 [R _{int} = 0.0569, R _{sigma} = 0.0403]
Data/restraints/parameters	3771/0/271
Goodness-of-fit on F ²	1.006
Final R indexes [I>=2σ (I)]	R ₁ = 0.0333, wR ₂ = 0.0606
Final R indexes [all data]	R ₁ = 0.0625, wR ₂ = 0.0691
Largest diff. peak/hole / e Å ⁻³	0.43/-0.38

7.2 X-Ray crystallographic data for 5y (CCDC 2191956)



Identification code	CCDC 2191956
Empirical formula	C ₁₉ H ₁₉ NO ₃ SSe
Formula weight	420.37
Temperature/K	296.15
Crystal system	monoclinic
Space group	P ₂ ₁ /c
a/Å	14.852(3)
b/Å	8.3709(17)
c/Å	15.745(3)
α/°	90
β/°	111.498(4)
γ/°	90
Volume/Å ³	1821.3(6)
Z	4
ρ _{calc} g/cm ³	1.533
μ/mm ⁻¹	2.194
F(000)	856.0
Crystal size/mm ³	0.2 × 0.15 × 0.1
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	5.254 to 55.202
Index ranges	-19 ≤ h ≤ 16, -10 ≤ k ≤ 10, -18 ≤ l ≤ 20
Reflections collected	10639
Independent reflections	4149 [R _{int} = 0.0436, R _{sigma} = 0.0554]
Data/restraints/parameters	4149/0/226
Goodness-of-fit on F ²	1.033
Final R indexes [I>=2σ (I)]	R ₁ = 0.0406, wR ₂ = 0.0854
Final R indexes [all data]	R ₁ = 0.0802, wR ₂ = 0.0967
Largest diff. peak/hole / e Å ⁻³	0.34/-0.40

7.3 X-Ray crystallographic data for 7m (CCDC 2227321)



Identification code	CCDC 2227321
Empirical formula	C ₁₉ H ₁₉ NO ₄ Se
Formula weight	404.31
Temperature/K	296.15
Crystal system	monoclinic
Space group	P ₂ ₁ /n
a/Å	6.0420(6)

b/Å	7.1534(7)
c/Å	41.598(4)
$\alpha/^\circ$	90
$\beta/^\circ$	91.970(2)
$\gamma/^\circ$	90
Volume/Å ³	1796.8(3)
Z	4
$\rho_{\text{calcg}}/\text{cm}^3$	1.495
μ/mm^{-1}	2.113
F(000)	824.0
Crystal size/mm ³	? × ? × ?
Radiation	MoKα ($\lambda = 0.71073$)
2Θ range for data collection/°	5.778 to 55.252
Index ranges	-7 ≤ h ≤ 7, -7 ≤ k ≤ 9, -46 ≤ l ≤ 54
Reflections collected	10475
Independent reflections	4080 [$R_{\text{int}} = 0.0368$, $R_{\text{sigma}} = 0.0504$]
Data/restraints/parameters	4080/0/226
Goodness-of-fit on F ²	1.036
Final R indexes [I>=2σ (I)]	$R_1 = 0.0442$, $wR_2 = 0.0780$
Final R indexes [all data]	$R_1 = 0.0790$, $wR_2 = 0.0873$
Largest diff. peak/hole / e Å ⁻³	0.30/-0.45