

Supporting Information for

Electrochemical Oxidative Cyclization of Alkenes, Boronic Acids, and Dichalcogenides to Access Chalcogenated Boronic Esters and 1,3-Diols

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1. General considerations

Unless otherwise noted, commercial reagents were purchased from Alfa Aesar, TCI, J&K or Adamas and used without further purification. All reactions were carried out using oven-dried glassware and all catalytic reactions proceeded without special care. Column chromatography was performed on 200-300 mesh silica gel (Huanghai, China).

^1H , ^{19}F and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra were recorded on an Bruker Ascend 400MHz spectrometer and Bruker Ultrashield 300MHz at ambient temperature. ^1H NMR spectra are referred to the TMS signal ($\delta = 0$ ppm) and ^{13}C NMR spectra are referred to the residual solvent signal ($\delta = 77.16$ ppm). Data for ^1H NMR are reported as follows: chemical shifts (δ ppm), multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constants (Hz), integration.

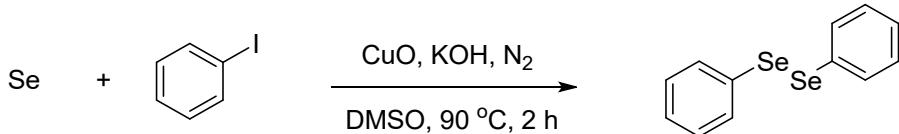
The instrument for electrolysis is ElectraSyn 2.0 Package (IKA). The anode electrode is vitreous carbon plate (52 mm \times 8 mm \times 2 mm) and cathodic electrode was platinum plate (52 mm \times 8 mm \times 2 mm). The data of HRMS was carried out on a waters G2-XS high-resolution mass spectrometer (HR-ESI-MS) or Agilent 7250 GC/QTOF. Melting point were recorded using a SGW X-4 Melting Point Apparatus.

Note: In the ^{13}C NMR spectral data, the carbons connected to boron are not listed due to quadrupole broadening and spin–spin coupling with boron.

2. Experimental procedures and characterization data

2.1 Experimental procedures

Synthesis of compounds 3 according to the following procedure¹:



To a stirred solution of Se⁰ metal (156 mg, 2.0 mmol) and iodobenzene (204 mg, 1.0 mmol) in dry DMSO (2.0 mL) was added CuO (10.0 mol%) followed by KOH (112 mg, 2.0 mmol) under nitrogen atmosphere at 90 °C for 2h. 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 (eluted with petroleum ether : ethyl acetate = 10:1) separation to give pure diselenide (yellow solid) in 96% yield.

All the selenide substrates were known or commercially available.

Synthesis of product 4 according to the following procedure:

As exemplified for 4a:

(E)-3-phenylprop-2-en-1-ol (0.3 mmol, 1.0 equiv), phenylboronic acid (0.3 mmol, 1.0 equiv), 1,2-diphenyldiselenane (0.3 mmol, 1.0 equiv), KBr (1.2 mmol), TFA (0.6 mmol), CH₃CN (4 mL) and H₂O (1 mL) were placed in a 10 mL undivided electrolytic cell with a vitreous carbon plate anode (52 mm×8 mm×2 mm) and a platinum plate cathode (52 mm×8 mm×2 mm). The electrolysis was carried out at room temperature under a constant current of 5 mA for 12 hours. When the reaction was finished, the resulting solution was quenched with 10 mL brine and extracted with 3×10 mL ethyl acetate. The extract was dried with Na₂SO₄. The solvent was removed with a rotary evaporator. The pure product was obtained by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 10: 1).

Gram-scale experiment for 4a:

(E)-3-phenylprop-2-en-1-ol (4.0 mmol, 1.0 equiv), phenylboronic acid (4.0 mmol, 1.0 equiv), 1,2-diphenyldiselenane (4.0 mmol, 1.0 equiv), KBr (16 mmol), TFA (8 mmol), CH₃CN (5 mL) and H₂O (2 mL) were placed in a 10 mL undivided electrolytic cell. The electrolysis was carried out at room temperature under a constant current of 5 mA for 24 hours. The resulting solution was quenched with 10 mL brine and extracted with 3×10 mL ethyl acetate. The extract was dried with Na₂SO₄. The solvent was removed with a rotary

evaporator. The pure product was obtained by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 10: 1). **4a**, 1.1 g, 71% yield.

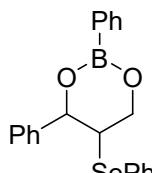
Synthesis of product **6** according to the following procedure:

As exemplified for **6a**:

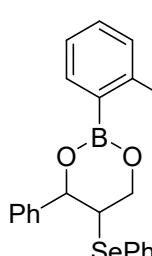
(*E*)-3-phenylprop-2-en-1-ol (0.3 mmol, 1.0 equiv), phenylboronic acid (0.3 mmol, 1.0 equiv), 1,2-di-*p*-tolyldisulfane (0.3 mmol, 1.0 equiv), KBr (1.2 mmol), AcOH (0.6 mmol), CH₃CN (4 mL) and H₂O (1 mL) were placed in a 10 mL undivided electrolytic cell with a vitreous carbon plate anode (52 mm×8 mm×2 mm) and a platinum plate cathode (52 mm×8 mm×2 mm). The electrolysis was carried out at room temperature under a constant current of 10 mA for 12 hours. When the reaction was finished, the resulting solution was quenched with 10 mL brine and extracted with 3×10 mL ethyl acetate. The extract was dried with Na₂SO₄. The solvent was removed with a rotary evaporator. The pure product was obtained by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 10: 1).

2.2 Characterization data

2,4-Diphenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (**4a**)

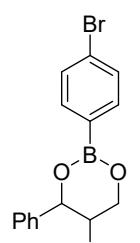
 Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **4a**. Brown oil (96.9 mg, 82%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.83 (d, *J* = 6.4 Hz, 2H), 7.46 – 7.36 (m, 3H), 7.39 – 7.27 (m, 7H), 7.27 (d, *J* = 7.4 Hz, 1H), 7.22 (d, *J* = 7.3 Hz, 2H), 5.15 (d, *J* = 7.8 Hz, 1H), 4.29 (dd, *J* = 11.8, 4.1 Hz, 1H), 4.10 (dd, *J* = 11.8, 8.6 Hz, 1H), 3.53 (td, *J* = 8.2, 4.1 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.6, 135.6, 134.1, 131.1, 129.3, 128.6, 128.5, 128.4, 127.8, 127.0, 126.7, 77.4, 64.6, 46.2. HR-QTOFMS (m/z): calcd for C₂₁H₁₉BO₂Se [M]: 394.0643, found: 394.0647.

4-Phenyl-5-(phenylselanyl)-2-(*o*-tolyl)-1,3,2-dioxaborinane (**4b**)

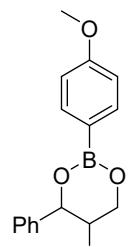
 Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **4b**. Yellow oil (91.8 mg, 75%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.81 (d, *J* = 6.6 Hz, 1H), 7.41 (d, *J* = 7.3 Hz, 2H), 7.38 – 7.24 (m, 7H), 7.26 – 7.18 (m, 1H), 7.18 – 7.12 (m, 2H), 5.16 (d, *J* = 7.4 Hz, 1H), 4.28 (dd, *J* = 11.9, 3.9 Hz, 1H), 4.09 (dd, *J* = 11.8, 8.2 Hz, 1H), 3.55 (td, *J* = 7.8, 4.0 Hz, 1H), 2.52 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 144.6, 140.7, 135.7, 135.4,

130.5, 130.2, 129.3, 128.5, 128.3, 127.0, 126.6, 124.8, 77.3, 64.2, 46.1, 22.8. HRMS GC/QTOF (m/z): calcd for $C_{22}H_{21}BO_2Se$ [M]: 408.0800, found: 408.0809.

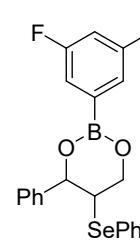
2-(4-Bromophenyl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4c)

 Flash column chromatography on silica gel (eluent: PE/EtOAc = 8/1, v/v) to afford **4c**. Brown solid (96.3 mg, 68%), mp 55.7–58.5 °C. 1H NMR (400 MHz, $CDCl_3$) δ ppm 7.67 (d, J = 8.2 Hz, 1H), 7.47 (d, J = 8.2 Hz, 2H), 7.42 – 7.28 (m, 7H), 7.26 (d, J = 7.3 Hz, 1H), 7.24 – 7.15 (m, 2H), 5.11 (d, J = 7.8 Hz, 1H), 4.27 (dd, J = 11.8, 4.2 Hz, 1H), 4.08 (dd, J = 11.8, 8.6 Hz, 1H), 3.51 (td, J = 8.3, 4.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 140.3, 135.8, 135.7, 130.9, 129.3, 128.6, 128.5, 126.8, 126.7, 126.0, 77.4, 64.6, 45.9. HRMS GC/QTOF (m/z): calcd for $C_{21}H_{18}BBrO_2Se$ [M]: 471.9748, found: 471.9756.

2-(4-Methoxyphenyl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4d)

 Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **4d**. Brown solid (96.7 mg, 76%), mp 84.2–85.0 °C. 1H NMR (400 MHz, $CDCl_3$) δ ppm 7.78 (d, J = 6.7 Hz, 2H), 7.41 – 7.31 (m, 7H), 7.25 (d, J = 7.5 Hz, 1H), 7.19 (t, J = 7.5 Hz, 2H), 6.87 (d, J = 7.8 Hz, 2H), 5.11 (d, J = 7.8 Hz, 1H), 4.26 (dd, J = 11.8, 4.0 Hz, 1H), 4.07 (dd, J = 11.8, 8.5 Hz, 1H), 3.78 (s, 3H), 3.50 (td, J = 8.1, 4.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 162.1, 140.7, 135.9, 135.6, 129.2, 128.5, 128.4, 128.3, 127.0, 126.7, 113.3, 77.3, 64.5, 55.1, 46.3. HRMS GC/QTOF (m/z): calcd for $C_{22}H_{21}BO_3Se$ [M]: 424.0749, found: 424.0750.

2-(3,5-Difluorophenyl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4e)

 Flash column chromatography on silica gel (eluent: PE/EtOAc = 8/1, v/v) to afford **4e**. Yellow solid (67.1 mg, 52%), mp 55.1–55.9 °C. 1H NMR (400 MHz, $CDCl_3$) δ ppm 7.39 – 7.27 (m, 9H), 7.27 – 7.14 (m, 3H), 6.83 (t, J = 9.0 Hz, 1H), 5.11 (d, J = 7.7 Hz, 1H), 4.27 (dd, J = 11.9, 4.1 Hz, 1H), 4.06 (dd, J = 11.9, 8.5 Hz, 1H), 3.50 (td, J = 8.1, 4.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 164.1 (d, J = 11.0 Hz), 161.6 (d, J = 11.0 Hz), 140.1, 135.7, 129.3, 128.6, 128.5, 126.7, 126.6, 116.4 – 116.0 (m), 106.3 (t, J = 25.2 Hz), 77.5, 64.6, 45.7. HRMS GC/QTOF (m/z): calcd for $C_{21}H_{17}BF_2O_2Se$ [M]: 430.0455, found: 430.0453.

2-(4-Chloro-3-methylphenyl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4f)

Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **4f**. Yellow oil (103.4 mg, 78%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.72 (d, *J* = 7.8 Hz, 1H), 7.44 – 7.30 (m, 7H), 7.27 (d, *J* = 7.2 Hz, 1H), 7.22 (d, *J* = 7.7 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 5.14 (d, *J* = 7.5 Hz, 1H), 4.27 (dd, *J* = 11.9, 4.0 Hz, 1H), 4.08 (dd, *J* = 11.9, 8.3 Hz, 1H), 3.54 (td, *J* = 7.9, 4.0 Hz, 1H), 2.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 146.7, 140.5, 136.8, 136.4, 135.7, 131.6, 130.1, 129.3, 128.6, 128.4, 126.9, 126.6, 125.0, 77.4, 64.3, 45.9, 22.6. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₀BClO₂Se [M]: 442.0410, found: 442.0410.

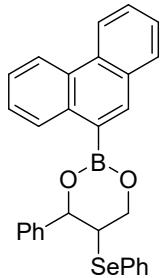
2-(3-Chloro-4-methylphenyl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4g)

Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **4g**. Yellow solid (92.8 mg, 70%), mp 88.7–89.5 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.78 (s, 1H), 7.59 (d, *J* = 7.5 Hz, 1H), 7.42 – 7.30 (m, 7H), 7.27 (d, *J* = 6.2 Hz, 1H), 7.20 (t, *J* = 7.3 Hz, 3H), 5.12 (d, *J* = 7.7 Hz, 1H), 4.27 (dd, *J* = 11.9, 4.0 Hz, 1H), 4.07 (dd, *J* = 11.8, 8.5 Hz, 1H), 3.52 (td, *J* = 8.1, 4.1 Hz, 1H), 2.37 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.4, 139.0, 135.7, 134.6, 134.3, 132.3, 130.6, 129.3, 128.6, 128.6, 128.4, 126.9, 126.7, 126.7, 77.4, 64.5, 46.0, 20.4. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₀BClO₂Se [M]: 442.0410, found: 442.0422.

2-(Naphthalen-2-yl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4h)

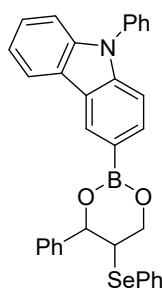
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **4h**. Yellow solid (86.2 mg, 65%), mp 79.3–80.2 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 8.80 (d, *J* = 7.8 Hz, 1H), 8.11 (d, *J* = 7.0 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.80 (d, *J* = 9.6 Hz, 1H), 7.49 – 7.29 (m, 10H), 7.25 (t, *J* = 7.3 Hz, 1H), 7.18 (t, *J* = 7.3 Hz, 2H), 5.21 (d, *J* = 7.5 Hz, 1H), 4.36 (dd, *J* = 11.9, 4.0 Hz, 1H), 4.16 (dd, *J* = 11.8, 8.3 Hz, 1H), 3.60 (td, *J* = 8.0, 4.1 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.6, 136.9, 135.7, 135.1, 133.5, 131.4, 129.3, 128.6, 128.6, 128.5, 128.4, 128.4, 127.0, 126.7, 126.2, 125.4, 125.1, 77.5, 64.4, 46.0. HRMS GC/QTOF (m/z): calcd for C₂₅H₂₁BO₂Se [M]: 444.0800, found: 444.0813.

2-(Phenanthren-9-yl)-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4i)



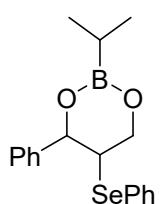
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **4i**. Brown solid (108.2 mg, 73%), mp 76.5–77.2 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 8.86 (d, *J* = 8.0 Hz, 1H), 8.66 (dd, *J* = 14.4, 8.2 Hz, 2H), 8.40 (s, 1H), 7.89 (d, *J* = 7.9 Hz, 1H), 7.68 – 7.51 (m, 4H), 7.47 – 7.31 (m, 7H), 7.27 (d, *J* = 7.1 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 2H), 5.27 (d, *J* = 7.4 Hz, 1H), 4.41 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.21 (dd, *J* = 11.9, 8.2 Hz, 1H), 3.65 (td, *J* = 7.8, 4.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.6, 137.4, 135.7, 134.6, 131.9, 131.2, 130.2, 129.4, 129.3, 129.2, 128.7, 128.6, 128.4, 127.7, 127.0, 126.7, 126.6, 126.5, 126.1, 122.8, 122.5, 77.6, 64.5, 46.0. HRMS GC/QTOF (m/z): calcd for C₂₉H₂₃BO₂Se [M]: 494.0956, found: 494.0955.

9-Phenyl-3-(4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinan-2-yl)-9H-carbazole (4j)



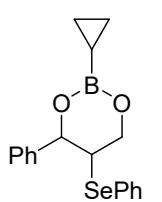
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **4j**. Brown solid (115.7 mg, 69%), mp 63.4.5–64.1 °C.. ¹H NMR (400 MHz, CDCl₃) δ ppm 8.68 (s, 1H), 8.13 (d, *J* = 7.7 Hz, 1H), 7.89 (d, *J* = 7.2 Hz, 1H), 7.58 – 7.47 (m, 4H), 7.44 – 7.28 (m, 11H), 7.29 – 7.21 (m, 2H), 7.22 – 7.13 (m, 2H), 5.17 (d, *J* = 7.8 Hz, 1H), 4.33 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.13 (dd, *J* = 11.8, 8.6 Hz, 1H), 3.55 (td, *J* = 8.2, 4.1 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.4, 138.6, 136.4, 136.4, 133.9, 130.0, 128.8, 128.6, 128.4, 126.6, 114.9, 114.7, 76.6, 51.5, 21.3. HRMS GC/QTOF (m/z): calcd for C₃₃H₂₆BNO₂Se [M]: 559.1222, found: 559.1222.

2-Isopropyl-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4k)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **4k**. Brown oil (73.7 mg, 65%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.36 (dt, *J* = 13.0, 6.5 Hz, 5H), 7.29 – 7.26 (m, 3H), 7.24 – 7.16 (m, 2H), 4.97 (d, *J* = 7.6 Hz, 1H), 4.11 (dd, *J* = 11.8, 4.1 Hz, 1H), 3.92 (dd, *J* = 11.8, 8.3 Hz, 1H), 3.43 (td, *J* = 8.0, 4.0 Hz, 1H), 1.52 – 1.38 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H), 0.80 (t, *J* = 7.7 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.6, 135.7, 129.3, 128.5, 128.3, 127.0, 126.6, 77.0, 64.0, 46.2, 17.6, 17.2. HR-ESI-MS (m/z): calcd for C₁₈H₂₂BO₂Se [M+H]⁺: 361.0878, found: 361.0875.

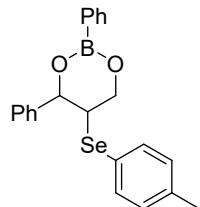
2-Cyclopropyl-4-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (4l)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **4l**. Brown oil (77.3 mg, 72%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.38 – 7.30 (m, 5H), 7.30 – 7.25 (m,

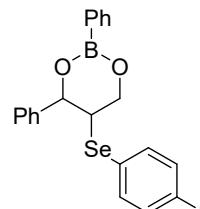
3H), 7.20 (dd, $J = 8.3, 6.8$ Hz, 2H), 4.92 (d, $J = 7.8$ Hz, 1H), 4.07 (dd, $J = 11.7, 4.1$ Hz, 1H), 3.89 (dd, $J = 11.7, 8.5$ Hz, 1H), 3.40 (td, $J = 8.1, 4.0$ Hz, 1H), 0.57 (dd, $J = 6.3, 2.9$ Hz, 2H), 0.50 (d, $J = 6.1$ Hz, 2H), -0.14 -- 0.32 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.7, 135.6, 129.2, 128.5, 128.4, 128.3, 127.0, 126.6, 76.9, 64.2, 46.2, 3.9, 3.9. HR-ESI-MS (m/z): calcd for $\text{C}_{18}\text{H}_{19}\text{BO}_2\text{NaSe} [\text{M}+\text{Na}]^+$: 381.0541, found: 381.0543.

2,4-Diphenyl-5-(*p*-tolylselanyl)-1,3,2-dioxaborinane (**5a**)



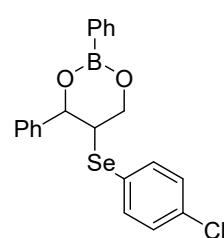
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **5a**. Brown solid (99.9 mg, 80%), mp 80.1-80.7 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.82 (d, $J = 6.9$ Hz, 2H), 7.43 (d, $J = 7.5$ Hz, 1H), 7.42 – 7.30 (m, 7H), 7.30 (d, $J = 7.9$ Hz, 2H), 7.03 (d, $J = 7.9$ Hz, 2H), 5.13 (d, $J = 7.8$ Hz, 1H), 4.27 (dd, $J = 11.8, 4.1$ Hz, 1H), 4.08 (dd, $J = 11.8, 8.5$ Hz, 1H), 3.48 (td, $J = 8.2, 4.1$ Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.7, 138.8, 136.0, 134.1, 131.1, 130.1, 128.5, 128.3, 127.7, 126.8, 123.1, 77.3, 64.6, 46.0, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{21}\text{BO}_2\text{Se} [\text{M}]$: 408.0800, found: 408.0804.

5-((4-Fluorophenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5b**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5b**. White solid (75.4 mg, 61%), mp 68.7-69.3 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.83 (d, $J = 7.2$ Hz, 2H), 7.46 – 7.41 (m, 1H), 7.34 (q, $J = 5.8, 5.1$ Hz, 9H), 6.89 (t, $J = 8.5$ Hz, 1H), 5.12 (d, $J = 7.8$ Hz, 1H), 4.28 (dd, $J = 11.9, 4.1$ Hz, 1H), 4.07 (dd, $J = 11.9, 8.7$ Hz, 1H), 3.46 (td, $J = 8.2, 4.1$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 163.2 (d, $J = 249.1$ Hz), 140.5, 138.1 (d, $J = 8.1$ Hz), 134.1, 131.1, 128.6, 128.4, 127.8, 126.8, 121.6 (d, $J = 3.5$ Hz), 116.5 (d, $J = 21.5$ Hz), 77.4, 64.4, 46.7. HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BFO}_2\text{Se} [\text{M}]$: 412.0549, found: 412.0549.

5-((4-Chlorophenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5c**)

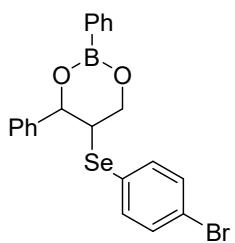


Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5c**. Yellow solid (83.5mg, 65%), mp 90.5-91.2 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.83 (d, $J = 7.4$ Hz, 2H), 7.44 (d, $J = 7.5$ Hz, 1H), 7.40 – 7.31 (m, 7H), 7.27 (d, $J = 8.0$ Hz, 2H), 7.17 (d, $J = 8.0$ Hz, 2H), 5.14 (d, $J = 7.7$ Hz, 1H), 4.29 (dd, $J = 11.9, 4.1$ Hz, 1H), 4.08 (dd, $J = 11.9, 8.6$ Hz, 1H), 3.50 (td, $J = 8.2, 4.1$ Hz, 1H). ^{13}C NMR (100 MHz,

CDCl_3) δ ppm 140.4, 137.0, 135.0, 134.1, 131.2, 129.5, 128.6, 128.5, 127.8, 126.7, 125.2, 77.4, 64.4, 46.7.

HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BClO}_2\text{Se}$ [M]: 428.0254, found: 428.0251.

5-((4-Bromophenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5d**)

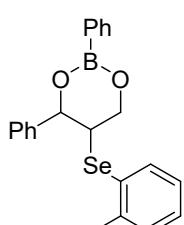


Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5d**.

Yellow solid (96.3 mg, 68%), mp 88.3–89.1 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.82 (d, J = 7.5 Hz, 2H), 7.44 (d, J = 6.9 Hz, 1H), 7.36 (t, J = 7.4 Hz, 9H), 7.32 (d, J = 1.9 Hz, 1H), 7.21 (d, J = 7.2 Hz, 1H), 5.15 (d, J = 7.7 Hz, 1H), 4.30 (dd, J = 12.0, 3.7 Hz, 1H), 4.10 (dd, J = 11.5, 9.1 Hz, 1H), 3.52 (td, J = 8.3, 4.2 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.4, 137.1, 134.1, 132.4, 131.2, 128.6, 128.5, 127.8, 126.7, 125.9, 123.2, 77.4, 64.4, 46.7.

HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BBrO}_2\text{Se}$ [M]: 471.9748, found: 471.9750.

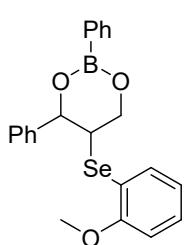
2,4-Diphenyl-5-(*o*-tolylselanyl)-1,3,2-dioxaborinane (**5e**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **5e**.

Yellow solid (91.8 mg, 75%), mp 78.9–79.5 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.84 (d, J = 6.6 Hz, 2H), 7.49 – 7.40 (m, 1H), 7.41 – 7.29 (m, 8H), 7.18 (d, J = 4.4 Hz, 2H), 7.02 (dt, J = 8.4, 4.3 Hz, 1H), 5.16 (d, J = 7.3 Hz, 1H), 4.23 (dd, J = 11.9, 3.9 Hz, 1H), 4.10 (dd, J = 11.9, 8.0 Hz, 1H), 3.55 (td, J = 7.7, 4.0 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 141.8, 140.6, 136.0, 134.1, 131.1, 130.3, 128.8, 128.6, 128.4, 128.4, 127.8, 126.7, 126.6, 77.2, 64.3, 45.8, 23.1. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{21}\text{BO}_2\text{Se}$ [M]: 408.0800, found: 408.0799.

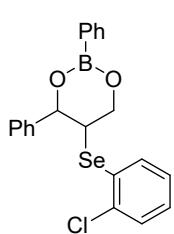
5-((2-Methoxyphenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5f**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **5f**.

Yellow solid (105.6 mg, 83%), mp 48.1–48.9 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.85 (d, J = 7.1 Hz, 2H), 7.42 (d, J = 7.4 Hz, 1H), 7.40 – 7.28 (m, 8H), 7.26 – 7.14 (m, 1H), 6.89 – 6.74 (m, 2H), 5.18 (d, J = 7.5 Hz, 1H), 4.25 (dd, J = 11.9, 4.0 Hz, 1H), 4.08 (dd, J = 11.8, 8.2 Hz, 1H), 3.80 (s, 3H), 3.74 (dt, J = 7.7, 4.0 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 159.1, 140.8, 135.9, 134.1, 131.0, 130.0, 128.4, 128.2, 127.7, 126.5, 121.3, 110.8, 77.3, 64.5, 55.9, 43.6. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{21}\text{BO}_3\text{Se}$ [M]: 424.0749, found: 424.0745.

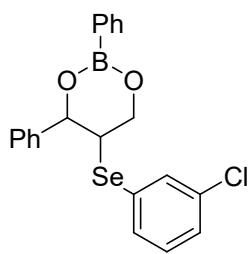
5-((2-Chlorophenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5g**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5g**.

Yellow solid (100.2 mg, 78%), mp 96.7–97.3 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.85 (d, *J* = 6.5 Hz, 2H), 7.49 – 7.40 (m, 1H), 7.40 – 7.26 (m, 9H), 7.22 – 7.13 (m, 1H), 7.09 – 6.99 (m, 1H), 5.21 (d, *J* = 7.2 Hz, 1H), 4.30 (dd, *J* = 11.9, 3.9 Hz, 1H), 4.11 (dd, *J* = 11.9, 8.0 Hz, 1H), 3.79 (td, *J* = 7.6, 4.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.4, 138.2, 135.9, 134.2, 131.2, 129.9, 129.6, 128.6, 128.4, 127.9, 127.8, 127.3, 126.5, 77.2, 64.0, 45.4. HRMS GC/QTOF (m/z): calcd for C₂₁H₁₈BClO₂Se [M]: 428.0254, found: 428.0263.

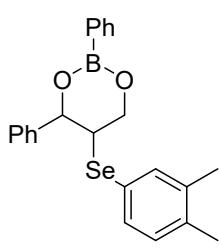
5-((3-Chlorophenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5h**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford

5h. Yellow solid (88.6 mg, 69%), mp 75.6–76.4 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.83 (d, *J* = 6.6 Hz, 2H), 7.44 (d, *J* = 7.5 Hz, 1H), 7.40 – 7.31 (m, 7H), 7.29 – 7.26 (m, 1H), 7.27 – 7.19 (m, 3H), 7.15 – 7.09 (m, 1H), 5.16 (d, *J* = 7.9 Hz, 1H), 4.33 (dd, *J* = 11.9, 4.2 Hz, 1H), 4.11 (dd, *J* = 11.8, 8.8 Hz, 1H), 3.55 (td, *J* = 4.6, 3.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.3, 134.9, 134.7, 134.2, 133.3, 131.2, 130.2, 128.7, 128.7, 128.6, 128.6, 127.8, 126.8, 77.5, 64.5, 46.8. HRMS GC/QTOF (m/z): calcd for C₂₁H₁₈BClO₂Se [M]: 428.0254, found: 428.0247.

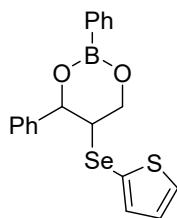
5-((3,4-Dimethylphenyl)selanyl)-2,4-diphenyl-1,3,2-dioxaborinane (**5i**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 14/1, v/v) to afford **5i**.

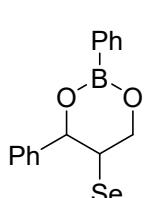
Yellow solid (101.3 mg, 80%), mp 51.4–52.2 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.82 (s, 2H), 7.44 – 7.29 (m, 8H), 7.19 – 7.08 (m, 2H), 6.96 (d, *J* = 7.8 Hz, 1H), 5.12 (d, *J* = 7.7 Hz, 1H), 4.28 (dd, *J* = 11.7, 4.3 Hz, 1H), 4.08 (dd, *J* = 11.6, 8.7 Hz, 1H), 3.44 (td, *J* = 8.4, 4.3 Hz, 1H), 2.19 (s, 3H), 2.15 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.8, 137.8, 137.4, 137.0, 134.1, 133.4, 131.0, 130.5, 128.5, 128.3, 127.7, 126.9, 77.4, 64.7, 46.0, 19.7, 19.6. HRMS GC/QTOF (m/z): calcd for C₂₃H₂₃BO₂Se [M]: 422.0956, found: 422.0956.

2,4-Diphenyl-5-(thiophen-2-ylselanyl)-1,3,2-dioxaborinane (**5j**)



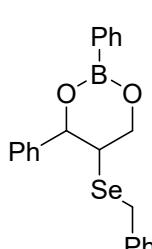
Flash column chromatography on silica gel (eluent: PE/EtOAc = 4/1, v/v) to afford **5j**. Brown oil (72.0 mg, 60%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.83 (d, J = 6.8 Hz, 2H), 7.50 – 7.27 (m, 9H), 7.16 – 6.83 (m, 2H), 5.15 (d, J = 7.6 Hz, 1H), 4.29 (dd, J = 11.9, 4.1 Hz, 1H), 4.14 – 4.01 (m, 1H), 3.41 (tt, J = 7.9, 3.9 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 138.2, 134.1, 132.5, 131.1, 128.7, 128.6, 128.4, 128.4, 127.8, 127.7, 126.7, 126.6, 76.7, 64.0, 46.8. HRMS GC/QTOF (m/z): calcd for $\text{C}_{19}\text{H}_{17}\text{BO}_2\text{SSe}$ [M]: 400.0208, found: 400.0211.

5-(Methylselanyl)-2,4-diphenyl-1,3,2-dioxaborinane (5k)



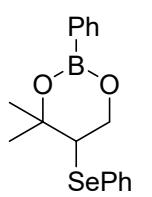
Flash column chromatography on silica gel (eluent: PE/EtOAc = 4/1, v/v) to afford **5k**. Brown oil (65.7 mg, 66%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.84 (d, J = 6.4 Hz, 2H), 7.47 – 7.37 (m, 5H), 7.41 – 7.32 (m, 4H), 5.07 (d, J = 9.0 Hz, 1H), 4.37 (dd, J = 11.6, 4.5 Hz, 1H), 4.05 (dd, J = 11.6, 10.2 Hz, 1H), 3.08 (td, J = 9.6, 4.5 Hz, 1H), 1.66 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.7, 134.1, 131.1, 128.5, 128.4, 127.7, 126.9, 78.4, 65.0, 42.9, 4.1. HRMS GC/QTOF (m/z): calcd for $\text{C}_{16}\text{H}_{17}\text{BO}_2\text{Se}$ [M]: 332.0487, found: 332.0485.

5-(Benzylselanyl)-2,4-diphenyl-1,3,2-dioxaborinane (5l)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 4/1, v/v) to afford **5l**. Yellow solid (66.1 mg, 54%), mp 68.4–69.3°C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.81 (d, J = 6.6 Hz, 2H), 7.41 (d, J = 4.6 Hz, 5H), 7.38 – 7.29 (m, 3H), 7.20 (dd, J = 13.1, 7.2 Hz, 3H), 7.05 (d, J = 6.5 Hz, 2H), 5.10 (d, J = 9.1 Hz, 1H), 4.21 (dd, J = 11.6, 4.6 Hz, 1H), 4.00 (dd, J = 11.6, 10.4 Hz, 1H), 3.34 (d, J = 11.7 Hz, 1H), 3.24 (d, J = 11.8 Hz, 1H), 3.06 – 2.95 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.7, 138.1, 134.1, 131.1, 129.0, 128.7, 128.7, 128.5, 128.4, 127.8, 127.2, 78.7, 65.2, 41.9, 27.8. HRMS GC/QTOF (m/z): calcd for $\text{C}_{23}\text{H}_{25}\text{BO}_2\text{Se}$ [M]: 408.0800, found: 408.0796.

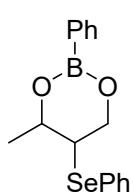
4,4-Dimethyl-2-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (5m)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **5m**. Brown oil (73.7 mg, 71%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.75 (d, J = 6.5 Hz, 2H), 7.64 – 7.54 (m, 2H), 7.44 – 7.35 (m, 1H), 7.36 – 7.24 (m, 5H), 4.23 (d, J = 8.4 Hz, 2H), 3.37 (t, J = 8.4 Hz, 1H), 1.64 (s, 3H), 1.41 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 134.7, 133.9, 130.8, 129.5, 128.7,

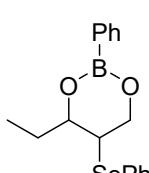
128.3, 127.6, 74.3, 63.9, 51.0, 29.9, 25.3. HRMS GC/QTOF (m/z): calcd for $C_{17}H_{19}BO_2Se$ [M]: 346.0643, found: 346.0645.

4-Methyl-2-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (5n)



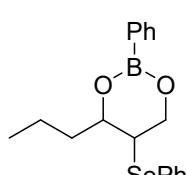
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5n**. Brown oil (59.8 mg, 60%). 1H NMR (400 MHz, $CDCl_3$) δ ppm 7.80 (d, J = 6.5 Hz, 2H), 7.65 – 7.57 (m, 2H), 7.47 – 7.38 (m, 1H), 7.39 – 7.26 (m, 5H), 4.27 (dd, J = 6.1, 3.7 Hz, 1H), 4.17 (dd, J = 11.8, 6.5 Hz, 1H), 3.70 (dt, J = 7.0, 3.7 Hz, 1H), 1.81 – 1.74 (m, 1H), 1.12 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 135.3, 134.0, 130.9, 129.4, 128.3, 127.7, 75.3, 64.8, 45.7, 27.7, 10.3. HRMS GC/QTOF (m/z): calcd for $C_{16}H_{17}BO_2Se$ [M]: 332.0487, found: 332.0487.

4-Ethyl-2-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (5o)



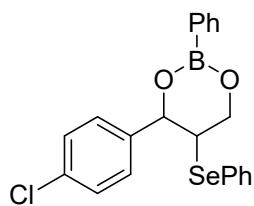
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **5o**. Brown oil (77.9 mg, 75%). 1H NMR (400 MHz, $CDCl_3$) δ ppm 7.80 (d, J = 6.5 Hz, 2H), 7.60 (d, J = 7.7 Hz, 2H), 7.47 – 7.38 (m, 1H), 7.39 – 7.31 (m, 2H), 7.34 – 7.24 (m, 3H), 4.35 – 4.19 (m, 2H), 4.17 (dd, J = 11.8, 6.5 Hz, 1H), 3.74 – 3.66 (m, 1H), 1.85 – 1.68 (m, 2H), 1.12 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 135.3, 134.0, 130.9, 129.4, 128.3, 128.0, 127.7, 75.3, 64.8, 45.7, 27.7, 10.3. HRMS GC/QTOF (m/z): calcd for $C_{17}H_{19}BO_2Se$ [M]: 346.0643, found: 346.0642.

2-Phenyl-5-(phenylselanyl)-4-propyl-1,3,2-dioxaborinane (5p)



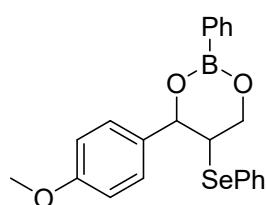
Flash column chromatography on silica gel (eluent: PE/EtOAc = 13/1, v/v) to afford **5p**. Brown oil (84.3 mg, 78%). 1H NMR (400 MHz, $CDCl_3$) δ ppm 7.76 (d, J = 7.8 Hz, 2H), 7.60 (d, J = 7.8 Hz, 2H), 7.45 – 7.36 (m, 1H), 7.39 – 7.25 (m, 5H), 4.30 (dd, J = 11.6, 4.5 Hz, 1H), 4.09 – 3.96 (m, 2H), 3.24 (td, J = 9.8, 4.5 Hz, 1H), 2.16 – 2.02 (m, 1H), 1.74 – 1.60 (m, 2H), 1.59 – 1.45 (m, 1H), 0.99 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 135.9, 133.9, 130.9, 129.4, 128.7, 127.7, 126.3, 74.6, 65.5, 43.4, 37.5, 18.3, 14.1. HRMS GC/QTOF (m/z): calcd for $C_{18}H_{21}BO_2Se$ [M]: 360.0800, found: 360.0801.

4-(4-Chlorophenyl)-2-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (5q)



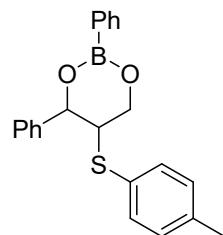
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5q**. Brown oil (97.6 mg, 76%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.80 (d, *J* = 7.6 Hz, 2H), 7.42 (d, *J* = 7.4 Hz, 1H), 7.38 – 7.31 (m, 5H), 7.28 (d, *J* = 8.1 Hz, 4H), 7.24 – 7.15 (m, 2H), 5.07 (d, *J* = 8.5 Hz, 1H), 4.30 (dd, *J* = 11.8, 4.3 Hz, 1H), 4.09 (dd, *J* = 11.8, 9.4 Hz, 1H), 3.43 (td, *J* = 9.0, 4.3 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 139.0, 135.6, 134.1, 134.1, 131.2, 129.3, 128.6, 128.6, 128.3, 127.8, 126.7, 77.0, 64.8, 46.0. HRMS GC/QTOF (m/z): calcd for C₂₁H₁₈BClO₂Se [M]: 428.0254, found: 428.0247.

4-(4-Methoxyphenyl)-2-phenyl-5-(phenylselanyl)-1,3,2-dioxaborinane (**5r**)



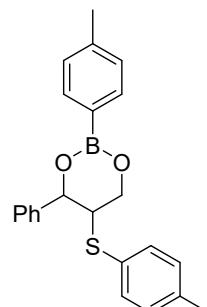
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **5r**. Brown oil (81.4 mg, 64%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.73 (d, *J* = 6.5 Hz, 2H), 7.33 (d, *J* = 6.9 Hz, 2H), 7.27 (d, *J* = 7.3 Hz, 2H), 7.22 – 7.18 (m, 3H), 7.18 – 7.09 (m, 3H), 6.81 (d, *J* = 8.6 Hz, 2H), 5.01 (d, *J* = 8.2 Hz, 1H), 4.22 (dd, *J* = 11.7, 4.2 Hz, 1H), 4.03 (dd, *J* = 11.7, 9.1 Hz, 1H), 3.73 (s, 3H), 3.44 (td, *J* = 8.7, 4.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 159.6, 135.7, 134.1, 132.8, 131.1, 129.3, 128.5, 128.0, 127.7, 126.9, 113.9, 77.1, 64.9, 55.4, 46.2. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₁BO₃Se [M]: 424.0749, found: 424.0750.

2,4-Diphenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (**6a**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6a**. Yellow solid (89.7 mg, 83%), mp 66.3–66.9 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.86 (d, *J* = 7.8 Hz, 2H), 7.43 (d, *J* = 7.9 Hz, 1H), 7.40 – 7.26 (m, 7H), 7.18 (d, *J* = 7.8 Hz, 2H), 7.05 (d, *J* = 7.8 Hz, 2H), 5.08 (d, *J* = 6.8 Hz, 1H), 4.22 (dd, *J* = 11.8, 3.9 Hz, 1H), 4.03 (dd, *J* = 11.8, 7.5 Hz, 1H), 3.40 (td, *J* = 7.2, 3.9 Hz, 1H), 2.30 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.5, 138.5, 134.2, 133.9, 131.1, 130.0, 128.9, 128.6, 128.3, 127.8, 126.6, 76.6, 63.5, 51.5, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₁BO₂S [M]: 360.1355, found: 360.1351.

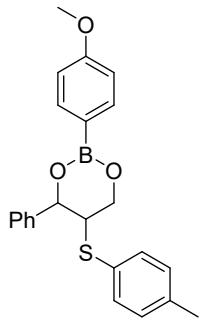
4-Phenyl-2-(*p*-tolyl)-5-(*p*-tolylthio)-1,3,2-dioxaborinane (**6b**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6b**. Yellow solid (88.7 mg, 79%), mp 70.1–70.8 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.75 (d, *J* = 7.9 Hz, 2H), 7.40 – 7.28 (m, 5H), 7.18 (d, *J* = 7.9 Hz, 4H), 7.05 (d, *J* = 7.8 Hz, 2H),

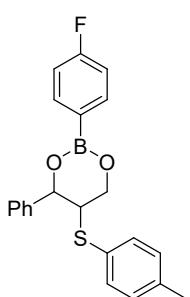
5.08 (d, $J = 6.9$ Hz, 1H), 4.22 (dd, $J = 11.7, 3.9$ Hz, 1H), 4.02 (dd, $J = 11.8, 7.5$ Hz, 1H), 3.39 (td, $J = 7.3, 4.0$ Hz, 1H), 2.37 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 141.2, 140.6, 138.5, 134.3, 133.9, 130.0, 129.0, 128.6, 128.6, 128.3, 126.6, 76.6, 63.5, 51.6, 21.9, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{23}\text{H}_{23}\text{BO}_2\text{S}$ [M]: 374.1512, found: 374.1511.

2-(4-Methoxyphenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (**6c**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6c**. Yellow solid (89.0 mg, 76%), mp 69.6–70.4 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.80 (d, $J = 8.5$ Hz, 2H), 7.39 – 7.30 (m, 5H), 7.18 (d, $J = 8.2$ Hz, 2H), 7.05 (d, $J = 8.2$ Hz, 2H), 6.89 (d, $J = 8.5$ Hz, 2H), 5.06 (d, $J = 6.9$ Hz, 1H), 4.21 (dd, $J = 11.7, 3.9$ Hz, 1H), 4.01 (dd, $J = 11.7, 7.5$ Hz, 1H), 3.81 (s, 3H), 3.39 (td, $J = 7.3, 4.0$ Hz, 1H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 162.1, 140.7, 138.4, 135.9, 133.8, 129.9, 128.9, 128.5, 128.3, 126.6, 113.3, 76.5, 63.51, 55.2, 51.6, 21.2. HRMS GC/QTOF (m/z): calcd for $\text{C}_{23}\text{H}_{23}\text{BO}_3\text{S}$ [M]: 390.1461, found: 390.1458.

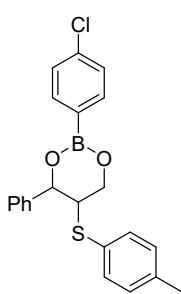
2-(4-Fluorophenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (**6d**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6d**. Brown oil (74.9 mg, 66%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.91 – 7.77 (m, 2H), 7.40 – 7.32 (m, 5H), 7.19 (d, $J = 7.8$ Hz, 2H), 7.10 – 6.99 (m, 4H), 5.08 (d, $J = 6.8$ Hz, 1H), 4.22 (dd, $J = 11.8, 4.0$ Hz, 1H), 4.02 (dd, $J = 11.8, 7.5$ Hz, 1H), 3.40 (td, $J = 7.2, 3.9$ Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.4, 138.6, 136.4 (d, $J = 8.1$ Hz), 133.9, 130.0, 128.71 (d, $J = 20.9$ Hz), 128.4, 126.6, 114.8 (d, $J = 20.1$ Hz), 76.6, 63.5, 51.5, 21.3.

HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{20}\text{BFO}_2\text{S}$ [M]: 378.1261, found: 378.1258.

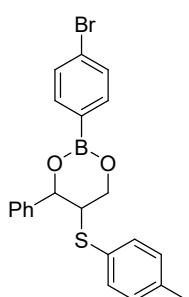
2-(4-Chlorophenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (**6e**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6e**. Brown oil (73.3 mg, 62%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.76 (d, $J = 8.2$ Hz, 2H), 7.40 – 7.29 (m, 7H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.05 (d, $J = 7.9$ Hz, 2H), 5.07 (d, $J = 6.9$ Hz, 1H), 4.22 (dd, $J = 11.8, 4.0$ Hz, 1H), 4.02 (dd, $J = 11.8, 7.5$ Hz, 1H), 3.40 (dt, $J = 7.3, 3.7$ Hz, 1H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.3, 138.6, 137.3, 135.6, 133.9,

130.0, 128.7, 128.6, 128.4, 128.0, 126.6, 76.6, 63.5, 51.4, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₀BClO₂S [M]: 394.0966, found: 394.0965.

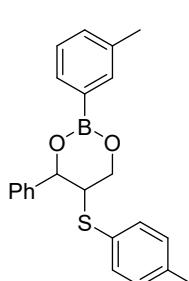
2-(4-Bromophenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6f)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6f**. Brown oil (98.6mg, 75%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.70 (d, *J* = 8.3 Hz, 2H), 7.49 (d, *J* = 8.3 Hz, 2H), 7.40 – 7.29 (m, 5H), 7.18 (d, *J* = 8.1 Hz, 2H), 7.06 (d, *J* = 7.8 Hz, 2H), 5.07 (d, *J* = 6.9 Hz, 1H), 4.22 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.02 (dd, *J* = 11.8, 7.5 Hz, 1H), 3.40 (td, *J* = 7.2, 4.0 Hz, 1H), 2.30 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.3, 138.6, 135.8, 133.9, 131.0, 130.0, 128.7, 128.6, 128.4, 126.6, 126.1, 76.7, 63.6, 51.4, 21.3.

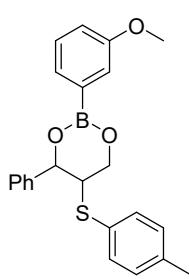
HRMS GC/QTOF (m/z): calcd for C₂₂H₂₀BBrO₂S [M]: 438.0460, found: 438.0466.

4-Phenyl-2-(*m*-tolyl)-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6g)



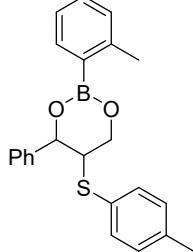
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6g**. Yellow solid (77.4 mg, 69%), mp 78.2–78.7 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.73 – 7.61 (m, 2H), 7.40 – 7.30 (m, 5H), 7.27 (d, *J* = 4.8 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 7.06 (d, *J* = 7.8 Hz, 2H), 5.09 (d, *J* = 6.7 Hz, 1H), 4.22 (dd, *J* = 11.8, 3.9 Hz, 1H), 4.03 (dd, *J* = 11.8, 7.3 Hz, 1H), 3.41 (td, *J* = 7.1, 3.9 Hz, 1H), 2.35 (s, 3H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.6, 138.5, 137.1, 134.8, 133.9, 131.9, 131.2, 130.0, 128.9, 128.6, 128.3, 127.7, 126.6, 76.5, 63.4, 51.6, 21.5, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₃H₂₃BO₂S [M]: 374.1512, found: 374.1510.

2-(3-Methoxyphenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6h)



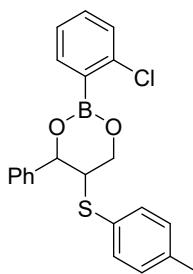
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6h**. Yellow solid (72.6 mg, 62%), mp 83.2–83.9 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.45 (d, *J* = 7.3 Hz, 1H), 7.41 – 7.31 (m, 6H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.20 (d, *J* = 8.1 Hz, 2H), 7.06 (d, *J* = 7.9 Hz, 2H), 7.04 – 6.96 (m, 1H), 5.09 (d, *J* = 6.8 Hz, 1H), 4.22 (dd, *J* = 11.8, 3.9 Hz, 1H), 4.03 (dd, *J* = 11.8, 7.4 Hz, 1H), 3.82 (s, 3H), 3.41 (td, *J* = 7.1, 4.0 Hz, 1H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 159.2, 140.5, 138.5, 133.9, 130.0, 129.0, 128.6, 128.3, 126.6, 126.6, 118.4, 117.5, 76.6, 63.5, 55.3, 51.5, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₃H₂₃BO₃S [M]: 390.1461, found: 390.1459.

4-Phenyl-2-(*o*-tolyl)-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6i)



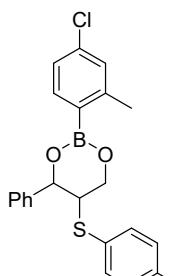
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6i**. Brown oil (87.6 mg, 78%). ^1H NMR (400 MHz, CDCl_3) δ ppm (d, $J = 6.7$ Hz, 1H), 7.41 – 7.26 (m, 6H), 7.22 (d, $J = 7.9$ Hz, 2H), 7.16 (d, $J = 7.4$ Hz, 2H), 7.07 (d, $J = 7.8$ Hz, 2H), 5.11 (d, $J = 6.5$ Hz, 1H), 4.22 (dd, $J = 11.8, 3.9$ Hz, 1H), 4.02 (dd, $J = 11.8, 7.1$ Hz, 1H), 3.43 (td, $J = 6.8, 3.9$ Hz, 1H), 2.54 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 144.6, 140.7, 138.5, 135.4, 134.0, 130.6, 130.2, 130.0, 129.0, 128.6, 128.2, 126.5, 124.9, 76.5, 63.2, 51.4, 22.8, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{23}\text{H}_{23}\text{BO}_2\text{S}$ [M]: 374.1512, found: 374.1520.

2-(2-Chlorophenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6j)



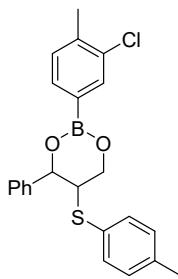
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6j**. Brown solid (70.9 mg, 60%), mp 72.3–72.8 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.73 (d, $J = 7.4$ Hz, 1H), 7.42 – 7.28 (m, 7H), 7.26 – 7.18 (m, 3H), 7.06 (d, $J = 7.9$ Hz, 2H), 5.11 (d, $J = 6.7$ Hz, 1H), 4.25 (dd, $J = 11.8, 3.9$ Hz, 1H), 4.04 (dd, $J = 11.8, 7.3$ Hz, 1H), 3.44 (td, $J = 7.0, 3.9$ Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.1, 139.0, 138.7, 136.0, 134.1, 131.5, 130.0, 129.7, 128.7, 128.6, 128.4, 126.6, 126.0, 76.9, 63.5, 51.3, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{20}\text{BClO}_2\text{S}$ [M]: 394.0966, found: 394.0966.

2-(4-Chloro-2-methylphenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6k)



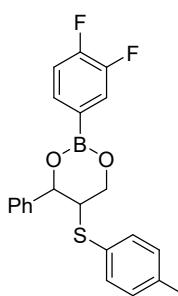
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6k**. Brown oil (80.8 mg, 66%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.74 (d, $J = 7.8$ Hz, 1H), 7.40 – 7.30 (m, 5H), 7.20 (d, $J = 7.9$ Hz, 2H), 7.13 (d, $J = 8.3$ Hz, 2H), 7.06 (d, $J = 7.9$ Hz, 2H), 5.09 (d, $J = 6.5$ Hz, 1H), 4.21 (dd, $J = 11.8, 3.9$ Hz, 1H), 4.01 (dd, $J = 11.8, 7.2$ Hz, 1H), 3.41 (td, $J = 7.0, 3.9$ Hz, 1H), 2.50 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 146.7, 140.4, 138.6, 136.9, 136.5, 134.0, 130.1, 130.0, 128.8, 128.6, 128.3, 126.5, 125.0, 76.6, 63.3, 51.3, 22.6, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{23}\text{H}_{22}\text{BClO}_2\text{S}$ [M]: 408.1122, found: 408.1123.

2-(3-Chloro-4-methylphenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6l)



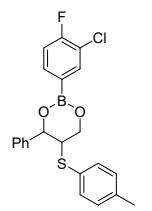
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6l**. Yellow solid (101.6 mg, 83%), mp 63.5–64.3 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.80 (s, 1H), 7.61 (d, *J* = 7.5 Hz, 1H), 7.41 – 7.30 (m, 5H), 7.25 – 7.16 (m, 3H), 7.06 (d, *J* = 7.8 Hz, 2H), 5.08 (d, *J* = 6.7 Hz, 1H), 4.21 (dd, *J* = 11.8, 3.9 Hz, 1H), 4.01 (dd, *J* = 11.8, 7.3 Hz, 1H), 3.40 (td, *J* = 7.1, 4.0 Hz, 1H), 2.39 (s, 3H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.3, 139.0, 138.6, 134.7, 134.3, 134.0, 132.3, 130.6, 130.0, 128.8, 128.6, 128.4, 126.5, 76.6, 63.5, 51.4, 21.3, 20.5. HRMS GC/QTOF (m/z): calcd for C₂₃H₂₂BClO₂S [M]: 408.1122, found: 408.1117.

2-(3,4-Difluorophenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6m)



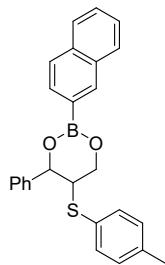
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6m**. Green solid (91.5 mg, 77%), mp 63.6–64.1 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.66 – 7.52 (m, 2H), 7.43 – 7.26 (m, 6H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 5.08 (d, *J* = 6.7 Hz, 1H), 4.22 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.02 (dd, *J* = 11.8, 7.4 Hz, 1H), 3.40 (dt, *J* = 7.0, 3.5 Hz, 1H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.2, 138.7, 134.0, 133.2, 130.8 – 130.7 (m), 130.1, 128.7, 128.7 (d, *J* = 2.1 Hz), 128.5, 126.5, 126.3, 122.7 (d, *J* = 15.1 Hz), 116.9 (d, *J* = 16.3 Hz), 76.7, 63.5, 51.4, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₂H₁₉BF₂O₂S [M]: 396.1167, found: 396.1178.

2-(3-Chloro-4-fluorophenyl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6n)



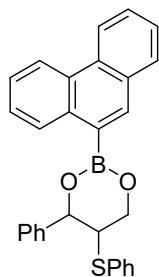
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6n**. Yellow solid (74.2 mg, 60%), mp 62.0–62.5 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.86 (d, *J* = 6.4 Hz, 1H), 7.74 – 7.66 (m, 1H), 7.43 – 7.31 (m, 5H), 7.20 (d, *J* = 8.1 Hz, 1H), 7.13 – 7.04 (m, 3H), 5.08 (d, *J* = 6.7 Hz, 1H), 4.22 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.02 (dd, *J* = 11.8, 7.3 Hz, 1H), 3.41 (td, *J* = 7.0, 4.0 Hz, 1H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 160.2 (d, *J* = 252.1 Hz), 158.9, 140.2, 138.7, 136.6, 134.3 (d, *J* = 7.5 Hz), 134.0, 130.1, 129.5, 128.7, 128.7, 128.5, 127.7, 126.5, 116.2 (d, *J* = 19.9 Hz), 76.7, 63.5, 51.4, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₂H₁₉BClFO₂S [M]: 412.0871, found: 412.0873.

2-(Naphthalen-2-yl)-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6o)



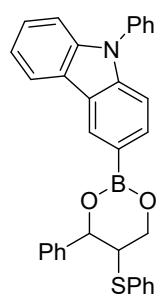
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6o**. Brown oil (83.7 mg, 68%). ¹H NMR (400 MHz, CDCl₃) δ ppm 8.81 (d, *J* = 7.4 Hz, 1H), 8.13 (d, *J* = 6.9 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.83 (d, *J* = 7.0 Hz, 1H), 7.49 – 7.43 (m, 3H), 7.39 (d, *J* = 6.1 Hz, 3H), 7.37 – 7.31 (m, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 7.8 Hz, 2H), 5.21 (d, *J* = 6.5 Hz, 1H), 4.32 (dd, *J* = 11.8, 3.9 Hz, 1H), 4.13 (dd, *J* = 11.8, 7.1 Hz, 1H), 3.50 (td, *J* = 6.8, 3.8 Hz, 1H), 2.30 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.5, 138.6, 136.9, 135.1, 134.0, 133.5, 131.4, 130.1, 128.9, 128.6, 128.6, 128.5, 128.3, 126.5, 126.2, 125.4, 125.1, 76.7, 63.4, 51.4, 21.3. HRMS GC/QTOF (m/z): calcd for C₂₆H₂₃BO₂S [M]: 410.1512, found: 410.1514.

2-(Phenanthren-9-yl)-4-phenyl-5-(phenylthio)-1,3,2-dioxaborinane (6p)



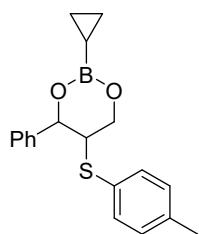
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6p**. Brown solid (100.4 mg, 75%), mp 70.1–71.5 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 8.88 (d, *J* = 8.0 Hz, 1H), 8.69 (dd, *J* = 13.5, 8.2 Hz, 2H), 8.43 (s, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.67 (t, *J* = 7.5 Hz, 1H), 7.65 – 7.53 (m, 3H), 7.45 – 7.38 (m, 4H), 7.38 – 7.31 (m, 3H), 7.30 – 7.22 (m, 3H), 5.27 (d, *J* = 6.5 Hz, 1H), 4.39 (dd, *J* = 11.8, 3.9 Hz, 1H), 4.19 (dd, *J* = 11.8, 7.1 Hz, 1H), 3.62 (td, *J* = 6.8, 3.9 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.4, 137.4, 134.6, 133.4, 132.7, 131.9, 131.2, 130.2, 129.5, 129.3, 129.2, 128.7, 128.4, 128.2, 127.7, 126.7, 126.6, 126.5, 126.1, 122.8, 122.6, 76.9, 63.5, 51.2. HRMS GC/QTOF (m/z): calcd for C₂₉H₂₃BO₂S [M]: 446.1512, found: 446.1512.

9-Phenyl-3-(4-phenyl-5-(phenylthio)-1,3,2-dioxaborinan-2-yl)-9*H*-carbazole (6q)



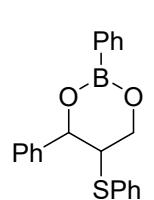
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6q**. Brown solid (101.2 mg, 66%), mp 76.2–76.9 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 8.69 (s, 1H), 8.16 (d, *J* = 7.7 Hz, 1H), 7.92 (d, *J* = 8.3 Hz, 1H), 7.63 – 7.51 (m, 4H), 7.47 – 7.31 (m, 9H), 7.28 (td, *J* = 6.8, 5.9, 3.7 Hz, 3H), 7.28 – 7.20 (m, 3H), 5.17 (d, *J* = 6.9 Hz, 1H), 4.32 (dd, *J* = 11.7, 4.0 Hz, 1H), 4.11 (dd, *J* = 11.7, 7.6 Hz, 1H), 3.53 (td, *J* = 7.4, 3.9 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 143.1, 141.2, 140.7, 137.7, 133.2, 132.9, 132.0, 130.0, 129.2, 128.6, 128.3, 128.0, 127.6, 127.2, 127.1, 126.7, 126.0, 123.7, 123.2, 120.6, 120.3, 109.9, 109.2, 76.8, 63.7, 51.5. HRMS GC/QTOF (m/z): calcd for C₃₃H₂₆BNO₂S [M]: 511.1777, found: 511.1771.

2-Cyclopropyl-4-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6r)



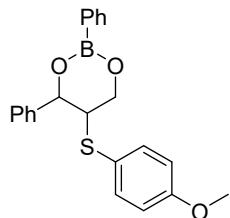
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6r**. Brown oil (70.2 mg, 72%). ¹H NMR (400 MHz, CDCl₃) δ ppm 7.38 – 7.24 (m, 5H), 7.19 – 7.11 (m, 2H), 7.04 (d, *J* = 7.8 Hz, 2H), 4.85 (d, *J* = 6.9 Hz, 1H), 4.01 (dd, *J* = 11.7, 3.9 Hz, 1H), 3.81 (dd, *J* = 11.7, 7.6 Hz, 1H), 3.26 (td, *J* = 7.2, 3.9 Hz, 1H), 2.30 (s, 3H), 0.58 (d, *J* = 9.2 Hz, 2H), 0.52 (d, *J* = 6.1 Hz, 2H), -0.13 – -0.26 (m, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.6, 138.4, 133.8, 129.9, 128.9, 128.5, 128.2, 126.5, 76.2, 63.2, 51.5, 21.2, 4.0, 3.9. HR-ESI-MS (m/z): calcd for C₁₉H₂₂BO₂S [M+H]⁺: 325.1434, found: 325.1433.

2,4-Diphenyl-5-(phenylthio)-1,3,2-dioxaborinane (6s)



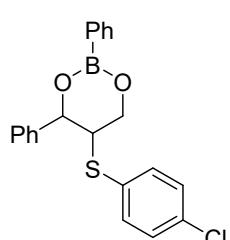
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6s**. Yellow solid (83.1 mg, 80%), mp 70.4–71.3 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.86 (d, *J* = 7.8 Hz, 2H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.41 – 7.30 (m, 7H), 7.29 – 7.26 (m, 2H), 7.25 – 7.21 (m, 3H), 5.11 (d, *J* = 6.9 Hz, 1H), 4.25 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.05 (dd, *J* = 11.8, 7.6 Hz, 1H), 3.48 (td, *J* = 7.4, 4.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.4, 134.2, 133.2, 132.8, 131.1, 129.2, 128.6, 128.4, 128.1, 127.8, 126.6, 76.7, 63.5, 51.3. HRMS GC/QTOF (m/z): calcd for C₂₁H₁₉BO₂S [M]: 346.1199, found: 346.1189.

5-((4-Methoxyphenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (6t)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6t**. Yellow solid (81.2 mg, 72%), mp 75.1–76.0 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.85 (d, *J* = 6.5 Hz, 2H), 7.43 (d, *J* = 7.3 Hz, 1H), 7.40 – 7.30 (m, 7H), 7.24 (d, *J* = 8.7 Hz, 2H), 6.82 – 6.75 (m, 2H), 5.07 (d, *J* = 6.8 Hz, 1H), 4.21 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.00 (dd, *J* = 11.8, 7.5 Hz, 1H), 3.76 (s, 3H), 3.32 (td, *J* = 7.2, 4.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 160.1, 140.6, 136.3, 134.2, 131.1, 128.6, 128.3, 127.7, 126.6, 122.7, 114.8, 76.5, 63.4, 55.4, 52.0. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₁BO₃S [M]: 376.1304, found: 376.1302.

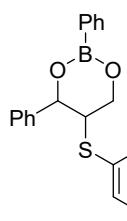
5-((4-Chlorophenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (6u)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6u**. Yellow solid (69.6 mg, 61%), mp 74.3–75.1 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.87 – 7.83 (m, 2H), 7.49 – 7.43 (m, 1H), 7.36 (q, *J* = 6.5, 5.9 Hz, 7H), 7.24 – 7.13 (m, 4H),

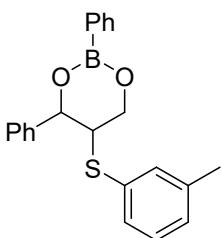
5.10 (d, $J = 6.9$ Hz, 1H), 4.26 (dd, $J = 11.8, 4.0$ Hz, 1H), 4.04 (dd, $J = 11.8, 7.7$ Hz, 1H), 3.44 (td, $J = 7.3, 4.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.2, 134.6, 134.4, 134.2, 131.3, 131.2, 129.4, 128.7, 128.5, 127.8, 126.6, 76.7, 63.4, 51.7. HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BClO}_2\text{S}$ [M]: 380.0809, found: 380.0813.

5-((4-Bromophenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (**6v**)



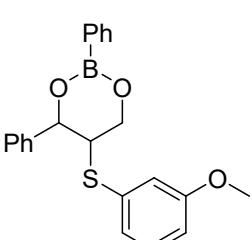
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6v**. White solid (91.6mg, 72%), mp 71.4–72.2 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.85 (d, $J = 7.3$ Hz, 2H), 7.45 (d, $J = 6.0$ Hz, 1H), 7.40 – 7.33 (m, 9H), 7.13 – 7.06 (m, 1H), 5.09 (d, $J = 6.8$ Hz, 1H), 4.25 (dd, $J = 11.9, 4.0$ Hz, 1H), 4.03 (dd, $J = 11.9, 7.6$ Hz, 1H), 3.44 (td, $J = 7.3, 4.1$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.2, 134.7, 134.2, 132.3, 132.0, 131.2, 128.7, 128.5, 127.8, 126.6, 122.5, 76.7, 63.4, 51.6. HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BBrO}_2\text{S}$ [M]: 424.0304, found: 424.0291.

2,4-Diphenyl-5-(*m*-tolylthio)-1,3,2-dioxaborinane (**6w**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6w**. Green solid (85.4 mg, 79%), mp 75.5–76.5 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.86 (d, $J = 6.5$ Hz, 2H), 7.48 – 7.42 (m, 1H), 7.39 – 7.32 (m, 7H), 7.15 – 7.00 (m, 4H), 5.10 (d, $J = 7.1$ Hz, 1H), 4.26 (dd, $J = 11.8, 4.1$ Hz, 1H), 4.05 (dd, $J = 11.8, 7.9$ Hz, 1H), 3.45 (td, $J = 7.6, 4.1$ Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.5, 139.0, 134.2, 133.8, 132.5, 131.1, 130.1, 129.0, 128.9, 128.5, 128.3, 127.8, 126.7, 76.8, 63.7, 51.3, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{21}\text{BO}_2\text{S}$ [M]: 360.1355, found: 360.1360.

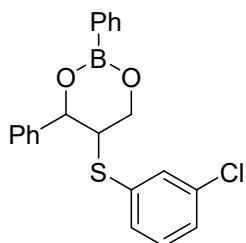
5-((3-Methoxyphenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (**6x**)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6x**. Brown oil (80.1 mg, 71%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.86 (d, $J = 6.6$ Hz, 2H), 7.50 – 7.41 (m, 1H), 7.41 – 7.31 (m, 7H), 7.21 – 7.11 (m, 1H), 6.88 (d, $J = 7.7$ Hz, 1H), 6.81 – 6.74 (m, 2H), 5.12 (d, $J = 6.9$ Hz, 1H), 4.27 (dd, $J = 11.7, 4.1$ Hz, 1H), 4.07 (dd, $J = 11.8, 7.7$ Hz, 1H), 3.74 (s, 3H), 3.50 (td, $J = 7.3, 4.0$ Hz, 1H). ^{13}C

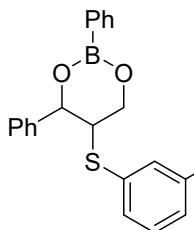
NMR (100 MHz, CDCl₃) δ ppm 159.9, 140.4, 134.2, 131.2, 130.0, 128.6, 128.4, 127.8, 126.6, 125.1, 118.1, 114.0, 76.7, 63.6, 55.5, 51.2. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₁BO₃S [M]: 376.1304, found: 376.1302.

5-((3-Chlorophenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (6y)



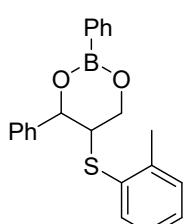
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6y**. Yellow solid (68.4 mg, 60%), mp 62.5–63.2 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.86 (d, *J* = 7.7 Hz, 2H), 7.45 (d, *J* = 6.7 Hz, 1H), 7.41 – 7.34 (m, 7H), 7.21 – 7.08 (m, 4H), 5.11 (d, *J* = 7.1 Hz, 1H), 4.29 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.06 (dd, *J* = 11.8, 7.9 Hz, 1H), 3.49 (td, *J* = 7.5, 3.9 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.1, 135.0, 134.8, 134.2, 132.3, 131.2, 130.7, 130.2, 128.7, 128.6, 128.1, 127.8, 126.6, 76.9, 63.5, 51.4. HRMS GC/QTOF (m/z): calcd for C₂₁H₁₈BClO₂S [M]: 380.0809, found: 380.0807.

5-((3-Bromophenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (6z)



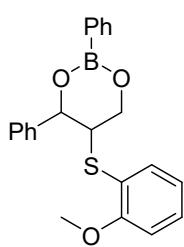
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6z**. Yellow solid (92.9 mg, 73%), mp 70.5–71.4 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.85 (d, *J* = 7.4 Hz, 2H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.41 – 7.28 (m, 9H), 7.16 (d, *J* = 8.0 Hz, 1H), 7.11 – 7.05 (m, 1H), 5.11 (d, *J* = 7.1 Hz, 1H), 4.29 (dd, *J* = 11.8, 4.0 Hz, 1H), 4.05 (dd, *J* = 11.8, 8.1 Hz, 1H), 3.49 (td, *J* = 7.6, 4.0 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 140.0, 135.3, 135.1, 134.2, 131.2, 131.2, 131.0, 130.5, 128.7, 128.6, 127.8, 126.6, 122.8, 76.9, 63.5, 51.5. HRMS GC/QTOF (m/z): calcd for C₂₁H₁₈BBrO₂S [M]: 424.0304, found: 424.0316.

2,4-Diphenyl-5-(*o*-tolylthio)-1,3,2-dioxaborinane (6aa)



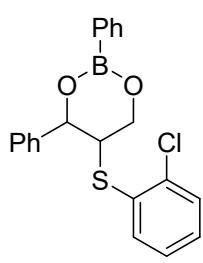
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6aa**. Yellow solid (69.1 mg, 64%), mp 81.1–82.0 °C. ¹H NMR (400 MHz, CDCl₃) δ ppm 7.87 (d, *J* = 6.5 Hz, 2H), 7.50 – 7.41 (m, 1H), 7.41 – 7.29 (m, 8H), 7.28 (d, *J* = 7.7 Hz, 1H), 7.20 – 7.13 (m, 2H), 5.11 (d, *J* = 6.4 Hz, 1H), 4.20 (dd, *J* = 11.8, 3.8 Hz, 1H), 4.04 (dd, *J* = 11.8, 7.0 Hz, 1H), 3.45 (td, *J* = 6.8, 3.9 Hz, 1H), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ ppm 141.2, 140.5, 134.2, 133.9, 132.1, 131.1, 130.7, 128.6, 128.3, 128.3, 127.8, 126.7, 126.4, 76.5, 63.2, 50.9, 20.9. HRMS GC/QTOF (m/z): calcd for C₂₂H₂₁BO₂S [M]: 360.1355, found: 360.1346.

5-((2-Methoxyphenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (6ab)



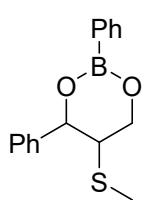
Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6ab**. Brown oil (75.6 mg, 67%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.87 (d, $J = 6.5$ Hz, 2H), 7.49 – 7.40 (m, 1H), 7.41 – 7.28 (m, 7H), 7.31 – 7.22 (m, 2H), 6.87 – 6.77 (m, 2H), 5.11 (d, $J = 6.6$ Hz, 1H), 4.21 (dd, $J = 11.8, 3.9$ Hz, 1H), 4.01 (dd, $J = 11.8, 7.1$ Hz, 1H), 3.84 (s, 3H), 3.68 (td, $J = 6.8, 3.9$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 159.4, 140.7, 135.4, 134.2, 131.1, 130.1, 128.5, 128.2, 127.7, 126.4, 121.1, 120.1, 111.1, 76.6, 63.3, 55.9, 48.7. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{21}\text{BO}_3\text{S}$ [M]: 376.1304, found: 376.1297.

5-((2-Chlorophenyl)thio)-2,4-diphenyl-1,3,2-dioxaborinane (6ac)



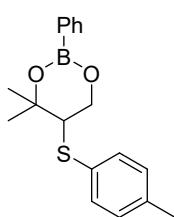
Flash column chromatography on silica gel (eluent: PE/EtOAc = 10/1, v/v) to afford **6ac**. Yellow solid (71.8 mg, 63%), mp 55.5–55.8 °C. ^1H NMR (400 MHz, CDCl_3) δ ppm 7.87 (d, $J = 6.5$ Hz, 2H), 7.46 (d, $J = 7.3$ Hz, 1H), 7.43 – 7.32 (m, 7H), 7.35 – 7.25 (m, 2H), 7.23 – 7.15 (m, 1H), 7.14 – 7.06 (m, 1H), 5.14 (d, $J = 6.3$ Hz, 1H), 4.26 (dd, $J = 11.9, 3.9$ Hz, 1H), 4.04 (dd, $J = 11.9, 7.0$ Hz, 1H), 3.69 (td, $J = 6.7, 3.9$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.2, 137.8, 135.0, 134.2, 131.7, 131.2, 130.3, 129.5, 128.6, 128.4, 127.8, 127.3, 126.4, 76.6, 63.0, 49.8. HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BClO}_2\text{S}$ [M]: 380.0809, found: 380.0817.

5-(Methylthio)-2,4-diphenyl-1,3,2-dioxaborinane (6ad)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6ad**. Brown oil (53.0 mg, 62%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.84 (d, $J = 6.4$ Hz, 2H), 7.47 – 7.38 (m, 3H), 7.41 – 7.32 (m, 4H), 7.33 (d, $J = 2.8$ Hz, 1H), 5.07 (d, $J = 9.0$ Hz, 1H), 4.37 (dd, $J = 11.6, 4.5$ Hz, 1H), 4.05 (dd, $J = 11.6, 10.2$ Hz, 1H), 3.08 (td, $J = 9.7, 4.4$ Hz, 1H), 1.66 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 140.7, 134.1, 131.1, 128.4, 128.4, 127.7, 126.9, 78.4, 65.0, 42.9, 4.1. HRMS GC/QTOF (m/z): calcd for $\text{C}_{16}\text{H}_{18}\text{BO}_2\text{S}$ [M + H] $^+$: 285.1121, found: 285.1121.

4,4-Dimethyl-2-phenyl-5-(*p*-tolylthio)-1,3,2-dioxaborinane (6ae)



Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6ae**. Brown oil (66.5 mg, 71%). ^1H NMR (400 MHz, CDCl_3) δ ppm (d, $J = 6.5$ Hz, 2H), 7.43 –

7.28 (m, 5H), 7.13 (d, J = 7.9 Hz, 2H), 4.18 (dd, J = 11.8, 5.3 Hz, 1H), 4.10 (t, J = 11.4 Hz, 1H), 3.24 (dd, J = 11.1, 5.3 Hz, 1H), 2.34 (s, 3H), 1.61 (s, 3H), 1.39 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 138.1, 133.9, 132.8, 130.8, 130.7, 130.1, 127.7, 74.3, 63.3, 55.2, 29.5, 24.5, 21.2. HRMS GC/QTOF (m/z): calcd for $\text{C}_{18}\text{H}_{21}\text{BO}_2\text{S}$ [M]: 312.1355, found: 312.1364.

2-Phenyl-5-(phenylthio)-4-(*p*-tolyl)-1,3,2-dioxaborinane (**6af**)

Flash column chromatography on silica gel (eluent: PE/EtOAc = 12/1, v/v) to afford **6af**. Brown oil (84.3 mg, 78%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.77 (d, J = 6.5 Hz, 2H), 7.42 – 7.33 (m, 1H), 7.29 (d, J = 7.3 Hz, 2H), 7.22 (d, J = 2.8 Hz, 1H), 7.17 (d, J = 4.3 Hz, 6H), 7.10 (d, J = 7.8 Hz, 2H), 5.00 (d, J = 7.0 Hz, 1H), 4.17 (dd, J = 11.8, 4.0 Hz, 1H), 3.97 (dd, J = 11.8, 7.6 Hz, 1H), 3.41 (td, J = 7.2, 3.9 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 138.1, 137.5, 134.2, 133.2, 132.7, 131.1, 129.3, 129.2, 128.1, 127.8, 126.5, 76.5, 63.6, 51.2, 21.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{22}\text{H}_{21}\text{BO}_2\text{S}$ [M]: 360.1355, found: 360.1346.

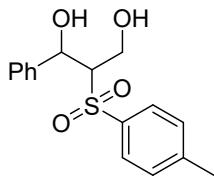
4-(4-Chlorophenyl)-2-phenyl-5-(phenylthio)-1,3,2-dioxaborinane (**6ag**)

Flash column chromatography on silica gel (eluent: PE/EtOAc = 9/1, v/v) to afford **6ag**. Brown oil (77.5 mg, 68%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.75 (d, J = 6.5 Hz, 2H), 7.38 (d, J = 7.4 Hz, 1H), 7.33 – 7.25 (m, 3H), 7.25 (d, J = 3.6 Hz, 3H), 7.18 (d, J = 2.5 Hz, 5H), 4.98 (d, J = 7.7 Hz, 1H), 4.20 (dd, J = 11.8, 4.2 Hz, 1H), 4.00 (dd, J = 11.8, 8.5 Hz, 1H), 3.34 (td, J = 8.1, 4.2 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 138.9, 134.2, 134.2, 133.2, 132.4, 131.3, 129.3, 128.7, 128.2, 128.2, 127.8, 76.4, 64.0, 51.3. HRMS GC/QTOF (m/z): calcd for $\text{C}_{21}\text{H}_{18}\text{BClO}_2\text{S}$ [M]: 380.0809, found: 380.0807.

1-Phenyl-2-(*p*-tolylthio)propane-1,3-diol (**7**)

Flash column chromatography on silica gel (eluent: PE/EtOAc = 3/1, v/v) to afford **7**. Brown oil (72.4 mg, 88%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.33 (d, J = 4.3 Hz, 4H), 7.31 – 7.21 (m, 3H), 7.07 (d, J = 7.9 Hz, 2H), 4.89 (d, J = 5.8 Hz, 1H), 3.82 (dd, J = 11.8, 4.5 Hz, 1H), 3.73 (dd, J = 11.8, 5.2 Hz, 1H), 3.36 (q, J = 5.2 Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 141.5, 138.0, 133.1, 130.0, 129.9, 128.5, 128.0, 126.4, 75.7, 62.4, 58.4, 21.2. HR-ESI-MS (m/z): calcd for $\text{C}_{16}\text{H}_{18}\text{O}_2\text{S}$ [M + H]⁺: 274.1028, found: 274.1016.

1-Phenyl-2-tosylpropane-1,3-diol (8)

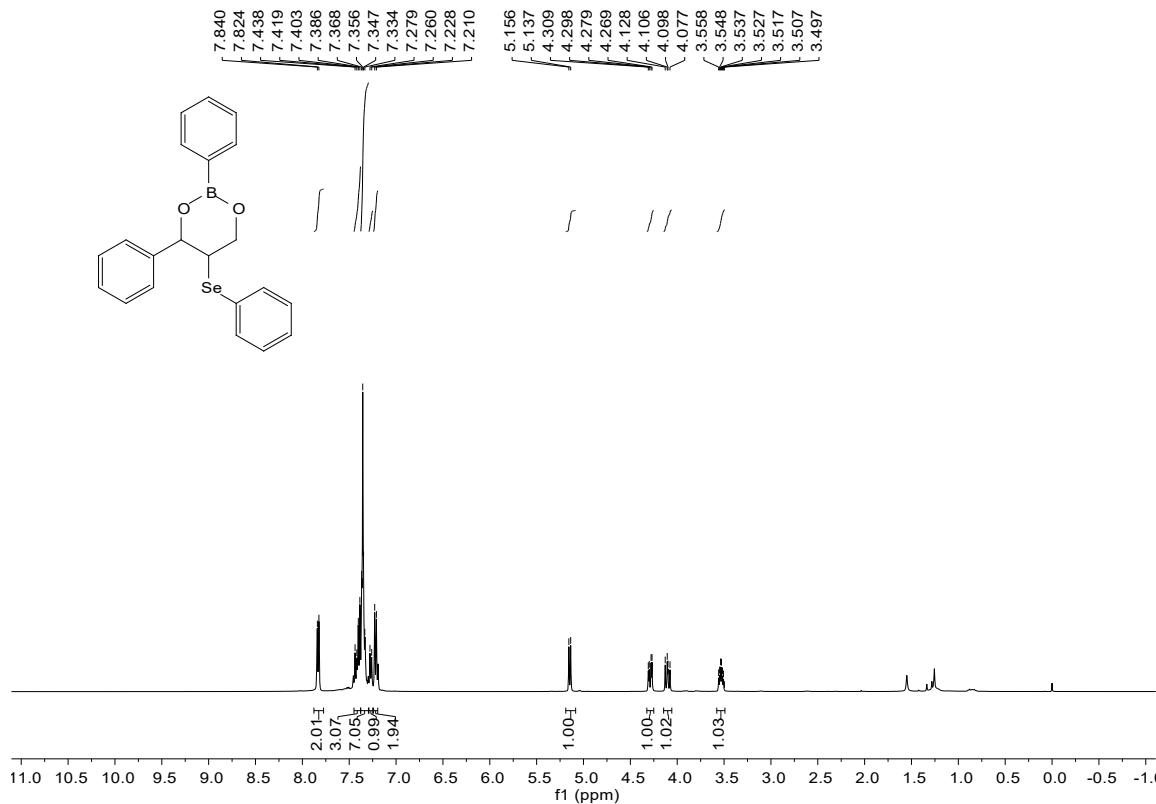


Flash column chromatography on silica gel (eluent: PE/EtOAc = 2/1, v/v) to afford **8**.

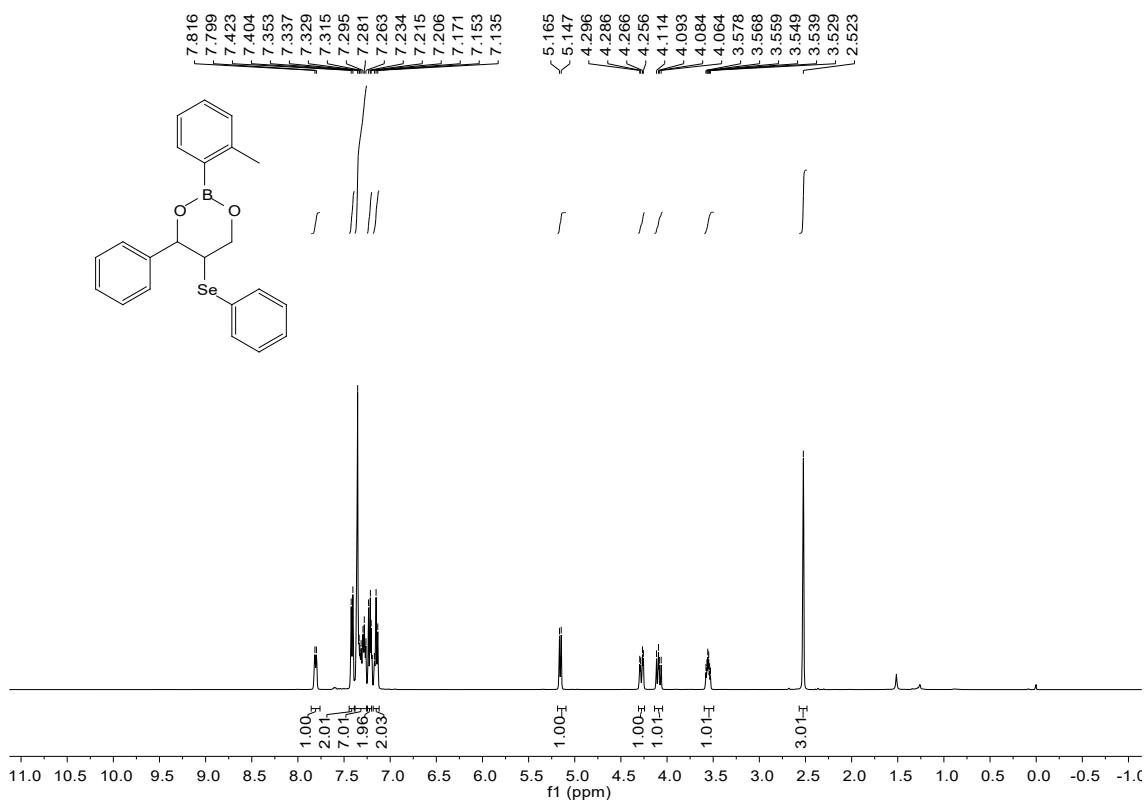
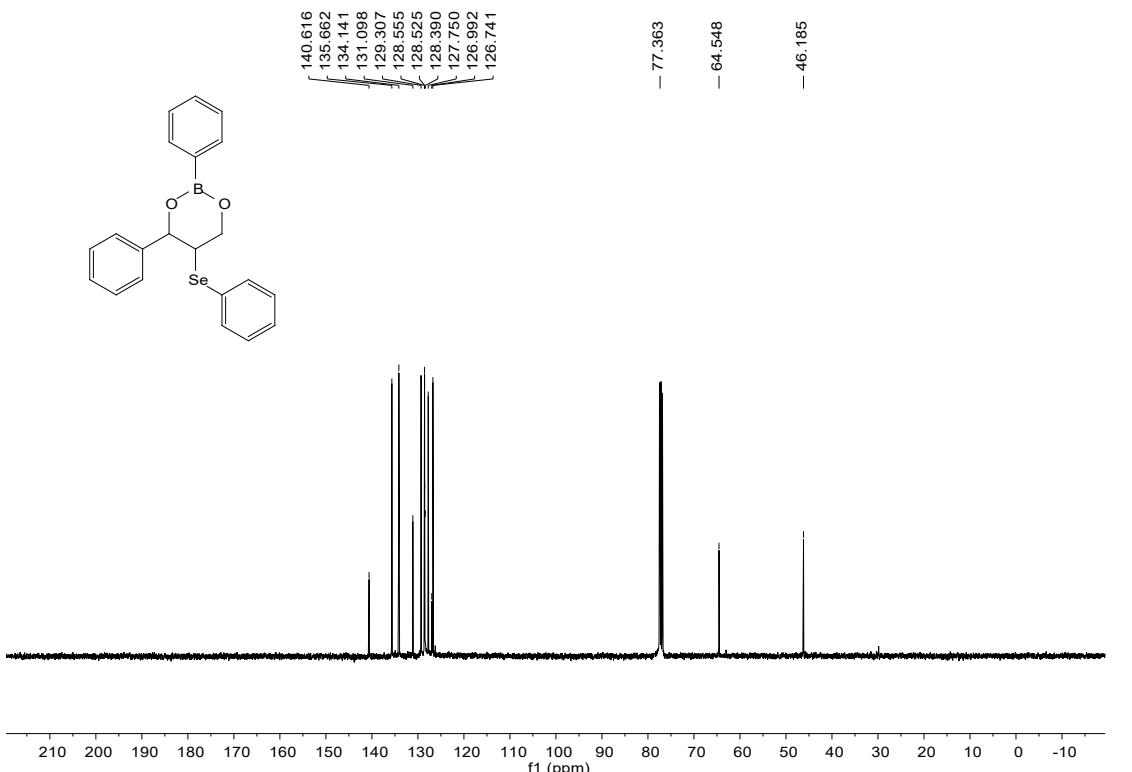
Yellow solid (66.1 mg, 67%). ^1H NMR (400 MHz, CDCl_3) δ ppm 7.89 (d, J = 8.3 Hz, 2H), 7.42 (d, J = 8.0 Hz, 2H), 7.37 – 7.25 (m, 3H), 7.22 (d, J = 7.0 Hz, 2H), 5.40 (s, 1H), 4.22 (dd, J = 13.3, 6.1 Hz, 1H), 3.97 (dd, J = 13.3, 2.8 Hz, 1H), 3.31 (dt, J = 6.1, 2.4 Hz, 1H), 2.49 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 145.8, 139.5, 134.7, 130.3, 128.8, 128.1, 125.5, 71.7, 70.5, 57.0, 21.9. HR-ESI-MS (m/z): calcd for $\text{C}_{16}\text{H}_{18}\text{O}_4\text{SNa} [\text{M} + \text{Na}]^+$: 329.0823, found: 329.0826.

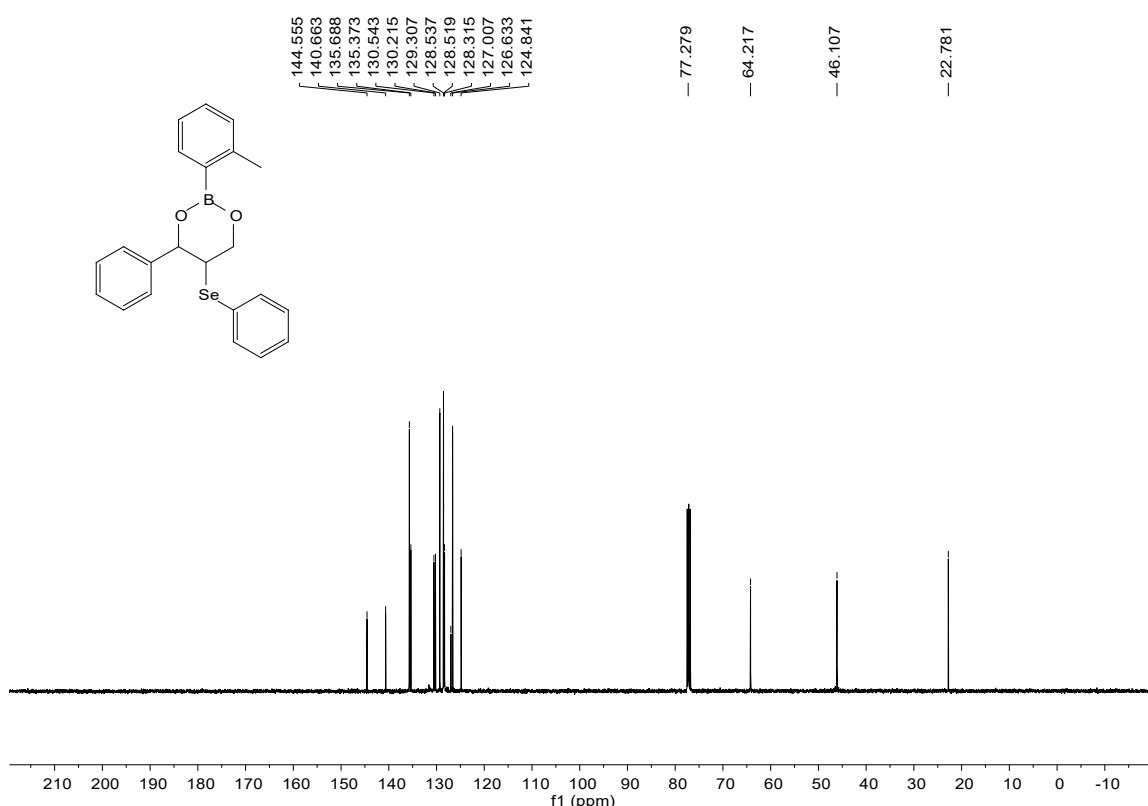
3. NMR spectra for new compounds

¹H NMR (400 MHz, CDCl₃) spectrum of compound 4a

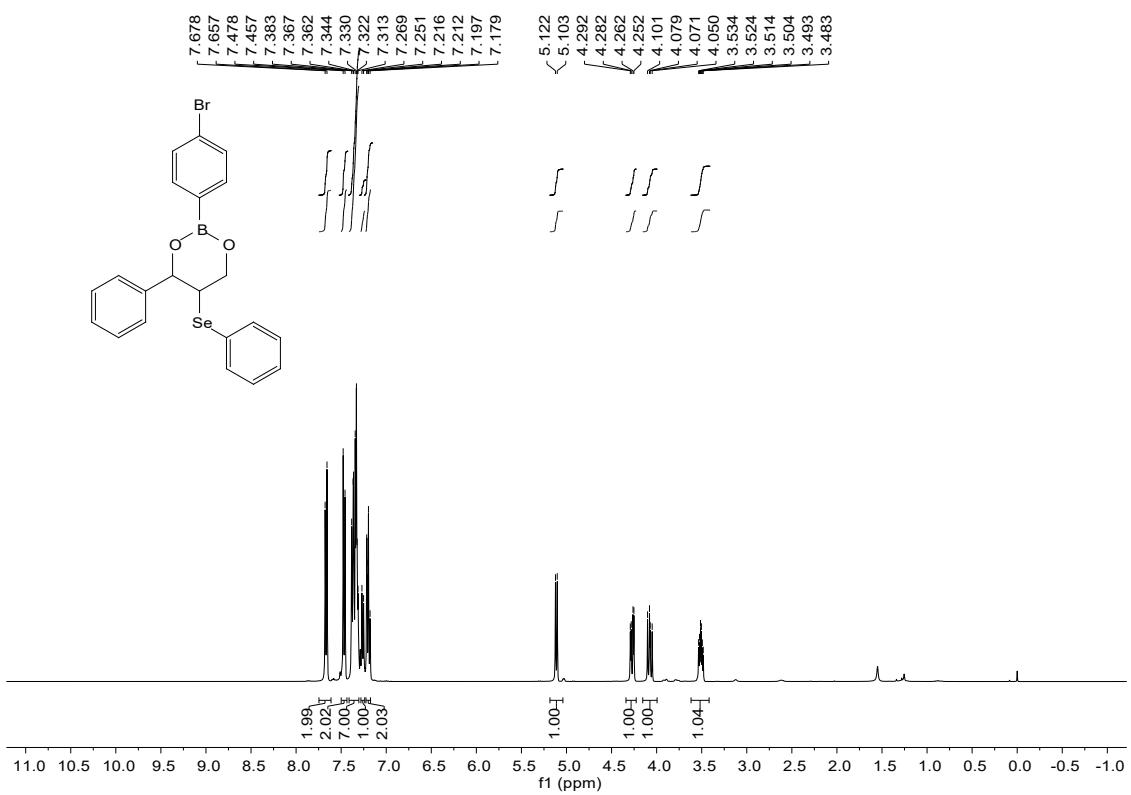


¹³C NMR (100 MHz, CDCl₃) spectrum of compound 4a

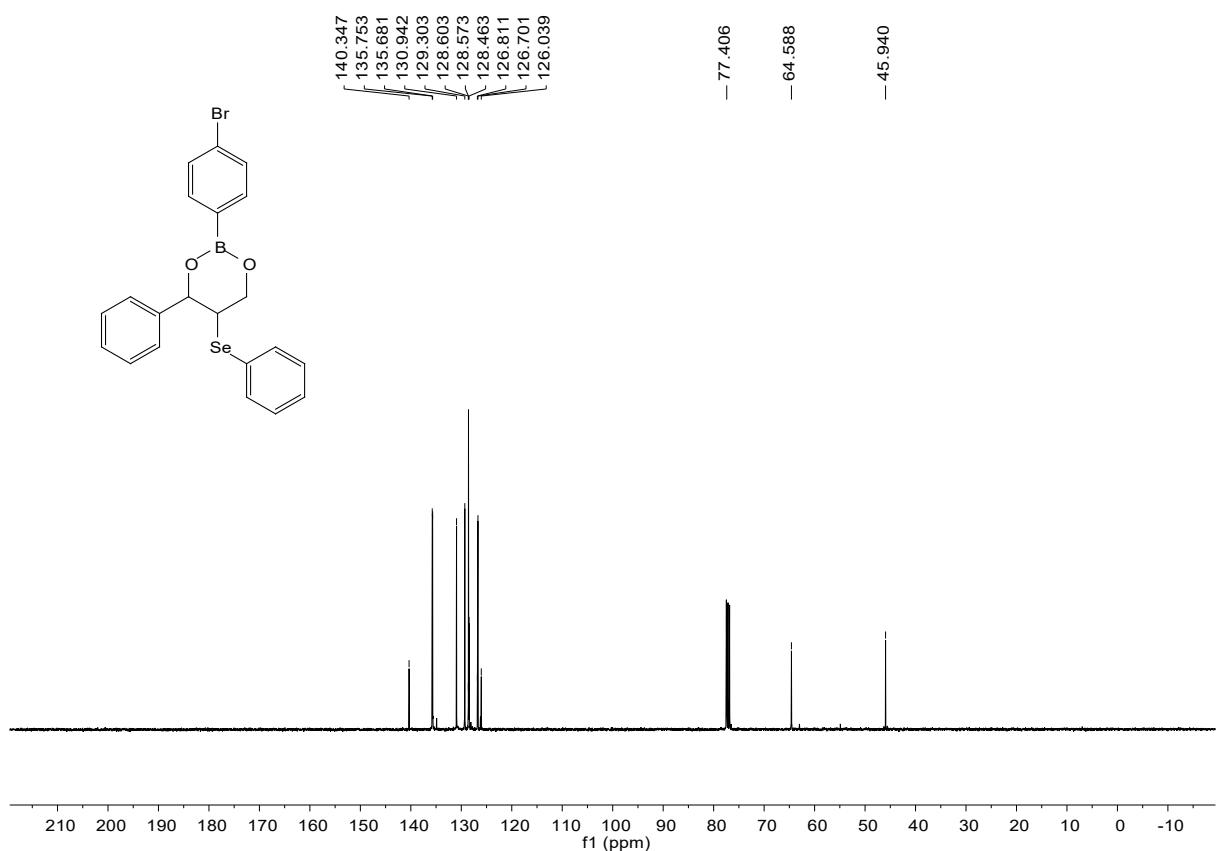




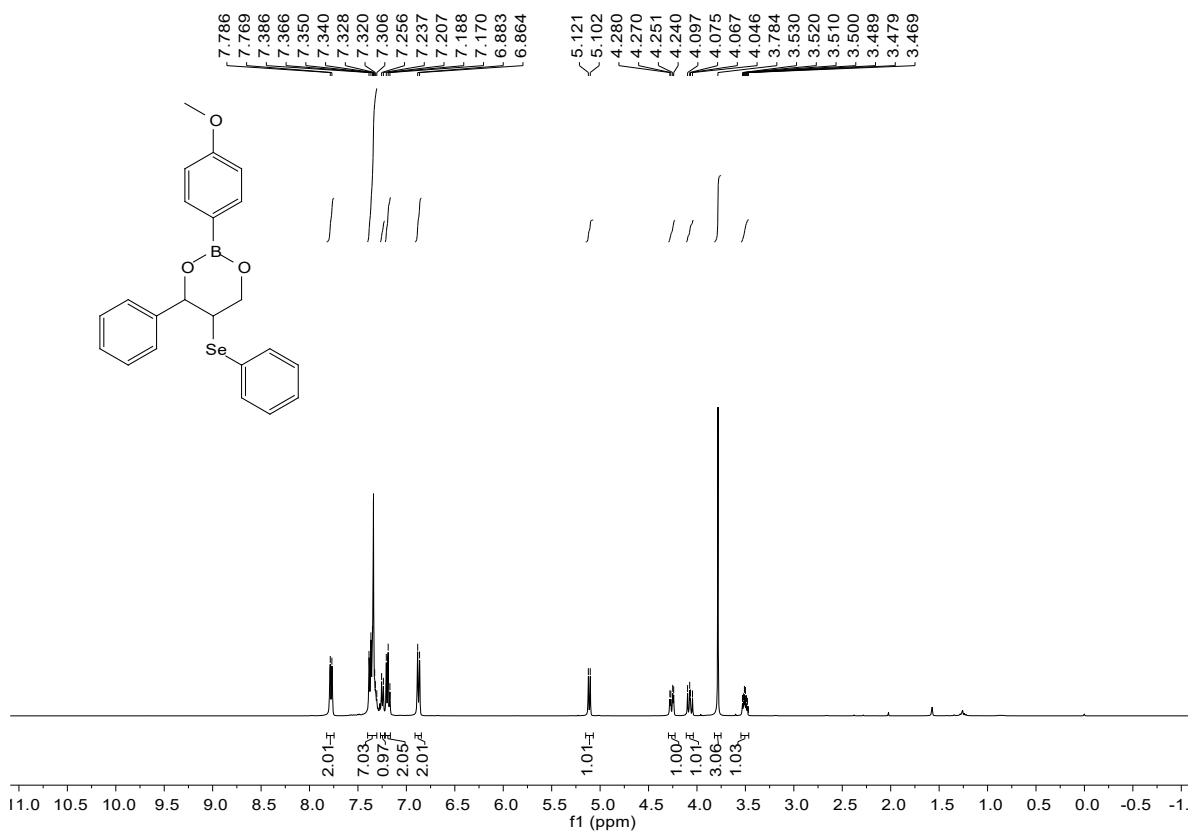
¹H NMR (400 MHz, CDCl₃) spectrum of compound 4c



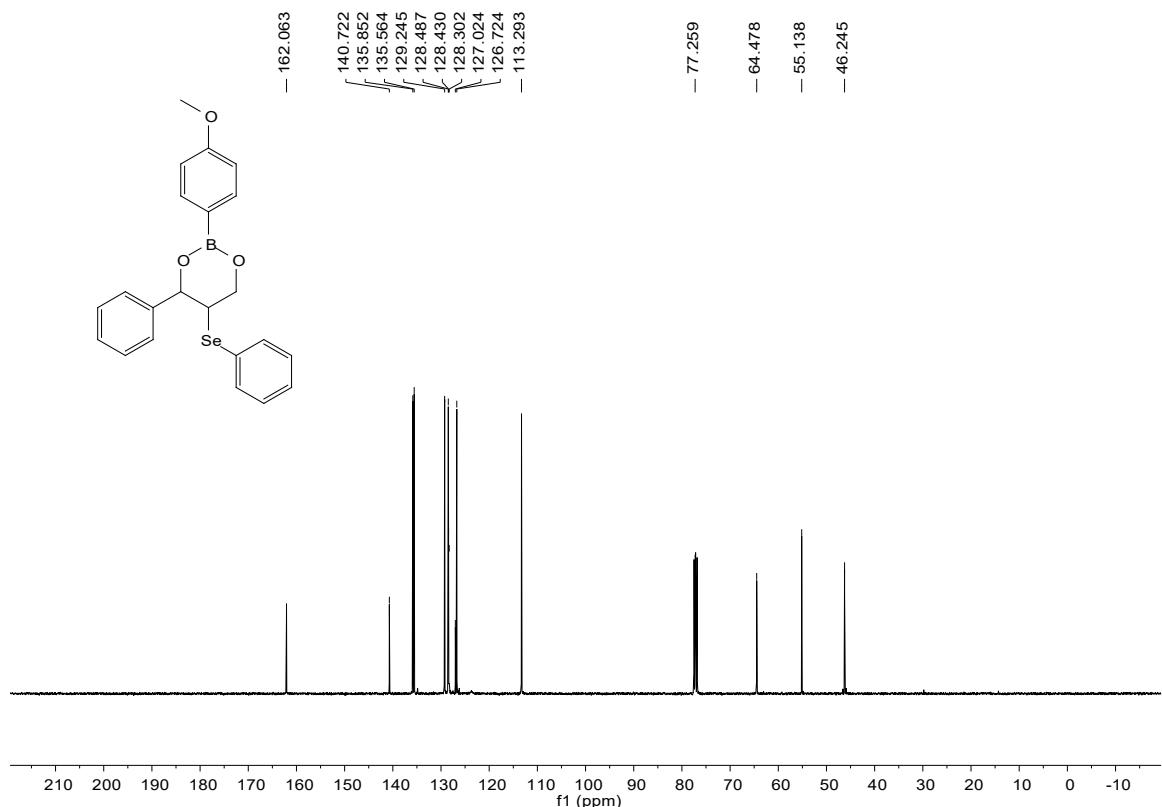
¹³C NMR (100 MHz, CDCl₃) spectrum of compound 4c



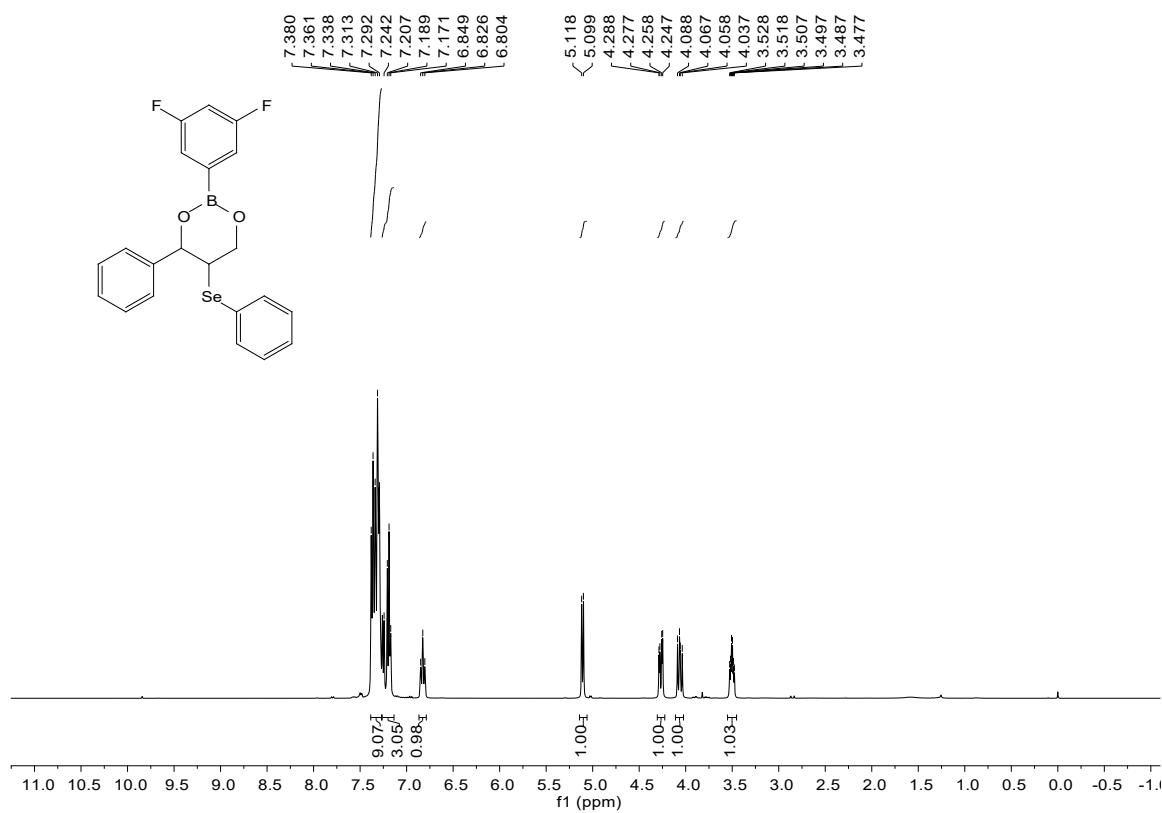
^1H NMR (400 MHz, CDCl_3) spectrum of compound **4d**



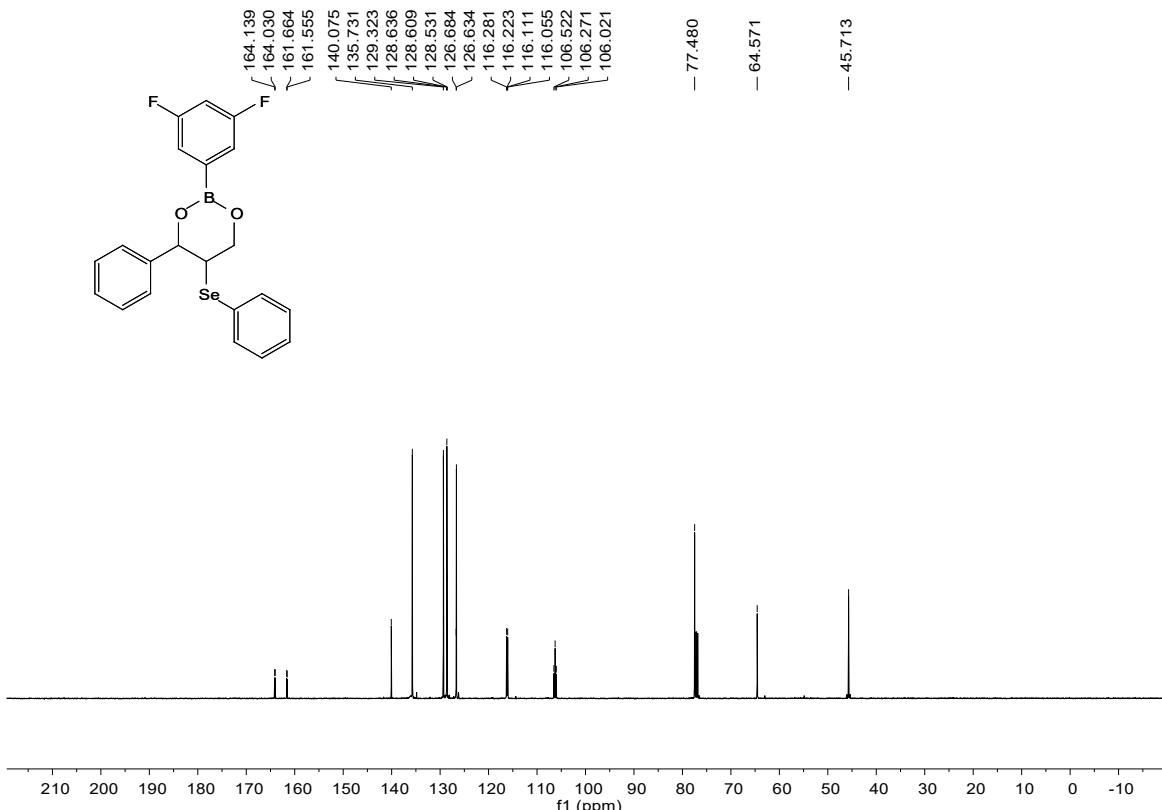
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **4d**



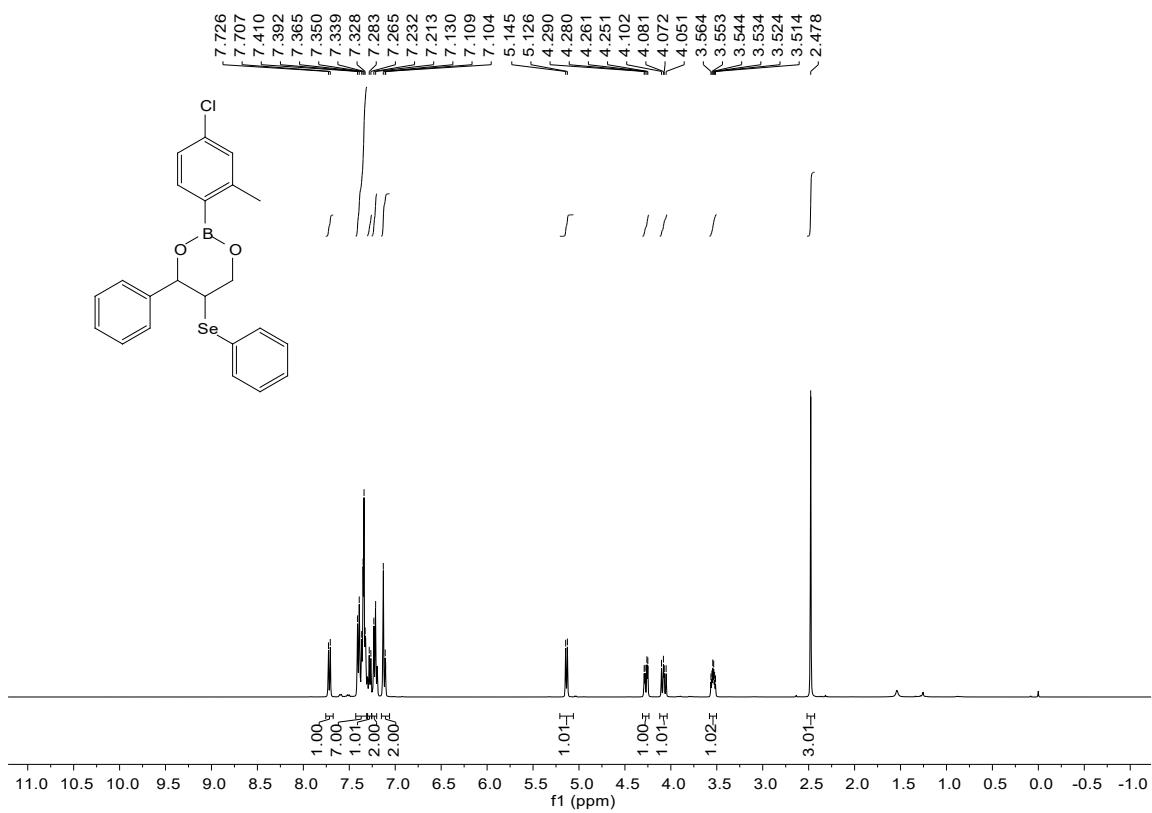
¹H NMR (400 MHz, CDCl₃) spectrum of compound **4e**



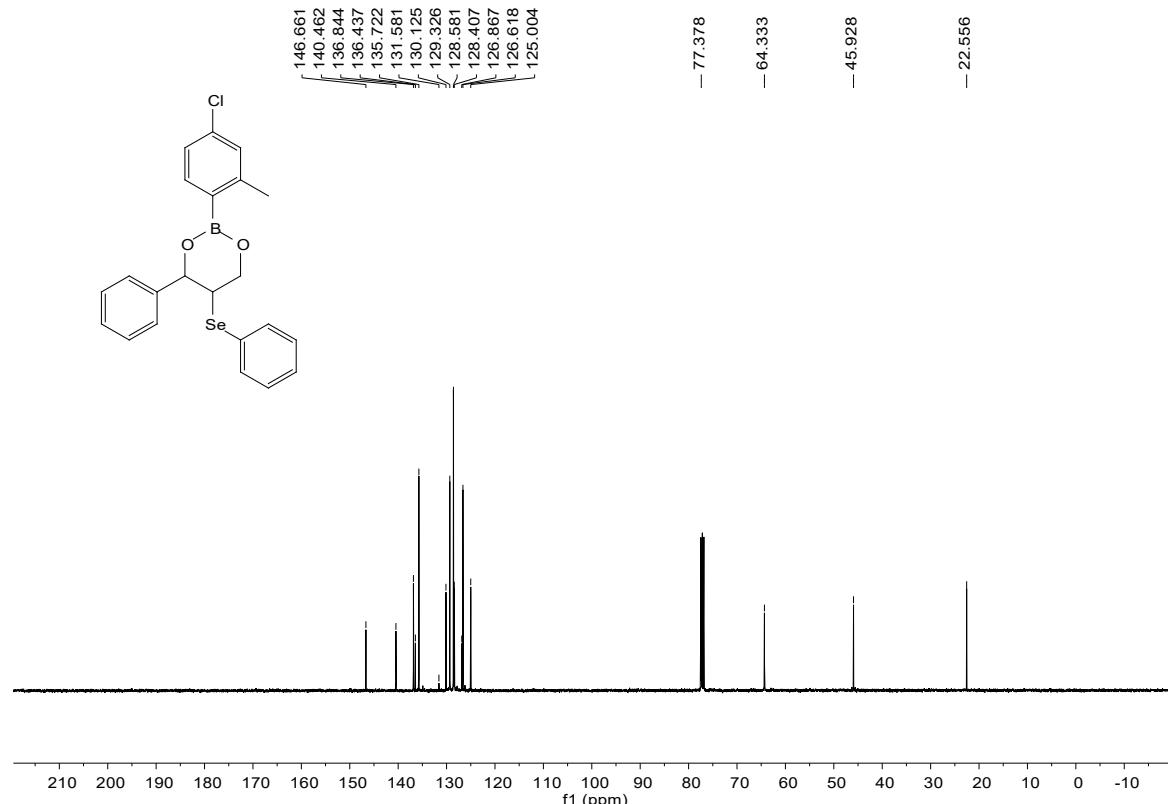
¹³C NMR (100 MHz, CDCl₃) spectrum of compound 4e



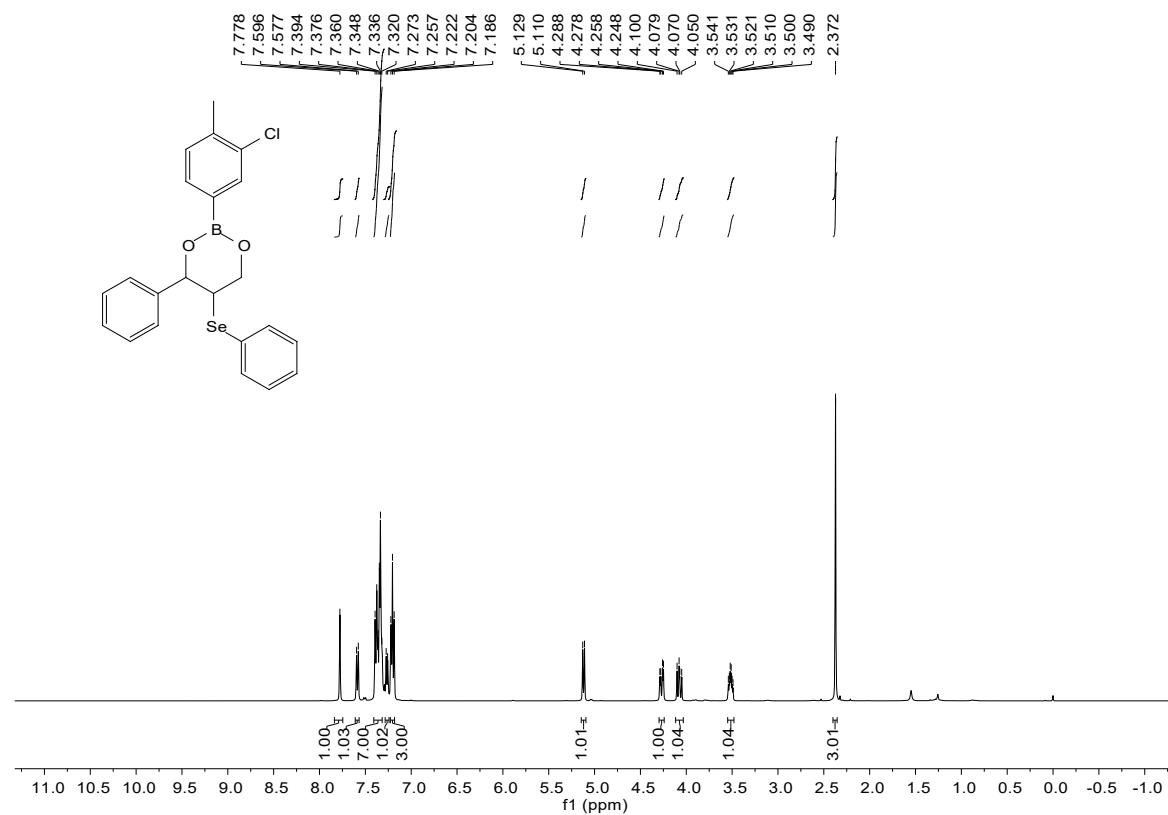
¹H NMR (400 MHz, CDCl₃) spectrum of compound 4f



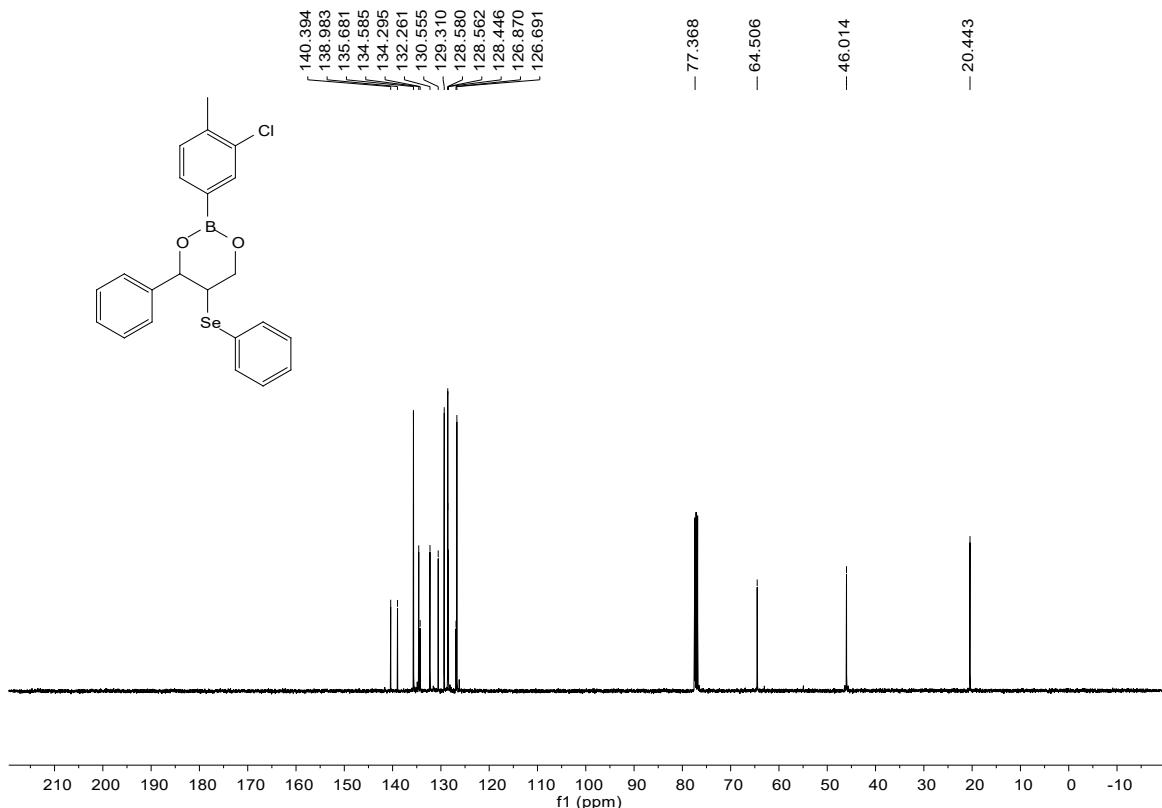
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **4f**



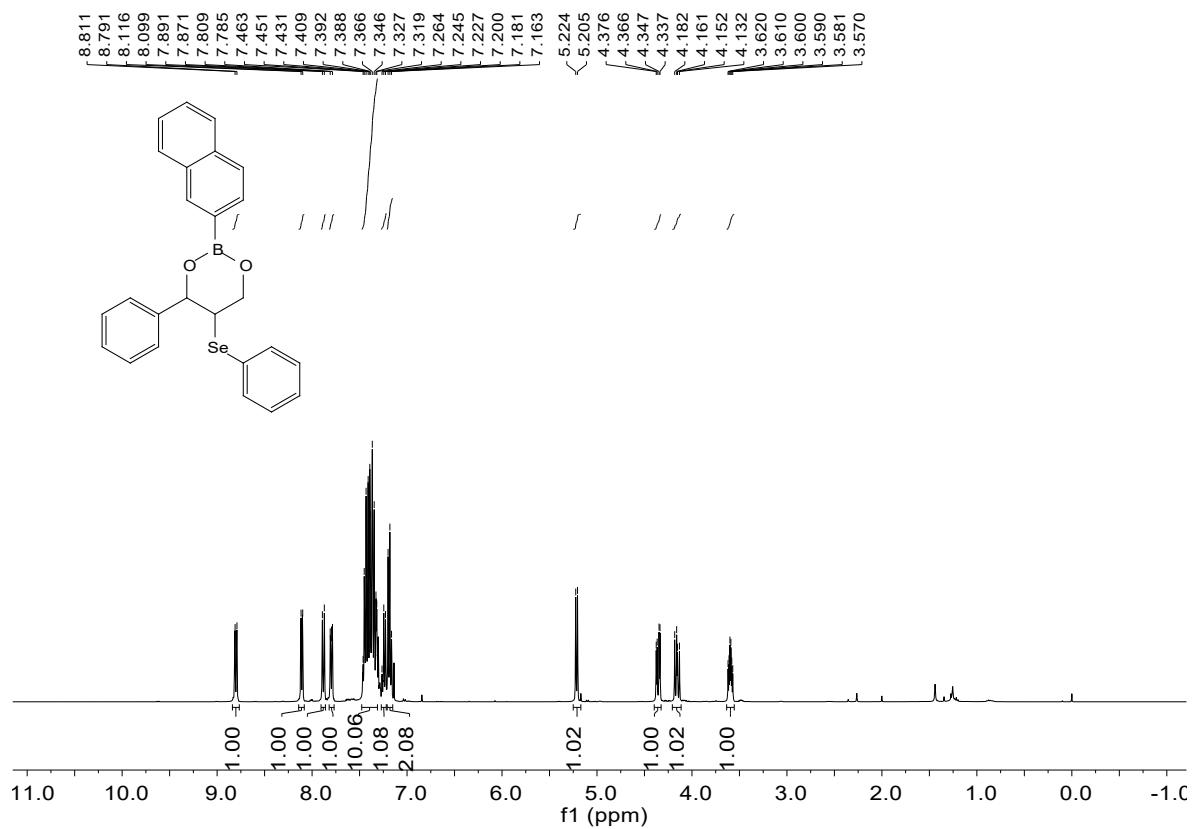
¹H NMR (400 MHz, CDCl₃) spectrum of compound **4g**



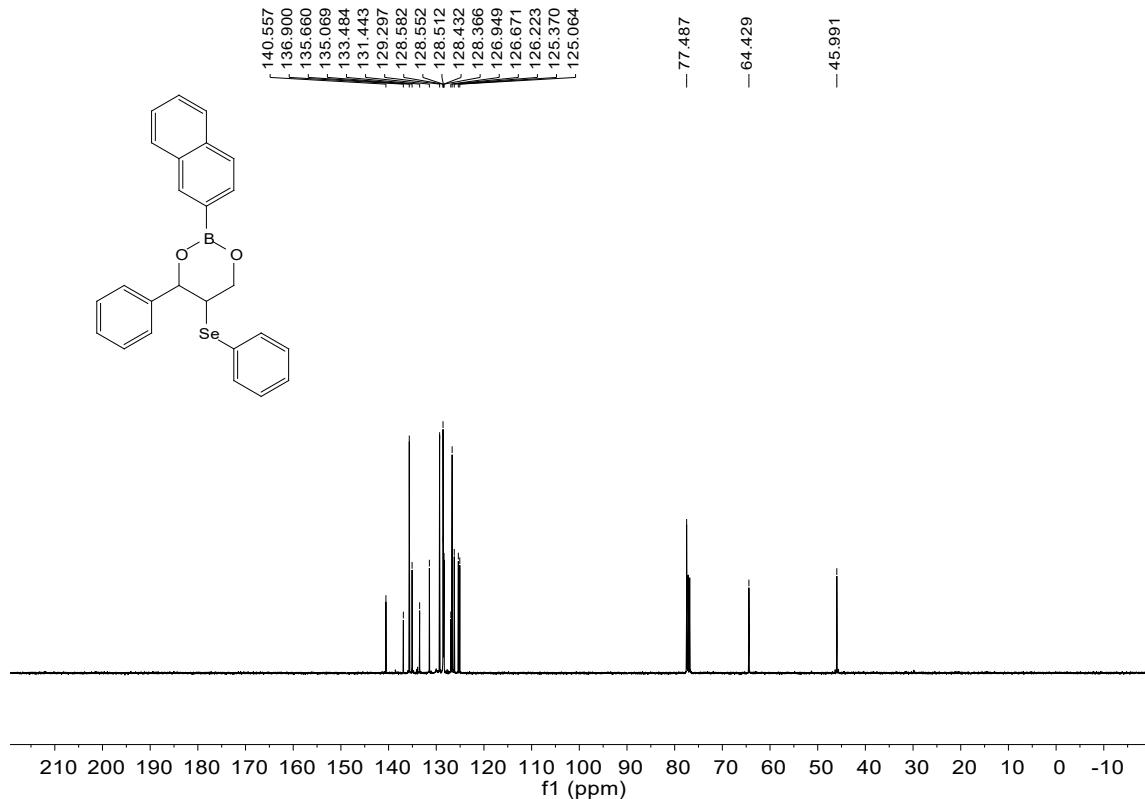
^{13}C NMR (100 MHz, CDCl_3) spectrum of compound **4g**



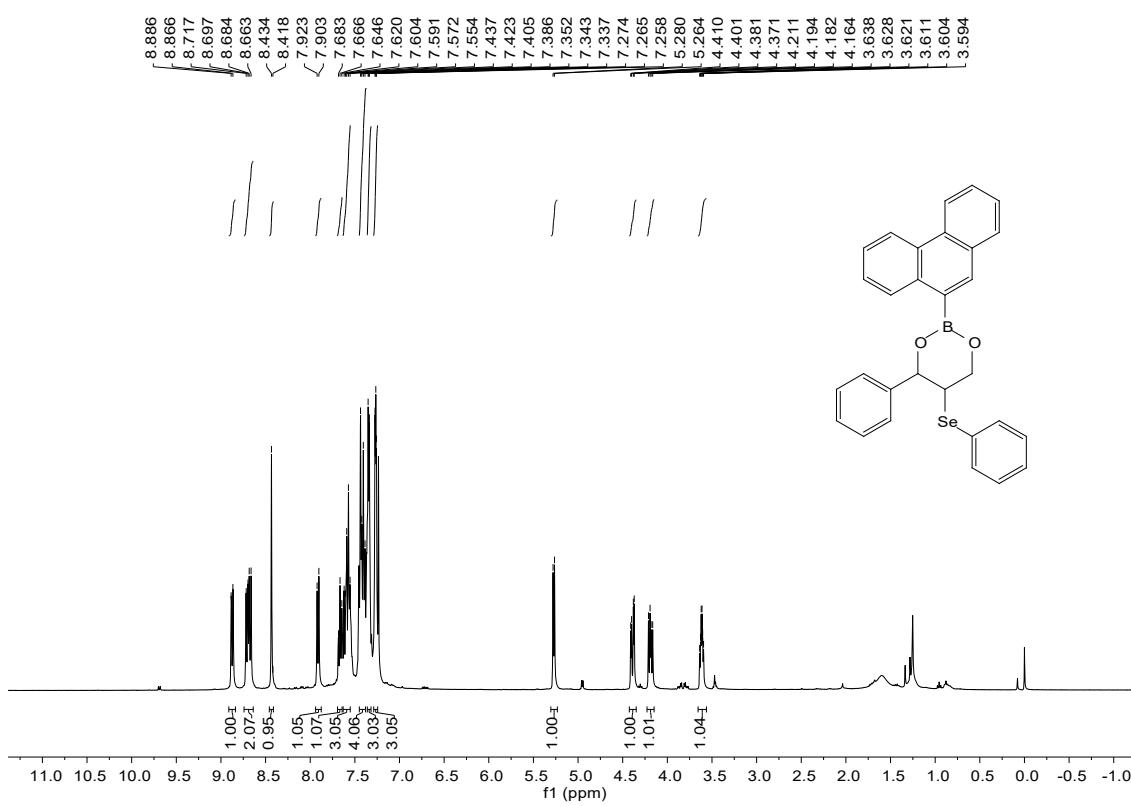
^1H NMR (400 MHz, CDCl_3) spectrum of compound **4h**



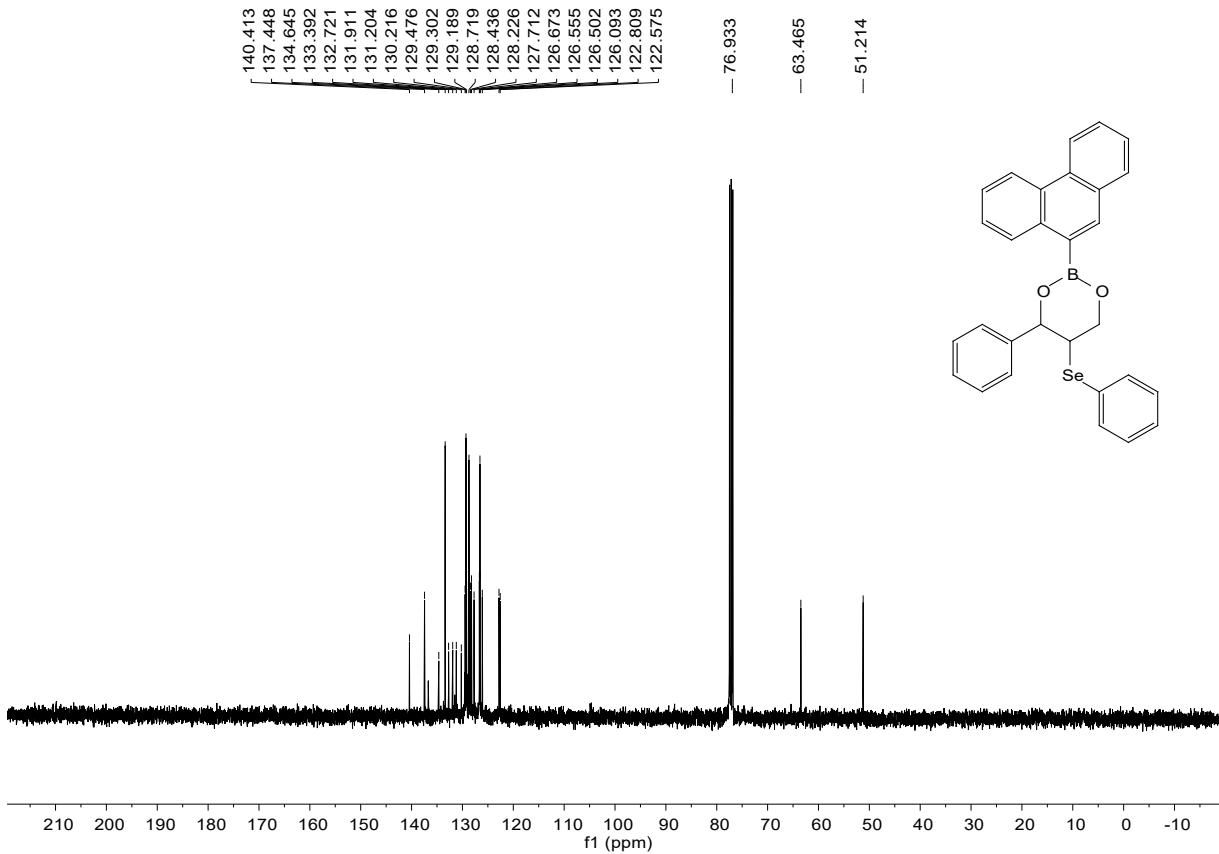
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **4h**



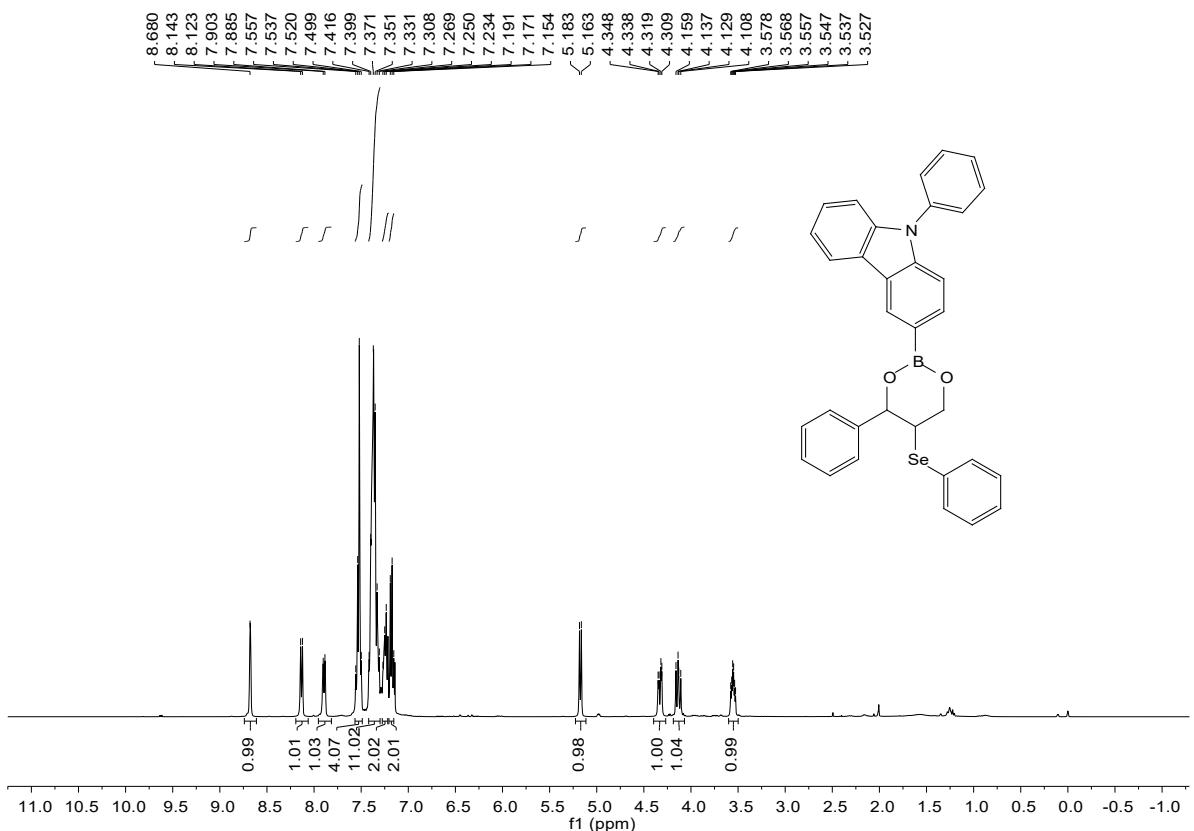
¹H NMR (400 MHz, CDCl₃) spectrum of compound **4i**



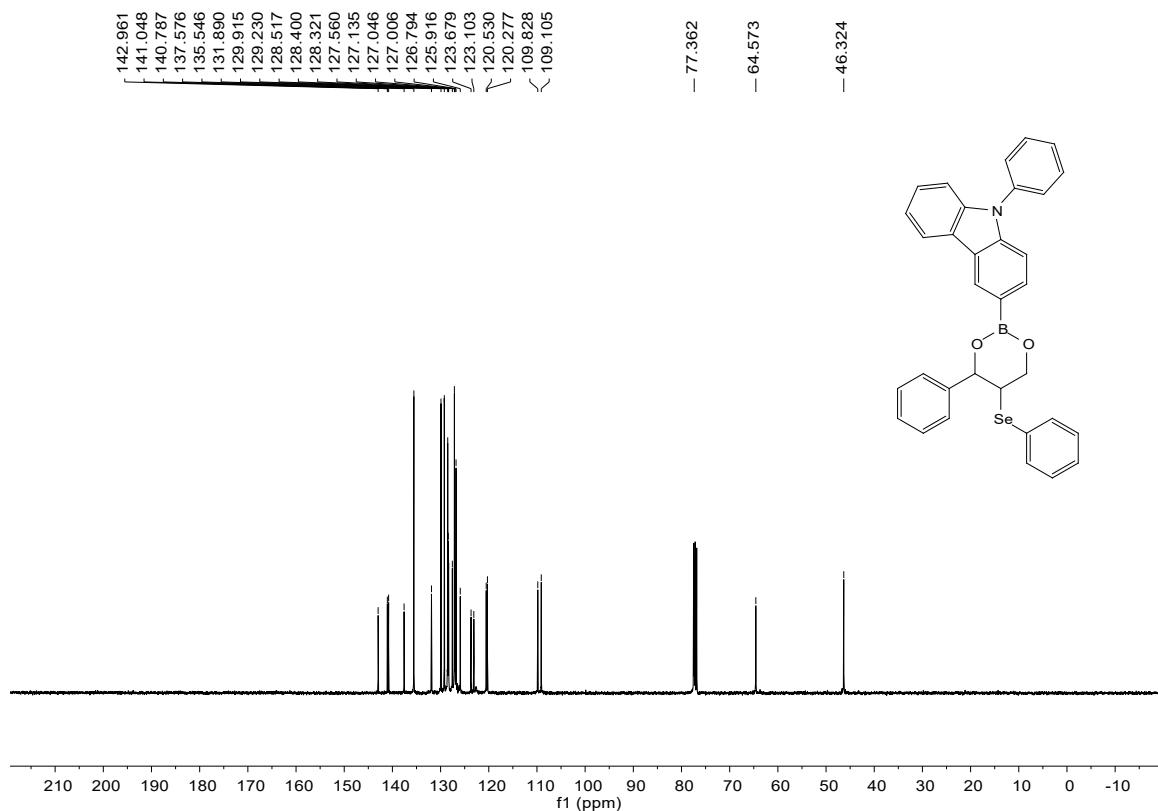
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **4i**



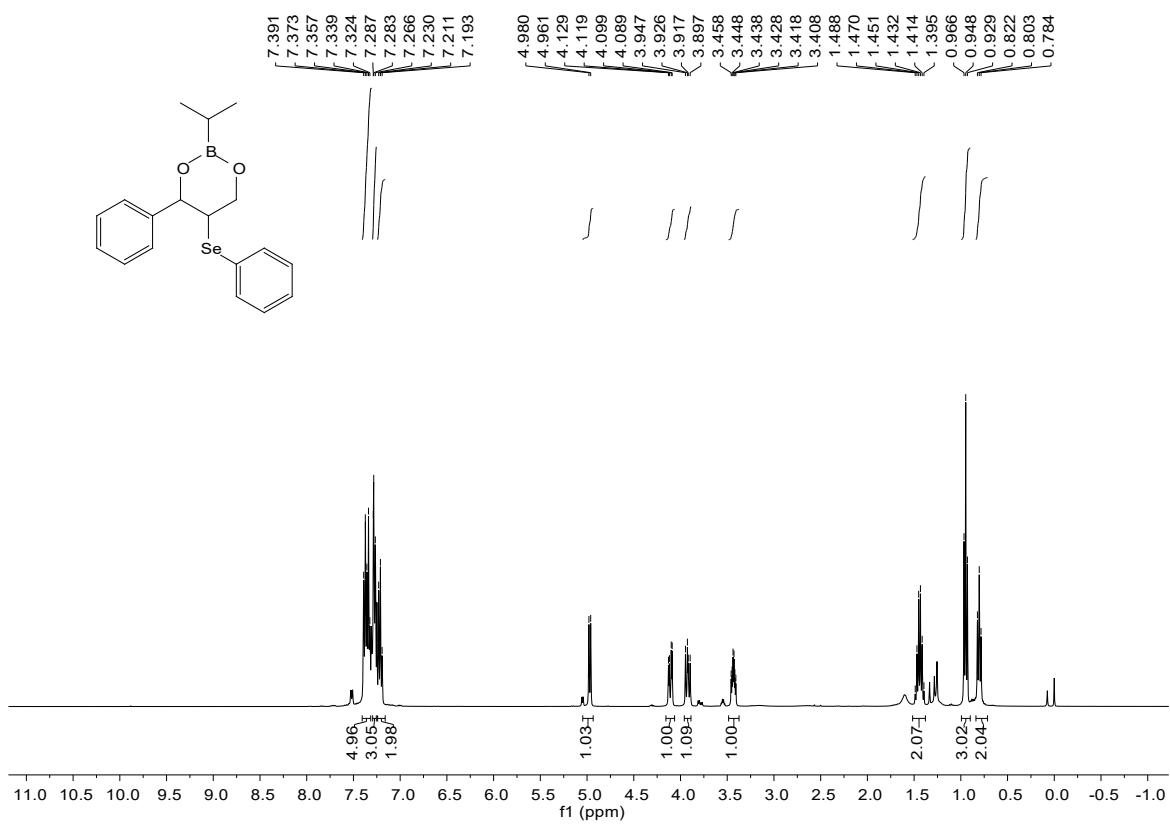
¹H NMR (400 MHz, CDCl₃) spectrum of compound 4j



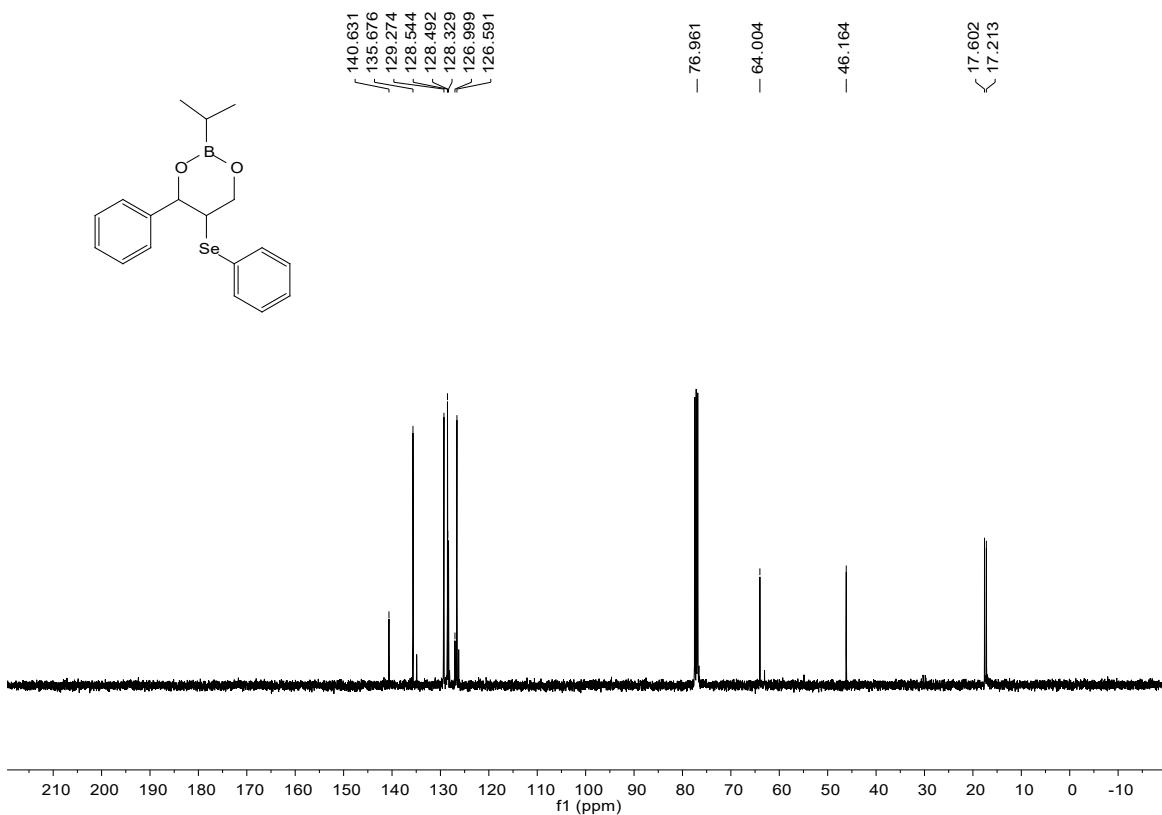
¹³C NMR (100 MHz, CDCl₃) spectrum of compound 4j



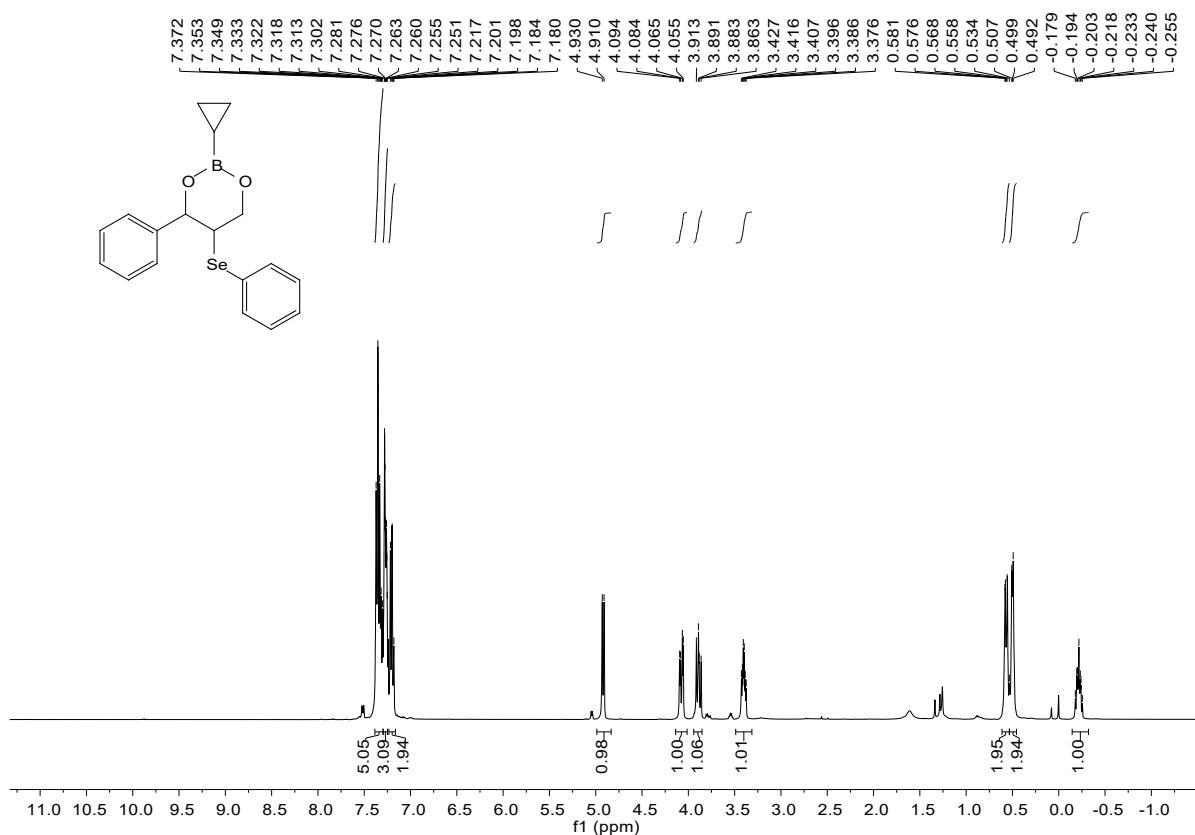
¹H NMR (400 MHz, CDCl₃) spectrum of compound **4k**



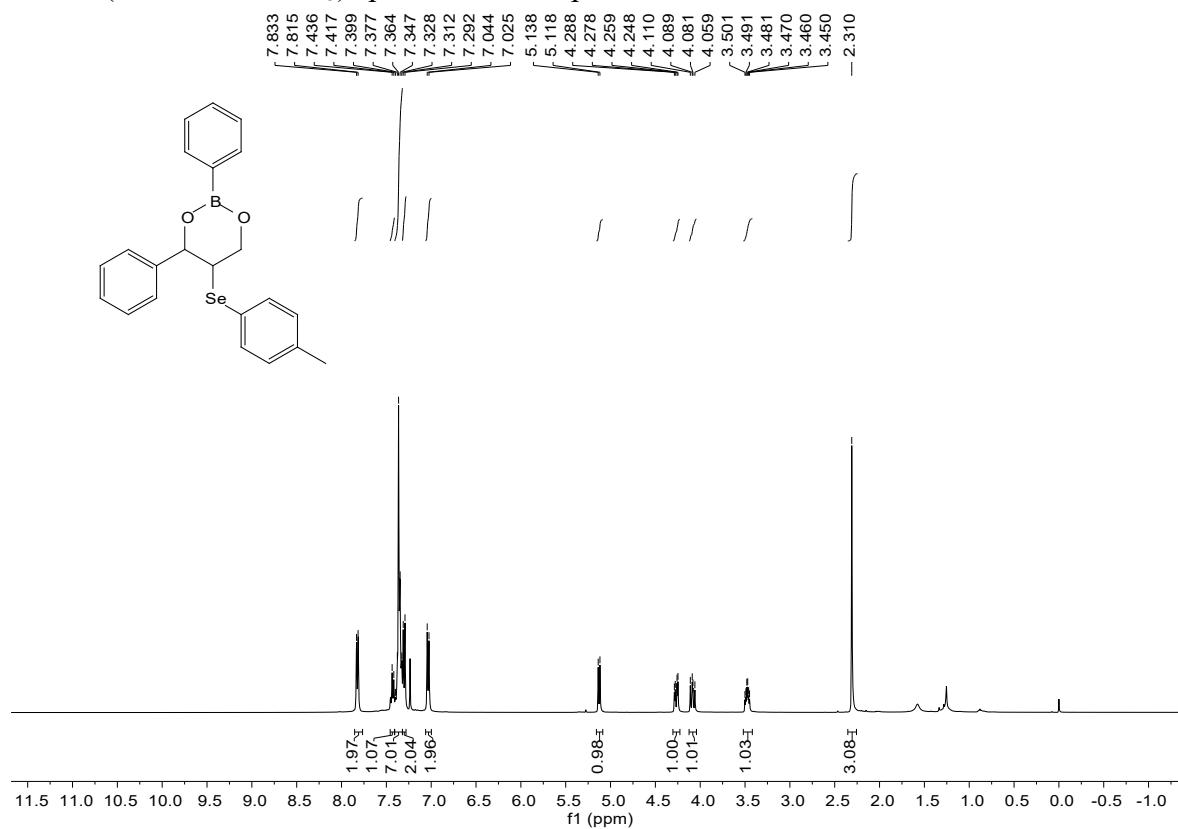
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **4k**



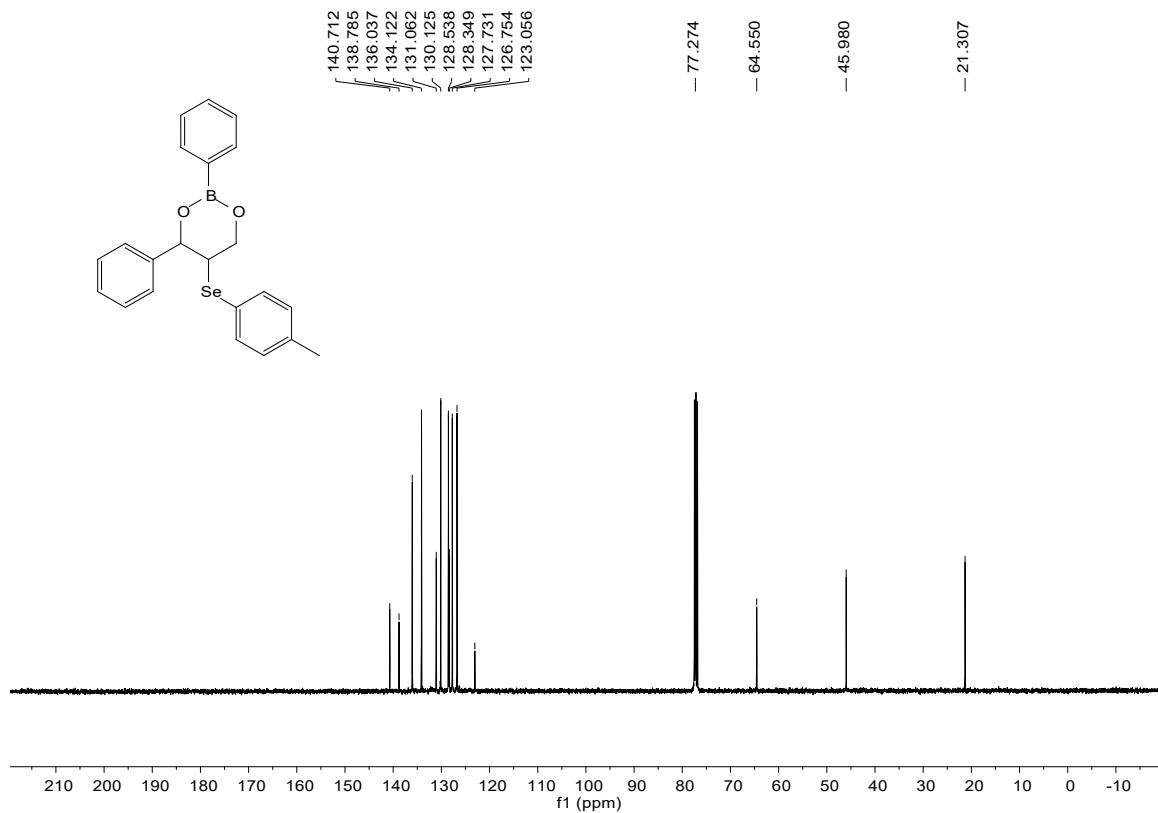
¹H NMR (400 MHz, CDCl₃) spectrum of compound **4I**



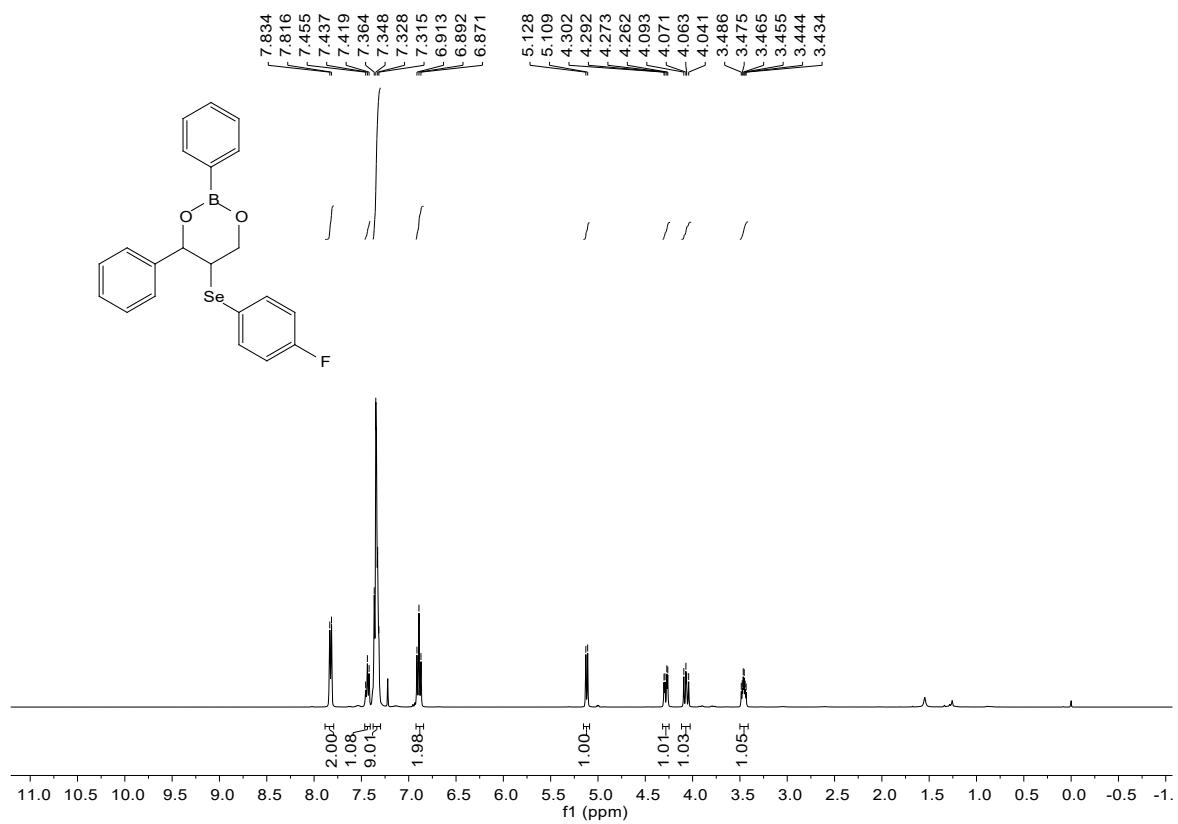
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5a**



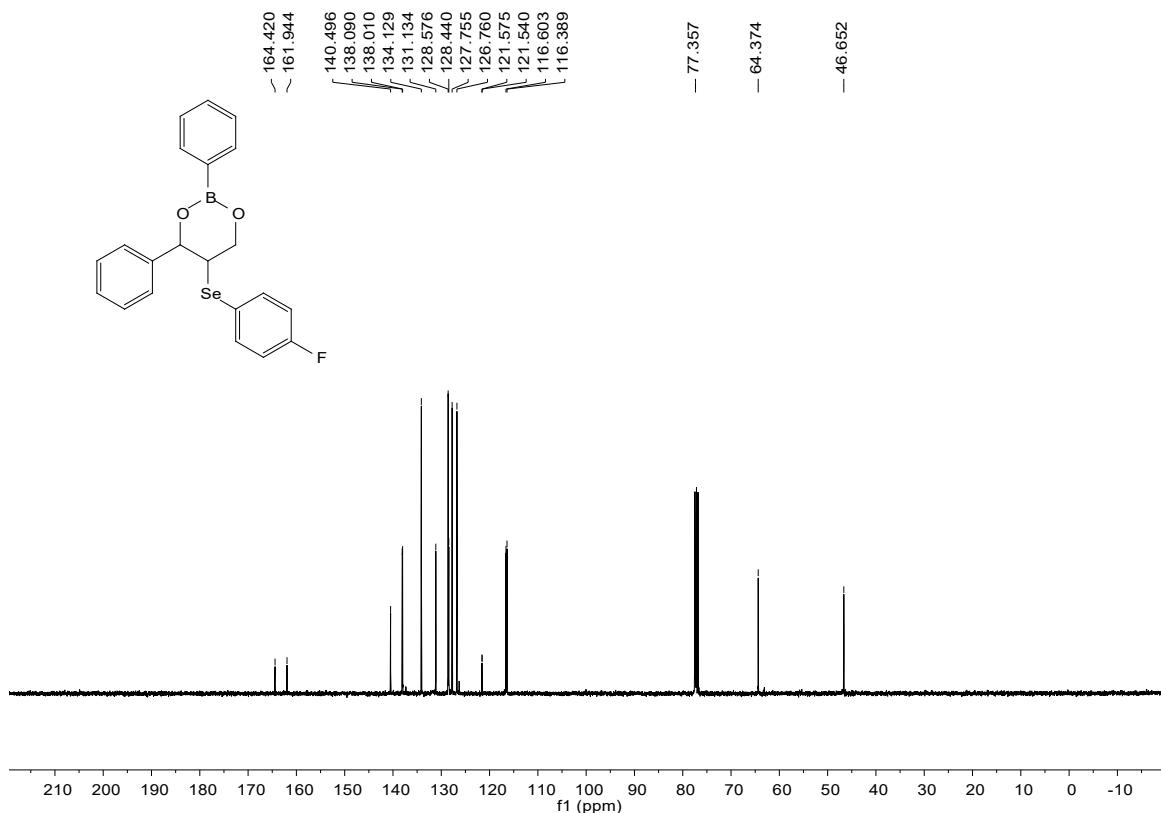
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5a**



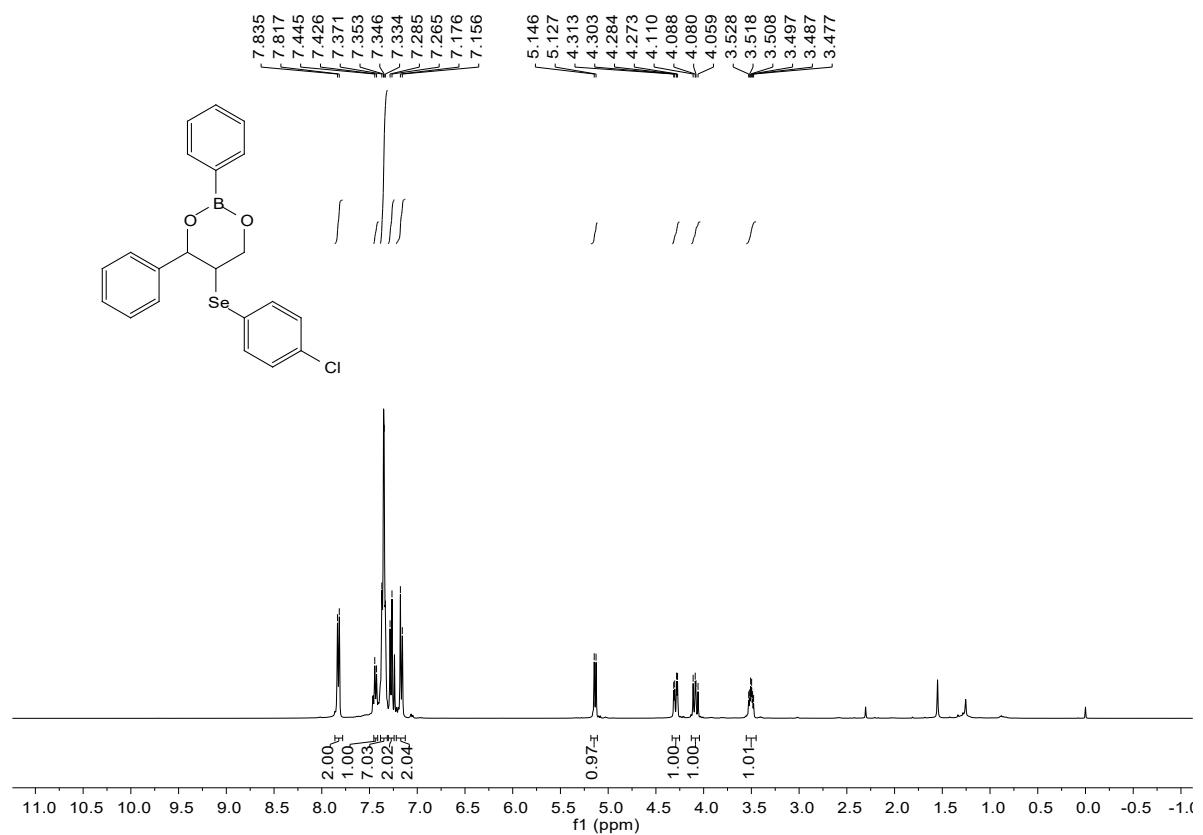
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5b**



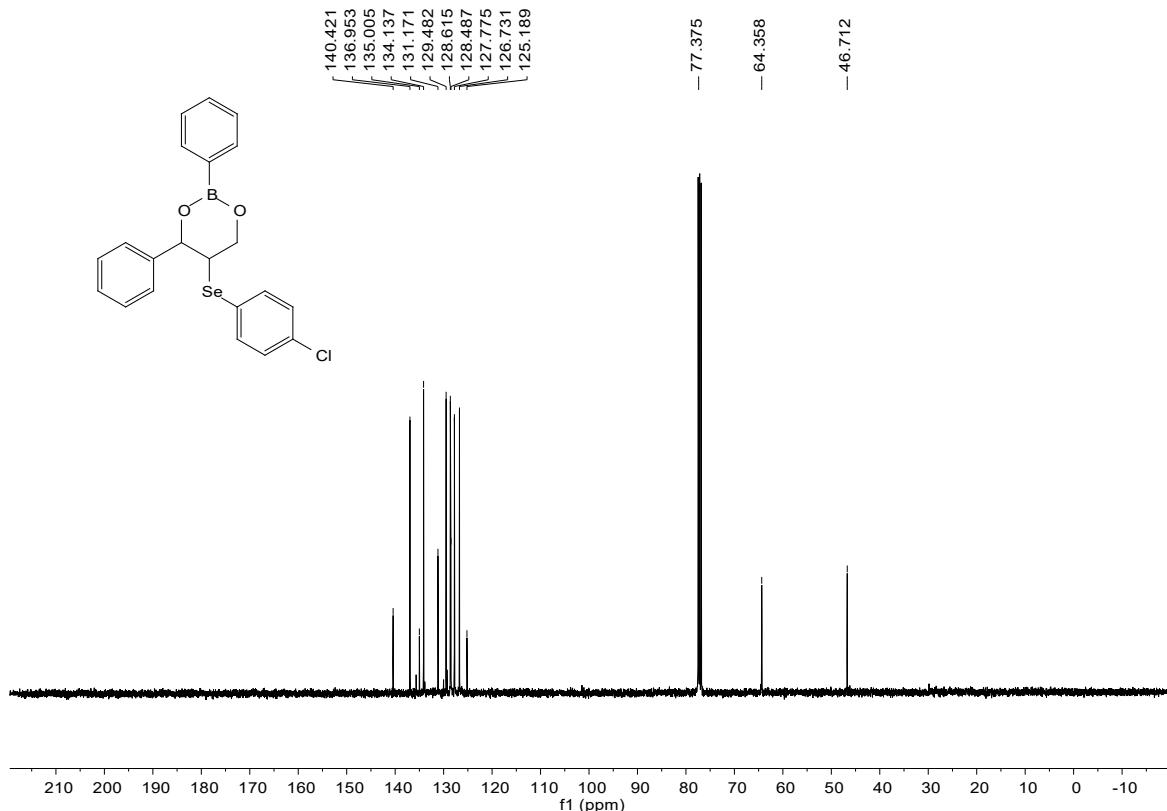
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5b**



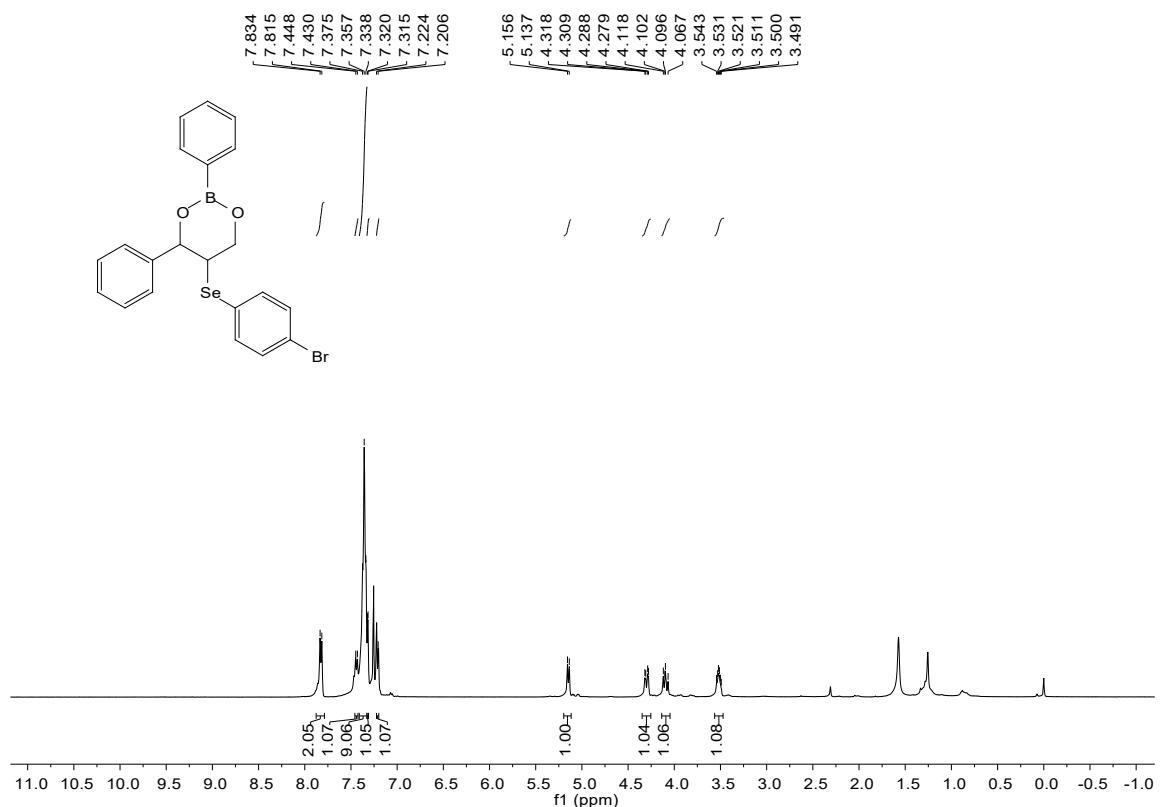
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5c**



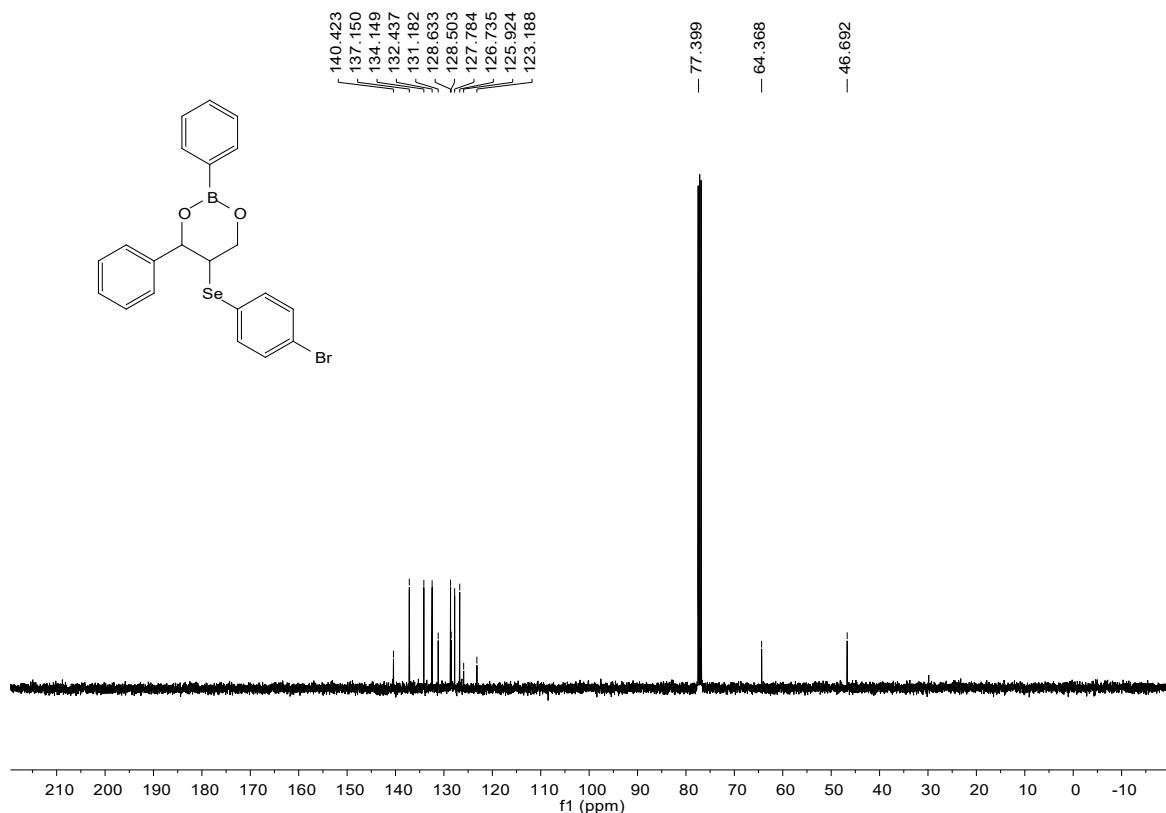
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5c**



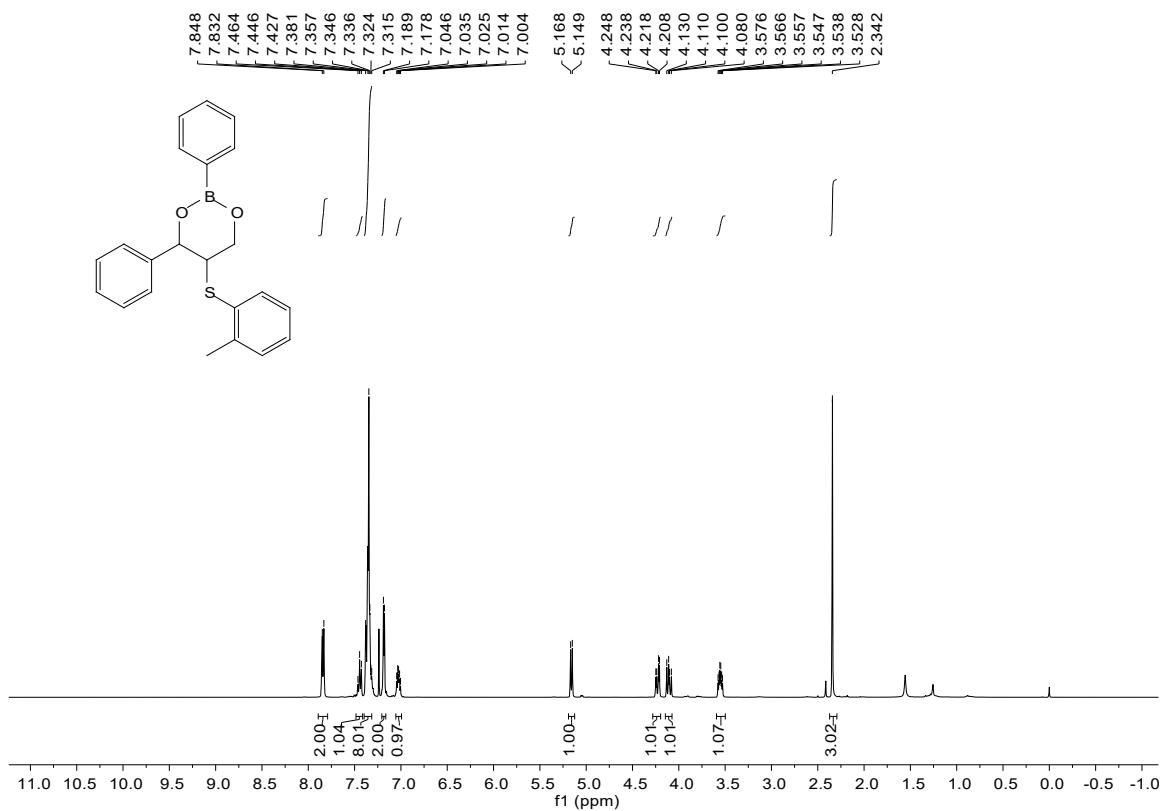
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5d**



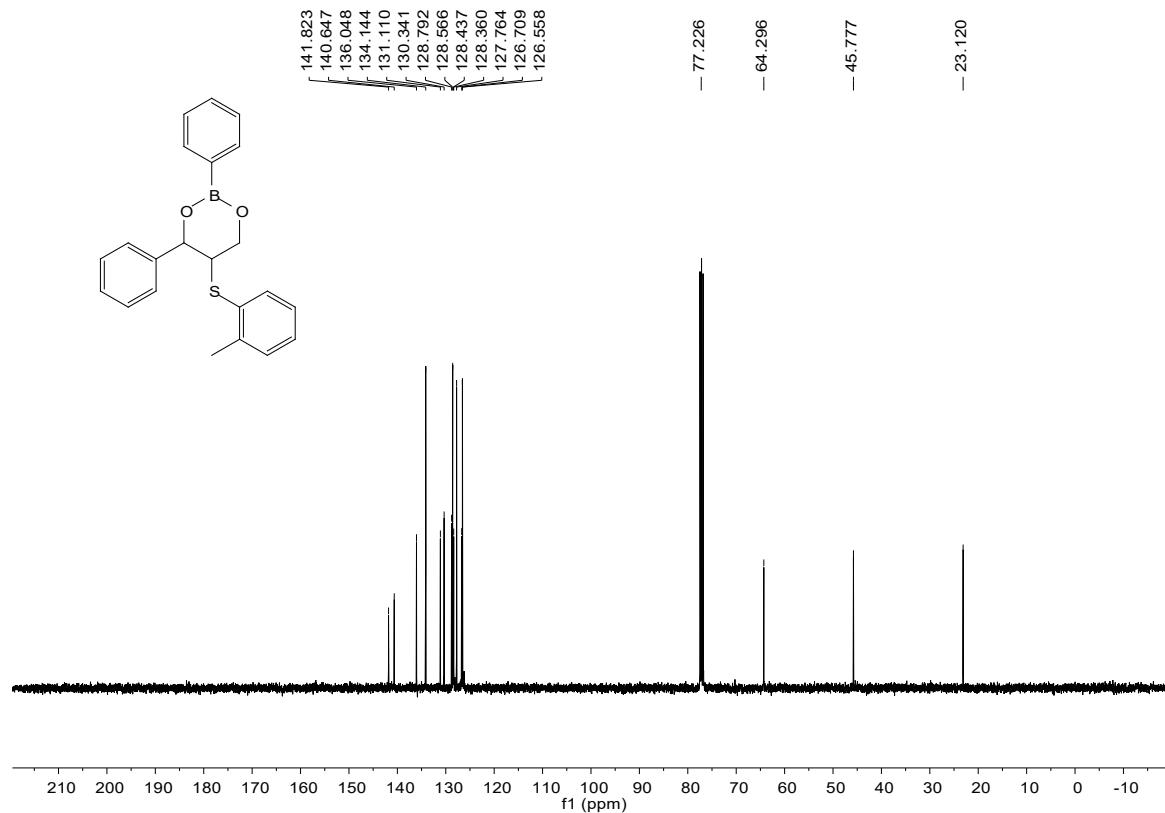
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5d**



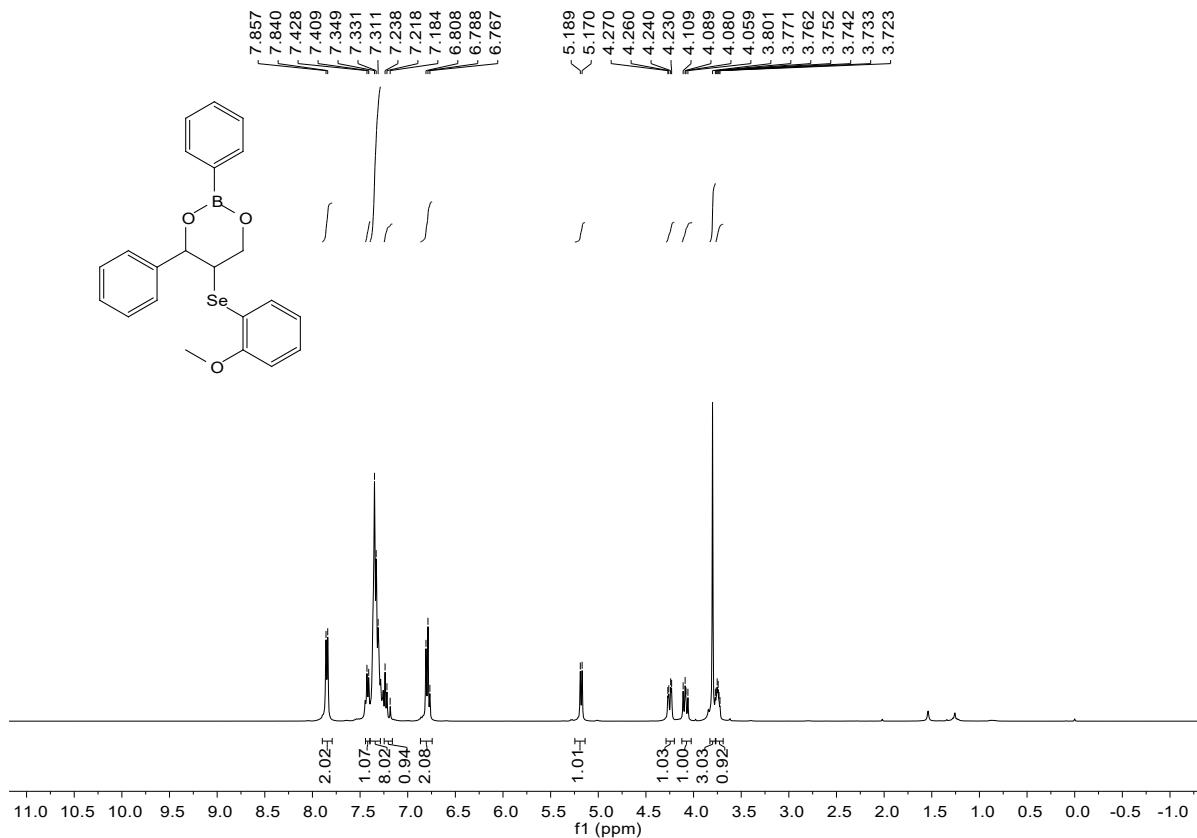
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5e**



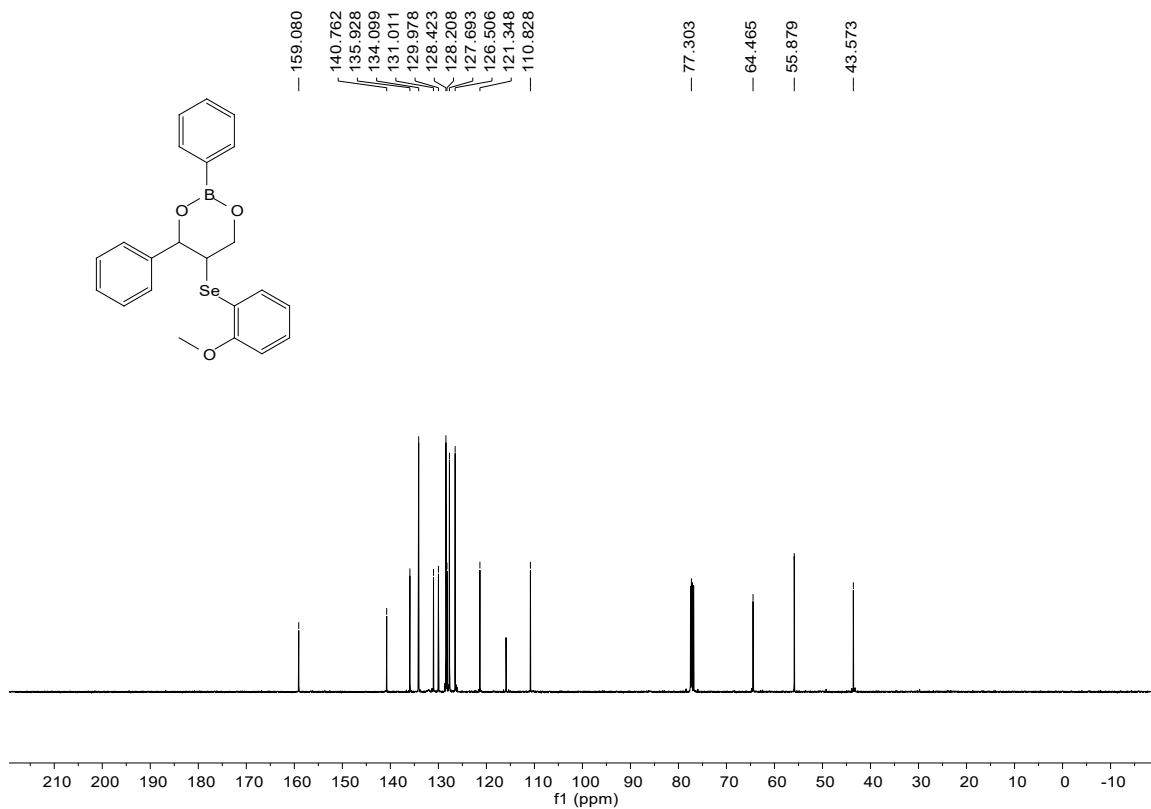
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5e**



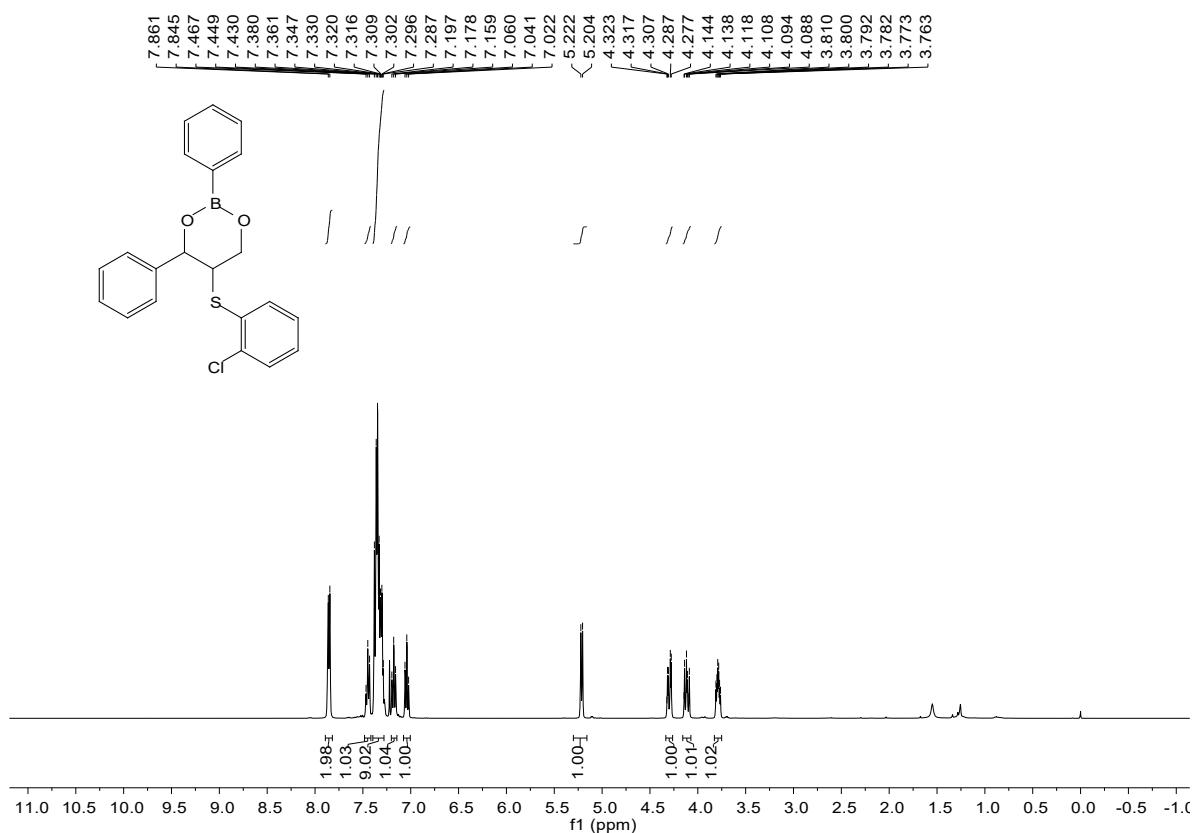
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5f**



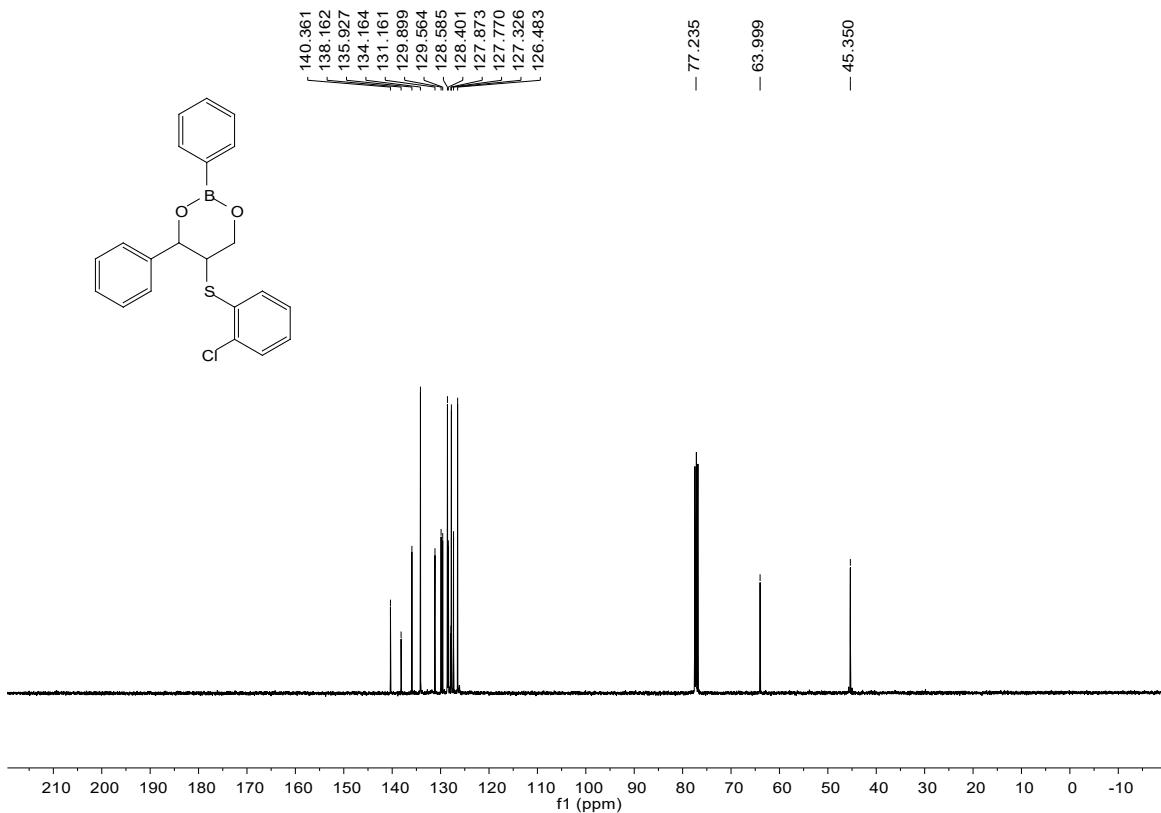
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5f**



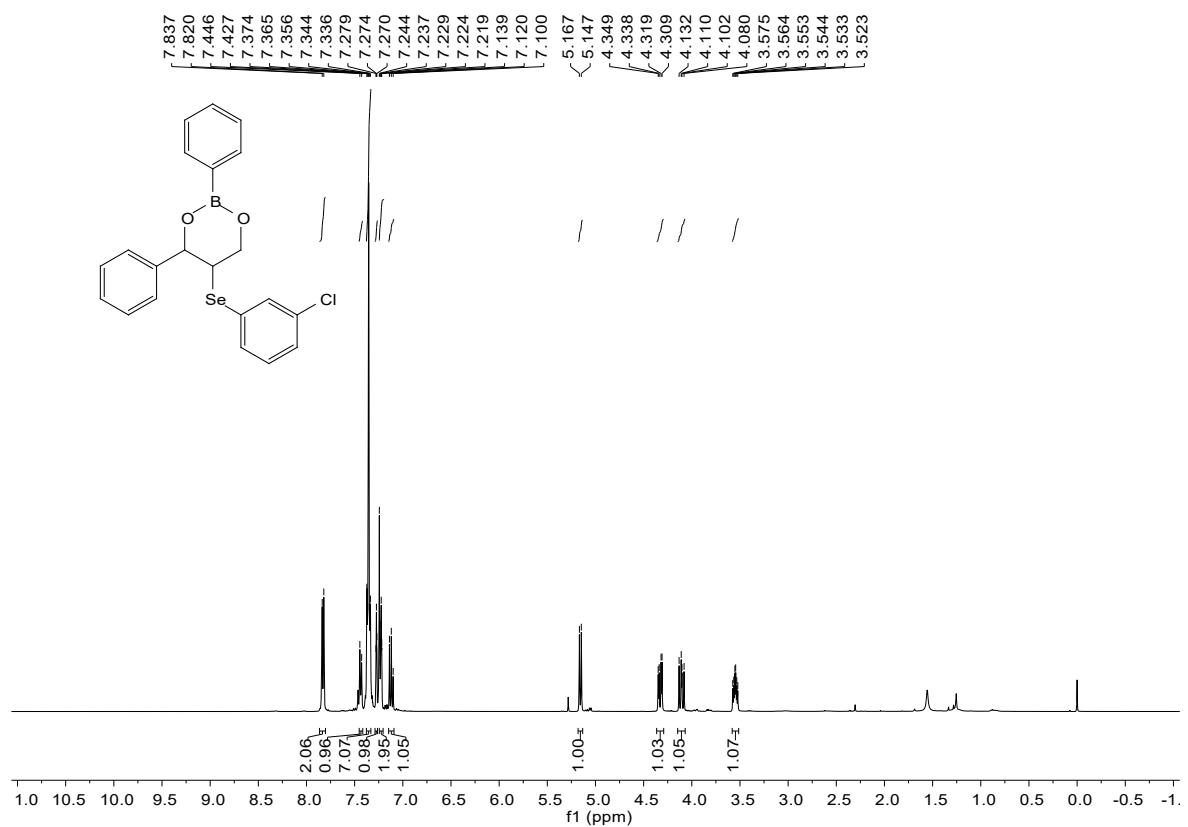
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5g**



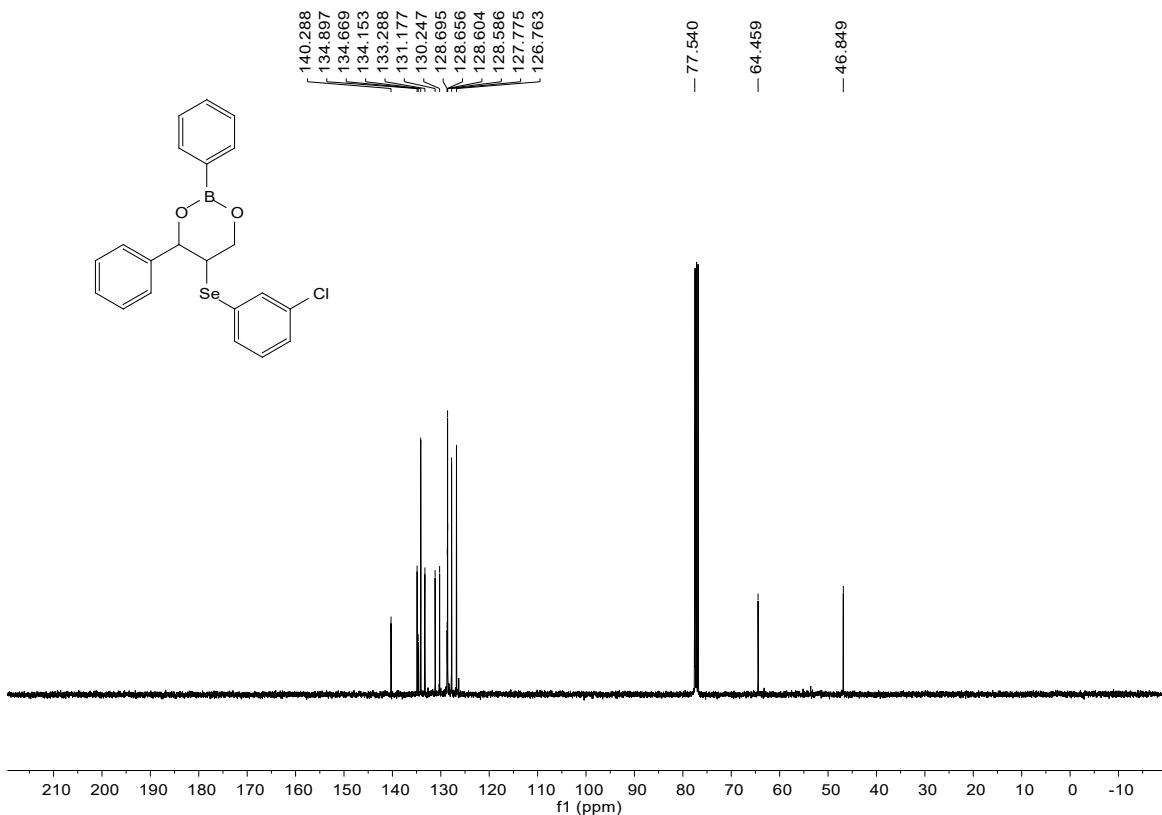
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5g**



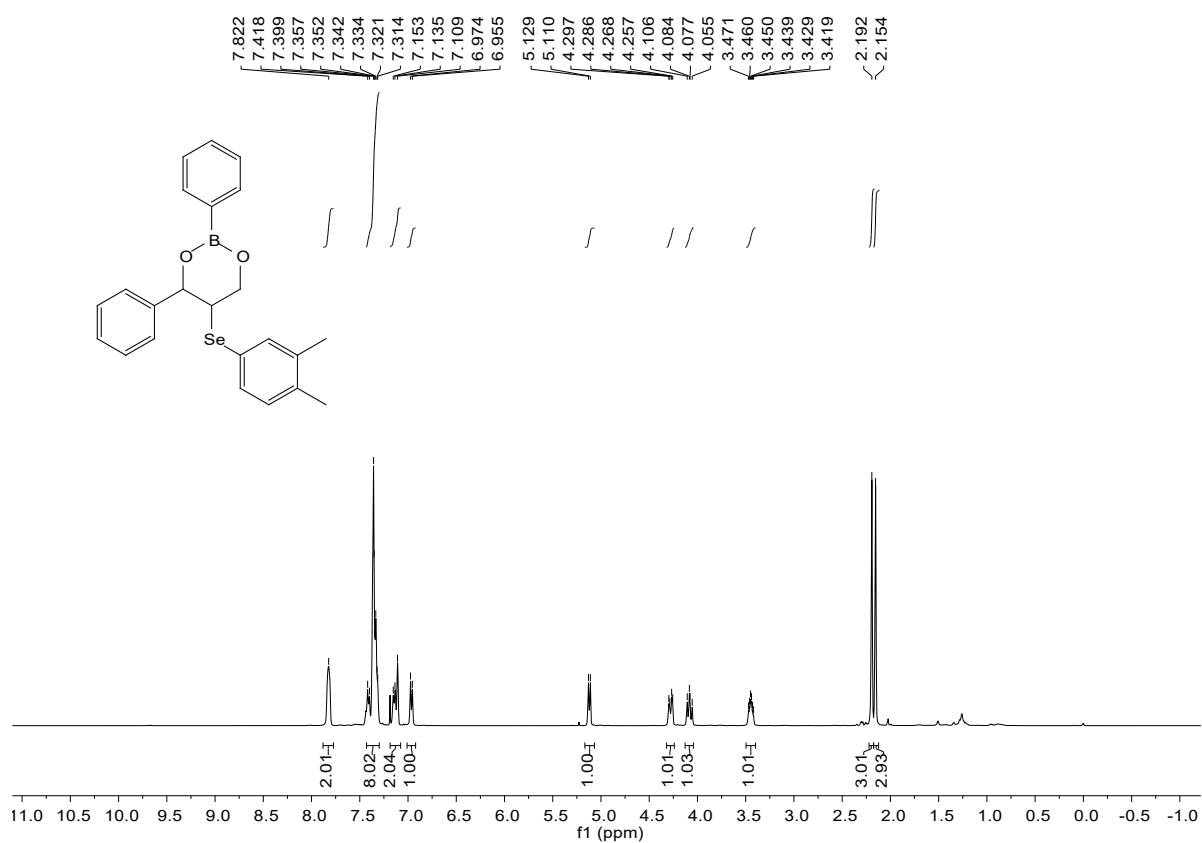
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5h**



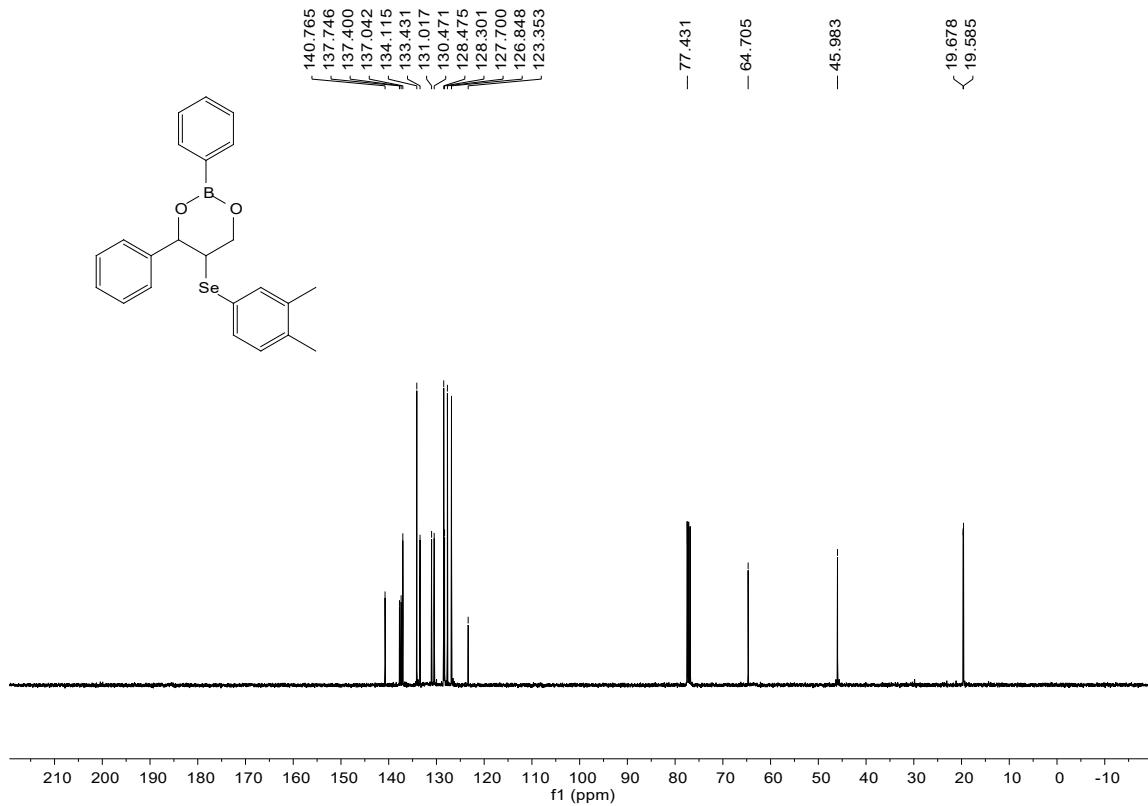
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5h**



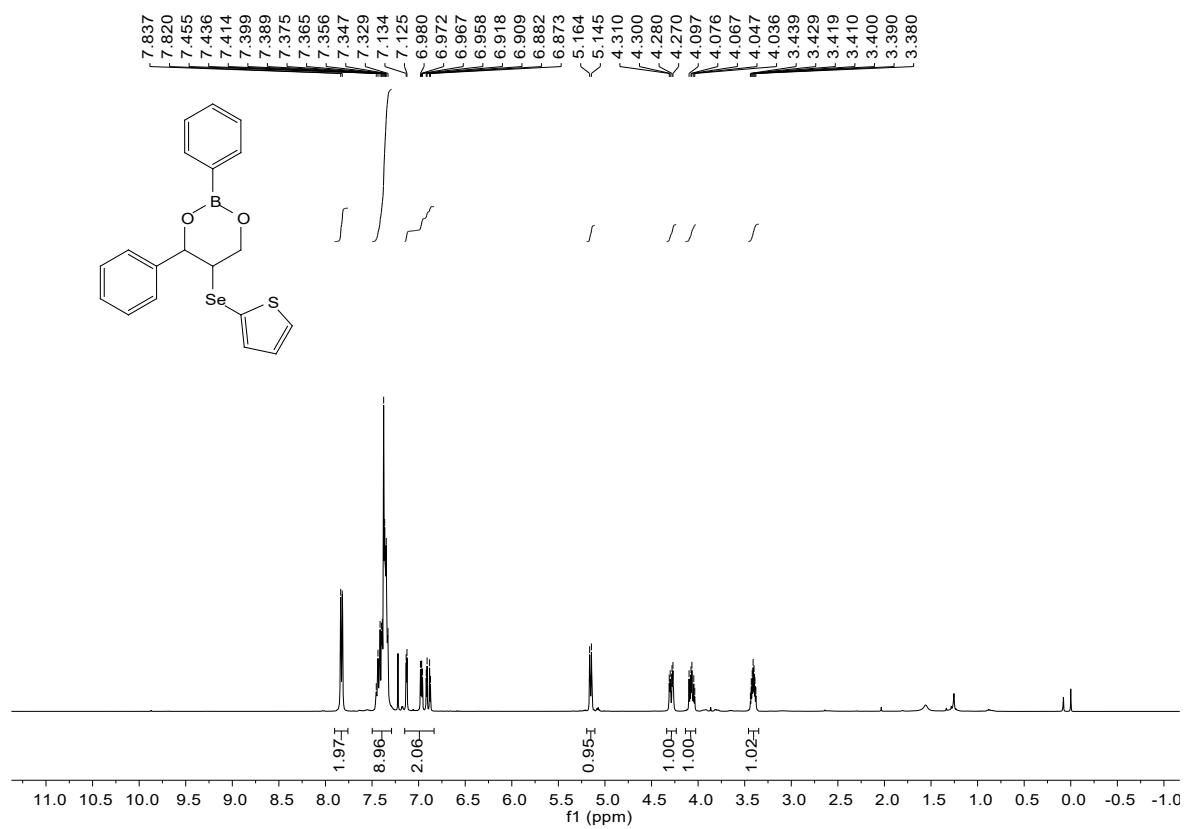
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5i**



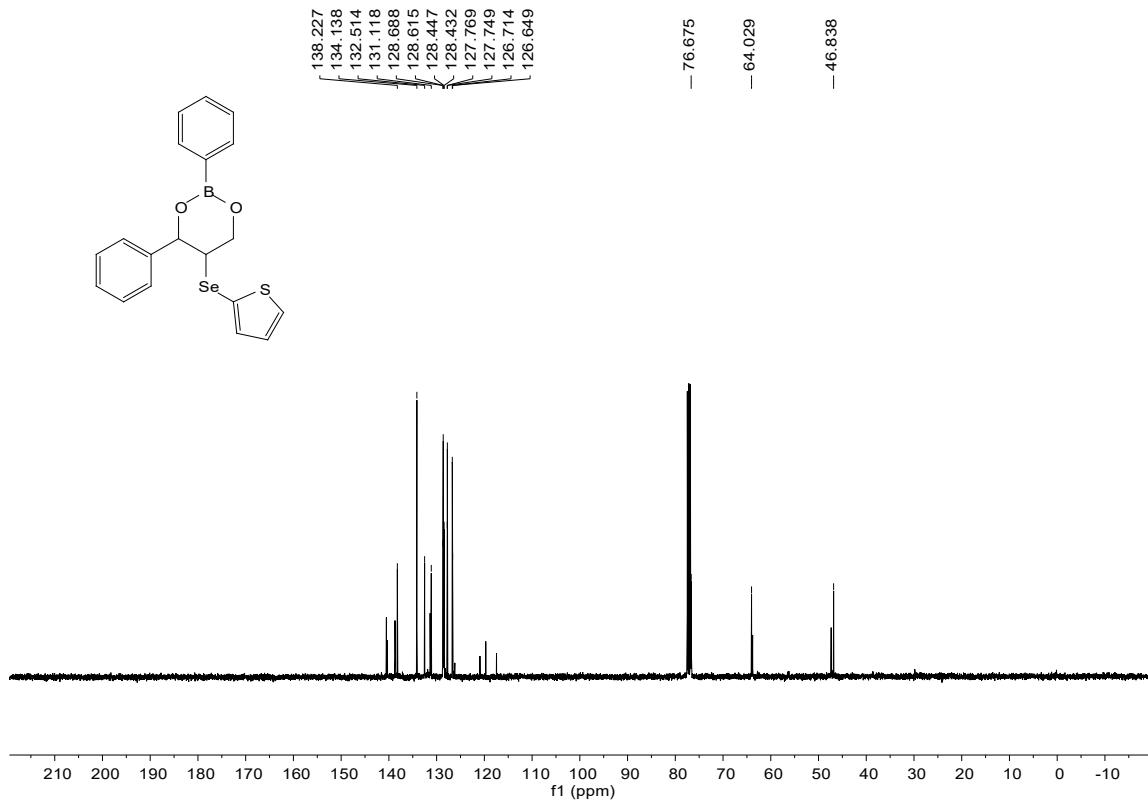
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5i**



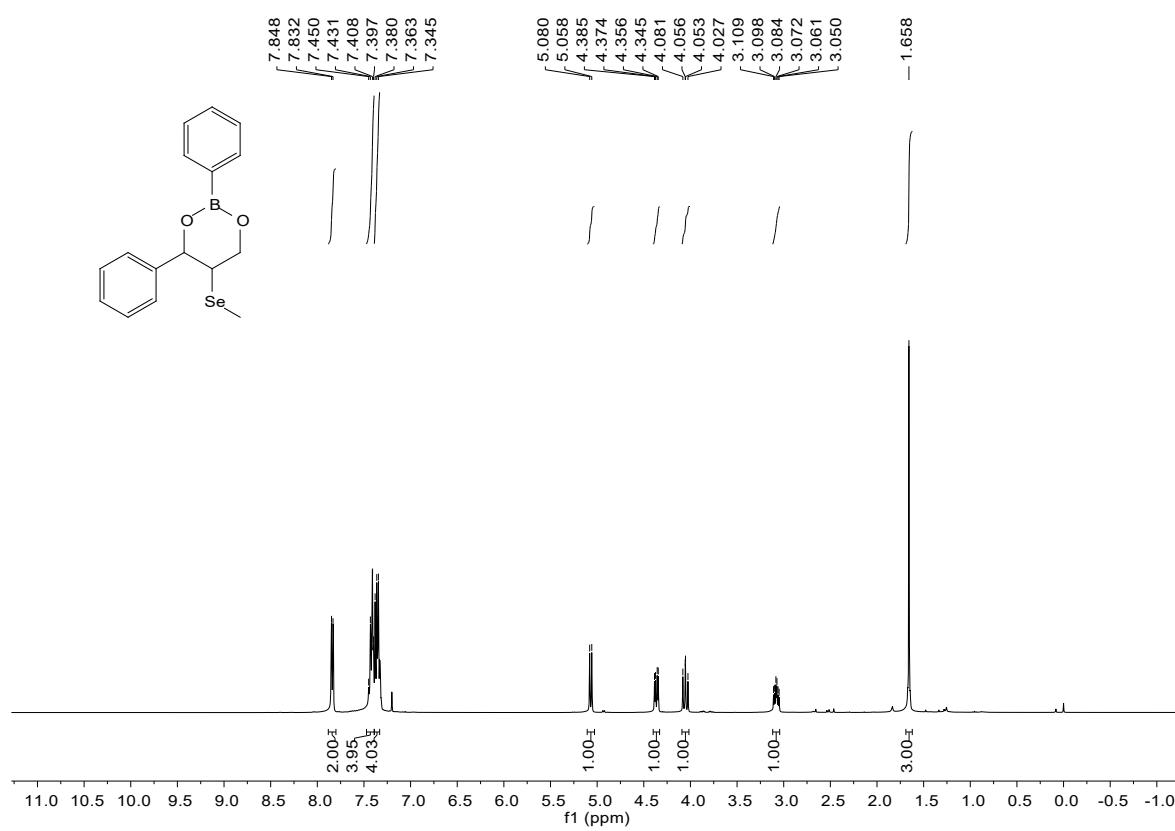
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5j**



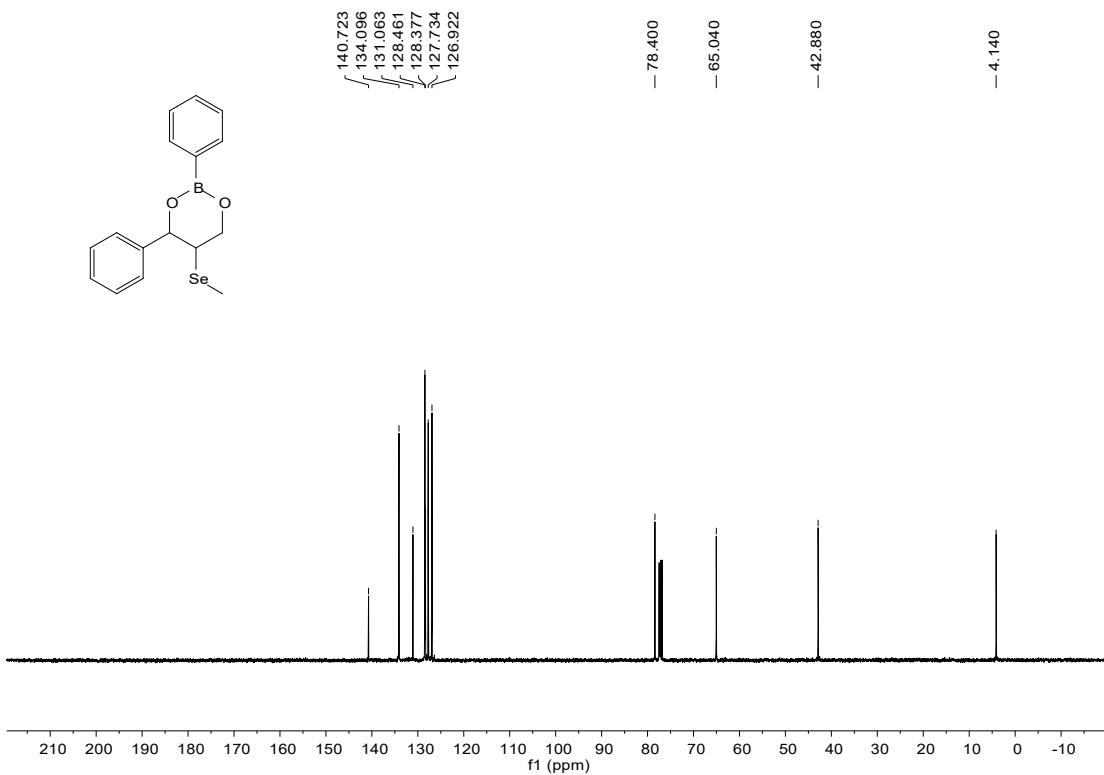
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5j**



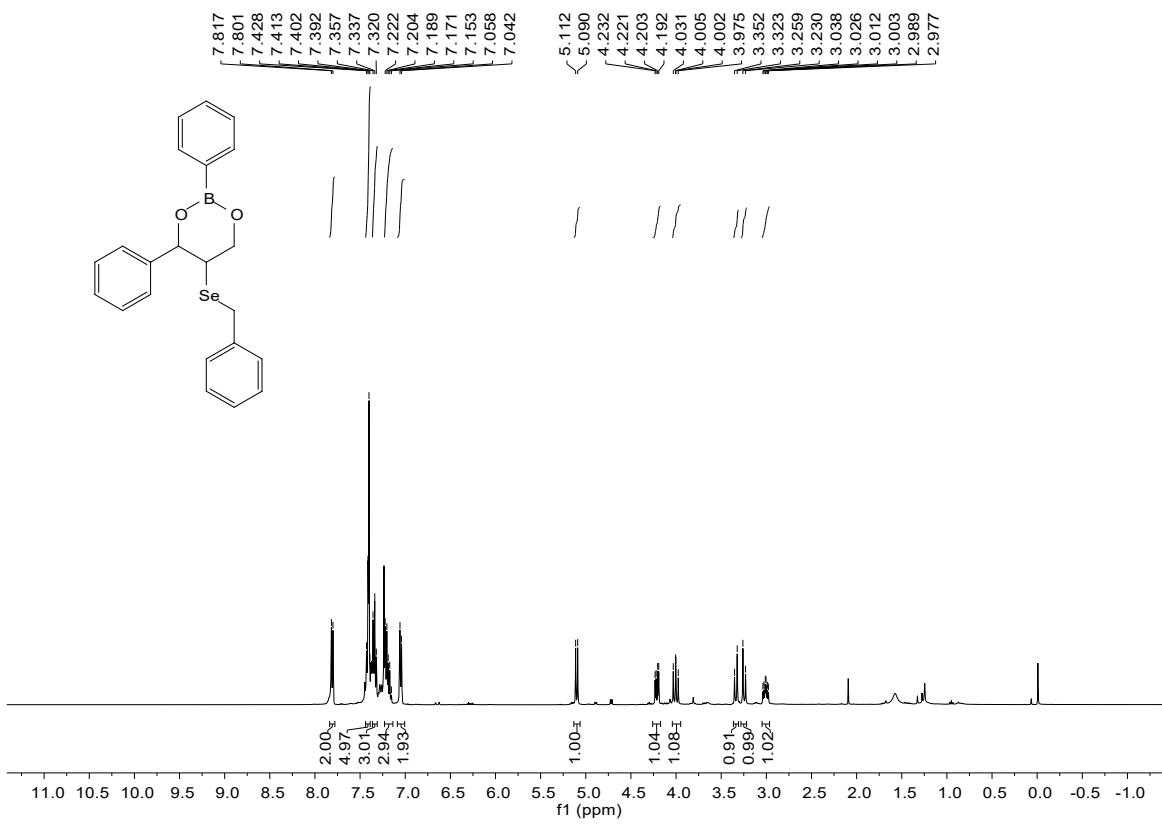
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5k**



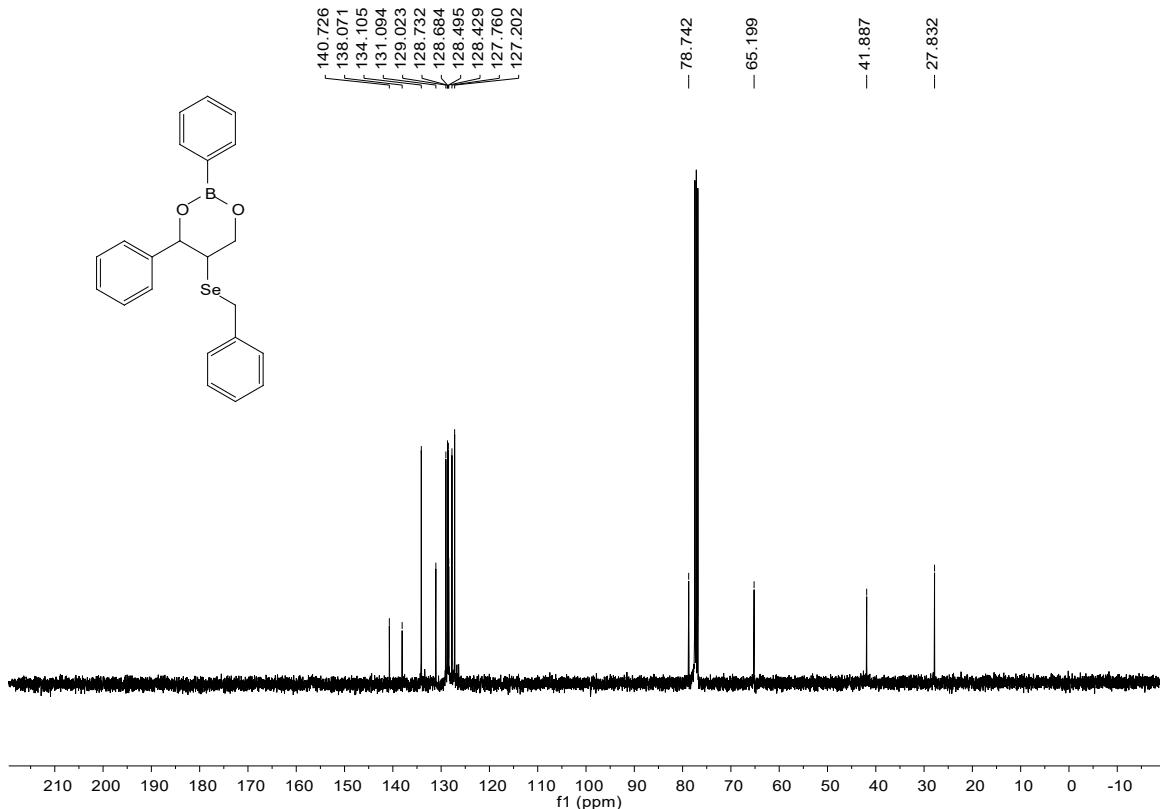
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5k**



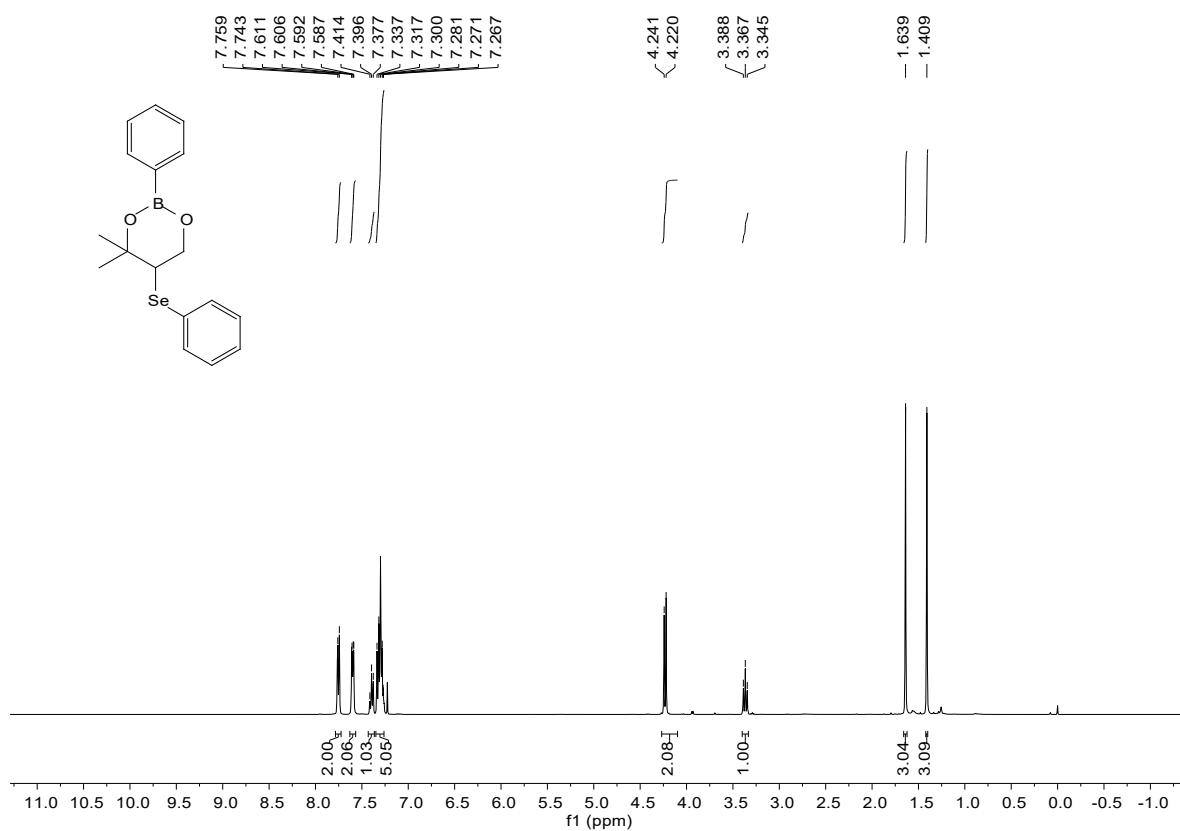
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5l**



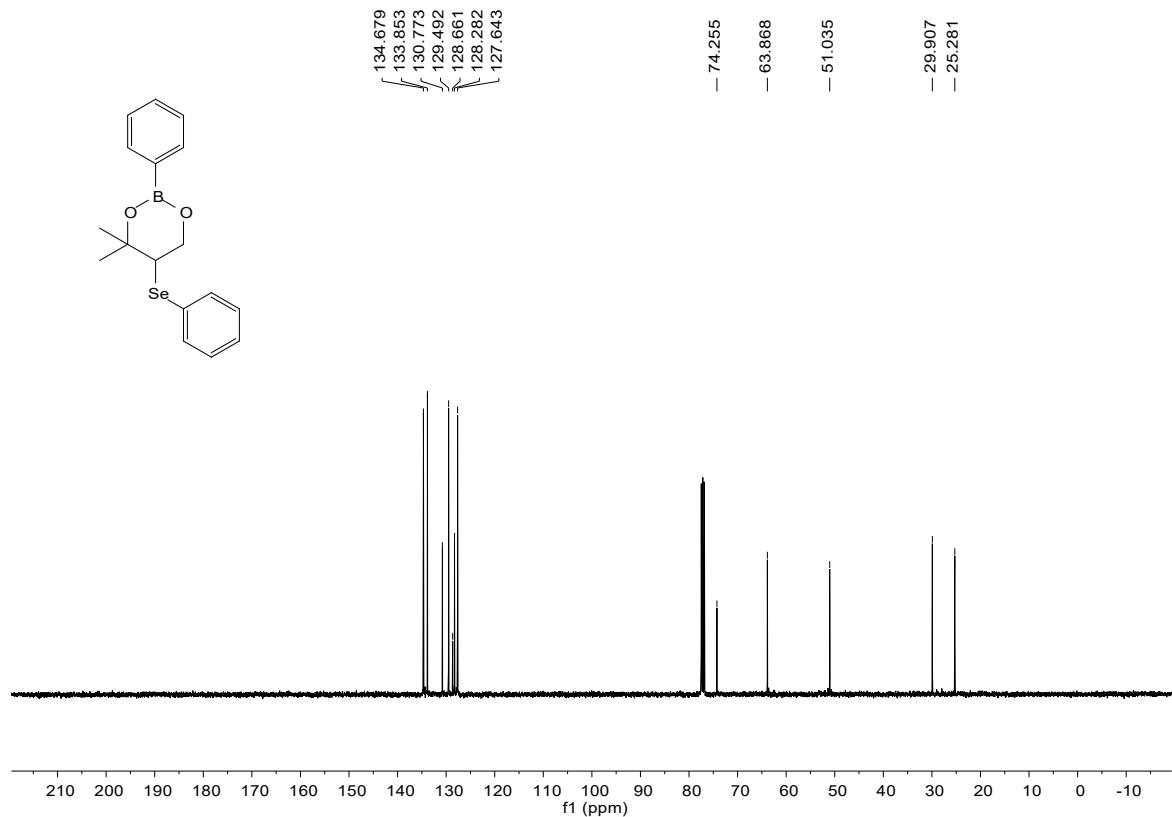
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5l**



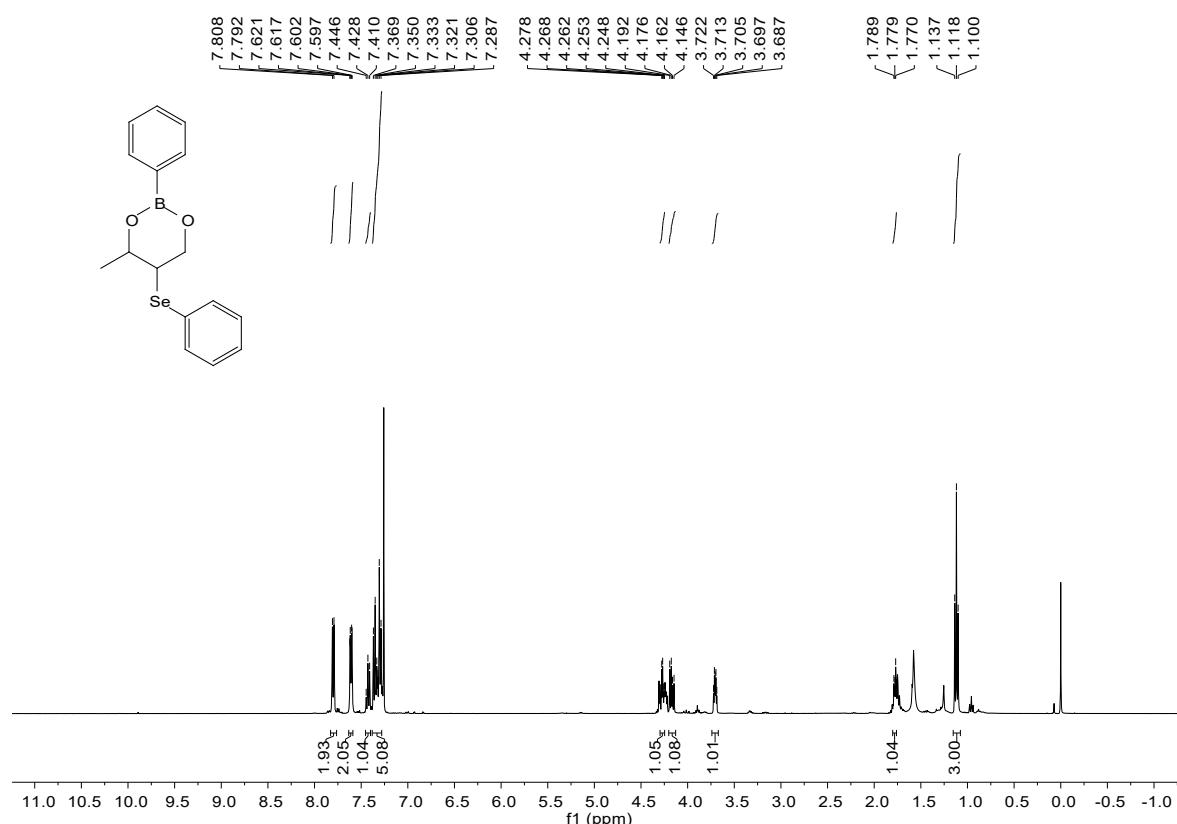
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5m**



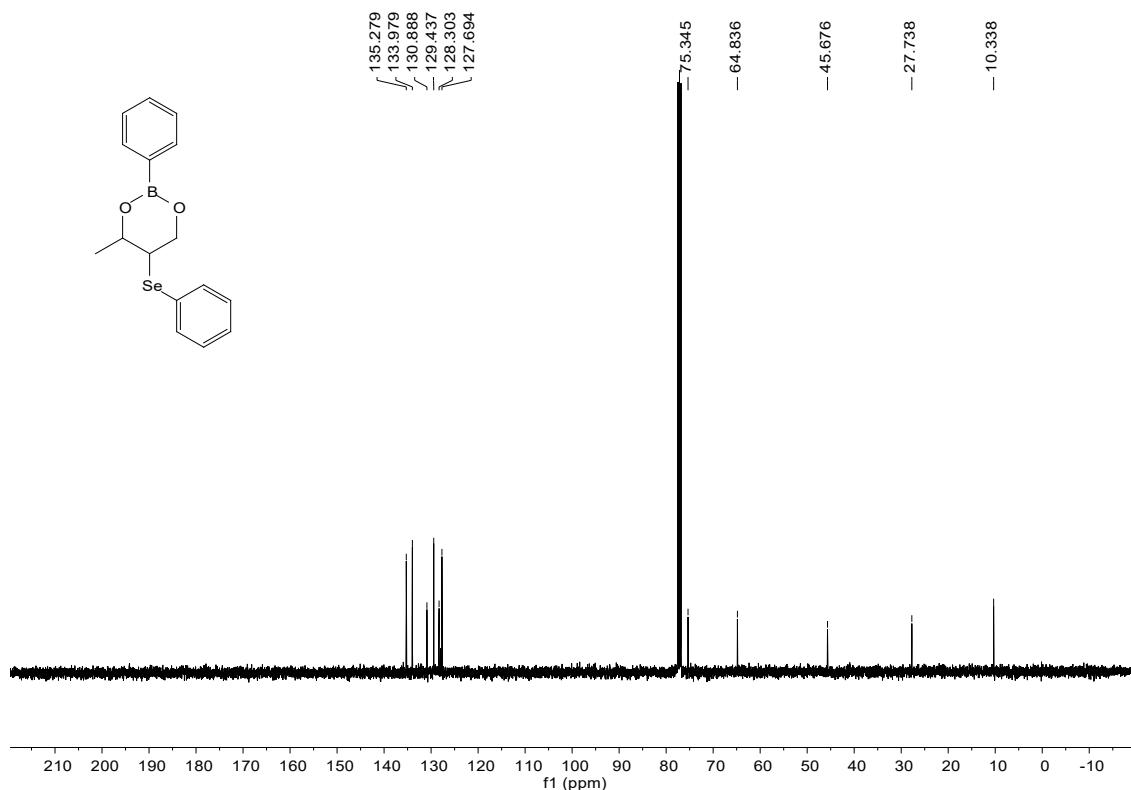
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5m**



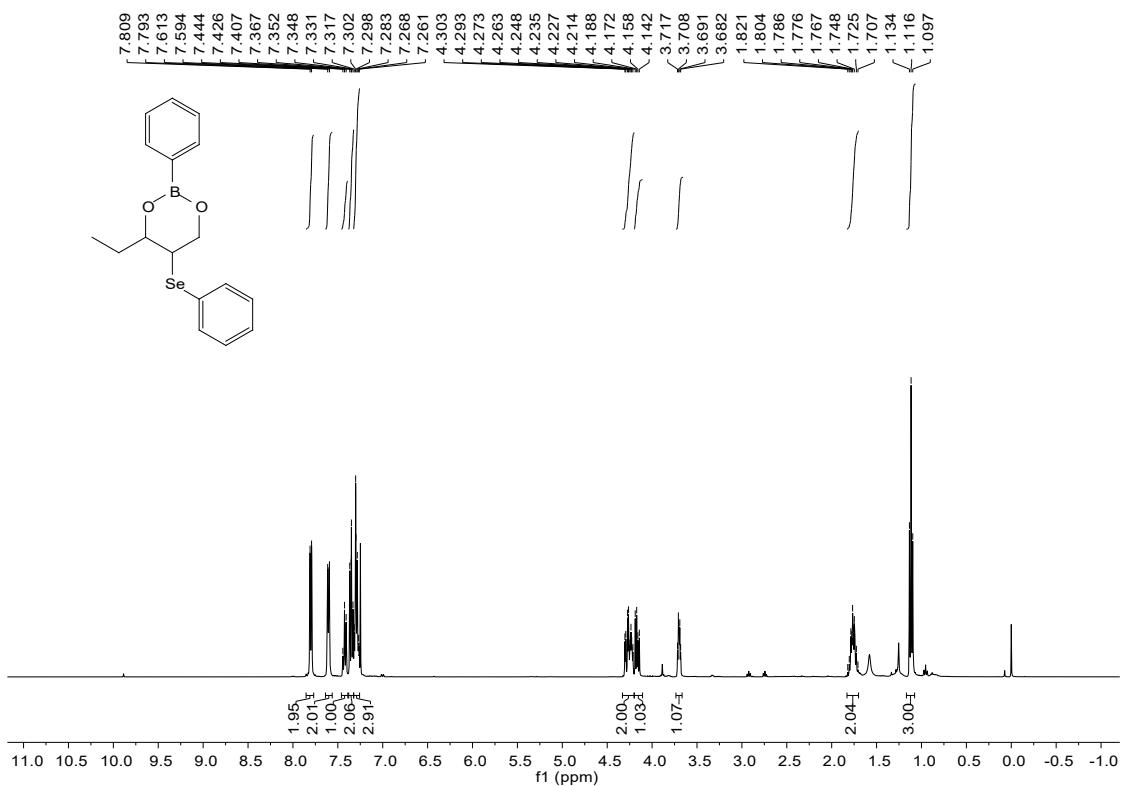
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5n**



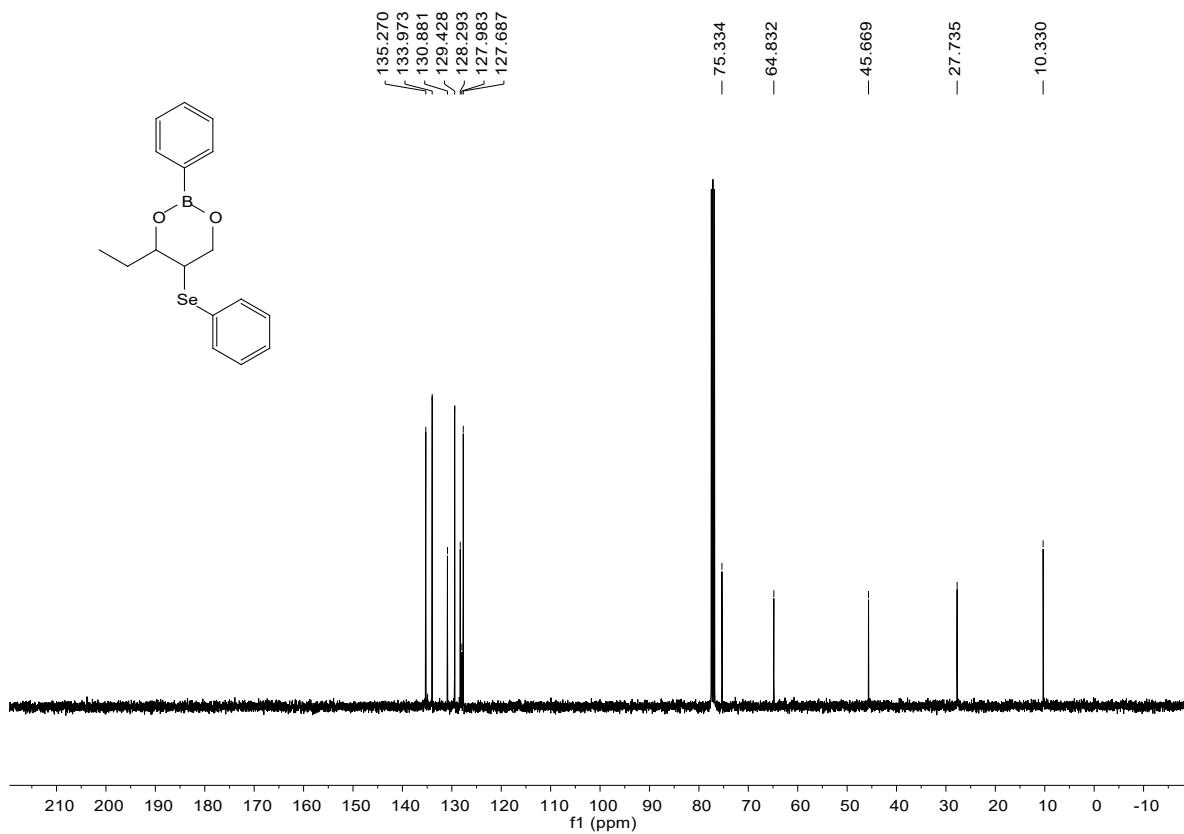
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5n**



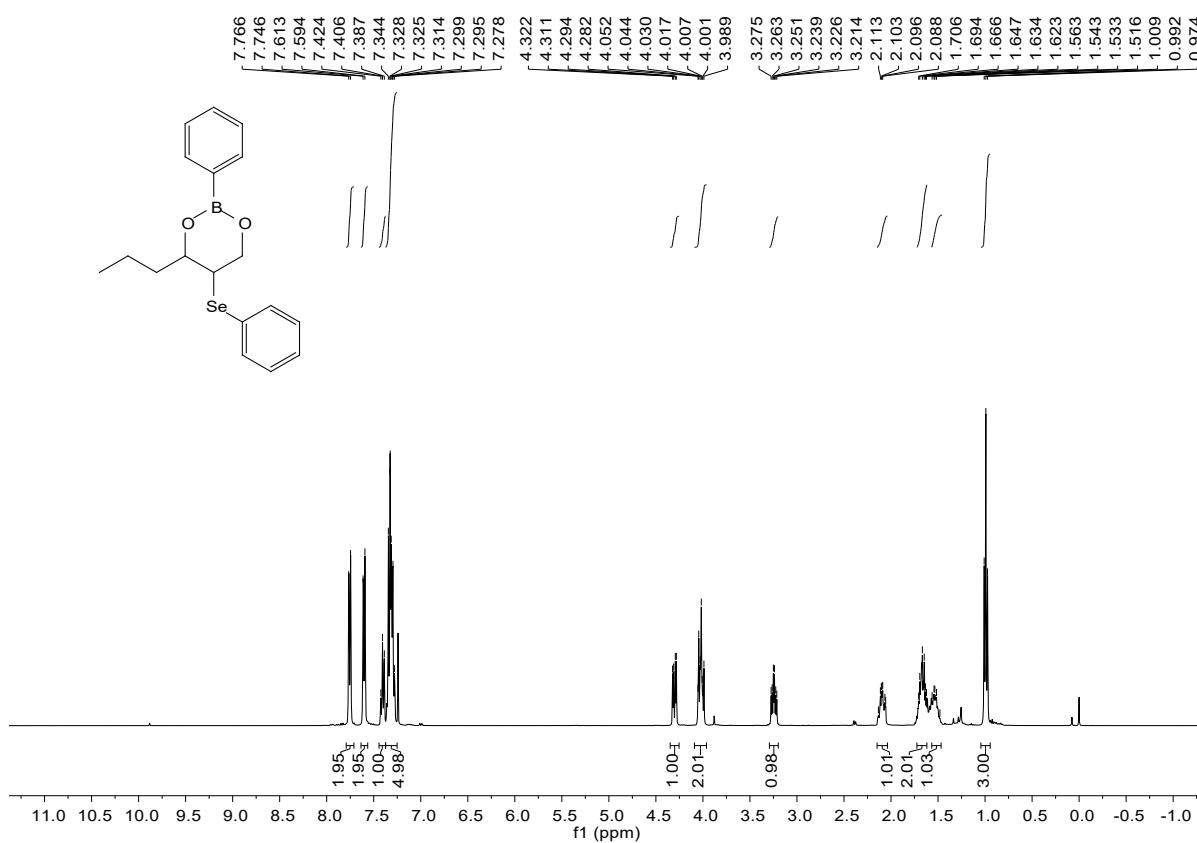
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5o**



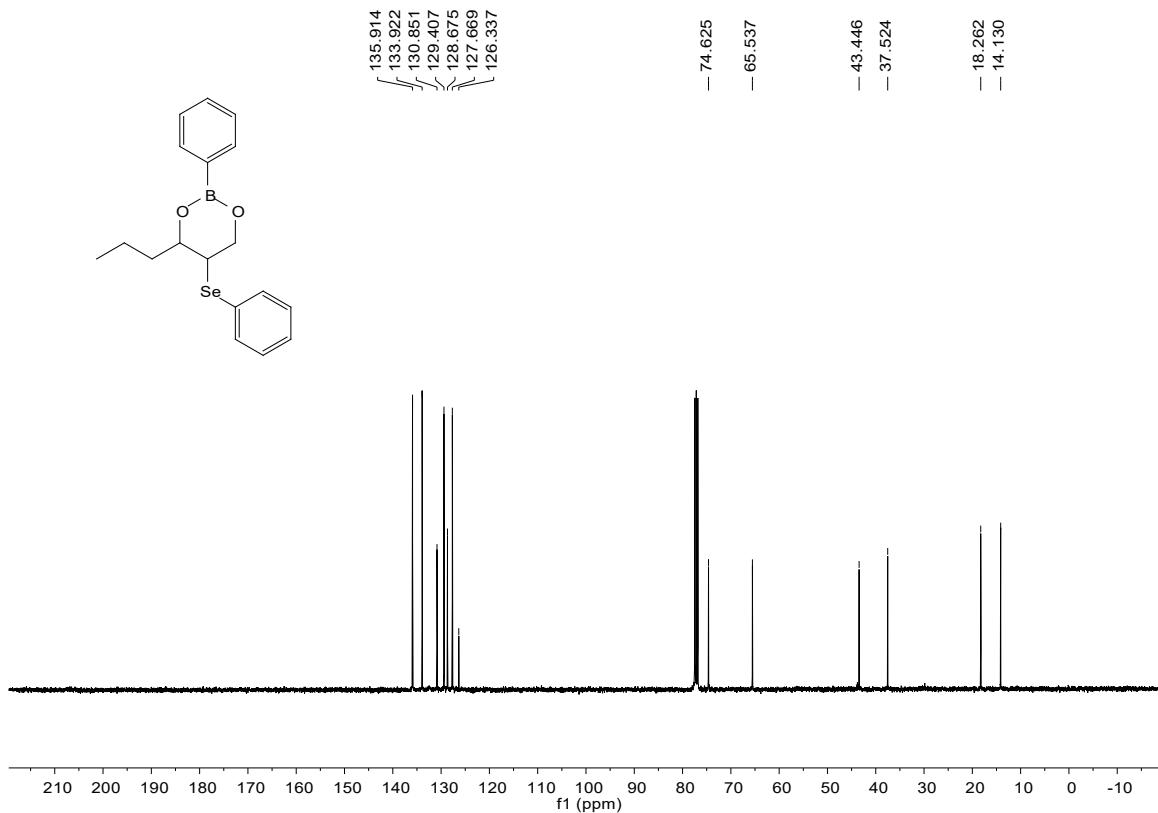
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5o**



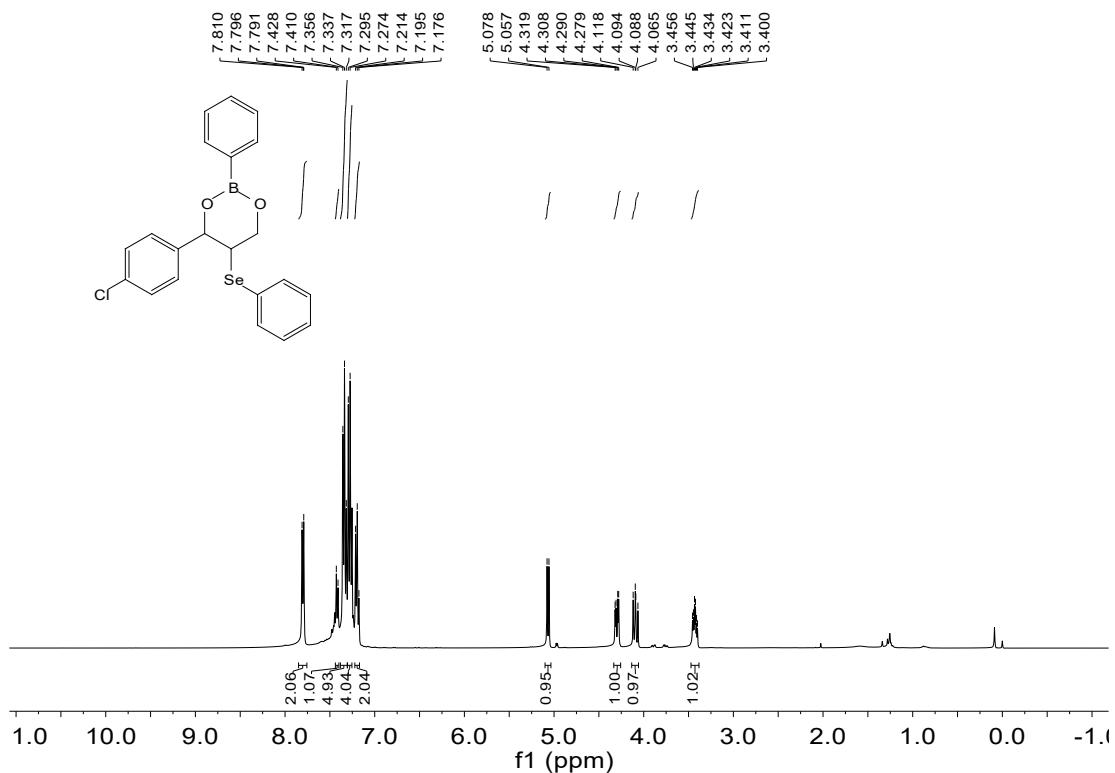
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5p**



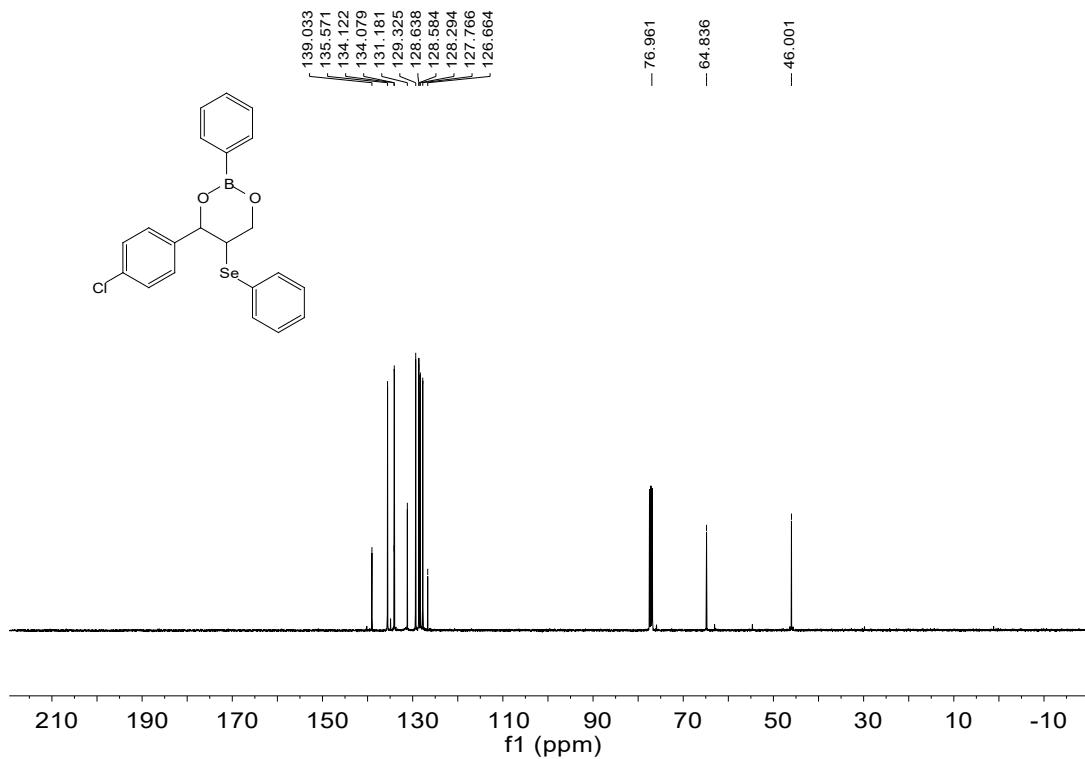
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5p**



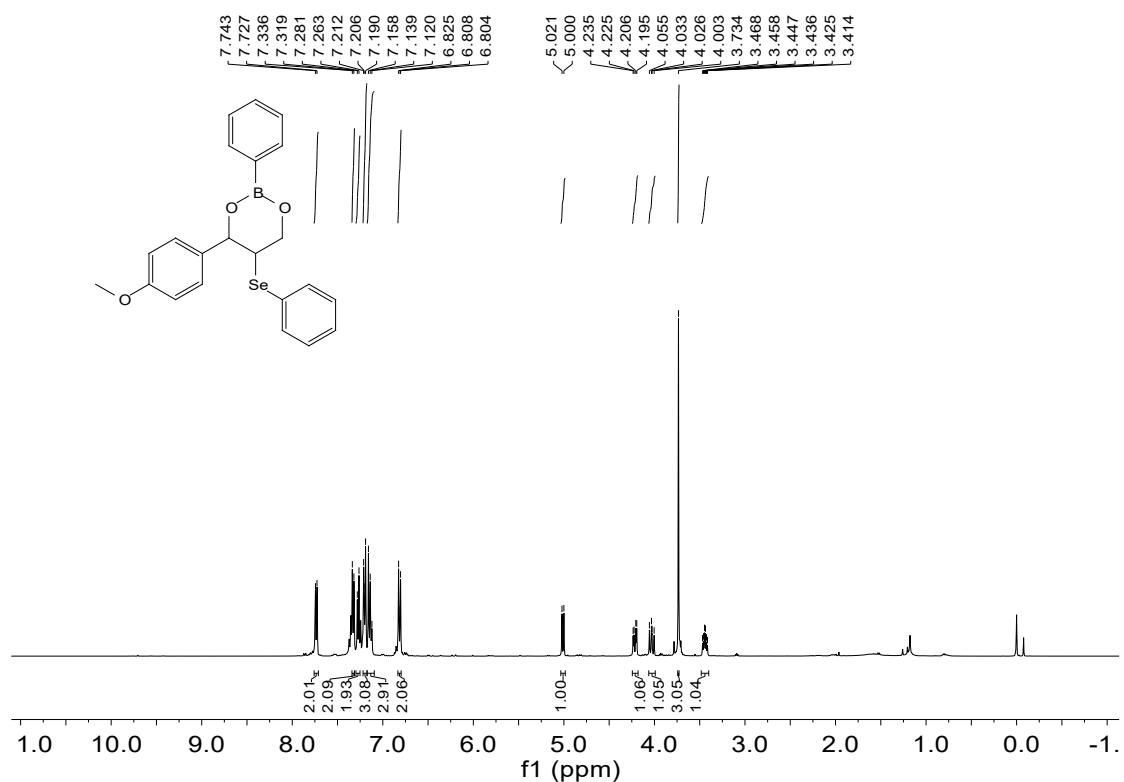
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5q**



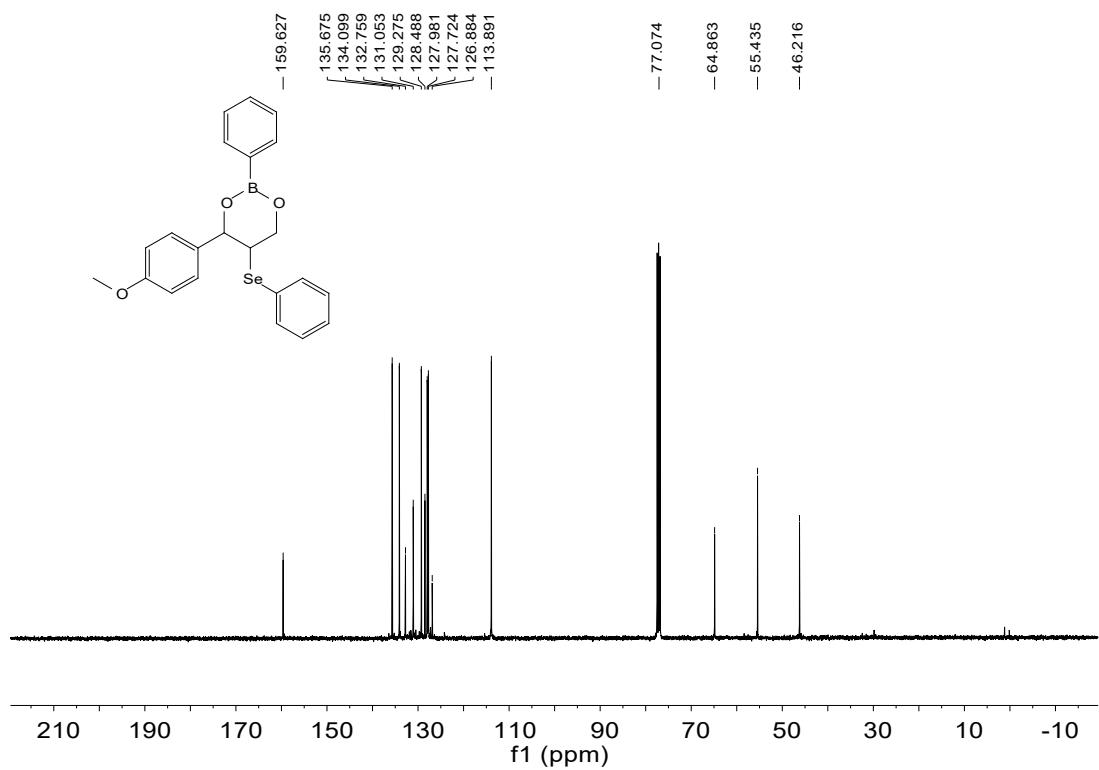
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5q**



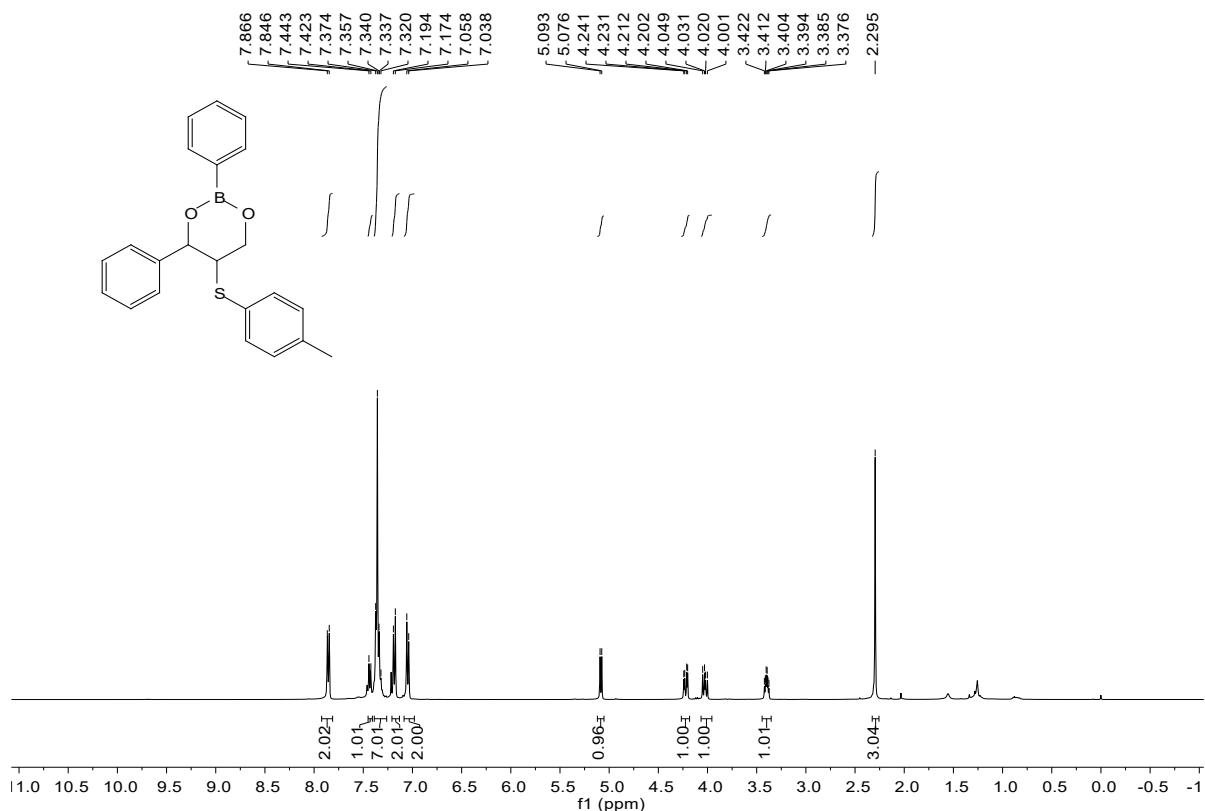
¹H NMR (400 MHz, CDCl₃) spectrum of compound **5r**



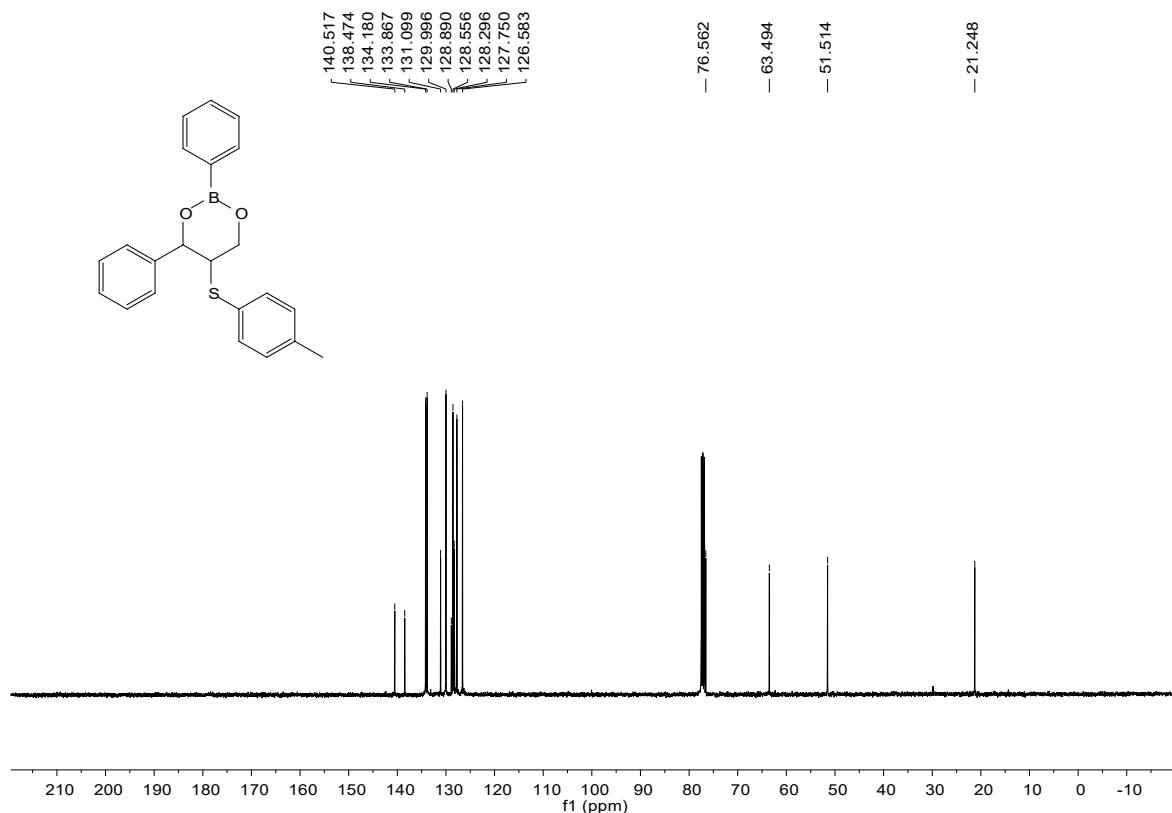
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **5r**



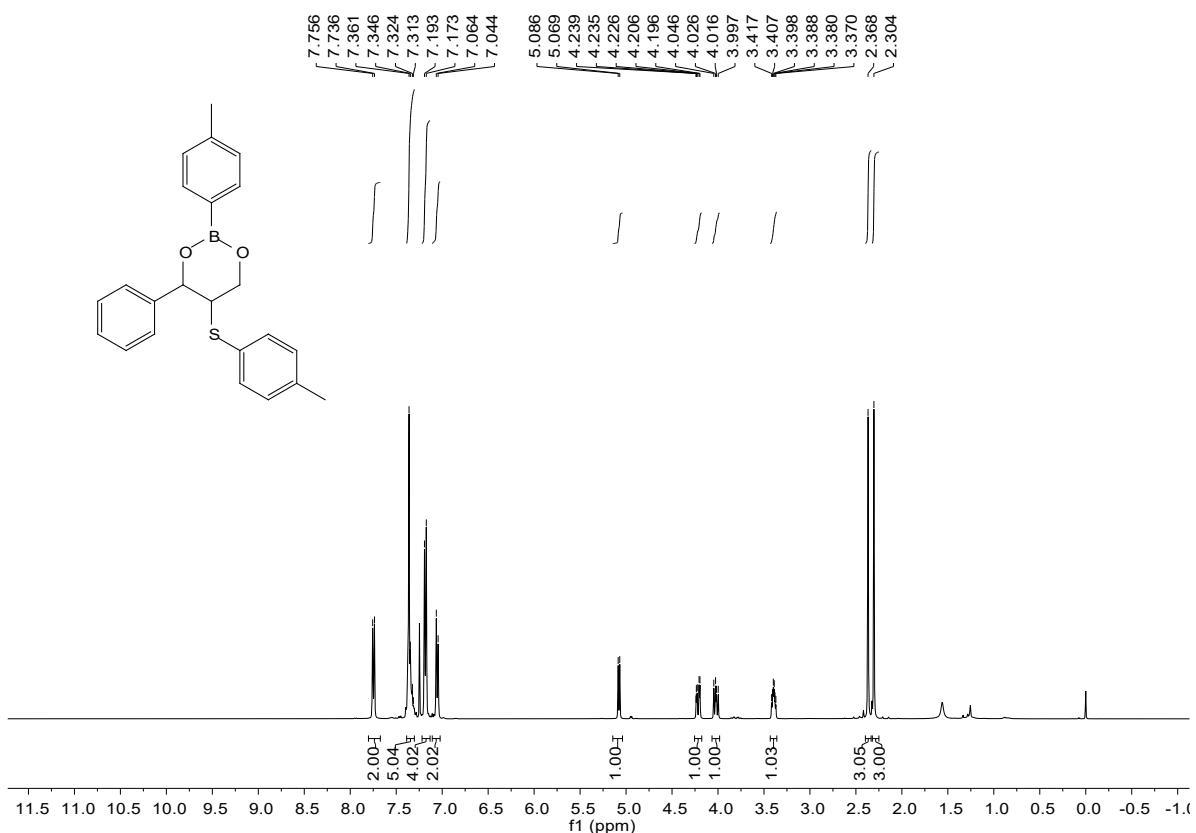
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6a**



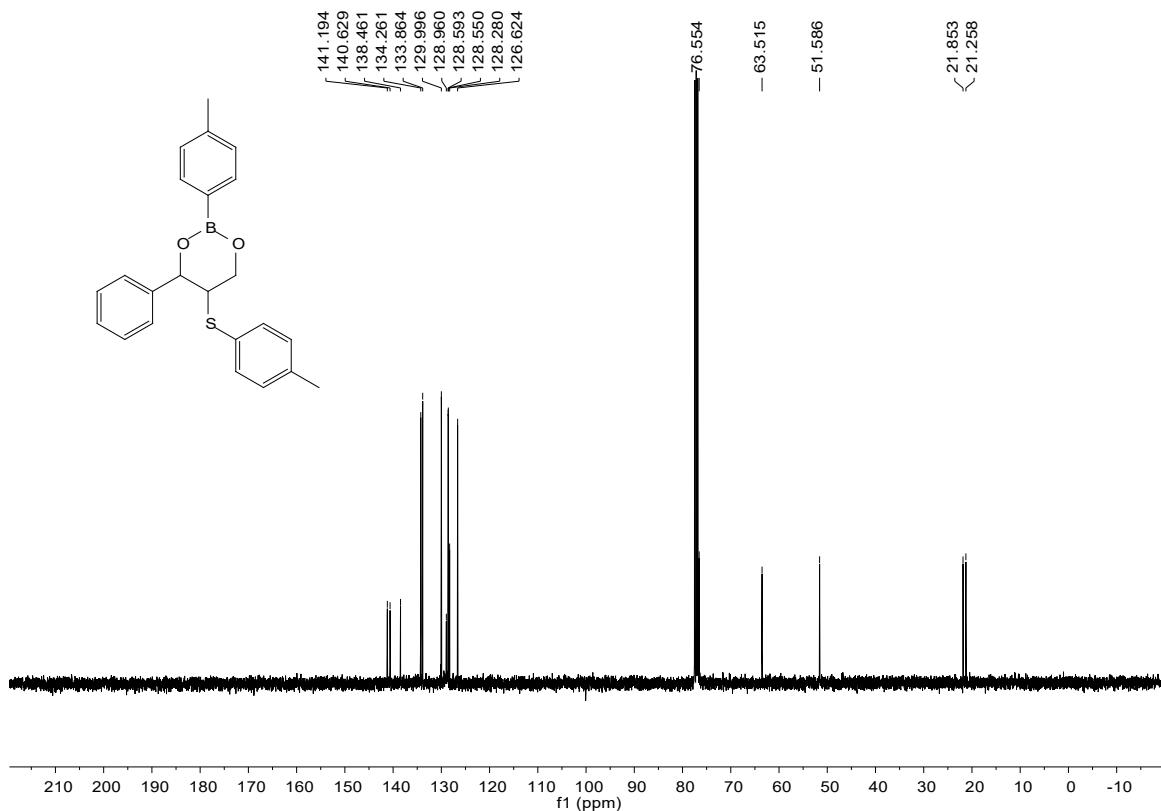
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6a**



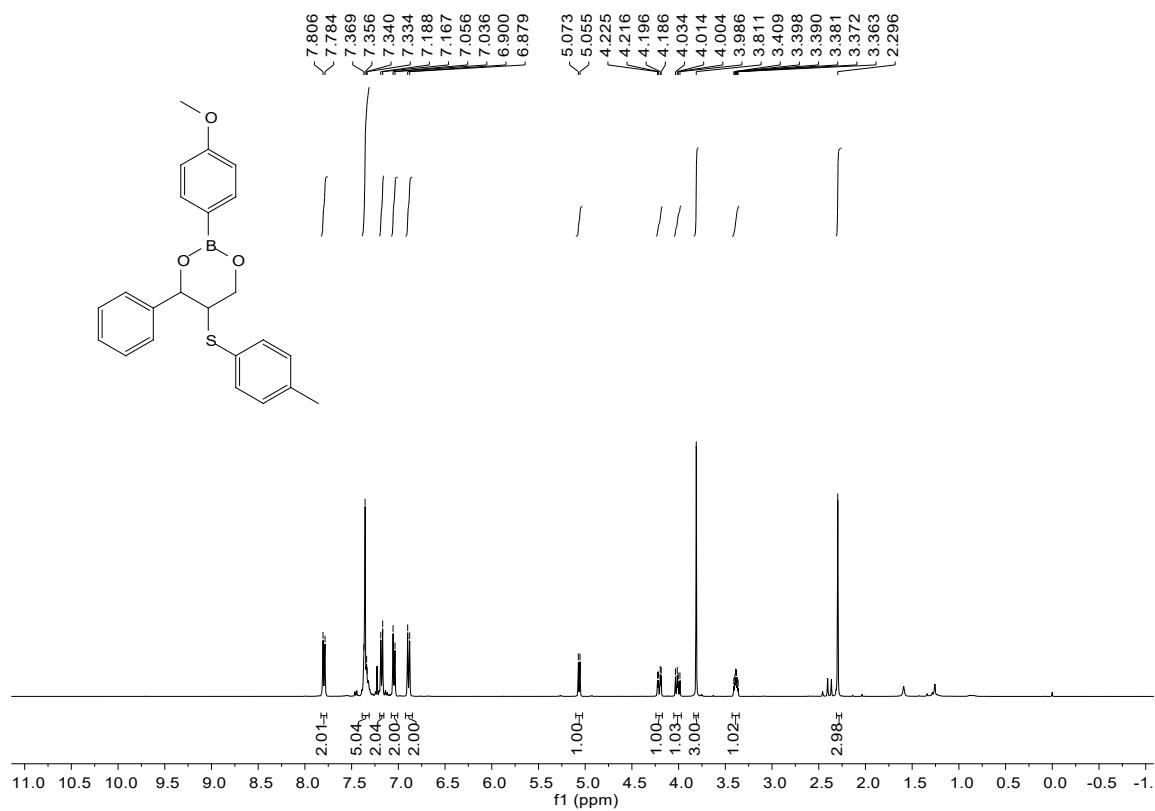
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6b**



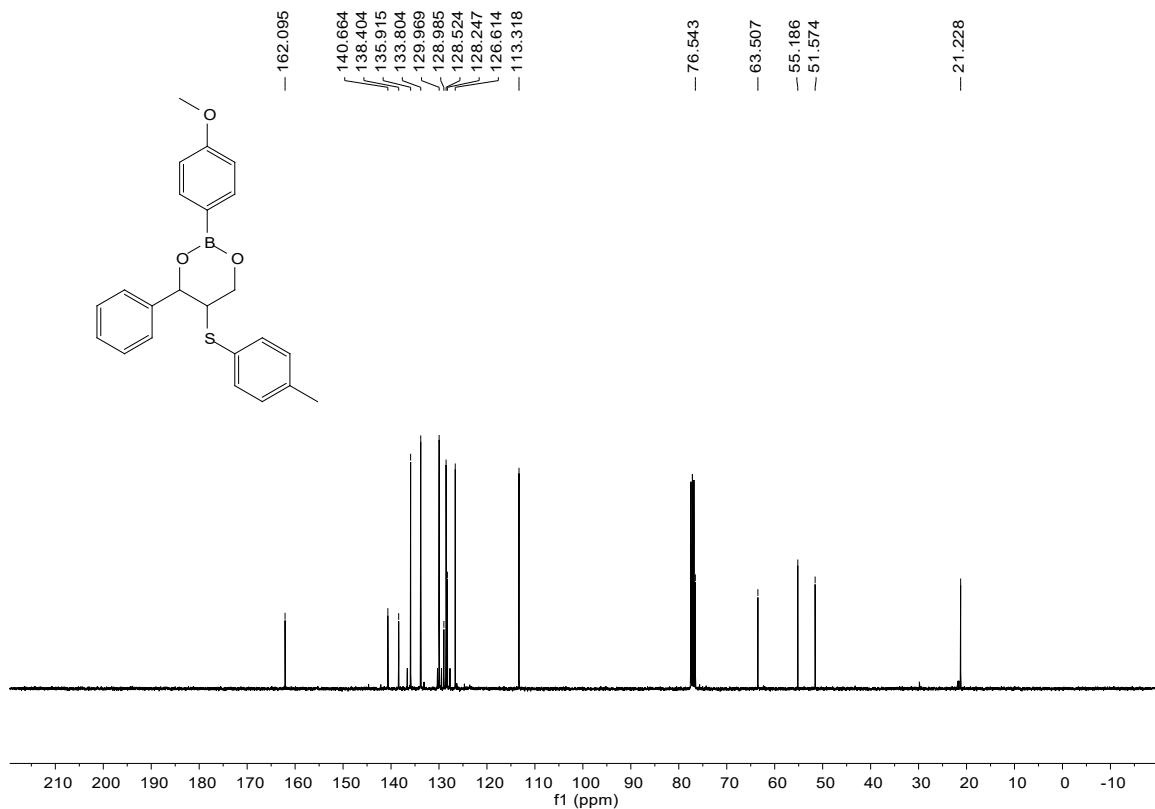
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6b**



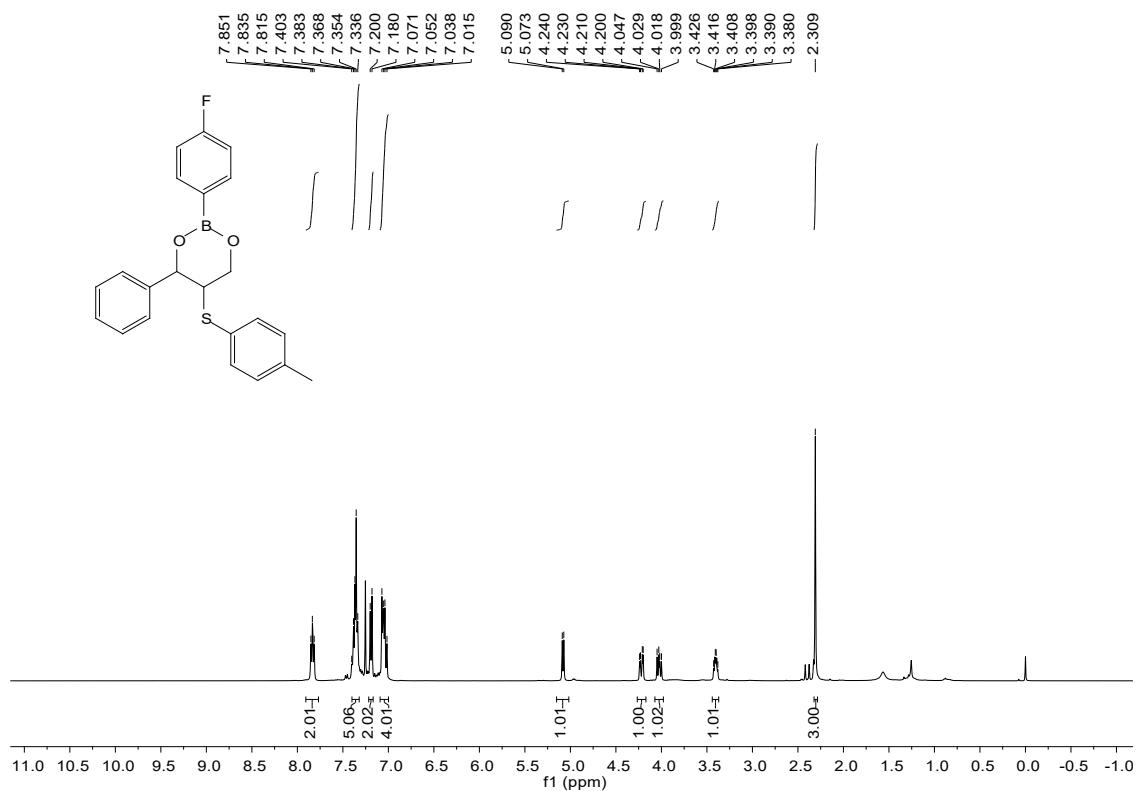
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6c**



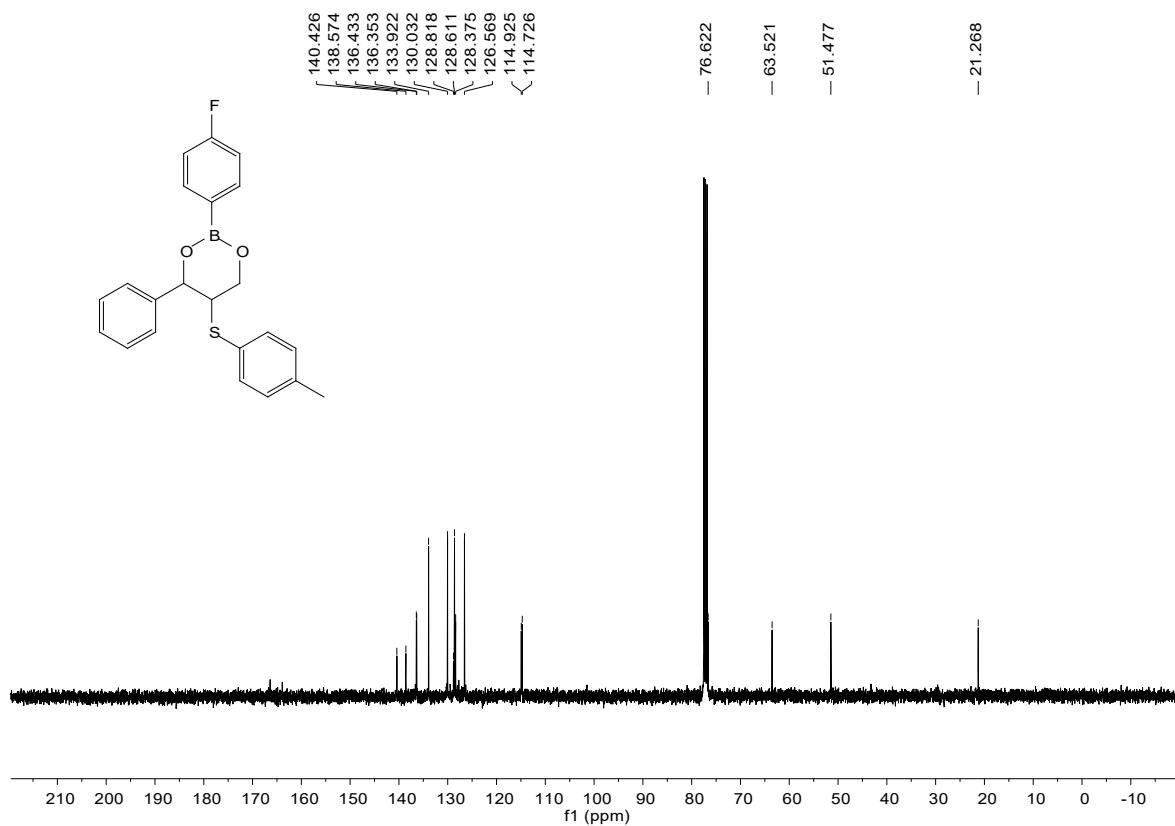
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6c**



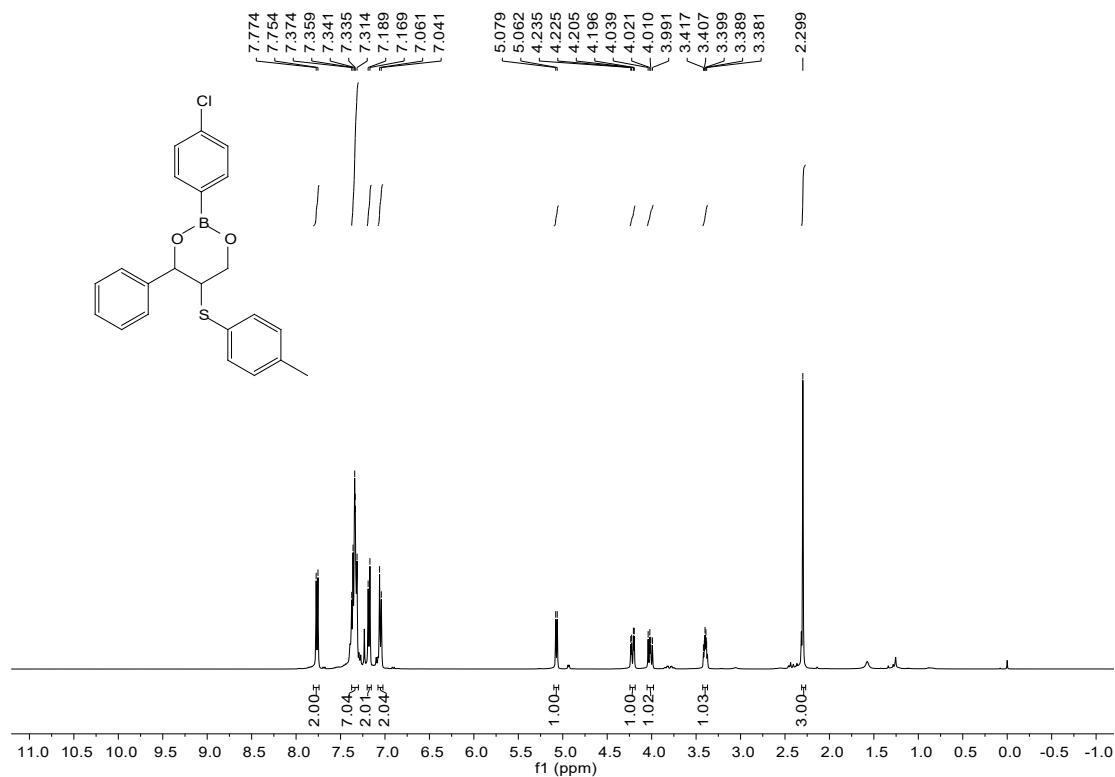
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6d**



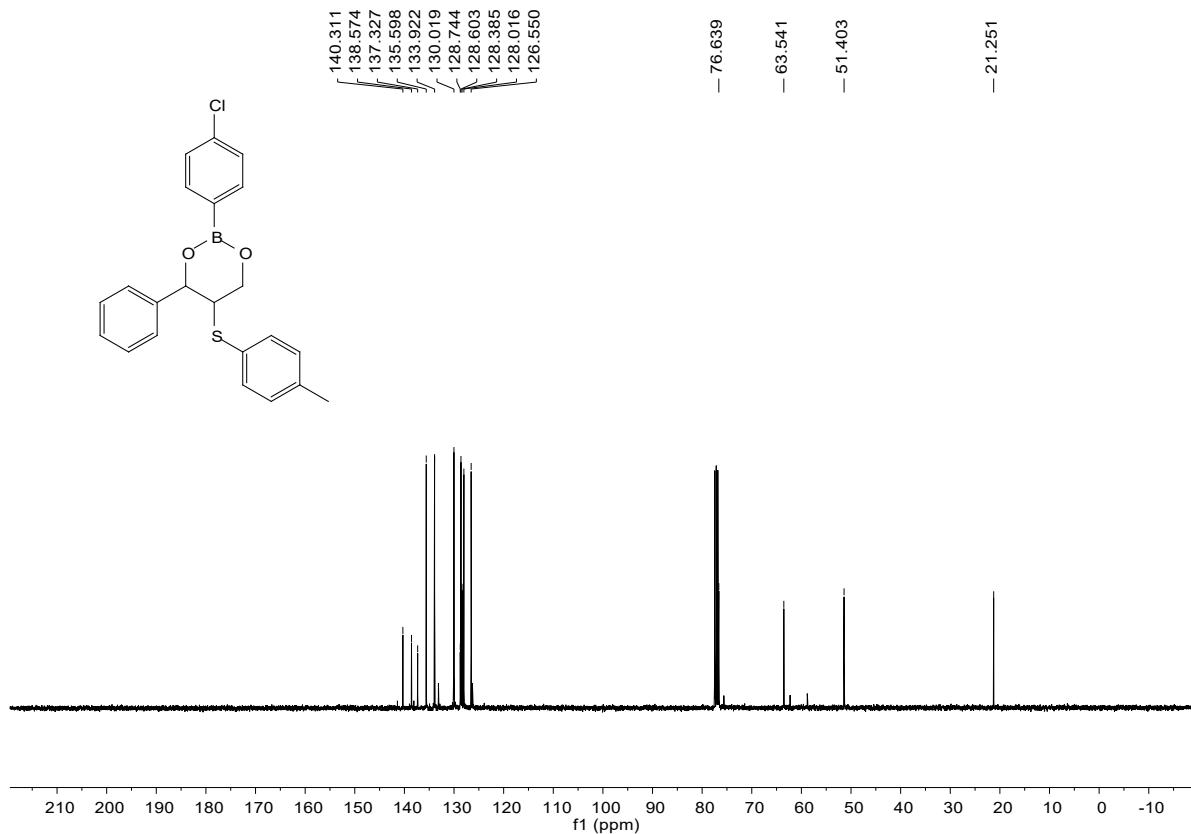
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6d**



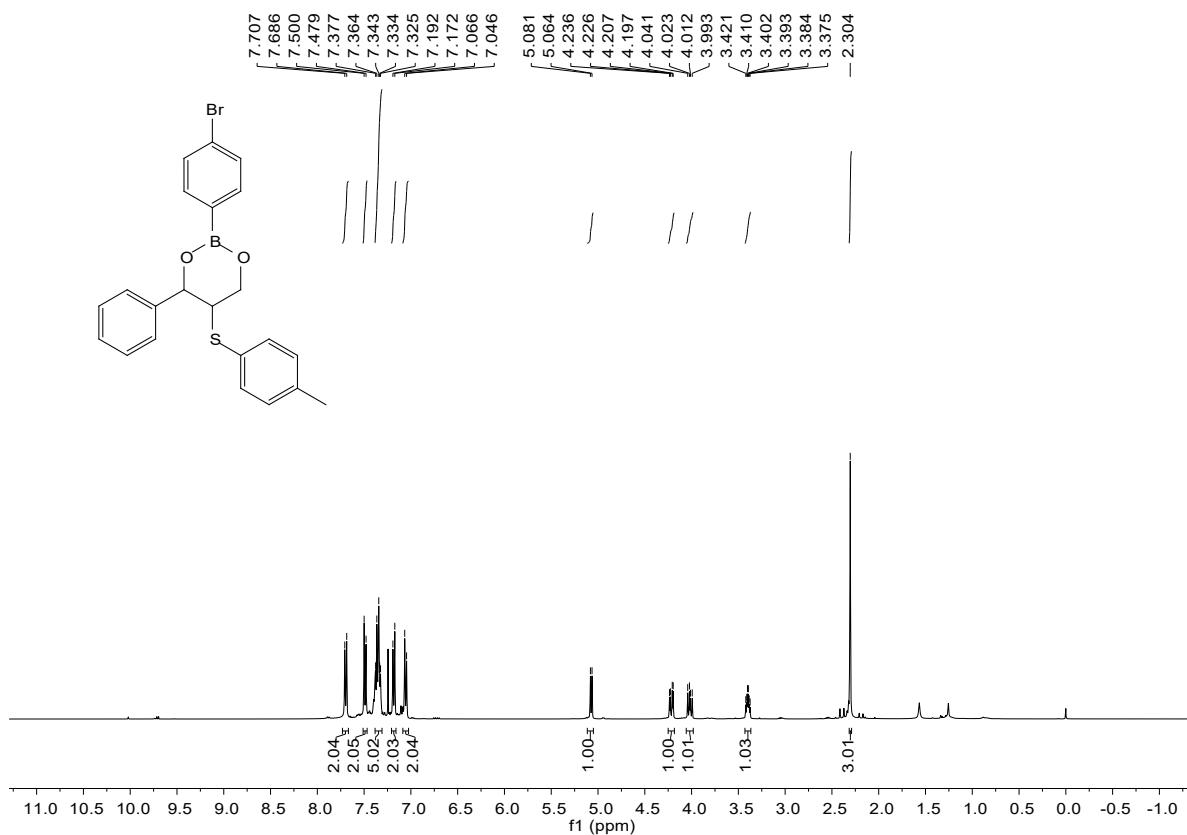
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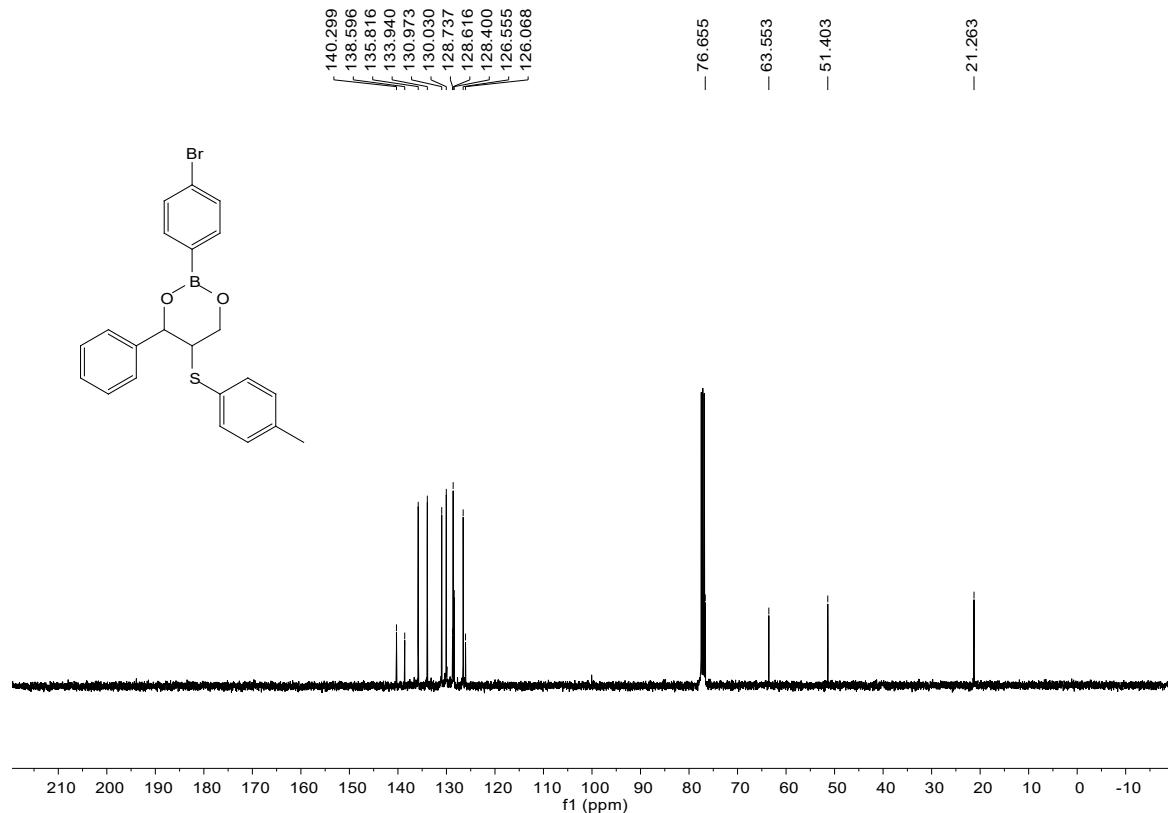
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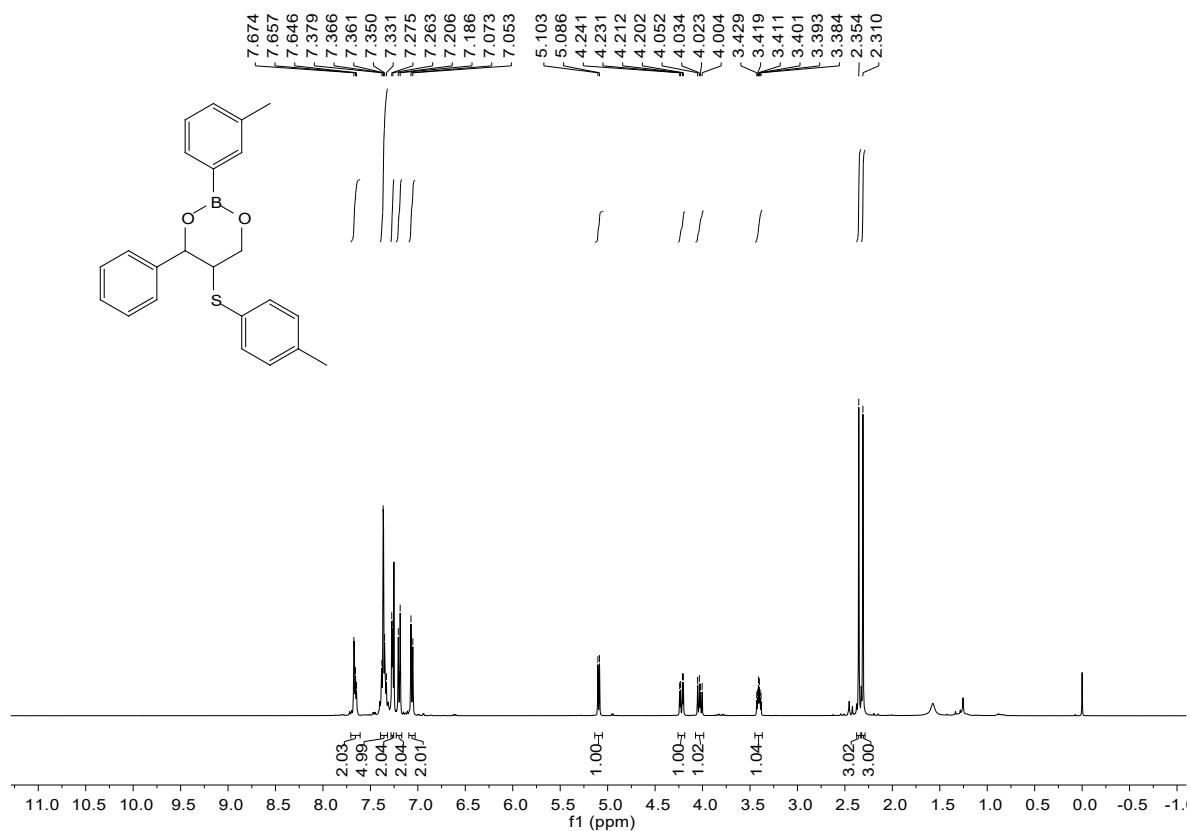
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6f**



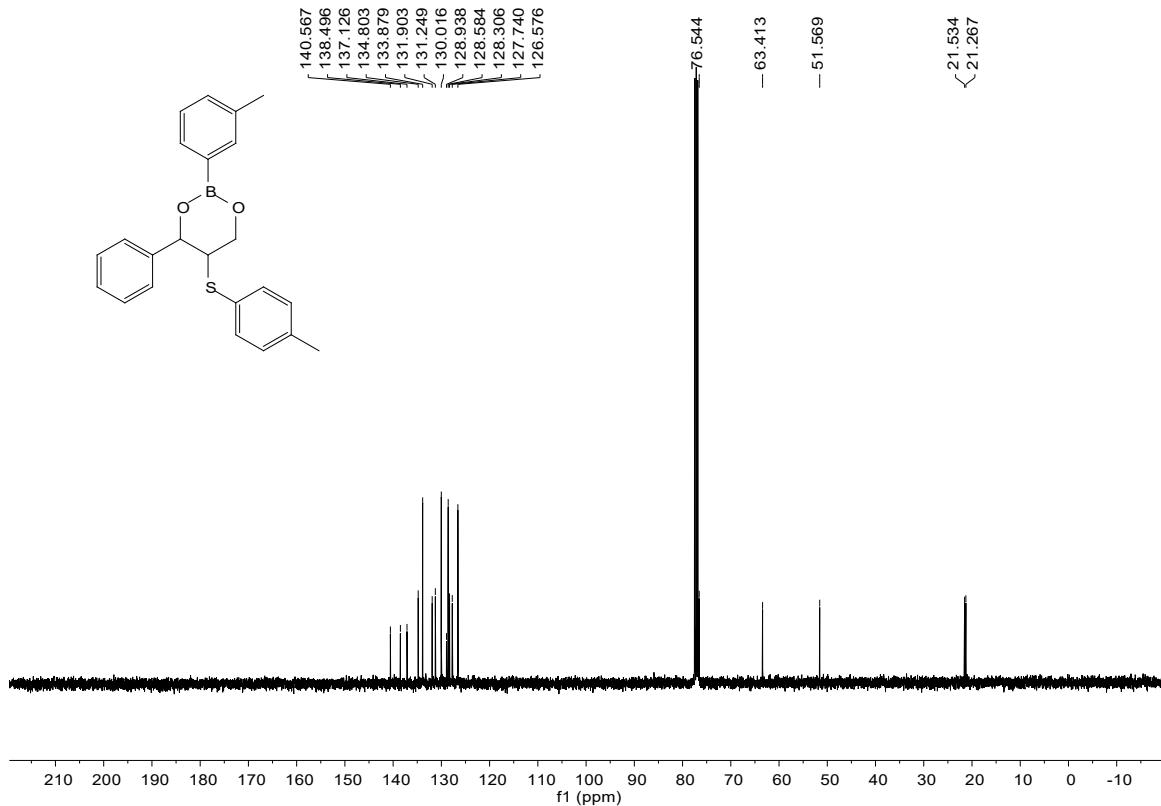
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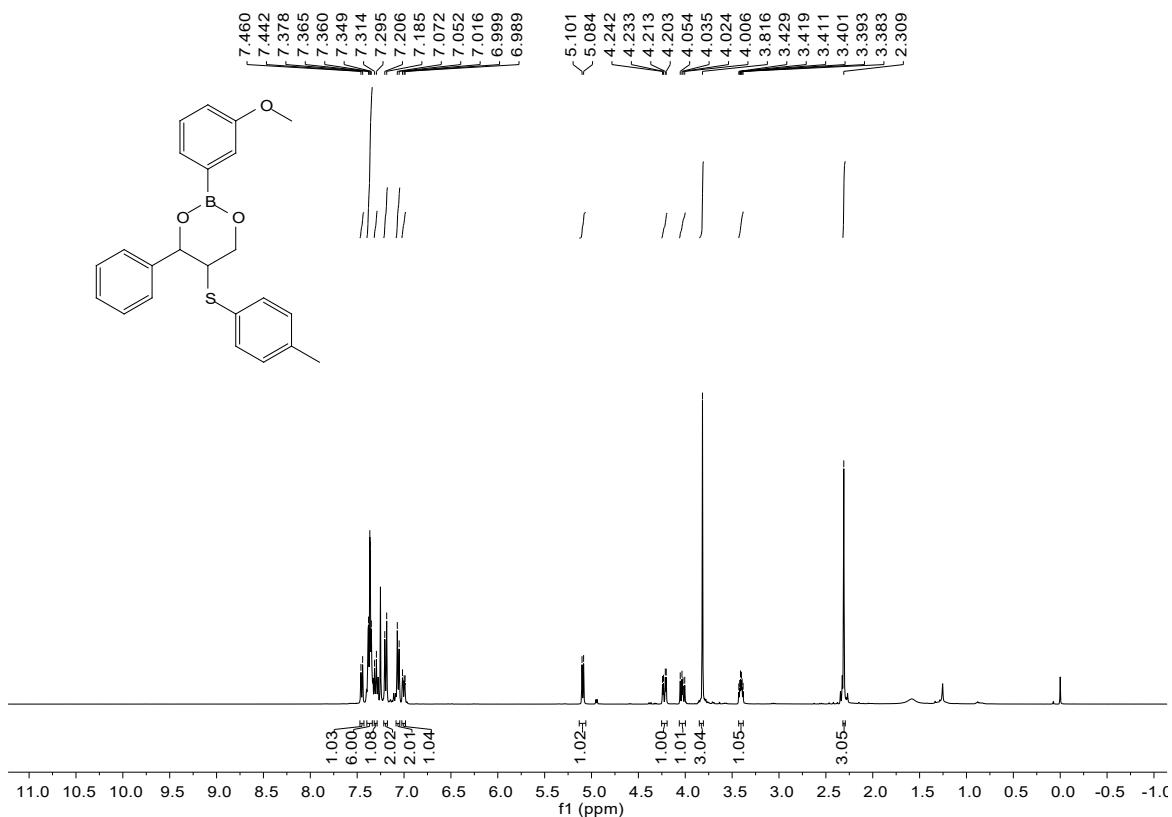
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6g**



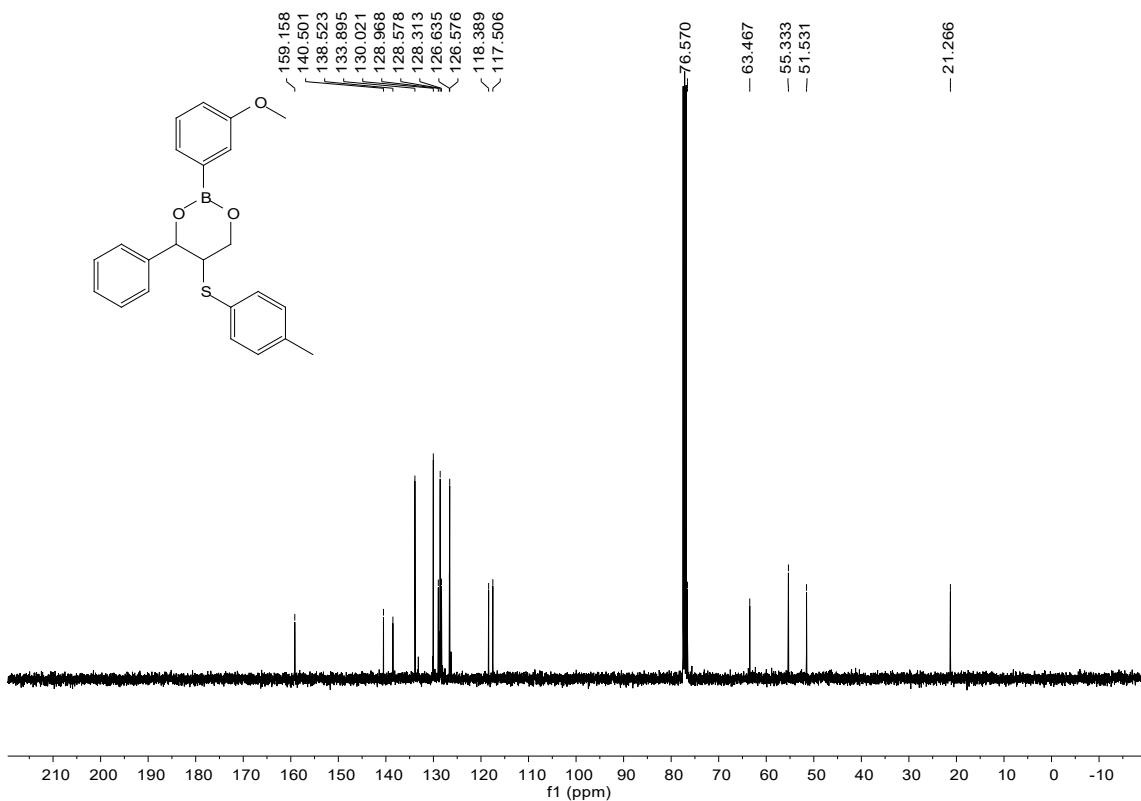
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6g**



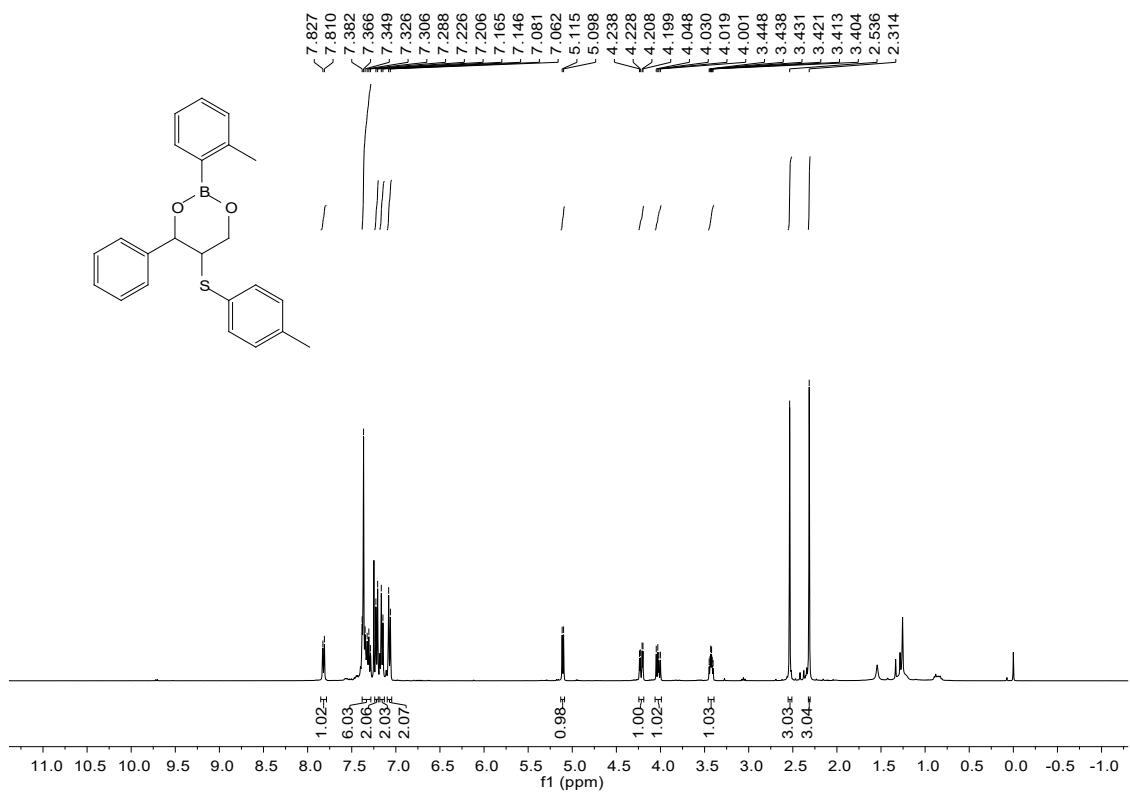
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6h**



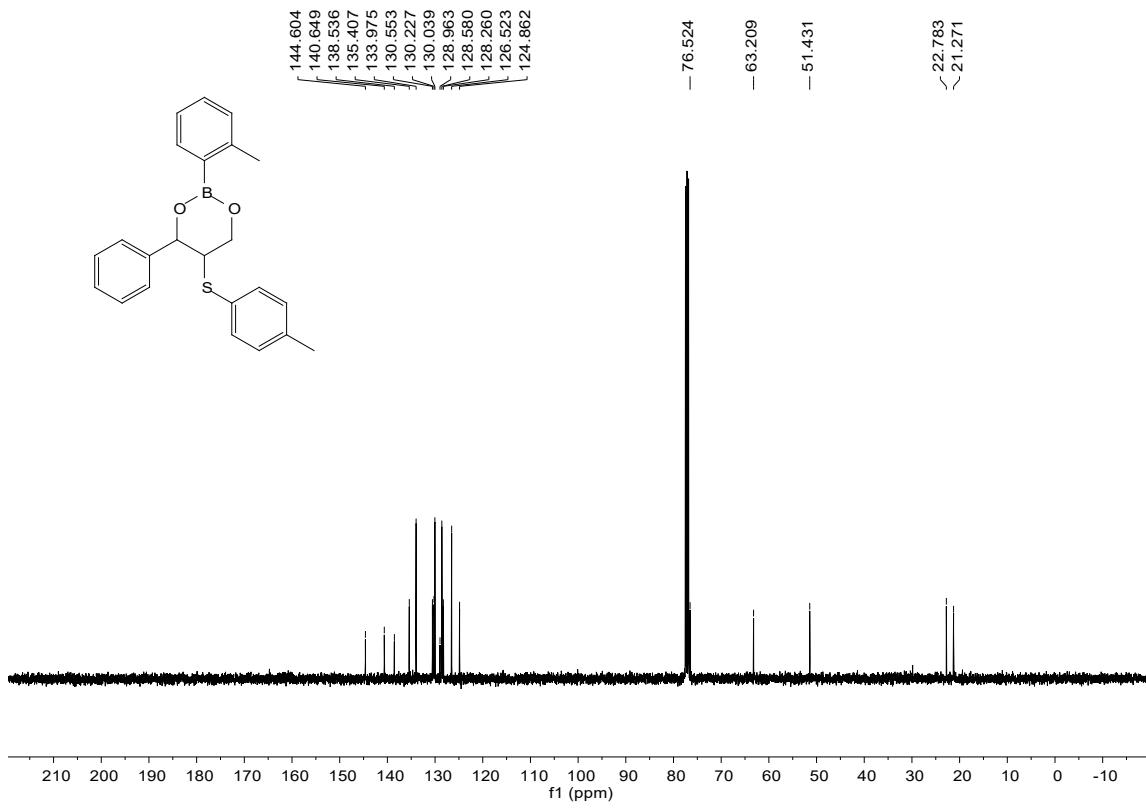
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6h**



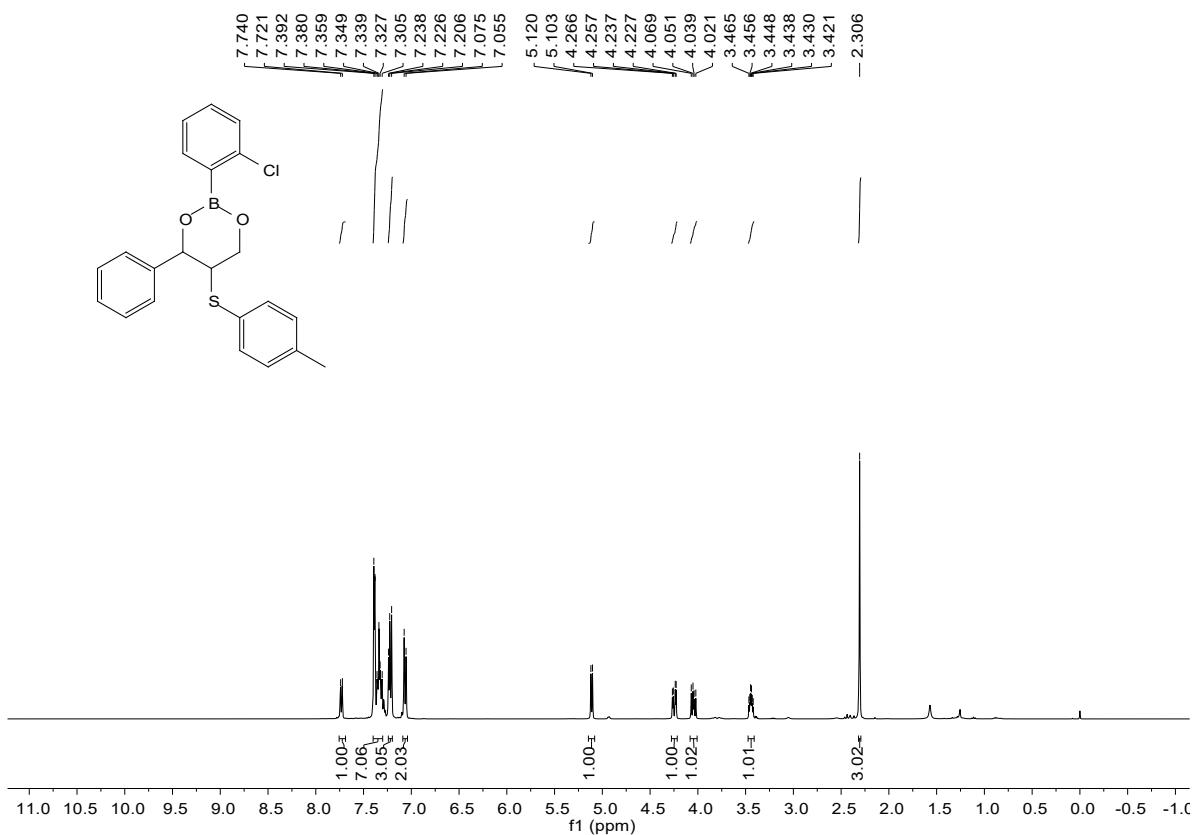
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6i**



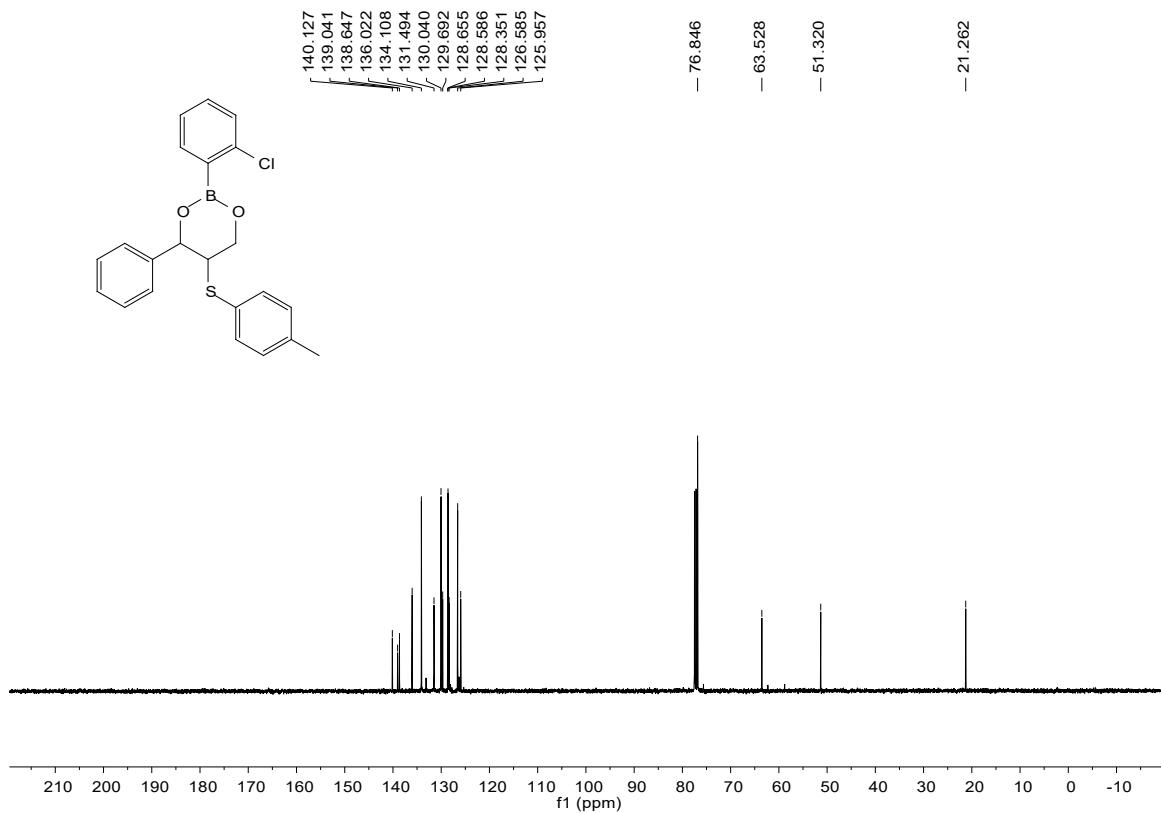
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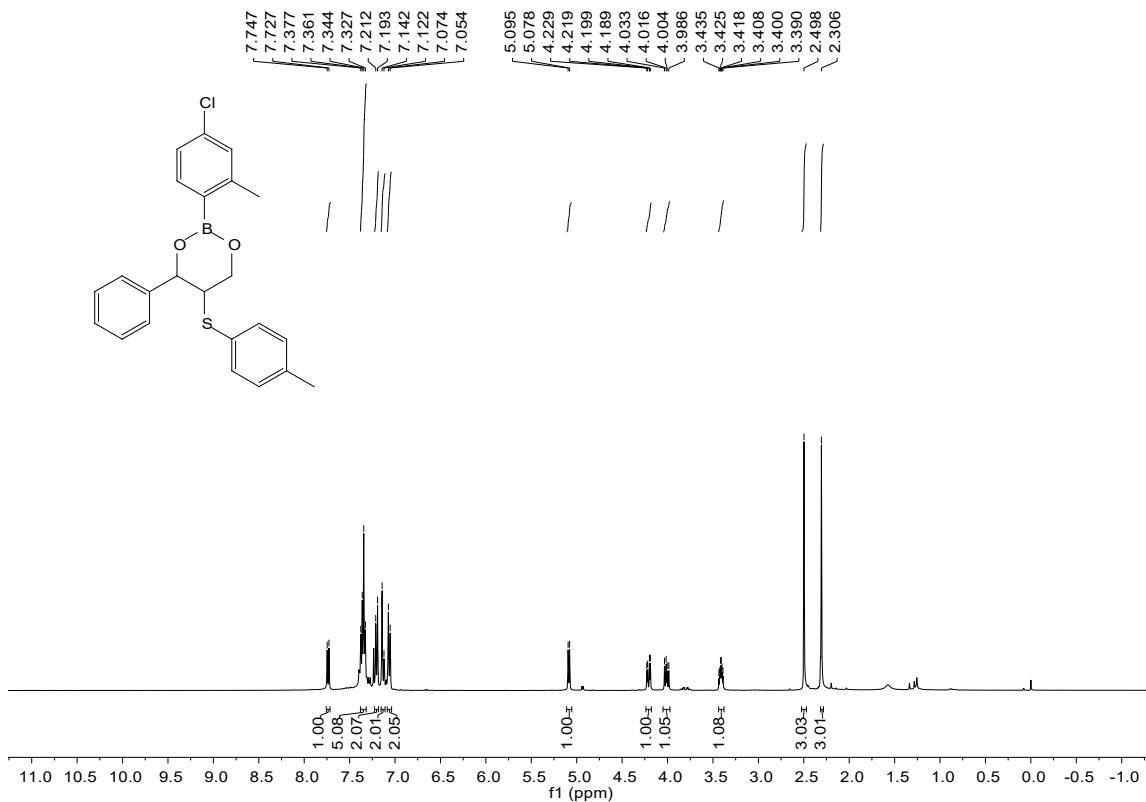
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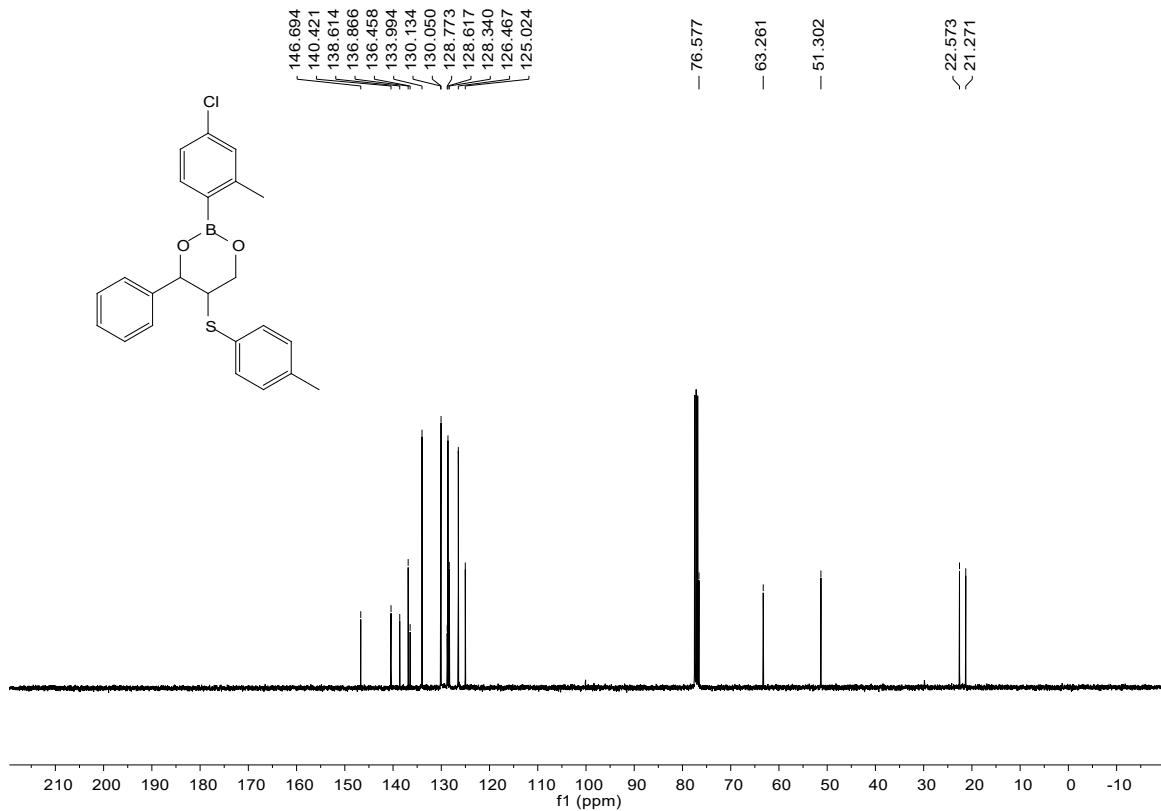
¹³C NMR (100 MHz, CDCl₃) spectrum of compound 6j



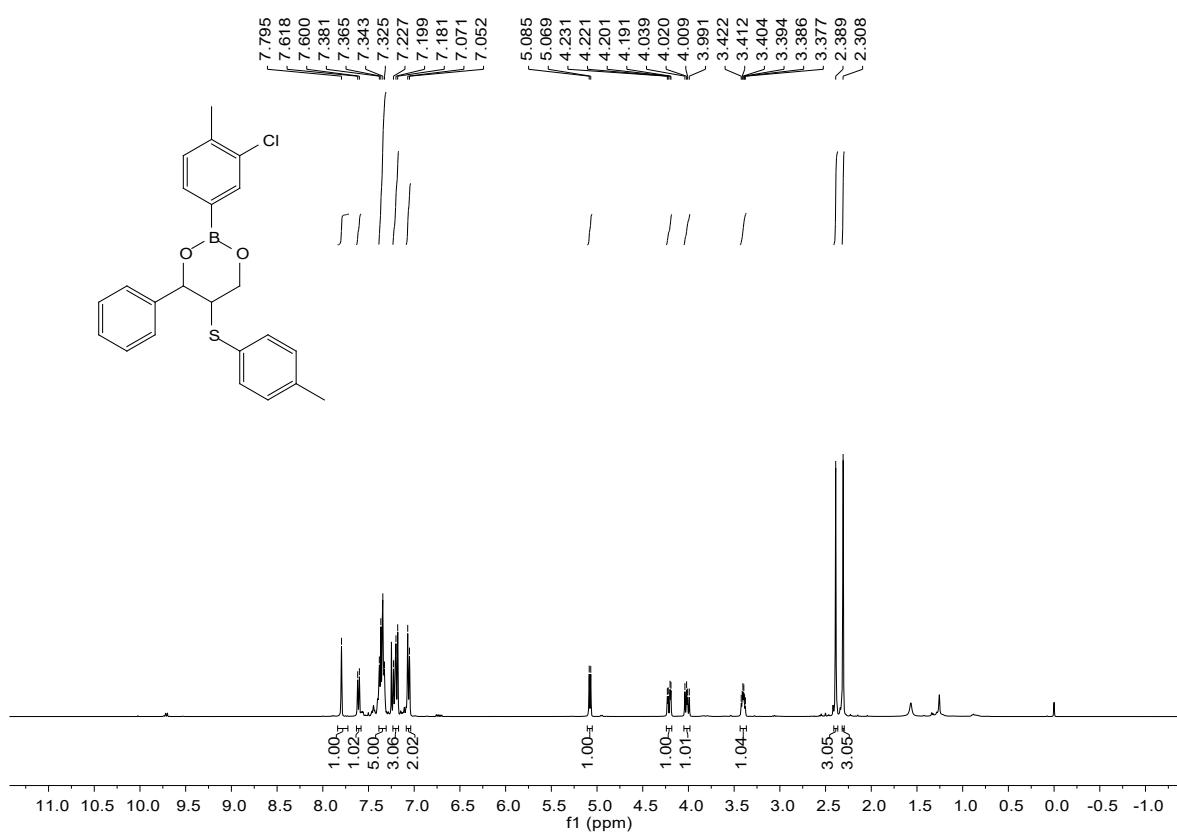
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6k**



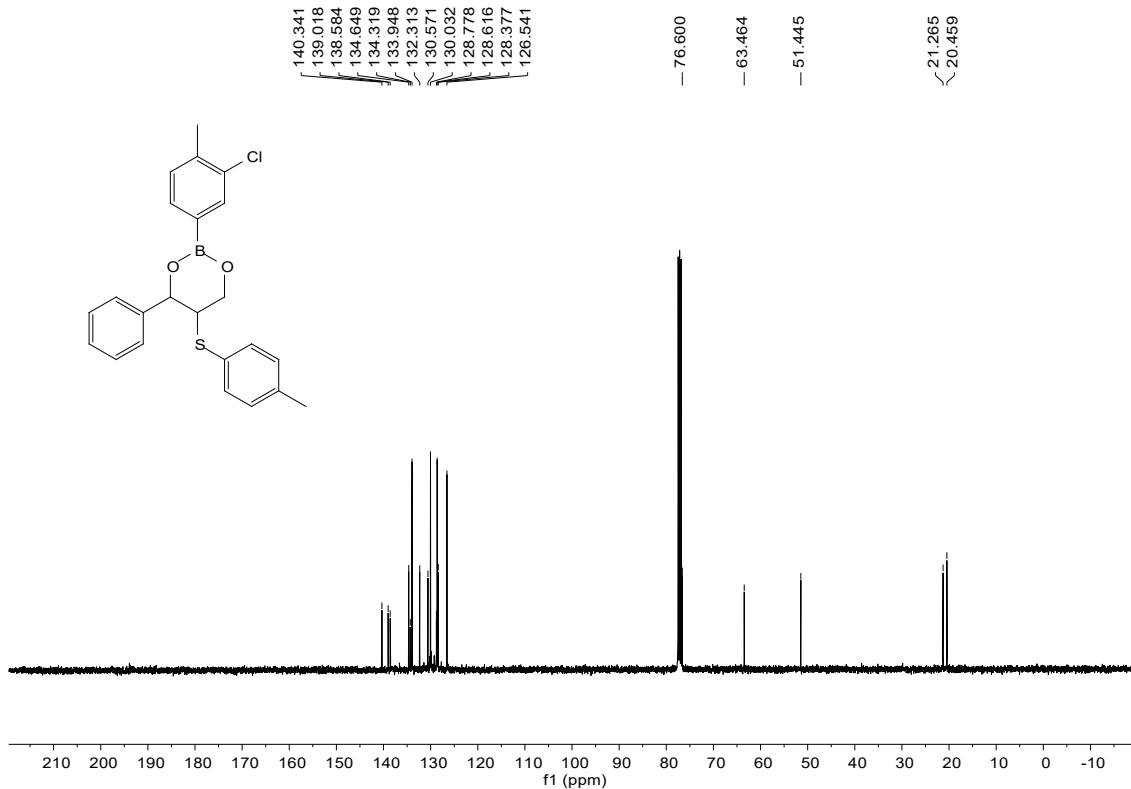
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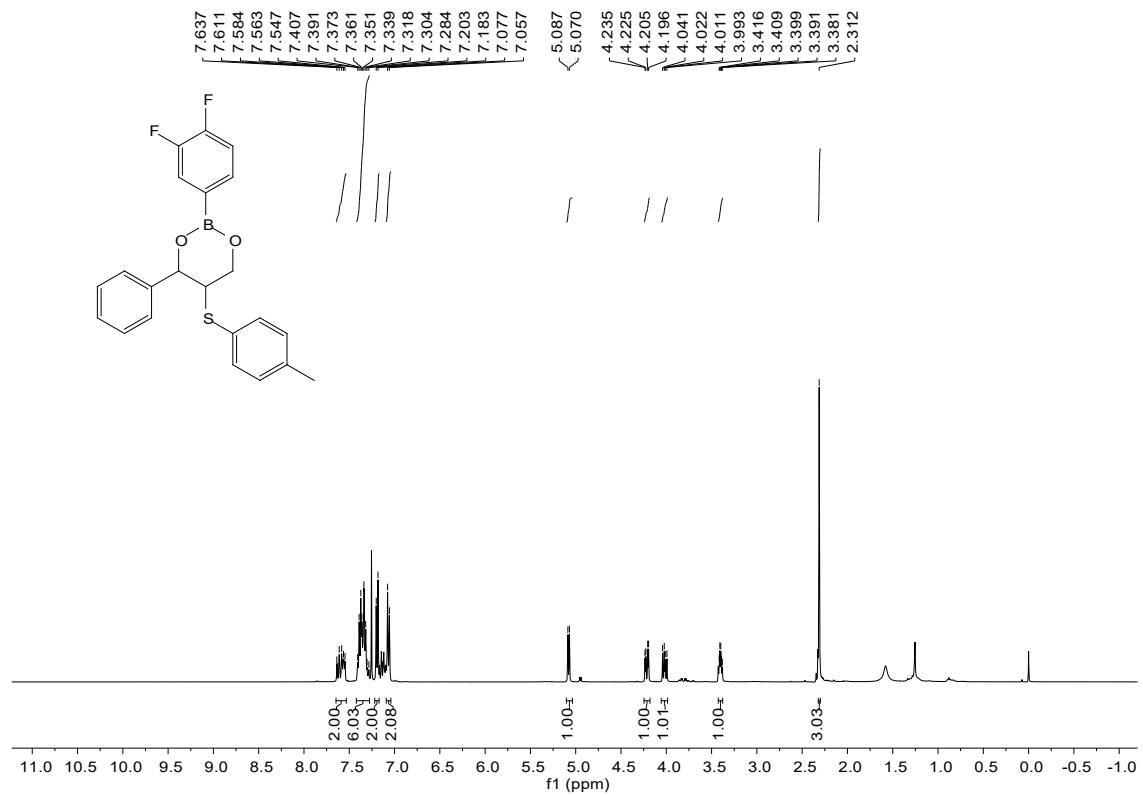
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6l**



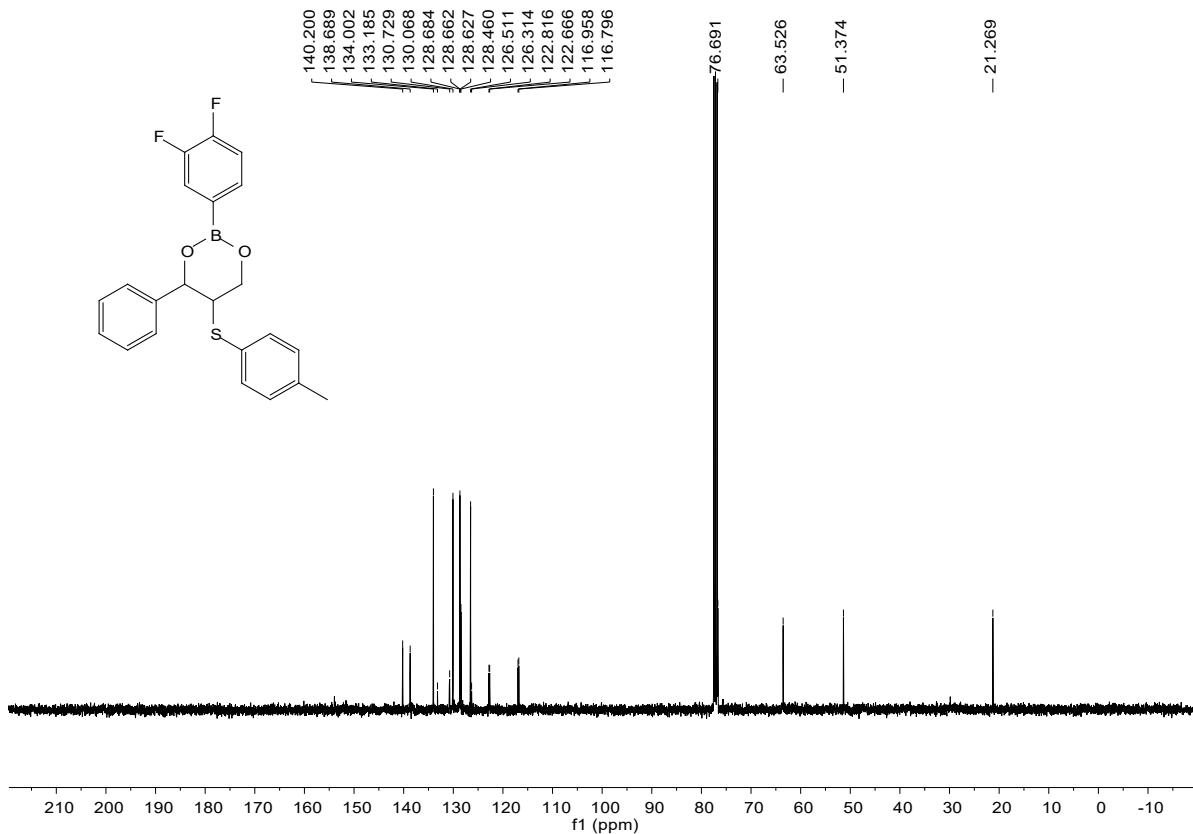
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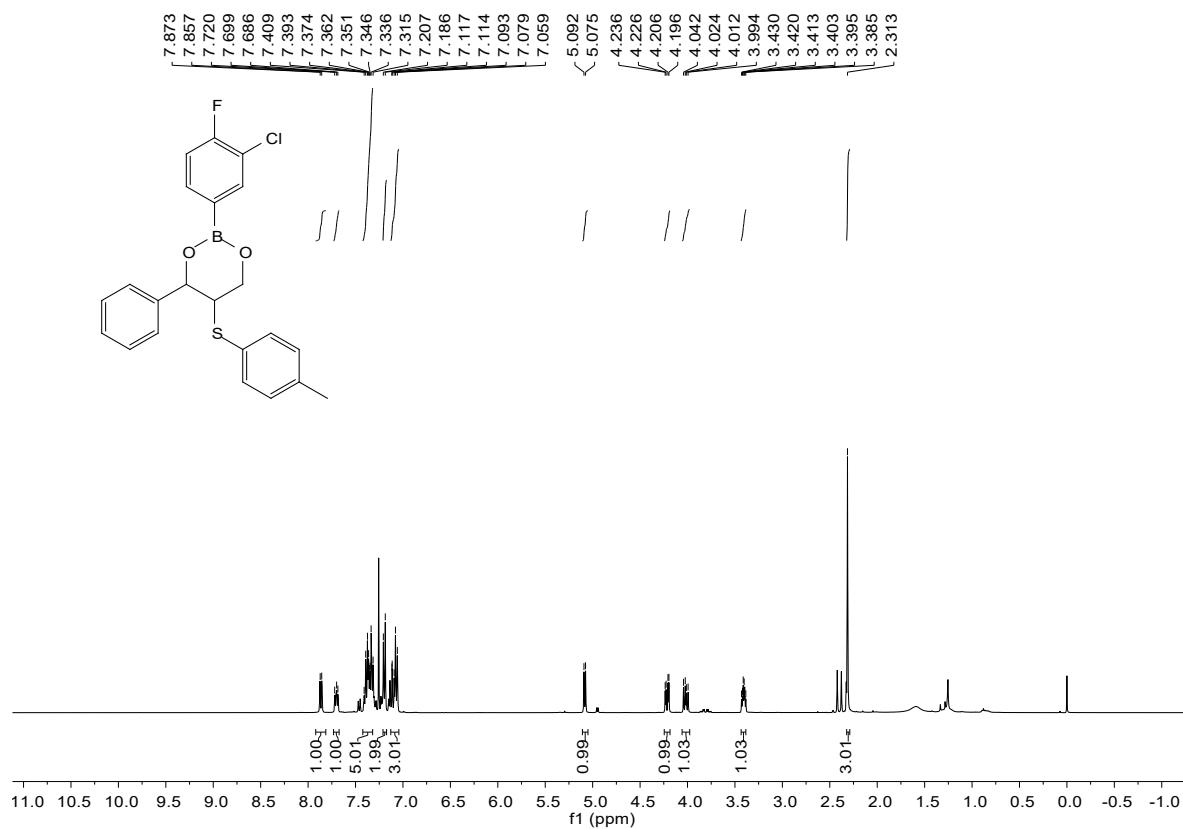
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6m**



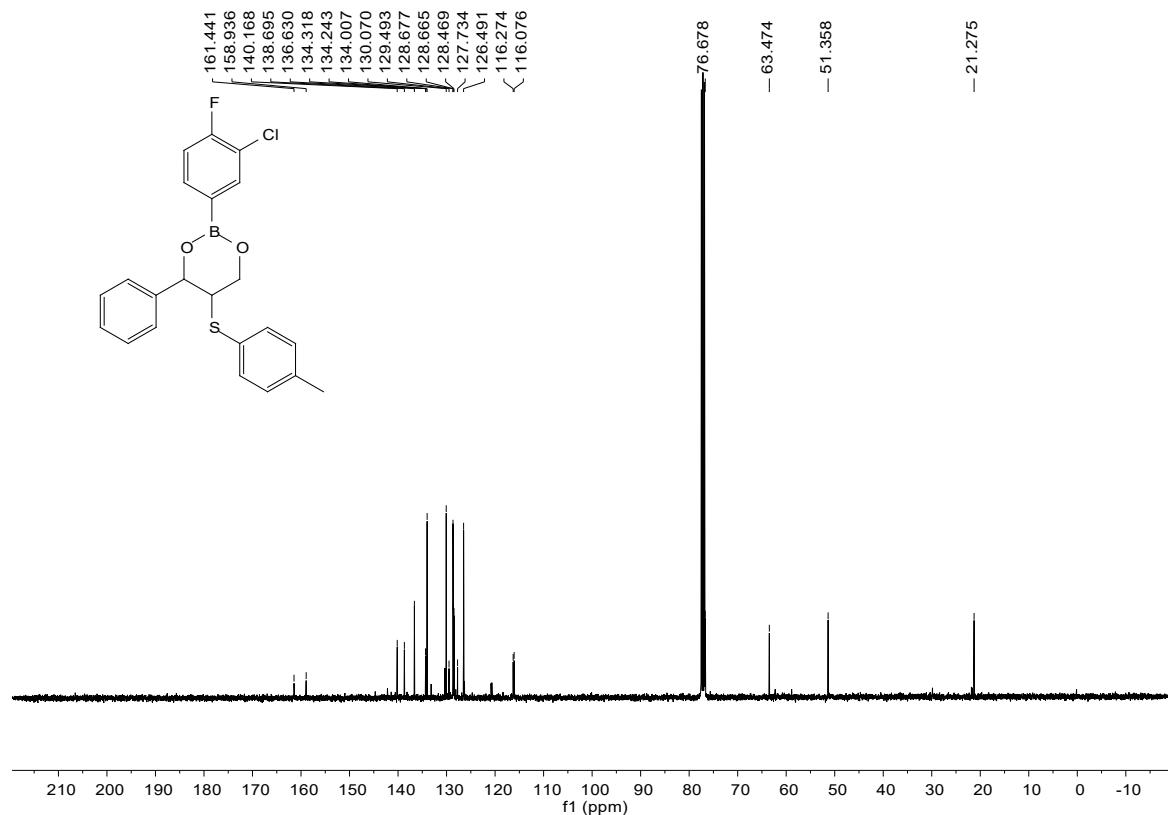
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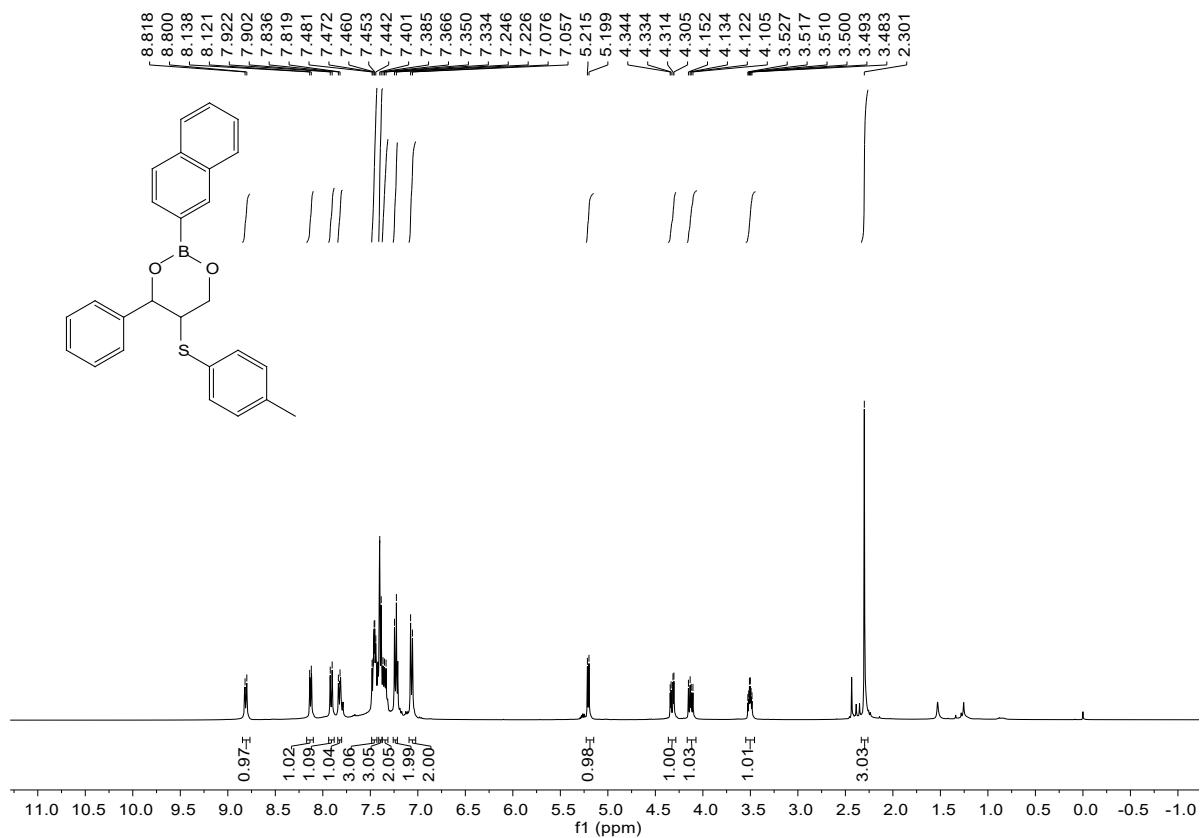
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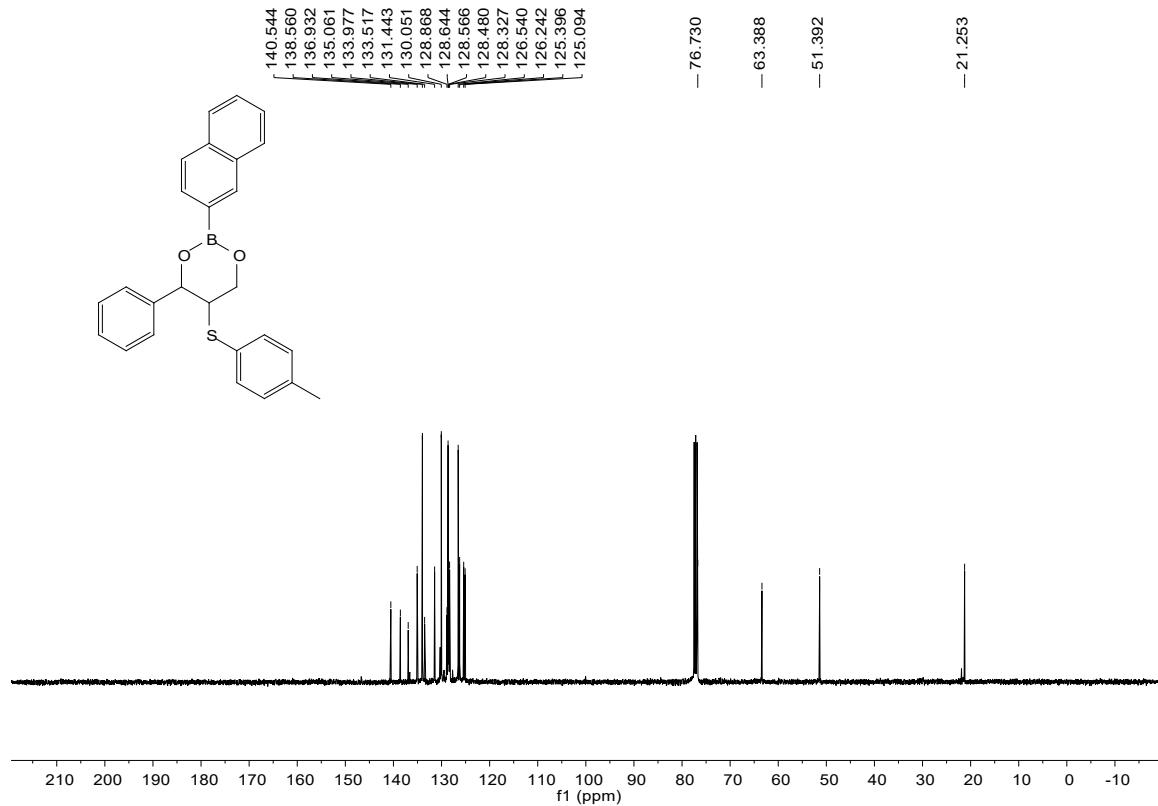
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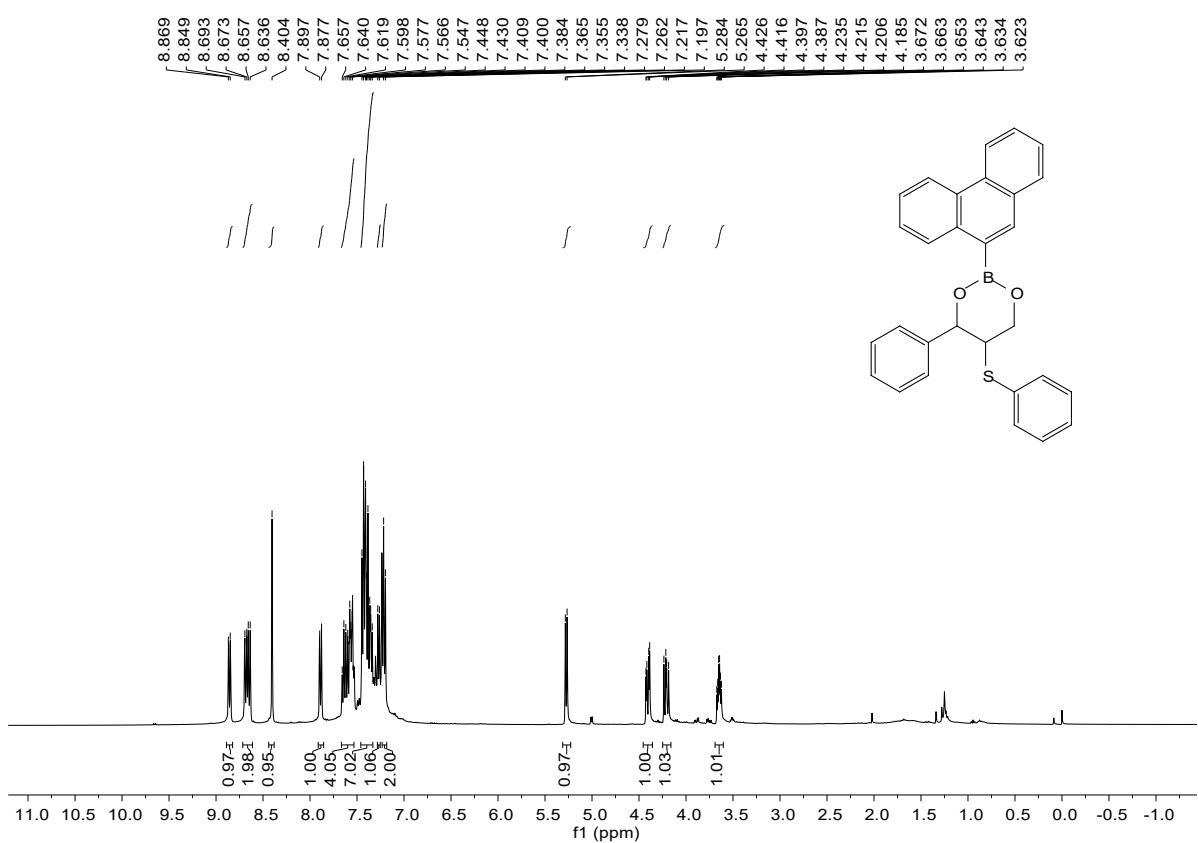
¹H NMR (400 MHz, CDCl₃) spectrum of compound **60**



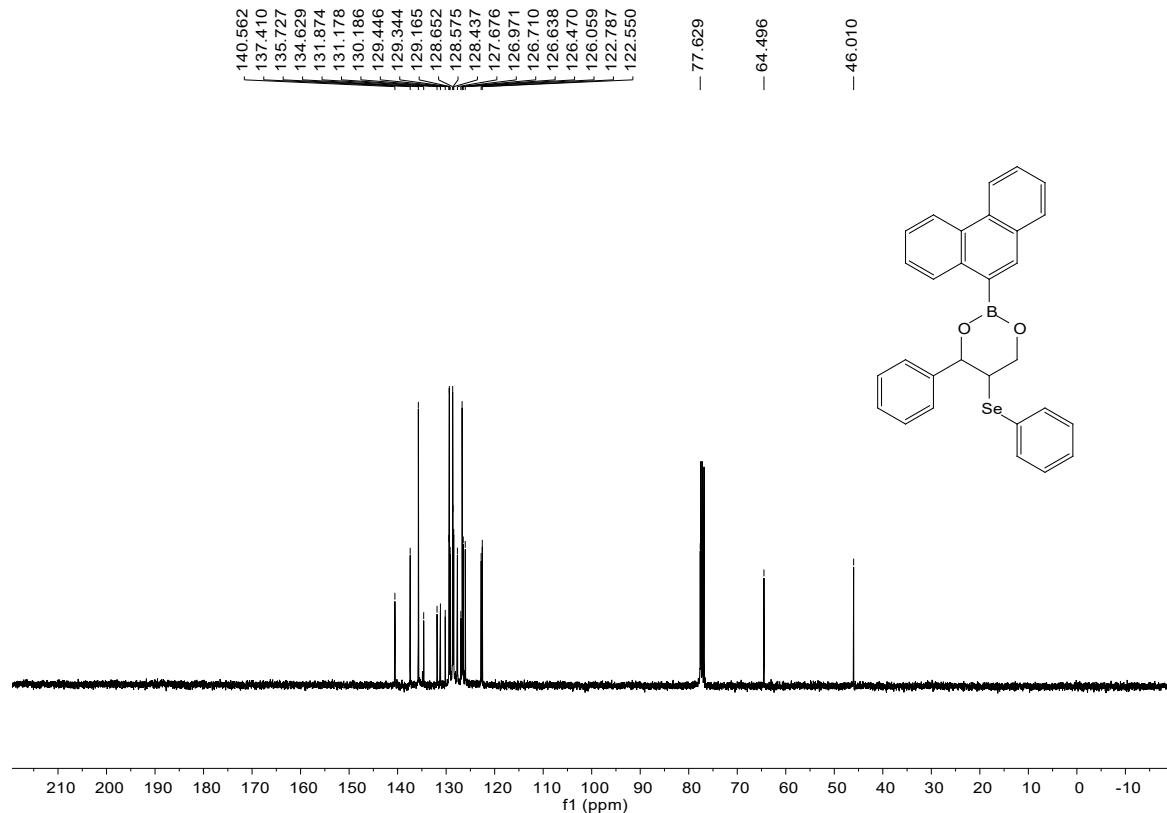
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **60**



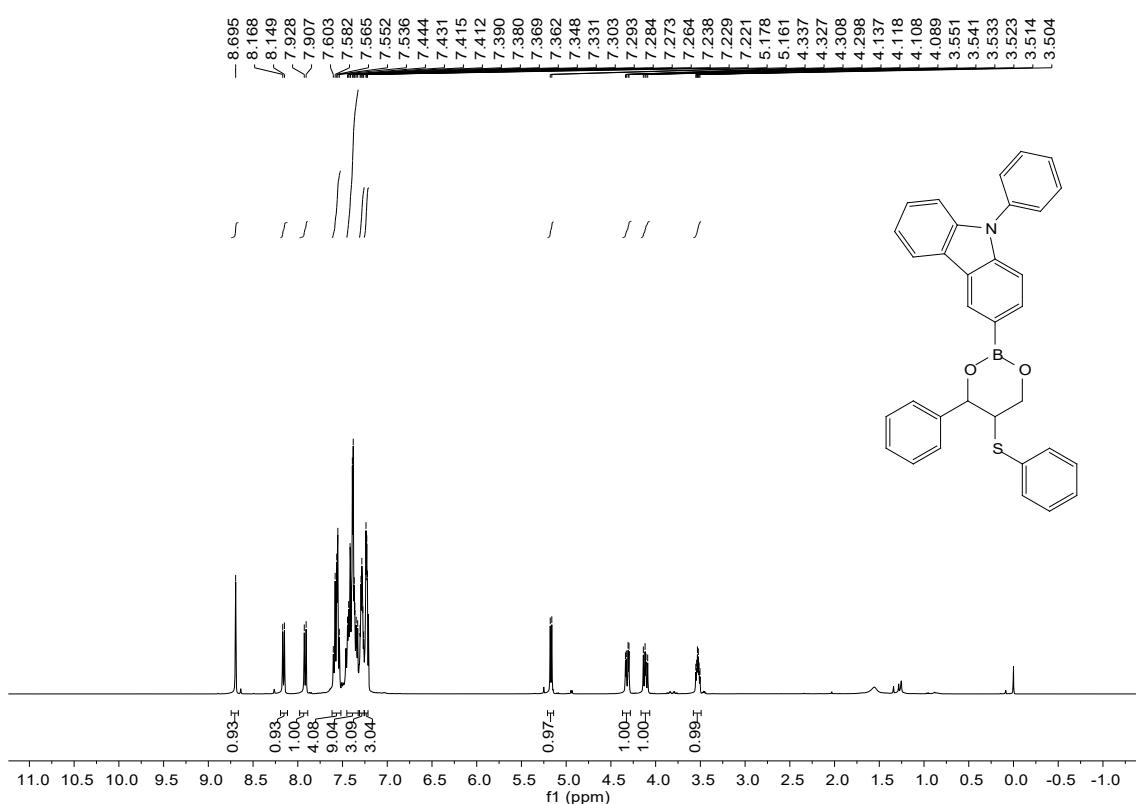
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6p**



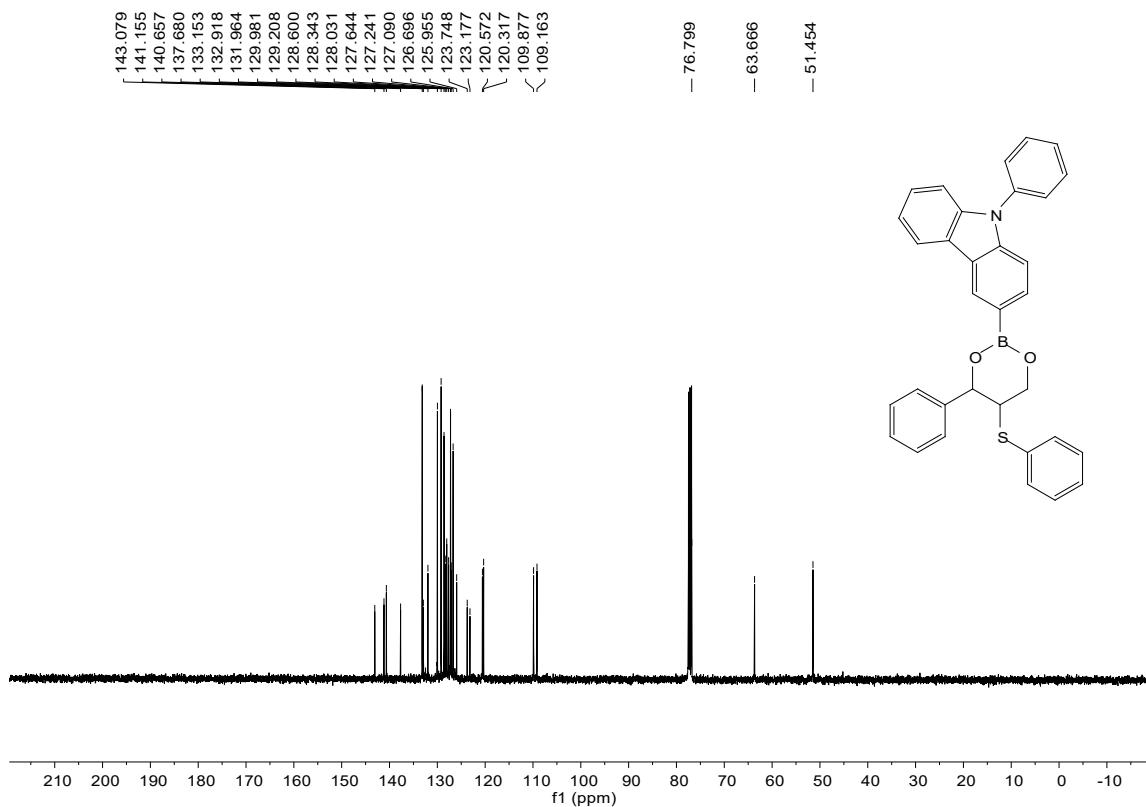
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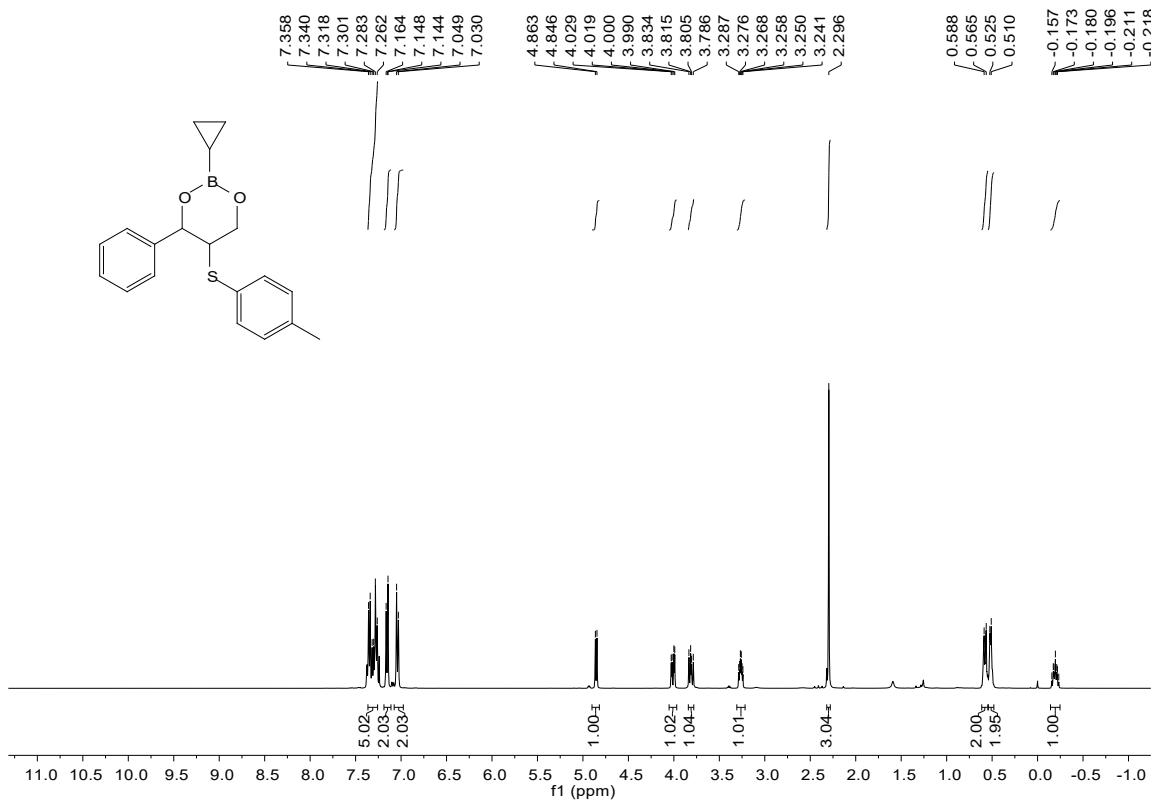
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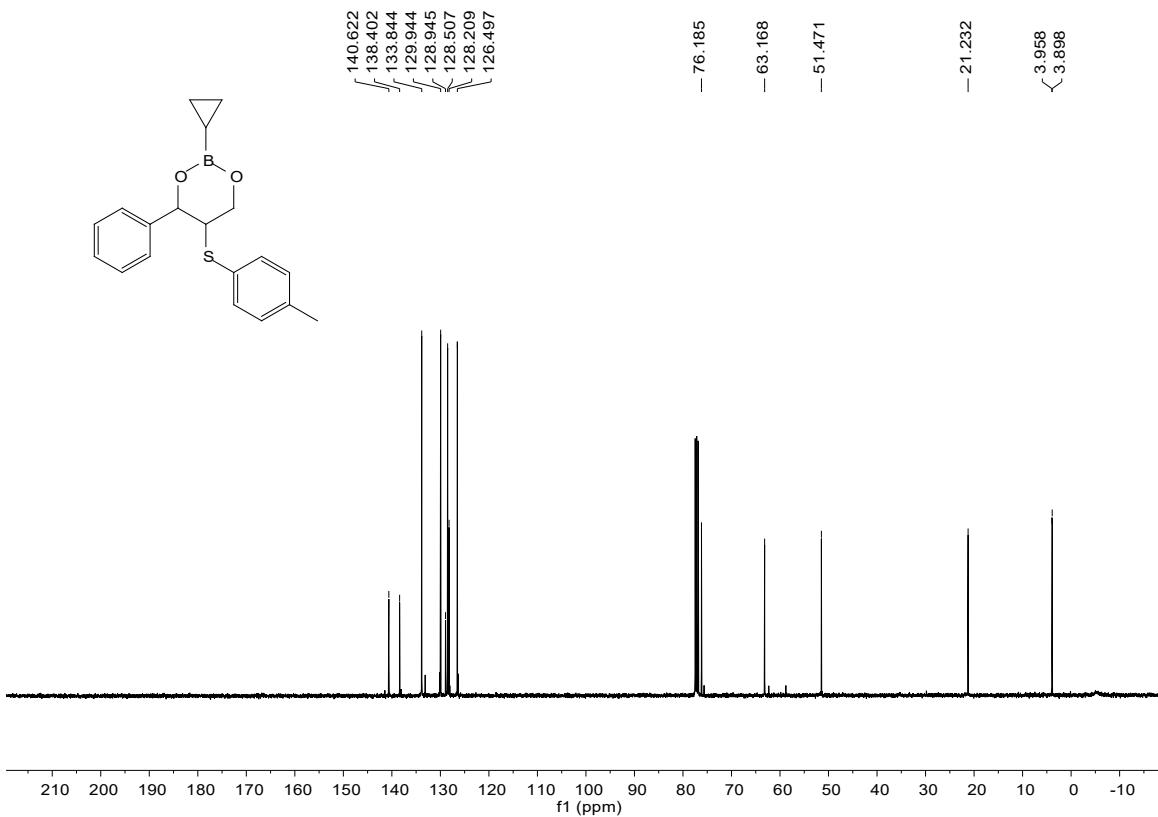
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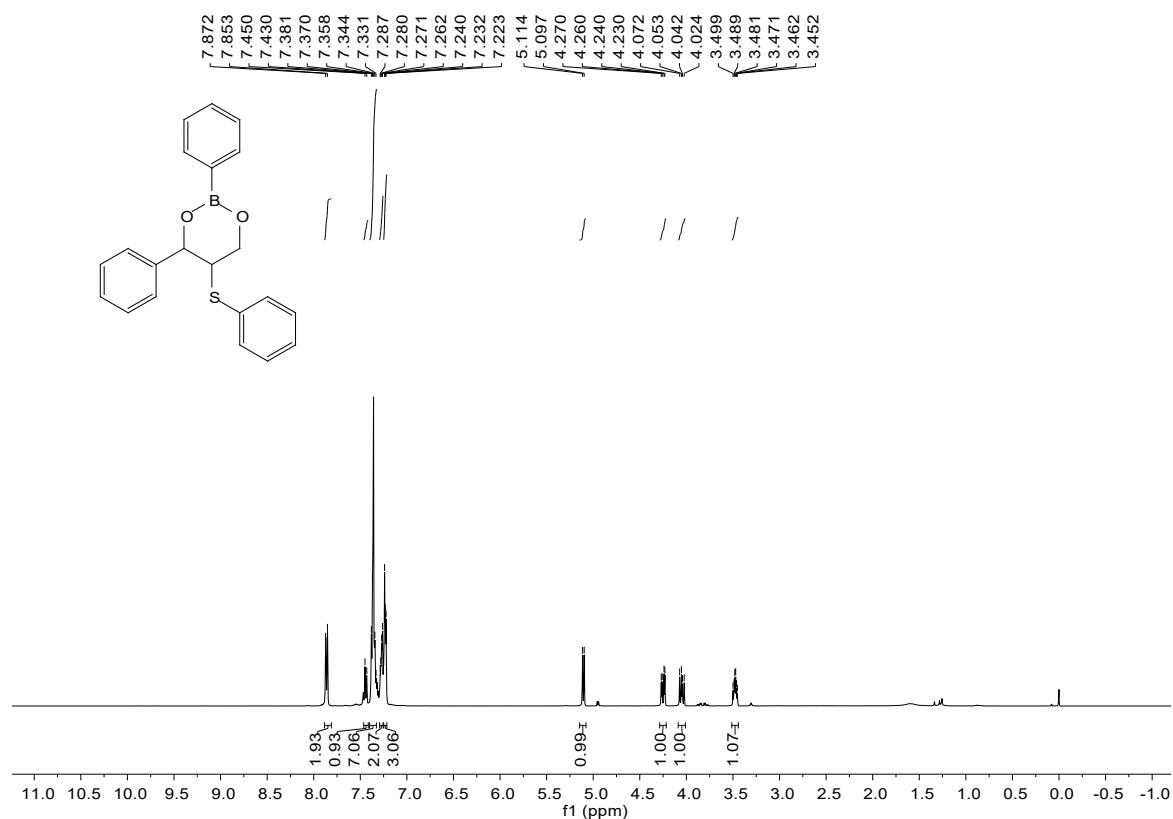
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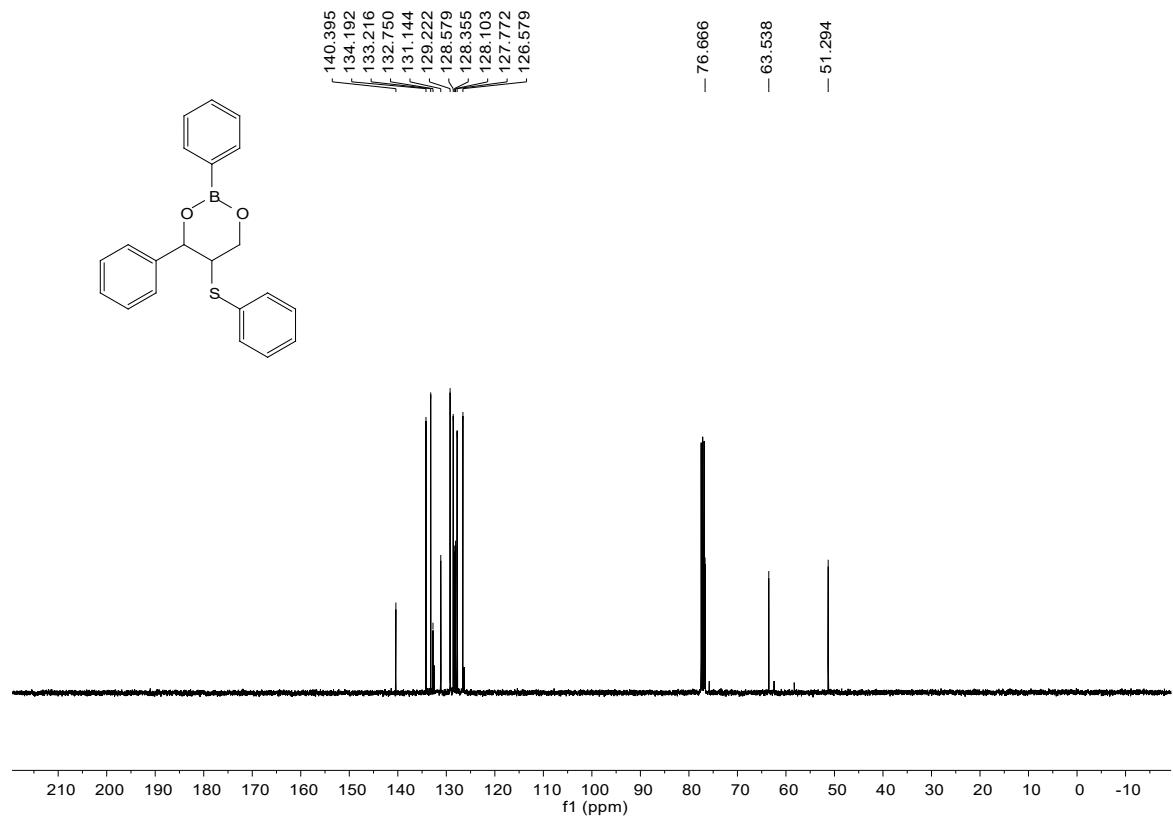
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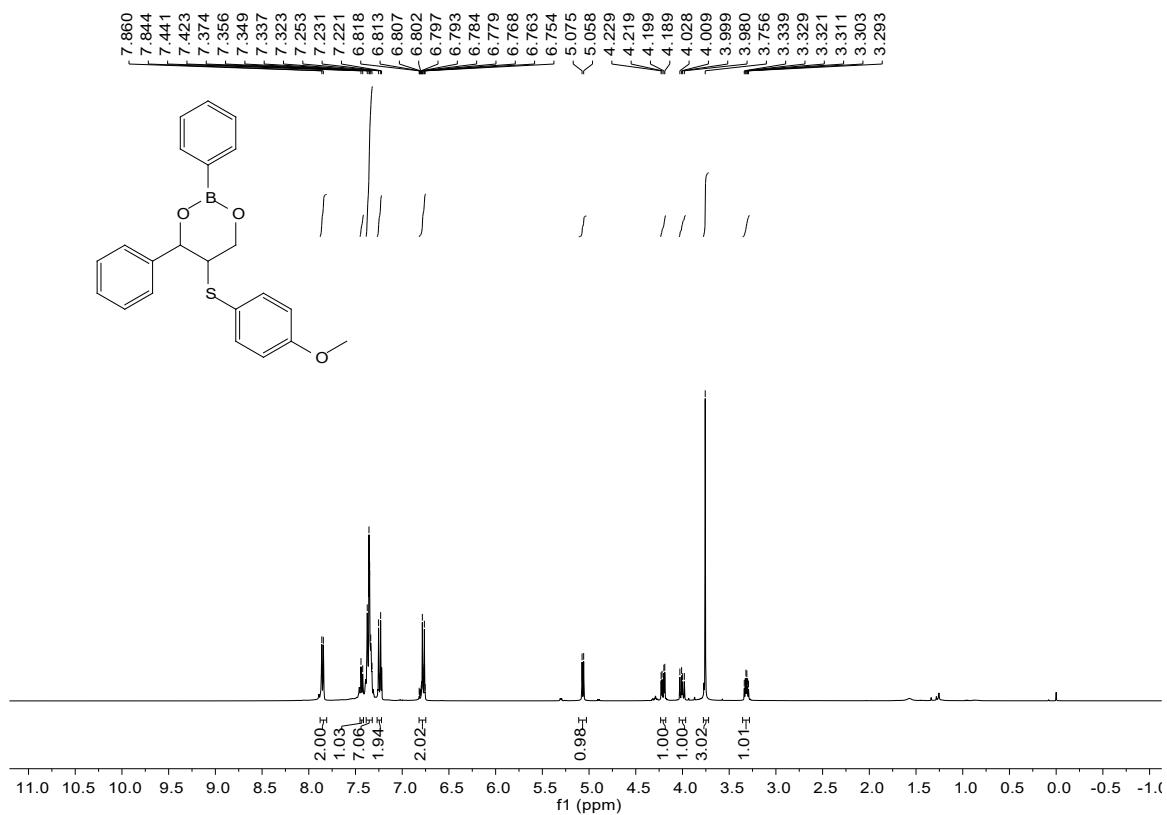
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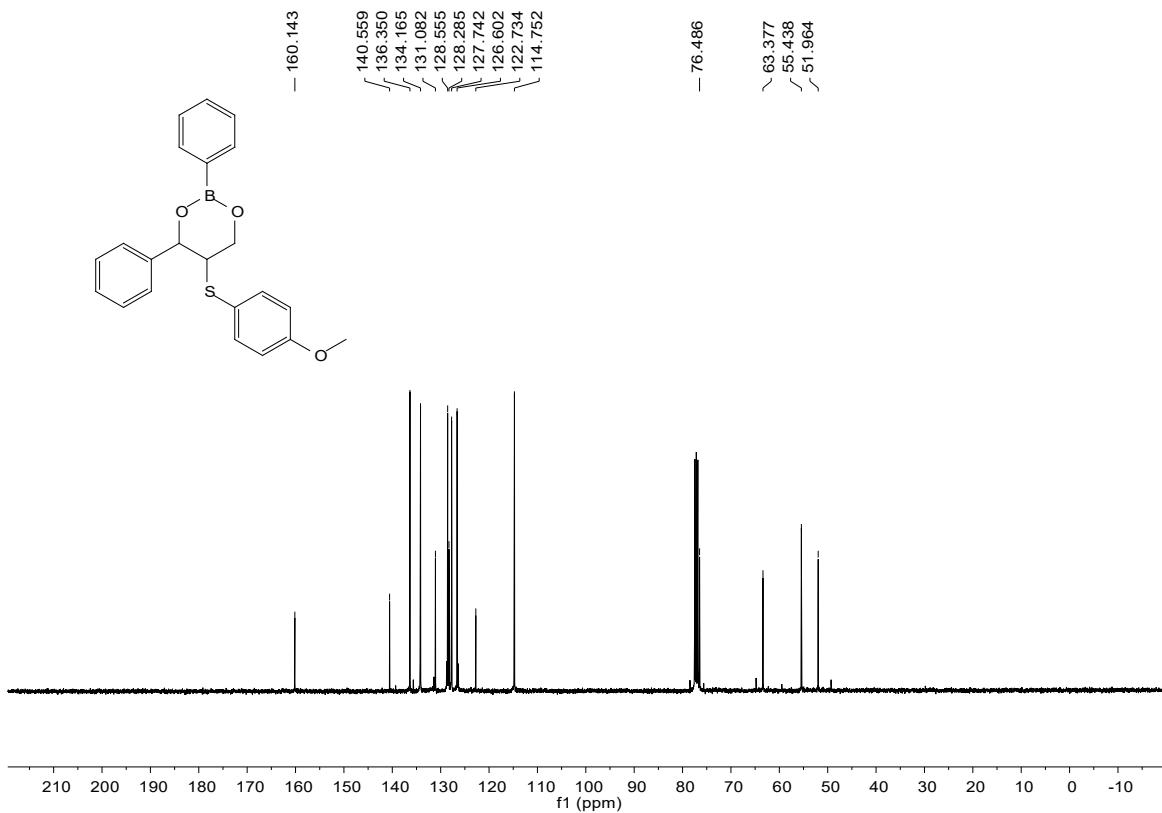
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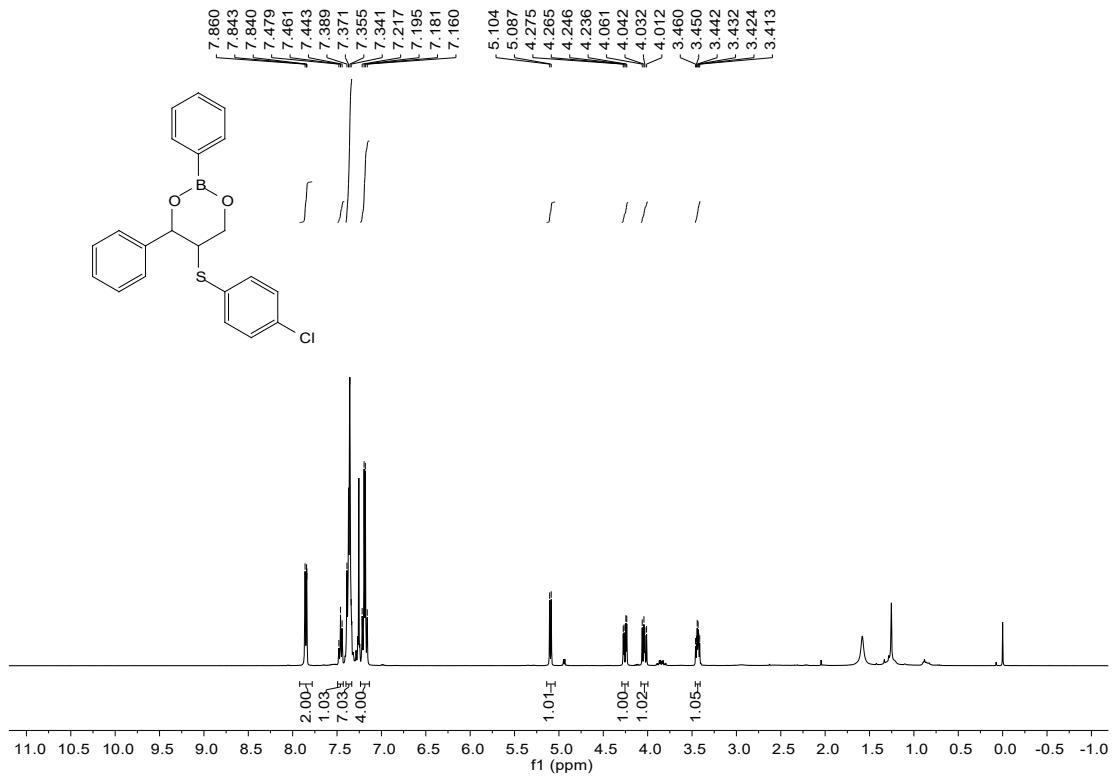
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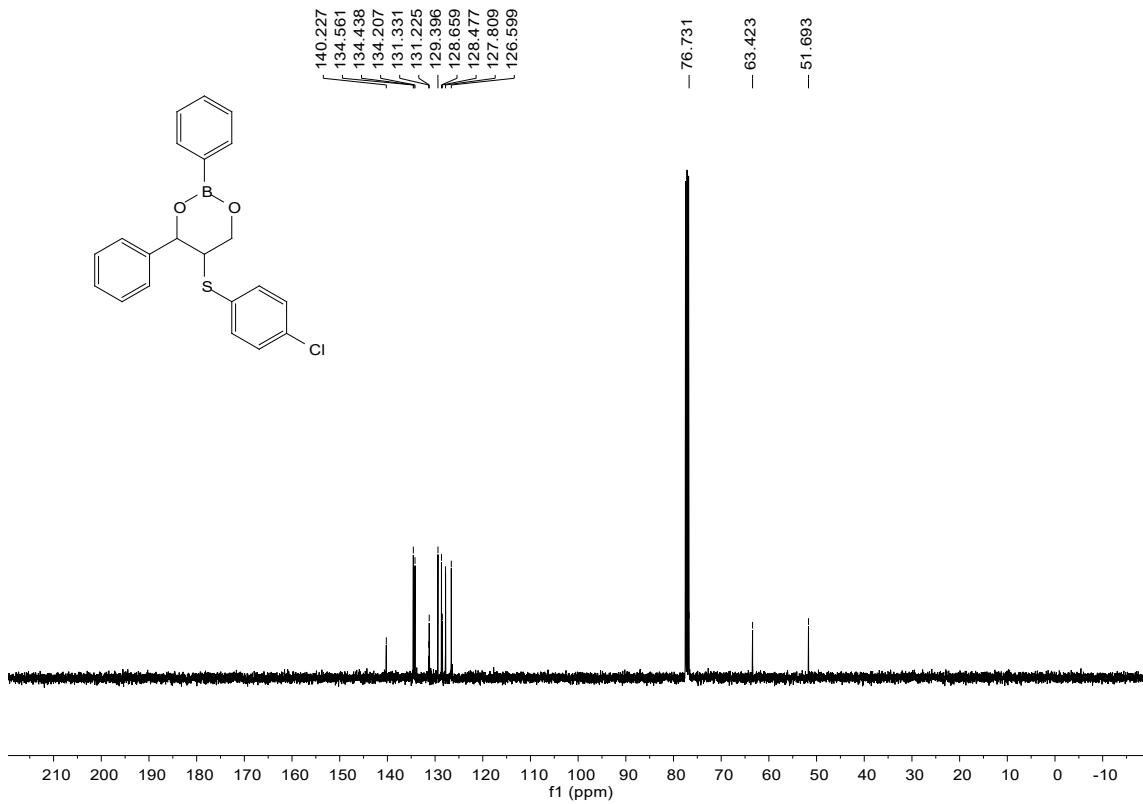
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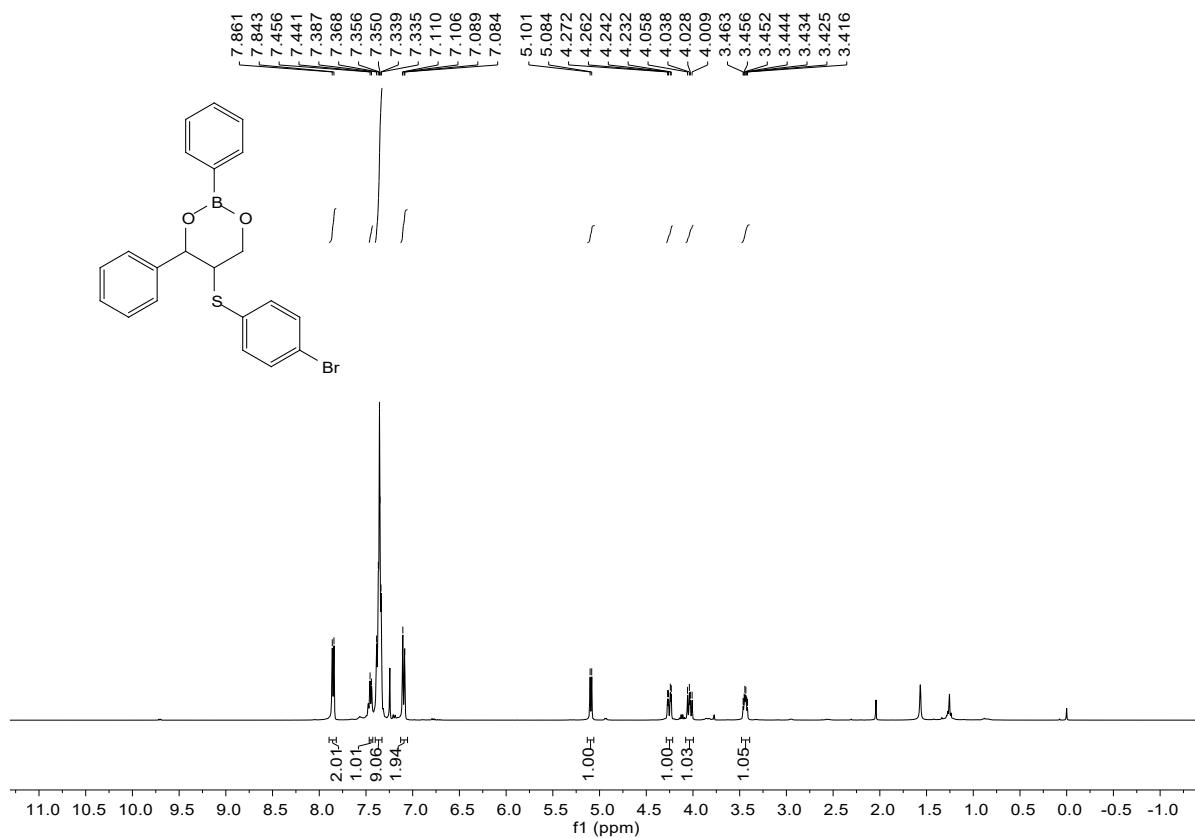
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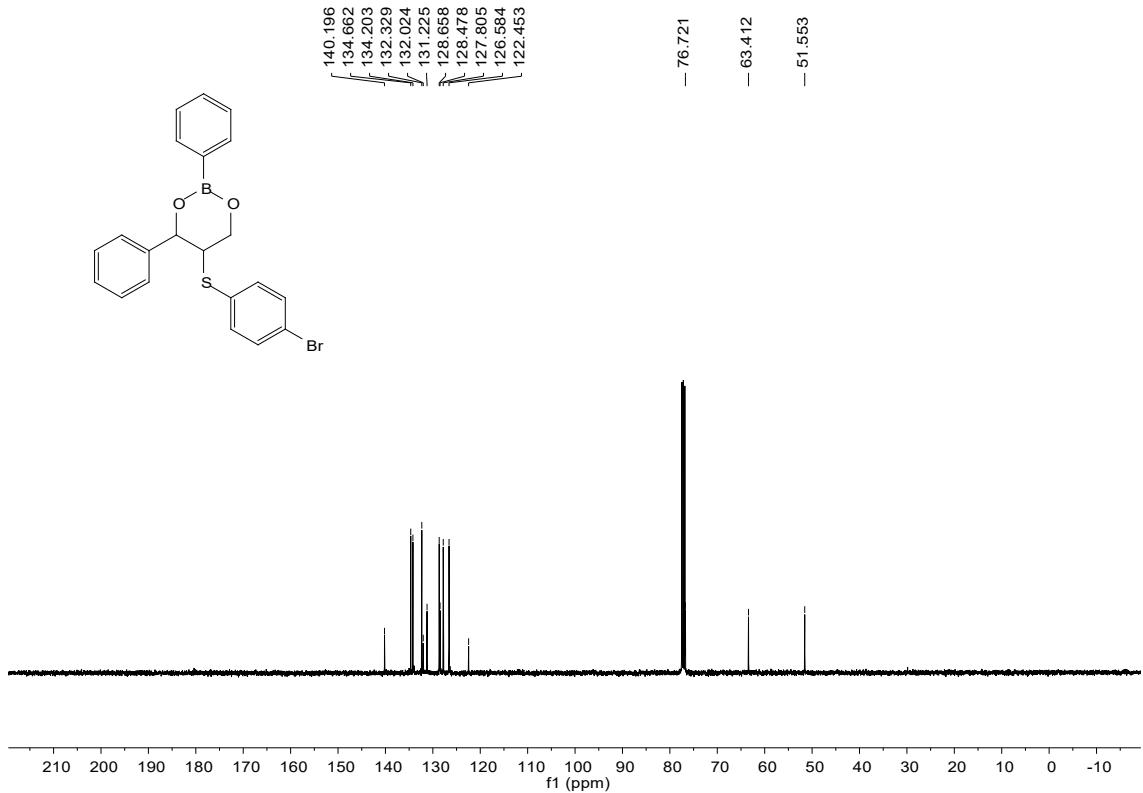
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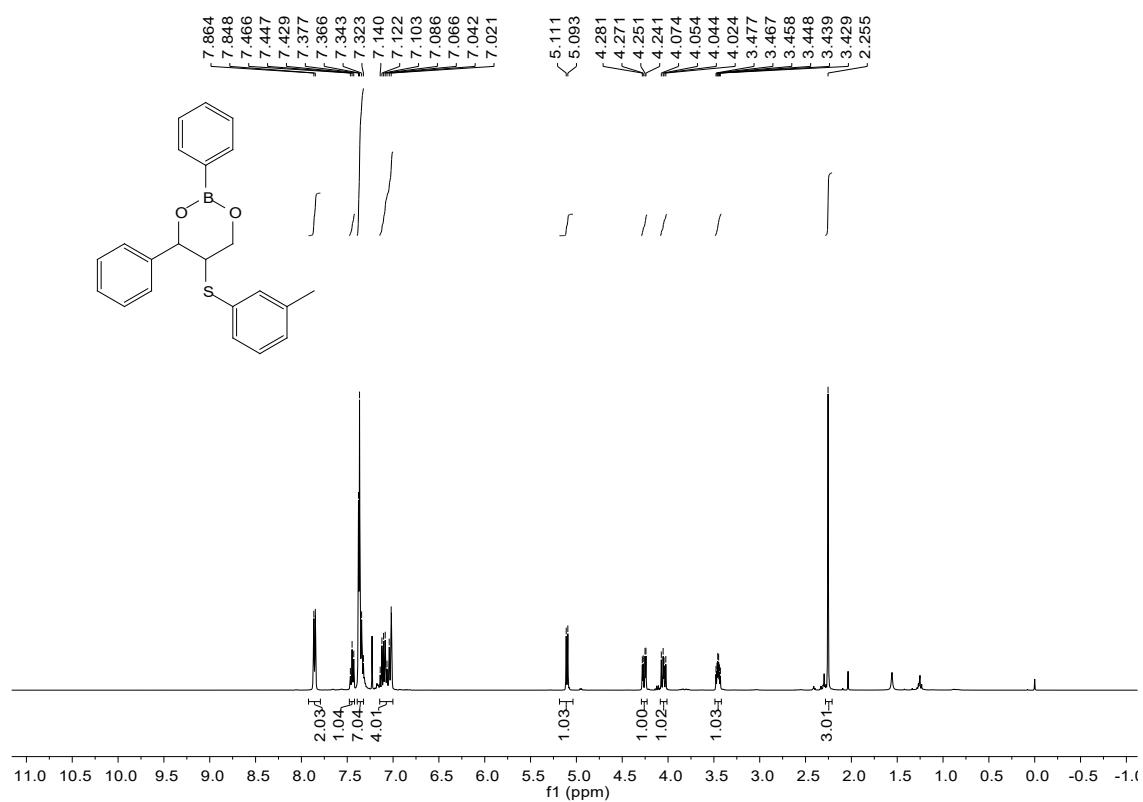
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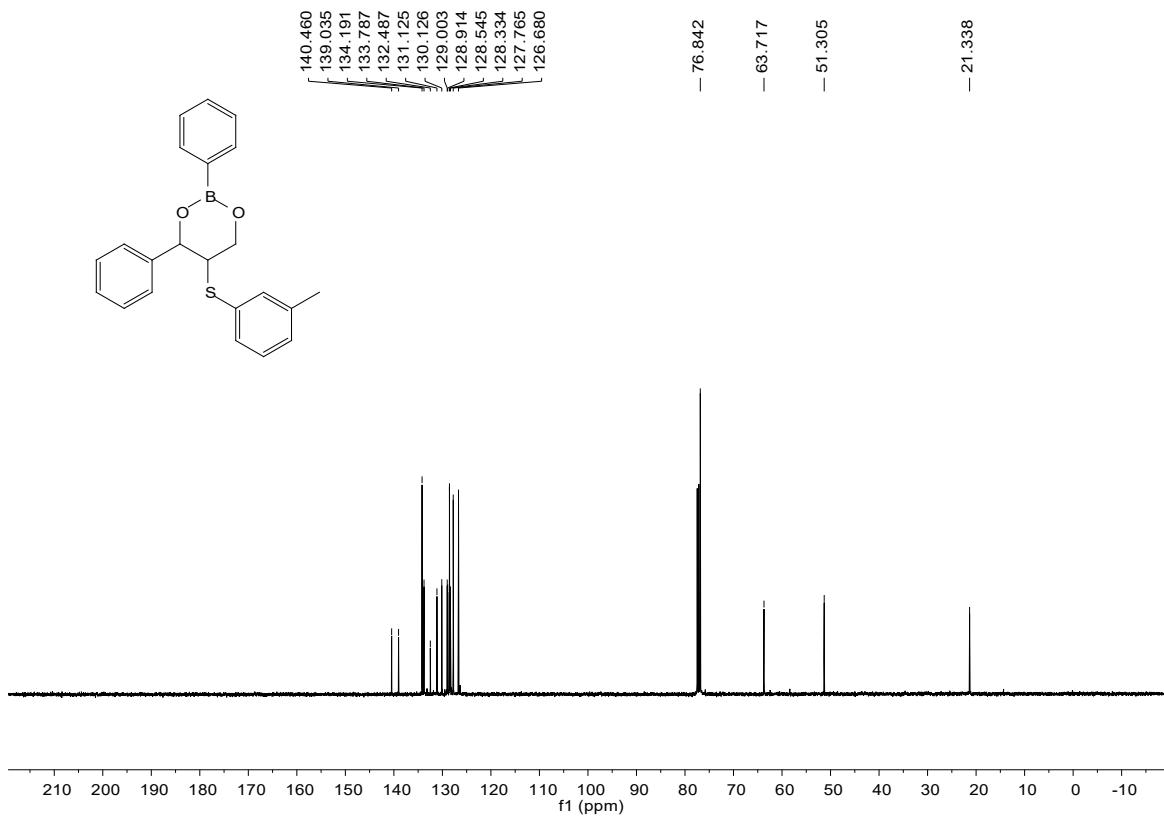
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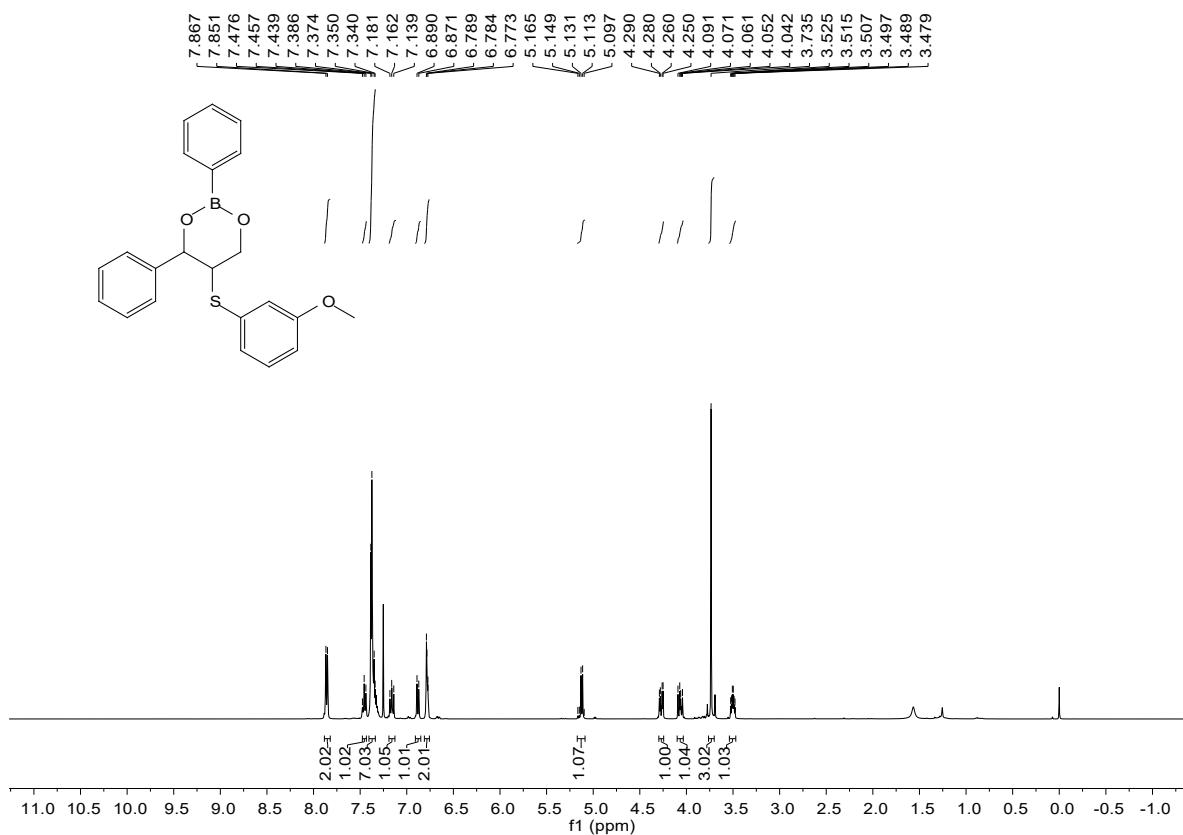
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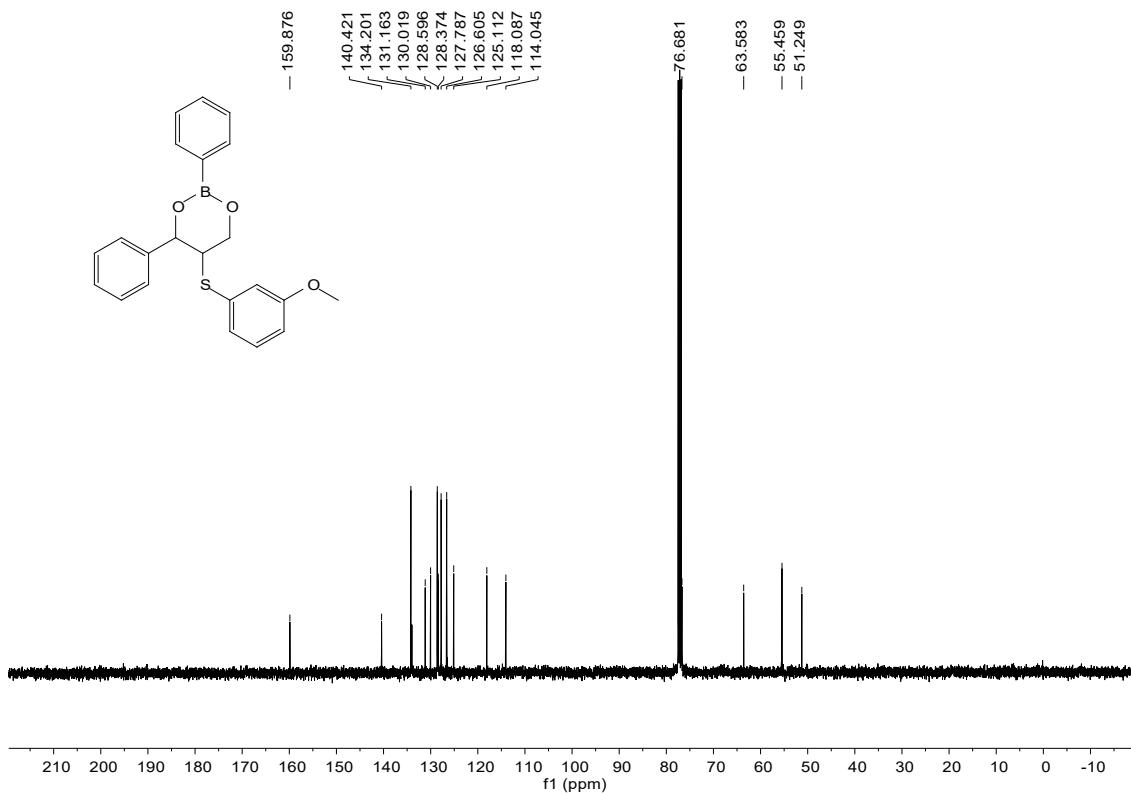
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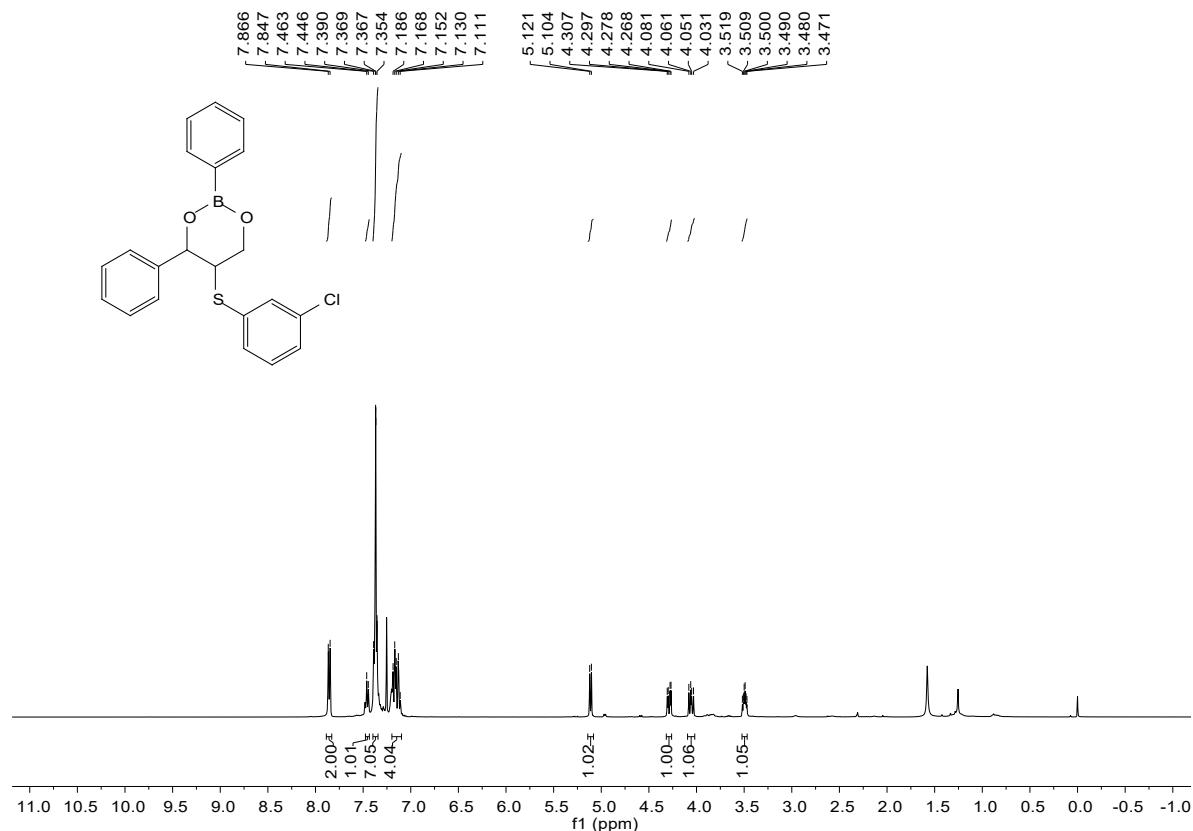
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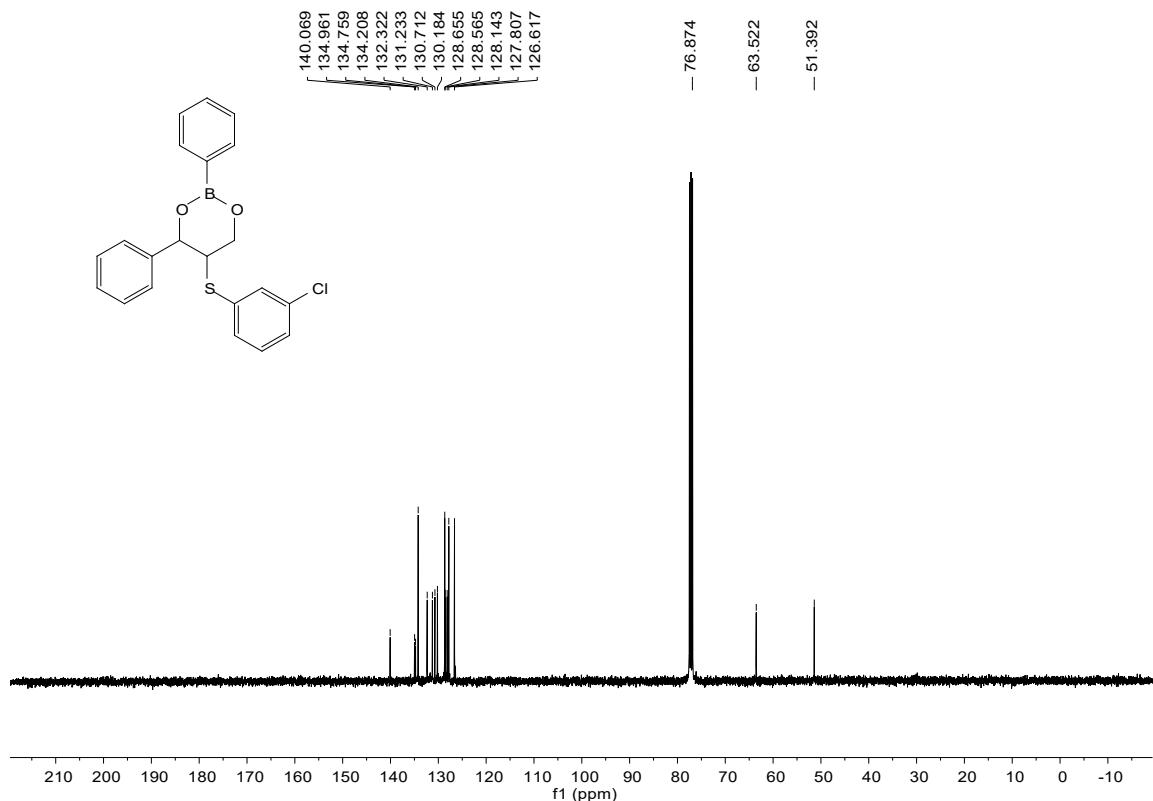
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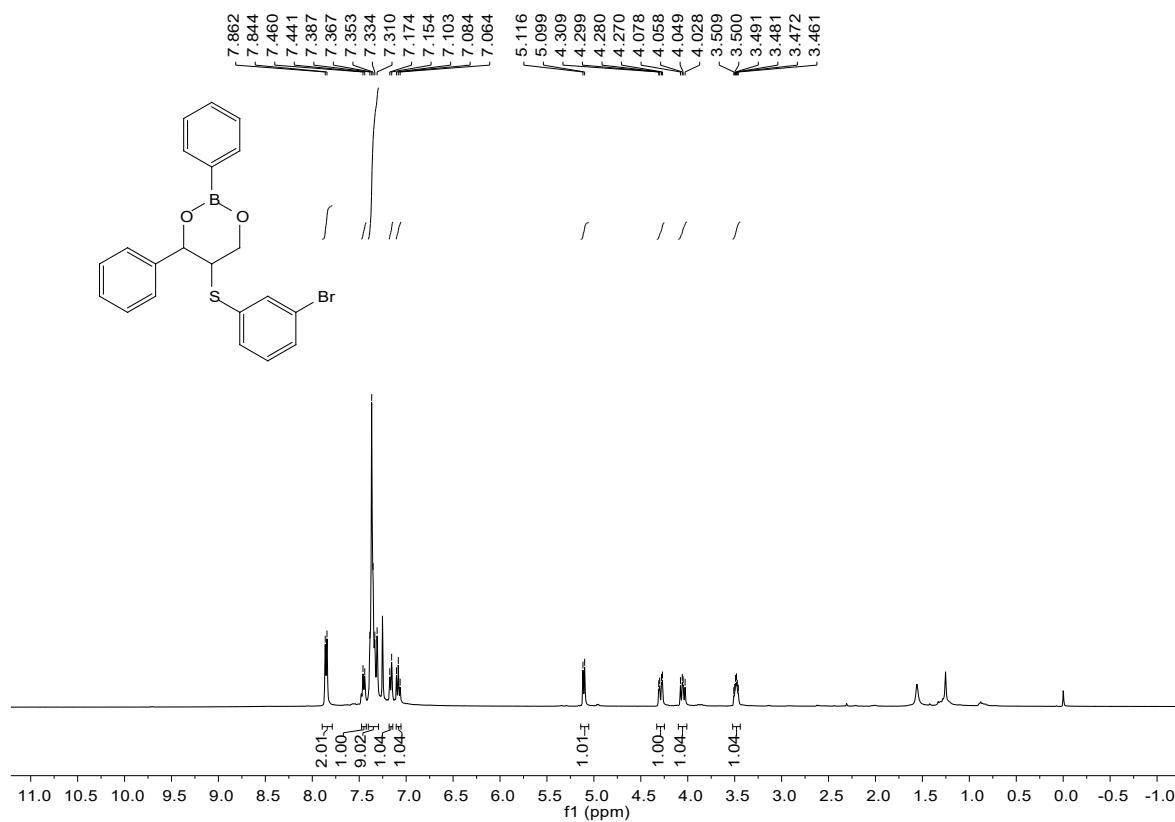
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6y**



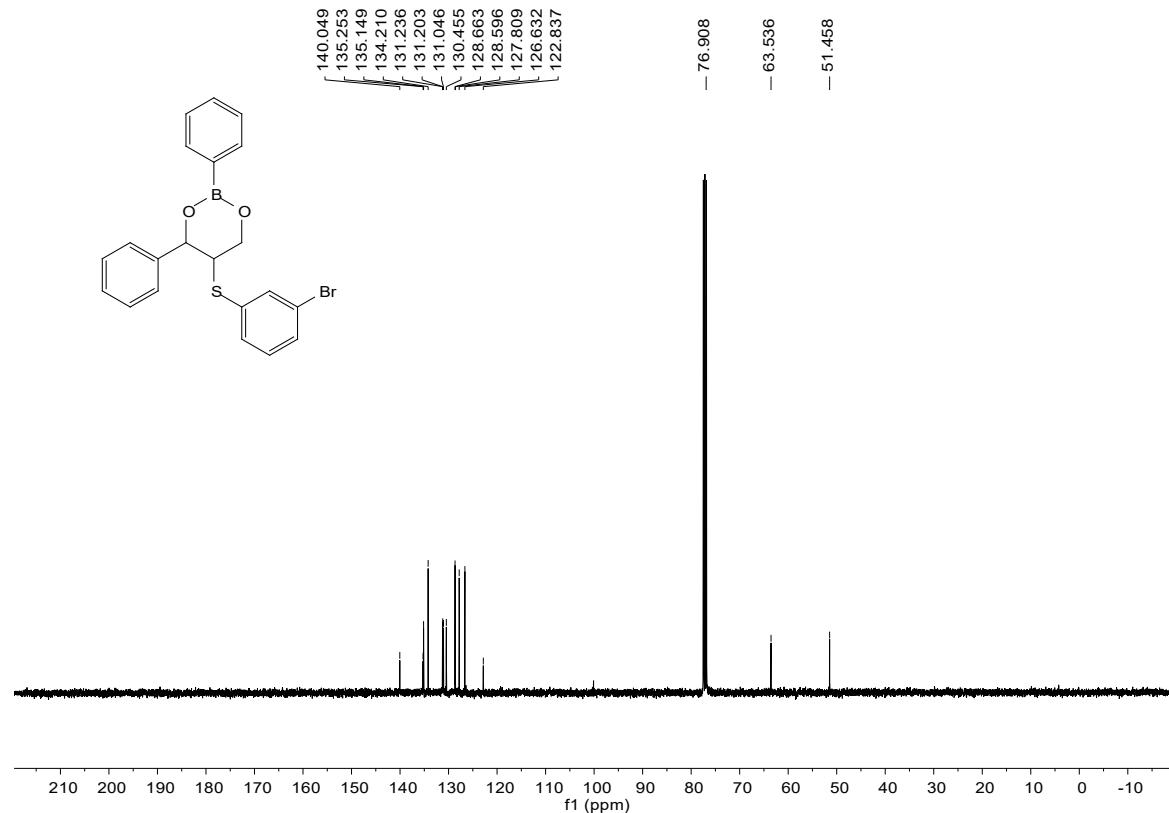
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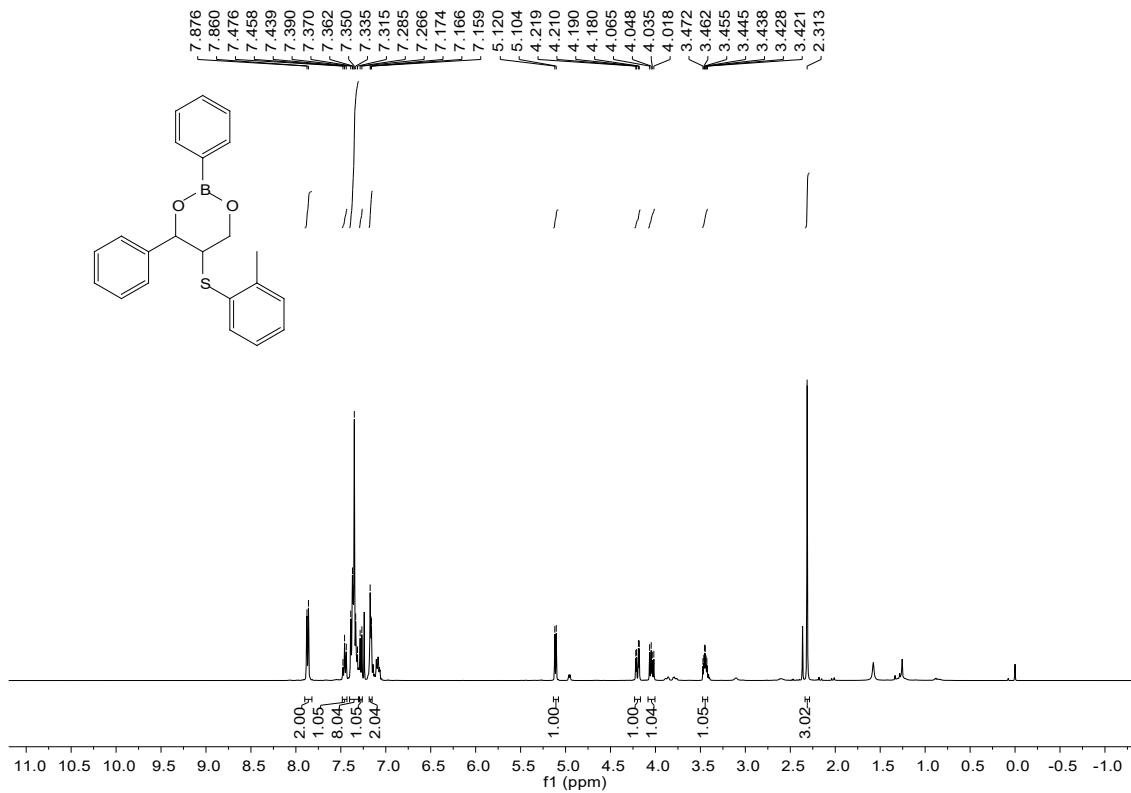
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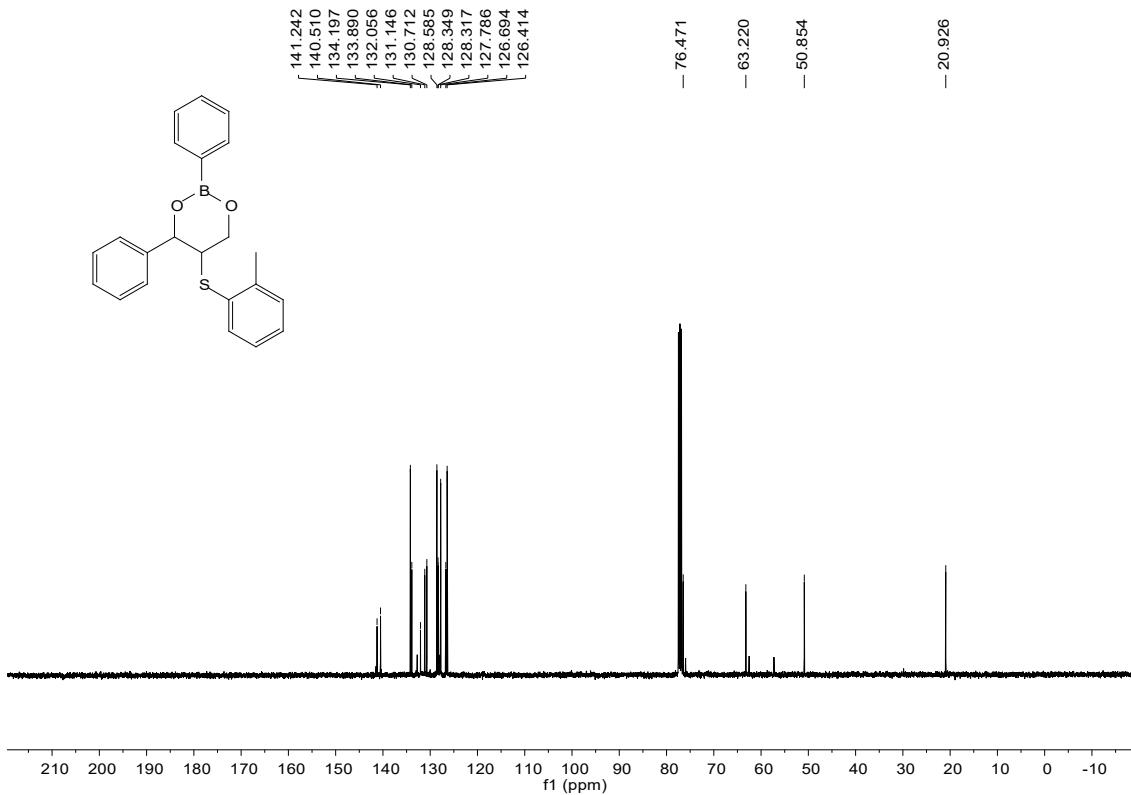
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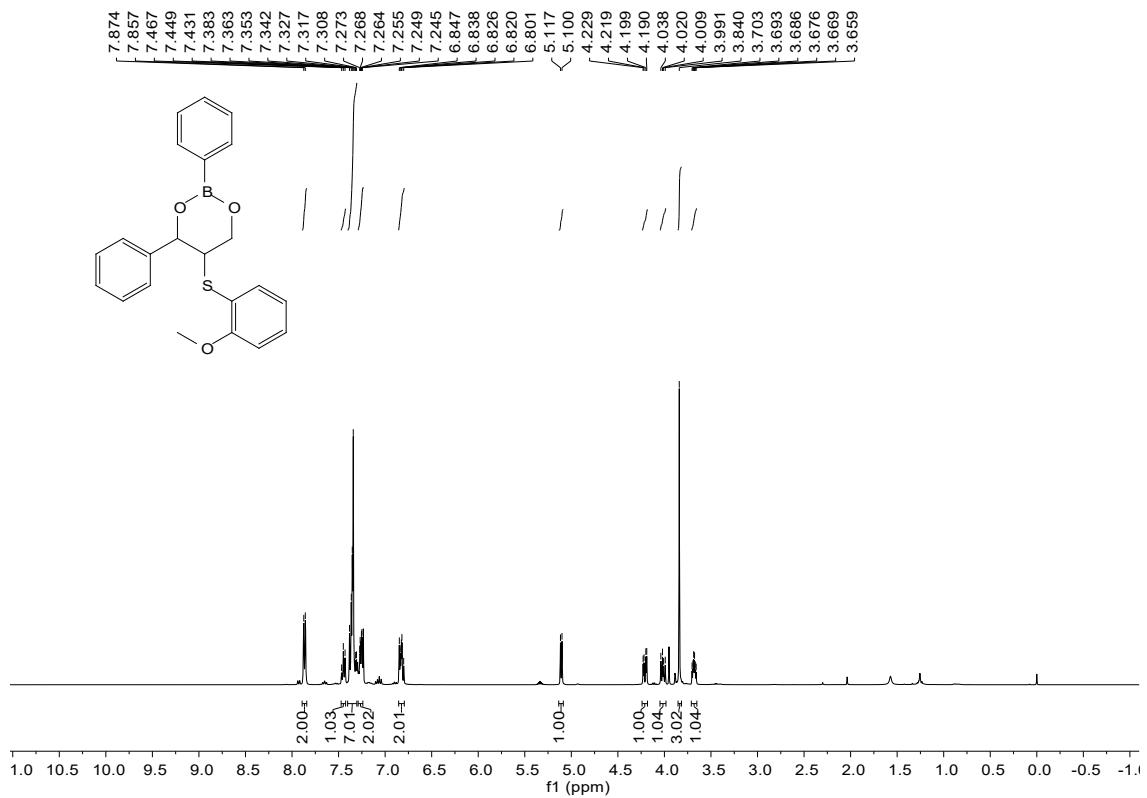
¹H NMR (400 MHz, CDCl₃) spectrum of compound **6aa**



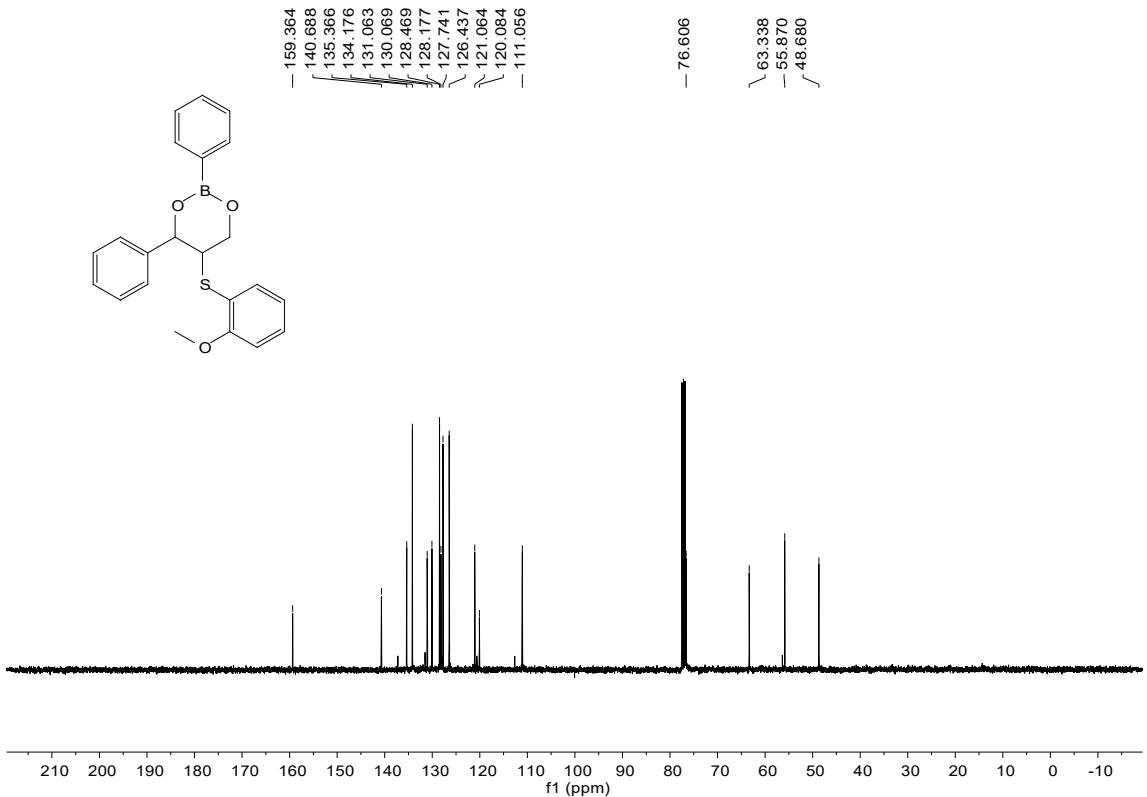
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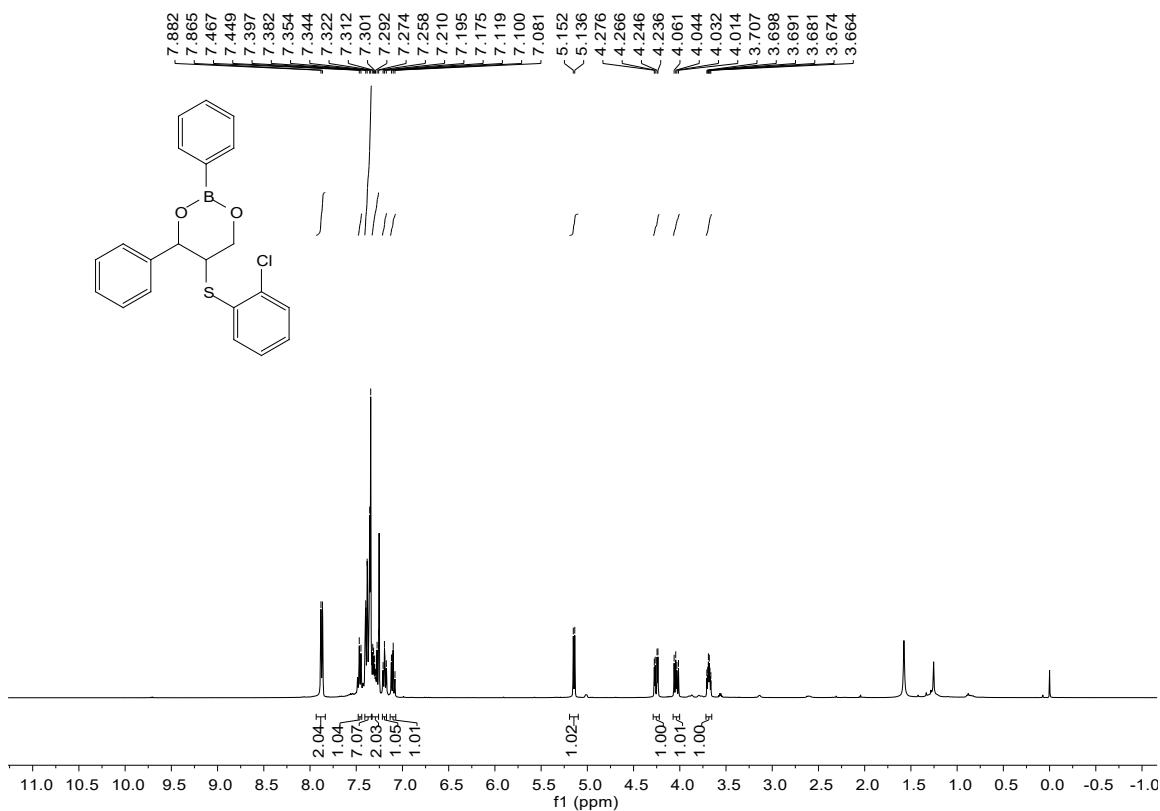
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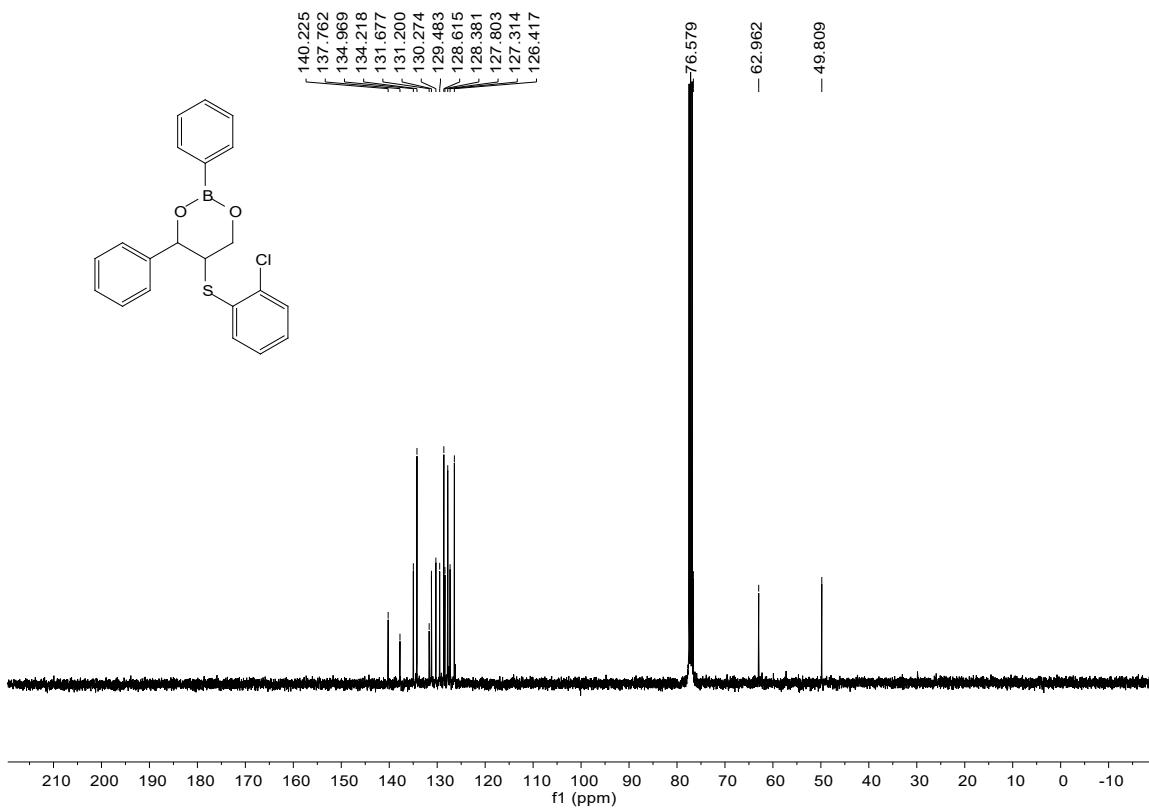
¹³C NMR (100 MHz, CDCl₃) spectrum of compound **6ab**



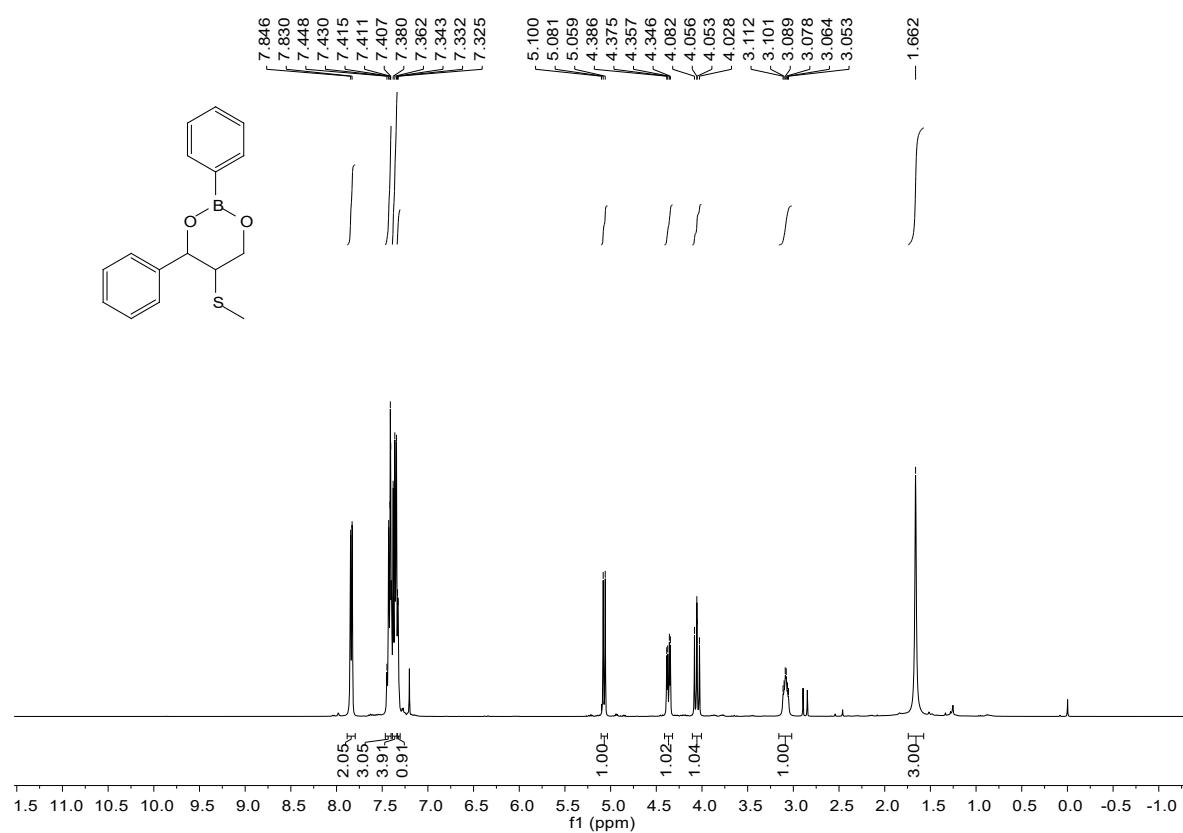
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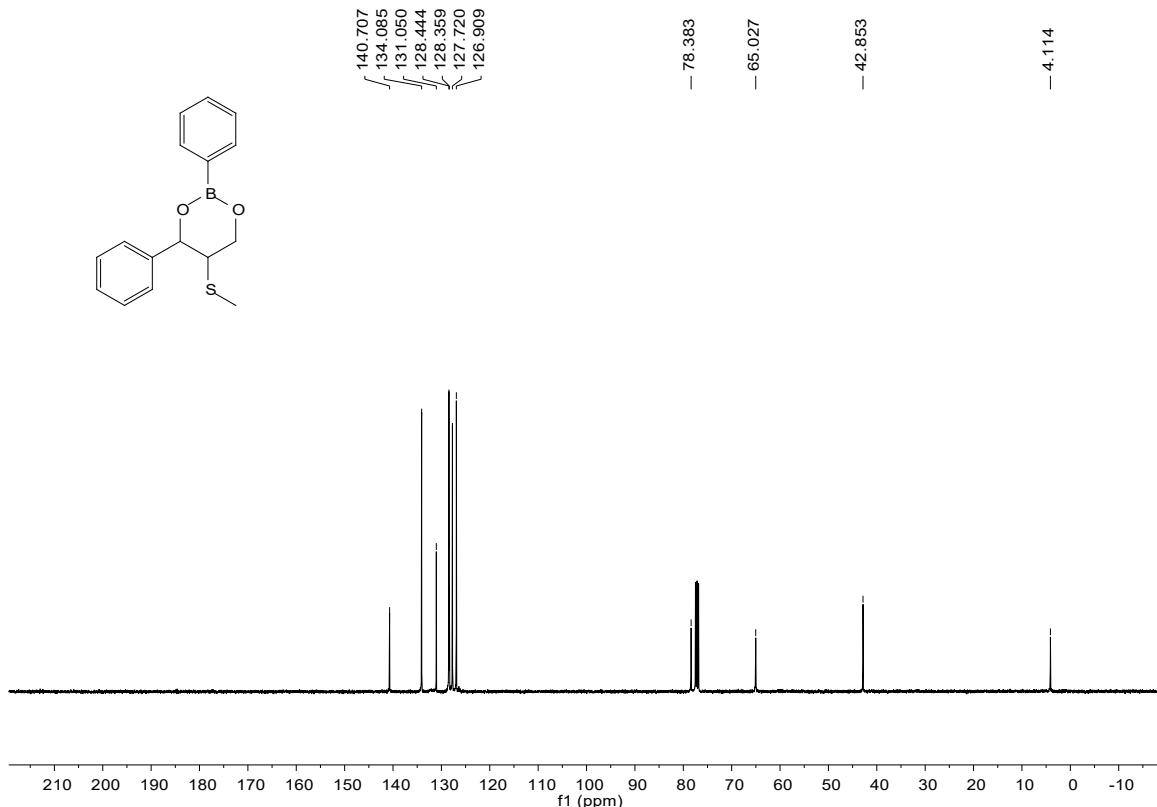
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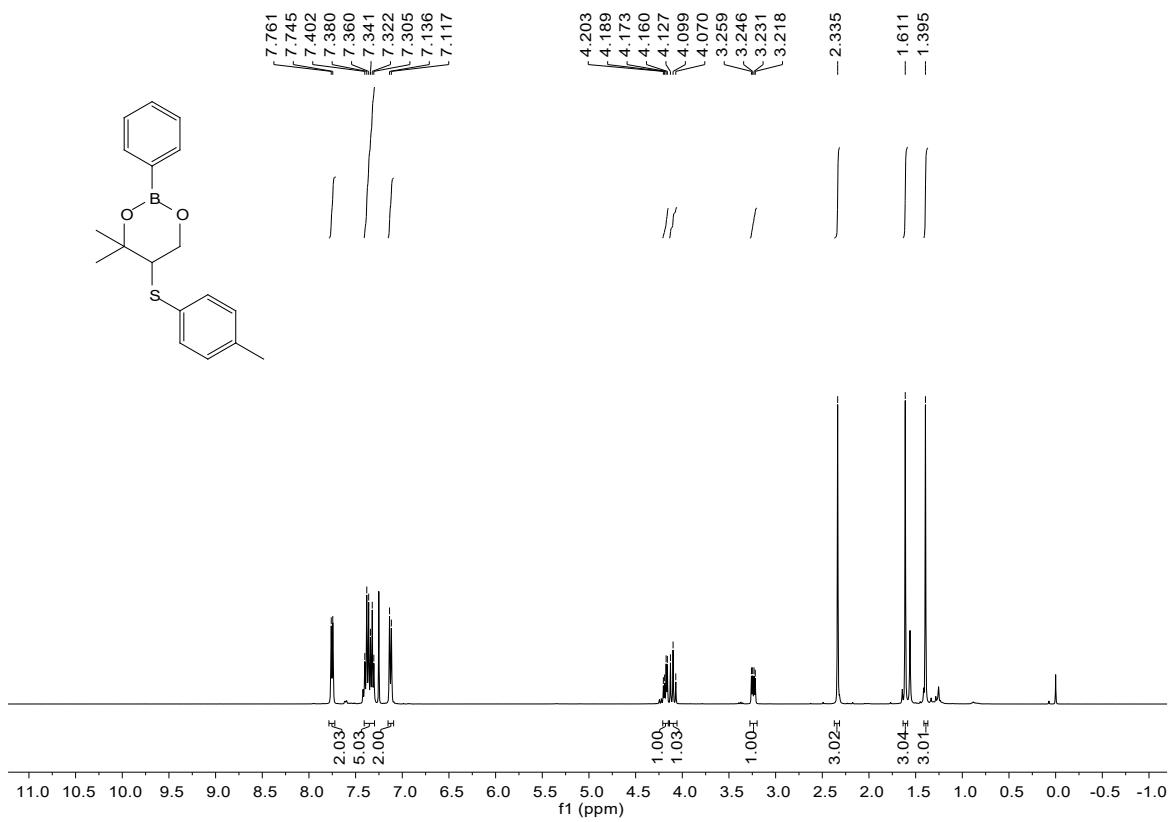
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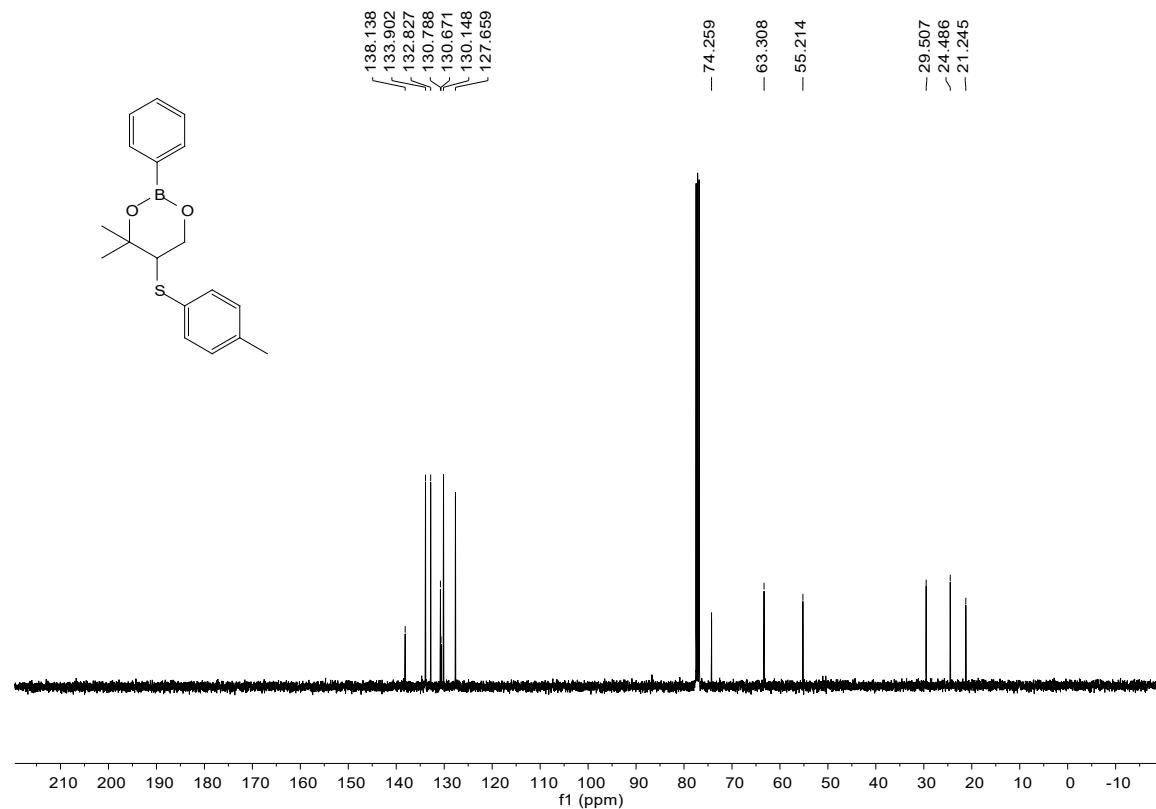
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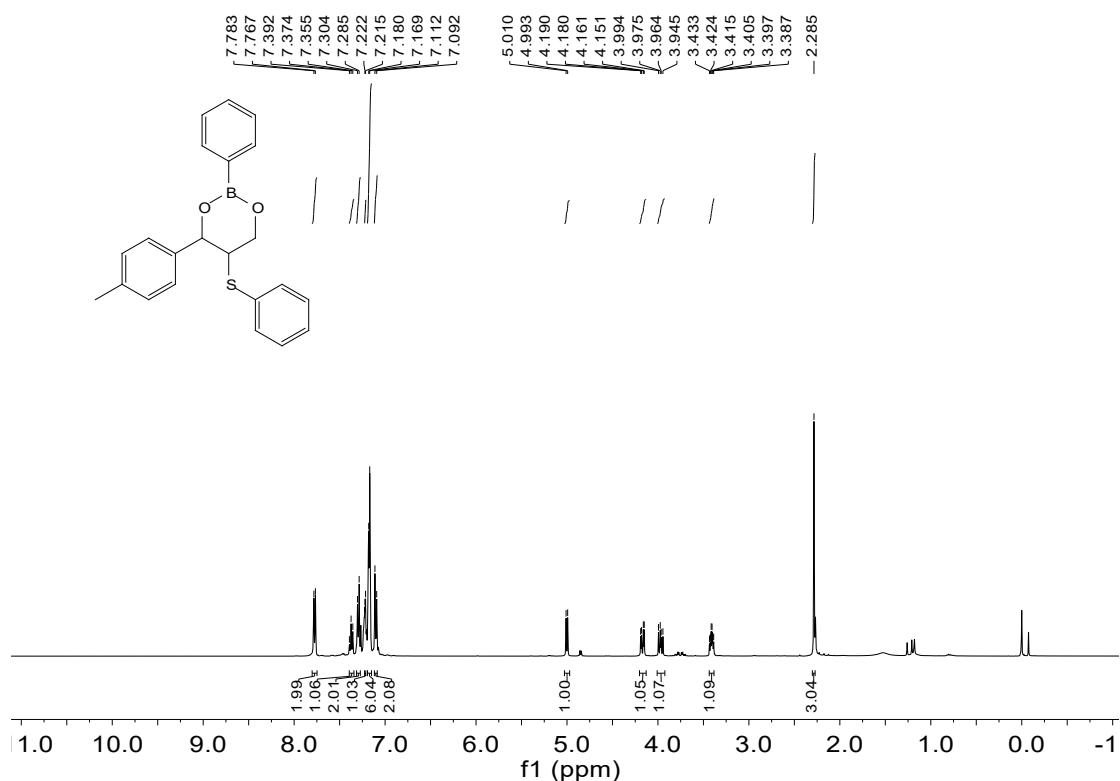
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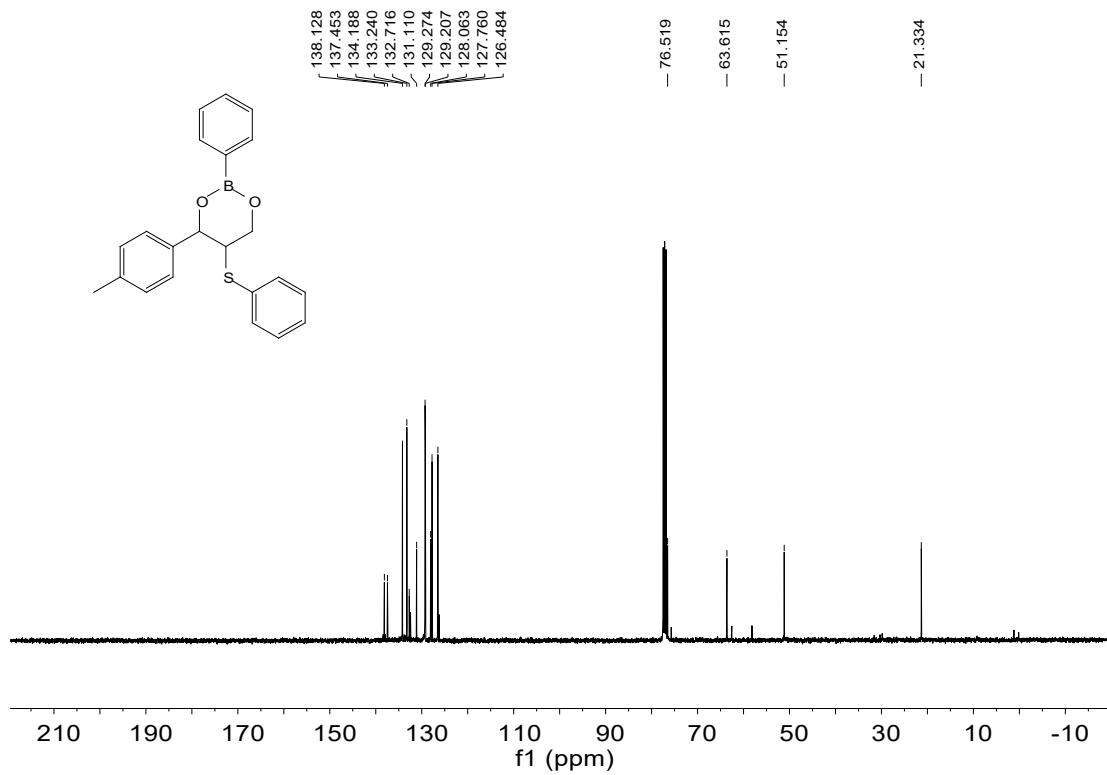
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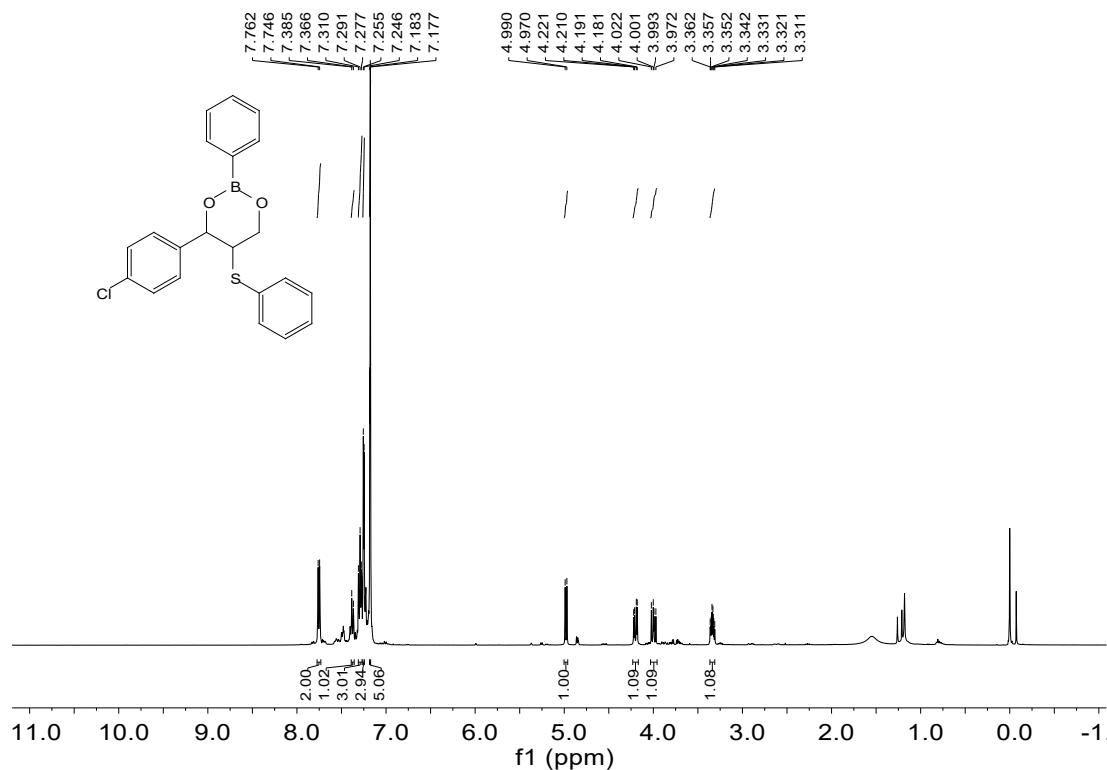
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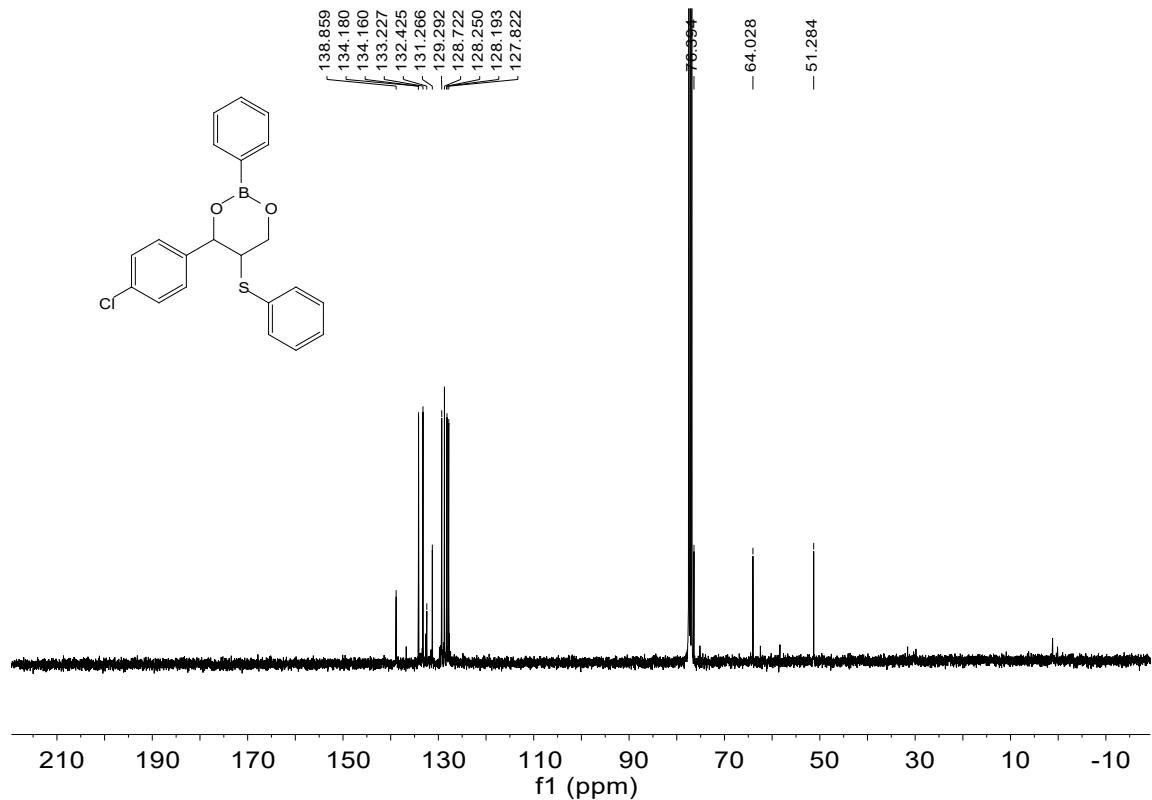
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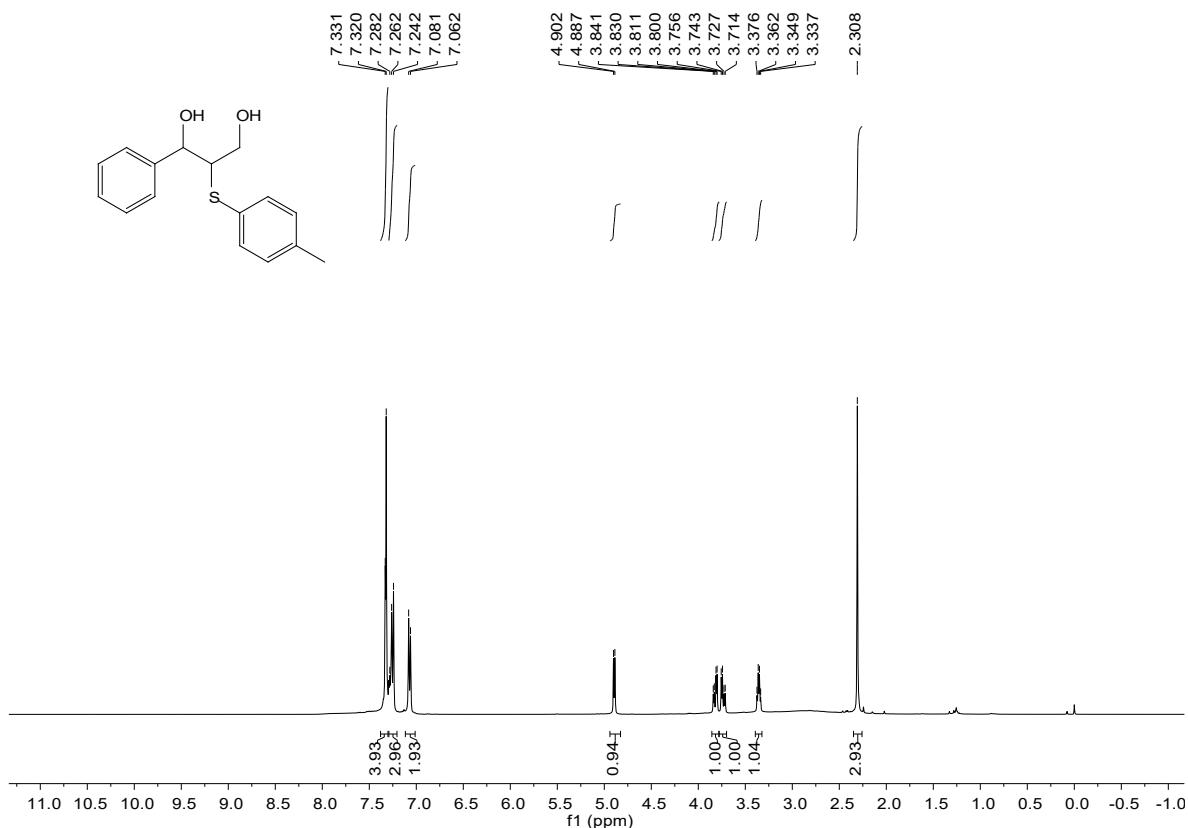
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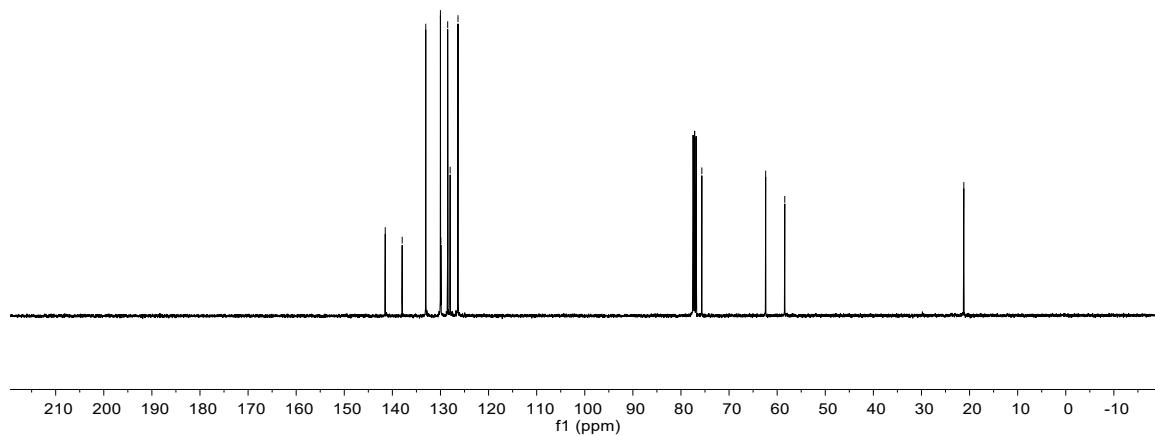
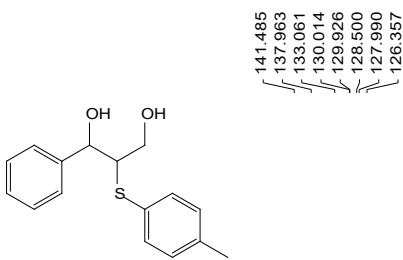
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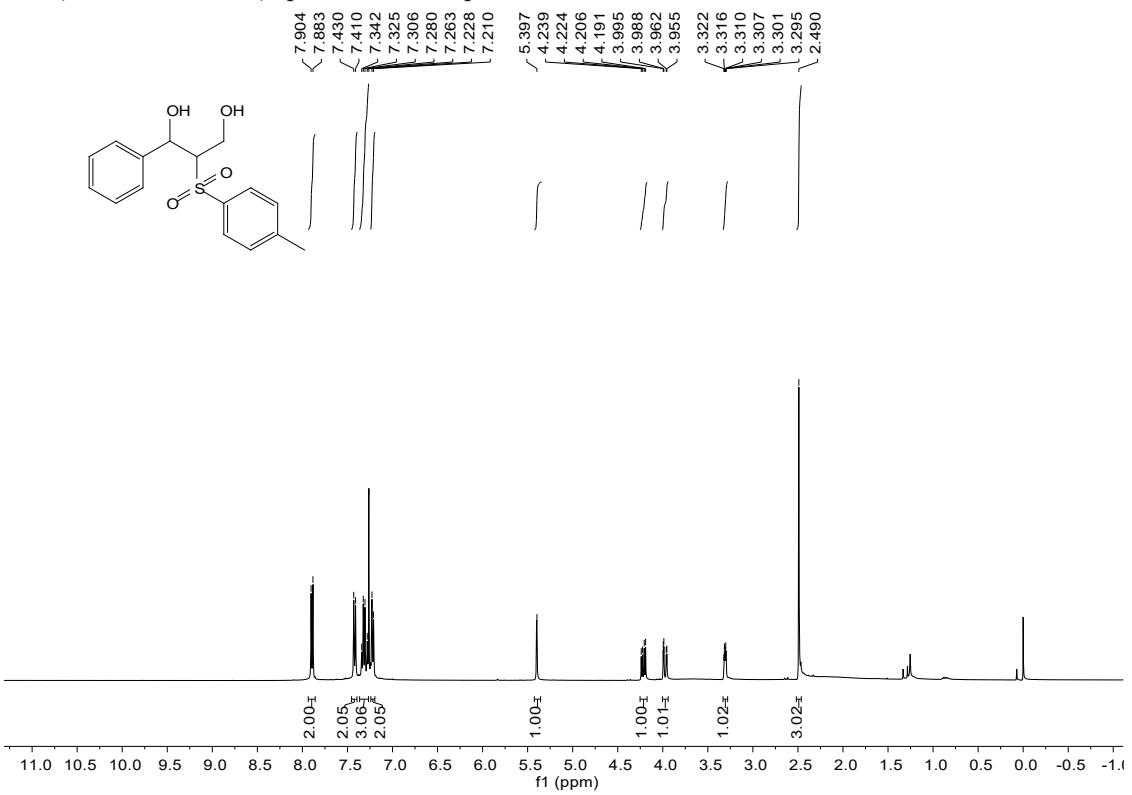
¹H NMR (400 MHz, CDCl₃) spectrum of compound 7



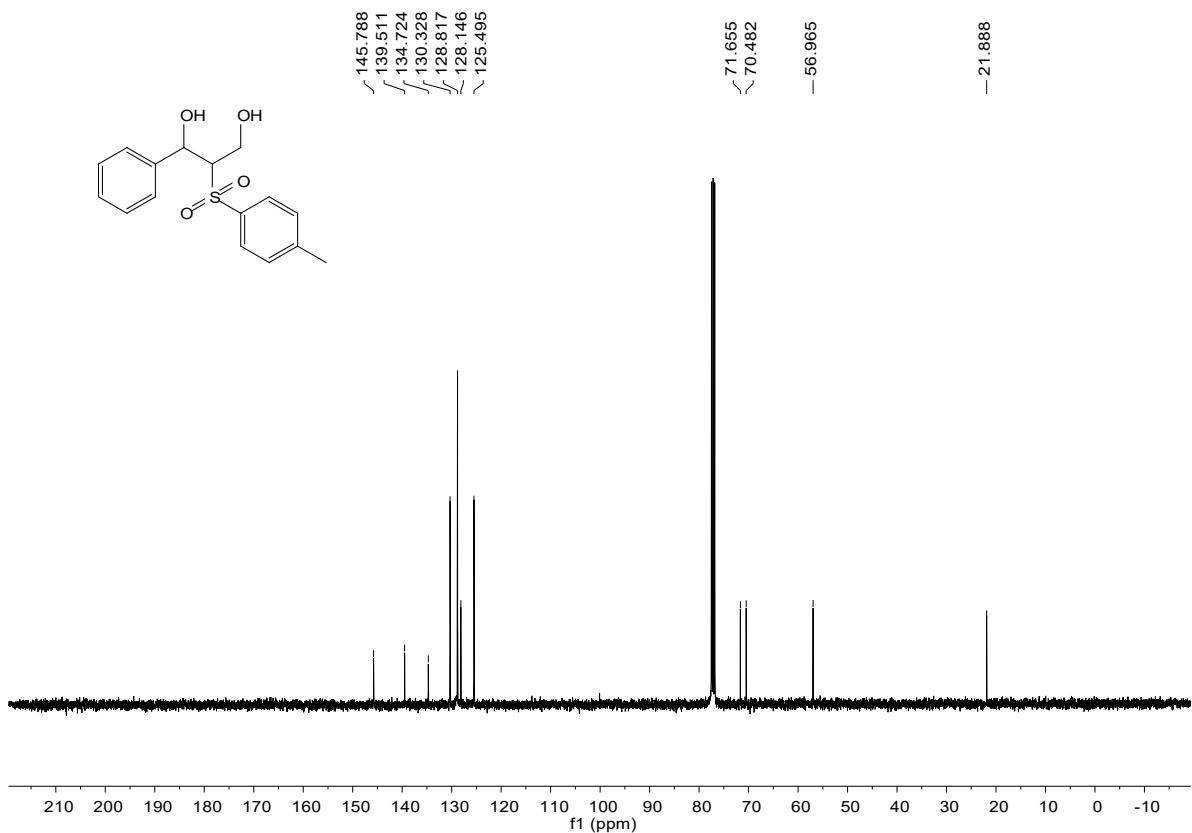
¹³C NMR (100 MHz, CDCl₃) spectrum of compound 7



¹H NMR (400 MHz, CDCl₃) spectrum of compound 8



¹³C NMR (100 MHz, CDCl₃) spectrum of compound 8



4. References

1. (a) J. Li, X. Liu, J. Deng and Y. Huang, *Chem. Commun.*, 2020, **56**, 735-738; (b) D. Singh, Anna M. Deobald and Leandro R. S. Camargo, *Org. Lett.*, 2010, **12**, 3288-3291.