Electronic Supplementary Material (ESI) for Dalton Transactions.

Regioselective B(3)-H bond activation based on an *o*-carboranyl dithiocarboxylate ligand and its derivatives

Contents

1.Experimental details

1.1.Materials:

All reagents and solvents were purchased from commercial sources (Aladdin, Sigma Aldrich, Energy Chemical and Adamas-beta) and used as supplied unless otherwise mentioned. The starting material $[Cp^*IrCl_2]_2$ (Cp^* = η^5-pentamethylcyclopentadienyl), $[Cp^*RhCl_2]_2$ and *o*-$C_2B_{10}H_{11}CSNHPh$ was prepared by a literature method.[16a,b]

1.2.Methods:

NMR spectra were recorded on Bruker AVANCE I 400 and VANCE-DMX 500 spectrometers. Spectra were recorded at room temperature and referenced to the residual protonated solvent for NMR spectra. Proton chemical shift (δH = 7.26 ppm in $CDCl_3$) and (δC = 77.16 ppm in $CDCl_3$) are reported relative to the solvent residual peak. Coupling constants are expressed in Hertz. Baseline subtraction is used in ^{11}B NMR spectra.

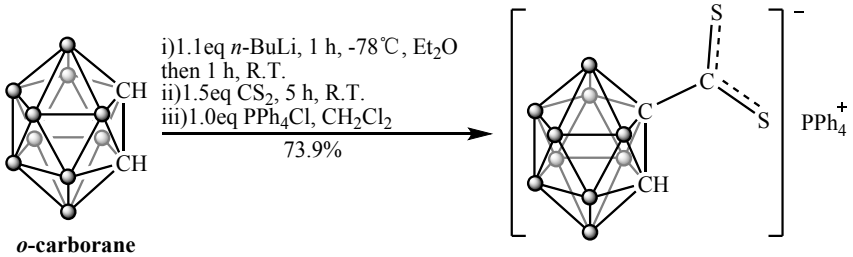
IR spectra of the solid samples (KBr tablets) in the range 400-4000/cm were recorded on a Nicolet AVATAR-360IR spectrometer.

Elemental analyses were performed on an Elementar Vario EL III analyzer.

X-Ray intensity data were collected on a CCD-Bruker SMART APEX system and a Bruker D8 Venture system.

1.3. Synthetic Procedures:

Scheme S1. Synthesis of Ligand 1.



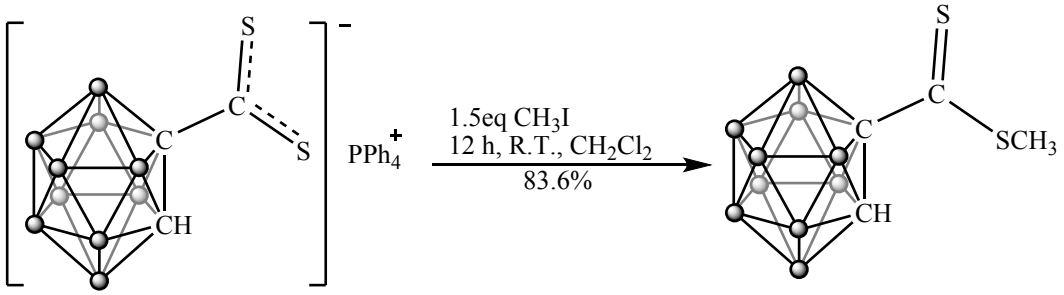
o-carborane

Ligand 1

Synthesis of *o*-$C_2B_{10}H_{11}CS_2PPh_4$ (Ligand 1). In a typical experiment, 660 μL of a *n*-BuLi solution (1.6 mol/L in *n*-hexane, 1.1 mmol, 1.1 equiv) was added to a Schlenk tube which contained a solution of 144.2 mg of *o*-carborane (1.0 mmol, 1.0 equiv) in 10 mL diethyl ether at -78 ℃ over a period of 1 h with continuous stirring, then the

Dewar flask was withdrawn and the stirring was continued at room temperature for another 1 h. 105 μL of CS_2 (1.5 mmol, 1.5 equiv) was added and the resulting mixture was stirred for 5 h. The reaction mixture was transferred to a flask and the ether was removed under vacuum. A thick orange-red oil was obtained, then 15 mL CH_2Cl_2 was added to the flask and the mixture was stirred (*solution A*). 374.8 mg of PPh_4Cl (1.0 mmol, 1.0 eq) was dissolved in 6 mL CH_2Cl_2, then this solution was mixed with *solution A*, whereupon the colour changed from orange-red to pink and a gray cloudy precipitate appeared immediately. The mixture was filtered with a sand-core funnel, then the solvent of the filtrate was removed under vacuum, and a pinkish solid was obtained. The solid was washed with *n*-hexane or petroleum ether and deionised water in a Buchner funnel. During this purification procedure, unreacted *o*-carborane was removed by washing with *n*-hexane, and excess PPh_4Cl was removed by washing with water. After air drying, 413.0 mg of **Ligand 1** was obtained as a light-pink powder, the yield based on *o*-carborane was 73.9%. **Ligand 1** is soluble in ethanol, dichloromethane and chloroform, but insoluble in water and common hydrocarbon solvents. ^{1}H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.45-3.50 (br, 10H, B-H), 5.41 (br, 1H, cage C-H), 7.60 (dd, J = 12.9, 7.7 Hz, 8H, phenyl C-H), 7.75 (td, J = 7.6, 3.4 Hz, 8H, phenyl C-H), 7.89 (t, J = 7.2 Hz, 4H, phenyl C-H). $^{13}C\{^{1}H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 61.79, 90.97 (cage C), 117.43 (d, J = 89.5 Hz, phenyl C), 130.84 (d, J = 12.9 Hz, phenyl C), 134.48 (d, J = 10.3 Hz, phenyl C), 135.87 (d, J = 3.0 Hz, phenyl C), 236.20 (dithiocarboxyl C). $^{11}B\{^{1}H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -4.47, -6.09, -10.44, -14.33. IR (KBr disk, cm^{-1}): ν = 3053.40, 3030.45, 2624.48, 2592.65, 2566.90, 2551.93, 1587.00, 1485.65, 1435.87, 1162.99, 1100.38, 1084.42, 1062.80, 1048.71, 1012.84, 997.02, 780.30, 753.89, 721.76, 689.23. Anal. calcd for **Ligand 1** ($C_{27}H_{31}B_{10}PS_2$): C, 58.04; H, 5.59; Found: C, 57.80; H, 5.45.

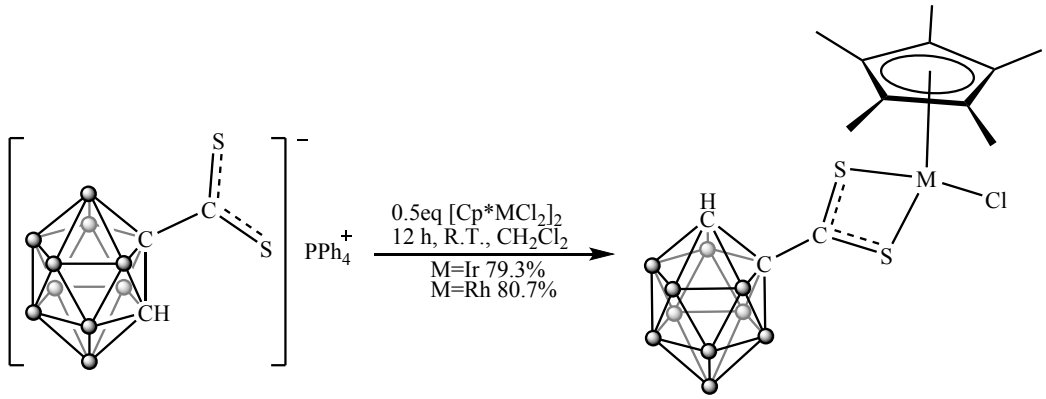
Scheme S2. Synthesis of Ligand 2.



Ligand 1 **Ligand 2**

Synthesis of *o*-$C_2B_{10}H_{11}CS_2CH_3$ (Ligand 2). In a typical experiment, 558.7 mg of **Ligand 1** (1.0 mmol, 1.0 equiv) was added to a flask with 10 mL CH_2Cl_2, then 98 μL of CH_3I (1.5 mmol, 1.5 equiv) was added to the solution and the flask was sealed. The mixture was stirred for 12 h at room temperature over which time its colour changed from pink to yellow. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with *n*-hexane gave **Ligand 2** as a yellow solid (R_f = 0.76 in pure *n*-hexane). 196.0 mg of **Ligand 2** was obtained, the yield based on **Ligand 1** was 83.6%. **Ligand 2** is soluble in common organic solvents. ^{1}H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.70-3.50 (br, 10H, B-H), 2.68 (s, 3H, S-methyl C-H), 4.86 (br, 1H, cage C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 22.85 (S-methyl C), 61.06, 82.23 (cage C), 219.99 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -3.22, -8.63, -10.47, -11.28, -13.32. IR (KBr disk, cm^{-1}): ν = 3043.70, 2915.62, 2627.43, 2603.34, 2571.21, 1199.82, 1132.65, 1088.44, 1042.24, 1013.38, 934.32. Anal. calcd for **Ligand 2** ($C_4H_{14}B_{10}S_2$): C, 20.50; H, 6.02; Found: C, 20.55; H, 6.08.

Scheme S3. Synthesis of 3 and 4.



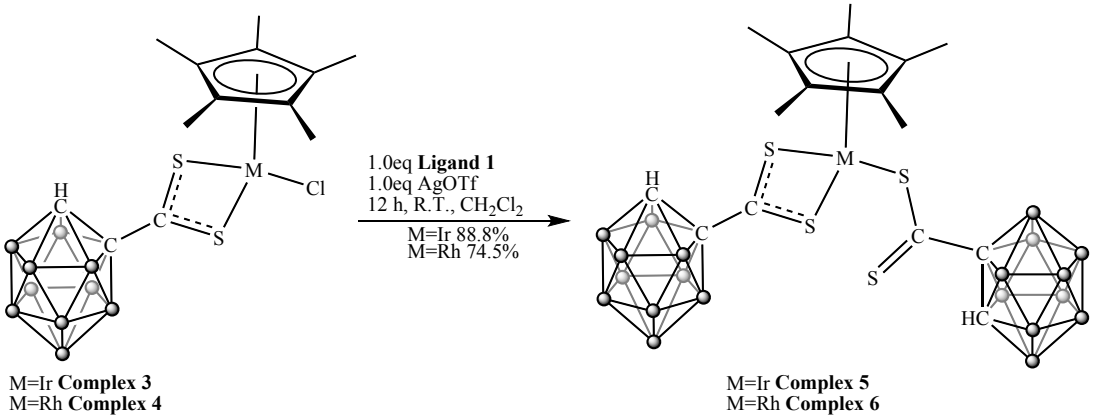
Ligand 1

M=Ir **Complex 3**
M=Rh **Complex 4**

Synthesis of Cp*Ir(*o*-$C_2B_{10}H_{11}CS_2$)Cl (3). In a typical experiment, 279.4 mg of **Ligand 1** (0.5 mmol, 1.0 equiv) was added to a 50 mL Schlenk tube with 10 mL CH_2Cl_2, and 199.2 mg of $[Cp^*IrCl_2]_2$ (0.25 mmol, 0.5 equiv) was then added, whereupon the colour immediately changed from pink to almost opaque black-green. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 1:1 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **3** as a black-green powder (R_f = 0.28 in 1:1 CH_2Cl_2 / *n*-hexane mixture). 231.0 mg of **3** was obtained, the yield based on **Ligand 1** was 79.3%. **3** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400MHz; $CDCl_3$, ppm): δ = 1.83 (s, 15H, Cp*-H), 1.70-3.25 (br, 10H, B-H), 4.25 (br, 1H, cage C-H). $^{13}C\{^1H\}$ NMR (101MHz; $CDCl_3$, ppm): δ = 9.53 (Cp*-C), 58.30, 88.45 (cage C), 90.77 (Cp*-C), 234.82 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -2.66, -8.82, -11.44, -13.18. IR (KBr disk, cm^{-1}): ν = 3052.19, 2958.37, 2919.12, 2606.72, 2554.33, 1449.73, 1377.69, 1240.27, 1146.46, 1081.88, 1040.01. Anal. calcd for **3** ($C_{13}H_{26}B_{10}S_2ClIr$): C, 26.82; H, 4.50; Found: C, 26.73; H, 4.45.

Synthesis of Cp*Rh(*o*-$C_2B_{10}H_{11}CS_2$)Cl (4). In a typical experiment, 167.6 mg of **Ligand 1** (0.3 mmol, 1.0 equiv) was added to a 50 mL Schlenk tube with 10 mL CH_2Cl_2, and 92.7 mg of $[Cp^*RhCl_2]_2$ (0.15mmol, 0.5 equiv) was then added, whereupon the colour immediately changed from pink to almost opaque black-red. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 3:1 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **4** as a deep-brown powder (R_f = 0.42 in 3:1 CH_2Cl_2-*n*-hexane mixture). 119.4 mg of **4** was obtained, the yield based on Ligand 1 was 80.7%. **4** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.78 (s, 15H, Cp*-H), 1.65-3.40 (br, 10H, B-H), 4.34 (br, 1H, cage C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 9.64 (Cp*-C), 57.99, 83.26 (cage C), 97.45 (Cp*-C), 237.58 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -2.79, -8.83, -11.46, -13.32. IR (KBr disk, cm^{-1}): ν = 2051.06, 2975.16, 2914.33, 2062.18, 2566.93, 1495.34, 1446.85, 1379.52, 1141.10, 1043.05, 1021.79. Anal. calcd for **4** ($C_{13}H_{26}B_{10}S_2ClRh$): C, 31.68; H, 5.32; Found: C, 31.41; H, 5.20.

Scheme S4. Synthesis of 5 and 6.

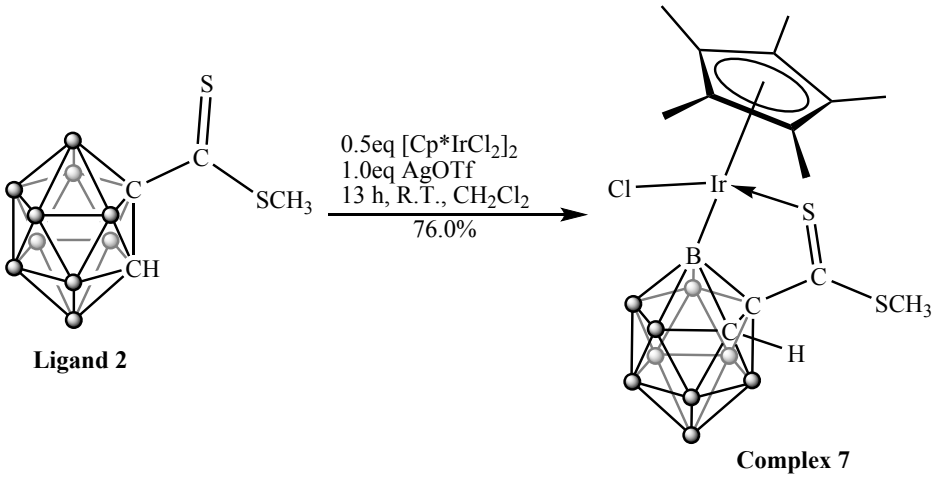


Synthesis of Cp*Ir(*o*-$C_2B_{10}H_{11}CS_2)_2$ (5). In a typical experiment, 27.9 mg of **Ligand 1** (0.05 mmol, 1.0 equiv) and 29.1 mg of **3** (0.05 mmol, 1.0 equiv) were added to a 25 mL Schlenk tube with 8 mL CH_2Cl_2, and 12.8 mg of AgOTf (0.05 mmol, 1.0 equiv) was then added. After the reagent addition was complete, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 1:5 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **5** as a dark-red powder (R_f = 0.47 in 1:3 CH_2Cl_2 / *n*-hexane mixture). 34.0 mg of **5** can be obtained, the yield based on **5** was 88.8%. **5** is soluble in CH_2Cl_2 and $CHCl_3$. ^{1}H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.84 (s, 15H, Cp*-H), 1.75-3.50 (br, 20H, B-H), 4.13, 5.00 (br, 1H, cage C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 9.40 (Cp*-C), 57.88, 63.24, 85.45, 88.43 (cage C), 93.52 (Cp*-C), 228.10 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -2.65, -3.66, -8.79, -11.55, -13.26. IR (KBr disk, cm^{-1}): ν = 3059.36, 3040.76, 2960.82, 2920.95, 2574.40, 1485.17, 1448.80, 1381.01, 1149.02, 1102.21, 1081.49, 1040.81, 1013.35. Anal. calcd for **5** ($C_{16}H_{37}B_{20}S_4Ir$): C, 25.08; H, 4.87; Found: C, 25.21; H, 4.90.

Synthesis of Cp*Rh(*o*-$C_2B_{10}H_{11}CS_2)_2$ (6). In a typical experiment, 55.9 mg of **Ligand 1** (0.1 mmol, 1.0 equiv) and 49.3 mg of **4** (0.1 mmol, 1.0 equiv) were added to a 25 mL Schlenk tube with 8 mL CH_2Cl_2, and 25.7 mg of AgOTf (0.1 mmol, 1.0 equiv) was then added. After the reagent addition was complete, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column

chromatography on silica gel. Elution with a 1:3 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **6** as a red powder (R_f = 0.32 in 1:2 CH_2Cl_2 / *n*-hexane mixture). 50.4 mg of **6** was obtained, the yield based on **6** was 74.5%. **6** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.78 (s, 15H, Cp*-H), 1.70-3.40 (br, 20H, B-H), 4.24, 5.00 (br, 1H, cage C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 9.65 (Cp*-C), 57.60, 62.84, 82.78, 85.85 (cage C), 99.62 (Cp*-C), 229.96, 234.70 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -2.77, -3.61, -8.81, -11.53, -13.41. IR (KBr disk, cm^{-1}): ν = 3058.95, 3040.32, 2962.37, 2906.56, 2573.47, 1485.89, 1444.90, 1380.58, 1261.53, 1143.09, 1099.26, 1080.75, 1013.05. Anal. calcd for **6** ($C_{16}H_{37}B_{20}S_4Rh$): C, 28.39; H, 5.51; Found: C, 28.45; H, 5.32.

Scheme S5. Synthesis of 7.

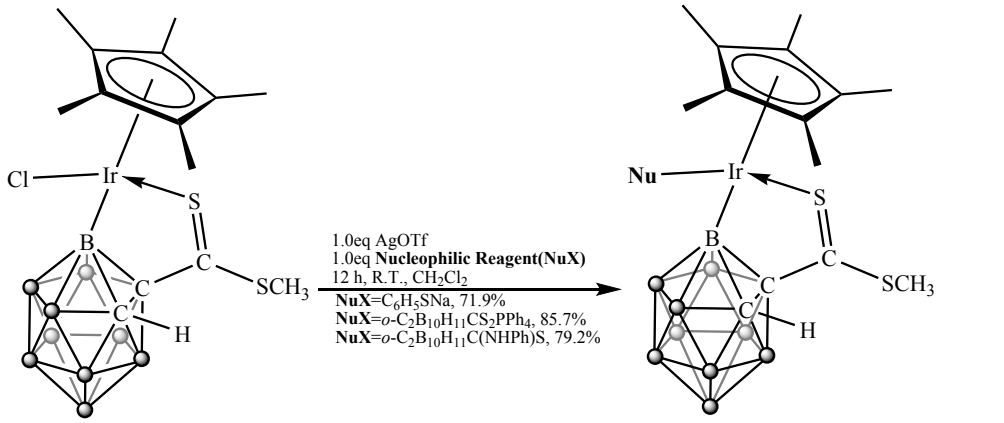


Ligand 2 **Complex 7**

Synthesis of Cp*Ir(*o*-$C_2B_{10}H_{10}CS_2CH_3$)Cl (7). In a typical experiment, 70.3 mg of **Ligand 2** (0.3 mmol, 1.0 equiv) and 119.5 mg of $[Cp^*IrCl_2]_2$ (0.15 mmol, 0.5 equiv) were added to a 50 mL Schlenk tube with 10 mL CH_2Cl_2, and 77.1 mg of AgOTf (0.3 mmol, 1.0 eq) was then added. After the reagent addition was complete, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 13 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 1:1 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **7** as a black powder (R_f = 0.31 in 1:1 CH_2Cl_2 / *n*-hexane mixture). 136.0 mg of **7** was obtained, the yield based on **Ligand 2** was 76.0%. **7** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.83 (s, 15H, Cp*-H), 1.70-3.40 (br, 9H, B-H), 4.58 (br, 1H, cage C-H), 2.75 (s, 3H, S-methyl C-H).

$^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 9.04 (Cp*-C), 22.25 (S-methyl C), 63.06, 85.79 (cage C), 96.05 (Cp*-C), 215.66 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = 3.27, -3.07, -7.51, -10.78, -11.85, -13.09. IR (KBr disk, cm^{-1}): ν = 3042.83, 2958.58, 2912.12, 2632.78, 2580.65, 1508.67, 1449.33, 1378.44, 1357.16, 1311.88, 1101.97, 1070.22, 1049.10, 1030.34, 1009.72, 993.87. Anal. calcd for **7** ($C_{14}H_{28}B_{10}S_2ClIr$): C, 28.20; H, 4.73; Found: C, 28.35; H, 4.62.

Scheme S6. Synthesis of 8 9 and 10.



Complex 7

Nu=$C_6H_5S^-$, **Complex 8**
Nu=*o*-$C_2B_{10}H_{11}CS_2^-$, **Complex 9**
Nu=*o*-$C_2B_{10}H_{11}C(NPh)S^-$, **Complex 10**

Synthesis of Cp*Ir(*o*-$C_2B_{10}H_{10}CS_2CH_3$)(SPh) (8). In a typical experiment, 59.6 mg of **7** (0.1 mmol, 1.0 equiv) and 14.7 mg of C_6H_5SNa (0.1 mmol, 1.0 equiv) were added to a 25 mL Schlenk tube with 8 mL CH_2Cl_2, and 25.7 mg of AgOTf (0.1 mmol, 1.0 equiv) was then added. After the reagent addition was complete, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 1:1 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **8** as a green-black powder (**8**: R_f = 0.38 in 1:2 CH_2Cl_2 / *n*-hexane mixture). 48.2 mg of **8** was obtained, the yield based on **7** was 71.9%. **8** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.82 (s, 15H, Cp*-H), 1.65-3.40 (br, 9H, B-H), 4.48 (br, 1H, cage C-H), 2.65 (s, 3H, S-methyl C-H), 6.84 (t, J = 7.3Hz, 1H, phenyl C-H), 7.03 (t, J = 7.8 Hz, 2H, phenyl C-H), 7.23 (dd, J = 8.3, 1.1 Hz, 2H, phenyl C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 8.87 (Cp*-C), 21.68 (S-methyl C), 62.27, 85.59 (cage C), 97.94 (Cp*-C), 122.34, 127.44, 130.49, 145.35 (phenyl C), 207.15 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$,

ppm): δ = 0.58, -3.35, -7.65, -11.29, -12.64, -13.38. IR (KBr disk, cm^{-1}): ν = 3027.90, 2962.88, 2915.88, 2590.00, 2563.79, 1575.68, 1472.64, 1432.27, 1411.89, 1261.62, 1097.36, 1023.48, 803.70, 734.11, 695.44, 688.90. Anal. calcd for **8** ($C_{20}H_{33}B_{10}S_3Ir$): C, 35.85; H, 4.96; Found: C, 35.74; H, 4.82.

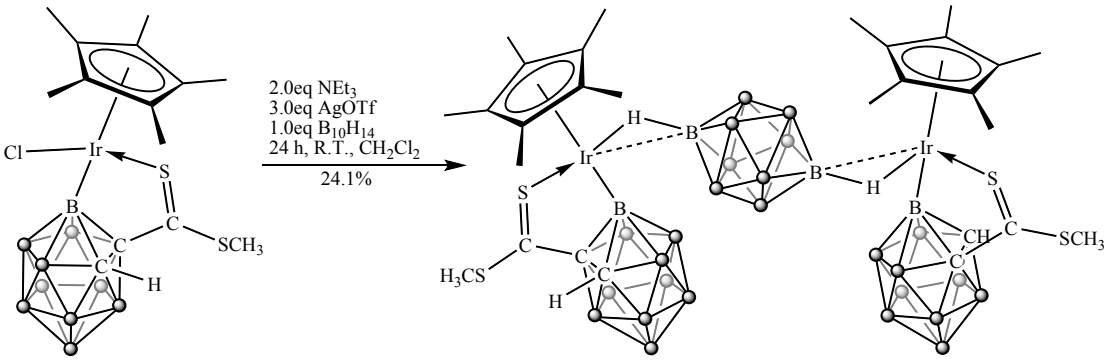
Synthesis of Cp*Ir(*o*-$C_2B_{10}H_{10}CS_2CH_3$)(*o*-$C_2B_{10}H_{11}CS_2$) (9). In a typical experiment, 59.6 mg of **7** (0.1 mmol, 1.0 equiv) and 55.9 mg of **Ligand 1** (0.1 mmol, 1.0 equiv) were added to a 25 mL Schlenk tube with 8 mL of CH_2Cl_2, and 25.7 mg of AgOTf (0.1 mmol, 1.0 equiv) was then added. After the reagent addition was complete, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 1:3 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **9** as a dark-red powder (**9**: R_f = 0.44 in 1:3 CH_2Cl_2 / *n*-hexane mixture). 66.9 mg of **9** was obtained, the yield based on **7** was 85.7%. **9** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.79 (s, 15H, Cp*-H), 1.75-3.40 (br, 19H, B-H), 3.15, 5.02 (br, 1H, cage C-H), 2.72 (s, 3H, S-methyl C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 8.79 (Cp*-C), 21.69 (S-methyl C), 63.13, 64.92, 85.61, 85.84 (cage C), 97.99 (Cp*-C), 207.64, 229.41 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -1.26, -3.49, -7.95, -9.30, -11.06, -13.11. IR (KBr disk, cm^{-1}): ν = 3045.52, 3027.78, 2965.60, 2908.48, 2854.88, 1415.05, 1380.71, 1100.80, 1081.42, 1069.63, 1036.17, 1012.09, 995.52. Anal. calcd for **9** ($C_{17}H_{39}B_{20}S_4Ir$): C, 26.17; H, 5.04; Found: C, 26.10; H, 5.12.

Synthesis of Cp*Ir(*o*-$C_2B_{10}H_{10}CS_2CH_3$)(*o*-$C_2B_{10}H_{11}CSNPh$) (10). In a typical experiment, 59.6 mg of **7** (0.1 mmol, 1.0 equiv), 27.9 mg of *o*-$C_2B_{10}H_{11}CSNHPh$ (0.1 mmol, 1.0 equiv) and 20 μL of NEt_3 were added to a 25 mL Schlenk tube with 8 mL of CH_2Cl_2, and 25.7 mg of AgOTf (0.1 mmol, 1.0 equiv) was then added. After the reagent addition was complete, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 12 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on neutral alumina gel. Elution with a 1:3 (volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **10** as a purple-black powder (**10**: R_f = 0.59 in 1:2 CH_2Cl_2 / *n*-hexane mixture). 66.5 mg **10** was

obtained, the yield based on **7** was 79.2%. **10** is soluble in CH_2Cl_2 and $CHCl_3$. ^1H NMR (400 MHz; $CDCl_3$, ppm): δ = 1.74 (s, 15H, Cp*-H), 1.60-3.45 (br, 19H, B-H), 3.71, 4.63 (br, 1H, cage C-H), 2.65 (s, 3H, S-methyl C-H), 6.51 (d, J = 7.5 Hz, 2H, phenyl C-H), 6.95 (t, J = 7.3 Hz, 1H, phenyl C-H), 7.20 (t, J = 7.7 Hz, 2H, phenyl C-H). $^{13}C\{^1H\}$ NMR (101 MHz; $CDCl_3$, ppm): δ = 8.88 (Cp*-C), 21.35 (S-methyl C), 60.96, 64.07, 85.82, 123.84 (cage C), 97.71 (Cp*-C), 119.08, 122.62, 128.92, 148.79 (phenyl C), 159.29 (thiamide C), 207.92 (dithiocarboxyl C). $^{11}B\{^1H\}$ NMR (160 MHz, $CDCl_3$, ppm): δ = -3.33, -8.15, -8.86, -9.61, -11.18, -13.03. IR (KBr disk, cm^{-1}): ν= 3067.79, 3054.26, 3045.18, 2914.96, 2606.89, 2575.80, 1580.08, 1482.27, 1446.36, 1378.39, 1103.02, 1085.97, 1072.76, 1046.50, 1015.43, 994.89, 806.76, 763.37, 722.14, 694.11. Anal. calcd for **10** ($C_{23}H_{44}B_{20}NS_3Ir$): C, 32.92; H, 5.29; N, 1.67. Found: C, 33.15; H, 5.40; N, 1.43.

Hint: While silica gel was used to separate **10**, the product will always be contaminated by *o*-$C_2B_{10}H_{11}CSNHPh$. Replace silica gel by neutral alumina gel can reduce the contamination largely.

Scheme S7. Synthesis of 11.



Complex 7 **Complex 11**

Synthesis of [Cp*Ir(*o*-$C_2B_{10}H_{10}CS_2CH_3$)]$_2$($B_{10}H_{10}$) (11). In a typical experiment, 59.6 mg of **7** (0.1 mmol, 1.0 equiv), 40 μL of NEt_3 and 12.2 mg of $B_{10}H_{14}$ (0.1 mmol, 1.0 equiv) were added to a 25 mL Schlenk tube with 8 mL of CH_2Cl_2, and 77.1 mg of AgOTf (0.3 mmol, 3.0 equiv) was then added. After the reagent addition was complete finished, the Schlenk tube was immediately covered with tin foil. The solution was stirred for 24 h at room temperature. The solvent was then removed and the residue was purified by column chromatography on silica gel. Elution with a 1:1

(volume ratio) CH_2Cl_2 / *n*-hexane mixture gave **11** as a brown powder (**11**: R_f = 0.73 in 2:1 CH_2Cl_2 / *n*-hexane mixture), then the crude product was washed three times with 2 mL hexane to remove contaminant **7**. 14.9 mg of **11** was obtained, the yield based on **7** was 24.1%. **11** is soluble in CH_2Cl_2 and $CHCl_3$. ^{1}H NMR (400 MHz; $CDCl_3$, ppm): δ = 0.10-0.80 (br, 8H, decaborate B-H), 1.97, 1.98 (s, 15H, Cp*-H), 2.10-3.40 (br, 18H, carborane B-H), 4.56, 4.61 (br, 1H, cage C-H), 2.67, 2.68 (s, 3H, S-methyl C-H), -8.28 (s, 2H, B-H-Ir bridging hydrogen). ^{13}C{^{1}H} NMR (101 MHz; $CDCl_3$, ppm): δ = 9.33 (Cp*-C), 22.28 (S-methyl C), 61.31, 86.11 (cage C), 99.30 (Cp*-C), 212.11 (dithiocarboxyl C). ^{11}B{^{1}H} NMR (160 MHz, $CDCl_3$, ppm): δ = -2.92, -8.44, -10.83, -12.76, -23.91. IR (KBr disk, cm^{-1}): ν = 3023.39, 2960.29, 2913.51, 2587.32, 2495.84, 1453.35, 1416.95, 1379.28, 1080.03, 1048.35, 1027.62, 1010.22. Anal. calcd for **11** ($C_{28}H_{66}B_{30}S_4Ir_2$): C, 27.12; H, 5.37; Found: C, 27.08; H, 5.32.

Hint: We strongly suggest NOT to adopt the ideal stoichiometric ratio of 2:1 (**7** versus decaborane) according to the composition of **11** because of the very close R_f values of **7** and **11**, which results in them being inseparable by chromatography. Using an excess of decaborane decreases the amount of unreacted **7**, making it easier to remove it by washing with hexane.

2.NMR spectra

Fig. S1a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **Ligand 1.**

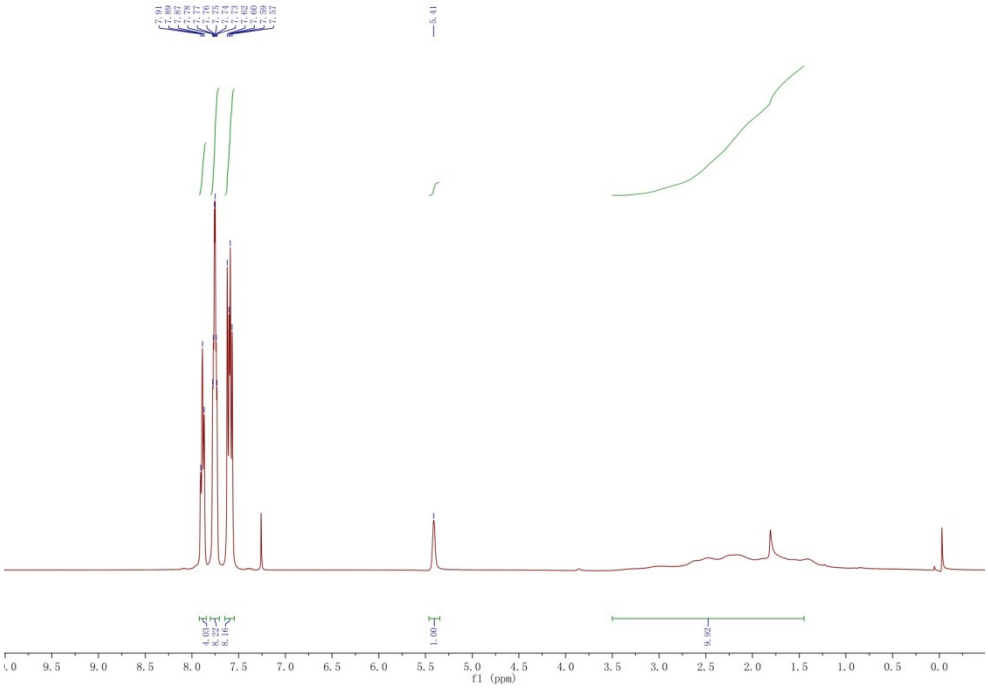


Fig. S1b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **Ligand 1.**

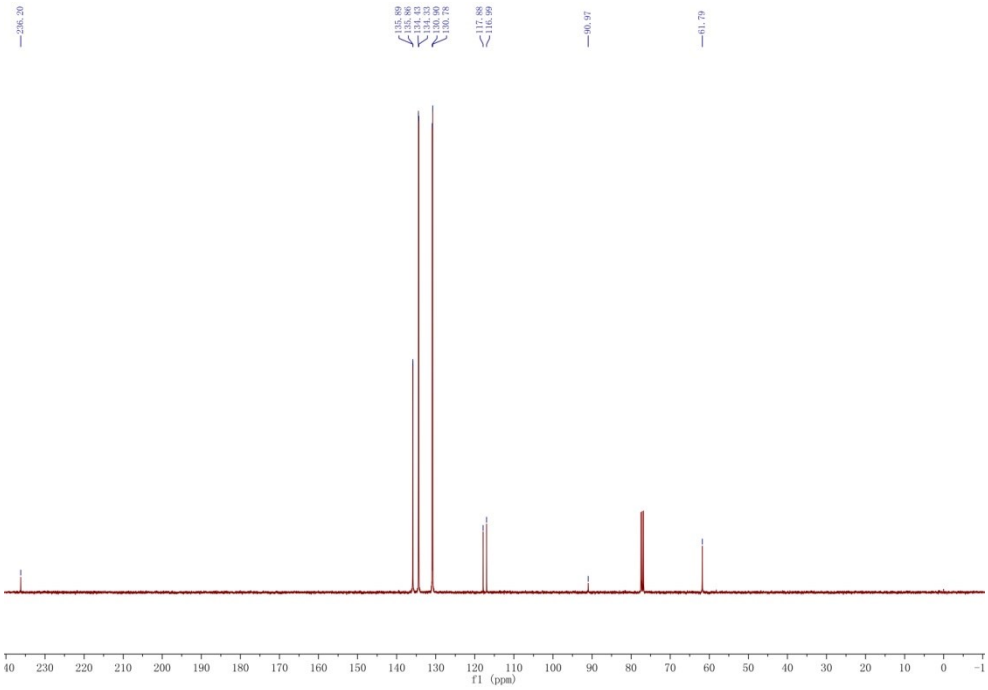


Fig. S1c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **Ligand 1.**

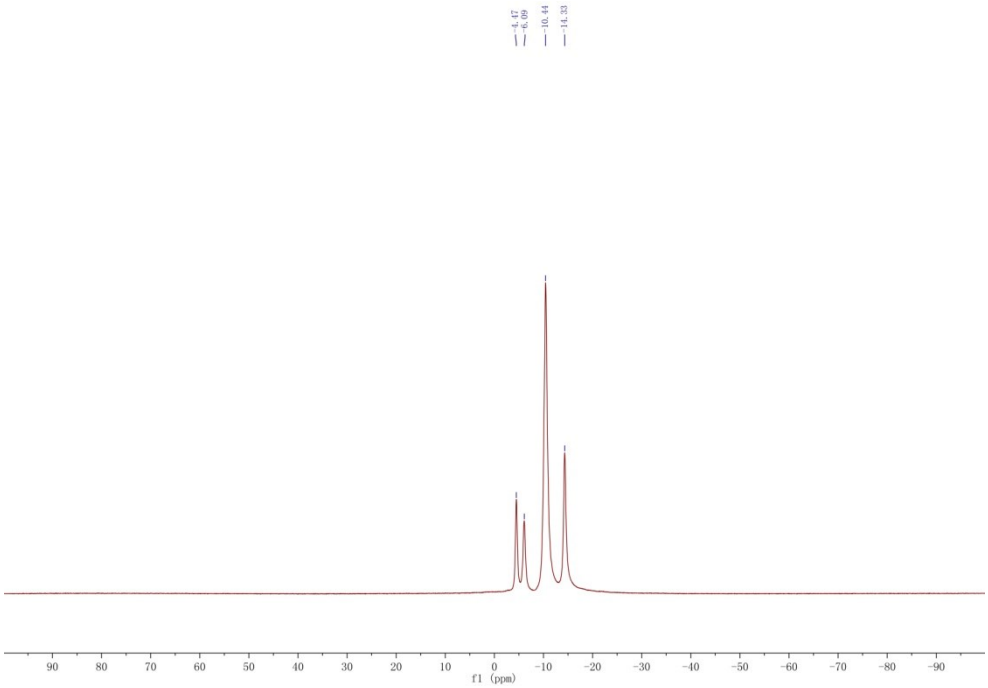


Fig. S2a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **Ligand 2.**

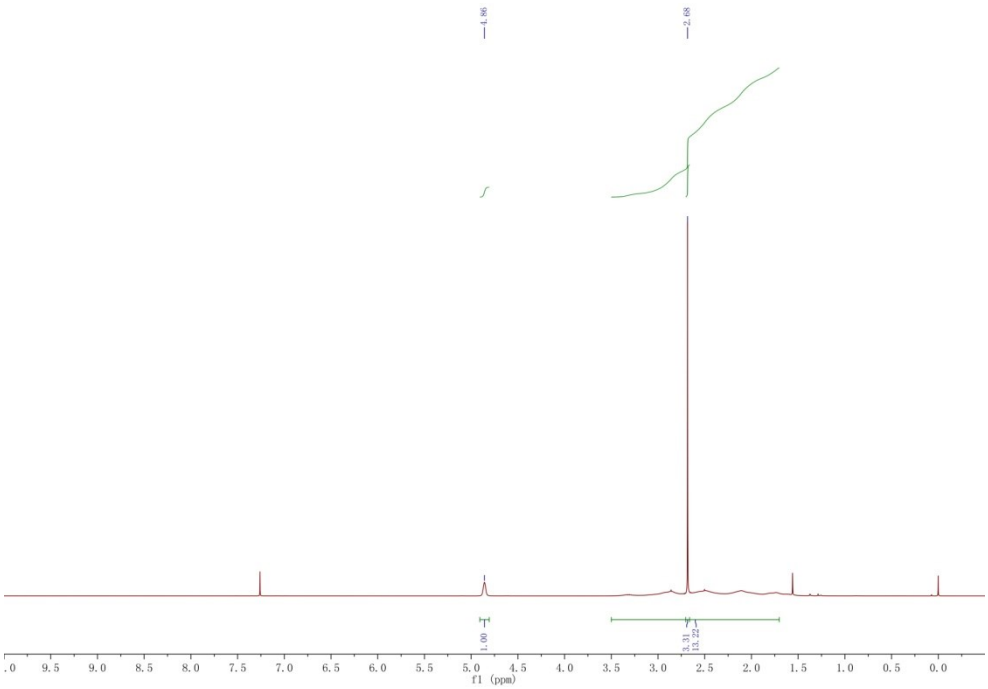


Fig. S2b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **Ligand 2.**

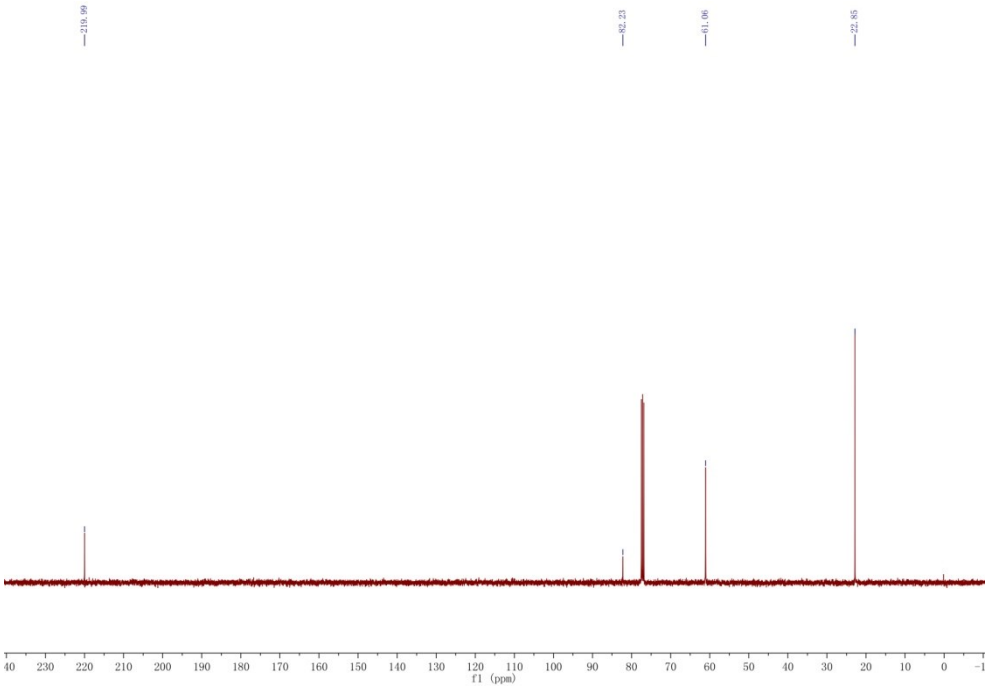


Fig. S2c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **Ligand 2.**

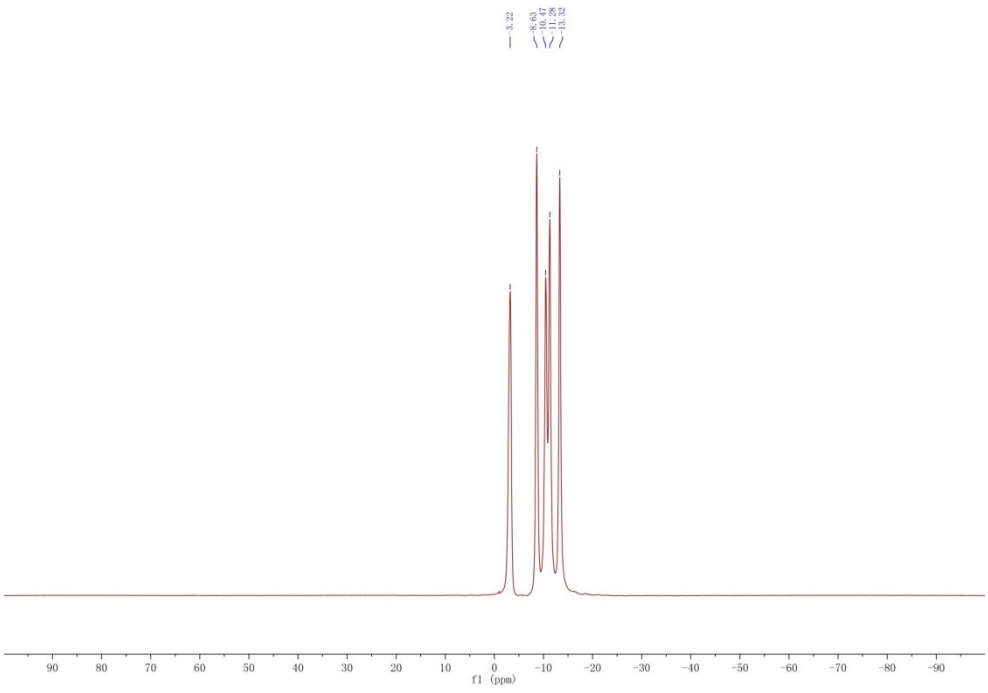


Fig. S3a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **3.**

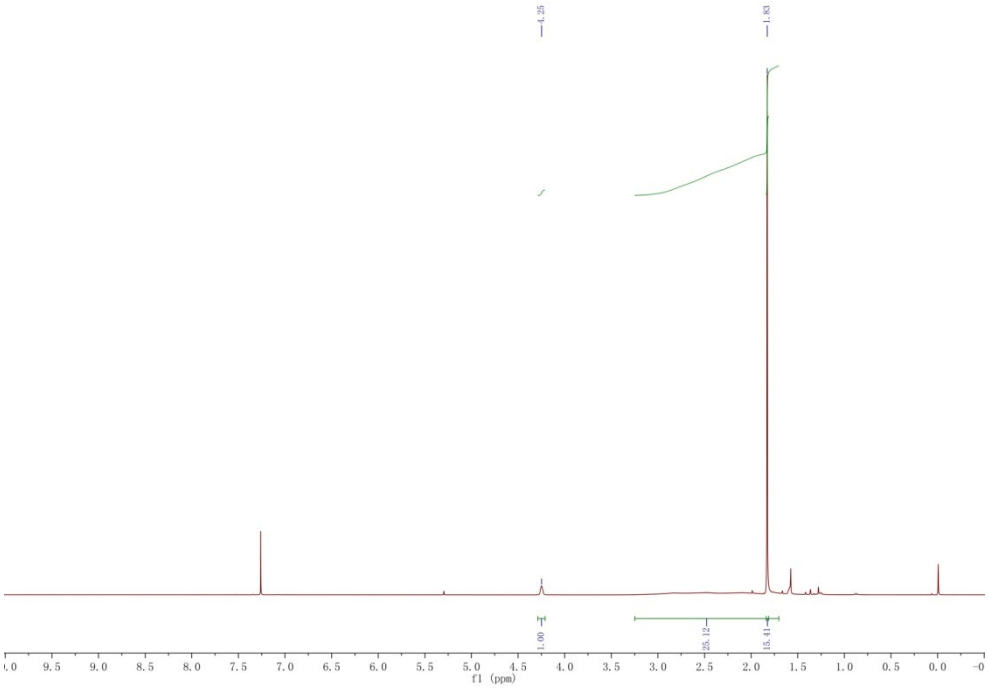


Fig. S3b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **3.**

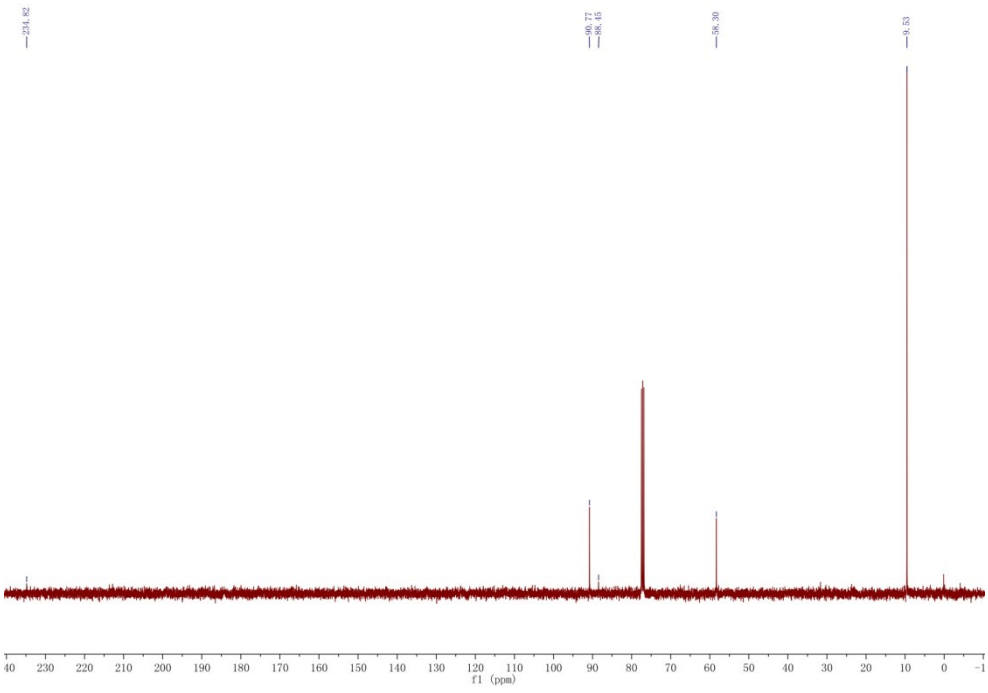


Fig. S3c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **3.**

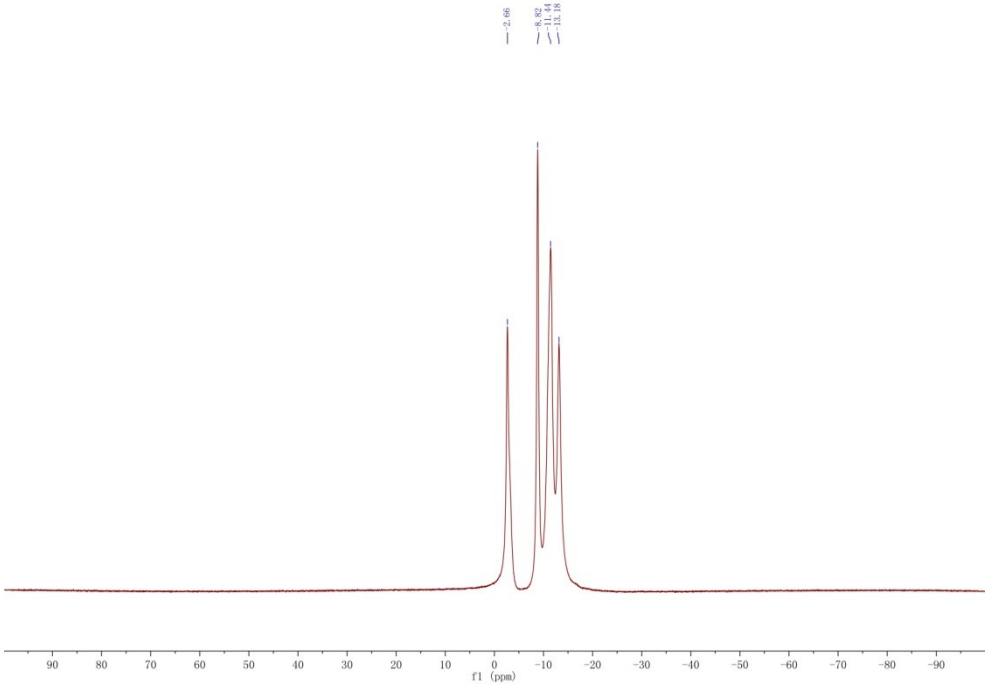


Fig. S4a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **4.**

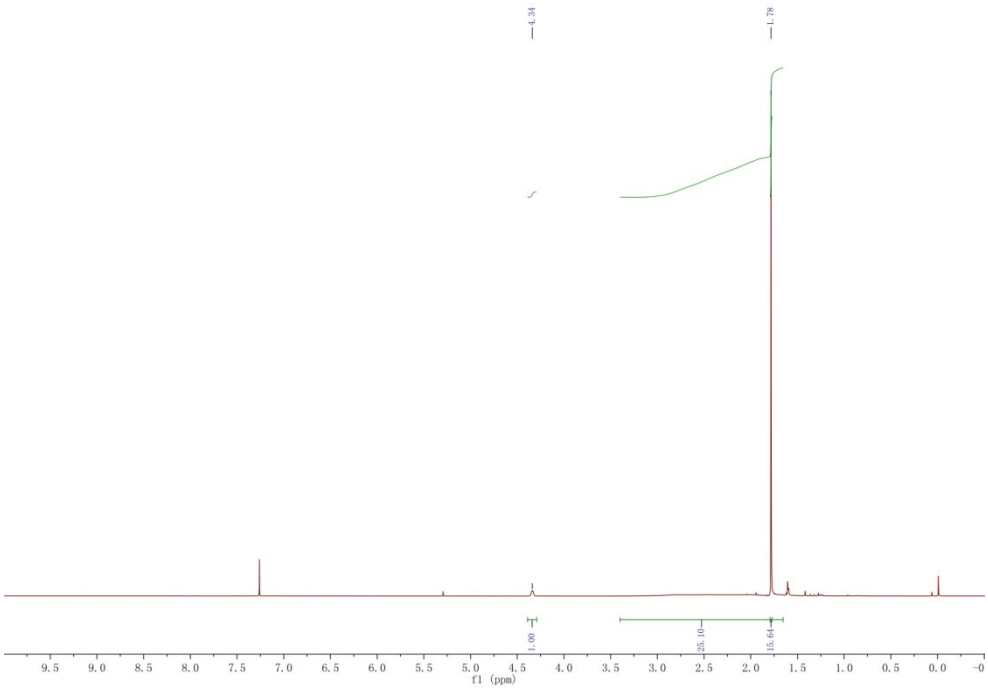


Fig. S4b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **4.**

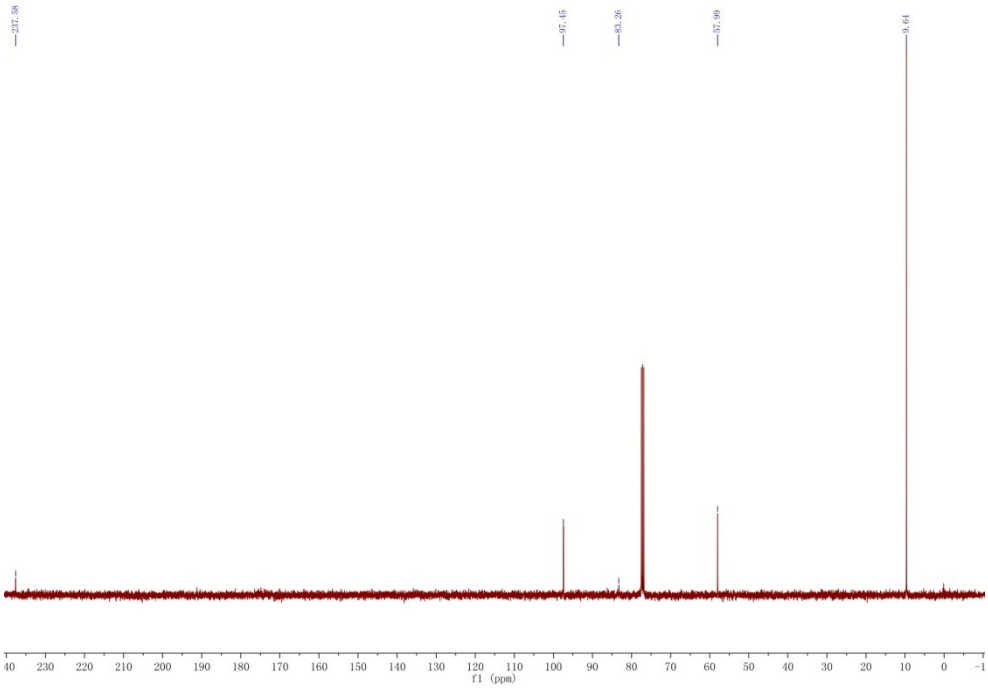


Fig. S4c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **4.**

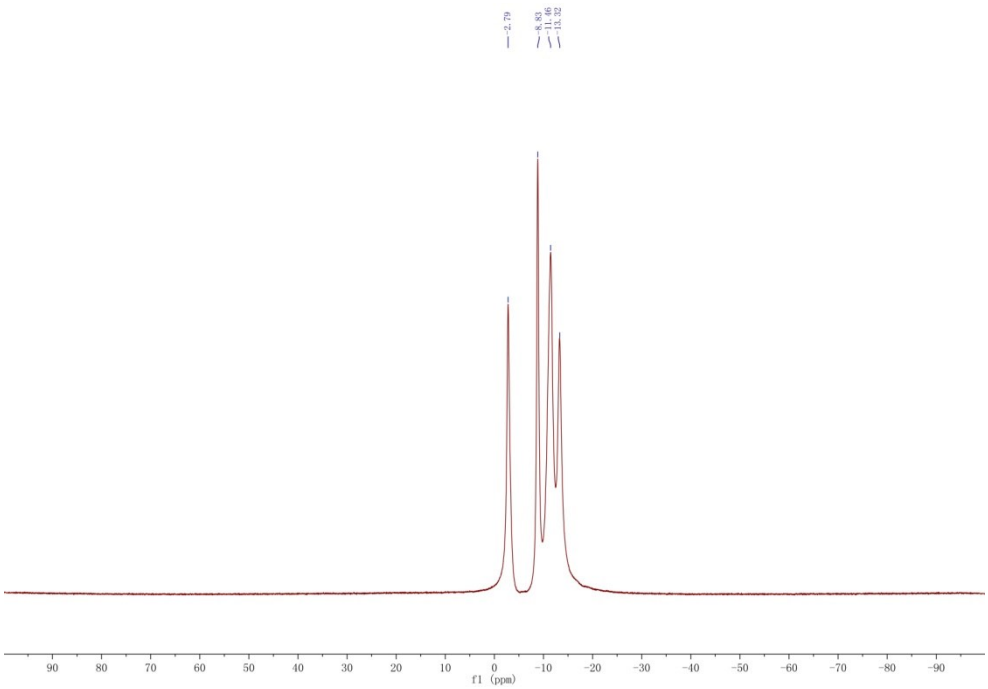


Fig. S5a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **5.**

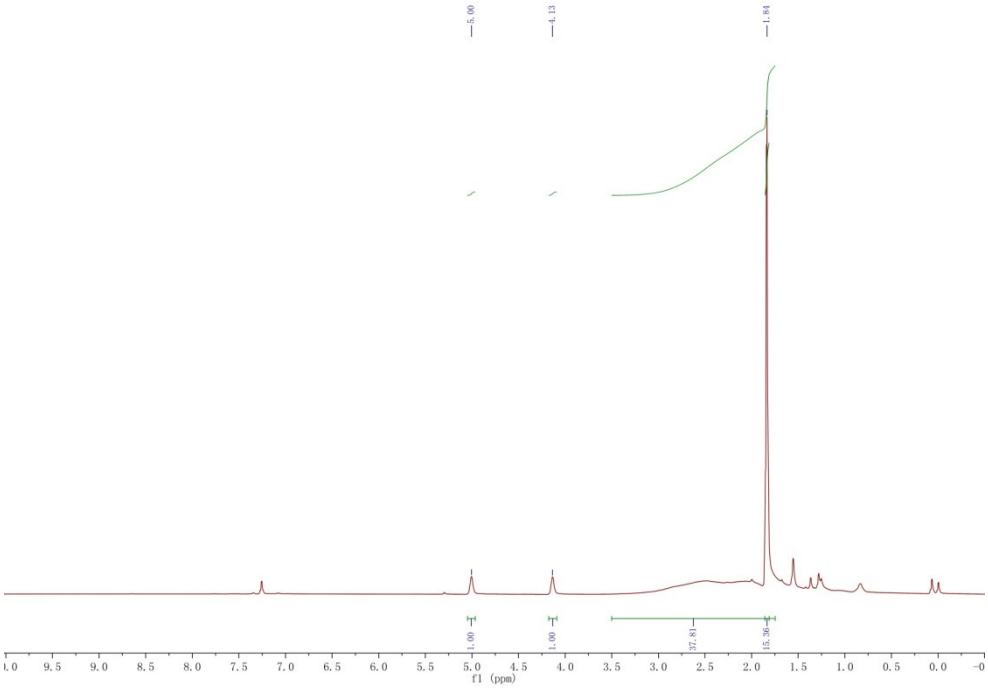


Fig. S5b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **5.**

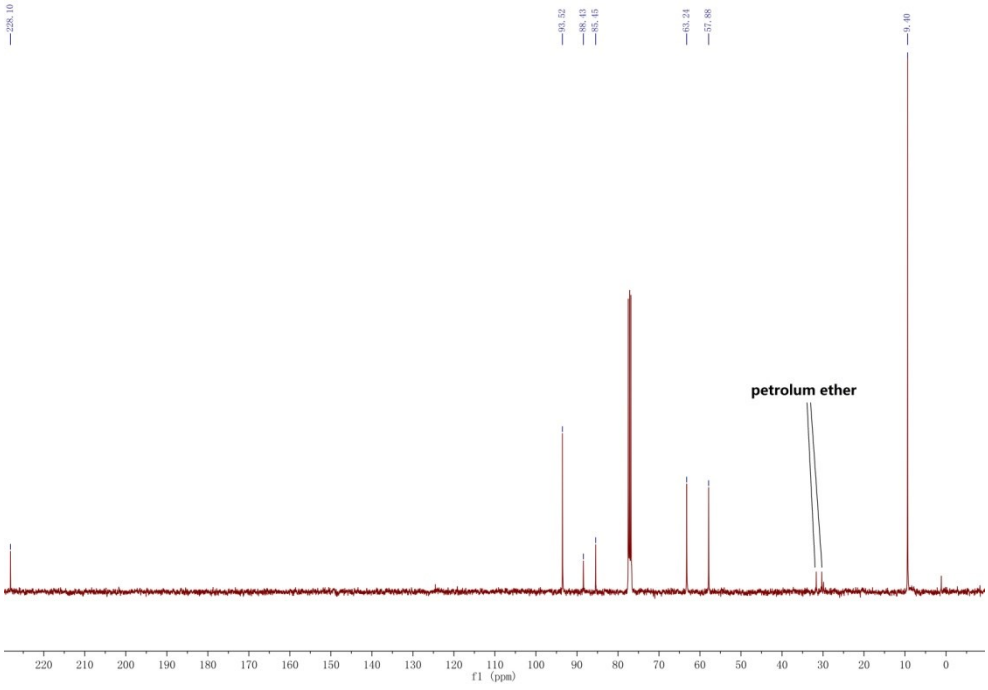


Fig. S5c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **5.**

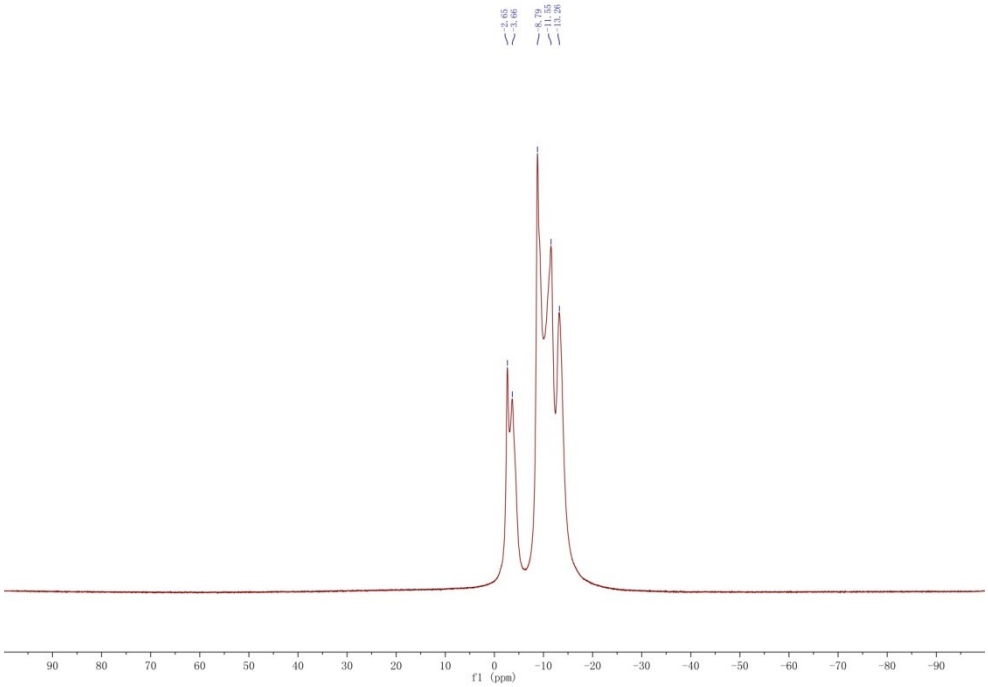


Fig. S6a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **6.**

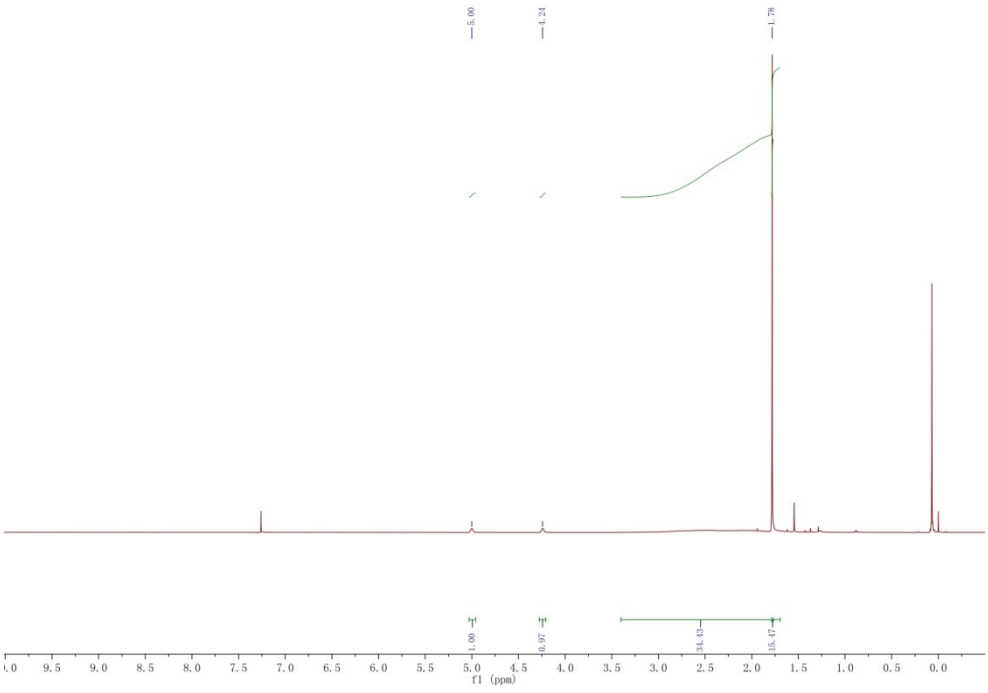


Fig. S6b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **6.**

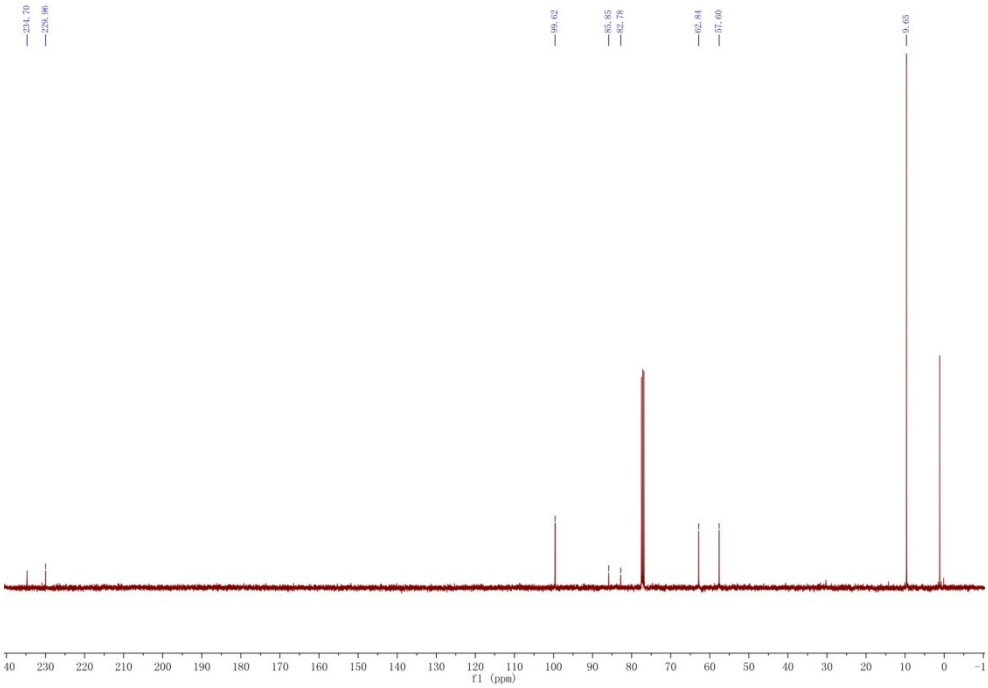


Fig. S6c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **6.**

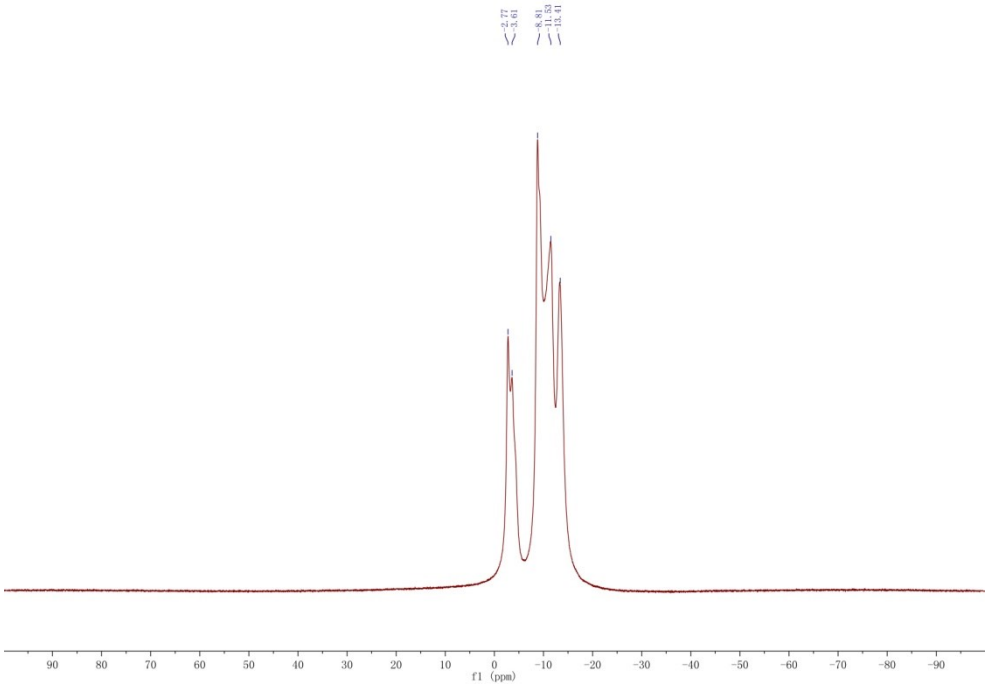


Fig. S7a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **7.**

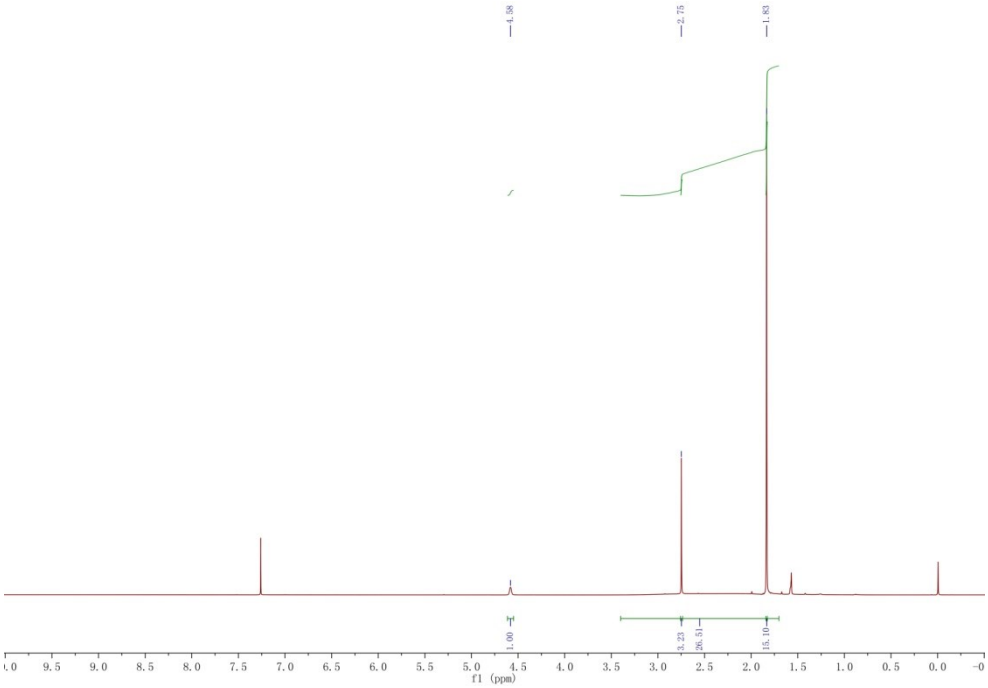


Fig. S7b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **7.**

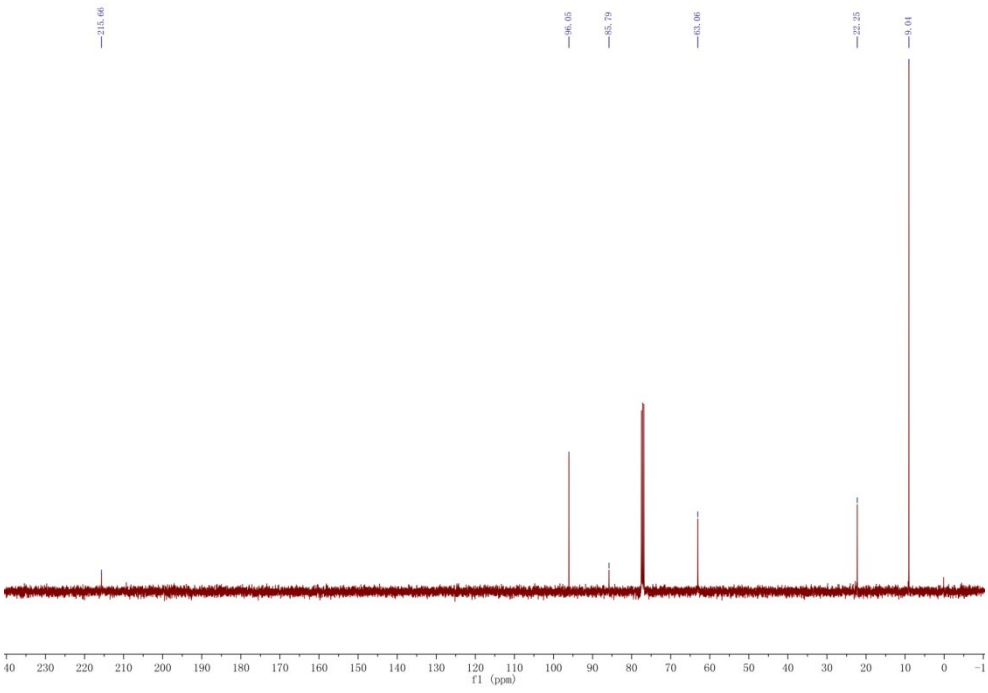


Fig. S7c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **7.**

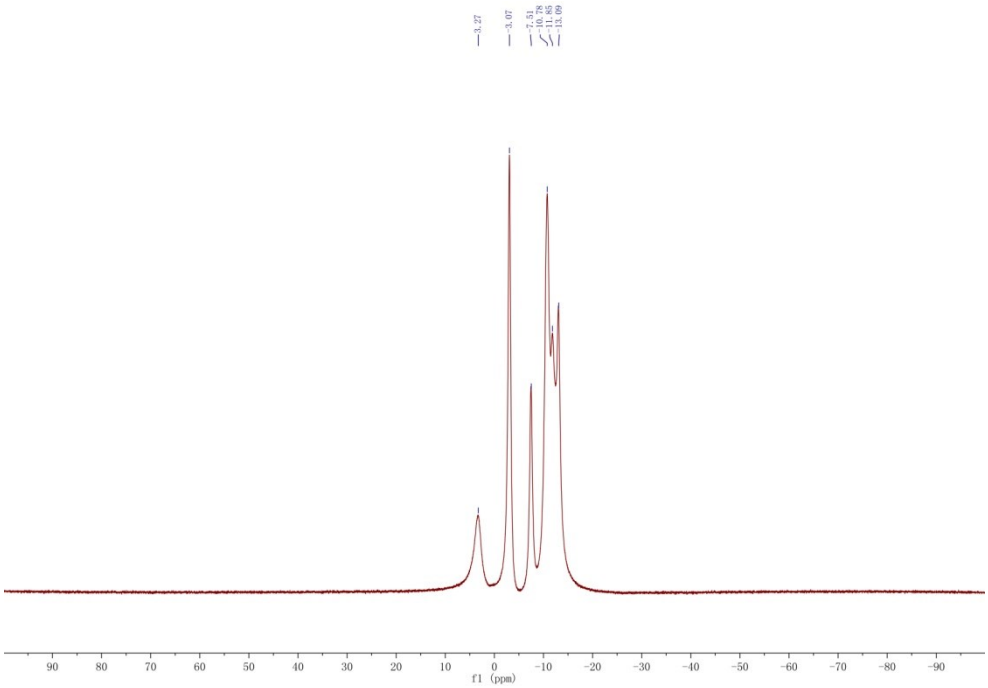


Fig. S8a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **8.**

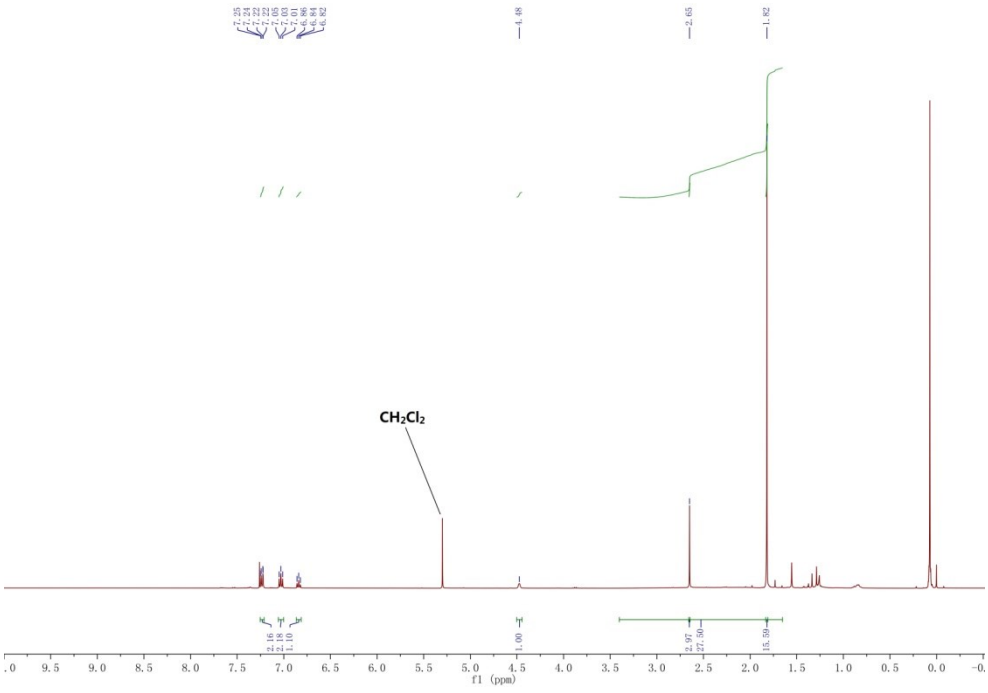


Fig. S8b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **8.**

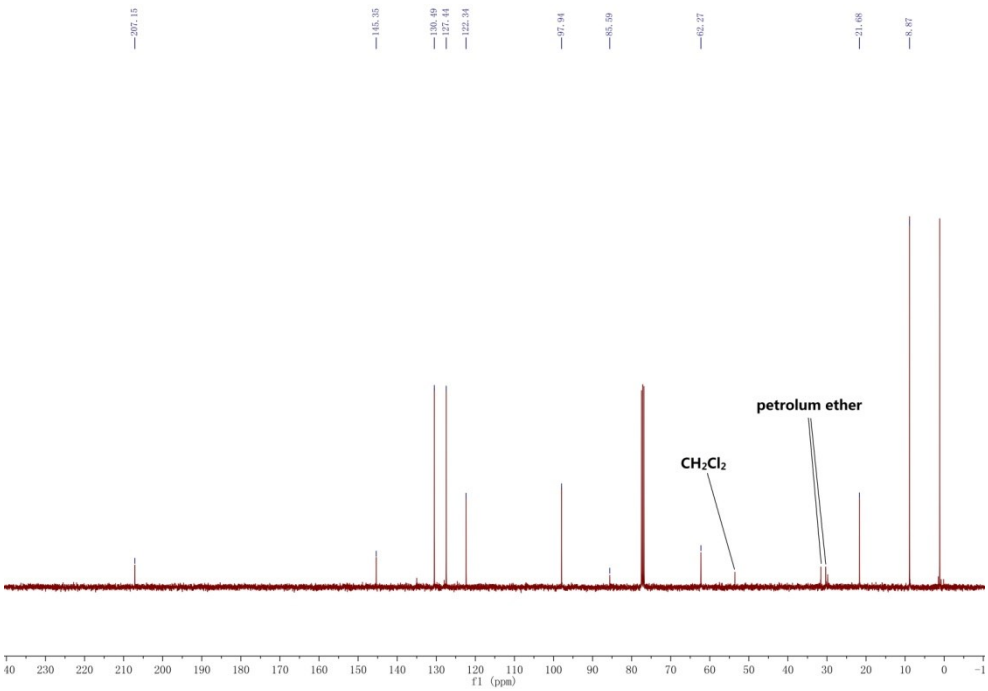


Fig. S8c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **8.**

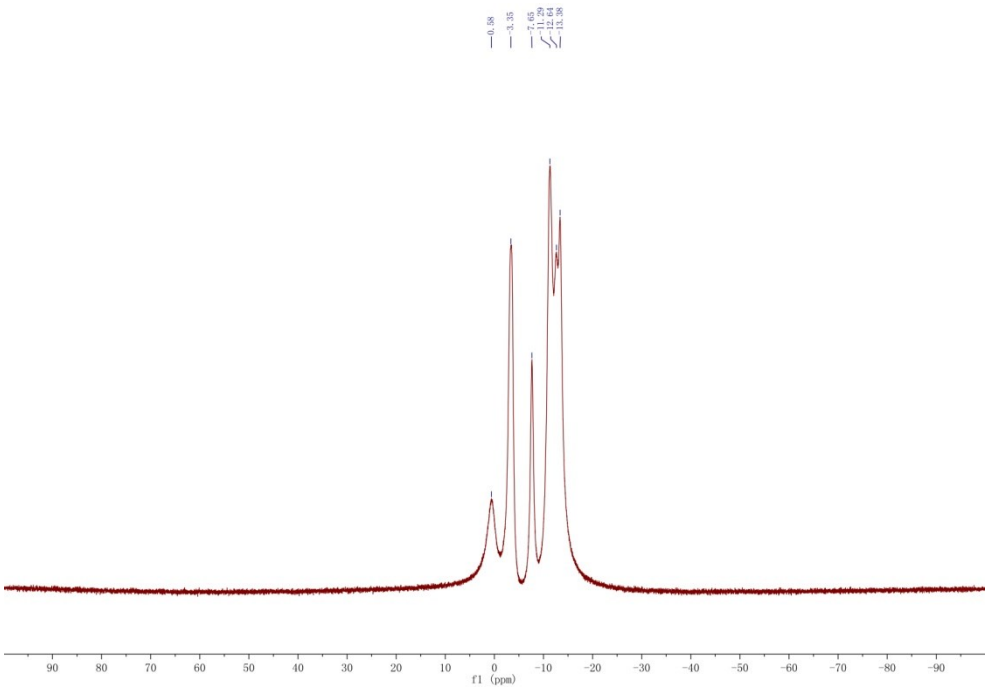


Fig. S9a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **9.**

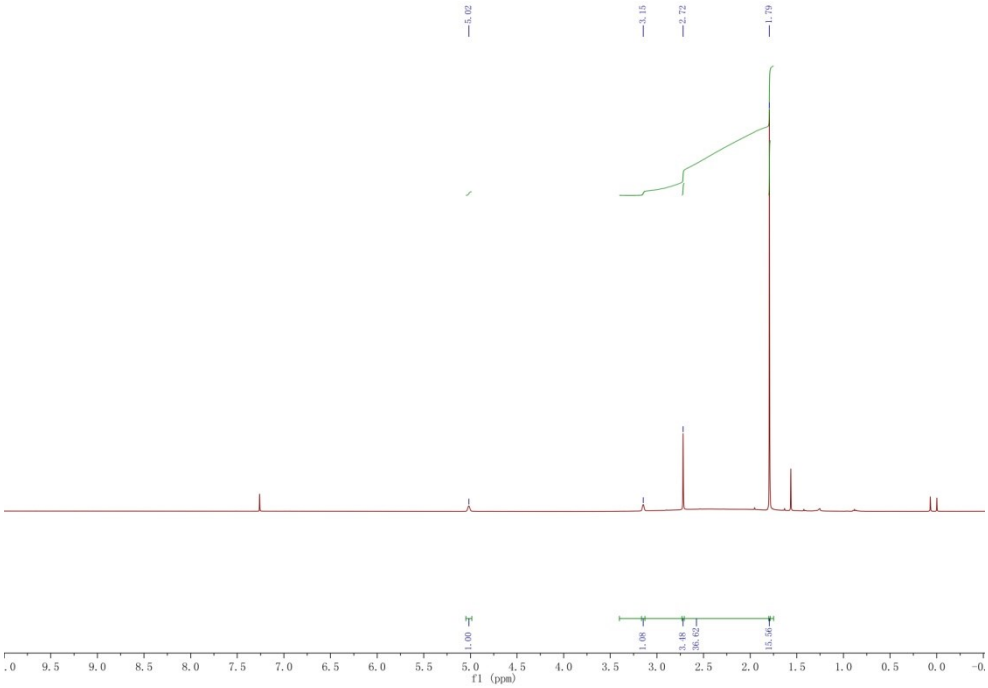


Fig. S9b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **9.**

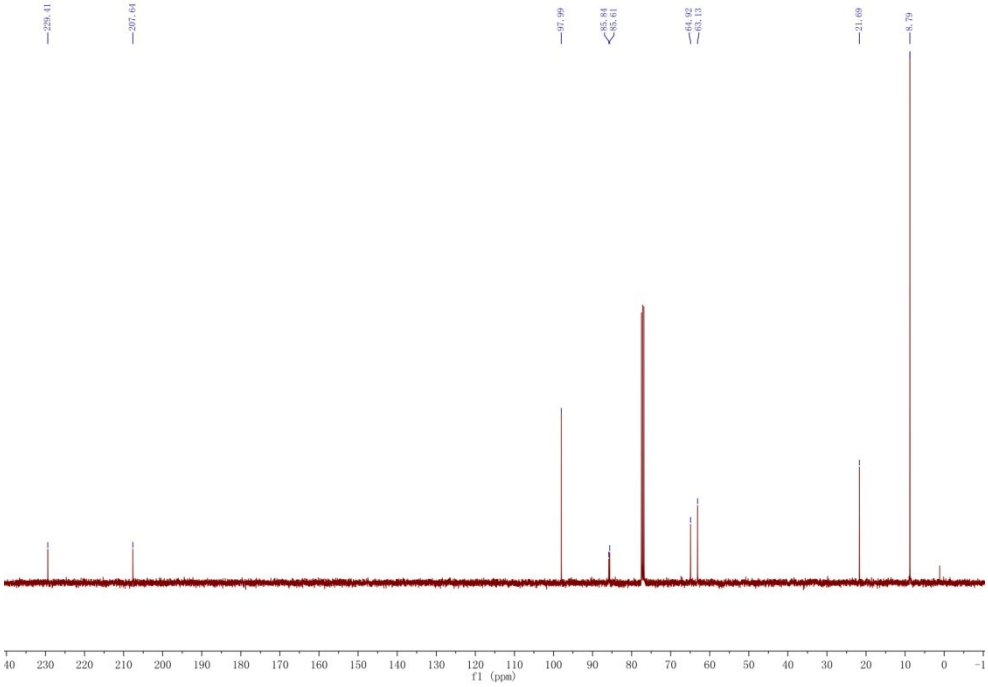


Fig. S9c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **9.**

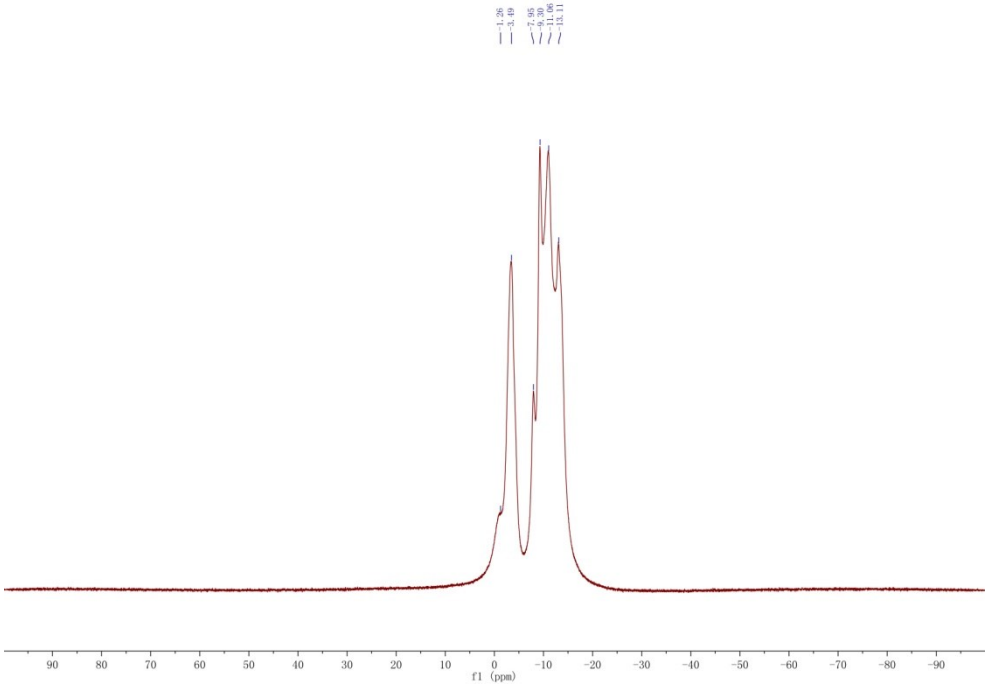


Fig. S10a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **10.**

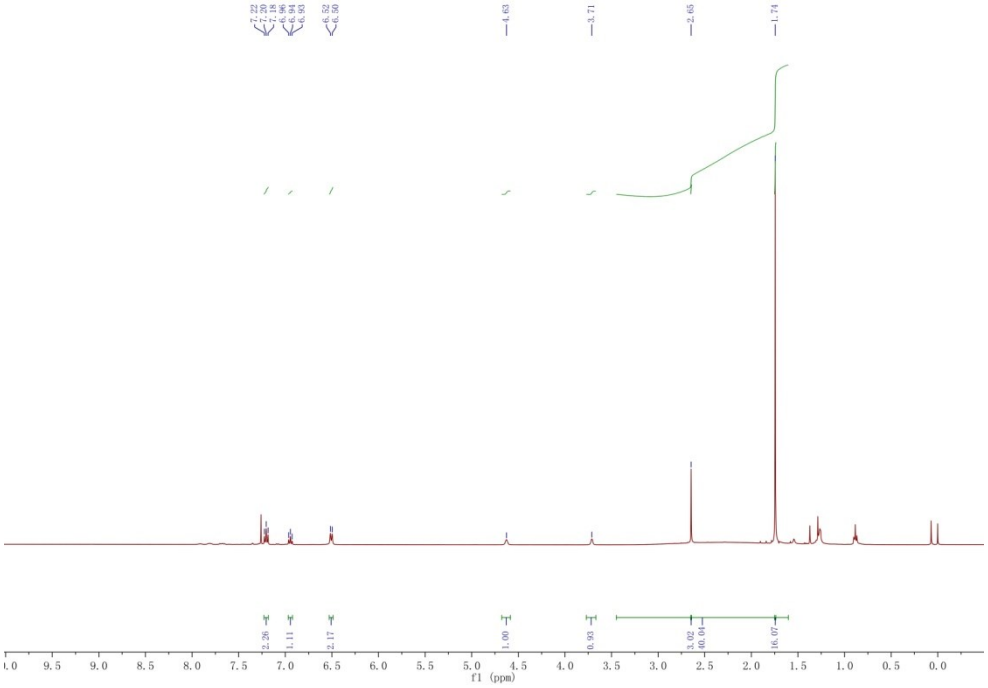


Fig. S10b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **10.**

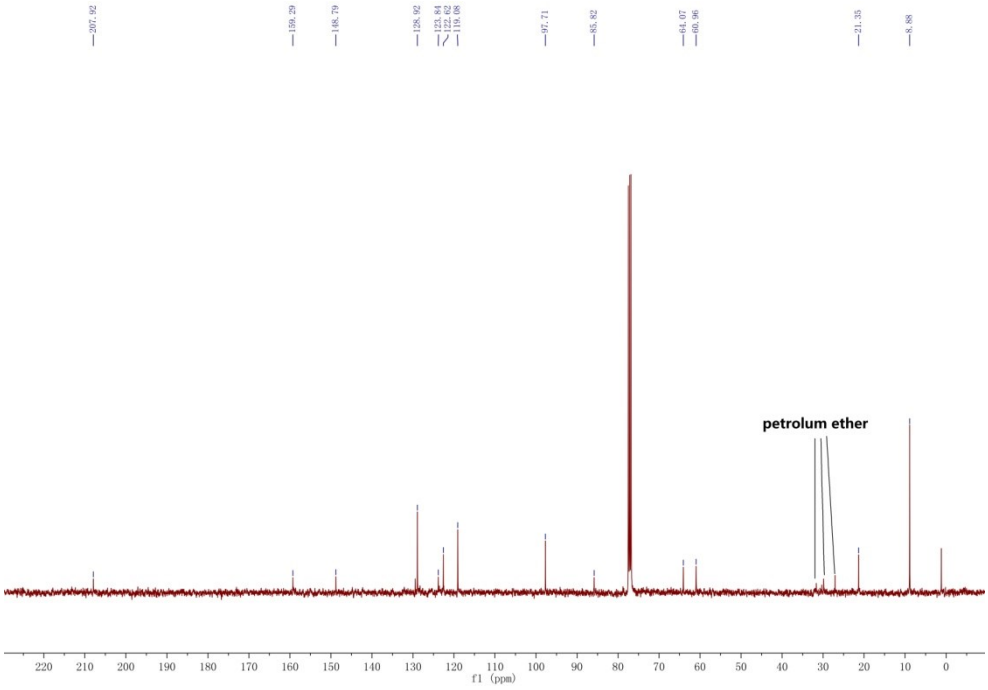


Fig. S10c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **10.**

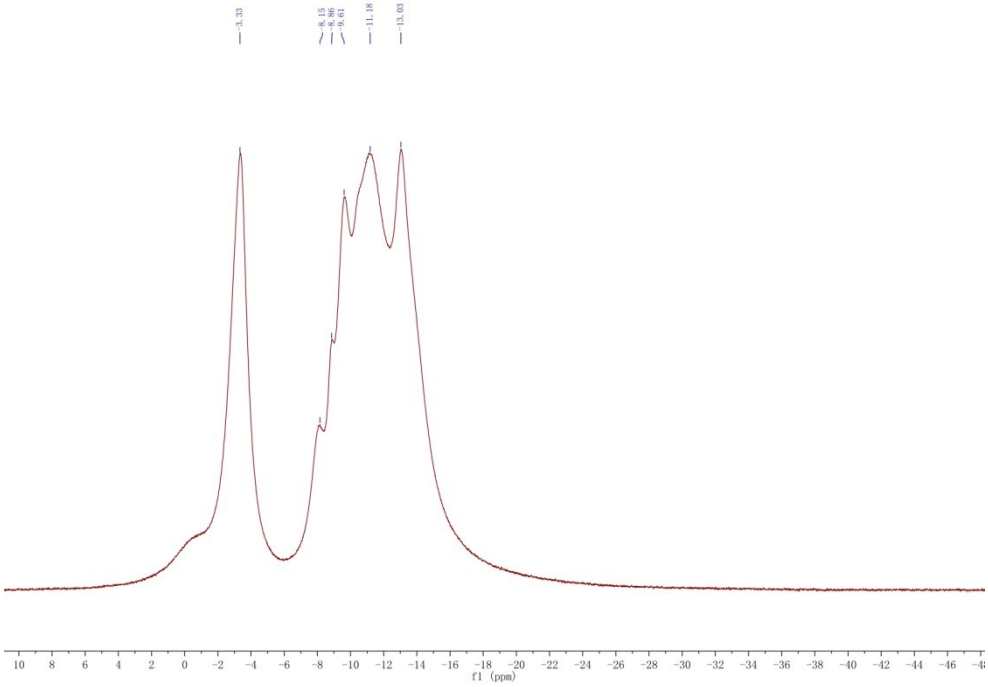


Fig. S11a. ^{1}H NMR(400MHz, $CDCl_3$, ppm) of **11.**

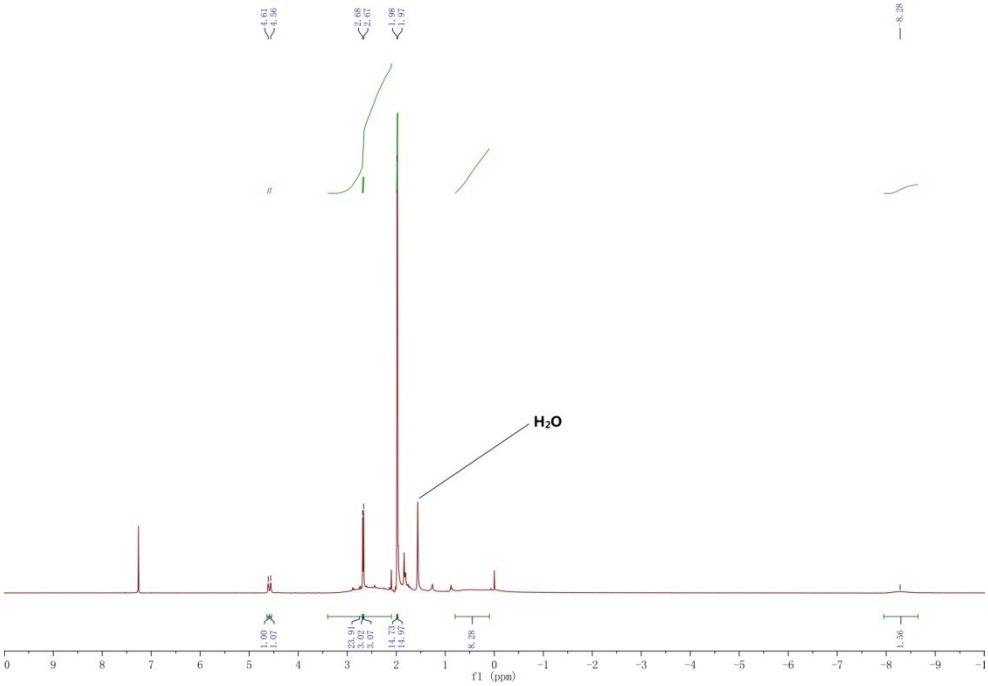


Fig. S11b. $^{13}C\{^{1}H\}$ NMR(101MHz, $CDCl_3$, ppm) of **11.**

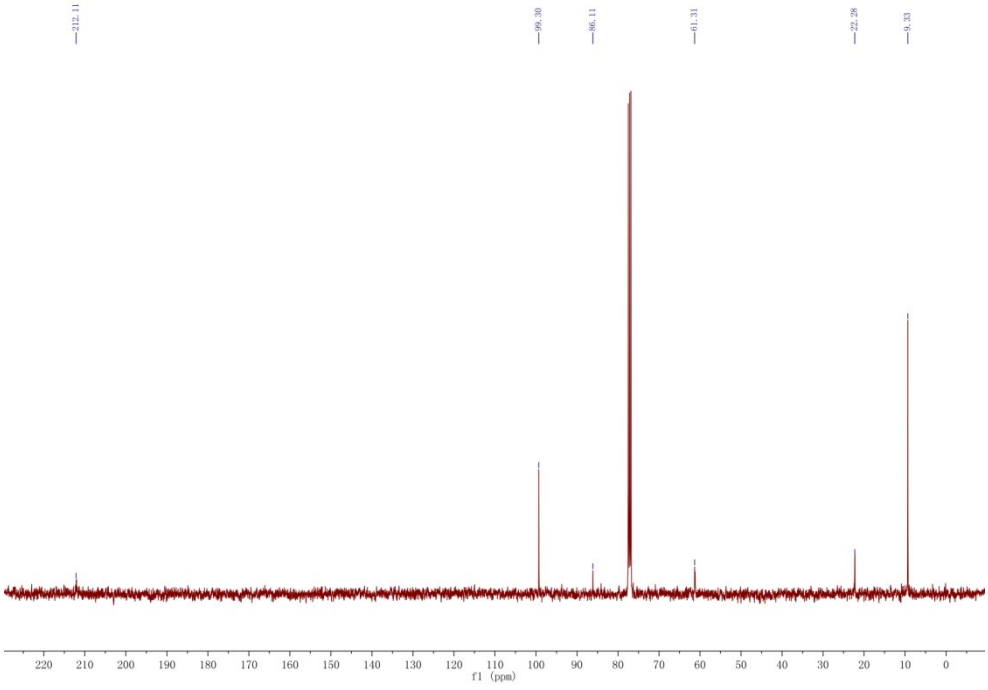
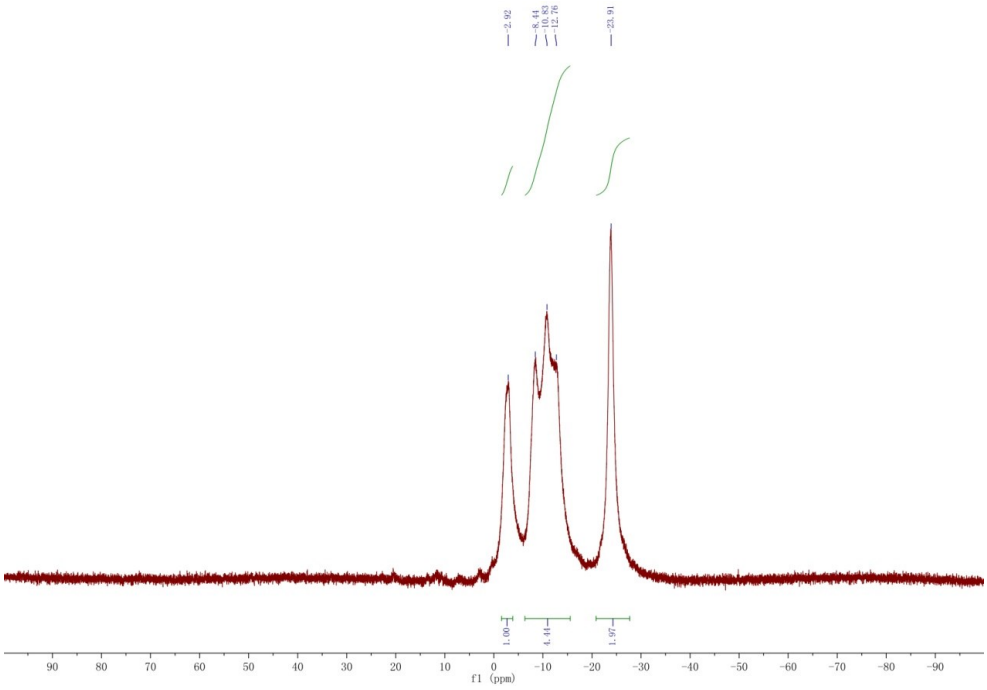


Fig. S11c. $^{11}B\{^{1}H\}$ NMR(160MHz, $CDCl_3$, ppm) of **11.**



3.X-Ray Crystallography details

Single crystal of **Ligand 1** suitable for X-Ray diffraction study was obtained by slow diffusion of hexane into chloroform solution of **Ligand 1** at room temperature.

Single crystal of **Ligand 2** suitable for X-Ray diffraction study was obtained by slow vaporization of hexane solution of **Ligand 2** at room temperature.

Single crystal of **3** to **10** suitable for X-Ray diffraction study were all obtained by slow diffusion of hexane into dichloromethane solution of corresponding complex at room temperature.

X-Ray intensity data of **3, 4, 5** were collected on a CCD-Bruker SMART APEX system. X-Ray intensity data of **Ligand 1, 2** and **6, 7, 8, 9, 10** were collected on a Bruker D8 Venture system.

Table S1. Crystal data for **Ligand 1**

Empirical formula	C27 H31 B10 P S2	
CCDC Number	2042333	
Formula weight	558.71	
Temperature	302(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 12.5156(5) Å	□= 90°.
	b = 8.7984(4) Å	□= 100.346(2)°.
	c = 28.8360(12) Å	□ = 90°.
Volume	3123.7(2) $Å^3$	
Z	2	
Density (calculated)	1.188 Mg/m^3	
Absorption coefficient	2.136 mm^{-1}	
F(000)	1160.0	

Crystal size	0.210 x 0.150 x 0.130 mm^3
Theta range for data collection	3.116 to 72.533°.
Index ranges	-15<=h<=15, -10<=k<=10, -35<=l<=35
Reflections collected	34916
Independent reflections	6182 [R(int) = 0.0737]
Completeness to theta = 72.533°	100.00 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.864 and 0.633
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6182 / 0 / 361
Goodness-of-fit on F^2	1.059
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1388
R indices (all data)	R1 = 0.0732, wR2 = 0.1547
Extinction coefficient	n/a
Largest diff. peak and hole	0.324 and -0.448 $e.Å^{-3}$

Table S2. Crystal data for **Ligand 2**

Empirical formula	C4 H14 B10 S2	
CCDC Number	2042334	
Formula weight	234.37	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 6.7430(5) Å	□= 90°.
	b = 13.4145(8) Å	□=
100.698(3)°.		
	c = 13.9386(10) Å	□ = 90°.

Volume	1238.89(15) $Å^3$
Z	4
Density (calculated)	1.257 Mg/m^3
Absorption coefficient	0.383 mm^{-1}
F(000)	480
Crystal size	0.250 x 0.220 x 0.180 mm^3
Theta range for data collection	2.125 to 27.113°.
Index ranges	-8<=h<=8, -17<=k<=17, -17<=l<=17
Reflections collected	9993
Independent reflections	2725 [R(int) = 0.0715]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.746 and 0.650
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2725 / 1 / 150
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.1018
R indices (all data)	R1 = 0.0760, wR2 = 0.1186
Extinction coefficient	n/a
Largest diff. peak and hole	0.241 and -0.307 $e.Å^{-3}$

Table S3. Crystal data for **3**

Empirical formula	C13 H26 B10 Cl Ir S2
CCDC Number	2042335
Formula weight	582.21
Temperature	293(2) K
Wavelength	1.34138 Å

Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 13.3076(4) Å □= 90°.
	b = 13.6172(4) Å □= 114.2730(10)°.
	c = 13.4642(4) Å □ = 90°.
Volume	2224.19(12) $Å^3$
Z	4
Density (calculated)	1.739 Mg/m^3
Absorption coefficient	9.573 mm^{-1}
F(000)	1120
Crystal size	0.022 x 0.021 x 0.015 mm^3
Theta range for data collection	4.219 to 55.058°.
Index ranges	-16<=h<=16, -16<=k<=16, -16<=l<=16
Reflections collected	21840
Independent reflections	4212 [R(int) = 0.0607]
Completeness to theta = 53.594°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.751 and 0.362
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4212 / 0 / 253
Goodness-of-fit on F^2	1.036
Final R indices [I>2sigma(I)]	R1 = 0.0280, wR2 = 0.0735
R indices (all data)	R1 = 0.0301, wR2 = 0.0753
Extinction coefficient	n/a
Largest diff. peak and hole	0.827 and -1.023 $e.Å^{-3}$

Table S4. Crystal data for **4**

Empirical formula	C13 H26 B10 Cl Rh S2
CCDC Number	2042336
Formula weight	492.92
Temperature	173(2) K
Wavelength	1.34138 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 13.3454(4) Å □= 90°.
	b = 13.2555(4) Å □= 114.9040(10)°.
	c = 13.4524(4) Å □ = 90°.
Volume	2158.45(11) $Å^3$
Z	4
Density (calculated)	1.517 Mg/m^3
Absorption coefficient	6.250 mm^{-1}
F(000)	992
Crystal size	0.180 x 0.080 x 0.020 mm^3
Theta range for data collection	3.176 to 58.985°.
Index ranges	-16<=h<=17, -16<=k<=16, -16<=l<=17
Reflections collected	37499
Independent reflections	4709 [R(int) = 0.0879]
Completeness to theta = 53.594°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.752 and 0.609
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4709 / 0 / 253
Goodness-of-fit on F^2	1.022

Final R indices [I>2sigma(I)]	R1 = 0.0297, wR2 = 0.0611
R indices (all data)	R1 = 0.0450, wR2 = 0.0662
Extinction coefficient	n/a
Largest diff. peak and hole	0.436 and -0.740 e.Å^{-3}

Table S5. Crystal data for **5**

Empirical formula	C16 H37 B20 Ir S4	
CCDC Number	2042337	
Formula weight	766.09	
Temperature	173(2) K	
Wavelength	1.34138 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 12.2831(5) Å	□= 90°.
	b = 19.6865(8) Å	□= 90.071(2)°.
	c = 13.4650(5) Å	□ = 90°.
Volume	3256.0(2) Å^3	
Z	4	
Density (calculated)	1.563 Mg/m^3	
Absorption coefficient	6.892 mm^{-1}	
F(000)	1496	
Crystal size	0.160 x 0.070 x 0.050 mm^3	
Theta range for data collection	3.130 to 54.969°.	
Index ranges	-14<=h<=14, -24<=k<=24, -13<=l<=16	
Reflections collected	48296	
Independent reflections	6186 [R(int) = 0.0585]	
Completeness to theta = 53.594°	99.9 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.751 and 0.592
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6186 / 2 / 383
Goodness-of-fit on F^2	1.067
Final R indices [I>2sigma(I)]	R1 = 0.0296, wR2 = 0.0643
R indices (all data)	R1 = 0.0352, wR2 = 0.0671
Extinction coefficient	n/a
Largest diff. peak and hole	3.392 and -1.152 e.$Å^{-3}$

Table S6. Crystal data for **6**

Empirical formula	C16 H37 B20 Rh S4	
CCDC Number	2042338	
Formula weight	676.80	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 12.2938(8) Å	□= 90°.
	b = 19.6540(12) Å	□= 90.096(2)°.
	c = 13.4549(8) Å	□ = 90°.
Volume	3251.0(3) $Å^3$	
Z	4	
Density (calculated)	1.383 Mg/m^3	
Absorption coefficient	6.702 mm^{-1}	
F(000)	1368	
Crystal size	0.390 x 0.320 x 0.280 mm^3	
Theta range for data collection	3.595 to 75.496°.	

Index ranges	-15<=h<=15, -24<=k<=24, -16<=l<=16
Reflections collected	93072
Independent reflections	6717 [R(int) = 0.0607]
Completeness to theta = 67.679°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.754 and 0.416
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6717 / 1 / 383
Goodness-of-fit on F^2	1.042
Final R indices [I>2sigma(I)]	R1 = 0.0719, wR2 = 0.1766
R indices (all data)	R1 = 0.0725, wR2 = 0.1770
Extinction coefficient	n/a
Largest diff. peak and hole	8.609 and -2.607 e.Å^{-3}

Table S7. Crystal data for **7**

Empirical formula	C14 H28 B10 Cl Ir S2	
CCDC Number	2042340	
Formula weight	596.23	
Temperature	300(2) K	
Wavelength	1.54184 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.4735(10) Å	□= 99.929(8)°.
	b = 15.2045(18) Å	□=
104.054(8)°.		
	c = 15.997(4) Å	□ =
105.830(5)°.		
Volume	2298.5(7) Å^3	

Z	4
Density (calculated)	1.723 Mg/m^3
Absorption coefficient	13.970 mm^{-1}
F(000)	1152
Crystal size	0.220 x 0.200 x 0.160 mm^3
Theta range for data collection	2.945 to 73.489°.
Index ranges	-13<=h<=13, -18<=k<=18, -19<=l<=19
Reflections collected	46708
Independent reflections	9186 [R(int) = 0.0571]
Completeness to theta = 67.684°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7538 and 0.3996
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	9186 / 72 / 525
Goodness-of-fit on F^2	1.027
Final R indices [I>2sigma(I)]	R1 = 0.0490, wR2 = 0.1295
R indices (all data)	R1 = 0.0510, wR2 = 0.1315
Extinction coefficient	n/a
Largest diff. peak and hole	7.332 and -2.572 $e.Å^{-3}$

Table S8. Crystal data for **8**

Empirical formula	C20 H33 B10 Ir S3
CCDC Number	2042339
Formula weight	669.94
Temperature	173(2) K
Wavelength	1.54178 Å
Crystal system	Triclinic
Space group	P-1

Unit cell dimensions	a = 8.1015(3) Å □= 94.7220(10)°.
	b = 9.9248(3) Å □= 99.0470(10)°.
	c = 17.6172(6) Å □ = 102.5030(10)°.
Volume	1355.74(8) $Å^3$
Z	2
Density (calculated)	1.641 Mg/m^3
Absorption coefficient	11.734 mm^{-1}
F(000)	656
Crystal size	0.210 x 0.180 x 0.150 mm^3
Theta range for data collection	2.558 to 71.497°.
Index ranges	-9<=h<=9, -12<=k<=12, -21<=l<=21
Reflections collected	27525
Independent reflections	5254 [R(int) = 0.0329]
Completeness to theta = 67.679°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.754 and 0.492
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5254 / 1 / 317
Goodness-of-fit on F^2	1.100
Final R indices [I>2sigma(I)]	R1 = 0.0154, wR2 = 0.0375
R indices (all data)	R1 = 0.0158, wR2 = 0.0377
Extinction coefficient	n/a
Largest diff. peak and hole	0.846 and -0.607 $e.Å^{-3}$

Table S9. Crystal data for **9**

Empirical formula	C17 H39 B20 Ir S4
CCDC Number	2042341
Formula weight	780.12
Temperature	173(2) K
Wavelength	1.54178 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 12.7778(15) Å □= 90°.
	b = 15.0383(13) Å □= 108.269(8)°.
	c = 18.354(4) Å □ = 90°.
Volume	3349.1(8) $Å^3$
Z	4
Density (calculated)	1.547 Mg/m^3
Absorption coefficient	10.112 mm^{-1}
F(000)	1528
Crystal size	0.140 x 0.100 x 0.030 mm^3
Theta range for data collection	3.729 to 71.492°.
Index ranges	-15<=h<=15, -18<=k<=18, -22<=l<=22
Reflections collected	84677
Independent reflections	6540 [R(int) = 0.0598]
Completeness to theta = 67.679°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.754 and 0.426
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6540 / 2 / 393
Goodness-of-fit on F^2	1.034

Final R indices [I>2sigma(I)]	R1 = 0.0232, wR2 = 0.0618
R indices (all data)	R1 = 0.0240, wR2 = 0.0625
Extinction coefficient	n/a
Largest diff. peak and hole	0.898 and -1.312 e.Å^{-3}

Table S10. Crystal data for **10**

Empirical formula	C23 H44 B20 Ir N S3	
CCDC Number	2042342	
Formula weight	839.17	
Temperature	173(2) K	
Wavelength	1.54184 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 9.176(2) Å	□= 90°.
	b = 22.801(5) Å	□= 90°.
	c = 34.588(7) Å	□ = 90°.
Volume	7237(3) Å^3	
Z	8	
Density (calculated)	1.540 Mg/m^3	
Absorption coefficient	8.889 mm^{-1}	
F(000)	3312	
Crystal size	0.250 x 0.220 x 0.180 mm^3	
Theta range for data collection	2.555 to 72.997°.	
Index ranges	-11<=h<=11, -24<=k<=28, -42<=l<=42	
Reflections collected	138695	
Independent reflections	7248 [R(int) = 0.0792]	
Completeness to theta = 67.684°	100.0 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.751 and 0.558
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7248 / 56 / 467
Goodness-of-fit on F^2	1.086
Final R indices [I>2sigma(I)]	R1 = 0.0264, wR2 = 0.0541
R indices (all data)	R1 = 0.0361, wR2 = 0.0582
Extinction coefficient	n/a
Largest diff. peak and hole	1.421 and -0.719 e.Å^{-3}

Table S11. Crystal data for **11.**

Empirical formula	C28 H66 B30 Ir2 S4	
CCDC Number	2042343	
Formula weight	1239.74	
Temperature	200(2) K	
Wavelength	1.54184 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.347(3) Å	a= 102.28(3)°.
	b = 15.930(5) Å	b= 95.76(2)°.
	c = 21.753(5) Å	g = 112.81(3)°.
Volume	2551.2(14) Å^3	
Z	2	
Density (calculated)	1.614 Mg/m^3	
Absorption coefficient	11.640 mm^{-1}	
F(000)	1204	
Crystal size	0.130 x 0.100 x 0.080 mm^3	
Theta range for data collection	2.123 to 73.989°.	

Index ranges	-10<=h<=10, -19<=k<=19, -27<=l<=27
Reflections collected	56759
Independent reflections	10329 [R(int) = 0.0443]
Completeness to theta = 67.684°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7538 and 0.5699
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10329 / 22 / 604
Goodness-of-fit on F^2	1.041
Final R indices [I>2sigma(I)]	R1 = 0.0278, wR2 = 0.0745
R indices (all data)	R1 = 0.0303, wR2 = 0.0762
Extinction coefficient	n/a
Largest diff. peak and hole	2.545 and -1.331 e.$Å^{-3}$

4.References

[1]C. White, A. Yates, P. M. Maitlis and D. M. Heinekey, *Inorg. Syth.*, 1992, **29**, 228-230.