Supporting Information

Synthesis of 1,3-diselenyl-dihydroisobenzofurans via electrochemical radical selenylation with substituted *o*-divinylbenzenes and diselenides

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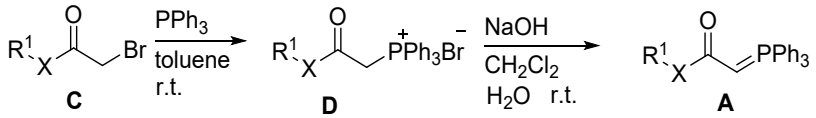
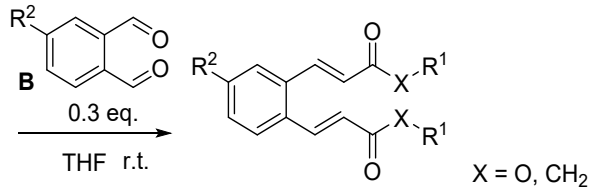
1.General Information.

All reactions were carried out under atmospheric conditions unless otherwise stated. Reagents and solvents were purchased commercially and used directly without further purification. ^{1}H NMR (600 MHz) and ^{13}C NMR (150 MHz) spectra were obtained on Bruker AV- 600 instrument in $CDCl_3$ with TMS as internal standard. Multiplicities were reported by use of the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. HRMS (ESI) spectra were recorded on a 1200-6520 Q-TOF/Agilent mass spectrometer using electrospray ionization. Flash column chromatography was performed using 200-300 mesh silica gel.

2. General Procedures for the Preparation of Substrates.

General Procedure for Syntheses of Compound 1a-1c, 1r



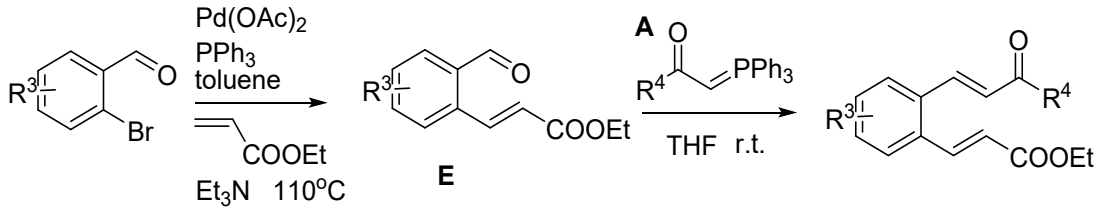


A solution of the appropriate compound **C** (26.3 mmol, 1.05 equiv.) in toluene (25 mL) was added dropwise over 10 min to a solution of triphenylphosphine (25.0 mmol, 1.0 equiv.) in toluene (75 mL). The reaction mixture was stirred at room temperature for 24 h, and the resulting phosphonium salt was filtered and oven-dried. The phosphonium salt **D** was obtained in quantitative yield, and was used without further purification. NaOH (1.2 g, 30 mmol) was dissolve in water (40 mL) was added to a suspension of the phosphonium salt **D** (5.5 mmol) in CH_2Cl_2 (50 mL). The mixture was stirred vigorously at room temperature for 1 h and then transferred to a separating funnel. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were dried (Na_2SO_4) and concentrated to give the corresponding stabilized phosphonium ylides **A**, which was used without further purification. [1]

To a solution of corresponding o-phthalaldehyde **B** (1.0 mmol, 1.0 equiv.) in THF (15 mL) was added the corresponding ester-stabilized phosphonium ylide **A** (2.4 mmol, 2.4 equiv.). The mixture was stirred at room temperature for 12 h, and concentrated in vacuo. Purification of the

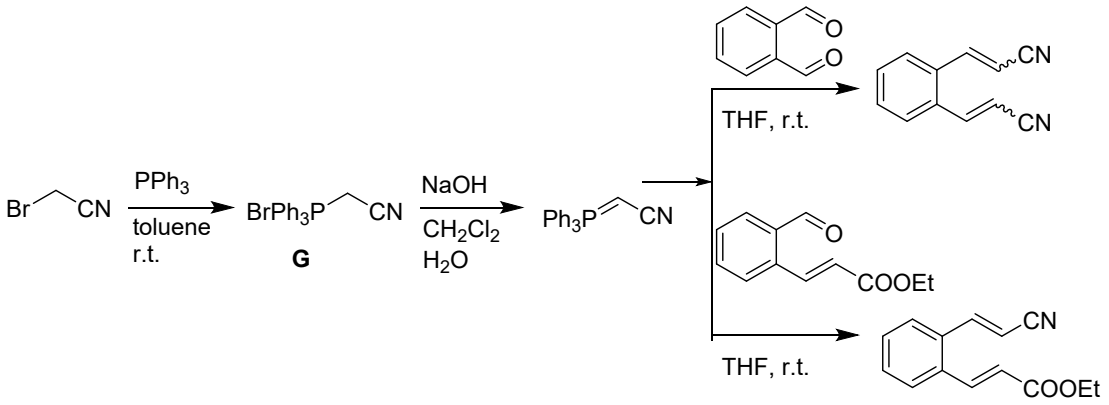
residue by column chromatography (Petroleum ether/EtOAc: 5/1) afforded the compound **1a-1c, 1r** . [2]

General Procedure for Syntheses of Compound 1d-1q



To a solution of the corresponding o-bromobenzaldehyde (1.0 equiv.) in anhydrous toluene (0.1 M) were successively added $Pd(OAc)_2$ (5 mol%), triphenylphosphine (10 mol%), ethyl acrylate (1.3 equiv.) and triethylamine (5.0 equiv.). The reaction mixture was heated at reflux for 12 h, cooled to rt, diluted with EtOAc and filtered through a thin pad of Celite. The filtrate was diluted with water and extracted with EtOAc. The organic layers were combined, dried over $MgSO_4$, and concentrated under vacuum. The dark thick oil obtained was purified by flash silica chromatography employing mixtures of Petroleum ether and EtOAc as eluents to get the corresponding (*E*)-ethyl 3-(2-formylphenyl)acrylate **E**.[3] To a solution of corresponding **(*E*)-ethyl 3-(2-formylphenyl)acrylate E** (1.0 mmol, 1.0 equiv.) in THF (15 mL) was added the appropriate ylide **A** (1.2 mmol, 1.25 equiv.). The mixture was stirred at room temperature for 12 h and concentrated in vacuo. Purification of the residue by column chromatography (Petroleum ether/EtOAc: 5/1) afforded the compound **1d-1q**.[2]

The Syntheses of Substrates 1s-1t.



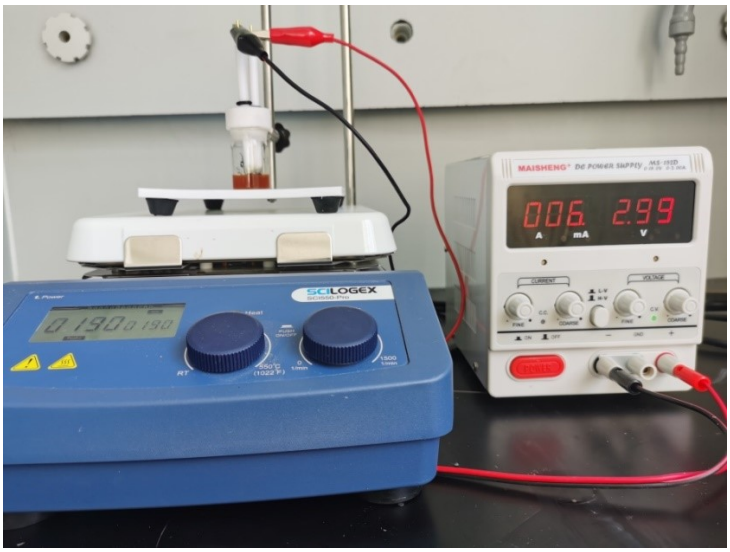
A solution of the 2-bromoacetonitrile (41.7 mmol, 1.05 equiv.) was added dropwise over 10 min to a solution of triphenylphosphine (39.8 mmol, 1.0 equiv.) in Petroleum ether (150 mL). The

reaction mixture was stirred at room temperature for 36 h, and the resulting phosphonium salt **G** was filtered and oven-dried. The phosphonium salt **G** was obtained in quantitative yield, and was used without further purification. NaOH (1.2 g, 30 mmol) dissolved in water (40 mL) was added to a suspension of the phosphonium salt **G** (5.5 mmol) in CH_2Cl_2 (50 mL). The mixture was stirred vigorously at room temperature for 1 h and then transferred to a separating funnel. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were dried (Na_2SO_4) and concentrated to give the corresponding stabilized phosphonium ylides, which was used without further purification. To a solution of corresponding *o*-phthalaldehyde (1.0 mmol, 1.0 equiv.) or **3-(2-formylphenyl)acrylate E** (1 mmol, 1.0 equiv.) in THF (15 mL) was added the corresponding ester-stabilized phosphonium ylide (2.4 equiv., for **E** 1.2 equiv.). The mixture was stirred at room temperature for 12 h, and concentrated in vacuo. Purification of the residue by column chromatography (EtOAc:hexane, 1:5) afforded the compound **1s-1t**.[4]

3. General Procedures for the Preparation of Products.

Procedure for the Synthesis of 3

A 10-mL three-necked round-bottomed flask (or 10 mL electrolytic bath) was charged with **1** (0.2 mmol), PhSeSePh **2a** (0.26 mmol, 81.8 mg), $LiBF_4$ (0.2 mmol, 18.7 mg). The flask was equipped with two platinum plates (1 cm x 1 cm) as anode and cathode respectively, then CH_3CN:H_2O 800:1 (3 mL) were added. The mixture was stirred at room temperature and electrolyzed at a constant current of 6 mA for 4 - 8 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent (eluent, petroleum ether/ethyl acetate = 15:1 to 10:1) to obtain the desired product **3**.

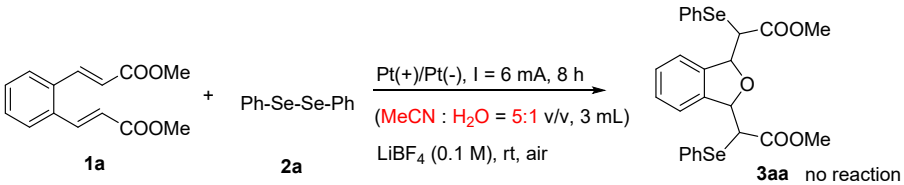


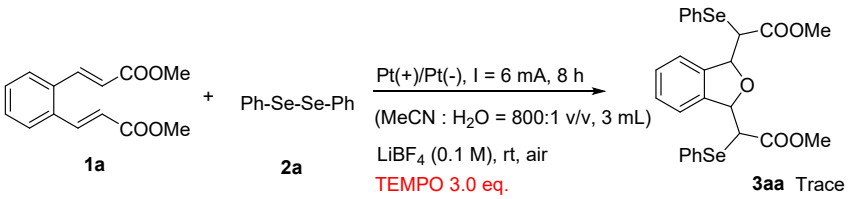
Gram-scale reaction: A 100-mL three-necked round-bottomed flask was charged with **1a** (4.06 mmol, 1.0 g), PhSeSePh **2a** (5.28 mmol, 1.6 g), $LiBF_4$ (4.06 mmol, 0.38 g). The flask was equipped with two platinum plates (2 cm x 2 cm) as anode and cathode respectively, then CH_3CN:H_2O 800:1 (60 mL) were added. The mixture was stirred at room temperature and electrolyzed at a constant current of 6 mA for 48 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ethyl acetate as eluent (eluent, petroleum ether/ethyl acetate = 15:1 to 10:1) to obtain the desired product **3aa** (3.33 mmol, 1.92 g).

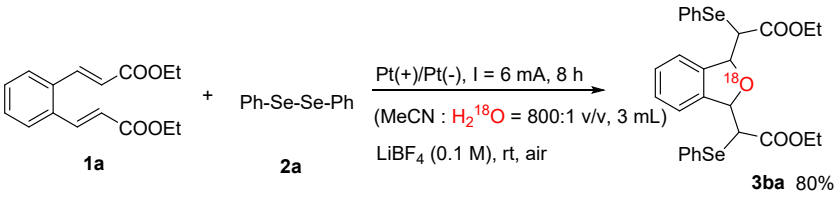
A 100-mL three-necked round-bottomed flask was charged with **1b** (3.65 mmol, 1.0 g), PhSeSePh **2a** (4.74 mmol, 1.5 g), $LiBF_4$ (3.65 mmol, 0.34 g). The flask was equipped with two platinum plates (2 cm x 2 cm) as anode and cathode respectively, then CH_3CN:H_2O 800:1 (55 mL) were added. The mixture was stirred at room temperature and electrolyzed at a constant current of 6 mA for 48 h. The reaction mixture was concentrated under reduced pressure, and the residue was purified through silica gel chromatography with petroleum/ ethyl acetate as eluent (eluent, petroleum ether/ethyl acetate = 15:1 to 10:1) to obtain the desired product **3ba** (2.85 mmol, 1.72 g).

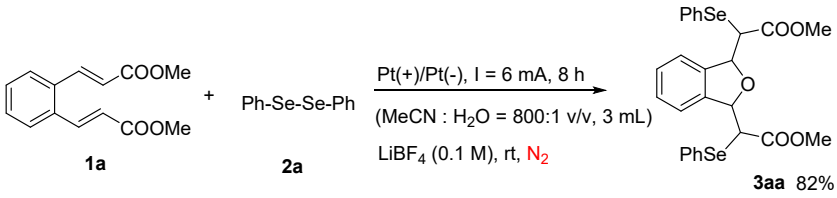
Mechanism study

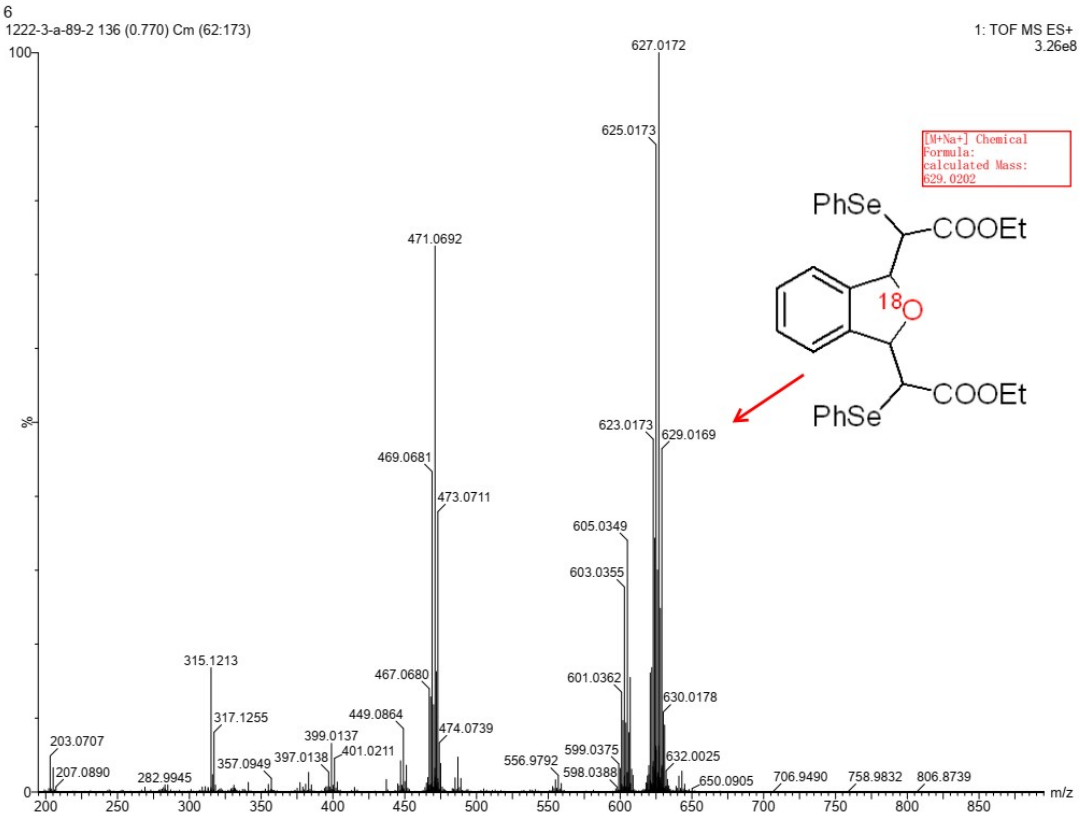
Control experiments:











This reaction did not work when the solvent is MeCN:H_2O (5:1 v/v), which means that the reaction is water sensitive and it could be a free radical pathway, probably because water could stop the radical reaction. To probe the possible reaction mechanism, the 2,2,6,6-tetramethylpiperidine N-oxide (TEMPO), a radical scavenger, was added to the reaction system, the formation of the desired product **3aa** was prohibited, indicating that a free radical was involved in the reaction. In addition, the $H_2{}^{18}O$-labeled experiment was also carried out which indicated that the oxygen atom in the framework of 1,3-dihydroisobenzofuran of the product was probably from H_2O in the system. The reation worked smoothly under N_2 atmosphere.

Cyclic voltammetry (CV) experiments

We probed the mechanism by means of a series of cyclic voltammetric (CV) analyses. As depicted in Fig. S1, the oxidative peak of **1a** was observed at 1.55 V *vs*. Ag/AgCl (curve b) in MeCN:H_2O (800:1 v/v) and the oxidative peak of **2a** at 0.75 V vs. Ag/AgCl (curve c). The compound **2a** was found to be oxidized at 0.55 V *vs*. Ag/AgCl (curve c). It could be concluded that the diphenyl diselenide **2a** was oxidized preferentially at a low potential which was lower than the oxidation potential of **1a**.

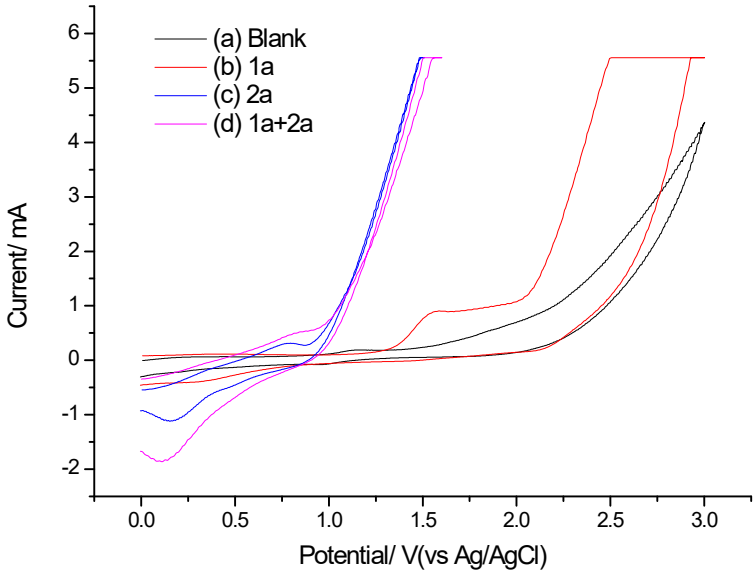
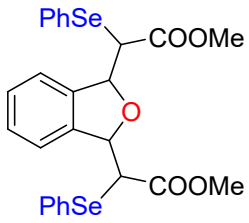


Figure S1. CVs (0.1 V/s) in MeCN:H_2O (800:1 v/v, 3 mL) and $LiBF_4$ (0.1 M) of: background (**a**); **1a** (0.2 mmol, **b**); **2a** (0.26 mmol, **c**); **1a** (0.2 mmol) + **2a** (0.26 mmol) (**d**). The voltammogram was obtained with Pt plates (1×1 cm) as the working and counter electrode and Ag/AgCl electrode as the reference electrode at room temperature.

4. Characterization of data for the electrolysis products.

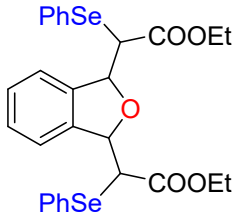


dimethyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3aa.

Yield = 84 %; 97 mg; colourless oil; R_f (EtOAc : PE = 1:10) = 0.33.

Major: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.59 – 7.47 (m, 4H), 7.35 (dd, *J* = 5.7, 3.2 Hz, 2H), 7.31 – 7.19 (m, 6H), 5.60 (d, *J* = 8.1 Hz, 2H), 3.98 (d, *J* = 8.2 Hz, 2H), 3.65 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.6, 139.5, 135.1, 129.1, 128.5, 128.3, 127.9, 123.7, 82.0, 52.1, 50.0. Minor: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.57 – 7.50 (m, 4H), 7.33 (dd, *J* = 5.7, 3.1 Hz, 2H), 7.30 – 7.21 (m, 6H), 5.80 (d, *J* = 5.9 Hz, 2H), 3.93 (d, *J* = 6.1 Hz, 2H), 3.63 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.4, 139.9, 134.8, 129.1, 128.6, 128.4, 128.3, 123.1, 82.4, 52.1, 50.1.

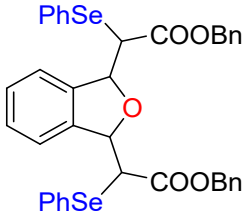
HRMS (ESI) calculated for $C_{26}H_{24}NaO_5Se_2$ [M+Na]+: 598.9846; found: 598.9832.



diethyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3ba.

Yield = 86 %; 104 mg; colourless oil; R_f (EtOAc : PE = 1:10) =0.47.

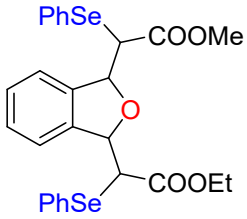
Major: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.61 – 7.55 (m, 4H), 7.40 – 7.35 (m, 2H), 7.31 (ddd, *J* = 6.5, 3.7, 1.2 Hz, 2H), 7.29 – 7.22 (m, 4H), 5.65 (d, *J* = 7.8 Hz, 2H), 4.21 – 4.08 (m, 4H), 4.04 (d, *J* = 7.8 Hz, 2H), 1.19 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.3, 139.6, 135.1, 129.0, 128.4, 128.3, 128.1, 123.7, 82.0, 61.1, 50.18, 14.0. Minor: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.59 – 7.51 (m, 4H), 7.32 (dd, *J* = 5.7, 3.1 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.26 – 7.23 (m, 4H), 5.80 (d, *J* = 5.9 Hz, 2H), 4.27 – 4.01 (m, 4H), 3.92 (d, *J* = 6.0 Hz, 2H), 1.13 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.0, 139.9, 134.8, 129.0, 128.7, 128.3, 128.2, 123.0, 82.5, 61.1, 50.3, 13.9. HRMS (ESI) calculated for $C_{28}H_{28}NaO_5Se_2$ [M+Na]+: 627.0159; found: 627.0161



dibenzyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3ca.

Yield = 74 %; 107 mg; colourless oil; R_f (EtOAc : PE = 1:10) = 0.45.

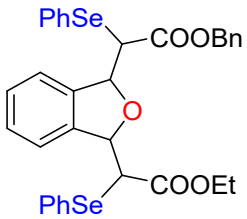
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.47 – 7.43 (m, 4H), 7.31 (m, 8H), 7.25 – 7.16 (m, 6H), 7.10 (m, 4H), 5.65 (d, *J* = 7.5 Hz, 2H), 5.13 (d, *J* = 12.4 Hz, 2H), 5.01 (d, *J* = 12.4 Hz, 2H), 4.09 (d, *J* = 7.6 Hz, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.1, 139.5, 135.6, 135.2, 129.0, 128.4, 128.4, 128.3, 128.1, 128.1, 127.9, 123.6, 82.0, 66.7, 49.9. HRMS (ESI) calculated for $C_{38}H_{32}NaO_5Se_2$ [M+Na]+: 751.0472; found: 751.0450



ethyl 2-(3-(2-methoxy-2-oxo-1-(phenylselanyl)ethyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3da.

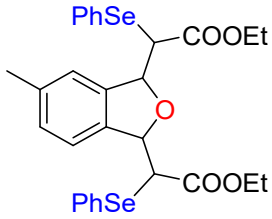
Yield = 87 %; 103 mg; colourless oil; R_f (EtOAc : PE = 1:10) = 0.49.

Major: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.83 (m,2H), 7.63 – 7.55 (m, 4H), 7.33 – 7.24 (m, 8H), 5.66 (d, *J* = 4.2 Hz, 2H), 4.20 – 4.12 (d,2H), 4.01 (d, *J* = 7.9 Hz, 2H), 3.68 (s, 3H), 1.20 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 171.3, 139.61, 139.56, 135.22, 135.17, 129.15, 129.11, 128.54, 128.44, 128.41, 128.39, 123.7 , 82.10, 82.0, 61.15, 52.12, 50.28 ,49.95, 14.04. Minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 – 7.68 (m, 2H), 7.61 – 7.59 (m, 4H), 7.40 – 7.33 (m, 8H), 5.84 (d, *J* = 5.9 Hz, 2H), 4.12 – 4.07 (d, 2H), 3.96 (d, *J* = 6.0, 3.7 Hz, 2H), 3.66 (s, 3H), 1.16 (t, *J* = 7.1 Hz,3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.50, 171.04, 139.98, 139.94, 134.86, 134.83, 128.74, 128.62, 128.36, 128.26 , 128.17, 127.95, 123.16, 123.09, 82.51, 82.46, 61.13, 52.19, 50.31, 50.19, 13.99. HRMS (ESI) calculated for $C_{27}H_{26}NaO_5Se_2$ [M+Na]+: 613.0003; found: 612.9992

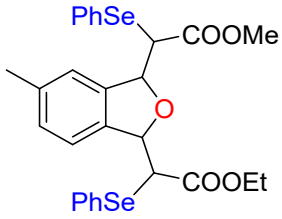


benzyl 2-(3-(2-ethoxy-2-oxo-1-(phenylselanyl)ethyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3ea.

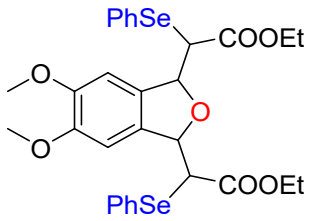
Yield = 70 %; 93 mg; colourless oil; R_f (EtOAc : PE = 1:10) = 0.45.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.59 (m, 2H), 7.58 – 7.42 (m, 4H), 7.40 – 6.98 (m, 13H), 5.89 – 5.54 (m, 2H), 5.23 – 4.95 (m, 2H), 4.15 – 3.86 (m, 4H), 1.20 – 0.90 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 170.9, 139.9, 139.6, 135.6, 135.2, 134.8, 129.1, 129.1, 128.5, 128.4, 128.2, 128.1, 128.0, 123.7, 123.1, 82.3, 66.8, 61.1, 50.2, 14.0. HRMS (ESI) calculated for $C_{33}H_{30}NaO_5Se_2$ [M+Na]+: 689.0316; found: 689.0306



diethyl 2,2'-(5-methyl-1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3fa.
Yield = 67 %; 83 mg; yellow oil; R_f (EtOAc : PE = 1:10) = 0.49.
Major: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 7.9 Hz, 1H), 7.56 (tt, J = 7.5, 4.0 Hz, 5H), 7.31 – 7.16 (m, 7H), 5.59 (dd, J = 7.4, 4.6 Hz, 2H), 4.16 – 4.06 (m, 4H), 4.01 (dd, J = 7.9, 5.6 Hz, 2H), 2.38 (s, 3H), 1.16 (td, J = 7.1, 1.5 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.40, 171.34 (s), 139.84 , 138.26 , 136.81 , 135.20 , 129.21 , 129.07 , 128.39 , 128.20 , 124.15 , 123.40 , 82.04 , 81.93 , 61.10 , 50.35 , 50.30 , 21.50 , 14.04. Minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.43 (m, 6H), 7.32 – 7.00 (m, 7H), 5.77 (d, J = 6.0 Hz, 2H), 4.20 – 4.06 (m, 6H), 2.36 (s, 3H), 1.15 – 1.10 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.14 , 171.06 , 140.22 , 138.19 , 137.19 , 135.15 , 134.79 , 128.88 , 128.33 , 128.23 , 123.54 , 122.76 , 82.49 , 82.38 , 61.07 , 50.57 , 50.45 , 21.50 , 13.98. HRMS (ESI) calculated for $C_{29}H_{30}NaO_5Se_2$ [M+Na]+: 641.0316; found: 641.0305



ethyl 2-(3-(2-methoxy-2-oxo-1-(phenylselanyl)ethyl)-5-methyl-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3ga.
Yield = 62 %; 75 mg; yellow oil; R_f (EtOAc : PE = 1:10) = 0.47.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.48 (m, 6H), 7.34 – 6.92 (m, 7H), 5.67 (dd, J = 76.5, 7.0 Hz, 2H), 4.29 – 3.84 (m, 4H), 3.78 – 3.54 (m, 3H), 2.37 (d, J = 7.4 Hz, 3H), 1.32 – 1.00 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 171.3, 139.7, 138.2, 136.7, 135.2, 134.8, 129.2, 129.1, 128.3, 124.1, 123.4, 82.1, 60.8, 52.1, 50.4, 50.0, 21.5, 14.0. HRMS (ESI) calculated for $C_{28}H_{28}NaO_5Se_2$ [M+Na]+: 627.0159; found: 627.0164



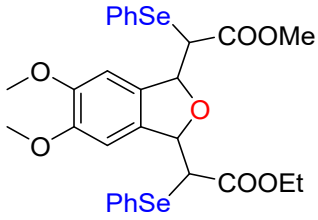
Diethyl 2,2'-(5,6-dimethoxy-1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3ha.

Yield = 63%; 83 mg; colourless oil; R_f (EtOAc : PE = 1:5) = 0.45.

Minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.50 (m, 4H), 7.32 – 7.20 (m, 8H), 5.77 (d, *J* = 5.6 Hz, 2H), 4.13 (ddd, *J* = 7.9, 6.4, 4.3 Hz, 4H), 3.93 (d, *J* = 5.7 Hz, 2H), 3.85 (s, 6H), 1.17 (t, *J* = 4.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.29, 149.2, 134.58 , 131.87 , 129.08 , 128.88 , 128.46 , 105.74 , 82.68 , 61.11 , 56.01 , 50.77 , 14.03.

Major: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.46 (m, 4H), 7.33 – 7.13 (m, 8H), 5.58 (d, *J* = 7.4 Hz, 2H), 4.22 – 4.10 (m, 4H), 4.07 (d, *J* = 7.4 Hz, 2H), 3.85 (s, 6H), 1.18 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.56 , 149.26 , 134.67 , 131.42 , 129.11 , 128.30 , 128.18 , 106.26 , 82.24 , 61.13 , 56.01 , 50.45 , 14.06.

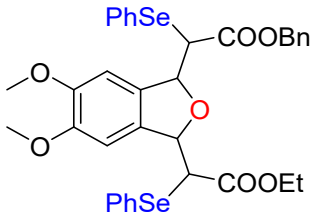
HRMS (ESI) calculated for $C_{30}H_{33}O_7Se_2$ [M+H]+:665.0551; found: 665.0539



ethyl 2-(5,6-dimethoxy-3-(2-methoxy-2-oxo-1-(phenylselanyl)ethyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3ia.

Yield = 72 %; 93 mg; colourless oil; R_f (EtOAc : PE = 1:5) = 0.47.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.40 (m, 3H), 7.37 – 7.02 (m, 9H), 5.89 – 5.43 (m, 2H), 4.06 (dddd, *J* = 39.0, 9.9, 6.3, 4.3 Hz, 4H), 3.85 (s, 6H), 3.67 (d, *J* = 4.0 Hz, 3H), 1.18 (dt, *J* = 10.4, 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 171.5, 149.2, 134.6, 131.3, 129.1, 129.1, 128.3, 128.3, 106.2, 82.2, 61.1, 56.0, 52.1, 50.5, 50.1, 14.0. HRMS (ESI) calculated for $C_{29}H_{30}NaO_7Se_2$ [M+Na]+: 673.0214; found: 673.0212.

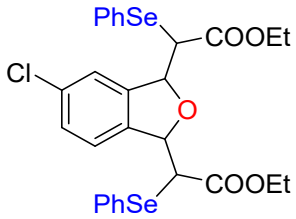


benzyl 2-(3-(2-ethoxy-2-oxo-1-(phenylselanyl)ethyl)-5,6-dimethoxy-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3ja.

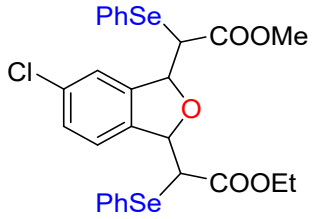
Yield = 48 %; 69 mg; colourless oil; R_f (EtOAc : PE = 1:5) = 0.45.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (m, 4H), 7.39 – 6.96 (m, 13H), 5.96 – 5.50 (m, 2H), 5.12 (dt, *J* = 10.3, 9.2 Hz, 2H), 4.25 – 3.90 (m, 4H), 3.87 – 3.55 (m, 6H), 1.30 –

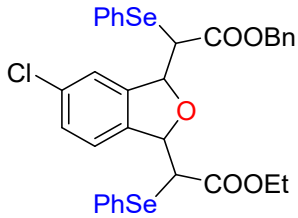
1.03 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 171.4, 149.2, 135.5, 134.8, 134.6, 131.3, 129.1, 128.4, 128.3, 128.2, 128.1, 106.2, 82.2, 66.8, 61.1, 55.9, 50.2, 14.0.
HRMS (ESI) calculated for $C_{35}H_{34}NaO_7Se_2$ [M+Na]+: 749.0527; found: 749.0526.



diethyl 2,2'-(5-chloro-1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3ka.
Yield = 72 %; 92 mg; pale yellow oil; R_f (EtOAc : PE = 1:10) = 0.35.
Major: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.50 (m, 6H), 7.42 – 7.08 (m, 7H), 5.58 (d, *J* = 7.7 Hz, 2H), 4.23 – 4.04 (m, 4H), 4.01 (dd, *J* = 7.7, 1.5 Hz, 2H), 1.29 – 1.02 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.30 , 171.17 , 141.55 , 138.12 , 135.29 , 135.12 , 134.33 , 129.17 , 128.62 , 128.46 , 128.00 , 124.84 , 124.07 , 81.77 , 81.72 , 61.27 , 49.94 , 49.83 , 14.0. Minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.52 (m, 6H), 7.36 – 7.16 (m, 7H), 5.88 – 5.71 (m, 2H), 4.16 – 4.09 (m, 4H), 3.89 (dd, *J* = 5.7, 2.5 Hz, 2H), 1.26 – 1.10 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.02 , 170.88 , 142.00 , 138.57 , 135.29 , 134.83 , 134.27 , 128.55 , 128.46 , 128.39 , 127.91 , 124.30 , 123.58 , 82.20 , 81.72 , 61.23 , 50.15 , 50.02 , 13.99. HRMS (ESI) calculated for $C_{28}H_{27}ClNaO_5Se_2$ [M+Na]+: 660.9770; found: 660.9779.



ethyl 2-(5-chloro-3-(2-methoxy-2-oxo-1-(phenylselanyl)ethyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3la.
Yield = 50 %; 62 mg; yellow oil; R_f (EtOAc : PE = 1:10) = 0.36.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.49 (m, 6H), 7.42 – 6.97 (m, 7H), 5.64 (t, *J* = 40.6 Hz, 2H), 4.25 – 3.84 (m, 4H), 3.69 (d, *J* = 12.3 Hz, 3H), 1.16 (dt, *J* = 10.8, 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 171.2, 141.4, 138.1, 135.3, 135.1, 134.3, 129.2, 129.1, 128.7, 128.6, 128.5, 124.8, 124.0, 81.8, 81.7, 61.2, 52.2, 50.0, 49.5, 14.0.
HRMS (ESI) calculated for $C_{27}H_{25}ClNaO_5Se_2$ [M+Na]+: 646.9613; found: 646.9619

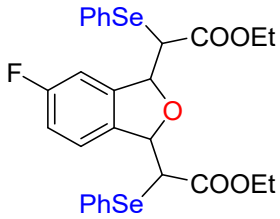


benzyl 2-(6-chloro-3-(2-ethoxy-2-oxo-1-(phenylselanyl)ethyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3ma.

Yield = 52 %; 73 mg; pale yellow oil; R_f (EtOAc : PE = 1:10) = 0.36.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.45 (m, 6H), 7.41 – 6.97 (m, 12H), 5.92 – 5.47 (m, 2H), 5.16 (dt, *J* = 22.1, 12.3 Hz, 2H), 4.23 – 3.62 (m, 4H), 1.23 – 0.96 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 171.0, 141.4, 138.1, 135.5, 135.3, 135.1, 134.3, 129.1, 128.6, 128.5, 128.2, 128.23, 127.7, 124.8, 124.0, 81.8, 81.6, 66.9, 61.2, 49.8, 49.7, 14.0. HRMS (ESI) calculated for $C_{33}H_{29}ClNaO_5Se_2$ [M+Na]+: 722.9926; found: 722.9916.



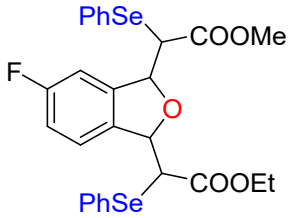
diethyl 2,2'-(5-fluoro-1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(phenylselanyl)acetate). Compound 3na.

Yield = 66 %; 82 mg; yellow oil; R_f (EtOAc : PE = 1:10) = 0.30.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 8.5, 5.0 Hz, 1H), 7.56 (ddd, *J* = 8.8, 4.1, 2.5 Hz,5H), 7.36 – 7.15 (m, 6H), 7.02 (td, *J* = 8.6, 2.4 Hz, 1H), 5.68 (dd, *J* = 77.7, 7.4 Hz, 2H), 4.30 – 4.04 (m, 4H), 4.03 – 3.76 (m, 2H), 1.18 (td, *J* = 7.1, 5.7 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.27 (d, *J* = 12.5 Hz), 162.87 (d, *J* = 246.2 Hz), 141.87 (d, *J* = 8.7 Hz), 135.15 (d, *J* = 2.1 Hz), 135.15 (d, *J* = 11.7 Hz), 129.15 (d, *J* = 2.0 Hz), 128.53 (d, *J* = 7.8 Hz), 128.02 (d, *J* = 13.6 Hz), 125.05 (d, *J* = 8.9 Hz), 115.56 (d, *J* = 22.8 Hz), 111.09 (d, *J* = 24.8 Hz), 81.6, 61.2, 49.9, 14.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ -113.3.

HRMS (ESI) calculated for $C_{28}H_{27}FNaO_5Se_2$ [M+Na]+: 645.0065; found: 645.0065

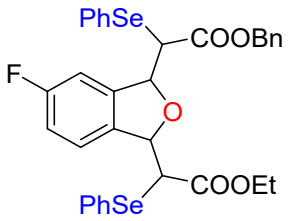


ethyl 2-(5-fluoro-3-(2-methoxy-2-oxo-1-(phenylselanyl)ethyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3oa.

Yield = 75 %; 91 mg; yellow oil; R_f (EtOAc : PE = 1:10) = 0.31.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 8.5, 5.0 Hz, 1H), 7.67 – 7.45 (m, 5H), 7.38 – 7.16 (m, 6H), 7.03 (td, *J* = 8.6, 2.3 Hz, 1H), 5.57 (dd, *J* = 7.6, 3.2 Hz, 2H), 4.24 – 4.06 (m, 2H), 4.06 – 3.86 (m, 2H), 3.66 (d, *J* = 5.3 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H).

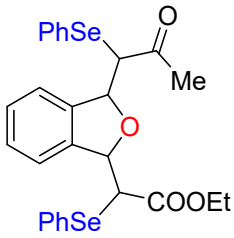
^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 171.3, 162.8, 141.8, 135.2, 135.0, 129.1, 128.5, 127.9, 125.0, 115.6, 111.1, 81.7, 81.6, 61.2, 52.2, 50.2, 49.5, 14.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ -113.3. HRMS (ESI) calculated for $C_{27}H_{25}FNaO_5Se_2$ [M+Na]+: 630.9909; found: 630.9904.



benzyl 2-(3-(2-ethoxy-2-oxo-1-(phenylselanyl)ethyl)-6-fluoro-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3pa.

Yield = 82 %; 112 mg; colourless oil; R_f (EtOAc : PE = 1:10) = 0.35.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.59 (m, 1H), 7.65 – 7.13 (m, 16H), 7.11 – 6.78 (m, 1H), 5.90 – 5.49 (m, 2H), 5.12 (dt, *J* = 26.9, 12.4 Hz, 2H), 4.38 – 3.58 (m, 4H), 1.23 – 0.96 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 171.0, 162.88 (d, *J* = 246.3 Hz), 141.80 (d, *J* = 8.7 Hz), 135.56 (d, *J* = 2.2 Hz), 135.3, 135.1, 134.9, 134.8, 129.19, 129.14, 128.6, 128.5, 128.4, 128.2, 127.7, 125.07 (d, *J* = 8.8 Hz), 115.70 (d, *J* = 4.4 Hz), 115.47 (d, *J* = 4.4 Hz), 111.08 (d, *J* = 24.8 Hz), 82.2, 81.7, 66.9, 61.2, 50.1, 49.7, 14.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ -113.2. HRMS (ESI) calculated for $C_{33}H_{30}FO_5Se_2$ [M+H]+: 685.0402; found: 685.0401.

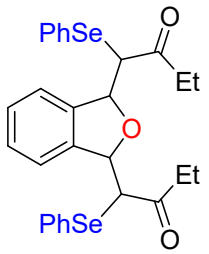


ethyl 2-(3-(2-oxo-1-(phenylselanyl)propyl)-1,3-dihydroisobenzofuran-1-yl)-2-(phenylselanyl)acetate. Compound 3qa.

Yield = 49 %; 56 mg; pale yellow oil; R_f (EtOAc : PE = 1:10) = 0.35.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.61 (m, 2H), 7.58 – 6.98 (m, 12H), 5.91 – 5.41 (m, 2H), 4.23 – 3.81 (m, 4H), 2.47 – 2.20 (m, 3H), 1.24 – 1.06 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.50, 171.1, 140.6, 139.9, 136.54, 135.0, 134.9, 129.5, 128.6, 128.4, 123.6, 123.1, 121.8 82.9, 81.5, 61.4, 58.8, 50.1, 28.4, 14.0.

HRMS (ESI) calculated for $C_{27}H_{26}O_4Se_2$ [M+Na]+: 597.0054; found: 597.0050.

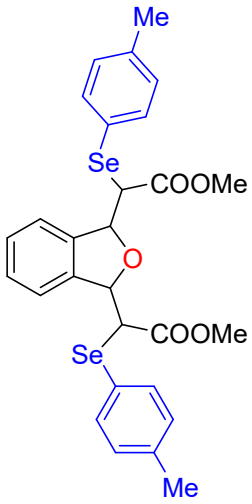


1,1'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(1-(phenylselanyl)butan-2-one). Compound 3ra.

Yield = 57 %; 65 mg; colourless oil; R_f (EtOAc : PE = 1:10) = 0.25.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 – 6.97 (m, 14H), 5.98 – 5.32 (m, 2H), 4.32 – 3.68 (m, 2H), 3.17 – 2.32 (m, 4H), 1.28 – 0.89 (m, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 206.3, 140.3, 136.1, 134.5, 129.4, 128.7, 123.6, 121.8, 81.6, 57.9, 35.2, 8.2.

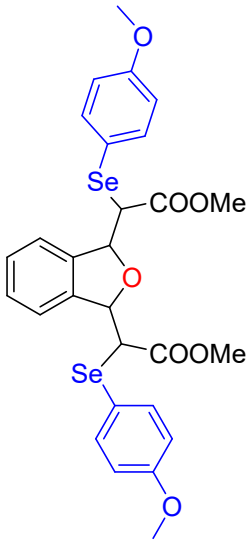
HRMS (ESI) calculated for $C_{28}H_{28}NaO_3Se_2$ [M+Na]+: 595.0261; found: 595.0252.



dimethyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(2-(p-tolylselanyl)acetate). Compound 3ac.

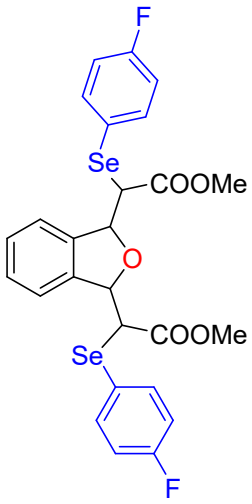
Yield = 58 %; 70 mg; yellow oil; R_f (EtOAc : PE = 1:15) = 0.23.

Major: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 5.5, 3.3 Hz, 2H), 7.48 – 7.29 (m, 6H), 7.03 (t, *J* = 6.0 Hz, 4H), 5.54 (d, *J* = 8.3 Hz, 2H), 3.87 (d, *J* = 8.3 Hz, 2H), 3.63 (s, 6H), 2.29 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.6, 139.6, 138.7, 135.6, 129.9, 128.3, 124.0, 123.7, 82.3, 52.1, 50.1, 21.2. Minor: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 5.5, 3.2 Hz, 2H), 7.43 – 7.28 (m, 6H), 7.03 (t, *J* = 6.0 Hz, 4H), 5.75 (d, *J* = 6.0 Hz, 2H), 3.83 (d, *J* = 6.1 Hz, 2H), 3.60 (s, 6H), 2.29 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.5, 139.9, 138.5, 135.3, 129.9, 128.2, 124.6, 123.1, 81.9, 52.0, 50.0, 21.2. HRMS (ESI) calculated for $C_{28}H_{28}NaO_5Se_2$ [M+Na]+: 627.0159; found: 627.0164



dimethyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(2-((4-methoxyphenyl)selanyl)acetate). Compound 3ad.

Yield = 70 %; 89 mg; pale yellow oil; R_f (EtOAc : PE = 1:5) = 0.2.
Major: ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 – 7.77 (m, 2H), 7.70 – 7.18 (m, 6H), 6.78 (d, *J* = 7.3 Hz, 4H), 5.54 (d, *J* = 8.2 Hz, 2H), 3.85 (d, *J* = 8.3 Hz, 2H), 3.78 (s, 6H), 3.64 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.5, 160.3, 139.6, 137.8, 128.3, 123.7, 117.5, 114.7, 81.8, 55.2, 52.0, 50.2, 50.2.Minor: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 – 7.59 (m, 2H), 7.50 – 7.26 (m, 6H), 6.78 (d, *J* = 7.3 Hz, 4H), 5.75 (d, *J* = 5.8 Hz, 2H), 3.81 (d, *J* = 6.4 Hz, 2H), 3.78 (s, 6H), 3.62 (s, 6H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.4, 160.2, 140.0, 137.5, 128.2, 123.2, 118.2, 114.7, 82.1, 55.2, 52.0, 50.2, 50.2.HRMS (ESI) calculated for $C_{28}H_{28}NaO_7Se_2$ [M+Na]+: 659.0058; found: 659.0060.



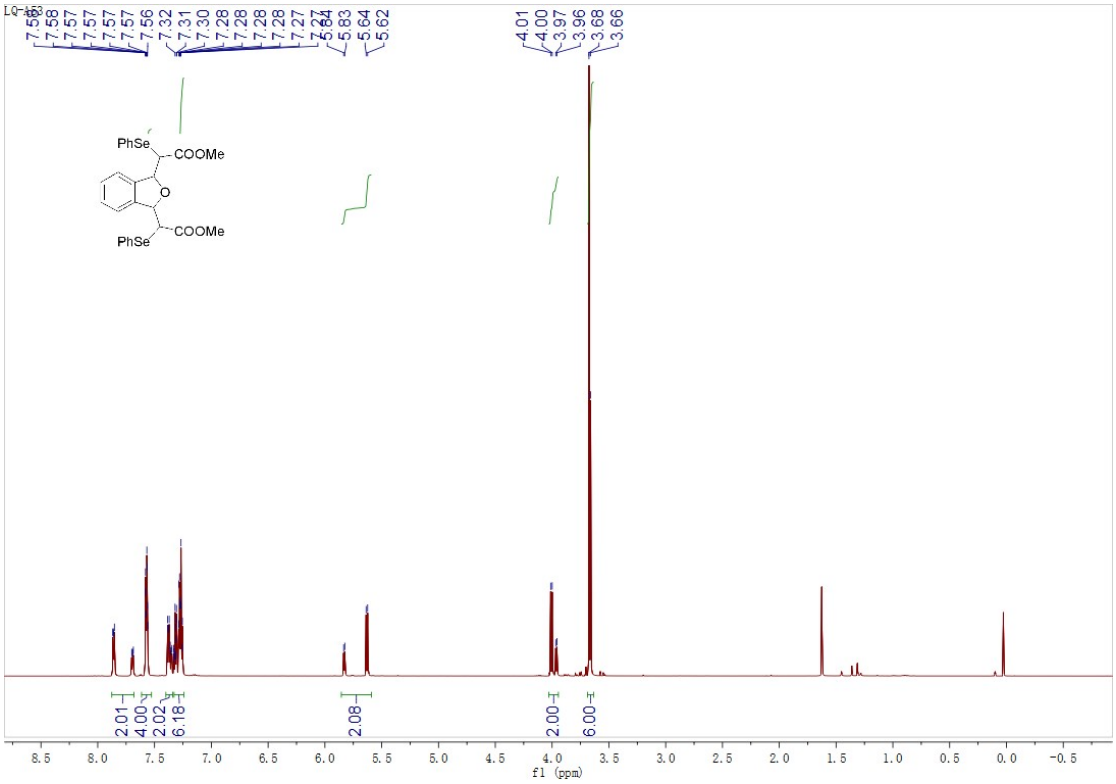
dimethyl 2,2'-(1,3-dihydroisobenzofuran-1,3-diyl)bis(2-((4-fluorophenyl)selanyl)acetate). Compound 3ae.
Yield = 65.4 %; 80 mg; yellow oil; R_f (EtOAc : PE = 1:15) = 0.25.
Major: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 23.5 Hz, 2H), 7.71 – 7.24 (m, 6H), 6.95 (t, *J* = 7.8 Hz, 4H), 5.57 (d, *J* = 7.6 Hz, 2H), 3.93 (d, *J* = 7.6 Hz, 2H), 3.66 (s, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.4, 163.2 (d, *J* = 249.6 Hz), 139.4, 137.8 (d, *J* = 8.2 Hz), 128.5, 123.6, 116.4 (d, *J* = 21.6 Hz), 81.9, 52.1, 50.2. ^{19}F NMR (565 MHz, $CDCl_3$) δ -112.0. Minor: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 23.5 Hz, 2H), 7.61 – 7.30 (m, 6H), 6.95 (t, *J* = 7.8 Hz, 4H), 5.79 (d, *J* = 4.7 Hz, 2H), 3.88 (d, *J* = 5.1 Hz, 2H), 3.63 (s, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.2, 163.1 (d, *J* = 249.6 Hz), 139.8, 137.5 (d, *J* = 8.2 Hz), 128.4, 122.2, 116.3 (d, *J* = 21.6 Hz), 82.3, 52.2, 50.4. ^{19}F NMR (565 MHz, $CDCl_3$) δ -112.3. HRMS (ESI) calculated for $C_{26}H_{22}F_2NaO_5Se_2$ [M+Na]+: 634.9658; found: 634.9660.

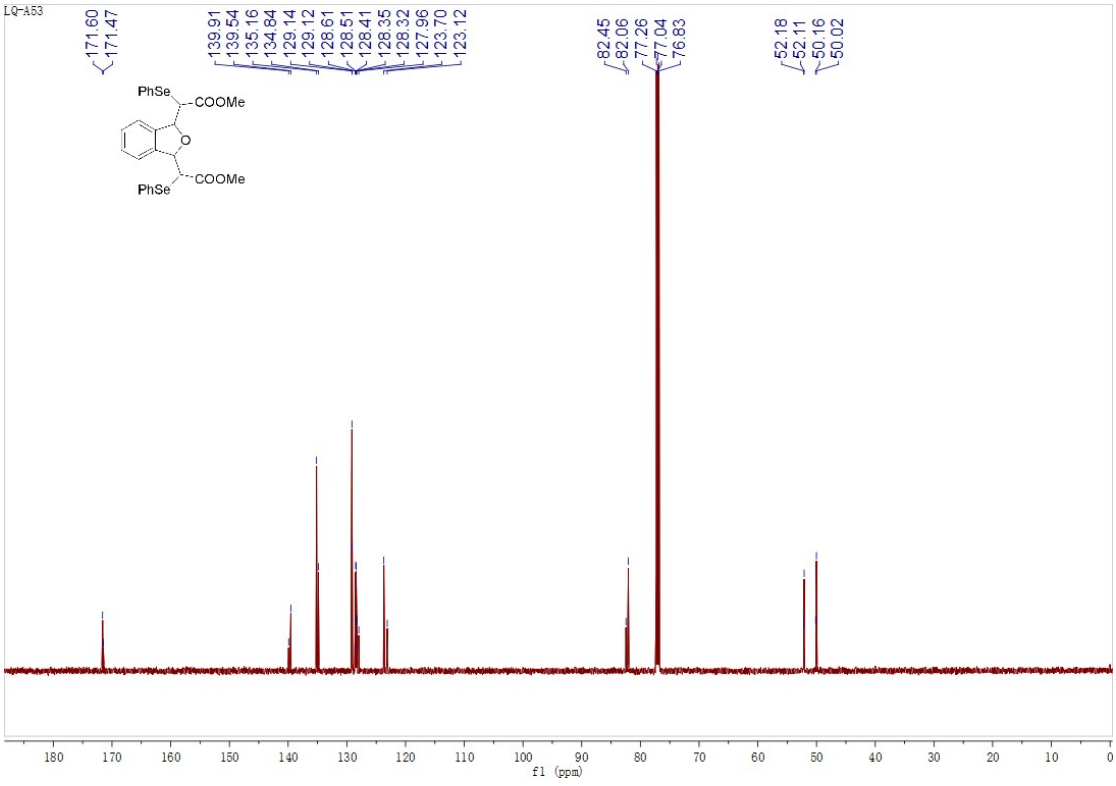
5. References

[1]H. Wei, Y. Li, K. Xiao, B. Cheng, H. Wang, L. Hu and H. Zhai, *Org. Lett.*, 2015, **17**, 5974.
[2]C. L. Oswald, J. A. Peterson and H. W. Lam, *Org. Lett.*, 2009, **11**, 4504.
[3]A. Matviitsuk, J. E. Taylor, D. B. Cordes, A. M. Z. Slawin and A. D. Smith, *Chem. Eur. J.*, 2016, **22**, 17748.
[4] Q. Liu, J. Wang, D. Li, Chao Yang and W. Xia, *J. Org. Chem.,* 2017, **82**, 1389.

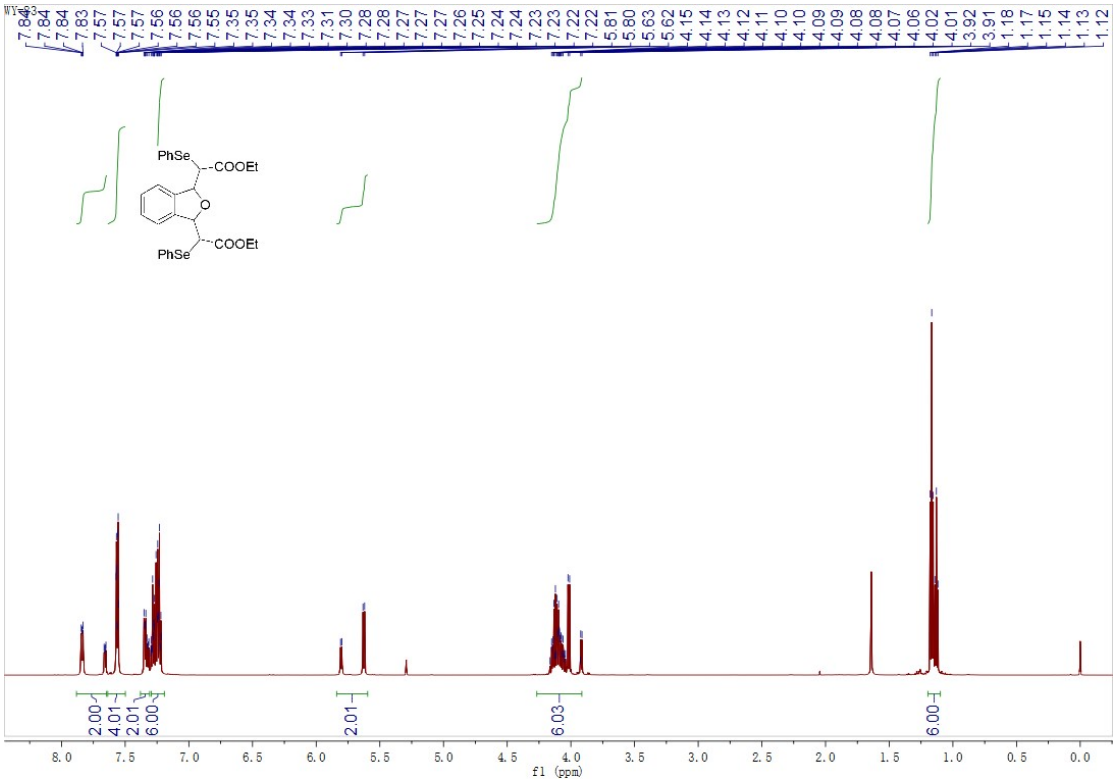
6. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra for Products.

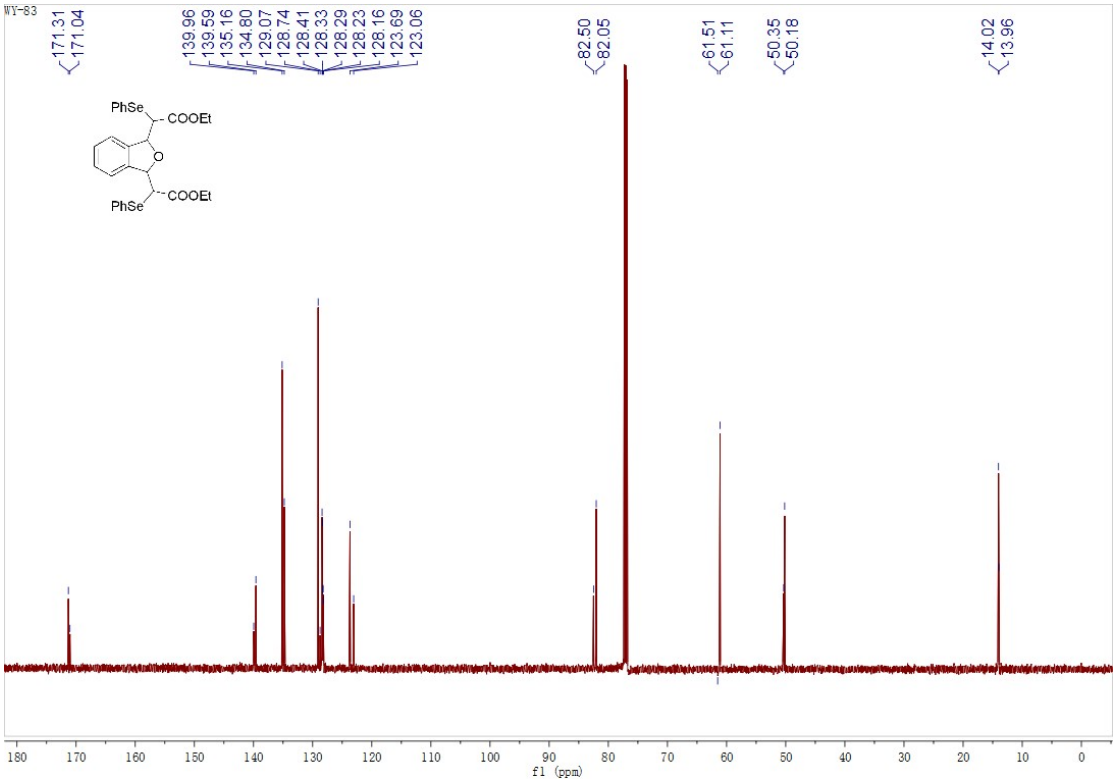
Compound 3aa ^{1}H NMR and ^{13}C NMR



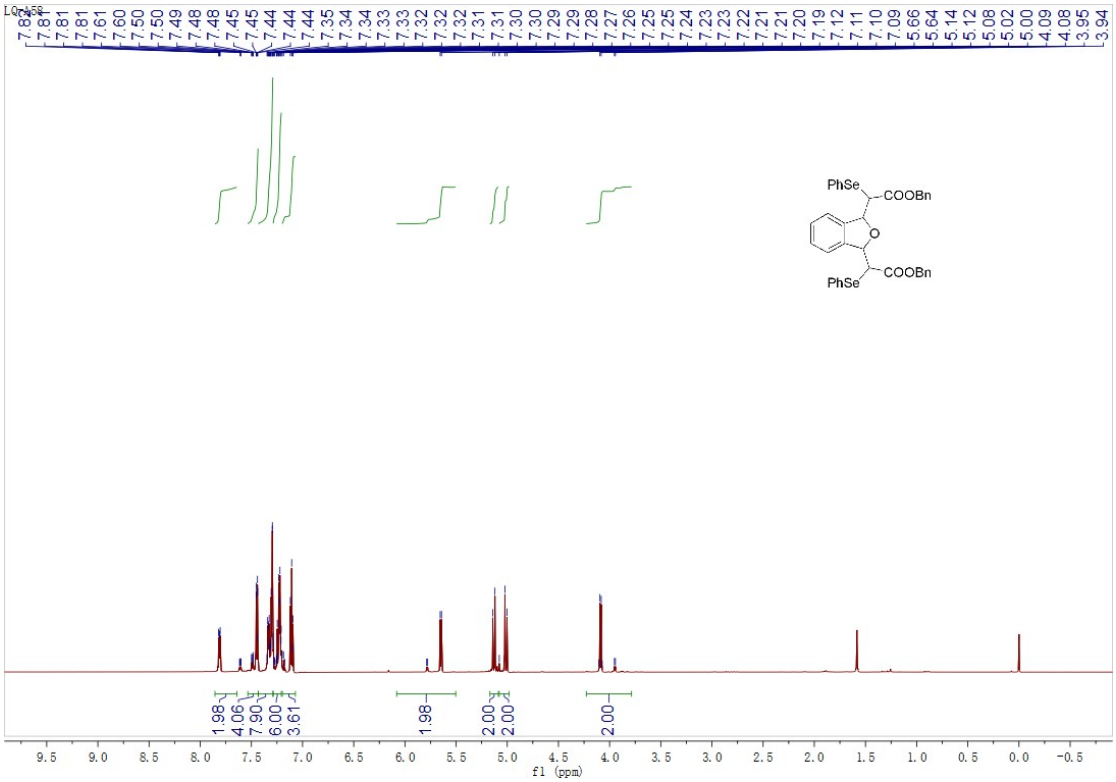


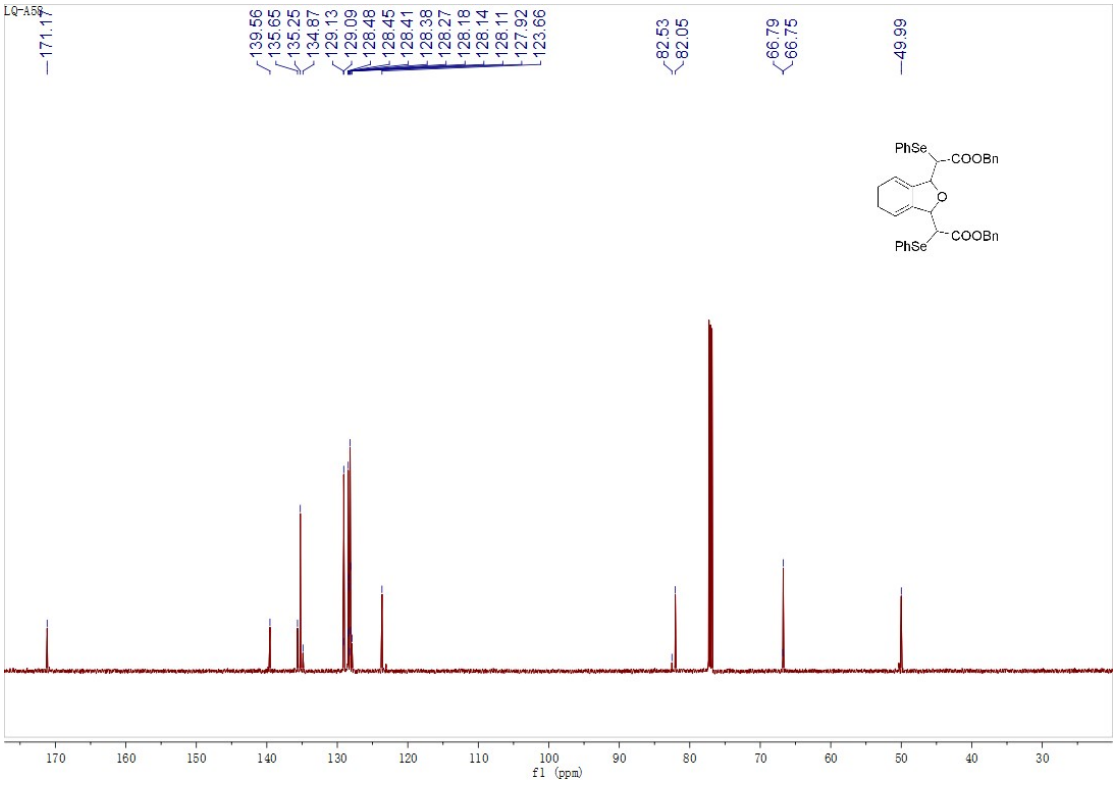
Compound 3ba ^{1}H NMR and ^{13}C NMR



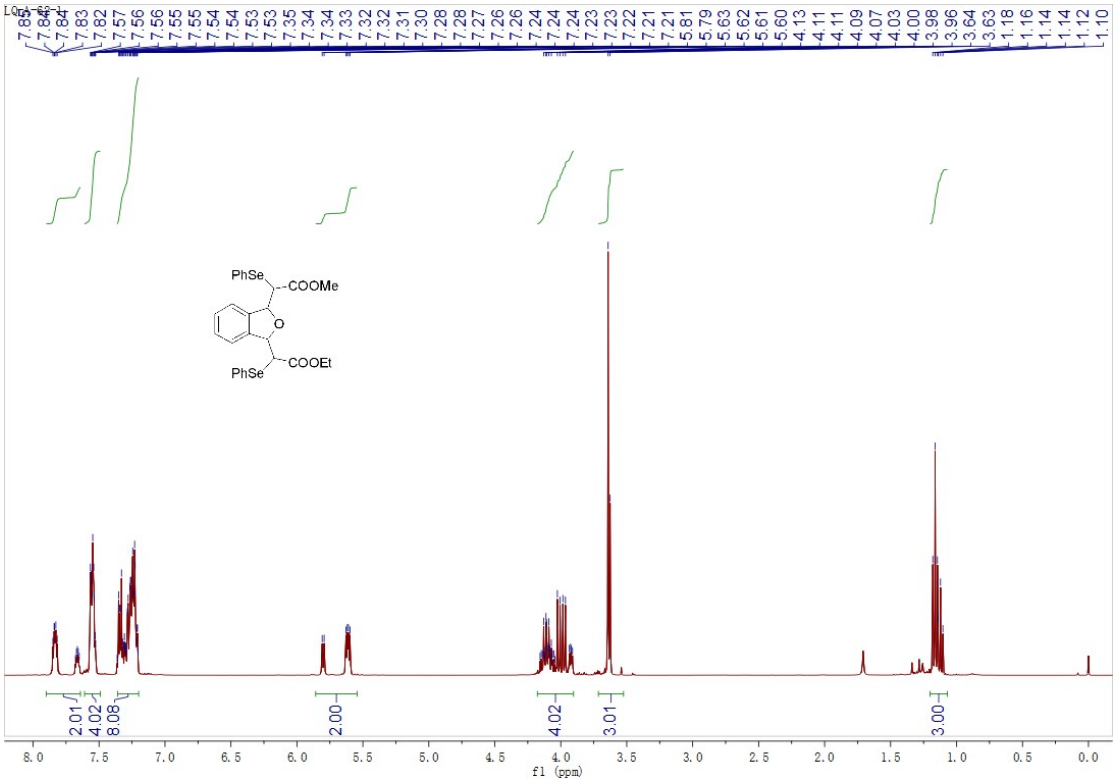


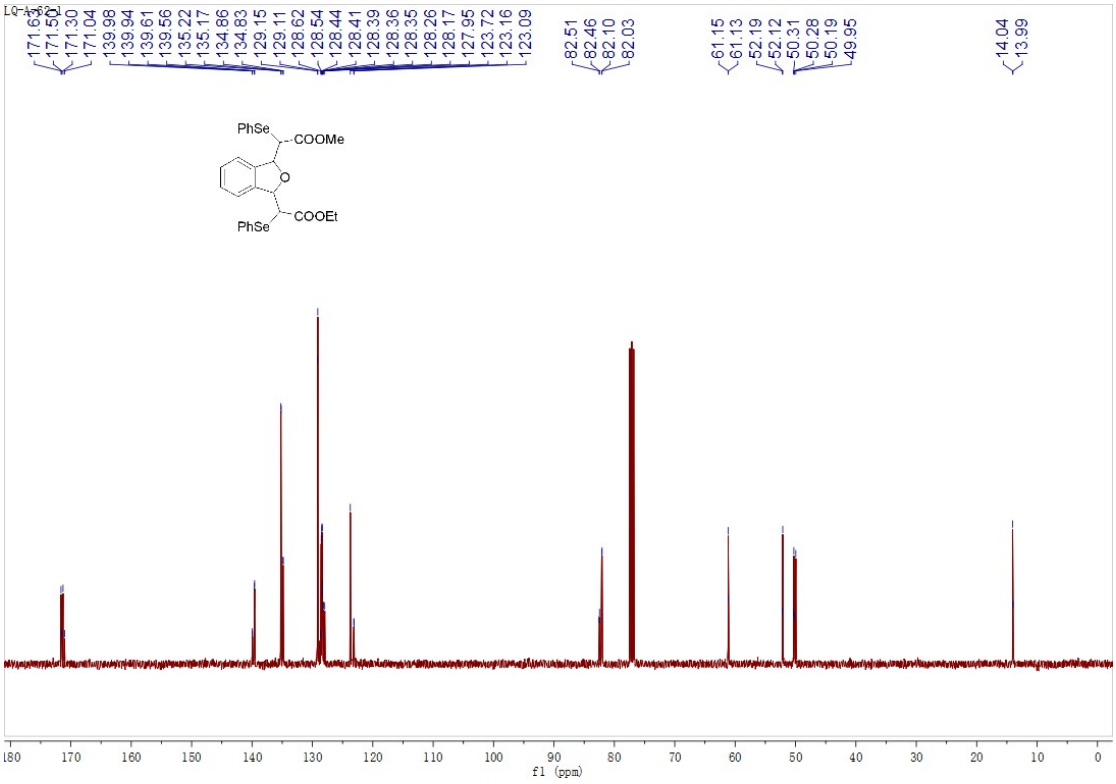
Compound 3ca ^{1}H NMR and ^{13}C NMR



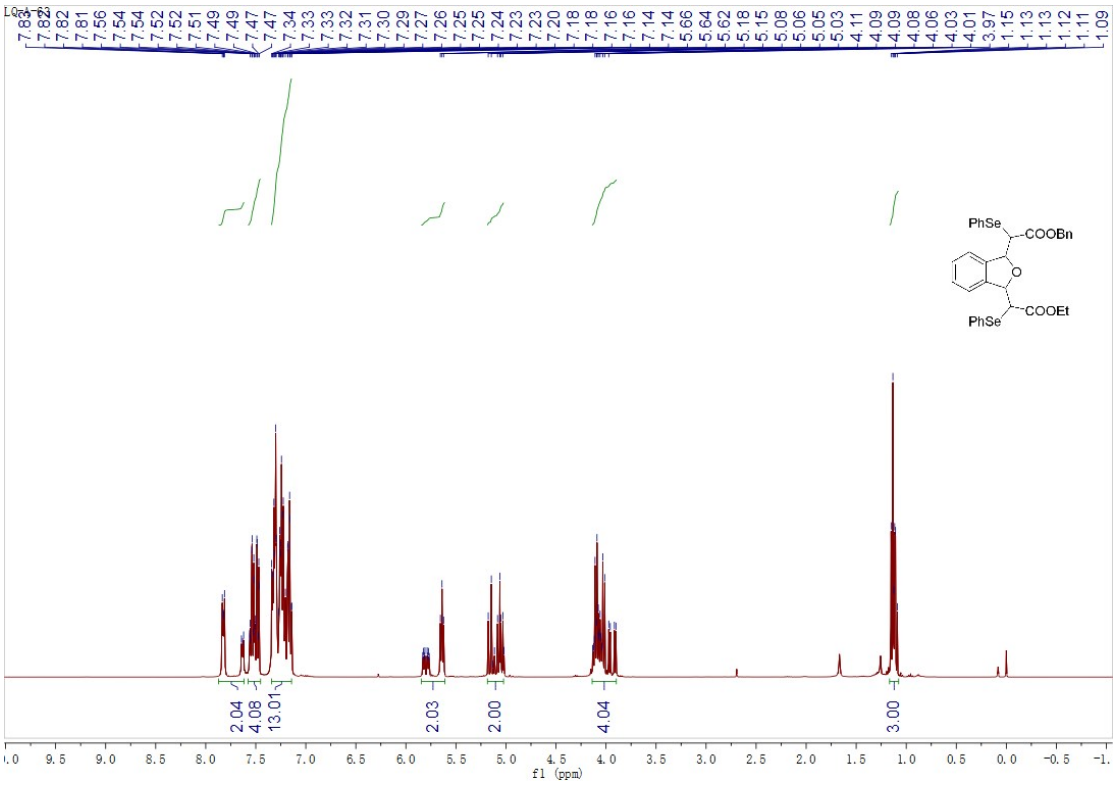


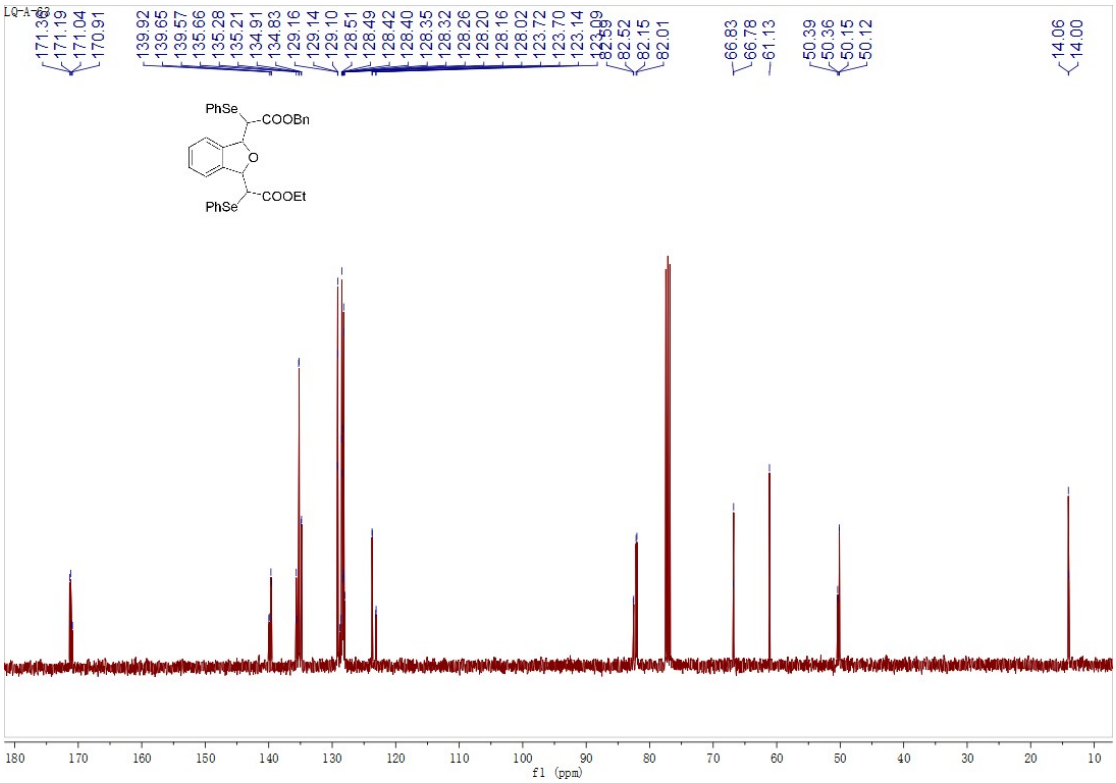
Compound 3da ^{1}H NMR and ^{13}C NMR



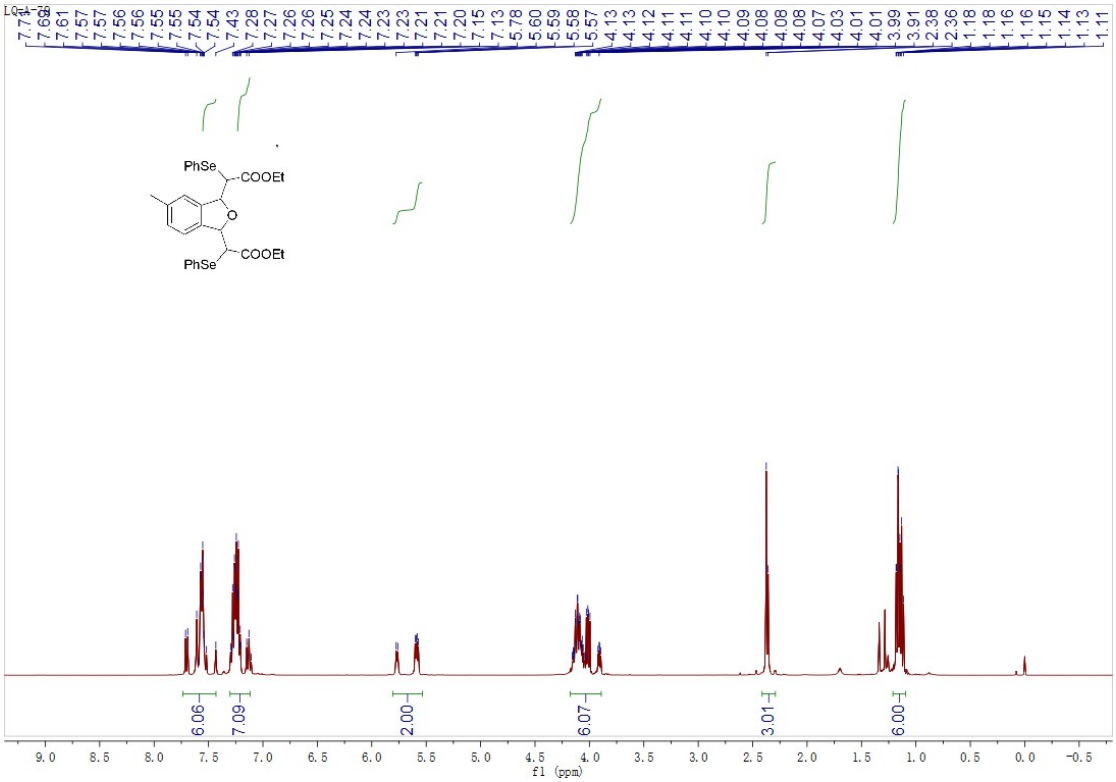


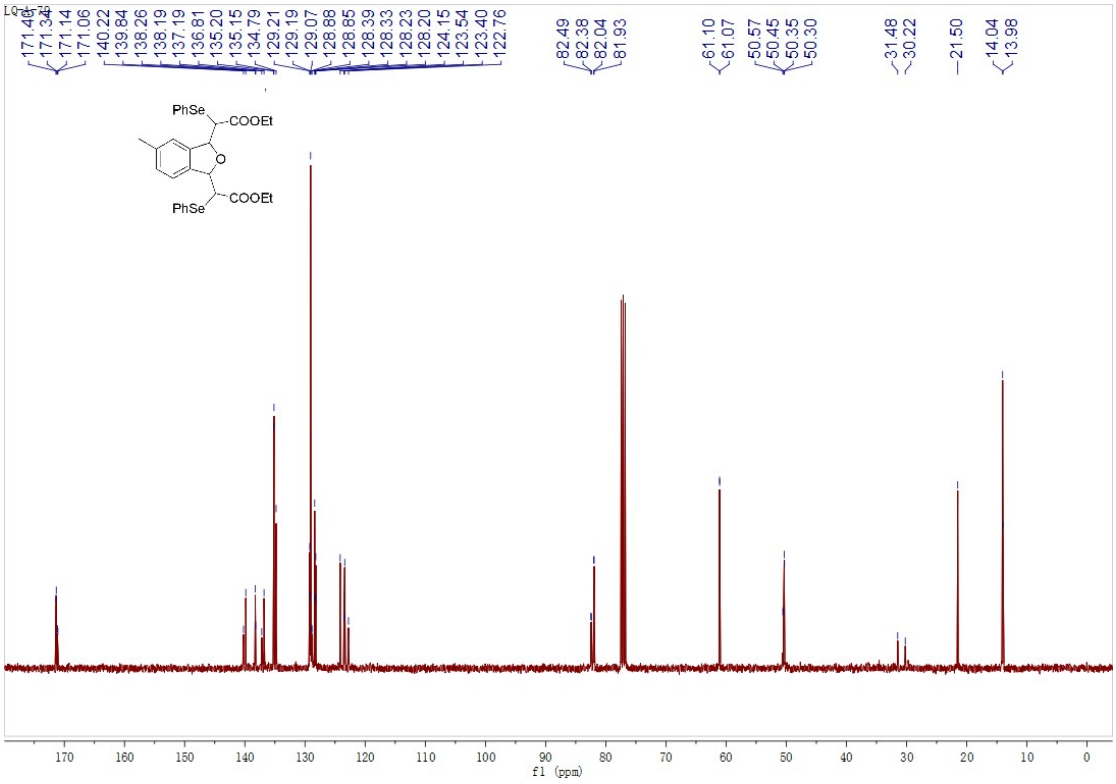
Compound 3ea ^{1}H NMR and ^{13}C NMR



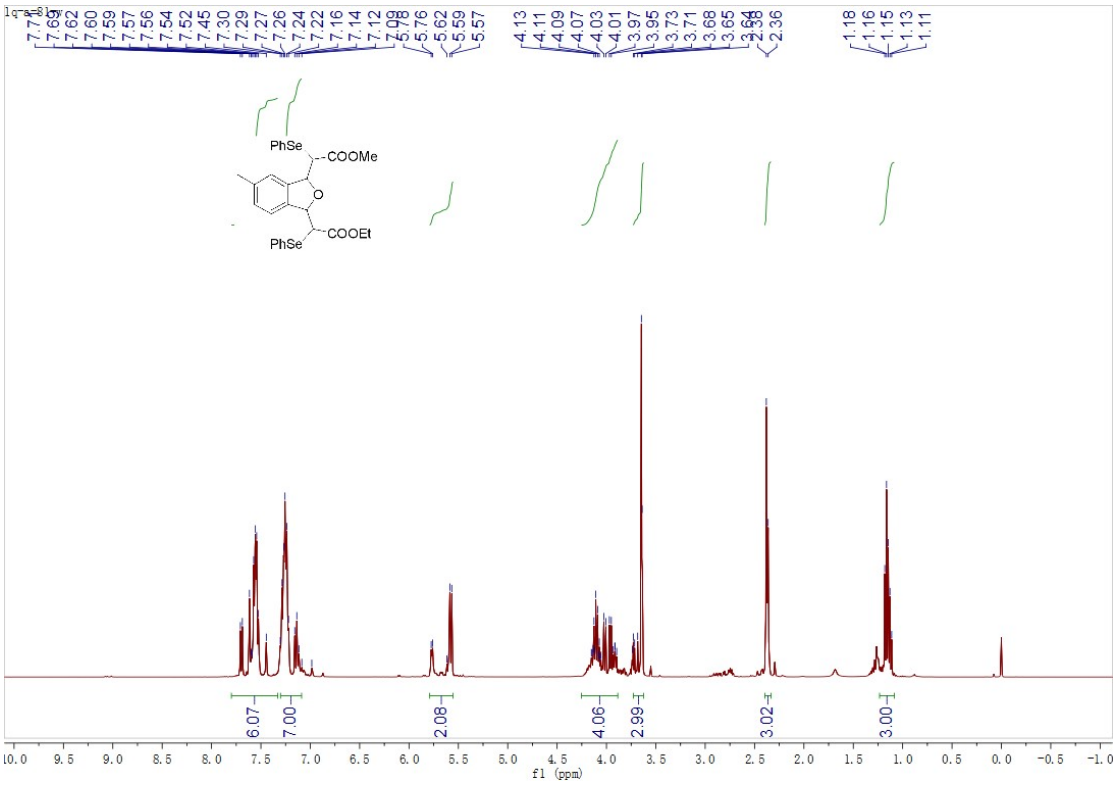


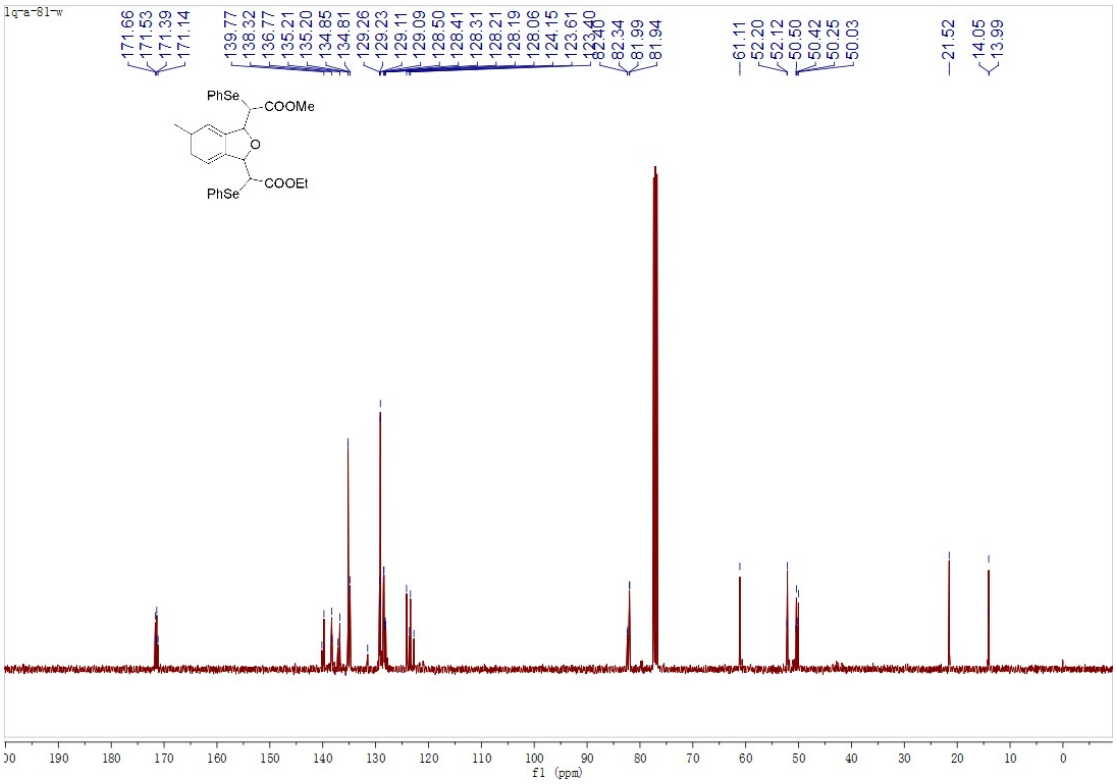
Compound 3fa ^{1}H NMR and ^{13}C NMR



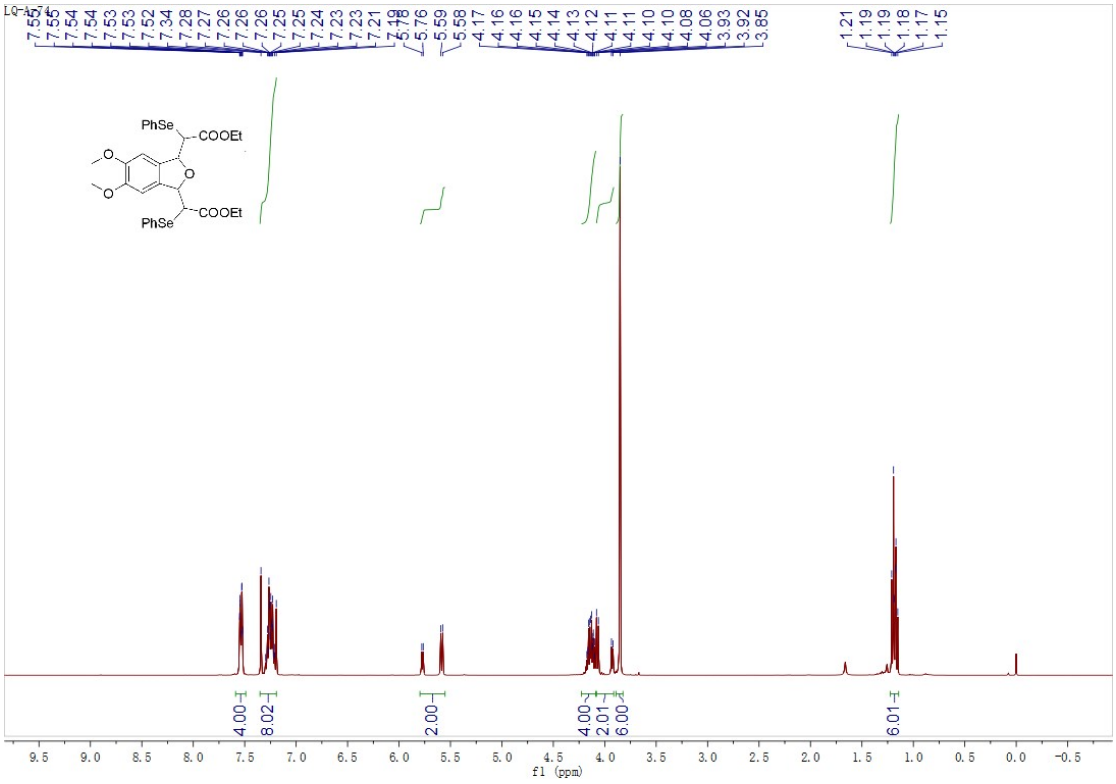


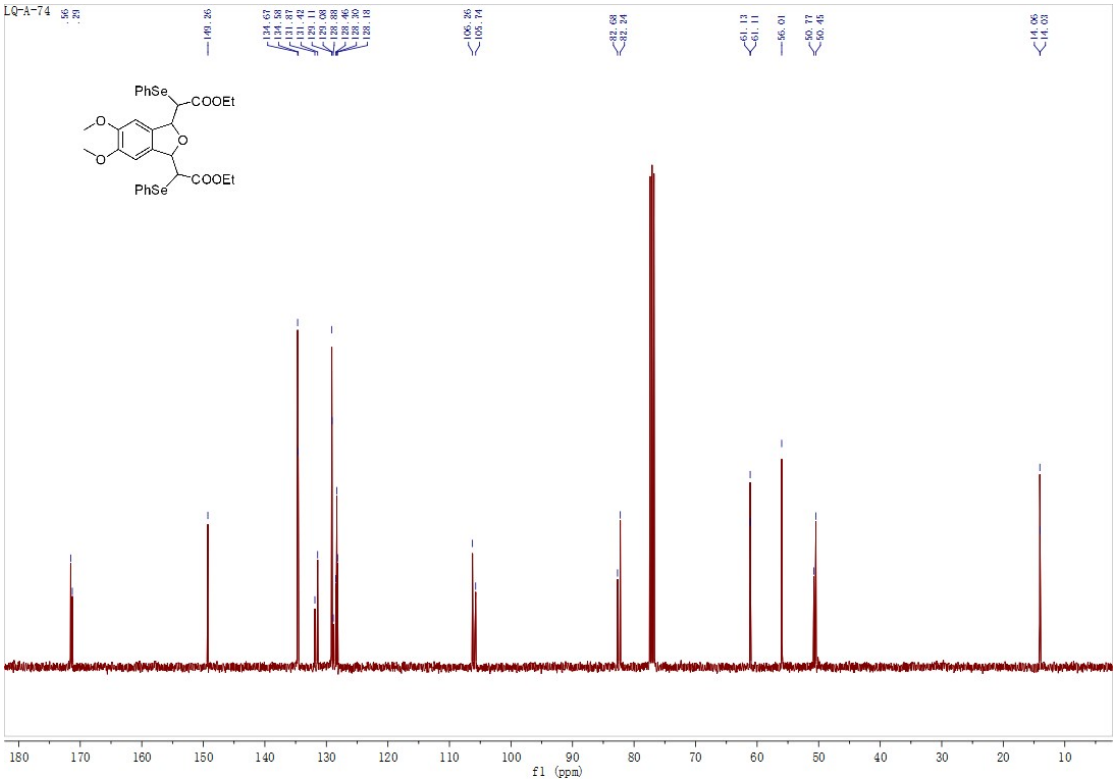
Compound 3ga ^{1}H NMR and ^{13}C NMR



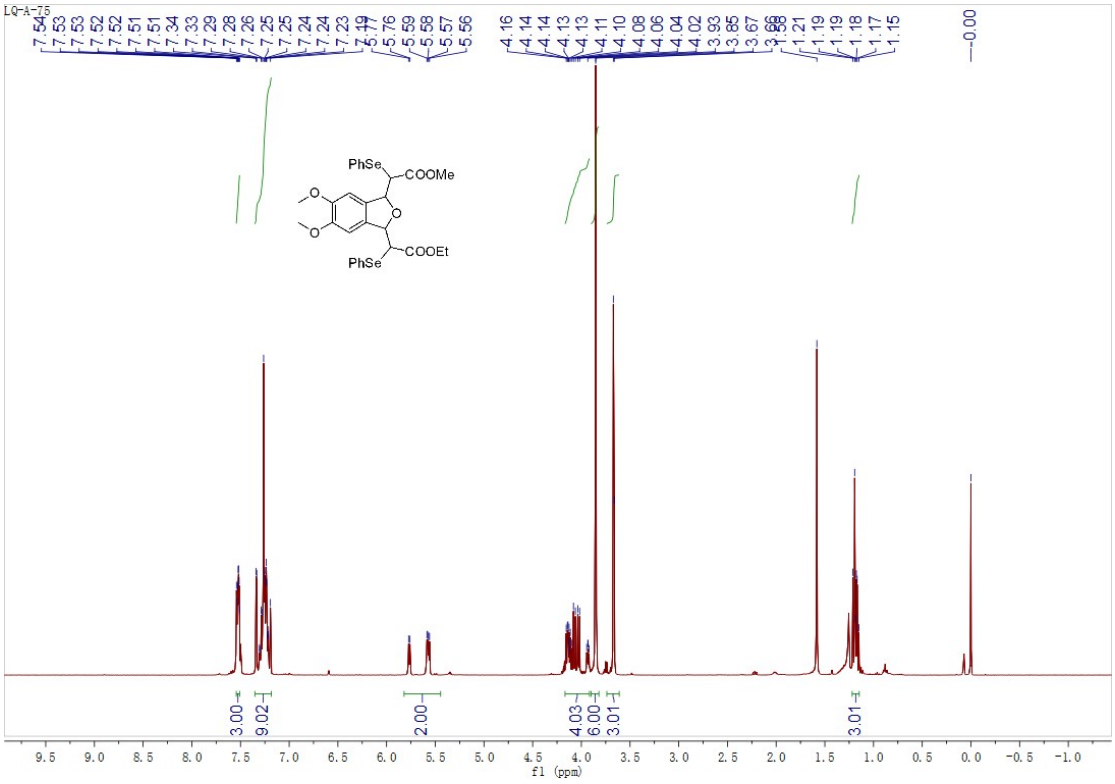


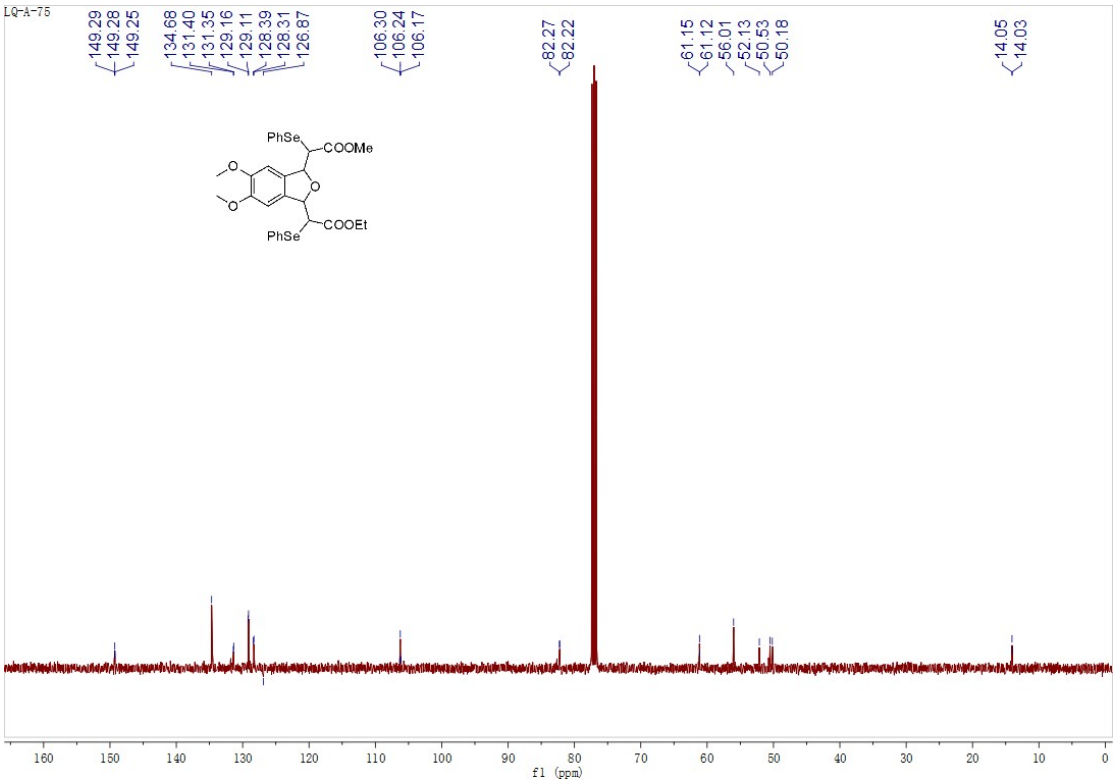
Compound 3ha ^{1}H NMR and ^{13}C NMR



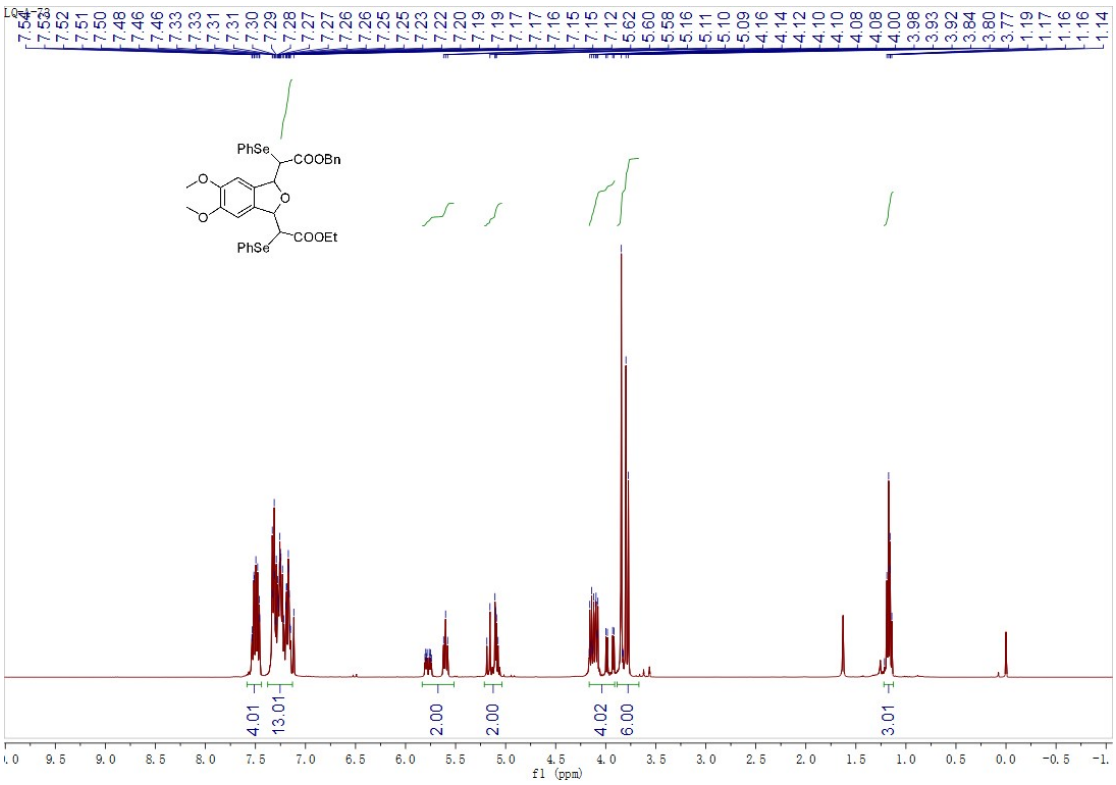


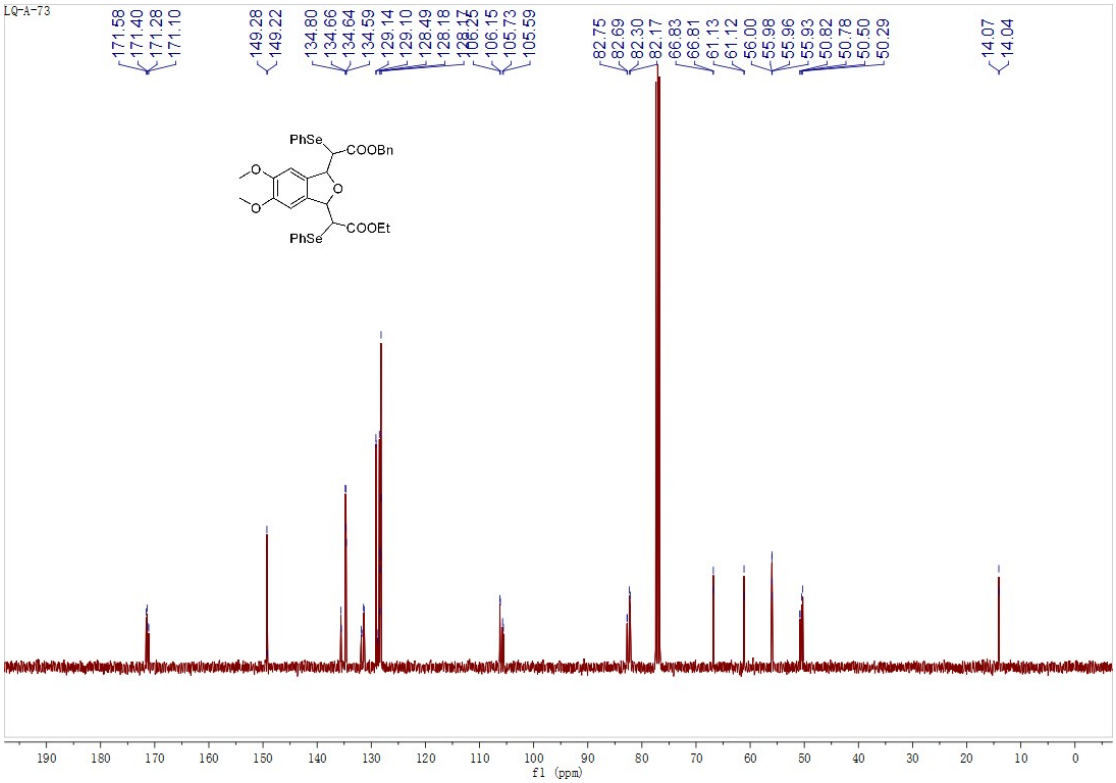
Compound 3ia ^{1}H NMR and ^{13}C NMR



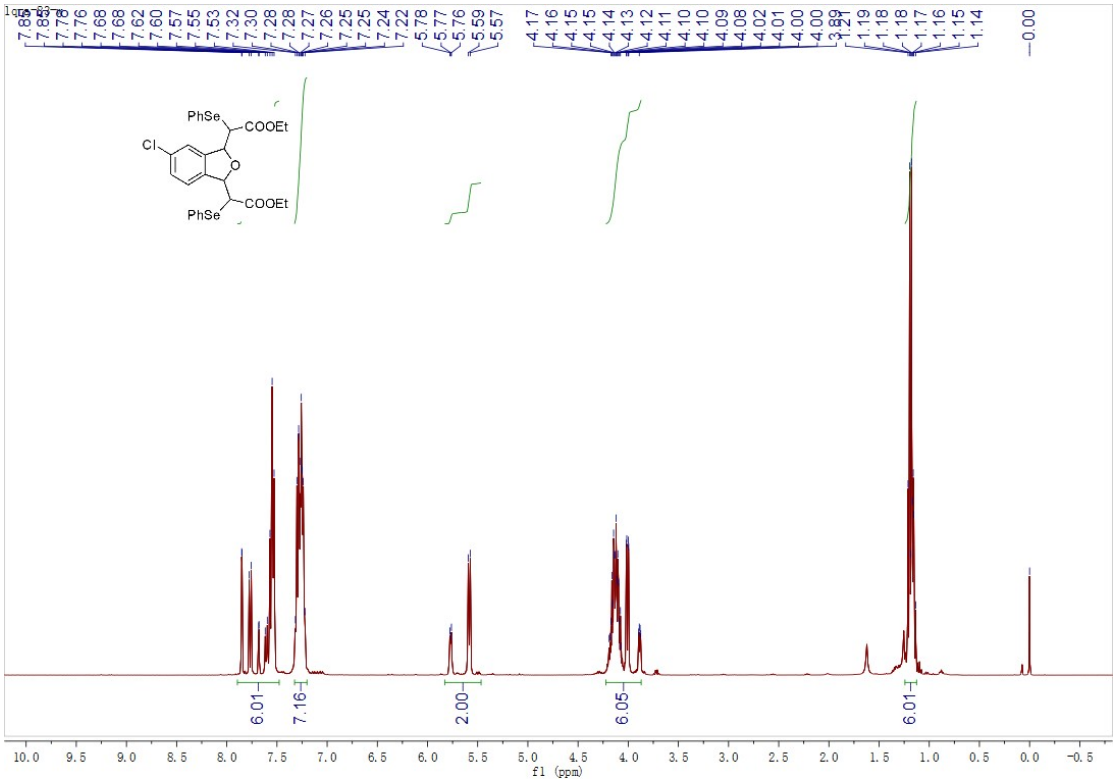


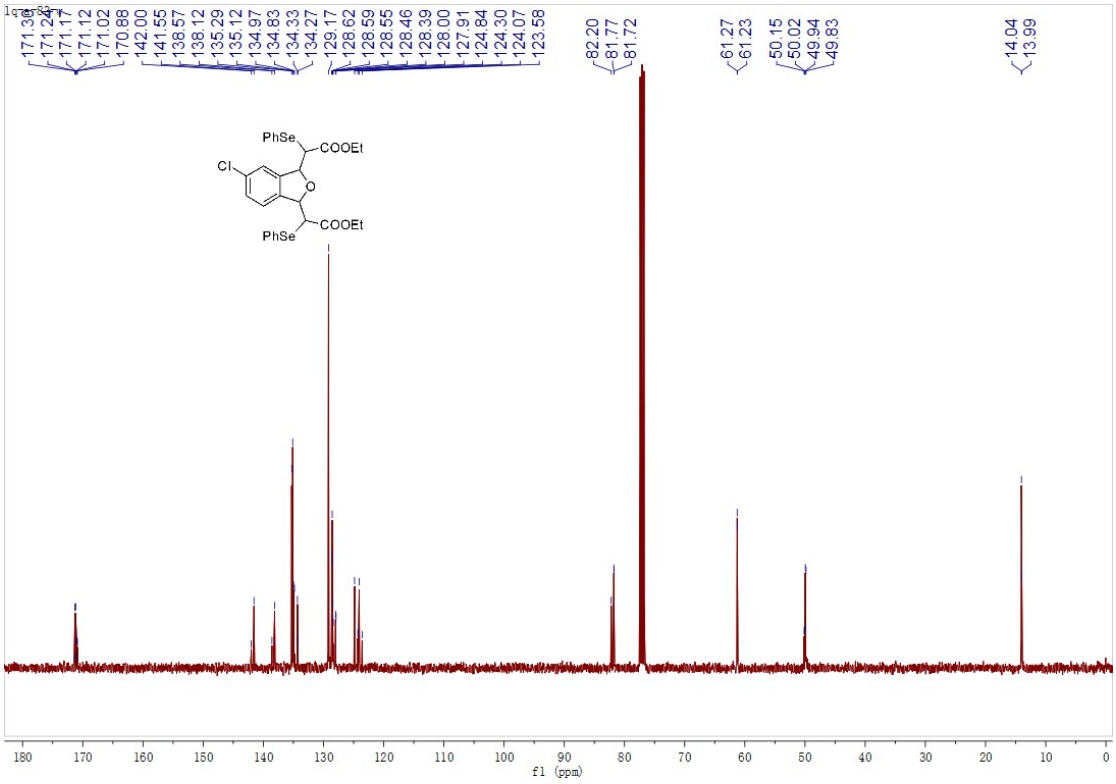
Compound 3ja ^{1}H NMR and ^{13}C NMR



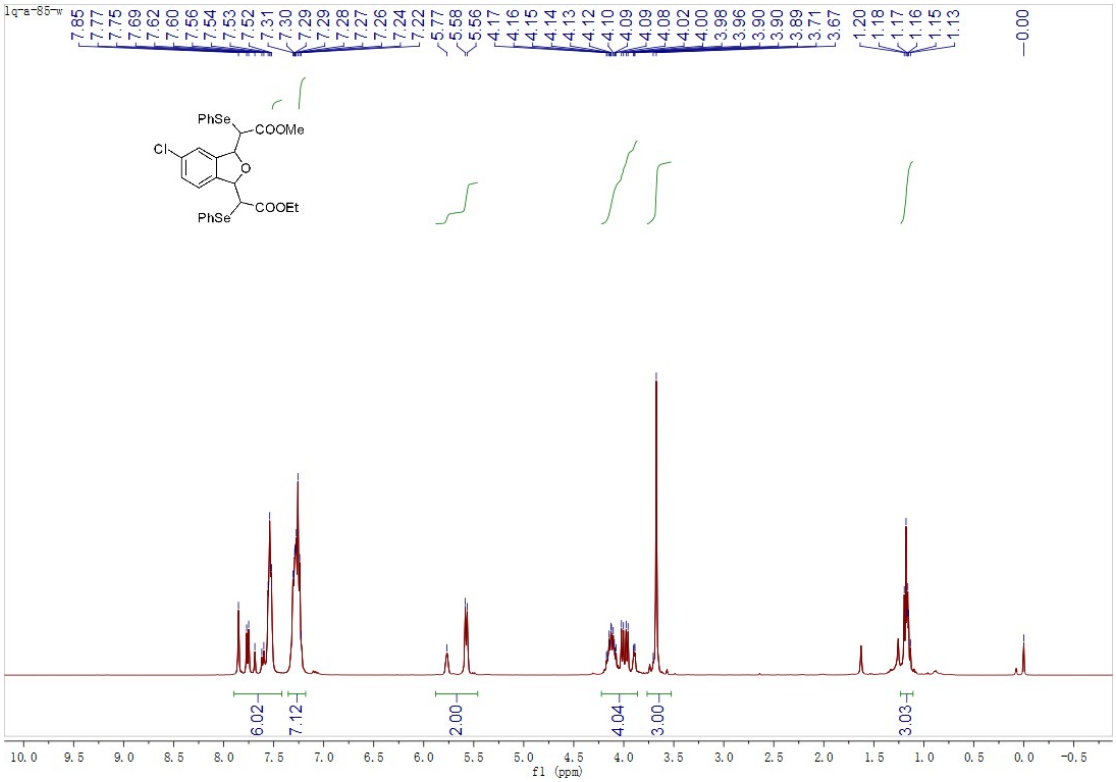


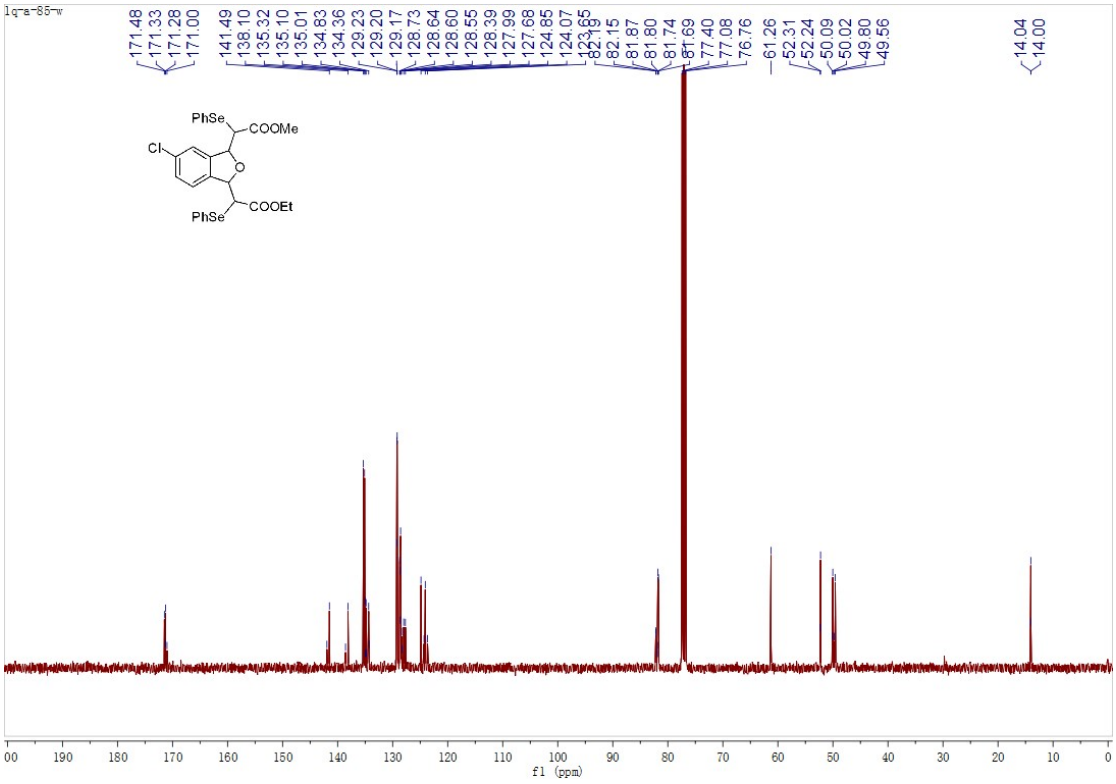
Compound 3ka ^{1}H NMR and ^{13}C NMR





Compound 3la ^{1}H NMR and ^{13}C NMR





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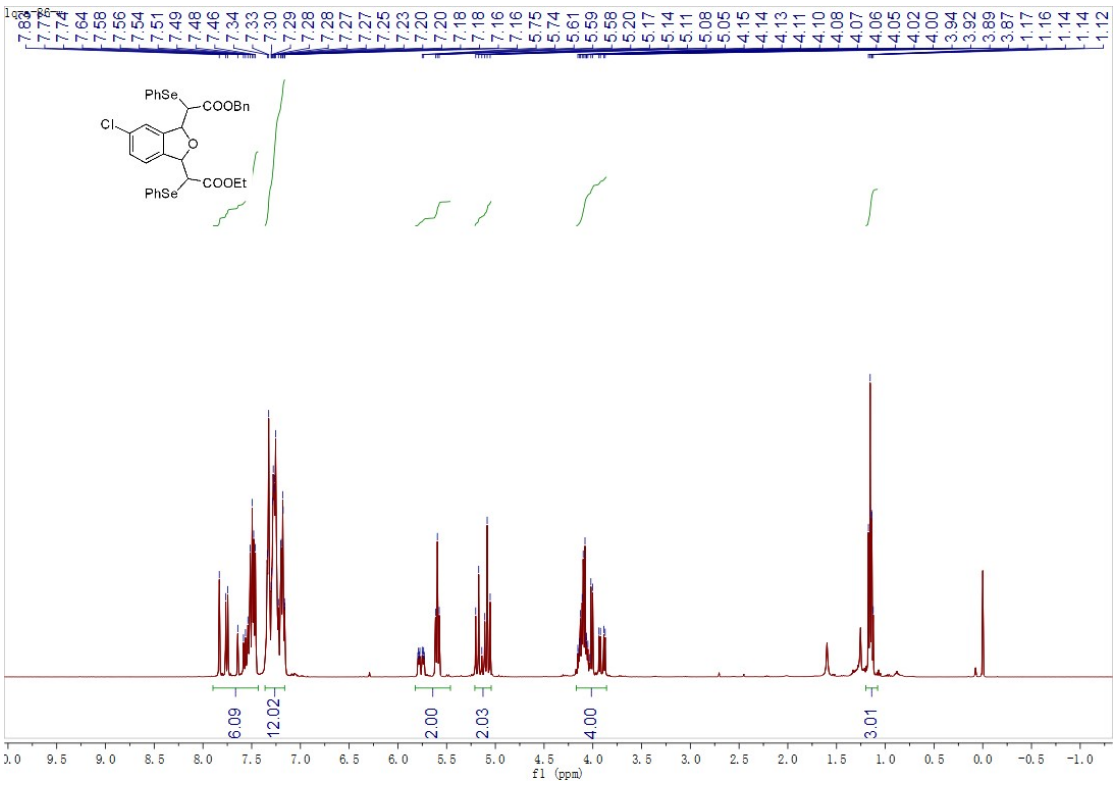
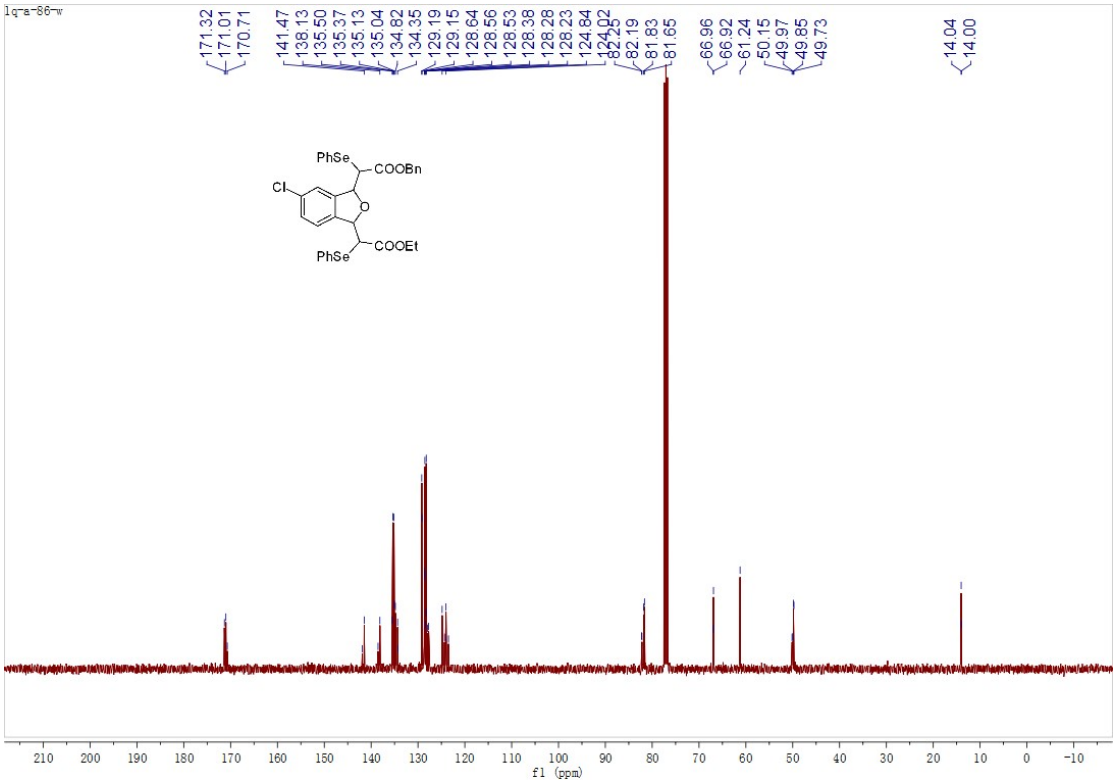
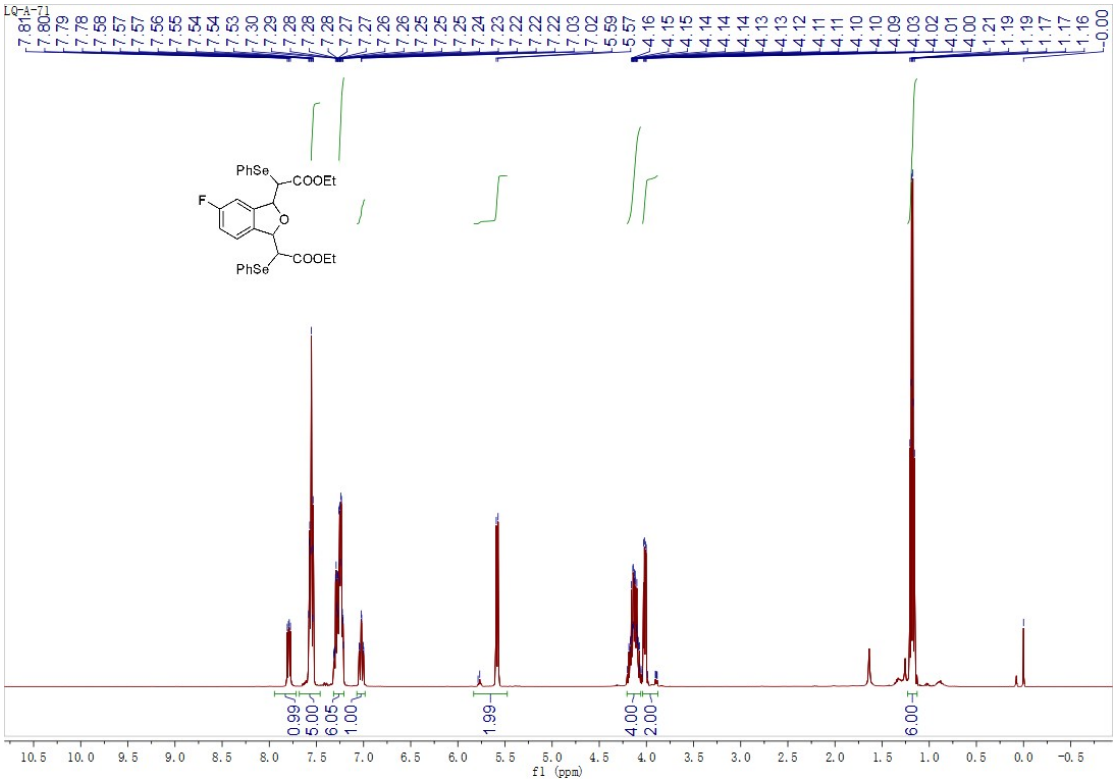
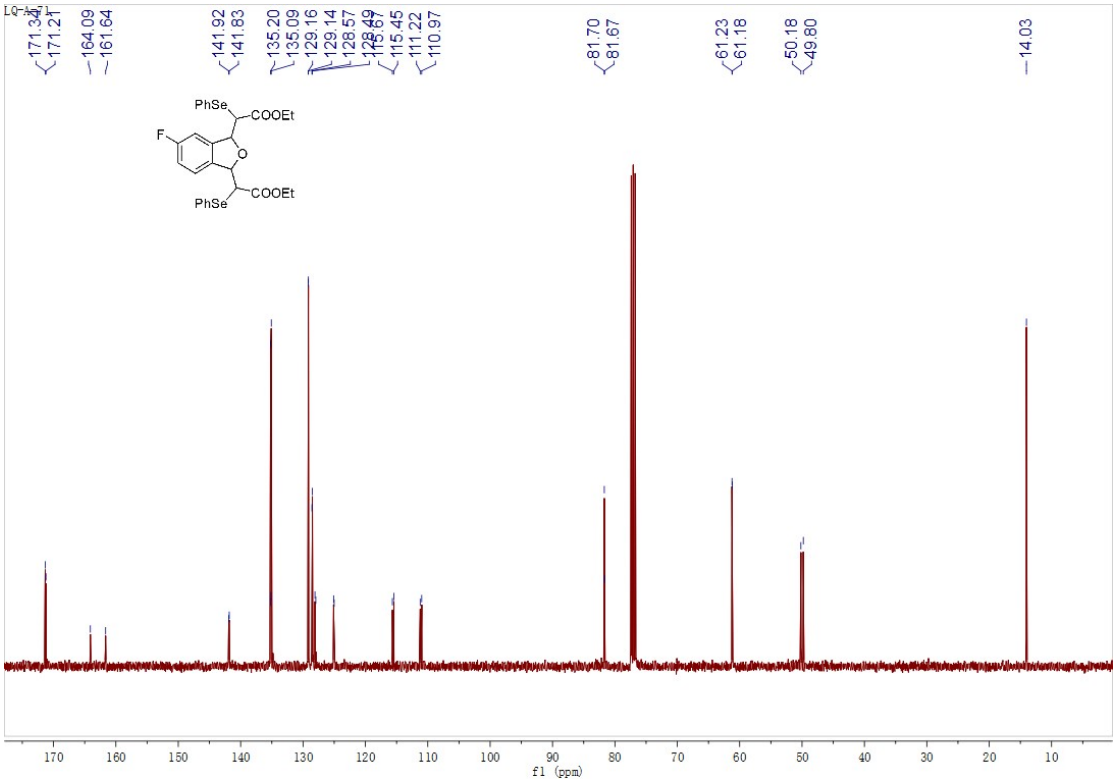
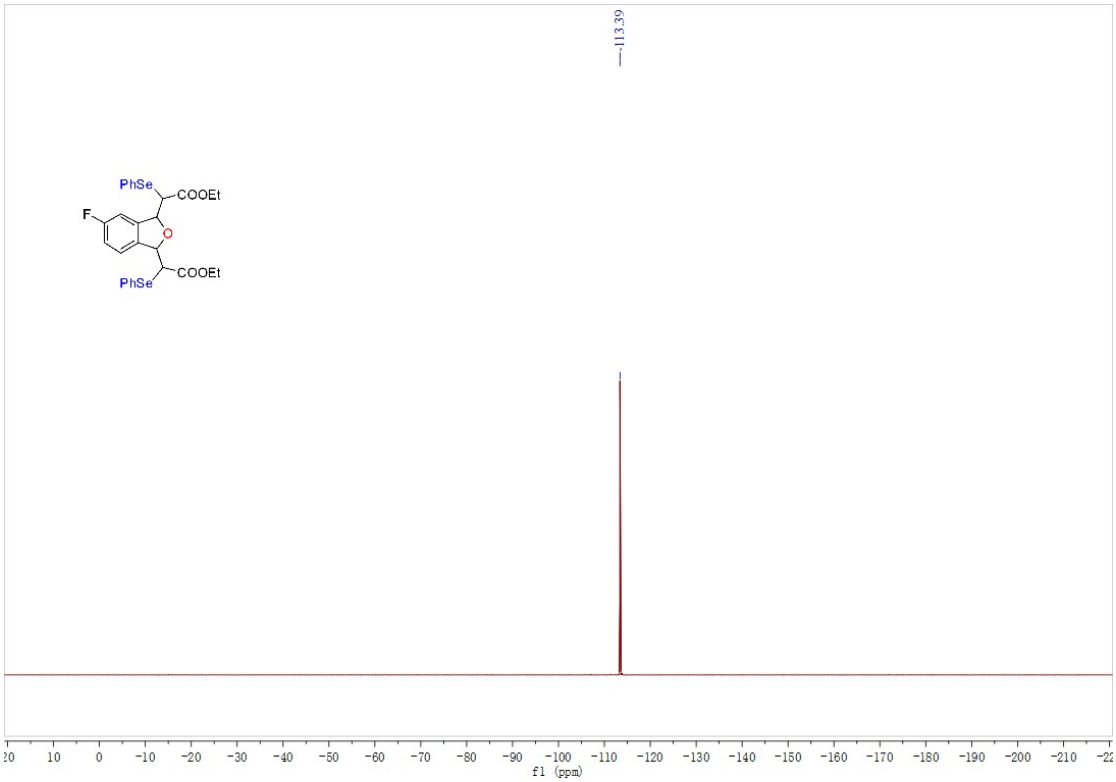




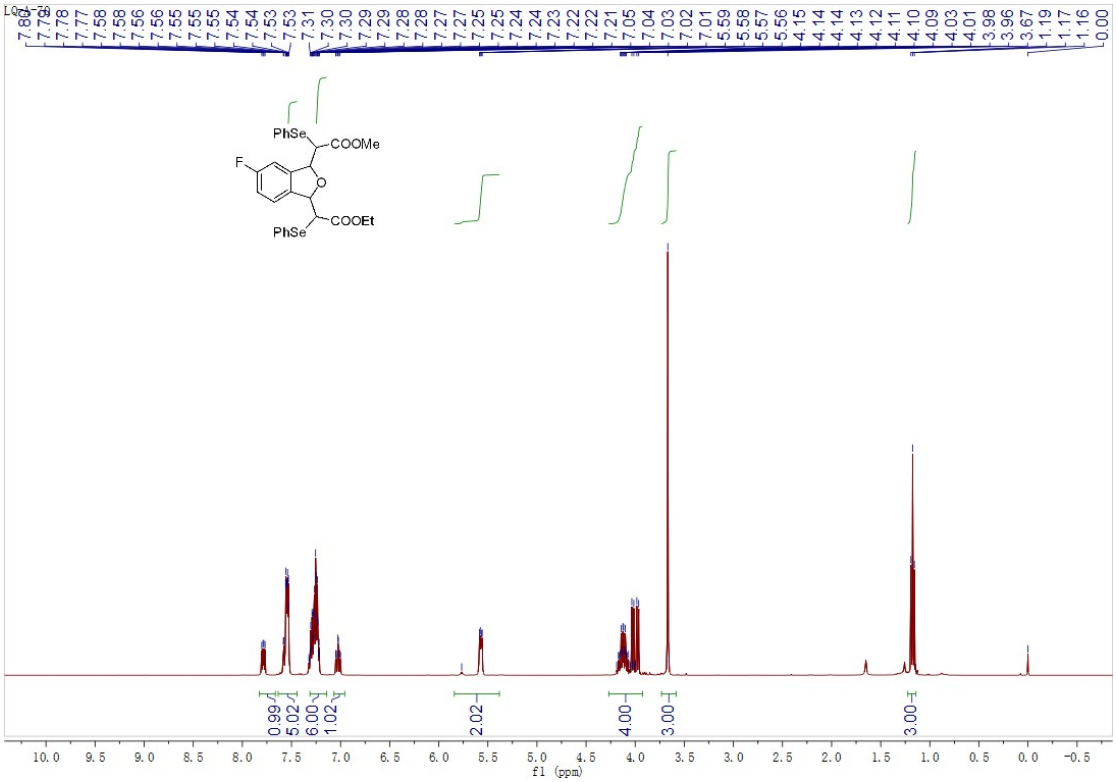
Compound 3na ^{1}H NMR, ^{13}C NMR and ^{19}F NMR

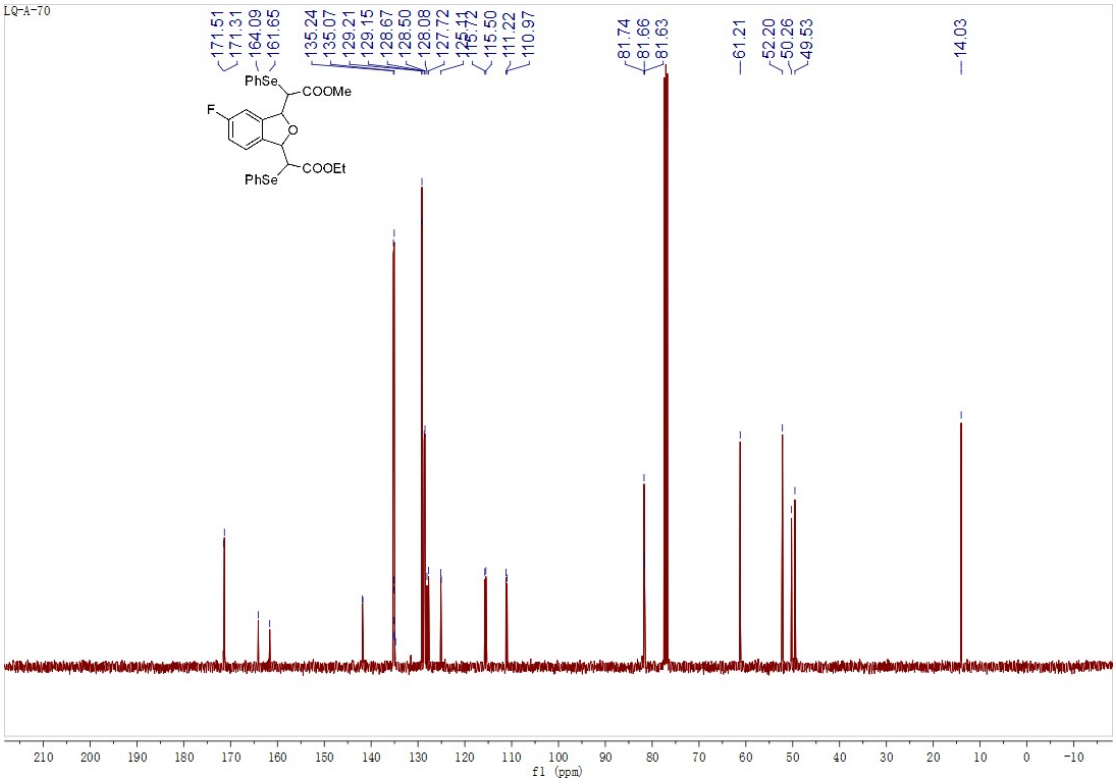


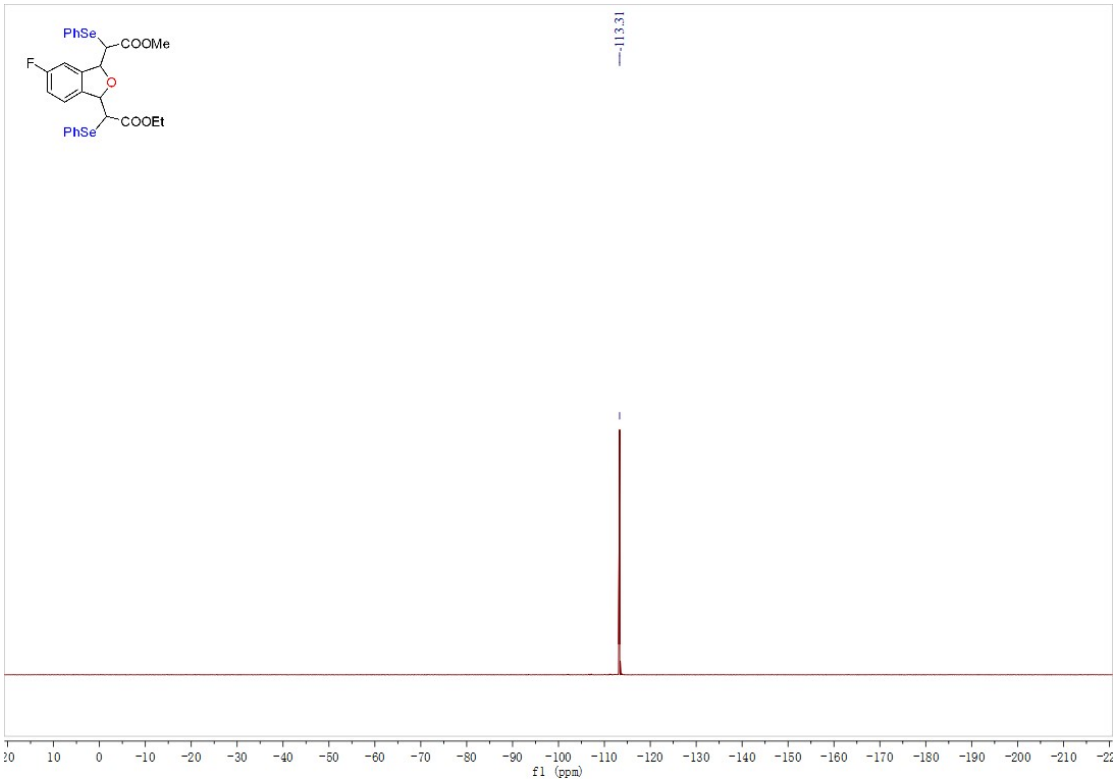



-113.39
PhSe
COOEt
F
O
COOEt
PhSe
f1 (ppm)

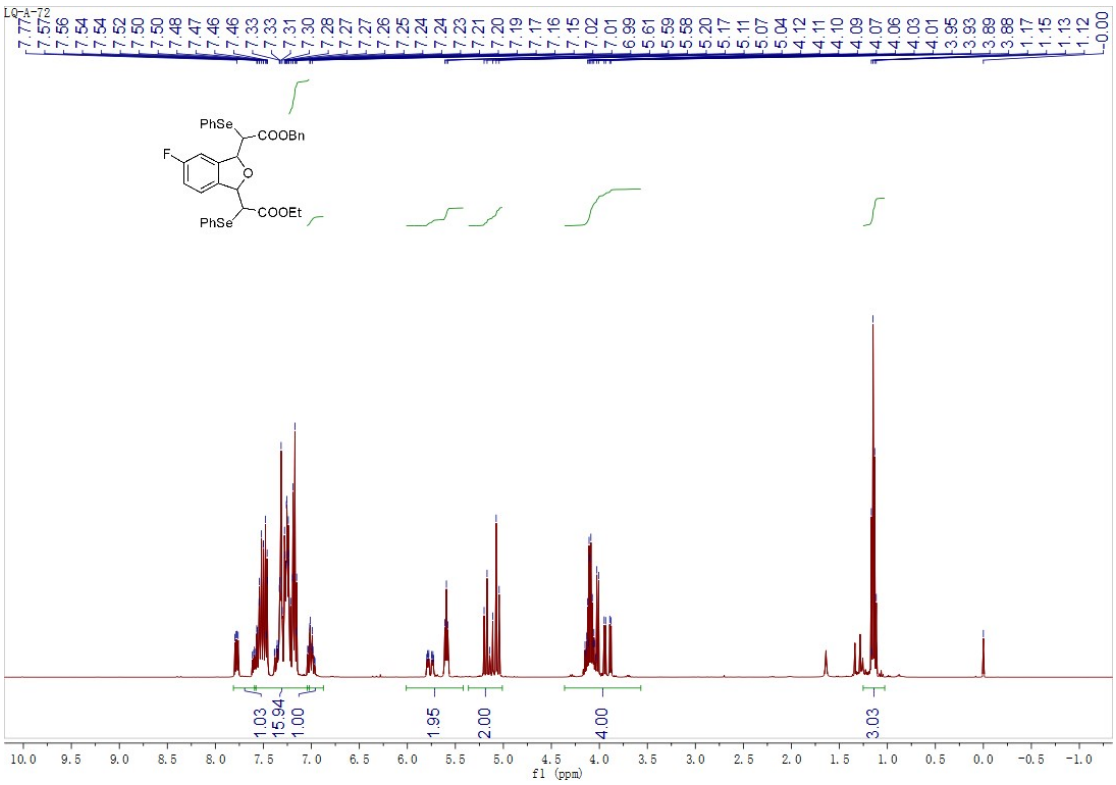
Compound 3oa ^{1}H NMR, ^{13}C NMR and ^{19}F NMR

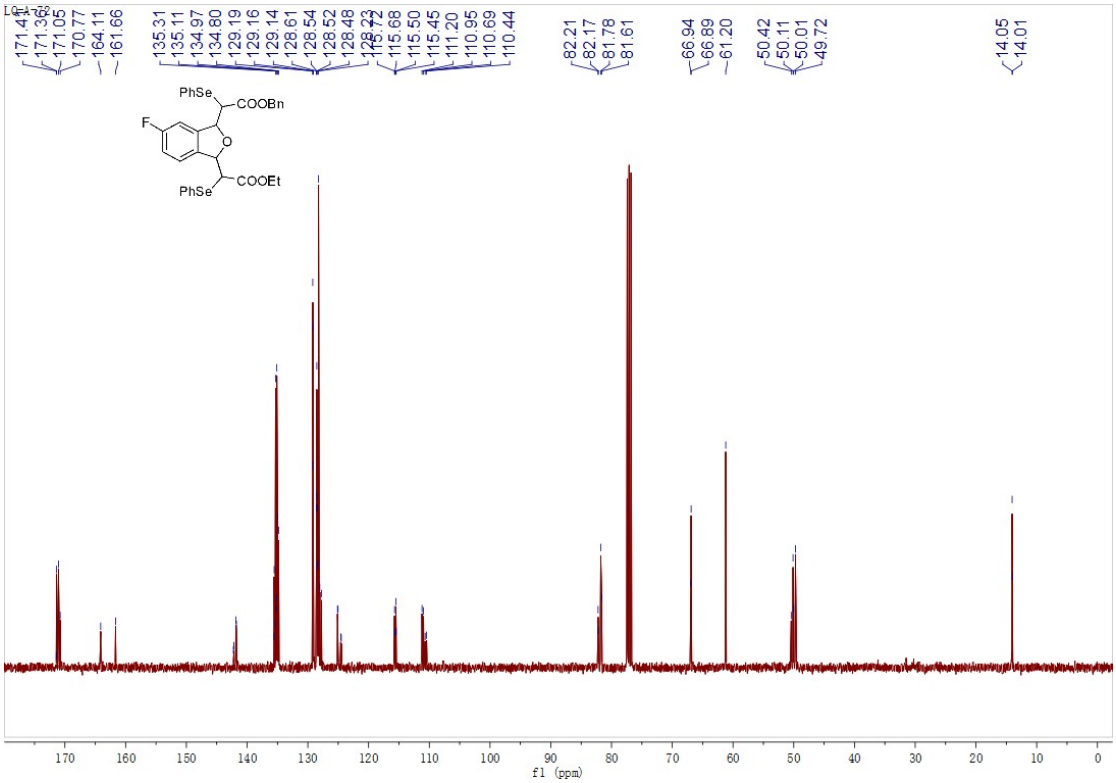


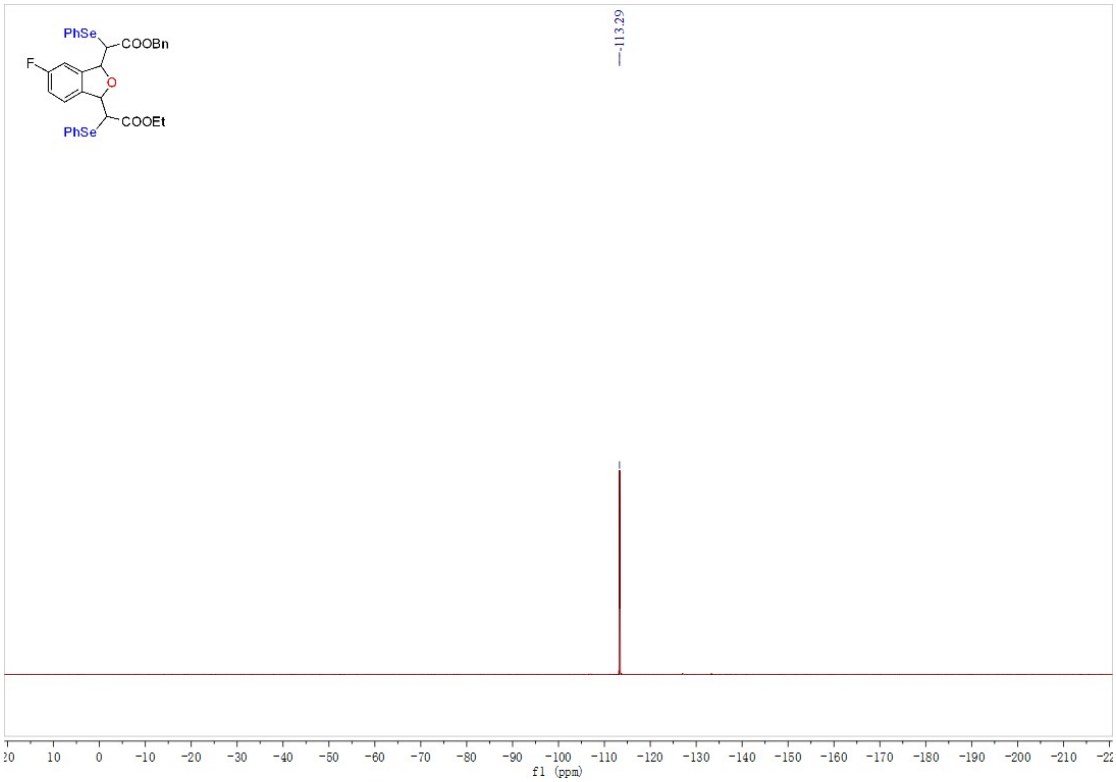



PhSe
COOMe
F
O
PhSe
COOEt
-113.31
f1 (ppm)

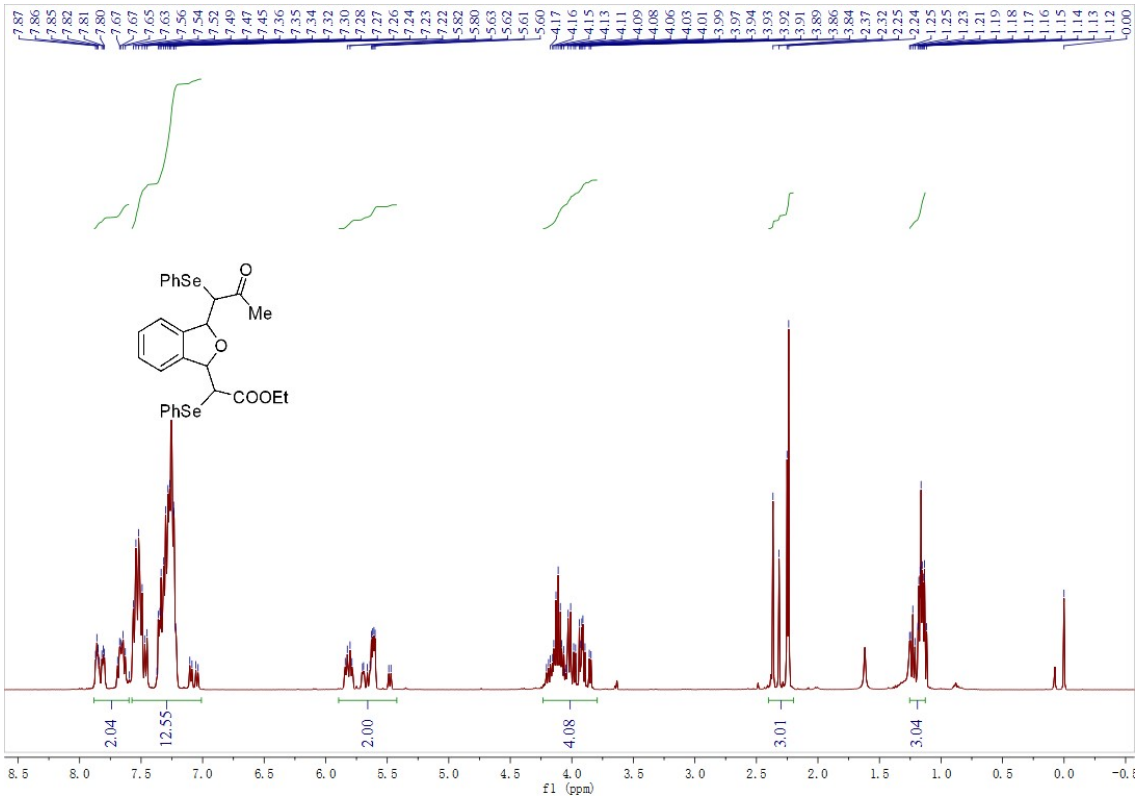
Compound 3pa ^{1}H NMR, ^{13}C NMR and ^{19}F NMR

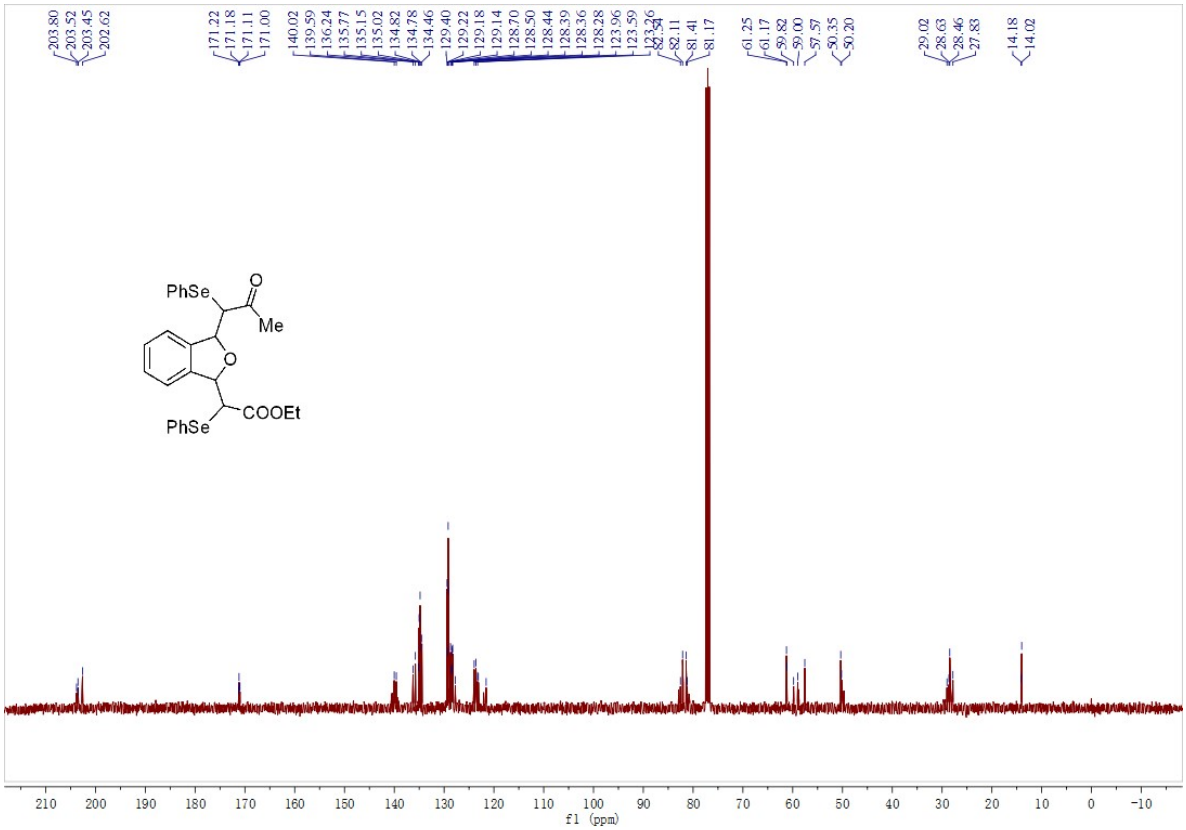




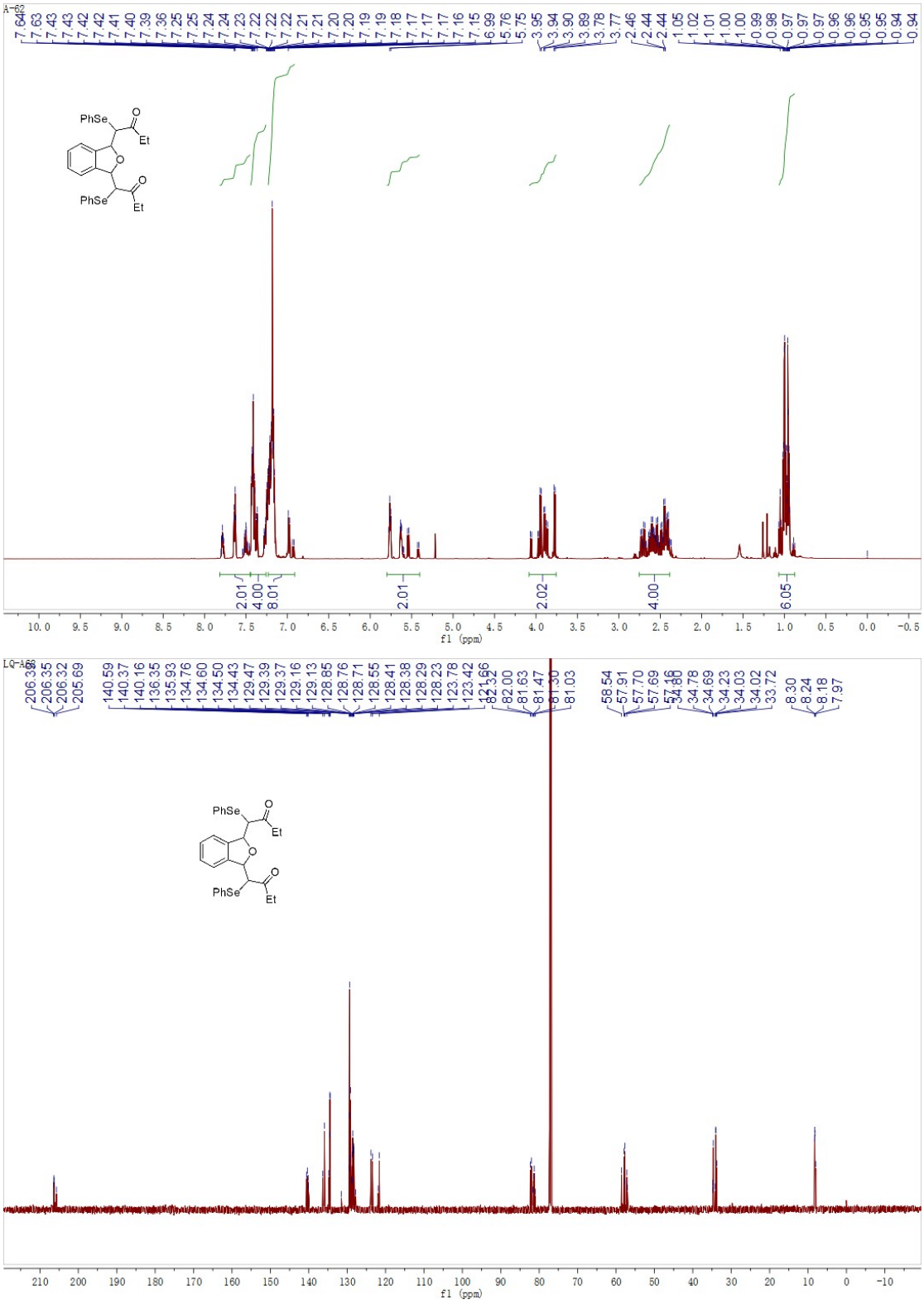

PhSe
COOBn
F
O
PhSe
COOEt
-113.29
f1 (ppm)

Compound 3qa ^{1}H NMR and ^{13}C NMR

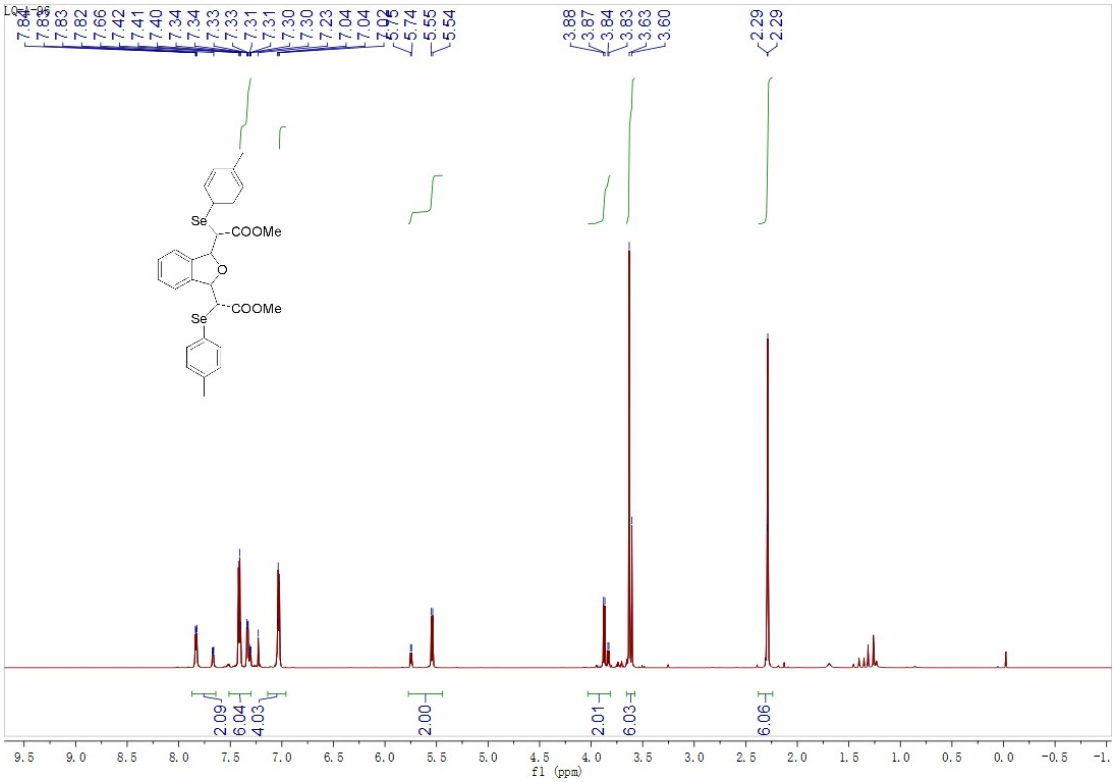


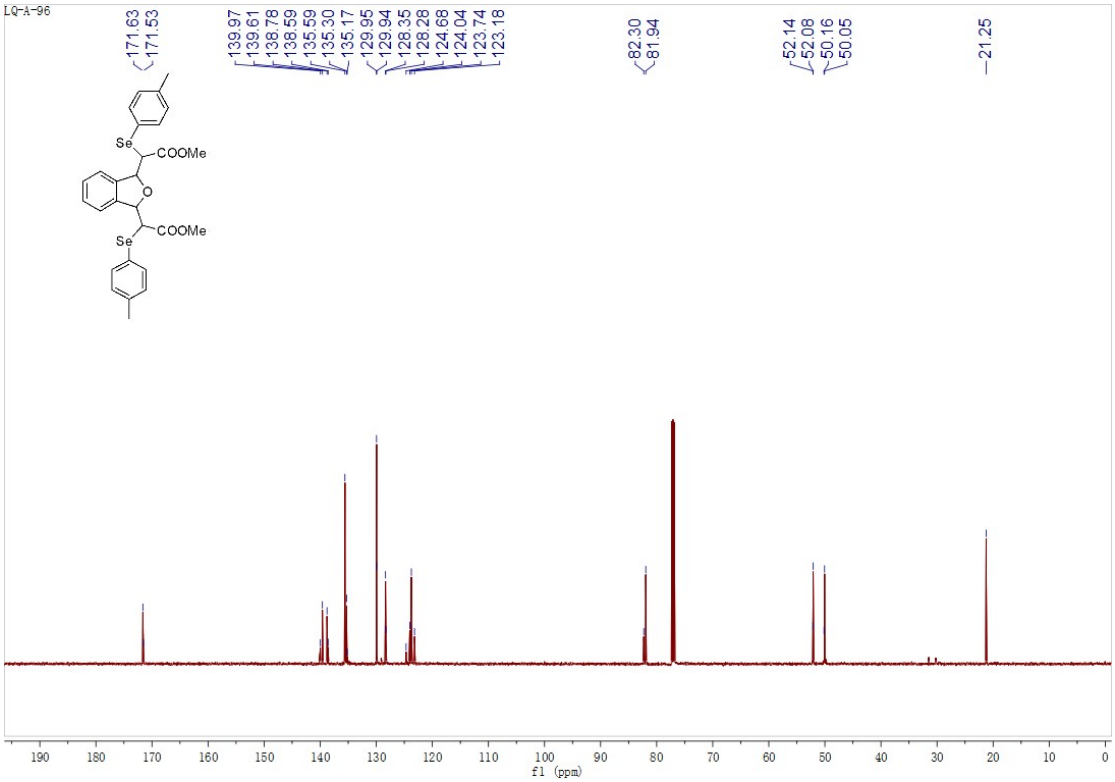


Compound 3ra ^{1}H NMR and ^{13}C NMR

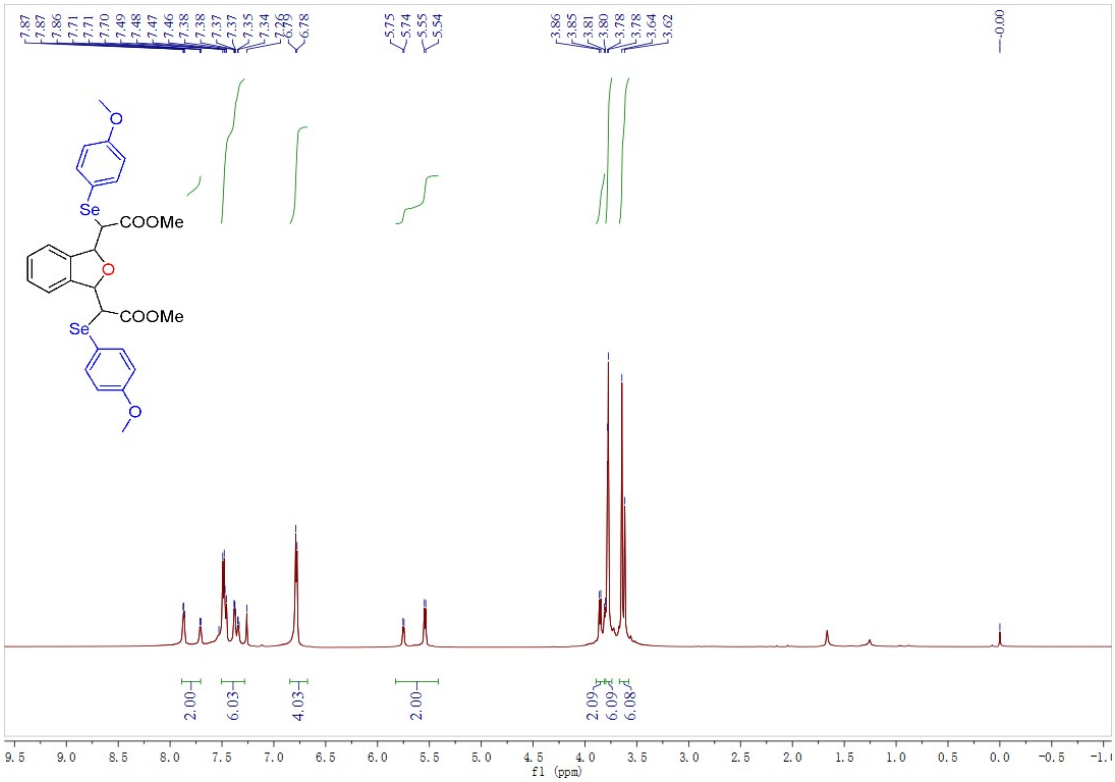


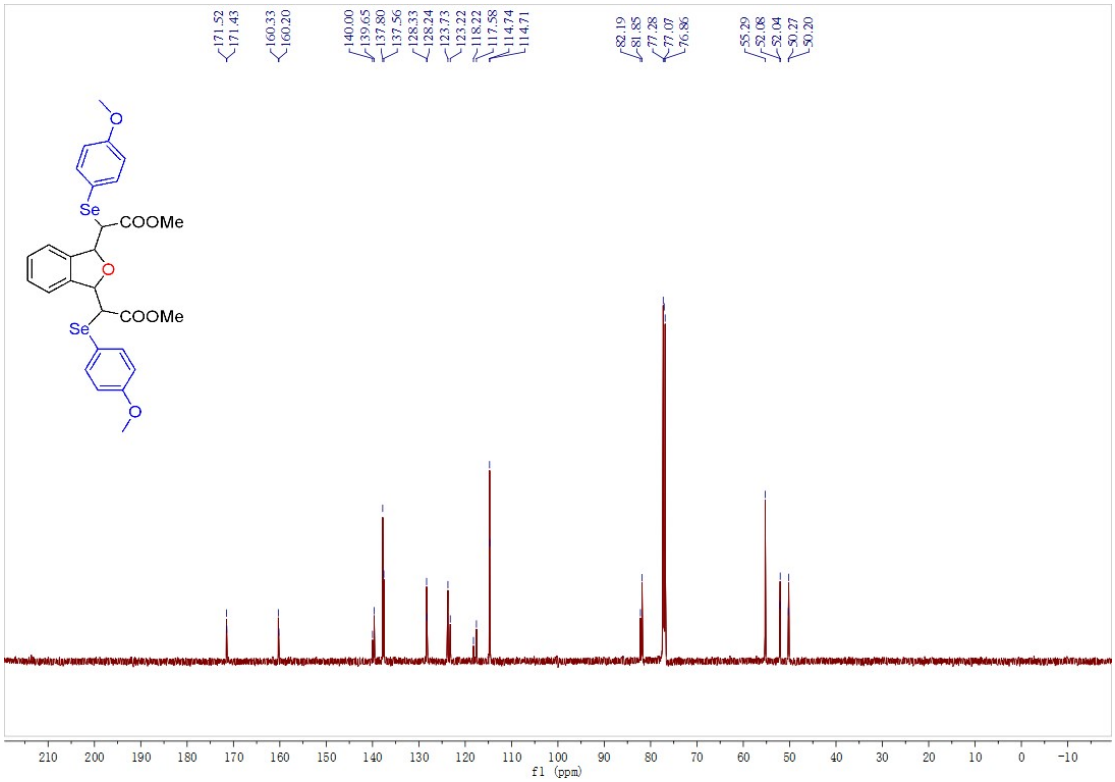
Compound 3ac ^{1}H NMR and ^{13}C NMR



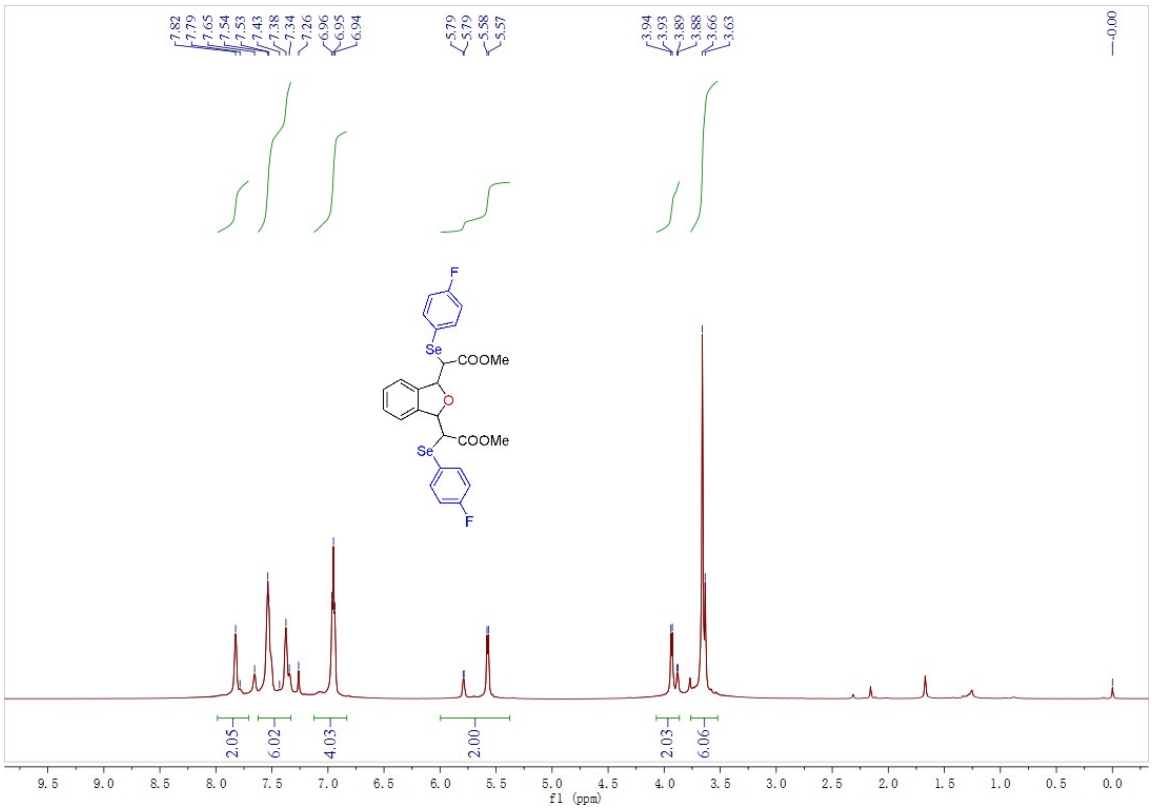


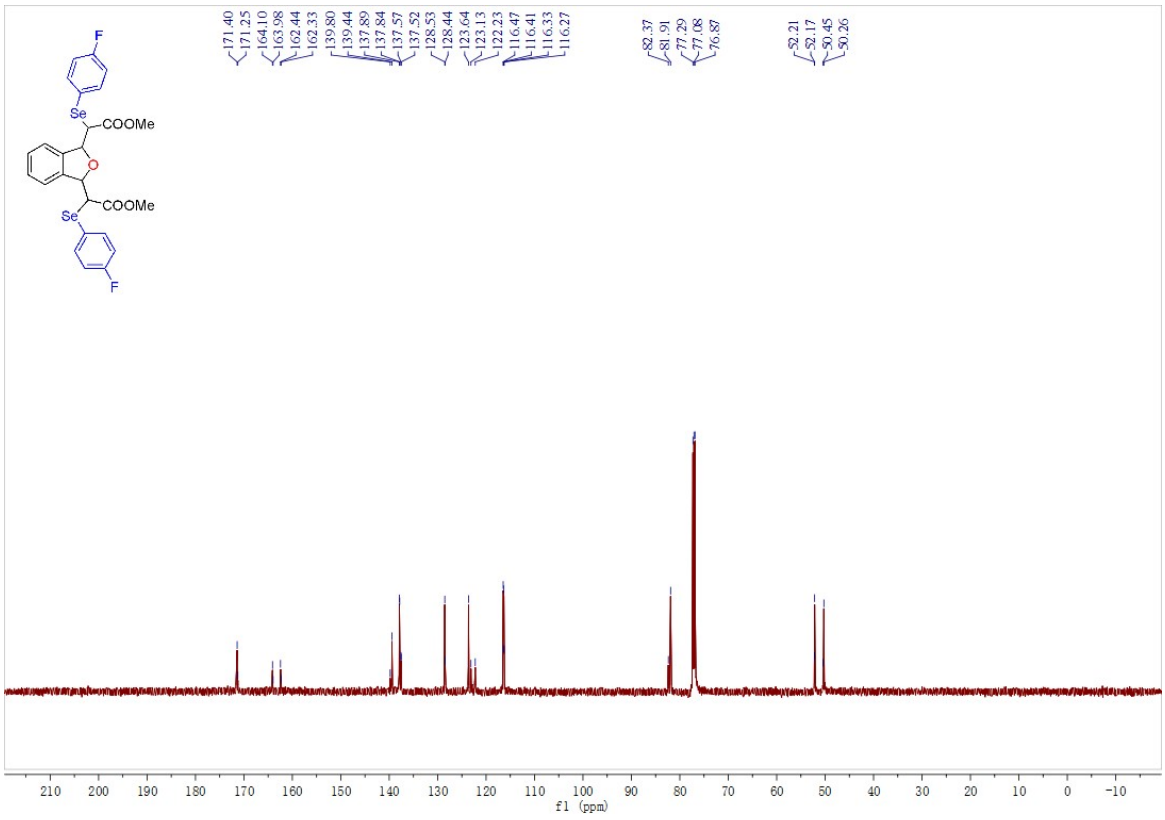
Compound 3ad ^{1}H NMR and ^{13}C NMR

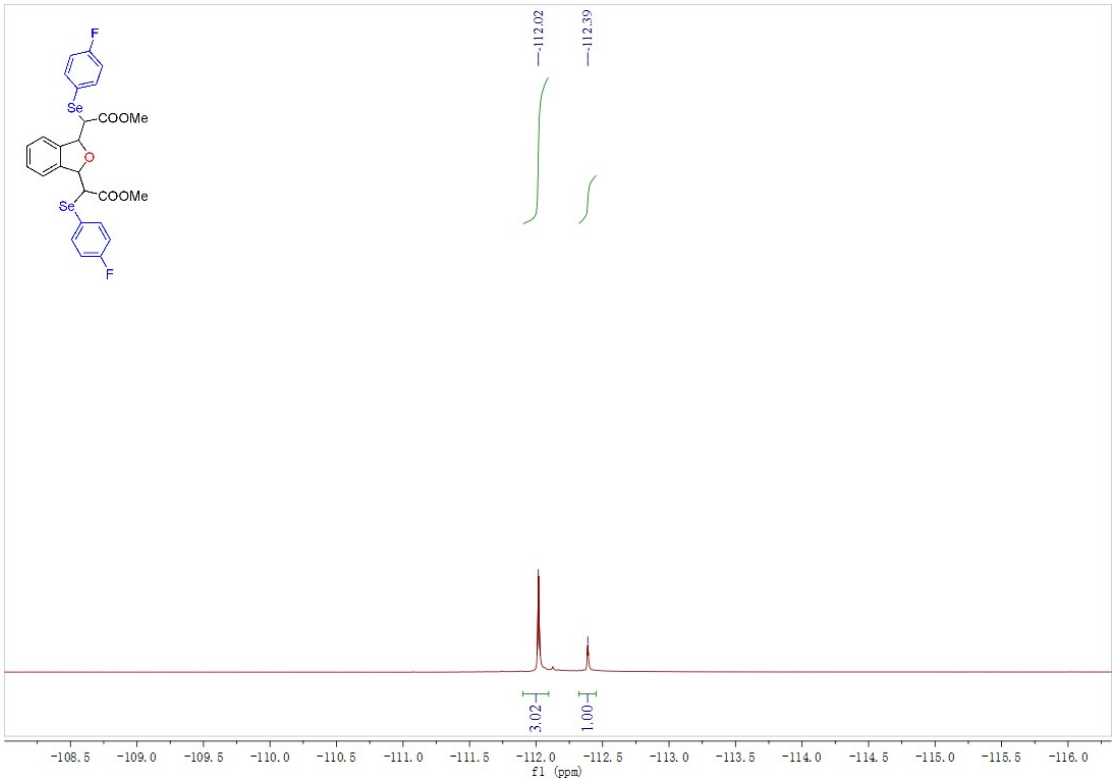




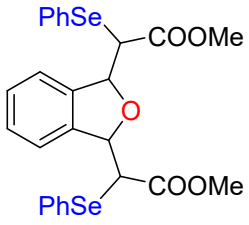
Compound 3ae ^{1}H NMR, ^{13}C NMR and ^{19}F NMR



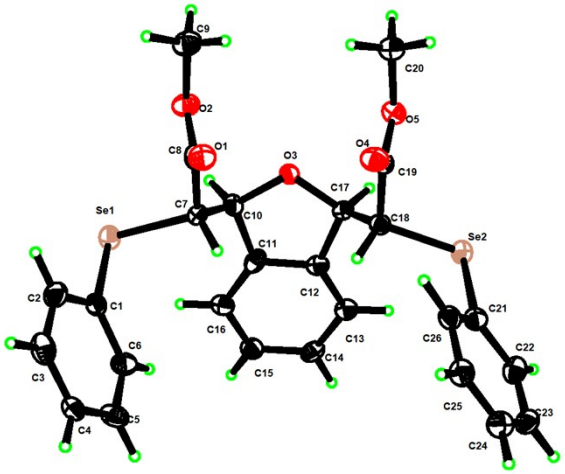



F
Se
COOMe
O
COOMe
Se
F
-112.02
-112.39
3.02
1.00
-108.5
-109.0
-109.5
-110.0
-110.5
-111.0
-111.5
-112.0
-112.5
-113.0
-113.5
-114.0
-114.5
-115.0
-115.5
-116.0
f1 (ppm)

7. X-ray crystallography data for 3aa.



3aa



^{1}H NMR of the collected crystal

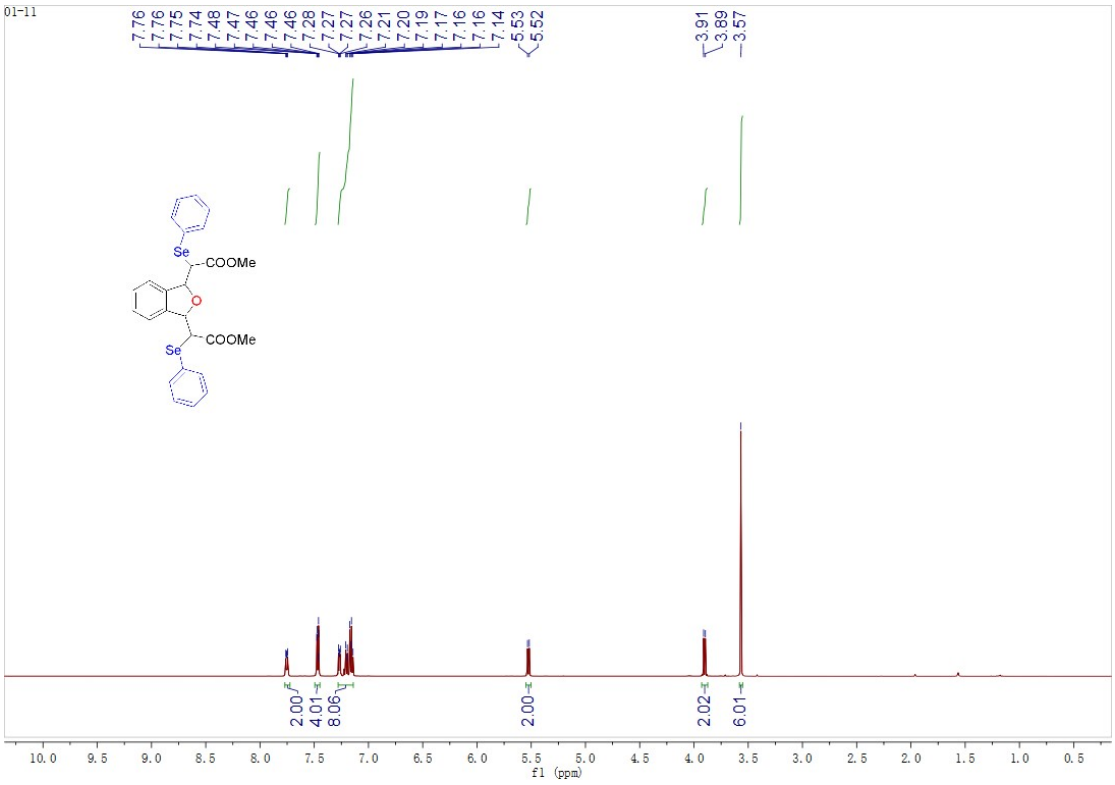


Table 1 Crystal data and structure refinement for 3aa (CCDC 2143795).

Identification code	**3aa**
Empirical formula	$C_{26}H_{24}O_5Se_2$
Formula weight	574.37
Temperature/K	150
Crystal system	triclinic
Space group	P-1

a/Å	9.754(3)
b/Å	9.783(3)
c/Å	27.883(9)
α/°	89.185(11)
β/°	88.372(13)
γ/°	62.580(9)
Volume/Å^3	2360.9(14)
Z	4
ρ_{calc}g/cm^3	1.616
μ/mm^{-1}	3.168
F(000)	1152.0
Crystal size/mm^3	0.15 × 0.08 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.384 to 51.582
Index ranges	-11 ≤ h ≤ 11, -11 ≤ k ≤ 11, -34 ≤ l ≤ 34
Reflections collected	25339
Independent reflections	8928 [R_{int} = 0.0808, R_{sigma} = 0.0936]
Data/restraints/parameters	8928/6/600
Goodness-of-fit on F^2	1.037
Final R indexes [I>=2σ (I)]	R_1 = 0.0917, wR_2 = 0.2493
Final R indexes [all data]	R_1 = 0.1385, wR_2 = 0.2987
Largest diff. peak/hole / e Å^{-3}	1.34/-0.82

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 3aa. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Se1	3118.7(10)	3014.4(10)	6091.0(3)	53.7(2)
Se2	7516.4(10)	7679.7(10)	6124.3(3)	52.7(2)
Se3	2659.7(9)	7706.8(9)	8843.1(3)	51.6(2)
Se4	-1734.0(10)	3072.4(10)	8898.8(3)	53.8(2)
O3	4977(6)	5764(6)	5523(2)	44.7(14)
O9	-3066(7)	6110(8)	9618(2)	65(2)
O5	4519(7)	8793(7)	5398(2)	55.6(17)
C1	2792(9)	3144(9)	6779(3)	47(2)
O10	-3486(8)	7270(7)	8899(2)	62(2)
O2	1889(7)	6145(8)	5408(2)	61(2)
O8	24(6)	5800(6)	9485(2)	47.7(15)
O1	1369(7)	7149(7)	6137(2)	60.6(19)

O4	3336(7)	9355(7)	6122(2)	59.9(19)
O7	-433(7)	8851(6)	9579(2)	56.4(18)
C32	2499(9)	7989(9)	8161(3)	48(2)
C37	2442(9)	4243(9)	9119(3)	46(2)
C41	2343(11)	1819(10)	9027(3)	54(2)
C17	6388(9)	5800(9)	5663(3)	46(2)
O6	-1522(7)	9374(7)	8858(2)	61.4(19)
C40	3890(10)	1172(10)	8874(3)	52(3)
C18	5875(9)	7167(9)	6047(3)	44.8(7)
C36	1430(8)	5851(8)	9318(3)	46(2)
C13	8850(9)	3577(10)	6009(3)	49(2)
C21	7366(9)	8026(9)	6804(3)	51(2)
C19	4440(10)	8559(9)	5869(3)	51(2)
C7	3610(9)	4778(9)	6014(3)	45(2)
C11	6508(9)	3408(10)	5853(3)	52(2)
C26	5945(10)	8635(9)	7062(3)	52(2)
C12	7355(9)	4232(10)	5866(3)	48(2)
C8	2172(9)	6141(10)	5866(3)	52(2)
C24	7239(11)	8520(10)	7783(4)	58(3)
C46	-2731(10)	6226(10)	9154(3)	54(2)
C44	-1261(9)	4792(9)	8988(3)	45(2)
C29	2255(10)	8297(11)	7165(4)	61(3)
C39	4686(10)	2025(10)	8822(3)	52(2)
C16	7211(10)	1852(10)	6005(3)	52(2)
C5	3825(11)	2684(11)	7568(4)	63(3)
C51	-2165(11)	2037(11)	7495(4)	60(3)
C14	9510(10)	2066(11)	6165(4)	60(3)
C33	949(8)	7233(9)	8937(3)	46(2)
C42	1607(10)	3419(10)	9165(3)	50(2)
C6	4056(11)	2596(10)	7076(4)	62(3)
C52	-1890(10)	1978(10)	7972(4)	59(3)
C15	8741(10)	1159(11)	6143(3)	57(3)
C34	-452(9)	8578(8)	9116(3)	48(2)
C2	1316(10)	3735(11)	6970(3)	59(3)
C49	-3153(10)	4811(10)	7520(3)	54(2)
C50	-2755(11)	3451(10)	7255(4)	60(3)
C45	-4421(12)	7382(15)	9810(4)	82(4)
C27	2912(10)	6728(10)	7872(4)	57(3)
C38	3962(9)	3575(10)	8952(3)	53(2)

C30	1873(11)	9539(11)	7468(4)	68(3)
C43	32(9)	4396(9)	9353(3)	46(2)
C47	-2192(8)	3317(9)	8225(3)	45(2)
C25	5942(10)	8832(10)	7552(4)	58(3)
C20	3106(12)	10038(12)	5194(4)	67(3)
C35	-1807(12)	10047(11)	9790(4)	66(3)
C3	1116(11)	3766(11)	7466(4)	66(3)
C23	8646(10)	7973(10)	7527(3)	56(3)
C28	2779(11)	6874(12)	7372(4)	65(3)
C10	4945(9)	4352(8)	5654(3)	45(2)
C31	2002(10)	9380(10)	7954(4)	59(3)
C9	584(11)	7472(13)	5221(4)	68(3)
C48	-2863(11)	4720(11)	8001(4)	60(3)
C4	2316(10)	3292(9)	7763(3)	57(2)
C22	8703(11)	7742(11)	7047(4)	61(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 3aa. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Se1	56.3(4)	54.0(4)	56.8(5)	-1.5(4)	0.7(4)	-30.6(3)
Se2	51.9(4)	54.2(4)	57.0(5)	0.0(4)	0.4(4)	-28.9(3)
Se3	50.9(4)	49.2(4)	59.4(5)	1.1(4)	-3.2(4)	-26.9(3)
Se4	56.0(4)	50.3(4)	60.1(5)	4.9(4)	-5.6(4)	-28.6(3)
O3	36(2)	41(2)	57(3)	10(2)	-6(2)	-16.7(17)
O9	50(3)	72(4)	59(4)	1(3)	0(3)	-17(3)
O5	59(3)	49(3)	57(3)	6(3)	-3(3)	-23(2)
C1	55(4)	45(3)	46(4)	2(3)	0(3)	-28(2)
O10	54(3)	43(3)	63(4)	-4(3)	4(3)	1(3)
O2	44(3)	59(3)	64(4)	2(3)	-4(3)	-10(3)
O8	37(2)	37(2)	63(3)	2(2)	7(2)	-12.1(18)
O1	50(3)	53(3)	67(4)	-2(3)	0(3)	-14(2)
O4	44(3)	52(3)	66(4)	5(3)	-2(3)	-8(2)
O7	61(3)	32(3)	63(4)	-11(3)	1(3)	-10(2)
C32	47(3)	47(3)	57(5)	-4(3)	0(3)	-27(2)
C37	38(3)	47(4)	49(4)	3(3)	-11(3)	-16(3)
C41	64(4)	47(4)	51(5)	10(4)	-6(4)	-25(3)

C17	43(3)	46(4)	48(4)	6(3)	-10(3)	-19(3)
O6	55(3)	54(3)	67(4)	10(3)	-12(3)	-18(2)
C40	41(4)	46(4)	49(5)	2(4)	0(4)	-3(3)
C18	44.3(8)	44.9(8)	44.4(9)	1.1(7)	-0.2(7)	-20.1(5)
C36	35(3)	36(3)	65(5)	4(3)	0(3)	-16(2)
C13	37(4)	52(4)	50(5)	-3(4)	4(3)	-13(3)
C21	36(4)	40(4)	67(5)	6(4)	-6(4)	-9(3)
C19	57(4)	50(4)	53(5)	6(4)	-6(4)	-28(3)
C7	44(3)	48(3)	48(4)	3(3)	-9(3)	-25(2)
C11	47(4)	64(4)	49(5)	-18(4)	4(3)	-28(3)
C26	46(4)	44(4)	65(5)	7(4)	-3(4)	-20(3)
C12	40(4)	56(4)	43(4)	1(4)	1(3)	-17(3)
C8	56(4)	58(4)	51(5)	-6(4)	8(4)	-34(3)
C24	62(5)	45(4)	57(5)	-13(4)	4(4)	-16(3)
C46	55(4)	56(4)	56(5)	5(4)	-2(4)	-29(3)
C44	40(3)	43(4)	45(4)	1(3)	1(3)	-13(3)
C29	55(4)	73(5)	63(6)	-3(4)	-8(4)	-34(3)
C39	40(4)	46(4)	57(5)	-1(4)	5(4)	-10(3)
C16	42(4)	56(4)	47(5)	4(4)	6(4)	-14(3)
C5	52(5)	56(5)	64(5)	19(4)	-1(4)	-10(4)
C51	59(5)	47(4)	69(6)	-5(4)	-7(4)	-20(3)
C14	43(4)	65(5)	66(6)	-1(4)	-15(4)	-18(3)
C33	24(3)	50(4)	52(4)	-17(3)	-6(3)	-6(3)
C42	54(4)	48(4)	44(4)	7(3)	-9(3)	-20(3)
C6	49(4)	48(4)	78(6)	18(4)	-16(4)	-12(3)
C52	60(4)	55(4)	63(5)	13(4)	-14(4)	-27(3)
C15	47(4)	56(4)	63(5)	8(4)	-2(4)	-18(3)
C34	43(3)	30(3)	70(5)	1(3)	13(4)	-16(2)
C2	48(4)	76(5)	59(5)	2(4)	0(4)	-33(3)
C49	55(4)	44(4)	60(5)	8(4)	-6(4)	-19(3)
C50	60(4)	45(4)	71(6)	-10(4)	-7(4)	-22(3)
C45	39(5)	91(8)	80(7)	-6(6)	-1(5)	2(5)
C27	54(4)	42(4)	72(6)	-2(4)	8(4)	-19(3)
C38	37(3)	53(4)	63(5)	9(4)	-3(4)	-17(3)
C30	77(5)	71(5)	71(6)	14(4)	-6(5)	-47(3)
C43	47(3)	46(3)	50(4)	3(3)	3(3)	-26(2)
C47	36(3)	51(4)	50(4)	0(3)	0(3)	-20(3)
C25	44(4)	52(4)	74(6)	0(4)	1(4)	-19(3)
C20	66(5)	65(5)	68(6)	11(5)	-15(5)	-28(4)

C35	71(5)	50(5)	66(6)	-3(4)	17(5)	-18(4)
C3	60(4)	56(4)	81(7)	-9(5)	21(4)	-29(3)
C23	43(4)	57(4)	66(6)	-1(4)	-10(4)	-21(3)
C28	52(5)	56(5)	76(6)	-11(5)	4(5)	-14(4)
C10	34(3)	28(3)	67(5)	3(3)	-2(3)	-8(2)
C31	60(4)	55(4)	74(6)	-1(4)	-7(4)	-36(3)
C9	45(5)	73(6)	70(6)	1(5)	-1(4)	-14(4)
C48	52(4)	51(4)	68(6)	1(4)	-9(4)	-15(3)
C4	72(4)	54(4)	56(5)	14(4)	-6(4)	-38(3)
C22	49(4)	56(4)	76(6)	-10(4)	1(4)	-23(3)

Table 4 Bond Lengths for 3aa.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Se1	C1	1.931(8)	C18	C19	1.524(10)
Se1	C7	1.999(9)	C36	C33	1.606(12)
Se2	C18	1.904(9)	C13	C12	1.364(12)
Se2	C21	1.921(10)	C13	C14	1.382(13)
Se3	C32	1.918(9)	C21	C26	1.410(12)
Se3	C33	1.937(9)	C21	C22	1.395(13)
Se4	C44	1.959(10)	C7	C8	1.486(10)
Se4	C47	1.929(9)	C7	C10	1.520(11)
O3	C17	1.457(10)	C11	C12	1.396(14)
O3	C10	1.438(10)	C11	C16	1.416(12)
O9	C46	1.340(11)	C11	C10	1.490(11)
O9	C45	1.430(12)	C26	C25	1.381(14)
O5	C19	1.338(11)	C24	C25	1.339(14)
O5	C20	1.478(11)	C24	C23	1.399(13)
C1	C6	1.391(13)	C46	C44	1.539(11)
C1	C2	1.375(12)	C44	C43	1.546(12)
O10	C46	1.186(10)	C29	C30	1.388(14)
O2	C8	1.315(11)	C29	C28	1.369(14)

O2	C9	1.441(11)	C39	C38	1.395(12)
O8	C36	1.458(10)	C16	C15	1.388(13)
O8	C43	1.424(10)	C5	C6	1.380(14)
O1	C8	1.196(10)	C5	C4	1.406(13)
O4	C19	1.210(10)	C51	C52	1.358(14)
O7	C34	1.324(11)	C51	C50	1.398(13)
O7	C35	1.430(11)	C14	C15	1.404(16)
C32	C27	1.374(13)	C33	C34	1.474(10)
C32	C31	1.345(12)	C42	C43	1.471(11)
C37	C36	1.524(11)	C52	C47	1.401(13)
C37	C42	1.387(14)	C2	C3	1.388(14)
C37	C38	1.385(11)	C49	C50	1.416(13)
C41	C40	1.396(13)	C49	C48	1.372(13)
C41	C42	1.443(12)	C27	C28	1.400(15)
C17	C18	1.607(12)	C30	C31	1.362(14)
C17	C12	1.496(11)	C47	C48	1.369(12)
O6	C34	1.220(10)	C3	C4	1.348(14)
C40	C39	1.380(15)	C23	C22	1.356(14)

Table 5 Bond Angles for 3aa.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	Se1	C7	97.4(4)	O1	C8	C7	123.2(8)
C18	Se2	C21	100.4(4)	C25	C24	C23	119.9(9)
C32	Se3	C33	96.8(4)	O9	C46	C44	111.1(7)
C47	Se4	C44	99.7(4)	O10	C46	O9	124.9(8)
C10	O3	C17	113.0(5)	O10	C46	C44	124.0(8)
C46	O9	C45	115.8(7)	C46	C44	Se4	109.6(6)
C19	O5	C20	115.3(7)	C46	C44	C43	110.1(7)
C6	C1	Se1	119.6(7)	C43	C44	Se4	111.8(5)
C2	C1	Se1	119.9(7)	C28	C29	C30	117.2(10)
C2	C1	C6	120.5(8)	C40	C39	C38	119.5(8)
C8	O2	C9	117.8(7)	C15	C16	C11	119.7(10)
C43	O8	C36	111.5(6)	C6	C5	C4	119.7(9)
C34	O7	C35	117.3(7)	C52	C51	C50	119.6(9)
C27	C32	Se3	119.2(7)	C13	C14	C15	122.3(8)
C31	C32	Se3	122.4(7)	C36	C33	Se3	108.8(5)
C31	C32	C27	118.4(9)	C34	C33	Se3	111.6(6)
C42	C37	C36	107.5(7)	C34	C33	C36	109.2(7)

C38	C37	C36	130.0(9)	C37	C42	C41	118.6(8)
C38	C37	C42	122.3(8)	C37	C42	C43	111.2(7)
C40	C41	C42	117.5(10)	C41	C42	C43	130.1(9)
O3	C17	C18	106.4(6)	C5	C6	C1	119.7(9)
O3	C17	C12	104.2(7)	C51	C52	C47	120.8(9)
C12	C17	C18	113.9(7)	C16	C15	C14	118.5(9)
C39	C40	C41	122.6(8)	O7	C34	C33	113.8(7)
C17	C18	Se2	109.6(5)	O6	C34	O7	123.3(7)
C19	C18	Se2	110.4(6)	O6	C34	C33	122.8(8)
C19	C18	C17	108.8(7)	C1	C2	C3	118.7(9)
O8	C36	C37	104.0(7)	C48	C49	C50	119.9(8)
O8	C36	C33	107.8(6)	C51	C50	C49	119.2(9)
C37	C36	C33	115.5(7)	C32	C27	C28	121.5(9)
C12	C13	C14	118.1(10)	C37	C38	C39	119.3(9)
C26	C21	Se2	122.5(7)	C31	C30	C29	122.6(9)
C22	C21	Se2	118.6(7)	O8	C43	C44	107.5(6)
C22	C21	C26	118.8(9)	O8	C43	C42	105.0(7)
O5	C19	C18	112.5(7)	C42	C43	C44	114.9(7)
O4	C19	O5	123.3(7)	C52	C47	Se4	116.6(6)
O4	C19	C18	124.2(8)	C48	C47	Se4	123.1(7)
C8	C7	Se1	107.4(6)	C48	C47	C52	120.1(8)
C8	C7	C10	113.3(7)	C24	C25	C26	121.8(9)
C10	C7	Se1	109.6(5)	C4	C3	C2	122.1(9)
C12	C11	C16	118.9(8)	C22	C23	C24	120.2(9)
C12	C11	C10	111.7(8)	C29	C28	C27	119.6(10)
C16	C11	C10	129.3(9)	O3	C10	C7	106.6(6)
C25	C26	C21	118.7(8)	O3	C10	C11	102.8(7)
C13	C12	C17	129.4(9)	C11	C10	C7	115.0(7)
C13	C12	C11	122.2(8)	C32	C31	C30	120.7(9)
C11	C12	C17	108.1(7)	C47	C48	C49	120.1(9)
O2	C8	C7	113.3(7)	C3	C4	C5	119.2(9)
O1	C8	O2	123.4(8)	C23	C22	C21	120.4(9)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 3aa.

Atom	*x*	*y*	*z*	U(eq)
H41	1796.98	1227.76	9038.94	65
H17	6920.78	5992.52	5377.78	55
H40	4415.16	103.24	8802.52	63

H18	5626.63	6821.09	6362.26	54
H36	1945.86	6044.64	9595.19	55
H13	9421.09	4144.59	6001.97	59
H7	3922.88	4996.08	6330.31	54
H26	5007.44	8904.54	6902.31	62
H24	7204.08	8670.61	8119.39	70
H44	-904.26	5028.16	8672.19	54
H29	2157.41	8428.88	6827.14	74
H39	5717.78	1559.34	8697.95	62
H16	6638.97	1285.94	6013.01	62
H5	4682.6	2336.15	7772.79	76
H51	-1957.89	1123.21	7325.88	72
H14	10519.32	1625	6290.16	72
H33	724.92	6896.27	8624.42	55
H6	5074.37	2162.22	6941.61	75
H52	-1488.95	1017.23	8134.78	71
H15	9255.1	94.08	6221.81	69
H2	449.58	4114.48	6767.81	71
H49	-3619.64	5785.85	7364.86	65
H50	-2887.95	3497.46	6917.79	72
H45A	-4986.64	8077.05	9548.5	123
H45B	-4122.01	7938.66	10039.74	123
H45C	-5082.25	7000.83	9972.1	123
H27	3296.06	5734.96	8013.08	69
H38	4506.38	4167.52	8927.26	63
H30	1506.26	10538.01	7332.63	81
H43	-176.63	3901.97	9643.61	55
H25	4994.25	9198.78	7729.58	70
H20A	3194.61	9996.43	4842.48	101
H20B	2978.4	11036.88	5304.06	101
H20C	2206.95	9905.01	5298.74	101
H35A	-2131.18	10992.75	9601.11	100
H35B	-1602.95	10233.22	10118.89	100
H35C	-2629.09	9735.4	9796.23	100
H3	101.54	4133.11	7599.27	79
H23	9564.73	7762.56	7689.94	67
H28	3050.75	5989.64	7179.17	78
H10	4794.92	3844.5	5366.02	54
H31	1738.63	10262.04	8147.13	71

H9A	653.51	7456.39	4869.26	102
H9B	583.32	8411.58	5338.57	102
H9C	-373.4	7452.62	5326.72	102
H48	-3129.61	5632.38	8179.27	72
H4	2143.22	3366.86	8101.03	69
H22	9659.3	7385.33	6874.5	73