

A General Photo-Induced Wide-Scope Regioselective Hydroboration of Alkenes without Using Photocatalyst or External Initiator

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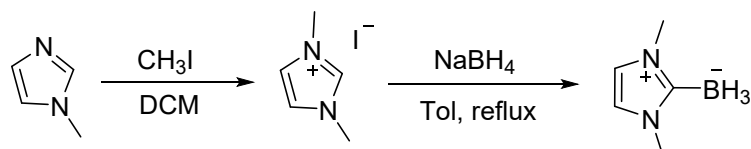
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1. General information

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. The Blue LEDs lamp was directly purchased from the supermarket. Reactions were monitored by thin-layer chromatography (TLC) using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm). ^1H , $^1\text{H}\{^{11}\text{B}\}$, and ^{13}C NMR spectra were recorded on Bruker Avance 600 and 400 spectrometers using CDCl_3 or DMSO as solvents. Chemical shifts (δ) are reported in ppm relative to the solvent peak. The ^{11}B and $^{11}\text{B}\{^1\text{H}\}$ NMR spectra were obtained at 128 or 193 MHz. All ^{11}B chemical shifts are referenced to $\text{BF}_3\cdot\text{OEt}_2$ (0.0 ppm), with a negative sign indicating an upfield shift. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), q (quartet), with coupling constants (J) in hertz (Hz).

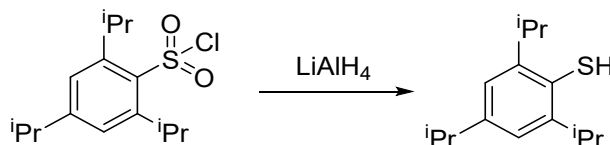
2. Synthesis of Starting Materials

2.1 Preparation of NHC-BH₃ using the modified literature method¹



A 100 mL Schlenk-tube was charged with 1-methylimidazole (8.2 g, 100 mmol, 1.0 equiv.) in CH_2Cl_2 (20 mL) to which methyl iodide (16.9 g, 120 mmol, 1.2 equiv.) was added dropwise over 10 min at 0 °C. The mixture was allowed to stir for 2 h at room temperature. The solvent was evaporated under the reduced pressure and obtained the crude product, and directly used for the next step without further purification. Then the mixture was added 50 mL toluene and NaBH_4 (4.53 g, 120 mmol, 1.2 equiv.), and the mixture was placed in an oil bath at 120 °C for 24 h. The hot reaction solvent was cautiously decanted from the insoluble mixture, and the remaining residue was extracted with hot toluene (2×20 mL). The organic extracts were combined, evaporated, and further recrystallized over water to give the 5.3 g pure product as a fine white crystal in a 48% yield.

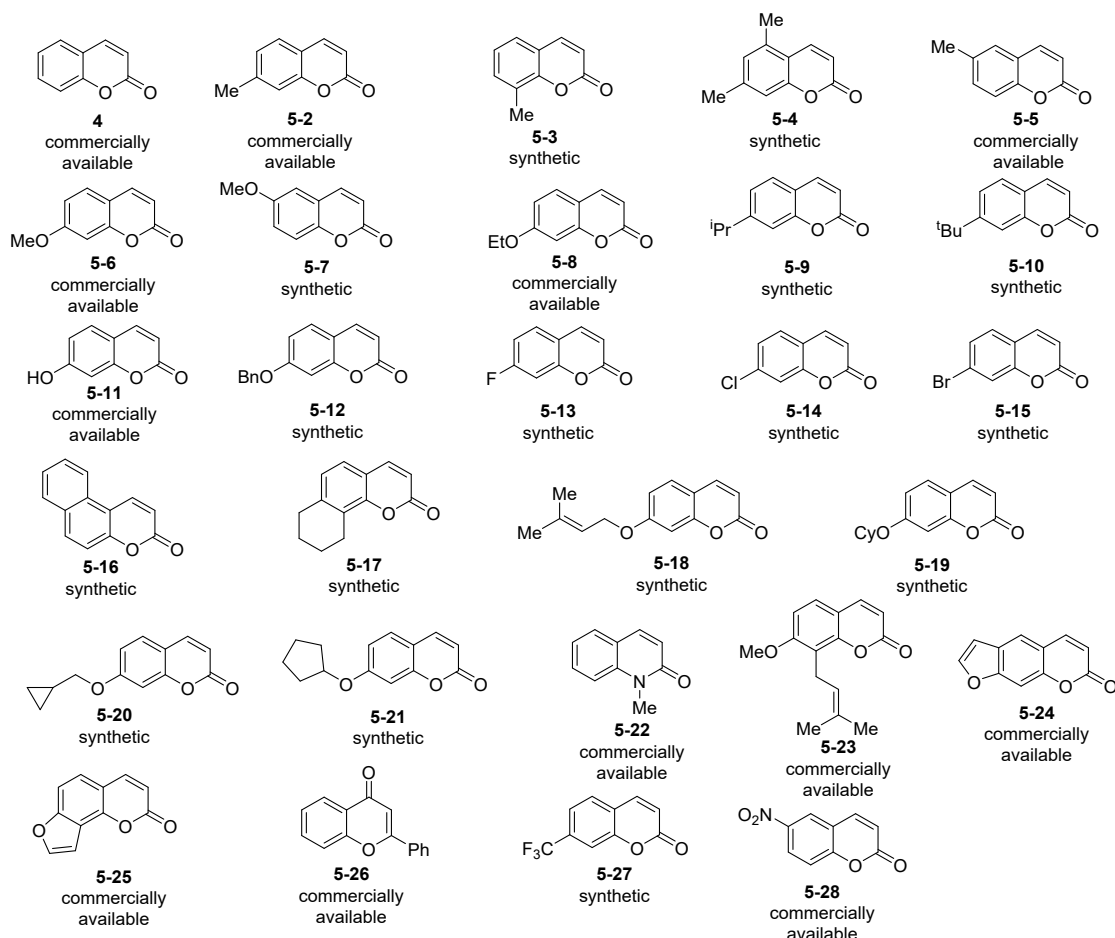
2.2 Preparation of TRIP thiol using the modified literature method²



A 100 mL Schlenk-tube was charged with lithium aluminum hydride (LiAlH_4) (1.52 g, 40.0 mmol, 2.0 equiv.). Dry Et_2O (20 mL) was then added and cooled to 0 °C. To this mixture, a solution of 2,4,6-triisopropylbenzene-1-sulfonyl chloride (5.96 g, 20 mmol, 1.0 equiv.) in Et_2O (20 mL) was added slowly. After completion of the addition, an additional load of LiAlH_4 (0.76 g, 20 mmol, 1.0 equiv.) was added. The reaction was allowed to warm to rt and it was stirred overnight. Upon completion, the reaction was cooled to 0 °C and diluted with 40 mL Et_2O . The reaction was quenched with water (4 mL), 4 mL 15% (w/w) NaOH solution, and 10 mL water. The reaction was stirred for

30 min at 0 °C before MgSO₄ was added. The resulting white slurry was allowed to stir for 30 minutes at rt. The white solids were removed via filtration, with Et₂O washing. The filtrate was then concentrated and distilled at reduced pressure to provide 3.8 g of 2,4,6-triisopropyl-benzenethiol as colorless oil in 80% yield.

2.3 Starting coumarin derivatives.



2.4 General Experimental Procedure for the Synthesis of Coumarins 5-3, 5-4, 5-7, 5-9, 5-10, 5-12 to 5-17.

A 100 mL Schlenk-tube was charged with phenol derivatives (10.0 mmol, 1.0 equiv.), trifluoromethanesulfonic acid (1.5 g, 10.0 mmol, 1.0 equiv.), and propiolic acid (350 mg, 5.0 mmol, 0.5 equiv.) in PhCl (25 mL), which was stirred at 100 °C for 3 h. After the starting material was consumed, monitored by TLC. The reaction was poured into H₂O, neutralized with NaHCO₃ solution, and extracted with DCM (3 × 15 mL), dried by anhydrous Na₂SO₄, and the solvent was evaporated under the reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain products.

2.5 General Experimental Procedure for the Synthesis of Coumarins 5-18 to 5-21.

A 100mL Schlenk-tube was charged with 7-hydroxycoumarin (810 mg, 5.0 mmol, 1.0 equiv.), alkyl or aryl halide (6.0 mmol, 1.2 equiv.), and potassium carbonate (2.07 g, 15.0 mmol, 3.0 equiv.) in DMF (15 mL), which was stirred at 100 °C for 10 h. After the starting material was consumed, monitored by TLC. The reaction was poured into H₂O, extracted with ethyl acetate (3 × 15 mL), and the combined extracts were washed

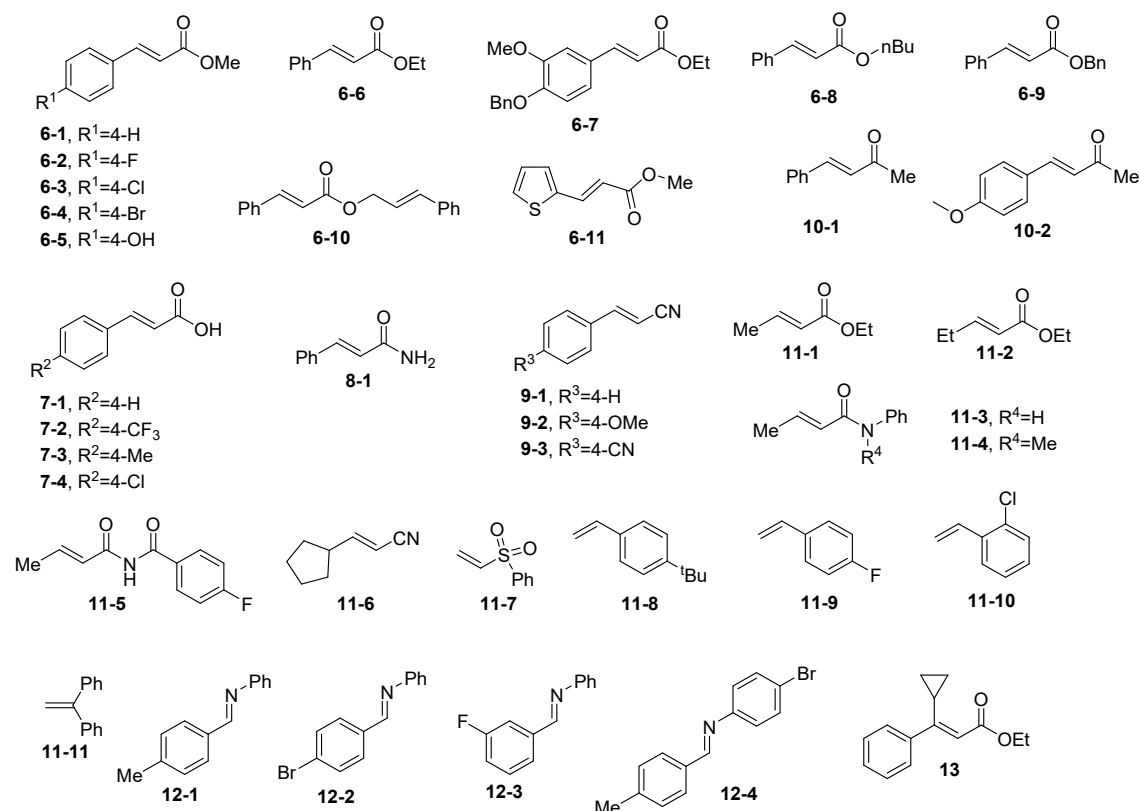
with water. The organic layer was dried over anhydrous Na₂SO₄ and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product.

2.6 General Experimental Procedure for the Synthesis of Coumarins 5-27¹⁵

A 10 mL Schlenk-tube was charged with (*E*)-3-(4-(trifluoromethyl)phenyl)acrylic acid (1 mmol, 1.0 equiv.), Xanthone (0.05 mmol, 0.05 equiv.), selectflour (1 mmol, 1 equiv.) in HFIP (5 mL) and DCE (1 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (400 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under the reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products.

2.7 Starting α,β -unsaturated esters, acids, ketones, amide, nitriles, styrenes, imines.

9-2, 9-3, 11-3, 11-4, 11-5, 12-1 to 12-4, 13 were synthetic, others were commercially available.



2.8 General Experimental Procedure for the Synthesis of 9-2, 9-3⁴.

To a mixture of NaH (2.4 mmol, 1.2 equiv.) in dry THF was added diethyl(cyanomethyl)phosphonate (2.4 mmol, 1.2 equiv.) at 0 °C under N₂ atmosphere. The mixture was stirred at room temperature for 0.5 h before adding the solution of aldehyde (2 mmol, 1.0 equiv.). The starting material was consumed, analyzed by TLC. The reaction was quenched with saturated NH₄Cl (aq.). The mixture was extracted with ethyl acetate (3 × 15 mL), the organic layer was dried over anhydrous Na₂SO₄ and evaporated under reduced pressure. The crude materials were purified by column

chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product.

2.9 General Experimental Procedure for the Synthesis of 11-3 to 11-5⁵.

A 100 mL Schlenk-tube was charged with aniline (3.0 mmol, 1.0 equiv.), potassium carbonate (420 mg, 3.0 mmol, 1.0 equiv.) in DCM (15 mL) to which crotonyl chloride (374 mg, 3.6 mmol, 1.2 equiv.) was added by syringe under N₂ atmosphere. The starting material was consumed, monitored by TLC. The reaction was poured into H₂O, extracted with ethyl acetate (3 × 15 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products **11-3**, **11-4**.

A 100 mL Schlenk-tube was charged with 4-fluorobenzamide (556 mg, 4.0 mmol, 2.0 equiv.) in THF (20 mL), NaH (192 mg, 4.8 mmol, 2.4 equiv.) was added at 0 °C. After stirring the reaction for 0.5 h, crotonyl chloride (210 mg, 2.0 mmol, 1.0 equiv.) was added at 0°C, after 10 minutes the solution was allowed to warm to room temperature and stirred for 4 h. The reaction was poured into H₂O, extracted with ethyl acetate (3 × 15 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product **11-5**.

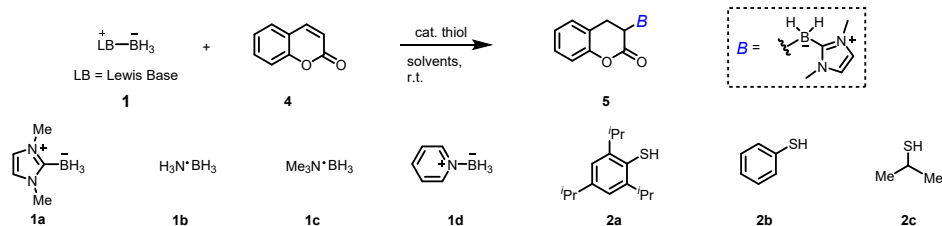
2.10 General Experimental Procedure for the Synthesis of 12-1 to 12-4⁶.

A 100 mL Schlenk-tube was charged with an aldehyde (10.0 mmol, 1 equiv.), aniline (10.0 mmol, 1 equiv.), and MgSO₄ (1.56 g, 13 mmol, 1.3 equiv.) in DCM (12 mL), which was stirred for 10h under N₂ atmosphere. The starting material was consumed, monitored by TLC. Then the mixture was filtered and the filter liquor was evaporated under reduced pressure. The final imines were recrystallized from n-hexane.

2.11 General Experimental Procedure for the Synthesis of 13¹³.

A 25 mL Schlenk-tube was charged with NaH (80 mg, 2 mmol, 2.0 equiv.) in dry THF, the mixture was cooled to 0 °C, triethylphosphonoacetate (448 mg, 2 mmol, 2.0 equiv.) was added dropwise to the solution before the resulting solution was stirred for 1 h at 0 °C. The cyclopropyl(phenyl)methanone (146 mg, 1 mmol, 1.0 equiv.) was added to the reaction mixture, which was stirred for 1 h at 0 °C and another 20 h at 40 °C oil bath. The organic phase was separated and the aqueous phase was extracted with Et₂O (3 × 10 mL). The combined organic phases were dried over MgSO₄, concentrated in vacuo, and purified by silica gel column chromatography.

3. Reaction condition optimization



Entry	Borane	Thiol	Equiv. of 1	Loading of thiol (mmol %)	Solvent	Conc. (M)	Atmosphere	Yield (%) ^a
1	1a	2a	1.3	10	THF	0.1	N ₂	90
2	1b	2a	1.3	10	THF	0.1	N ₂	0
3	1c	2a	1.3	10	THF	0.1	N ₂	0
4	1d	2a	1.3	10	THF	0.1	N ₂	0
5	1a	2b	1.3	10	THF	0.1	N ₂	82
6	1a	2c	1.3	10	THF	0.1	N ₂	0
7	1a	2a	1.3	10	MeCN	0.1	N ₂	87
8	1a	2a	1.3	10	Toluene	0.1	N ₂	41
9	1a	2a	1.3	10	1,4-Dioxane	0.1	N ₂	59
10	1a	2a	1.3	10	DCE	0.1	N ₂	87
11	1a	2a	1.3	10	MeOH	0.1	N ₂	86
12	1a	2a	1.3	10	H ₂ O	0.1	N ₂	70
13	1a	2a	1.3	20	THF	0.1	N ₂	92
14	1a	2a	1.3	5	THF	0.1	N ₂	78
15	1a	2a	1.5	10	THF	0.1	N ₂	90
16	1a	2a	1.1	10	THF	0.1	N ₂	85
17	1a	2a	1.3	10	THF	0.1	Air	16
18	1a	2a	1.3	10	THF	0.2	N ₂	93
19	1a	2a	1.3	10	THF	0.2	N ₂	0 ^b
20	1a	2a	1.3	0	THF	0.2	N ₂	0

Table S1: Unless otherwise specified, reaction conditions were: coumarin (0.2 mmol, 1 equiv.), borane (0.26 mmol, 1.3 equiv.), thiol (0.02 mmol, 0.1 equiv.), and solvents (2 mL) were irradiated with blue LEDs for 24 h under nitrogen atmosphere. yields were determined by ¹H NMR using 1,3,5-methoxybenzen as the internal standard. ^bThe reaction was set up in dark. DCE, 1,2-dichloroethane.

4. General Experimental Procedure for the hydroboration of alkenes.

4.1 General Experimental Procedure for the hydroboration of coumarins.

A 25 mL Schlenk-tube was charged with coumarins (2 mmol, 1.0 equiv.), NHC-borane (286 mg, 2.6 mmol, 1.3 equiv.), 2,4,6-triisopropyl-thiophenol (47 mg, 0.2 mmol, 0.1 equiv.) in dry THF (10 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature.

General purified procedure of product **5a**.

After the reaction was illuminated for 24 h, the mixture was filtered and obtained the product directly. The product was washed with petroleum ether, and the product was

separated from the mother liquor, which was filtered again.

General purified procedure of products **5b**, **5f**, **5n**, **5o**, **5r**, **5v**.

After the reaction was illuminated for 24 h, the mixture was added 5 mL petroleum ether and remained stationary for 6-24 h until the product was separated from the solvent. Then the final product was filtered directly.

General purified procedure of product **5c**.

After the reaction was illuminated for 24 h, the mixture was added 10 mL petroleum ether and remained stationary for 6 h until the product was separated from the solvent. Then the final product was filtered directly.

General purified procedure of products **5d**, **5h**, **5i**.

After the reaction was illuminated for 24 h, THF was evaporated under reduced pressure. The residue was poured into 30 mL petroleum ether and stirred for 6 h. The crude product was filtered and added 1 mL ethyl acetate, which was stirred for 2 h and filtered to obtain the final product.

General purified procedure of products **5e**, **5l**, **5t**, **5x**, **5y**.

After the reaction was illuminated for 24 h, the product was separated from THF and filtered directly.

General purified procedure of products **5g**, **5s**, **5w**, **5z**.

After the reaction was illuminated for 24 h, THF was evaporated under reduced pressure. The residue was poured into 30 mL petroleum ether and stirred for 6 h. The crude product was filtered and added 2 mL ethyl acetate, which was stirred for 2 h and filtered to obtain the final product.

General purified procedure of product **5j**.

After the reaction was illuminated for 24 h, THF was evaporated under reduced pressure. The residue was poured into 30 mL petroleum ether and stirred for 6 h. The crude product was filtered and added 3 mL ethyl acetate and 7 mL petroleum ether, then stirred for 2 h and filtered to obtain the final product.

General purified procedure of product **5k**.

After the reaction was illuminated for 24 h, the crude product was separated from the THF and filtered. The crude product was added to 4 mL ethyl acetate and stirred for 4 h. The mixture was filtered to offer the final product.

General purified procedure of product **5m**.

After the reaction was illuminated for 24 h, the mixture was added 7 mL petroleum ether and remained stationary for 12 h until the product was separated from the solvent. The mixture was filtered to obtain the final product, and the filter liquor was poured into 15 mL petroleum ether. The final product was separated from the filter liquor and filtered again.

General purified procedure of product **5p**.

After the reaction was illuminated for 24 h, THF was evaporated under reduced pressure. The residue was poured into 30 mL petroleum ether and stirred for 6 h. The crude product was filtered and added 3 mL ethyl acetate, which was stirred for 2 h and filtered to obtain the final product.

General purified procedure of product **5q**.

After the reaction was illuminated for 24 h, the mixture was added 5 mL petroleum

ether and remained stationary for 12 h until the product was separated from the solvent, then the final product was filtered directly. The filtered liquor was evaporated under reduced pressure. The mixture was added 3 mL ethyl acetate and stirred for 4 h. The mixture was also filtered to obtain the final product.

General purified procedure of product **5u**.

After the reaction was illuminated for 24 h, the mixture was filtered and obtained the final product directly. The filtered liquor was added 15 mL petroleum ether, and the crude product was separated from filter liquor, which was filtered. The crude product was added 2 mL ethyl acetate, which was stirred for 2 h and filtered to obtain the final product.

4.2 General Experimental Procedure for the hydroboration of coumarins 5-27, 5-28.

A 25 mL Schlenk-tube was charged with coumarins (0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 1.3 equiv.), 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.1 equiv.) in dry THF (2 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products **5aa**.

4.3 General Experimental Procedure for the hydroboration of 6-1 to 6-11, 7-1 to 7-4, 8-1, 9-1 to 9-3, 10-1, 10-2, 11-3, 11-4, 11-6, 11-7, 11-11

A 10 mL Schlenk-tube was charged with alkenes (0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 0.13 equiv.), 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.01 equiv.) in dry THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products **6a-k, 7a-d, 8a, 9a-c, 10a-b, 11c-d, 11f, 11g, 11k**.

4.4 General Experimental Procedure for the hydroboration of 11-1, 11-2.

A 10 mL Schlenk-tube was charged with alkenes (0.4 mmol, 1.0 equiv.), NHC-borane (57.2 mg, 0.52 mmol, 0.13 equiv.), 2,4,6-triisopropyl-thiophenol (9.4 mg, 0.04 mmol, 0.01 equiv.) in dry THF (3 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products **11a, 11b**.

4.5 General Experimental Procedure for the hydroboration of 11-5.

A 10 mL Schlenk-tube was charged with alkenes (41 mg, 0.2 mmol, 1.0 equiv.), NHC-borane (66 mg, 0.6 mmol, 3 equiv.), 2,4,6-triisopropyl-thiophenol (14.2 mg, 0.06 mmol, 0.03 equiv.) in DCE (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube

was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product **11e**.

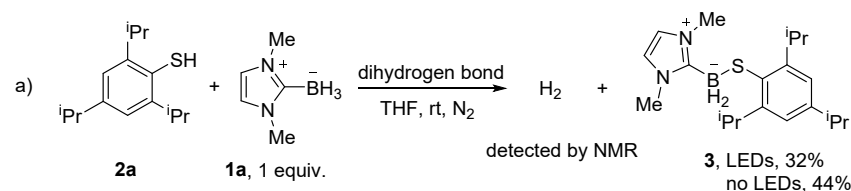
4.6 General Experimental Procedure for the hydroboration of **11-8** to **11-10**.

A 10 mL Schlenk-tube was charged with styrenes (0.2 mmol, 1.0 equiv.), NHC-borane (44 mg, 0.4 mmol, 2 equiv.), 2,4,6-triisopropyl-thiophenol (9.4 mg, 0.04 mmol, 0.02 equiv.) in THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products **11h-j**

4.7 General Experimental Procedure for the hydroboration of **12-1** to **12-4**.

A 10 mL Schlenk-tube was charged with imines (0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 0.13 equiv.), 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.01 equiv.) in dry THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the products **12a-d**.

5. The synthesis of boryl sulphide and its photochemical reaction



A 10 mL Schlenk-tube was charged with NHC-borane (165 mg, 1.5 mmol, 1.0 equiv.) and 2,4,6-triisopropyl-thiophenol (354 mg, 1.5 mmol, 1.0 equiv.) in THF (5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. The resulting gas was passed through Tol-*d*₈ and detected by ¹H NMR, showing that H₂ was produced. Then the reaction was added 10 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product 165 mg **3** in 32% isolated yield.

A 10 mL Schlenk-tube was charged with NHC-borane (165 mg, 1.5 mmol, 1.0 equiv.) and 2,4,6-triisopropyl-thiophenol (354 mg, 1.5 mmol, 1.0 equiv.) in THF (5 mL) with magnetic stirring under N₂ atmosphere. The mixture was stirred for 24 h at room temperature under dark conditions. The resulting gas was passed through Tol-*d*₈ and detected by ¹H NMR, showing that H₂ was produced. Then the reaction was added 10

mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product 227 mg **3** in 44% isolated yield.

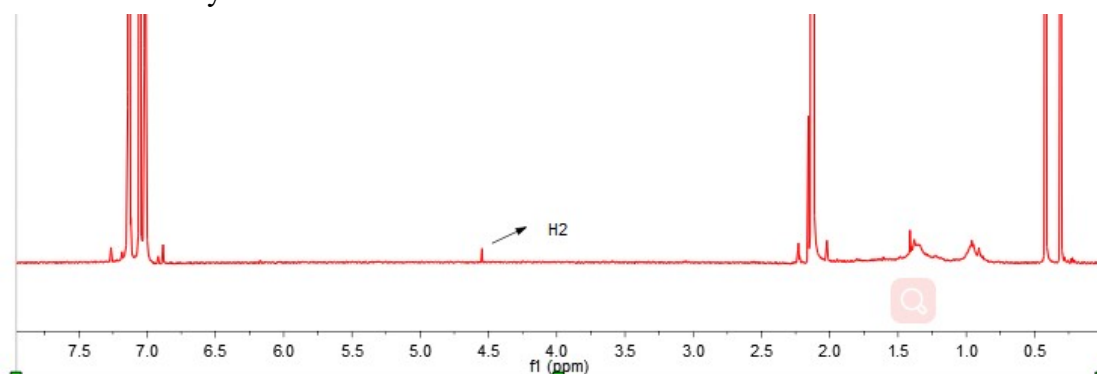
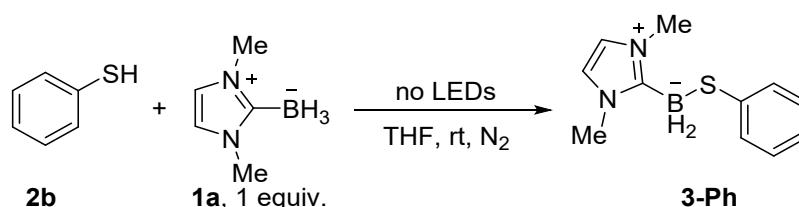
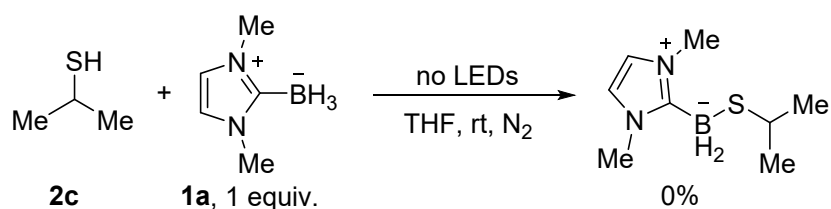


Figure S1. The ¹H NMR spectra of the H₂ in Tol-*d*₈



A 10 mL Schlenk-tube was charged with NHC-borane (22 mg, 0.2 mmol, 1.0 equiv.) and thiophenol (22 mg, 0.2 mmol, 1.0 equiv.) in THF (2 mL) with magnetic stirring under N₂ atmosphere. The mixture was stirred for 24 h at room temperature. Then the reaction was added 3 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product 11 mg **3-Ph** in a 25% isolated yield.



A 10 mL Schlenk-tube was charged with NHC-borane (22 mg, 0.2 mmol, 1.0 equiv.) and 2-mercapto-2-methylpropane (15.2 mg, 0.2 mmol, 1.0 equiv.) in THF (2 mL) with magnetic stirring under N₂ atmosphere. The mixture was stirred for 24 h at room temperature under dark conditions. No targeted compound was generated by TLC and ¹¹B NMR was detected.

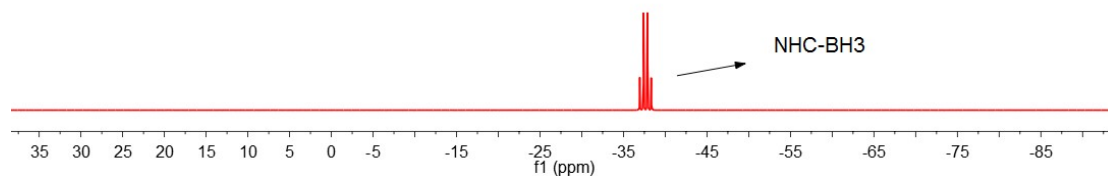
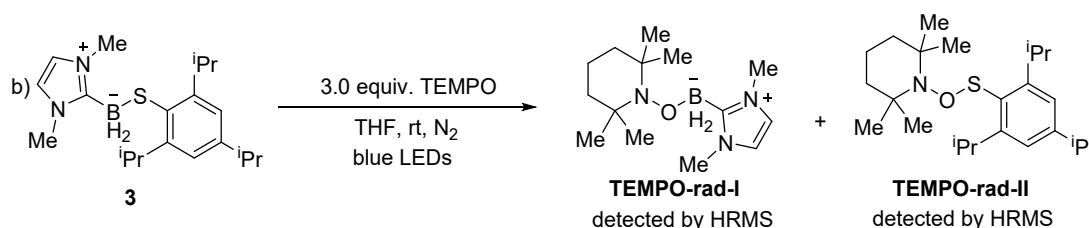


Figure S2. The ^{11}B NMR spectra of the reaction mixture with propane-2-thiol and **1a** in THF.



A 10 mL Schlenk-tube was charged with compound **3** (34 mg, 0.1 mmol, 1.0 equiv.) and TEMPO (47 mg, 0.3 mmol, 3.0 equiv.) in THF (2 mL) with magnetic stirring under N_2 atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) and the mixture was stirred for 24 h at room temperature. We found the boryl radical-trapping and thiyl radical-trapping products by HRMS.

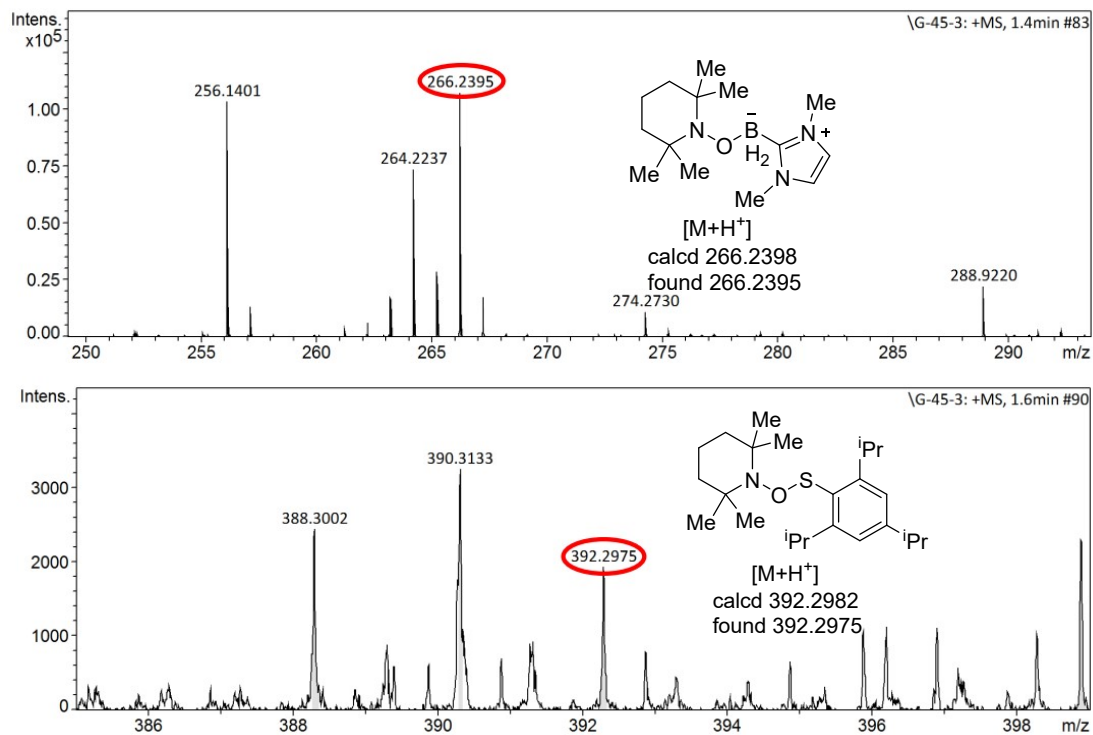
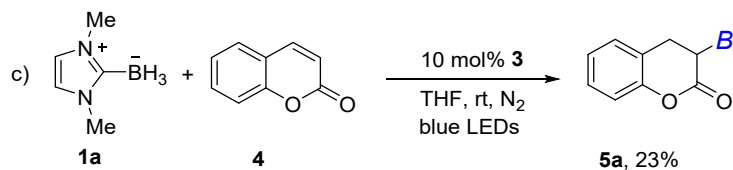
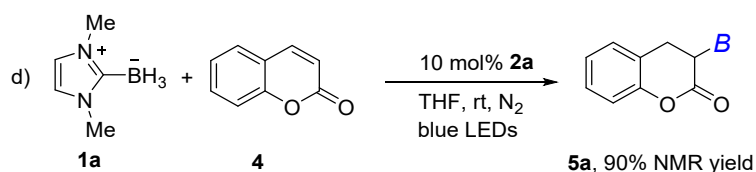


Figure S3. HRMS data of the reaction mixture



A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 1.3 equiv.) and compound **3** (6.9 mg, 0.02 mmol, 0.1 equiv.) in THF (2 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 10 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the 12 mg product in 23% yield.



A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 1.3 equiv.) and compound **3** (6.9 mg, 0.02 mmol, 0.1 equiv.) in THF (2 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 10 h at room temperature. The solvent was evaporated under the reduced pressure and the mixture was subjected to ¹H NMR to determine the yield (90%) using 1,3,5-methoxybenzen as the internal standard.

6. The UV/vis absorption and fluorescence spectroscopy.

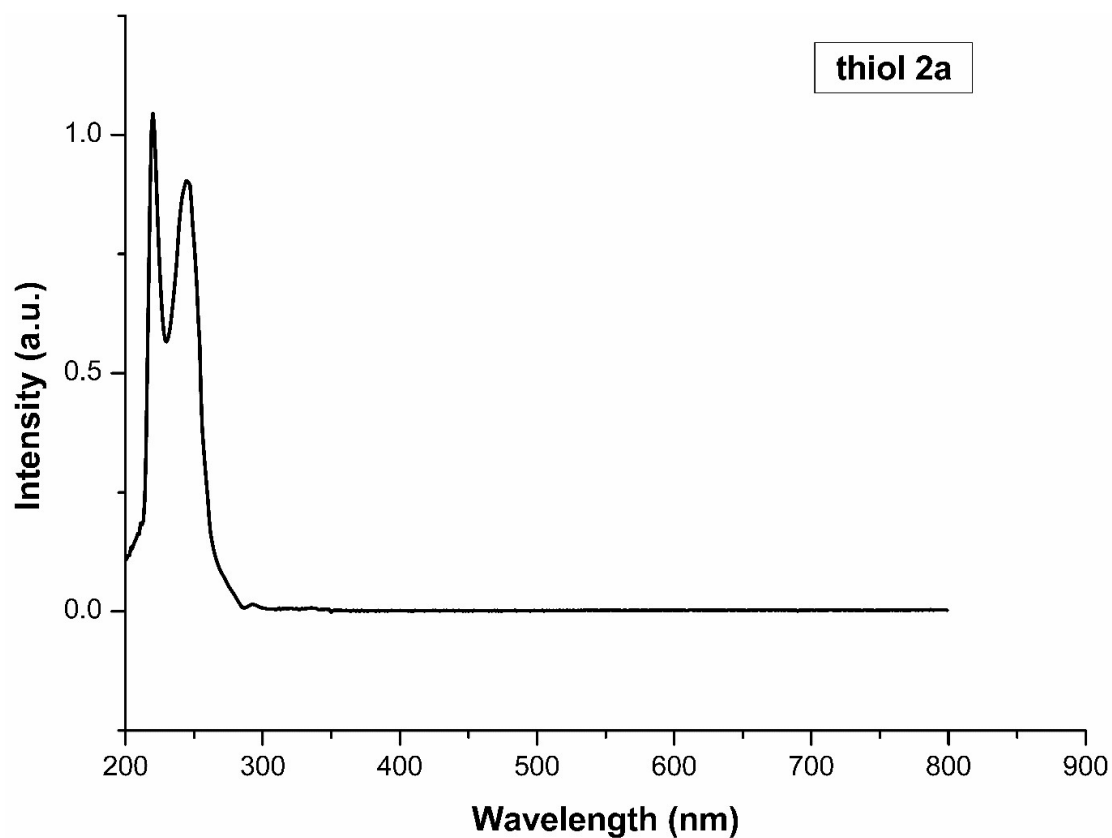


Figure S4. UV/vis absorption of thiol 2a

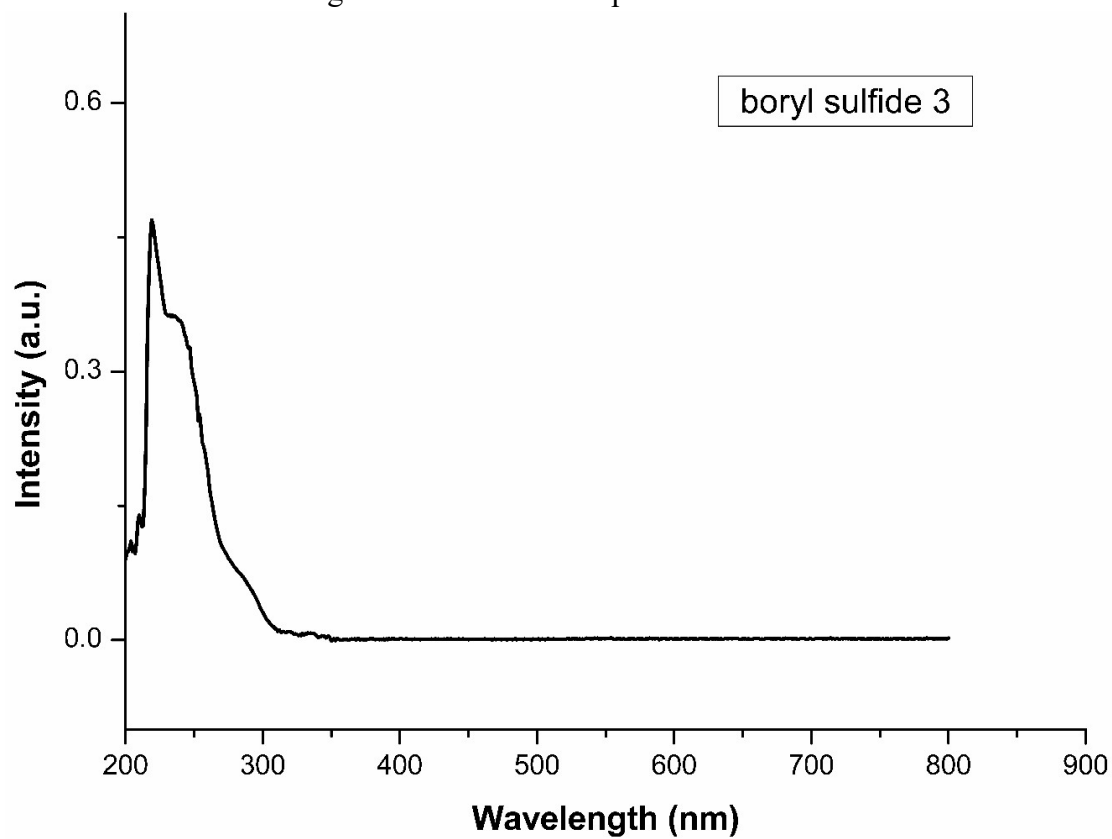


Figure S5. UV/vis absorption of boryl sulphide 3

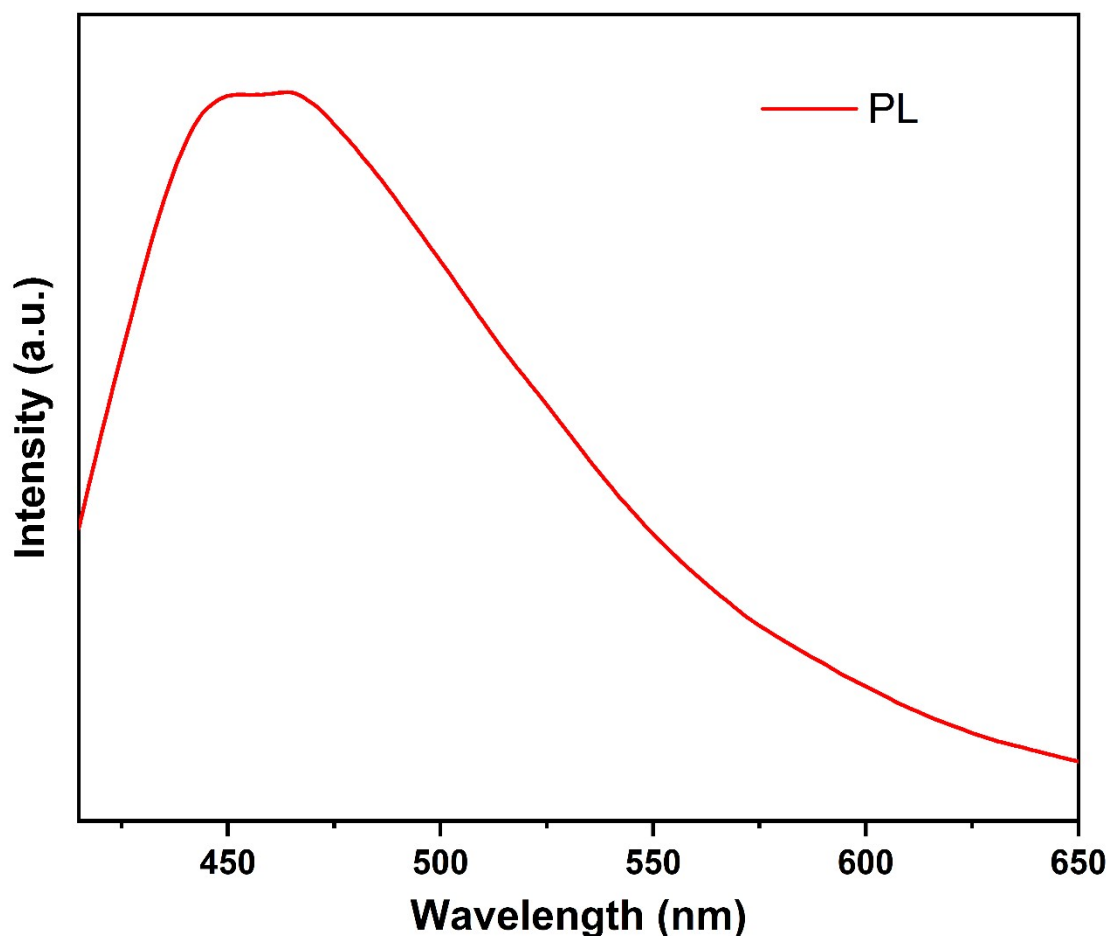
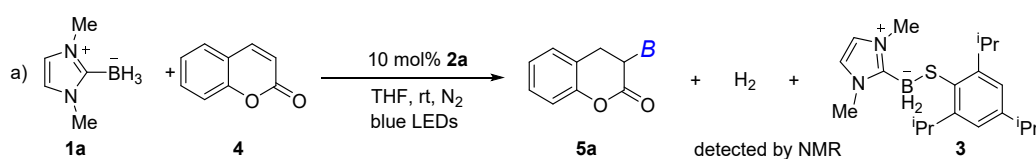


Figure S6. fluorescence spectroscopy of boryl sulphide **3**

6. Mechanistic Experiments.

(I) Monitoring the catalytic reaction



A 100 mL Schlenk-tube was charged with coumarin (1.46 g, 10 mmol, 1.0 equiv.), NHC-borane (1.43 g, 13 mmol, 1.3 equiv.) and 2,4,6-triisopropyl-thiophenol (236 mg, 1.0 mmol, 0.1 equiv.) in THF (70 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 10 h at room temperature. The resulting gas was passed through Tol-*d*₈ and detected by ¹H NMR, showing that H₂ was produced. The mixture was detected by ¹¹B NMR and ¹¹B {¹H} NMR, showing that boryl sulphide **3** was generated in the reaction process.

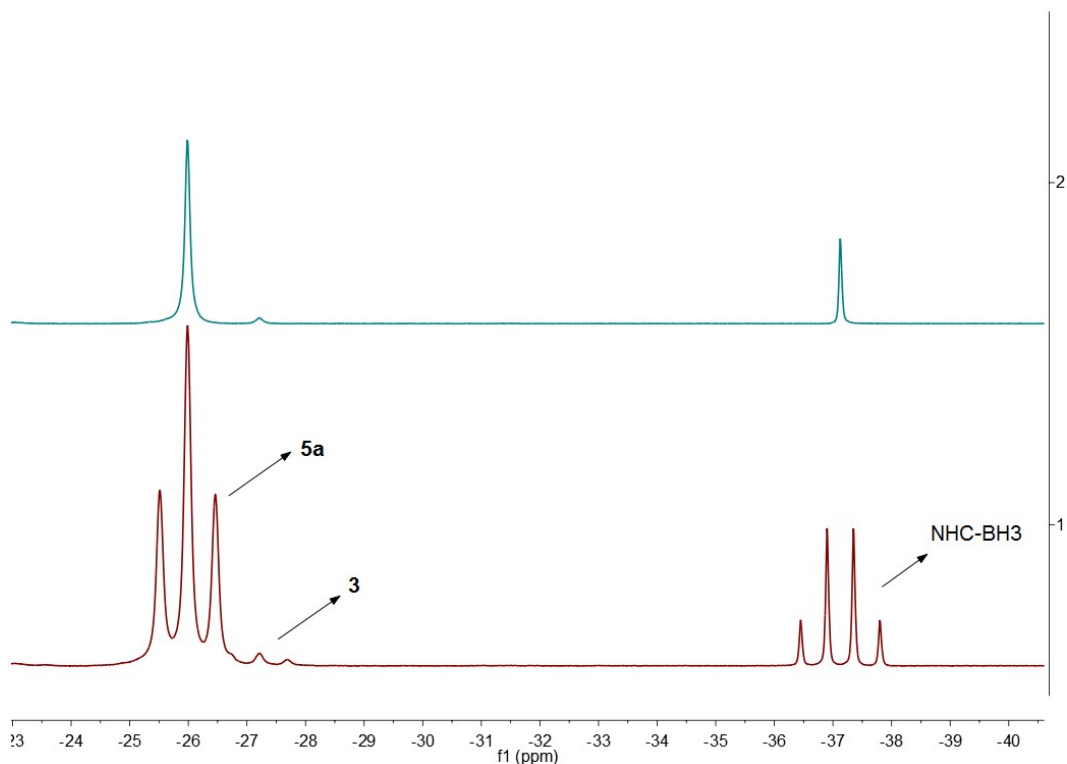


Figure S7. The ^{11}B NMR (a) and ^{11}B $\{^1\text{H}\}$ NMR (b) spectra of the reaction mixture in THF.

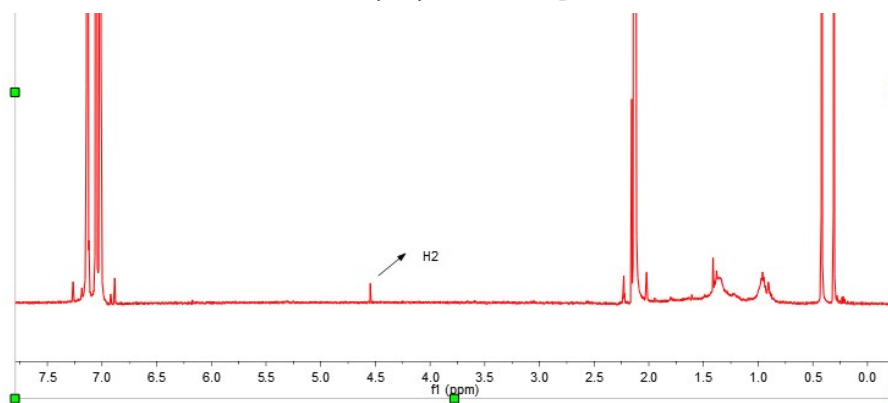
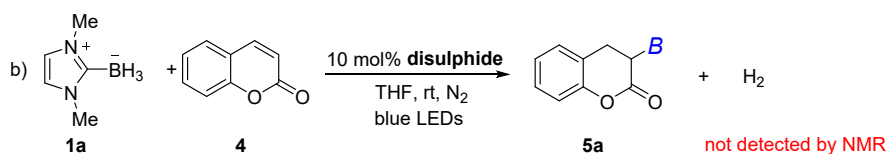
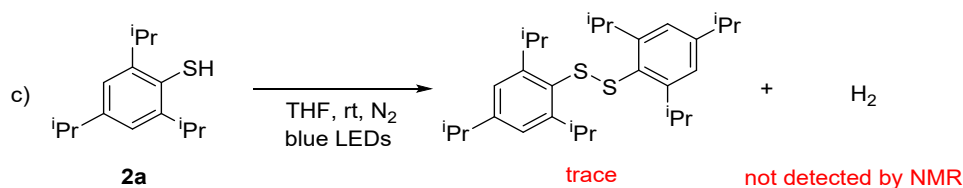


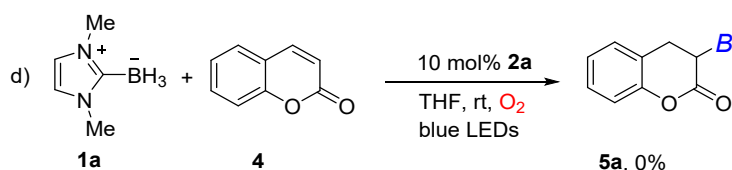
Figure S8. The ^1H NMR spectra of the H_2 in $\text{Tol-}d_8$



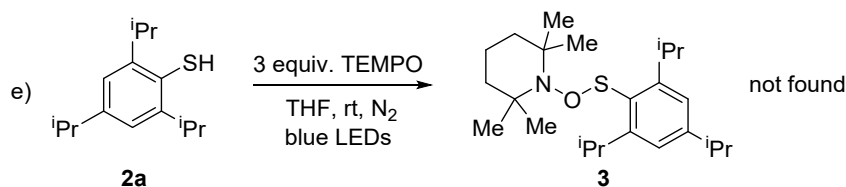
A 100 mL Schlenk-tube was charged with coumarin (1.46 g, 10 mmol, 1.0 equiv.), NHC-borane (1.43 g, 13 mmol, 1.3 equiv.) and disulphide (235 mg, 0.5 mmol, 0.05 equiv.) in THF (70 mL) with magnetic stirring under N_2 atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 10 h at room temperature. The H_2 was not detected by ^1H NMR.



A 10 mL Schlenk-tube was charged with 2,4,6-triisopropylthiophenol (470 mg) in THF (3 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) and the mixture was stirred for 10 h at room temperature. The H₂ was not detected by ¹H NMR. And the only trace disulphide was detected by TLC detected.



A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 1.3 equiv.) and 2,4,6-triisopropylthiophenol (4.7 mg, 0.02 mmol, 0.1 equiv.) in THF (1.5 mL) with magnetic stirring under O₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) and the mixture was stirred for 24 h at room temperature. No desired product was formed by TLC detected.

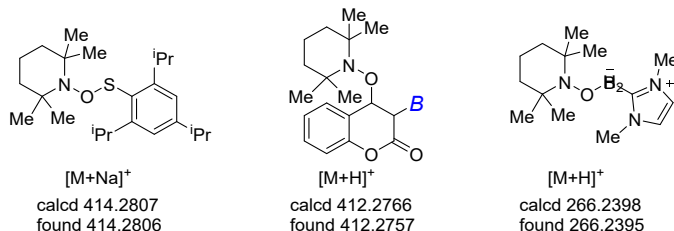


A 10 mL Schlenk-tube was charged with 2,4,6-triisopropylthiophenol (0.1 mmol, 24 mg) and TEMPO (0.3 mmol, 3.0 equiv., 47 mg) in THF (2 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) and the mixture was stirred for 24 h at room temperature. No thiyl radical-trapping product was formed by HRMS detected.

(II) Control experiments and radical-trapping experiments



Detected compounds:



A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 1.3 equiv.), 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.1 equiv.) and TEMPO (94 mg, 0.6 mmol, 3.0 equiv.) in THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. No desired product was formed and we found the boryl radical, thiyl radical, and intermediate A radical-trapping products.

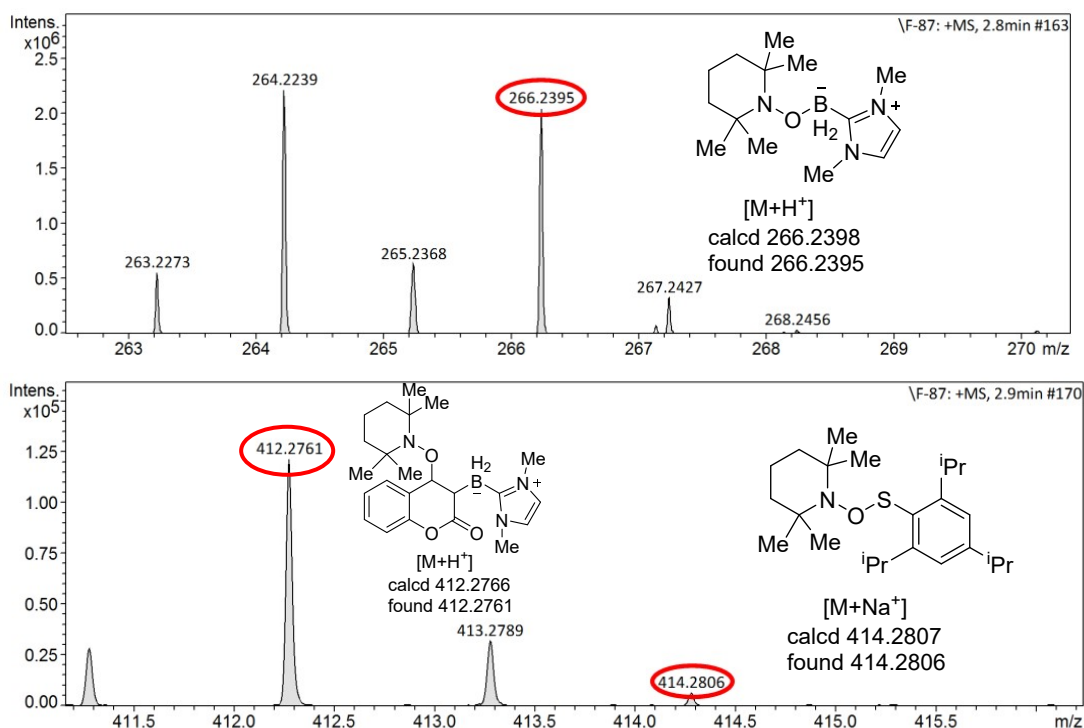
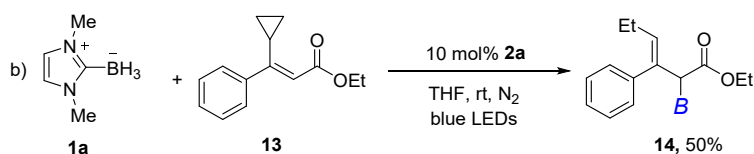


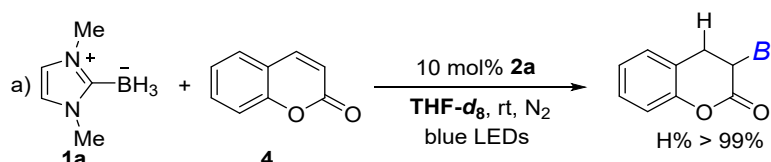
Figure S9. HRMS data of the reaction mixture



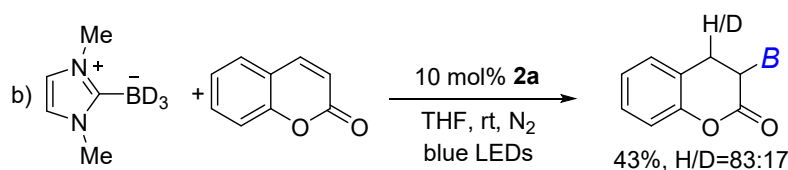
A 10 mL Schlenk-tube was charged with **13** (22 mg, 0.1 mmol, 1.0 equiv.), NHC-borane (14.3 mg, 0.13 mmol, 1.3 equiv.), 2,4,6-triisopropyl-thiophenol (2.4 mg, 0.01

mmol, 0.1 equiv.) in THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. Then the reaction was added 5 mL H₂O, extracted with DCM (3 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated under reduced pressure. The residue was purified by column chromatography (Petroleum ether: EtOAc) on silica gel to obtain the product **14** in 50% yield.

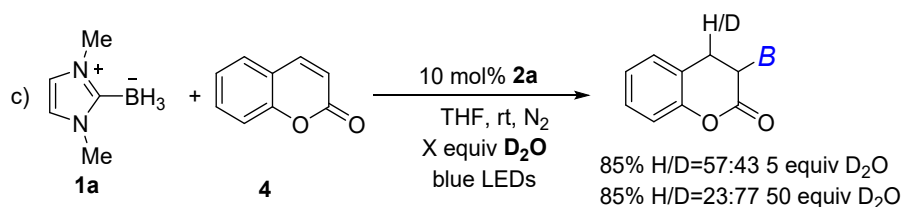
(III) Deuterium-labeled experiments



A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv.), NHC-borane (28.6 mg, 0.26 mmol, 1.3 equiv.) and 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.1 equiv.) in THF-*d*₈ (1.0 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. The solvent was evaporated under the reduced pressure and detected by ¹H NMR, indicating no deuterium hydrogen in the product.

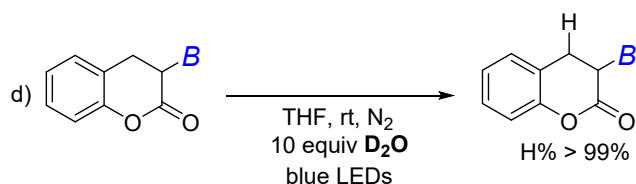


A 10 mL Schlenk-tube was charged with coumarin (14.6 mg, 0.1 mmol, 1.0 equiv.), NHC-BD₃ (14.7 mg, 0.13 mmol, 1.3 equiv.) and 2,4,6-triisopropyl-thiophenol (2.4 mg, 0.01 mmol, 0.1 equiv.) in THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) lamp and the mixture was stirred for 24 h at room temperature. The solvent was evaporated under the reduced pressure and detected by ¹H NMR, indicating 17% (based on the integration value in ¹H NMR spectrum) deuterium hydrogen at the β-position of the product.



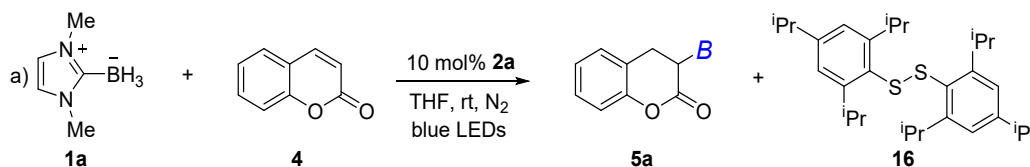
A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv.), NHC-BH₃ (28.6 mg, 0.26 mmol, 1.3 equiv.), 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.1 equiv.) and 5 or 50 Equiv. D₂O in THF (1.5 mL) with magnetic stirring

under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) and the mixture was stirred for 24 h at room temperature. The reaction mixture was dried over anhydrous Na₂SO₄, extracted with DCM (3 × 10 mL), and evaporated under reduced pressure. The data was detected by ¹H NMR, indicating 43% (based on the integration value in ¹H NMR spectrum) deuterium hydrogen at the β-position of the product when the reaction was added 5 Equiv. D₂O and 77% deuterium hydrogen at the β-position of the product when the reaction was added 50 Equiv. D₂O.



A 10 mL Schlenk-tube was charged with **5a** (51.2 mg, 0.2 mmol, 1.0 equiv.), 10 Equiv. D₂O (20 mg, 1 mmol, 10 equiv.) in THF (1.5 mL) with magnetic stirring under N₂ atmosphere. The tube was placed at a distance (app. 5 cm) from 10W blue LEDs (410-420 nm) and the mixture was stirred for 24 h at room temperature. The reaction mixture was dried over anhydrous Na₂SO₄, extracted with DCM (3 × 10 mL), and evaporated under reduced pressure. The data was detected by ¹H NMR, indicating no deuterium hydrogen in the product.

(IV) Formation of disulphide



A 10 mL Schlenk-tube was charged with coumarin (29.2 mg, 0.2 mmol, 1.0 equiv), NHC-boranes (28.6 mg, 0.26 mmol, 1.3 equiv) and 2,4,6-triisopropyl-thiophenol (4.7 mg, 0.02 mmol, 0.1 equiv) in THF (1.5 mL) with magnetic stirring under N₂ atmosphere. We cannot detect the disulfide compound **16** by TLC, nevertheless, we can detect the boryl sulphide **3** by TLC in the reaction process. Fortunately, we detected the disulphide compound **16** by HRMS in the reaction process. These data further support our proposed mechanism.

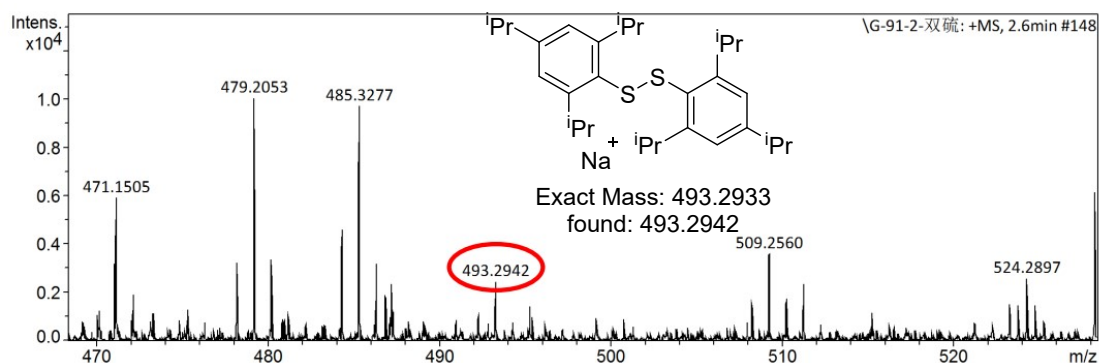


Figure S10. HRMS data of the disulphide **16** of the reaction mixture

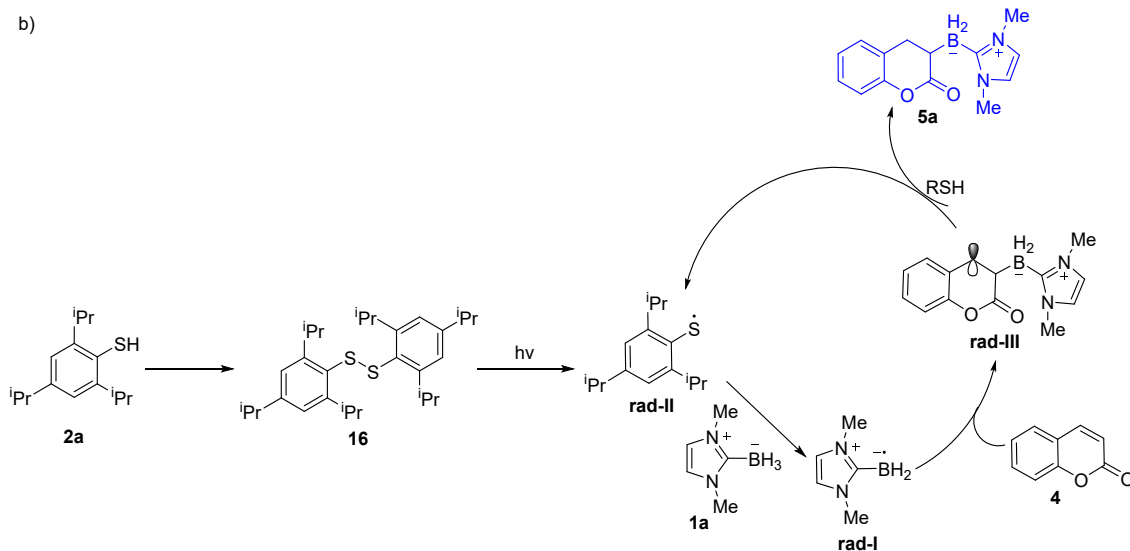
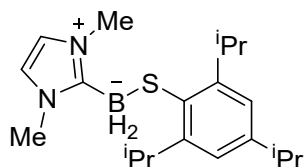


Figure S11. Disulphide mechanism

At this stage, we could not rule out another pathway through disulphide compounds (Figure S8). However, the absence of mediators for S-S bond formation and failure of isolation of disulphide species may at least insinuate this pathway is not a major route for the formation of the products.

7. Characterization data of products.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)((2,4,6-triisopropylphenyl)thio)dihydroborate (**3**): Colorless liquid (227 mg, 44% yield); Gradient eluent: EtOAc/petroleum ether: 1/1 to 2/1.

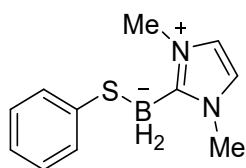
¹H NMR (600 MHz, CDCl₃) δ 6.83 (s, 2H), 6.72 (s, 2H), 3.83 (dq, *J* = 13.8, 6.9 Hz, 2H), 3.38 (s, 6H), 2.81 (dq, *J* = 13.8, 6.9 Hz, 1H), 2.36 (dd, *J* = 175.1, 69.5 Hz, 2H), 1.21 (d, *J* = 6.9 Hz, 6H), 1.09 (d, *J* = 6.9 Hz, 12H).

¹³C NMR (151 MHz, CDCl₃) δ 152.21, 146.07, 135.24, 120.41, 120.37, 35.47, 34.15, 31.21, 24.26, 24.19. ¹¹B NMR (193 MHz, CDCl₃) δ -23.23 (t, *J* = 96.9 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -23.31.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 6.83 (s, 2H), 6.72 (s, 2H), 3.84 (hept, *J* = 6.9 Hz, 2H), 3.39 (s, 6H), 2.81 (dq, *J* = 13.8, 6.9 Hz, 1H), 2.37 (s, 2H), 1.21 (d, *J* = 6.9 Hz, 6H), 1.09 (d, *J* = 6.9 Hz, 12H).

HRMS (ESI-TOF) *m/z*: [M + Na]⁺ cauld. for C₂₀H₃₃BN₂NaS 367.2355; found: 367.2343.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(phenylthio)dihydroborate (**3-Ph**): Colorless liquid (11 mg, 25% yield); Gradient eluent: EtOAc/petroleum ether: 1/4 to 1/2.

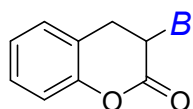
$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.36 (d, $J = 7.2$ Hz, 2H), 7.10 (t, $J = 7.7$ Hz, 2H), 6.98 (t, $J = 7.3$ Hz, 1H), 6.81 (s, 2H), 3.73 (s, 6H), 2.85 – 2.33 (m, 2H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 142.68, 131.18, 127.95, 123.37, 120.82, 36.15.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -24.38 (t, $J = 100.4$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.38. $^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.36 (d, $J = 7.2$ Hz, 2H), 7.10 (t, $J = 7.7$ Hz, 2H), 6.98 (t, $J = 7.3$ Hz, 1H), 6.81 (s, 2H), 3.73 (s, 6H), 2.62 (s, 2H).

Characterization agrees with previous reports for this compound.¹⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(2-oxochroman-3-yl)dihydroborate(**5a**): White solid (450 mg, 88% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.14 (dd, $J = 16.3, 7.7$ Hz, 2H), 7.01 (t, $J = 6.9$ Hz, 1H), 6.91 (d, $J = 7.9$ Hz, 1H), 6.78 (s, 2H), 3.69 (s, 6H), 3.26 (d, $J = 14.0$ Hz, 1H), 2.74 (d, $J = 15.2$ Hz, 1H), 2.39 (s, 1H), 1.56 – 1.19 (m, 2H).

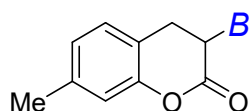
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 178.26, 152.38, 128.46, 126.82, 124.08, 123.32, 120.67, 115.44, 35.99, 30.64.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -25.86 (t, $J = 92.1$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.09.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.14 (dd, $J = 16.3, 7.8$ Hz, 2H), 7.01 (t, $J = 7.4$ Hz, 1H), 6.90 (d, $J = 8.0$ Hz, 1H), 6.78 (s, 2H), 3.69 (s, 6H), 3.26 (dd, $J = 15.2, 6.1$ Hz, 1H), 2.75 (d, $J = 15.2$ Hz, 1H), 2.39 (dd, $J = 12.7, 6.1$ Hz, 1H), 1.48 (d, $J = 37.7$ Hz, 2H).

Characterization agrees with previous reports for this compound.¹



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-methyl-2-oxochroman-3-yl)dihydroborate (**5b**): White solid (480 mg, 89% yield).

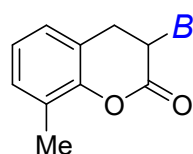
$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 6.96 (s, 1H), 6.92 (d, $J = 8.2$ Hz, 1H), 6.79 (d, $J = 8.3$ Hz, 1H), 6.78 (s, 2H), 3.68 (s, 6H), 3.22 (d, $J = 12.0$ Hz, 1H), 2.69 (d, $J = 15.2$ Hz, 1H), 2.35 (s, 1H), 2.29 (s, 3H), 1.58 – 1.18 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 178.49, 150.27, 132.62, 129.03, 127.25, 123.70, 120.62, 115.14, 35.99, 30.67, 20.80.

^{11}B NMR (193 MHz, CDCl_3) -26.08 (t, $J = 91.2$ Hz). $^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.08.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.96 (s, 1H), 6.92 (d, $J = 8.1$ Hz, 1H), 6.80 (d, $J = 4.8$ Hz, 1H), 6.78 (s, 2H), 3.68 (s, 6H), 3.22 (dd, $J = 15.2, 6.2$ Hz, 1H), 2.69 (d, $J = 15.2$ Hz, 1H), 2.35 (dd, $J = 13.0, 6.0$ Hz, 1H), 2.29 (s, 3H), 1.48 (d, $J = 41.6$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{19}\text{BN}_2\text{NaO}_2$ 293.1437; found: 293.1434.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(8-methyl-2-oxochroman-3-yl)dihydroborate (5c): White solid (415 mg, 77% yield).

^1H NMR (600 MHz, CDCl_3) δ 6.98 (d, $J = 7.3$ Hz, 2H), 6.90 (t, $J = 7.4$ Hz, 1H), 6.77 (s, 2H), 3.68 (s, 6H), 3.25 (d, $J = 14.2$ Hz, 1H), 2.71 (d, $J = 15.2$ Hz, 1H), 2.39 (s, 1H), 2.25 (s, 3H), 1.59 – 1.19 (m, 2H).

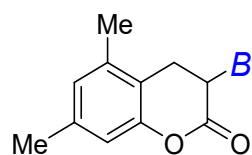
^{13}C NMR (151 MHz, CDCl_3) δ 178.48, 150.51, 128.37, 125.98, 124.38, 123.70, 122.79, 120.63, 35.95, 30.83, 15.82.

^{11}B NMR (193 MHz, CDCl_3) δ -26.17 (t, $J = 90.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.17.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.97 (s, 2H), 6.89 (dd, $J = 13.0, 6.2$ Hz, 1H), 6.76 (d, $J = 2.6$ Hz, 2H), 3.66 (dd, $J = 7.2, 3.2$ Hz, 6H), 3.23 (d, $J = 15.1$ Hz, 1H), 2.69 (d, $J = 15.1$ Hz, 1H), 2.38 (s, 1H), 2.24 (s, 3H), 1.47 (d, $J = 48.9$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{19}\text{BN}_2\text{NaO}_2$ 293.1437; found: 293.1431.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(5,7-dimethyl-2-oxochroman-3-yl)dihydroborate (5d): White solid (375 mg, 66% yield).

^1H NMR (600 MHz, CDCl_3) δ 6.79 (s, 2H), 6.71 (s, 1H), 6.58 (s, 1H), 3.68 (s, 6H), 2.97 (d, $J = 11.4$ Hz, 1H), 2.78 (d, $J = 15.5$ Hz, 1H), 2.37 (s, 1H), 2.26 (s, 3H), 2.23 (s, 3H), 1.62 – 1.26 (m, 2H).

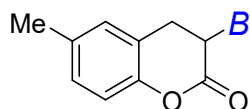
^{13}C NMR (151 MHz, CDCl_3) δ 178.55, 152.32, 136.12, 136.10, 125.76, 120.60, 119.29, 113.87, 35.98, 27.69, 21.02, 19.17.

^{11}B NMR (193 MHz, CDCl_3) δ -25.81 (t, $J = 91.5$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.81.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.79 (s, 2H), 6.71 (s, 1H), 6.57 (s, 1H), 3.68 (s, 6H), 2.97 (dd, $J = 15.4, 6.1$ Hz, 1H), 2.78 (d, $J = 15.5$ Hz, 1H), 2.37 (d, $J = 6.3$ Hz, 1H), 2.26 (s, 3H), 2.23 (s, 3H), 1.50 (s, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{16}\text{H}_{21}\text{BN}_2\text{NaO}_2$ 307.1594; found: 307.1593.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(6-methyl-2-oxochroman-3-yl)dihydroborate (**5e**): White solid (470 mg, 87% yield).

^1H NMR (600 MHz, CDCl_3) δ 6.96 (s, 1H), 6.92 (d, $J = 8.2$ Hz, 1H), 6.79 (d, $J = 8.3$ Hz, 1H), 6.78 (s, 2H), 3.68 (s, 6H), 3.22 (d, $J = 12.0$ Hz, 1H), 2.69 (d, $J = 15.2$ Hz, 1H), 2.35 (s, 1H), 2.29 (s, 3H), 1.65 – 1.13 (m, 2H).

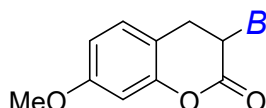
^{13}C NMR (151 MHz, CDCl_3) δ 178.49, 150.27, 132.62, 129.03, 127.25, 123.70, 120.62, 115.14, 35.99, 30.67, 20.80.

^{11}B NMR (193 MHz, CDCl_3) δ -25.85 (d, $J = 91.2$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.32.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.96 (s, 1H), 6.92 (d, $J = 8.1$ Hz, 1H), 6.80 (d, $J = 4.5$ Hz, 1H), 6.78 (s, 2H), 3.68 (s, 6H), 3.22 (dd, $J = 15.2, 6.2$ Hz, 1H), 2.69 (d, $J = 15.2$ Hz, 1H), 2.35 (dd, $J = 13.0, 6.0$ Hz, 1H), 2.29 (s, 3H), 1.48 (d, $J = 41.6$ Hz, 2H).

Characterization agrees with previous reports for this compound.⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-methoxy-2-oxochroman-3-yl)dihydroborate (**5f**): White solid (516 mg, 90% yield).

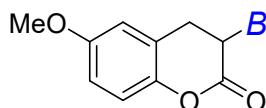
^1H NMR (600 MHz, CDCl_3) δ 7.04 (d, $J = 8.1$ Hz, 1H), 6.79 (s, 2H), 6.58 (d, $J = 8.1$ Hz, 1H), 6.49 (s, 1H), 3.77 (s, 3H), 3.69 (s, 6H), 3.18 (d, $J = 11.0$ Hz, 1H), 2.67 (d, $J = 14.7$ Hz, 1H), 2.35 (s, 1H), 1.76 – 1.24 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 178.17, 158.65, 153.01, 128.76, 120.62, 116.06, 108.84, 101.59, 55.40, 36.02, 29.89.

^{11}B NMR (193 MHz, CDCl_3) δ -26.00 (t, $J = 91.1$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.00. $^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.04 (d, $J = 8.0$ Hz, 1H), 6.58 (d, $J = 8.0$ Hz, 1H), 6.49 (s, 1H), 3.77 (s, 3H), 3.69 (s, 6H), 3.18 (d, $J = 14.8$ Hz, 1H), 2.67 (d, $J = 14.8$ Hz, 1H), 2.35 (s, 1H), 1.49 (d, $J = 28.8$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{19}\text{BN}_2\text{NaO}_3$ 309.1386; found: 309.1377.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(6-methoxy-2-oxochroman-3-yl)dihydroborate (**5g**): White solid (516 mg, 90% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 6.83 (d, $J = 8.7$ Hz, 1H), 6.79 (s, 2H), 6.70 (s, 1H), 6.67 (d, $J = 8.7$ Hz, 1H), 3.77 (s, 3H), 3.68 (s, 6H), 3.23 (d, $J = 11.7$ Hz, 1H), 2.69 (d, $J = 15.2$ Hz, 1H), 2.34 (s, 1H), 1.70 – 1.24 (m, 2H).

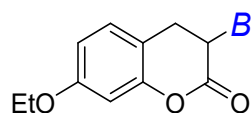
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 178.45, 155.46, 146.42, 125.03, 120.64, 116.01, 113.53, 111.95, 55.57, 35.99, 31.02.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -26.12 (t, $J = 91.8$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.13.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.83 (d, $J = 8.6$ Hz, 1H), 6.79 (s, 2H), 6.69 (s, 1H), 6.67 (d, $J = 8.6$ Hz, 1H), 3.77 (s, 3H), 3.69 (s, 6H), 3.23 (d, $J = 15.2$ Hz, 1H), 2.69 (d, $J = 15.2$ Hz, 1H), 2.34 (s, 1H), 1.47 (d, $J = 37.9$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{19}\text{BN}_2\text{NaO}_3$ 309.1386; found: 309.1381.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-ethoxy-2-oxochroman-3-yl)dihydroborate (**5h**): White solid (482 mg, 80% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.02 (d, $J = 8.2$ Hz, 1H), 6.78 (s, 2H), 6.56 (dd, $J = 8.2$, 2.4 Hz, 1H), 6.48 (d, $J = 2.3$ Hz, 1H), 3.99 (q, $J = 7.0$ Hz, 2H), 3.68 (s, 6H), 3.17 (d, $J = 10.1$ Hz, 1H), 2.65 (d, $J = 14.9$ Hz, 1H), 2.36 (s, 1H), 1.85 – 1.40 (m, 2H), 1.39 (t, $J = 7.0$ Hz, 3H).

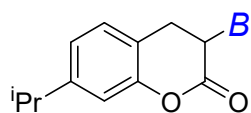
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 178.23, 158.00, 152.97, 128.72, 120.62, 115.92, 109.43, 102.12, 63.57, 36.01, 29.88, 14.84.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -26.01 (t, $J = 90.9$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.01.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.02 (d, $J = 8.2$ Hz, 1H), 6.78 (s, 2H), 6.56 (d, $J = 8.2$ Hz, 1H), 6.48 (s, 1H), 3.99 (q, $J = 6.9$ Hz, 2H), 3.68 (s, 6H), 3.17 (dd, $J = 14.9$, 6.1 Hz, 1H), 2.65 (d, $J = 14.9$ Hz, 1H), 2.35 (dd, $J = 12.3$, 6.0 Hz, 1H), 1.49 (dd, $J = 21.2$, 9.8 Hz, 2H), 1.39 (t, $J = 7.0$ Hz, 3H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{16}\text{H}_{21}\text{BN}_2\text{NaO}_3$ 323.1543; found: 323.1536.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-isopropyl-2-oxochroman-3-yl)dihydroborate (**5i**): White solid (482 mg, 80% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.05 (d, $J = 7.6$ Hz, 1H), 6.87 (d, $J = 7.6$ Hz, 1H), 6.76 (s, 2H), 6.75 (s, 1H), 3.68 (s, 6H), 3.21 (d, $J = 11.9$ Hz, 1H), 2.85 (dt, $J = 13.8$, 6.9 Hz, 1H), 2.70 (d, $J = 15.2$ Hz, 1H), 2.38 (s, 1H), 1.61 (s, 2H), 1.23 (s, 3H), 1.22 (s, 3H).

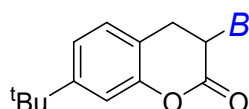
^{13}C NMR (151 MHz, CDCl_3) δ 178.48, 152.28, 147.93, 128.09, 121.36, 121.11, 120.58, 113.38, 36.03, 33.69, 30.37, 24.00, 23.96. ^{11}B NMR (193 MHz, CDCl_3) δ -25.94 (t, $J = 92.0$ Hz).

^{11}B NMR (193 MHz, CDCl_3) δ -25.94 (t, $J = 91.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.94.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.05 (d, $J = 7.6$ Hz, 1H), 6.87 (d, $J = 7.6$ Hz, 1H), 6.76 (s, 2H), 6.75 (s, 1H), 3.68 (s, 6H), 3.21 (dd, $J = 15.1, 6.2$ Hz, 1H), 2.85 (dt, $J = 13.8, 6.9$ Hz, 1H), 2.70 (d, $J = 15.2$ Hz, 1H), 2.38 (dd, $J = 12.2, 6.0$ Hz, 1H), 1.51 (d, $J = 32.4$ Hz, 2H), 1.23 (s, 3H), 1.22 (s, 3H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{17}\text{H}_{23}\text{BN}_2\text{NaO}_2$ 321.1750; found: 321.1741.



(7-(tert-butyl)-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**5j**): White solid (312 mg, 63% yield).

^1H NMR (600 MHz, CDCl_3) δ 7.03 (d, $J = 19.1$ Hz, 2H), 6.90 (s, 1H), 6.75 (s, 2H), 3.67 (s, 6H), 3.20 (d, $J = 7.8$ Hz, 1H), 2.69 (d, $J = 14.4$ Hz, 1H), 2.38 (s, 1H), 1.64 (dd, $J = 83.1, 34.0$ Hz, 2H), 1.28 (s, 9H).

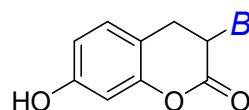
^{13}C NMR (151 MHz, CDCl_3) δ 178.48, 152.09, 150.34, 127.79, 120.74, 120.61, 120.19, 112.56, 36.02, 34.49, 31.36, 30.28.

^{11}B NMR (193 MHz, CDCl_3) δ -25.94 (t, $J = 92.6$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.92.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.03 (d, $J = 19.2$ Hz, 2H), 6.89 (s, 1H), 6.75 (s, 2H), 3.67 (s, 6H), 3.20 (d, $J = 9.9$ Hz, 1H), 2.69 (d, $J = 14.9$ Hz, 1H), 2.38 (s, 1H), 1.50 (d, $J = 29.5$ Hz, 2H), 1.28 (s, 9H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{18}\text{H}_{25}\text{BN}_2\text{NaO}_2$ 335.1907; found: 335.1897.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-hydroxy-2-oxochroman-3-yl)dihydroborate (**5k**): White solid (480 mg, 88% yield).

^1H NMR (600 MHz, DMSO) δ 7.27 (s, 2H), 6.84 (d, $J = 8.1$ Hz, 1H), 6.36 (dd, $J = 8.1, 2.3$ Hz, 1H), 6.21 (d, $J = 2.3$ Hz, 1H), 3.83 (s, 1H), 3.54 (s, 6H), 2.95 (dd, $J = 14.8, 5.7$ Hz, 1H), 2.32 (d, $J = 14.8$ Hz, 1H), 2.15 (d, $J = 5.6$ Hz, 1H), 1.49 – 1.09 (m, 2H).

^{13}C NMR (151 MHz, DMSO) δ 176.87, 153.24, 128.91, 123.91, 121.72, 113.27, 110.82, 102.85, 35.73, 29.54.

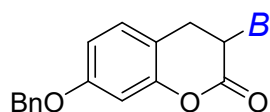
^{11}B NMR (193 MHz, DMSO) δ -25.54 (t, $J = 89.9$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, DMSO) δ -25.82.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, DMSO) δ 7.27 (s, 2H), 6.84 (d, $J = 8.1$ Hz, 1H), 6.36 (dd, $J = 8.1, 2.3$ Hz, 1H), 6.21 (d, $J = 2.3$ Hz, 1H), 3.84 (s, 1H), 3.55 (s, 6H), 2.97 – 2.92 (m,

1H), 2.32 (d, $J = 14.8$ Hz, 1H), 2.15 (dd, $J = 11.5, 5.7$ Hz, 1H), 1.35 (s, 2H).

HRMS (ESI-TOF) m/z : $[M + Na]^+$ cacl. for $C_{14}H_{17}BN_2NaO_3$ 295.1230; found: 295.1220.



(7-(benzyloxy)-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**5l**): White solid (631 mg, 87% yield).

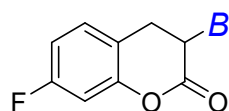
1H NMR (600 MHz, $CDCl_3$) δ 7.43 (d, $J = 7.5$ Hz, 2H), 7.38 (t, $J = 7.5$ Hz, 2H), 7.32 (t, $J = 7.3$ Hz, 1H), 7.04 (d, $J = 8.2$ Hz, 1H), 6.75 (s, 2H), 6.66 (dd, $J = 8.3, 2.5$ Hz, 1H), 6.56 (d, $J = 2.5$ Hz, 1H), 5.03 (s, 2H), 3.67 (s, 6H), 3.18 (d, $J = 16.6$ Hz, 1H), 2.67 (d, $J = 15.0$ Hz, 1H), 2.37 (d, $J = 4.3$ Hz, 1H), 1.60 – 1.17 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.21, 157.79, 152.96, 137.04, 128.79, 128.57, 127.95, 127.57, 120.61, 116.39, 109.89, 102.55, 70.15, 36.01, 29.90.

^{11}B NMR (193 MHz, $CDCl_3$) δ -26.02 (t, $J = 89.8$ Hz).

$^{11}B\{^1H\}$ NMR (193 MHz, $CDCl_3$) δ -26.01.

$^1H\{^{11}B\}$ NMR (600 MHz, $CDCl_3$) δ 7.43 (d, $J = 7.3$ Hz, 2H), 7.38 (t, $J = 7.5$ Hz, 2H), 7.32 (t, $J = 7.3$ Hz, 1H), 7.04 (d, $J = 8.2$ Hz, 1H), 6.75 (s, 2H), 6.66 (dd, $J = 8.2, 2.4$ Hz, 1H), 6.55 (d, $J = 2.4$ Hz, 1H), 5.03 (s, 2H), 3.67 (s, 6H), 3.18 (dd, $J = 14.9, 6.1$ Hz, 1H), 2.67 (d, $J = 15.0$ Hz, 1H), 2.37 (dd, $J = 12.4, 6.0$ Hz, 1H), 1.49 (d, $J = 34.2$ Hz, 2H). **HRMS (ESI-TOF)** m/z : $[M + Na]^+$ cacl. for $C_{21}H_{23}BN_2NaO_3$ 385.1699; found: 385.1687.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-fluoro-2-oxochroman-3-yl) dihydroborate (**5m**): White solid (438 mg, 80% yield).

1H NMR (600 MHz, $CDCl_3$) δ 7.09 (t, $J = 6$ Hz, 1H), 6.80 (s, 2H), 6.73 (t, $J = 8.4$ Hz, 1H), 6.65 (d, $J = 9.5$ Hz, 1H), 3.69 (s, 6H), 3.20 (d, $J = 11.7$ Hz, 1H), 2.72 (d, $J = 15.0$ Hz, 1H), 2.36 (s, 1H), 1.73 – 1.25 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 177.50, 161.50 (d, $J = 11.7$ Hz), 152.96 (d, $J = 12.0$ Hz), 129.09 (d, $J = 9.1$ Hz), 120.73, 119.77 (d, $J = 3.0$ Hz), 109.90 (d, $J = 21.1$ Hz), 103.24 (d, $J = 25.7$ Hz), 35.99, 30.01.

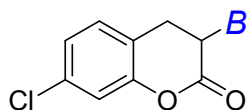
^{11}B NMR (193 MHz, $CDCl_3$) δ -26.08 (t, $J = 91.4$ Hz).

$^{11}B\{^1H\}$ NMR (193 MHz, $CDCl_3$) δ -26.08.

$^1H\{^{11}B\}$ NMR (600 MHz, $CDCl_3$) δ 7.09 (t, $J = 6$ Hz, 1H), 6.80 (s, 2H), 6.72 (td, $J = 8.4, 2.4$ Hz, 1H), 6.65 (dd, $J = 9.5, 2.4$ Hz, 1H), 3.69 (s, 6H), 3.20 (dd, $J = 15.0, 6.0$ Hz, 1H), 2.72 (d, $J = 15.1$ Hz, 1H), 2.36 (dd, $J = 12.8, 5.9$ Hz, 1H), 1.47 (d, $J = 43.7$ Hz, 2H).

^{19}F NMR (565 MHz, $CDCl_3$) δ -115.82.

HRMS (ESI-TOF) m/z : $[M + Na]^+$ cacl. for $C_{14}H_{16}BFN_2NaO_2$ 297.1187; found: 297.1185.



(7-chloro-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**5n**): White solid (516 mg, 89% yield).

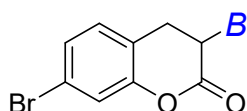
$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.08 (d, $J = 8.0$ Hz, 1H), 6.99 (dd, $J = 8.0, 2.0$ Hz, 1H), 6.92 (d, $J = 2.0$ Hz, 1H), 6.81 (s, 2H), 3.69 (s, 6H), 3.20 (d, $J = 13.4$ Hz, 1H), 2.72 (d, $J = 15.3$ Hz, 1H), 2.35 (s, 1H), 1.74 – 1.35 (m, 2H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 177.46, 152.87, 131.78, 129.36, 123.35, 122.74, 120.72, 115.83, 36.02, 30.21. $^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -25.87 (t, $J = 91.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.10.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.08 (d, $J = 8.0$ Hz, 1H), 6.99 (dd, $J = 8.0, 2.0$ Hz, 1H), 6.92 (d, $J = 1.9$ Hz, 1H), 6.81 (s, 2H), 3.69 (s, 6H), 3.20 (dd, $J = 15.3, 6.2$ Hz, 1H), 2.72 (d, $J = 15.3$ Hz, 1H), 2.35 (dd, $J = 13.4, 5.6$ Hz, 1H), 1.46 (d, $J = 45.9$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{14}\text{H}_{16}\text{BClN}_2\text{NaO}_2$ 313.0891; found: 313.0883.



(7-bromo-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**5o**): White solid (560 mg, 89% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.13 (d, $J = 7.9$ Hz, 1H), 7.06 (s, 1H), 7.02 (d, $J = 8.0$ Hz, 1H), 6.81 (s, 2H), 3.69 (s, 6H), 3.17 (d, $J = 11.4$ Hz, 1H), 2.71 (d, $J = 15.2$ Hz, 1H), 2.35 (s, 1H), 1.53 (dd, $J = 146.2, 54.4$ Hz, 2H).

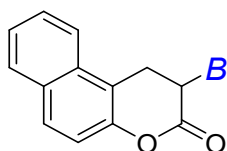
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 177.41, 153.02, 129.75, 126.25, 123.27, 120.73, 119.35, 118.66, 36.02, 30.27.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -26.10 (t, $J = 91.8$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.10.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.14 (d, $J = 7.9$ Hz, 1H), 7.07 (s, 1H), 7.03 (d, $J = 7.9$ Hz, 1H), 6.81 (s, 2H), 3.69 (s, 6H), 3.18 (dd, $J = 15.3, 5.9$ Hz, 1H), 2.71 (d, $J = 15.3$ Hz, 1H), 2.36 (d, $J = 5.8$ Hz, 1H), 1.46 (d, $J = 43.2$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{14}\text{H}_{16}\text{BBrN}_2\text{NaO}_2$ 357.0386; found: 357.0385.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(3-oxo-2,3-dihydro-1H-benzo[f]chromen-2-yl)dihydroborate (**5p**): White solid (560 mg, 89% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.91 (d, $J = 8.4$ Hz, 1H), 7.80 (d, $J = 8.1$ Hz, 1H), 7.66 (d, $J = 8.8$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 1H), 7.39 (t, $J = 7.4$ Hz, 1H), 7.13 (d, $J = 8.8$ Hz, 1H), 3.63 (s, 6H), 3.35 (s, 2H), 2.57 (s, 1H), 1.58 (dd, $J = 172.2, 75.8$ Hz, 2H).

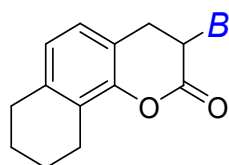
^{13}C NMR (151 MHz, CDCl_3) δ 178.05, 149.50, 132.22, 130.60, 128.46, 127.49, 126.37, 124.21, 123.27, 120.65, 117.06, 116.41, 35.98, 27.08.

^{11}B NMR (193 MHz, CDCl_3) δ -25.46 (t, $J = 91.6$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.45.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.91 (d, $J = 8.4$ Hz, 1H), 7.80 (d, $J = 8.1$ Hz, 1H), 7.66 (d, $J = 8.8$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 1H), 7.39 (t, $J = 7.4$ Hz, 1H), 7.13 (d, $J = 8.8$ Hz, 1H), 6.73 (s, 2H), 3.63 (s, 6H), 3.35 (s, 2H), 2.57 (s, 1H), 1.57 (d, $J = 30.5$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{18}\text{H}_{19}\text{BN}_2\text{NaO}_2$ 329.1437; found: 329.1431.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(2-oxo-3,4,7,8,9,10-hexahydro-2H-benzo [h]chromen-3-yl)dihydroborate (**5q**): White solid (434 mg, 70% yield).

^1H NMR (600 MHz, CDCl_3) δ 6.88 (d, $J = 7.6$ Hz, 1H), 6.76 (s, 2H), 6.74 (d, $J = 7.6$ Hz, 1H), 3.68 (s, 6H), 3.20 (d, $J = 11.8$ Hz, 1H), 2.78 (dd, $J = 17.3, 6.0$ Hz, 1H), 2.73 (s, 2H), 2.67 (d, $J = 15.1$ Hz, 1H), 2.58 (dd, $J = 18.1, 5.7$ Hz, 1H), 2.35 (s, 1H), 1.79 – 1.72 (m, 4H), 1.65 – 1.22 (m, 2H).

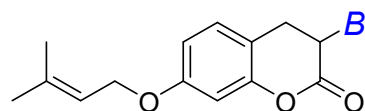
^{13}C NMR (151 MHz, CDCl_3) δ 178.68, 149.92, 136.06, 125.03, 123.92, 123.64, 120.57, 120.33, 36.01, 30.67, 29.41, 23.05, 22.87, 22.72.

^{11}B NMR (193 MHz, CDCl_3) δ -26.05 (t, $J = 91.1$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.04.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.88 (d, $J = 7.6$ Hz, 1H), 6.76 (s, 2H), 6.74 (d, $J = 7.6$ Hz, 1H), 3.68 (s, 6H), 3.20 (dd, $J = 15.1, 6.1$ Hz, 1H), 2.81 – 2.75 (m, 1H), 2.73 (s, 2H), 2.67 (d, $J = 15.1$ Hz, 1H), 2.58 (d, $J = 17.5$ Hz, 1H), 2.36 (dd, $J = 12.1, 6.0$ Hz, 1H), 1.80 – 1.72 (m, 4H), 1.49 (d, $J = 42.6$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{18}\text{H}_{23}\text{BN}_2\text{NaO}_2$ 333.1750; found: 333.1754.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-((3-methylbut-2-en-1-yl)oxy)-2-oxochroman-3-yl)dihydroborate (**5r**): White solid (630 mg, 93% yield).

^1H NMR (600 MHz, CDCl_3) δ 7.03 (d, $J = 8.2$ Hz, 1H), 6.78 (s, 2H), 6.59 (dd, $J = 8.3, 2.5$ Hz, 1H), 6.51 (d, $J = 2.4$ Hz, 1H), 5.48 (t, $J = 6.7$ Hz, 1H), 4.47 (d, $J = 6.7$ Hz, 2H), 3.69 (s, 6H), 3.17 (d, $J = 13.7$ Hz, 1H), 2.66 (d, $J = 14.9$ Hz, 1H), 2.36 (s, 1H), 1.79 (s, 3H), 1.74 (s, 3H), 1.68 – 1.26 (m, 2H).

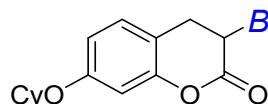
^{13}C NMR (151 MHz, CDCl_3) δ 178.28, 157.93, 152.95, 138.13, 128.70, 120.62, 119.70, 116.01, 109.62, 102.37, 64.94, 36.03, 29.89, 25.85, 18.22.

¹¹B NMR (193 MHz, CDCl₃) δ -26.01 (t, *J* = 90.7 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -26.00.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.03 (d, *J* = 8.2 Hz, 1H), 6.79 (s, 2H), 6.59 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.51 (d, *J* = 2.2 Hz, 1H), 5.48 (t, *J* = 6.7 Hz, 1H), 4.47 (d, *J* = 6.7 Hz, 2H), 3.69 (s, 6H), 3.18 (dd, *J* = 14.9, 6.0 Hz, 1H), 2.66 (d, *J* = 14.9 Hz, 1H), 2.36 (dd, *J* = 12.1, 5.9 Hz, 1H), 1.79 (s, 3H), 1.74 (s, 3H), 1.50 (d, *J* = 29.4 Hz, 2H).

HRMS (ESI-TOF) m/z: [M + Na]⁺ cacl. for C₁₉H₂₅BN₂NaO₃ 363.1856; found: 363.1854.



(7-(cyclohexyloxy)-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**5s**): White solid (453 mg, 64% yield).

¹H NMR (600 MHz, CDCl₃) δ 7.01 (d, *J* = 8.2 Hz, 1H), 6.78 (s, 2H), 6.57 (dd, *J* = 8.2, 2.4 Hz, 1H), 6.49 (d, *J* = 2.4 Hz, 1H), 4.16 (dt, *J* = 12.8, 6.3 Hz, 1H), 3.69 (s, 6H), 3.17 (d, *J* = 19.4 Hz, 1H), 2.66 (d, *J* = 14.9 Hz, 1H), 2.36 (d, *J* = 4.4 Hz, 1H), 1.98 (d, *J* = 10.3 Hz, 2H), 1.79 (dd, *J* = 8.2, 3.9 Hz, 2H), 1.58 (s, 2H), 1.49 (dd, *J* = 19.4, 12.9 Hz, 2H), 1.39 – 1.32 (m, 2H), 1.32 – 1.27 (m, 2H).

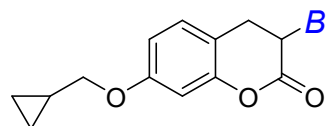
¹³C NMR (151 MHz, CDCl₃) δ 178.30, 156.80, 152.96, 128.68, 120.61, 115.87, 111.11, 103.59, 75.74, 36.03, 31.89, 31.81, 29.91, 25.64, 23.85, 23.82.

¹¹B NMR (193 MHz, CDCl₃) δ -25.98 (t, *J* = 92.4 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -25.98.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.01 (d, *J* = 8.2 Hz, 1H), 6.78 (s, 2H), 6.57 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.48 (d, *J* = 2.2 Hz, 1H), 4.16 (dt, *J* = 12.6, 6.1 Hz, 1H), 3.69 (s, 6H), 3.17 (dd, *J* = 14.9, 6.1 Hz, 1H), 2.66 (d, *J* = 15.0 Hz, 1H), 2.36 (dd, *J* = 12.3, 6.0 Hz, 1H), 1.98 (d, *J* = 10.1 Hz, 2H), 1.83 – 1.76 (m, 2H), 1.58 (d, *J* = 3.7 Hz, 2H), 1.51 – 1.48 (m, 2H), 1.39 – 1.33 (m, 2H), 1.33 – 1.26 (m, 2H).

HRMS (ESI-TOF) m/z: [M + Na]⁺ cacl. for C₂₀H₂₇BN₂NaO₃ 377.2012; found: 377.2013.



(7-(cyclopropylmethoxy)-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**5t**): White solid (581 mg, 89% yield).

¹H NMR (600 MHz, CDCl₃) δ 7.02 (d, *J* = 8.2 Hz, 1H), 6.79 (s, 2H), 6.58 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.49 (d, *J* = 1.6 Hz, 1H), 3.76 (d, *J* = 6.9 Hz, 2H), 3.69 (s, 6H), 3.17 (d, *J* = 10.2 Hz, 1H), 2.66 (d, *J* = 14.9 Hz, 1H), 2.35 (s, 1H), 1.74 – 1.34 (m, 2H), 1.28 – 1.23 (m, 1H), 0.63 (q, *J* = 5.3 Hz, 2H), 0.34 (q, *J* = 5.0 Hz, 2H).

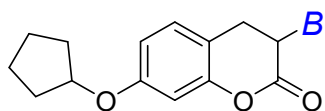
¹³C NMR (151 MHz, CDCl₃) δ 178.28, 158.08, 152.96, 128.73, 120.62, 116.03, 109.52, 102.30, 72.95, 36.03, 29.88, 10.28, 3.19.

¹¹B NMR (193 MHz, CDCl₃) δ -26.02 (t, *J* = 89.1 Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.02.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.02 (d, $J = 8.2$ Hz, 1H), 6.78 (s, 2H), 6.58 (d, $J = 8.2$ Hz, 1H), 6.49 (s, 1H), 3.76 (d, $J = 6.9$ Hz, 2H), 3.69 (s, 6H), 3.17 (dd, $J = 14.9, 6.0$ Hz, 1H), 2.66 (d, $J = 14.9$ Hz, 1H), 2.35 (d, $J = 6.0$ Hz, 1H), 1.48 (d, $J = 28.7$ Hz, 2H), 1.28 – 1.22 (m, 1H), 0.63 (d, $J = 7.9$ Hz, 2H), 0.34 (d, $J = 4.8$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{18}\text{H}_{23}\text{BN}_2\text{NaO}_3$ 349.1699; found: 349.1696.



(7-(cyclopentyloxy)-2-oxochroman-3-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**5u**): White solid (585 mg, 86% yield).

^1H NMR (600 MHz, CDCl_3) δ 7.01 (d, $J = 8.2$ Hz, 1H), 6.78 (s, 2H), 6.54 (dd, $J = 8.2, 2.4$ Hz, 1H), 6.46 (d, $J = 2.2$ Hz, 1H), 4.72 – 4.68 (m, 1H), 3.69 (s, 6H), 3.17 (d, $J = 11.6$ Hz, 1H), 2.65 (d, $J = 14.9$ Hz, 1H), 2.36 (s, 1H), 1.90 – 1.77 (m, 6H), 1.63 – 1.58 (m, 2H), 1.56 – 1.16 (m, 2H).

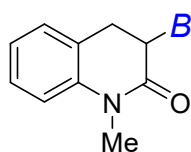
^{13}C NMR (151 MHz, CDCl_3) δ 178.34, 157.18, 152.91, 128.65, 120.61, 115.56, 110.62, 103.07, 79.42, 36.03, 32.86, 32.81, 29.87, 24.06.

^{11}B NMR (193 MHz, CDCl_3) δ -25.98 (t, $J = 90.5$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.98.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.00 (d, $J = 8.2$ Hz, 1H), 6.78 (s, 2H), 6.53 (d, $J = 8.2$ Hz, 1H), 6.45 (s, 1H), 4.69 (s, 1H), 3.68 (s, 6H), 3.17 (dd, $J = 14.8, 5.4$ Hz, 1H), 2.65 (d, $J = 14.9$ Hz, 1H), 2.36 (d, $J = 5.2$ Hz, 1H), 1.91 – 1.76 (m, 6H), 1.60 (s, 2H), 1.50 (d, $J = 26.0$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{19}\text{H}_{25}\text{BN}_2\text{NaO}_3$ 363.1856; found: 363.1845.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-methyl-2-oxo-1,2,3,4-tetrahydroquinolin-3-yl)dihydroborate (**5v**): White solid (585 mg, 86% yield).

^1H NMR (600 MHz, CDCl_3) δ 7.15 (t, $J = 7.9$ Hz, 1H), 7.12 (d, $J = 7.2$ Hz, 1H), 6.93 (t, $J = 7.3$ Hz, 1H), 6.83 (d, $J = 8.0$ Hz, 1H), 6.74 (s, 2H), 3.61 (s, 6H), 3.24 (s, 3H), 3.21 (s, 1H), 2.63 (d, $J = 14.7$ Hz, 1H), 2.25 (s, 1H), 1.63 – 1.12 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 180.15, 141.19, 128.00, 126.09, 121.69, 120.27, 112.93, 35.82, 32.51, 29.10.

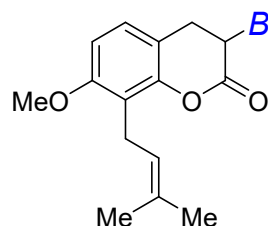
^{11}B NMR (193 MHz, CDCl_3) δ -26.83 (t, $J = 89.2$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.82.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.17 – 7.13 (m, 1H), 7.12 (d, $J = 6.4$ Hz, 1H), 6.92 (t, $J = 7.2$ Hz, 1H), 6.83 (d, $J = 8.0$ Hz, 1H), 6.74 (s, 2H), 3.61 (s, 6H), 3.24 (s, 3H), 3.21 (dd, $J = 14.8, 5.7$ Hz, 1H), 2.63 (d, $J = 14.8$ Hz, 1H), 2.25 (d, $J = 5.6$ Hz, 1H), 1.36

(d, $J = 45.1$ Hz, 2H).

Characterization agrees with previous reports for this compound.³



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-methoxy-8-(3-methylbut-2-en-1-yl)-2-oxochroman-3-yl)dihydroborate (**5w**): White solid (606 mg, 86% yield).

¹H NMR (600 MHz, CDCl₃) δ 6.93 (d, $J = 8.2$ Hz, 1H), 6.77 (s, 2H), 6.55 (d, $J = 8.2$ Hz, 1H), 5.18 (t, $J = 6.4$ Hz, 1H), 3.80 (s, 3H), 3.67 (s, 6H), 3.41 (dd, $J = 14.0, 7.4$ Hz, 1H), 3.32 (dd, $J = 14.0, 6.5$ Hz, 1H), 3.18 (d, $J = 9.9$ Hz, 1H), 2.67 (d, $J = 14.8$ Hz, 1H), 2.32 (s, 1H), 1.77 (s, 3H), 1.63 (s, 3H), 1.60 – 1.07 (m, 2H).

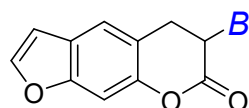
¹³C NMR (151 MHz, CDCl₃) δ 178.36, 156.31, 150.56, 131.03, 125.44, 122.93, 120.56, 116.99, 116.54, 105.44, 55.80, 35.95, 30.49, 25.81, 22.26, 17.91.

¹¹B NMR (193 MHz, CDCl₃) δ -26.13 (t, $J = 91.0$ Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -26.13.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 6.93 (d, $J = 8.2$ Hz, 1H), 6.77 (s, 2H), 6.55 (d, $J = 8.2$ Hz, 1H), 5.18 (t, $J = 6.9$ Hz, 1H), 3.80 (s, 3H), 3.67 (s, 6H), 3.41 (dd, $J = 14.0, 7.4$ Hz, 1H), 3.32 (dd, $J = 14.0, 6.5$ Hz, 1H), 3.19 (dd, $J = 14.7, 5.9$ Hz, 1H), 2.67 (d, $J = 14.7$ Hz, 1H), 2.32 (dd, $J = 11.7, 5.9$ Hz, 1H), 1.77 (s, 3H), 1.63 (s, 3H), 1.46 (d, $J = 41.6$ Hz, 2H).

HRMS (ESI-TOF) m/z : [M + Na]⁺ cacl. for C₂₀H₂₇BN₂NaO₃ 377.2012; found: 377.2000.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(7-oxo-6,7-dihydro-5H-furo[3,2-g]chromen-6-yl)dihydroborate (**5x**): White solid (400 mg, 68% yield).

¹H NMR (600 MHz, CDCl₃) δ 7.54 (d, $J = 2.2$ Hz, 1H), 7.34 (s, 1H), 7.09 (s, 1H), 6.78 (s, 2H), 6.68 (dd, $J = 2.1, 0.8$ Hz, 1H), 3.68 (s, 6H), 3.34 (d, $J = 12.0$ Hz, 1H), 2.84 (d, $J = 14.8$ Hz, 1H), 2.39 (s, 1H), 1.56 – 1.12 (m, 2H).

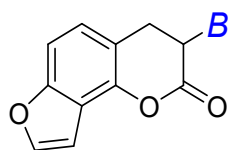
¹³C NMR (151 MHz, CDCl₃) δ 178.39, 153.75, 150.37, 144.74, 123.11, 120.63, 120.04, 119.61, 106.27, 98.70, 36.01, 30.87.

¹¹B NMR (193 MHz, CDCl₃) δ -26.25 (t, $J = 91.5$ Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -26.25.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.54 (d, $J = 2.1$ Hz, 1H), 7.34 (s, 1H), 7.09 (s, 1H), 6.78 (s, 2H), 6.68 (d, $J = 2.0$ Hz, 1H), 3.68 (s, 6H), 3.34 (dd, $J = 14.7, 5.8$ Hz, 1H), 2.84 (d, $J = 14.8$ Hz, 1H), 2.39 (dd, $J = 12.9, 5.5$ Hz, 1H), 1.47 (d, $J = 59.3$ Hz, 2H).

HRMS (ESI-TOF) m/z : [M + Na]⁺ cacl. for C₁₆H₁₇BN₂NaO₃ 319.1230; found: 319.1222.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(2-oxo-3,4-dihydro-2H-furo[2,3-h]chromen-3-yl)dihydroborate (**5y**): White solid (528 mg, 89% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.52 (d, $J = 2.1$ Hz, 1H), 7.17 (d, $J = 8.2$ Hz, 1H), 7.06 (d, $J = 8.2$ Hz, 1H), 6.86 (d, $J = 2.0$ Hz, 1H), 6.74 (s, 2H), 3.67 (s, 6H), 3.35 (d, $J = 11.6$ Hz, 1H), 2.80 (d, $J = 15.0$ Hz, 1H), 2.45 (s, 1H), 1.62 – 1.16 (m, 2H).

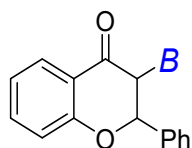
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 178.12, 154.97, 144.98, 144.40, 124.45, 120.62, 116.55, 116.46, 106.20, 103.34, 36.01, 30.35.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -25.90 (t, $J = 91.4$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.90.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.52 (d, $J = 2.0$ Hz, 1H), 7.16 (d, $J = 8.2$ Hz, 1H), 7.06 (d, $J = 8.2$ Hz, 1H), 6.86 (d, $J = 1.3$ Hz, 1H), 6.73 (s, 2H), 3.67 (s, 6H), 3.35 (dd, $J = 15.0, 6.2$ Hz, 1H), 2.80 (d, $J = 15.0$ Hz, 1H), 2.46 (dd, $J = 12.5, 6.0$ Hz, 1H), 1.52 (d, $J = 45.8$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{16}\text{H}_{17}\text{BN}_2\text{NaO}_3$ 319.1230; found: 319.1221.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(4-oxo-2-phenylchroman-3-yl)dihydroborate (**5z**): White solid (266 mg, 40% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.89 (dd, $J = 7.8, 1.6$ Hz, 1H), 7.52 (d, $J = 7.9$ Hz, 2H), 7.44 (ddd, $J = 8.7, 7.2, 1.7$ Hz, 1H), 7.36 (t, $J = 7.7$ Hz, 2H), 7.24 (d, $J = 7.3$ Hz, 1H), 7.10 (d, $J = 8.7$ Hz, 1H), 7.00 – 6.95 (m, 1H), 6.66 (s, 2H), 5.74 (s, 1H), 3.38 (s, 6H), 2.47 (s, 1H), 1.44 – 1.18 (m, 2H).

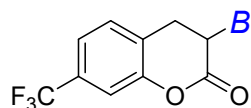
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 202.36, 161.35, 141.07, 134.36, 127.72, 126.81, 126.60, 125.57, 121.25, 120.42, 120.35, 117.60, 82.52, 35.70.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -30.05 (t, $J = 91.1$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -30.05.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.89 (dd, $J = 7.8, 1.6$ Hz, 1H), 7.52 (d, $J = 7.7$ Hz, 2H), 7.44 (ddd, $J = 8.7, 7.3, 1.7$ Hz, 1H), 7.36 (t, $J = 7.7$ Hz, 2H), 7.24 (d, $J = 7.4$ Hz, 1H), 7.10 (d, $J = 8.3$ Hz, 1H), 6.98 (t, $J = 7.8$ Hz, 1H), 6.66 (s, 2H), 5.74 (s, 1H), 3.38 (s, 6H), 2.47 (s, 1H), 1.35 (d, $J = 37.4$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{20}\text{H}_{21}\text{BN}_2\text{NaO}_2$ 355.1594; found: 355.1588.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(2-oxo-7-(trifluoromethyl)chroman-3-yl) dihydroborate (**5aa**): White solid (40 mg, 61% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.27 (d, $J = 0.8$ Hz, 2H), 7.14 (s, 1H), 6.81 (s, 2H), 3.69 (s, 6H), 3.28 (d, $J = 12.3$ Hz, 1H), 2.82 (d, $J = 15.6$ Hz, 1H), 2.38 (s, 1H), 1.62 – 1.27 (m, 2H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 177.20, 152.44, 129.31 (q, $J = 33.2$ Hz), 129.00, 128.38, 124.0 (q, $J = 271.8$ Hz), 120.76, 120.06 (q, $J = 3.5$ Hz), 112.55 (q, $J = 4.0$ Hz), 36.01, 30.68.

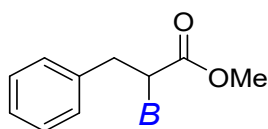
$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ 26.10 (t, $J = 92.6$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.10.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.27 (d, $J = 0.9$ Hz, 2H), 7.14 (s, 1H), 6.81 (s, 2H), 3.69 (s, 6H), 3.28 (dd, $J = 15.6, 6.2$ Hz, 1H), 2.82 (d, $J = 15.6$ Hz, 1H), 2.38 (dd, $J = 13.6, 5.3$ Hz, 1H), 1.52 – 1.37 (m, 2H).

$^{19}\text{F NMR}$ (565 MHz, CDCl_3) δ -62.30.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{15}\text{H}_{16}\text{BF}_3\text{N}_2\text{NaO}_2$ 347.1155; found: 347.1151.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-methoxy-1-oxo-3-phenylpropan-2-yl) dihydroborate (**6a**): Colorless liquid (41 mg, 75% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.20 – 7.17 (m, 4H), 7.08 (dt, $J = 5.8, 3.5$ Hz, 1H), 6.80 (s, 2H), 3.72 (s, 6H), 3.37 (s, 3H), 3.10 (dd, $J = 14.3, 10.2$ Hz, 1H), 2.71 (dd, $J = 14.3, 4.1$ Hz, 1H), 2.19 (s, 1H), 1.70 (dd, $J = 89.0, 71.9$ Hz, 2H).

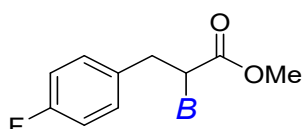
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 181.96, 144.72, 128.46, 127.90, 125.05, 120.43, 50.38, 39.09, 35.99.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -24.79 (t, $J = 89.9$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.02.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.19 (d, $J = 4.7$ Hz, 4H), 7.10 – 7.06 (m, 1H), 6.79 (s, 2H), 3.72 (s, 6H), 3.37 (s, 3H), 3.11 (dd, $J = 14.3, 10.2$ Hz, 1H), 2.71 (dd, $J = 14.3, 4.4$ Hz, 1H), 2.19 (td, $J = 10.4, 5.1$ Hz, 1H), 1.62 (d, $J = 17.5$ Hz, 2H).

Characterization agrees with previous reports for this compound.⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(3-(4-fluorophenyl)-1-methoxy-1-oxopropan-2-yl) dihydroborate (**6b**): Colorless liquid (48 mg, 83% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.13 (dd, $J = 8.6, 5.7$ Hz, 2H), 6.88 – 6.83 (m, 2H),

6.81 (s, 2H), 3.71 (s, 6H), 3.34 (s, 3H), 3.04 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.66 (dd, $J = 14.3, 4.1$ Hz, 1H), 2.13 (s, 1H), 1.60 (dd, $J = 172.9, 90.9$ Hz, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 181.78, 160.86 (d, $J = 241.6$ Hz), 140.36, 129.74 (d, $J = 7.6$ Hz), 120.46, 114.47 (d, $J = 21.1$ Hz) 50.36, 38.28, 35.97.

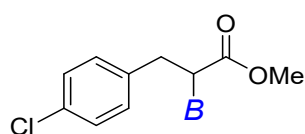
^{11}B NMR (193 MHz, CDCl_3) δ -24.87 (t, $J = 90.3$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.10.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.13 (dd, $J = 8.3, 5.8$ Hz, 2H), 6.86 (t, $J = 8.8$ Hz, 2H), 6.80 (s, 2H), 3.71 (s, 6H), 3.34 (s, 3H), 3.04 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.66 (dd, $J = 14.4, 4.3$ Hz, 1H), 2.13 (td, $J = 10.5, 5.1$ Hz, 1H), 1.60 (d, $J = 20.2$ Hz, 2H).

^{19}F NMR (565 MHz, CDCl_3) δ -119.14.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{20}\text{BFN}_2\text{NaO}_2$ 313.1500; found: 313.1497.



(3-(4-chlorophenyl)-1-methoxy-1-oxopropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**6c**): Colorless liquid (40 mg, 65% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

^1H NMR (600 MHz, CDCl_3) δ 7.16 – 7.10 (m, 4H), 6.81 (s, 2H), 3.71 (s, 6H), 3.34 (s, 3H), 3.04 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.66 (dd, $J = 14.4, 4.2$ Hz, 1H), 2.13 (s, 1H), 1.60 (dd, $J = 167.4, 80.3$ Hz, 2H).

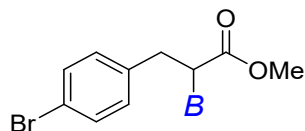
^{13}C NMR (151 MHz, CDCl_3) δ 181.67, 143.24, 130.61, 129.88, 127.92, 120.46, 50.39, 38.45, 35.99.

^{11}B NMR (193 MHz, CDCl_3) δ -24.86 (t, $J = 90.3$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.09.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.13 (q, $J = 8.4$ Hz, 4H), 6.81 (s, 2H), 3.71 (s, 6H), 3.34 (s, 3H), 3.04 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.66 (dd, $J = 14.4, 4.3$ Hz, 1H), 2.13 (td, $J = 10.6, 5.0$ Hz, 1H), 1.59 (d, $J = 20.9$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{20}\text{BClN}_2\text{NaO}_2$ 329.1204; found: 329.1194.



(3-(4-bromophenyl)-1-methoxy-1-oxopropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**6d**): Colorless liquid (45 mg, 64% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

^1H NMR (600 MHz, CDCl_3) δ 7.31 – 7.28 (m, 2H), 7.06 (d, $J = 8.3$ Hz, 2H), 6.81 (s, 2H), 3.71 (s, 6H), 3.34 (s, 3H), 3.03 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.65 (dd, $J = 14.3, 4.2$ Hz, 1H), 2.12 (s, 1H), 1.74 (dd, $J = 161.4, 83.9$ Hz, 2H).

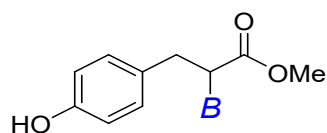
^{13}C NMR (151 MHz, CDCl_3) δ 181.65, 143.76, 130.86, 130.34, 120.46, 118.71, 50.40, 38.50, 36.00.

¹¹B NMR (193 MHz, CDCl₃) δ -25.09 (t, *J* = 90.2 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -25.09.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.29 (d, *J* = 8.2 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 2H), 6.81 (s, 2H), 3.72 (s, 6H), 3.34 (s, 3H), 3.03 (dd, *J* = 14.3, 10.3 Hz, 1H), 2.65 (dd, *J* = 14.4, 4.3 Hz, 1H), 2.13 (td, *J* = 10.5, 5.1 Hz, 1H), 1.59 (d, *J* = 22.5 Hz, 2H).

HRMS (ESI-TOF) *m/z*: [M + Na]⁺ cacl. for C₁₅H₂₀BBrN₂NaO₂ 373.0699; found: 373.0693.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(3-(4-hydroxyphenyl)-1-methoxy-1-oxopropan-2-yl)dihydroborate (**6e**): White solid (45 mg, 64% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 6.96 (d, *J* = 8.4 Hz, 2H), 6.75 (s, 2H), 6.58 (d, *J* = 8.5 Hz, 2H), 3.69 (s, 6H), 3.41 (s, 3H), 2.98 (dd, *J* = 14.5, 10.7 Hz, 1H), 2.62 (dd, *J* = 14.5, 4.0 Hz, 1H), 2.19 (d, *J* = 4.8 Hz, 1H), 1.62 (dd, *J* = 179.8, 79.5 Hz, 2H).

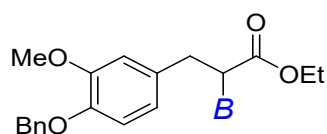
¹³C NMR (151 MHz, CDCl₃) δ 183.23, 153.85, 135.92, 129.20, 120.51, 114.91, 50.73, 38.10, 35.94.

¹¹B NMR (193 MHz, CDCl₃) δ -24.99 (t, *J* = 90.3 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -24.99.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 6.96 (d, *J* = 8.1 Hz, 2H), 6.75 (s, 2H), 6.58 (d, *J* = 8.1 Hz, 2H), 3.69 (s, 6H), 3.41 (s, 3H), 2.99 (dd, *J* = 14.4, 10.7 Hz, 1H), 2.62 (dd, *J* = 14.5, 4.0 Hz, 1H), 2.19 (td, *J* = 10.3, 5.1 Hz, 1H), 1.61 (d, *J* = 23.7 Hz, 2H).

HRMS (ESI-TOF) *m/z*: [M + Na]⁺ cacl. for C₁₅H₂₁BN₂NaO₃ 311.1543; found: 311.1531.



(3-(4-(benzyloxy)-3-methoxyphenyl)-1-ethoxy-1-oxopropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**6f**): Colorless liquid (60 mg, 71% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.41 (d, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.26 (t, *J* = 7.3 Hz, 1H), 6.80 (d, *J* = 1.9 Hz, 1H), 6.77 (s, 2H), 6.73 (d, *J* = 8.2 Hz, 1H), 6.66 (dd, *J* = 8.2, 1.9 Hz, 1H), 5.08 (s, 2H), 3.83 (s, 3H), 3.83 – 3.74 (m, 2H), 3.70 (s, 6H), 3.05 (dd, *J* = 14.4, 10.2 Hz, 1H), 2.65 (dd, *J* = 14.4, 4.2 Hz, 1H), 2.15 (s, 1H), 1.62 (dd, *J* = 180.0, 91.1 Hz, 2H), 0.97 (t, *J* = 7.1 Hz, 3H).

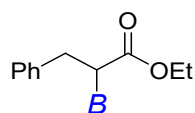
¹³C NMR (151 MHz, CDCl₃) δ 181.73, 149.14, 145.72, 138.51, 137.70, 128.44, 127.64, 127.33, 120.39, 120.24, 114.10, 112.64, 71.24, 58.56, 55.87, 38.79, 36.01, 14.40.

¹¹B NMR (193 MHz, CDCl₃) δ -24.99 (t, *J* = 89.7 Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.03.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.41 (d, $J = 7.6$ Hz, 2H), 7.33 (t, $J = 7.5$ Hz, 2H), 7.26 (t, $J = 7.3$ Hz, 1H), 6.80 (s, 1H), 6.77 (s, 2H), 6.73 (d, $J = 8.1$ Hz, 1H), 6.66 (d, $J = 8.2$ Hz, 1H), 5.08 (s, 2H), 3.83 (s, 3H), 3.78 (td, $J = 18.1, 7.2$ Hz, 2H), 3.70 (s, 6H), 3.05 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.65 (dd, $J = 14.4, 4.1$ Hz, 1H), 2.15 (td, $J = 10.2, 5.2$ Hz, 1H), 1.61 (d, $J = 21.3$ Hz, 2H), 0.97 (t, $J = 7.1$ Hz, 3H).

Characterization agrees with previous reports for this compound.⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-ethoxy-1-oxo-3-phenylpropan-2-yl) dihydroborate (**6g**): Colorless liquid (43 mg, 75% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

^1H NMR (600 MHz, CDCl_3) δ 7.22 – 7.17 (m, 4H), 7.10 – 7.06 (m, 1H), 6.80 (s, 2H), 3.81 (tdd, $J = 10.8, 7.1, 3.7$ Hz, 2H), 3.73 (s, 6H), 3.11 (dd, $J = 14.3, 10.2$ Hz, 1H), 2.72 (dd, $J = 14.3, 4.0$ Hz, 1H), 2.19 (s, 1H), 1.70 – 1.35 (m, 2H), 0.98 (t, $J = 7.1$ Hz, 3H).

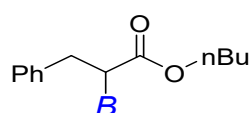
^{13}C NMR (151 MHz, CDCl_3) δ 181.62, 144.87, 128.55, 127.84, 124.98, 120.35, 58.54, 39.14, 36.04, 14.33.

^{11}B NMR (193 MHz, CDCl_3) δ -24.77 (t, $J = 89.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.00.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.22 – 7.17 (m, 4H), 7.08 (t, $J = 6.5$ Hz, 1H), 6.80 (s, 2H), 3.81 (ddd, $J = 26.2, 10.8, 7.1$ Hz, 2H), 3.73 (s, 6H), 3.12 (dd, $J = 14.3, 10.2$ Hz, 1H), 2.72 (dd, $J = 14.3, 4.3$ Hz, 1H), 2.19 (td, $J = 10.4, 5.2$ Hz, 1H), 1.64 (s, 2H), 0.98 (t, $J = 7.1$ Hz, 3H).

Characterization agrees with previous reports for this compound.⁴



(1-butoxy-1-oxo-3-phenylpropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**6h**): Colorless liquid (48 mg, 76% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

^1H NMR (600 MHz, CDCl_3) δ 7.21 – 7.16 (m, 4H), 7.07 (dd, $J = 7.4, 5.1$ Hz, 1H), 6.79 (s, 2H), 3.84 – 3.79 (m, 1H), 3.72 (s, 6H), 3.71 – 3.67 (m, 1H), 3.11 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.71 (dd, $J = 14.4, 4.1$ Hz, 1H), 2.19 (td, $J = 10.3, 5.4$ Hz, 1H), 1.63 (s, 2H), 1.33 (dd, $J = 14.9, 7.0$ Hz, 2H), 1.21 – 1.14 (m, 2H), 0.82 (t, $J = 7.4$ Hz, 3H).

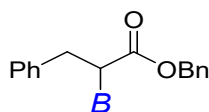
^{13}C NMR (151 MHz, CDCl_3) δ 181.75, 144.83, 128.49, 127.83, 124.98, 120.38, 62.67, 39.16, 36.01, 30.93, 19.14, 13.79.

^{11}B NMR (193 MHz, CDCl_3) δ -24.98 (t, $J = 90.0$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.98.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.21 – 7.16 (m, 4H), 7.07 (dd, $J = 7.4, 5.1$ Hz, 1H), 6.79 (s, 2H), 3.84 – 3.79 (m, 1H), 3.72 (s, 6H), 3.71 – 3.67 (m, 1H), 3.11 (dd, $J =$

14.3, 10.3 Hz, 1H), 2.71 (dd, $J = 14.4, 4.1$ Hz, 1H), 2.19 (td, $J = 10.3, 5.4$ Hz, 1H), 1.63 (s, 2H), 1.33 (dd, $J = 14.9, 7.0$ Hz, 2H), 1.21 – 1.14 (m, 2H), 0.82 (t, $J = 7.4$ Hz, 3H). Characterization agrees with previous reports for this compound.⁴



(1-(benzyloxy)-1-oxo-3-phenylpropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**6i**): Colorless liquid (60 mg, 71% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

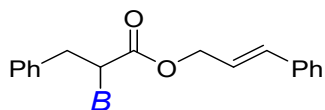
¹H NMR (600 MHz, CDCl₃) δ 7.25 – 7.22 (m, 3H), 7.21 – 7.16 (m, 4H), 7.13 – 7.07 (m, 3H), 6.65 (s, 2H), 4.84 (q, $J = 12.6$ Hz, 2H), 3.61 (s, 6H), 3.15 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.76 (dd, $J = 14.4, 4.3$ Hz, 1H), 2.28 (td, $J = 10.4, 5.2$ Hz, 1H), 1.64 (s, 2H). **¹³C NMR (151 MHz, CDCl₃)** δ 181.36, 144.73, 137.31, 128.57, 128.21, 127.92, 127.47, 125.08, 120.32, 64.56, 39.24, 35.94.

¹¹B NMR (193 MHz, CDCl₃) δ -24.97 (t, $J = 89.9$ Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -24.97.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.25 – 7.22 (m, 3H), 7.21 – 7.16 (m, 4H), 7.13 – 7.07 (m, 3H), 6.65 (s, 2H), 4.84 (q, $J = 12.6$ Hz, 2H), 3.61 (s, 6H), 3.15 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.76 (dd, $J = 14.4, 4.3$ Hz, 1H), 2.28 (td, $J = 10.4, 5.2$ Hz, 1H), 1.64 (s, 2H).

Characterization agrees with previous reports for this compound.⁴



(1-(cinnamyloxy)-1-oxo-3-phenylpropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**6j**): Colorless liquid (58 mg, 78% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.28 – 7.25 (m, 4H), 7.20 (dd, $J = 6.5, 3.9$ Hz, 3H), 7.16 (t, $J = 7.6$ Hz, 2H), 7.05 (t, $J = 7.2$ Hz, 1H), 6.66 (s, 2H), 6.38 (d, $J = 15.9$ Hz, 1H), 5.97 (dt, $J = 15.9, 6.2$ Hz, 1H), 4.41 (dddd, $J = 37.5, 13.2, 6.2, 1.3$ Hz, 2H), 3.65 (s, 6H), 3.13 (dd, $J = 14.3, 10.3$ Hz, 1H), 2.73 (dd, $J = 14.3, 4.1$ Hz, 1H), 2.24 (s, 1H), 1.50 (dd, $J = 169.5, 83.7$ Hz, 2H).

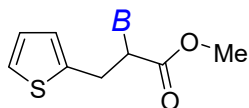
¹³C NMR (151 MHz, CDCl₃) δ 181.31, 144.78, 136.62, 132.55, 128.59, 127.94, 127.80, 126.46, 125.11, 124.73, 120.41, 63.32, 39.23, 36.05.

¹¹B NMR (193 MHz, CDCl₃) δ -24.89 (t, $J = 90.0$ Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -24.89.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.31 – 7.27 (m, 4H), 7.22 (d, $J = 7.2$ Hz, 3H), 7.18 (t, $J = 7.5$ Hz, 2H), 7.08 (t, $J = 7.2$ Hz, 1H), 6.68 (s, 2H), 6.41 (d, $J = 15.9$ Hz, 1H), 6.03 – 5.96 (m, 1H), 4.43 (ddd, $J = 35.1, 13.2, 6.2$ Hz, 2H), 3.68 (s, 6H), 3.15 (dd, $J = 14.2, 10.4$ Hz, 1H), 2.75 (dd, $J = 14.3, 3.9$ Hz, 1H), 2.30 – 2.23 (m, 1H), 1.66 (s, 2H).

Characterization agrees with previous reports for this compound.⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-methoxy-1-oxo-3-(thiophen-2-yl)propan-2-yl)dihydroborate (**6k**): Colorless liquid (26 mg, 46% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.00 (s, 1H), 6.83 (d, $J = 2.8$ Hz, 1H), 6.82 (s, 2H), 6.75 (s, 1H), 3.74 (s, 6H), 3.42 (s, 3H), 3.31 (t, $J = 12.6$ Hz, 1H), 2.89 (d, $J = 15.3$ Hz, 1H), 2.20 (s, 1H), 1.76 – 1.36 (m, 2H).

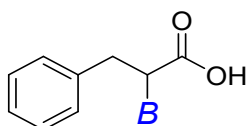
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 181.59, 148.14, 126.37, 123.58, 122.25, 120.46, 50.51, 36.02, 33.32.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -25.17 (t, $J = 89.8$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.17.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.00 (s, 1H), 6.83 (d, $J = 2.5$ Hz, 1H), 6.81 (s, 2H), 6.75 (s, 1H), 3.74 (s, 6H), 3.42 (s, 3H), 3.31 (t, $J = 12.7$ Hz, 1H), 2.90 (d, $J = 15.3$ Hz, 1H), 2.20 (s, 1H), 1.61 (d, $J = 33.7$ Hz, 2H).

Characterization agrees with previous reports for this compound.⁴



(1-carboxy-2-phenylethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**7a**): White solid (27 mg, 52% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 2/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.22 (s, 4H), 7.11 (s, 1H), 6.68 (s, 2H), 3.63 (s, 6H), 3.05 (t, $J = 12.4$ Hz, 1H), 2.73 (d, $J = 14.5$ Hz, 1H), 2.17 (s, 1H), 1.91 – 1.40 (m, 2H).

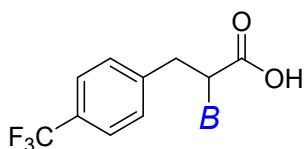
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 188.54, 144.74, 128.59, 127.87, 125.07, 120.50, 38.65, 35.85.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -24.59 (t, $J = 90.5$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.60.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.22 (s, 4H), 7.11 (s, 1H), 6.68 (s, 2H), 3.62 (s, 6H), 3.05 (t, $J = 11.8$ Hz, 1H), 2.73 (d, $J = 14.5$ Hz, 1H), 2.17 (s, 1H), 1.65 (d, $J = 38.3$ Hz, 2H).

Characterization agrees with previous reports for this compound.³



(1-carboxy-2-(4-(trifluoromethyl)phenyl)ethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**7b**): White solid (35 mg, 58% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 2/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.46 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 8.0$ Hz, 2H), 6.71 (s, 2H), 3.62 (s, 6H), 3.07 (dd, $J = 14.4, 10.6$ Hz, 1H), 2.77 (dd, $J = 14.5, 3.7$ Hz, 1H),

2.14 (s, 1H), 1.61 (dd, $J = 152.6, 78.3$ Hz, 2H).

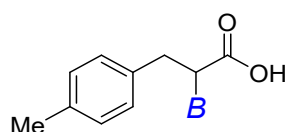
^{13}C NMR (151 MHz, CDCl_3) δ 188.33, 148.92, 128.92, 126.4 (q, $J = 252.7$ Hz), 127.32 (q, $J = 33.2$ Hz), 124.66 (q, $J = 3.0$ Hz), 120.53, 38.50, 35.79.

^{11}B NMR (193 MHz, CDCl_3) δ -24.64 (t, $J = 91.6$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.63.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.46 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 7.9$ Hz, 2H), 6.71 (s, 2H), 3.62 (s, 6H), 3.07 (dd, $J = 14.4, 10.7$ Hz, 1H), 2.77 (dd, $J = 14.5, 3.7$ Hz, 1H), 2.14 (d, $J = 4.6$ Hz, 1H), 1.63 (d, $J = 39.0$ Hz, 2H). ^{19}F NMR (565 MHz, CDCl_3) δ -62.03.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{18}\text{BF}_3\text{N}_2\text{NaO}_2$ 349.1311; found: 349.1306.



(1-carboxy-2-(p-tolyl)ethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**7c**): White solid (38 mg, 70% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 2/1.

^1H NMR (600 MHz, CDCl_3) δ 7.12 (d, $J = 7.9$ Hz, 2H), 7.03 (d, $J = 7.9$ Hz, 2H), 6.69 (s, 2H), 3.63 (s, 6H), 3.02 (dd, $J = 14.5, 10.5$ Hz, 1H), 2.68 (dd, $J = 14.6, 3.8$ Hz, 1H), 2.29 (s, 3H), 2.16 (s, 1H), 1.63 (dd, $J = 192.3, 73.8$ Hz, 2H).

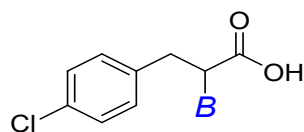
^{13}C NMR (151 MHz, CDCl_3) δ 188.69, 141.66, 134.28, 128.56, 128.44, 120.49, 38.20, 35.85, 21.01.

^{11}B NMR (193 MHz, CDCl_3) δ -24.58 (t, $J = 88.9$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.58.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.12 (d, $J = 7.8$ Hz, 2H), 7.03 (d, $J = 7.8$ Hz, 2H), 6.69 (s, 2H), 3.63 (s, 6H), 3.02 (dd, $J = 14.5, 10.5$ Hz, 1H), 2.68 (dd, $J = 14.6, 3.8$ Hz, 1H), 2.29 (s, 3H), 2.15 (d, $J = 4.8$ Hz, 1H), 1.65 (d, $J = 38.5$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{15}\text{H}_{21}\text{BN}_2\text{NaO}_2$ 295.1594; found: 295.1587.



(1-carboxy-2-(4-chlorophenyl)ethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**7d**): White solid (38 mg, 65% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 2/1.

^1H NMR (600 MHz, CDCl_3) δ 7.19 – 7.13 (m, 4H), 6.75 (s, 2H), 3.65 (s, 6H), 2.99 (dd, $J = 14.5, 10.5$ Hz, 1H), 2.69 (dd, $J = 14.5, 4.2$ Hz, 1H), 2.11 (s, 1H), 1.62 (dd, $J = 83.4, 39.0$ Hz, 2H).

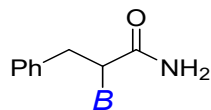
^{13}C NMR (151 MHz, CDCl_3) δ 187.98, 143.14, 130.62, 130.03, 127.85, 120.53, 38.03, 35.87.

^{11}B NMR (193 MHz, CDCl_3) δ -24.70 (t, $J = 88.8$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.70.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.16 (q, J = 8.6 Hz, 4H), 6.75 (s, 2H), 3.65 (s, 6H), 2.99 (dd, J = 14.4, 10.5 Hz, 1H), 2.69 (dd, J = 14.5, 4.1 Hz, 1H), 2.11 (dd, J = 9.6, 5.0 Hz, 1H), 1.63 (d, J = 36.5 Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{14}\text{H}_{18}\text{BClN}_2\text{NaO}_2$ 315.1048; found: 315.1036.



(1-amino-1-oxo-3-phenylpropan-2-yl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**8a**): Colorless liquid (26 mg, 50% yield); Gradient eluent: DCM/MeOH: 50/1 to 20/1.

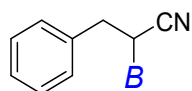
^1H NMR (600 MHz, CDCl_3) δ 7.18 (d, J = 4.4 Hz, 4H), 7.07 (dt, J = 8.7, 4.4 Hz, 1H), 6.74 (s, 2H), 5.28 (s, 2H), 3.69 (s, 6H), 2.94 (dd, J = 14.4, 10.1 Hz, 1H), 2.71 (dd, J = 14.4, 4.7 Hz, 1H), 2.08 (s, 1H), 1.63 (dd, J = 178.7, 89.0 Hz, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 185.28, 144.20, 128.37, 127.95, 125.17, 120.57, 39.66, 36.13. ^{11}B NMR (193 MHz, CDCl_3) δ -24.58 (t, J = 88.9 Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.58.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.18 (d, J = 4.4 Hz, 4H), 7.07 (dt, J = 8.6, 4.4 Hz, 1H), 6.74 (s, 2H), 5.27 (s, 2H), 3.69 (s, 6H), 2.94 (dd, J = 14.4, 10.0 Hz, 1H), 2.71 (dd, J = 14.5, 4.8 Hz, 1H), 2.08 (dq, J = 10.0, 5.0 Hz, 1H), 1.63 (d, J = 2.7 Hz, 2H).

Characterization agrees with previous reports for this compound.⁴



(1-cyano-2-phenylethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**9a**): Colorless liquid (33 mg, 69% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 7.28 (d, J = 4.6 Hz, 4H), 7.18 (dt, J = 8.6, 3.8 Hz, 1H), 6.86 (s, 2H), 3.83 (s, 6H), 2.87 (dd, J = 14.0, 4.6 Hz, 1H), 2.82 (dd, J = 13.9, 10.4 Hz, 1H), 1.88 (s, 1H), 1.82 – 1.45 (m, 2H).

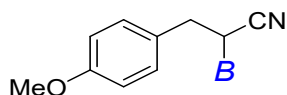
^{13}C NMR (151 MHz, CDCl_3) δ 142.51, 129.10, 128.55, 128.25, 125.97, 120.95, 40.02, 36.40.

^{11}B NMR (193 MHz, CDCl_3) δ 25.90 (t, J = 90.9 Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.90.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.29 – 7.26 (m, 4H), 7.17 (ddd, J = 8.6, 5.2, 3.4 Hz, 1H), 6.85 (s, 2H), 3.83 (s, 6H), 2.86 (dd, J = 14.0, 4.9 Hz, 1H), 2.81 (dd, J = 13.9, 10.3 Hz, 1H), 1.87 (qd, J = 9.9, 4.8 Hz, 1H), 1.69 (d, J = 40.4 Hz, 2H).

Characterization agrees with previous reports for this compound.⁴



(1-cyano-2-(4-methoxyphenyl)ethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**9b**): Colorless liquid (44 mg, 82% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.19 (d, $J = 8.6$ Hz, 2H), 6.85 (s, 2H), 6.82 (d, $J = 8.6$ Hz, 2H), 3.82 (s, 6H), 3.77 (s, 3H), 2.80 (dd, $J = 14.0, 4.6$ Hz, 1H), 2.74 (dd, $J = 13.9, 10.4$ Hz, 1H), 1.83 (s, 1H), 1.78 – 1.30 (m, 2H).

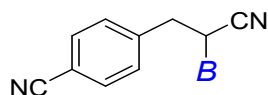
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 157.87, 134.73, 129.48, 129.23, 120.96, 113.66, 55.26, 39.16, 36.38.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -25.97 (t, $J = 90.4$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.97.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.19 (d, $J = 8.5$ Hz, 2H), 6.85 (s, 2H), 6.82 (d, $J = 8.5$ Hz, 2H), 3.82 (s, 6H), 3.77 (s, 3H), 2.80 (dd, $J = 14.1, 4.8$ Hz, 1H), 2.74 (dd, $J = 13.9, 10.5$ Hz, 1H), 1.83 (td, $J = 12.1, 4.7$ Hz, 1H), 1.67 (d, $J = 35.3$ Hz, 2H).

Characterization agrees with previous reports for this compound.⁴



(1-cyano-2-(4-cyanophenyl)ethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**9c**): Colorless liquid (18 mg, 34% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.57 (d, $J = 8.0$ Hz, 2H), 7.39 (d, $J = 8.0$ Hz, 2H), 6.89 (s, 2H), 3.85 (s, 6H), 2.93 (dd, $J = 14.0, 4.9$ Hz, 1H), 2.87 (dd, $J = 13.7, 10.4$ Hz, 1H), 1.84 (ddd, $J = 13.9, 9.4, 4.7$ Hz, 1H), 1.66 (d, $J = 44.8$ Hz, 2H).

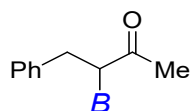
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 147.93, 132.11, 129.47, 128.30, 121.05, 119.26, 109.82, 40.00, 36.41.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -26.00 (t, $J = 90.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.99.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.57 (d, $J = 8.0$ Hz, 2H), 7.39 (d, $J = 8.0$ Hz, 2H), 6.89 (s, 2H), 3.85 (s, 6H), 2.93 (dd, $J = 14.0, 4.9$ Hz, 1H), 2.87 (dd, $J = 13.7, 10.4$ Hz, 1H), 1.84 (ddd, $J = 13.9, 9.4, 4.7$ Hz, 1H), 1.66 (d, $J = 44.8$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{15}\text{H}_{17}\text{BN}_4\text{Na}$ 287.1444; found: 287.1443.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(3-oxo-1-phenylbutan-2-yl) dihydroborate (**10a**): Colorless liquid (38 mg, 65% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.17 (t, $J = 7.5$ Hz, 2H), 7.11 (d, $J = 7.2$ Hz, 2H), 7.07

(t, $J = 7.3$ Hz, 1H), 6.79 (s, 2H), 3.70 (s, 6H), 3.09 (dd, $J = 14.5, 9.7$ Hz, 1H), 2.65 (dd, $J = 14.6, 4.5$ Hz, 1H), 2.58 (s, 1H), 2.00 (s, 3H), 1.89 – 1.47 (m, 2H).

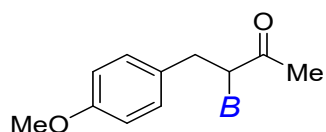
^{13}C NMR (151 MHz, CDCl_3) δ 217.52, 144.39, 128.27, 127.95, 125.06, 120.63, 38.34, 36.15, 28.30.

^{11}B NMR (193 MHz, CDCl_3) δ -25.33 (t, $J = 90.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.33.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.17 (t, $J = 7.5$ Hz, 2H), 7.11 (d, $J = 7.3$ Hz, 2H), 7.06 (t, $J = 7.3$ Hz, 1H), 6.79 (s, 2H), 3.70 (s, 6H), 3.09 (dd, $J = 14.6, 9.6$ Hz, 1H), 2.65 (dd, $J = 14.6, 4.7$ Hz, 1H), 2.58 (td, $J = 10.0, 5.0$ Hz, 1H), 2.00 (s, 3H), 1.61 (d, $J = 31.5$ Hz, 2H).

Characterization agrees with previous reports for this compound.³



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-(4-methoxyphenyl)-3-oxobutan-2-yl) dihydroborate (**10b**): Colorless liquid (38 mg, 65% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 7.03 (d, $J = 8.6$ Hz, 2H), 6.80 (s, 2H), 6.72 (d, $J = 8.6$ Hz, 2H), 3.73 (s, 3H), 3.70 (s, 6H), 3.01 (dd, $J = 14.5, 9.7$ Hz, 1H), 2.58 (dd, $J = 14.5, 4.5$ Hz, 1H), 2.53 (s, 1H), 1.99 (s, 3H), 1.66 (dd, $J = 107.0, 74.3$ Hz, 2H).

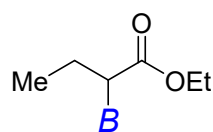
^{13}C NMR (151 MHz, CDCl_3) δ 217.69, 157.18, 136.60, 129.09, 120.64, 113.38, 55.23, 37.43, 36.15, 28.30.

^{11}B NMR (193 MHz, CDCl_3) δ -25.36 (t, $J = 90.2$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.36.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.03 (d, $J = 8.5$ Hz, 2H), 6.79 (s, 2H), 6.72 (d, $J = 8.6$ Hz, 2H), 3.73 (s, 3H), 3.70 (s, 6H), 3.01 (dd, $J = 14.5, 9.7$ Hz, 1H), 2.58 (dd, $J = 14.5, 4.7$ Hz, 1H), 2.53 (td, $J = 9.9, 5.0$ Hz, 1H), 1.98 (s, 3H), 1.59 (d, $J = 31.1$ Hz, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl. for $\text{C}_{16}\text{H}_{23}\text{BN}_2\text{NaO}_2$ 309.1750; found: 309.1743.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-ethoxy-1-oxobutan-2-yl)dihydroborate (**11a- α**): Colorless liquid (5 mg, 5% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 6.81 (s, 2H), 3.92 – 3.81 (m, 2H), 3.74 (s, 6H), 1.73 (dd, $J = 10.7, 5.6$ Hz, 2H), 1.59 (dd, $J = 73.3, 27.3$ Hz, 2H), 1.39 (dd, $J = 10.0, 6.1$ Hz, 1H), 1.05 (t, $J = 7.1$ Hz, 3H), 0.88 (t, $J = 7.1$ Hz, 3H).

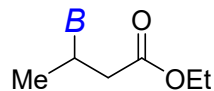
^{13}C NMR (151 MHz, CDCl_3) δ 182.51, 120.28, 58.39, 36.05, 26.32, 14.96, 14.46.

^{11}B NMR (193 MHz, CDCl_3) δ -25.07 (t, $J = 89.3$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.31.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.81 (s, 2H), 3.92 – 3.81 (m, 2H), 3.74 (s, 6H), 1.72 (d, $J = 5.0$ Hz, 2H), 1.53 (d, $J = 48.3$ Hz, 2H), 1.41 – 1.37 (m, 1H), 1.05 (t, $J = 7.1$ Hz, 3H), 0.88 (t, $J = 7.1$ Hz, 3H).

Characterization agrees with previous reports for this compound.³



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(4-ethoxy-4-oxobutan-2-yl)dihydroborate (**11a- β**): Colorless liquid (57 mg, 64% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 6.79 (s, 2H), 3.99 (q, $J = 7.1$ Hz, 2H), 3.74 (s, 6H), 2.18 (dd, $J = 14.0, 6.3$ Hz, 1H), 2.06 (dd, $J = 13.5, 9.1$ Hz, 1H), 1.61 – 1.21 (m, 2H), 1.19 (t, $J = 7.1$ Hz, 3H), 1.17 (dt, $J = 13.0, 6.5$ Hz, 1H), 0.79 (d, $J = 6.0$ Hz, 3H).

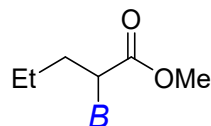
^{13}C NMR (151 MHz, CDCl_3) δ 176.42, 120.17, 59.36, 44.61, 36.05, 22.39, 14.39.

^{11}B NMR (193 MHz, CDCl_3) δ -24.06 (t, $J = 84.4$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.06.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.82 (s, 2H), 4.03 (q, $J = 7.1$ Hz, 2H), 3.78 (s, 6H), 2.21 (dd, $J = 14.0, 6.5$ Hz, 1H), 2.10 (dd, $J = 14.0, 8.8$ Hz, 1H), 1.36 – 1.32 (m, 1H), 1.22 (t, $J = 7.1$ Hz, 3H), 1.17 (dt, $J = 13.0, 6.5$ Hz, 1H), 0.83 (d, $J = 6.9$ Hz, 3H).

Characterization agrees with previous reports for this compound.³



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-methoxy-1-oxopentan-2-yl)dihydroborate (**11b- α**): Colorless liquid (5 mg, 6% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 6.82 (s, 2H), 3.74 (s, 6H), 3.43 (s, 3H), 1.83 (s, 1H), 1.74 (dd, $J = 18.1, 9.6$ Hz, 1H), 1.65 – 1.36 (m, 2H), 1.34 – 1.29 (m, 2H), 1.26 – 1.22 (m, 1H), 0.85 (t, $J = 7.1$ Hz, 3H).

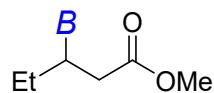
^{13}C NMR (151 MHz, CDCl_3) δ 182.89, 120.33, 50.29, 36.03, 35.66, 23.52, 14.36.

^{11}B NMR (193 MHz, CDCl_3) δ -25.19 (t, $J = 89.6$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.19.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.81 (s, 2H), 3.74 (s, 6H), 3.43 (s, 3H), 1.83 (dt, $J = 9.8, 4.9$ Hz, 1H), 1.74 (dd, $J = 17.9, 9.7$ Hz, 1H), 1.54 (dt, $J = 44.9, 7.9$ Hz, 2H), 1.34 – 1.30 (m, 2H), 1.26 – 1.22 (m, 1H), 0.85 (t, $J = 7.1$ Hz, 3H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl'd. for $\text{C}_{11}\text{H}_{21}\text{BN}_2\text{NaO}_2$ 247.1594; found: 247.1586.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-methoxy-1-oxopentan-3-yl)dihydroborate (**11b-β**): Colorless liquid (54 mg, 60% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 6.77 (s, 2H), 3.73 (s, 6H), 3.48 (s, 3H), 2.25 (dd, $J = 12.7, 5.5$ Hz, 1H), 2.01 (dd, $J = 14.0, 8.4$ Hz, 1H), 1.61 – 1.23 (m, 2H), 1.21 (dd, $J = 13.9, 7.0$ Hz, 2H), 0.99 (s, 1H), 0.81 (t, $J = 7.4$ Hz, 3H).

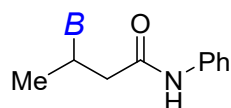
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 177.07, 120.16, 50.78, 41.75, 35.99, 30.00, 13.22.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -25.31 (t, $J = 84.4$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.31.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.77 (s, 2H), 3.73 (s, 6H), 3.48 (s, 3H), 2.25 (dd, $J = 14.1, 6.8$ Hz, 1H), 2.01 (dd, $J = 14.0, 8.3$ Hz, 1H), 1.34 (s, 1H), 1.21 (dq, $J = 13.4, 6.6$ Hz, 3H), 0.99 (dt, $J = 12.5, 6.3$ Hz, 1H), 0.81 (t, $J = 7.4$ Hz, 3H).

Characterization agrees with previous reports for this compound.⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(4-oxo-4-(phenylamino)butan-2-yl)dihydroborate (**11c**): White solid (30 mg, 55% yield); Gradient eluent: EtOAc/petroleum ether: 1/1 to 2/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.83 (s, 1H), 7.53 (d, $J = 7.9$ Hz, 2H), 7.28 (d, $J = 7.7$ Hz, 2H), 7.03 (t, $J = 7.4$ Hz, 1H), 6.79 (s, 2H), 3.76 (s, 6H), 2.32 (dd, $J = 13.8, 7.3$ Hz, 1H), 2.24 (dd, $J = 13.6, 6.8$ Hz, 1H), 1.76 – 1.35 (m, 2H), 1.19 (s, 1H), 0.84 (d, $J = 6.6$ Hz, 3H).

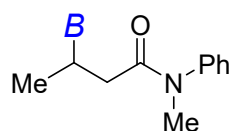
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 174.65, 138.77, 128.82, 123.38, 120.32, 119.60, 48.69, 36.14, 22.57.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -24.03 (t, $J = 84.0$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.03.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.82 (s, 1H), 7.53 (d, $J = 7.9$ Hz, 2H), 7.29 – 7.26 (m, 2H), 7.03 (t, $J = 7.3$ Hz, 1H), 6.79 (s, 2H), 3.76 (s, 6H), 2.32 (dd, $J = 13.9, 7.4$ Hz, 1H), 2.24 (dd, $J = 13.9, 6.9$ Hz, 1H), 1.58 – 1.40 (m, 2H), 1.18 (td, $J = 13.1, 6.6$ Hz, 1H), 0.84 (d, $J = 6.9$ Hz, 3H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{15}\text{H}_{22}\text{BN}_3\text{NaO}$ 294.1754; found: 294.1750.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(4-(methyl(phenyl)amino)-4-oxobutan-2-yl)dihydroborate (**11d**): White solid (28 mg, 50% yield); Gradient eluent: EtOAc/petroleum ether: 1/1 to 3/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.34 (t, $J = 7.7$ Hz, 2H), 7.24 (d, $J = 7.2$ Hz, 1H), 7.17 (d, $J = 7.5$ Hz, 2H), 6.74 (s, 2H), 3.65 (s, 6H), 3.23 (s, 3H), 2.07 (s, 1H), 1.92 (s, 1H),

1.16 (d, $J = 51.5$ Hz, 3H), 0.72 (d, $J = 4.6$ Hz, 3H).

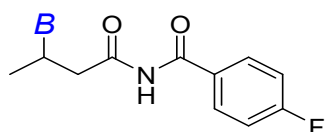
^{13}C NMR (151 MHz, CDCl_3) δ 176.19, 145.18, 129.23, 127.69, 126.90, 120.03, 43.72, 37.32, 35.99, 22.19.

^{11}B NMR (193 MHz, CDCl_3) δ -24.09 (t, $J = 84.4$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -24.09.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.34 (t, $J = 7.7$ Hz, 2H), 7.24 (d, $J = 7.2$ Hz, 1H), 7.17 (d, $J = 7.5$ Hz, 2H), 6.74 (s, 2H), 3.65 (s, 6H), 3.23 (s, 3H), 2.07 (s, 1H), 1.92 (s, 1H), 1.16 (d, $J = 51.5$ Hz, 3H), 0.72 (d, $J = 4.6$ Hz, 3H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ calcd. for $\text{C}_{16}\text{H}_{24}\text{BN}_3\text{NaO}$ 308.1910; found: 308.1907.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(4-(4-fluorobenzamido)-4-oxobutan-2-yl)dihydroborate (**11e**): White solid (18 mg, 28% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 8.88 (s, 1H), 7.90 (ddd, $J = 8.2, 5.1, 2.5$ Hz, 2H), 7.15 – 7.11 (m, 2H), 6.84 (s, 2H), 3.78 (s, 6H), 2.67 (dd, $J = 14.0, 6.5$ Hz, 1H), 2.52 (dd, $J = 14.0, 7.4$ Hz, 1H), 1.41 – 1.28 (m, 2H), 1.22 – 1.17 (m, 1H), 0.85 (d, $J = 6.9$ Hz, 3H).

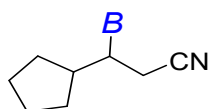
^{13}C NMR (151 MHz, CDCl_3) δ 176.69, 165.37 (d, $J = 253.7$ Hz), 164.34, 130.35 (d, $J = 9.1$ Hz), 130.08 (d, $J = 3.0$ Hz), 120.35, 115.92 (d, $J = 21.1$ Hz), 48.06, 36.15, 22.25.

^{11}B NMR (193 MHz, CDCl_3) δ -23.91 (t, $J = 84.2$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -23.91.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 8.87 (s, 1H), 7.90 (dd, $J = 8.7, 5.3$ Hz, 2H), 7.13 (t, $J = 8.5$ Hz, 2H), 6.84 (s, 2H), 3.78 (s, 6H), 2.67 (dd, $J = 14.1, 6.6$ Hz, 1H), 2.52 (dd, $J = 14.1, 7.4$ Hz, 1H), 1.59 – 1.45 (m, 2H), 1.20 (dd, $J = 11.9, 6.2$ Hz, 1H), 0.86 (d, $J = 6.9$ Hz, 3H).

Characterization agrees with previous reports for this compound.⁵



(1-cyano-2-cyclopentylethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**11f**): Colorless liquid (23 mg, 49% yield); Gradient eluent: EtOAc/petroleum ether: 1/2 to 1/1.

^1H NMR (600 MHz, CDCl_3) δ 6.83 (s, 2H), 3.78 (s, 6H), 2.23 (d, $J = 15.6$ Hz, 1H), 2.12 (dd, $J = 16.3, 8.5$ Hz, 1H), 1.80 – 1.72 (m, 2H), 1.65 – 1.60 (m, 1H), 1.58 – 1.51 (m, 2H), 1.46 (dd, $J = 20.0, 6.1$ Hz, 2H), 1.44 – 1.15 (m, 2H), 1.11 – 1.03 (m, 2H), 0.87 (s, 1H).

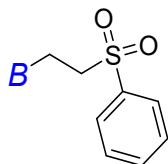
^{13}C NMR (151 MHz, CDCl_3) δ 122.67, 120.50, 46.98, 36.10, 32.07, 31.68, 25.48, 25.43, 23.33.

^{11}B NMR (193 MHz, CDCl_3) δ -26.00 (t, $J = 85.2$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -26.00. $^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.84

(s, 2H), 3.79 (s, 6H), 2.25 (dd, $J = 16.4, 4.8$ Hz, 1H), 2.13 (dd, $J = 16.4, 8.6$ Hz, 1H), 1.81 – 1.74 (m, 2H), 1.66 – 1.62 (m, 1H), 1.56 (dd, $J = 18.5, 12.2$ Hz, 2H), 1.49 (dd, $J = 12.7, 5.2$ Hz, 2H), 1.42 (d, $J = 45.9$ Hz, 2H), 1.14 – 1.06 (m, 2H), 0.92 – 0.86 (m, 1H).

Characterization agrees with previous reports for this compound.⁴



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(2-(phenylsulfonyl)ethyl)dihydroborate (**11g**): Colorless liquid (21 mg, 39% yield); Gradient eluent: EtOAc/petroleum ether: 1/1 to 2/1.

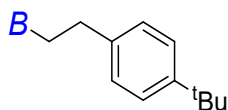
¹H NMR (600 MHz, CDCl₃) δ 7.87 (d, $J = 7.2$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.7$ Hz, 2H), 6.80 (s, 2H), 3.69 (s, 6H), 3.09 – 3.01 (m, 2H), 1.57 – 1.22 (m, 2H), 0.67 (s, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 139.90, 132.85, 128.83, 128.11, 120.44, 60.23, 35.92.

¹¹B NMR (193 MHz, CDCl₃) δ -27.88 (t, $J = 86.0$ Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -27.88. ¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.87 (d, $J = 7.3$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.6$ Hz, 2H), 6.80 (s, 2H), 3.69 (s, 6H), 3.07 – 3.02 (m, 2H), 1.33 (s, 2H), 0.70 – 0.63 (m, 2H).

HRMS (ESI-TOF) m/z : [M + Na]⁺ cacl. for C₁₃H₁₉BN₂NaO₂S 301.1158; found: 301.1155.



(4-(tert-butyl)phenethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**11h**): Colorless liquid (11 mg, 20% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1.

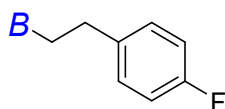
¹H NMR (600 MHz, CDCl₃) δ 7.23 (d, $J = 8.1$ Hz, 2H), 7.12 (d, $J = 8.1$ Hz, 2H), 6.75 (s, 2H), 3.76 (s, 6H), 2.54 – 2.50 (m, 2H), 1.55 – 1.38 (m, 2H), 1.29 (s, 9H), 0.78 (s, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 147.12, 145.06, 127.48, 124.74, 119.97, 38.14, 35.94, 34.23, 31.49.

¹¹B NMR (193 MHz, CDCl₃) δ -27.04 (t, $J = 83.5$ Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -27.05. ¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.23 (d, $J = 8.1$ Hz, 2H), 7.12 (d, $J = 8.0$ Hz, 2H), 6.75 (s, 2H), 3.76 (s, 6H), 2.55 – 2.49 (m, 2H), 1.57 (d, $J = 31.6$ Hz, 2H), 1.29 (s, 9H), 0.78 (td, $J = 11.4, 5.7$ Hz, 2H).

HRMS (ESI-TOF) m/z : [M + Na]⁺ cacl. for C₁₇H₂₇BN₂Na 293.2165; found: 293.2158.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(4-fluorophenethyl)dihydroborate (11i):
Colorless liquid (13 mg, 28% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.12 (dd, $J = 8.3, 5.8$ Hz, 2H), 6.88 (t, $J = 8.8$ Hz, 2H), 6.77 (s, 2H), 3.75 (s, 6H), 2.55 – 2.47 (m, 2H), 1.61 – 1.26 (m, 2H), 0.73 (s, 2H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 160.56 (d, $J = 240.1$ Hz), 143.68, 129.05 (d, $J = 7.6$ Hz), 120.04, 114.43 (d, $J = 21.1$ Hz), 37.87, 35.93.

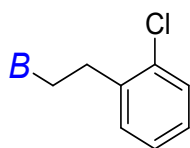
$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -27.18 (t, $J = 84.0$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -27.17.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.12 (dd, $J = 8.2, 5.9$ Hz, 2H), 6.87 (t, $J = 8.8$ Hz, 2H), 6.76 (s, 2H), 3.75 (s, 6H), 2.53 – 2.49 (m, 2H), 1.51 (s, 2H), 0.73 (tt, $J = 11.7, 5.9$ Hz, 2H).

$^{19}\text{F NMR}$ (565 MHz, CDCl_3) δ -120.00.

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl'd. for $\text{C}_{13}\text{H}_{18}\text{BFN}_2\text{Na}$ 255.1445; found: 255.1455.



(2-chlorophenethyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (11j):
Colorless liquid (16 mg, 32% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1;

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.28 (dd, $J = 7.6, 1.3$ Hz, 1H), 7.25 (d, $J = 7.1$ Hz, 1H), 7.12 (td, $J = 7.5, 0.9$ Hz, 1H), 7.02 (td, $J = 7.7, 1.6$ Hz, 1H), 6.79 (s, 2H), 3.79 (s, 6H), 2.62 – 2.54 (m, 2H), 1.79 – 1.45 (m, 2H), 0.74 (s, 2H).

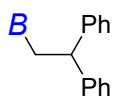
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 145.38, 133.45, 129.77, 128.94, 126.46, 125.92, 120.08, 36.05, 35.96.

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -27.19 (t, $J = 83.9$ Hz).

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -27.19.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.27 (dd, $J = 7.6, 1.1$ Hz, 1H), 7.24 (d, $J = 7.9$ Hz, 1H), 7.12 (td, $J = 7.5, 0.8$ Hz, 1H), 7.01 (td, $J = 7.7, 1.5$ Hz, 1H), 6.78 (s, 2H), 3.78 (s, 6H), 2.61 – 2.54 (m, 2H), 1.62 – 1.50 (m, 2H), 0.77 – 0.70 (m, 2H).

HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ cacl'd. for $\text{C}_{13}\text{H}_{18}\text{BClN}_2\text{Na}$ 271.1149; found: 271.1140.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(2,2-diphenylethyl)dihydroborate (**11k**): Colorless liquid (27 mg, 47% yield); Gradient eluent: EtOAc/petroleum ether: 1/5 to 1/3.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.21 (d, $J = 7.8$ Hz, 4H), 7.16 (t, $J = 7.6$ Hz, 4H), 7.03 (t, $J = 7.3$ Hz, 2H), 6.58 (s, 2H), 3.84 (t, $J = 7.5$ Hz, 1H), 3.52 (s, 6H), 1.53 (dd, $J = 94.9, 71.7$ Hz, 2H), 1.32 (s, 2H).

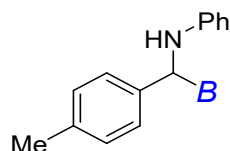
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 149.29, 127.81, 127.74, 124.91, 119.82, 54.34, 35.66.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -27.93 (t, $J = 83.7$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -27.93.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.21 (d, $J = 7.6$ Hz, 4H), 7.16 (t, $J = 7.5$ Hz, 4H), 7.03 (t, $J = 7.2$ Hz, 2H), 6.58 (s, 2H), 3.84 (t, $J = 7.6$ Hz, 1H), 3.52 (s, 6H), 1.46 (s, 2H), 1.32 (dd, $J = 11.6, 5.5$ Hz, 2H).

Characterization agrees with previous reports for this compound.¹



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)((phenylamino)(p-tolyl)methyl)dihydroborate (**12a**): White solid (41 mg, 67% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.01 (t, $J = 7.8$ Hz, 2H), 6.87 (d, $J = 7.9$ Hz, 2H), 6.76 (d, $J = 7.9$ Hz, 2H), 6.71 (s, 2H), 6.49 (t, $J = 8.7$ Hz, 3H), 3.71 (s, 1H), 3.37 (s, 6H), 2.24 (s, 3H), 1.56 (dd, $J = 176.0, 87.8$ Hz, 2H).

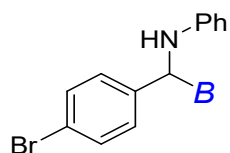
$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 150.31, 148.81, 132.50, 128.77, 128.49, 124.46, 120.31, 114.97, 112.88, 35.57, 20.98.

$^{11}\text{B NMR}$ (193 MHz, CDCl_3) δ -23.05 (t, $J = 87.8$ Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -23.05.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.01 (t, $J = 7.3$ Hz, 2H), 6.87 (d, $J = 7.3$ Hz, 2H), 6.76 (d, $J = 7.6$ Hz, 2H), 6.49 (t, $J = 8.8$ Hz, 3H), 3.71 (s, 1H), 3.38 (s, 6H), 2.24 (s, 3H), 1.63 (dd, $J = 91.7, 85.0$ Hz, 2H).

Characterization agrees with previous reports for this compound.⁶



((4-bromophenyl)(phenylamino)methyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl)dihydroborate (**12b**): White solid (64 mg, 87% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1.

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.18 (d, $J = 8.3$ Hz, 2H), 7.02 (t, $J = 7.8$ Hz, 2H), 6.78 (d, $J = 8.3$ Hz, 2H), 6.75 (s, 2H), 6.52 (t, $J = 7.3$ Hz, 1H), 6.45 (d, $J = 8.1$ Hz, 2H), 3.69 (s, 1H), 3.41 (s, 6H), 1.68 (dd, $J = 187.8, 105.2$ Hz, 2H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 151.60, 149.88, 130.76, 128.84, 126.45, 120.51,

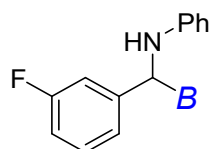
116.43, 115.41, 112.88, 35.67.

¹¹B NMR (193 MHz, CDCl₃) δ -23.04 (t, *J* = 88.5 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -23.04.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.18 (d, *J* = 8.3 Hz, 2H), 7.02 (t, *J* = 7.8 Hz, 2H), 6.78 (d, *J* = 8.2 Hz, 2H), 6.75 (s, 2H), 6.52 (t, *J* = 7.3 Hz, 1H), 6.45 (d, *J* = 8.0 Hz, 2H), 3.71 – 3.67 (m, 1H), 3.42 (s, 6H), 1.87 – 1.52 (m, 2H).

Characterization agrees with previous reports for this compound.⁶



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)((3-fluorophenyl) (phenylamino) methyl) dihydroborate (**12c**): White solid (50 mg, 81% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.03 (dd, *J* = 8.3, 7.5 Hz, 2H), 7.01 – 6.98 (m, 1H), 6.74 (s, 2H), 6.69 – 6.62 (m, 2H), 6.60 (d, *J* = 7.7 Hz, 1H), 6.52 (t, *J* = 7.2 Hz, 1H), 6.48 (d, *J* = 7.7 Hz, 2H), 3.72 (s, 1H), 3.42 (s, 6H), 2.02 – 1.35 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 163.28 (d, *J* = 244.6 Hz) 155.86 (d, *J* = 6.1 Hz), 149.97, 129.02 (d, *J* = 7.6 Hz), 128.84, 120.49, 120.03 (d, *J* = 1.5 Hz), 115.39, 112.86, 111.19 (d, *J* = 21.1 Hz), 110.19 (d, *J* = 22.7 Hz), 35.60.

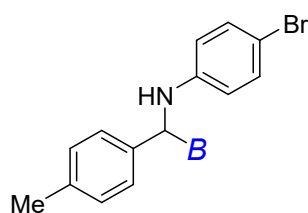
¹¹B NMR (193 MHz, CDCl₃) δ -23.09 (d, *J* = 88.4 Hz).

¹¹B{¹H} NMR (193 MHz, CDCl₃) δ -23.09.

¹H{¹¹B} NMR (600 MHz, CDCl₃) δ 7.03 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.01 – 6.98 (m, 1H), 6.74 (s, 2H), 6.69 – 6.62 (m, 2H), 6.61 (d, *J* = 7.6 Hz, 1H), 6.52 (t, *J* = 7.2 Hz, 1H), 6.48 (d, *J* = 7.7 Hz, 2H), 3.75 – 3.71 (m, 1H), 3.42 (s, 6H), 1.87 – 1.54 (m, 2H).

¹⁹F NMR (565 MHz, CDCl₃) δ -114.84.

HRMS (ESI-TOF) *m/z*: [M + Na]⁺ cacl. for C₁₈H₂₁BN₃Na 332.1710; found: 332.1702.



(((4-bromophenyl)amino)(p-tolyl)methyl)(1,3-dimethyl-1H-imidazol-3-ium-2-yl) dihydroborate (**12d**): White solid (58 mg, 76% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/1.

¹H NMR (600 MHz, CDCl₃) δ 7.05 (d, *J* = 8.8 Hz, 2H), 6.86 (d, *J* = 7.9 Hz, 2H), 6.72 (s, 2H), 6.70 (d, *J* = 7.9 Hz, 2H), 6.35 (d, *J* = 8.8 Hz, 2H), 3.65 (s, 1H), 3.36 (s, 6H), 2.23 (s, 3H), 1.67 (dd, *J* = 186.3, 100.6 Hz, 2H).

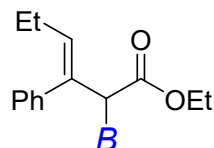
¹³C NMR (151 MHz, CDCl₃) δ 149.11, 148.06, 132.76, 131.34, 128.58, 124.39, 120.36, 114.46, 106.26, 35.57, 20.97.

^{11}B NMR (193 MHz, CDCl_3) δ -23.13 (t, J = 87.9 Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -23.13.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.05 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 7.8 Hz, 2H), 6.72 (s, 2H), 6.70 (d, J = 7.6 Hz, 2H), 6.35 (d, J = 8.7 Hz, 2H), 3.67 – 3.63 (m, 1H), 3.36 (s, 6H), 2.23 (s, 3H), 1.85 – 1.50 (m, 2H).

Characterization agrees with previous reports for this compound.⁶



(*Z*)-(1,3-dimethyl-1H-imidazol-3-ium-2-yl)(1-ethoxy-1-oxo-3-phenylhex-3-en-2-yl)dihydroborate (**14**): Colorless liquid (16 mg, 50% yield); Gradient eluent: EtOAc/petroleum ether: 1/1 to 2/1.

^1H NMR (600 MHz, CDCl_3) δ 7.22 (t, J = 7.6 Hz, 2H), 7.12 (t, J = 7.7 Hz, 3H), 6.78 (s, 2H), 5.90 (t, J = 7.3 Hz, 1H), 3.88 (q, J = 7.1 Hz, 2H), 3.69 (s, 6H), 2.89 (s, 1H), 2.02 – 1.94 (m, 2H), 1.79 – 1.56 (m, 2H), 1.03 (t, J = 7.1 Hz, 3H), 0.94 (t, J = 7.5 Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 180.02, 144.52, 141.04, 128.84, 128.60, 127.42, 125.51, 120.32, 58.88, 36.08, 22.83, 14.91, 14.34.

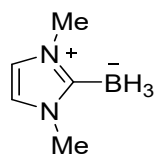
^{11}B NMR (193 MHz, CDCl_3) δ -25.25 (t, J = 89.8 Hz).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -25.25.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 7.22 (t, J = 7.6 Hz, 2H), 7.12 (t, J = 7.1 Hz, 3H), 6.78 (s, 2H), 5.90 (t, J = 7.3 Hz, 1H), 3.88 (q, J = 7.1 Hz, 2H), 3.69 (s, 6H), 2.89 (t, J = 5.9 Hz, 1H), 2.01 – 1.93 (m, 2H), 1.82 – 1.74 (m, 2H), 1.03 (t, J = 7.1 Hz, 3H), 0.93 (t, J = 7.5 Hz, 3H).

Characterization agrees with previous reports for this compound.¹²

8. Characterization data of starting materials.



(1,3-dimethyl-1H-imidazol-3-ium-2-yl)trihydroborate: White solid (5.3 g, 48% yield).

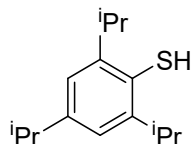
^1H NMR (600 MHz, CDCl_3) δ 6.79 (s, 2H), 3.71 (s, 6H), 0.99 (dd, J = 55.1, 27.9 Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 119.95, 35.94. ^{11}B NMR (193 MHz, CDCl_3) δ -37.49 (q, J = 85.95).

$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3) δ -37.48.

$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3) δ 6.79 (s, 2H), 3.71 (s, 6H), 0.99 (dd, J = 55.1, 27.9 Hz, 3H).

Characterization agrees with previous reports for this compound.¹

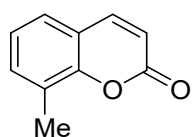


2,4,6-triisopropylbenzenethiol: Colorless liquid (3.8 g, 80% yield).

¹H NMR (600 MHz, CDCl₃) δ 7.03 (s, 2H), 3.58 – 3.50 (m, 2H), 3.10 (s, 1H), 2.90 (dq, *J* = 13.8, 6.9 Hz, 1H), 1.29 (dd, *J* = 11.1, 6.9 Hz, 19H).

¹³C NMR (151 MHz, CDCl₃) δ 148.14, 147.12, 124.33, 121.36, 34.19, 31.84, 24.09, 23.28.

Characterization agrees with previous reports for this compound.²

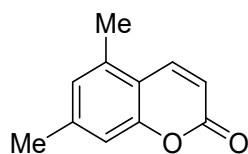


8-methyl-2H-chromen-2-one (**5-3**): White solid (800 mg, 50% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.69 (d, *J* = 9.5 Hz, 1H), 7.38 (d, *J* = 7.4 Hz, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 6.41 (d, *J* = 9.5 Hz, 1H), 2.46 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 161.06, 152.41, 143.84, 133.18, 126.37, 125.59, 124.01, 118.58, 116.33, 15.42.

Characterization agrees with previous reports for this compound.⁷

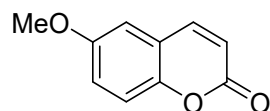


5,7-dimethyl-2H-chromen-2-one (**5-4**): White solid (835 mg, 48% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.86 (d, *J* = 10.2 Hz, 1H), 6.97 (s, 1H), 6.92 (s, 1H), 6.35 (d, *J* = 9.7 Hz, 1H), 2.47 (s, 3H), 2.39 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 161.15, 154.78, 142.76, 140.38, 135.67, 126.99, 115.34, 115.08, 114.74, 21.69, 18.20.

Characterization agrees with previous reports for this compound.⁷



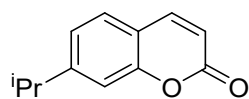
6-methoxy-2H-chromen-2-one (**5-7**): White solid (915 mg, 52% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.66 (d, *J* = 9.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.11 (dd, *J* = 9.0, 2.5 Hz, 1H), 6.92 (s, 1H), 6.43 (d, *J* = 9.5 Hz, 1H), 3.85 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 161.01, 156.10, 148.48, 143.21, 119.47, 119.18,

117.92, 117.1, 110.03, 55.86.

Characterization agrees with previous reports for this compound.⁸

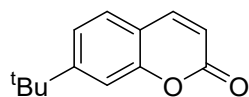


7-isopropyl-2H-chromen-2-one (**5-9**): White solid (564 mg, 30% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.66 (d, *J* = 9.5 Hz, 1H), 7.38 (d, *J* = 7.9 Hz, 1H), 7.18 – 7.11 (m, 2H), 6.33 (d, *J* = 9.5 Hz, 1H), 2.97 (dt, *J* = 13.7, 6.8 Hz, 1H), 1.27 (s, 3H), 1.26 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 161.16, 154.32, 154.04, 143.39, 127.70, 123.13, 116.79, 115.52, 114.49, 34.30, 23.65.

Characterization agrees with previous reports for this compound.⁷

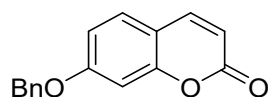


7-(tert-butyl)-2H-chromen-2-one (**5-10**): Yellow solid (707 mg, 35% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.68 (d, *J* = 9.5 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 1H), 7.34 (d, *J* = 1.5 Hz, 1H), 7.32 (dd, *J* = 8.1, 1.7 Hz, 1H), 6.36 (d, *J* = 9.5 Hz, 1H), 1.35 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 161.23, 156.41, 154.14, 143.23, 127.33, 121.98, 116.41, 115.74, 113.75, 35.30, 31.07.

Characterization agrees with previous reports for this compound.⁷

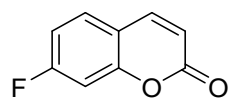


7-(benzyloxy)-2H-chromen-2-one (**5-12**): Yellow solid (1600 mg, 63% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.62 (d, *J* = 9.5 Hz, 1H), 7.44 – 7.35 (m, 6H), 6.91 (dd, *J* = 8.6, 2.4 Hz, 1H), 6.87 (d, *J* = 2.3 Hz, 1H), 6.23 (d, *J* = 9.5 Hz, 1H), 5.11 (s, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 161.89, 161.21, 155.82, 143.43, 135.80, 128.85, 128.79, 128.41, 127.55, 126.98, 113.25, 112.76, 101.93, 70.53.

Characterization agrees with previous reports for this compound.⁷



7-fluoro-2H-chromen-2-one (**5-13**): Yellow solid (705 mg, 43% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

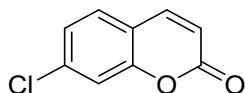
¹H NMR (600 MHz, CDCl₃) δ 7.68 (d, *J* = 9.6 Hz, 1H), 7.47 (dd, *J* = 8.6, 6.0 Hz, 1H),

7.07 – 7.00 (m, 2H), 6.37 (d, $J = 9.6$ Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 164.55 (d, $J = 253.7$ Hz), 160.25, 155.29 (d, $J = 12.1$ Hz), 142.86, 129.41 (d, $J = 10.6$ Hz), 115.57 (d, $J = 3.0$ Hz), 115.46 (d, $J = 4.5$ Hz), 112.58 (d, $J = 24.2$ Hz) 104.65 (d, $J = 25.7$ Hz).

^{19}F NMR (565 MHz, CDCl_3) δ -104.97.

Characterization agrees with previous reports for this compound.⁸

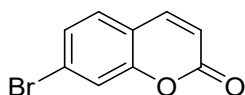


7-chloro-2H-chromen-2-one (**5-14**): Yellow solid (540 mg, 30% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

^1H NMR (600 MHz, CDCl_3) δ 7.67 (d, $J = 9.6$ Hz, 1H), 7.42 (d, $J = 8.3$ Hz, 1H), 7.35 (d, $J = 1.8$ Hz, 1H), 7.27 (d, $J = 2.0$ Hz, 1H), 7.26 (s, 1H), 6.42 (d, $J = 9.6$ Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 159.96, 154.38, 142.64, 137.85, 128.64, 125.06, 117.42, 117.29, 116.67.

Characterization agrees with previous reports for this compound.⁷

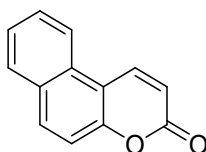


7-bromo-2H-chromen-2-one (**5-15**): Yellow solid (780 mg, 35% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

^1H NMR (600 MHz, CDCl_3) δ 7.66 (d, $J = 9.6$ Hz, 1H), 7.49 (d, $J = 1.7$ Hz, 1H), 7.40 (dd, $J = 8.2, 1.8$ Hz, 1H), 7.34 (d, $J = 8.3$ Hz, 1H), 6.42 (d, $J = 9.6$ Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 159.93, 154.28, 142.79, 128.81, 127.91, 125.82, 120.20, 117.76, 116.88.

Characterization agrees with previous reports for this compound.⁷

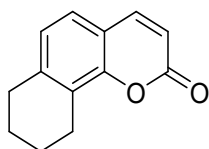


3H-benzo[f]chromen-3-one (**5-16**): Yellow solid (784 mg, 40% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

^1H NMR (600 MHz, CDCl_3) δ 8.49 (d, $J = 9.7$ Hz, 1H), 8.22 (d, $J = 8.4$ Hz, 1H), 7.98 (d, $J = 9.0$ Hz, 1H), 7.91 (d, $J = 8.1$ Hz, 1H), 7.69 (t, $J = 7.7$ Hz, 1H), 7.57 (t, $J = 7.5$ Hz, 1H), 7.46 (d, $J = 9.0$ Hz, 1H), 6.58 (d, $J = 9.7$ Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 160.93, 153.91, 139.11, 133.15, 130.30, 129.04, 128.31, 126.08, 121.37, 117.10, 115.68, 113.01.

Characterization agrees with previous reports for this compound.⁷

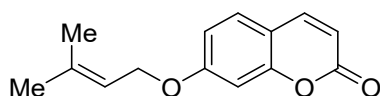


7,8,9,10-tetrahydro-2H-benzo[h]chromen-2-one (**5-17**): Yellow solid (700 mg, 35% yield); Gradient eluent: EtOAc/petroleum ether: 1/20 to 1/10.

¹H NMR (600 MHz, CDCl₃) δ 7.64 (d, *J* = 9.4 Hz, 1H), 7.19 (d, *J* = 7.9 Hz, 1H), 6.99 (d, *J* = 7.9 Hz, 1H), 6.32 (d, *J* = 9.5 Hz, 1H), 2.89 (s, 2H), 2.83 (t, *J* = 5.4 Hz, 2H), 1.86 – 1.79 (m, 4H).

¹³C NMR (151 MHz, CDCl₃) δ 161.40, 152.20, 143.95, 142.49, 125.51, 125.29, 124.38, 116.12, 114.93, 29.94, 22.51, 22.46, 22.07.

Characterization agrees with previous reports for this compound.⁷

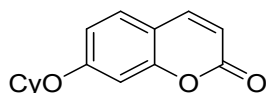


7-((3-methylbut-2-en-1-yl)oxy)-2H-chromen-2-one (**5-18**): White solid (1600 mg, 70% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.62 (d, *J* = 9.5 Hz, 1H), 7.35 (d, *J* = 8.6 Hz, 1H), 6.84 (dd, *J* = 8.6, 2.2 Hz, 1H), 6.81 (s, 1H), 6.23 (d, *J* = 9.4 Hz, 1H), 5.46 (t, *J* = 6.7 Hz, 1H), 4.57 (d, *J* = 6.7 Hz, 2H), 1.80 (s, 3H), 1.76 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 162.14, 161.30, 155.88, 143.47, 139.30, 128.71, 118.64, 113.22, 112.97, 112.44, 101.57, 65.44, 25.83, 18.30.

Characterization agrees with previous reports for this compound.⁷

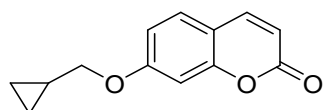


7-(cyclohexyloxy)-2H-chromen-2-one (**5-19**): White solid (854 mg, 35% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, DMSO) δ 7.98 (d, *J* = 9.5 Hz, 1H), 7.60 (d, *J* = 8.6 Hz, 1H), 6.99 (d, *J* = 2.3 Hz, 1H), 6.94 (dd, *J* = 8.6, 2.4 Hz, 1H), 6.27 (d, *J* = 9.5 Hz, 1H), 4.50 (td, *J* = 8.5, 3.9 Hz, 1H), 1.98 – 1.92 (m, 2H), 1.71 (dd, *J* = 8.9, 4.1 Hz, 2H), 1.58 – 1.51 (m, 1H), 1.49 – 1.37 (m, 4H), 1.27 (tt, *J* = 16.2, 8.3 Hz, 1H).

¹³C NMR (151 MHz, DMSO) δ 161.10, 160.82, 155.96, 144.80, 130.02, 113.91, 112.75, 112.60, 102.52, 75.52, 31.55, 25.46, 23.54.

Characterization agrees with previous reports for this compound.⁷



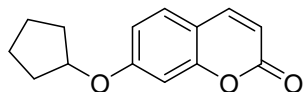
7-(cyclopropylmethoxy)-2H-chromen-2-one (**5-20**): Colorless liquid (1663 mg, 77% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.61 (d, *J* = 9.5 Hz, 1H), 7.35 (d, *J* = 8.6 Hz, 1H), 6.83

(dd, $J = 8.6, 2.4$ Hz, 1H), 6.77 (d, $J = 2.3$ Hz, 1H), 6.22 (d, $J = 9.4$ Hz, 1H), 3.84 (d, $J = 7.0$ Hz, 2H), 1.28 (tt, $J = 10.2, 5.1$ Hz, 1H), 0.68 – 0.64 (m, 2H), 0.37 (q, $J = 4.9$ Hz, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 162.26, 161.28, 155.89, 143.47, 128.75, 113.00, 122.97, 112.44, 101.43, 73.34, 9.99, 3.31.

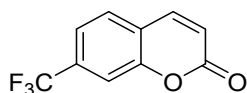
Characterization agrees with previous reports for this compound.⁷



7-(cyclopentyloxy)-2H-chromen-2-one (**5-21**): Colorless liquid (1725 mg, 75% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

^1H NMR (600 MHz, CDCl_3) δ 7.61 (d, $J = 9.4$ Hz, 1H), 7.33 (d, $J = 8.4$ Hz, 1H), 6.80 – 6.74 (m, 2H), 6.20 (d, $J = 9.4$ Hz, 1H), 4.79 – 4.75 (m, 1H), 1.93 (dt, $J = 12.3, 6.3$ Hz, 2H), 1.88 – 1.82 (m, 2H), 1.81 – 1.74 (m, 2H), 1.67 – 1.59 (m, 2H).

^{13}C NMR (151 MHz, CDCl_3) δ 161.52, 161.36, 155.86, 143.53, 128.69, 113.79, 112.70, 112.13, 102.25, 80.21, 32.80, 24.07.



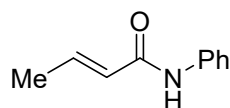
7-(trifluoromethyl)-2H-chromen-2-one (**5-27**): Colorless liquid (50 mg, 23% yield); Gradient eluent: EtOAc/petroleum ether: 1/10 to 1/5.

^1H NMR (600 MHz, CDCl_3) δ 7.75 (d, $J = 9.6$ Hz, 1H), 7.62 (d, $J = 8.0$ Hz, 1H), 7.56 (s, 1H), 7.52 (d, $J = 8.1$ Hz, 1H), 6.53 (d, $J = 9.6$ Hz, 1H).

^{13}C NMR (151 MHz, CDCl_3) δ 159.60, 153.69, 142.25, 133.47 (q, $J = 33.2$ Hz), 128.65, 123.15 (q, $J = 91.1$ Hz), 121.36, 121.04 (q, $J = 3.5$ Hz), 118.97, 114.32 (q, $J = 4.0$ Hz).

^{19}F NMR (565 MHz, CDCl_3) δ -62.92.

Characterization agrees with previous reports for this compound.¹⁵

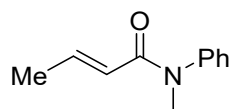


(*E*)-N-phenylbut-2-enamide (**11-3**): White solid (362 mg, 75% yield); Gradient eluent: EtOAc/petroleum ether: 1/5 to 1/4.

^1H NMR (600 MHz, CDCl_3) δ 8.12 (s, 1H), 7.59 (d, $J = 7.3$ Hz, 2H), 7.28 (t, $J = 7.9$ Hz, 2H), 7.08 (t, $J = 7.3$ Hz, 1H), 6.95 (dq, $J = 13.8, 6.9$ Hz, 1H), 6.02 (dd, $J = 15.1, 1.6$ Hz, 1H), 1.83 (dd, $J = 6.9, 1.6$ Hz, 3H).

^{13}C NMR (151 MHz, CDCl_3) δ 164.59, 141.34, 138.23, 128.92, 125.66, 124.23, 120.26, 17.82.

Characterization agrees with previous reports for this compound.⁹

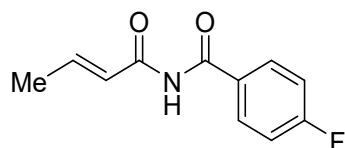


(*E*)-*N*-methyl-*N*-phenylbut-2-enamide (**11-4**): Colorless liquid (315 mg, 60% yield); Gradient eluent: EtOAc/petroleum ether: 1/5 to 1/4.

¹H NMR (600 MHz, CDCl₃) δ 7.37 (t, *J* = 7.7 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 7.3 Hz, 2H), 6.88 (dq, *J* = 13.8, 6.9 Hz, 1H), 5.71 (d, *J* = 15.0 Hz, 1H), 3.29 (s, 3H), 1.68 (dd, *J* = 6.9, 1.5 Hz, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 166.15, 143.72, 141.15, 129.52, 127.42, 127.34, 122.72, 37.35, 17.92.

Characterization agrees with previous reports for this compound.¹⁰

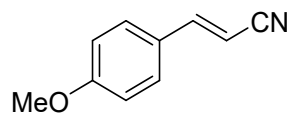


(*E*)-*N*-(but-2-enoyl)-4-fluorobenzamide (**11-5**): White solid (267 mg, 64% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 8.80 (s, 1H), 7.95 – 7.90 (m, 2H), 7.24 – 7.16 (m, 3H), 7.13 (dd, *J* = 15.3, 1.5 Hz, 1H), 1.99 (dd, *J* = 6.8, 1.5 Hz, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 167.47, 165.74 (d, *J* = 255.2 Hz), 164.83, 147.4, 130.46 (d, *J* = 9.1 Hz), 129.22 (d, *J* = 3.0 Hz), 124.07, 116.14 (d, *J* = 22.7 Hz), 18.61. ¹⁹F NMR (565 MHz, CDCl₃) δ -104.88.

Characterization agrees with previous reports for this compound.⁶

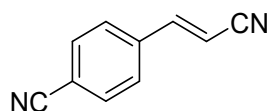


(*E*)-3-(4-methoxyphenyl)acrylonitrile (**9-2**): Colorless liquid (127 mg, 40% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.38 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 16.6 Hz, 1H), 6.90 (d, *J* = 8.8 Hz, 2H), 5.70 (d, *J* = 16.6 Hz, 1H), 3.83 (s, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 162.08, 150.05, 129.13, 126.36, 118.77, 114.55, 93.34, 55.48.

Characterization agrees with previous reports for this compound.¹¹



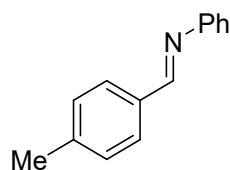
(*E*)-4-(2-cyanovinyl)benzonitrile (**9-3**): White solid (93 mg, 30% yield); Gradient eluent: EtOAc/petroleum ether: 1/3 to 1/2.

¹H NMR (600 MHz, CDCl₃) δ 7.71 (d, *J* = 8.3 Hz, 2H), 7.56 (d, *J* = 8.3 Hz, 2H), 7.41 (d, *J* = 16.7 Hz, 1H), 6.01 (d, *J* = 16.7 Hz, 1H).

¹³C NMR (151 MHz, CDCl₃) δ 148.26, 137.47, 132.89, 127.82, 117.98, 117.13,

114.46, 100.33.

Characterization agrees with previous reports for this compound.¹¹

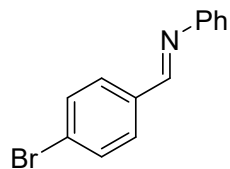


(*E*)-*N*-phenyl-1-(*p*-tolyl)methanimine (**12-1**): White solid (585 mg, 30% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.43 (s, 1H), 7.80 (d, *J* = 8.1 Hz, 2H), 7.42 – 7.36 (m, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 7.25 – 7.19 (m, 3H), 2.43 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 160.40, 152.28, 141.89, 133.69, 129.53, 129.13, 128.83, 125.76, 120.89, 21.65.

Characterization agrees with previous reports for this compound.⁶

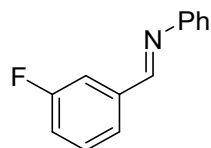


(*E*)-1-(4-bromophenyl)-*N*-phenylmethanimine (**12-2**): White solid (1032 mg, 40% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.40 (s, 1H), 7.80 – 7.74 (m, 2H), 7.62 – 7.57 (m, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.25 (dd, *J* = 6.5, 1.7 Hz, 1H), 7.21 (dd, *J* = 7.4, 1.0 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 158.94, 151.68, 135.14, 132.06, 130.17, 129.23, 126.26, 125.91, 120.87.

Characterization agrees with previous reports for this compound.⁶



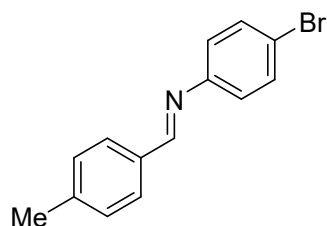
(*E*)-1-(3-fluorophenyl)-*N*-phenylmethanimine (**12-3**): White solid (756 mg, 38% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.43 (d, *J* = 1.2 Hz, 1H), 7.69 – 7.65 (m, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.47 – 7.37 (m, 3H), 7.27 – 7.15 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 163.14 (d, *J* = 248.5 Hz), 158.86 (d, *J* = 3.0 Hz), 151.54, 138.55 (d, *J* = 7.1 Hz), 130.32 (d, *J* = 8.1 Hz), 129.23, 126.34, 125.01 (d, *J* = 3.0 Hz), 120.89, 118.33 (d, *J* = 22.2 Hz), 114.70 (d, *J* = 22.2 Hz).

¹⁹F NMR (565 MHz, CDCl₃) δ -112.52.

HRMS (ESI-TOF) *m/z*: [M + H]⁺ cacl. for C₁₃H₁₁FN 200.0870; found: 200.0872.

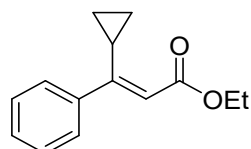


(*E*)-*N*-(4-bromophenyl)-1-(*p*-tolyl)methanimine (**12-4**): White solid (819 mg, 30% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 8.39 (s, 1H), 7.78 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.5$ Hz, 2H), 7.28 (d, $J = 7.9$ Hz, 2H), 7.08 (d, $J = 8.5$ Hz, 2H), 2.42 (s, 3H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 160.74, 151.23, 142.24, 133.42, 132.17, 129.60, 128.92, 122.62, 119.10, 21.68.

Characterization agrees with previous reports for this compound.⁶



Ethyl (*E*)-3-cyclopropyl-3-phenylacrylate (**13**): Colorless liquid (22 mg, 10% yield).

$^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.32 – 7.28 (m, 3H), 7.16 – 7.11 (m, 2H), 5.79 (s, 1H), 4.22 (q, $J = 7.1$ Hz, 2H), 3.17 – 3.08 (m, 1H), 1.31 (t, $J = 7.1$ Hz, 3H), 0.91 – 0.86 (m, 2H), 0.50 – 0.43 (m, 2H).

$^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 166.95, 163.18, 138.85, 128.04, 127.76, 127.73, 118.79, 59.78, 14.36, 13.51, 6.91.

9. The crystallographic data

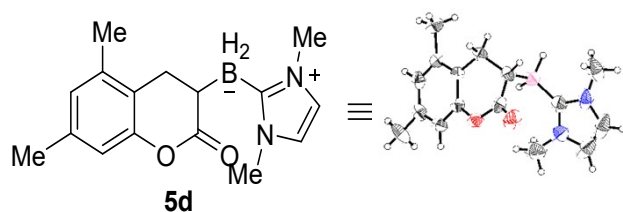


Figure S12. The single-crystal structure of **5d**

Table S2

Empirical formula	$\text{C}_{16}\text{H}_{21}\text{BN}_2\text{O}_2$
Formula weight	284.16
Temperature/K	293(2)
Crystal system	Triclinic
Space group	P-1
$a/\text{\AA}$	9.0836(6)
$b/\text{\AA}$	9.1558(8)
$c/\text{\AA}$	11.0863(8)

$\alpha/^\circ$	114.228(8)
$\beta/^\circ$	106.840(6)
$\gamma/^\circ$	93.503(6)
Volume/ \AA^3	787.46(12)
Z	2
$\rho_{\text{calc}}/\text{g}/\text{cm}^3$	1.198
μ/mm^{-1}	0.622
F(000)	304.0
Crystal size/ mm^3	$0.05 \times 0.02 \times 0.02$
Radiation	Cu K α ($\lambda = 1.54184$)
Index ranges	$-10 \leq h \leq 11, -11 \leq k \leq 11, -13 \leq l \leq 13$
2 Θ range for data collection/ $^\circ$	9.324 to 143.132
Reflections collected	5210
Independent reflections	2990 [$R_{\text{int}} = 0.0219, R_{\text{sigma}} = 0.0332$]
Data/restraints/parameters	2990/0/202
Goodness-of-fit on F^2	1.058
Final R indexes [$I \geq 2\sigma(I)$][a]	$R_1 = 0.0514, wR_2 = 0.1436$
Final R indexes [all data]	$R_1 = 0.0670, wR_2 = 0.1538$
Largest diff. peak/hole / $e \text{\AA}^{-3}$	0.19/-0.19

$$[a]R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}; wR_2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}^{1/2}$$

Table S3

Bond Lengths			Bond Angles			
C7	C6	1.504(2)	C9	C8	C7	113.11(17)
C8	C7	1.533(3)	C7	C8	B11	109.51(17)
C9	C8	1.466(3)	C9	C8	B11	108.62(16)
C8	B11	1.675(3)	C6	C7	C8	110.97(15)
C12	B11	1.601(3)	C12	B11	C8	111.82(17)

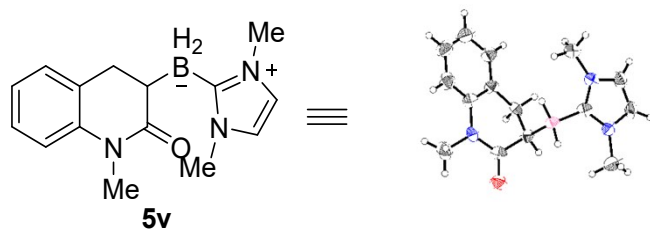


Figure S13. The single-crystal structure of **5v**

Table S4

Empirical formula	$\text{C}_{15}\text{H}_{20}\text{BN}_3\text{O}$
Formula weight	269.15
Temperature/K	293(2)
Crystal system	orthorhombic

Space group	P2 ₁ 2 ₁ 2 ₁
a/Å	6.2427(5)
b/Å	7.8871(6)
c/Å	29.178(2)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	1436.64(19)
Z	4
ρ _{calc} /g/cm ³	1.244
μ/mm ⁻¹	0.620
F(000)	576.0
Crystal size/mm ³	0.01 × 0.01 × 0.01
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	11.622 to 143.074
Index ranges	-7 ≤ h ≤ 4, -9 ≤ k ≤ 8, -31 ≤ l ≤ 35
Reflections collected	3502
Independent reflections	2428 [R _{int} = 0.0312, R _{sigma} = 0.0526]
Data/restraints/parameters	2428/0/192
Goodness-of-fit on F ²	1.096
Final R indexes [I ≥ 2σ (I)] ^[a]	R ₁ = 0.0452, wR ₂ = 0.1025
Final R indexes [all data]	R ₁ = 0.0650, wR ₂ = 0.1264
Largest diff. peak/hole / e Å ⁻³	0.21/-0.25
Flack parameter	0.5

[a] $R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$; $wR_2 = \left\{ \frac{\sum [w(F_o - F_c)^2]}{\sum [w(F_o)^2]} \right\}^{1/2}$

Table S5

Bond Lengths			Bond Angles			
C4	C3	1.506(5)	C4	C3	C2	110.6(3)
C3	C2	1.540(5)	C1	C2	C3	110.8(4)
C2	C1	1.488(6)	C3	C2	B11	113.4(3)
C2	B11	1.671(7)	C1	C2	B11	111.3(3)
C12	B11	1.612(7)	C12	B11	C2	106.9(4)

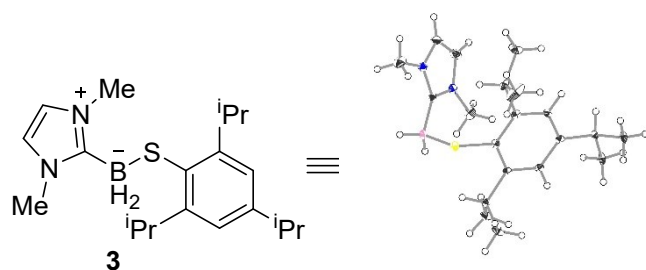


Figure S14. The single-crystal structure of **3**

Table S6

Empirical formula	C ₂₀ H ₃₃ BN ₂ S
Formula weight	344.35
Temperature/K	100.01(10)
Crystal system	Monoclinic
Space group	P2 ₁ /n
a/Å	7.8039(2)
b/Å	17.1774(4)
c/Å	14.8916(3)
α/°	90
β/°	91.570(2)
γ/°	90
Volume/Å ³	1995.48(8)
Z	4
ρ _{calc} /g/cm ³	1.146
μ/mm ⁻¹	1.439
F(000)	752.0
Crystal size/mm ³	0.01 × 0.01 × 0.01
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	7.86 to 142.88
Index ranges	-9 ≤ h ≤ 5, -20 ≤ k ≤ 20, -18 ≤ l ≤ 17
Reflections collected	9265
Independent reflections	3794 [R _{int} = 0.0324, R _{sigma} = 0.0320]
Data/restraints/parameters	3794/0/225
Goodness-of-fit on F ²	1.135
Final R indexes [I ≥ 2σ (I)] ^[a]	R ₁ = 0.0505, wR ₂ = 0.1330
Final R indexes [all data]	R ₁ = 0.0532, wR ₂ = 0.1347
Largest diff. peak/hole / e Å ⁻³	0.51/-0.46

$$[a]R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}; wR_2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}^{1/2}$$

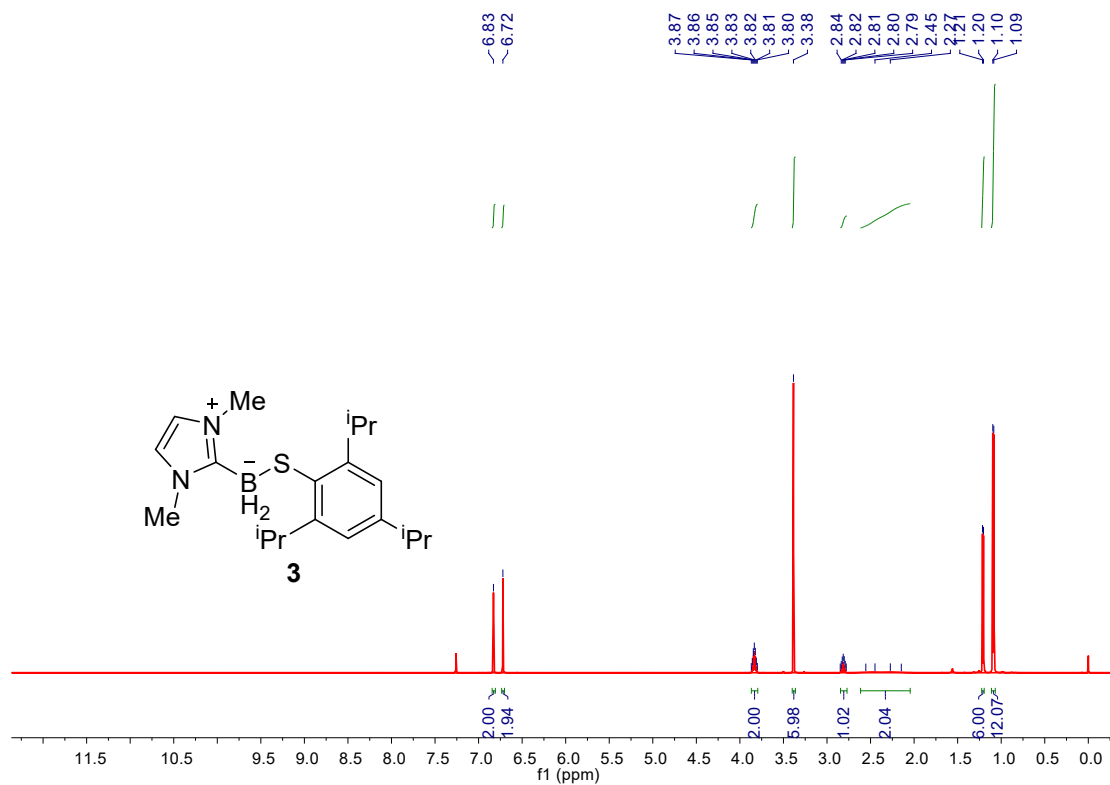
Table S7

Bond Lengths			Bond Angles			
C9	B8	1.606(3)	C9	B8	S7	109.23(14)
S7	B8	1.947(2)	C1	S7	B8	102.53(9)
S7	C1	1.791(2)	C2	C1	S7	119.36(15)
			C6	C1	S7	120.70(15)

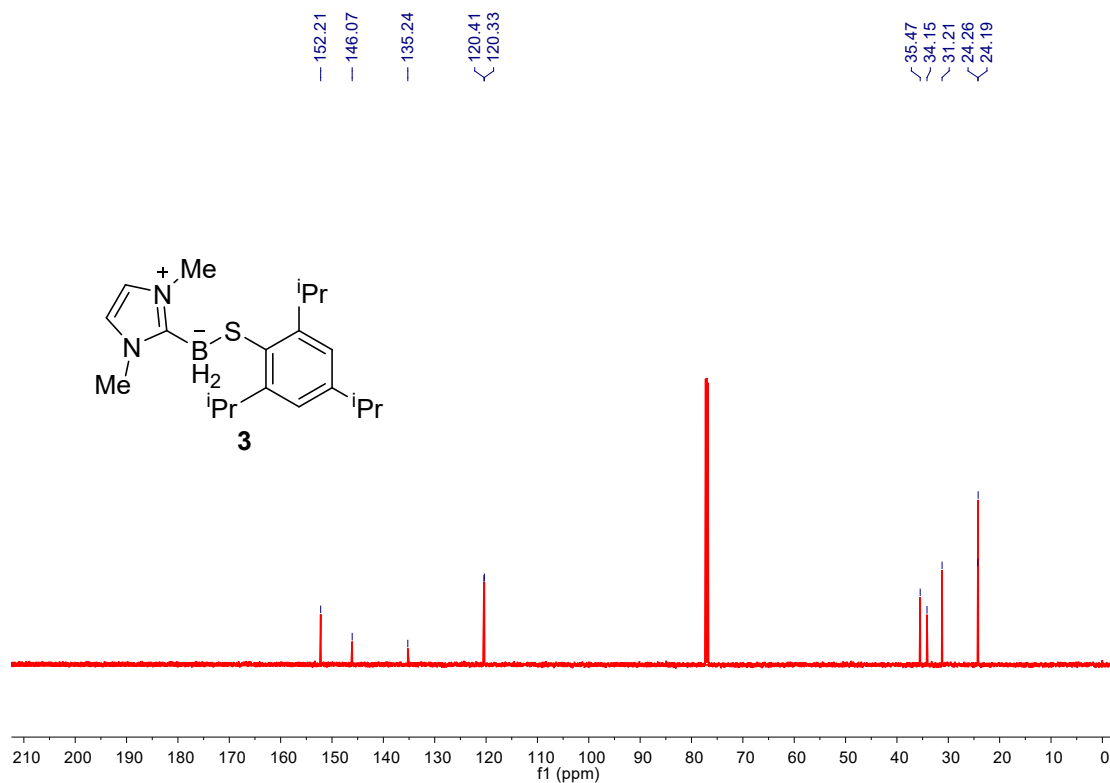
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11. NMR Spectra

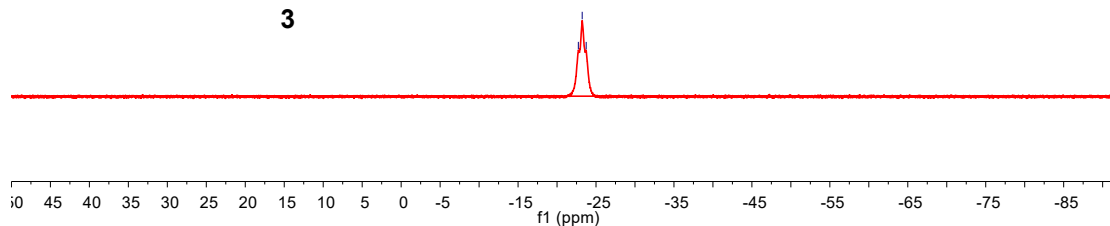
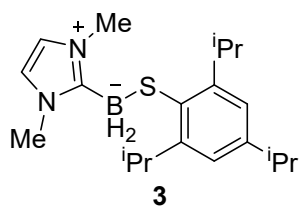


¹H NMR (600 MHz, CDCl₃)



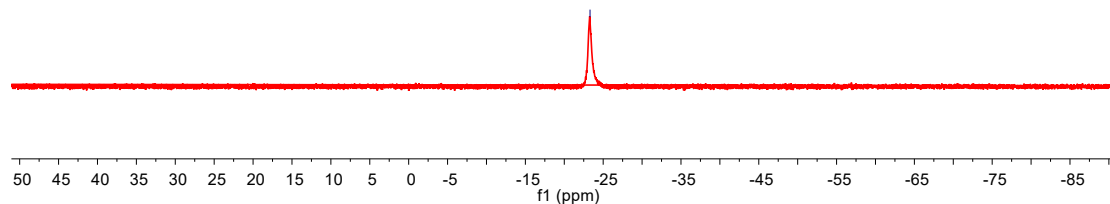
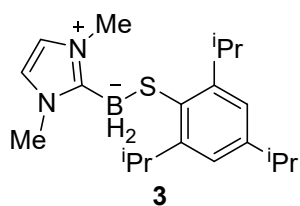
¹³C NMR (151 MHz, CDCl₃)

-22.73
-23.23
-23.74

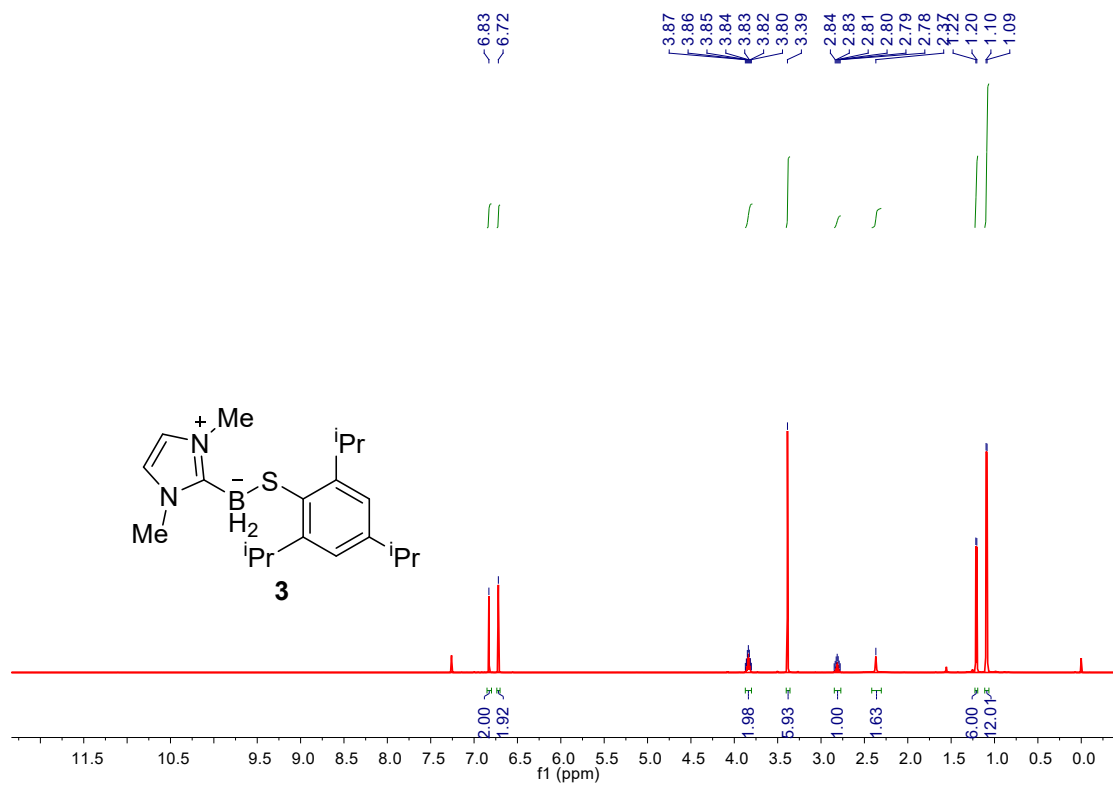


^{11}B NMR (193 MHz, CDCl_3)

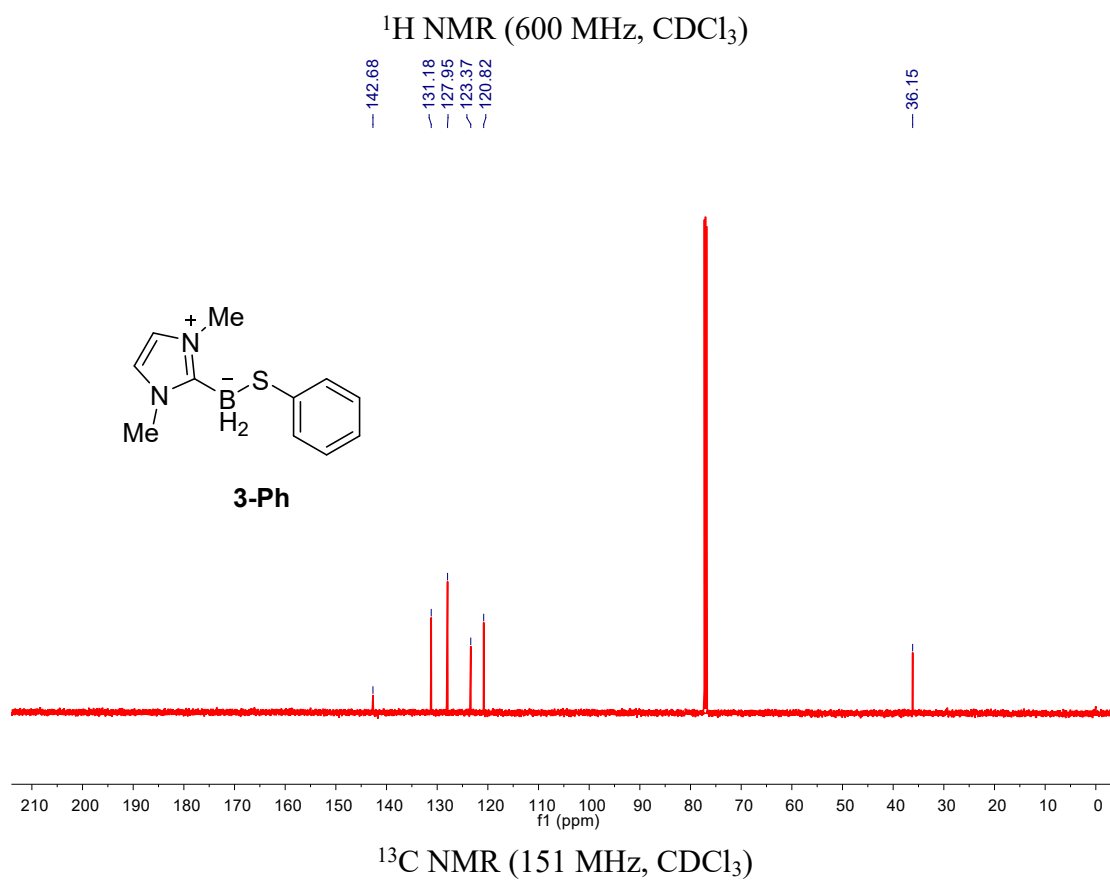
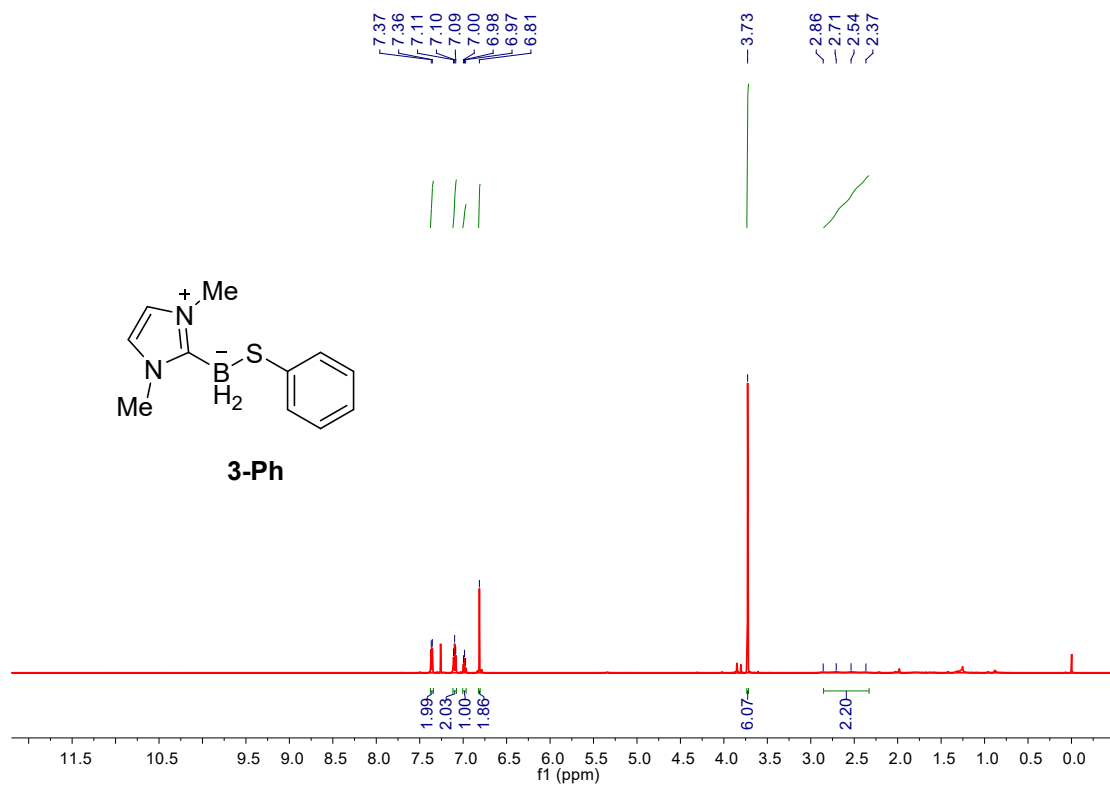
-23.31



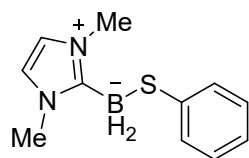
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



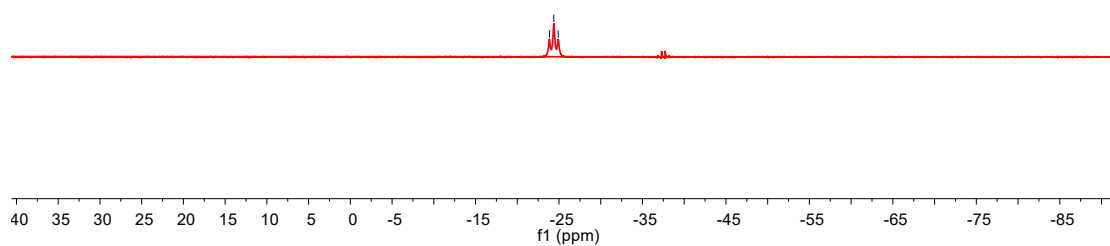
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



-23.86
-24.38
-24.91

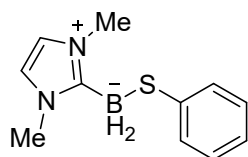


3-Ph

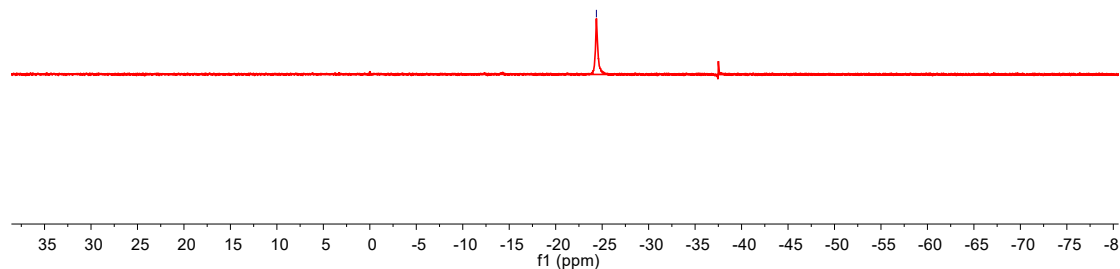


^{11}B NMR (193 MHz, CDCl_3)

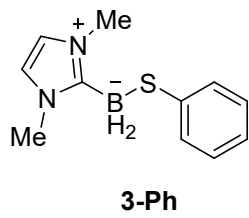
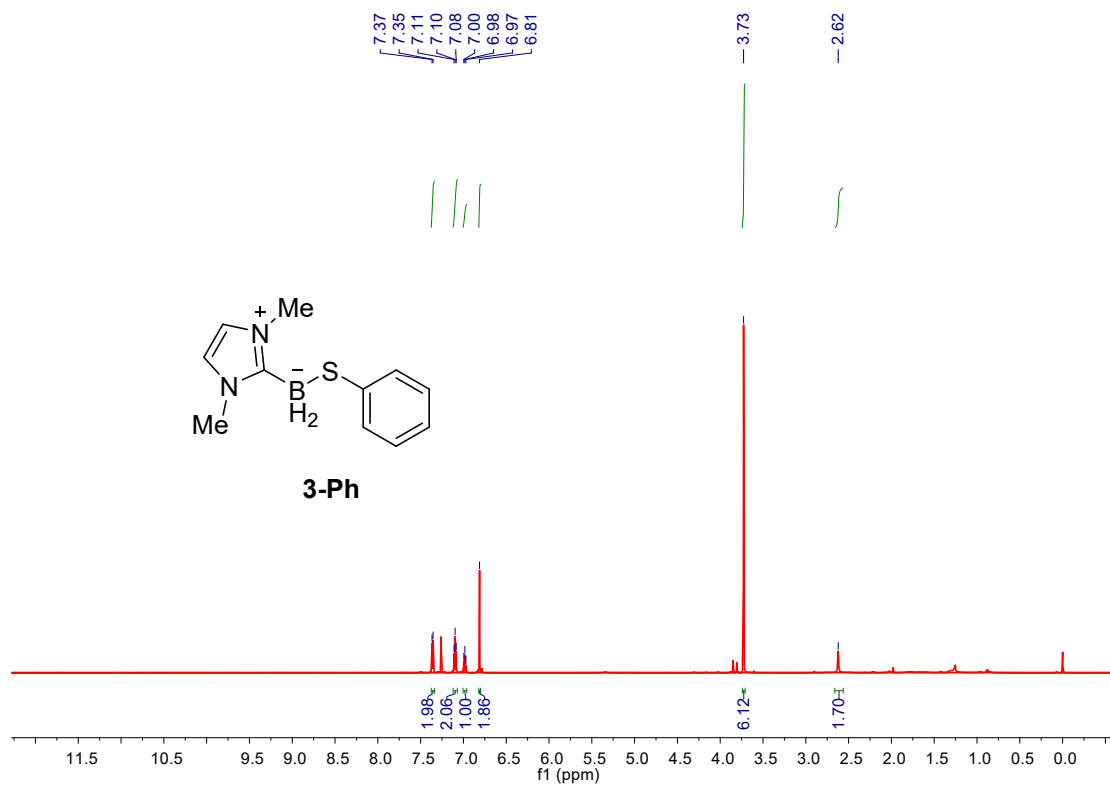
-24.38



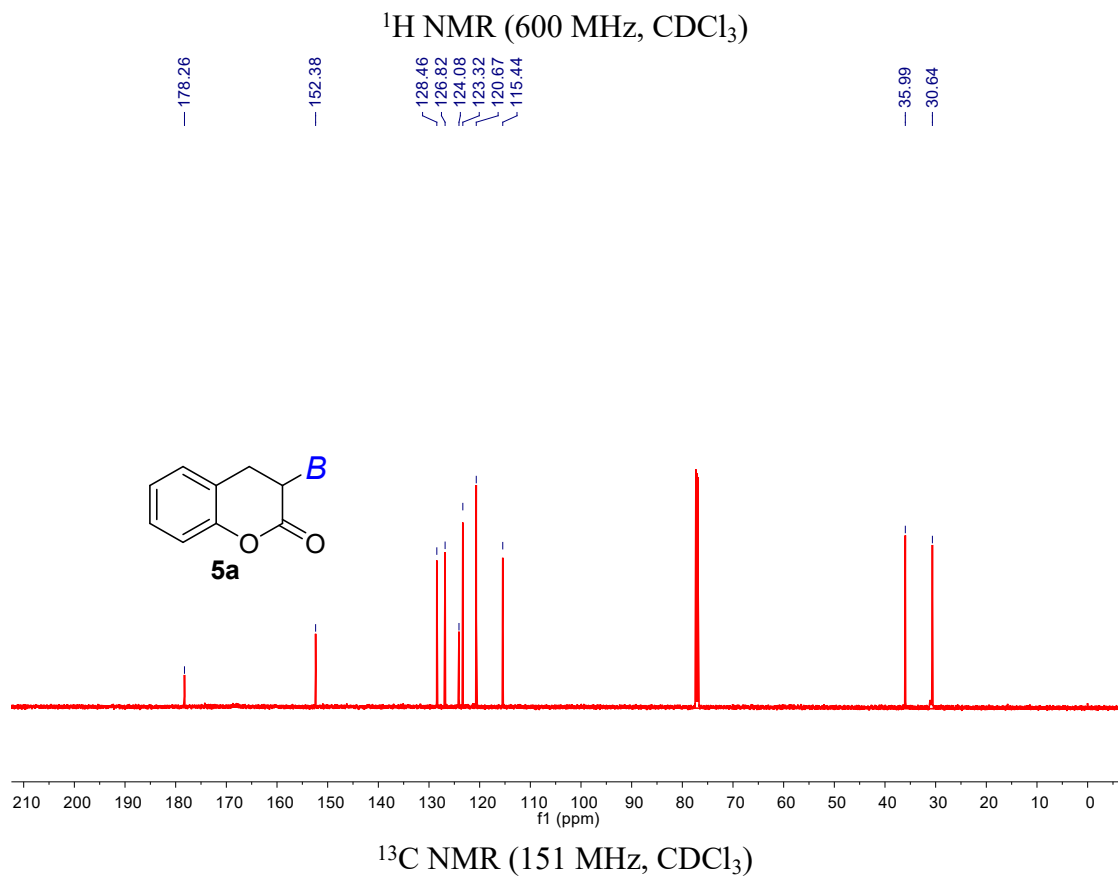
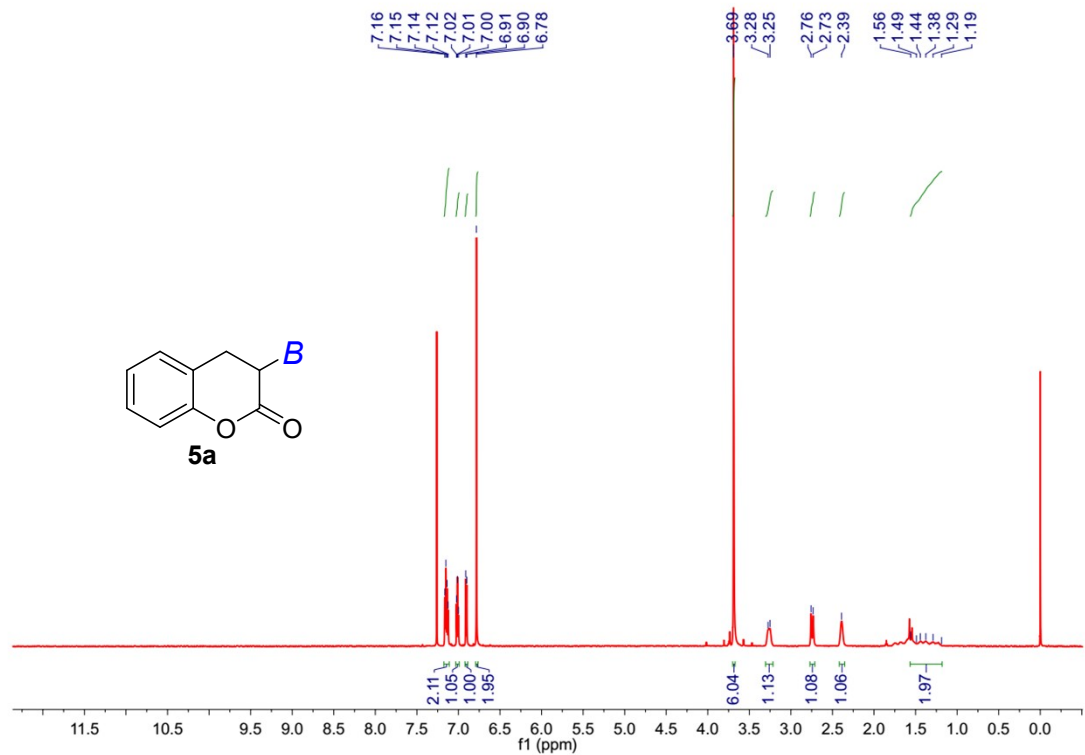
3-Ph

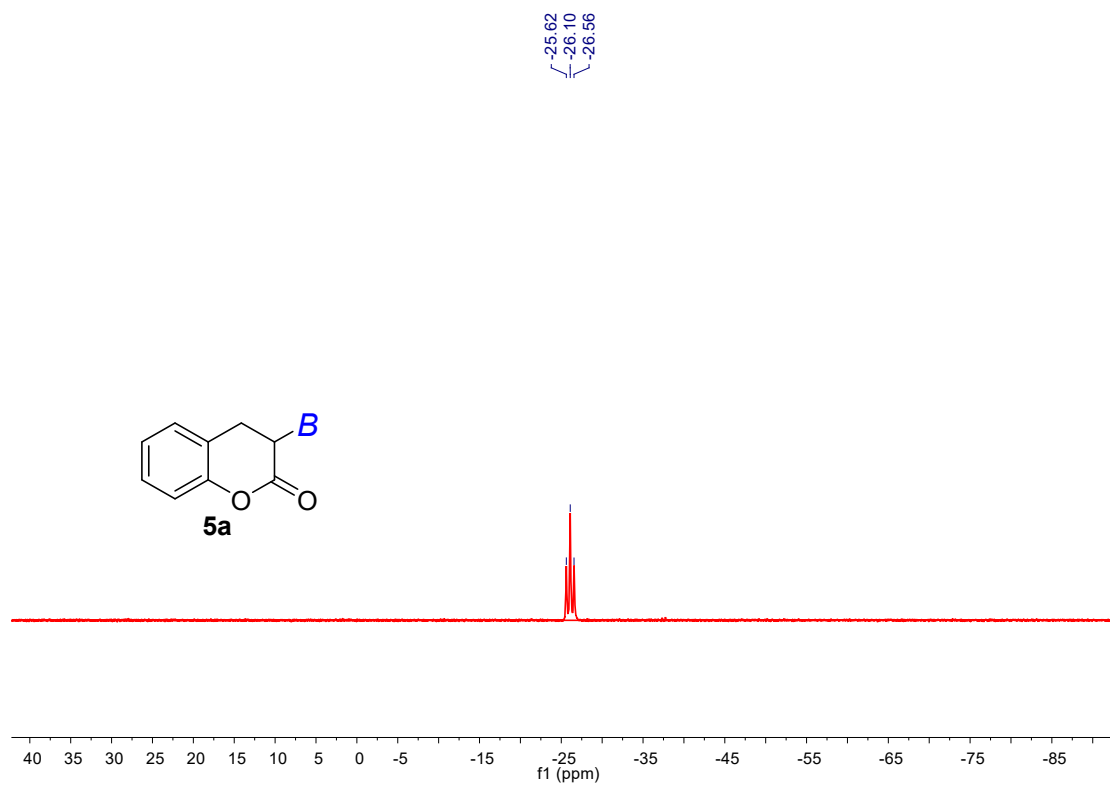


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

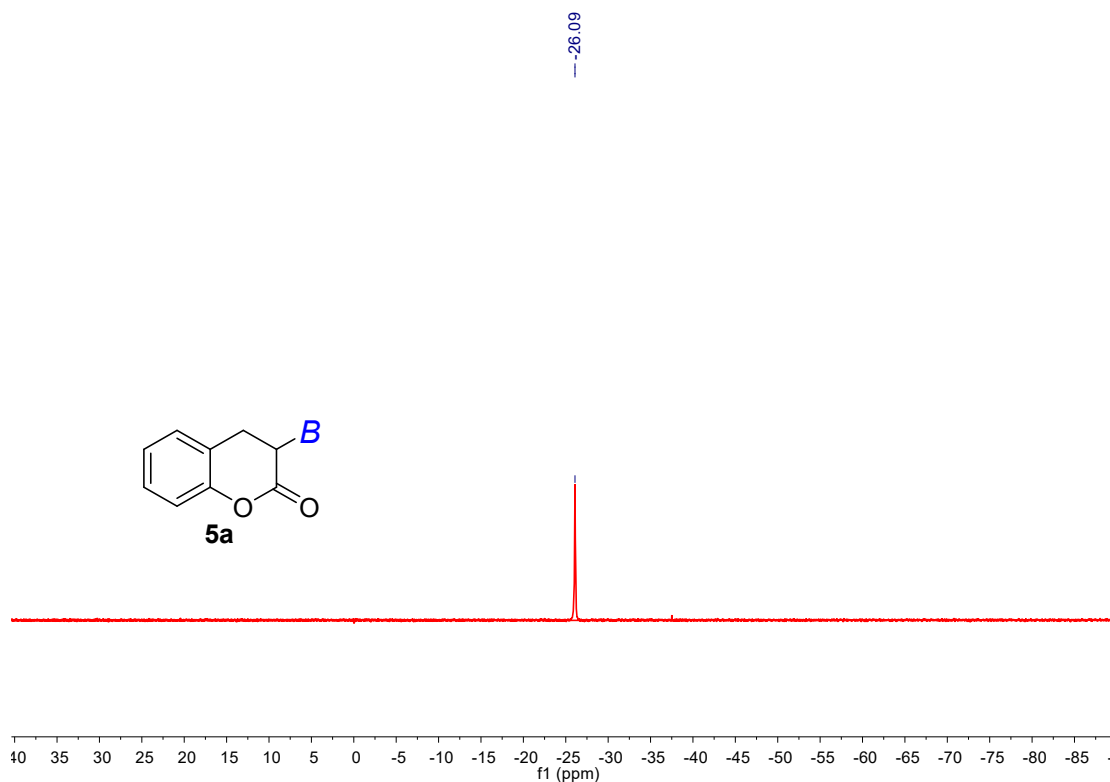


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

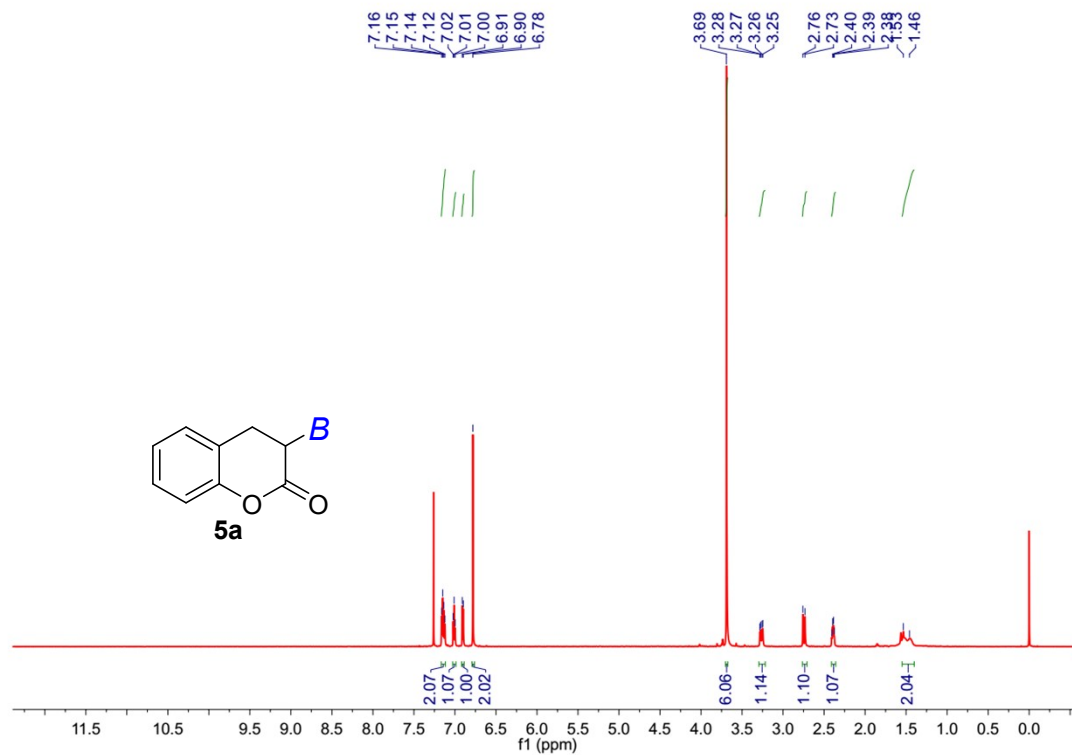




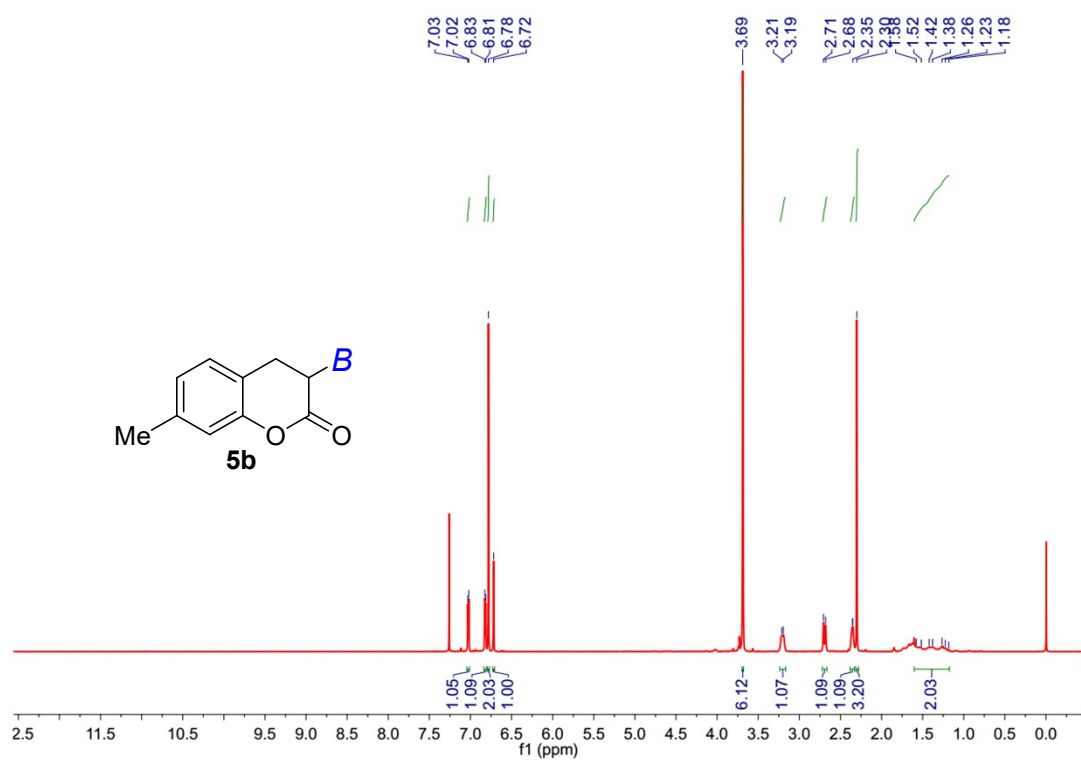
^{11}B NMR (193 MHz, CDCl_3)



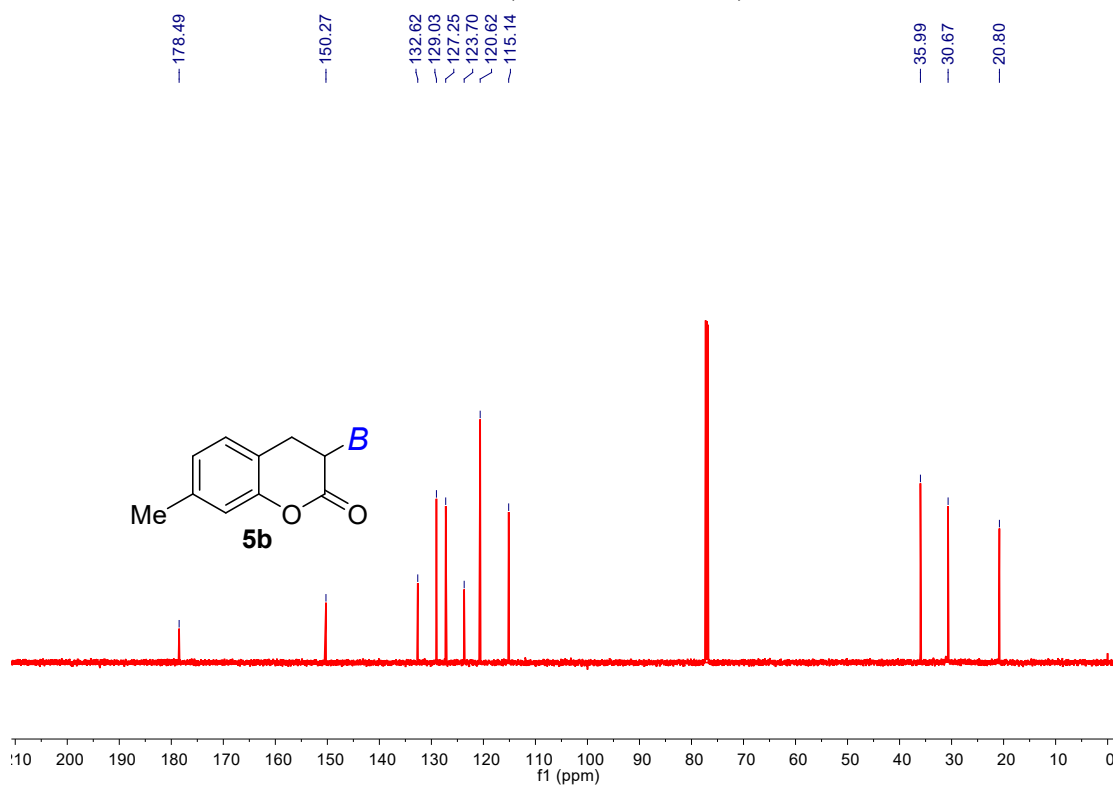
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



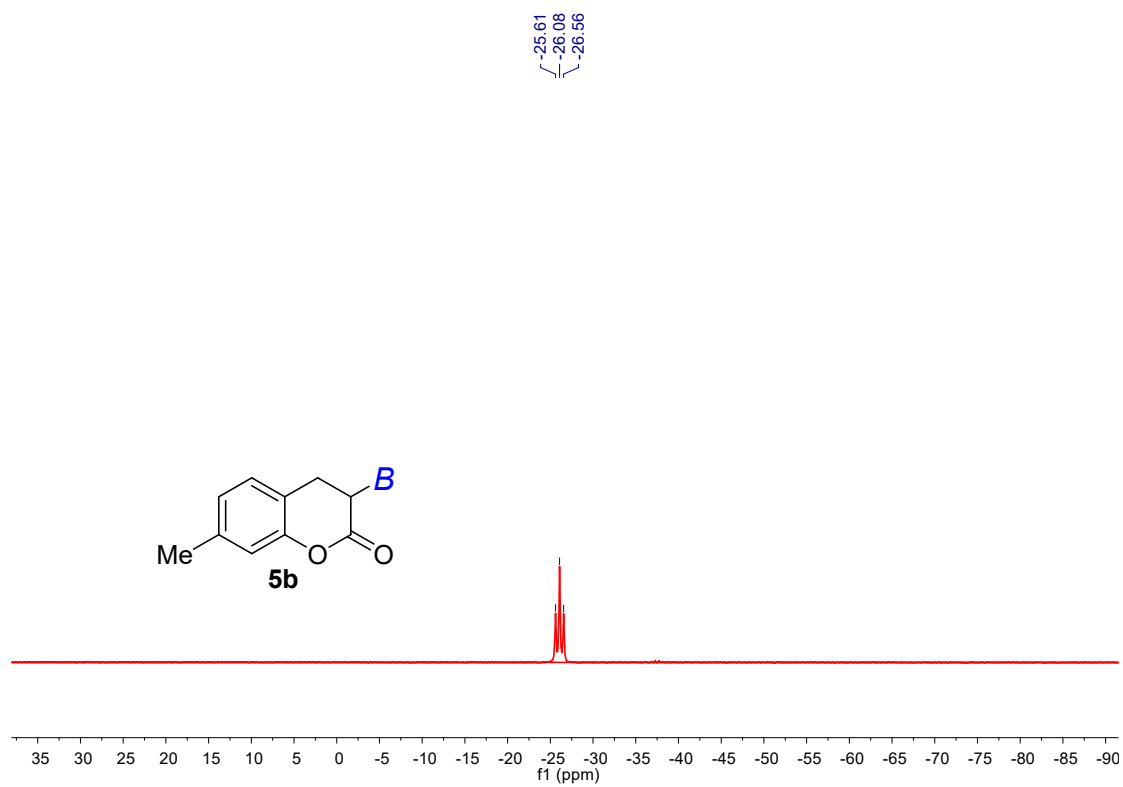
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



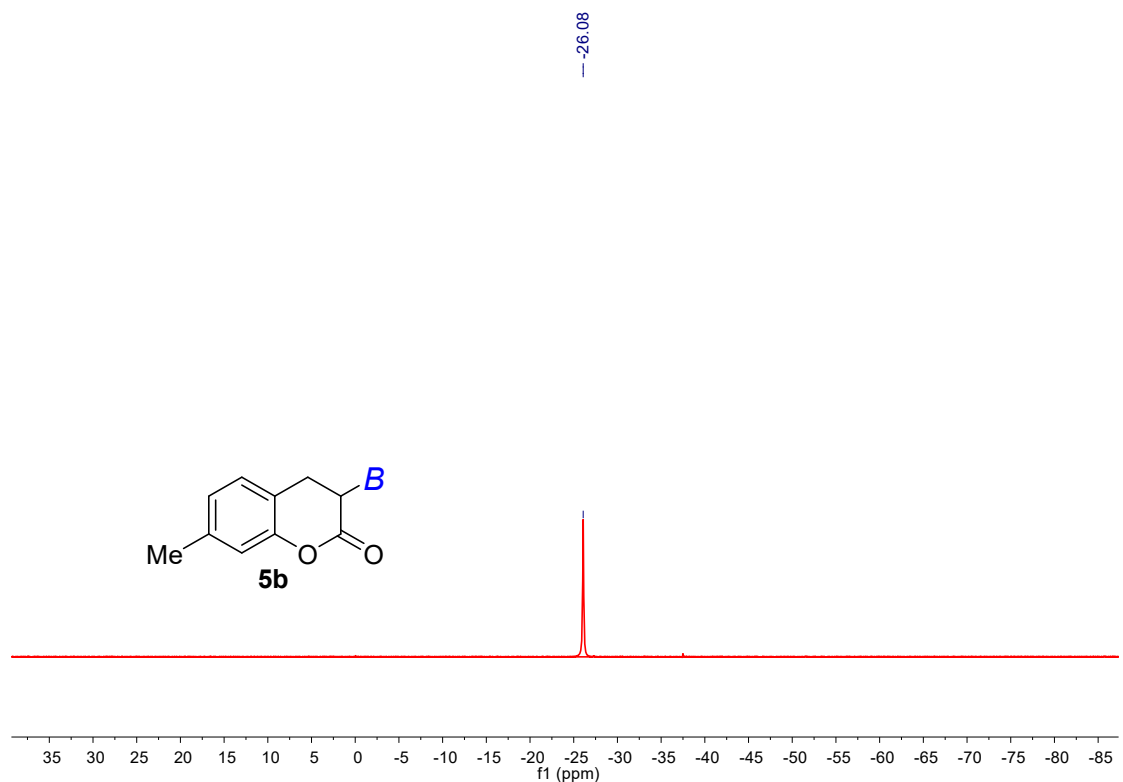
¹H NMR (600 MHz, CDCl₃)



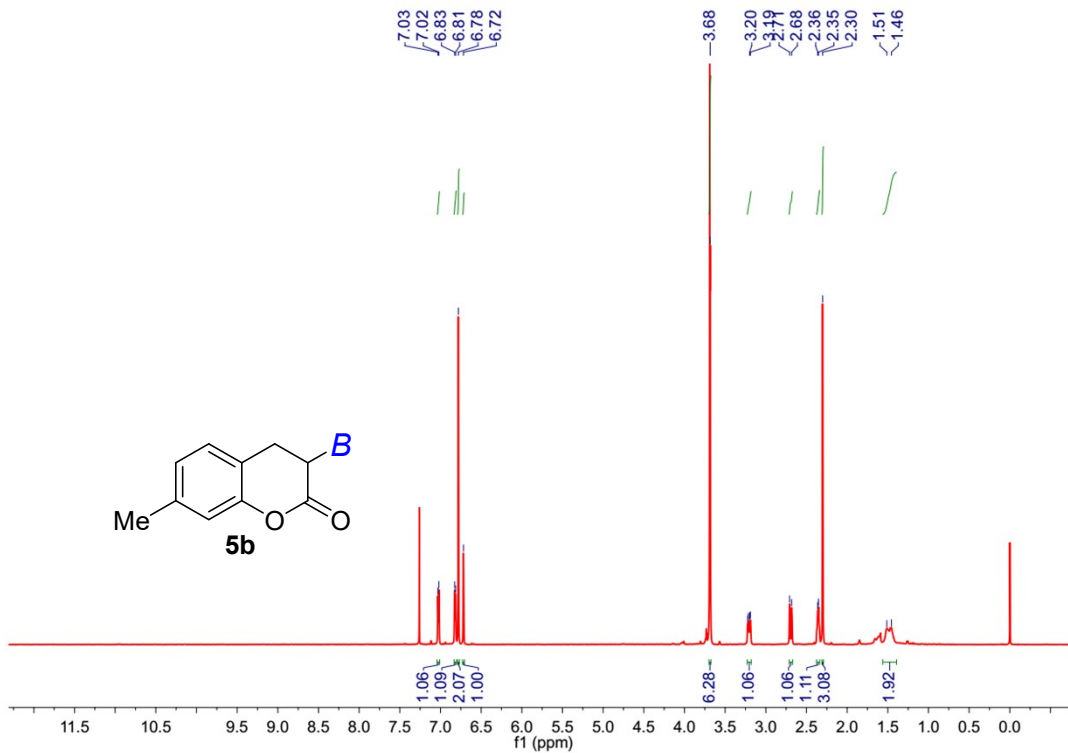
¹³C NMR (151 MHz, CDCl₃)



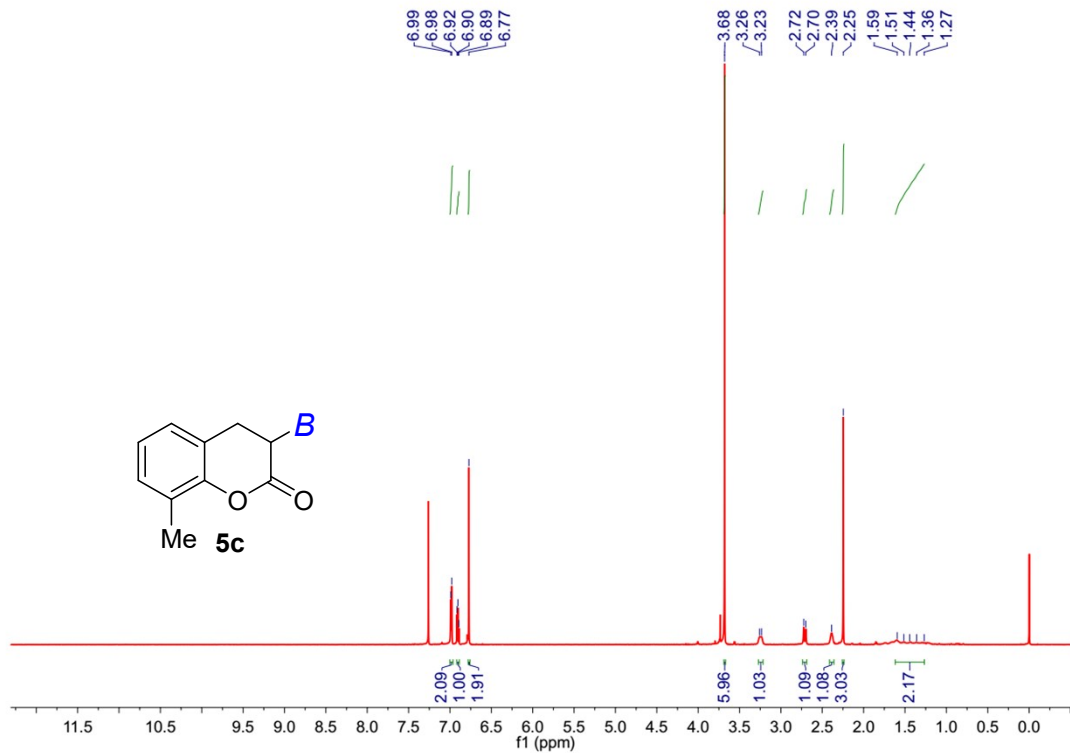
^{11}B NMR (193 MHz, CDCl_3)



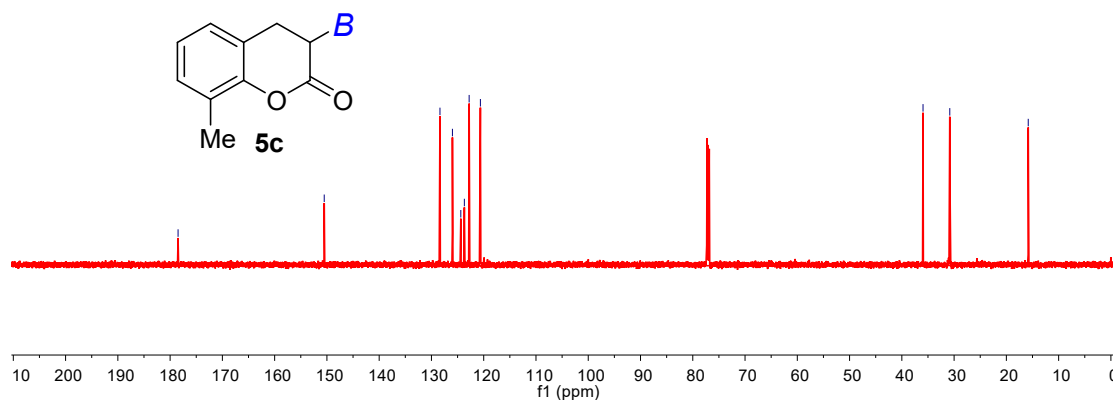
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

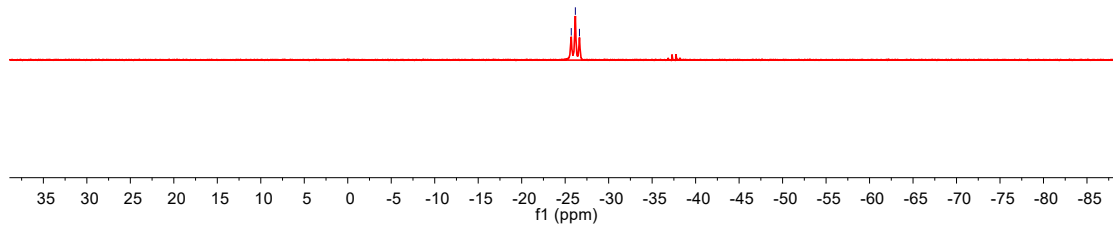
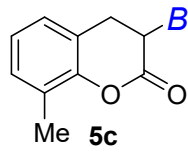


¹H NMR (600 MHz, CDCl₃)



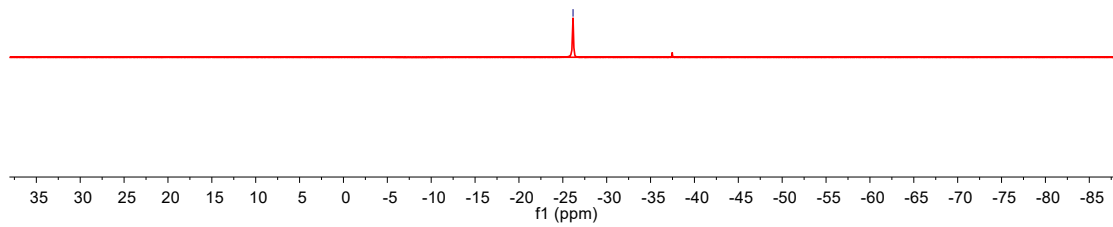
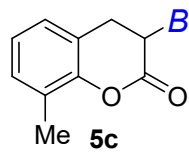
¹³C NMR (151 MHz, CDCl₃)

-25.70
-26.17
-26.64

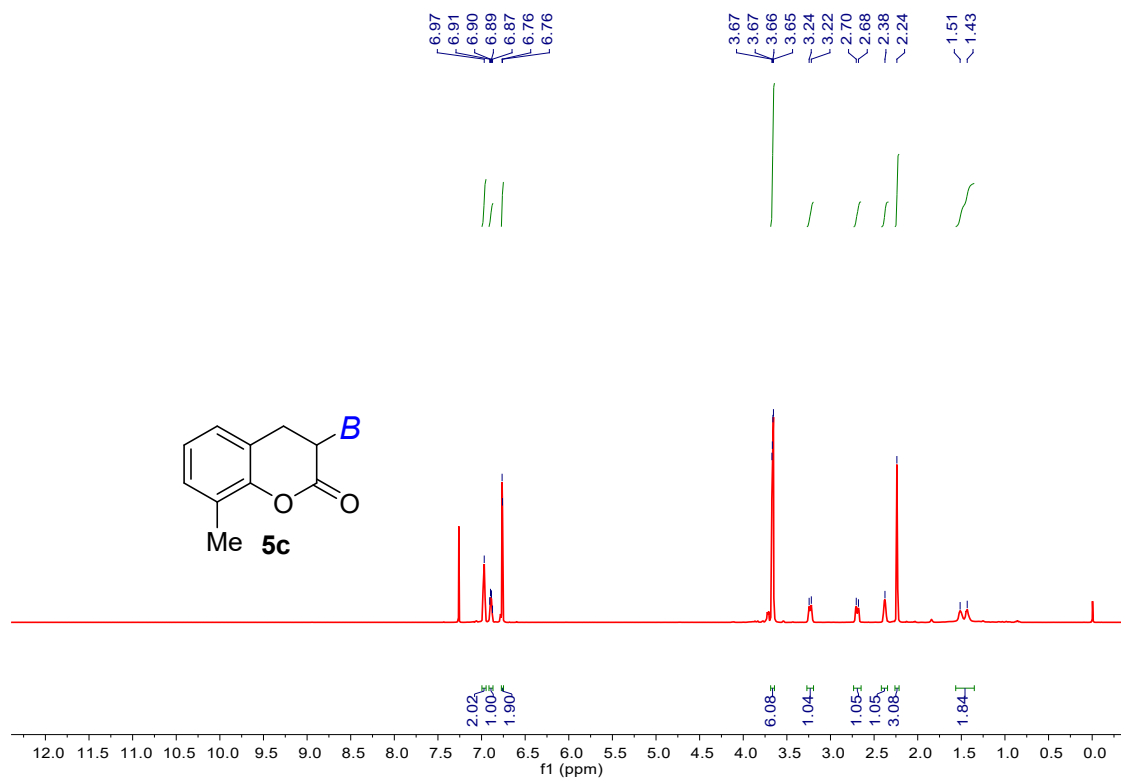


^{11}B NMR (193 MHz, CDCl_3)

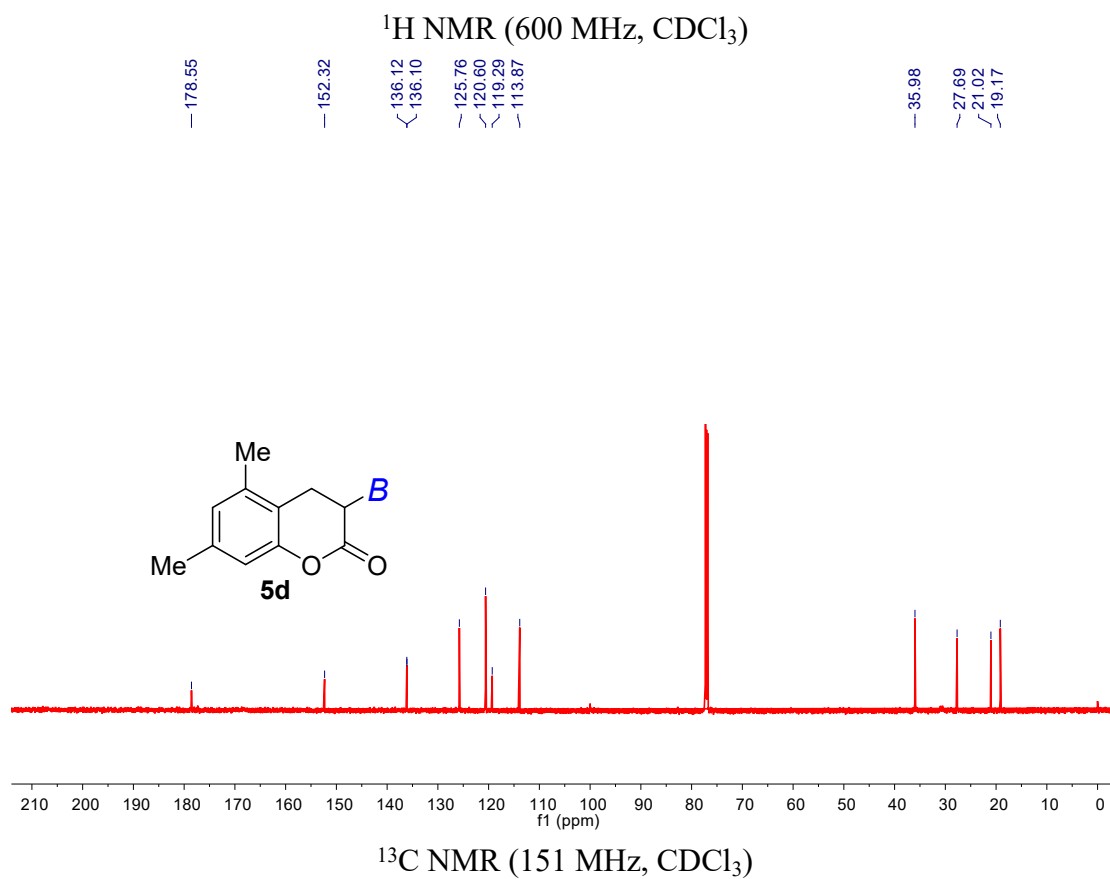
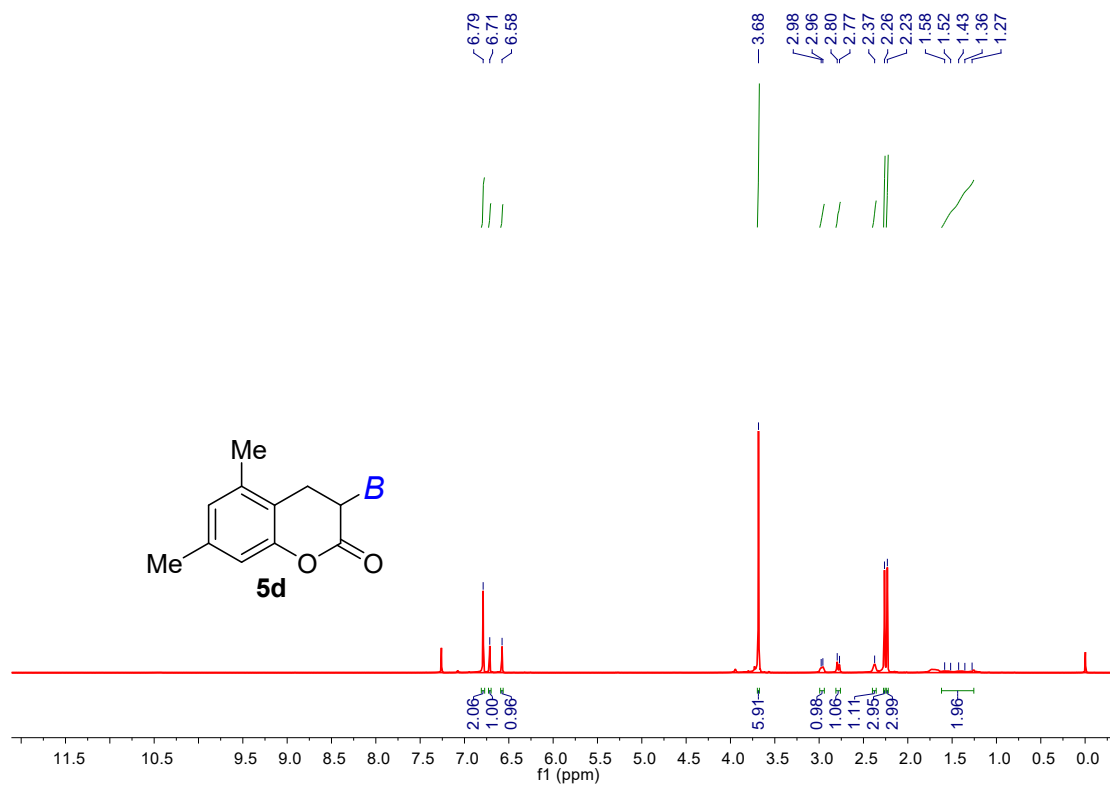
-26.17

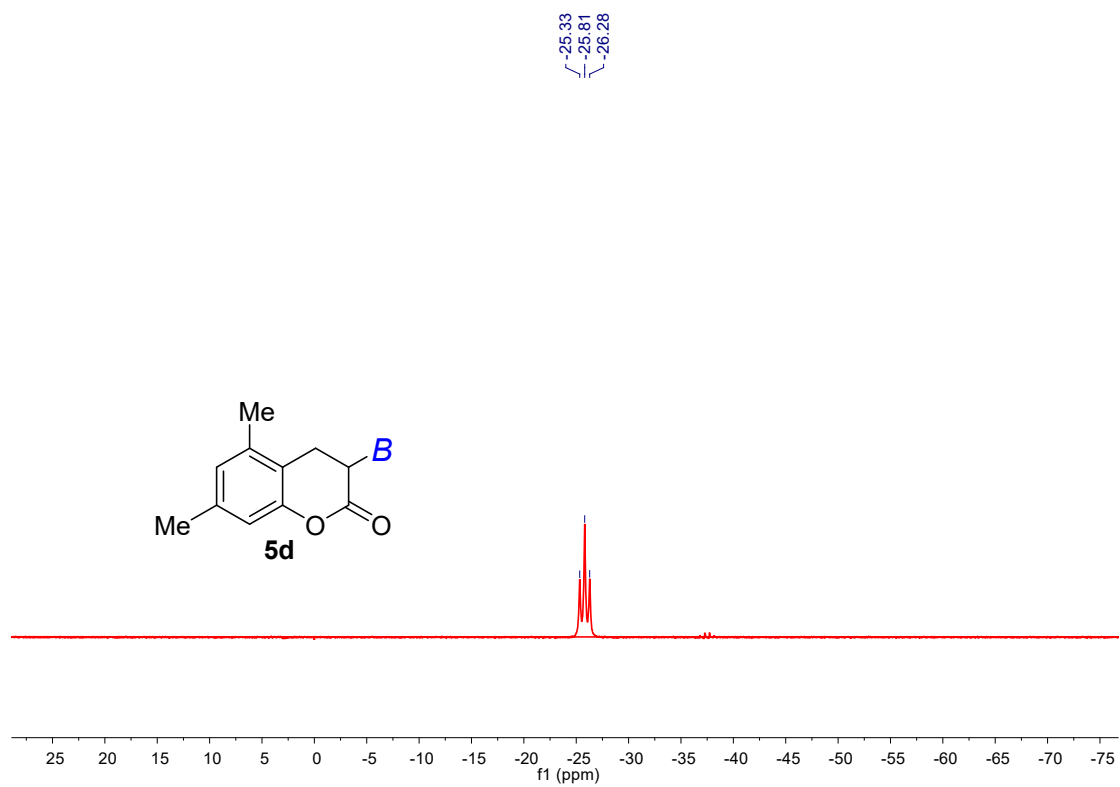


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

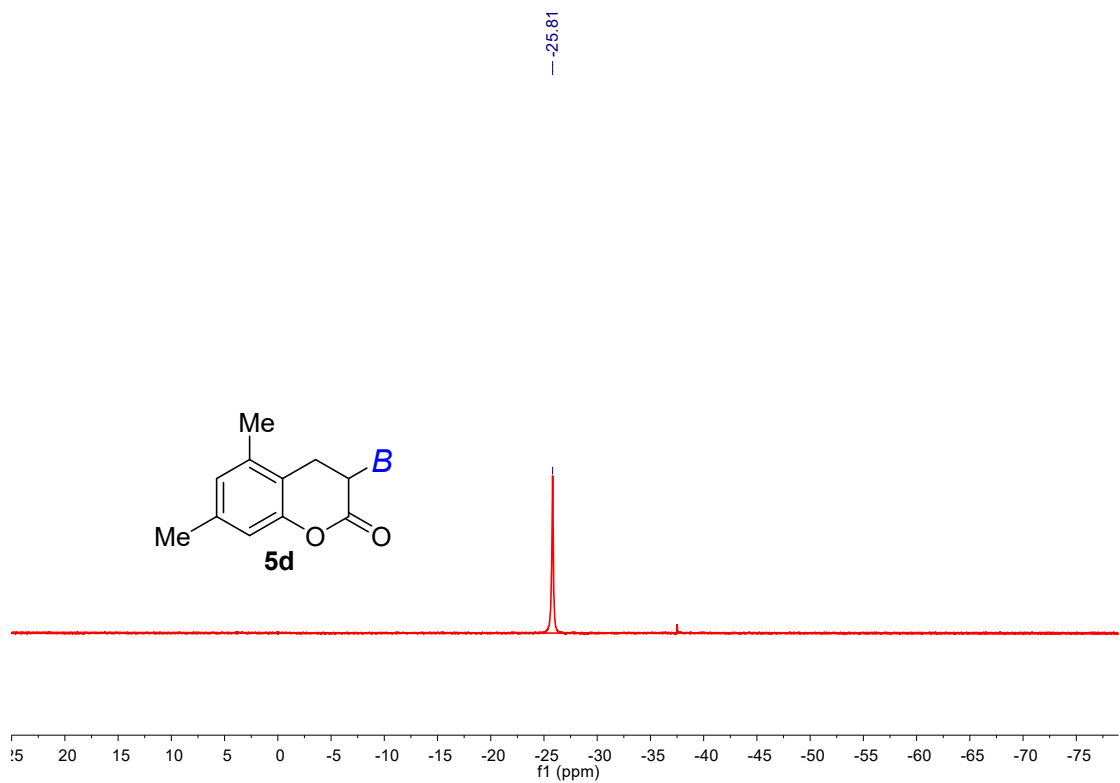


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

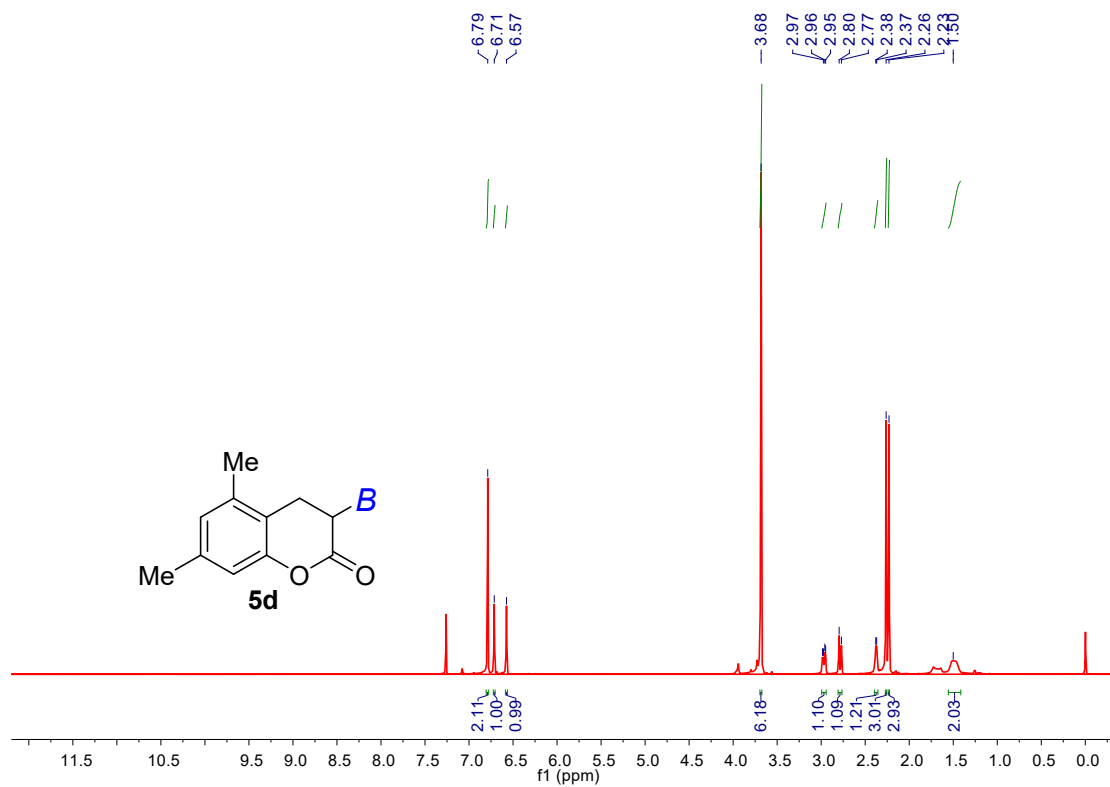




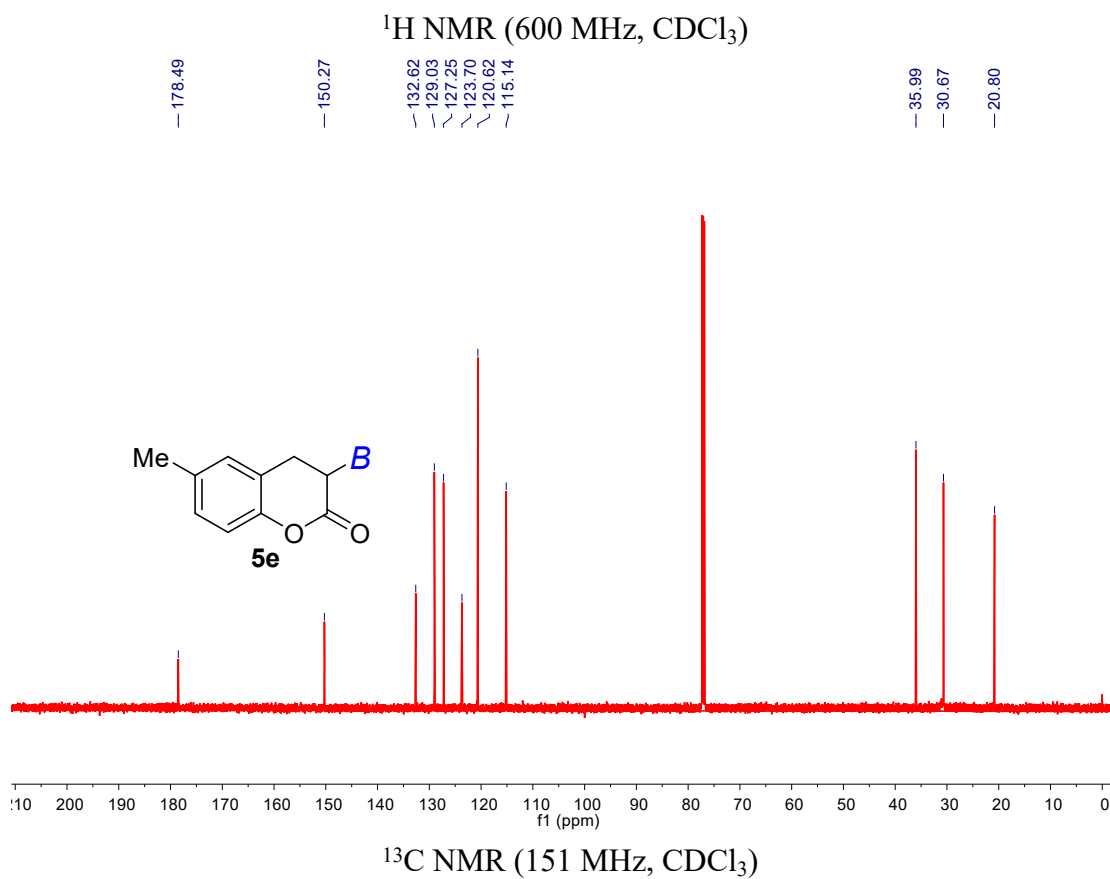
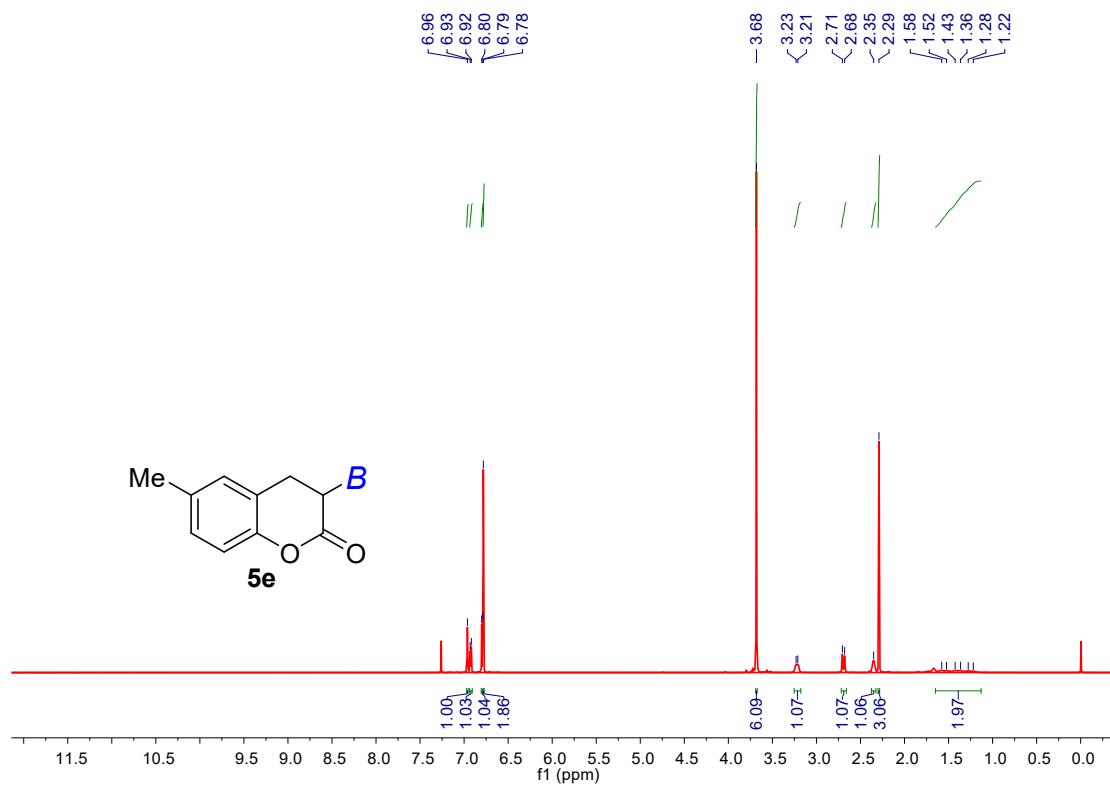
^{11}B NMR (193 MHz, CDCl_3)

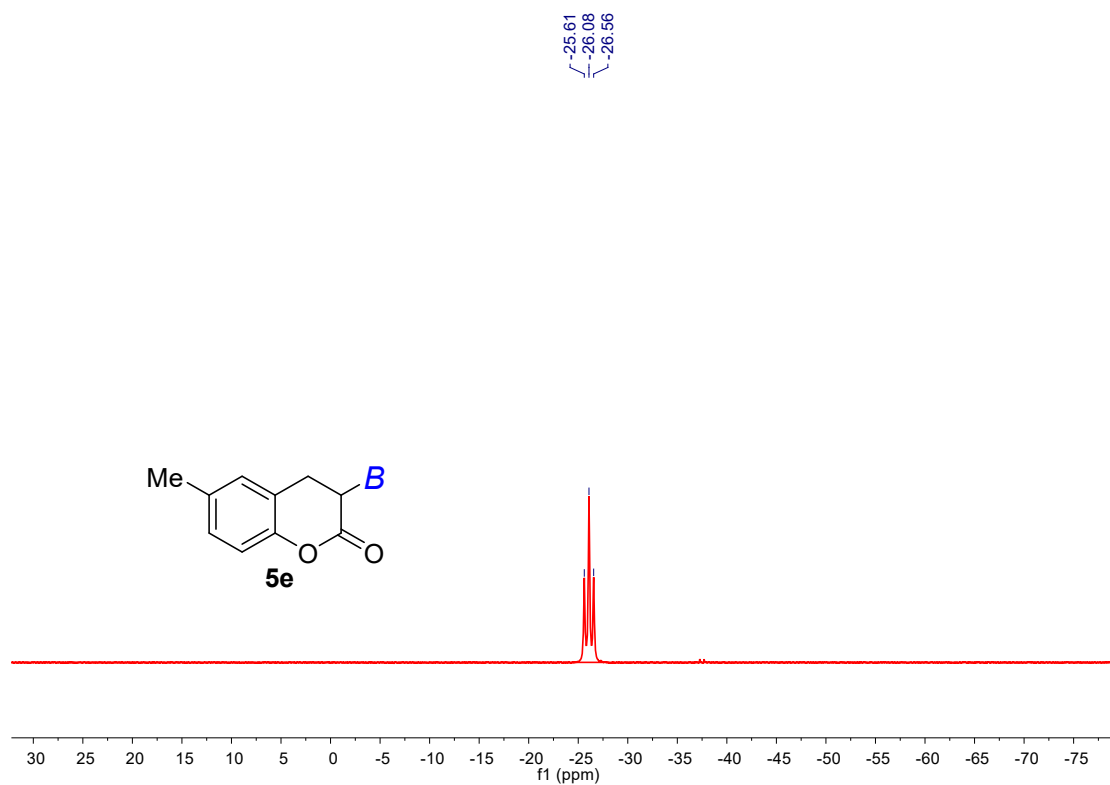


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

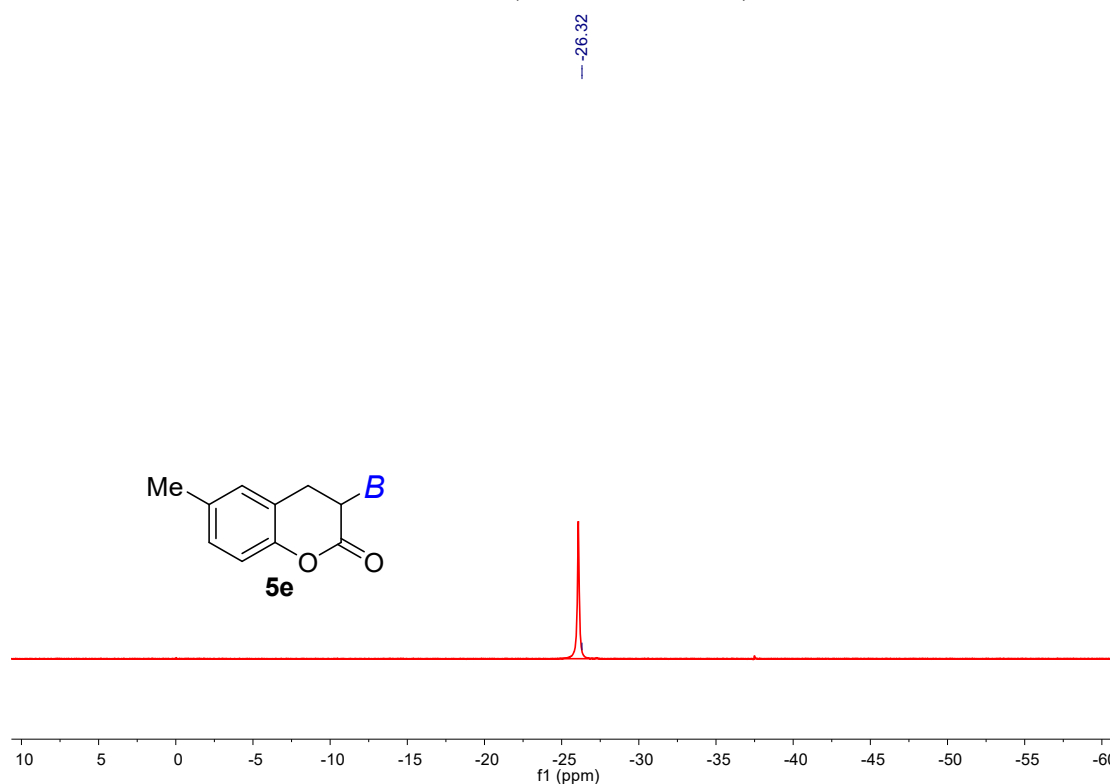


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

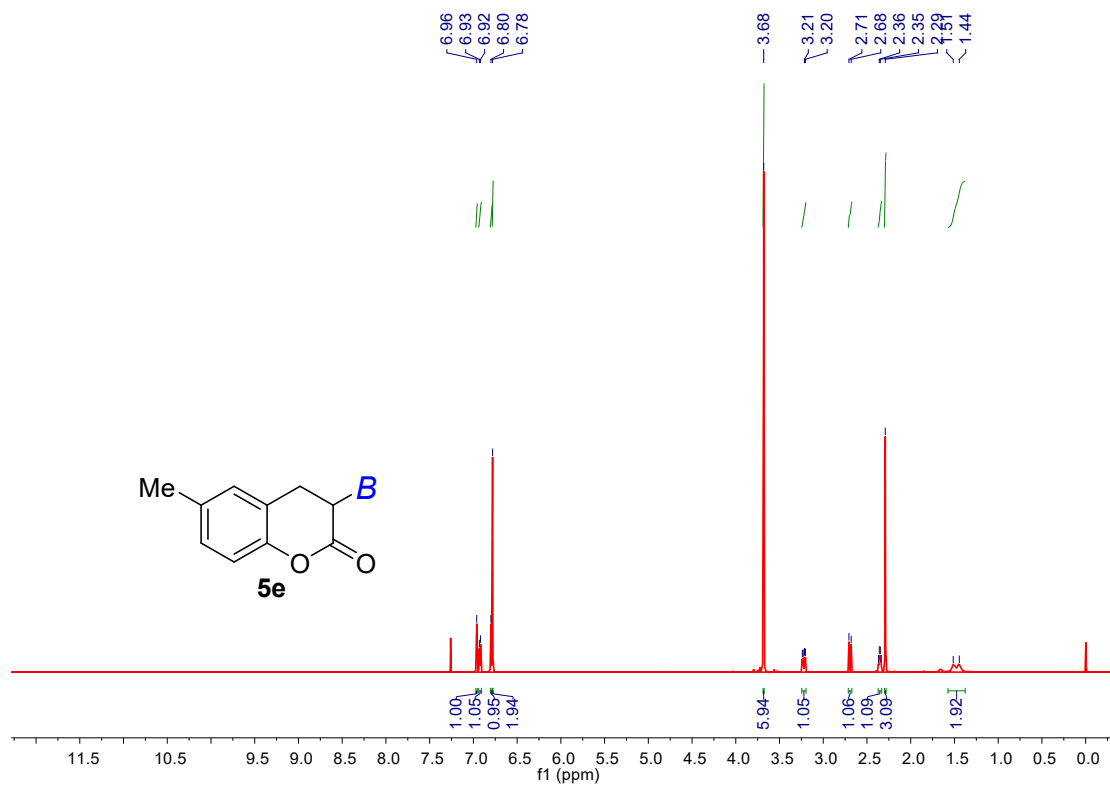




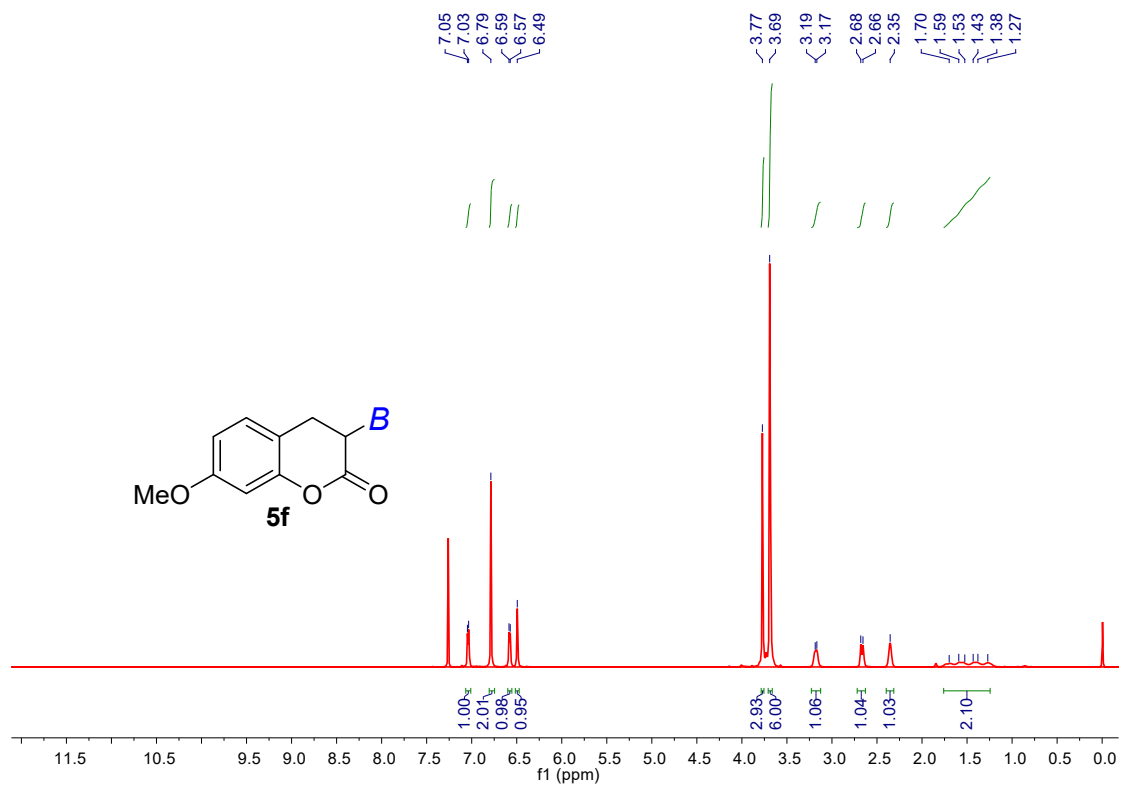
^{11}B NMR (193 MHz, CDCl_3)



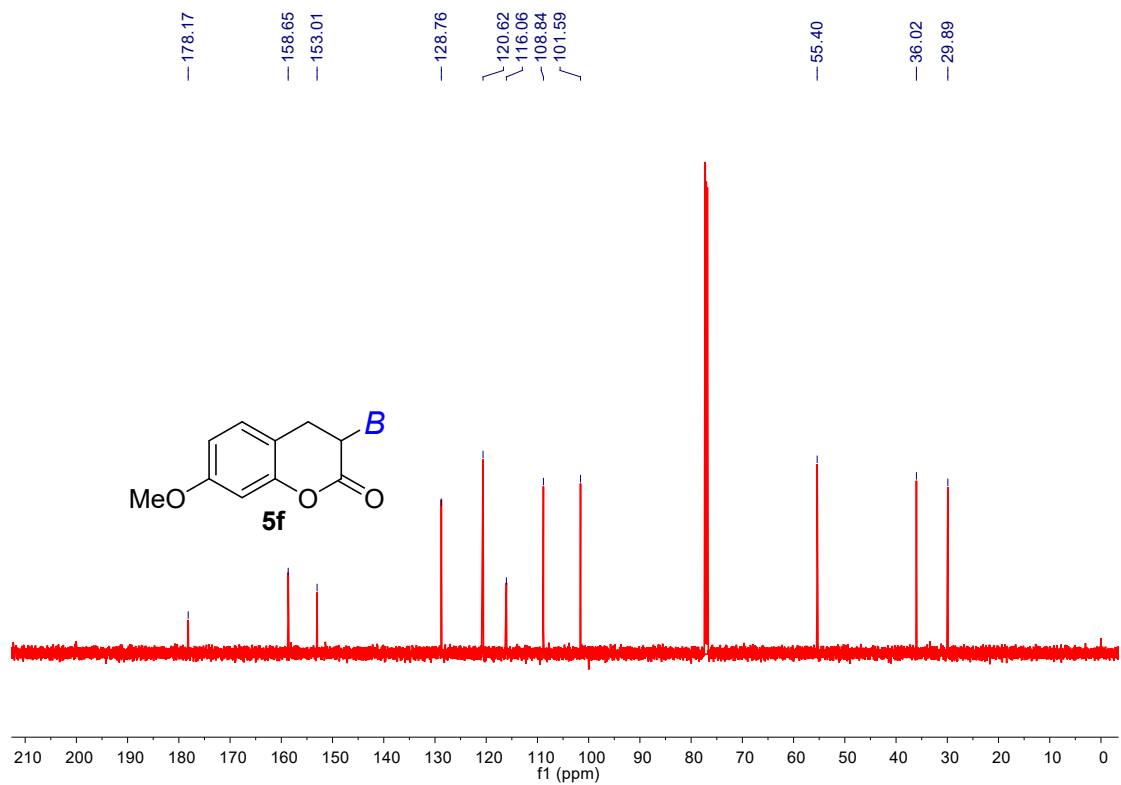
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



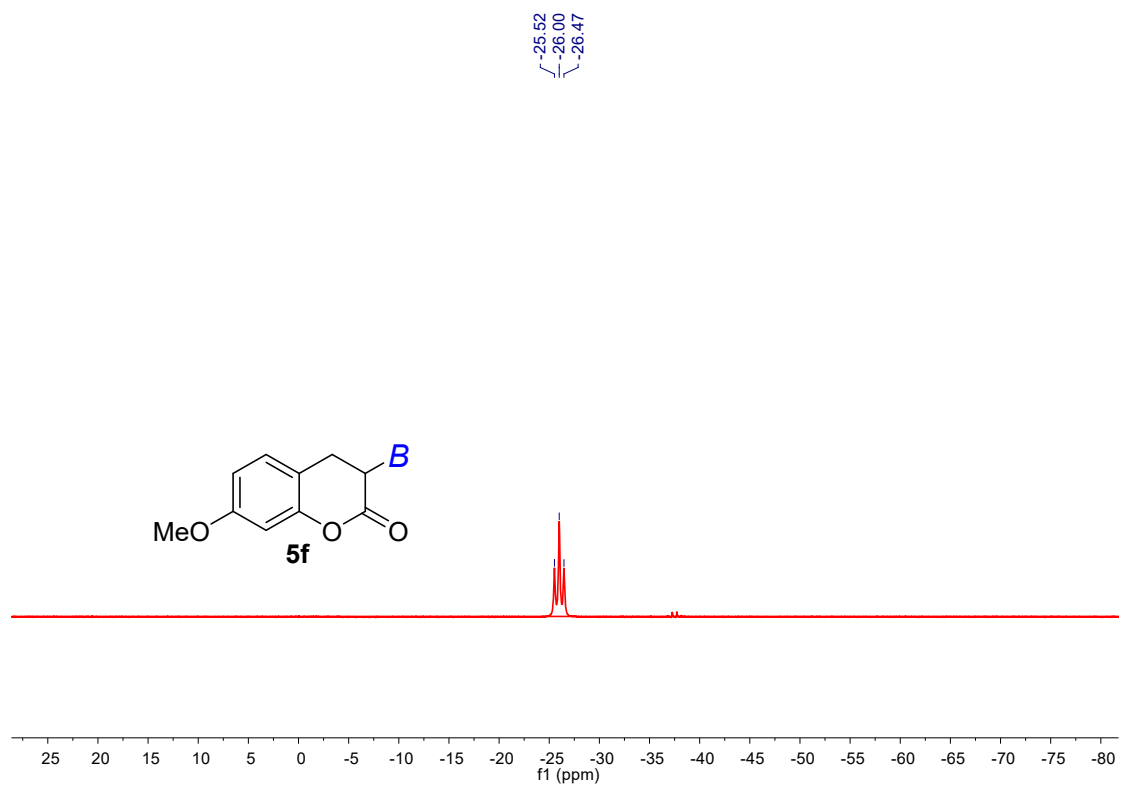
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



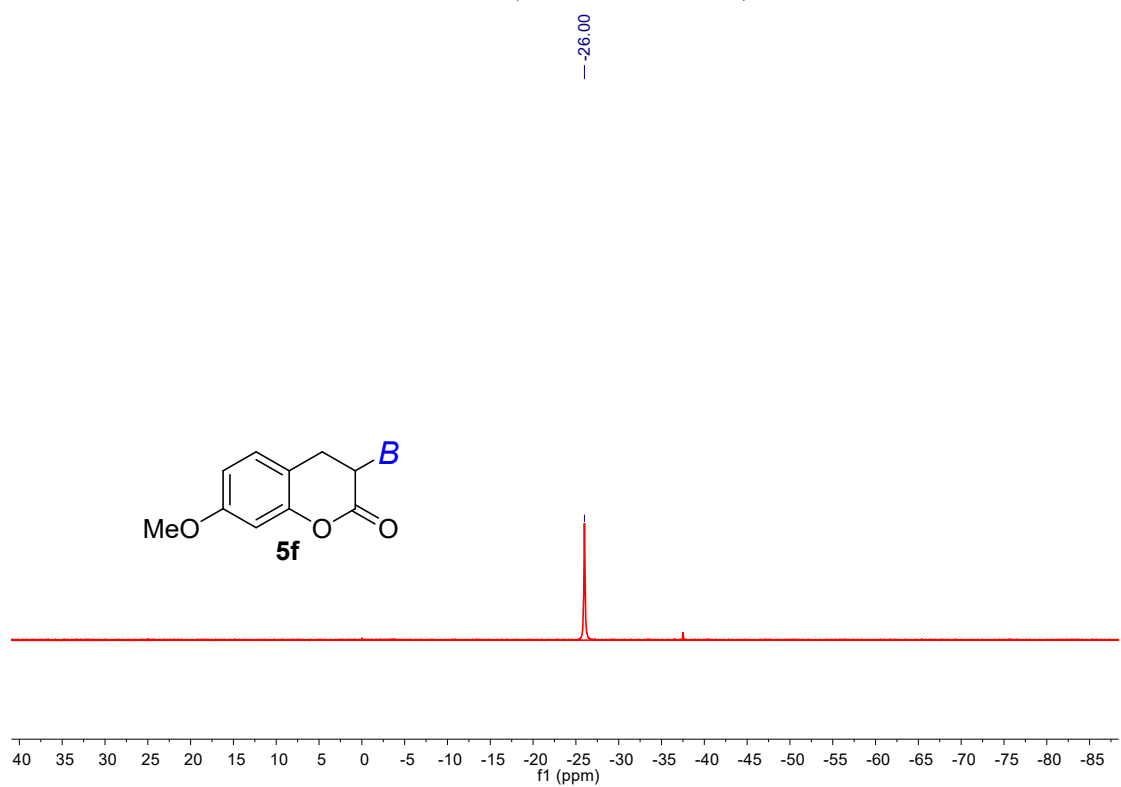
¹H NMR (600 MHz, CDCl₃)



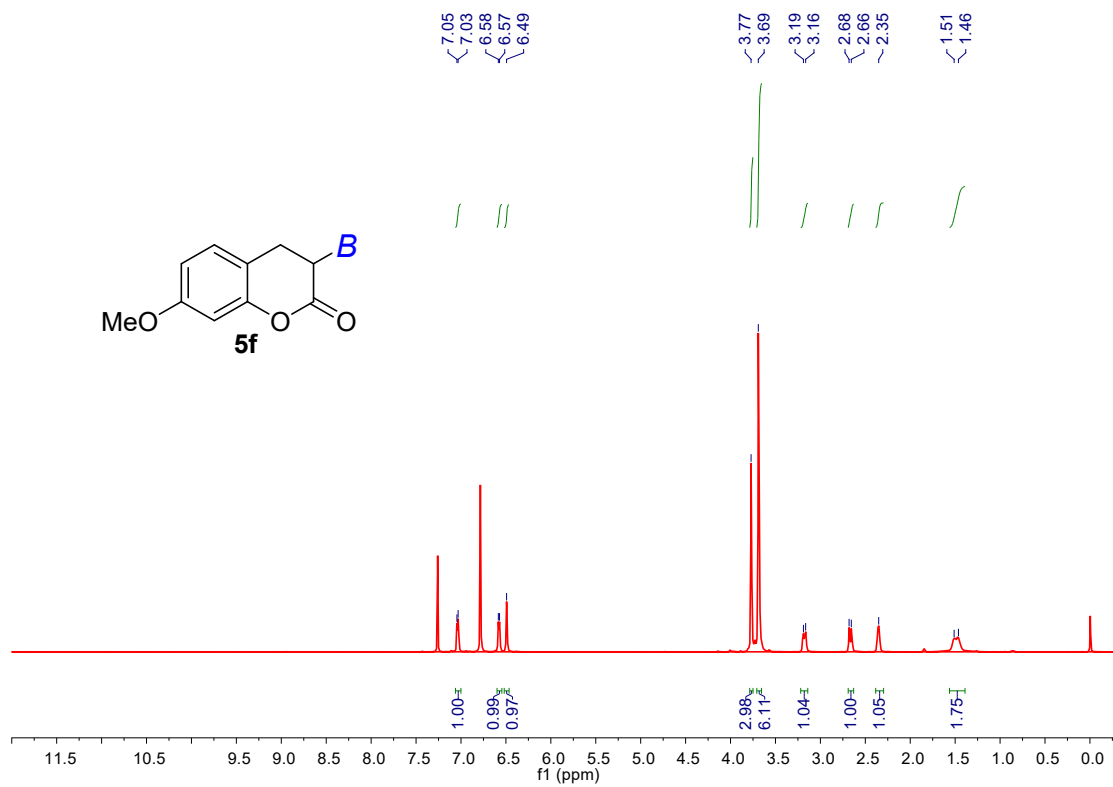
¹³C NMR (151 MHz, CDCl₃)



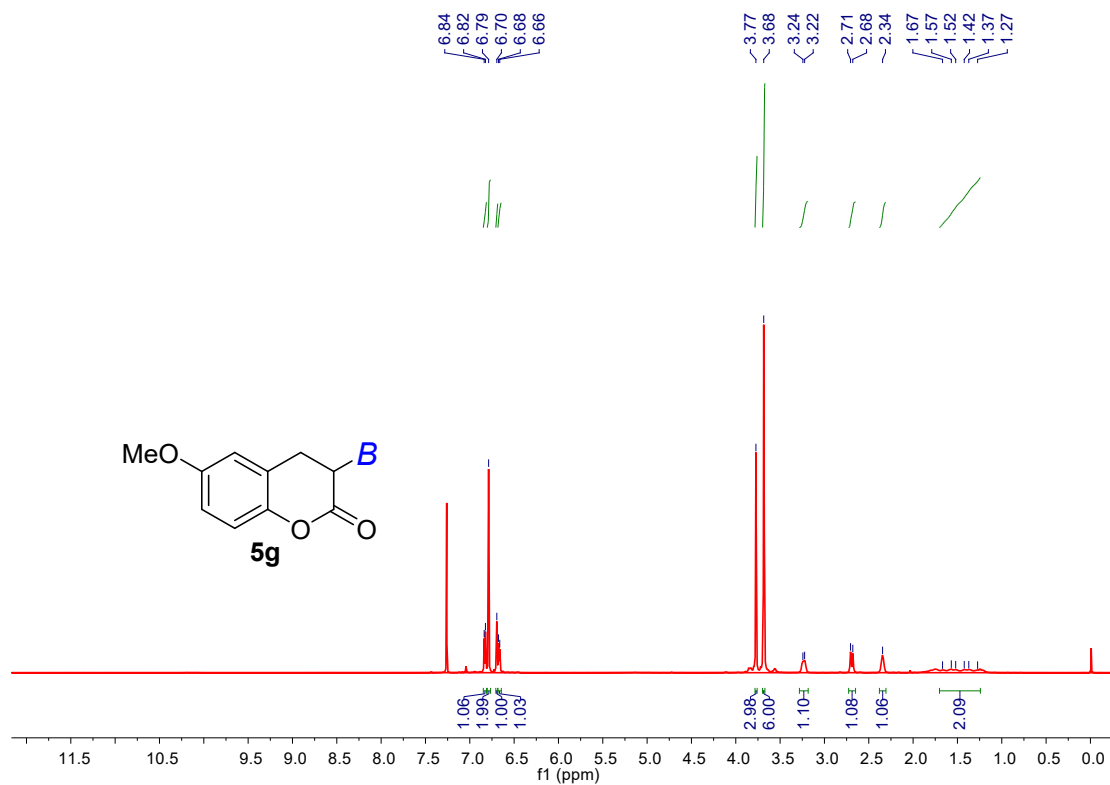
^{11}B NMR (193 MHz, CDCl_3)



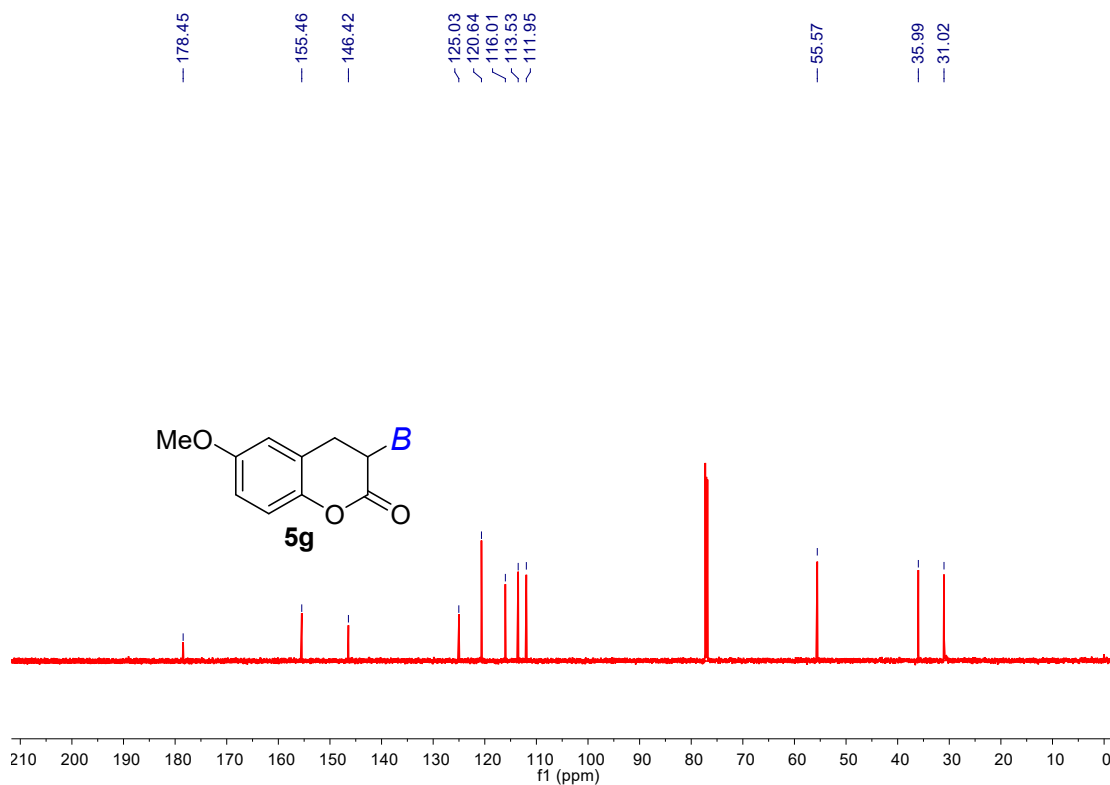
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

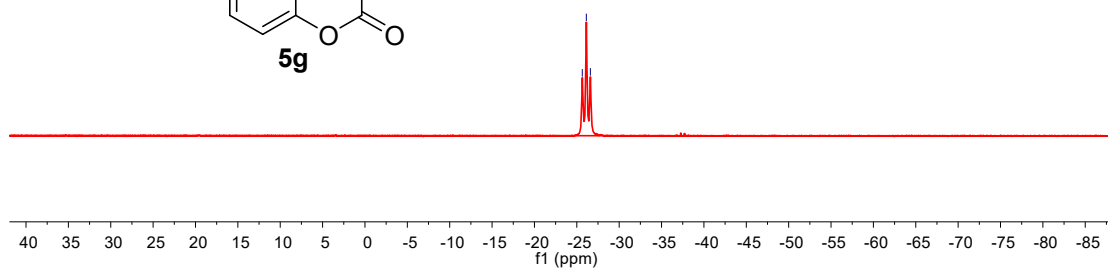
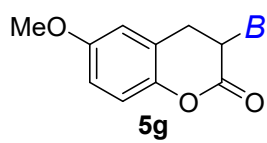


¹H NMR (600 MHz, CDCl₃)



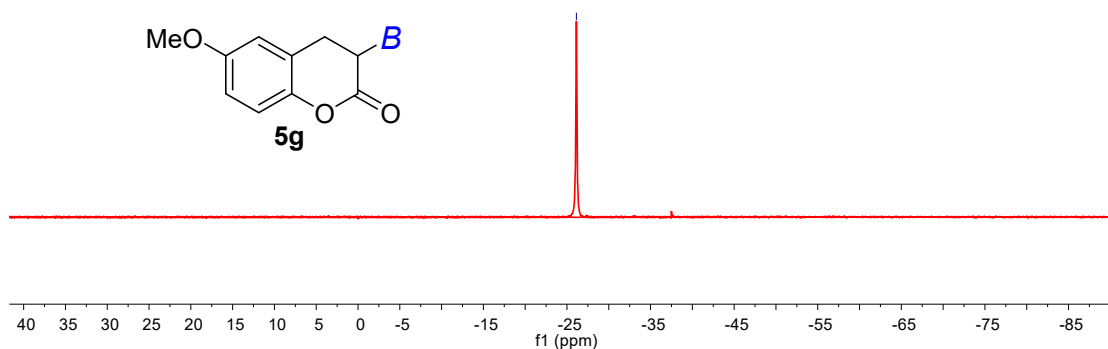
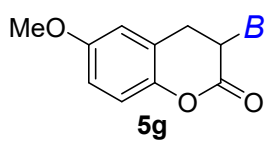
¹³C NMR (151 MHz, CDCl₃)

-25.65
-26.12
-26.60

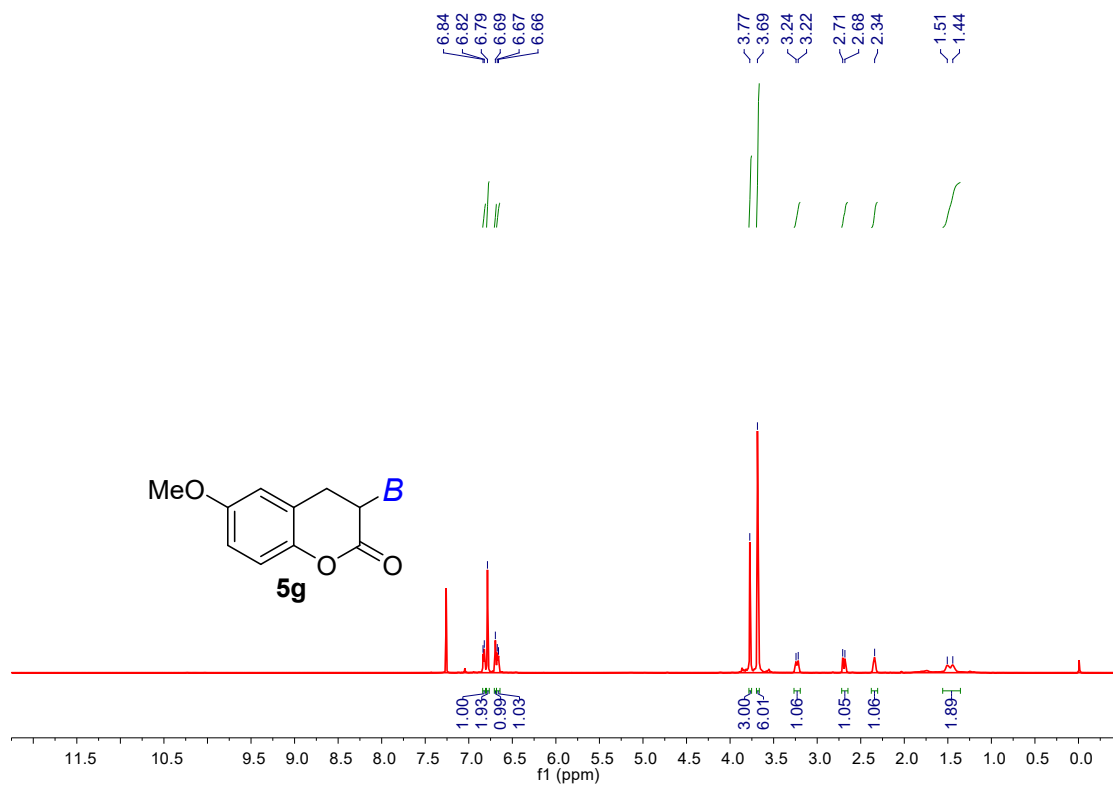


^{11}B NMR (193 MHz, CDCl_3)

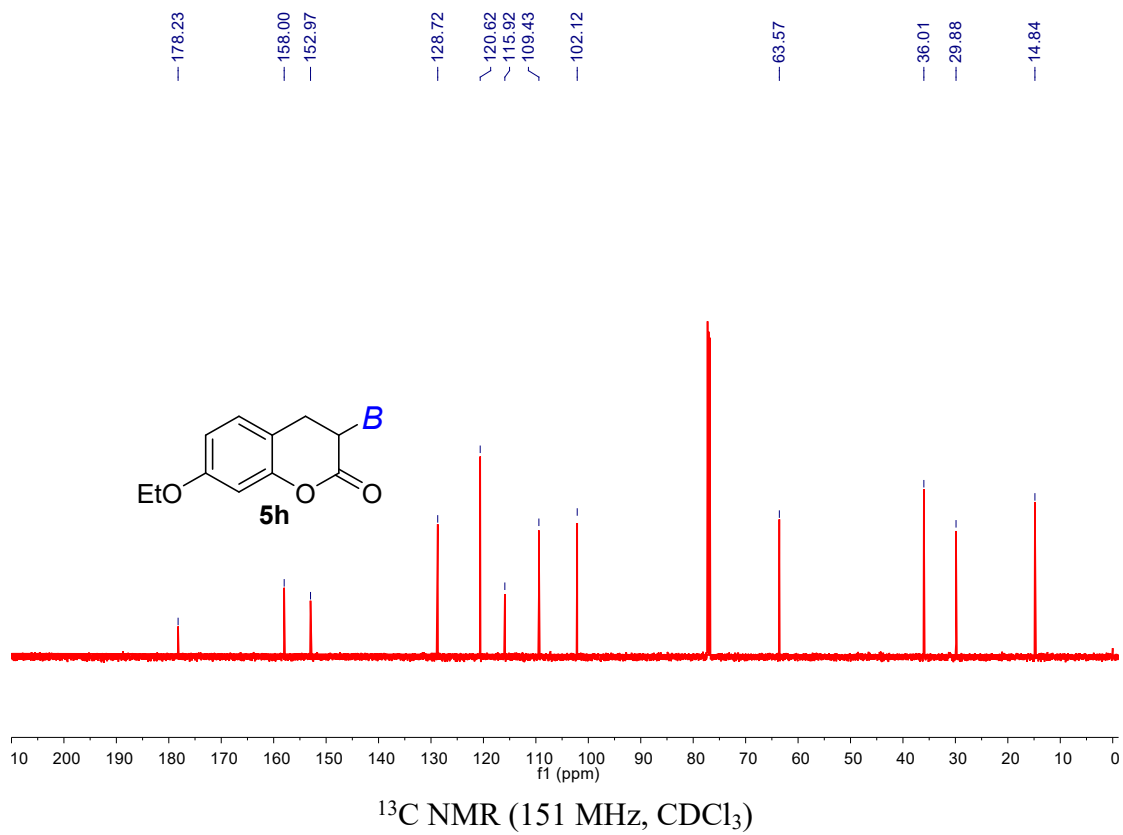
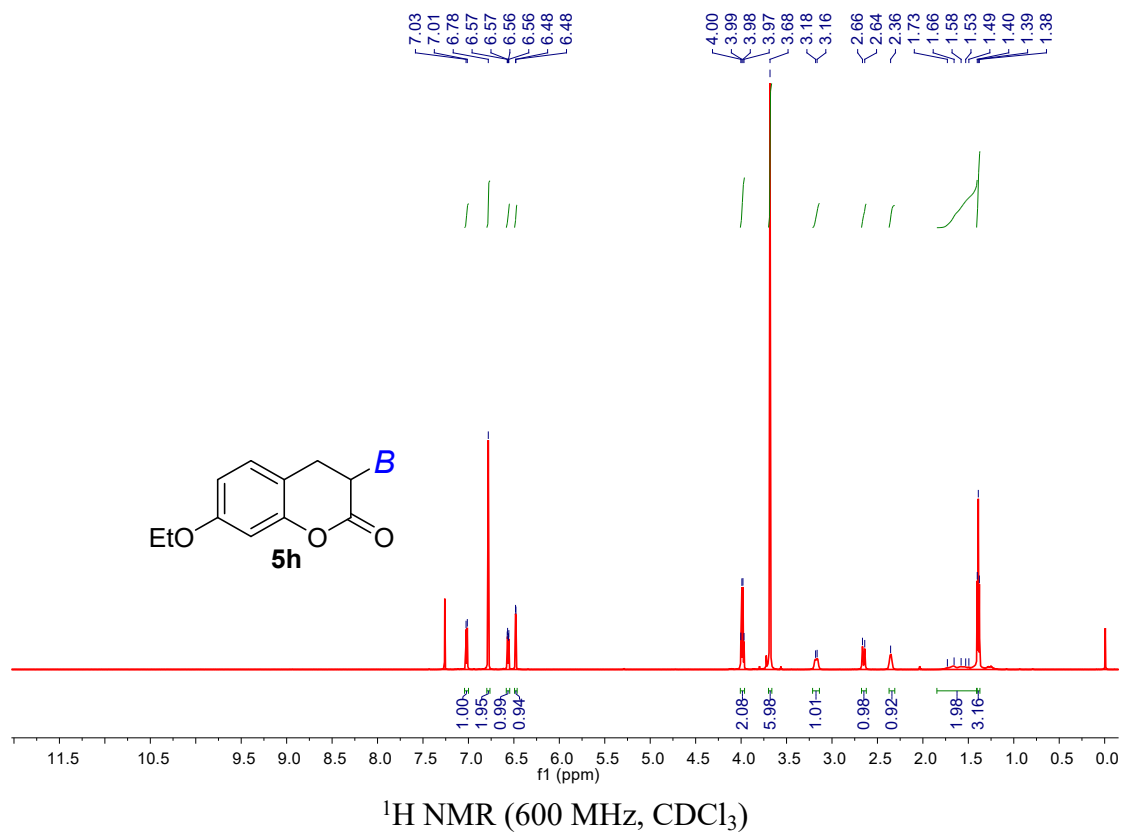
-26.13



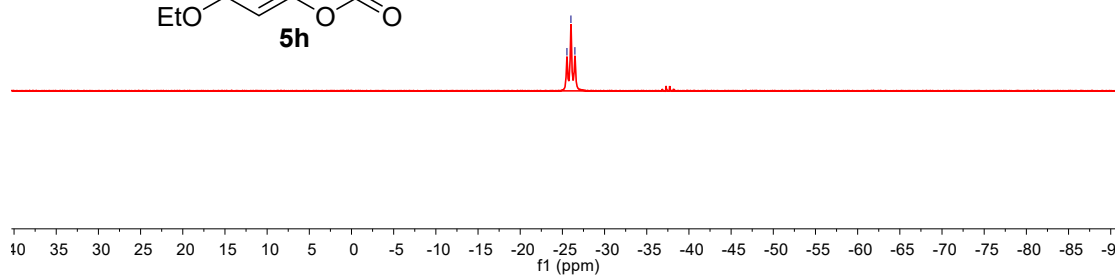
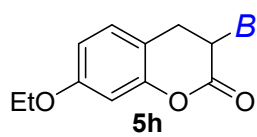
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

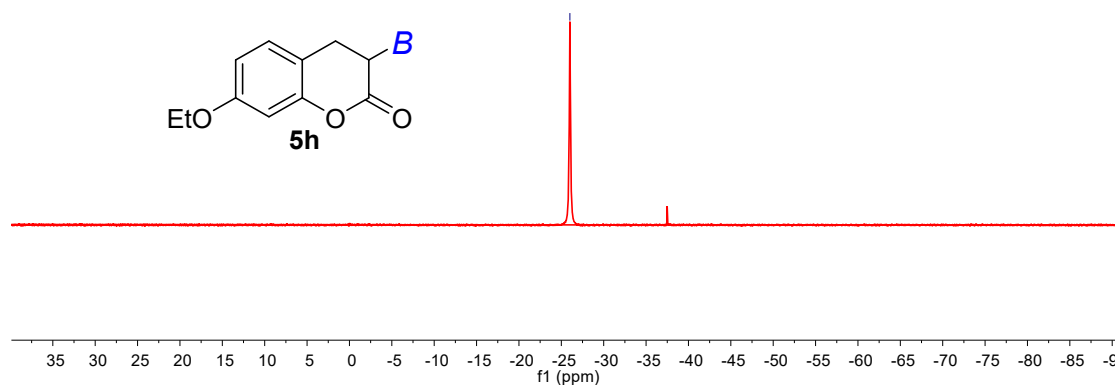
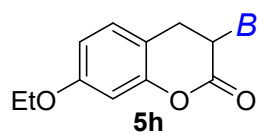


-25.54
-26.01
-26.48

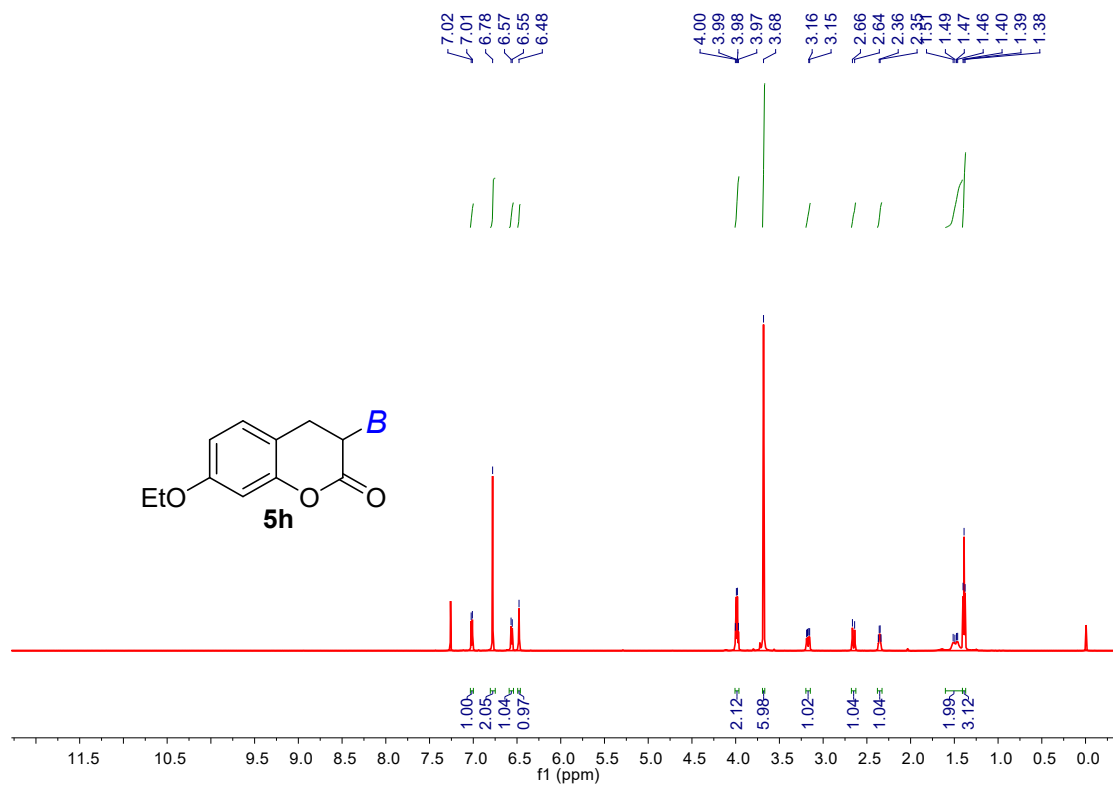


^{11}B NMR (193 MHz, CDCl_3)

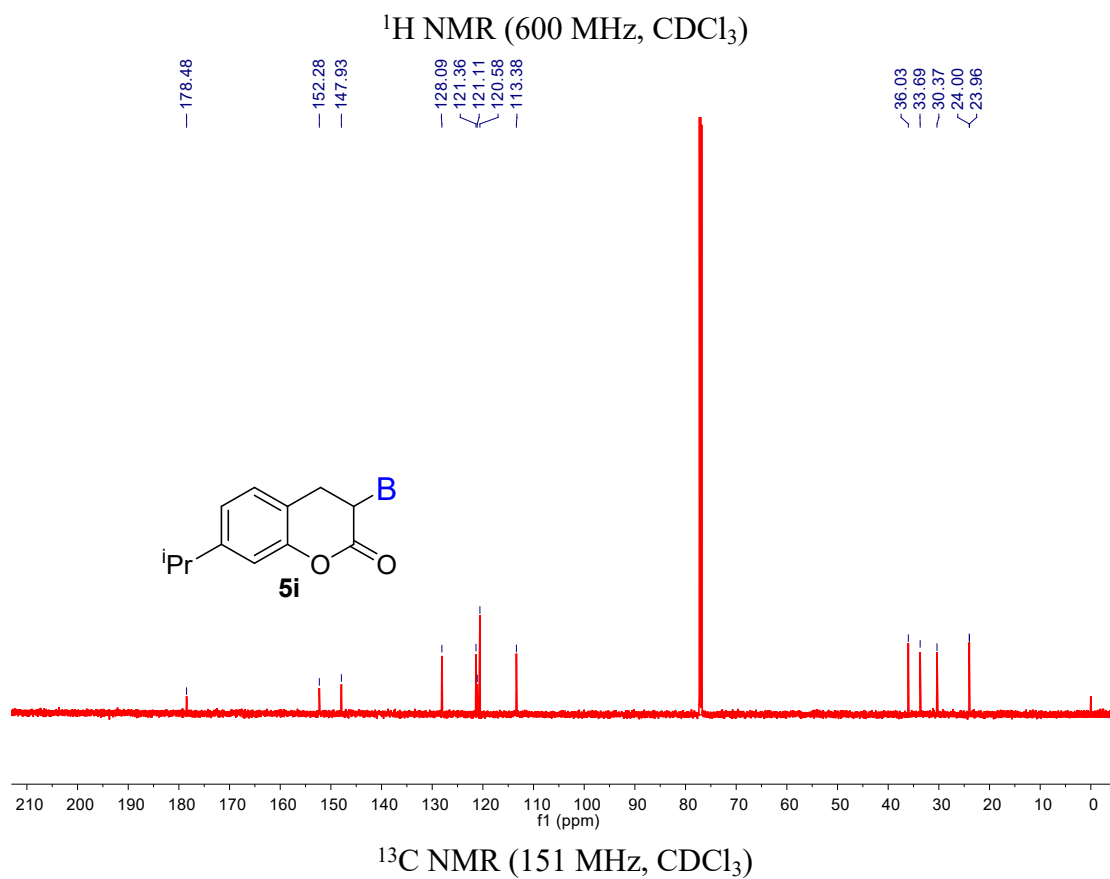
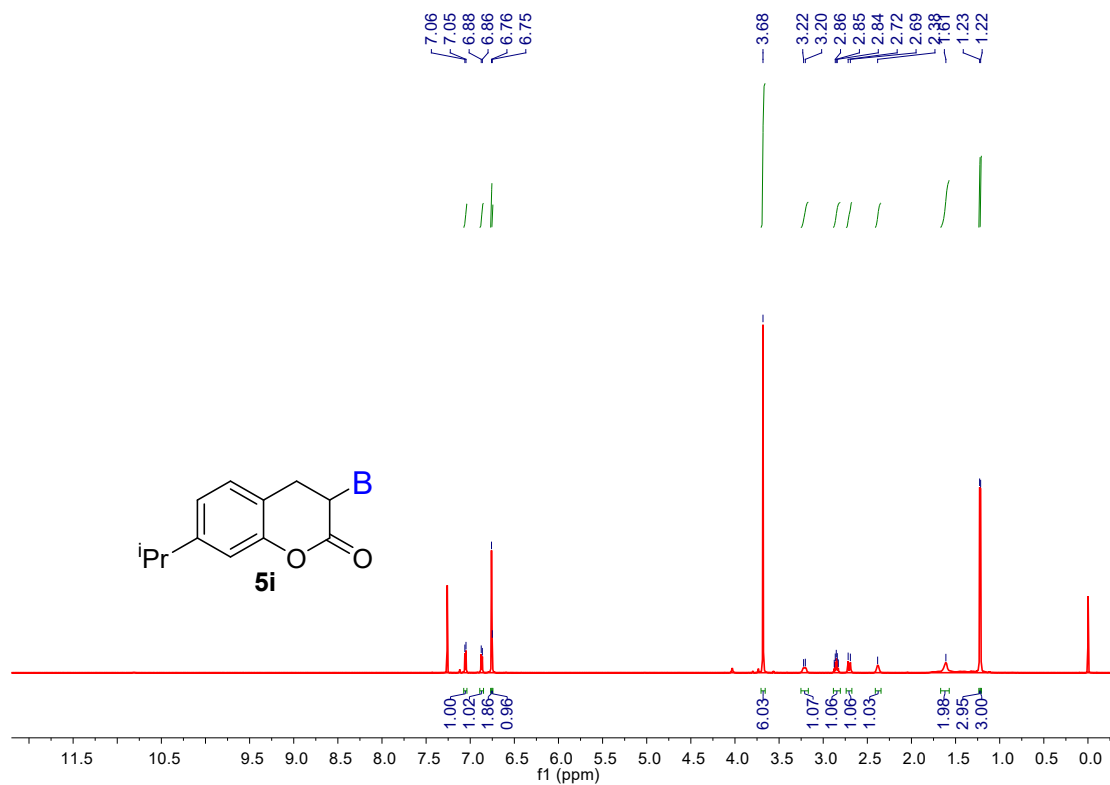
-26.01

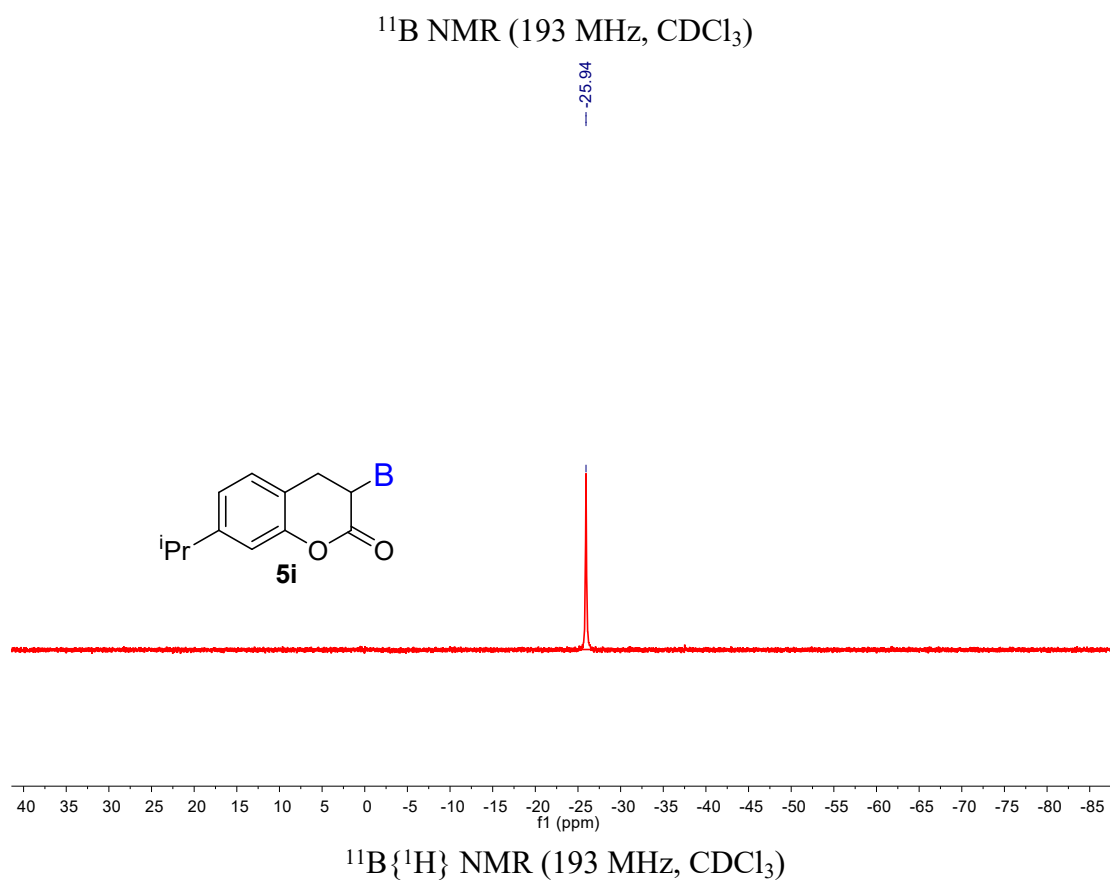
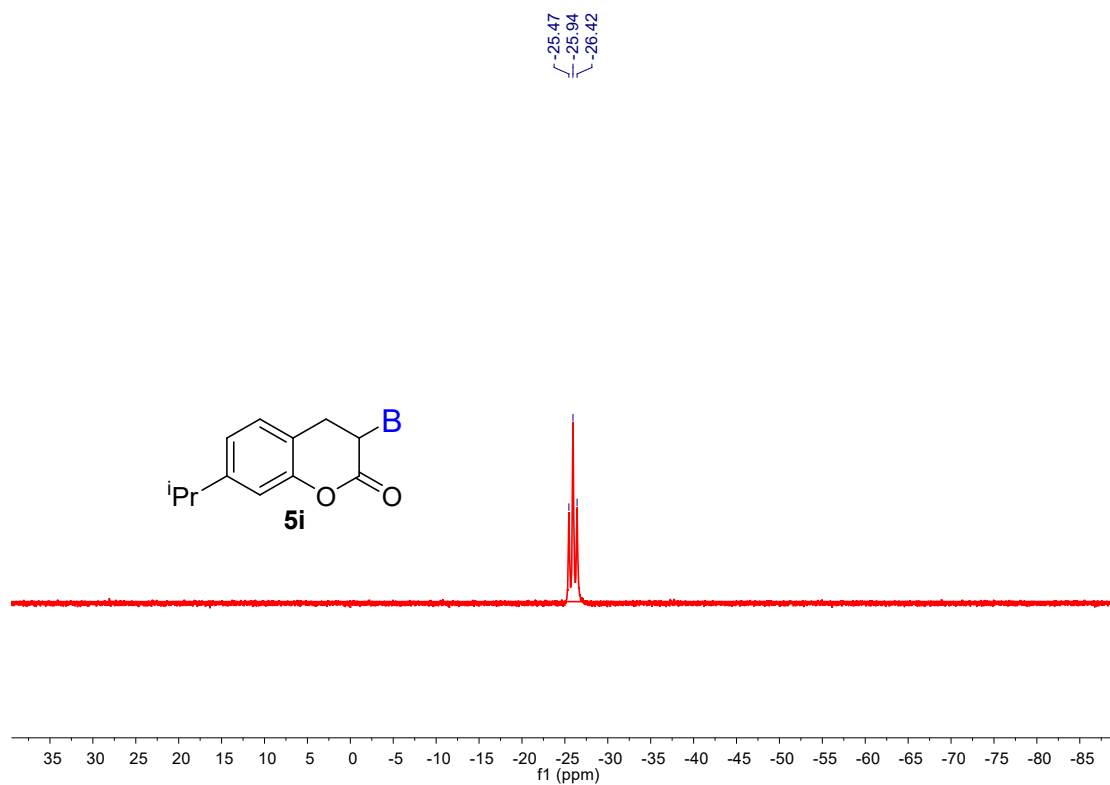


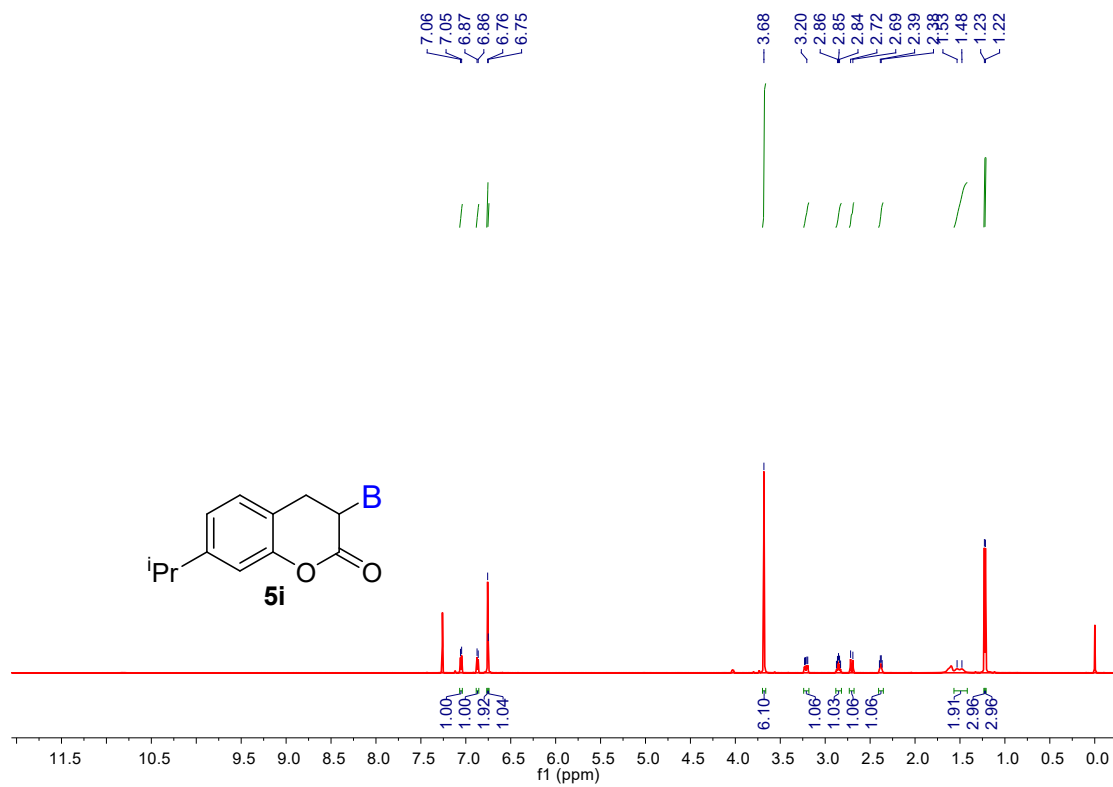
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



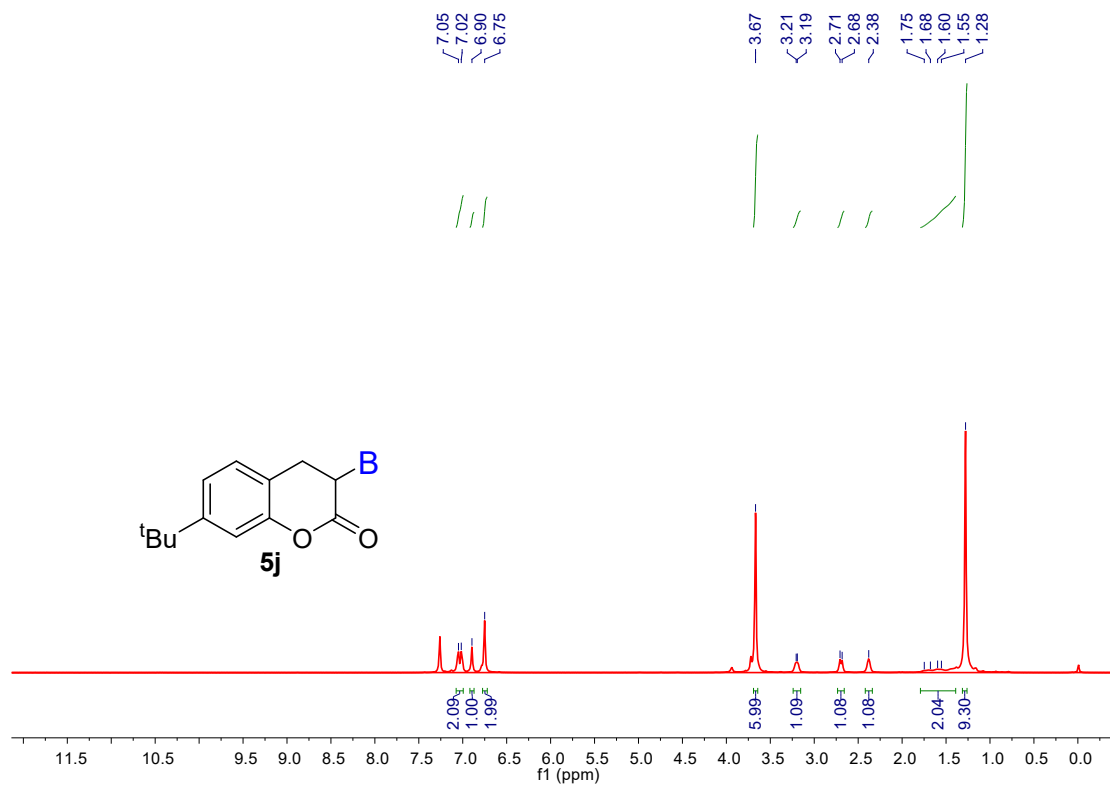
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



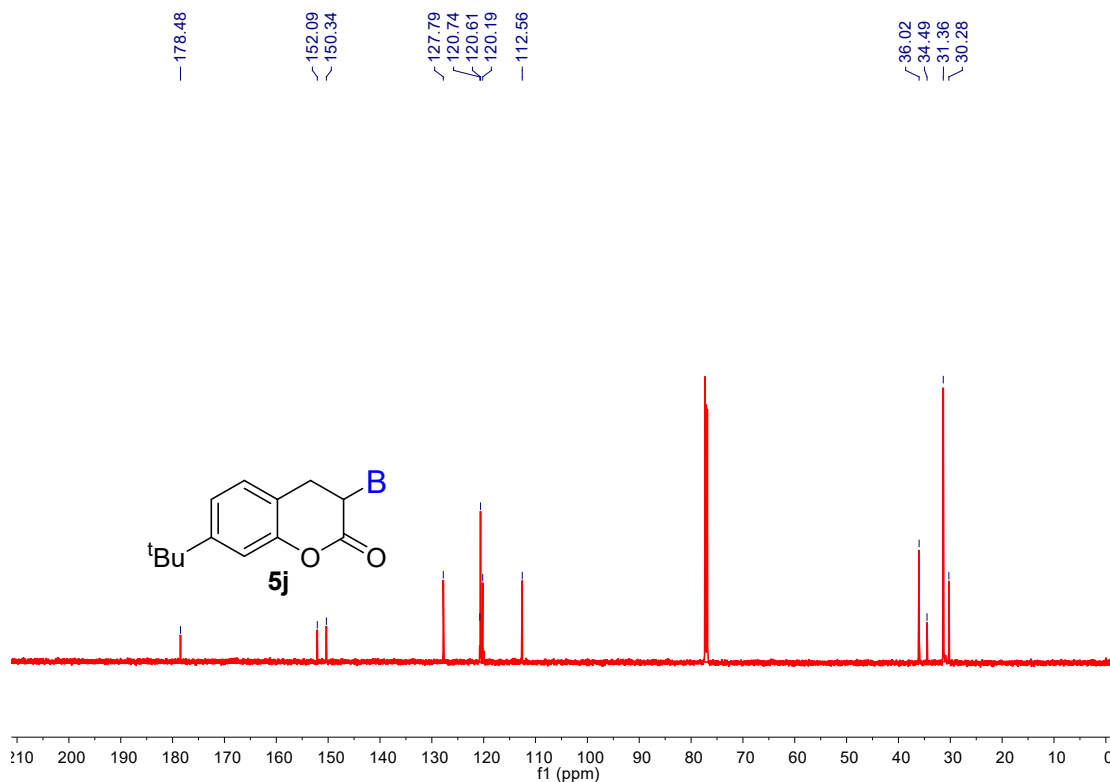




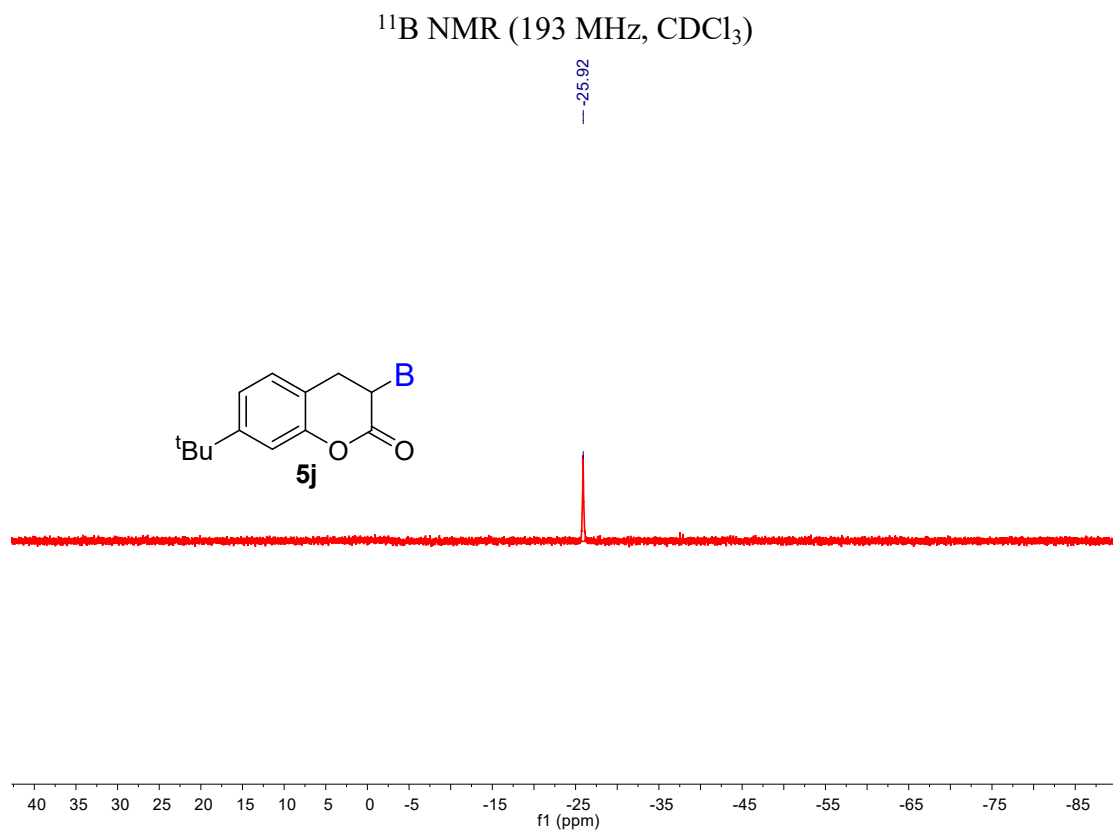
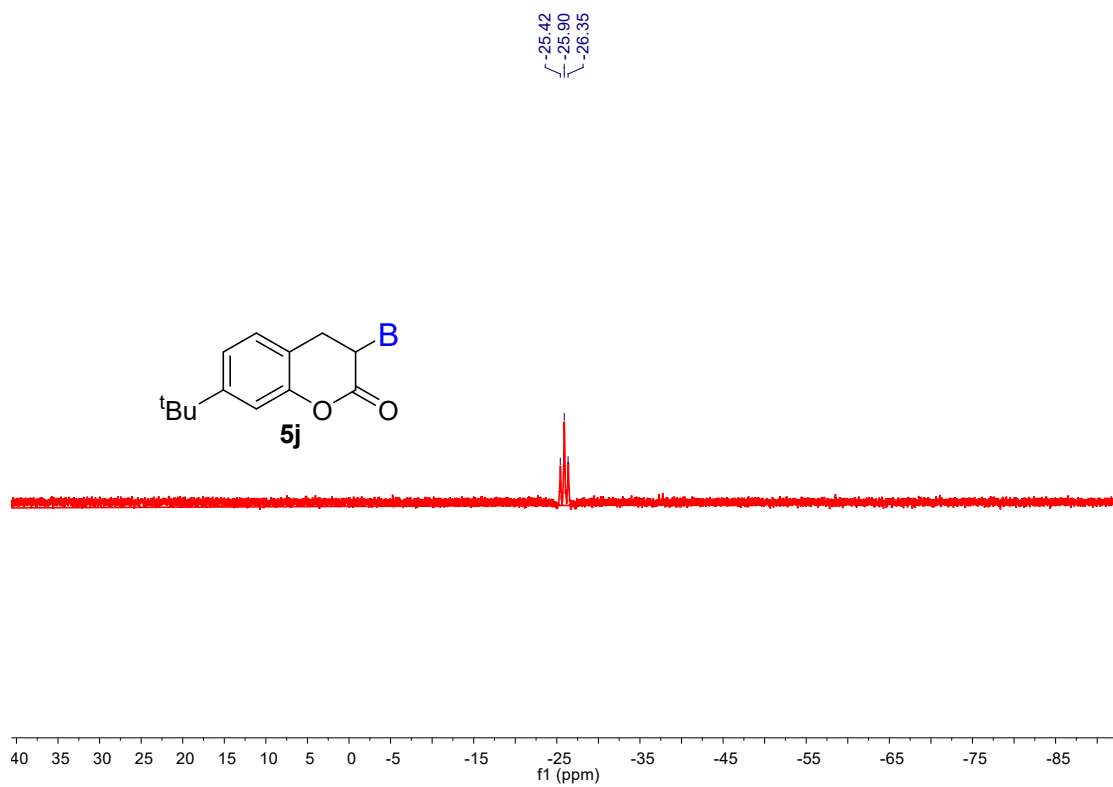
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

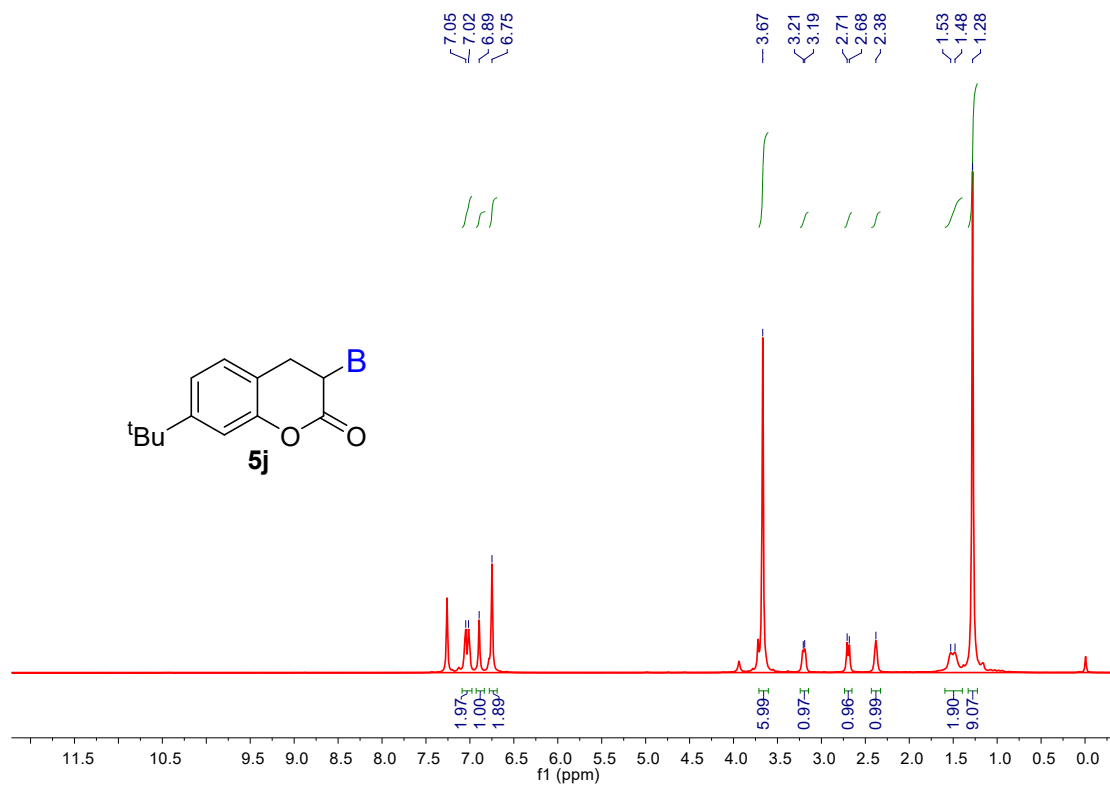


¹H NMR (600 MHz, CDCl₃)

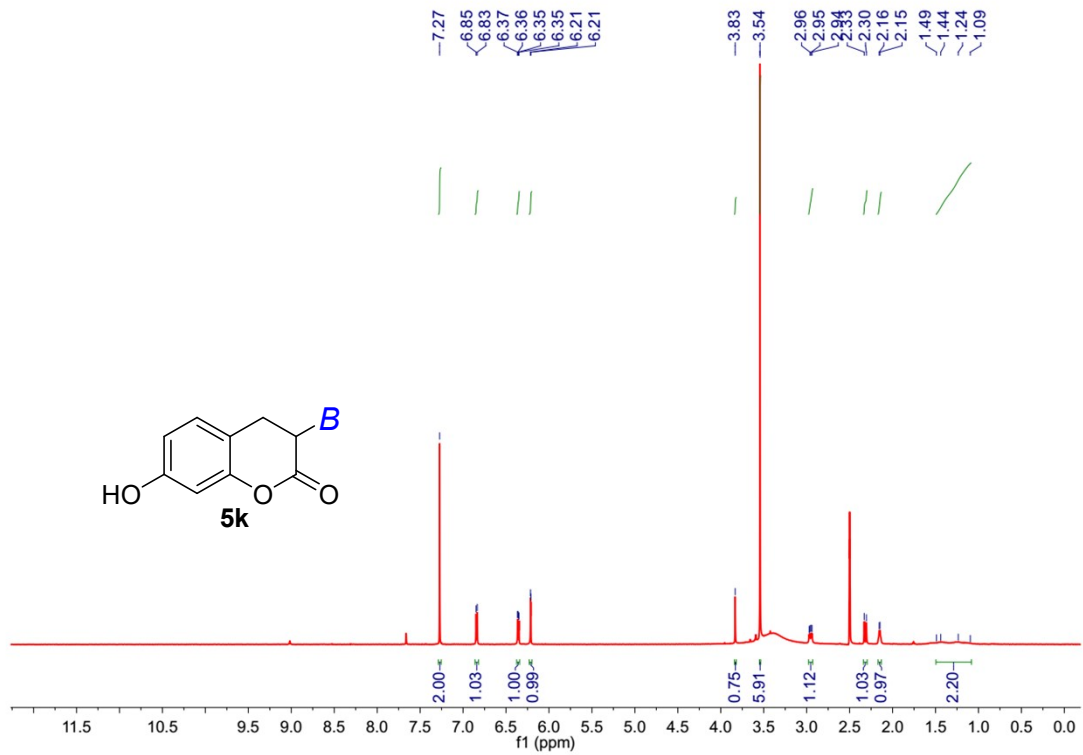


¹³C NMR (151 MHz, CDCl₃)





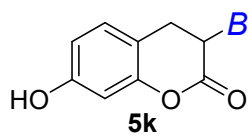
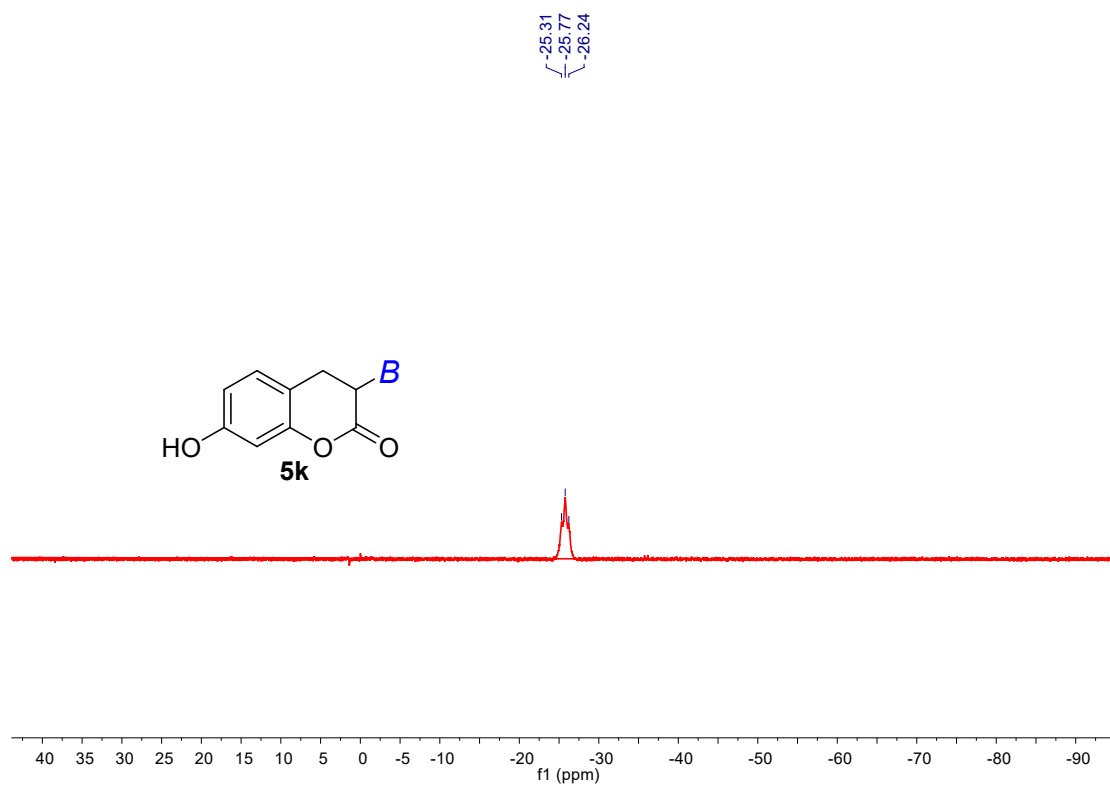
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



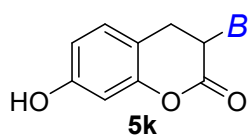
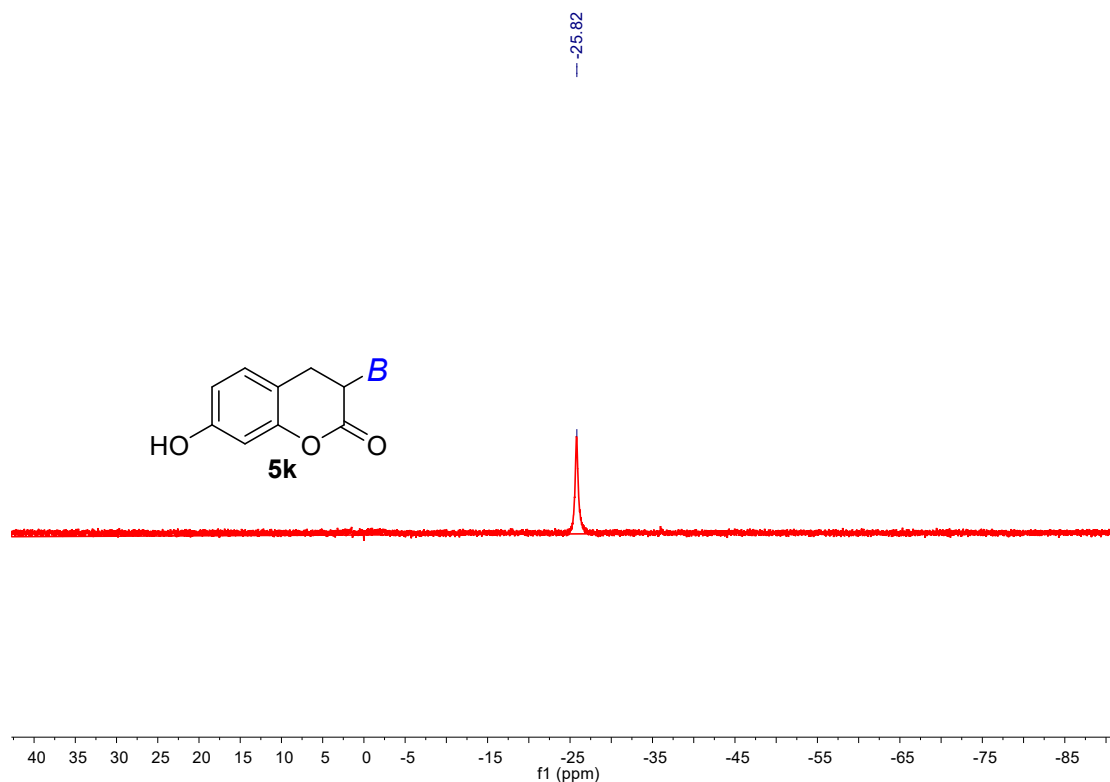
¹H NMR (600 MHz, DMSO-*d*₆)



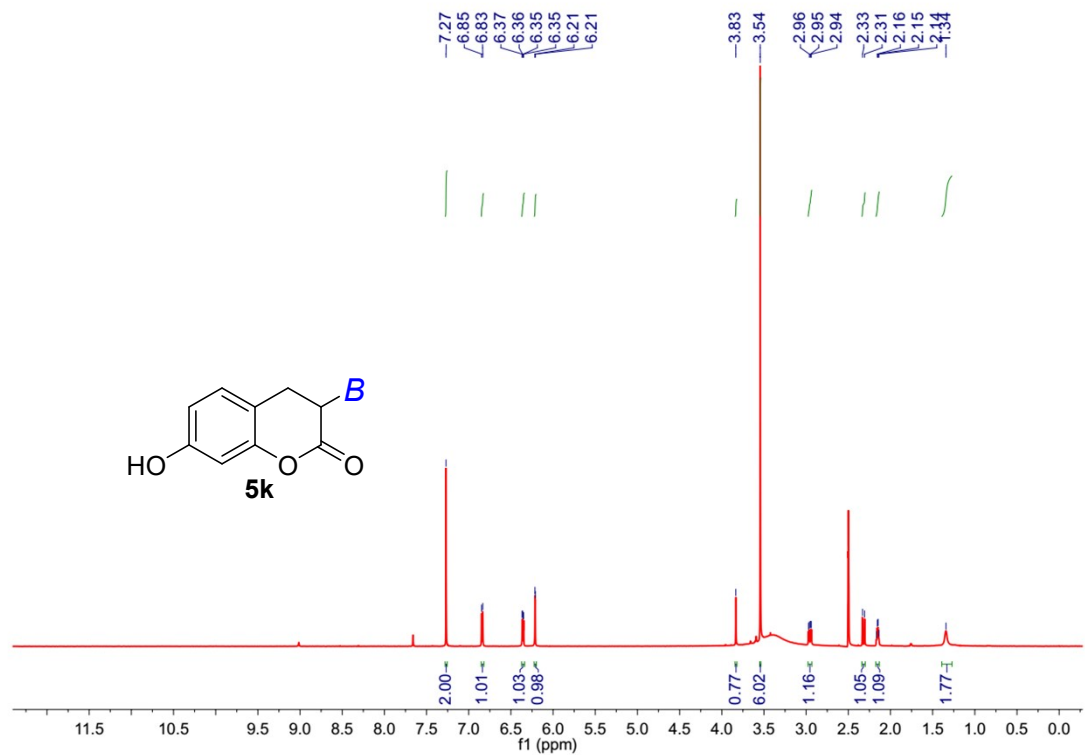
¹³C NMR (151 MHz, DMSO-*d*₆)



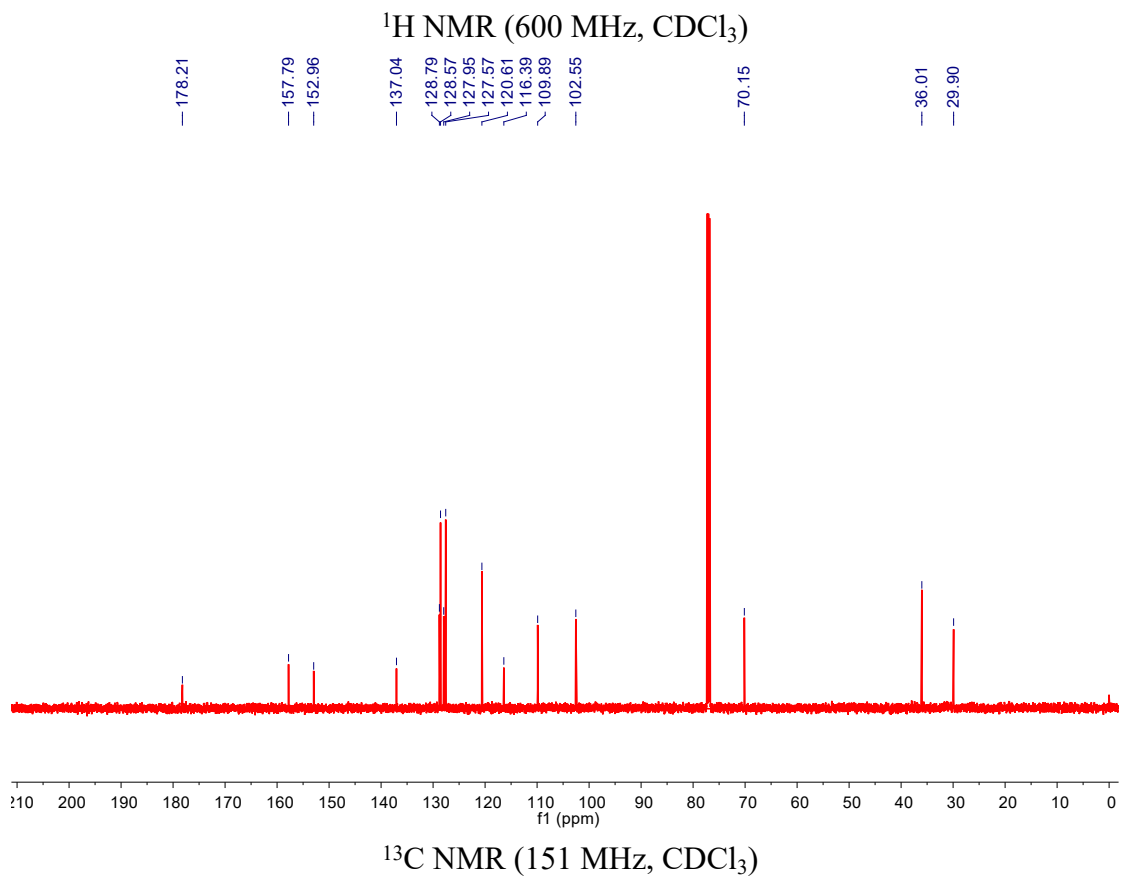
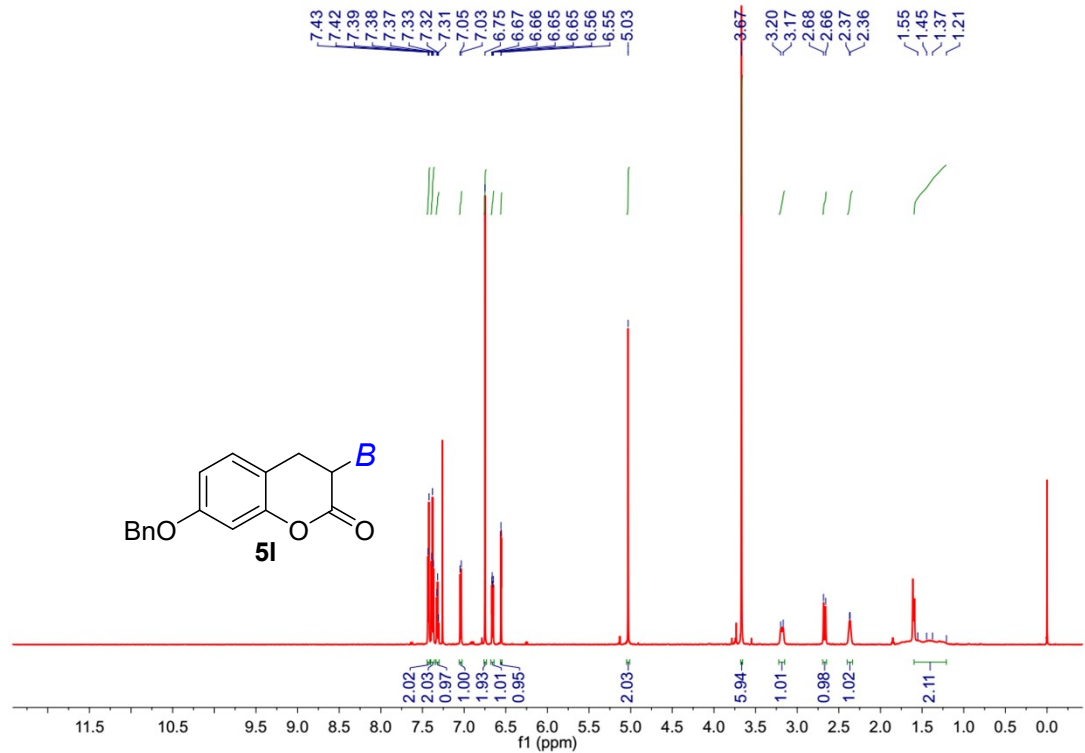
¹¹B NMR (193 MHz, DMSO-*d*₆)

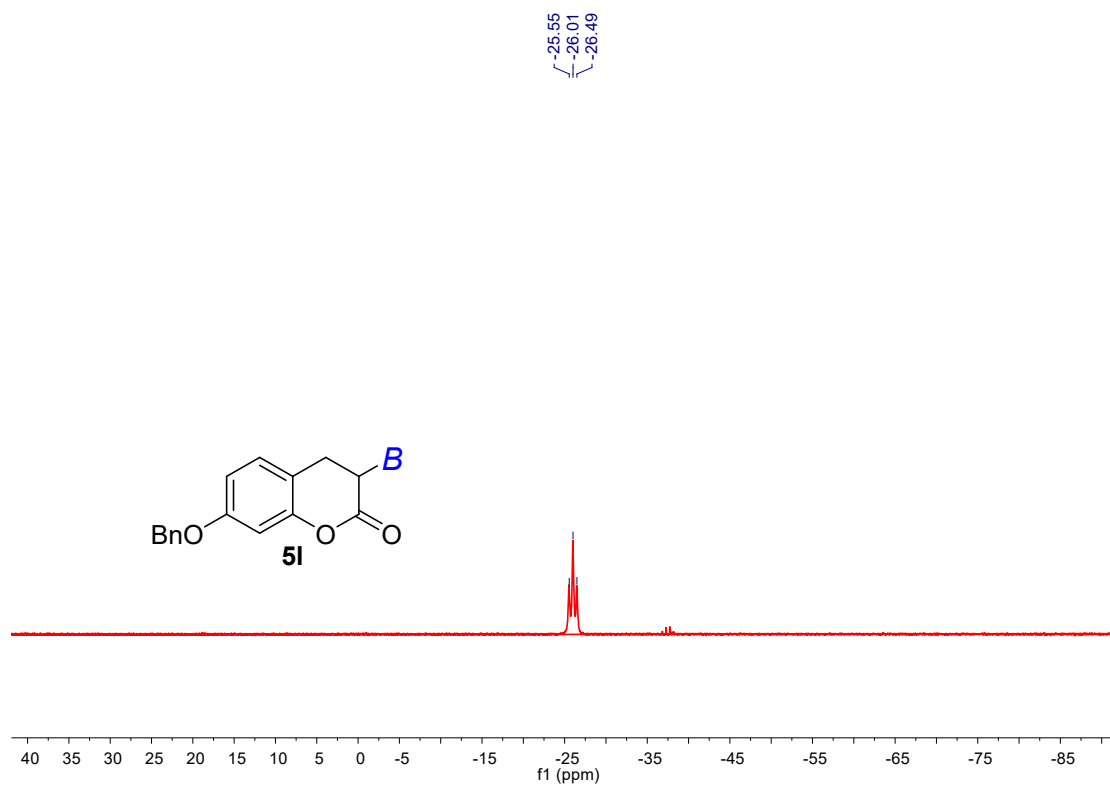


¹¹B{¹H} NMR (193 MHz, DMSO-*d*₆)

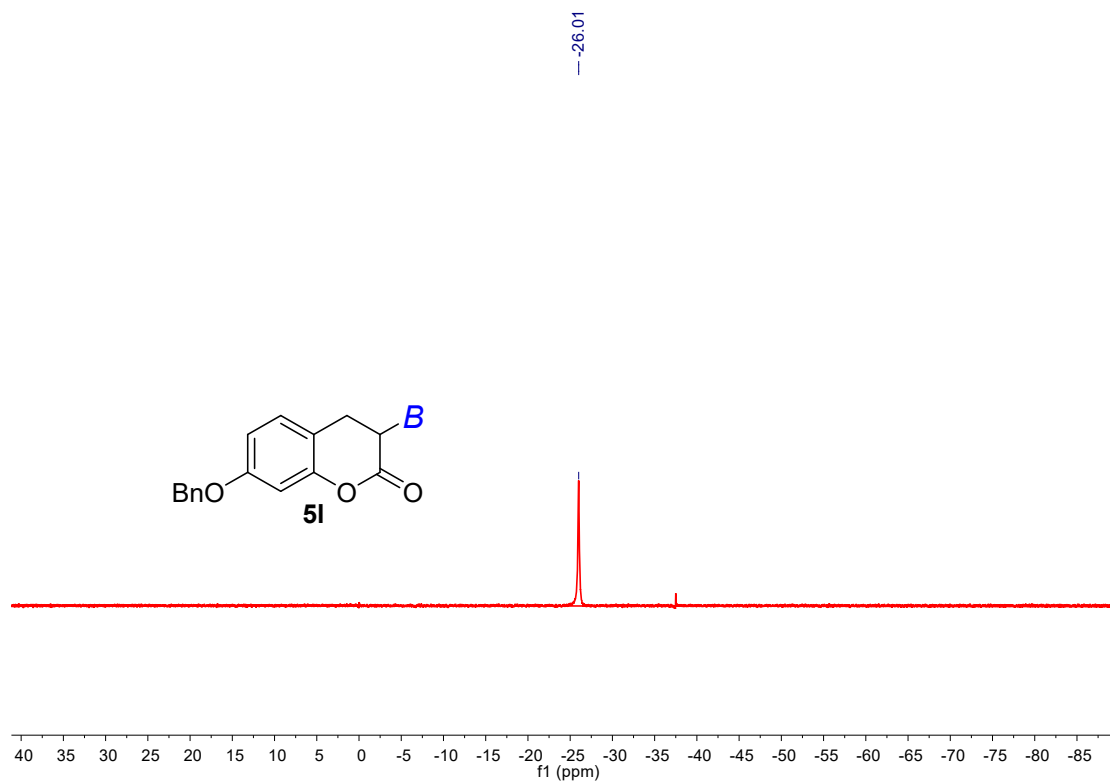


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, $\text{DMSO-}d_6$)

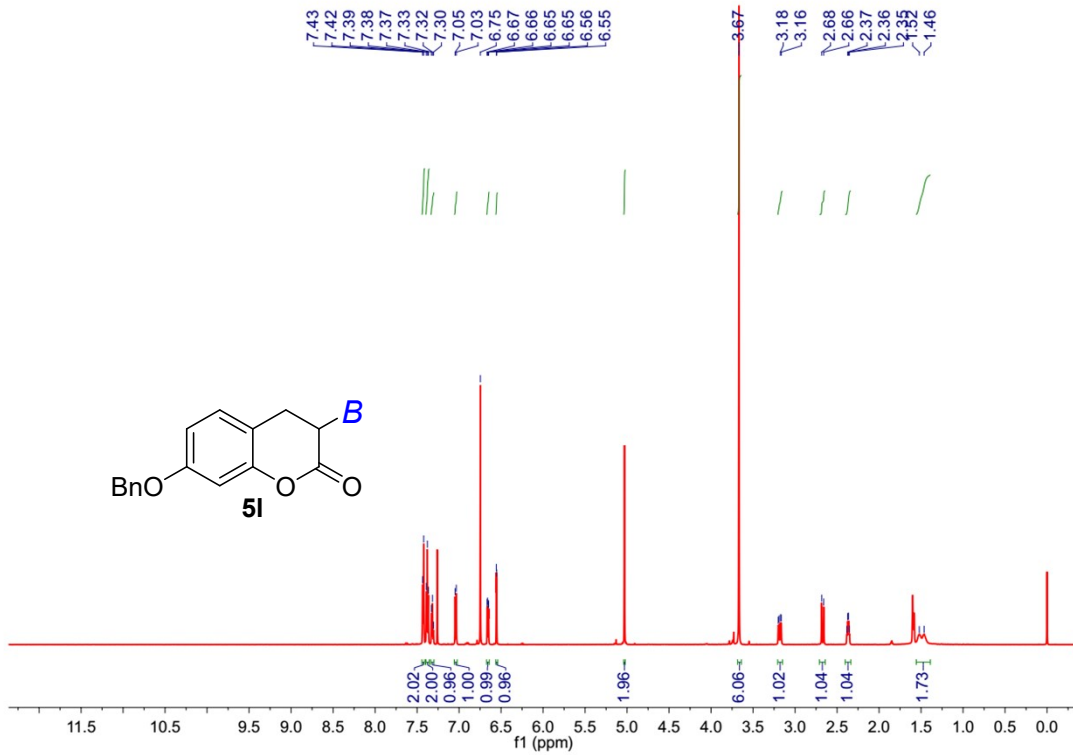




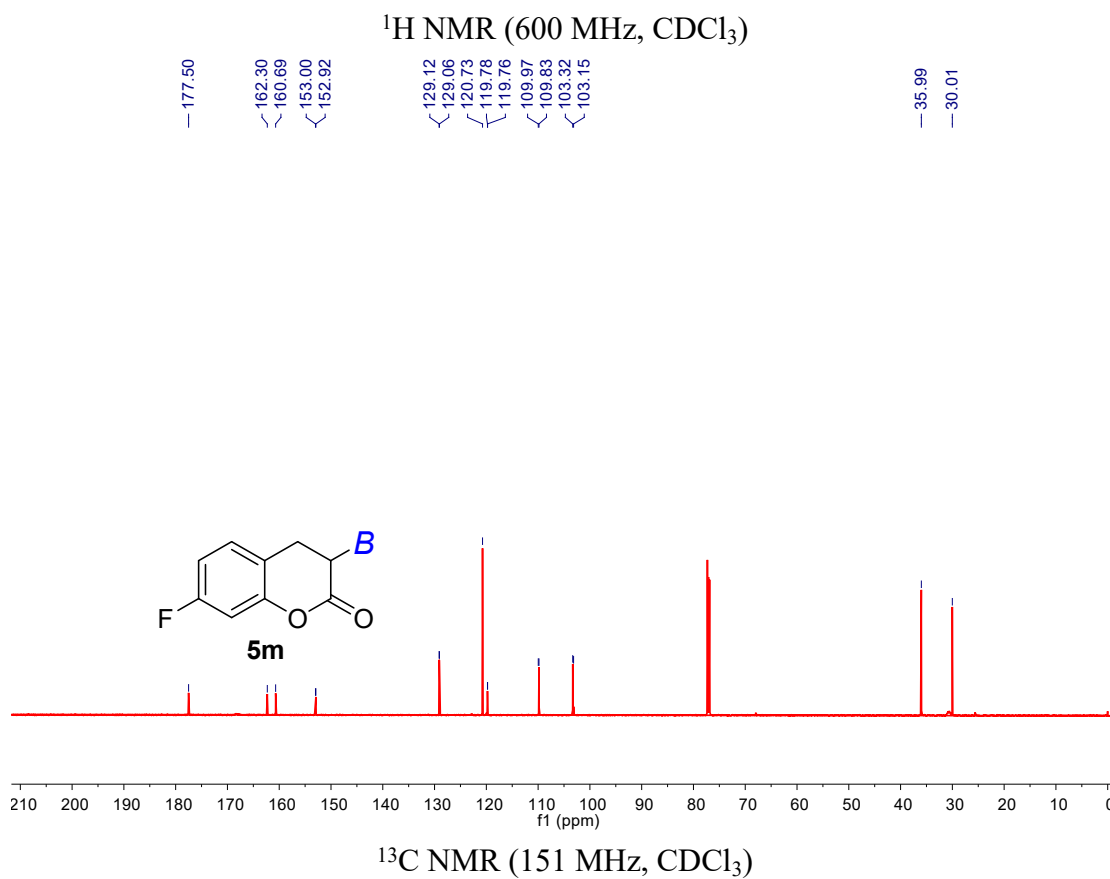
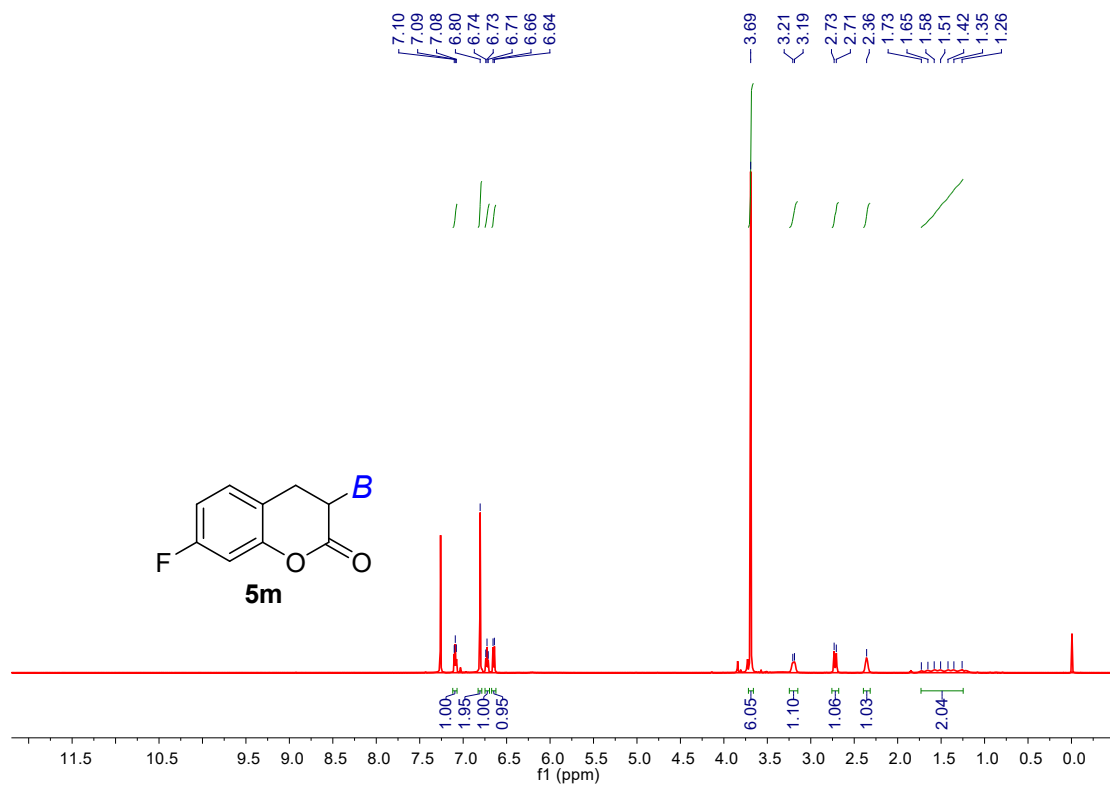
^{11}B NMR (193 MHz, CDCl_3)

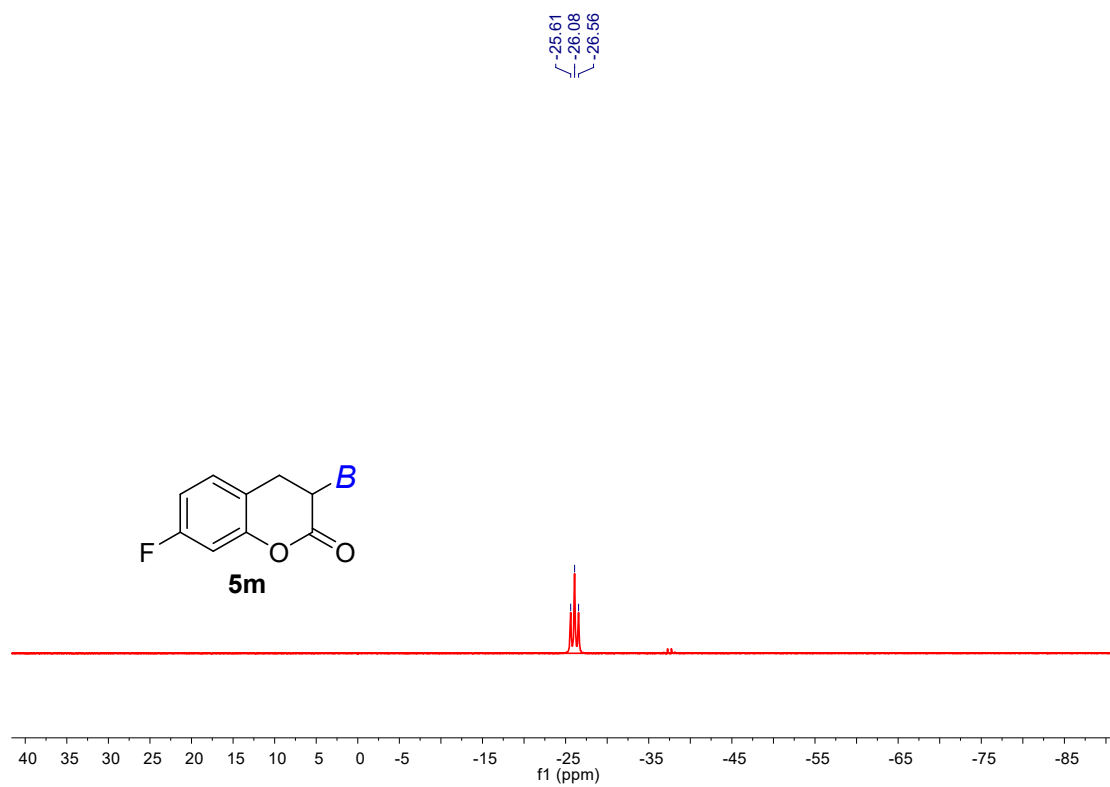


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

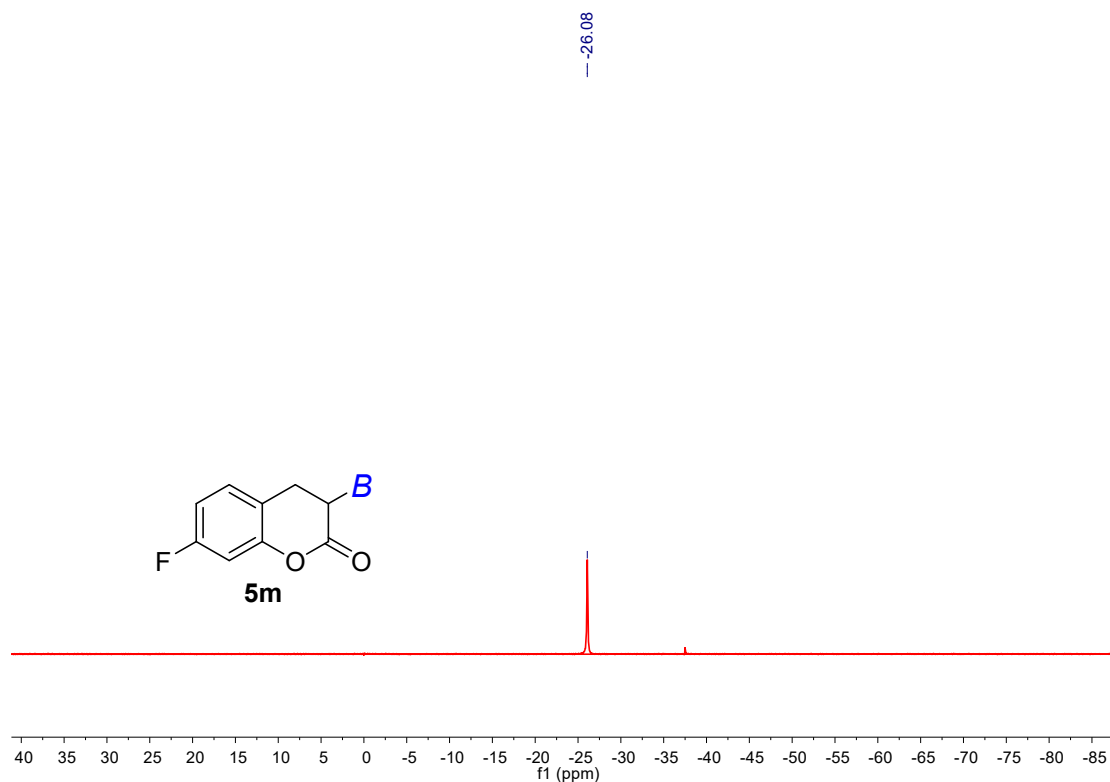


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

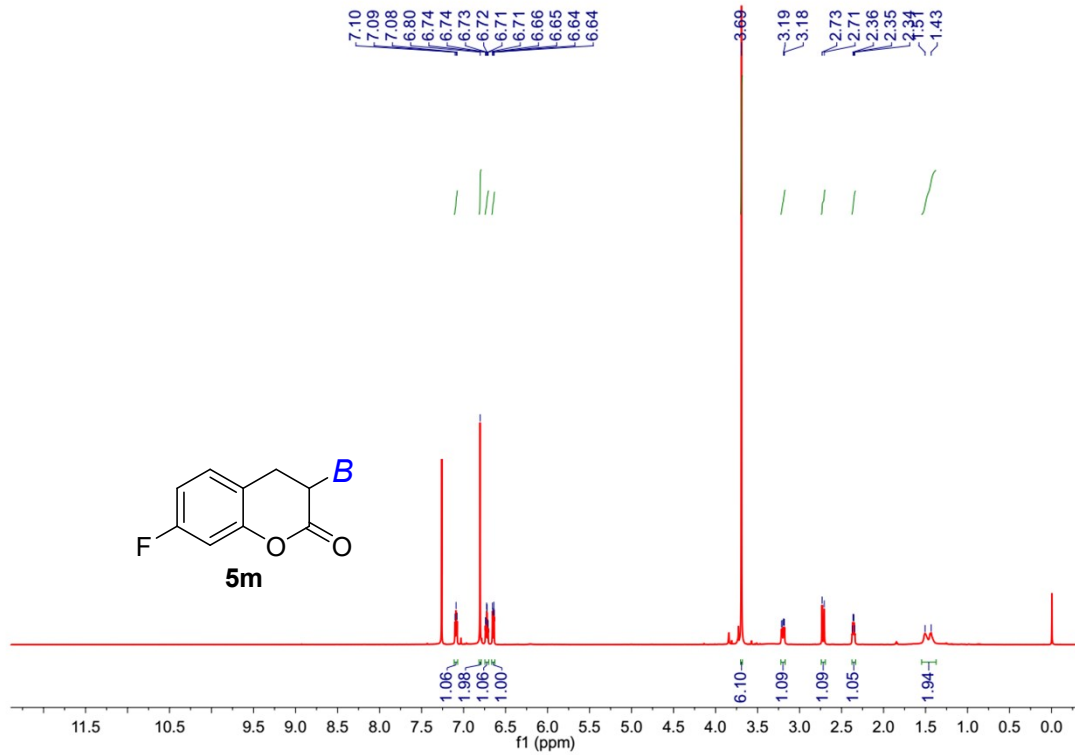




^{11}B NMR (193 MHz, CDCl_3)

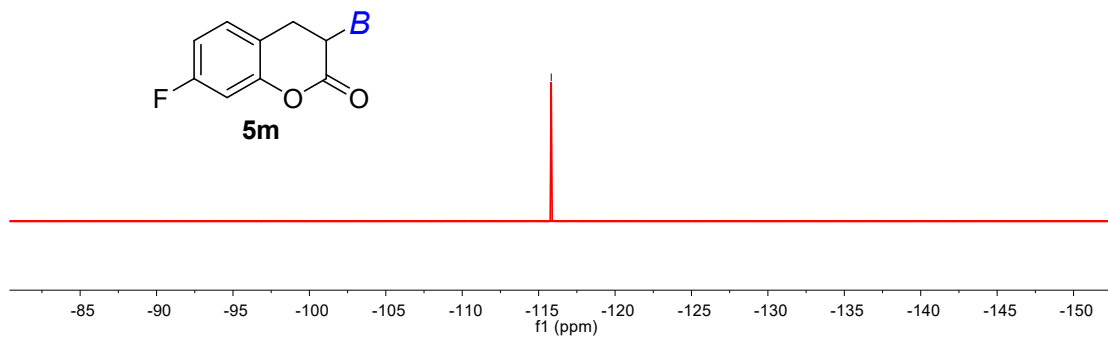


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

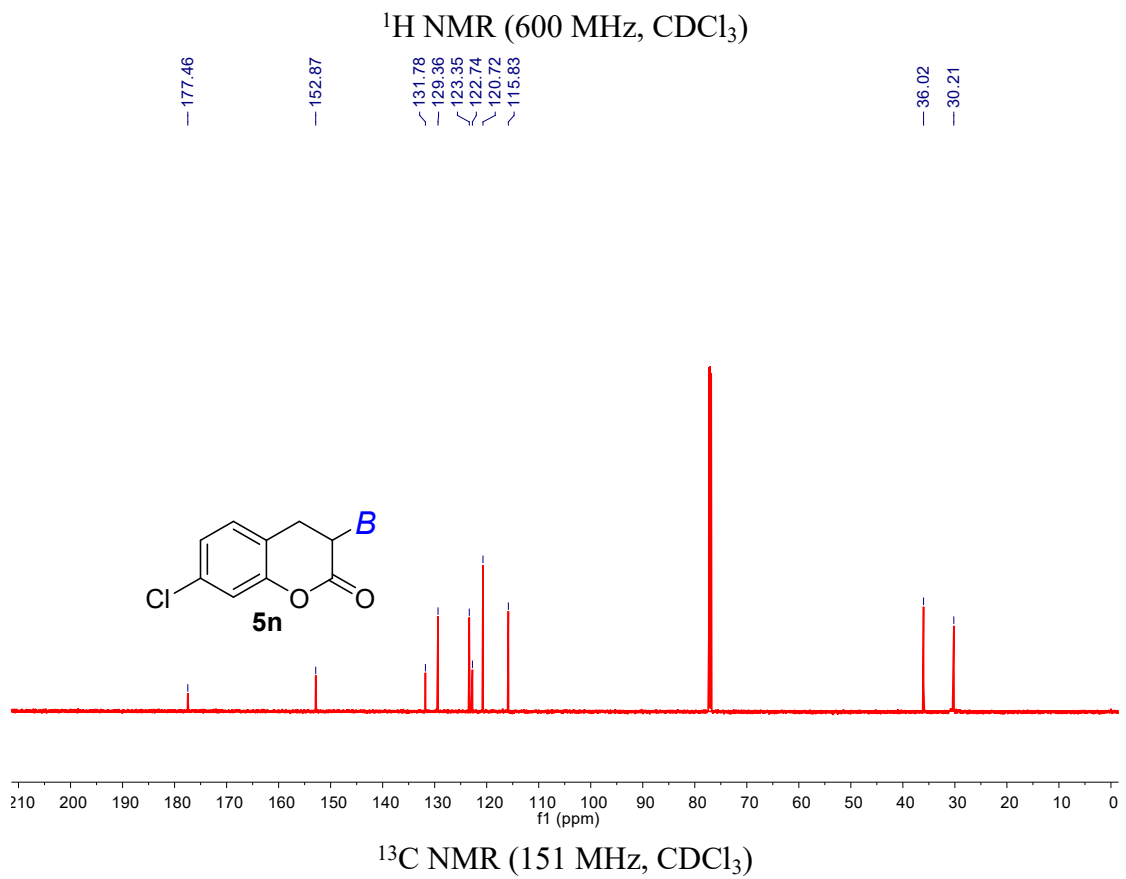
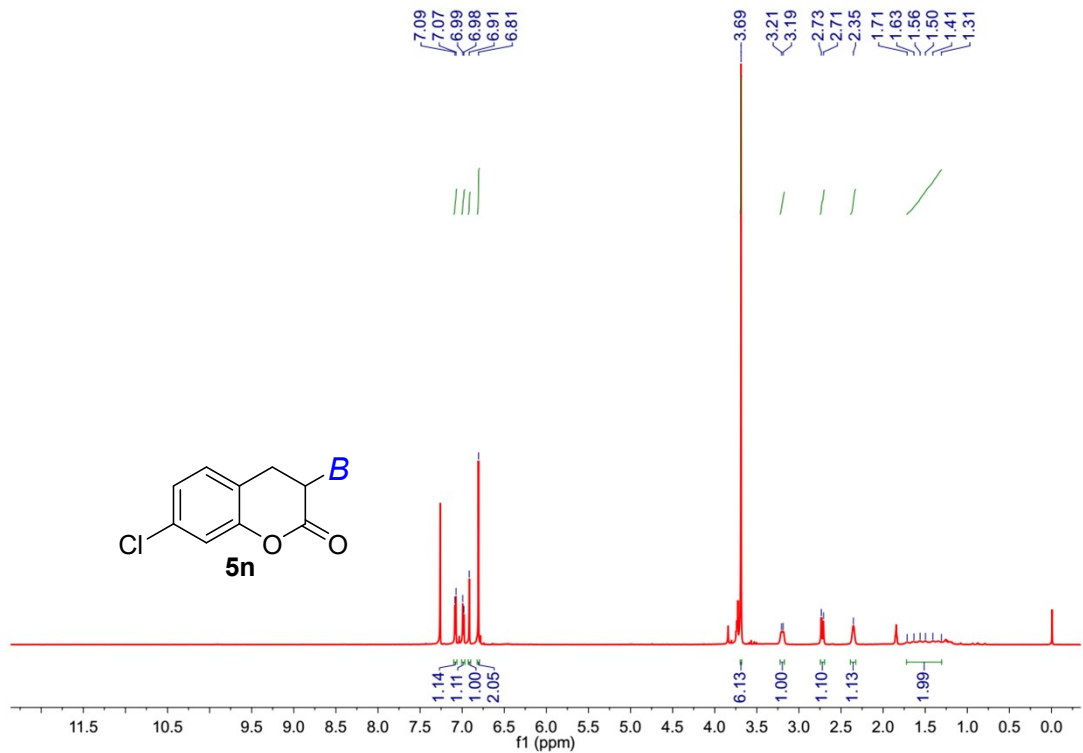


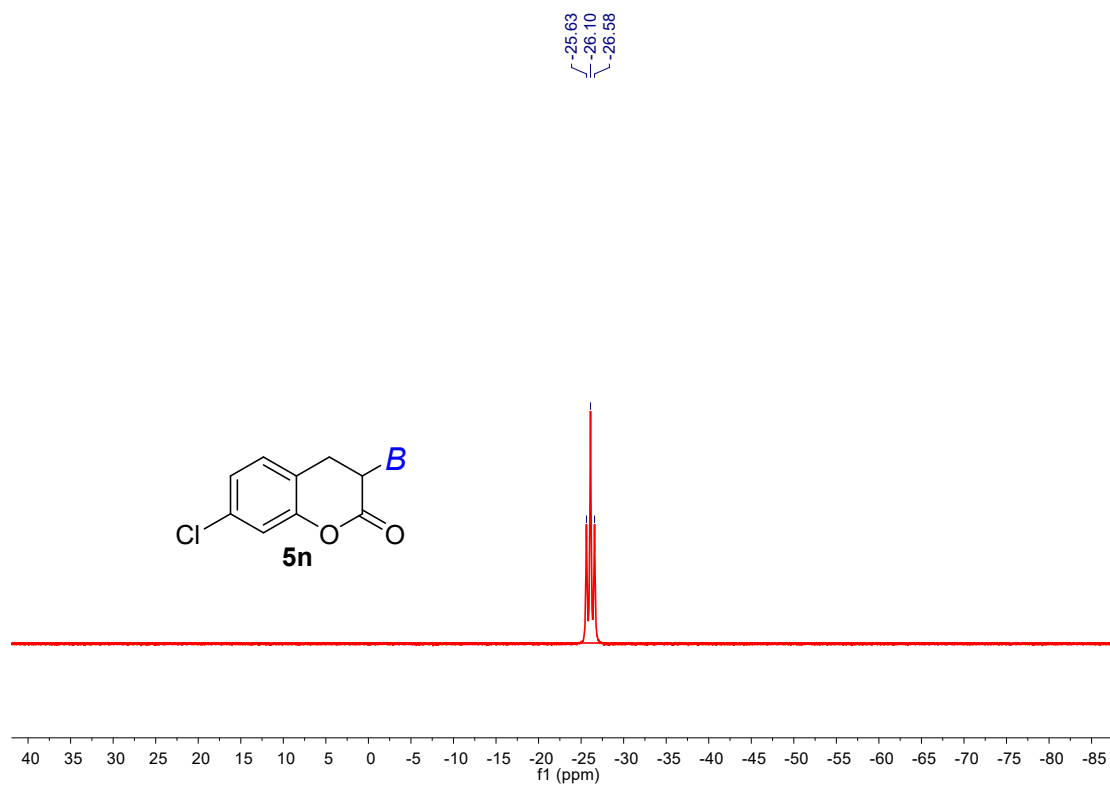
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

---115.82

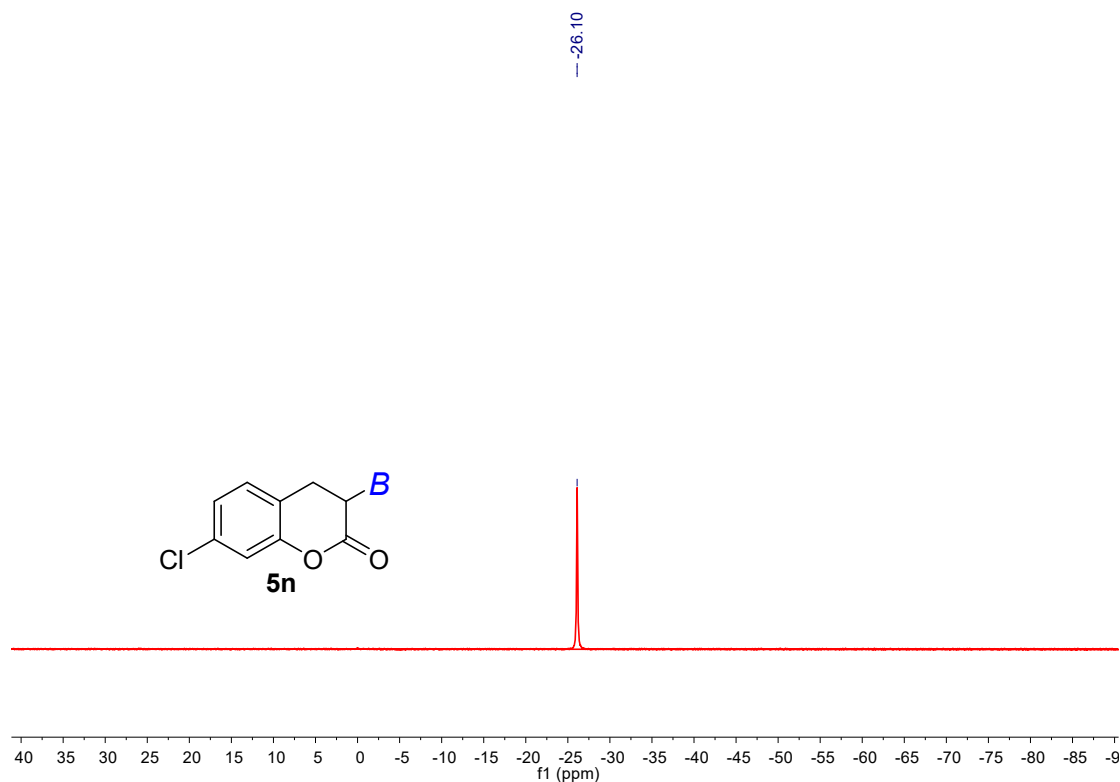


^{19}F NMR (565 MHz, CDCl_3)

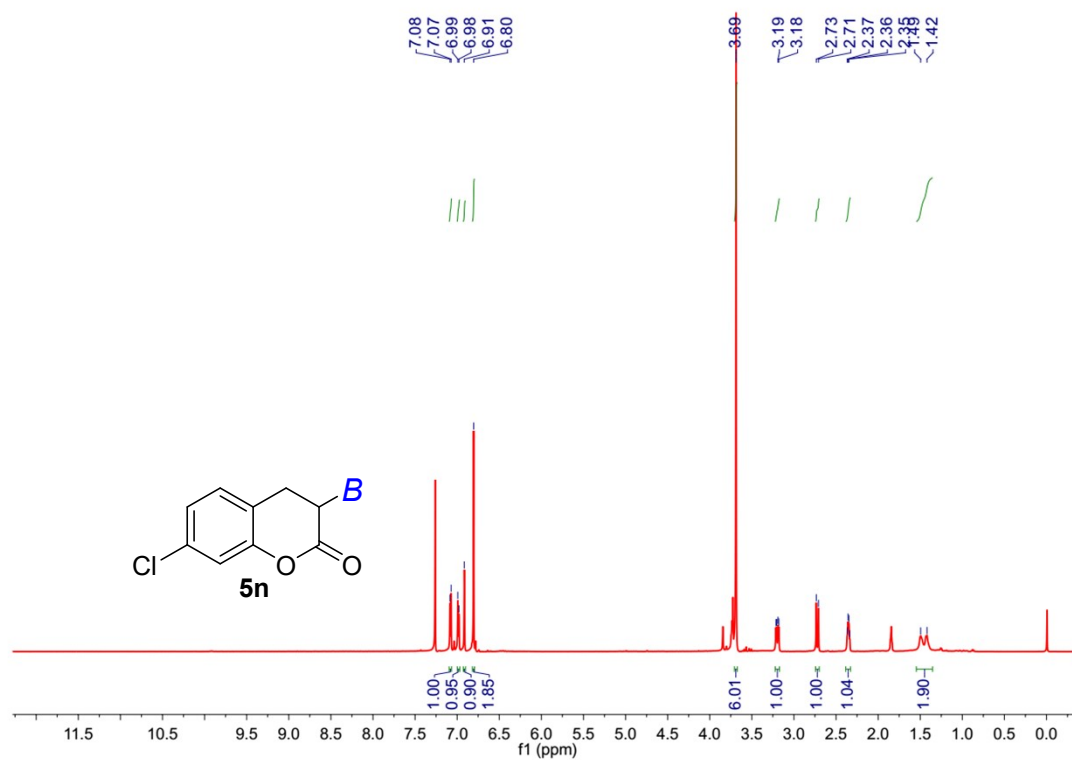




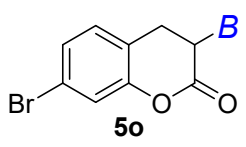
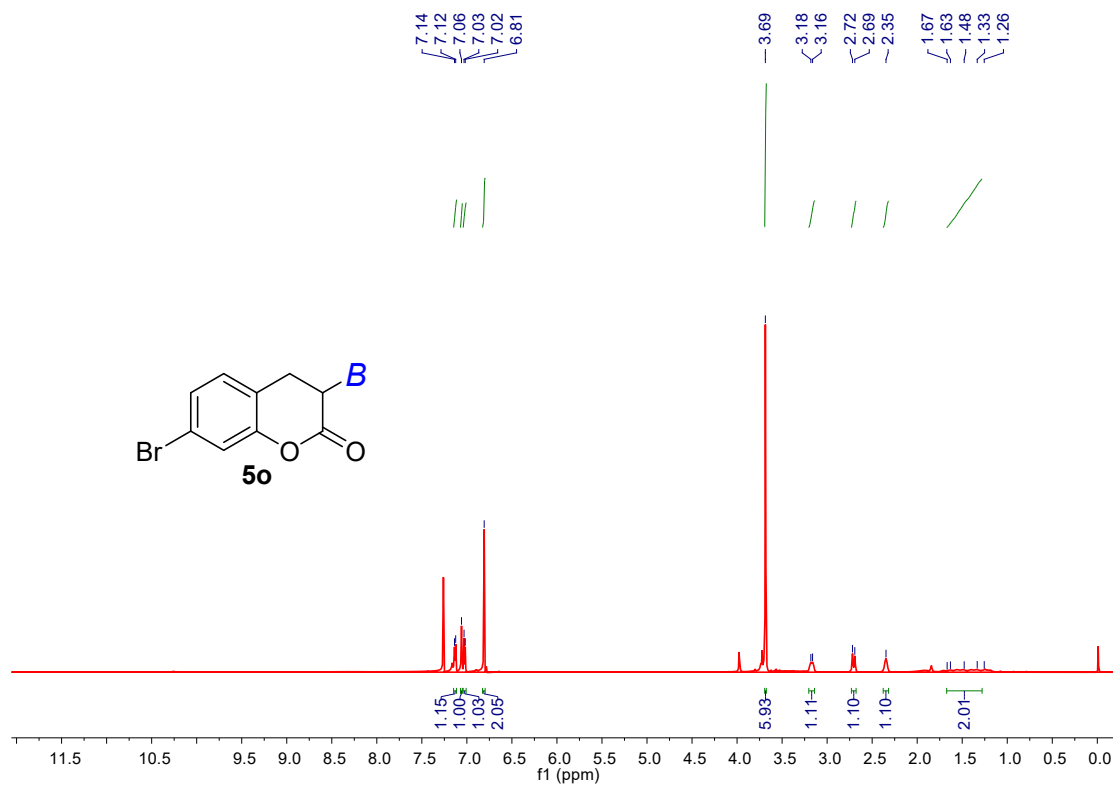
^{11}B NMR (193 MHz, CDCl_3)



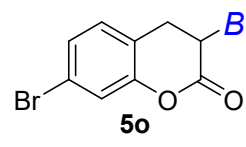
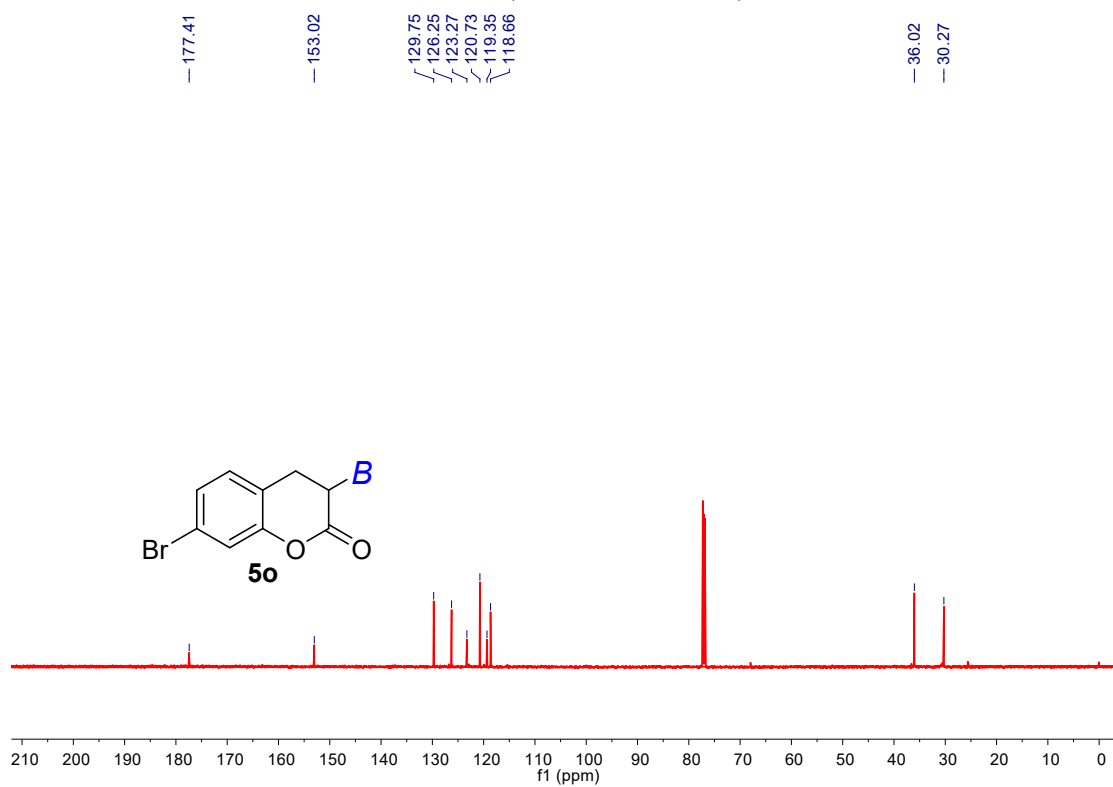
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



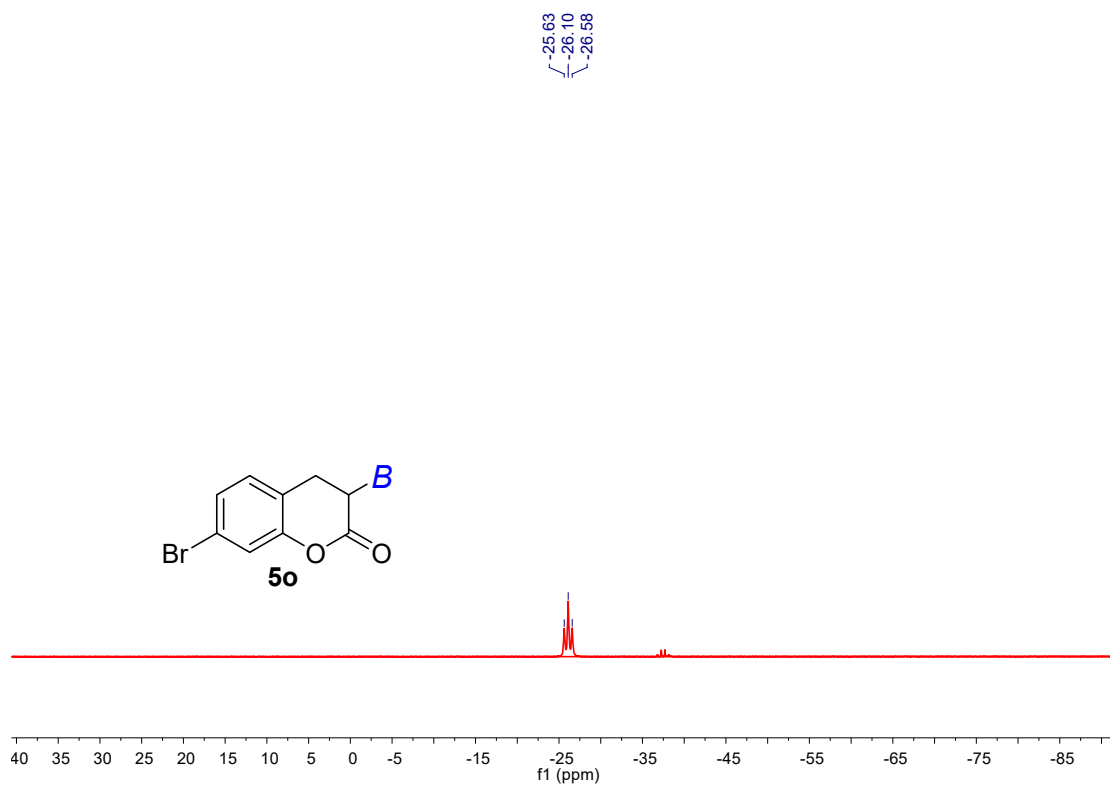
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



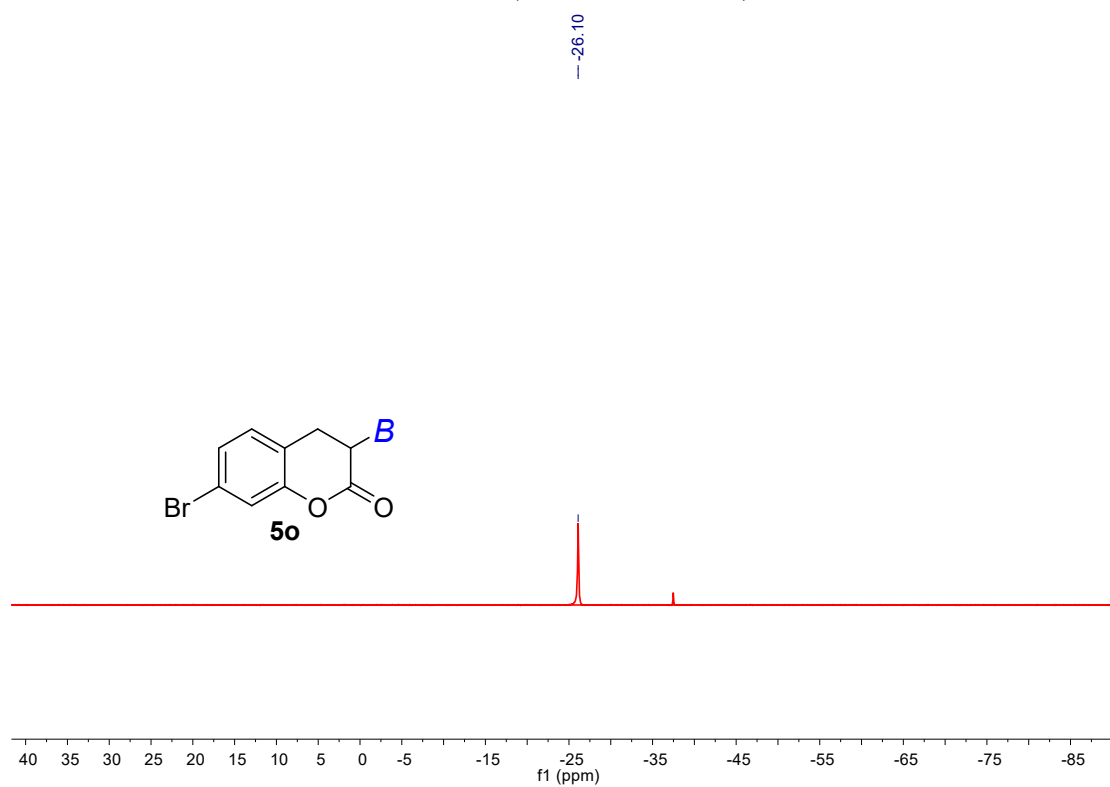
¹H NMR (600 MHz, CDCl₃)



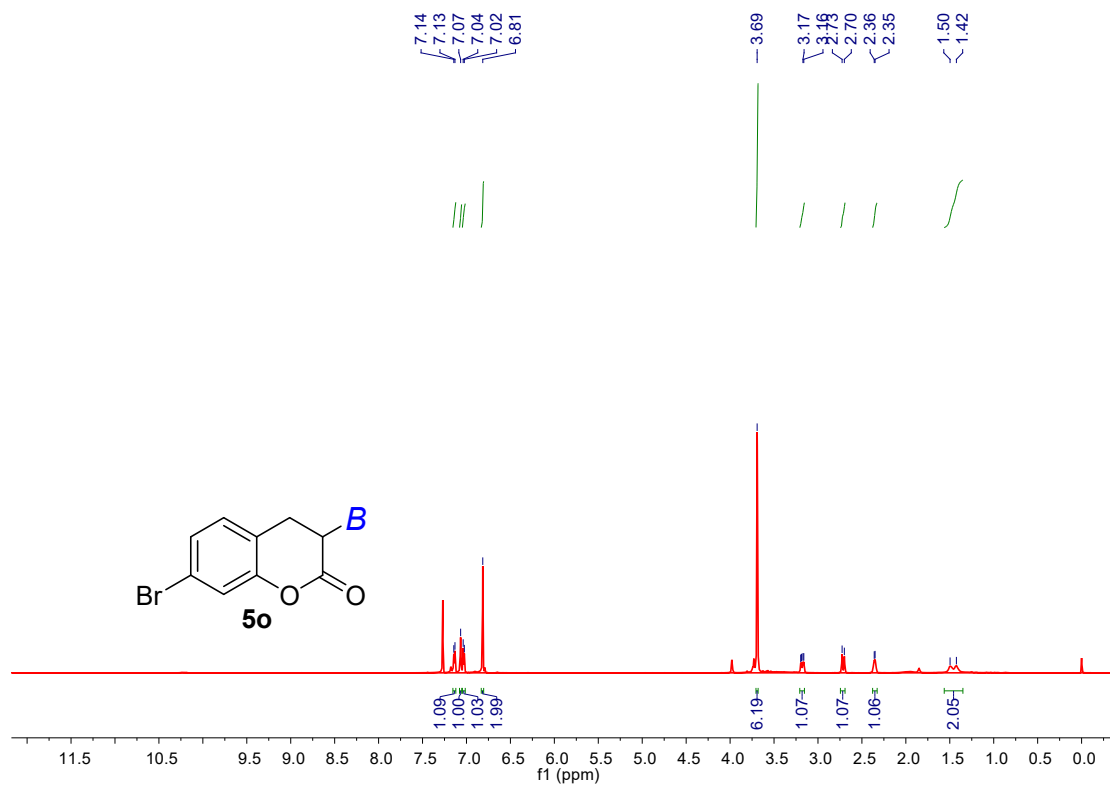
¹³C NMR (151 MHz, CDCl₃)



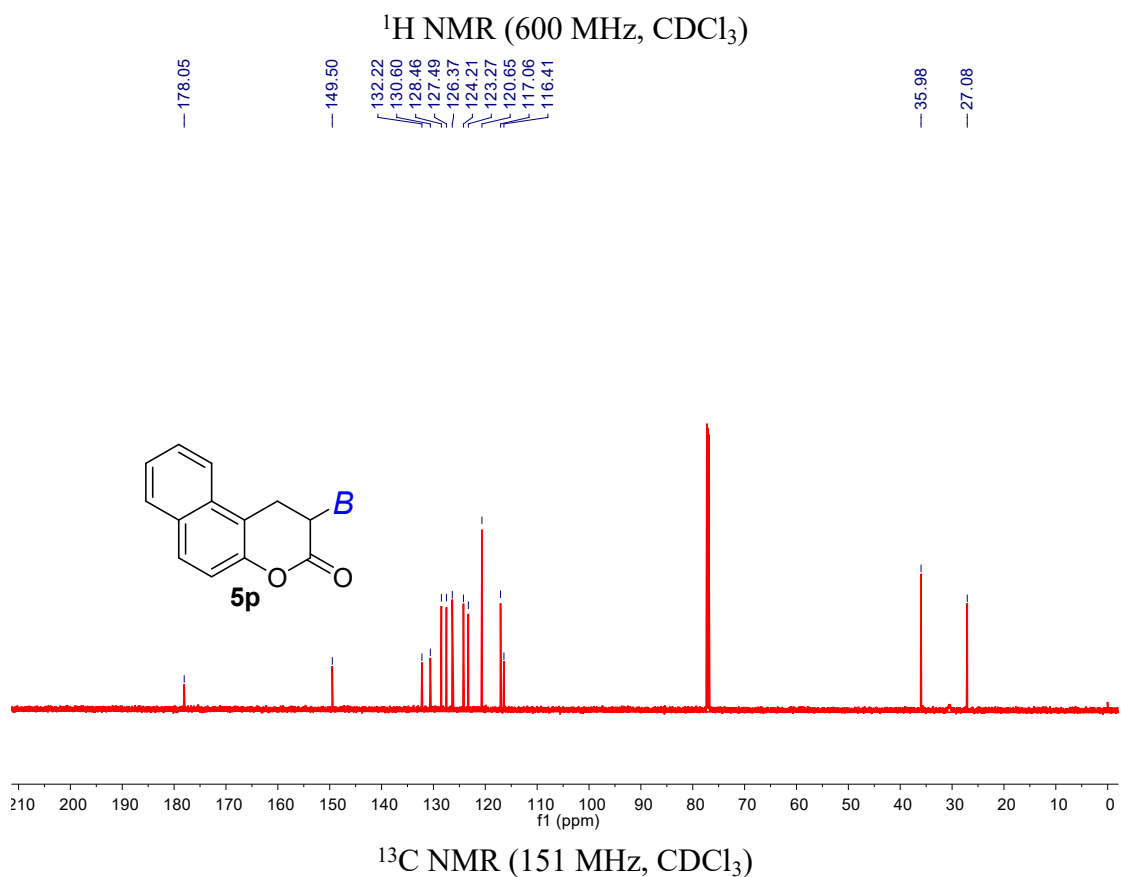
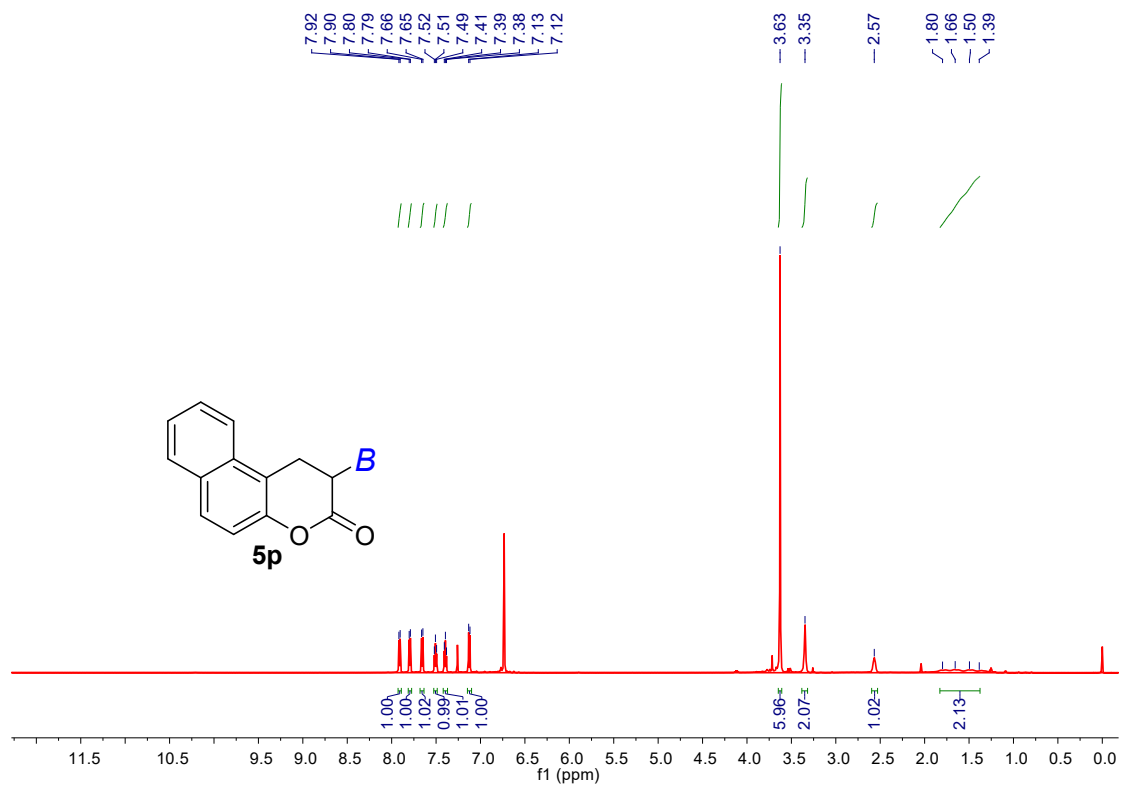
^{11}B NMR (193 MHz, CDCl_3)



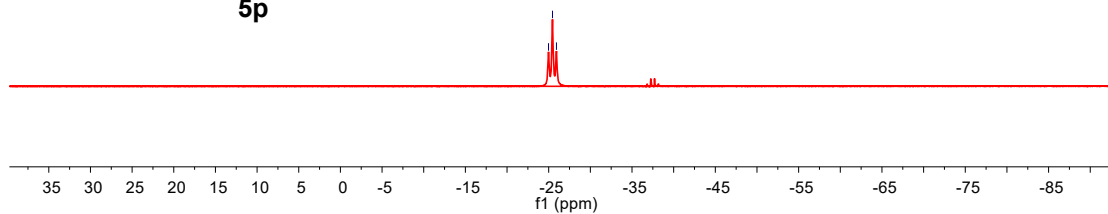
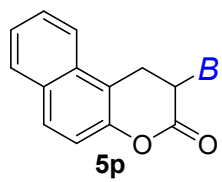
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

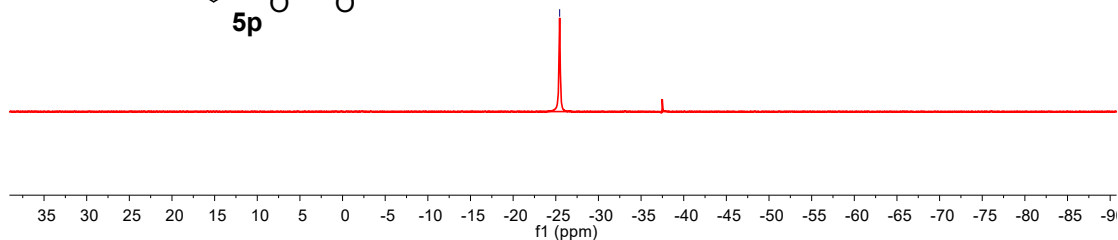
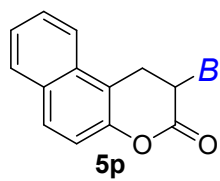


-24.98
-25.46
-25.93

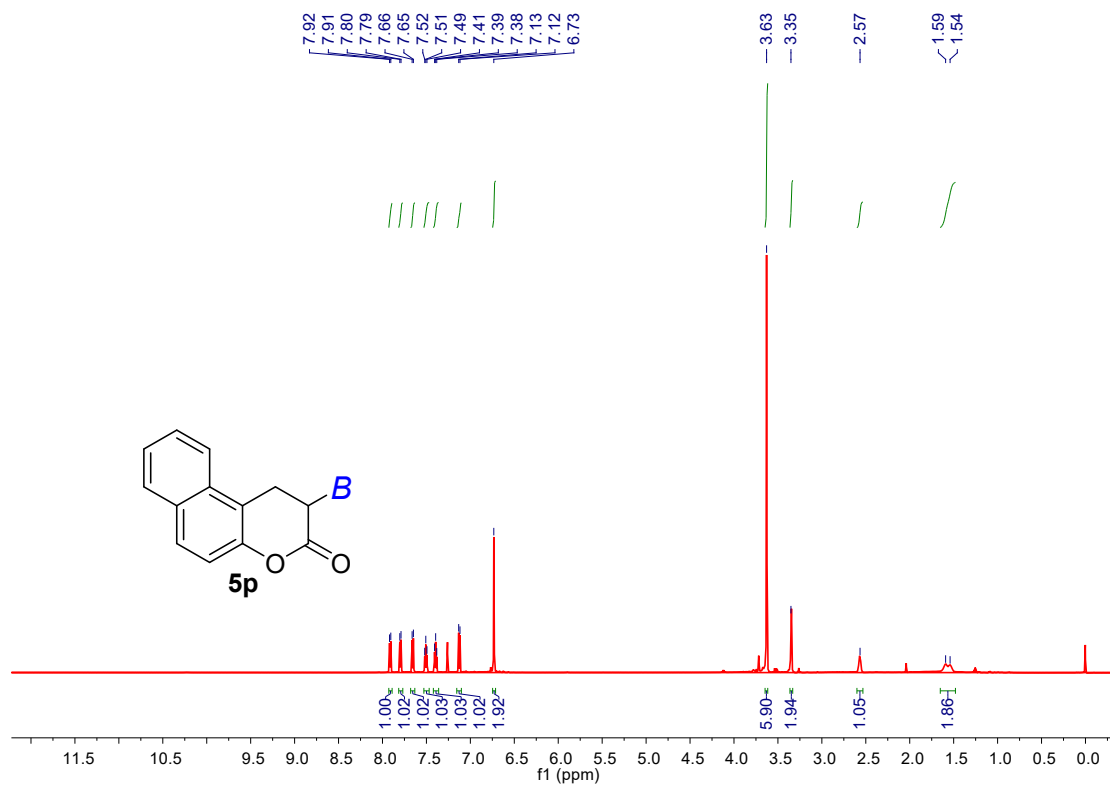


^{11}B NMR (193 MHz, CDCl_3)

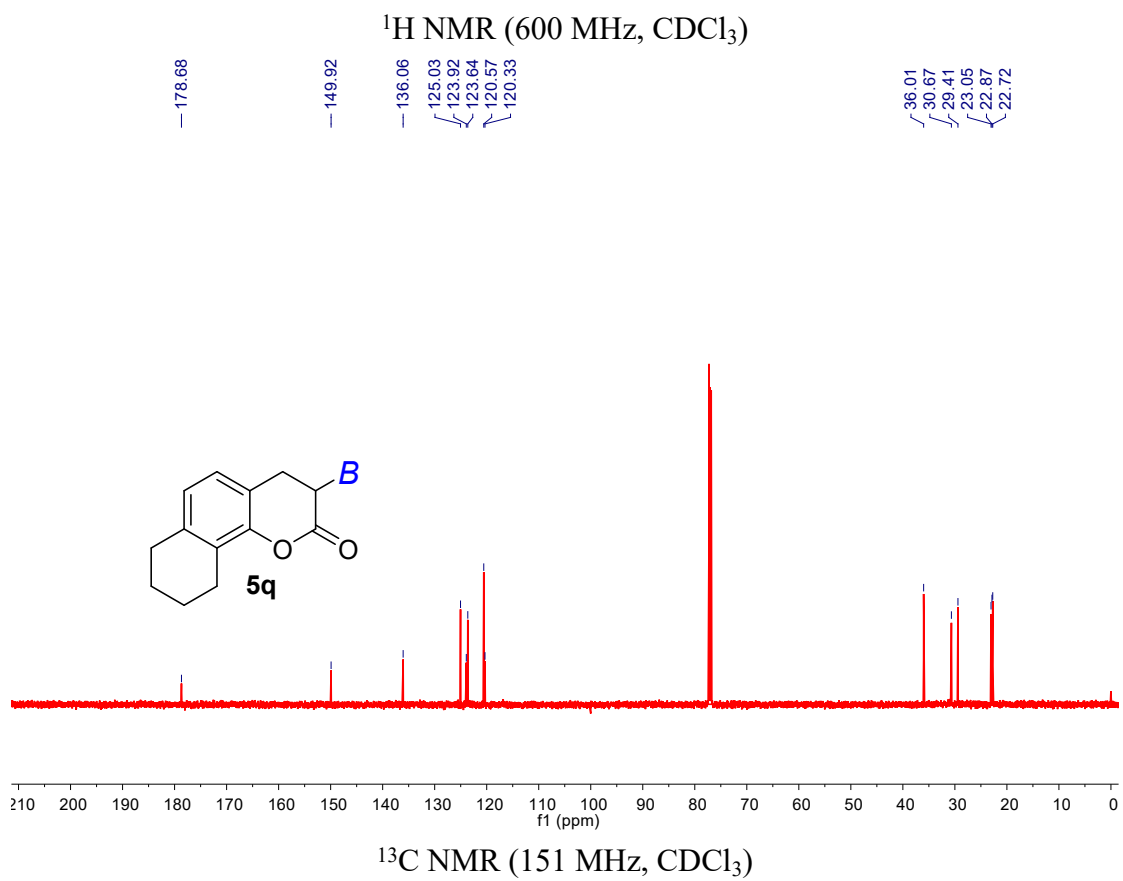
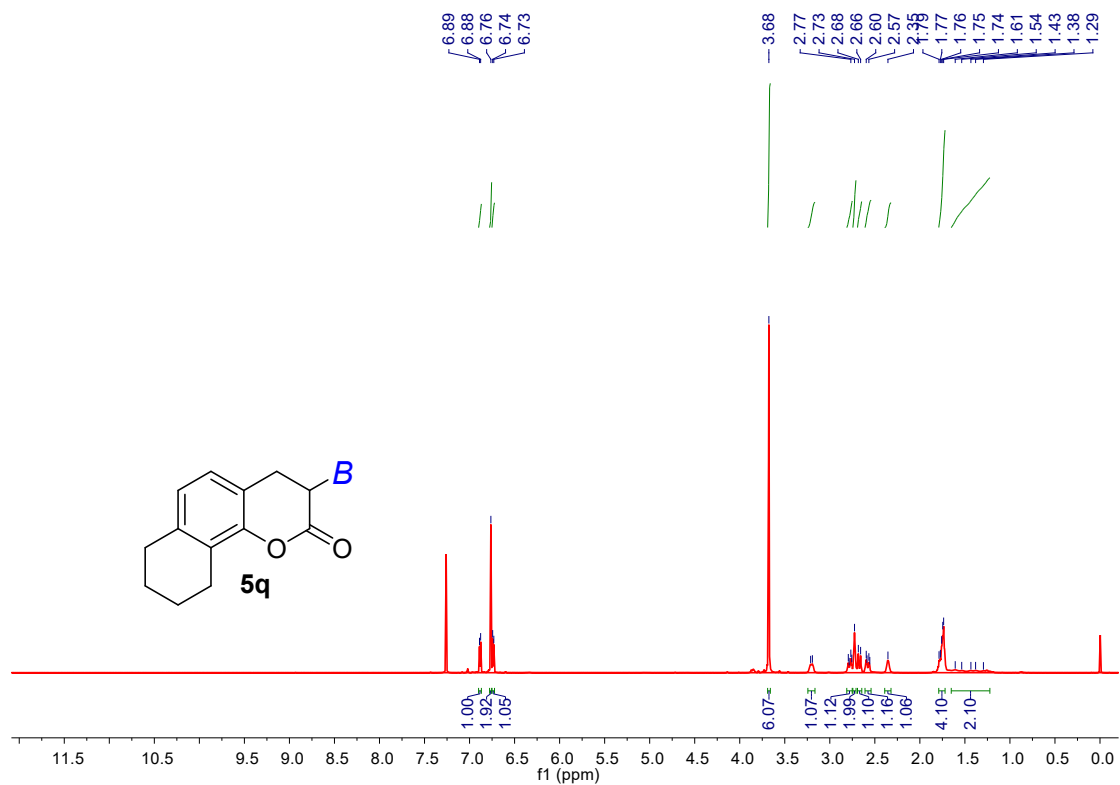
-25.45

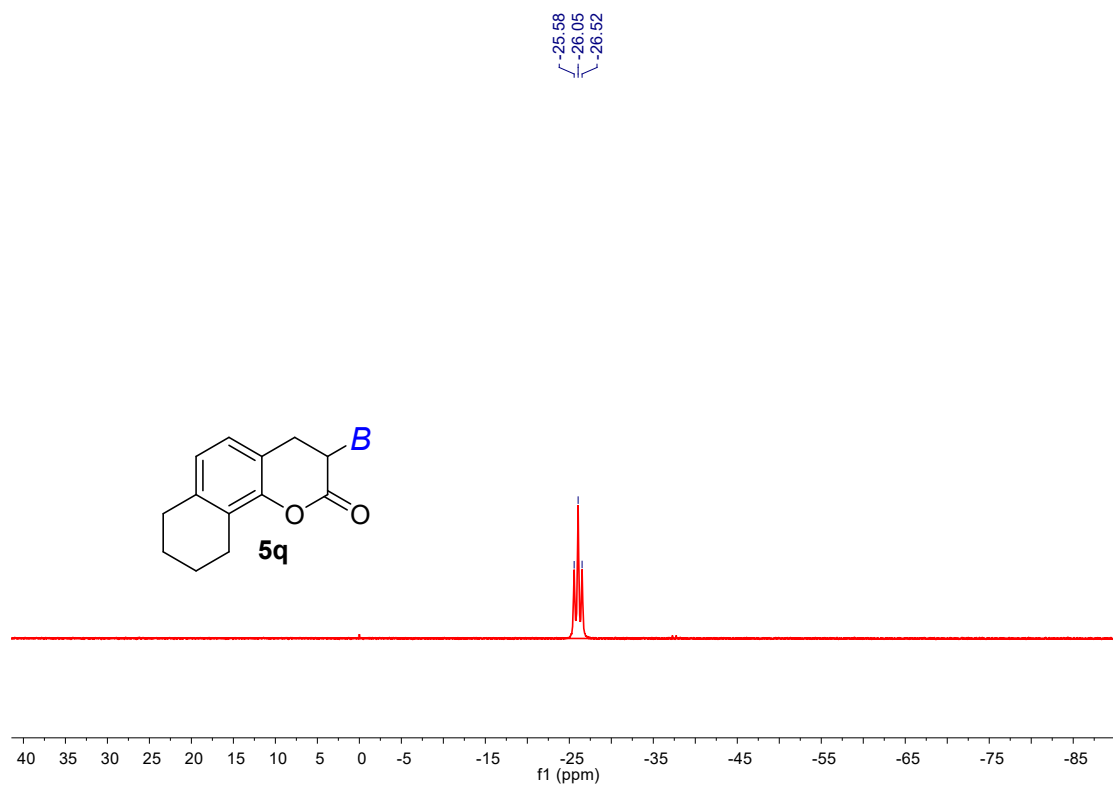


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

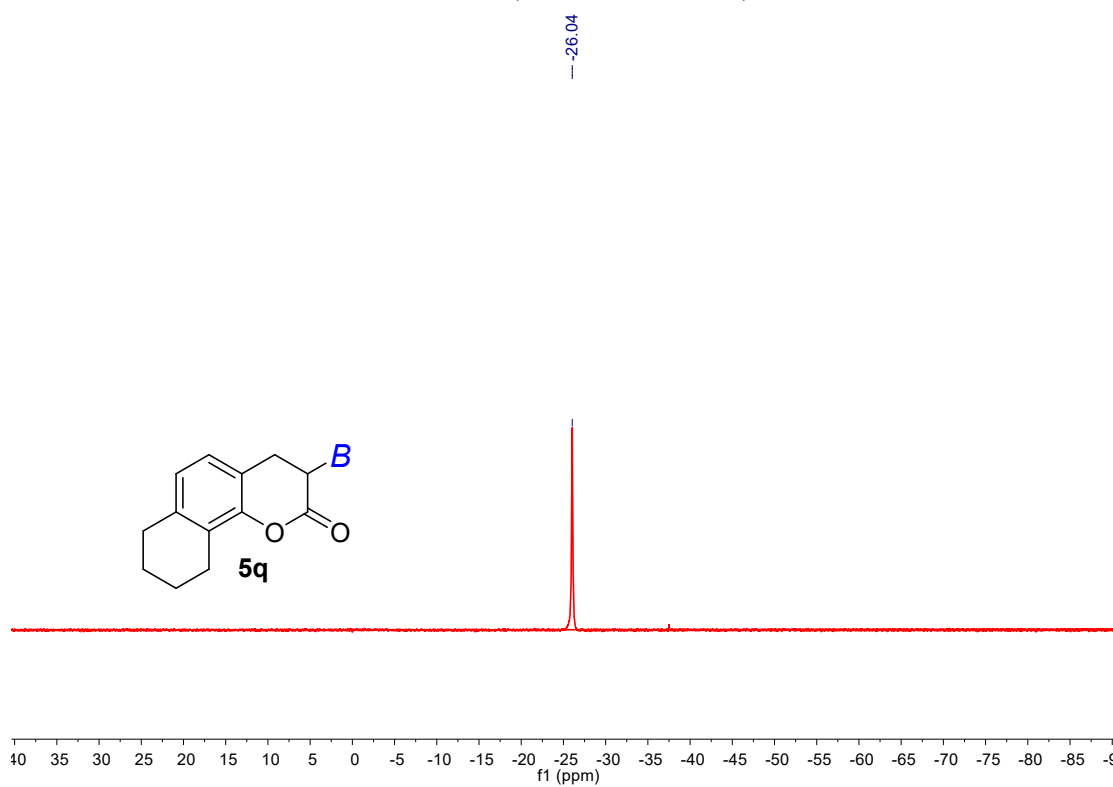


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

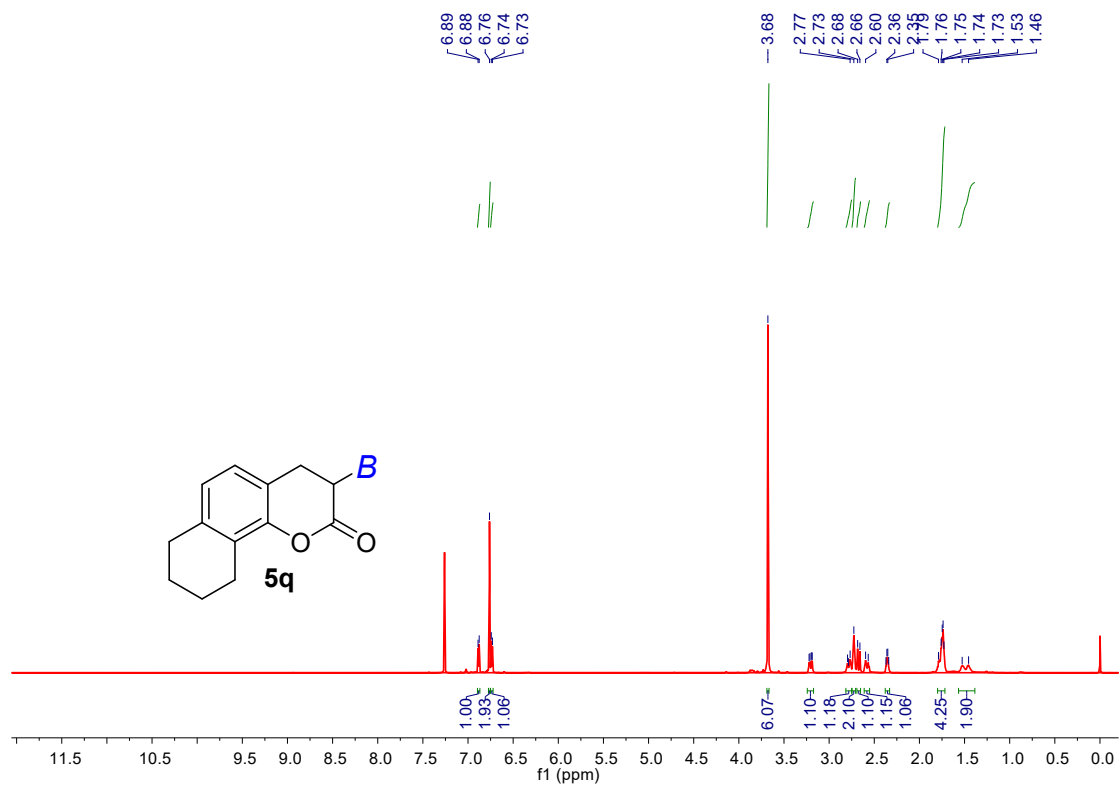




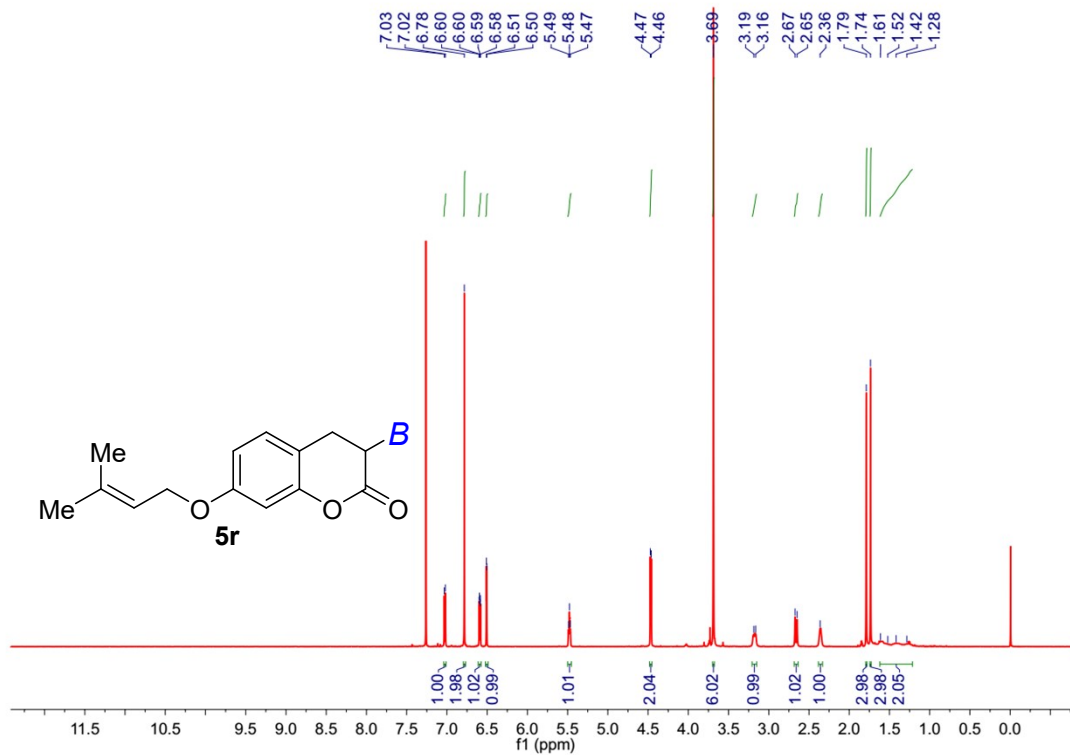
^{11}B NMR (193 MHz, CDCl_3)



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

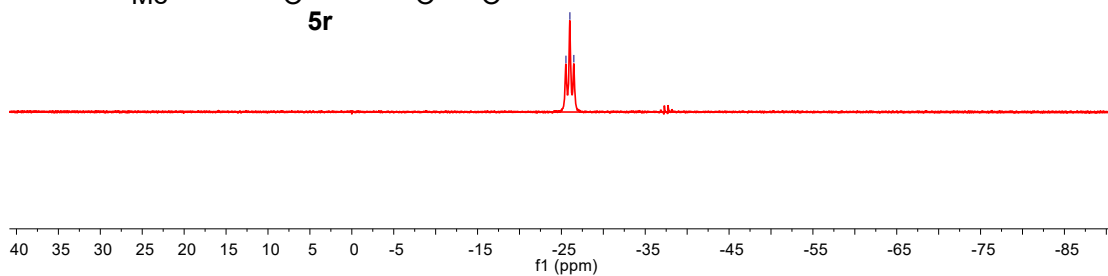
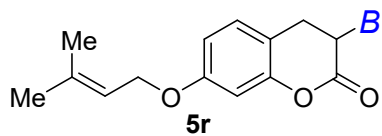


¹H NMR (600 MHz, CDCl₃)



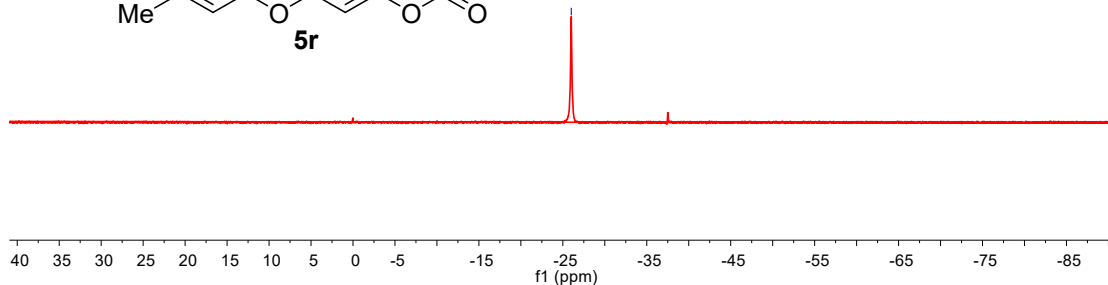
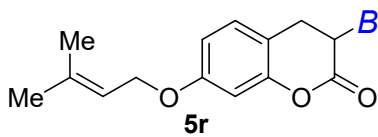
¹³C NMR (151 MHz, CDCl₃)

-25.54
-26.02
-26.49

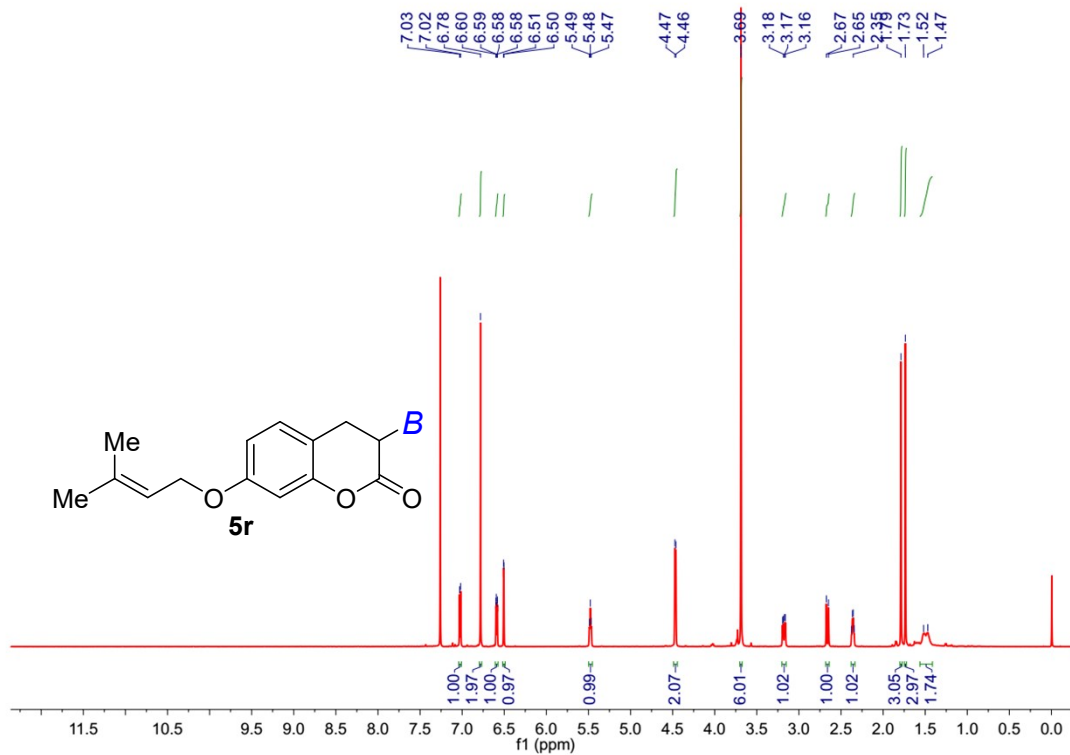


^{11}B NMR (193 MHz, CDCl_3)

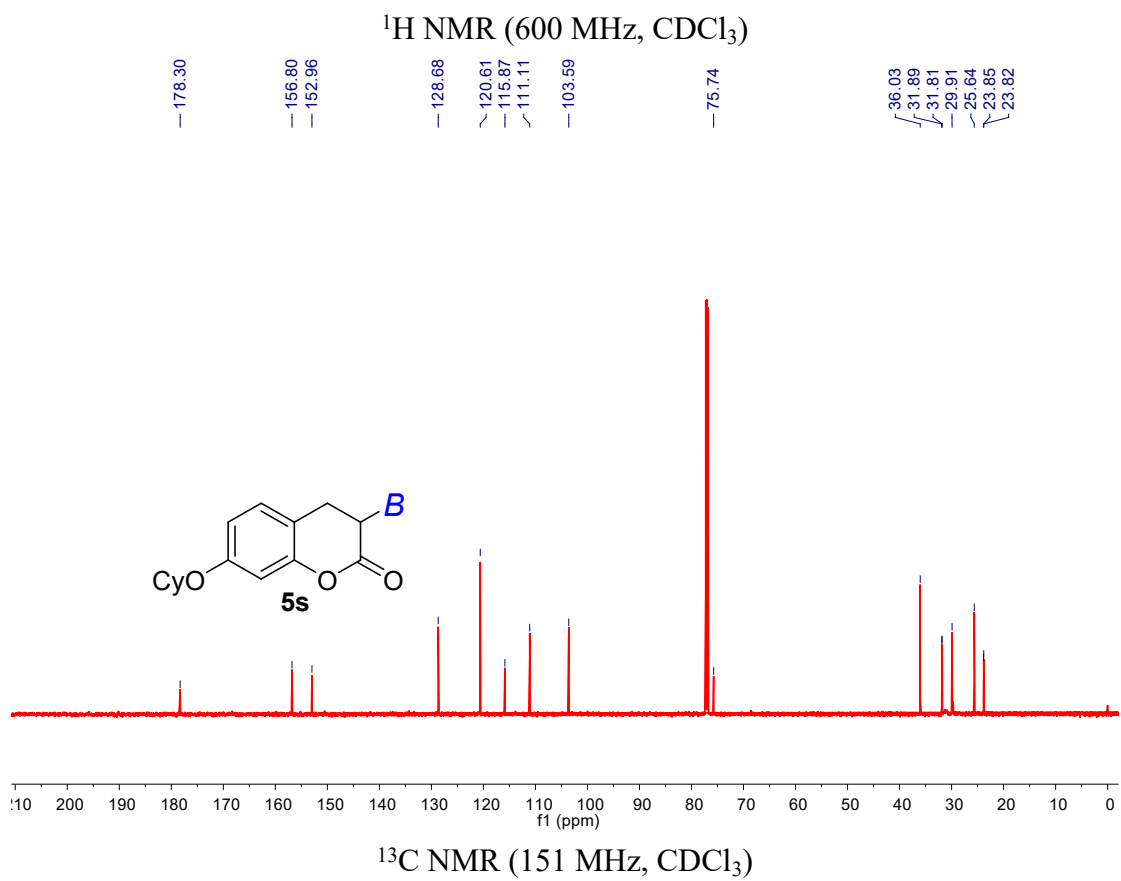
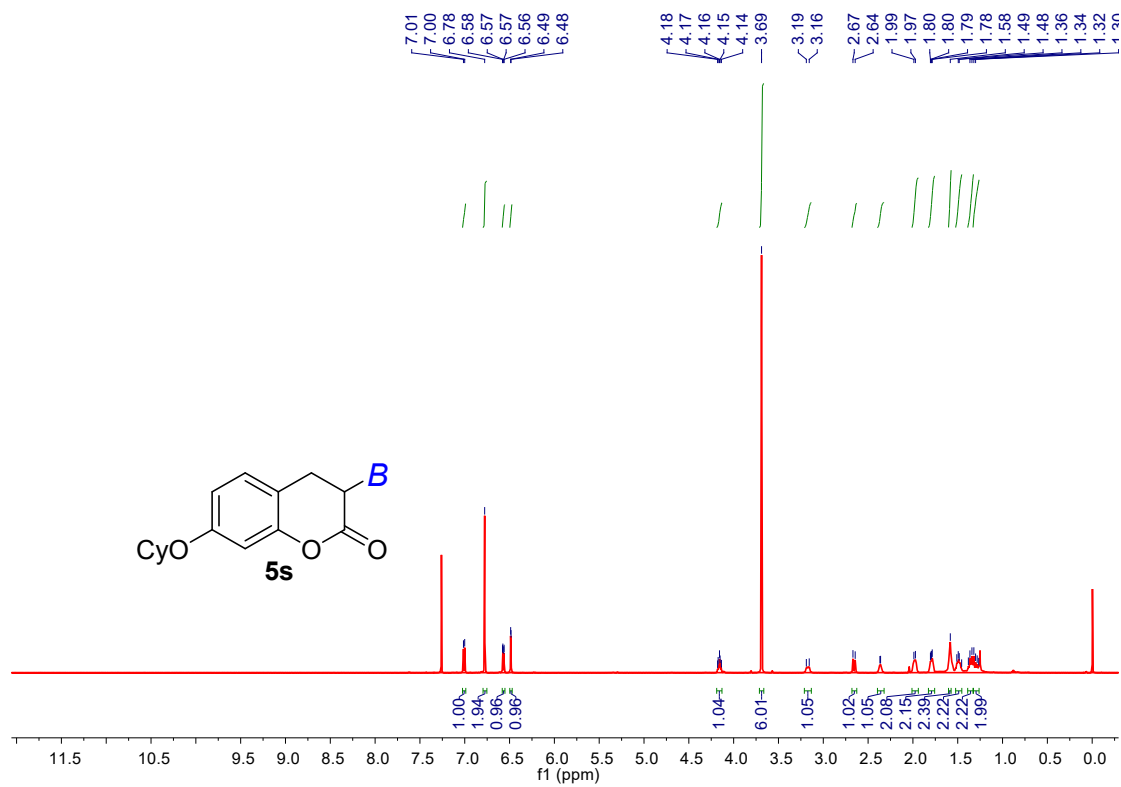
-26.00

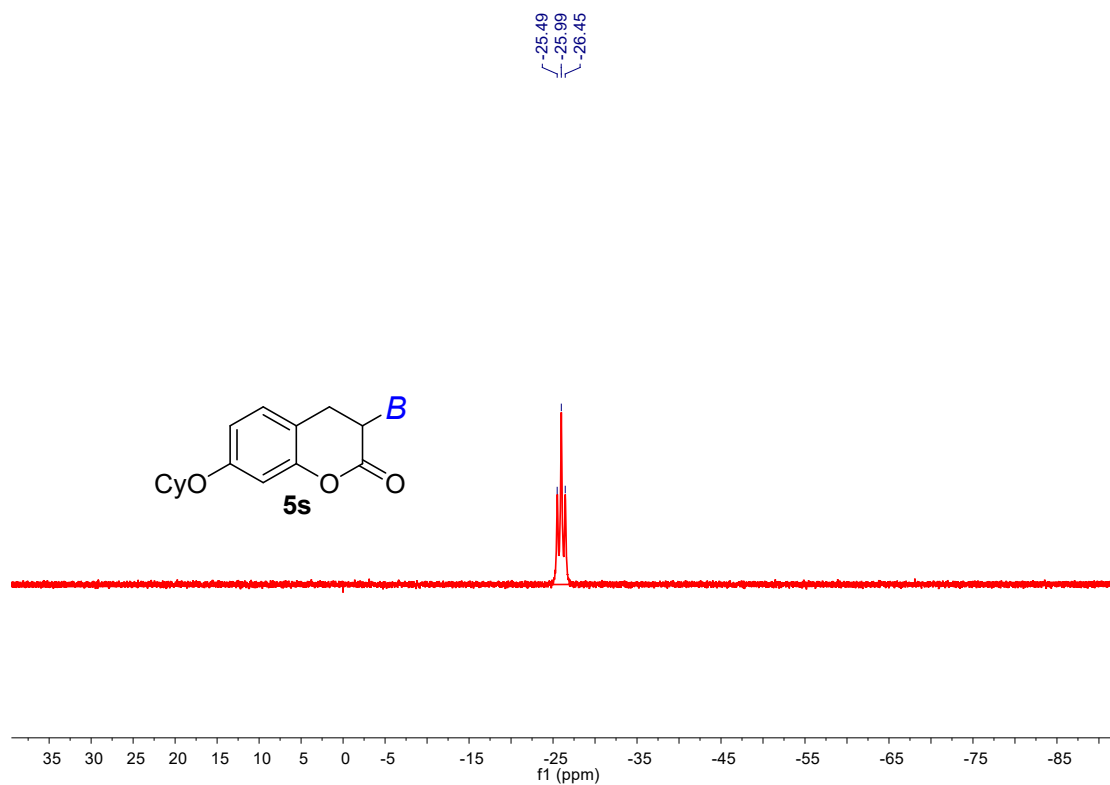


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

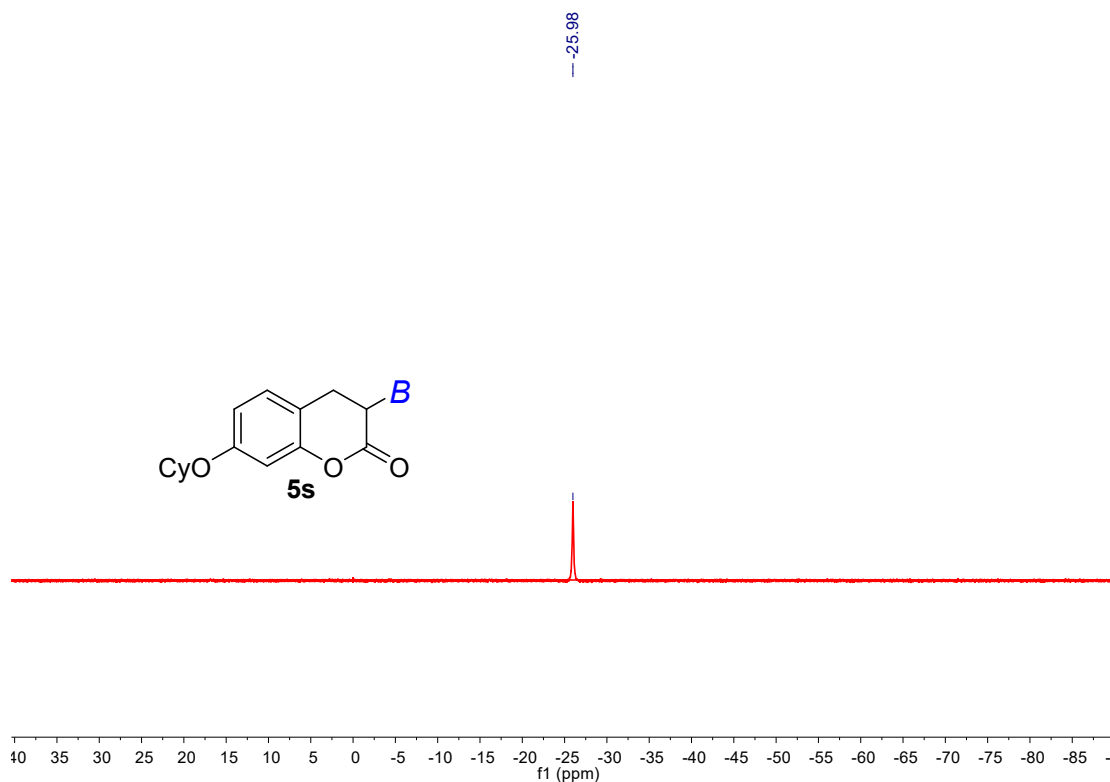


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

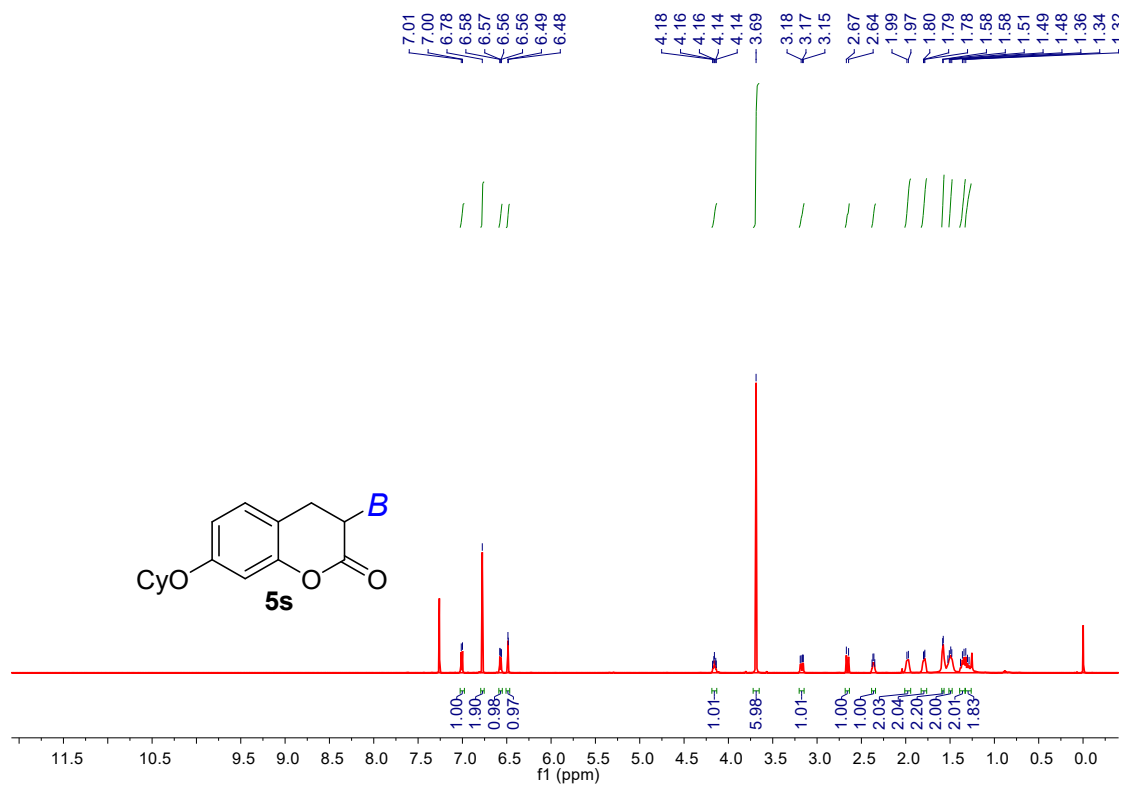




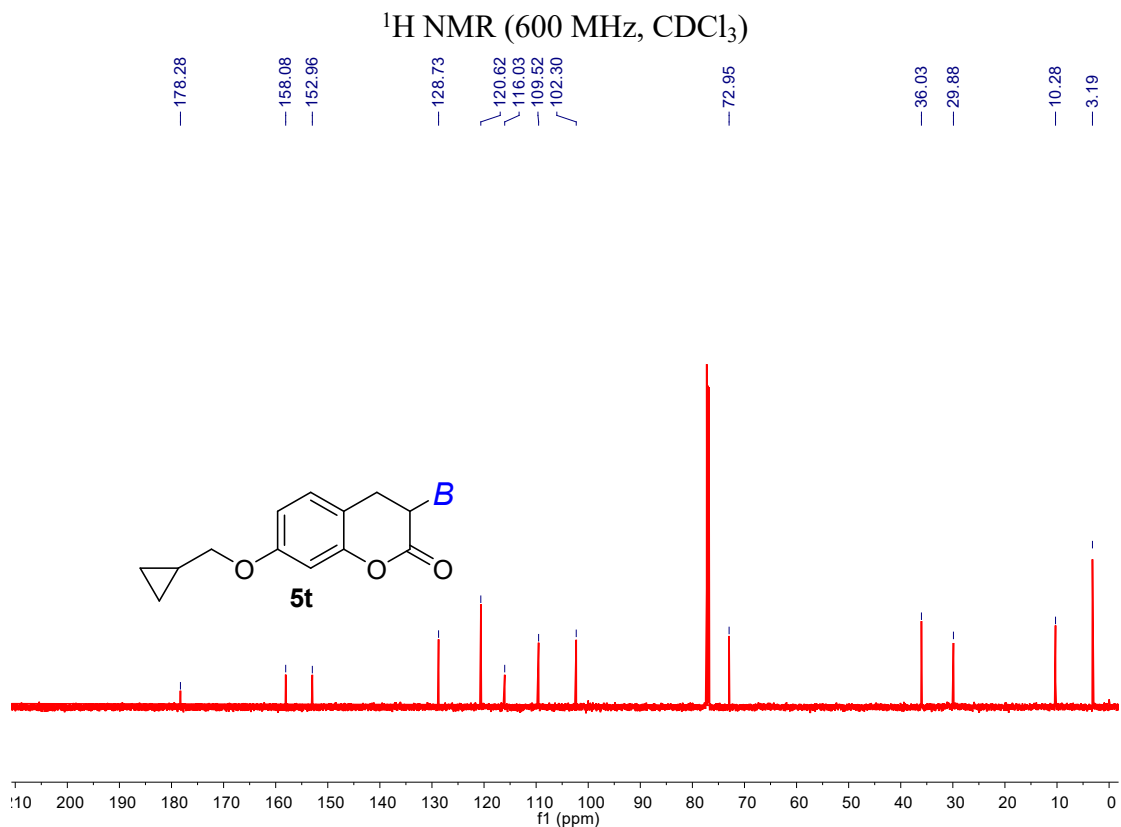
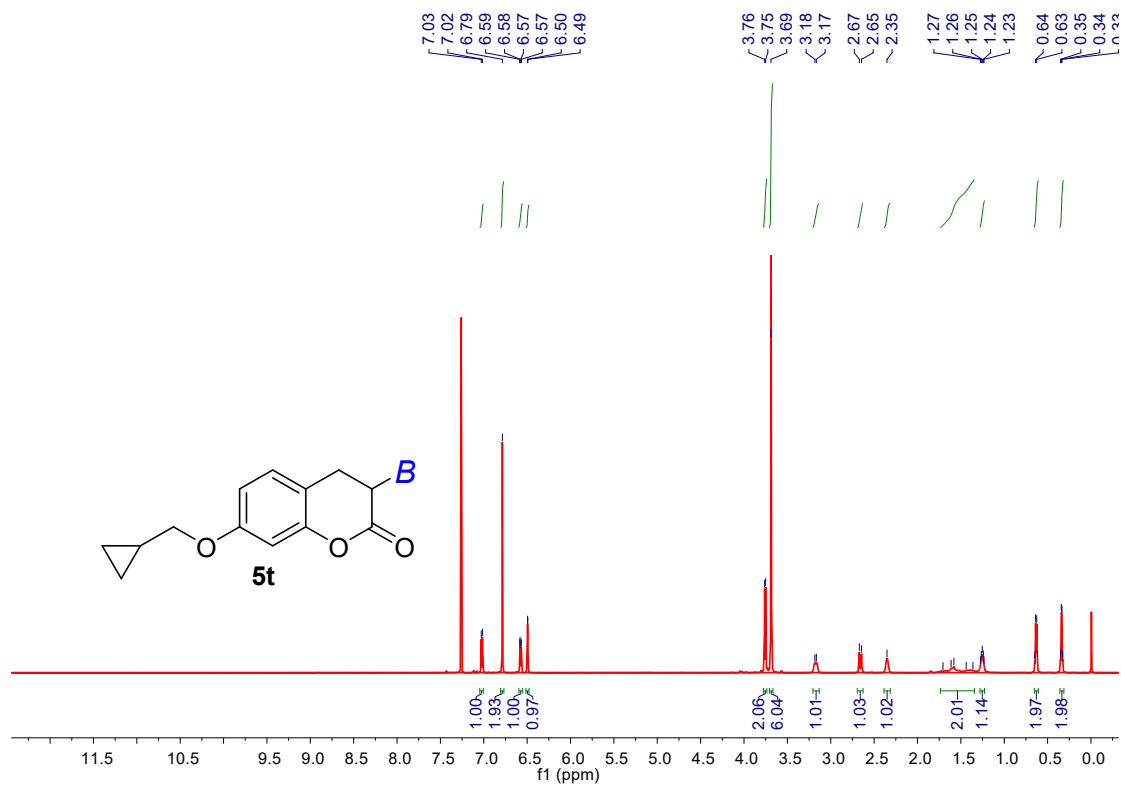
¹¹B NMR (193 MHz, CDCl₃)

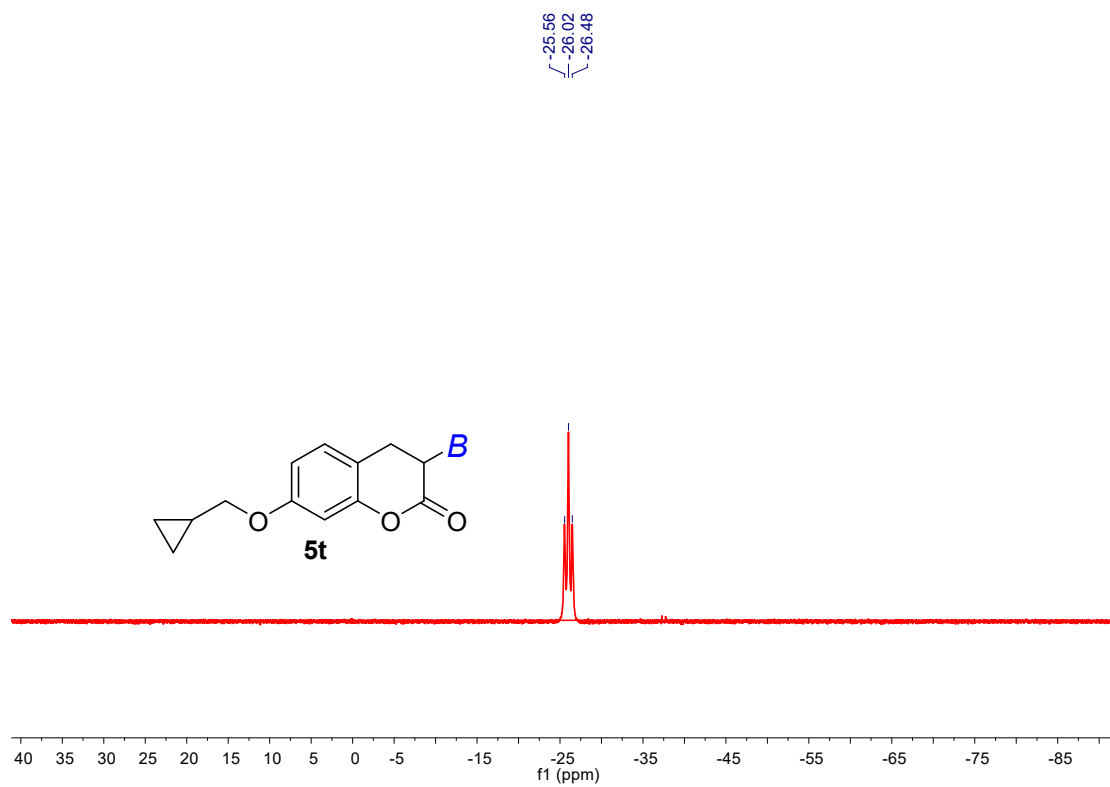


¹¹B{¹H} NMR (193 MHz, CDCl₃)

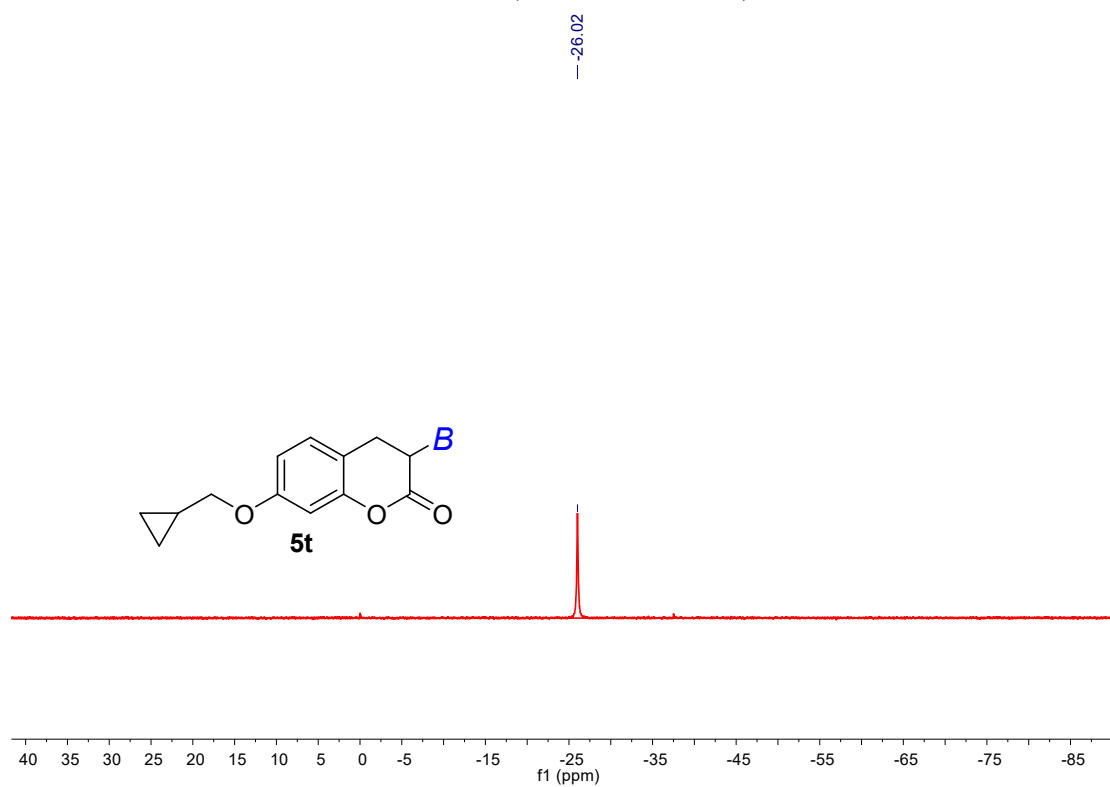


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

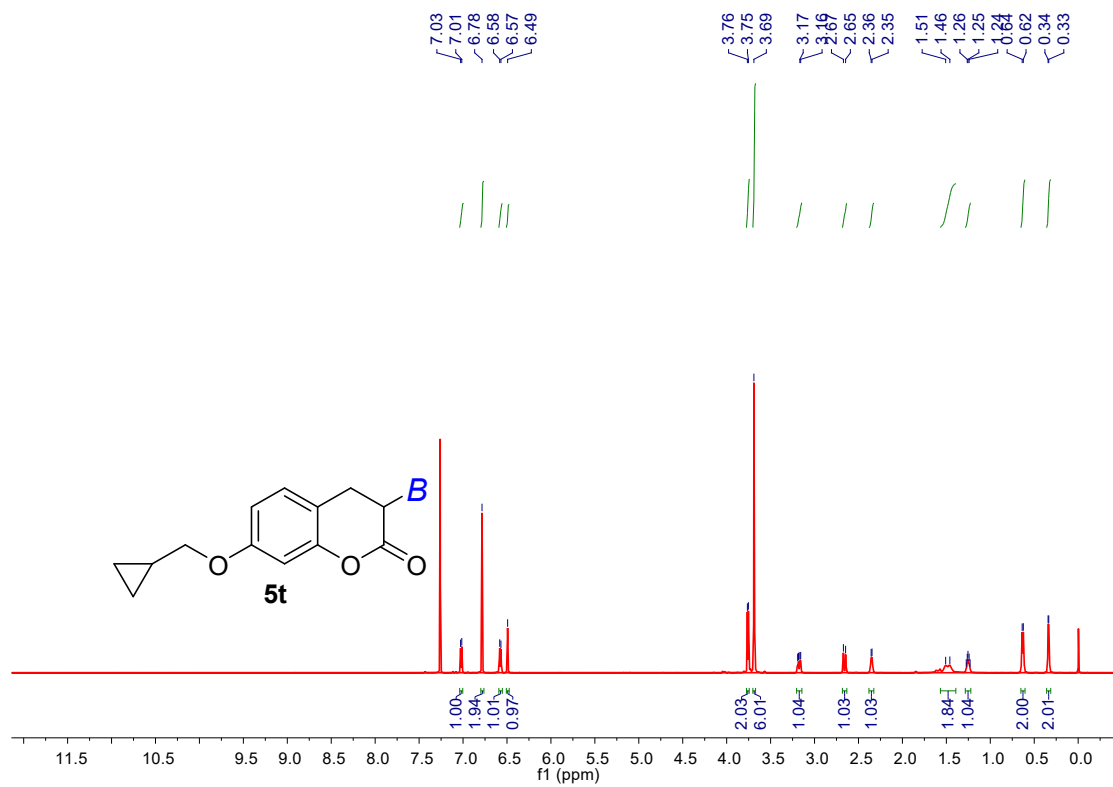




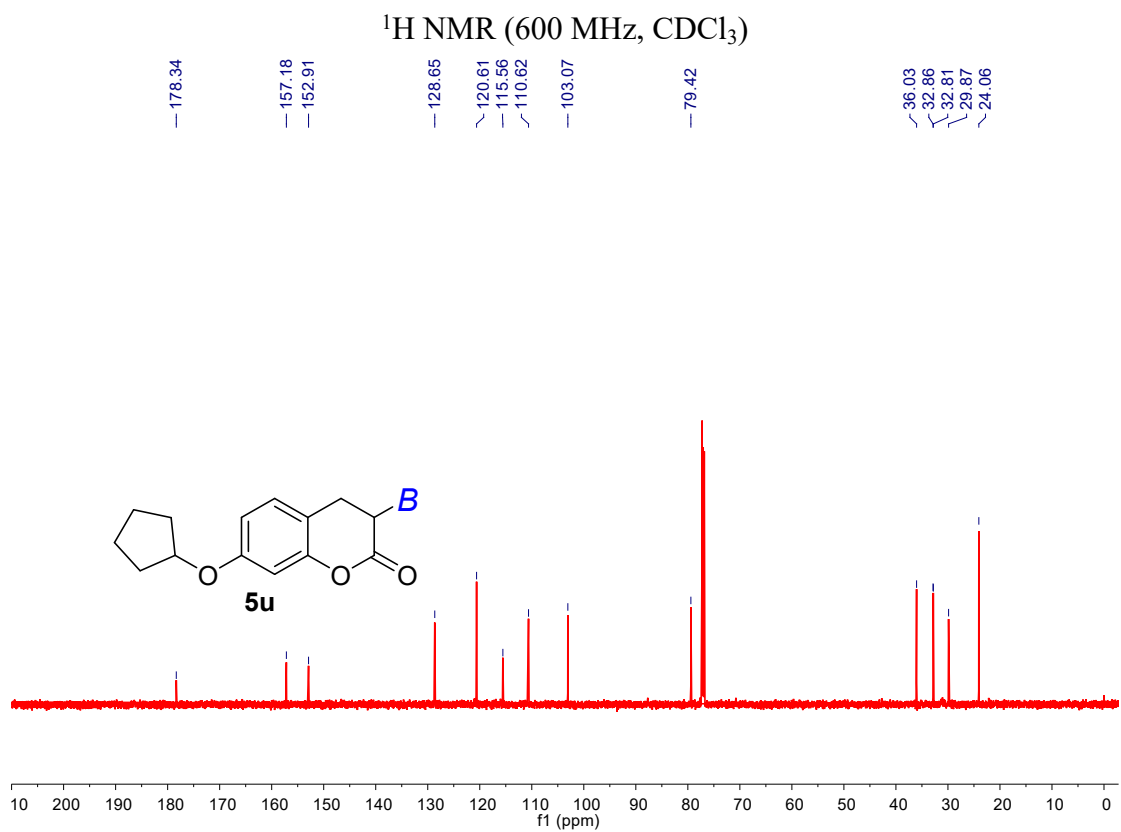
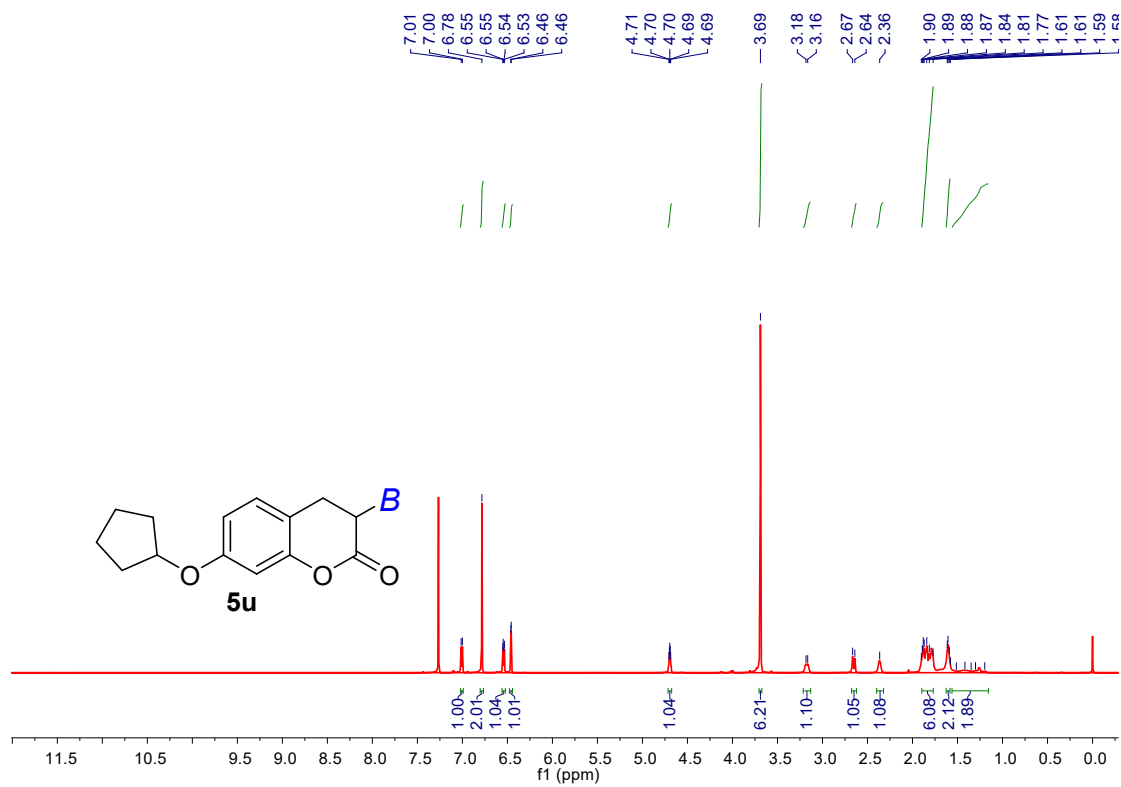
^{11}B NMR (193 MHz, CDCl_3)

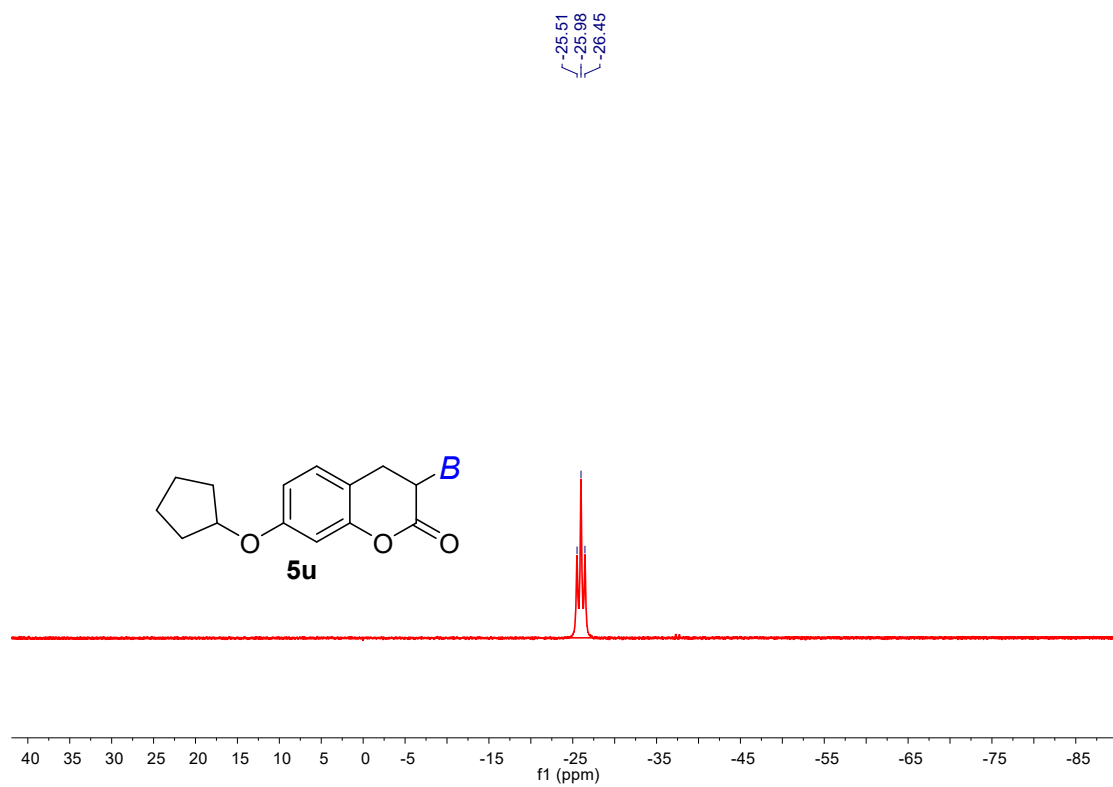


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

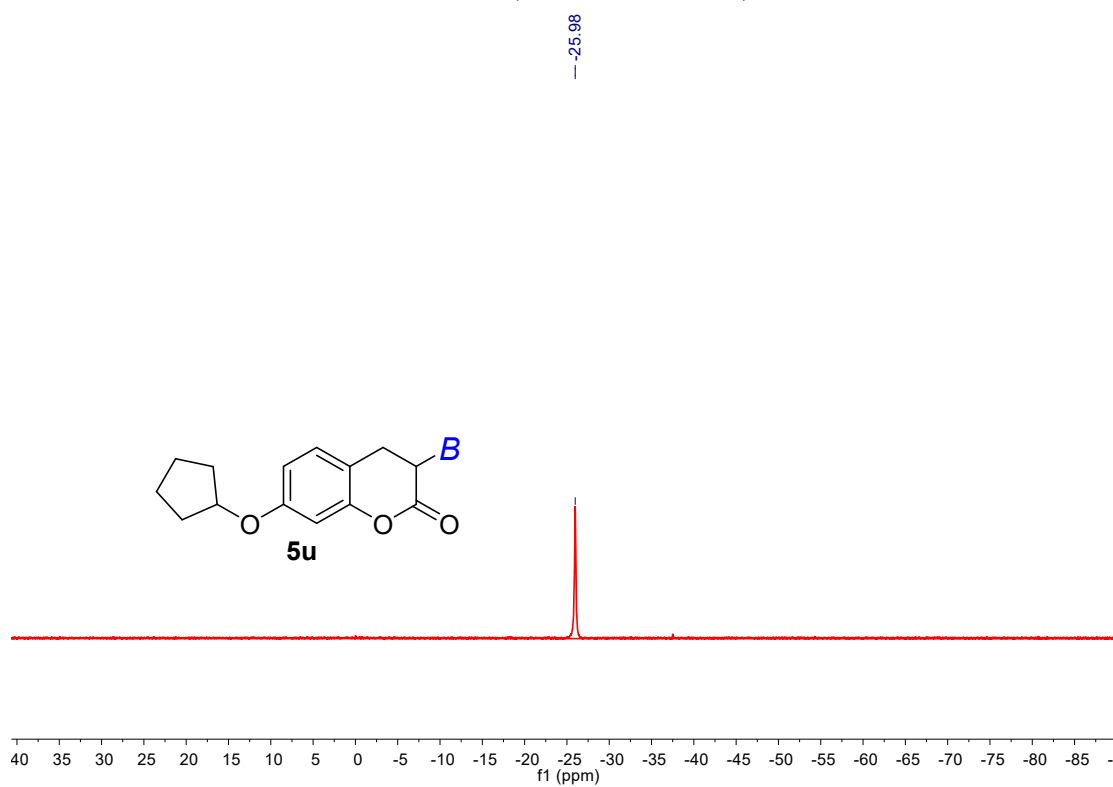


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

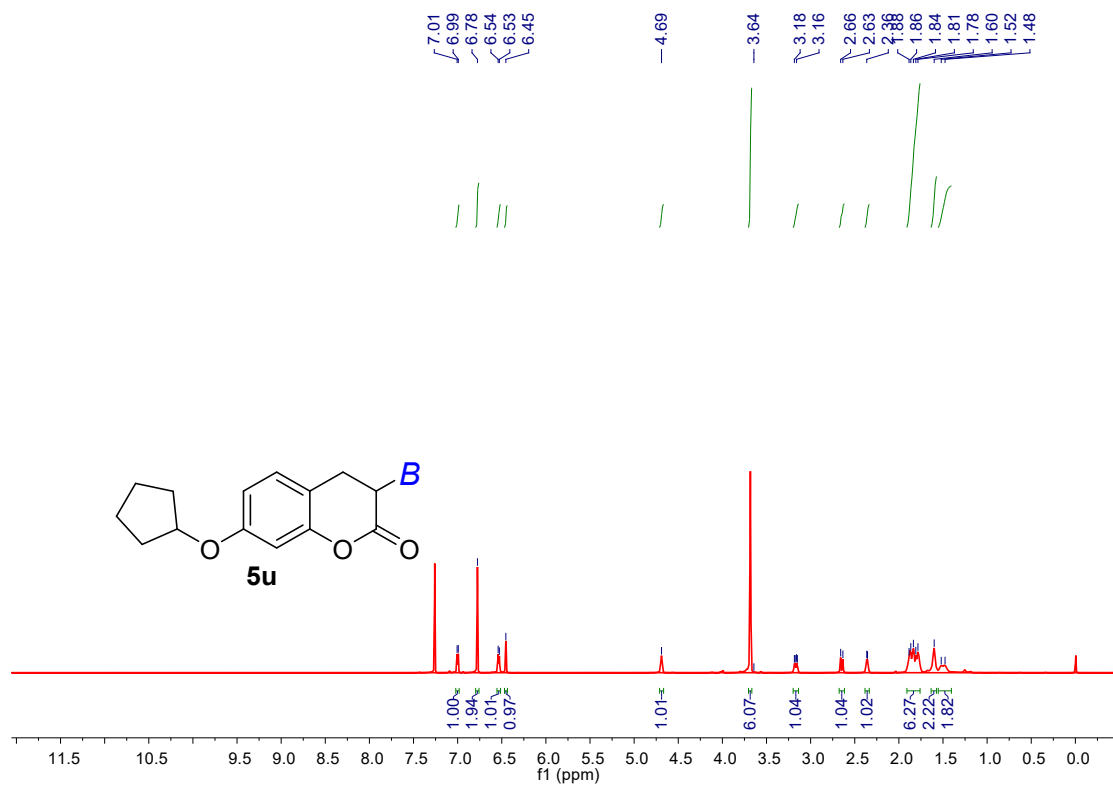




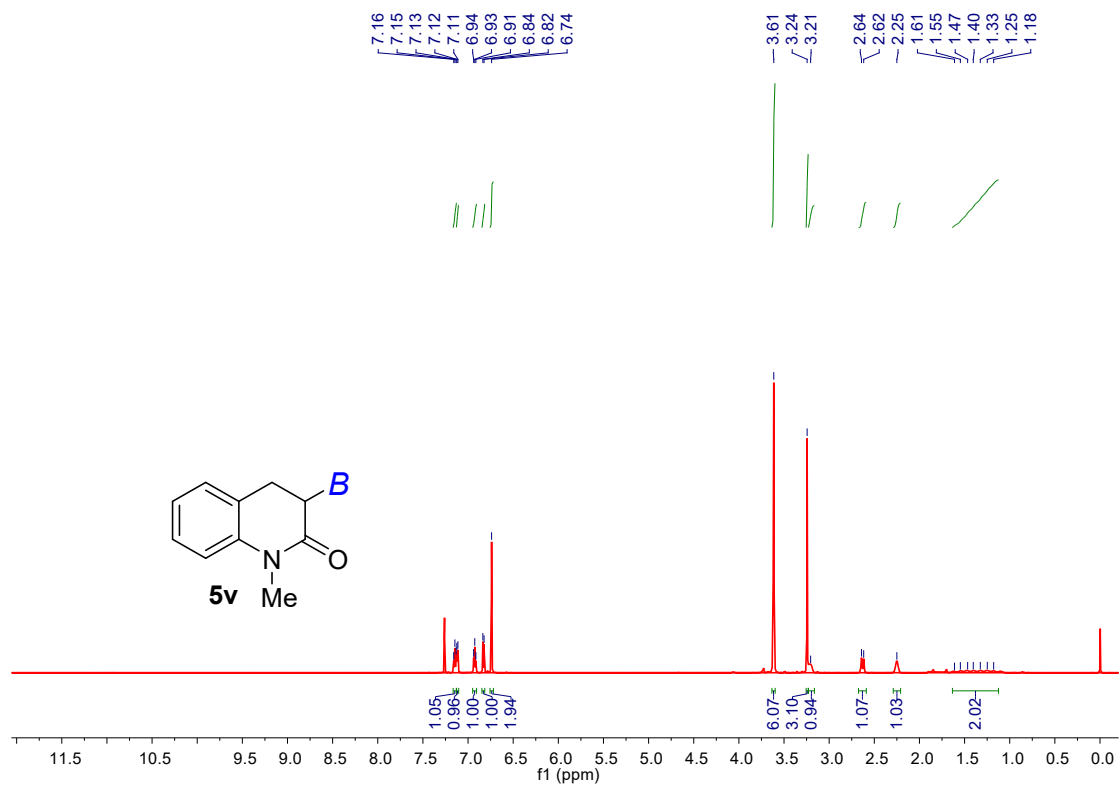
¹¹B NMR (193 MHz, CDCl₃)



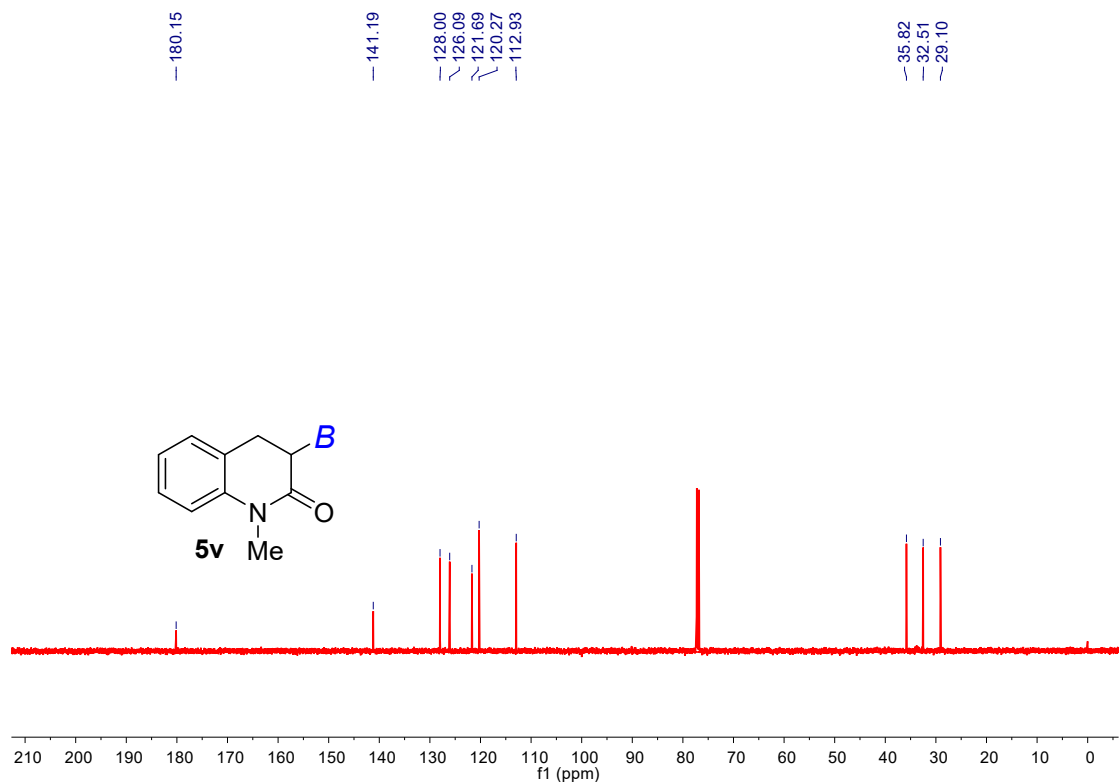
¹¹B{¹H} NMR (193 MHz, CDCl₃)



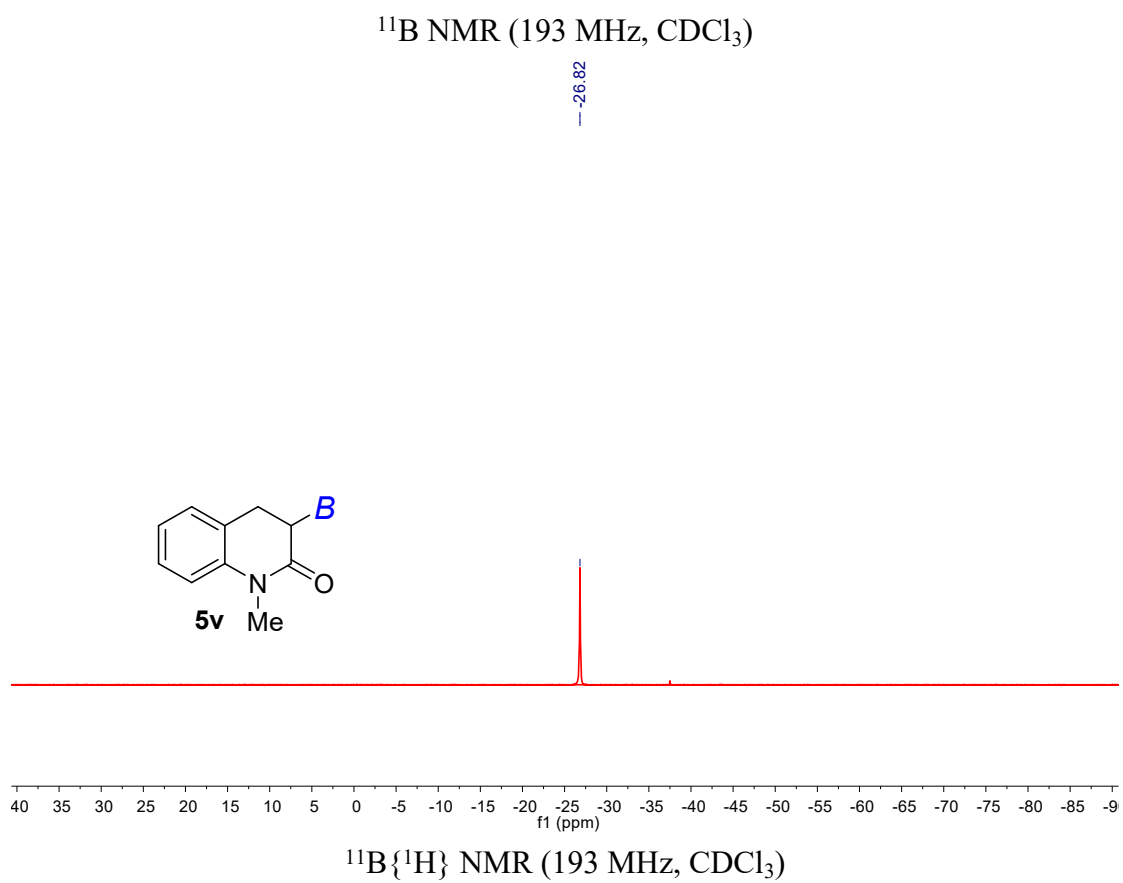
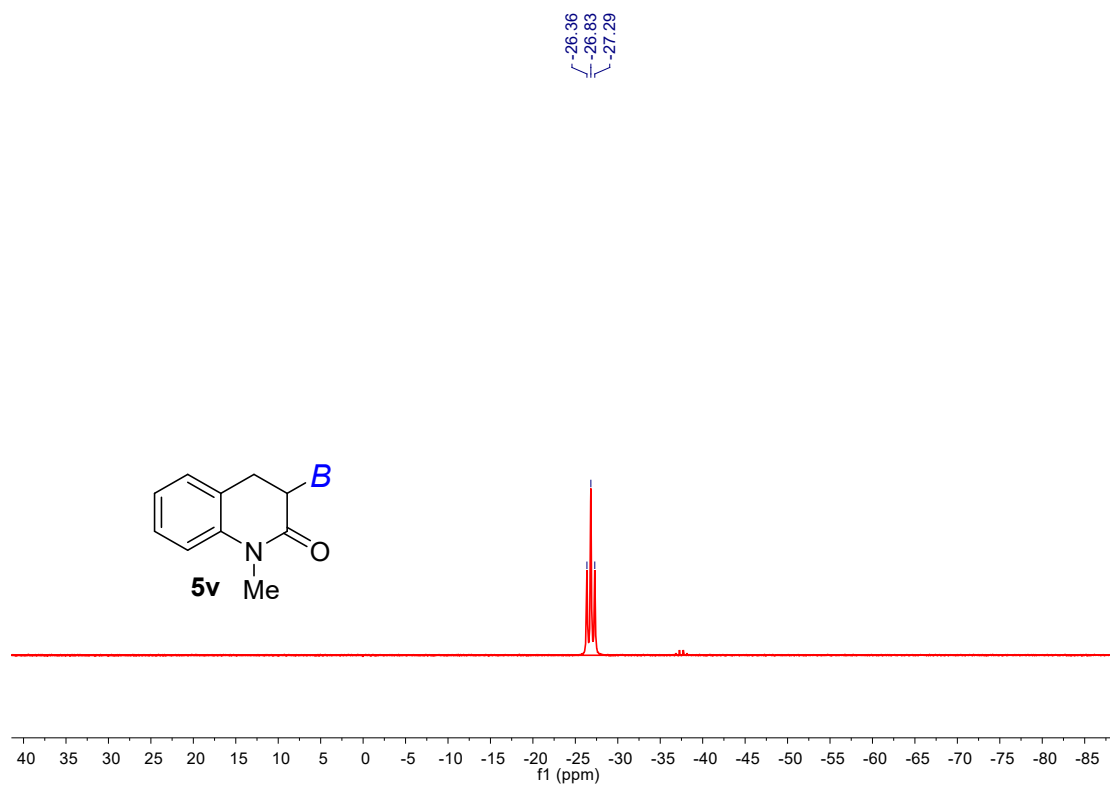
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

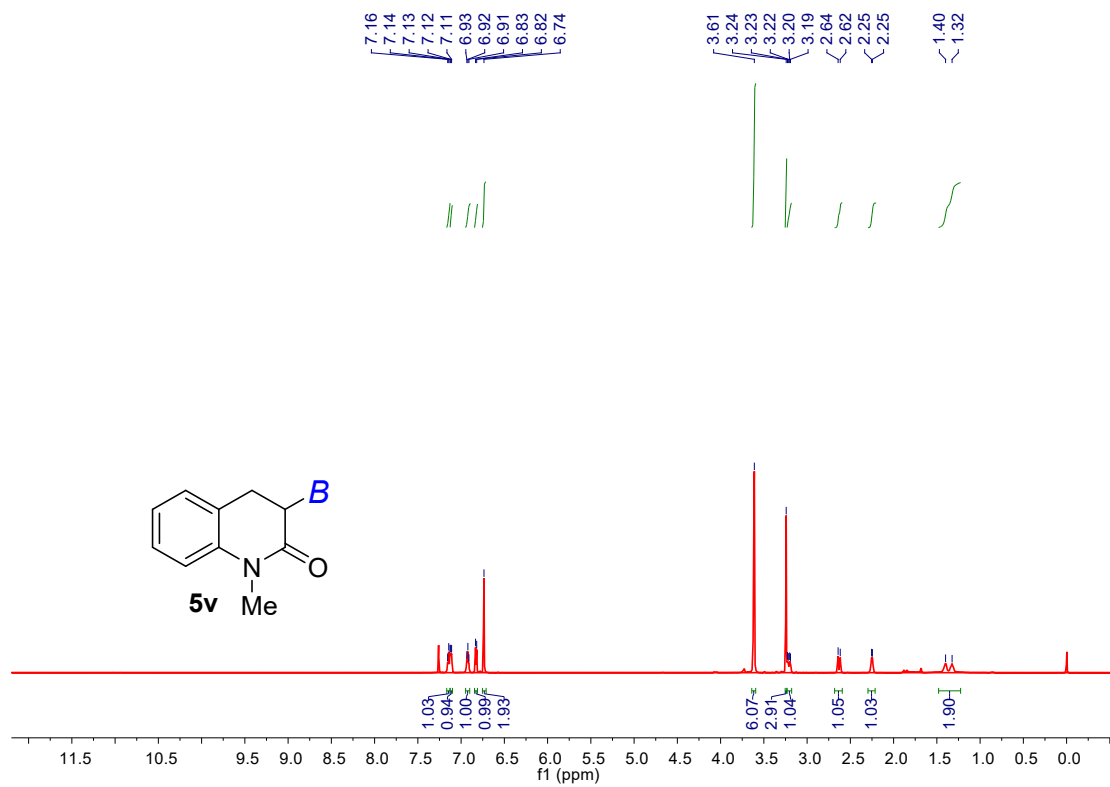


$^1\text{H NMR}$ (600 MHz, CDCl_3)

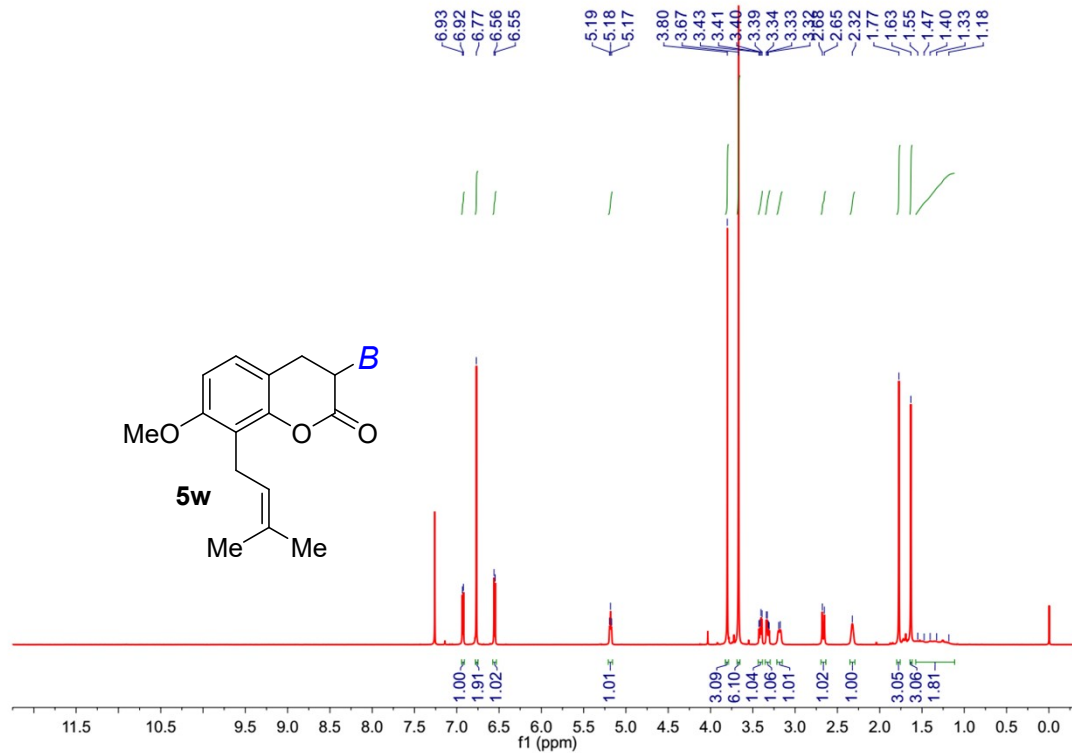


$^{13}\text{C NMR}$ (151 MHz, CDCl_3)

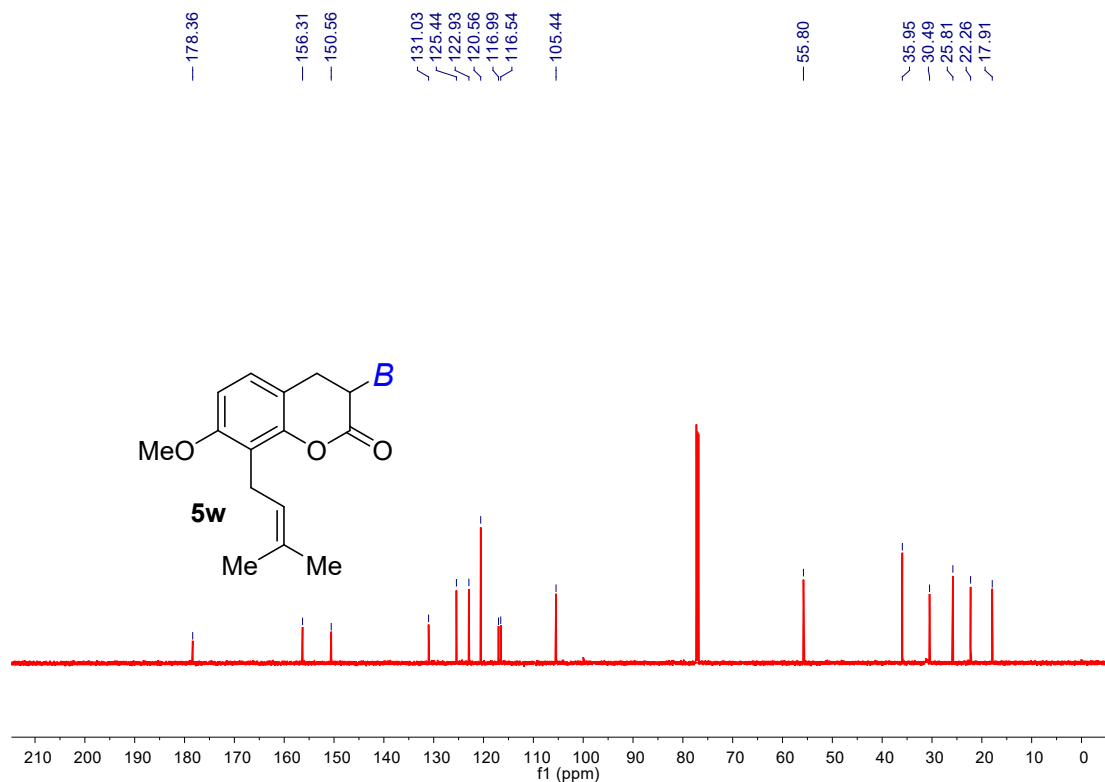




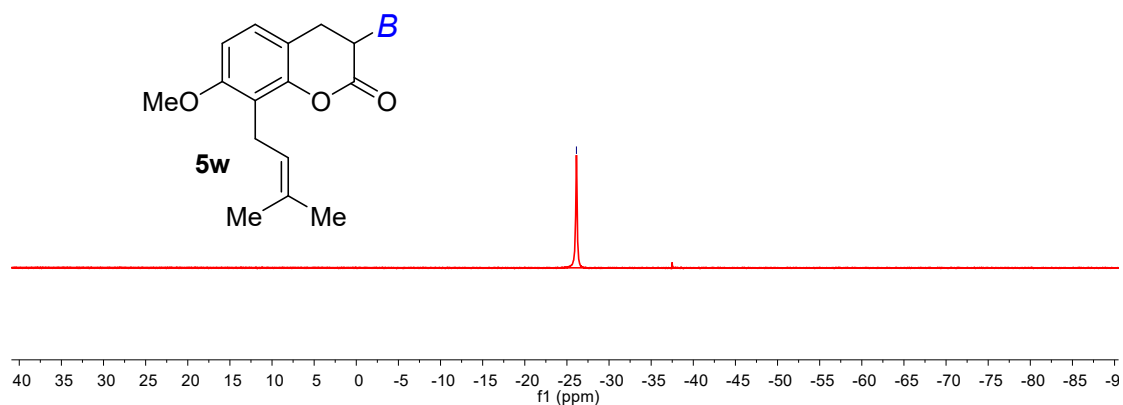
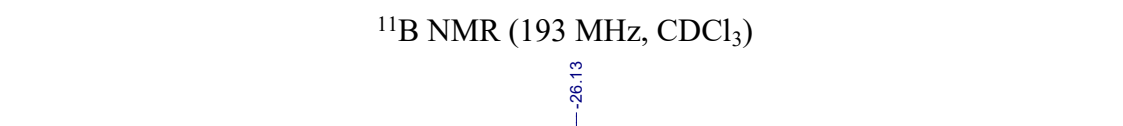
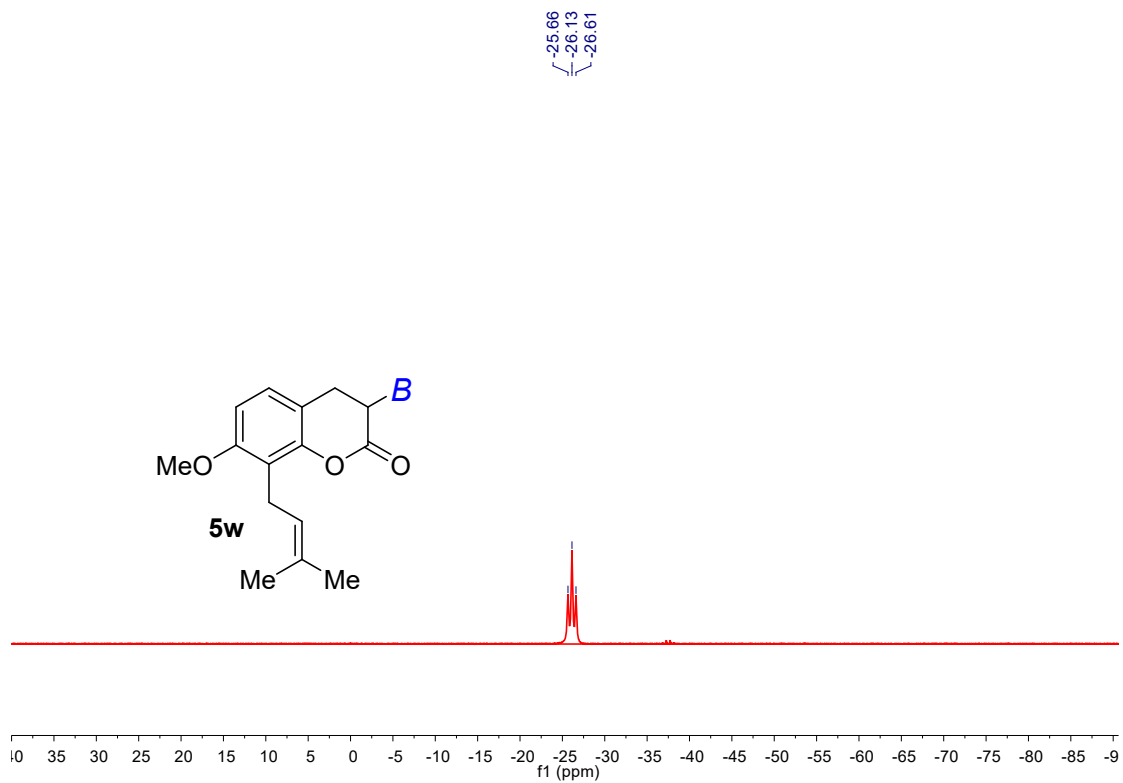
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

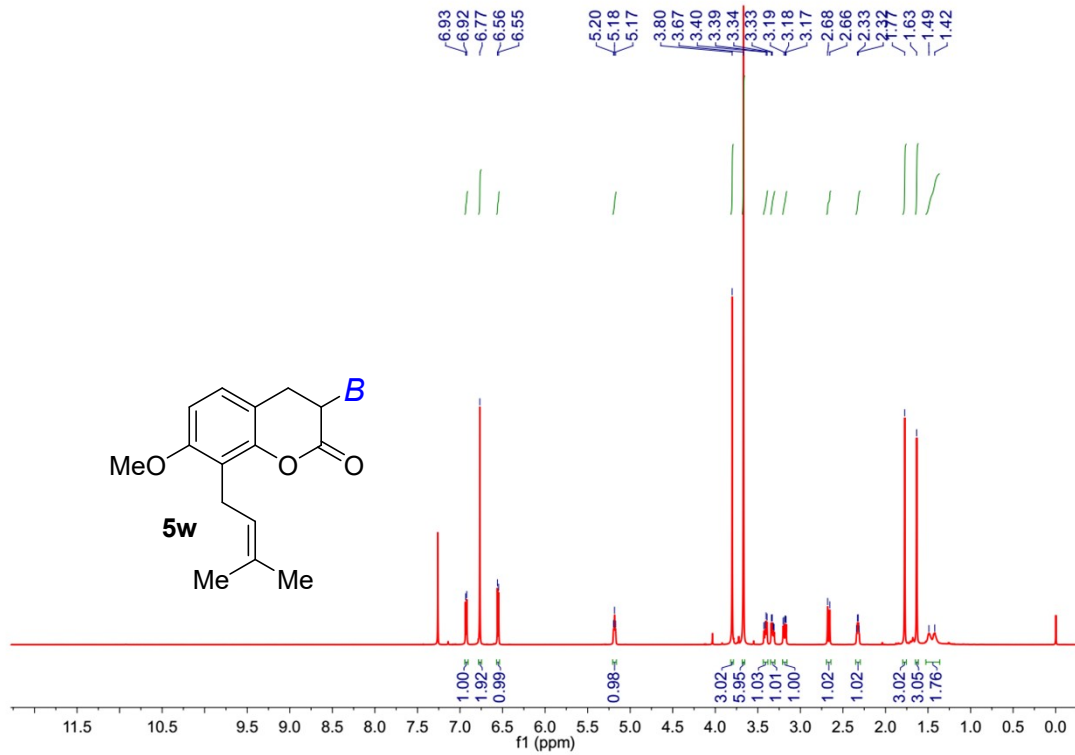


¹H NMR (600 MHz, CDCl₃)

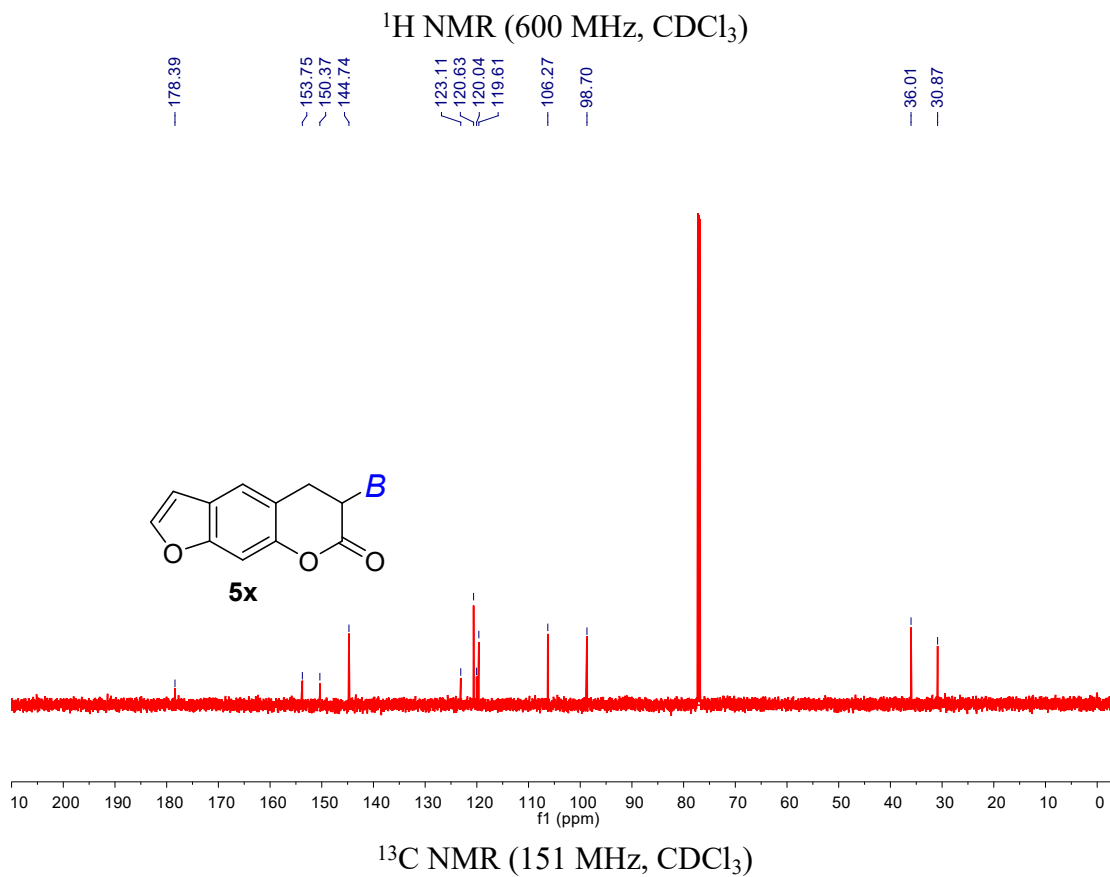
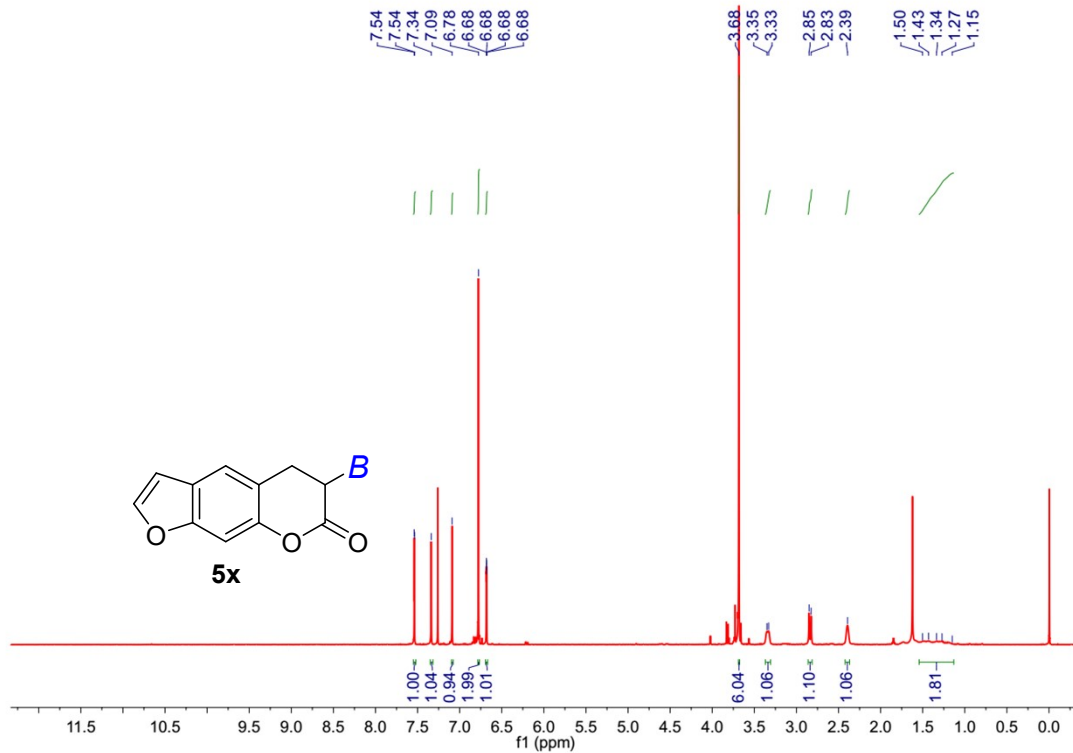


¹³C NMR (151 MHz, CDCl₃)

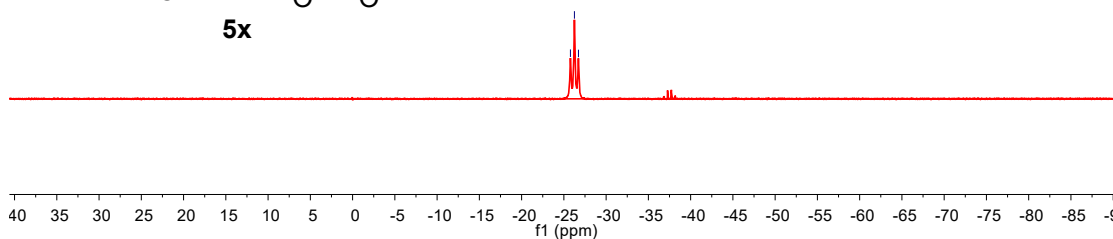
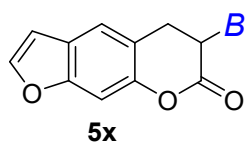




$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

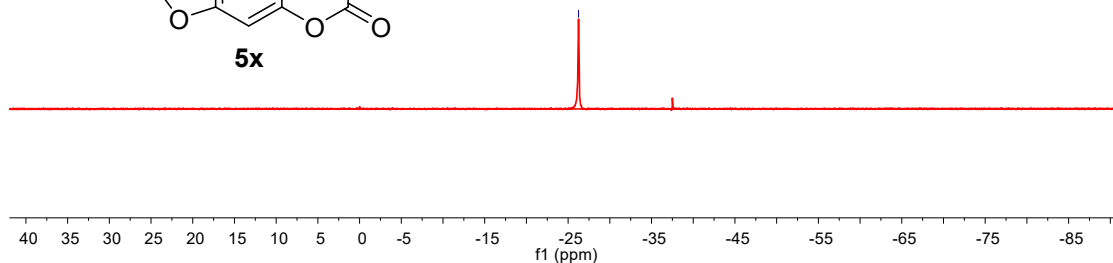
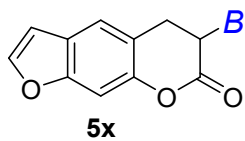


-25.78
-26.25
-26.73

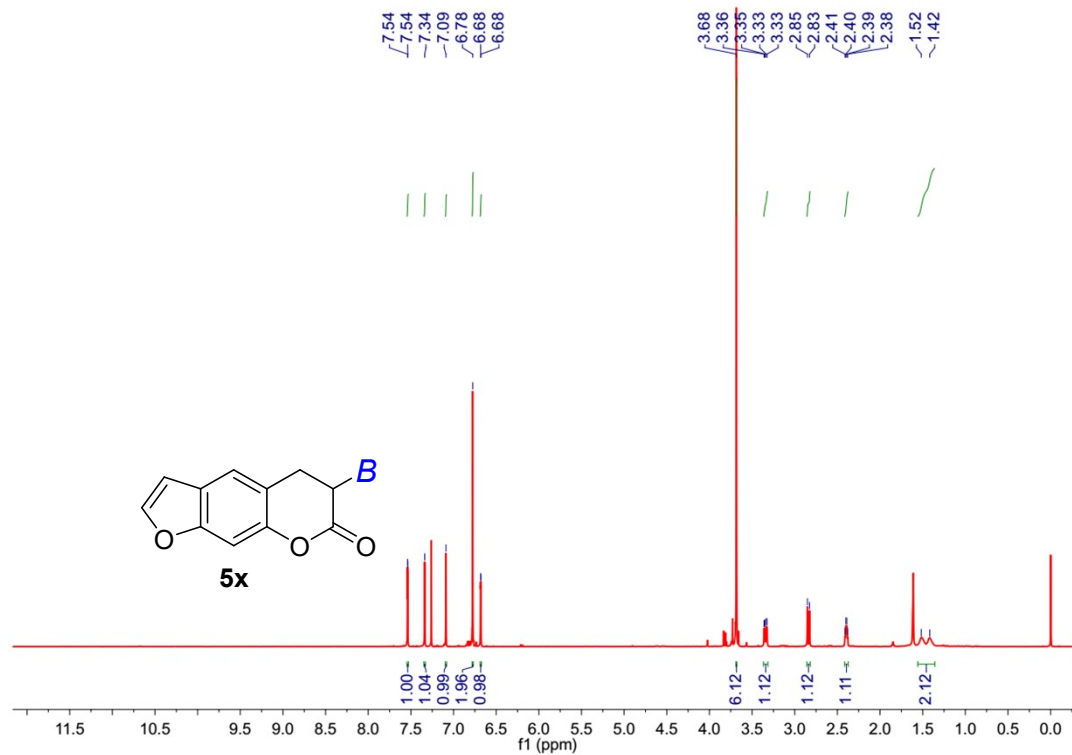


^{11}B NMR (193 MHz, CDCl_3)

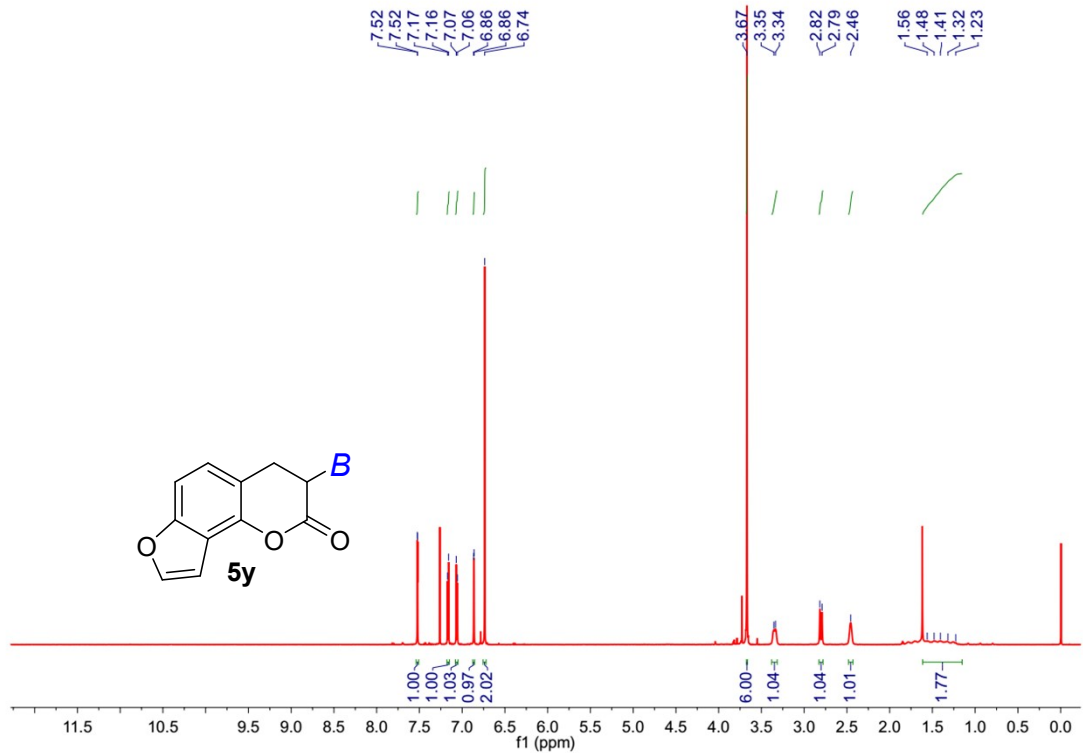
-26.25



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

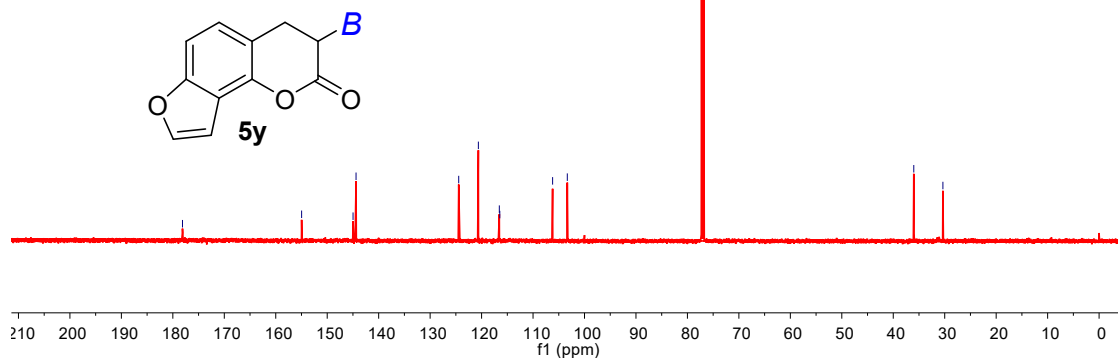


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

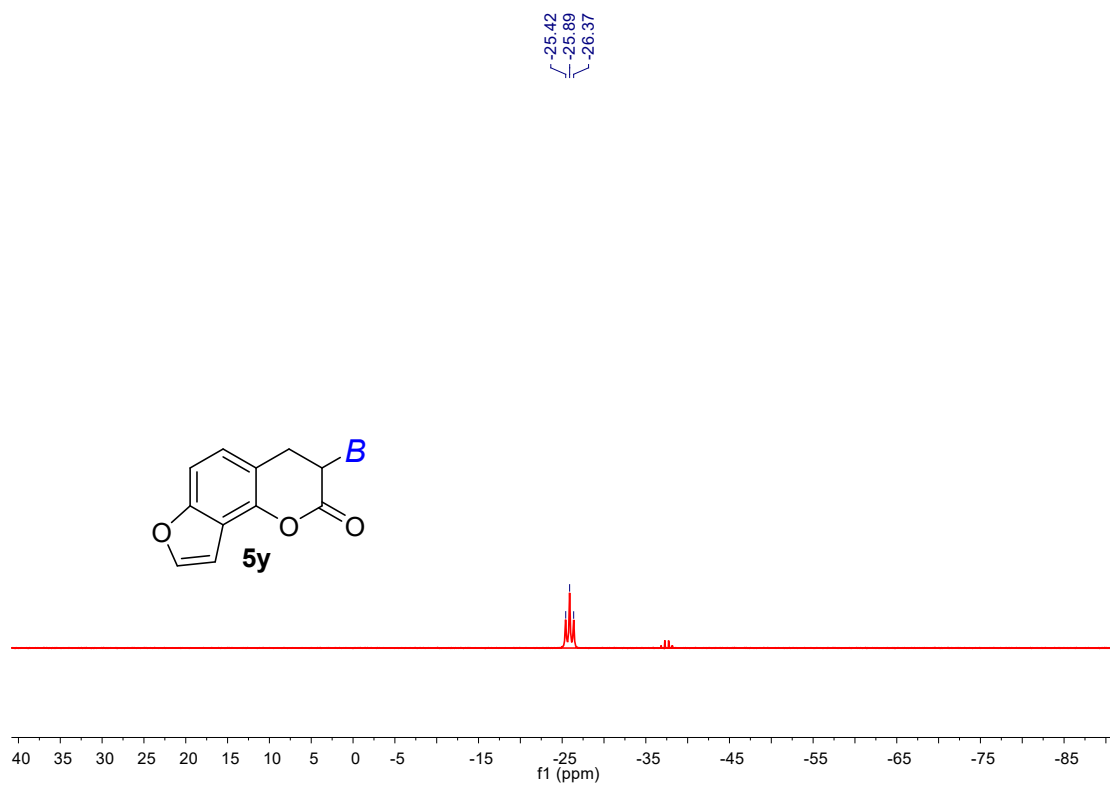


¹H NMR (600 MHz, CDCl₃)

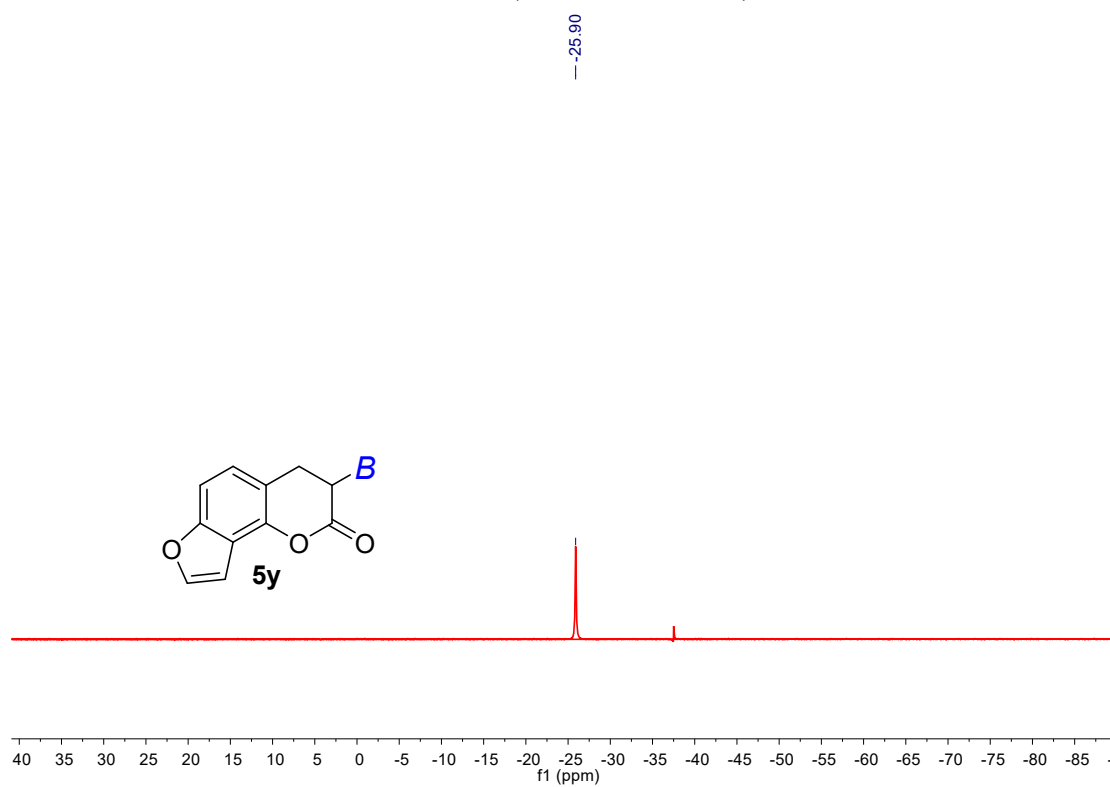
— 178.12 — 154.97 — 144.98 — 144.40 — 124.45 — 120.62 — 116.55 — 116.46 — 106.20 — 103.34 — 36.01 — 30.35



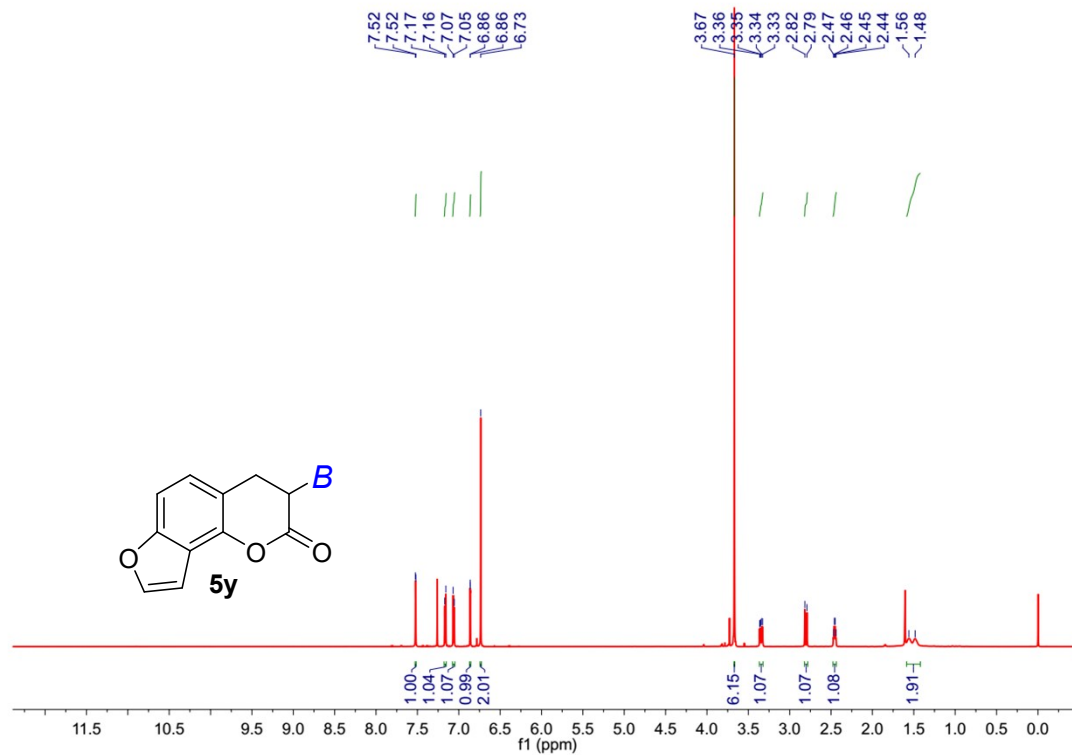
¹³C NMR (151 MHz, CDCl₃)



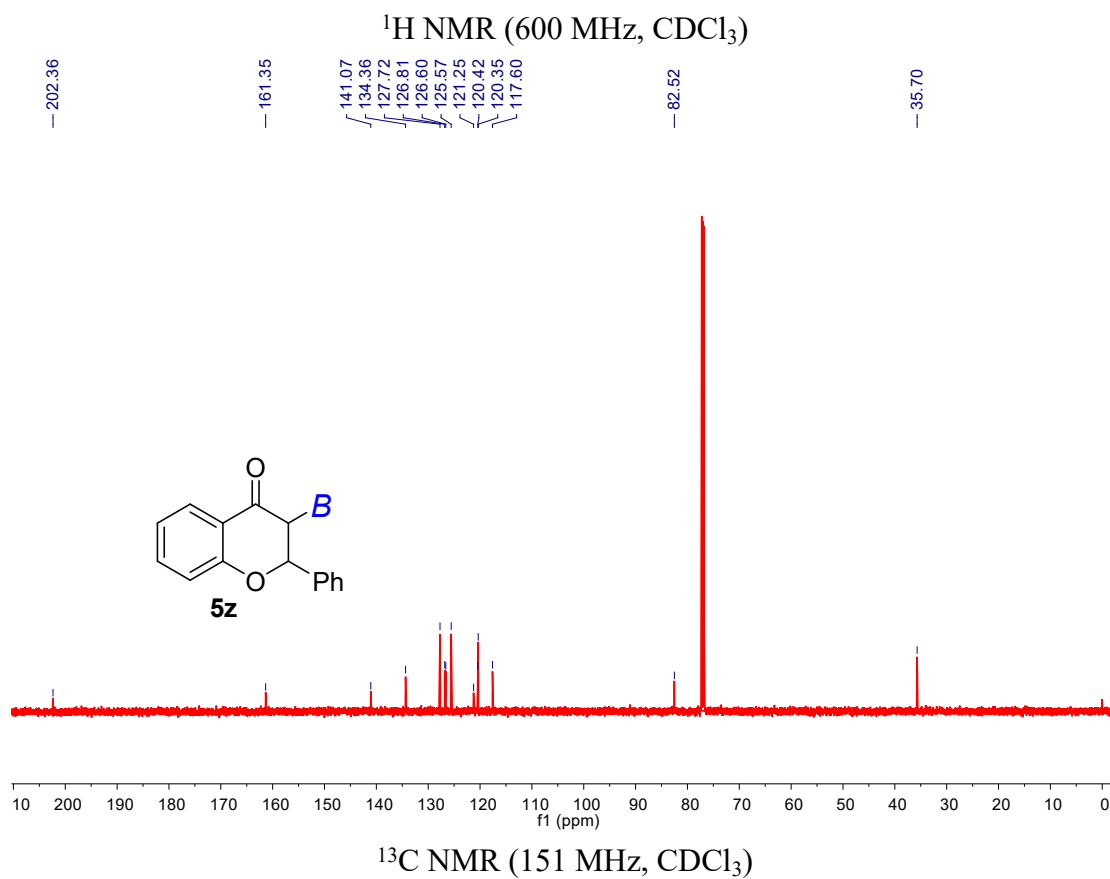
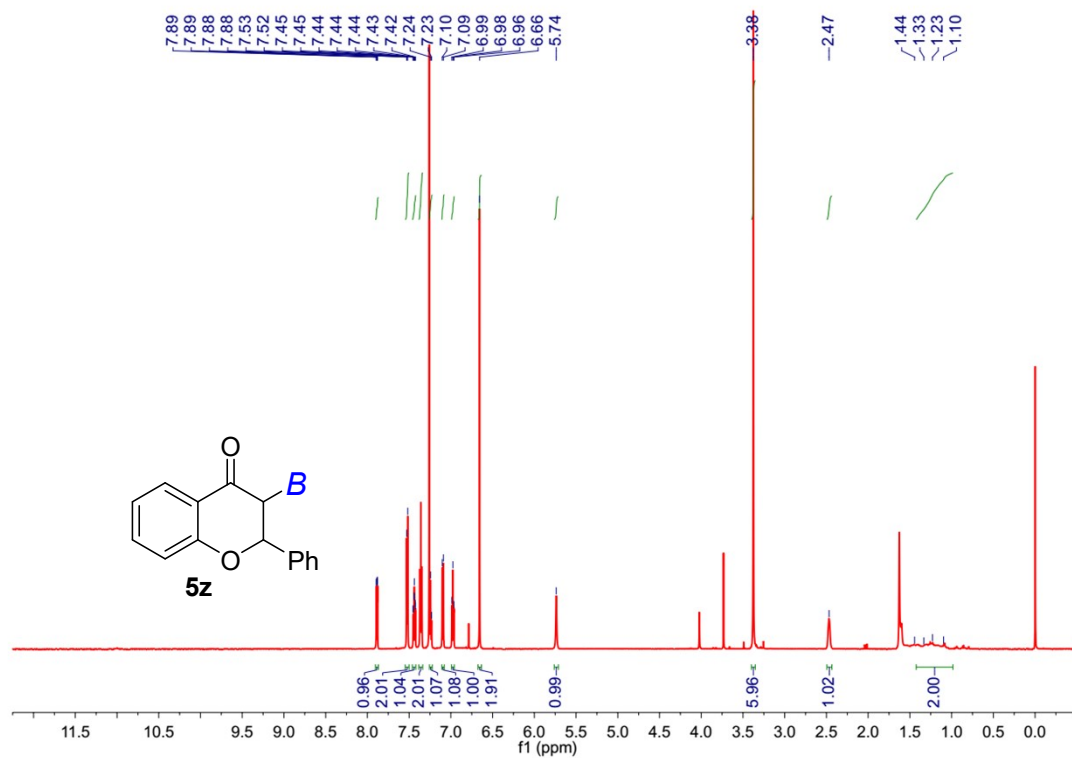
^{11}B NMR (193 MHz, CDCl_3)

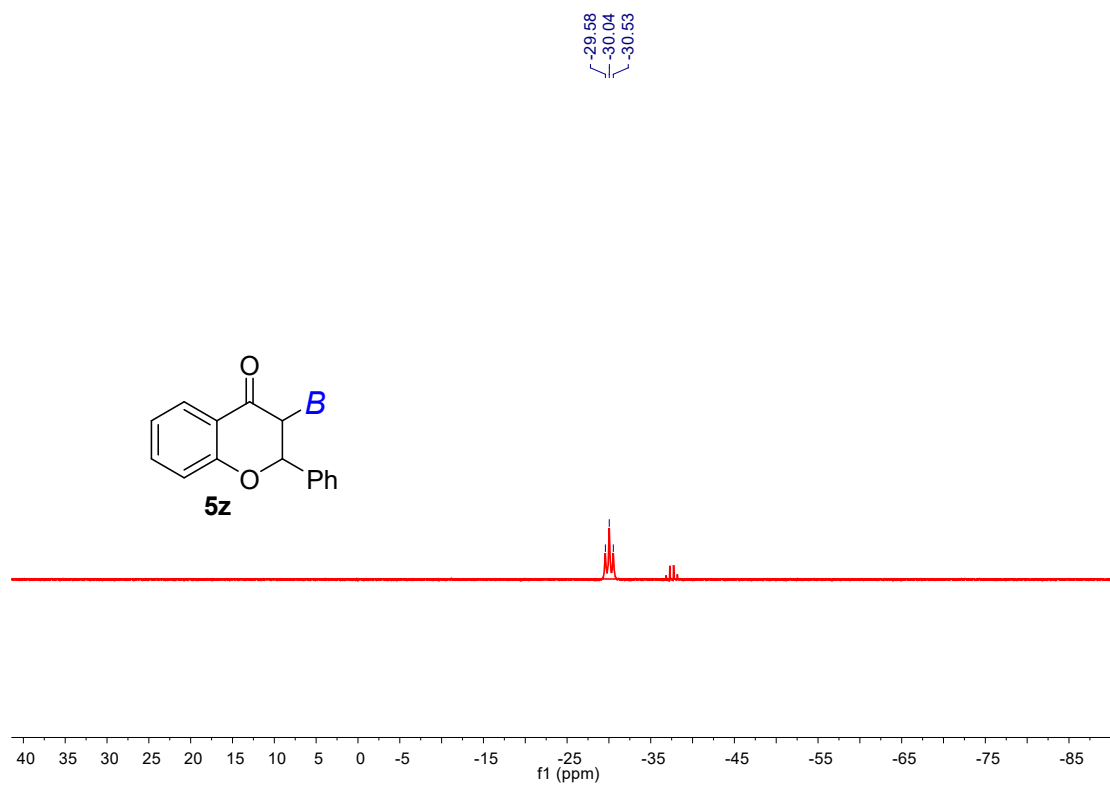


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

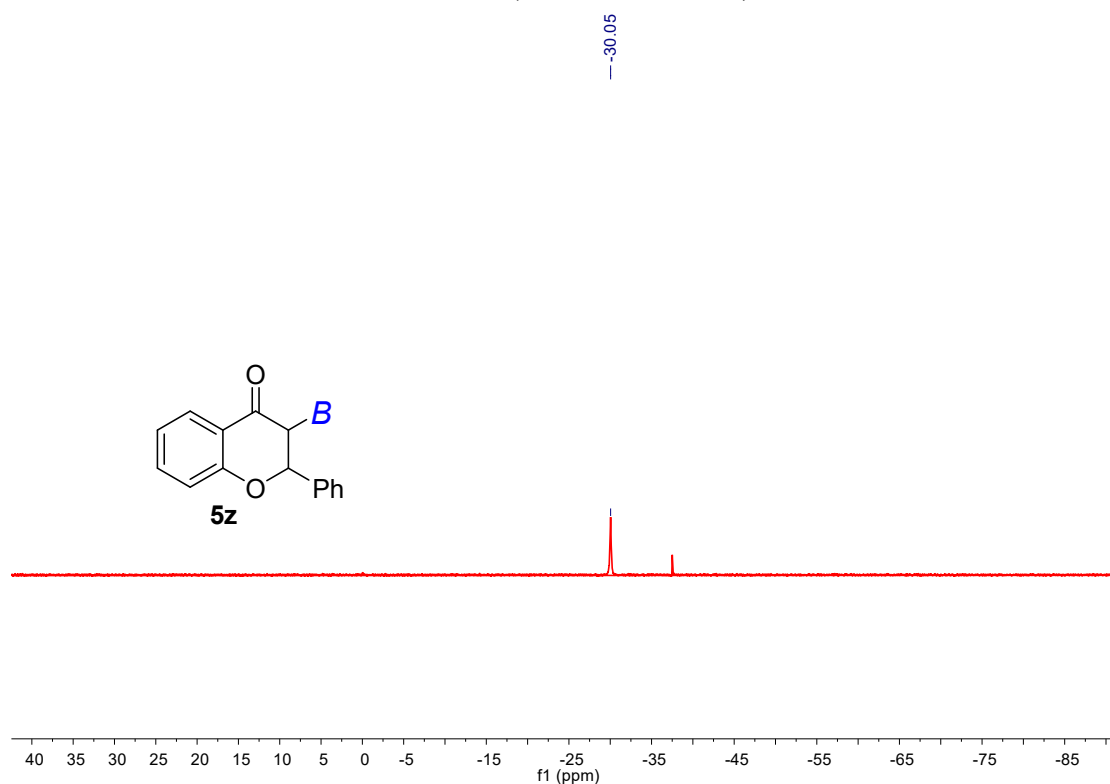


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

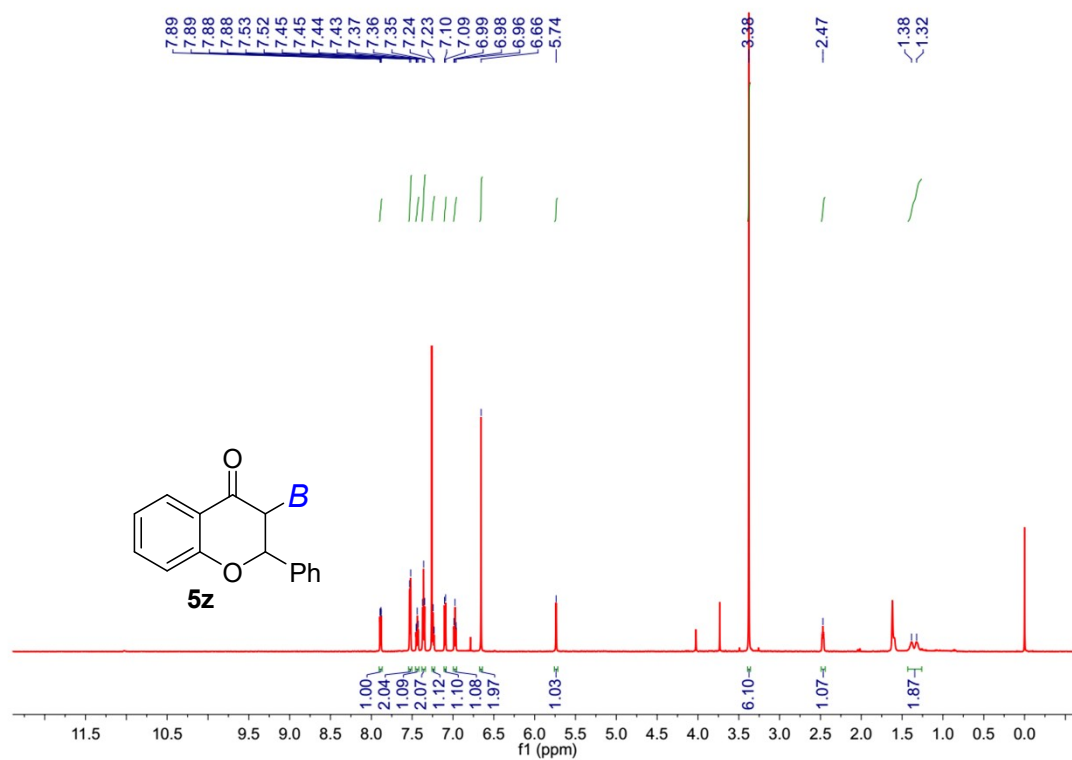




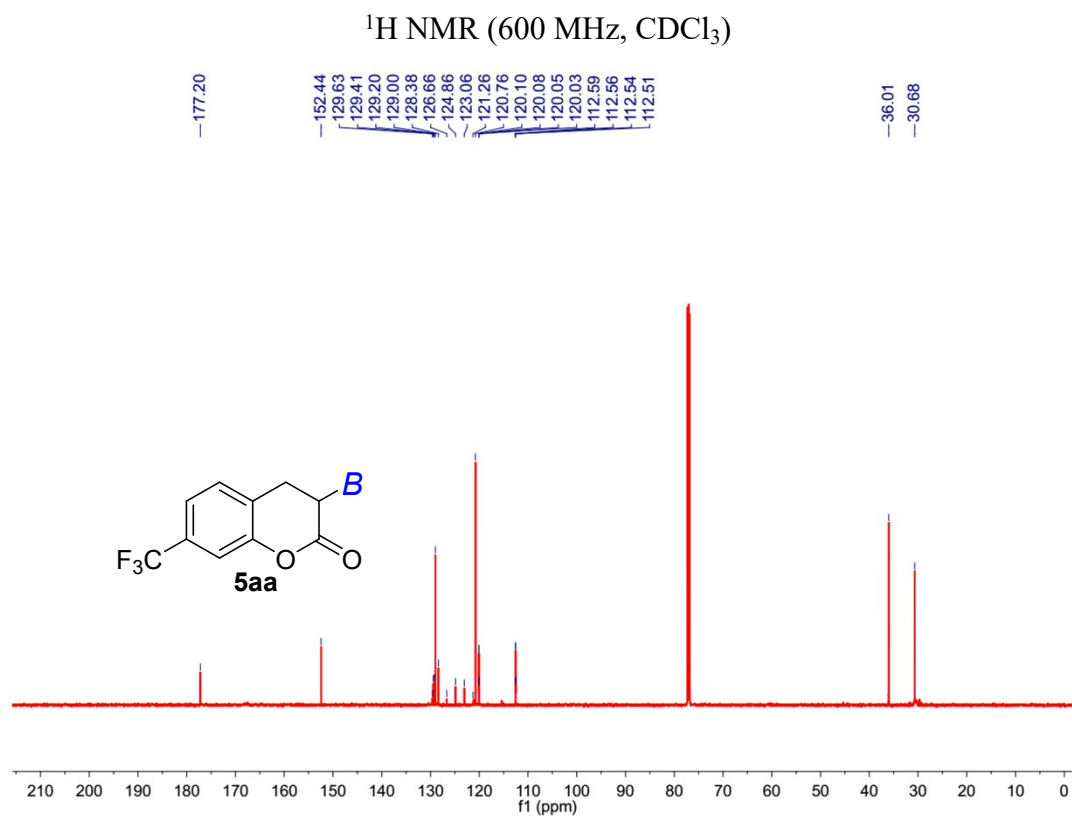
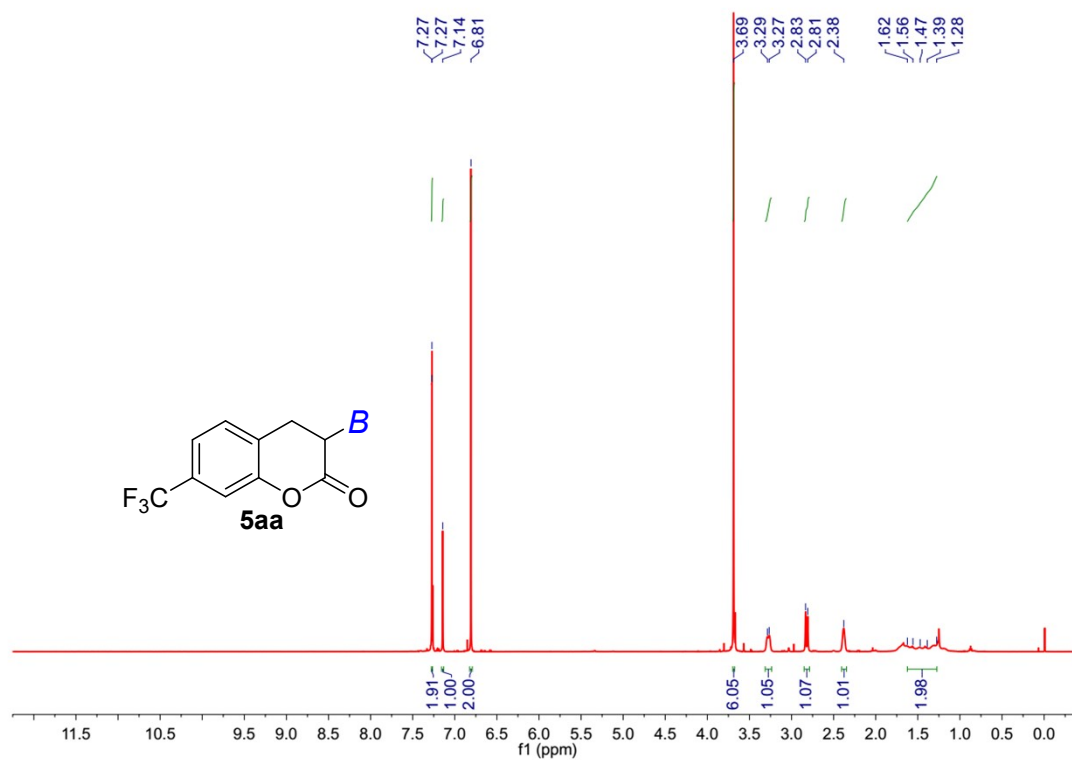
^{11}B NMR (193 MHz, CDCl_3)

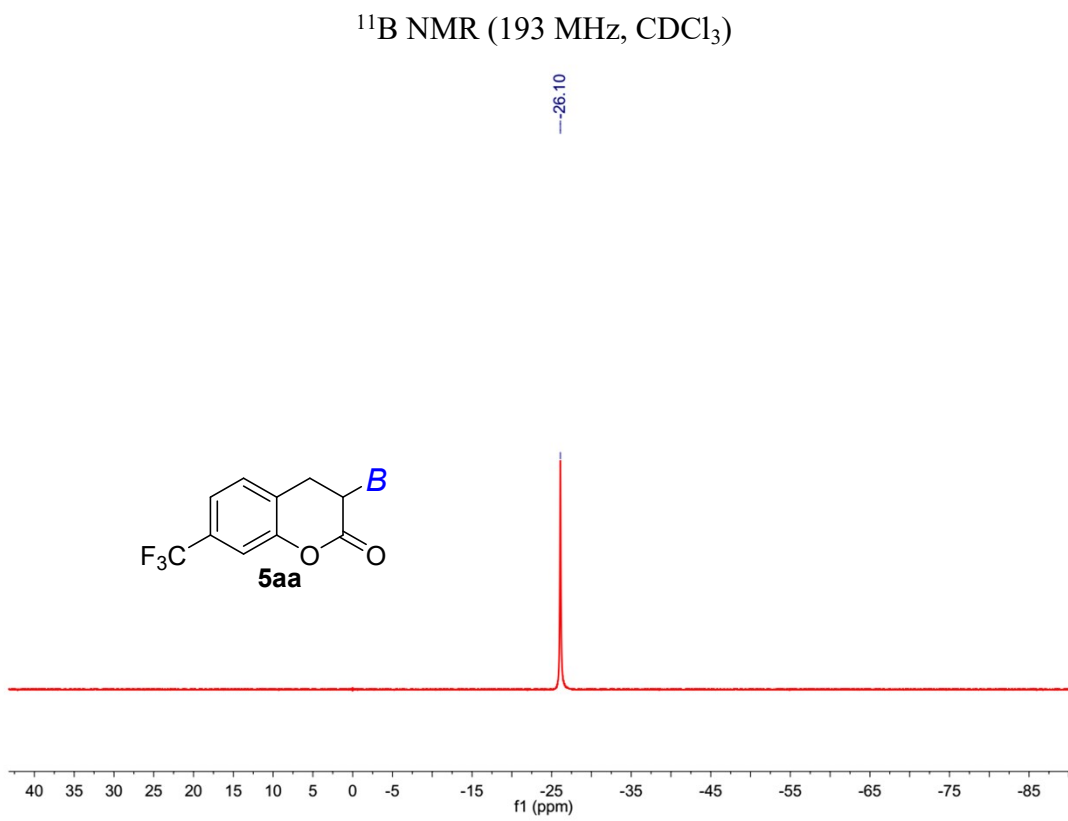
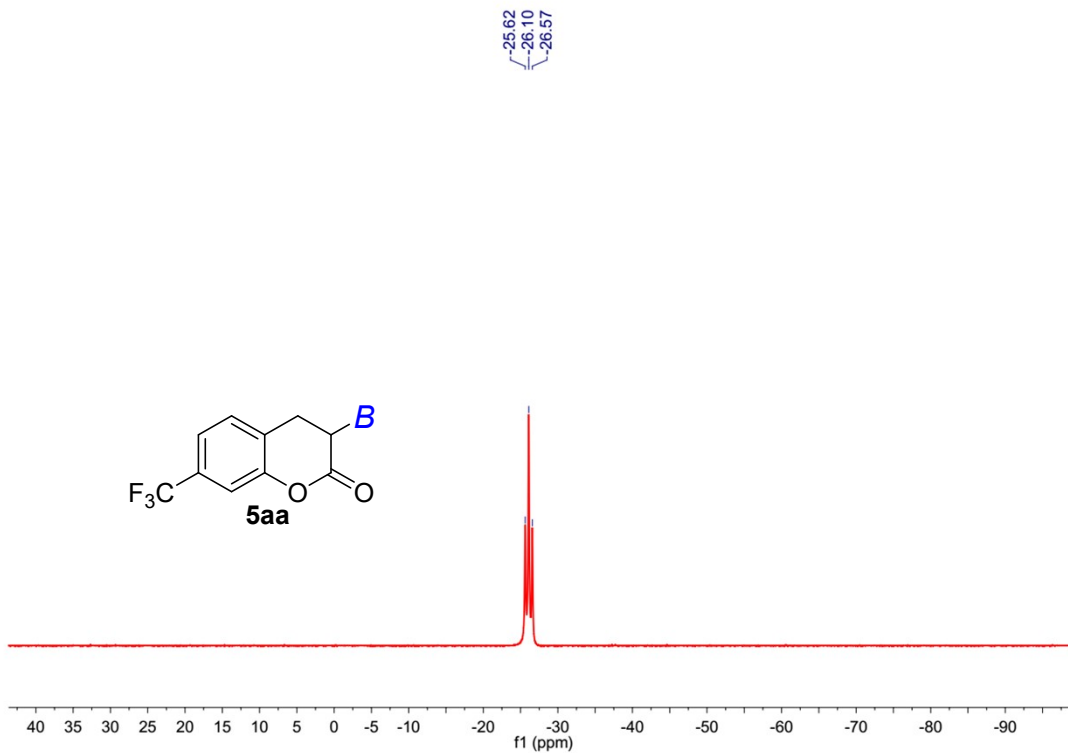


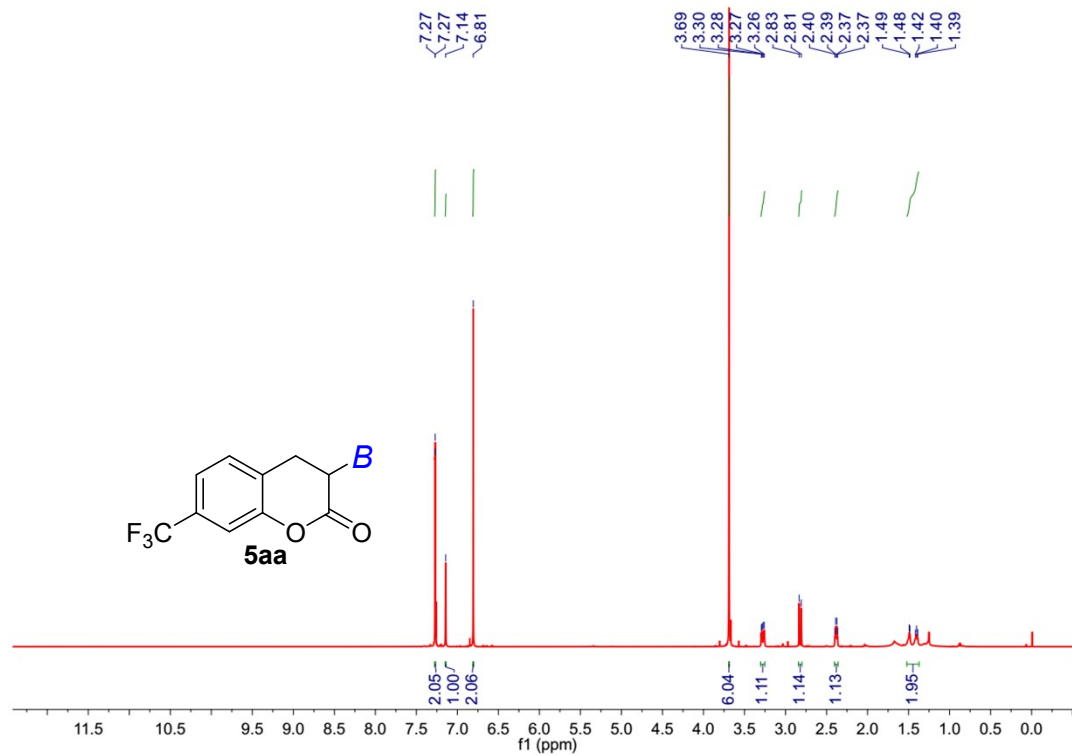
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

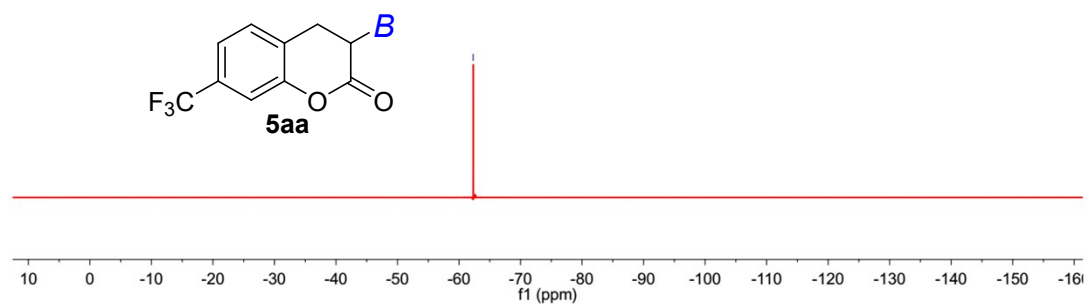




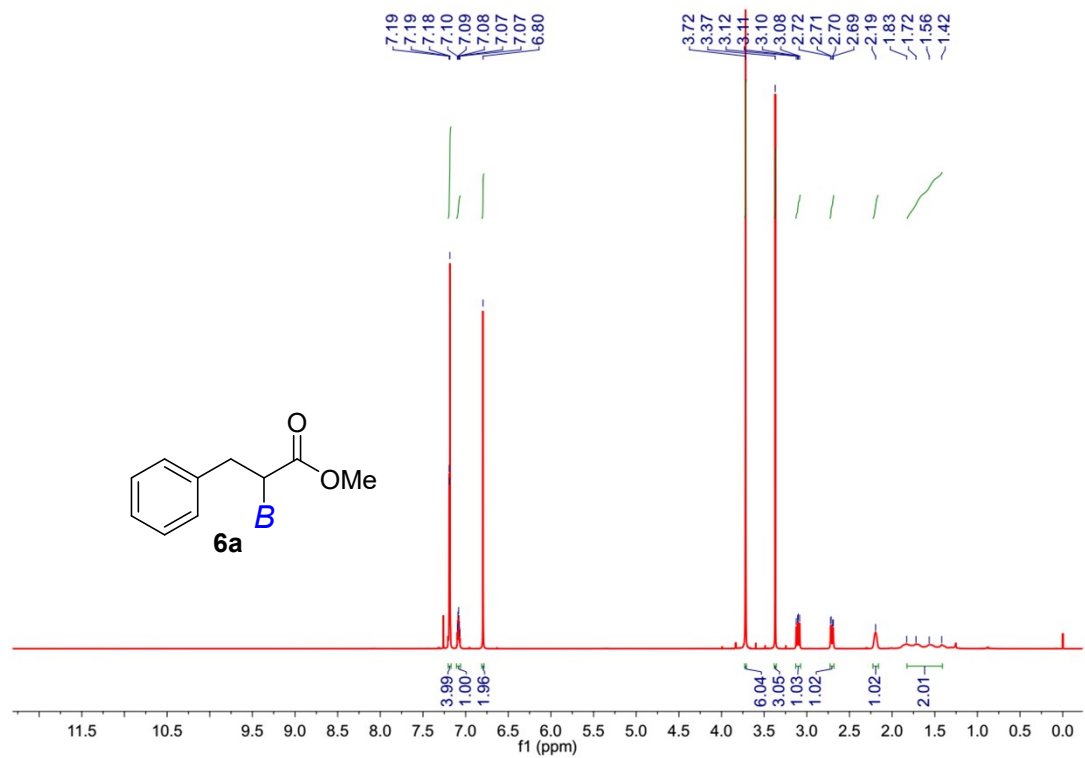


$^1H\{^{11}B\}$ NMR (600 MHz, $CDCl_3$)

---62.30



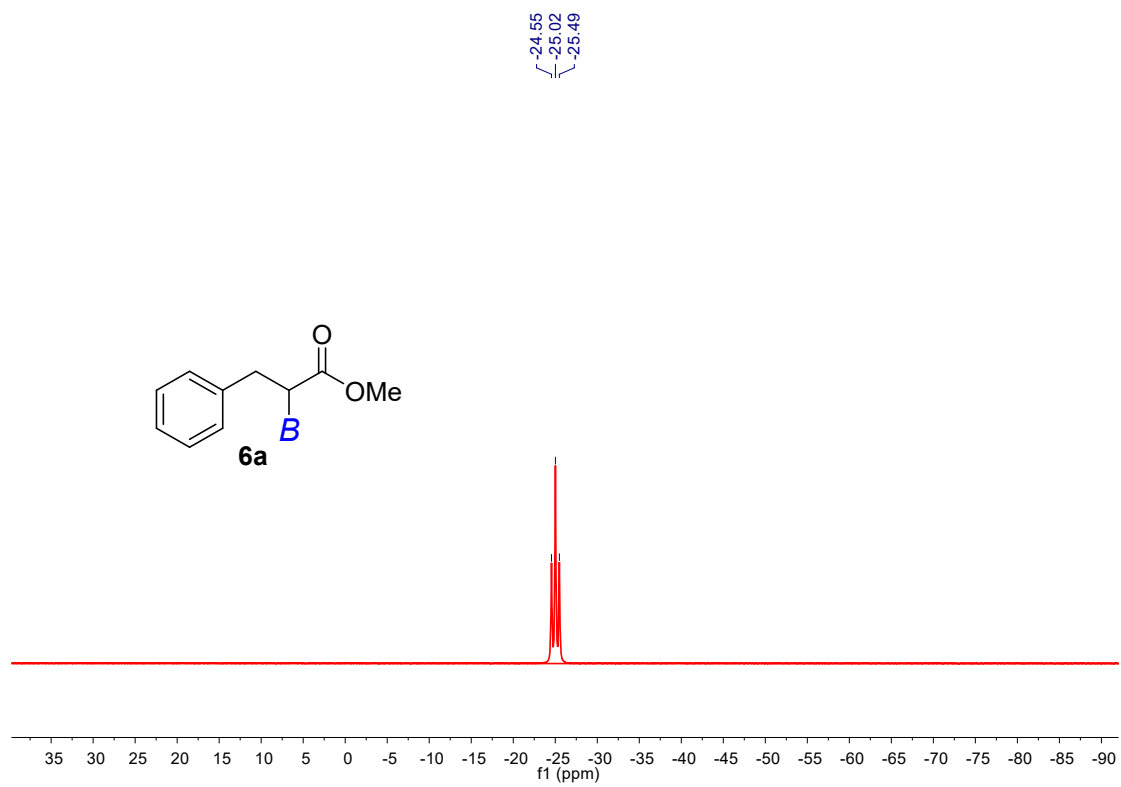
^{19}F NMR (565 MHz, $CDCl_3$)



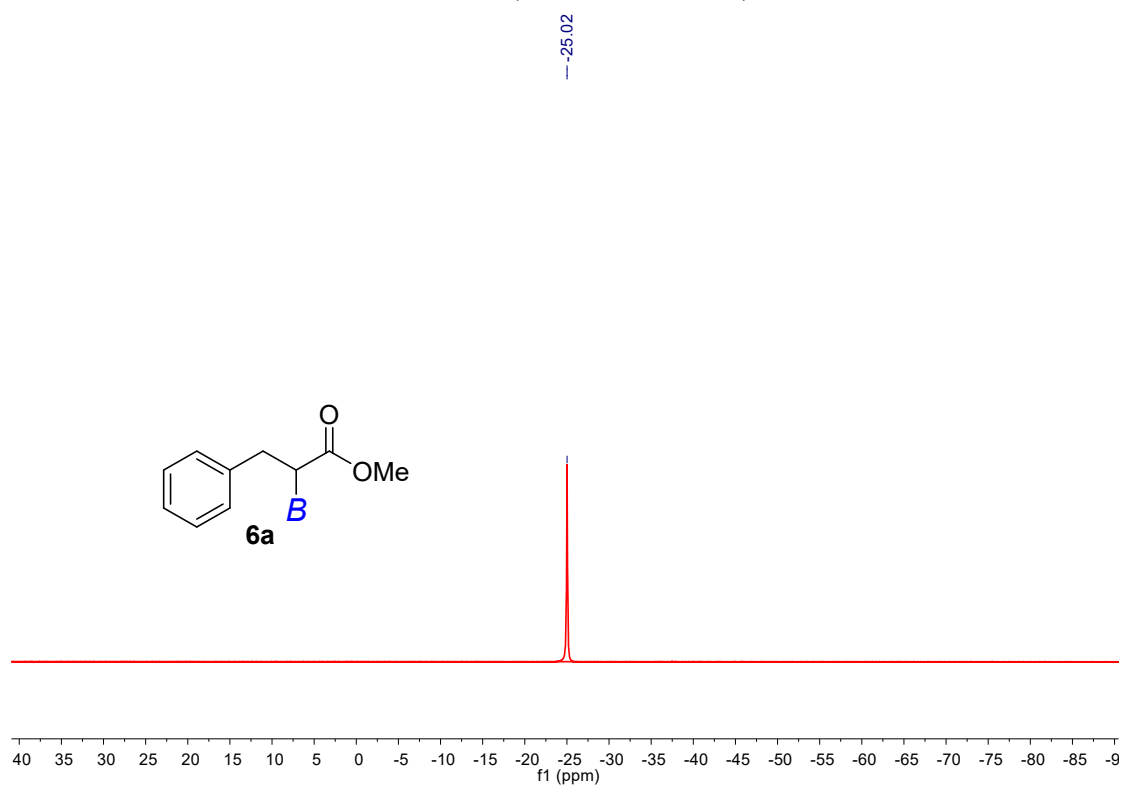
¹H NMR (600 MHz, CDCl₃)



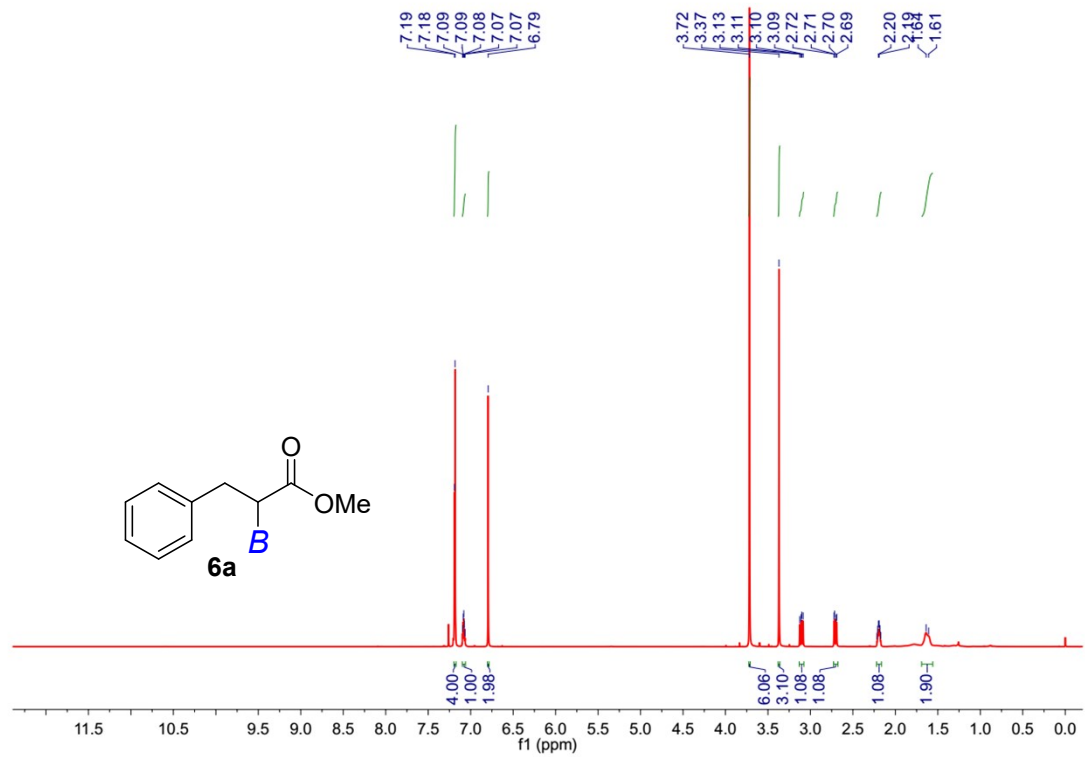
¹³C NMR (151 MHz, CDCl₃)



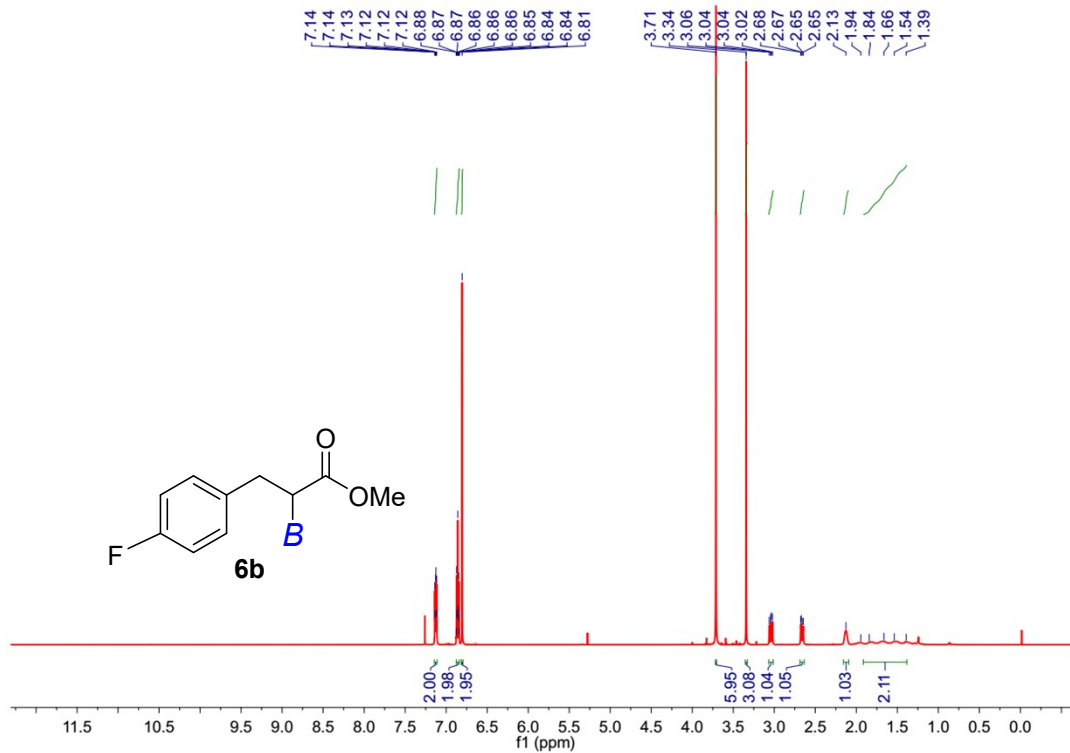
^{11}B NMR (193 MHz, CDCl_3)



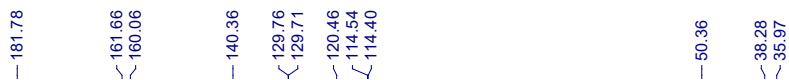
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



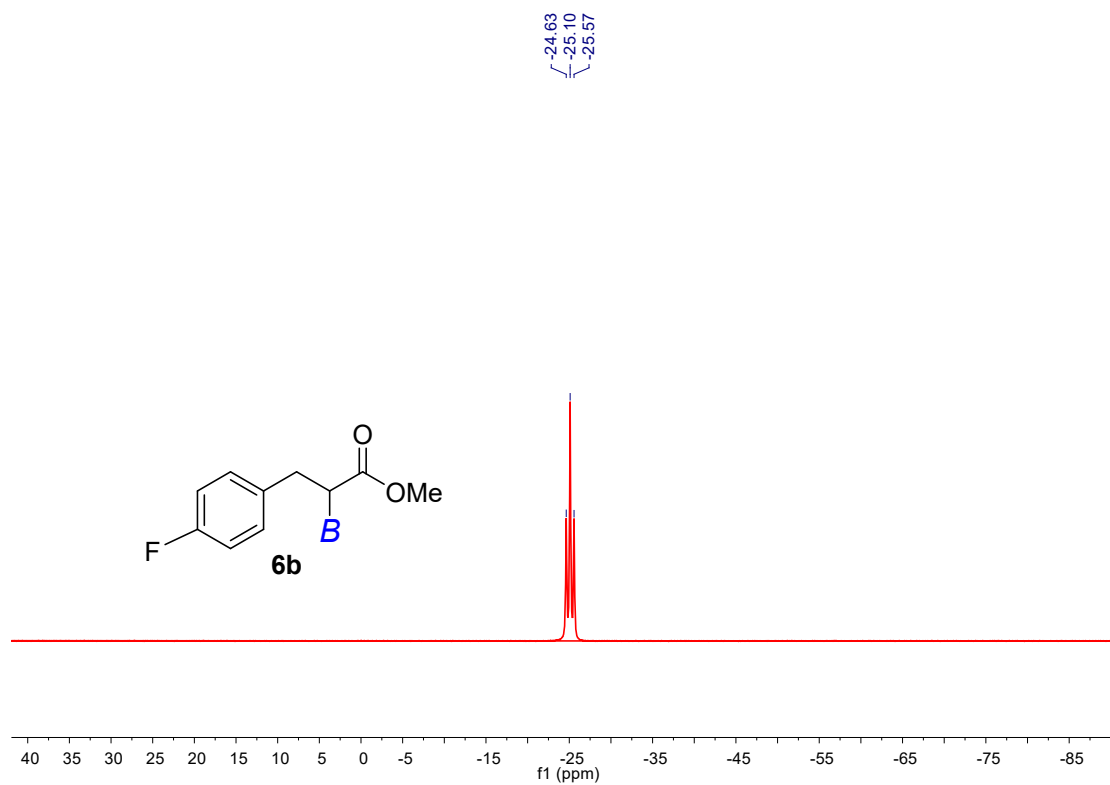
¹H{¹¹B} NMR (600 MHz, CDCl₃)



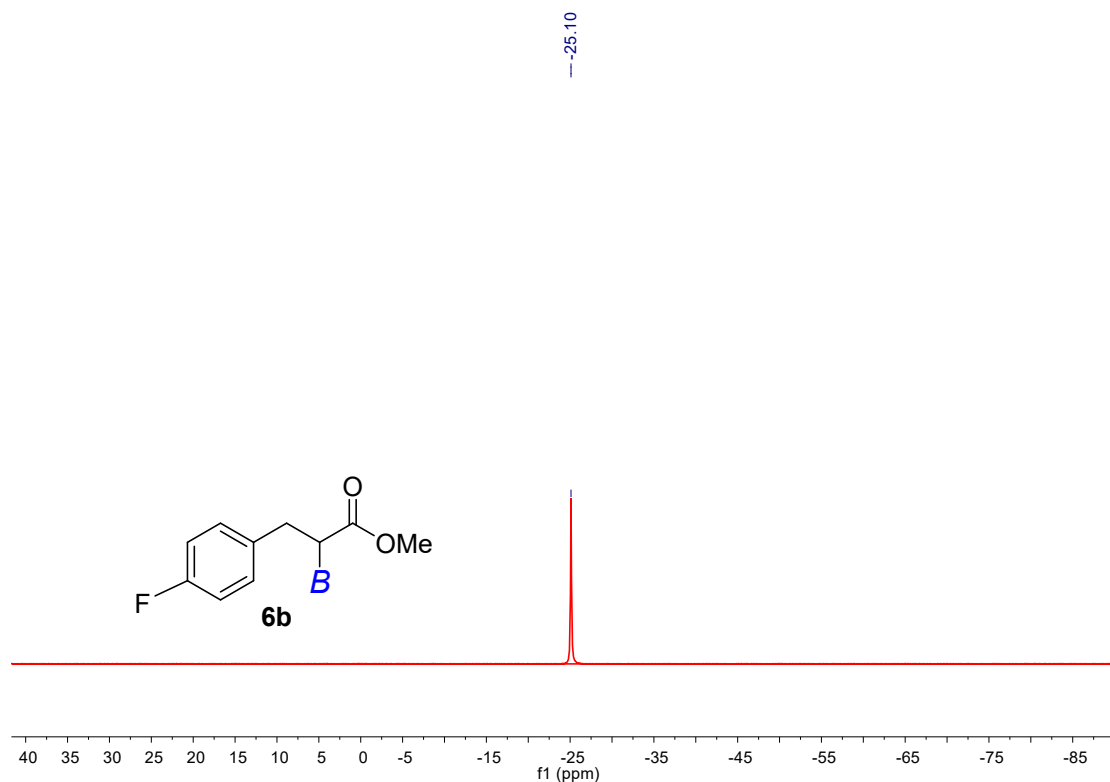
¹H NMR (600 MHz, CDCl₃)



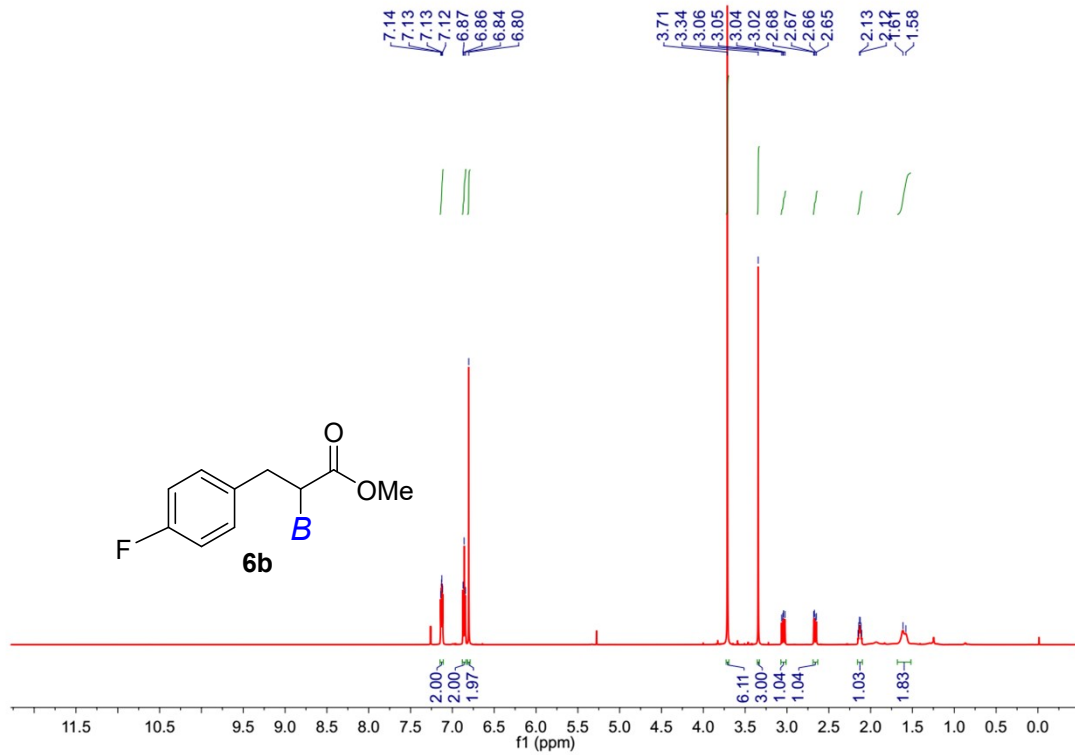
¹³C NMR (151 MHz, CDCl₃)



^{11}B NMR (193 MHz, CDCl_3)

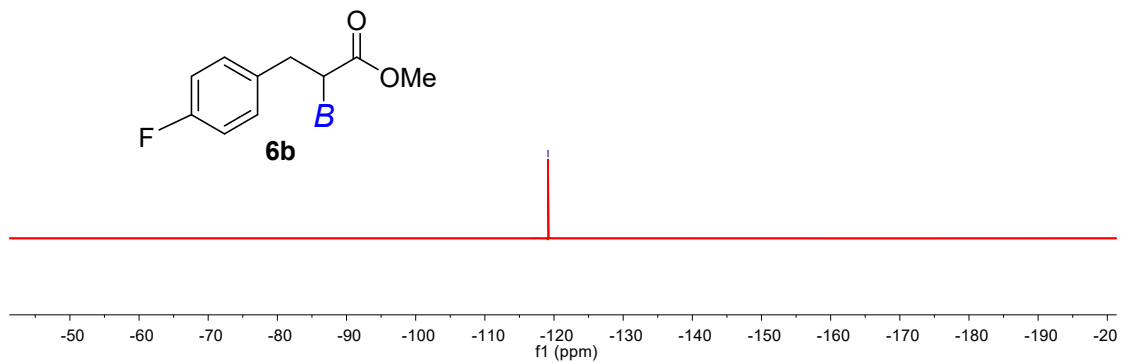


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

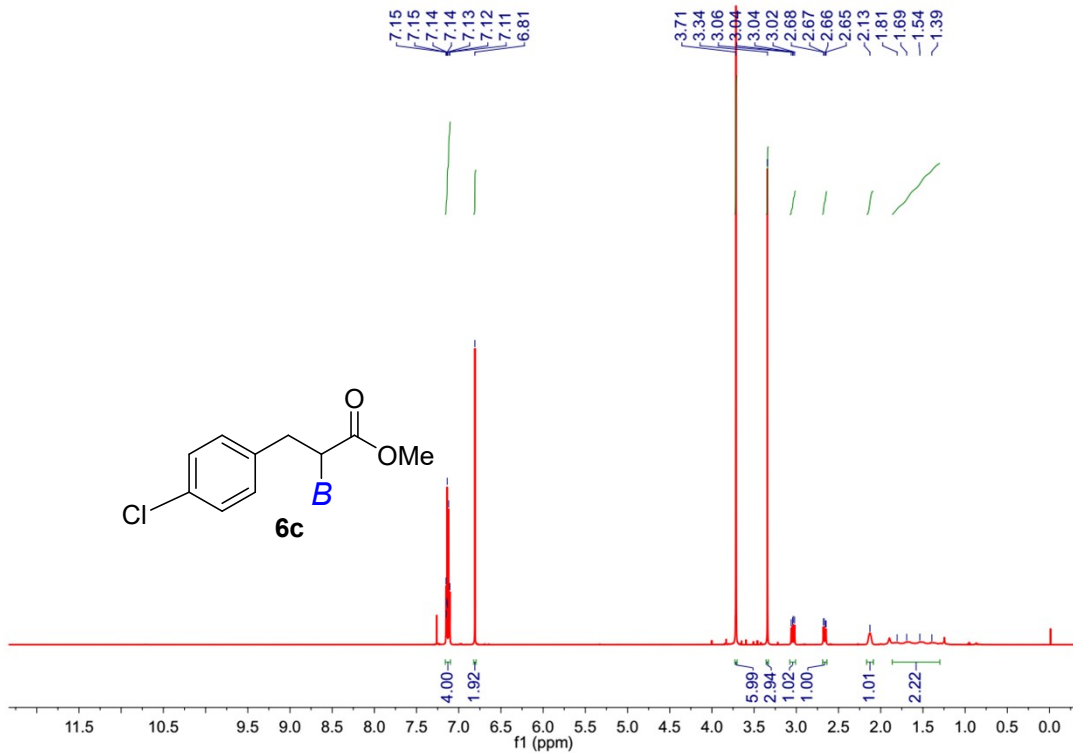


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

-119.14



^{19}F NMR (565 MHz, CDCl_3)



¹H NMR (600 MHz, CDCl₃)

— 181.67

— 143.24

— 130.61

— 129.88

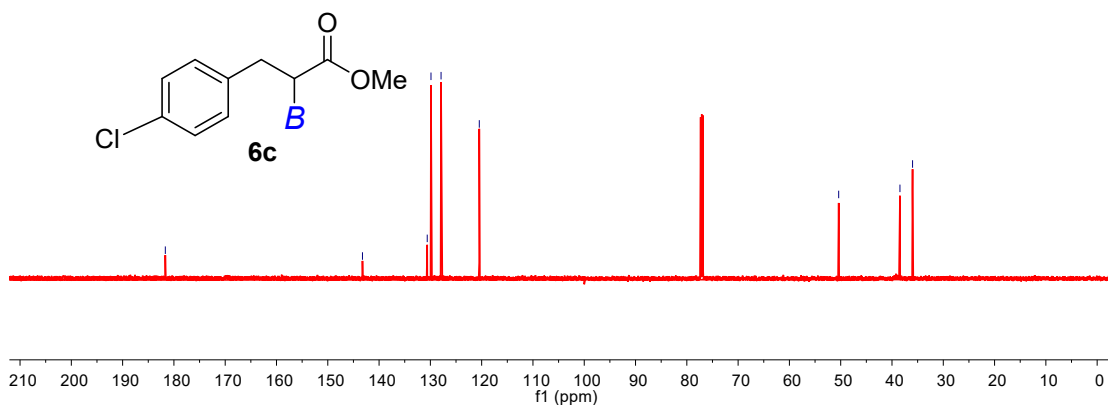
— 127.92

— 120.46

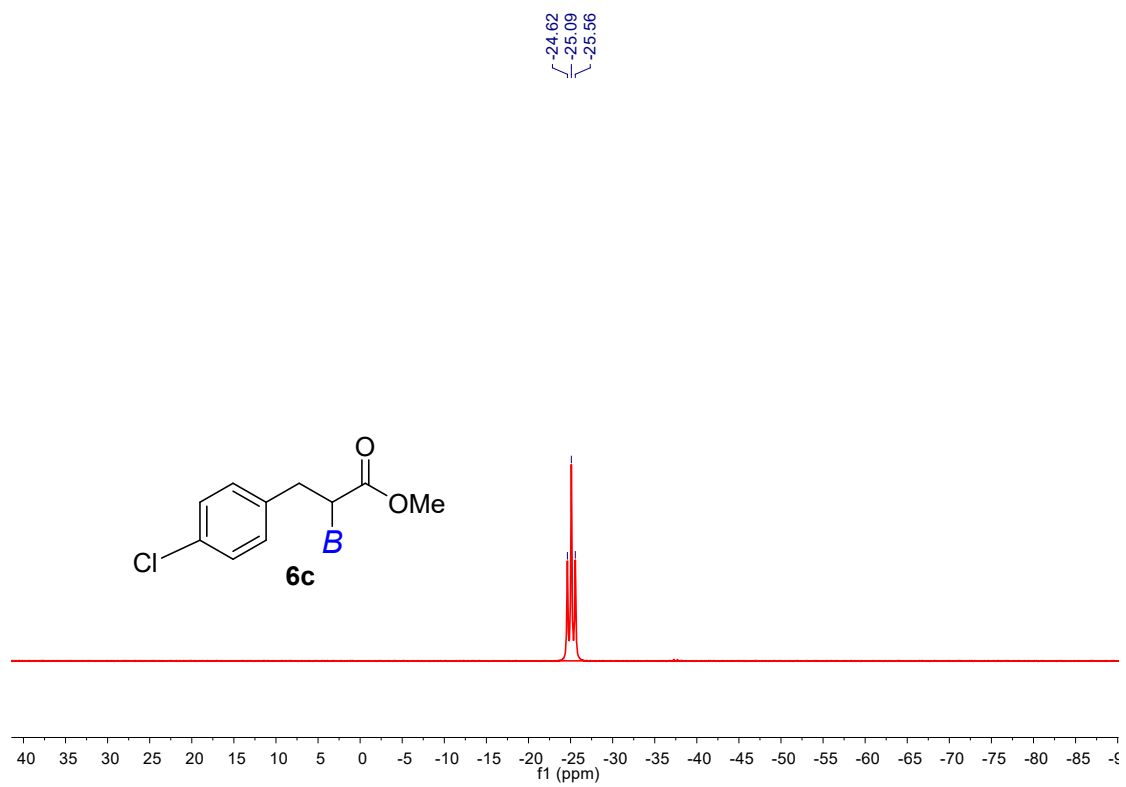
— 50.39

— 38.45

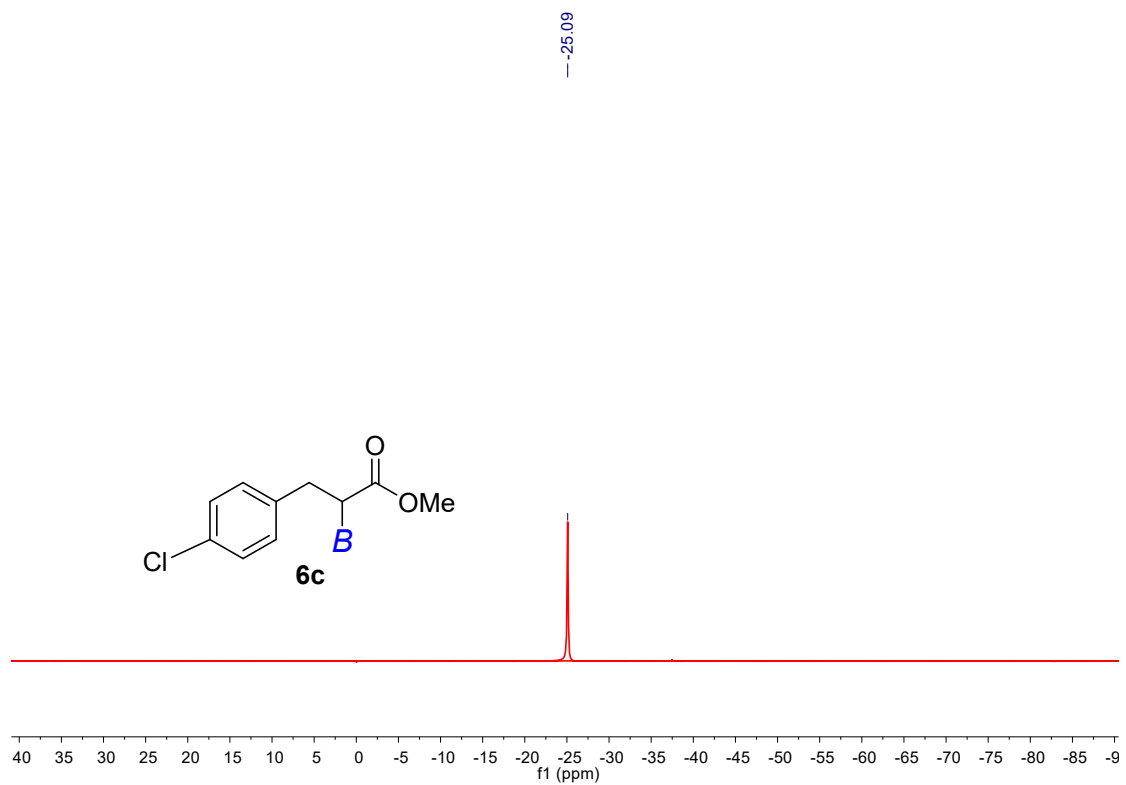
— 35.99



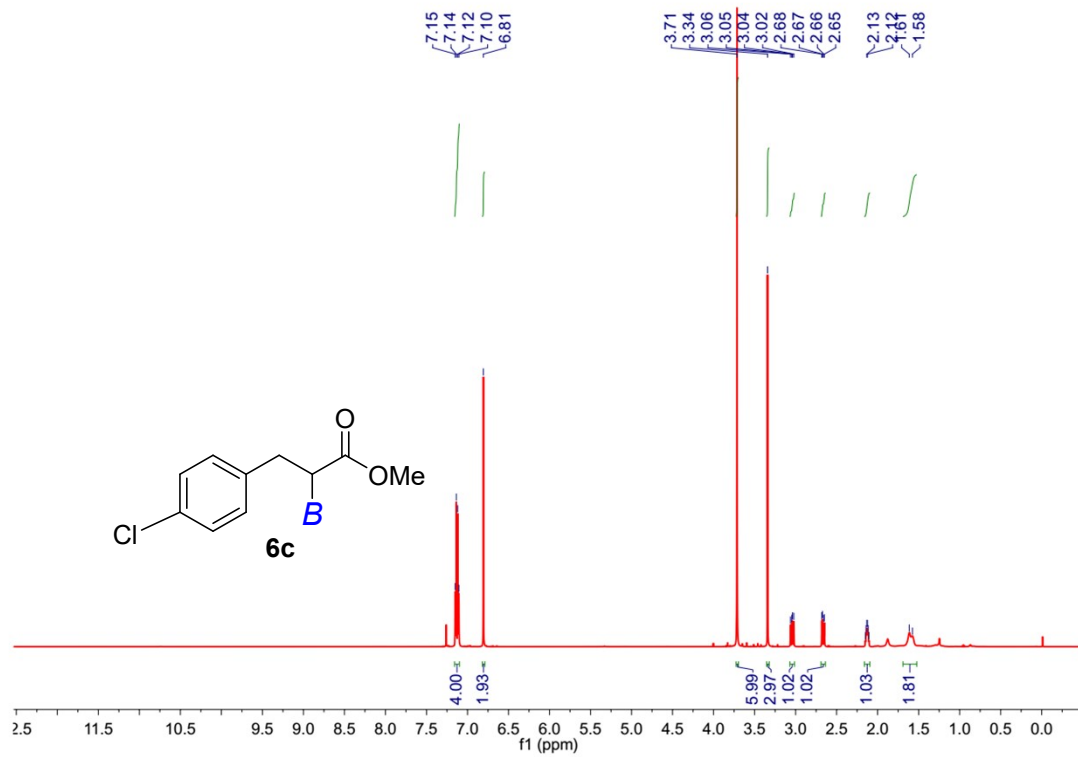
¹³C NMR (151 MHz, CDCl₃)



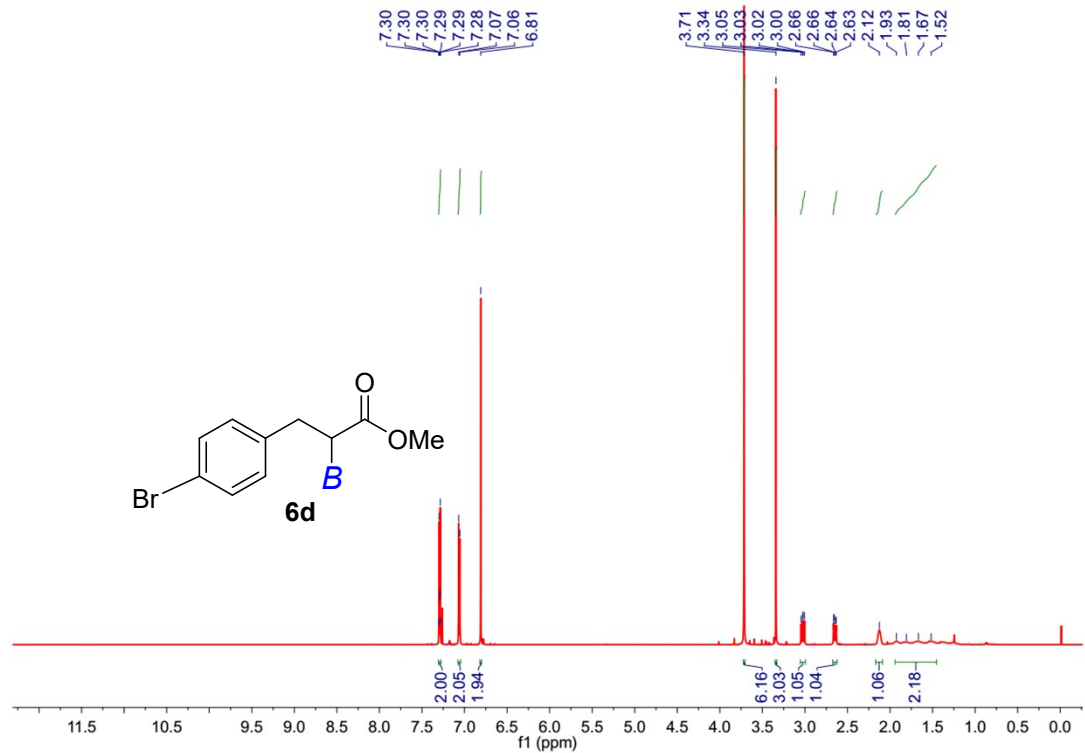
^{11}B NMR (193 MHz, CDCl_3)



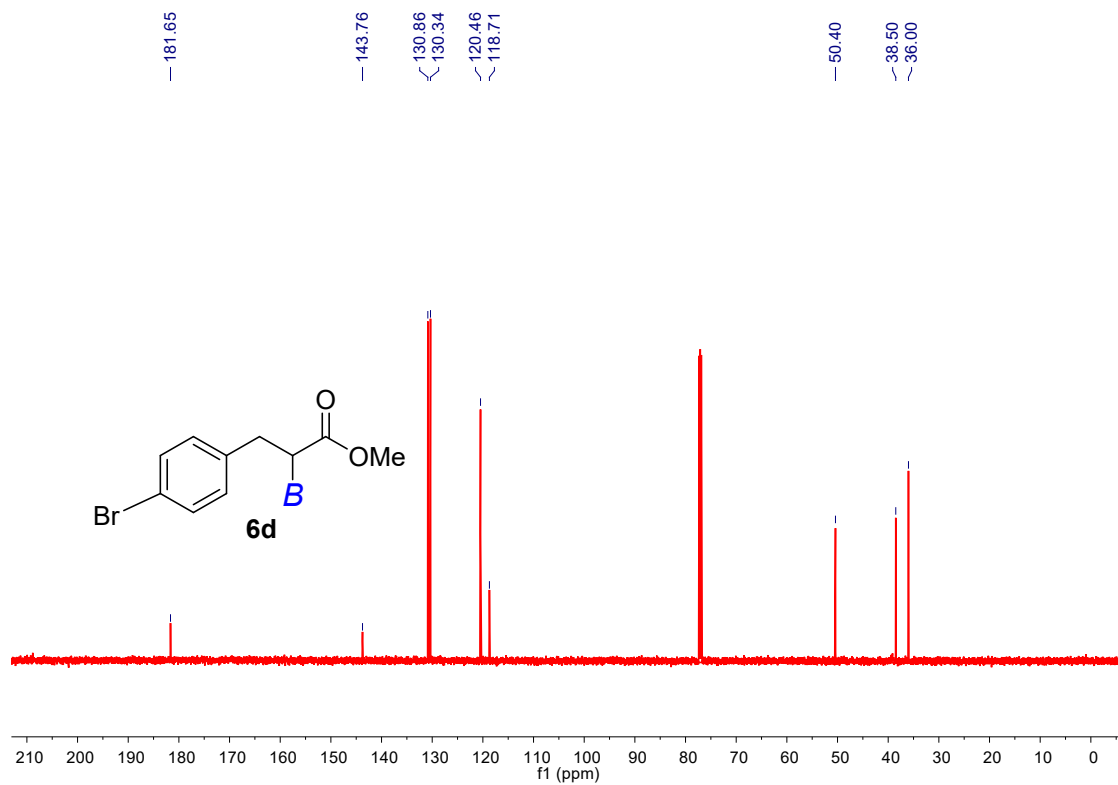
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



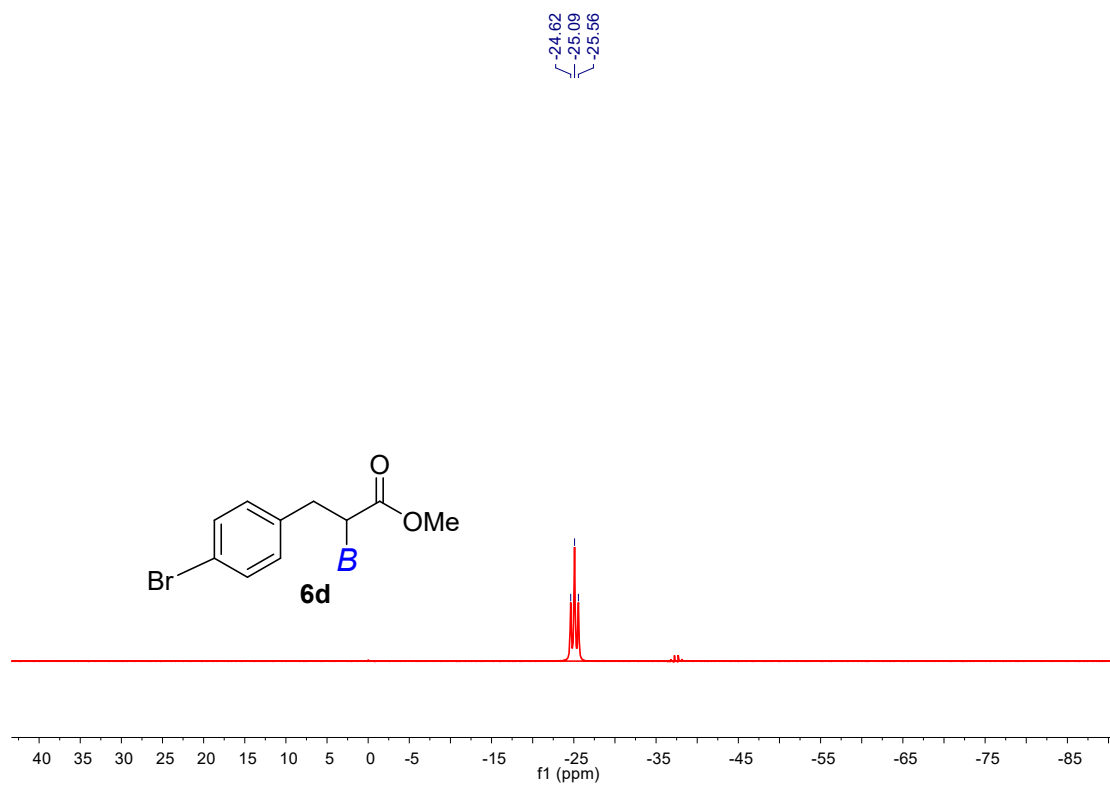
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



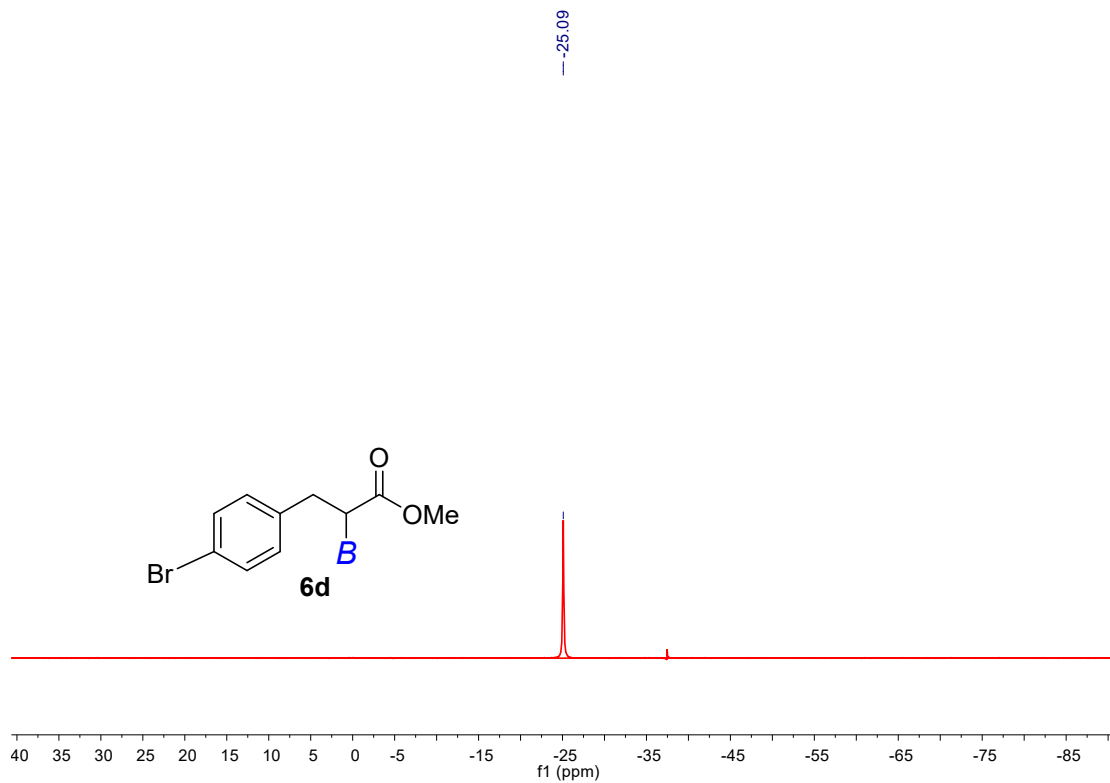
¹H NMR (600 MHz, CDCl₃)



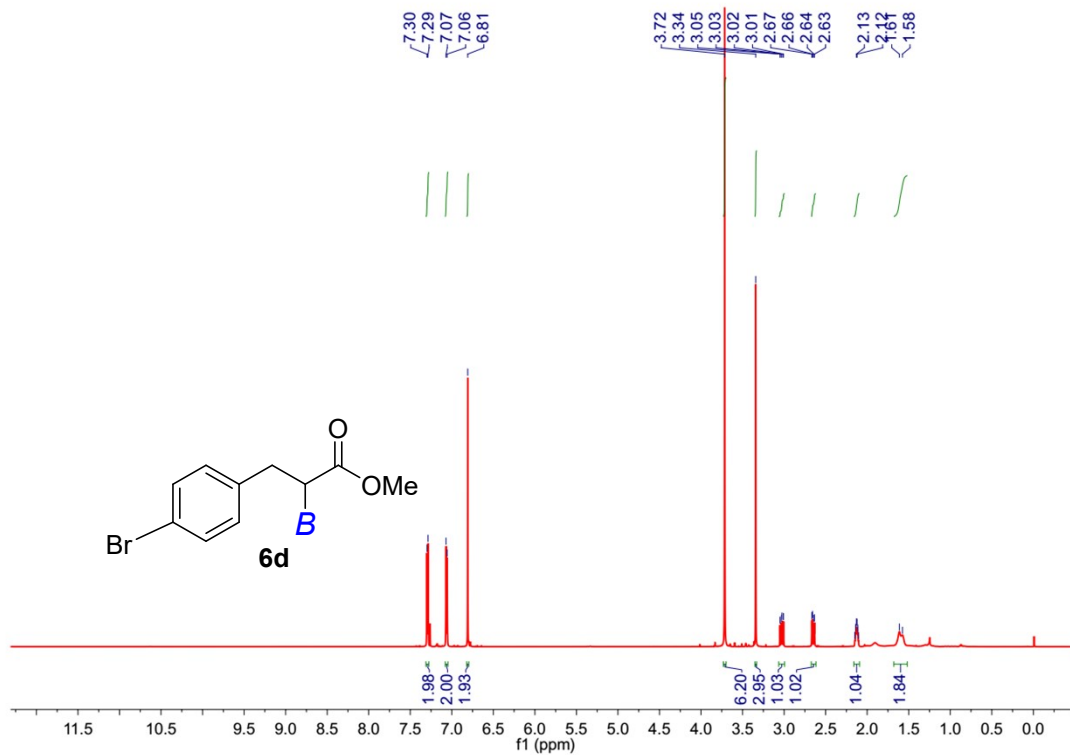
¹³C NMR (151 MHz, CDCl₃)



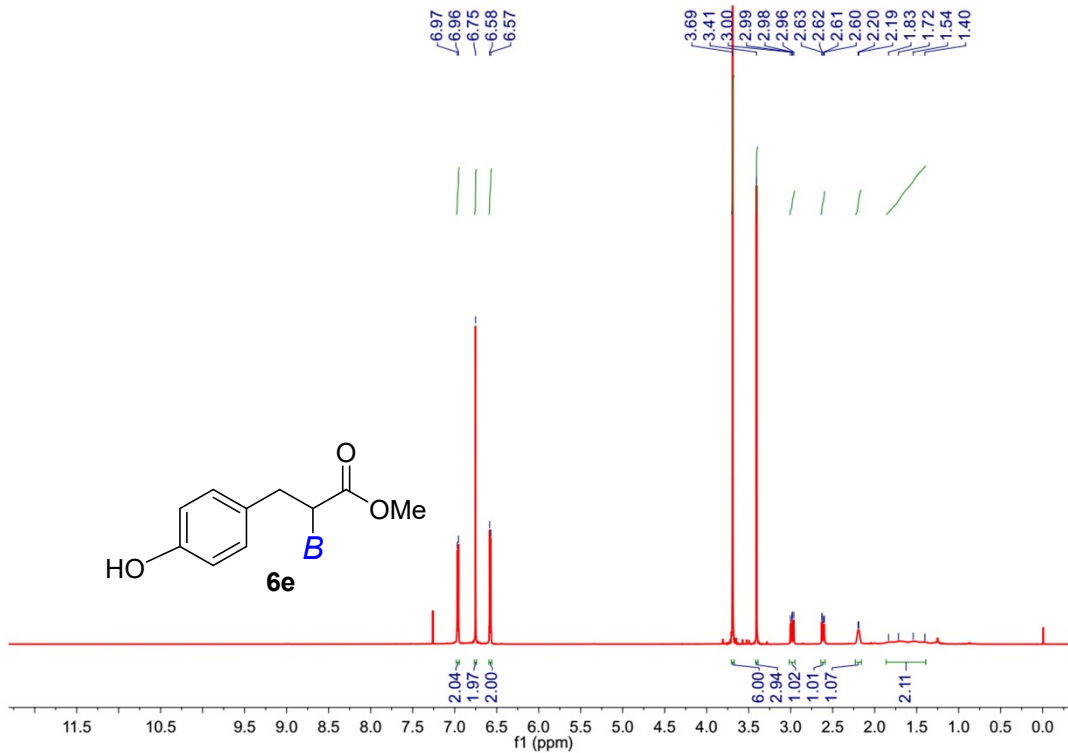
^{11}B NMR (193 MHz, CDCl_3)



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

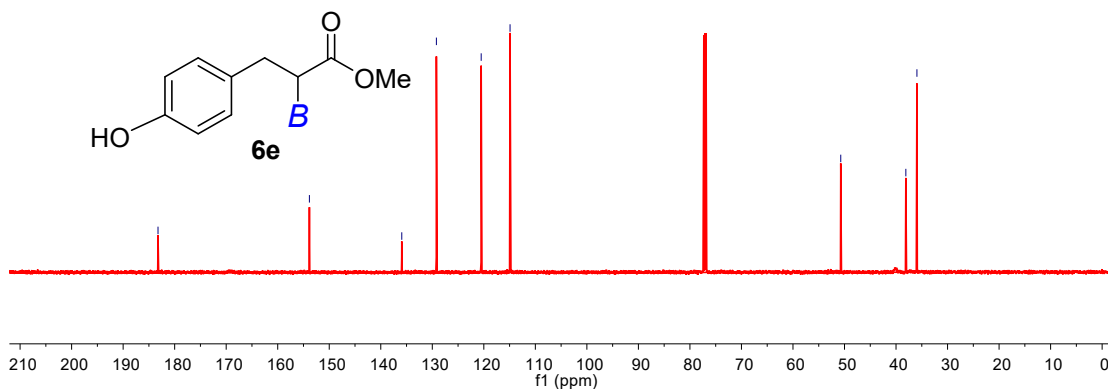


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

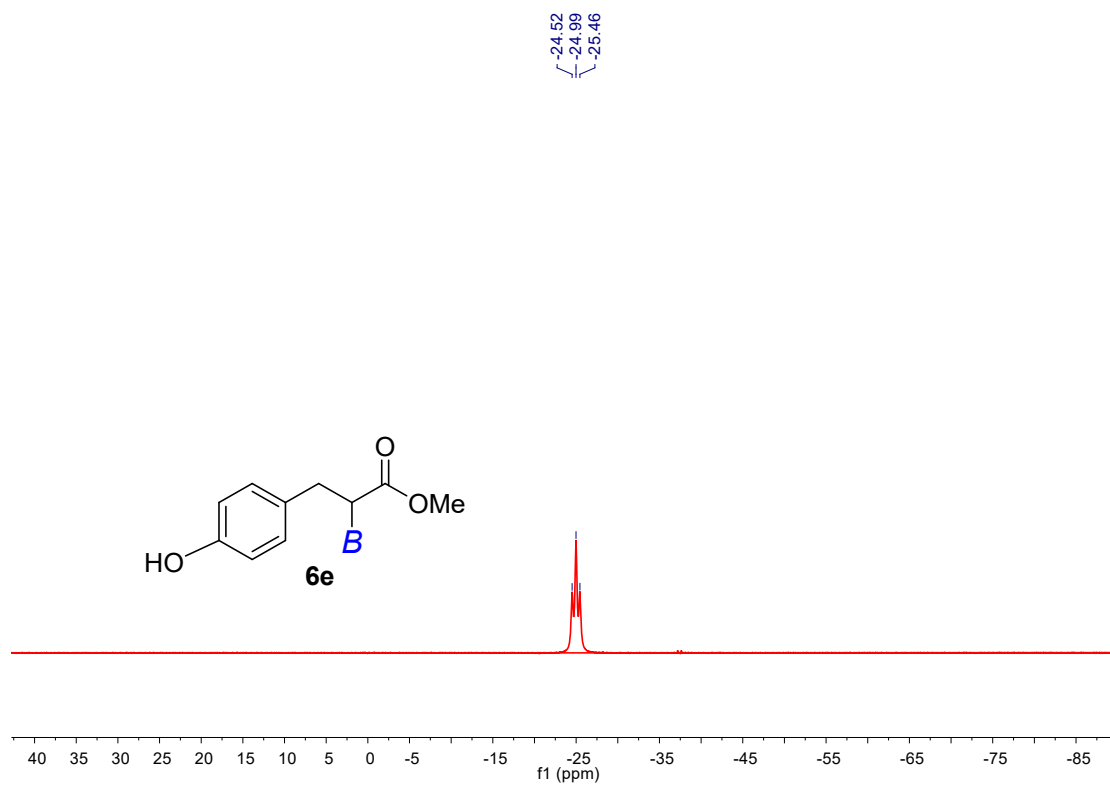


¹H NMR (600 MHz, CDCl₃)

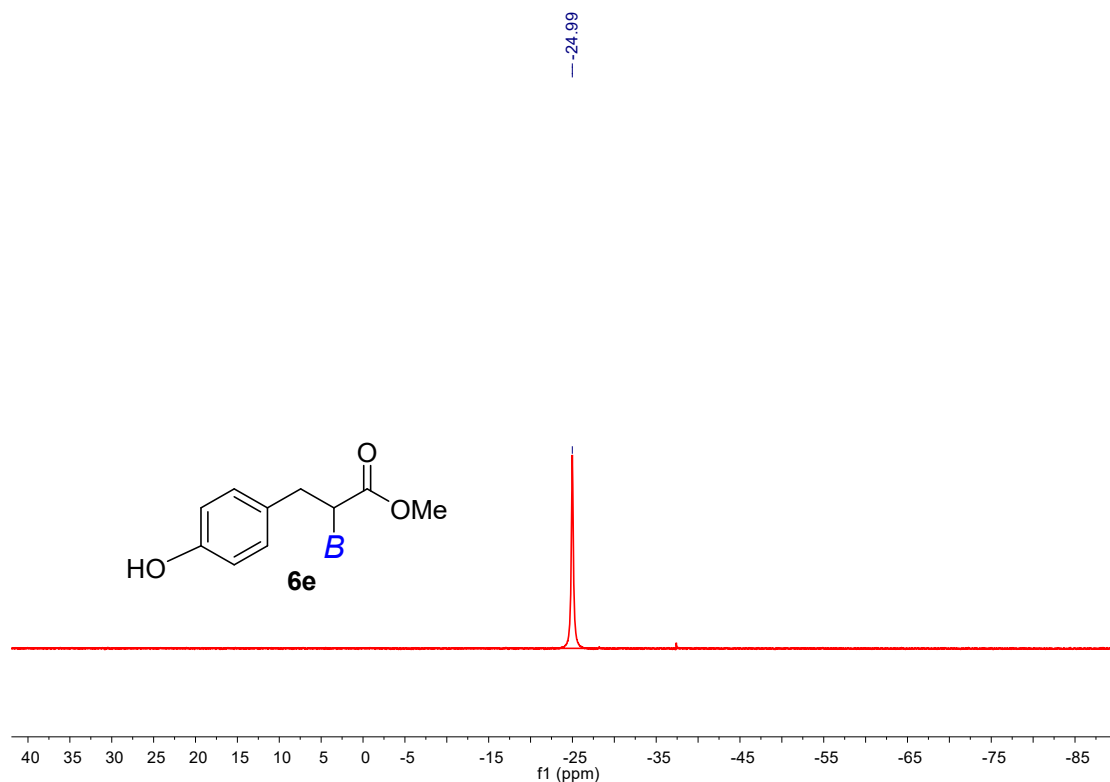
183.23, 153.85, 135.92, 129.20, 120.51, 114.91, 50.73, 38.10, 35.94



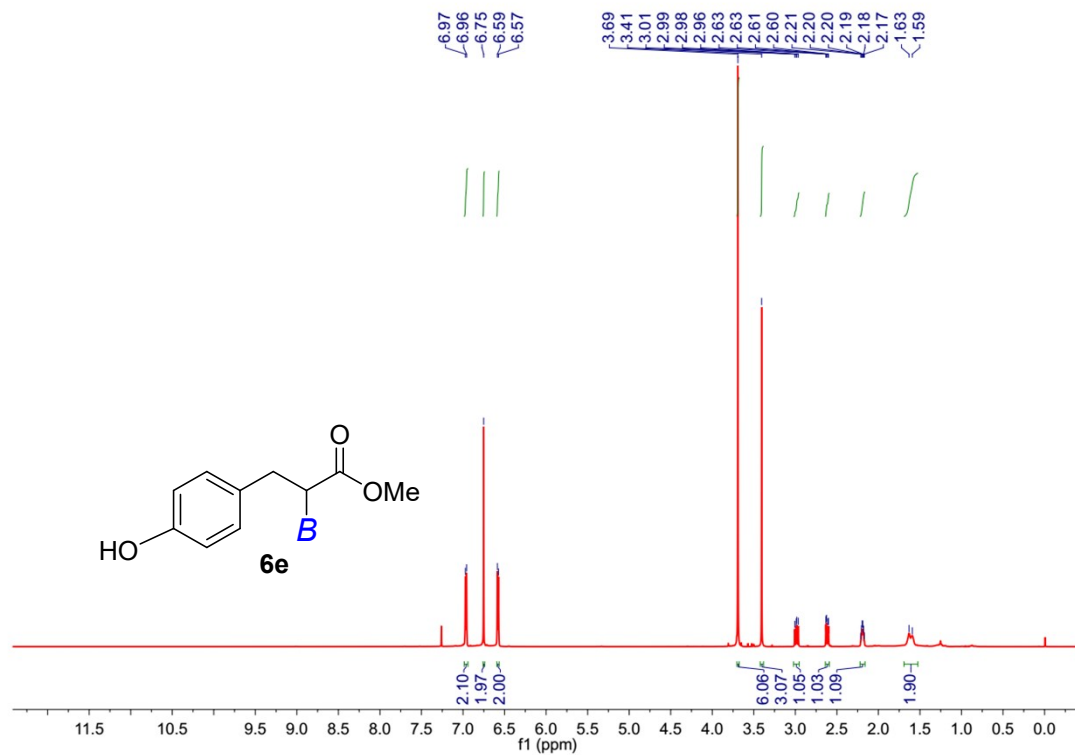
¹³C NMR (151 MHz, CDCl₃)



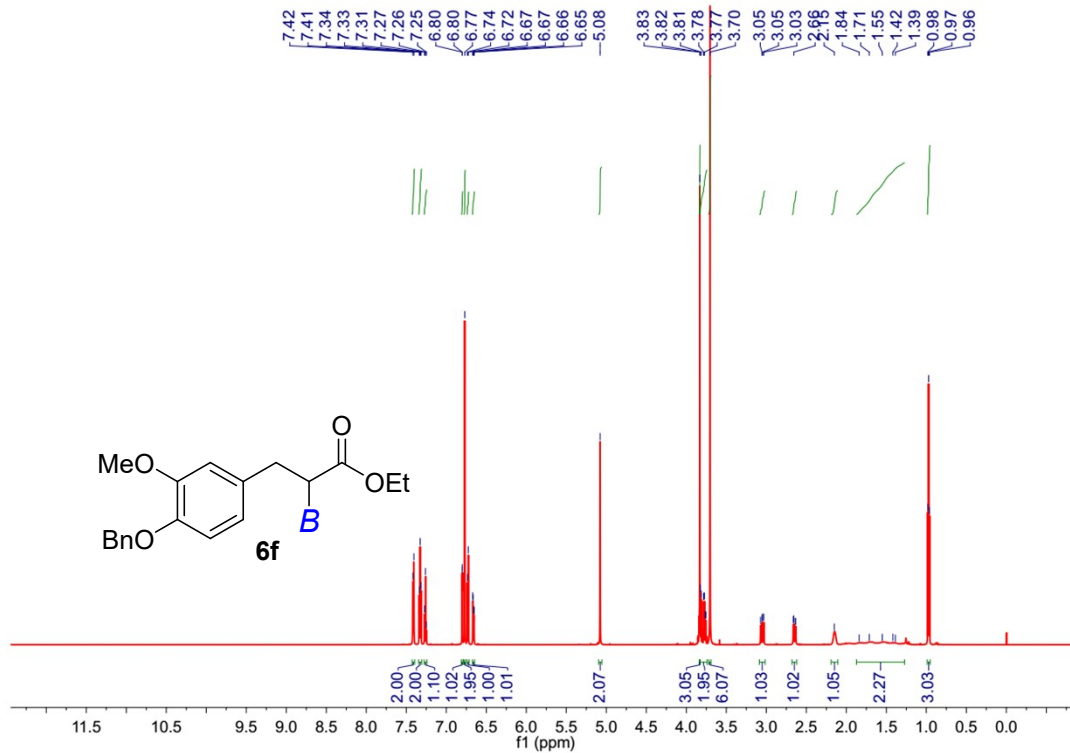
¹¹B NMR (193 MHz, CDCl₃)



¹¹B{¹H} NMR (193 MHz, CDCl₃)



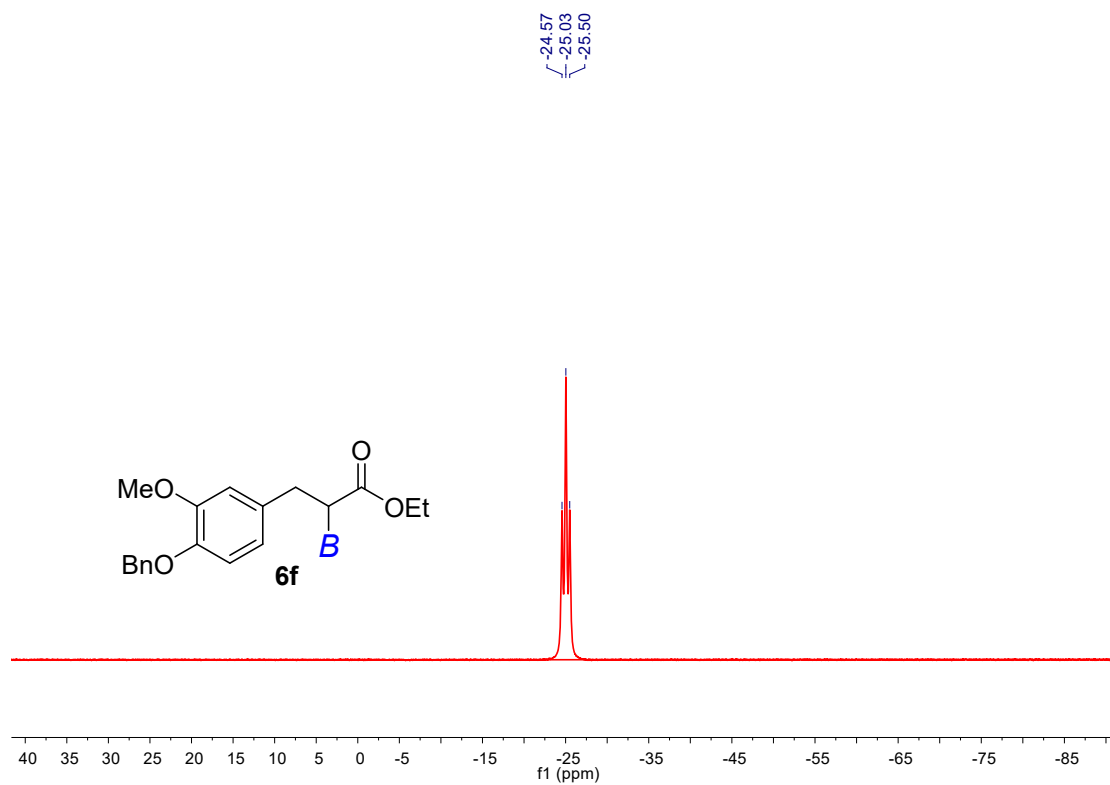
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



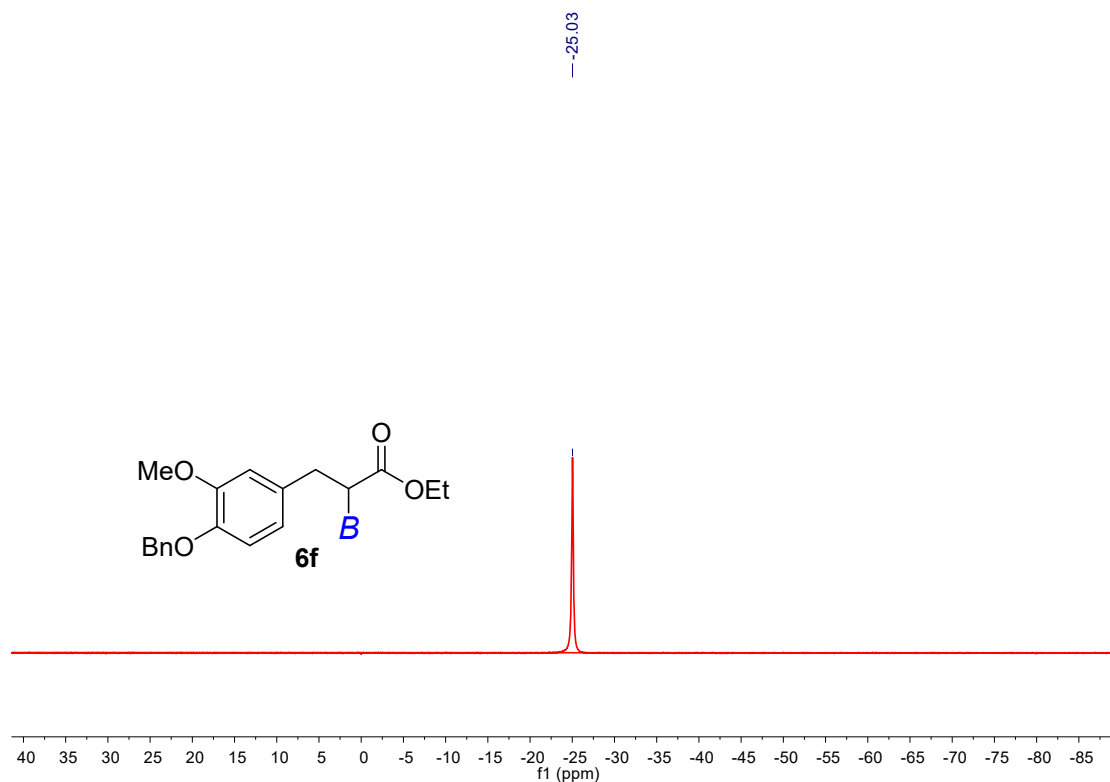
1H NMR (600 MHz, CDCl₃)



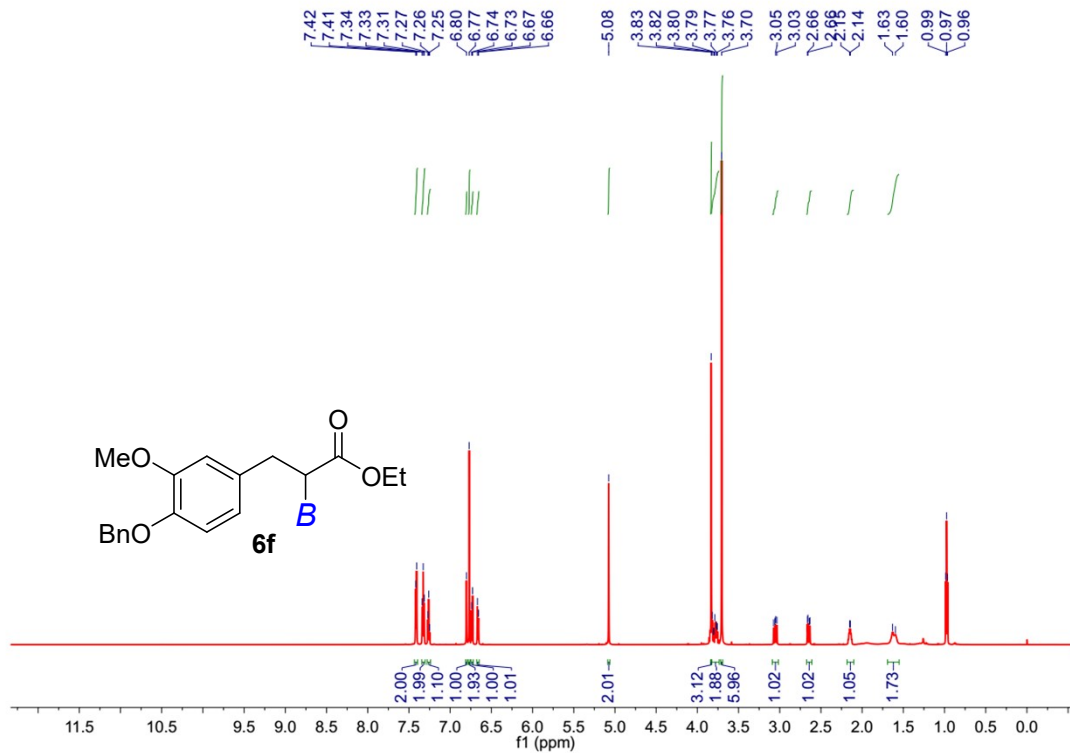
13C NMR (151 MHz, CDCl₃)



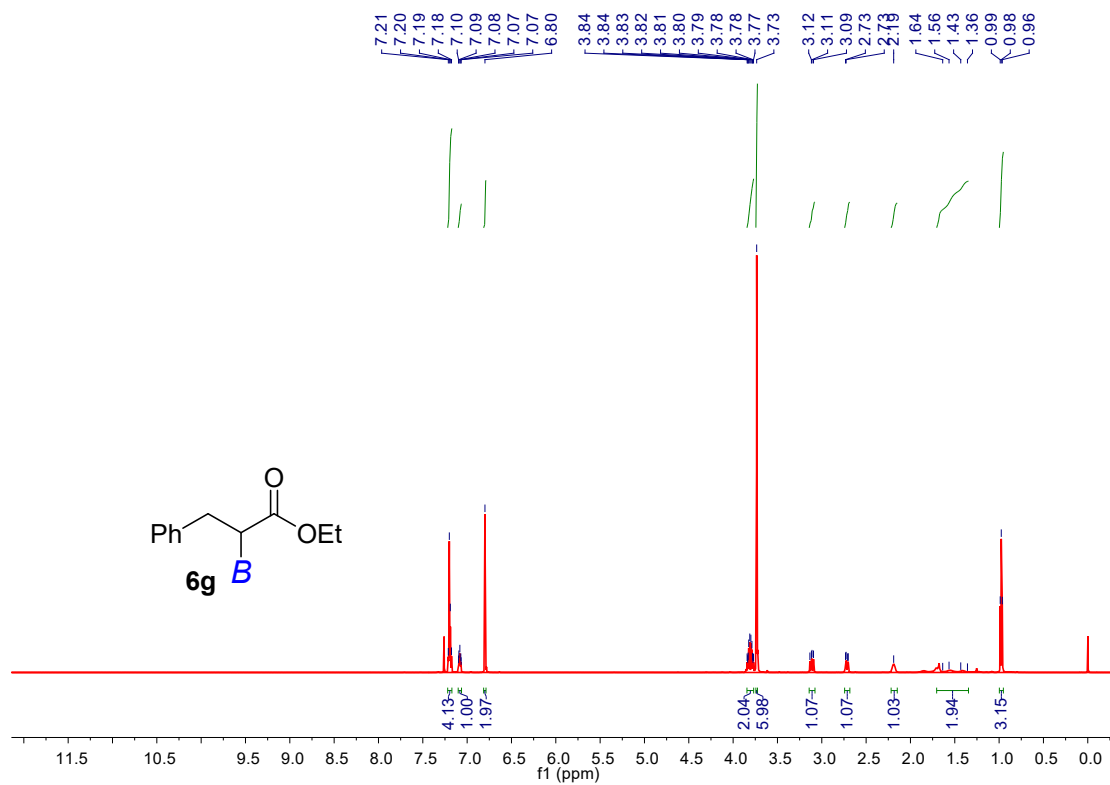
^{11}B NMR (193 MHz, CDCl_3)



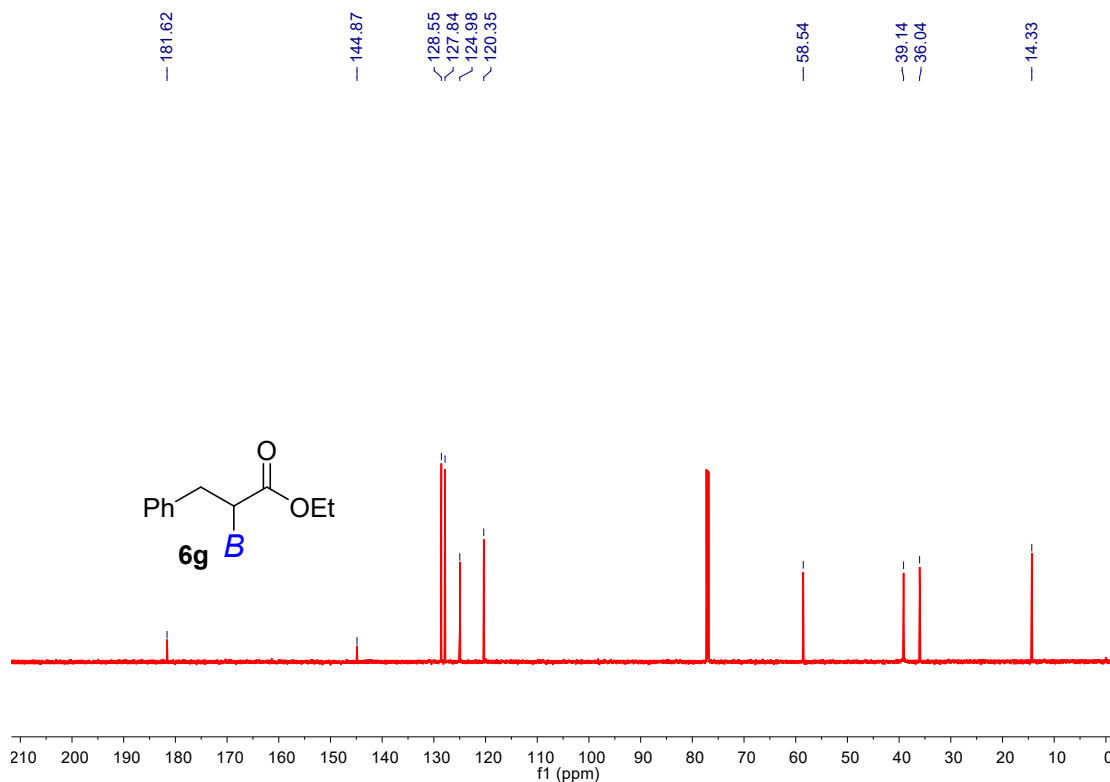
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



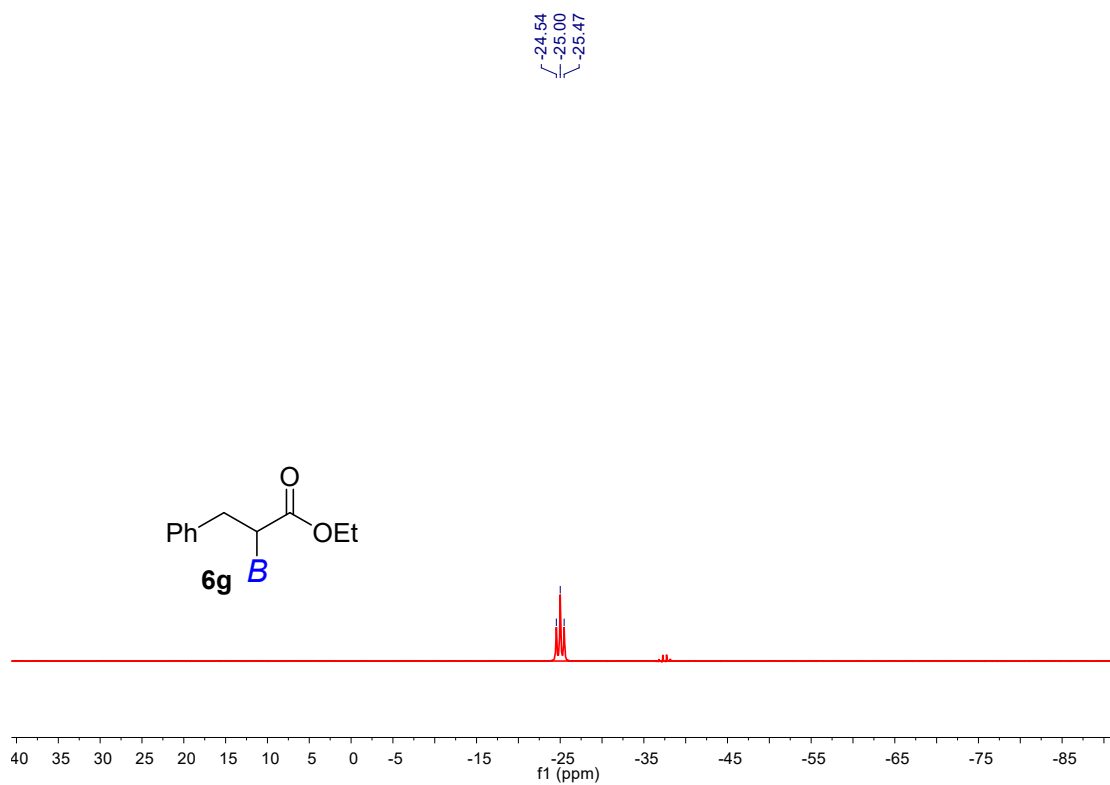
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



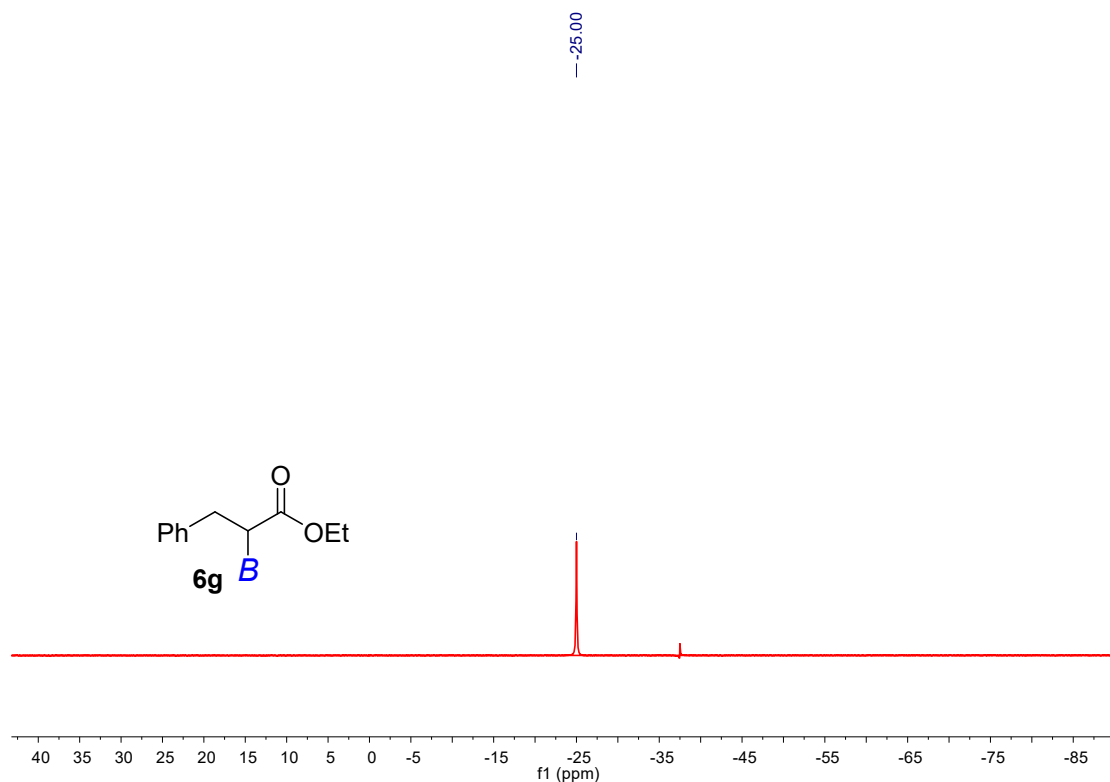
¹H NMR (600 MHz, CDCl₃)



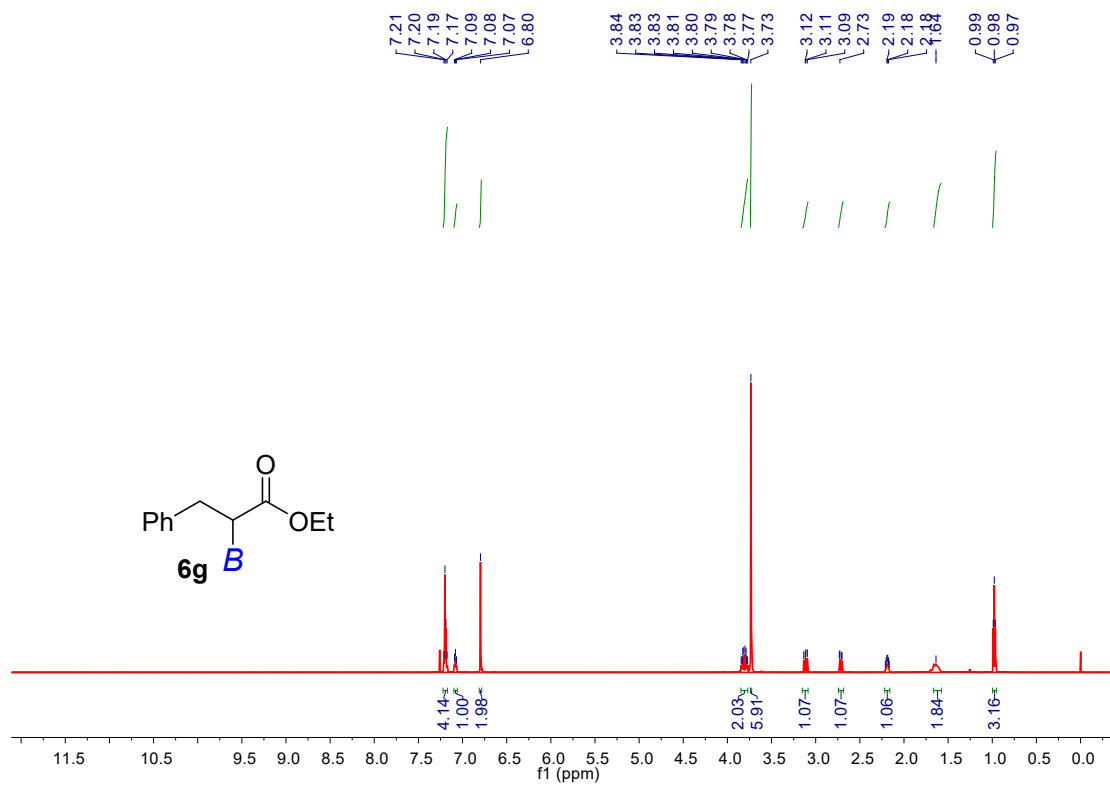
¹³C NMR (151 MHz, CDCl₃)



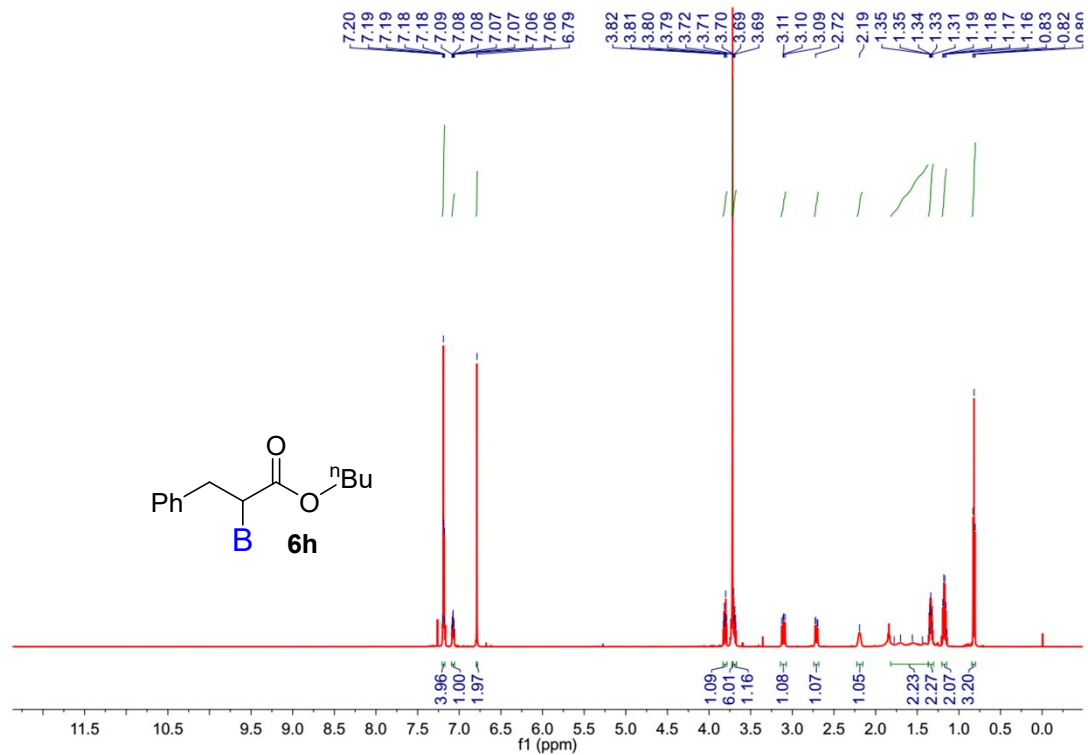
^{11}B NMR (193 MHz, CDCl_3)



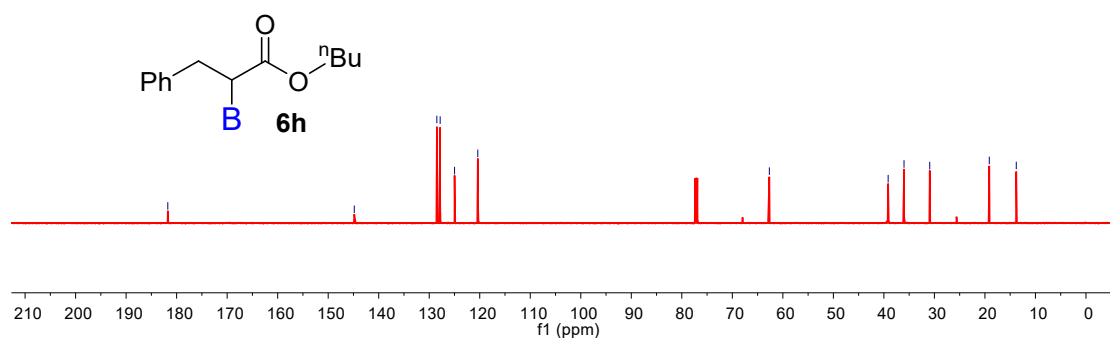
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



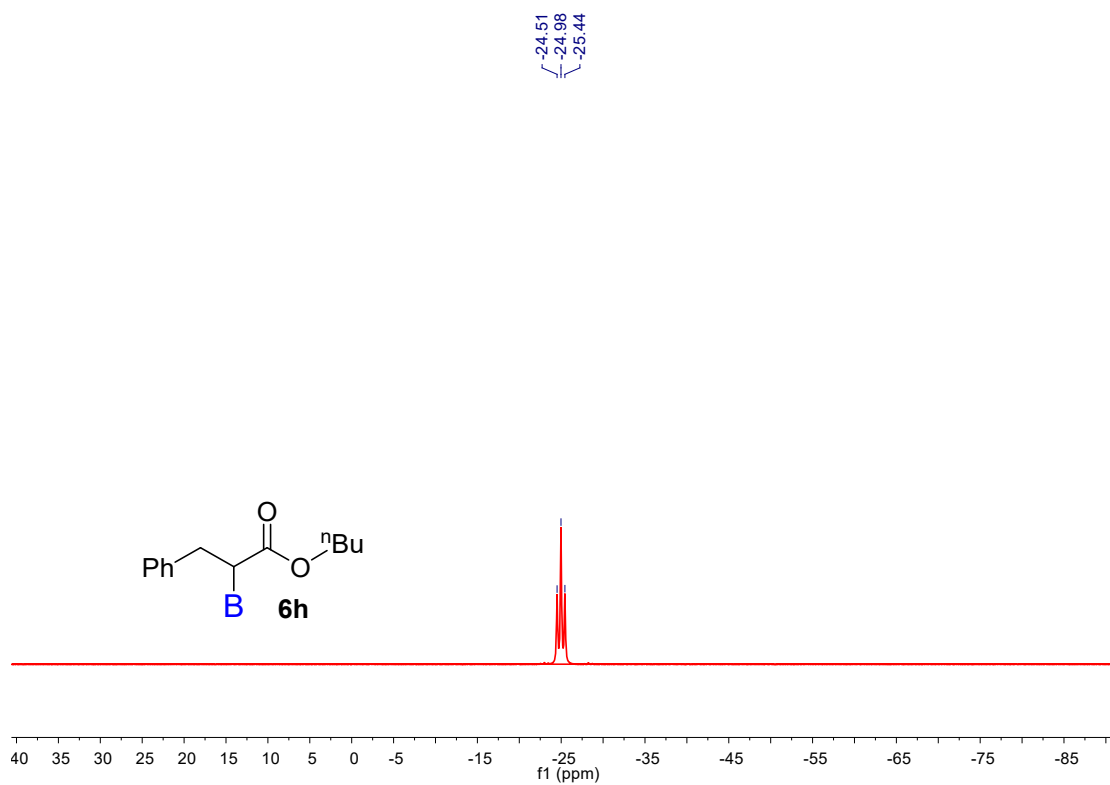
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



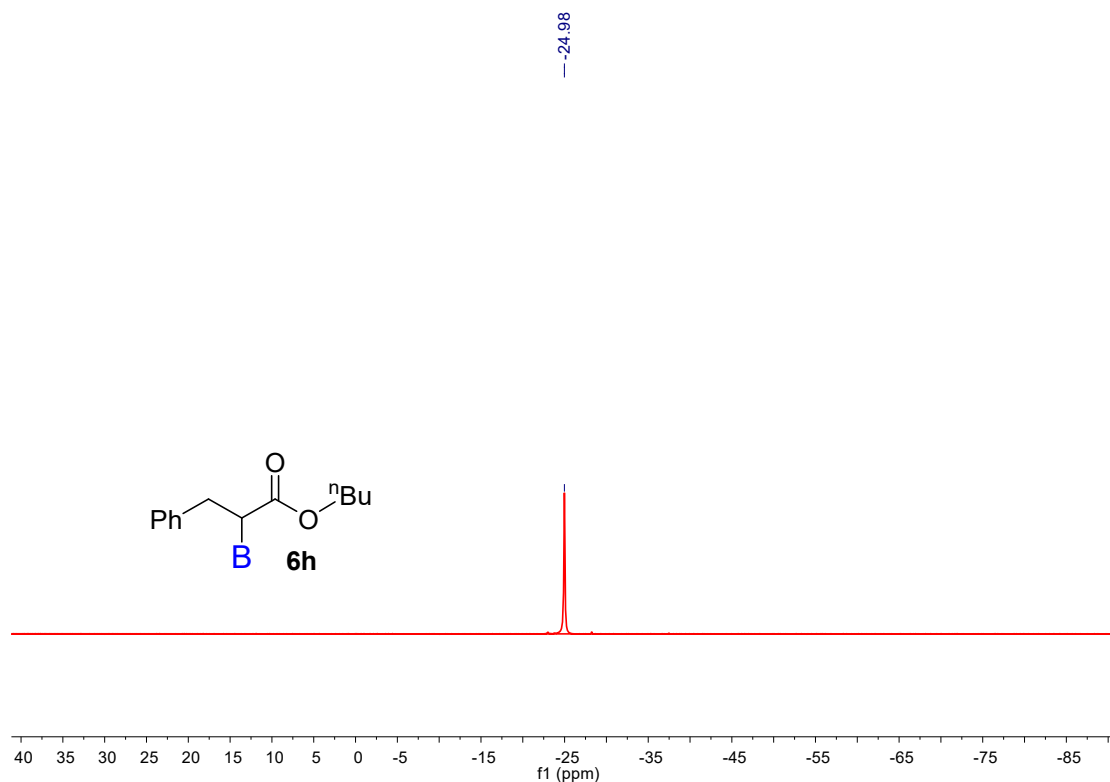
— 181.75
 — 144.83
 — 128.49
 — 127.83
 — 124.98
 — 120.38
 — 62.67
 — 39.16
 — 36.01
 — 30.93
 — 19.14
 — 13.79



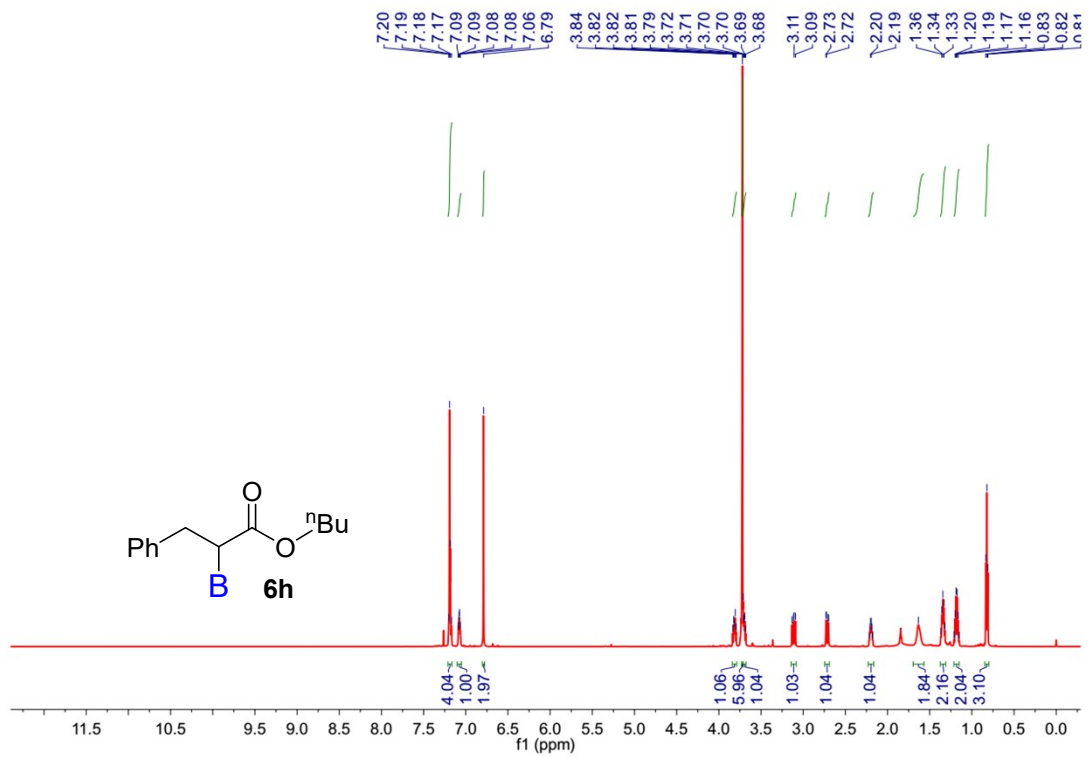
— 181.75
 — 144.83
 — 128.49
 — 127.83
 — 124.98
 — 120.38
 — 62.67
 — 39.16
 — 36.01
 — 30.93
 — 19.14
 — 13.79



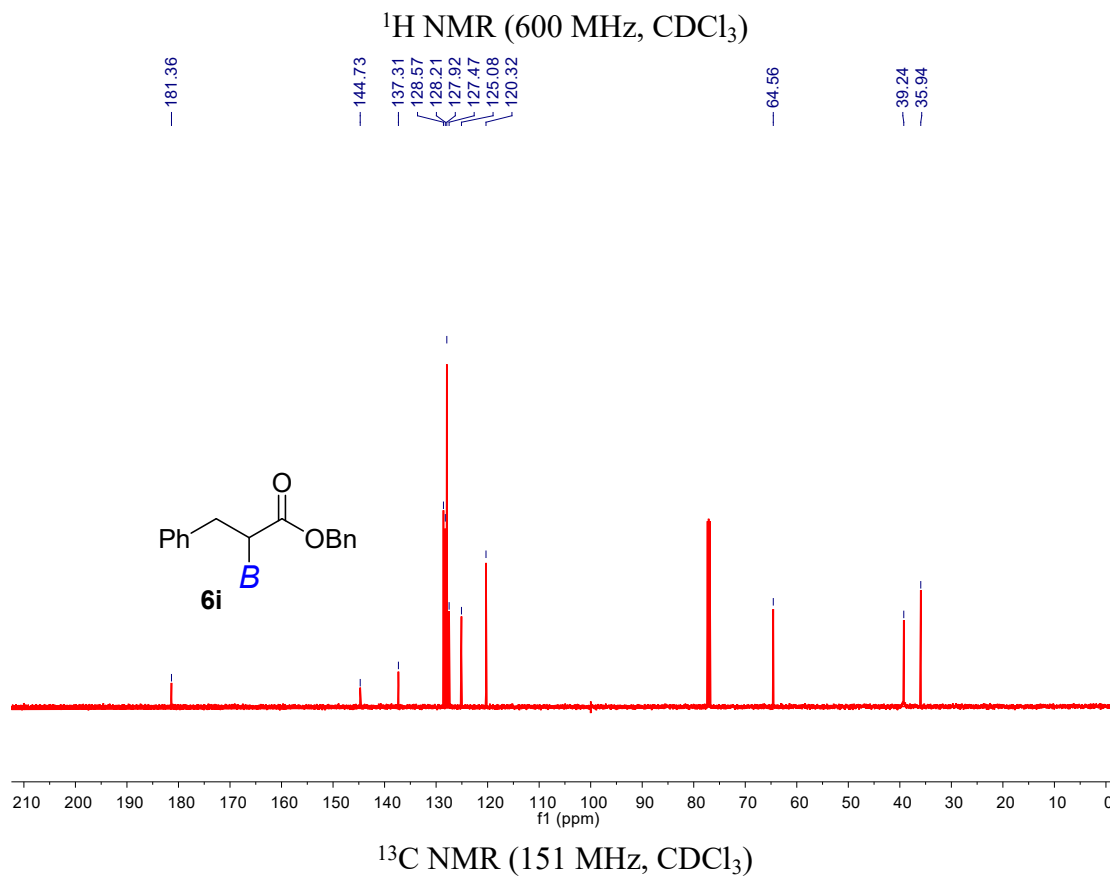
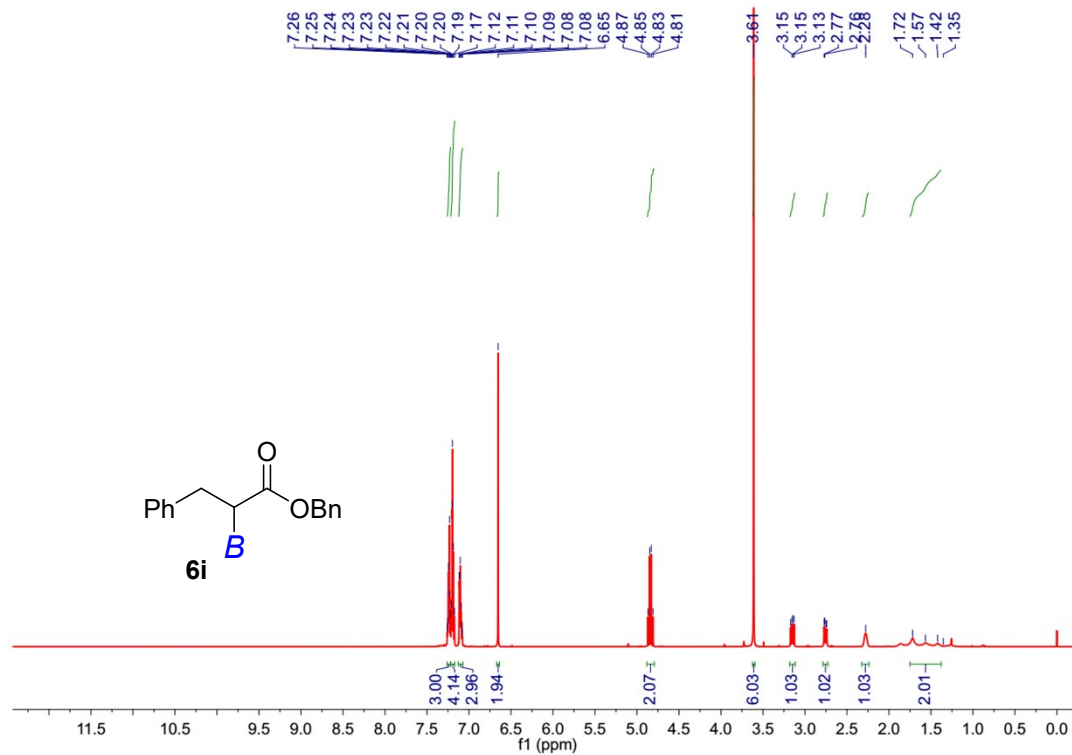
^{11}B NMR (193 MHz, CDCl_3)

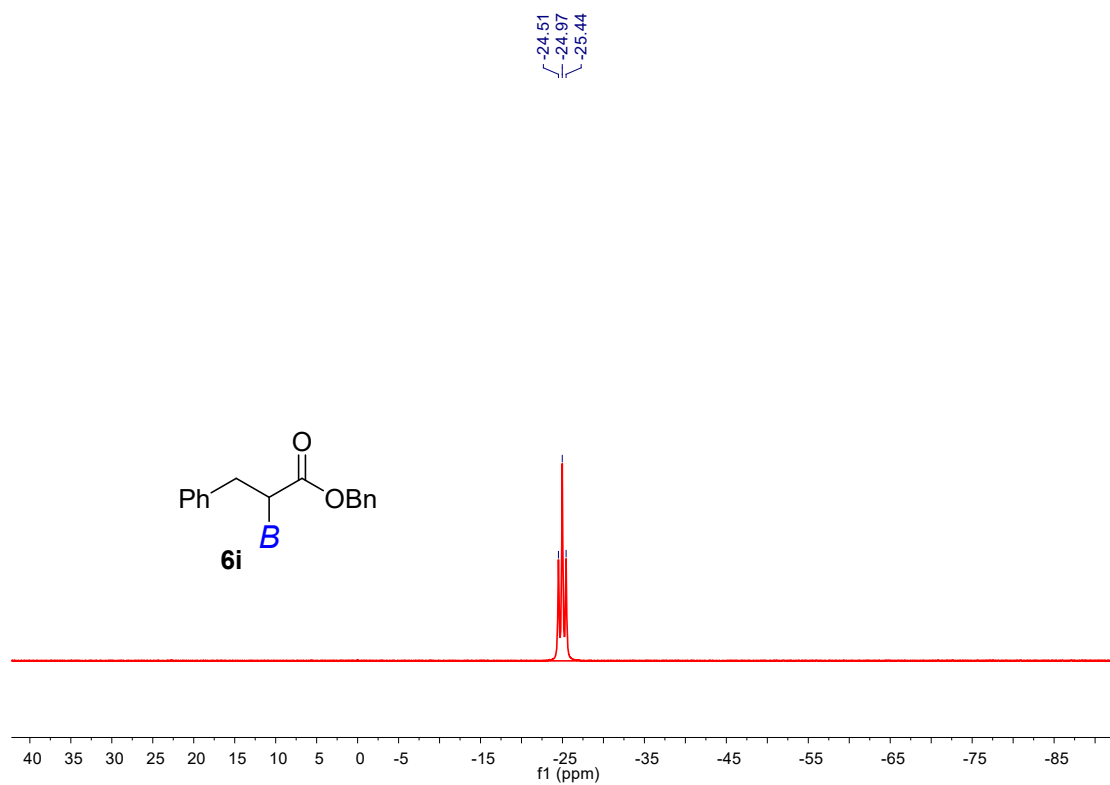


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

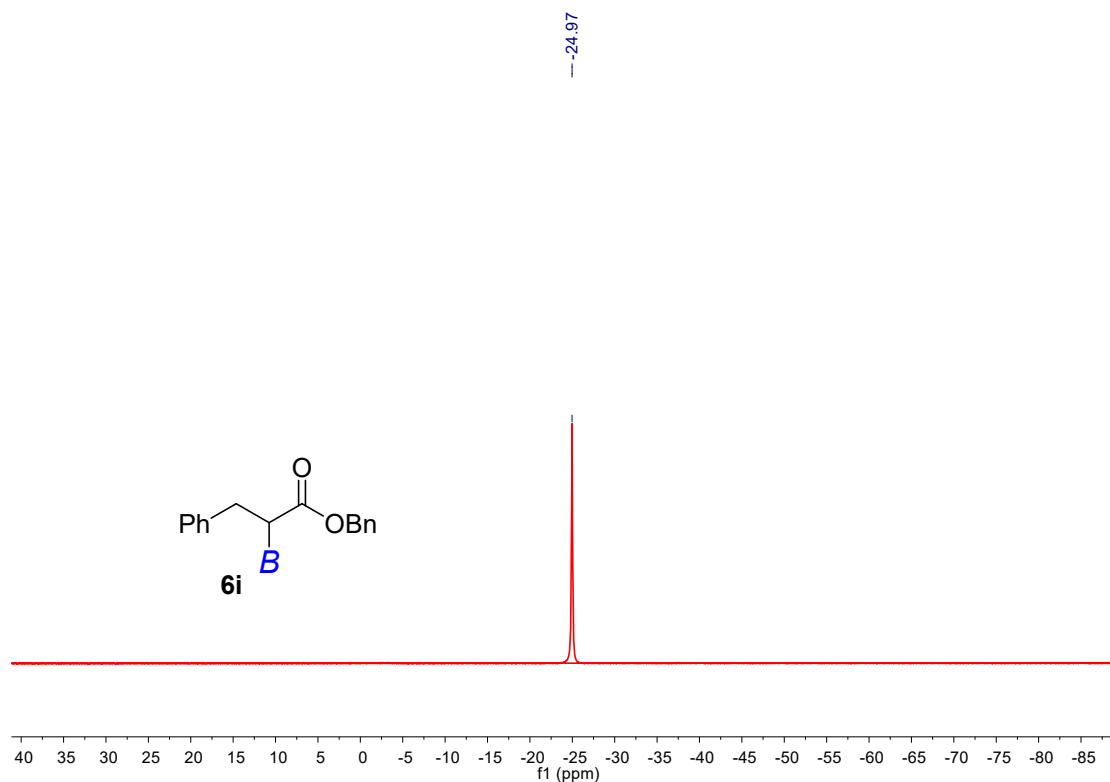


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

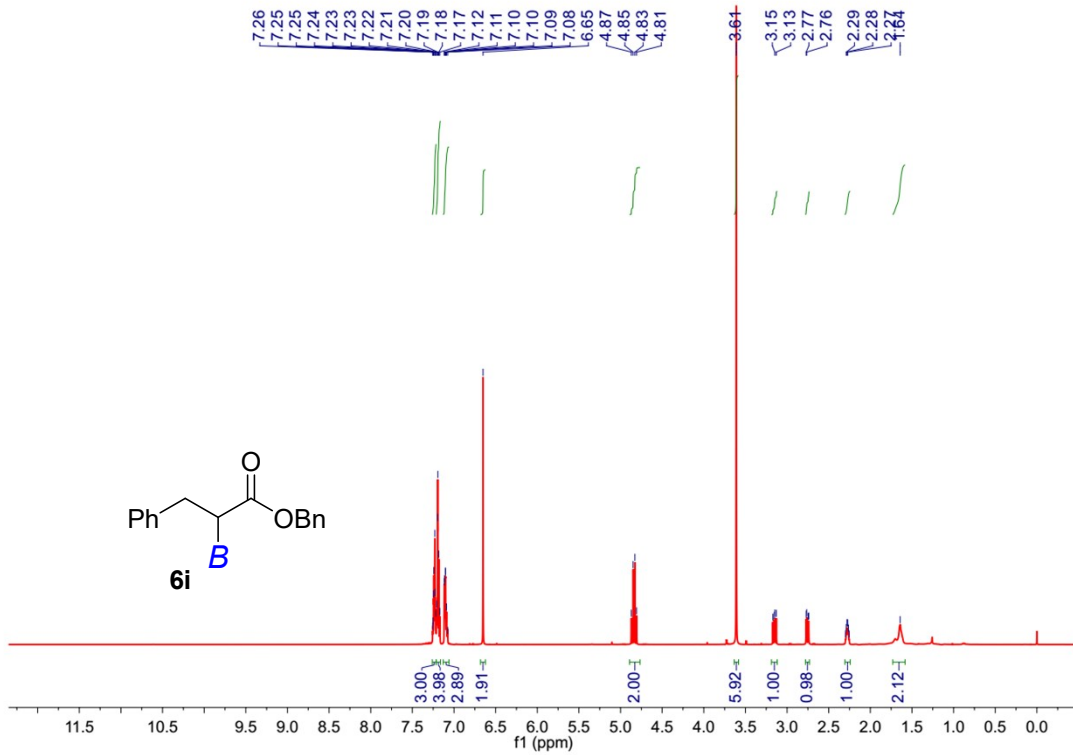




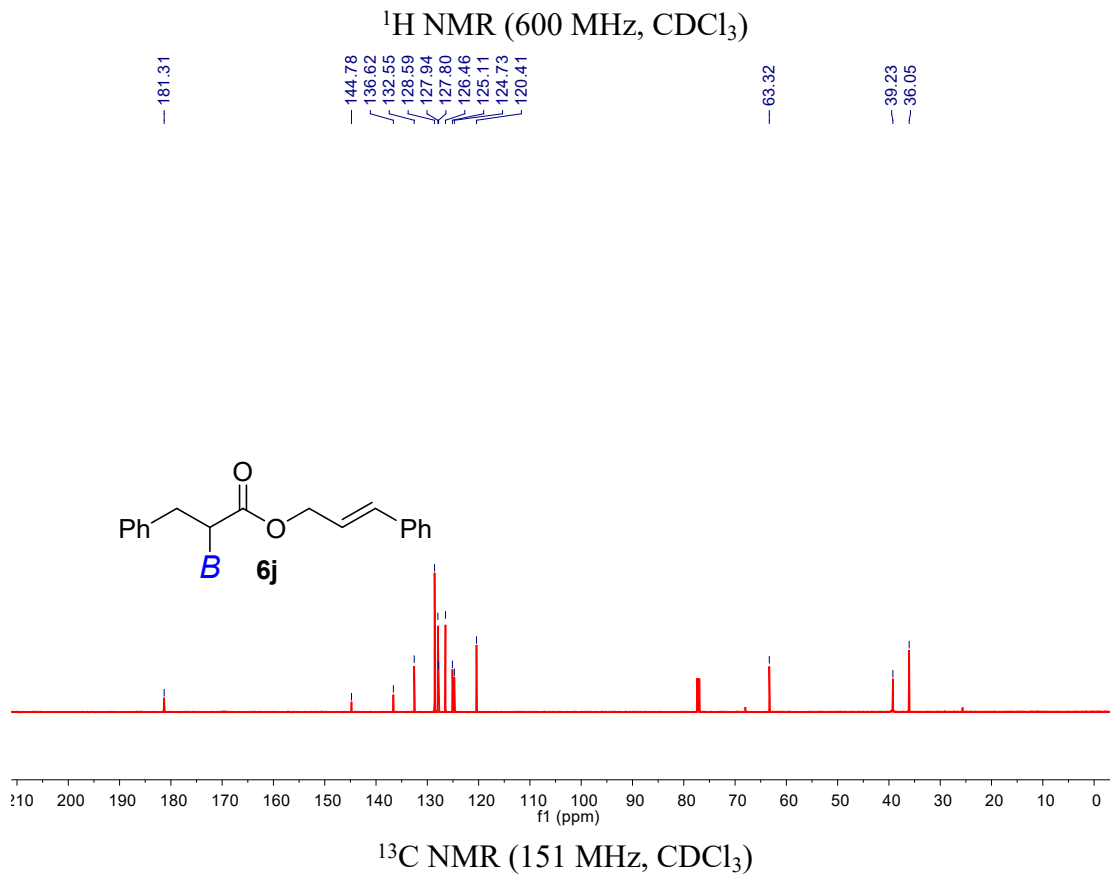
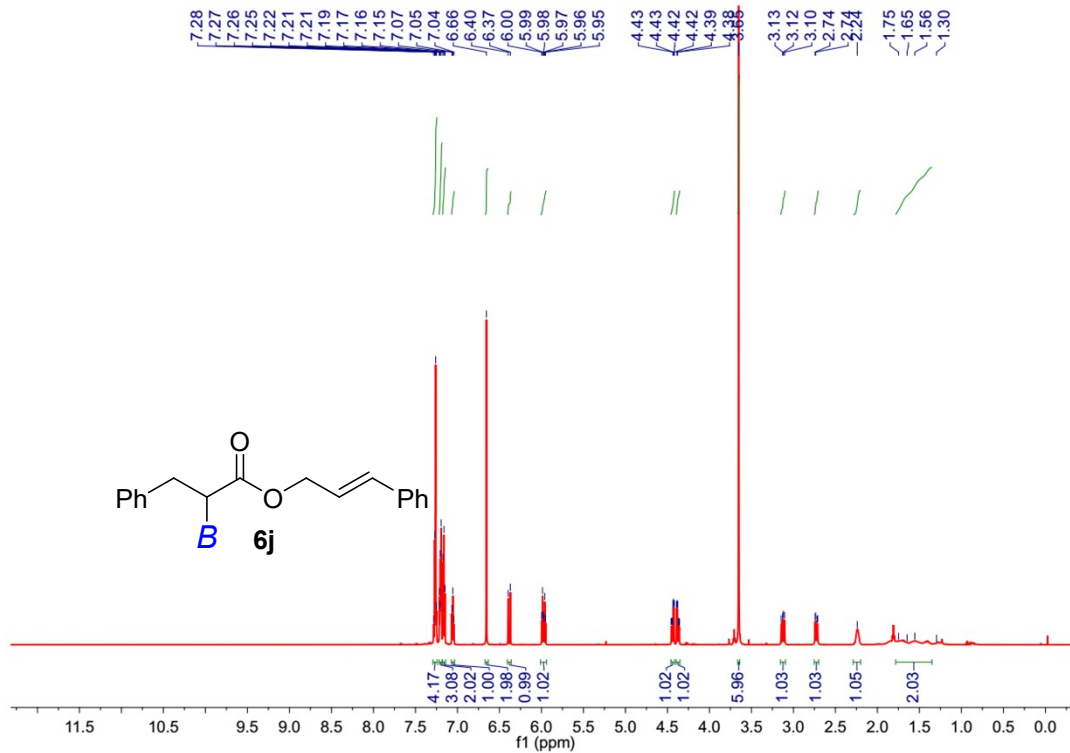
^{11}B NMR (193 MHz, CDCl_3)

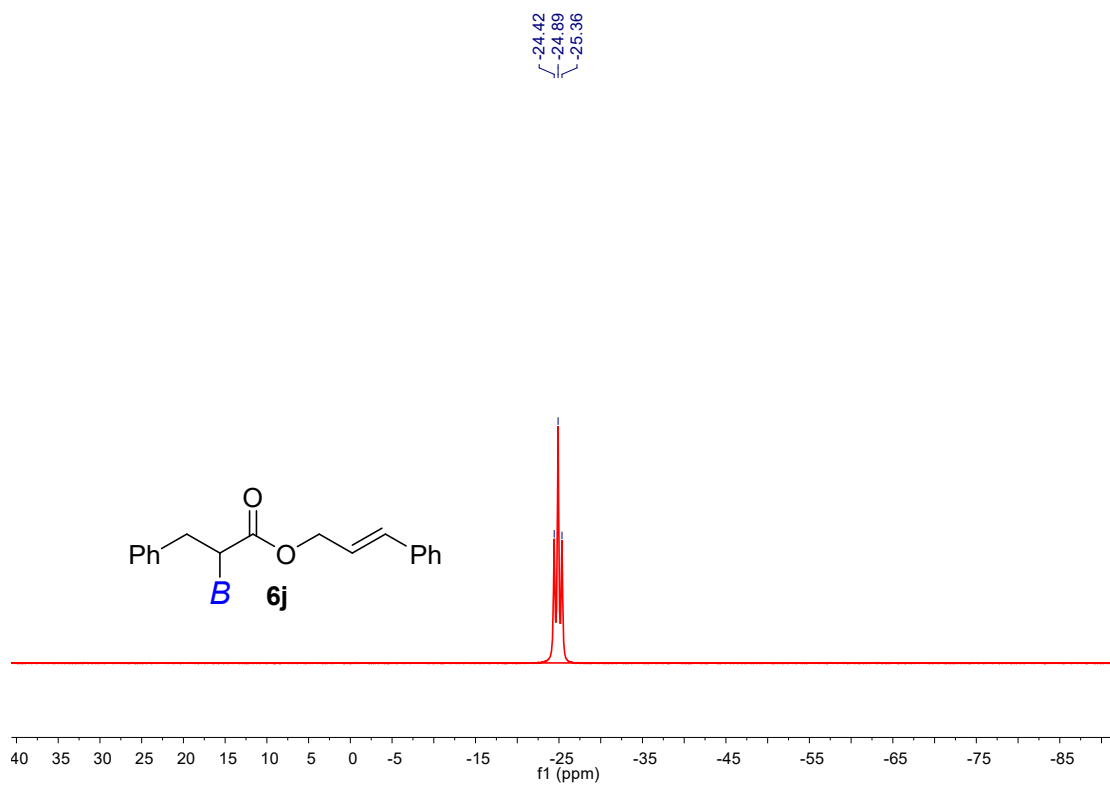


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

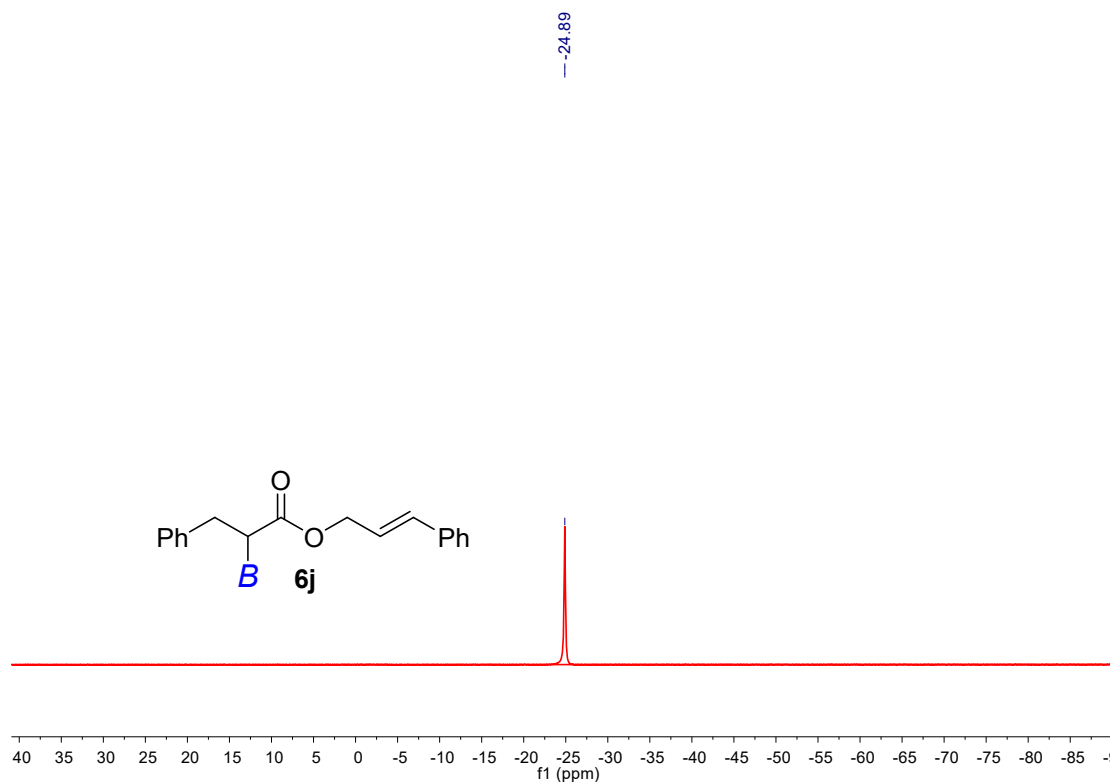


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

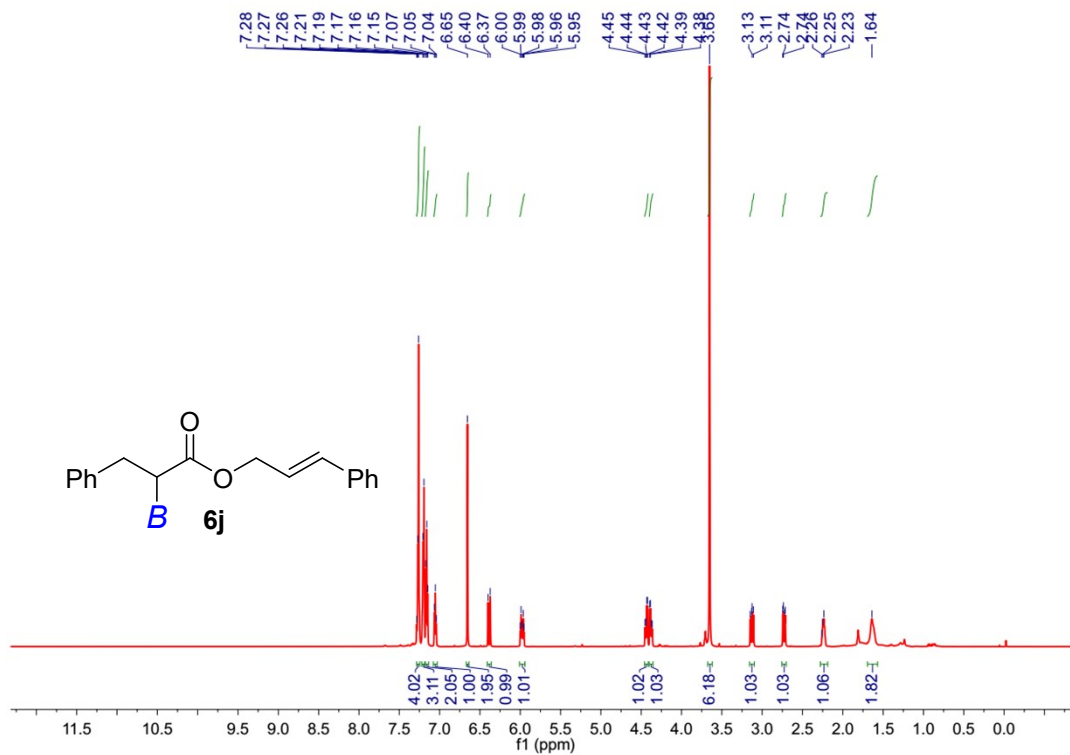




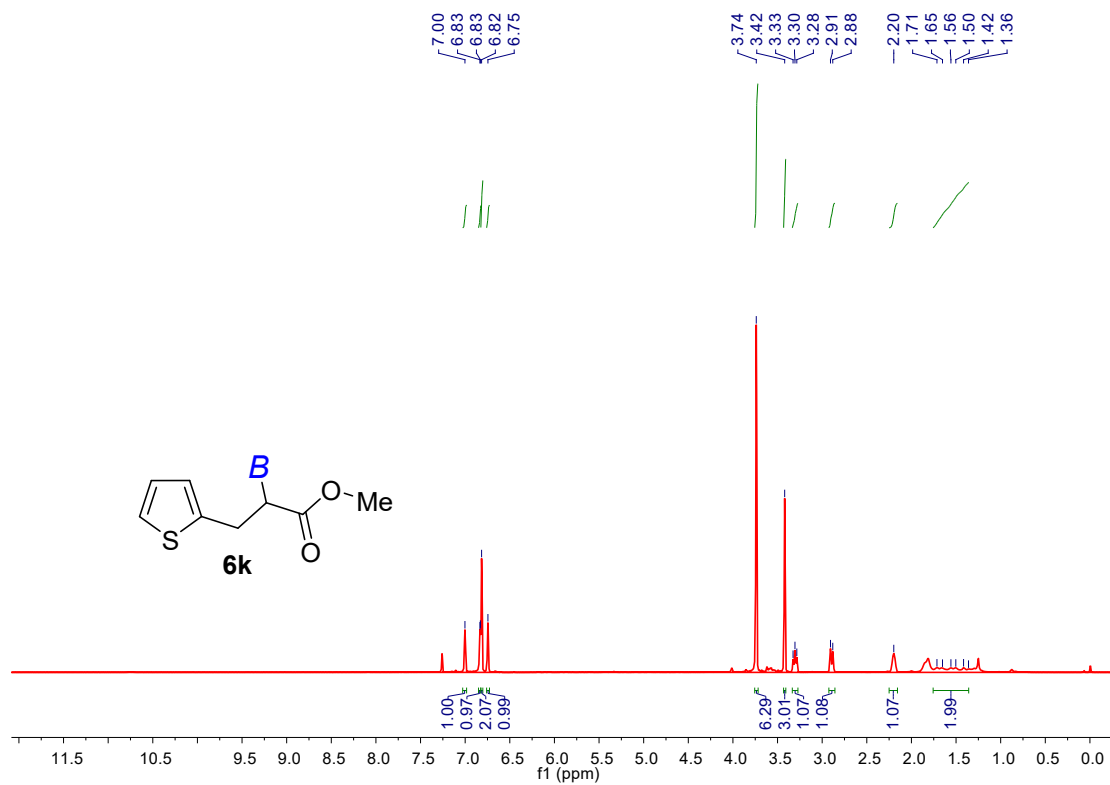
^{11}B NMR (193 MHz, CDCl_3)



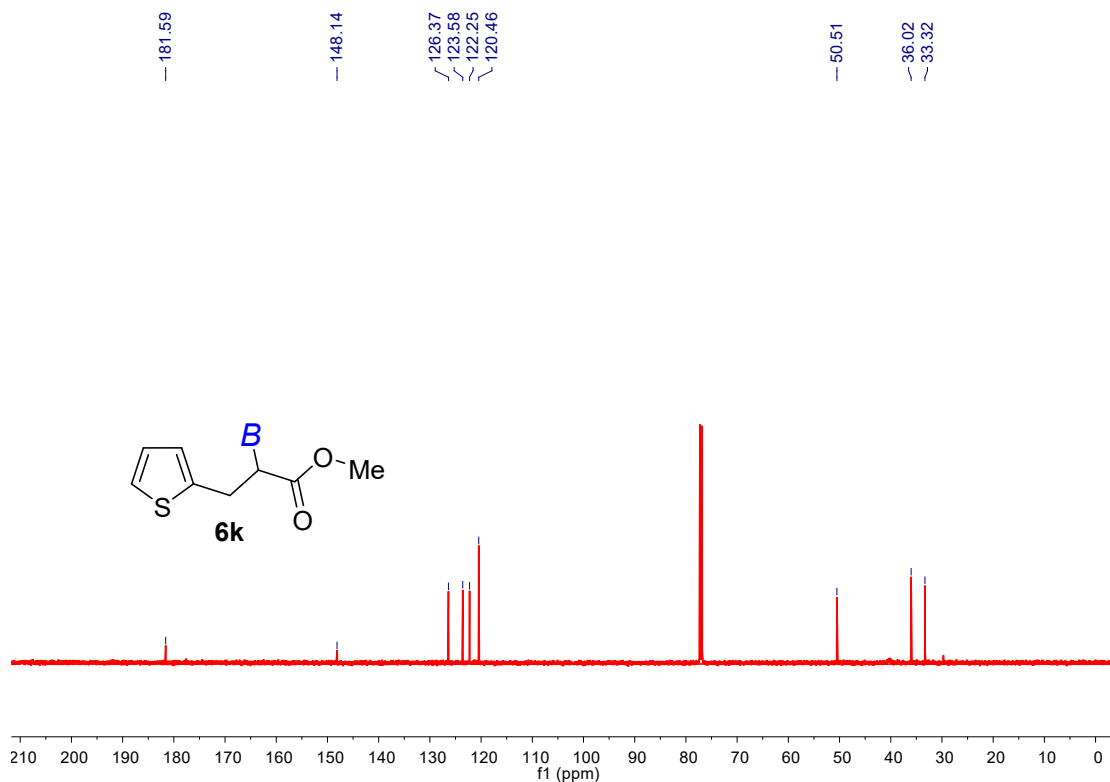
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



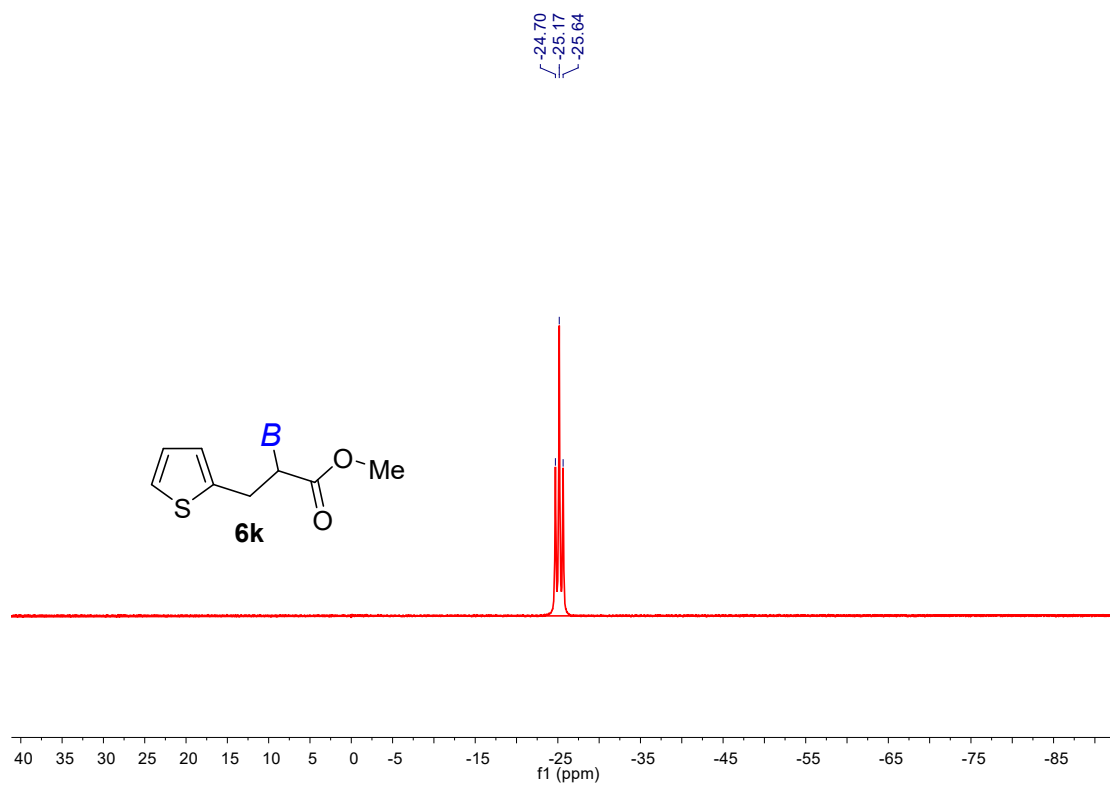
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



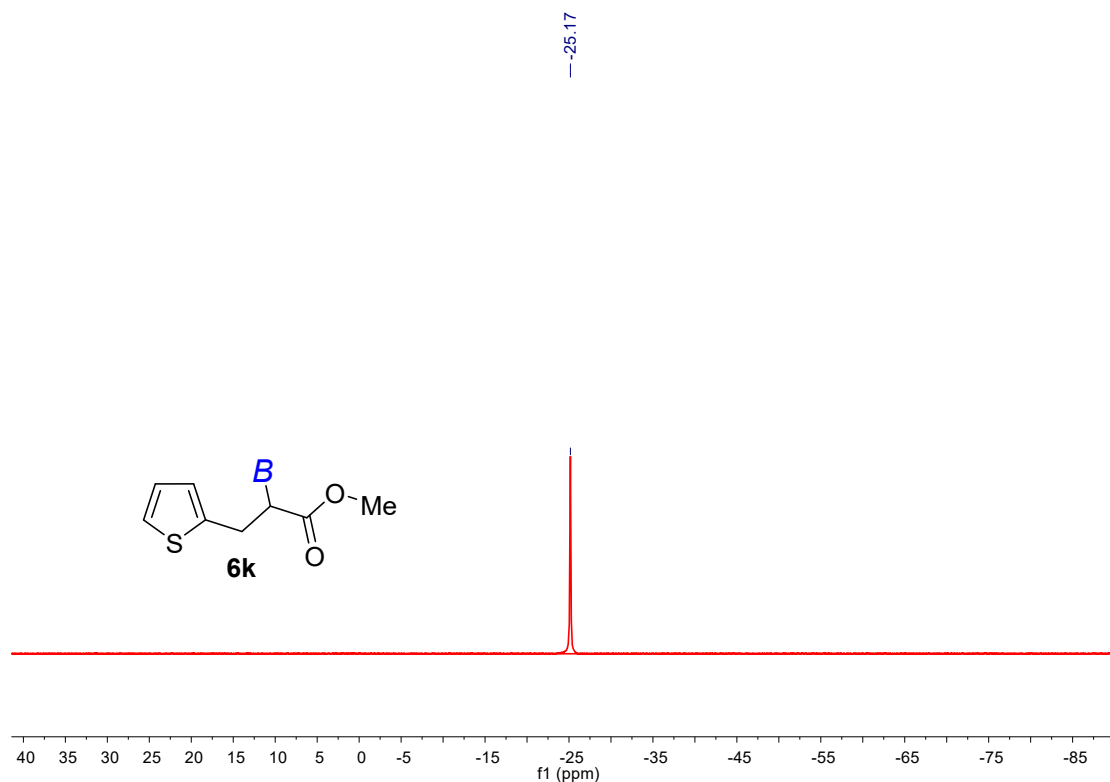
¹H NMR (600 MHz, CDCl₃)



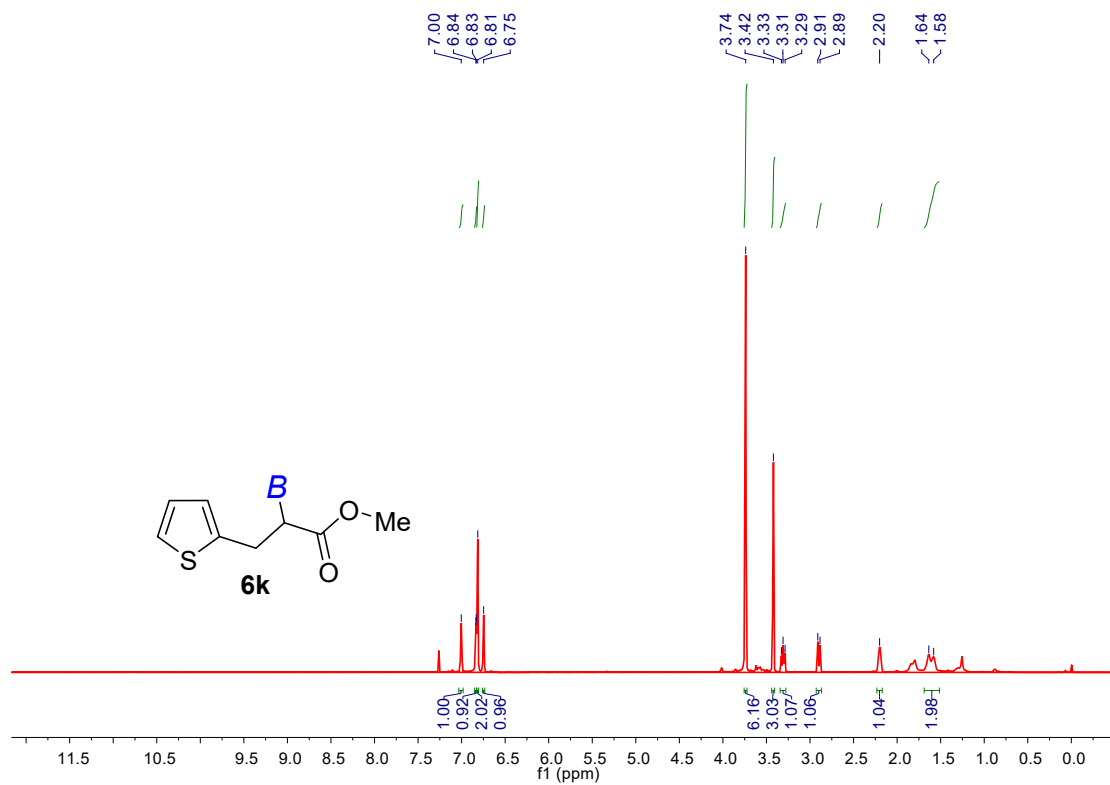
¹³C NMR (151 MHz, CDCl₃)



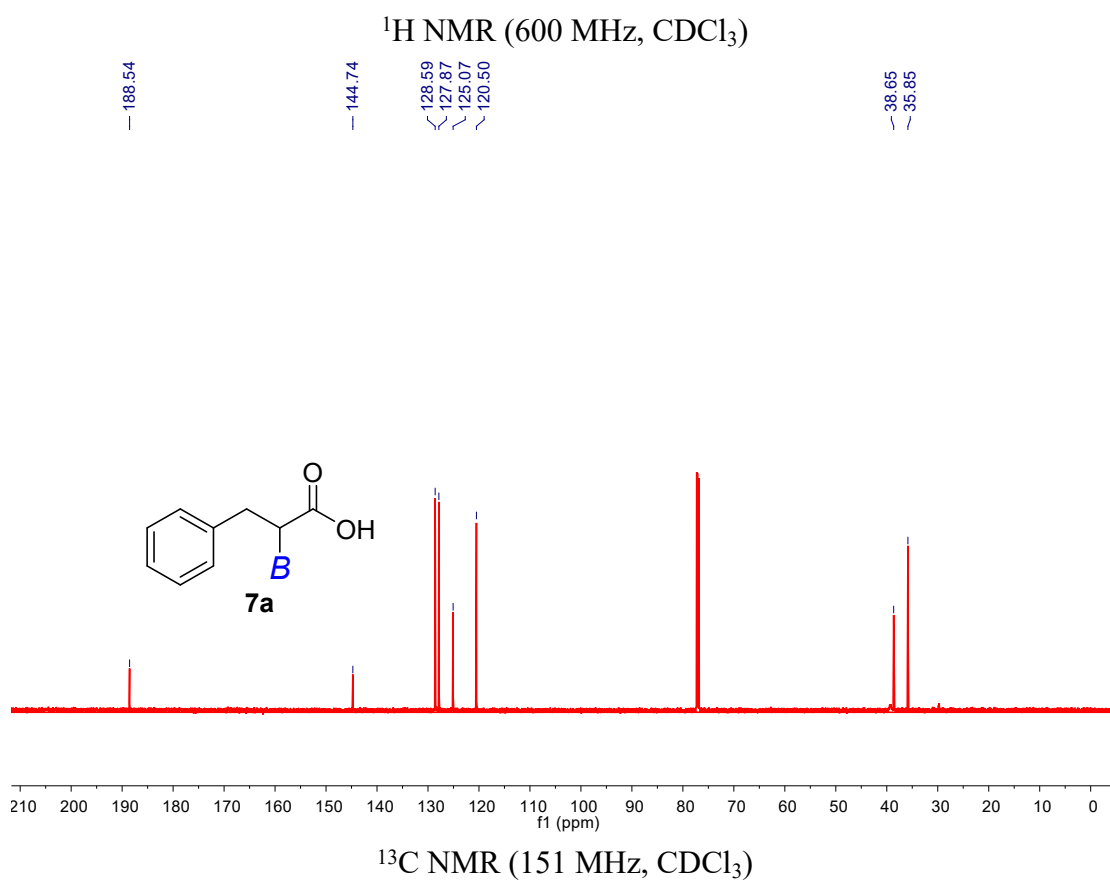
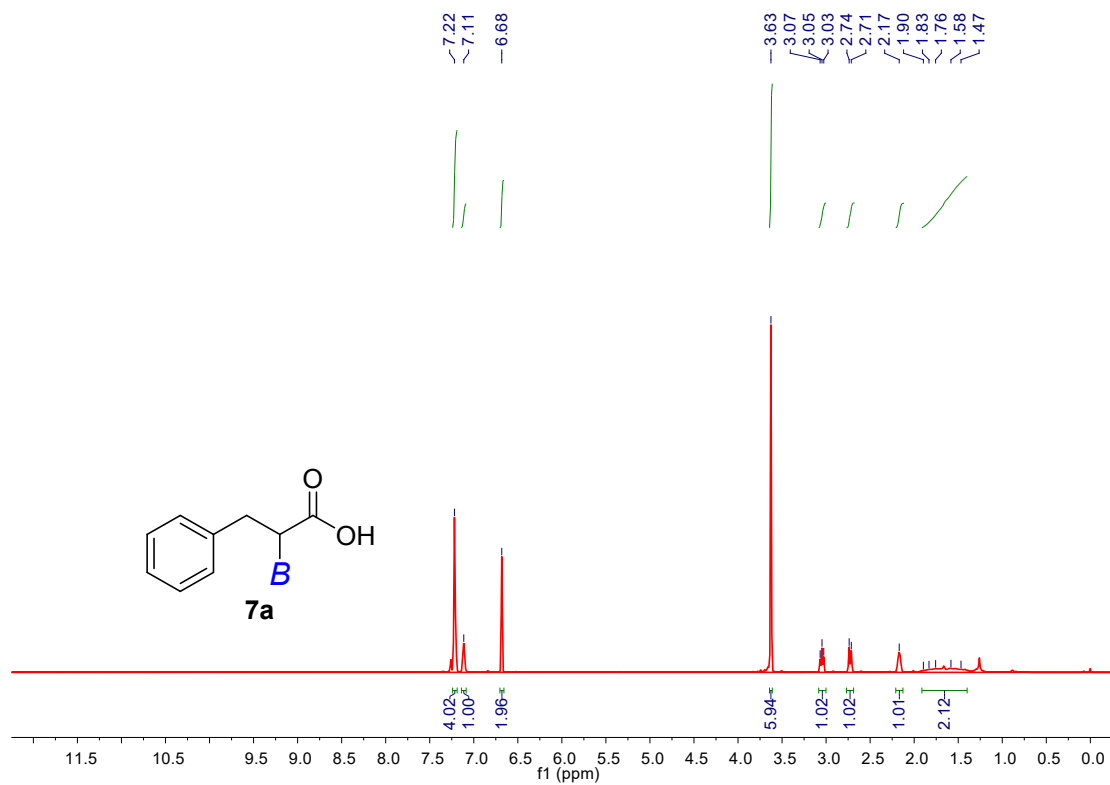
^{11}B NMR (193 MHz, CDCl_3)

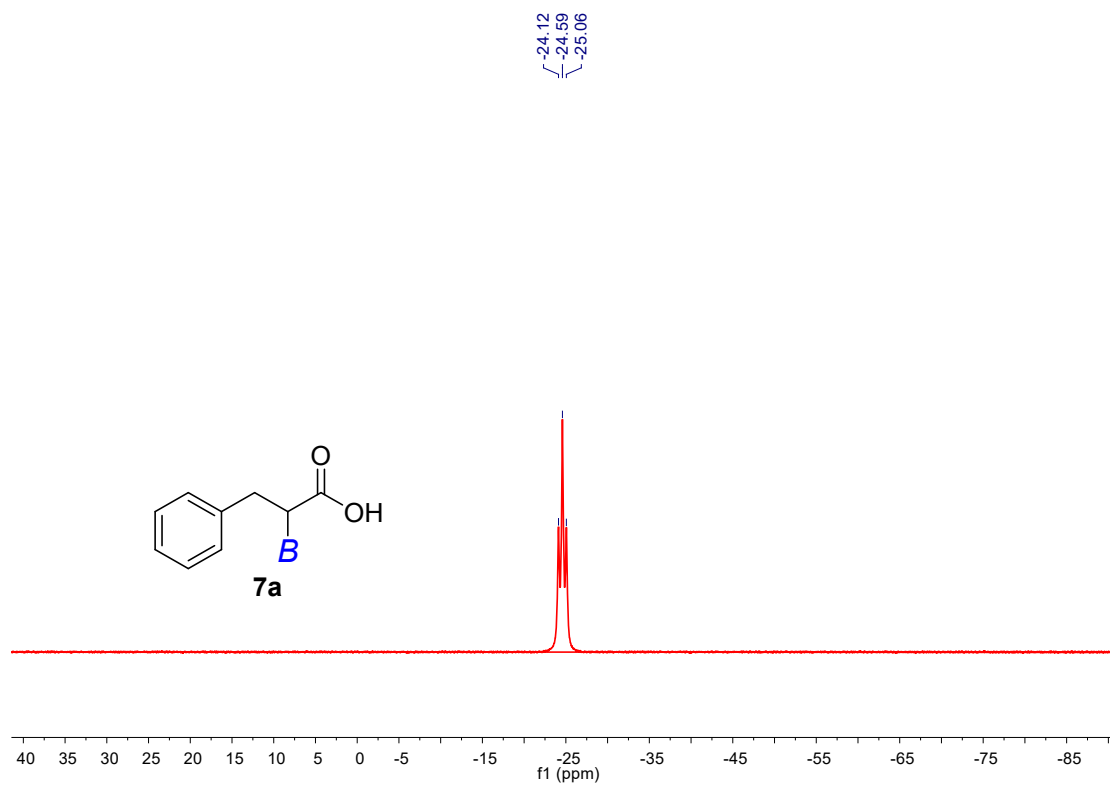


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

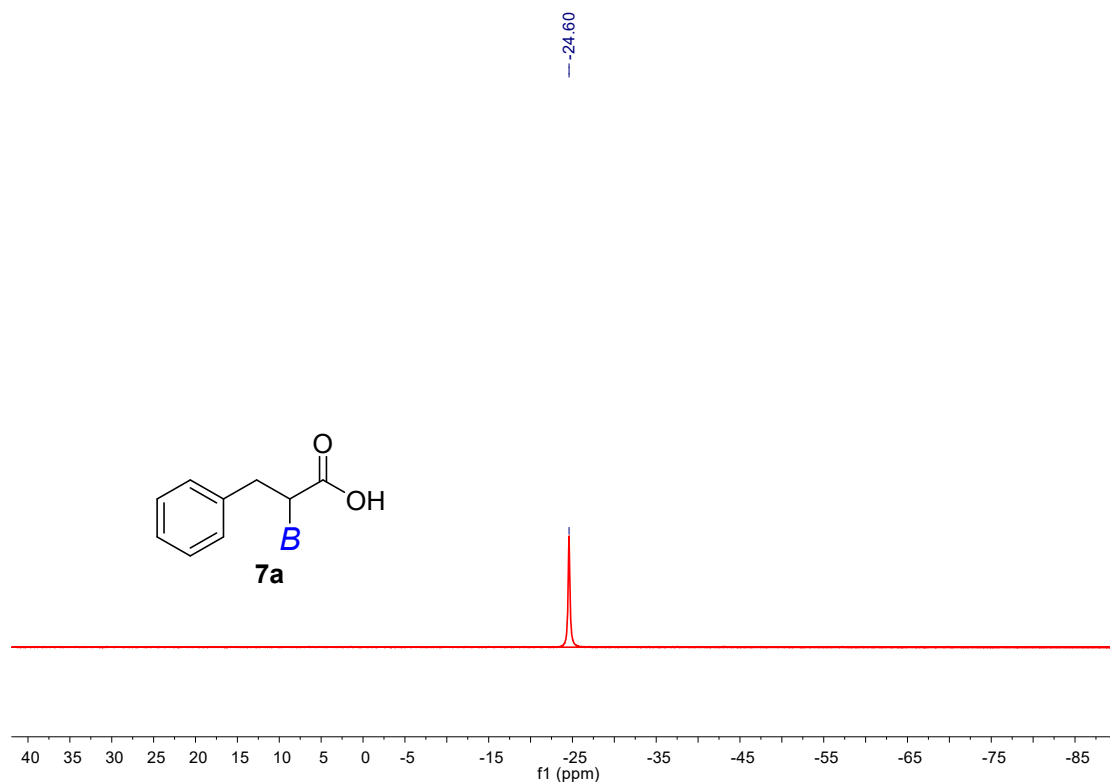


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

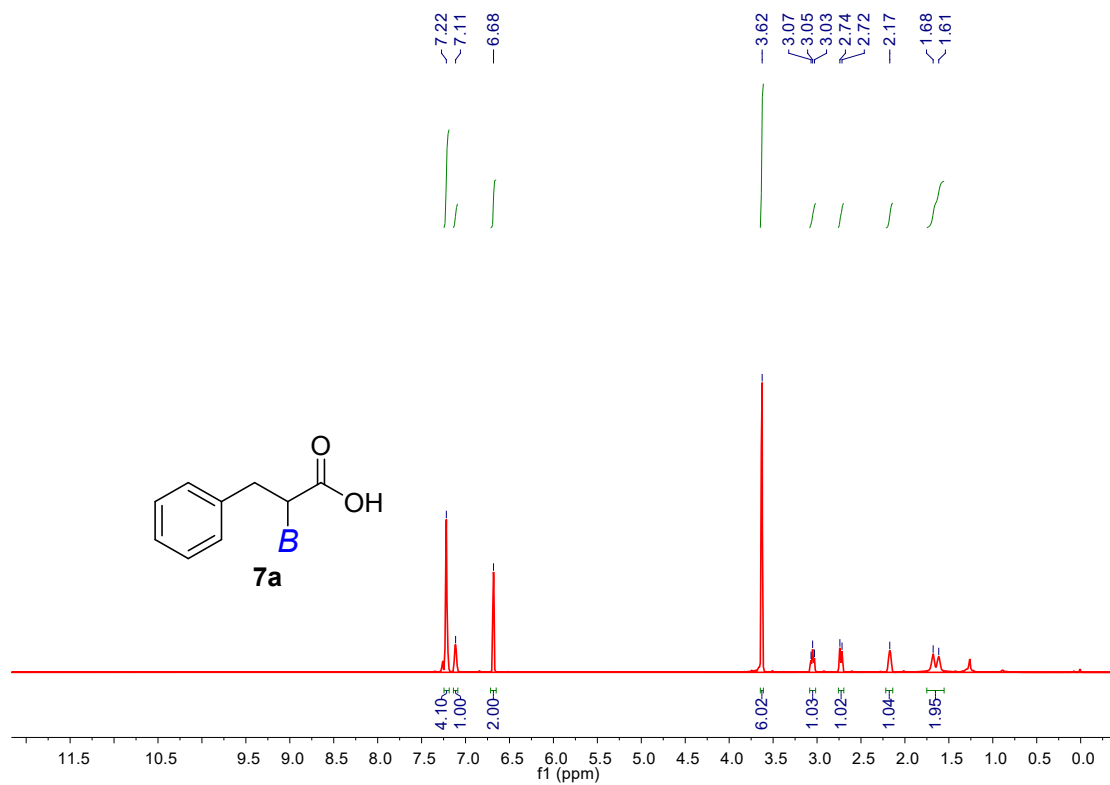




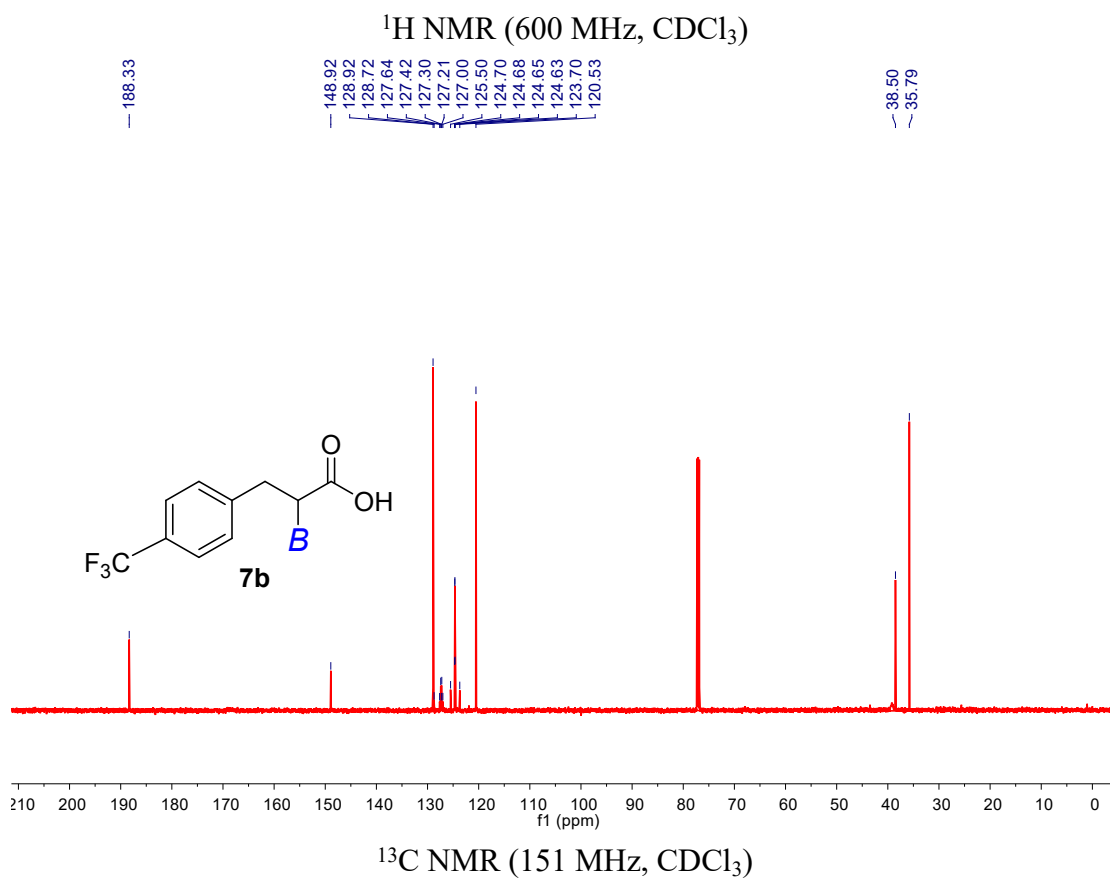
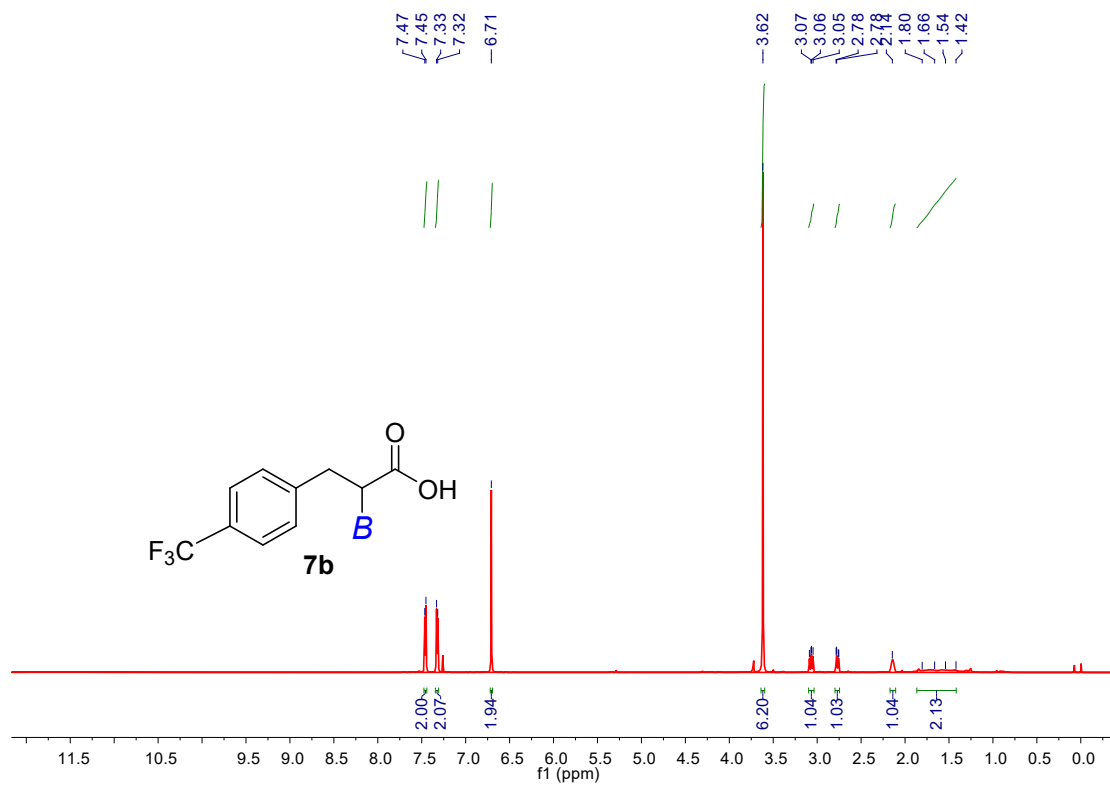
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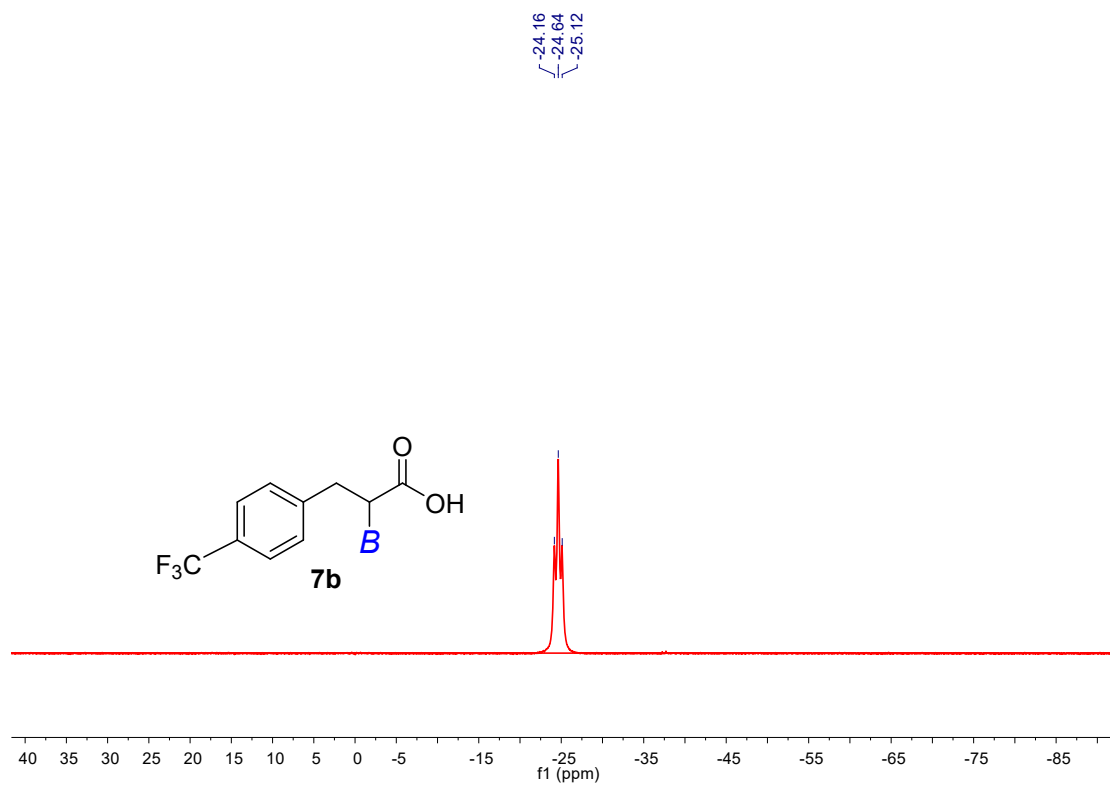


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

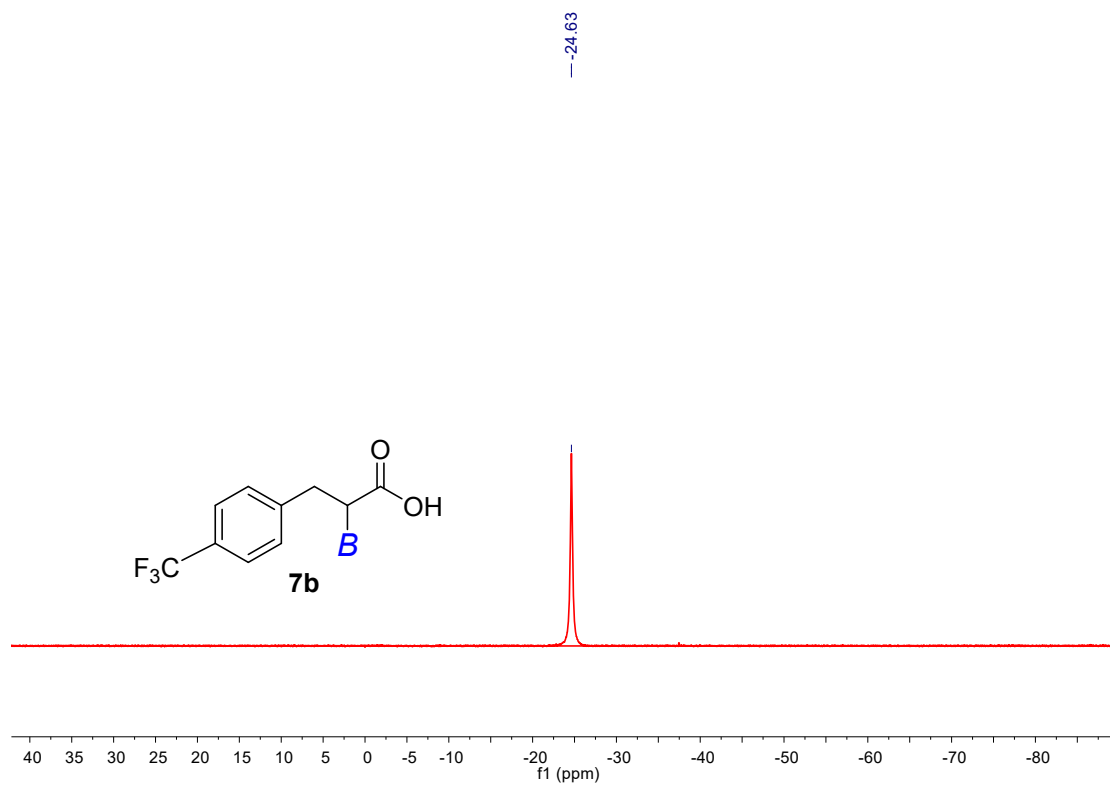


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

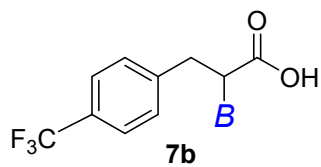
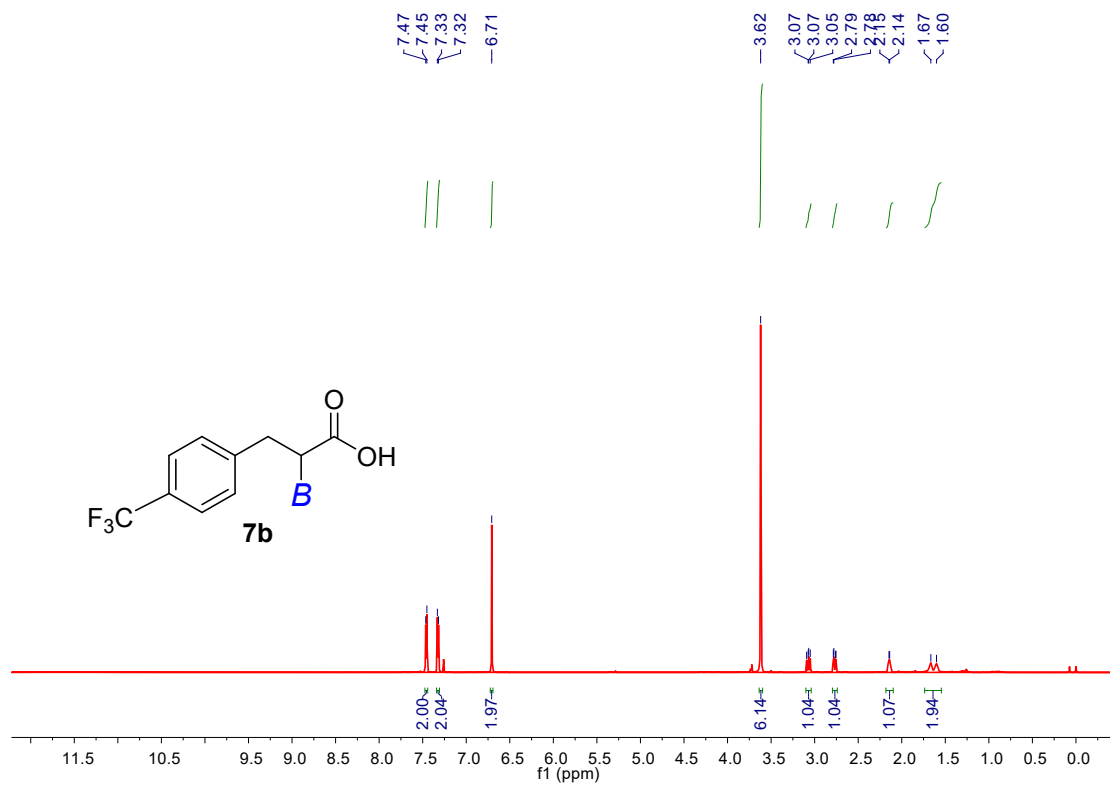




^{11}B NMR (193 MHz, CDCl_3)

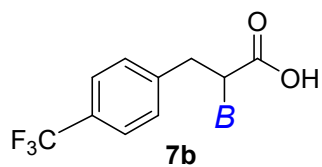
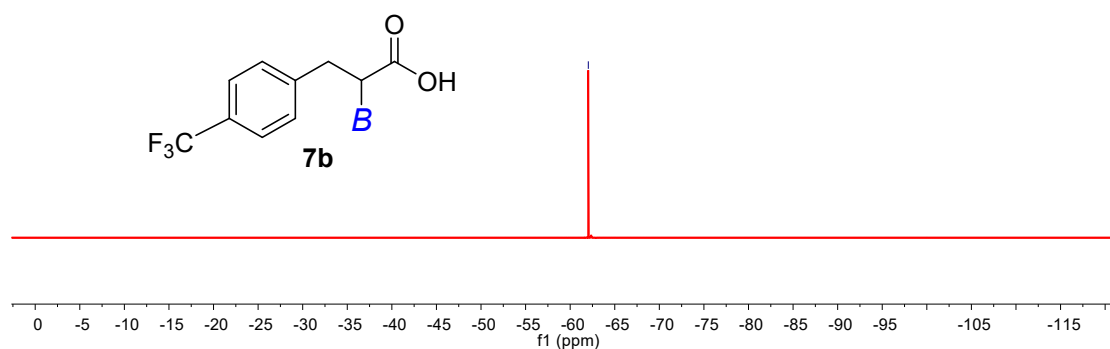


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

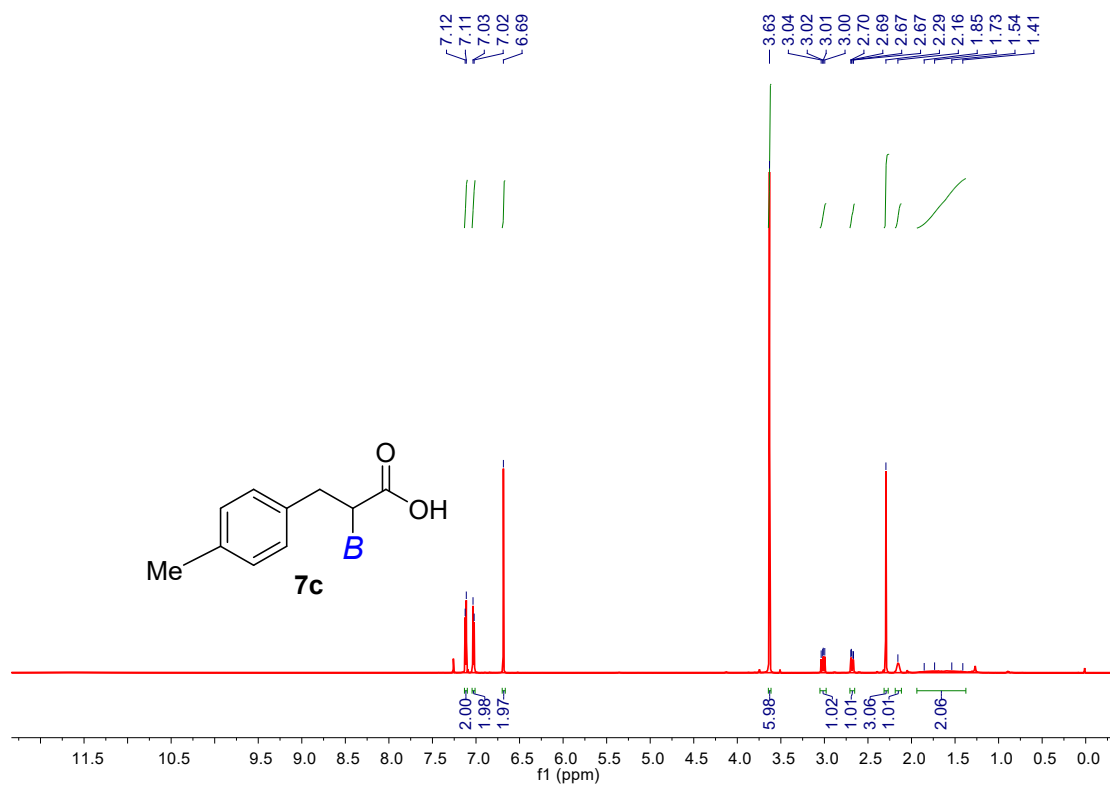


¹H{¹¹B} NMR (600 MHz, CDCl₃)

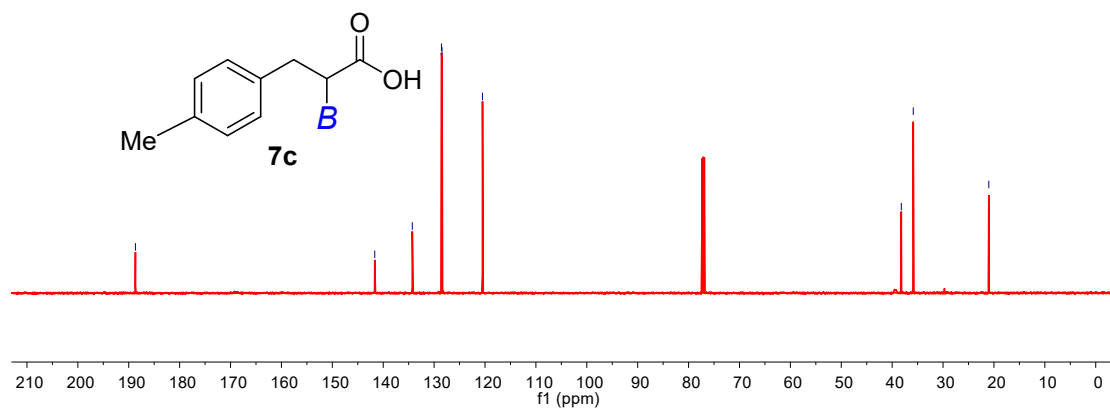
-62.03



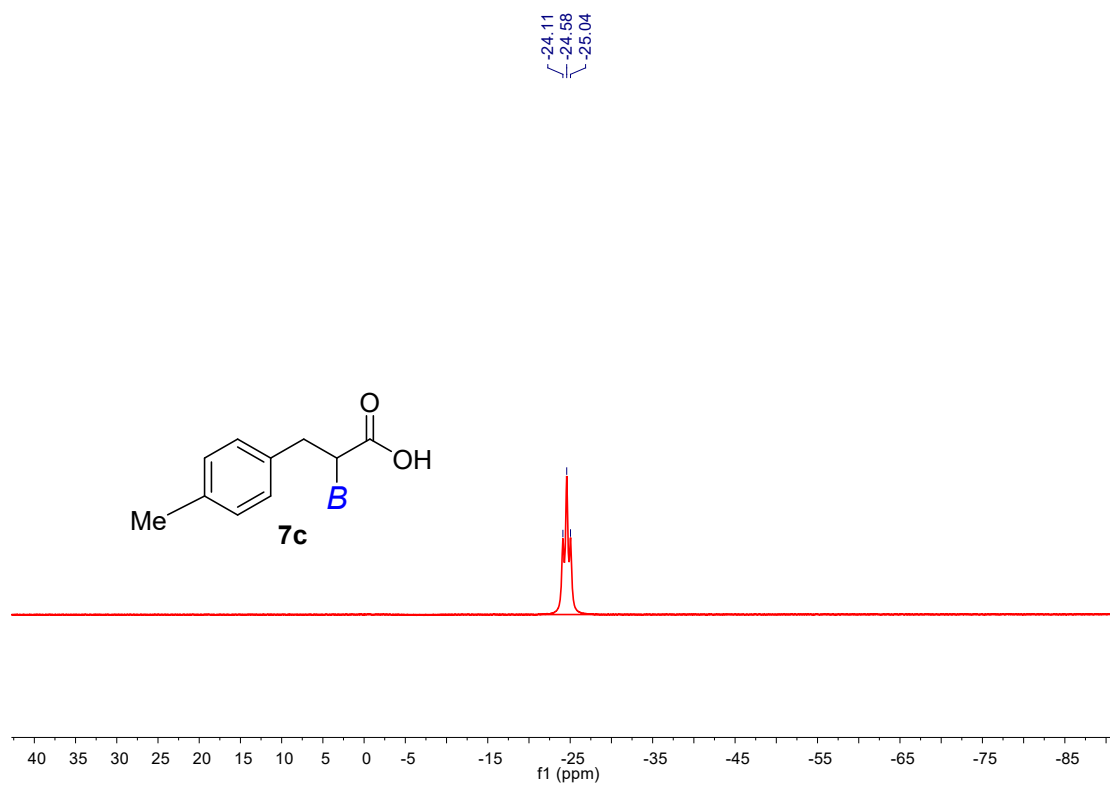
¹⁹F NMR (565 MHz, CDCl₃)



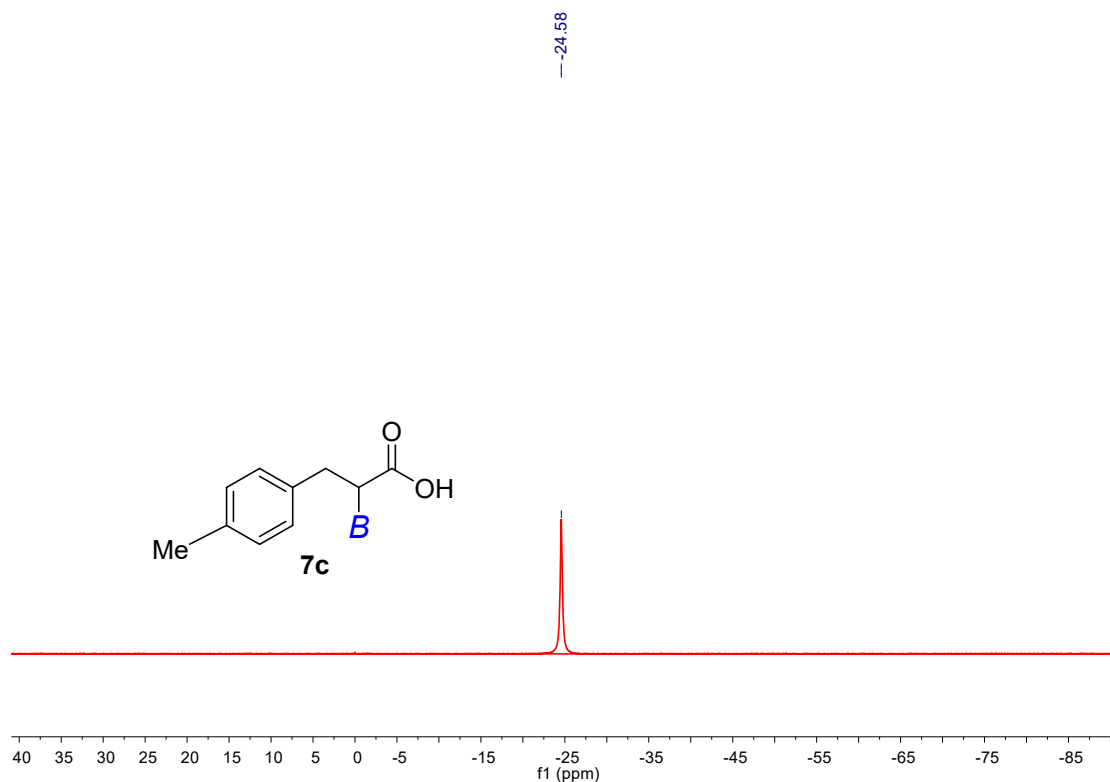
— 188.69
 ~ 141.66
 ~ 134.28
 ~ 128.56
 ~ 128.44
 — 120.49
 ~ 38.20
 ~ 35.85
 — 21.01



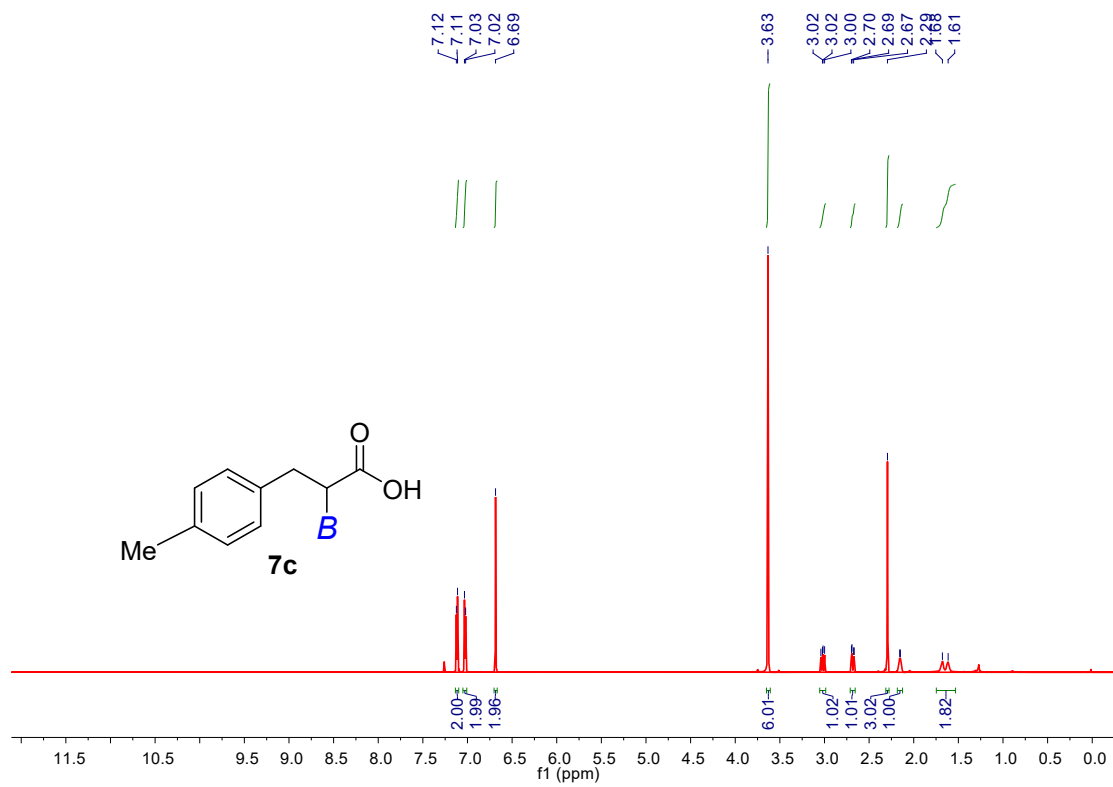
¹³C NMR (151 MHz, CDCl₃)



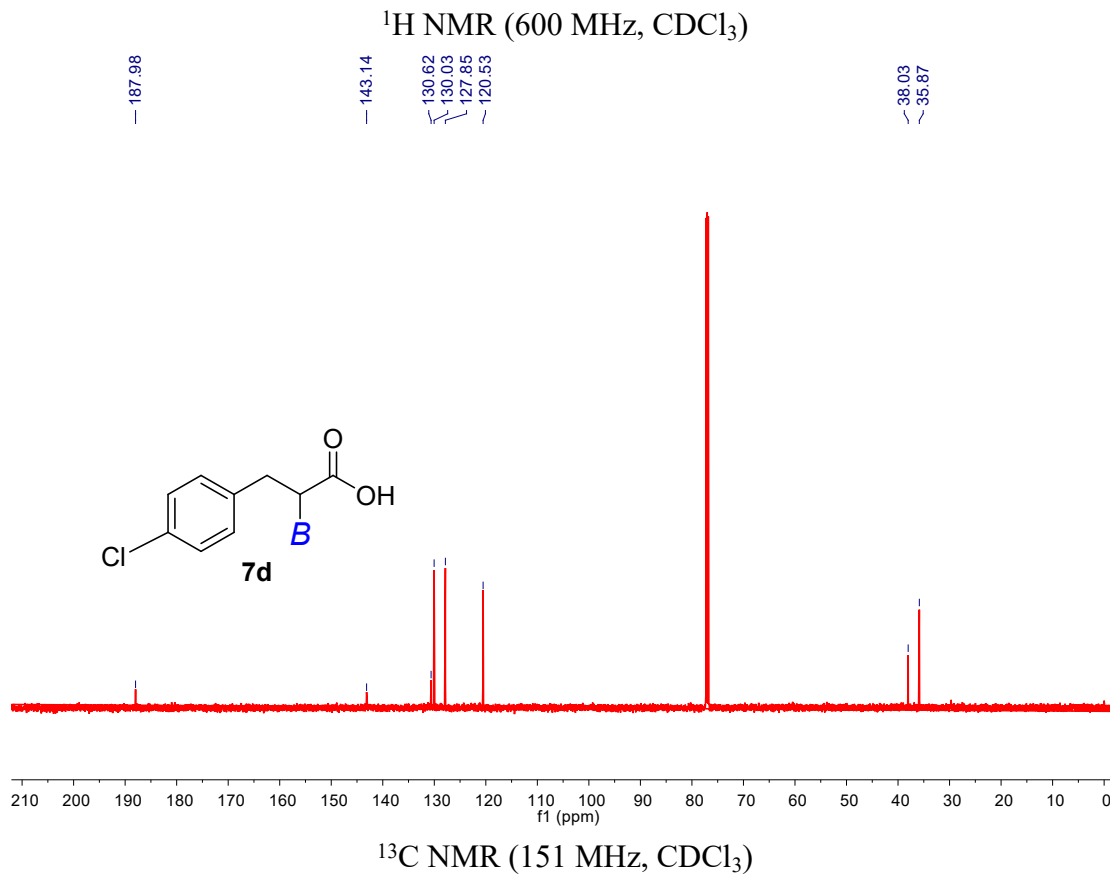
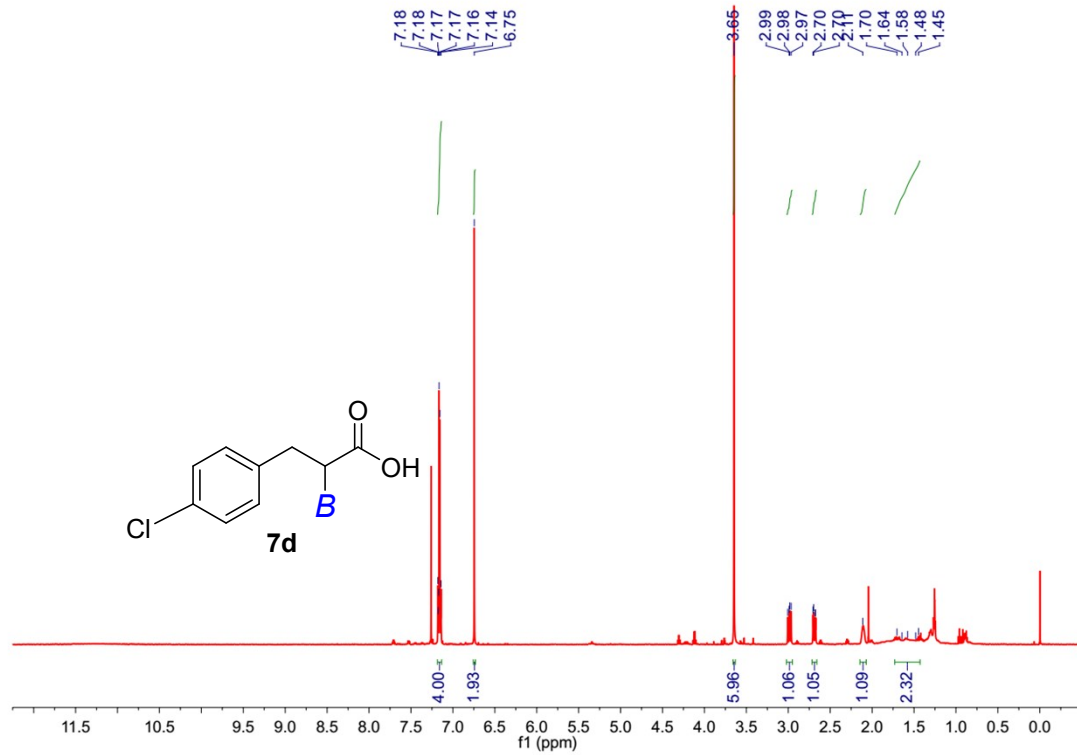
¹¹B NMR (193 MHz, CDCl₃)

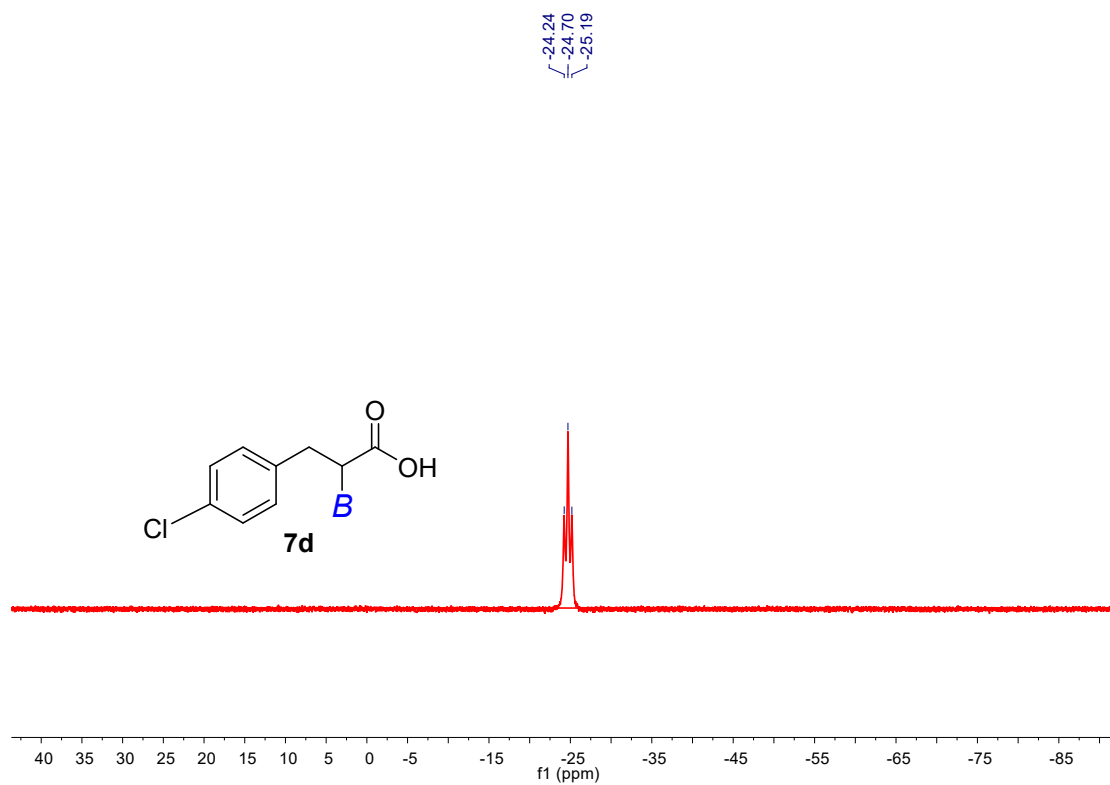


¹¹B{¹H} NMR (193 MHz, CDCl₃)

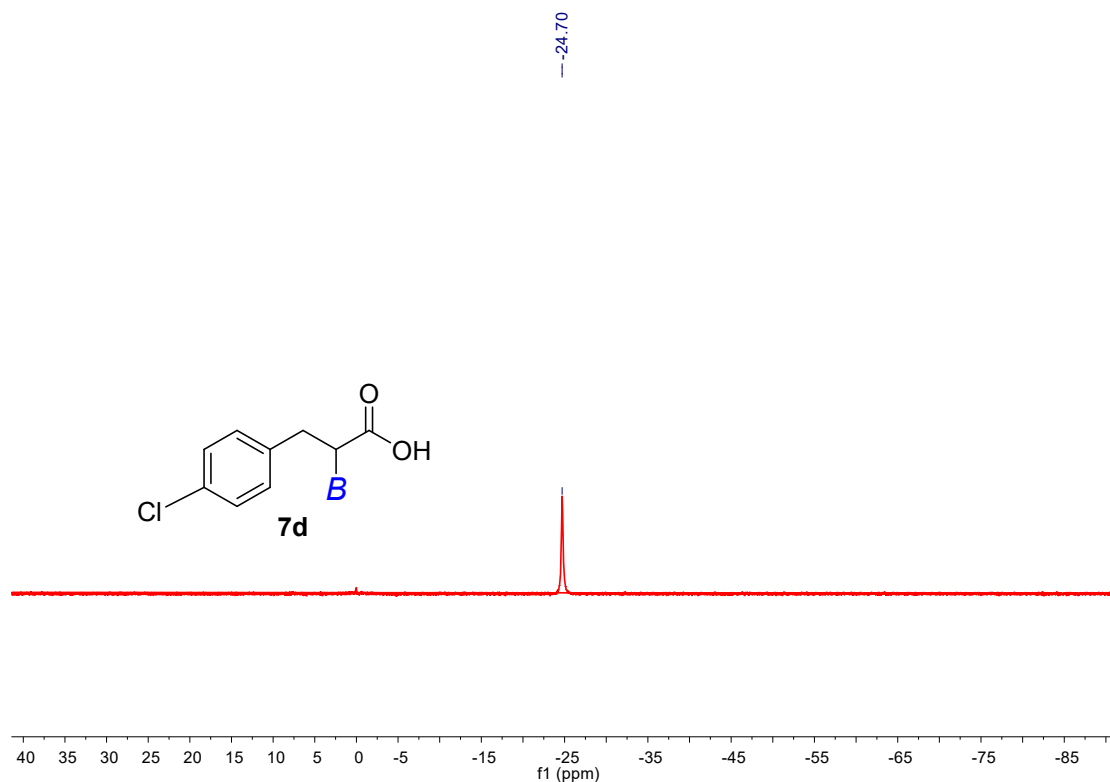


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

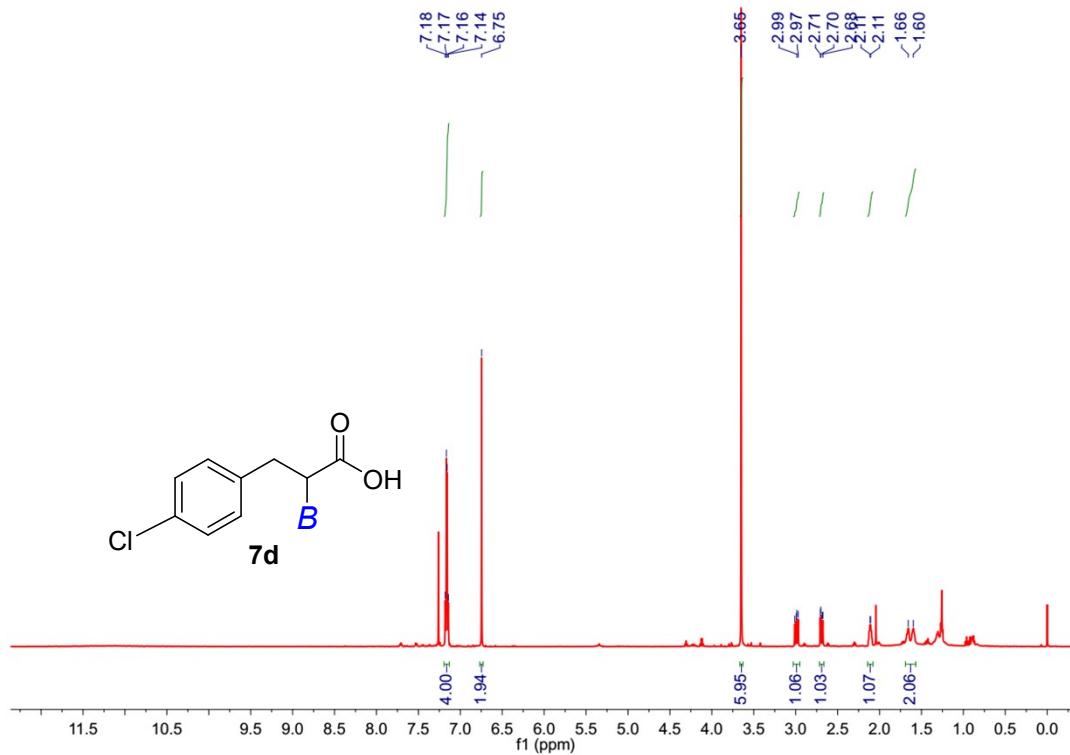




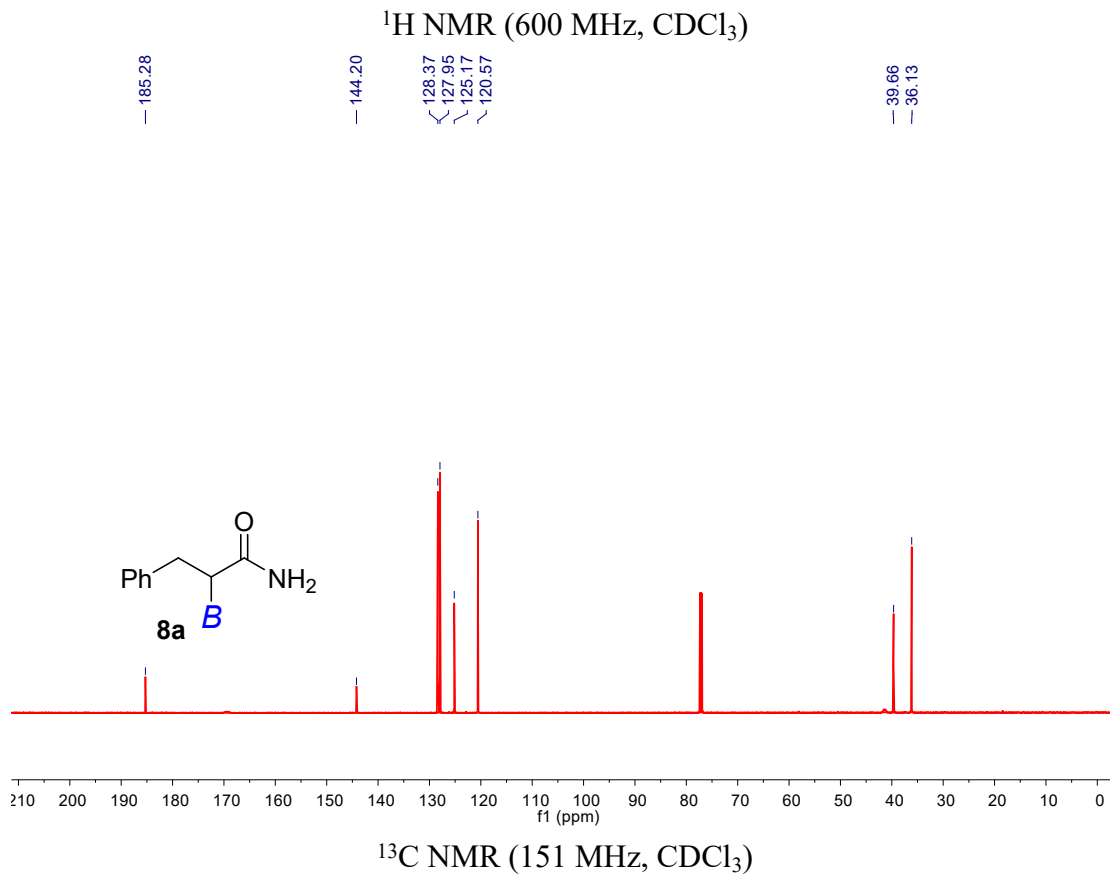
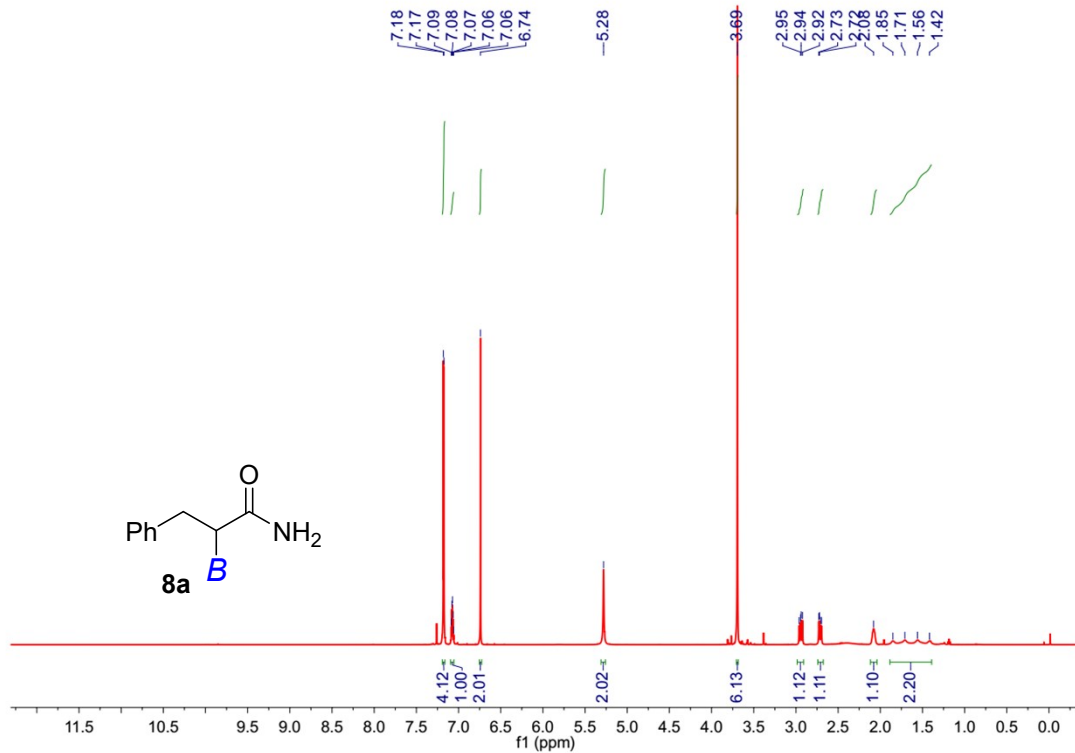
^{11}B NMR (193 MHz, CDCl_3)

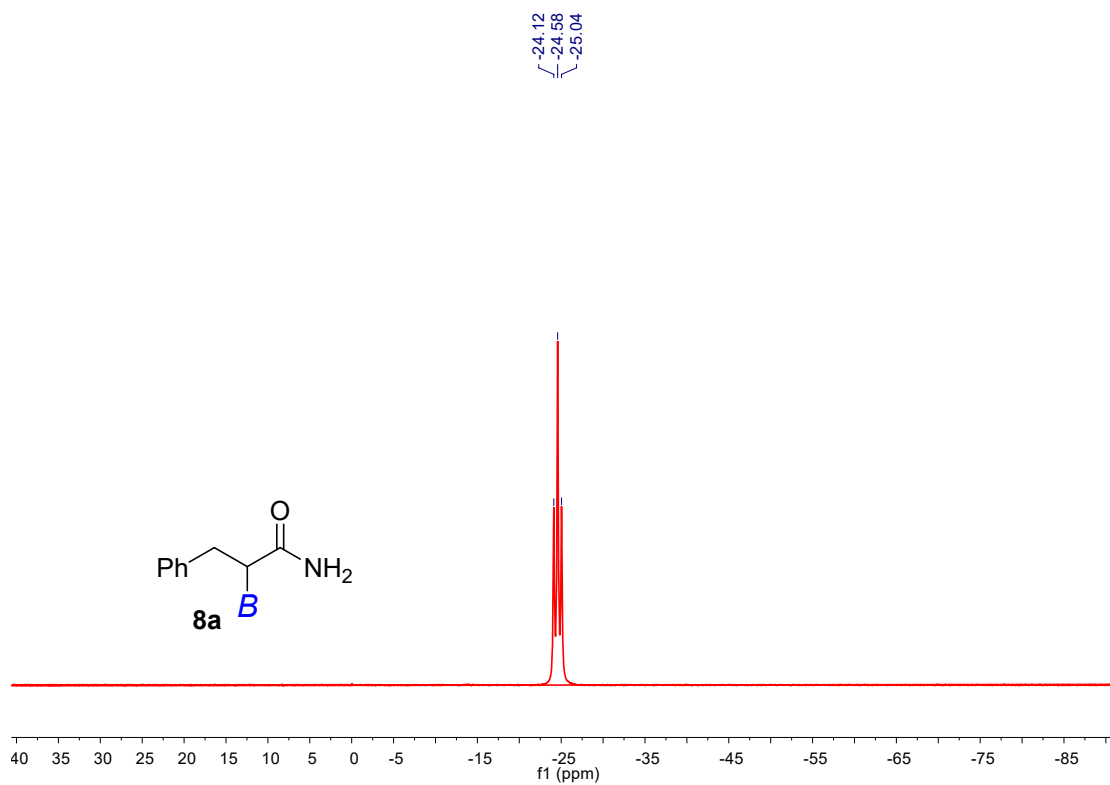


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

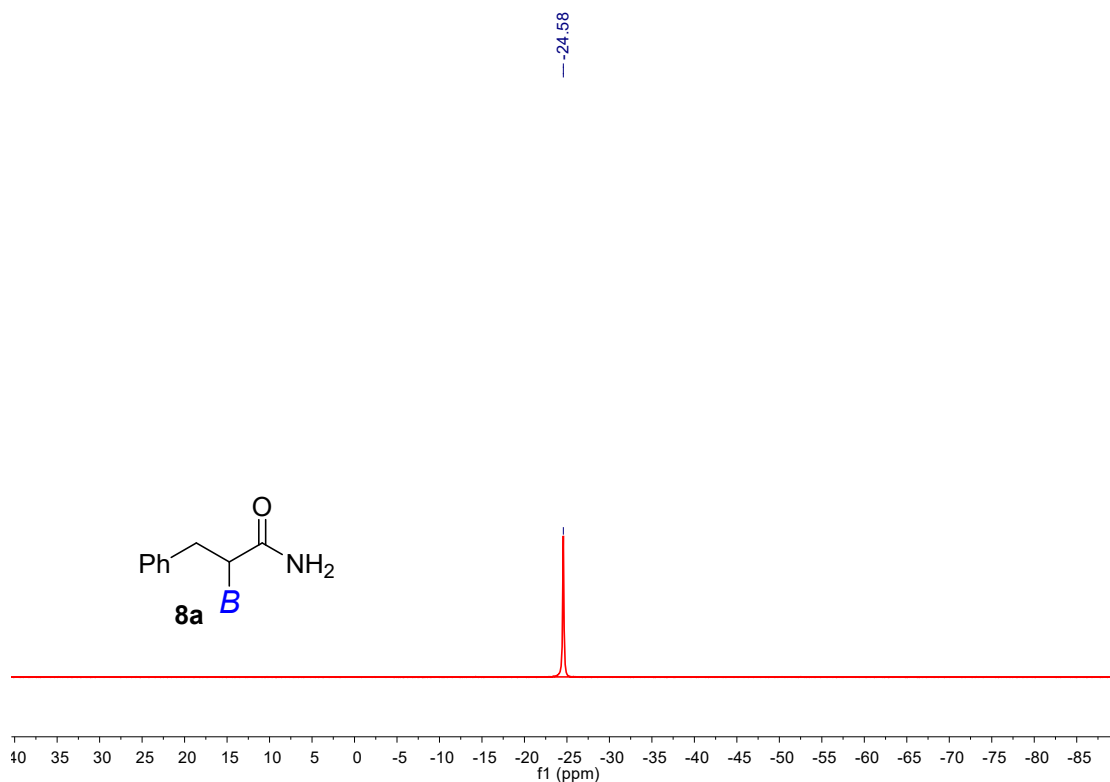


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

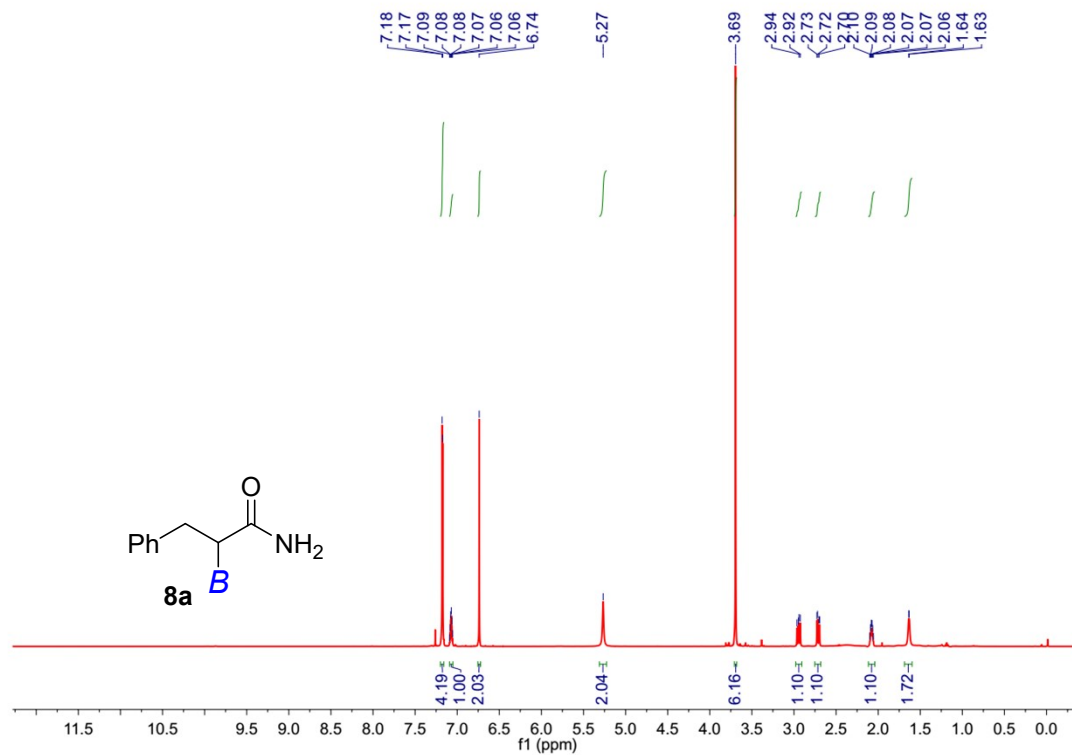




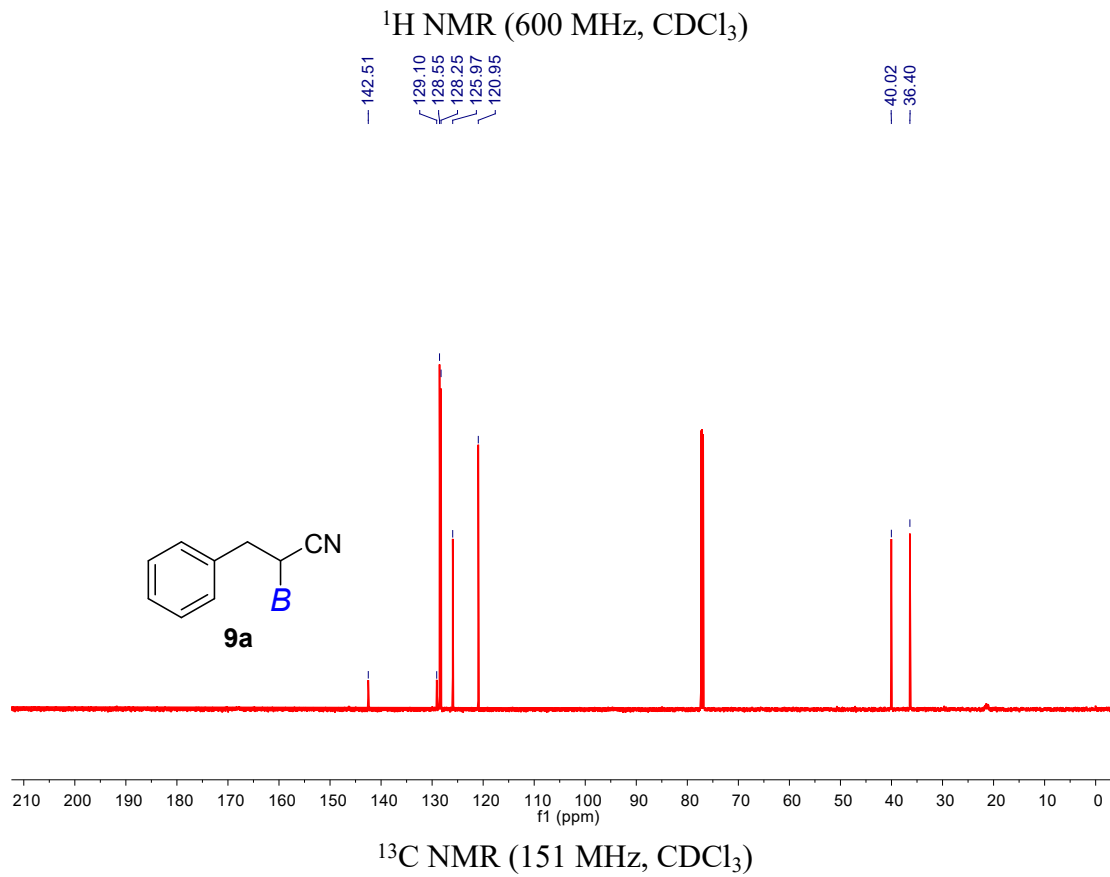
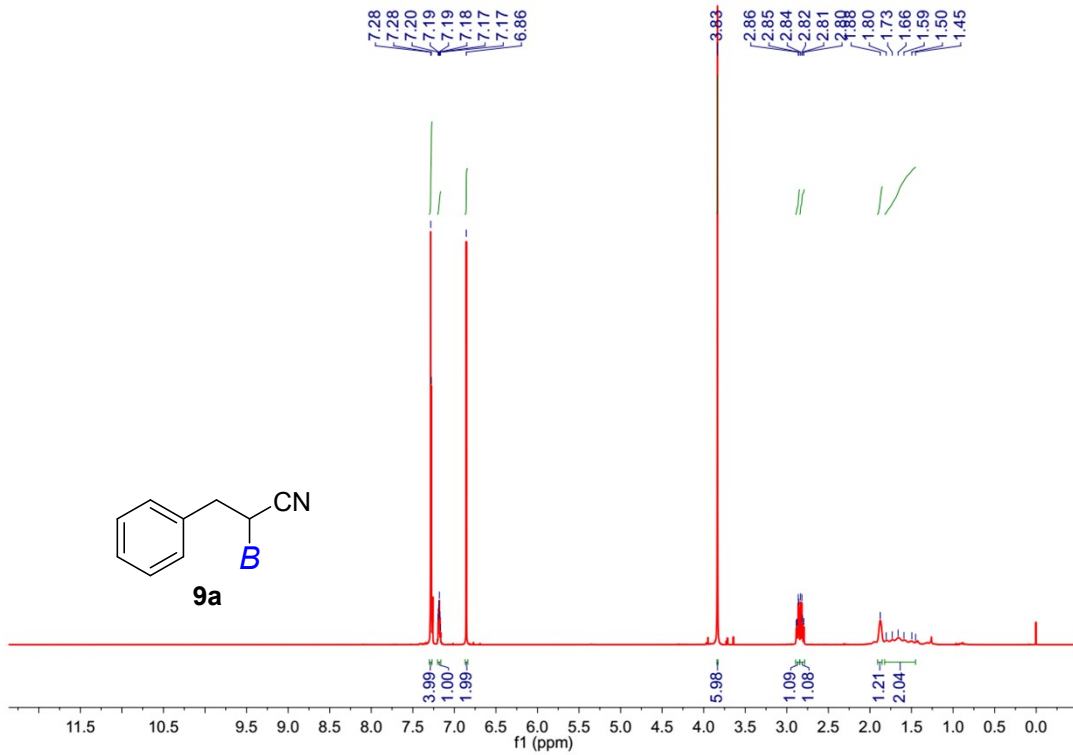
^{11}B NMR (193 MHz, CDCl_3)

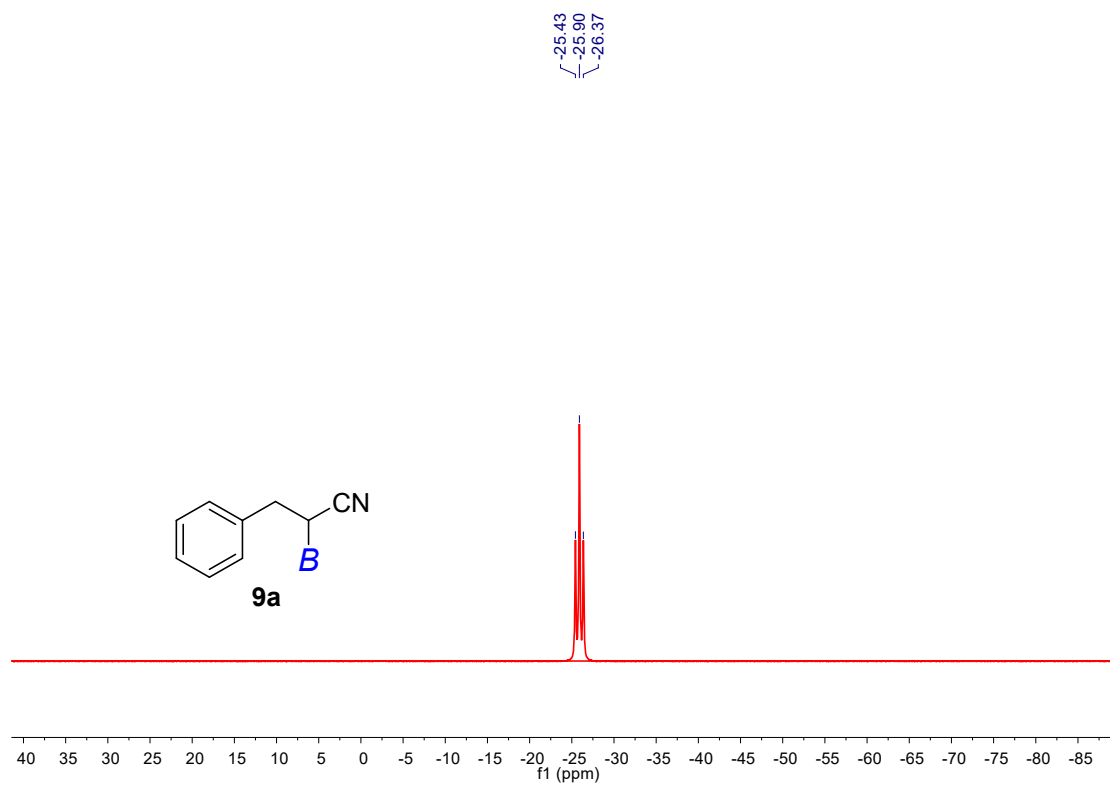


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

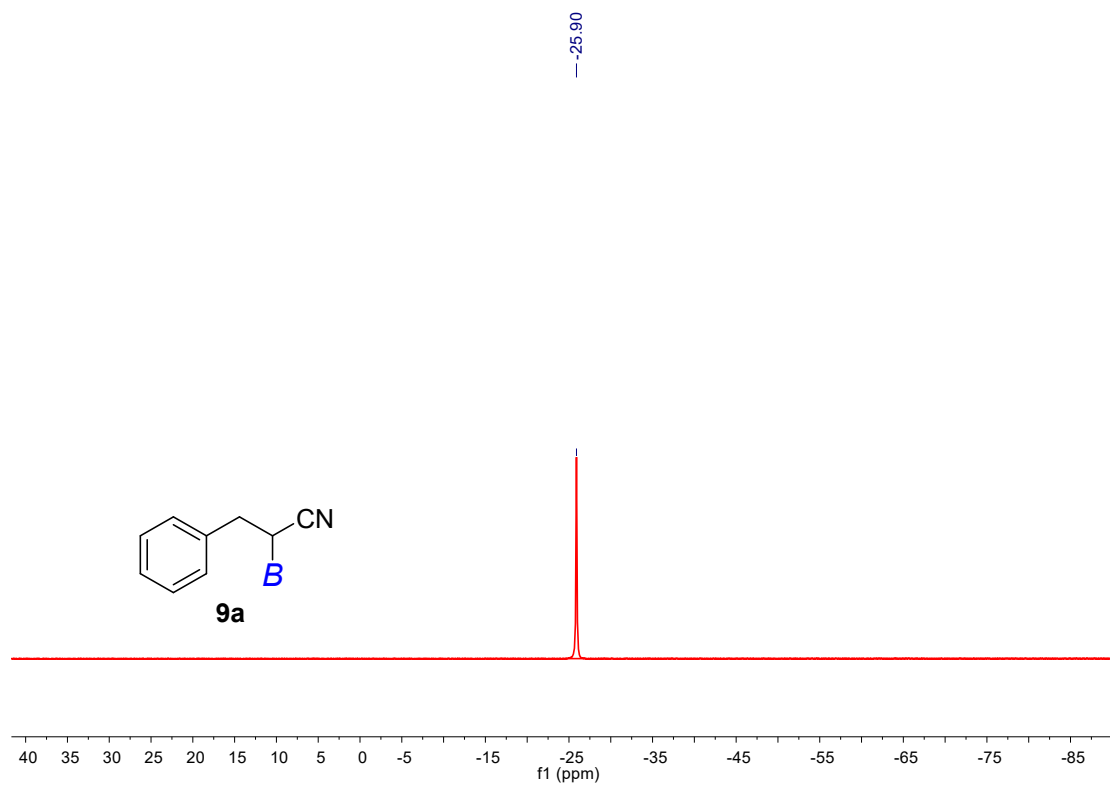


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

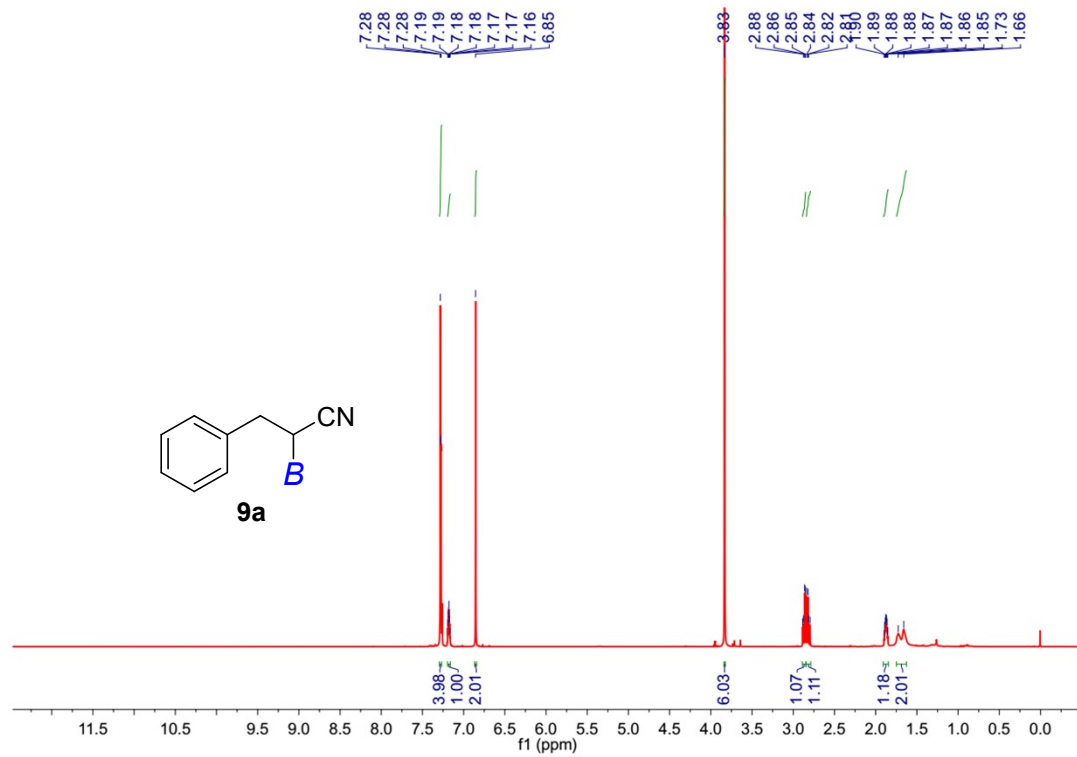




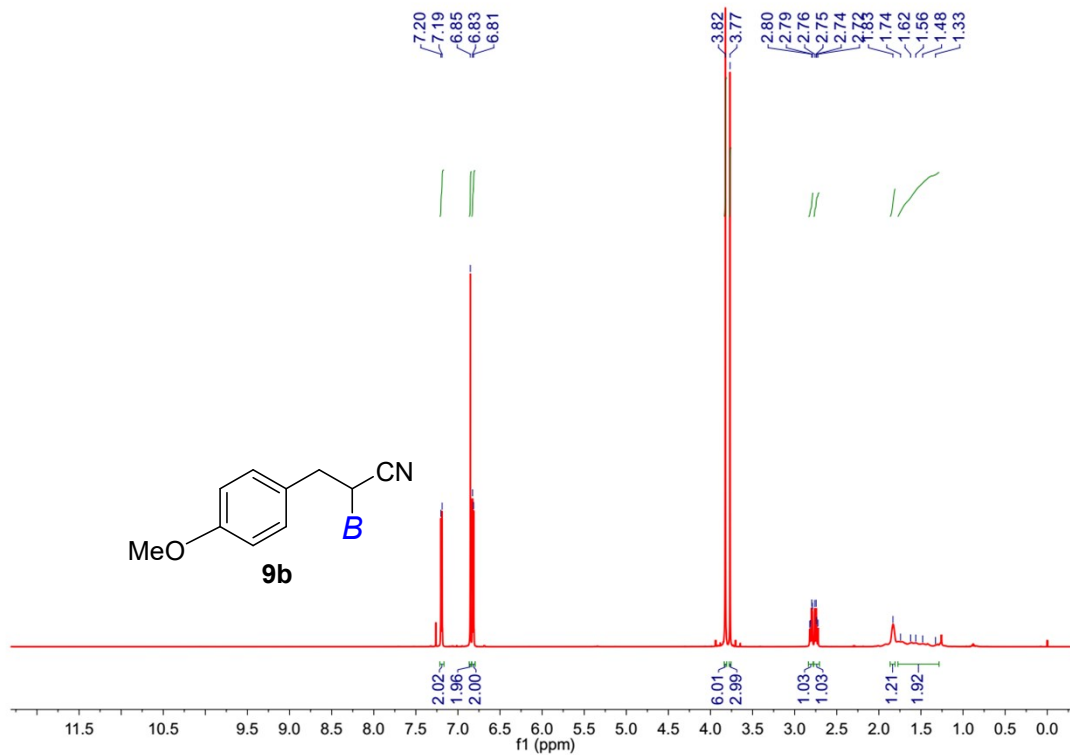
^{11}B NMR (193 MHz, CDCl_3)



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

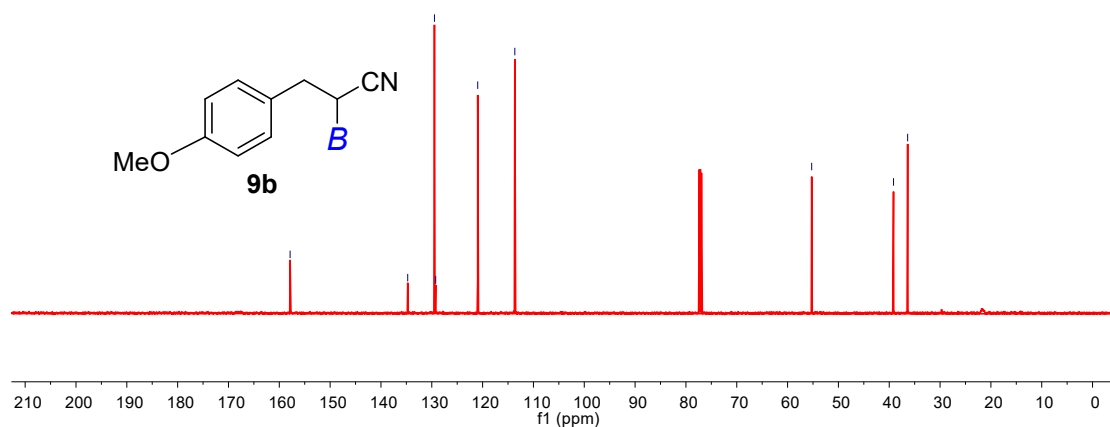


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

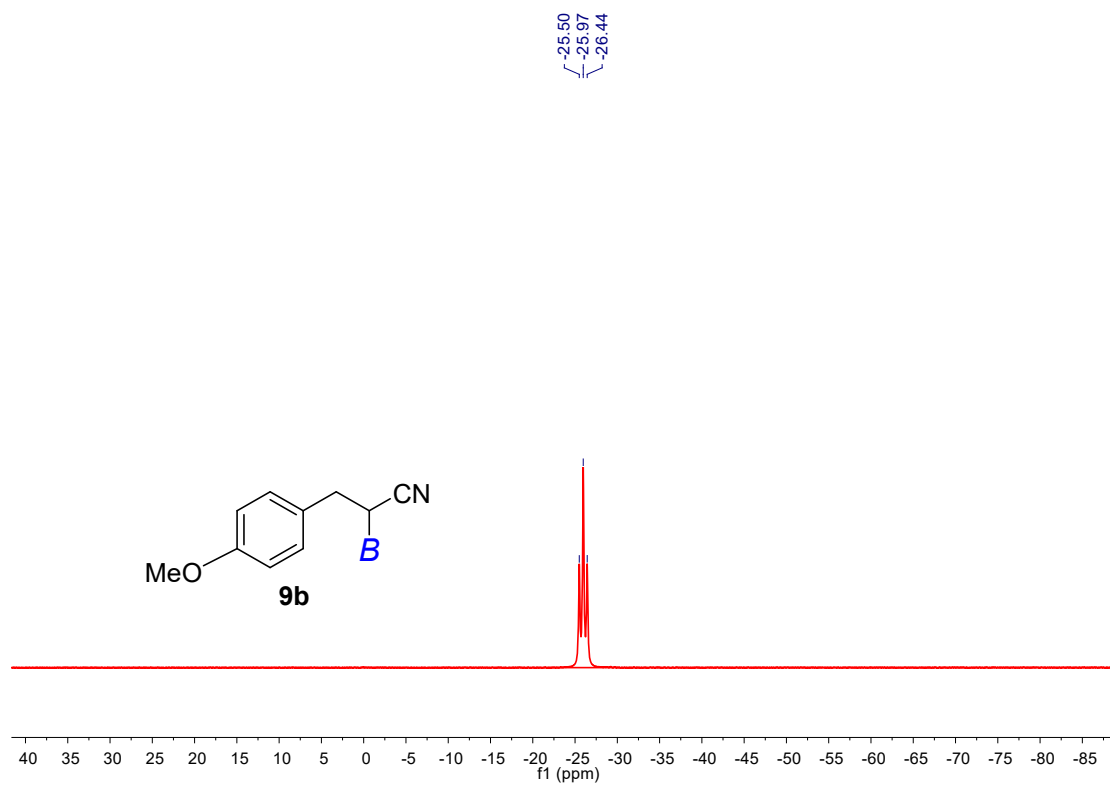


¹H NMR (600 MHz, CDCl₃)

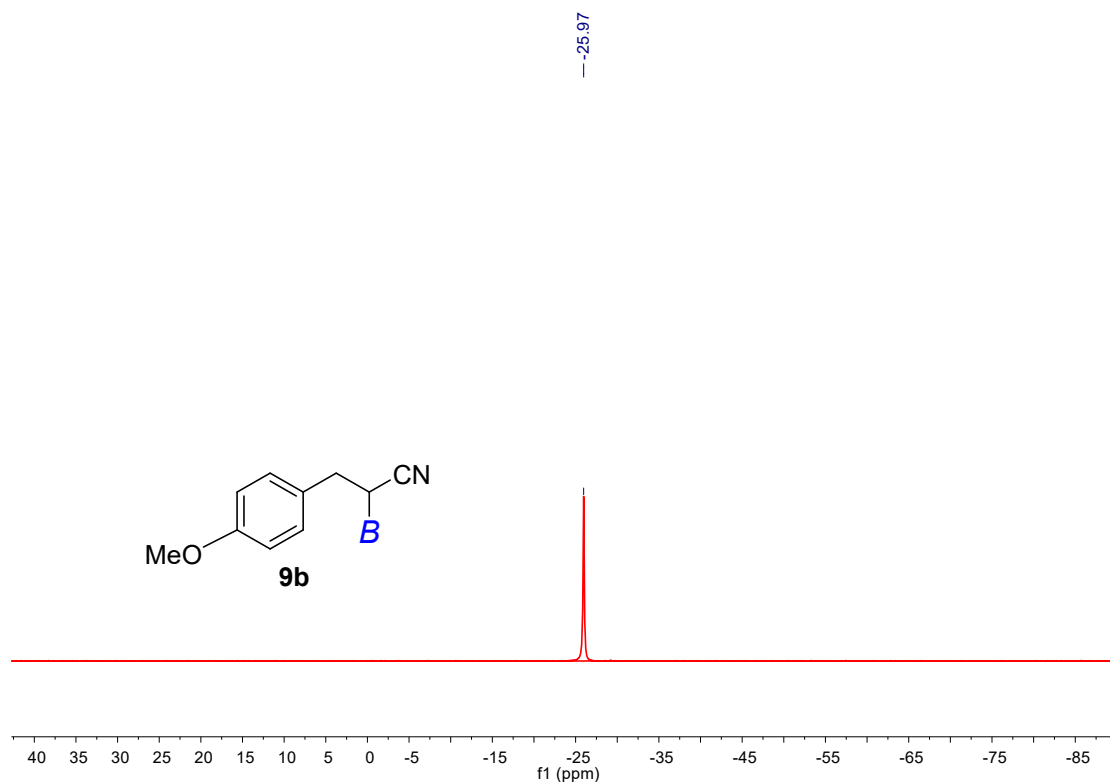
157.87
 134.73
 129.48
 129.23
 120.96
 113.66
 55.26
 39.16
 36.38



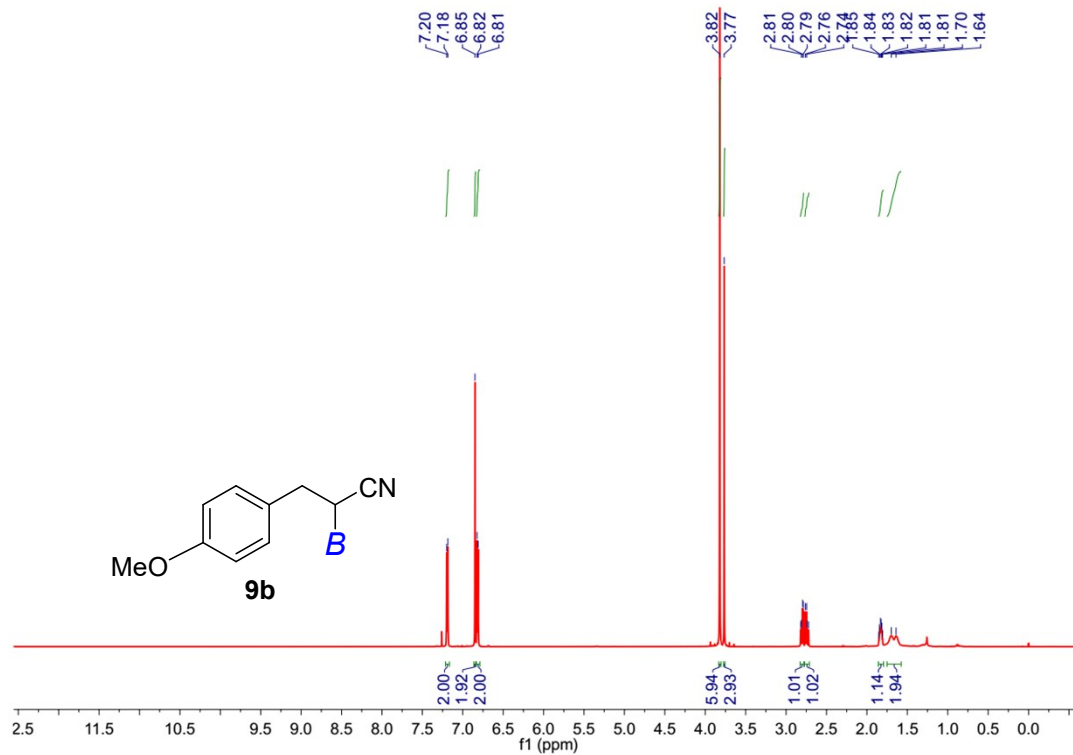
¹³C NMR (151 MHz, CDCl₃)



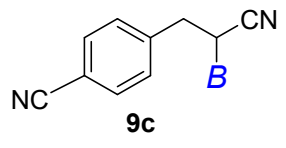
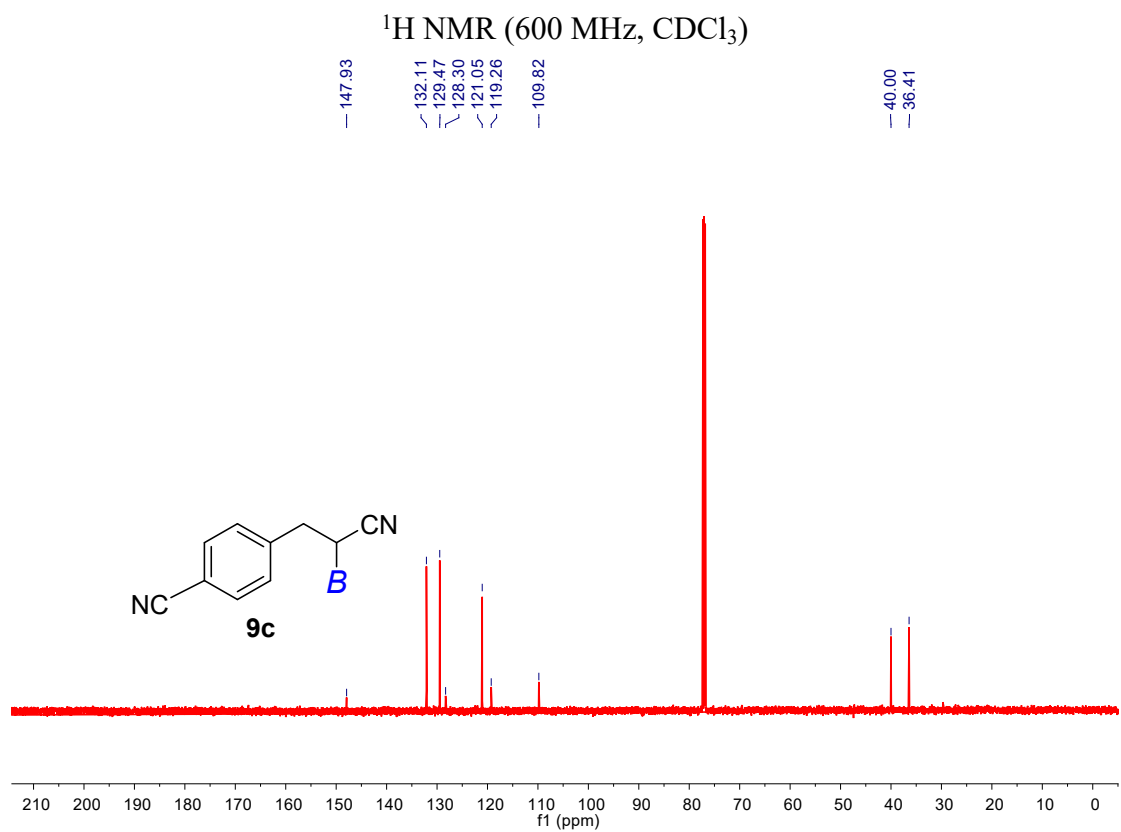
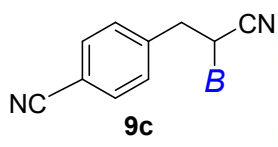
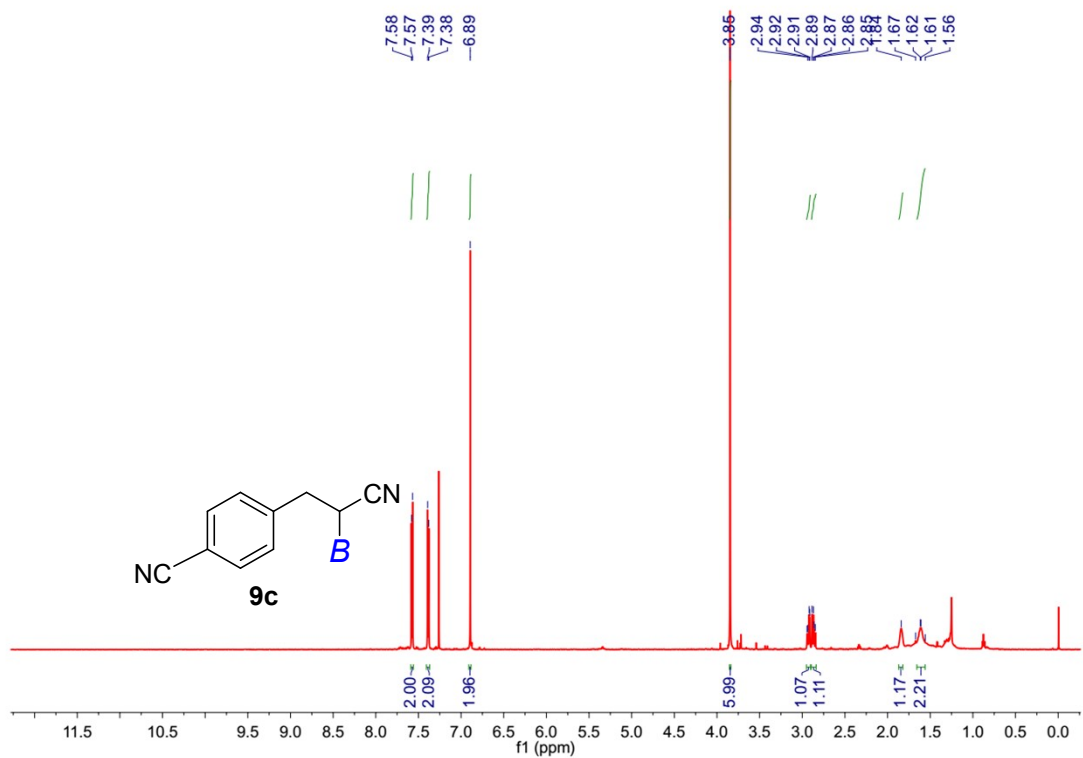
^{11}B NMR (193 MHz, CDCl_3)

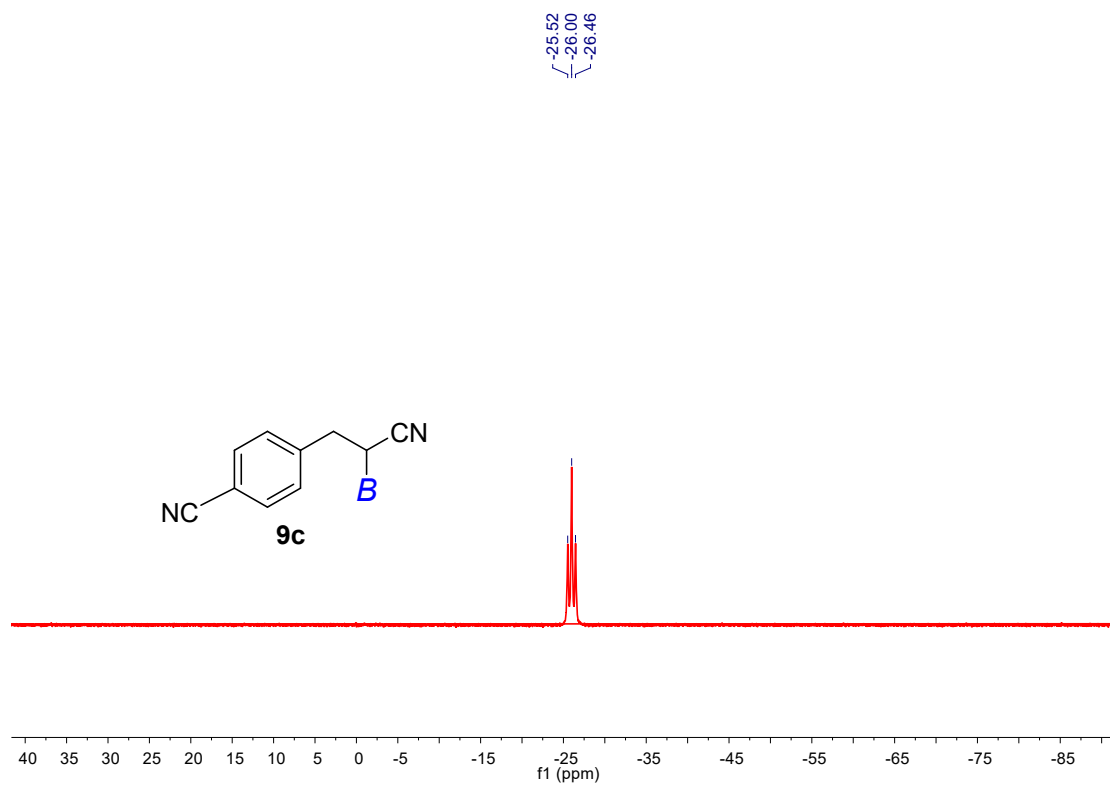


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

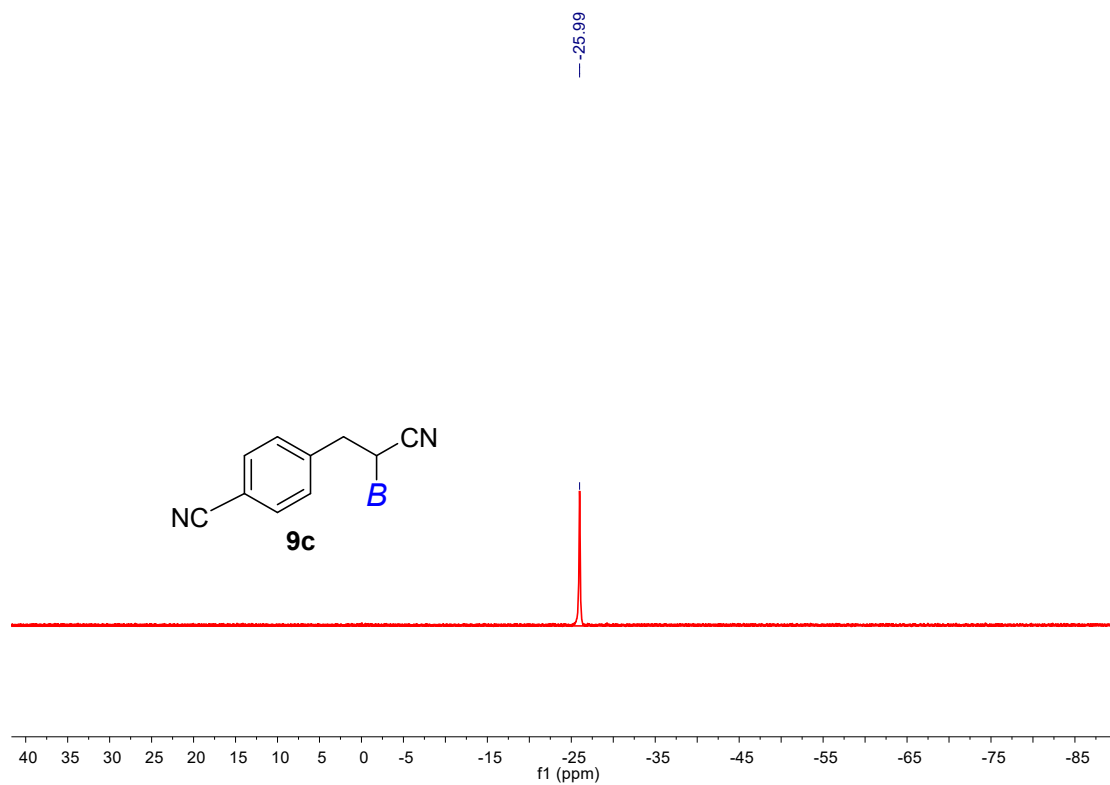


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

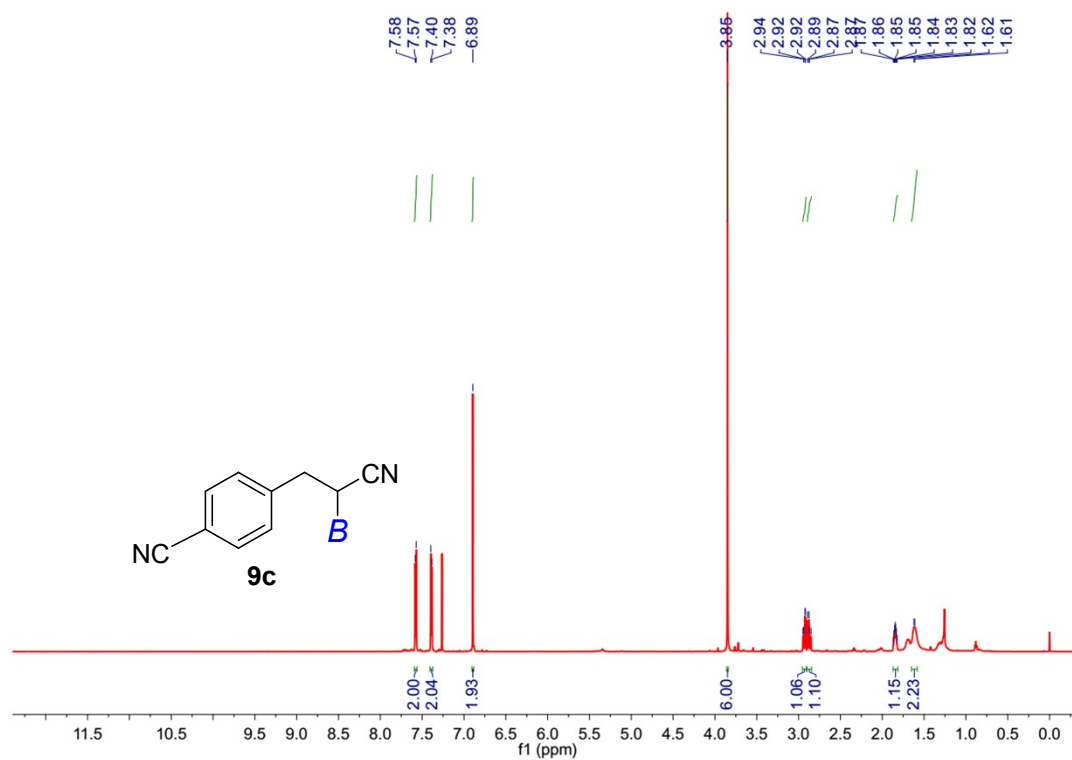




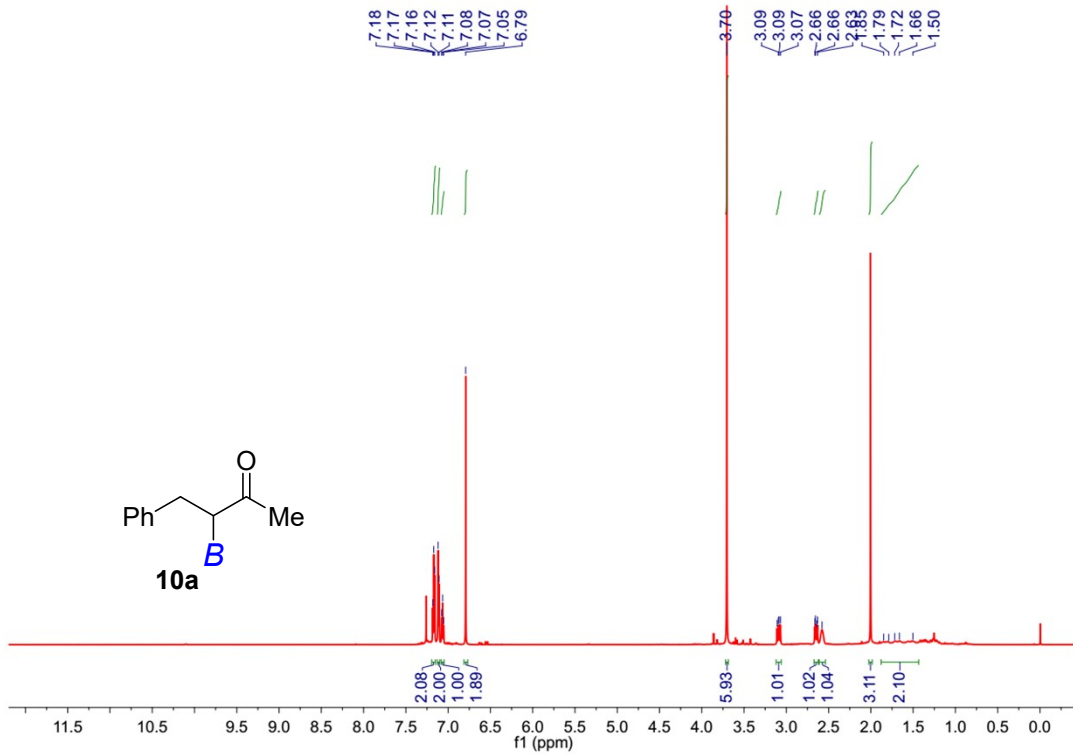
^{11}B NMR (193 MHz, CDCl_3)



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



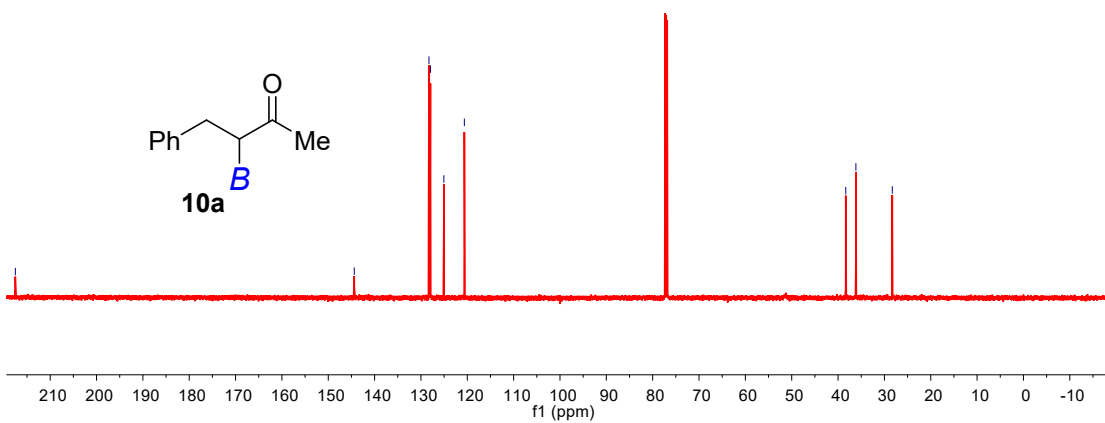
— 217.52

¹H NMR (600 MHz, CDCl₃)

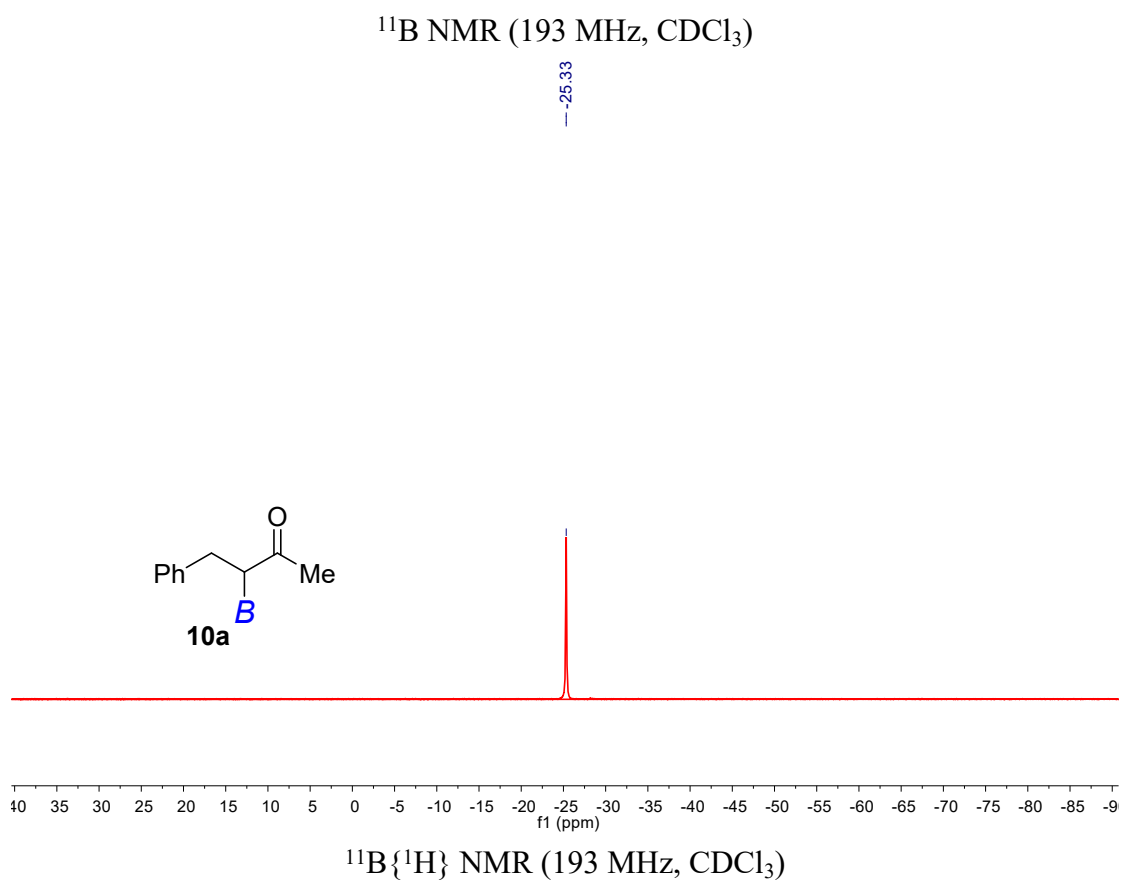
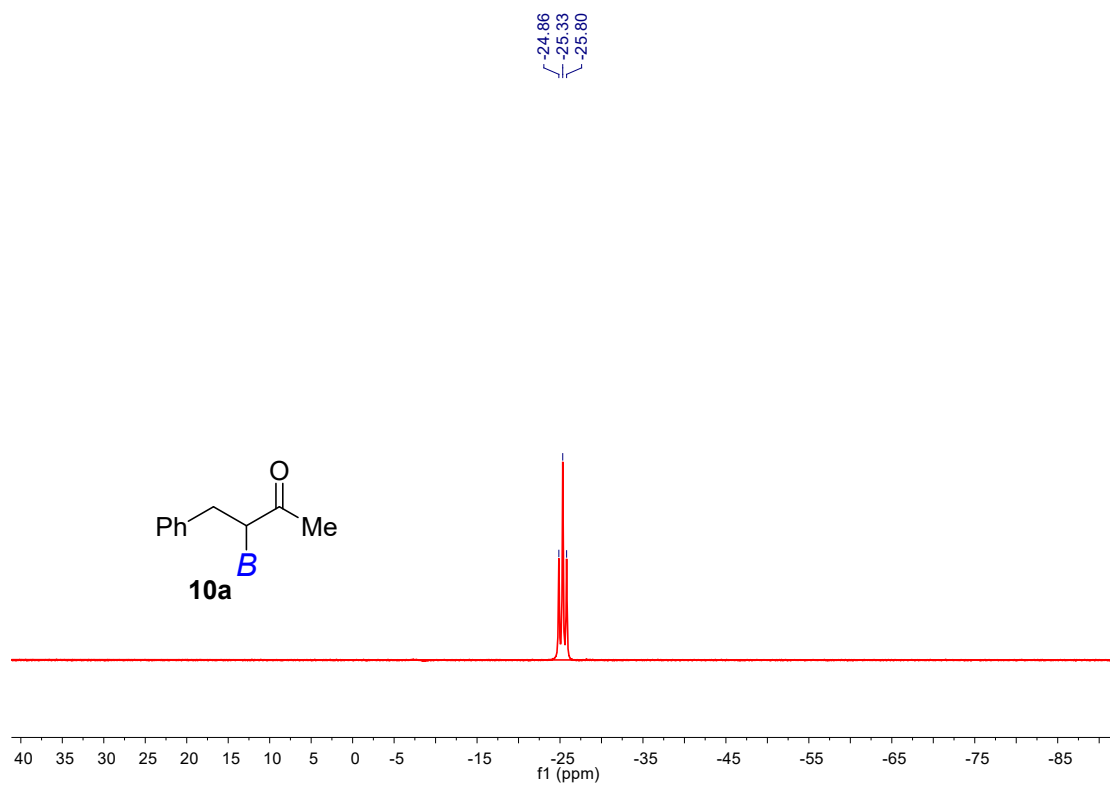
— 144.39

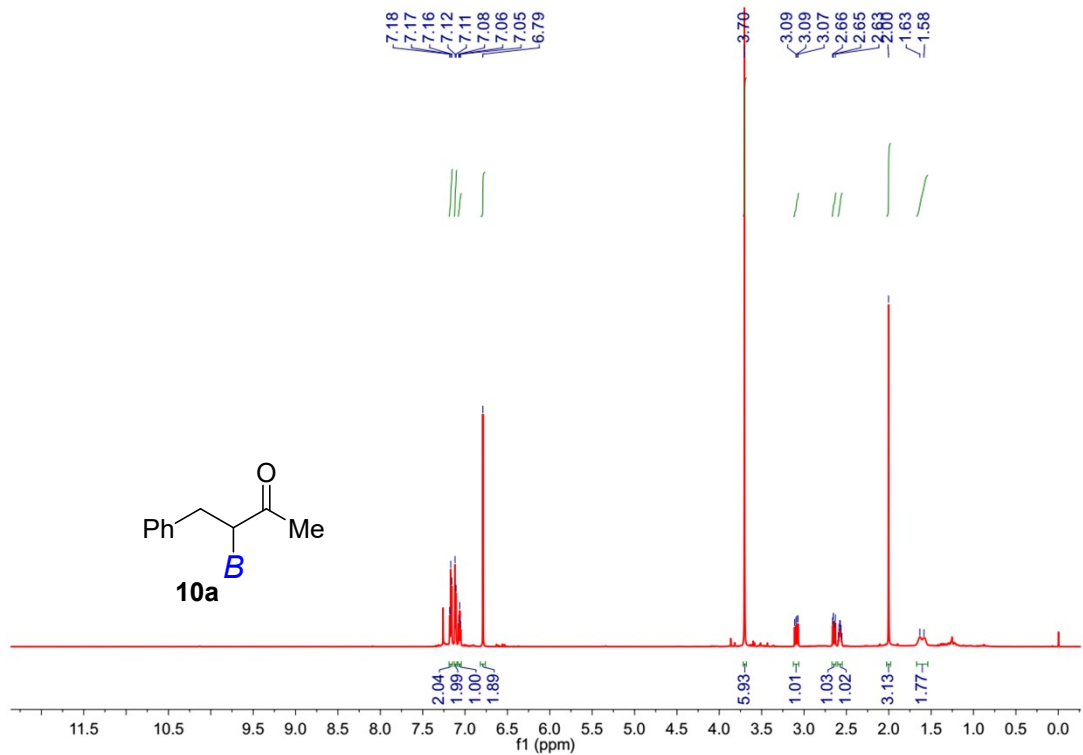
128.27
127.95
125.06
120.63

38.34
36.15
28.30

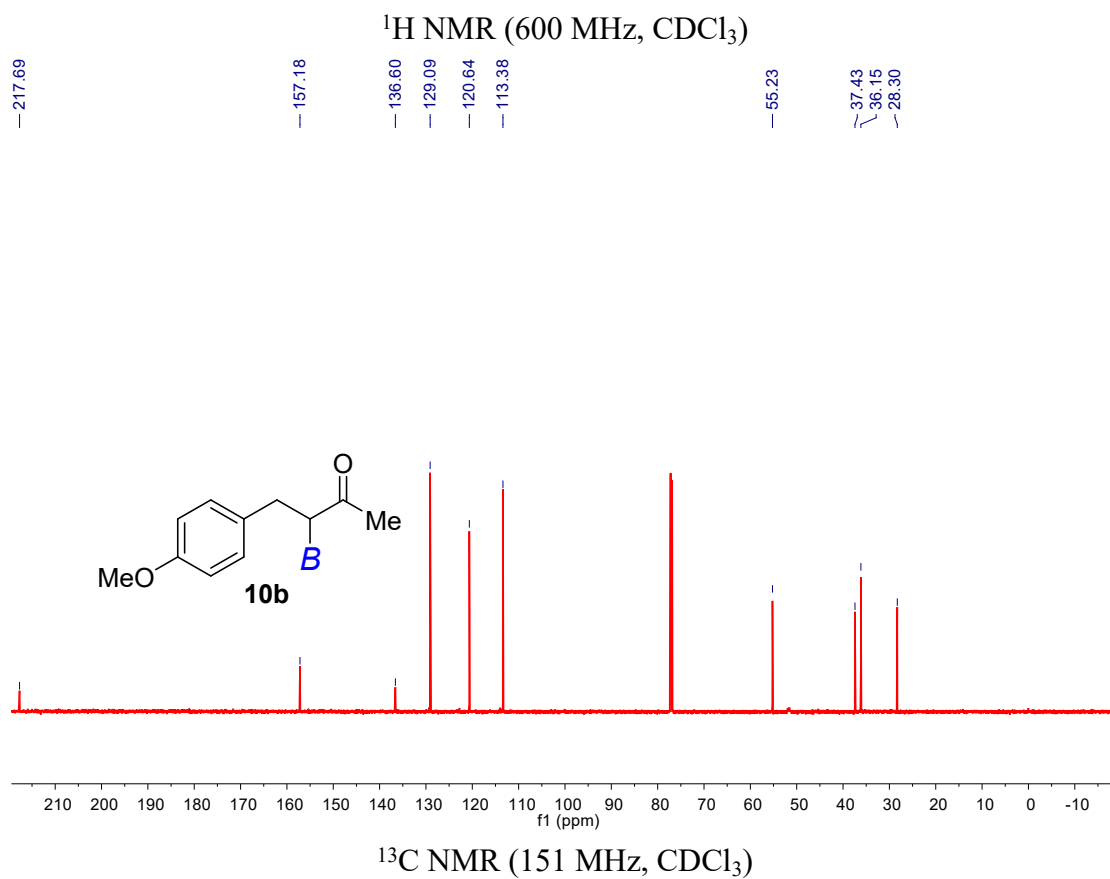
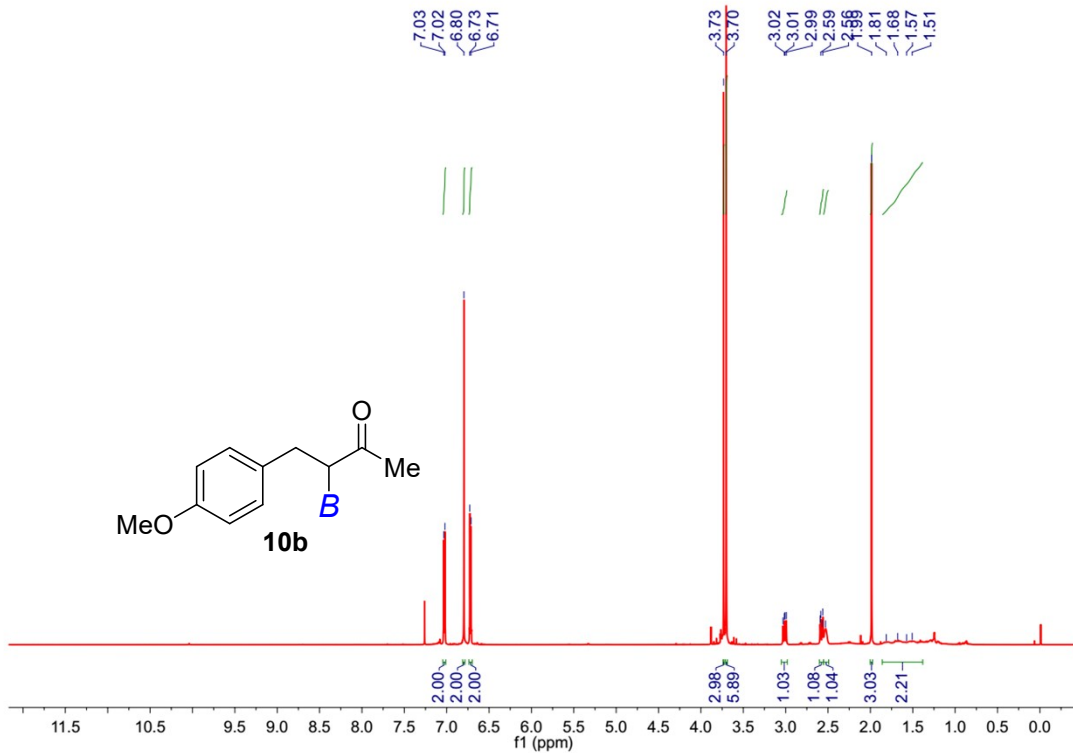


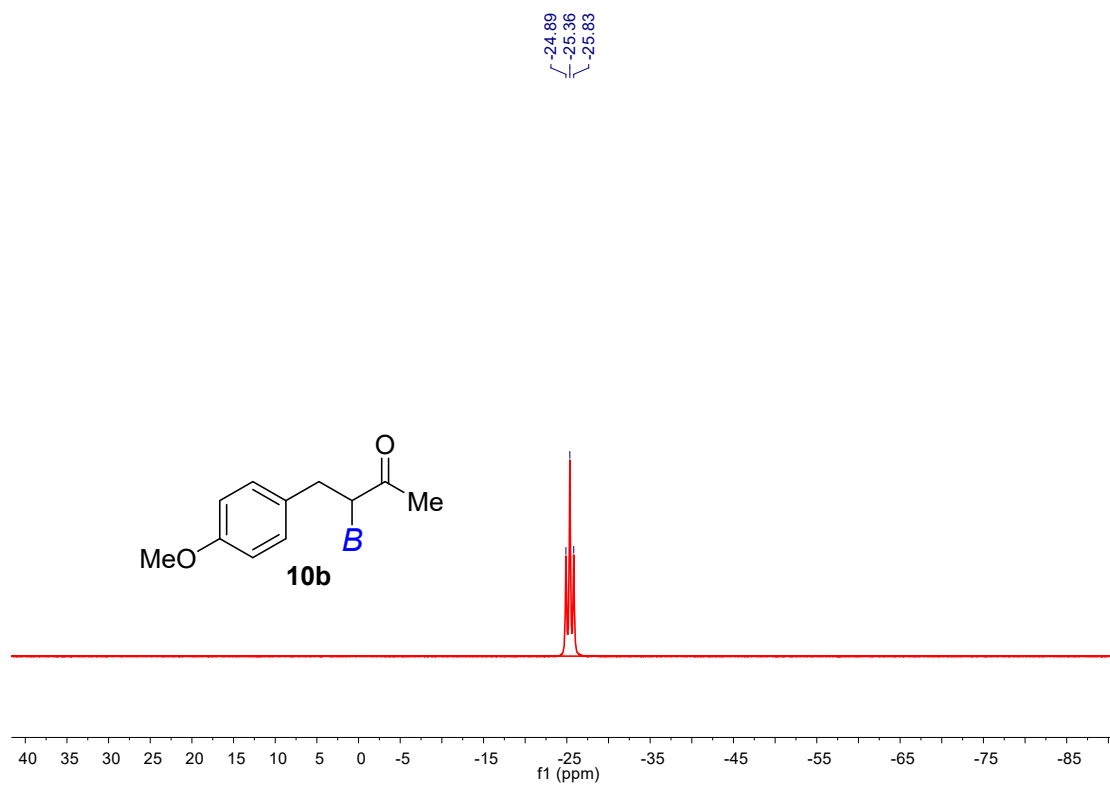
¹³C NMR (151 MHz, CDCl₃)



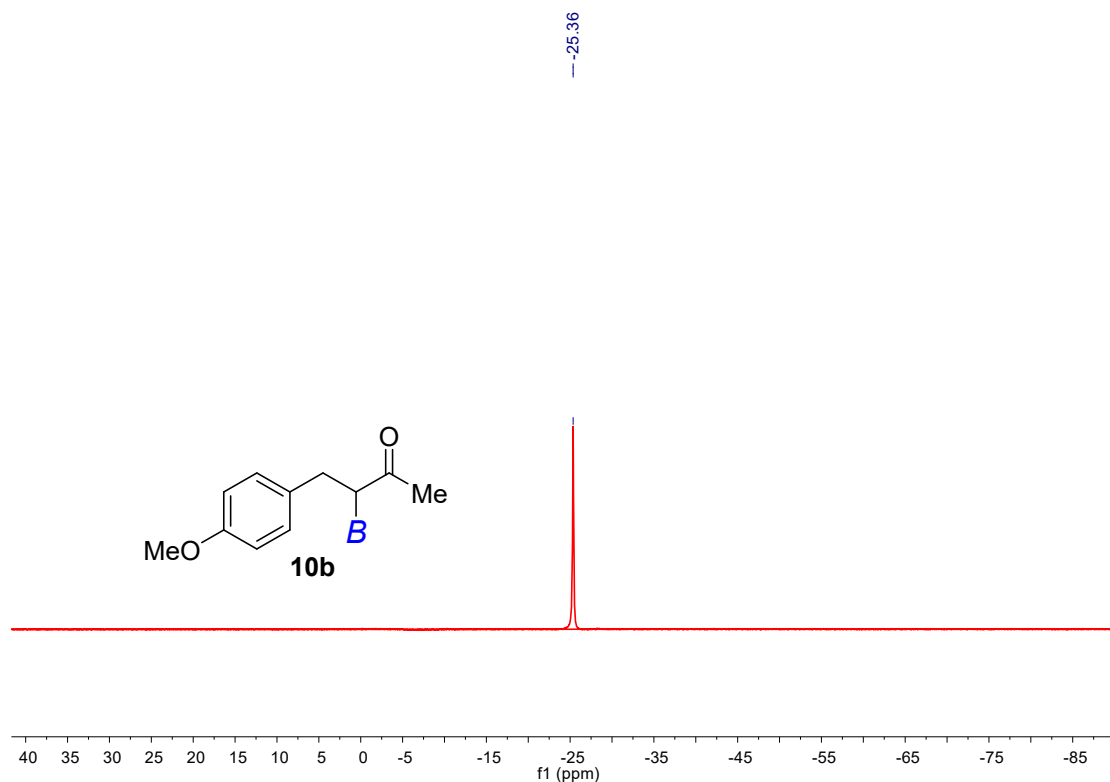


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

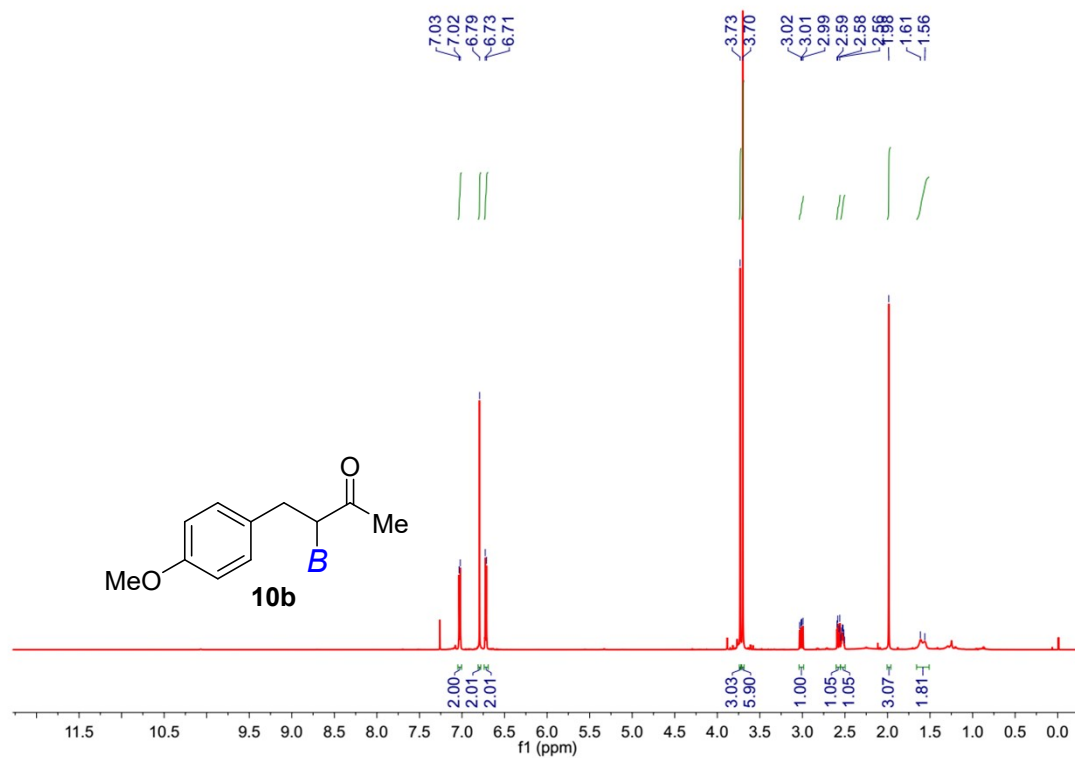




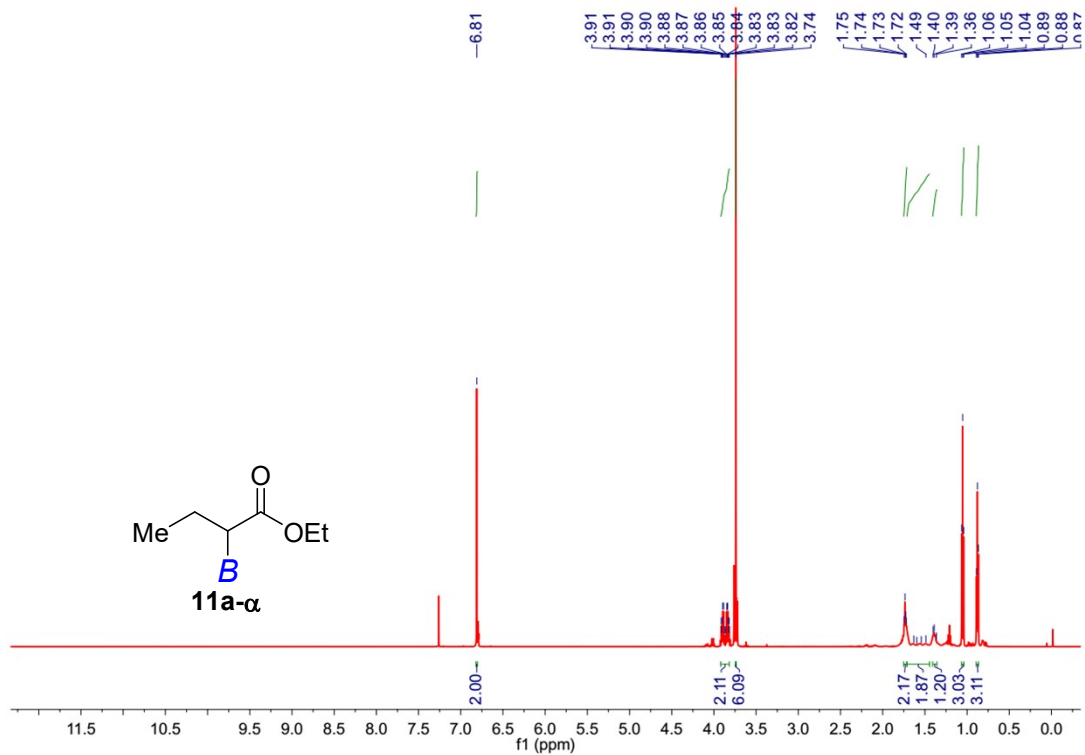
¹¹B NMR (193 MHz, CDCl₃)



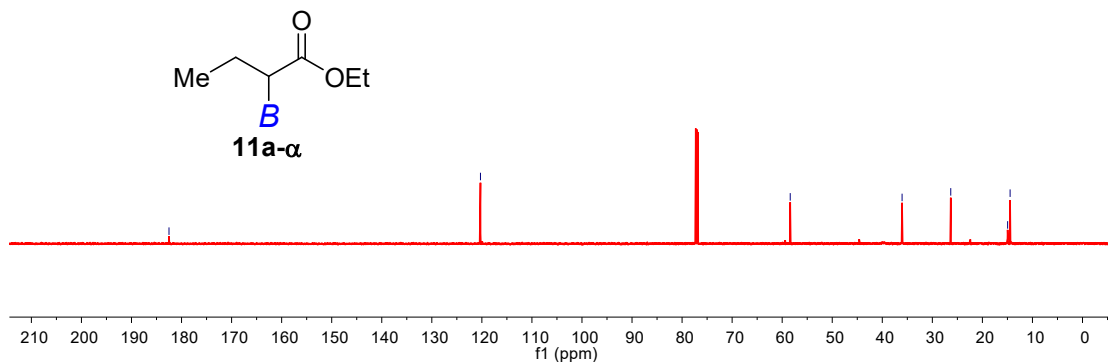
¹¹B{¹H} NMR (193 MHz, CDCl₃)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

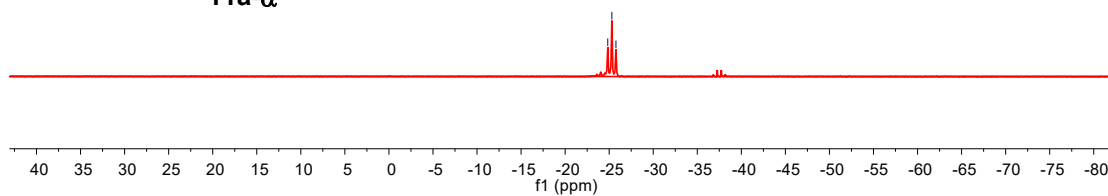
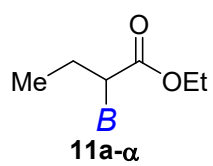


¹H NMR (600 MHz, CDCl₃)



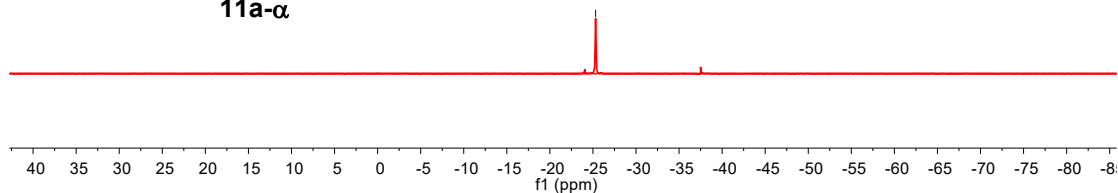
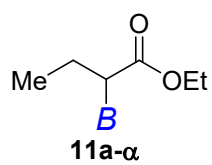
¹³C NMR (151 MHz, CDCl₃)

-24.84
-25.30
-25.77

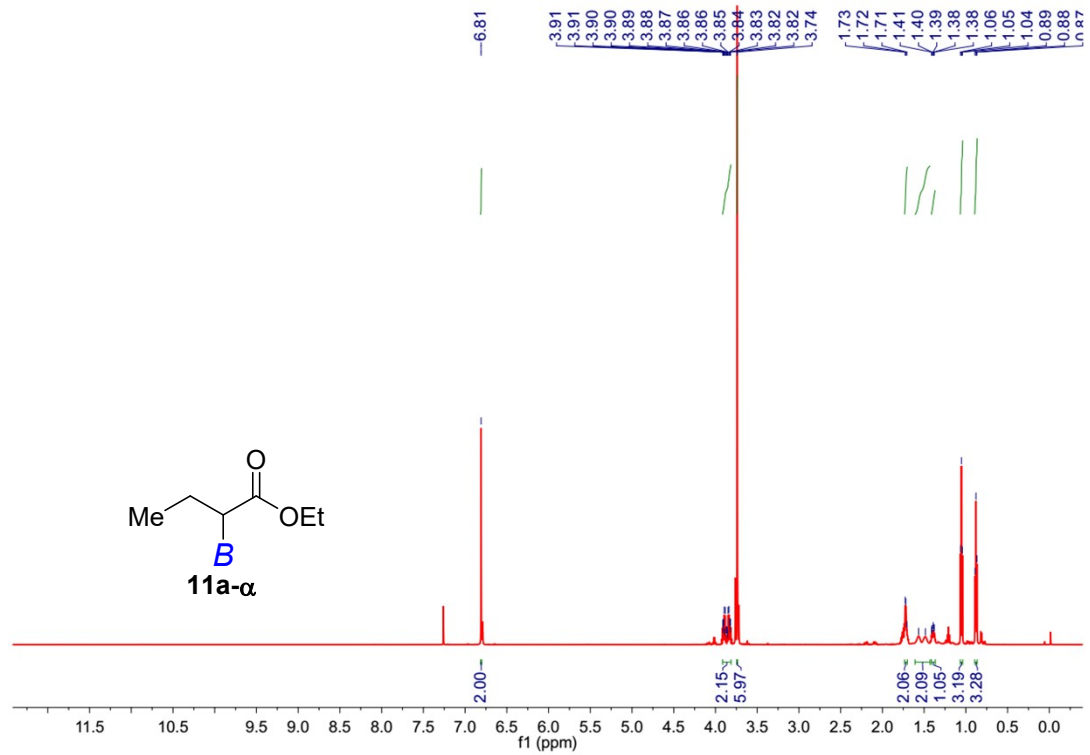


^{11}B NMR (193 MHz, CDCl_3)

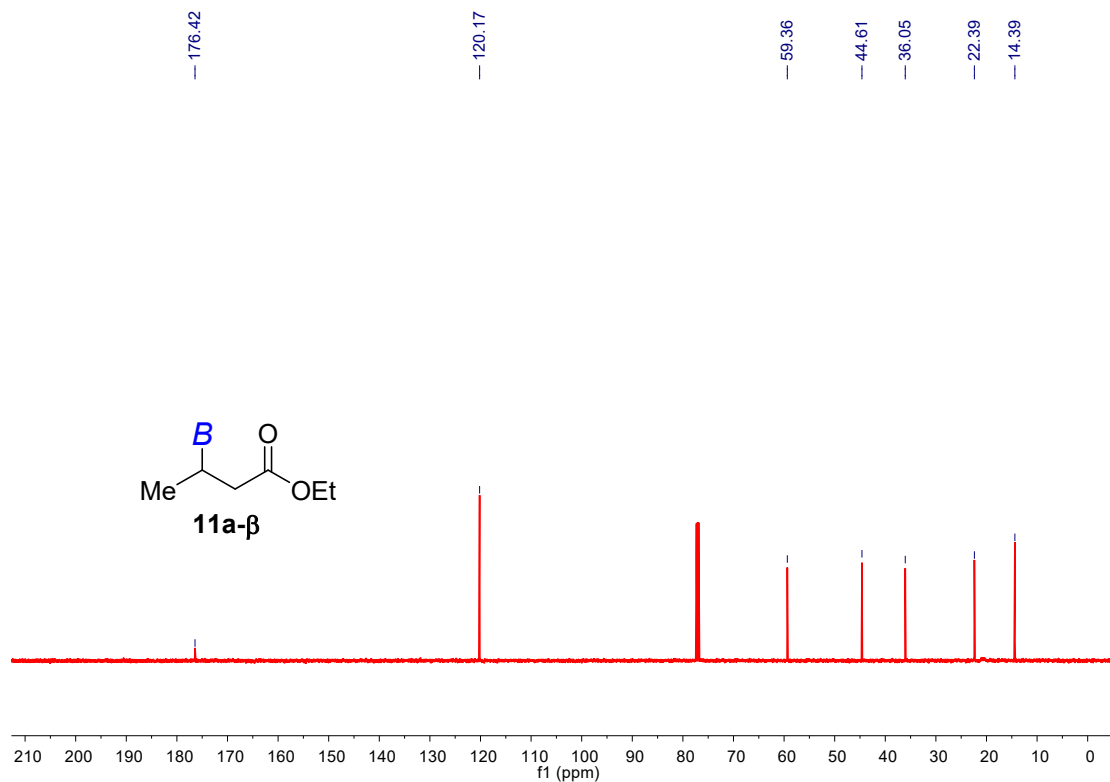
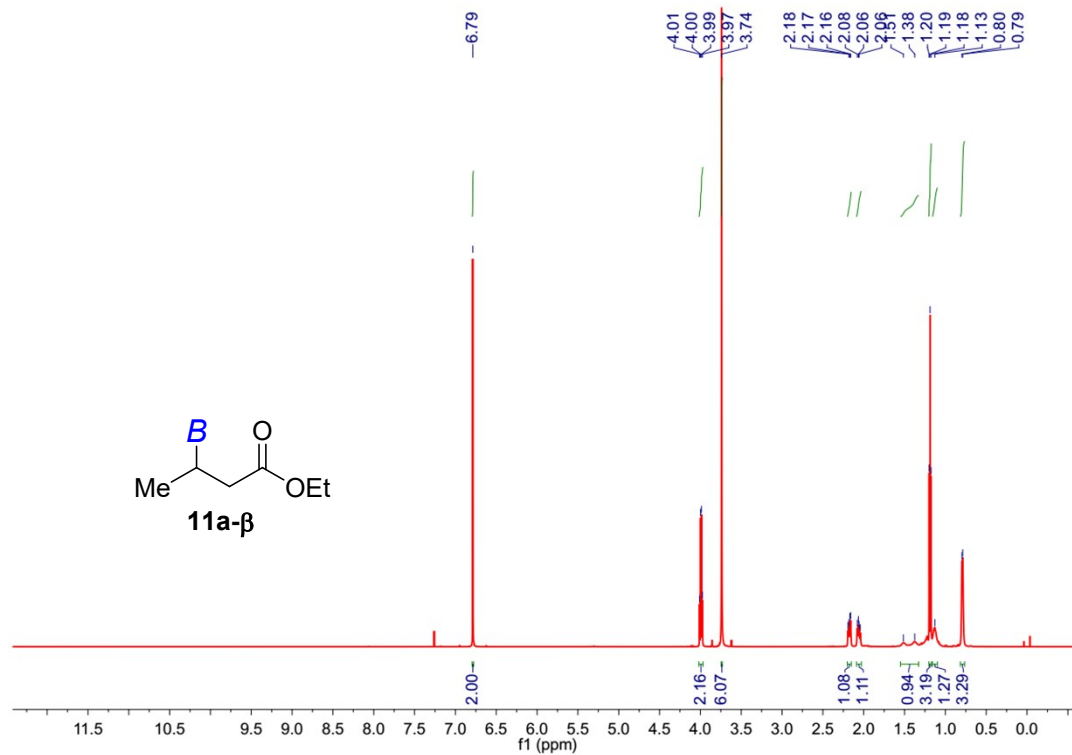
-25.31

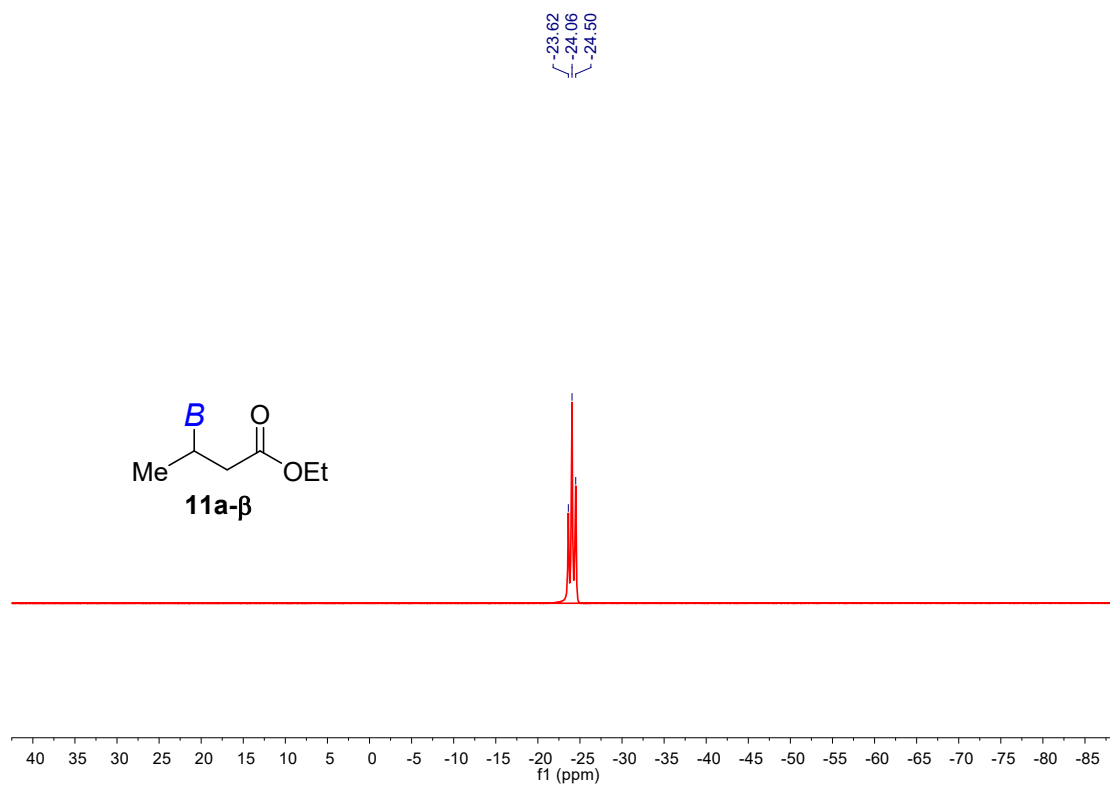


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

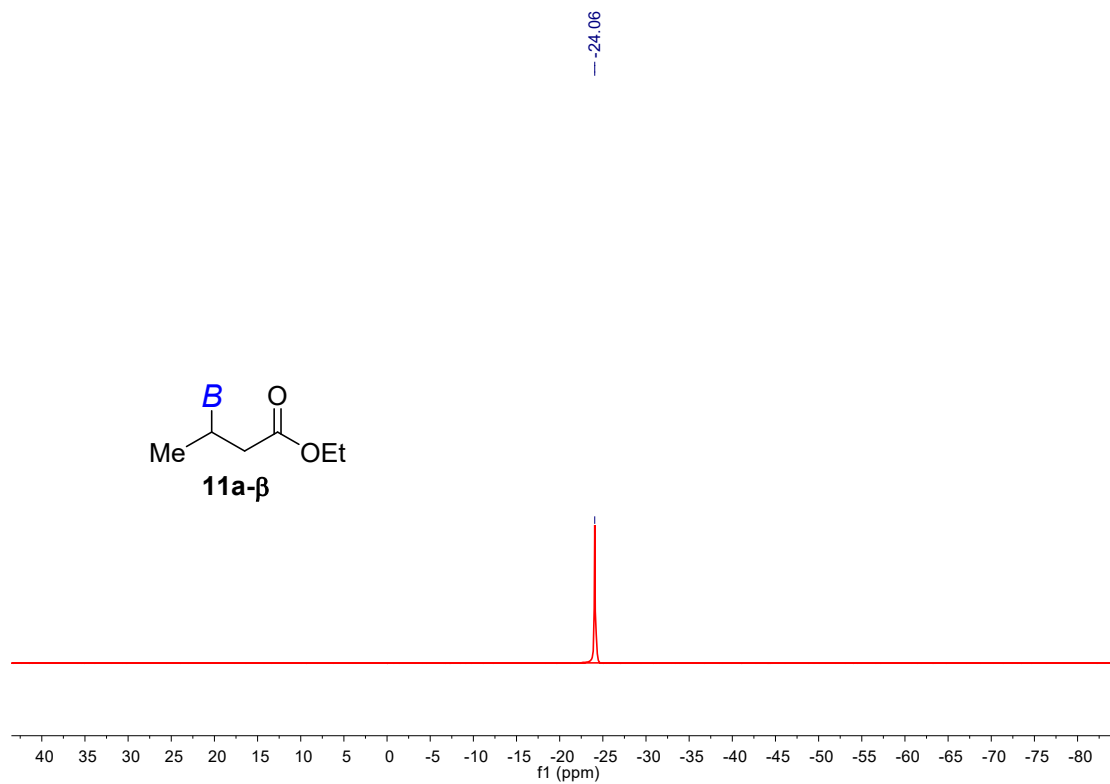


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

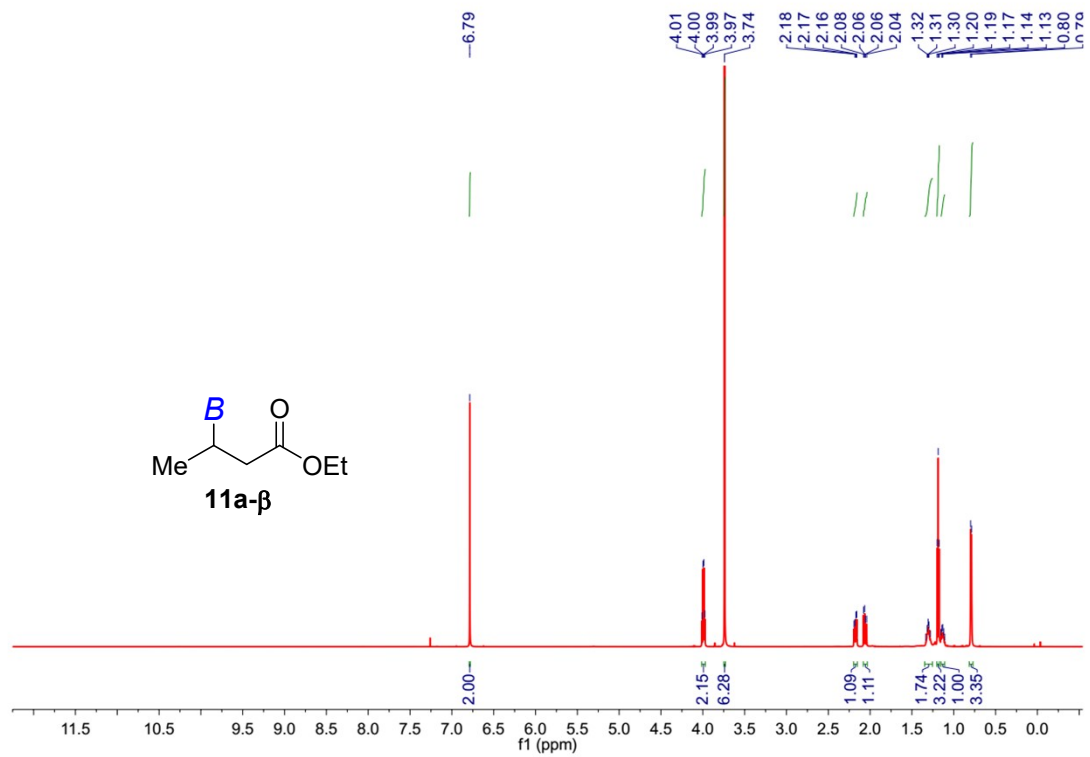




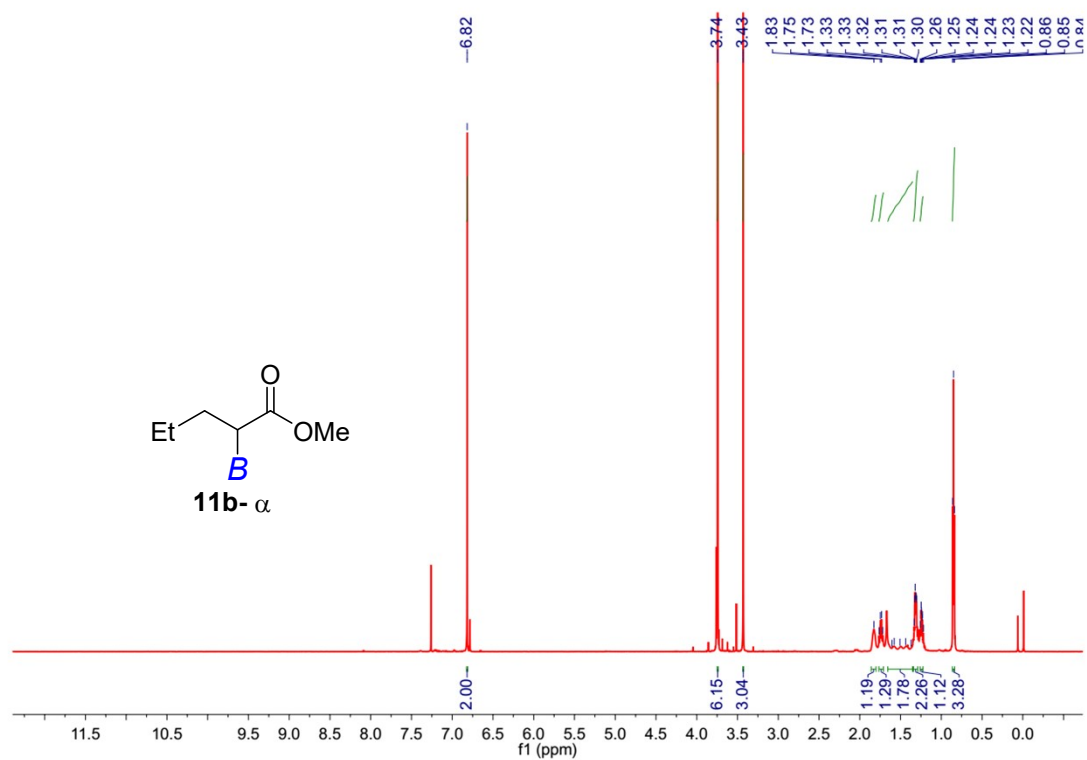
^{11}B NMR (193 MHz, CDCl_3)



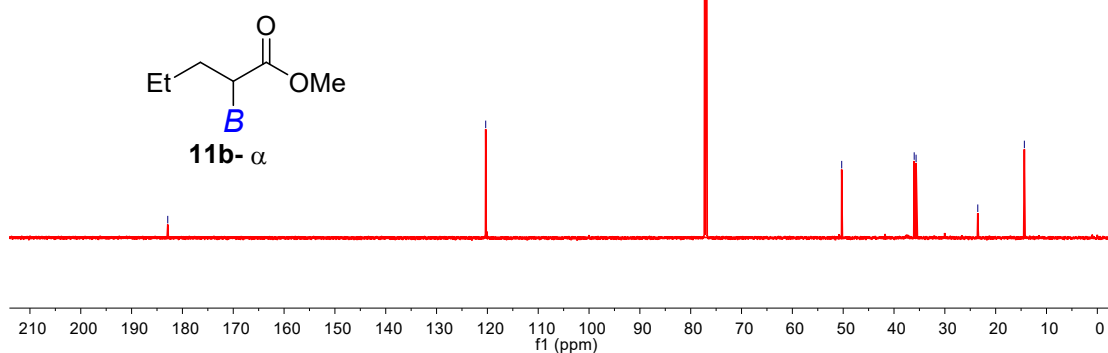
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



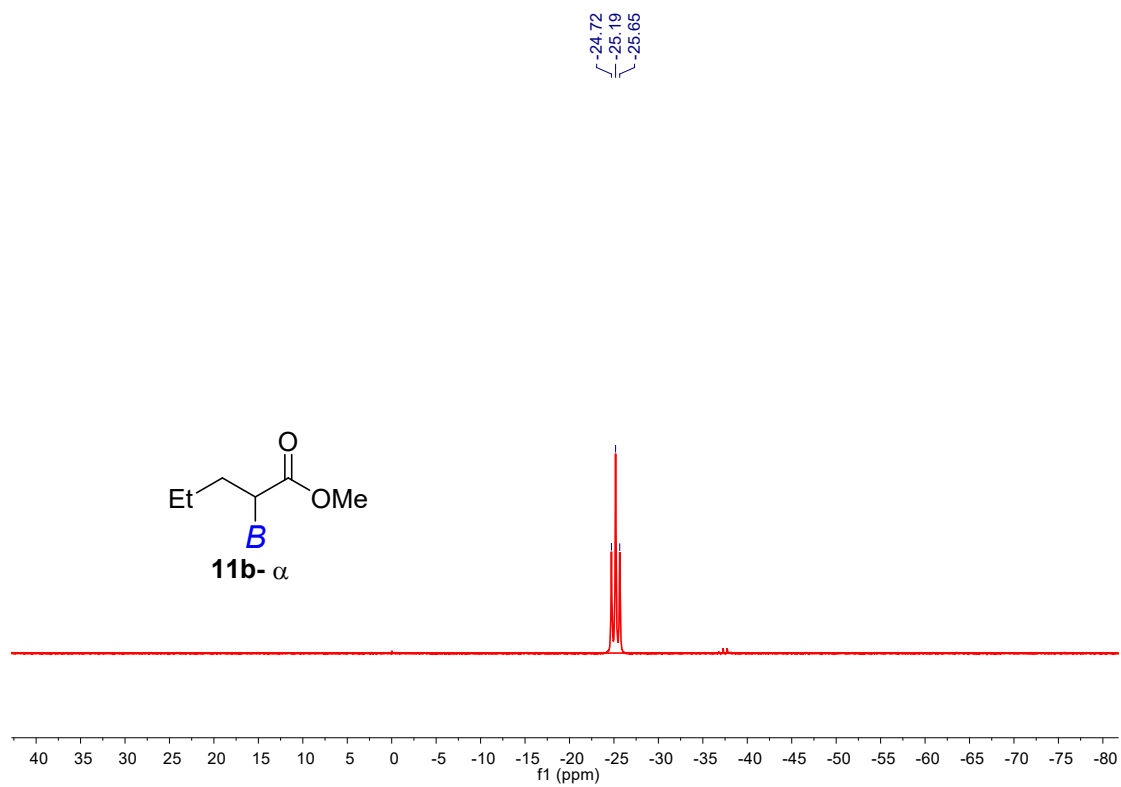
¹H{¹¹B} NMR (600 MHz, CDCl₃)



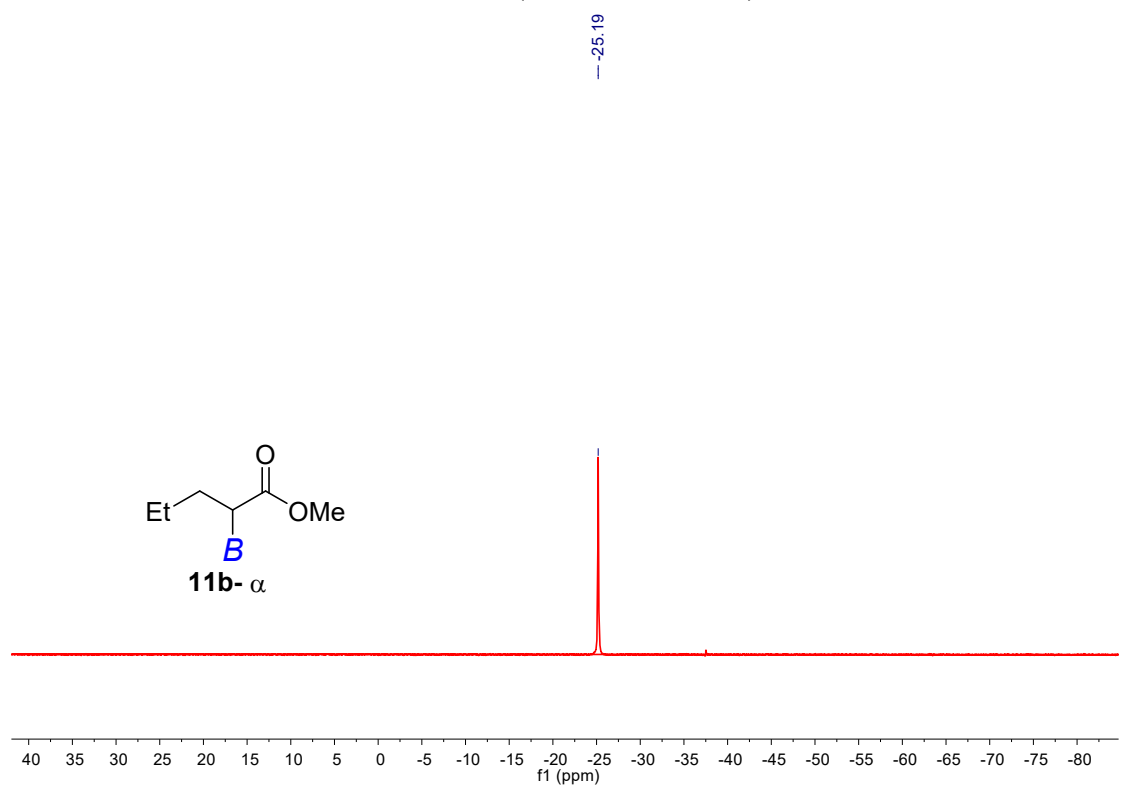
$^1\text{H NMR}$ (600 MHz, CDCl_3)



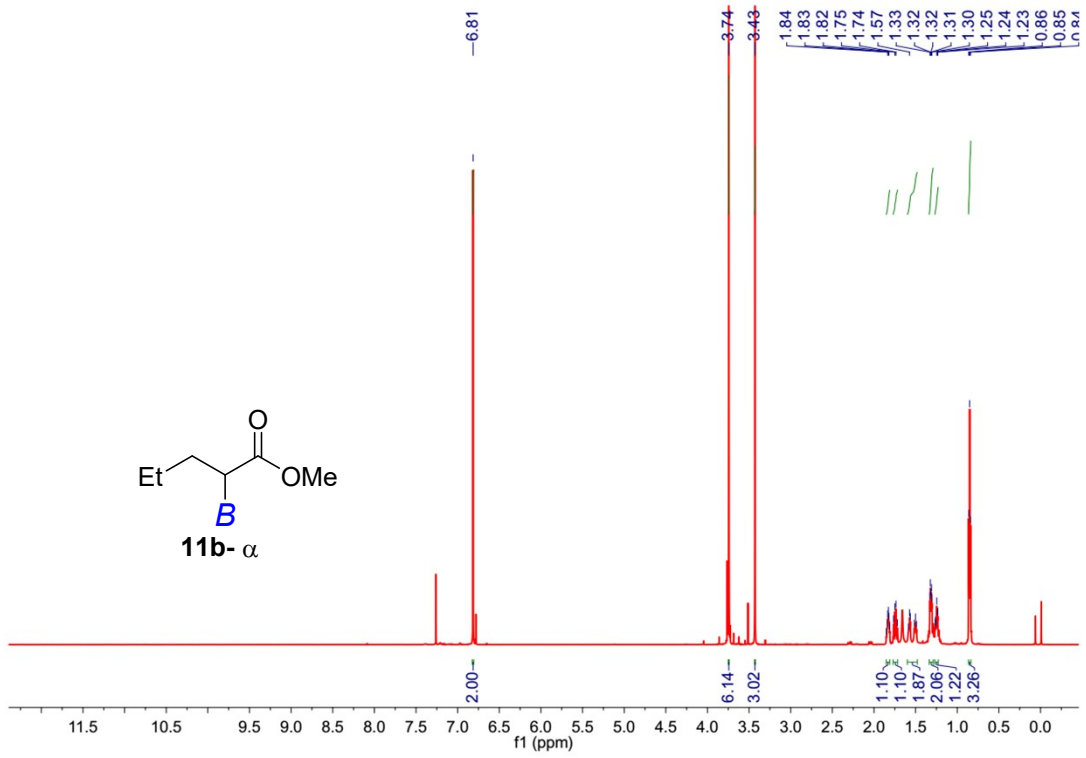
$^{13}\text{C NMR}$ (151 MHz, CDCl_3)



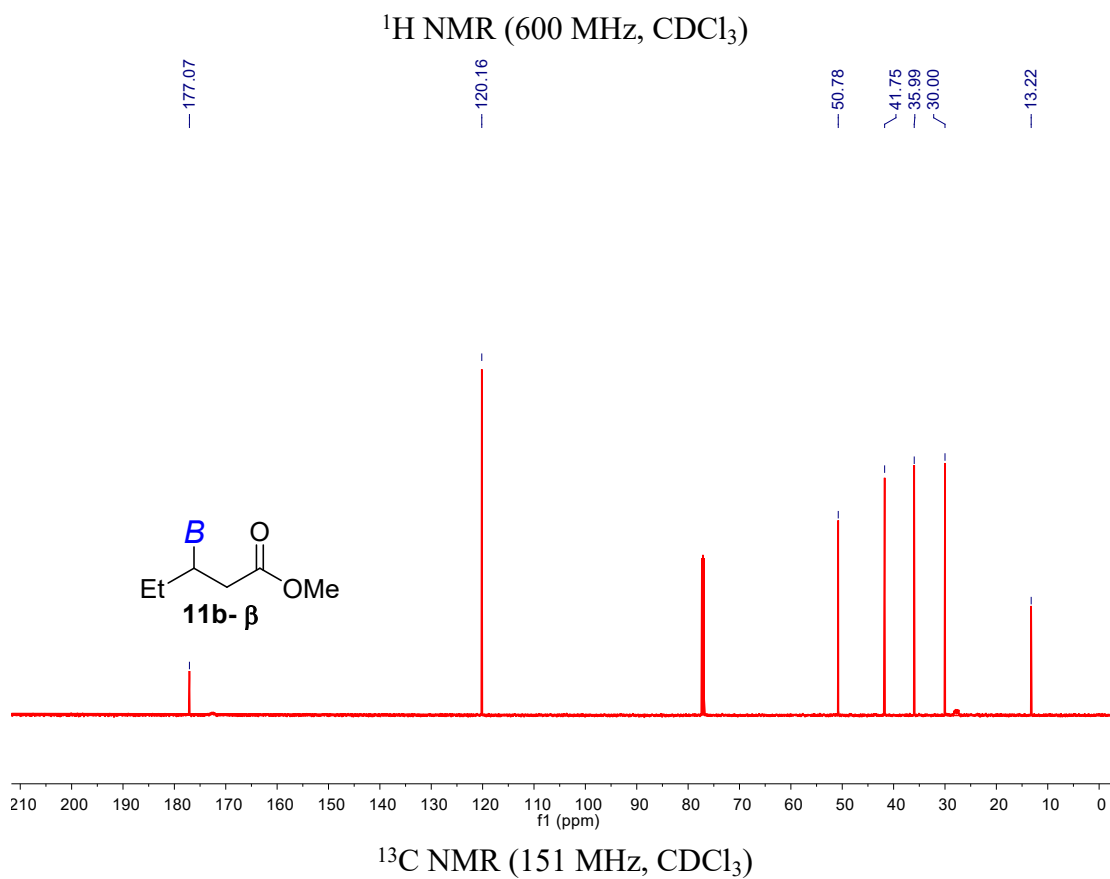
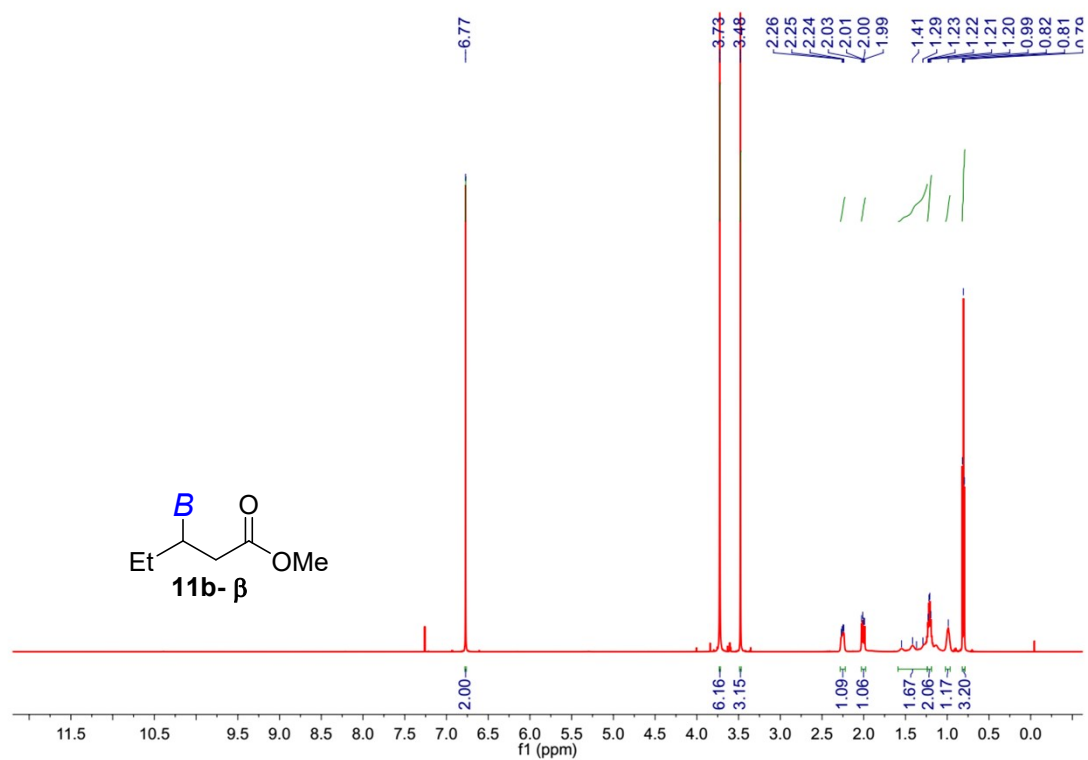
^{11}B NMR (193 MHz, CDCl_3)

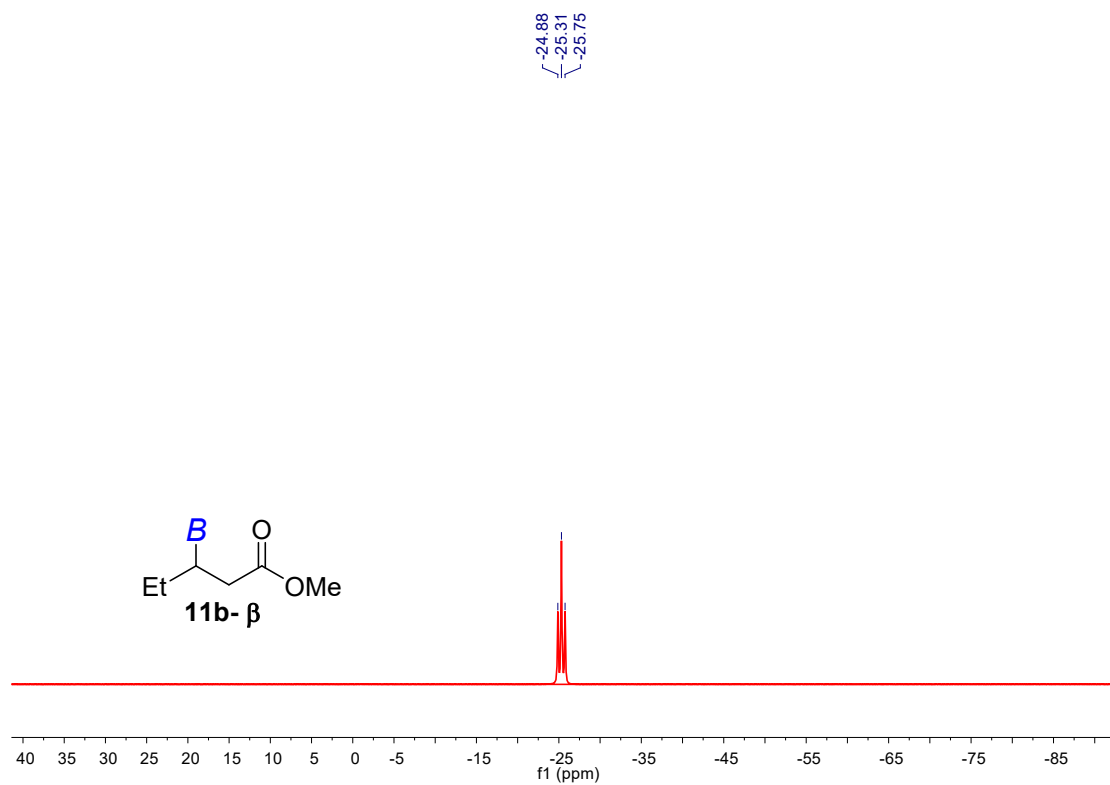


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

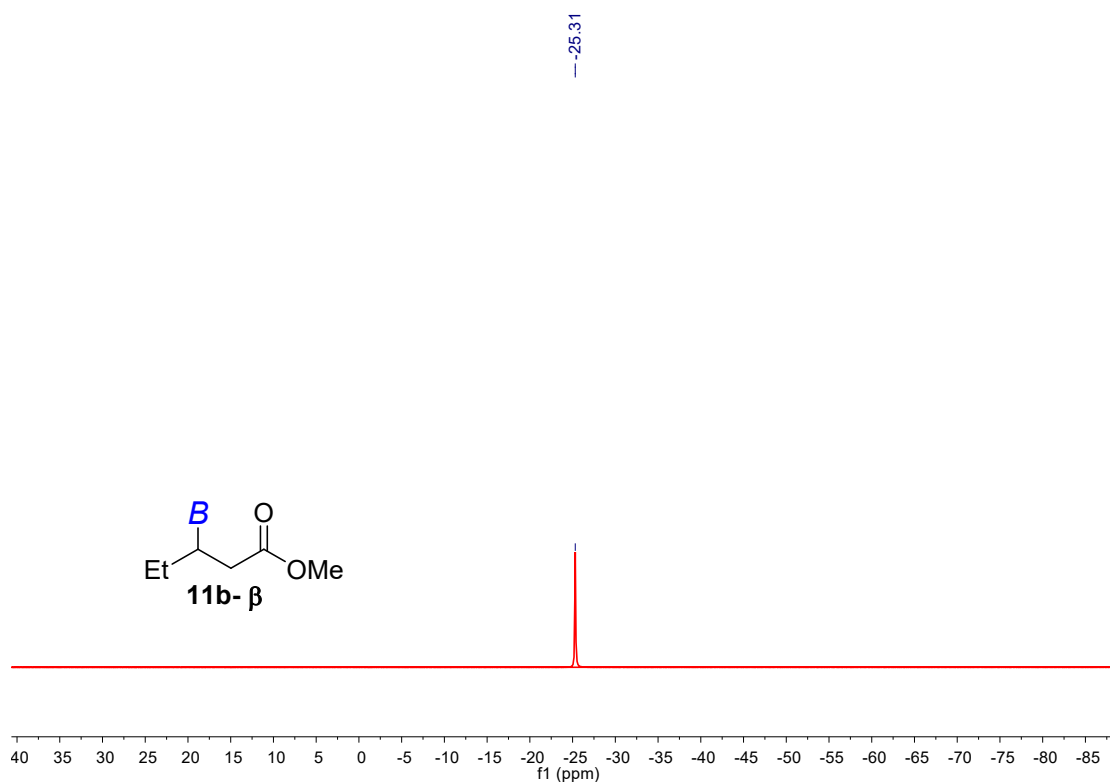


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

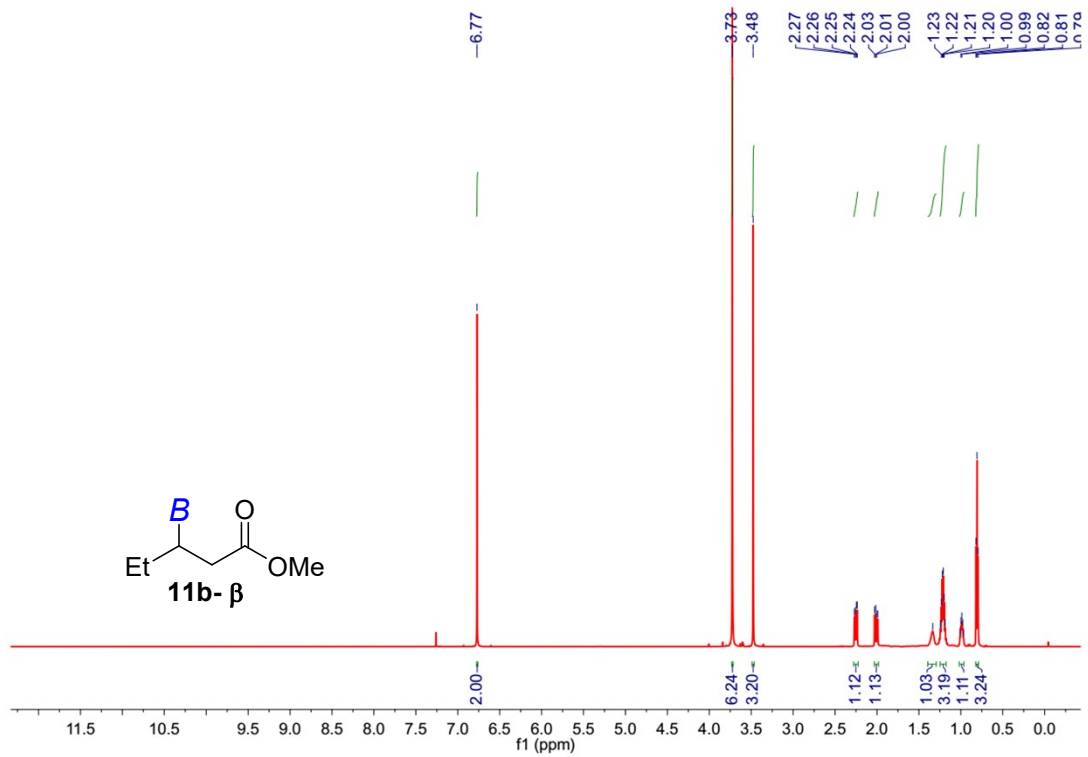




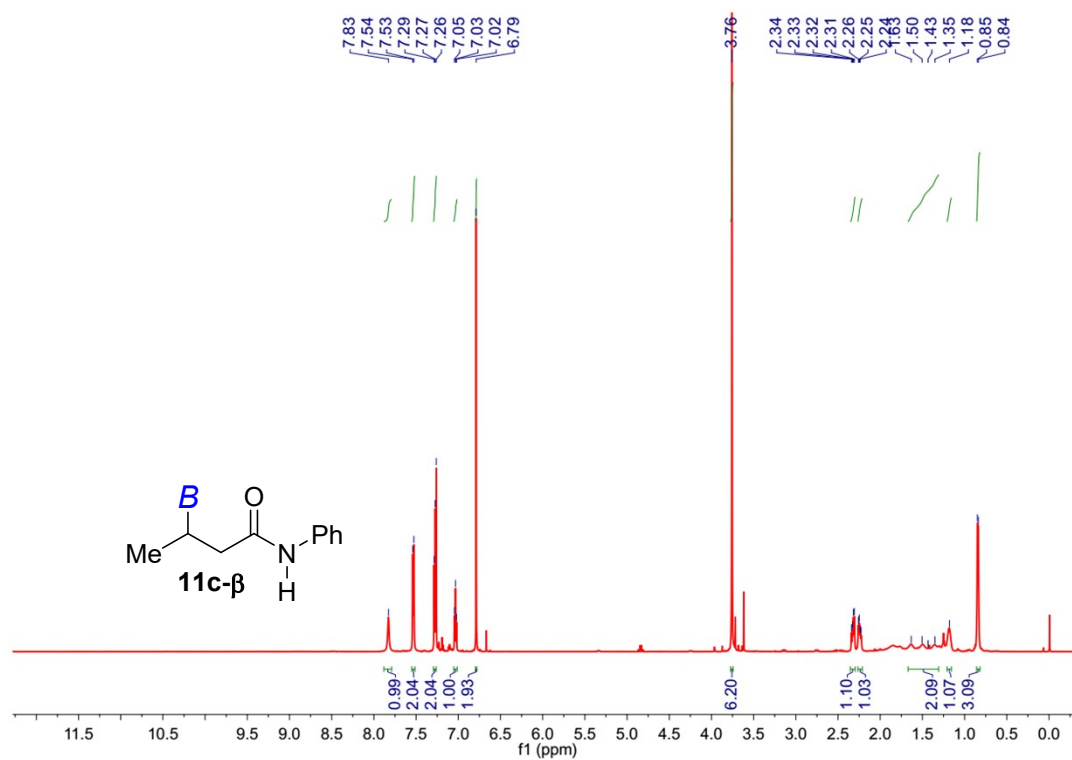
^{11}B NMR (193 MHz, CDCl_3)



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



¹H NMR (600 MHz, CDCl₃)

— 174.65

— 138.77

— 128.82

— 123.38

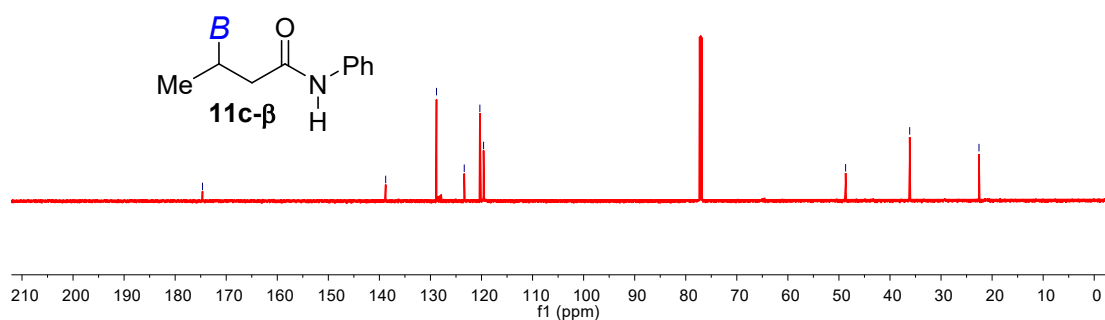
— 120.32

— 119.60

— 48.69

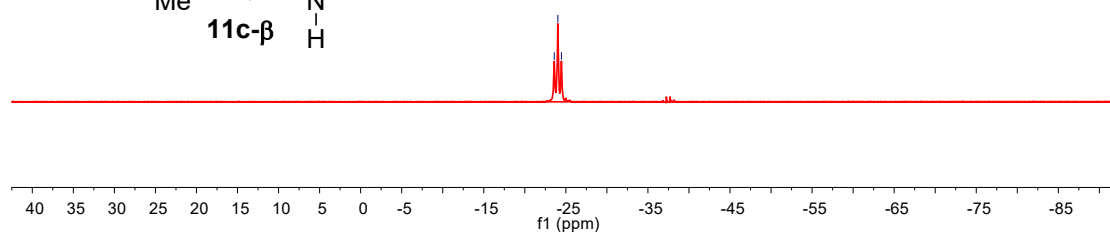
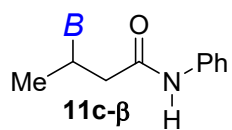
— 36.14

— 22.57



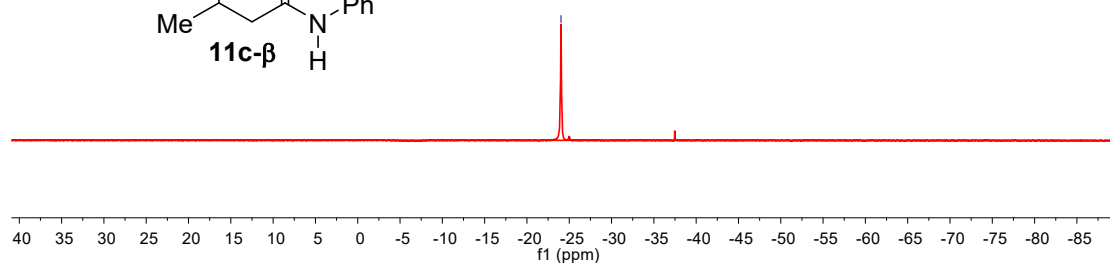
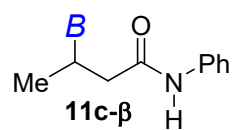
¹³C NMR (151 MHz, CDCl₃)

-23.59
-24.03
-24.46

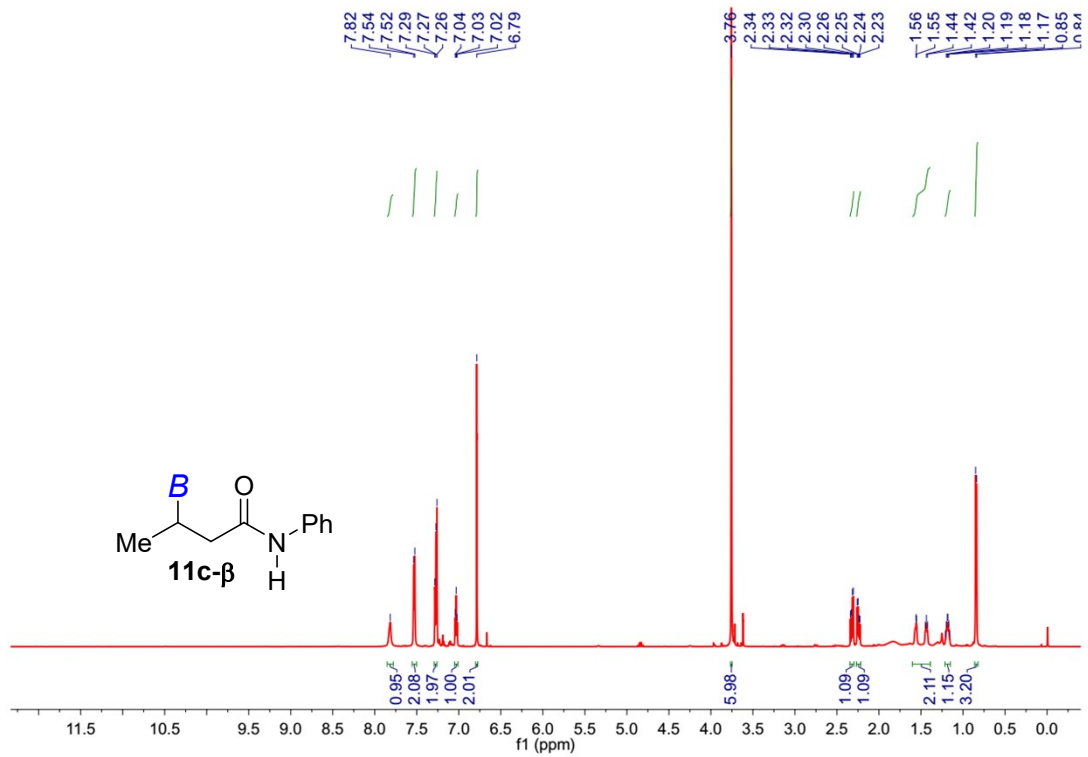


^{11}B NMR (193 MHz, CDCl_3)

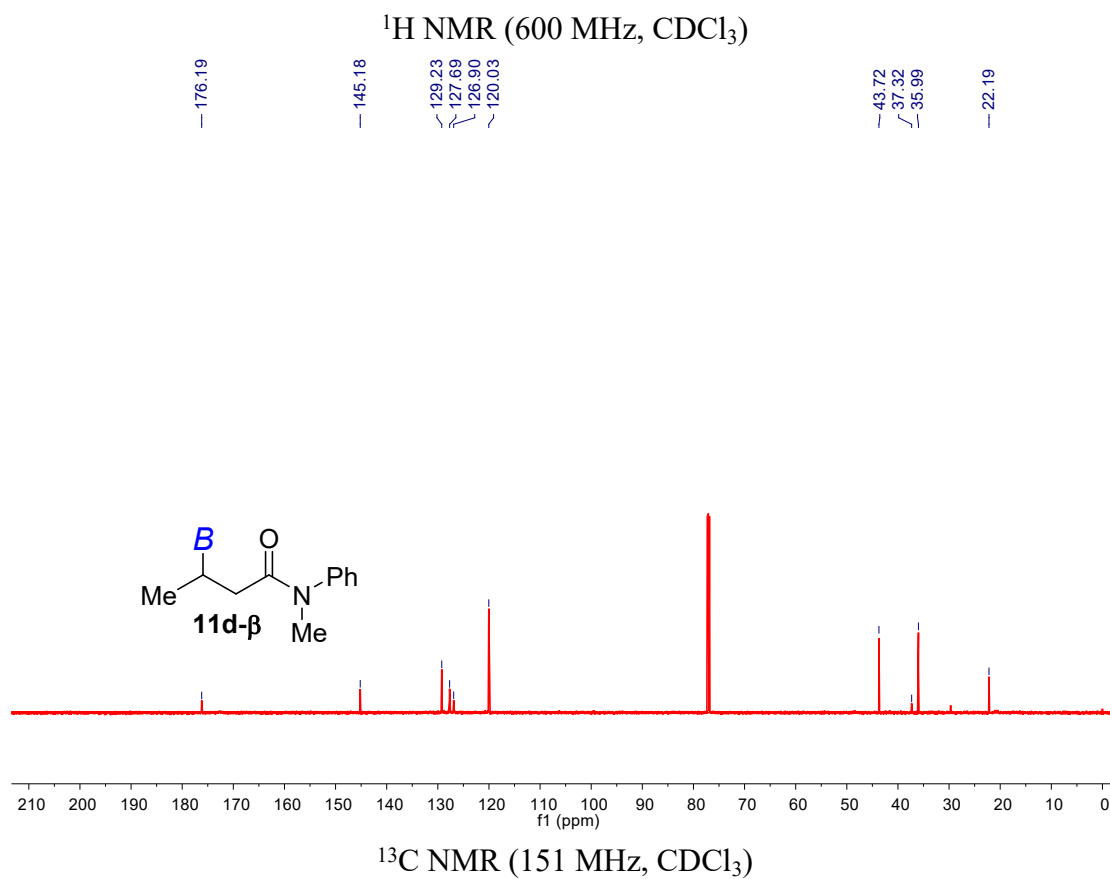
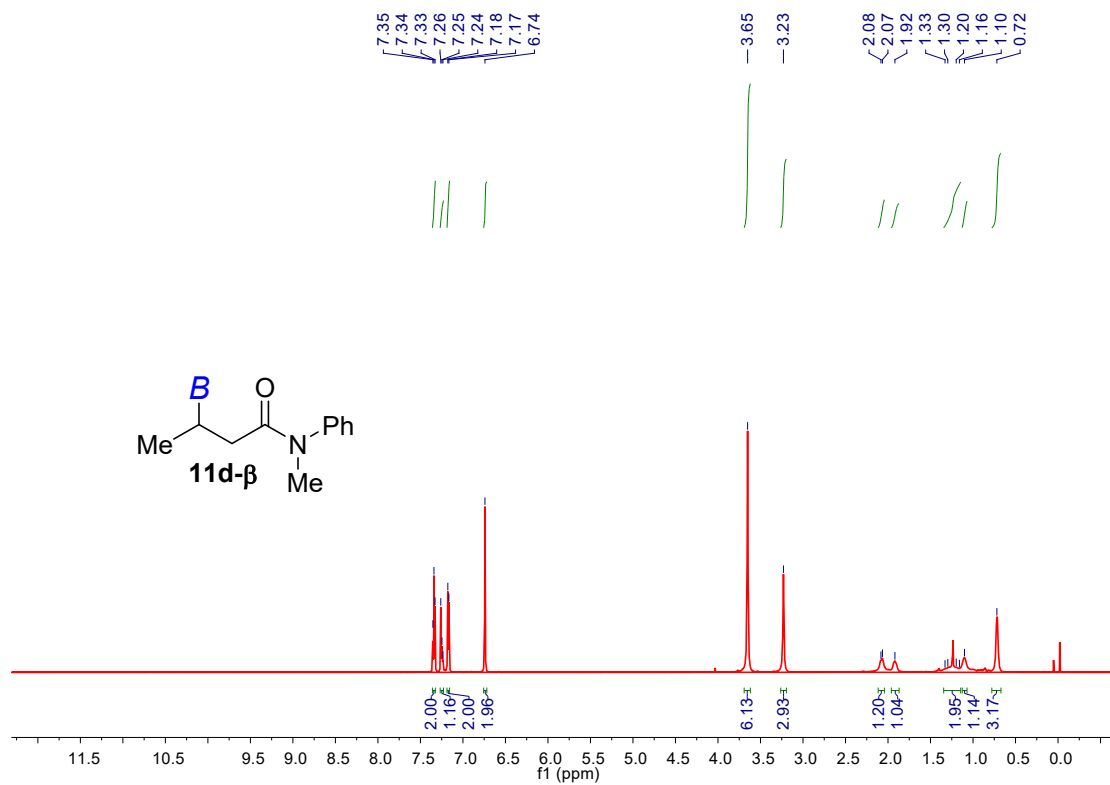
-24.03

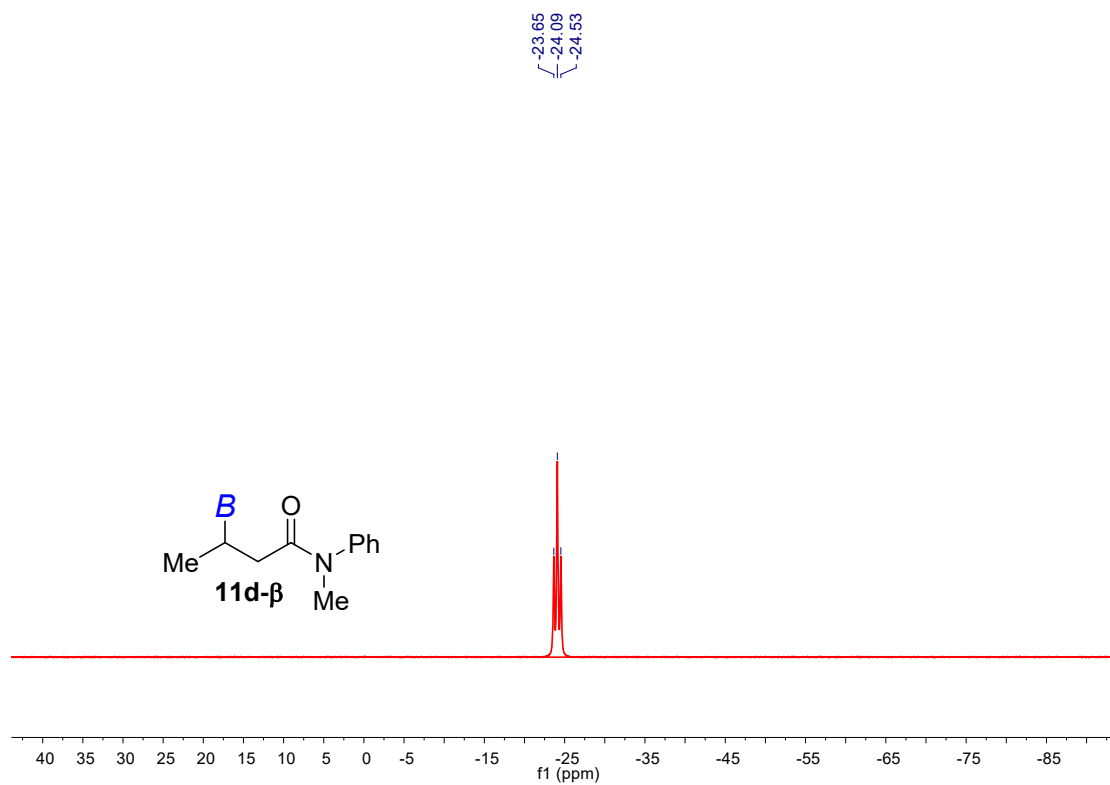


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

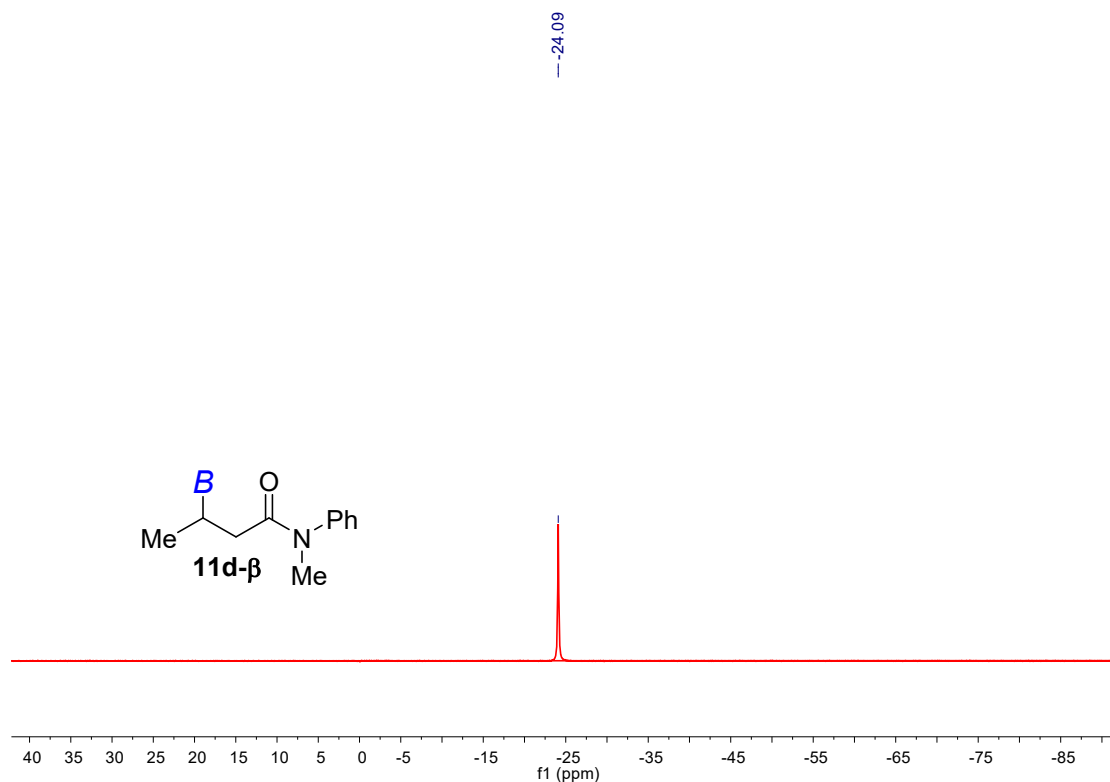


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

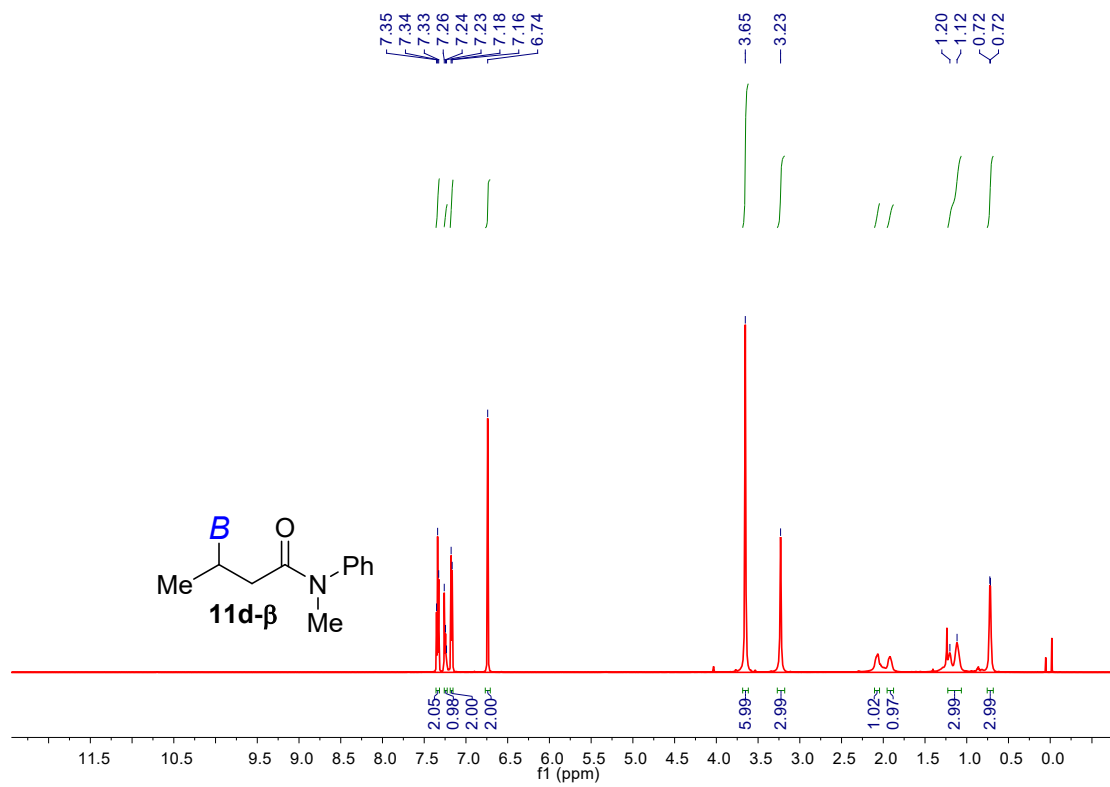




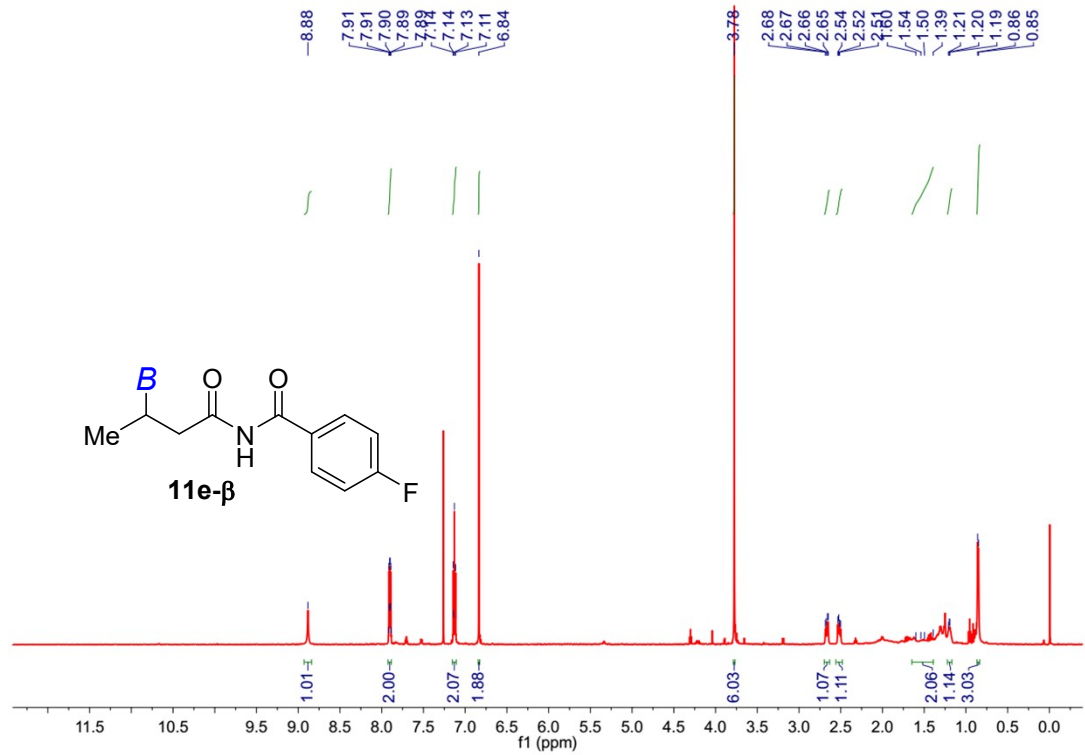
^{11}B NMR (193 MHz, CDCl_3)



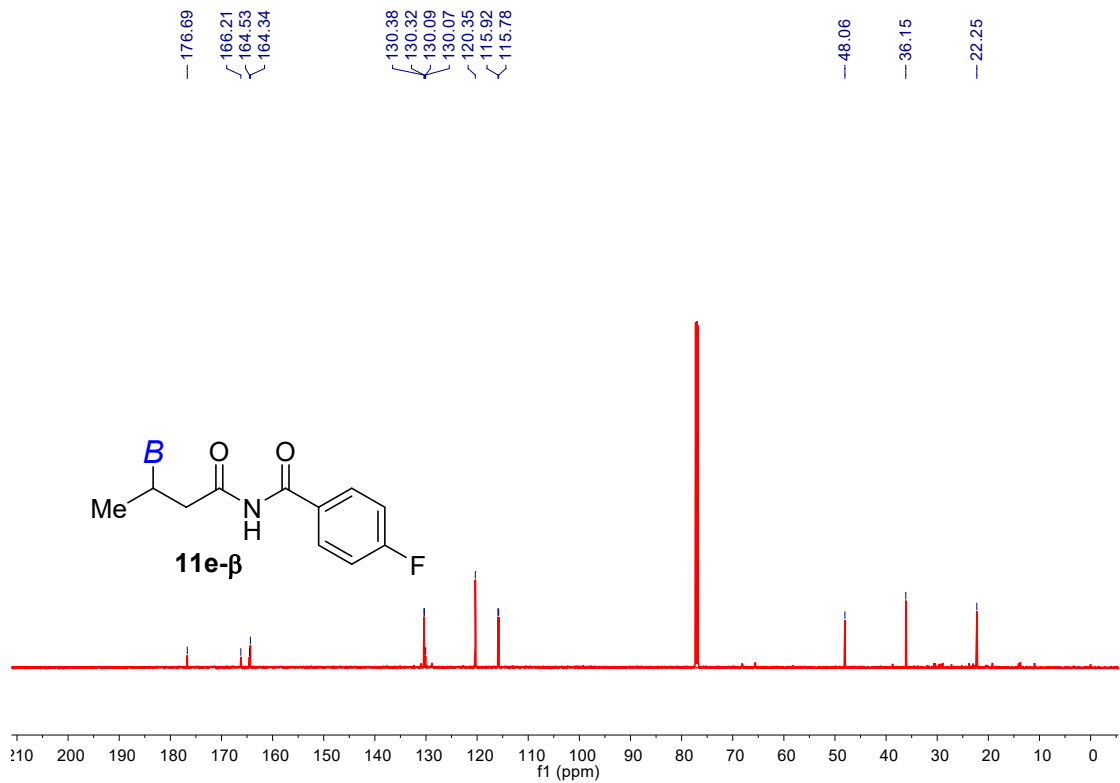
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



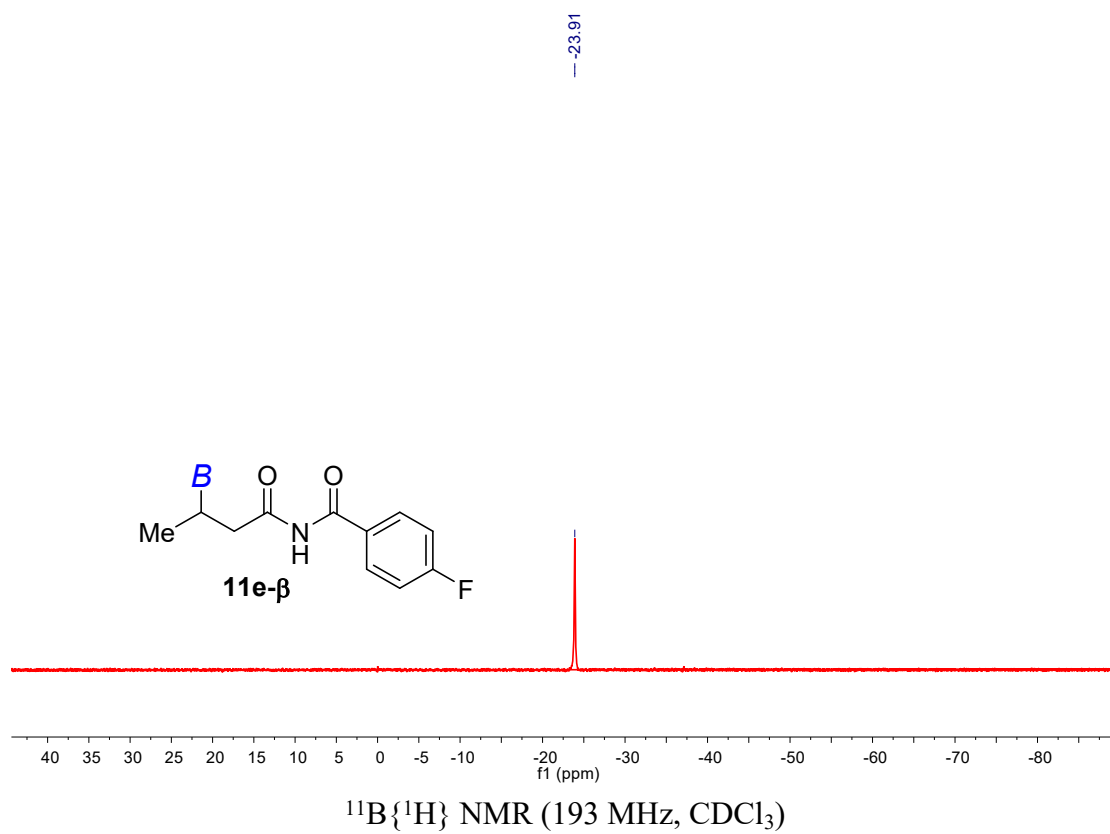
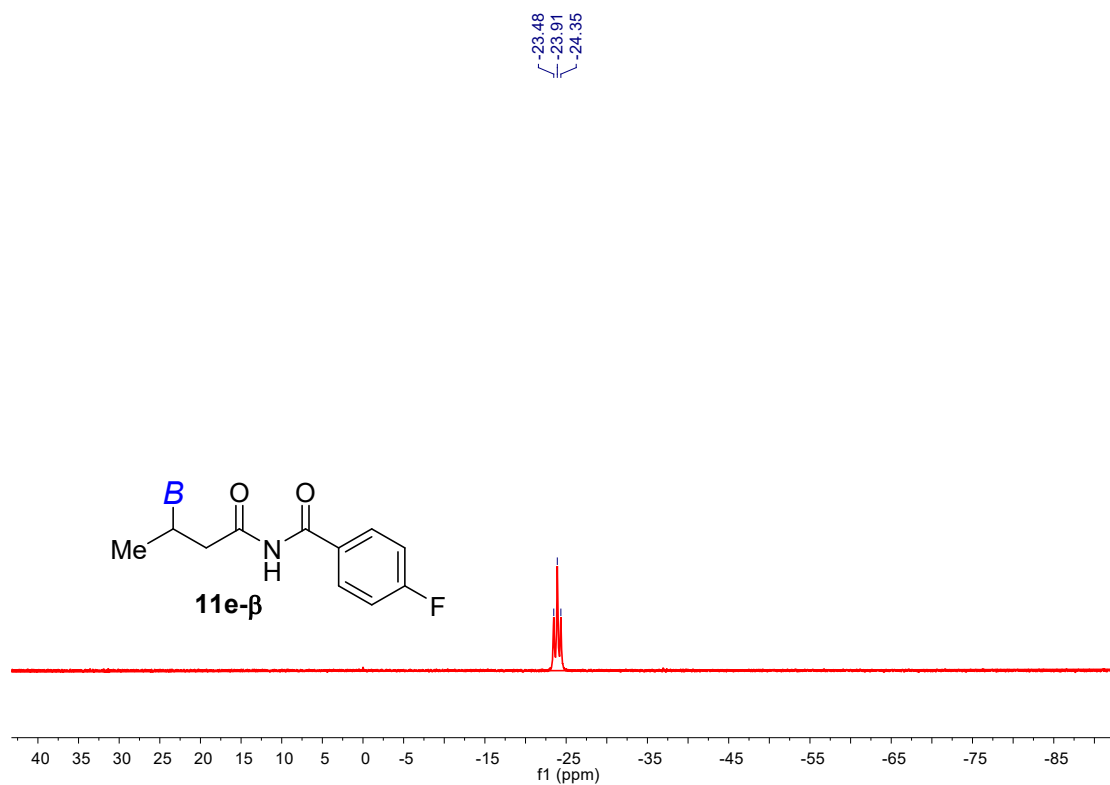
¹H{¹¹B} NMR (600 MHz, CDCl₃)

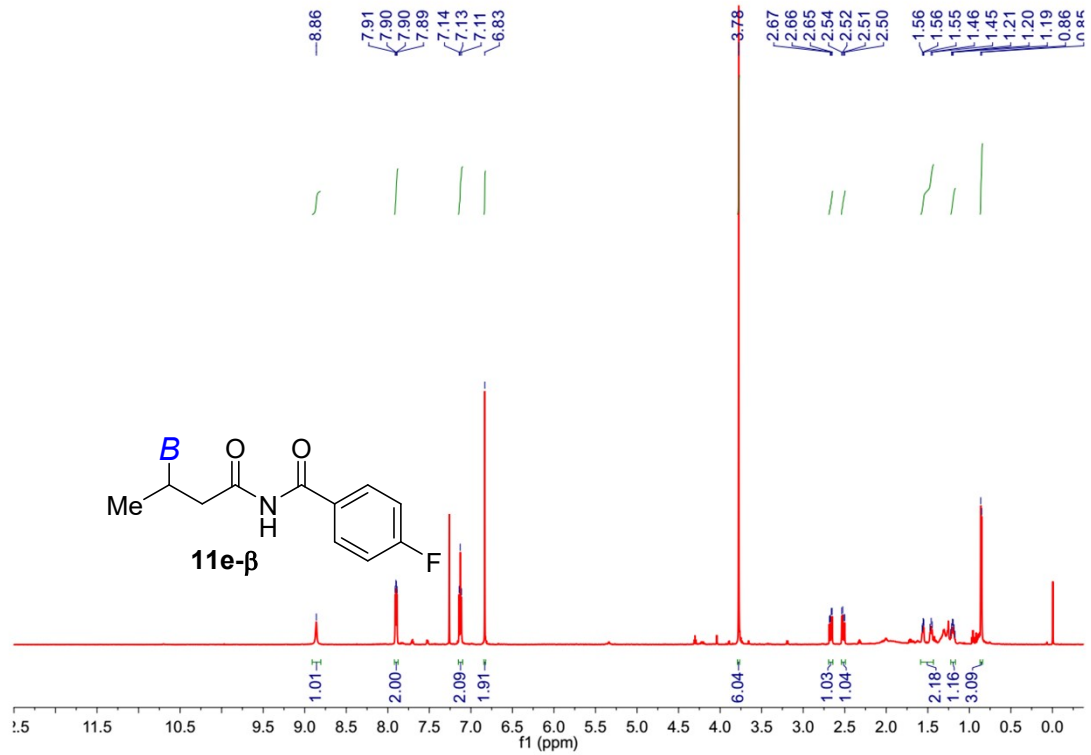


¹H NMR (600 MHz, CDCl₃)

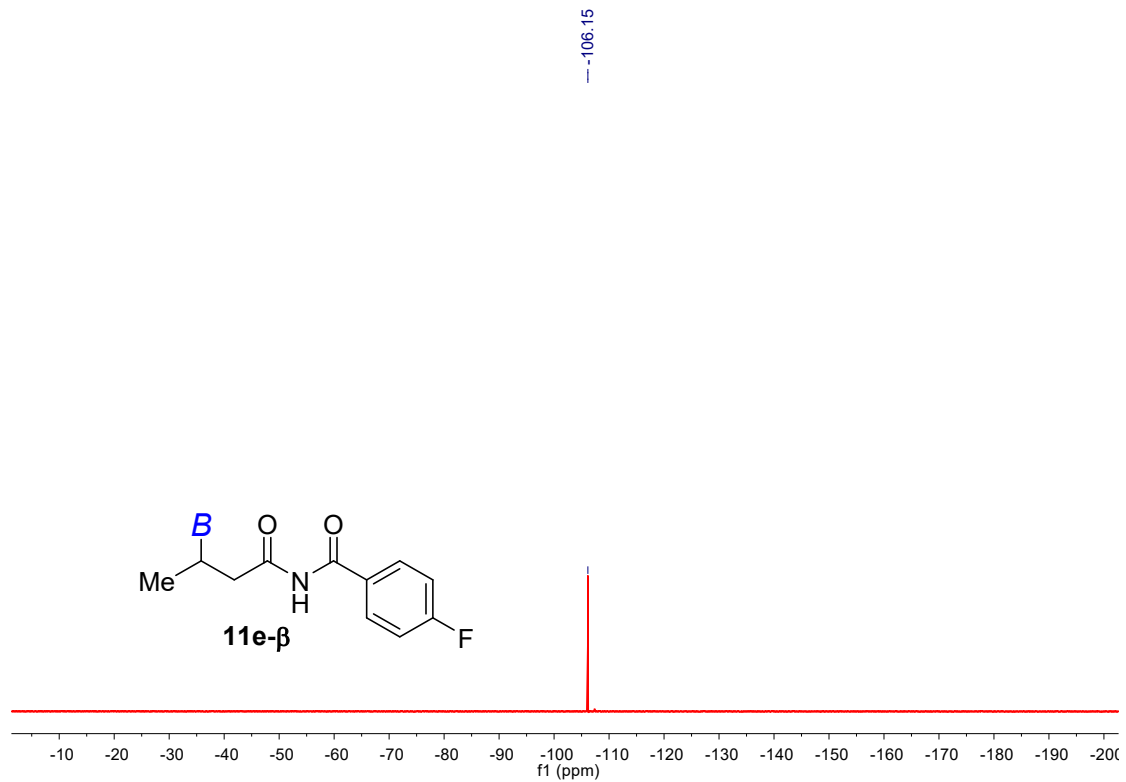


¹³C NMR (151 MHz, CDCl₃)

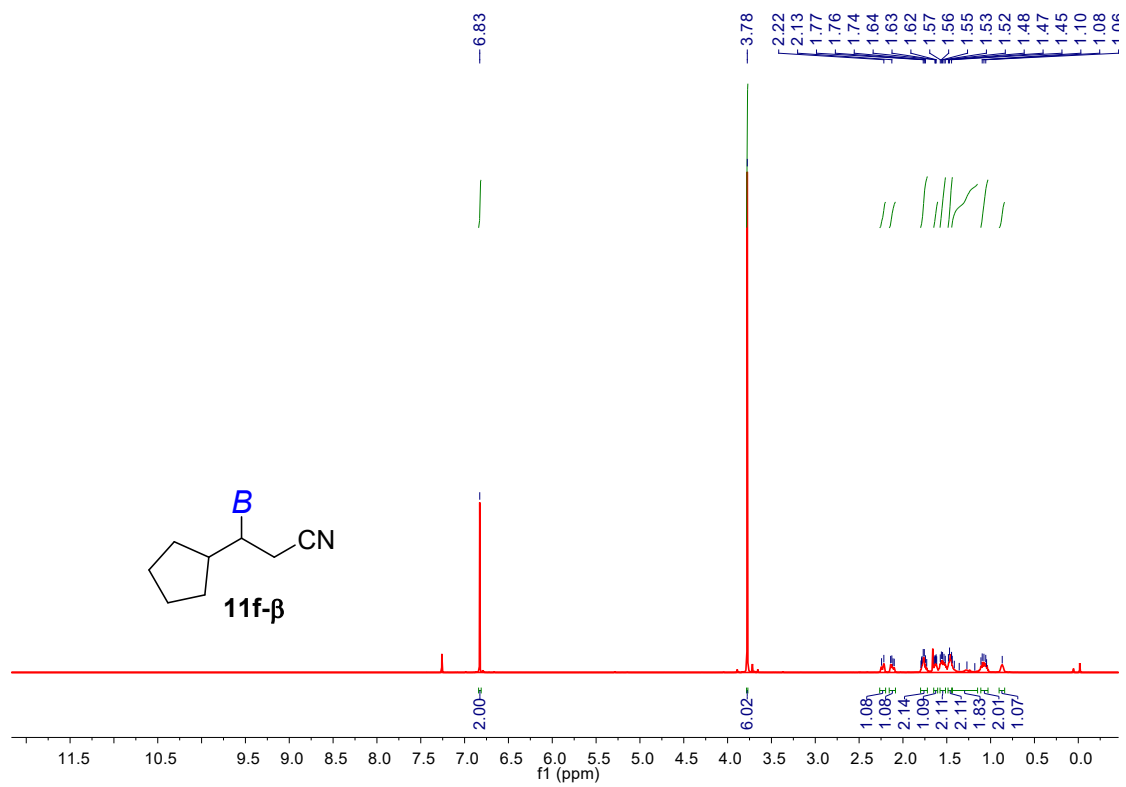




¹H{¹B} NMR (600 MHz, CDCl₃)



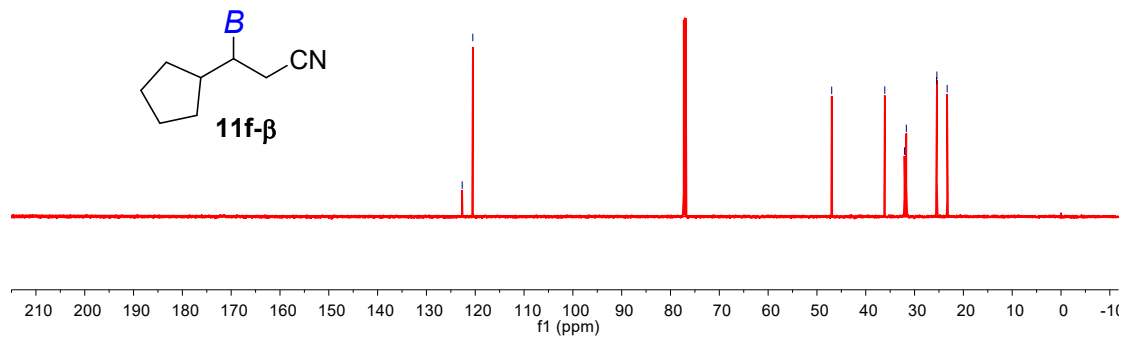
¹⁹F NMR (565 MHz, CDCl₃)



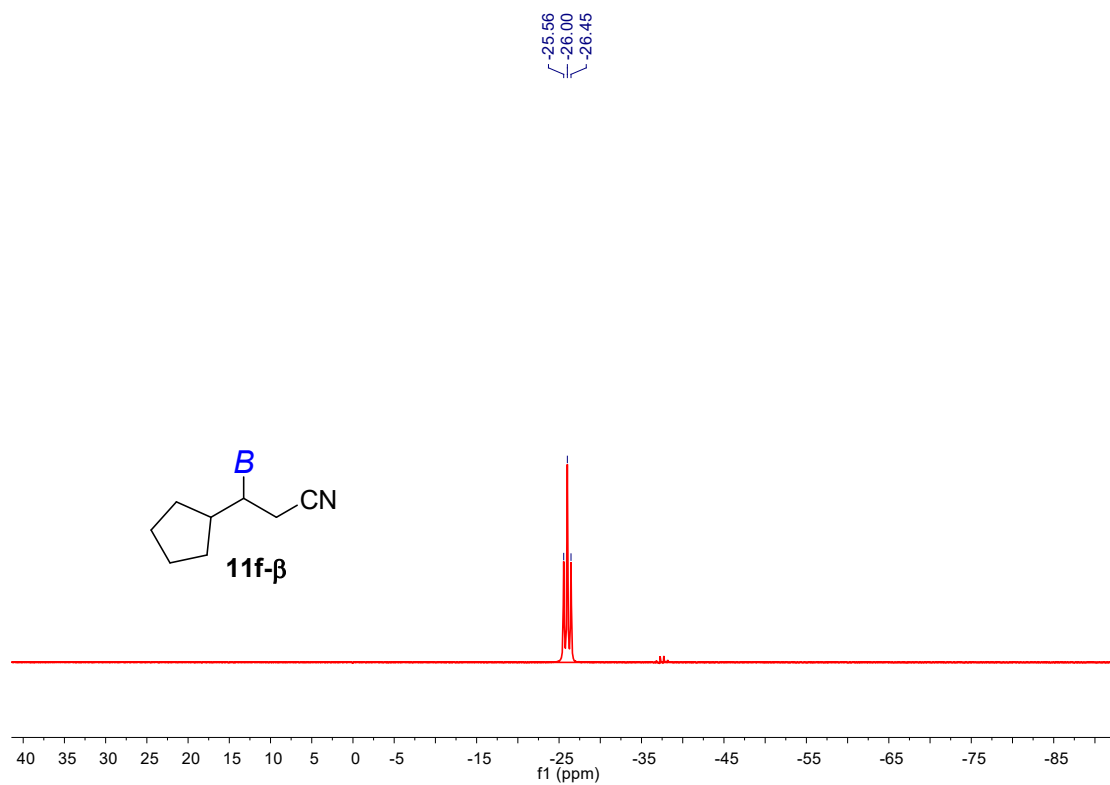
¹H NMR (600 MHz, CDCl₃)

122.67
120.50

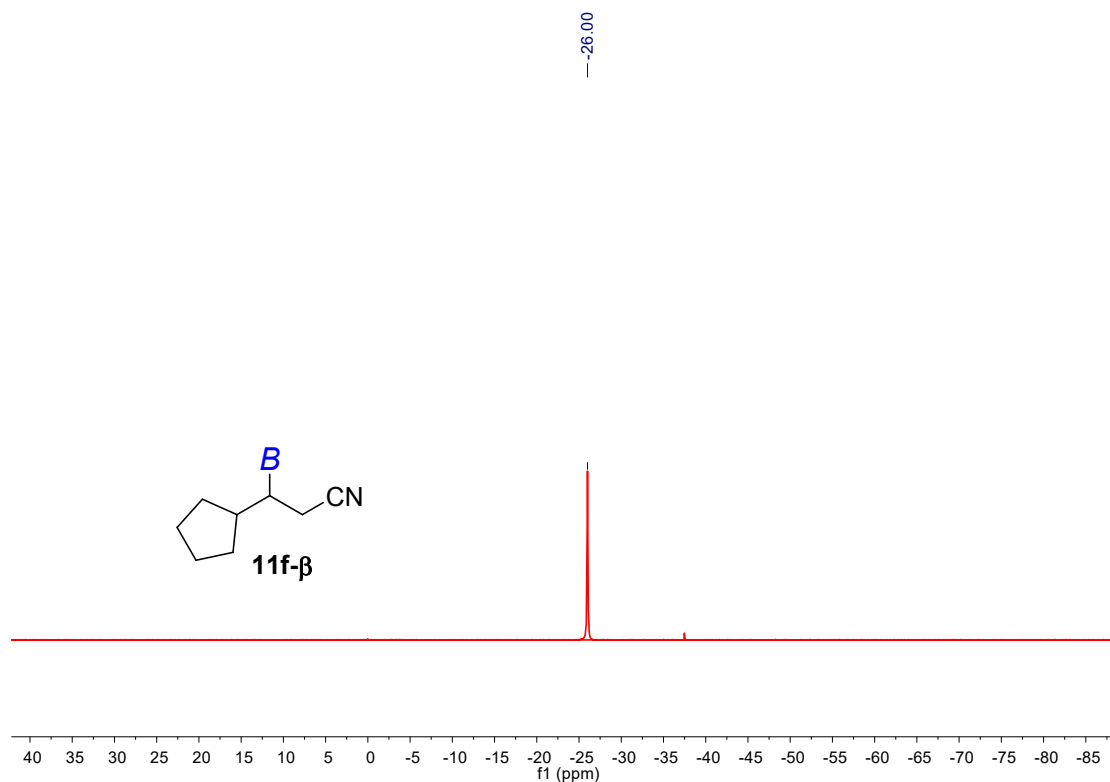
46.98
36.10
32.07
31.68
25.48
25.43
23.33



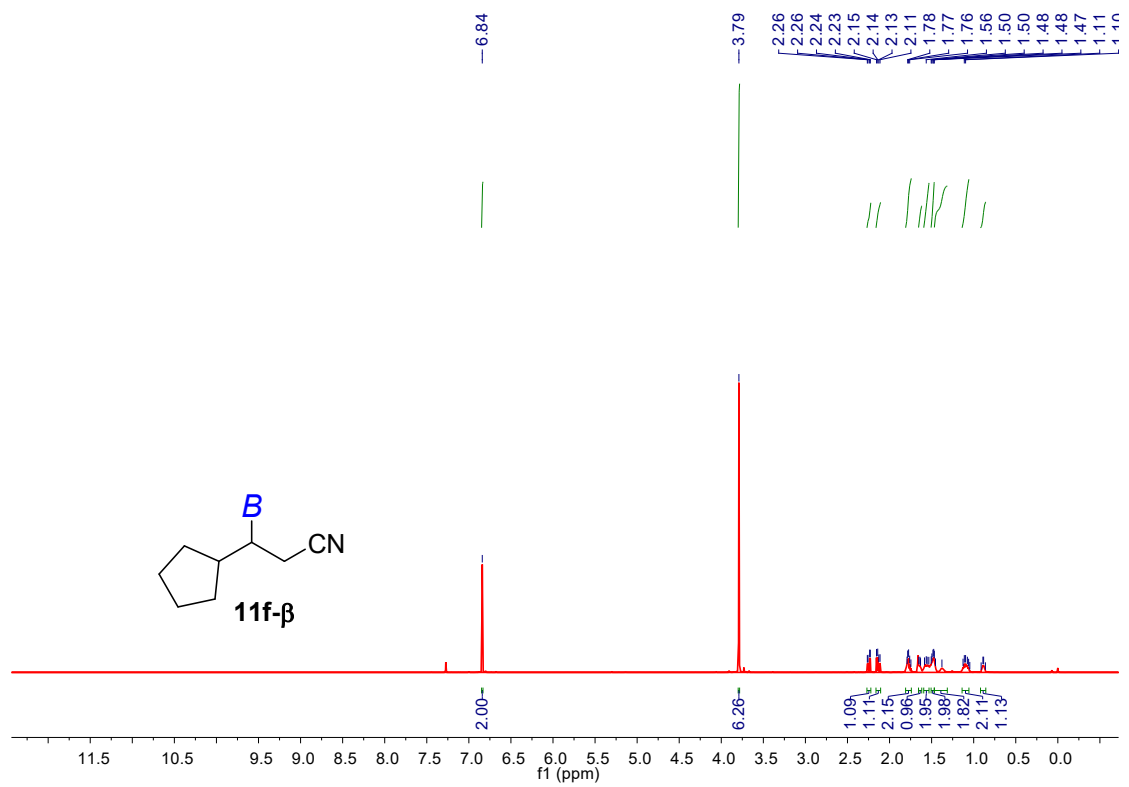
¹³C NMR (151 MHz, CDCl₃)



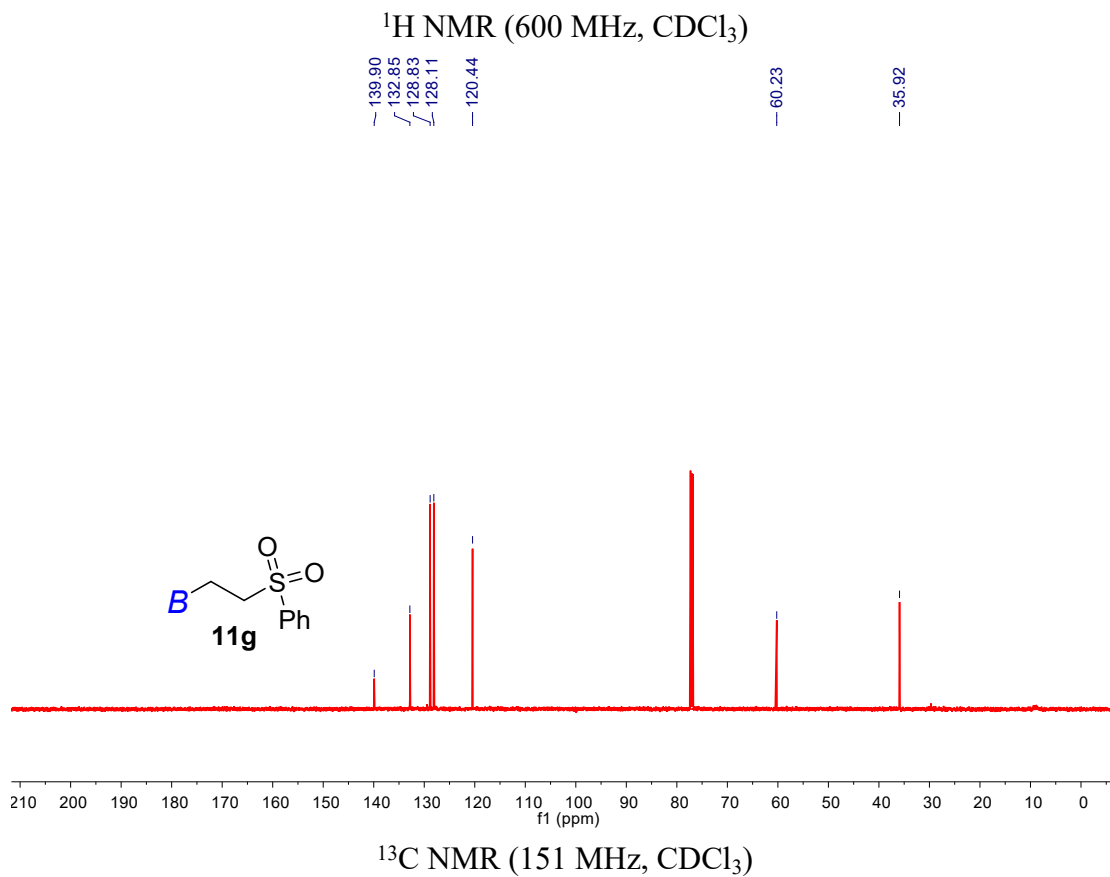
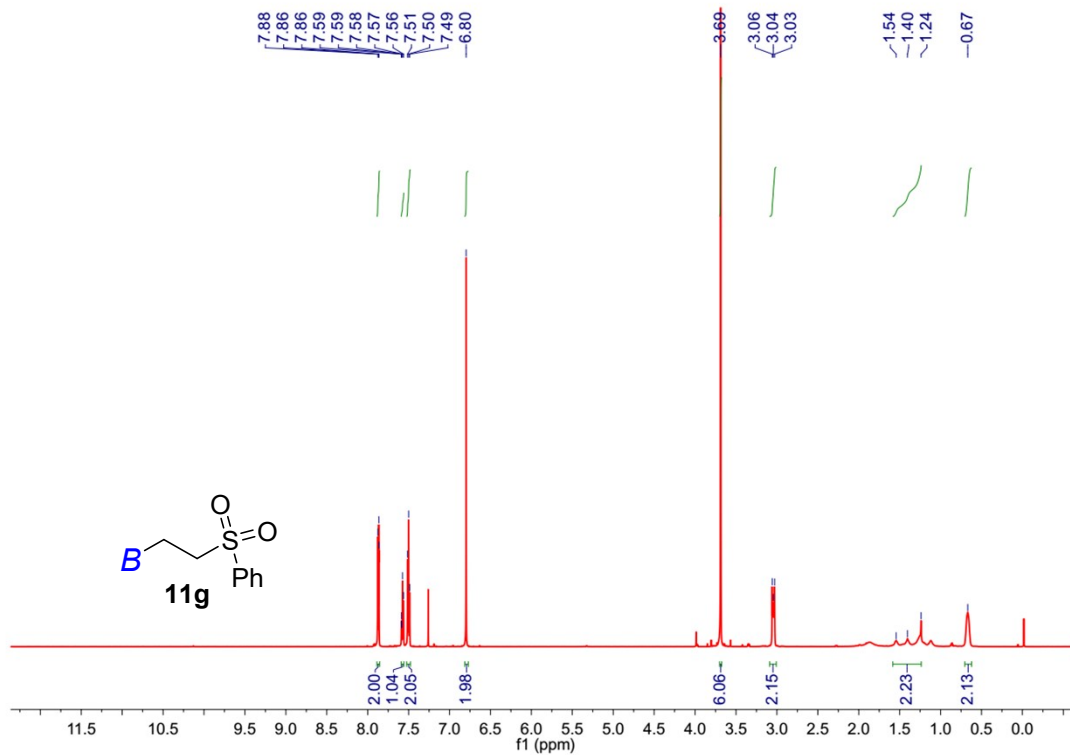
^{11}B NMR (193 MHz, CDCl_3)

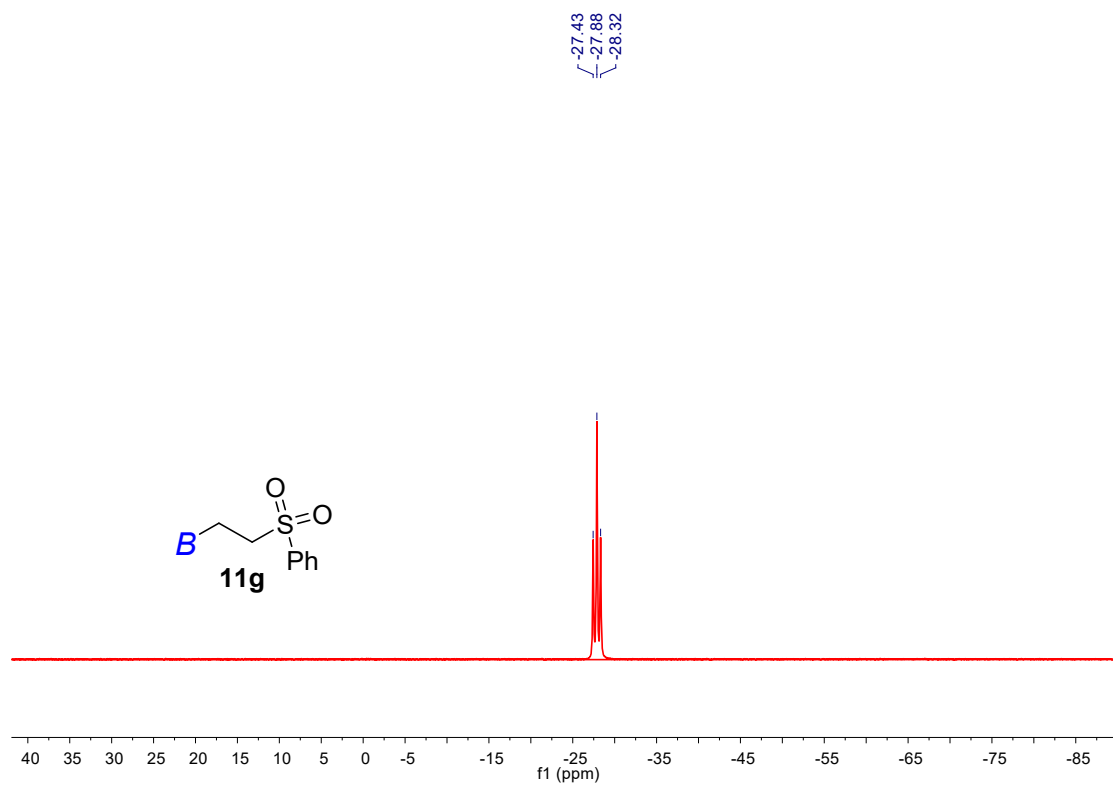


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

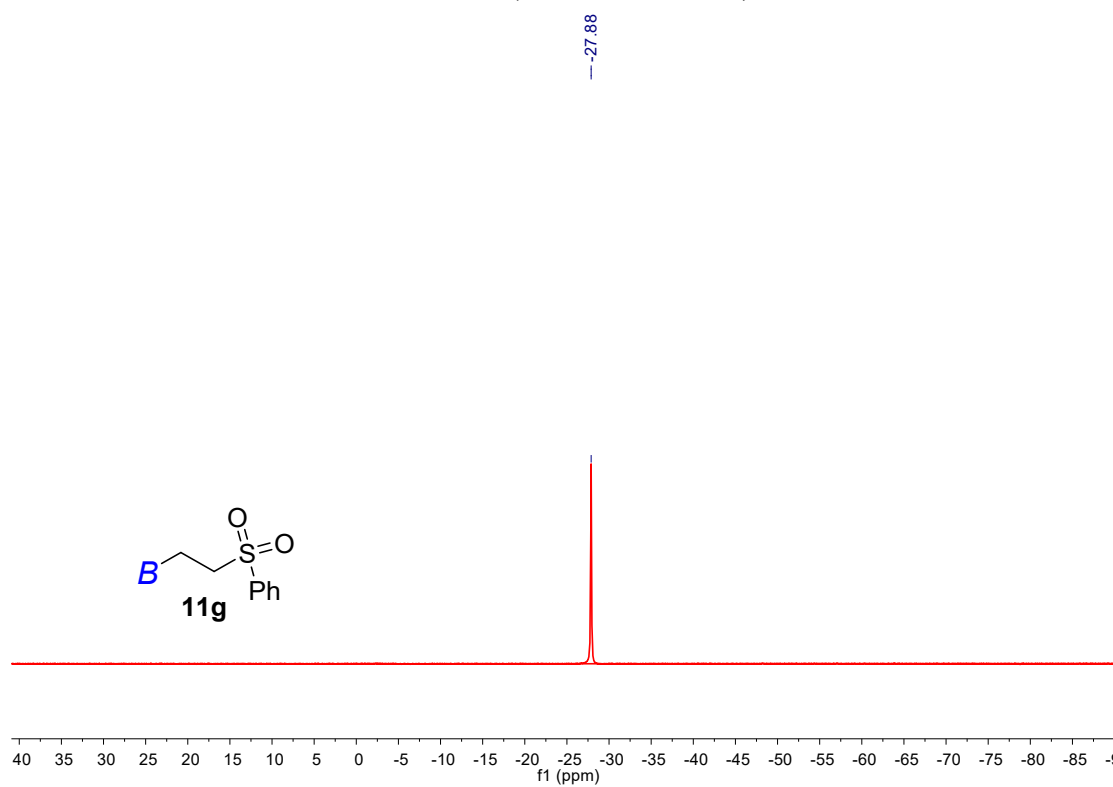


¹H{¹¹B} NMR (600 MHz, CDCl₃)

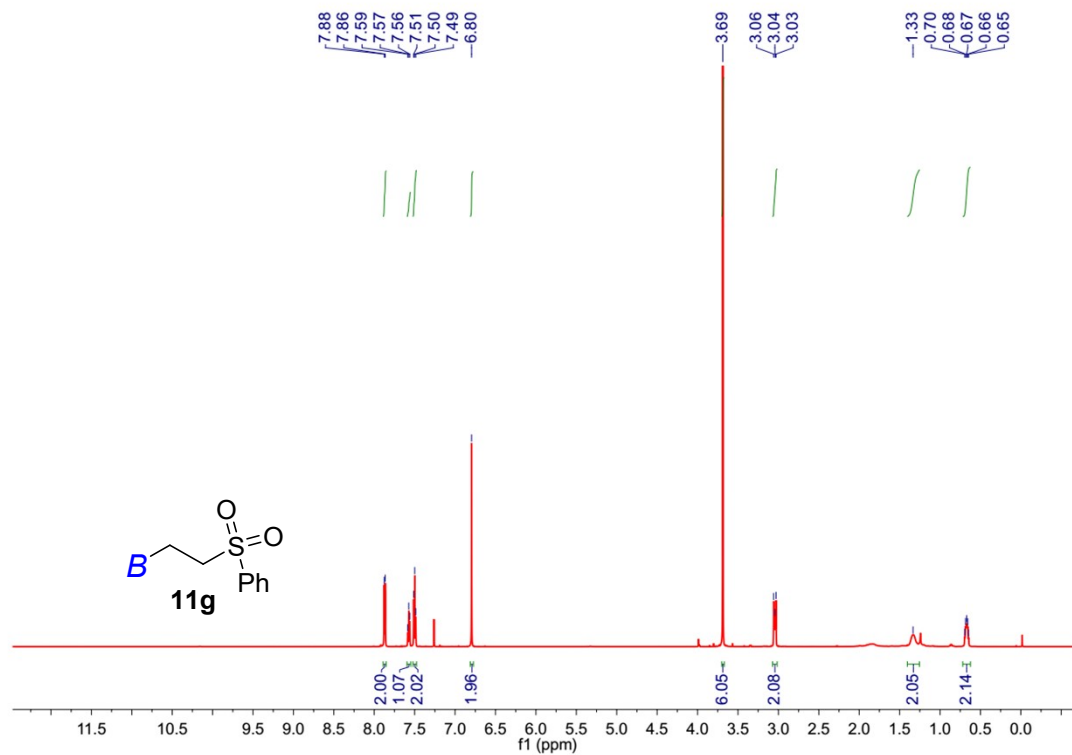




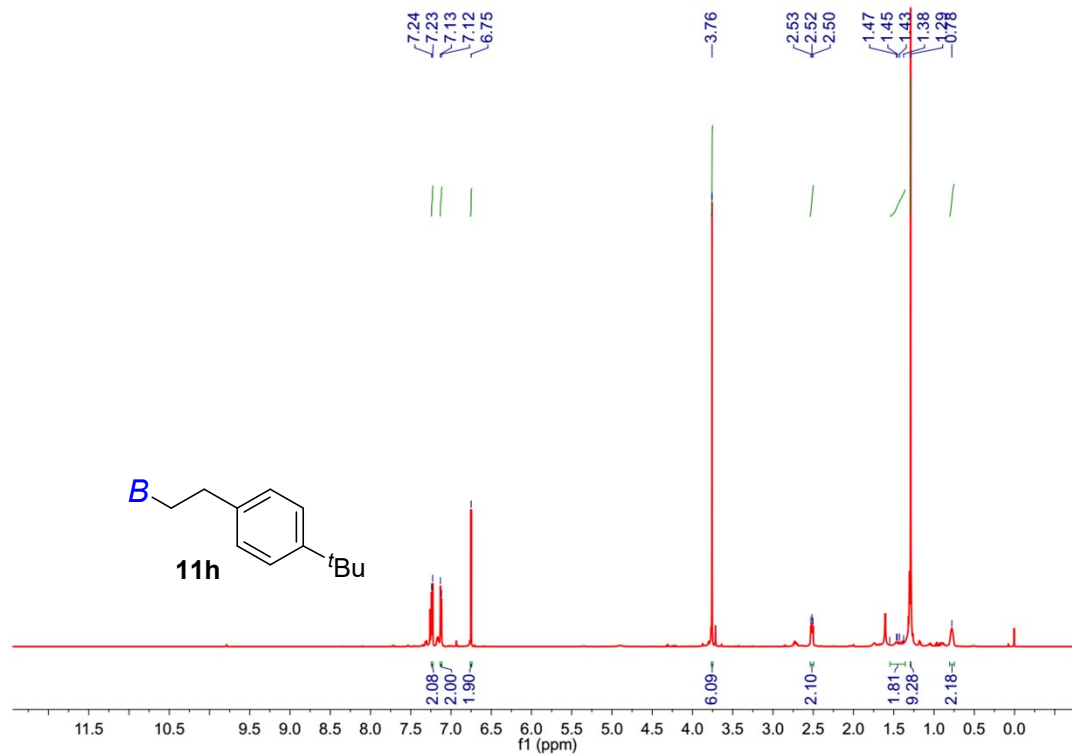
^{11}B NMR (193 MHz, CDCl_3)



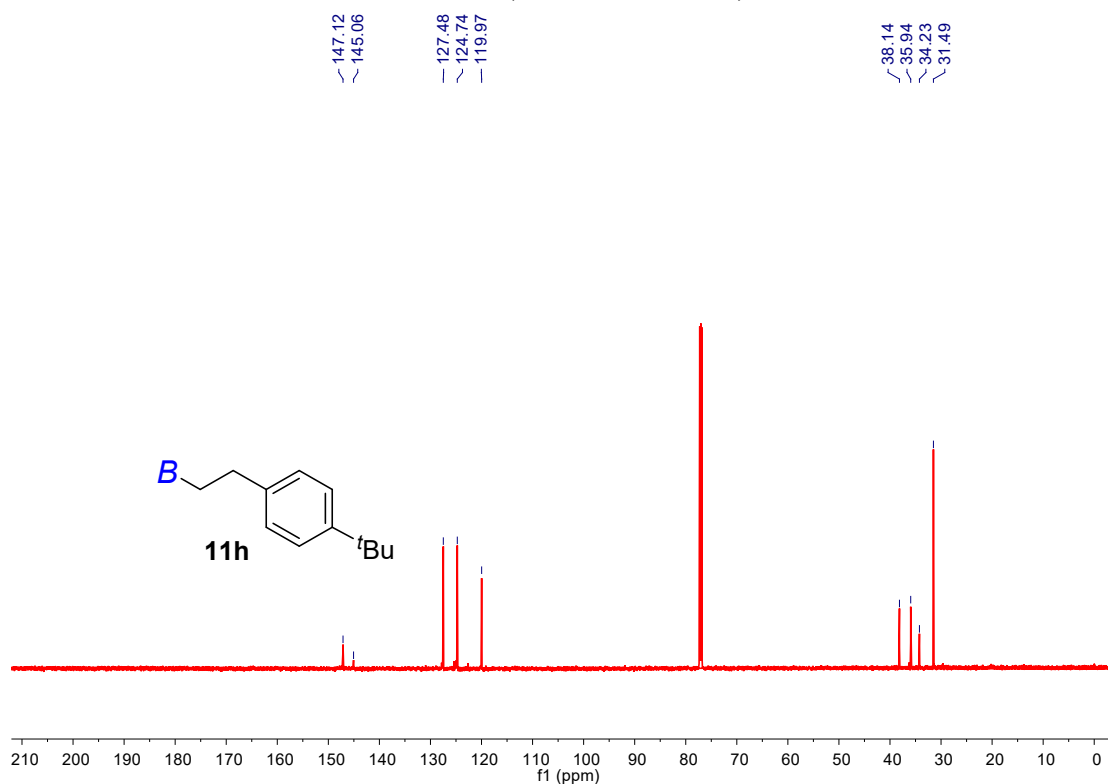
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



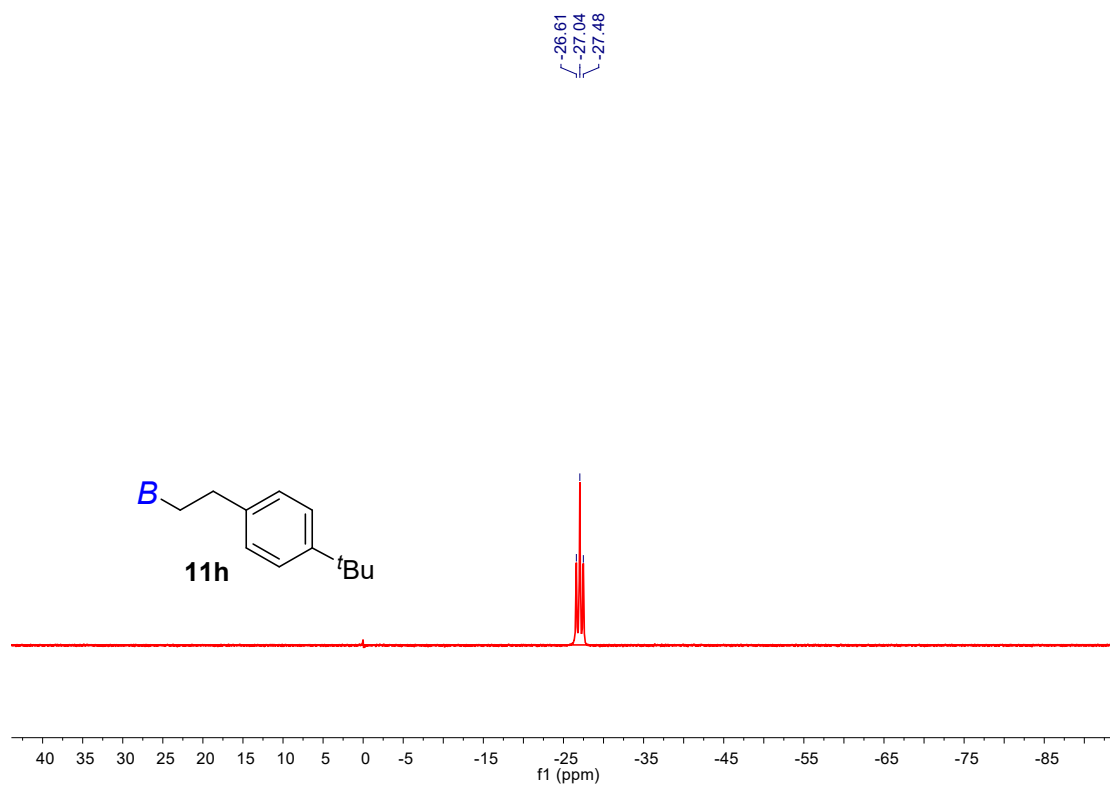
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



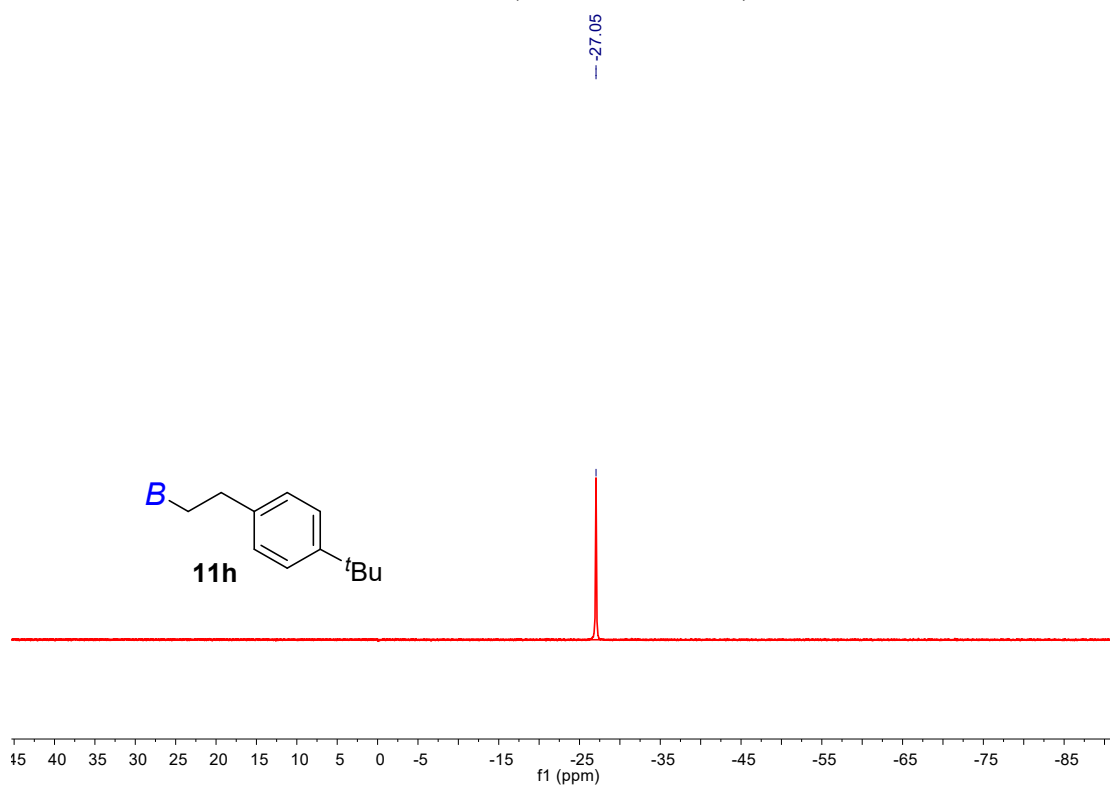
^1H NMR (600 MHz, CDCl_3)



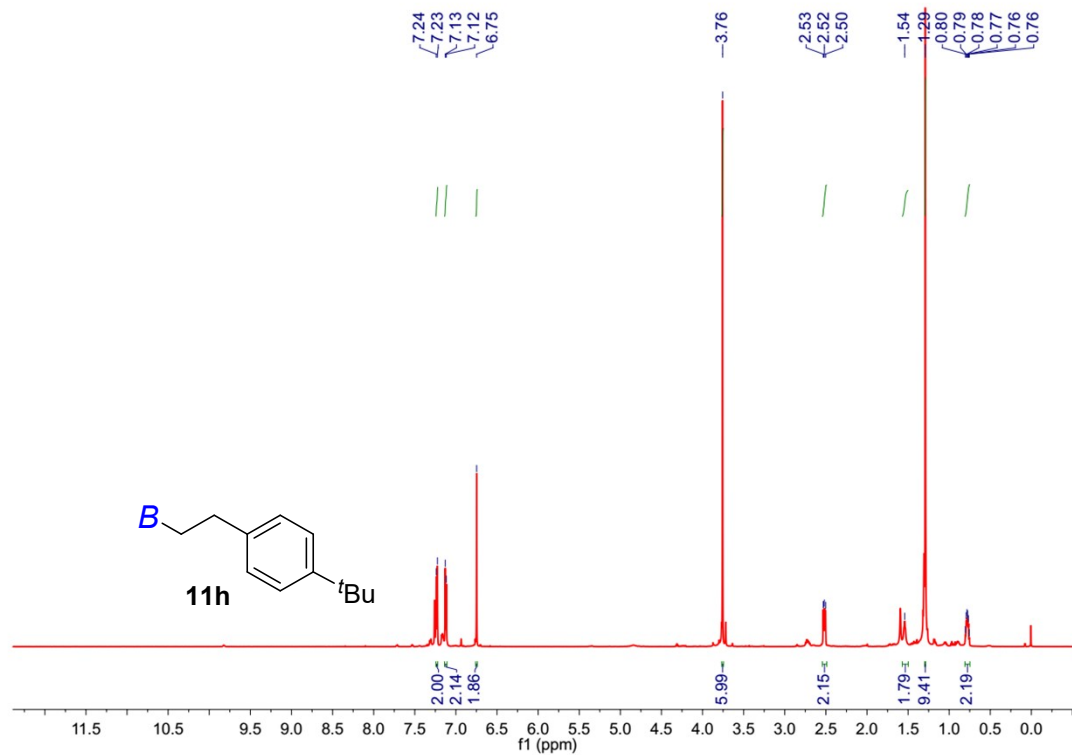
^{13}C NMR (151 MHz, CDCl_3)



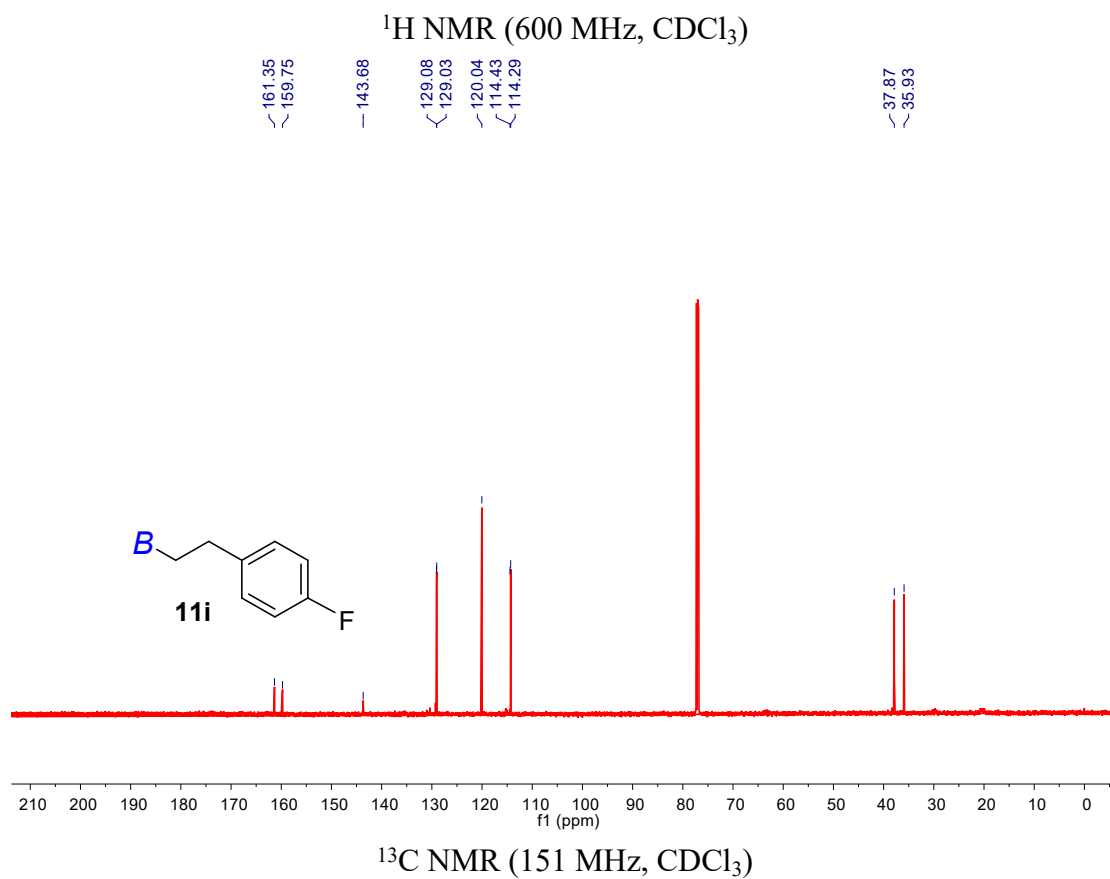
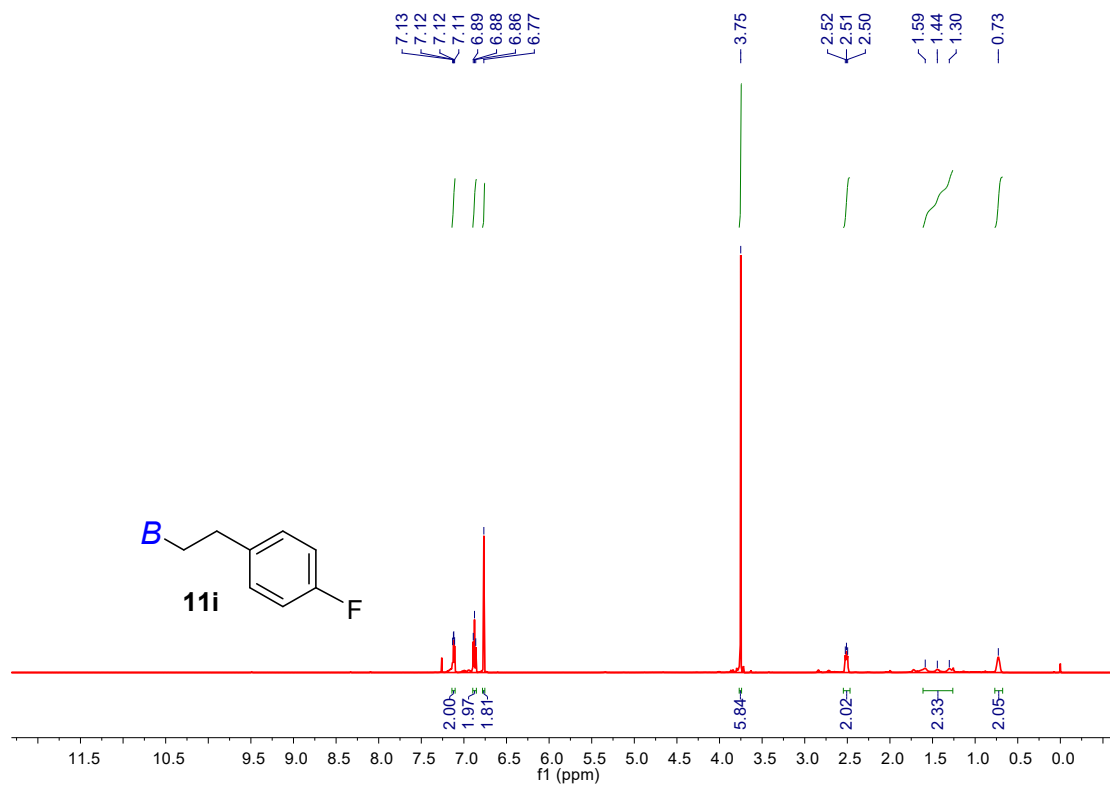
^{11}B NMR (193 MHz, CDCl_3)



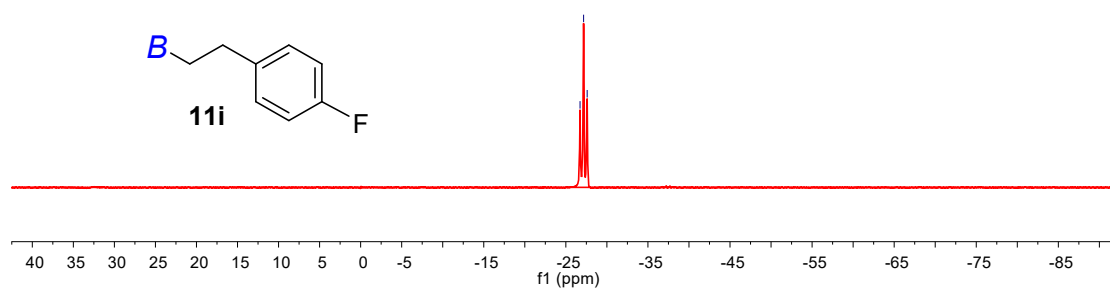
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

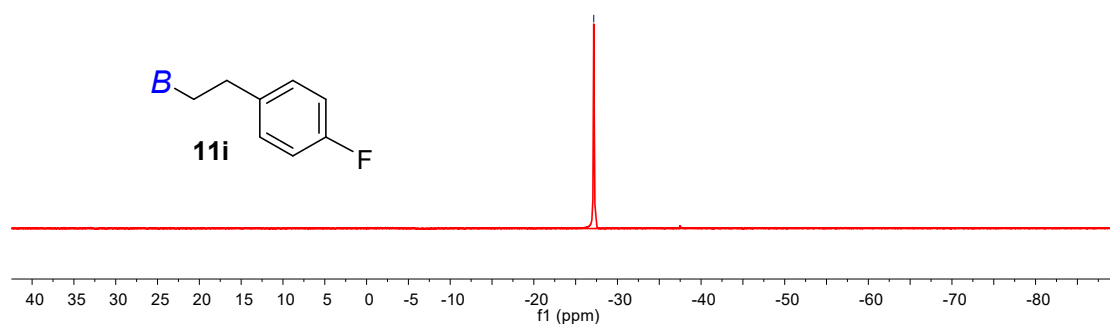


-26.73
-27.16
-27.60

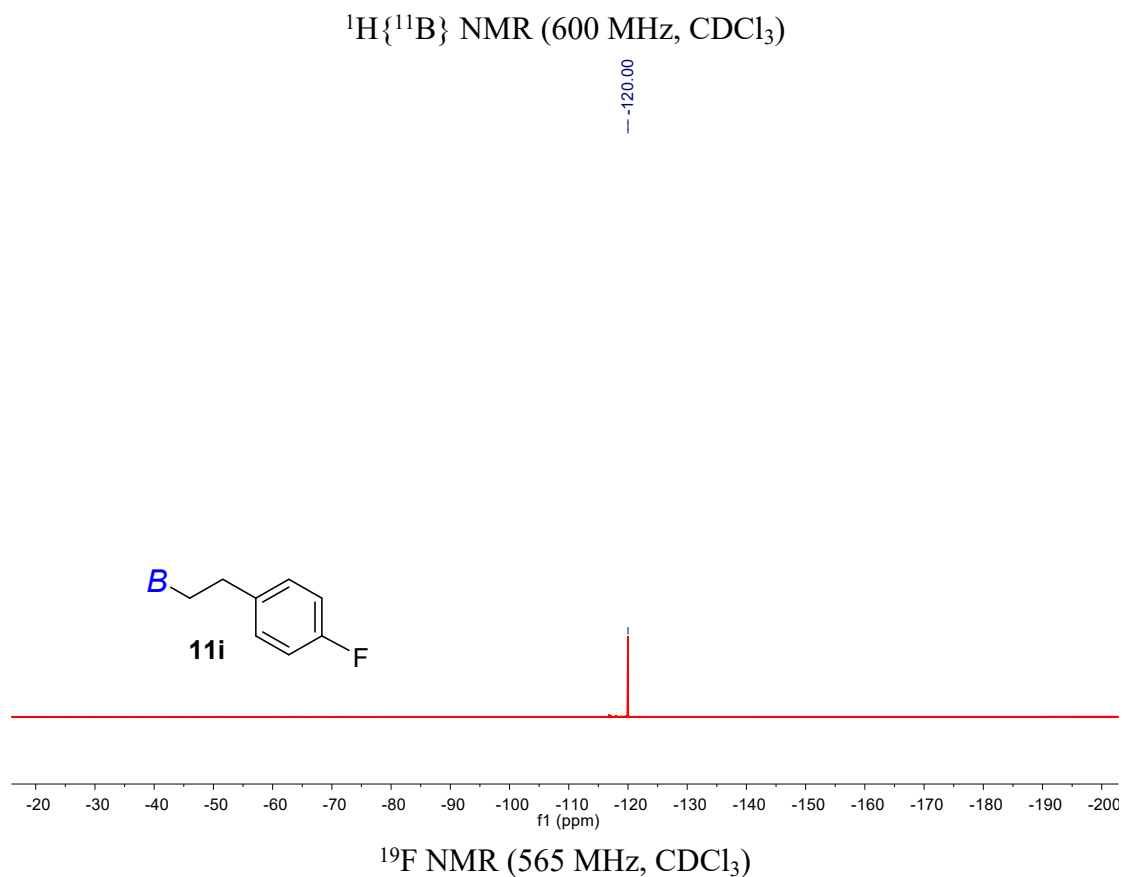
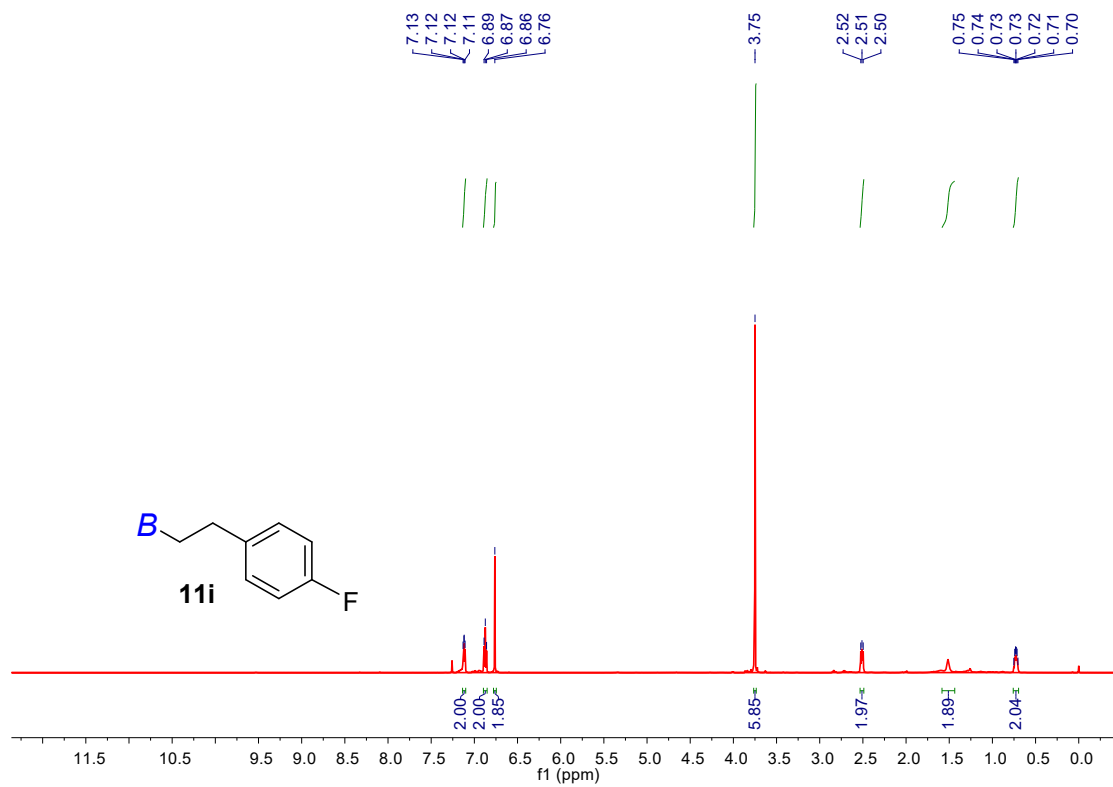


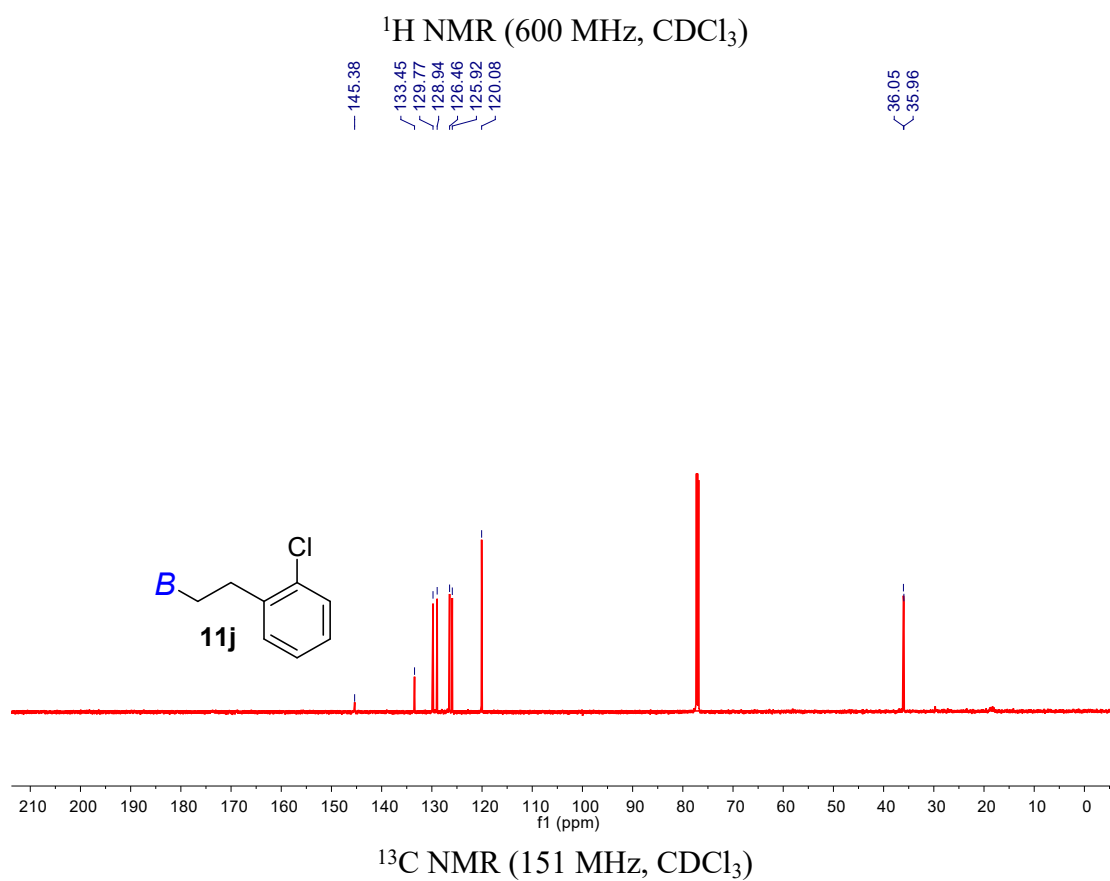
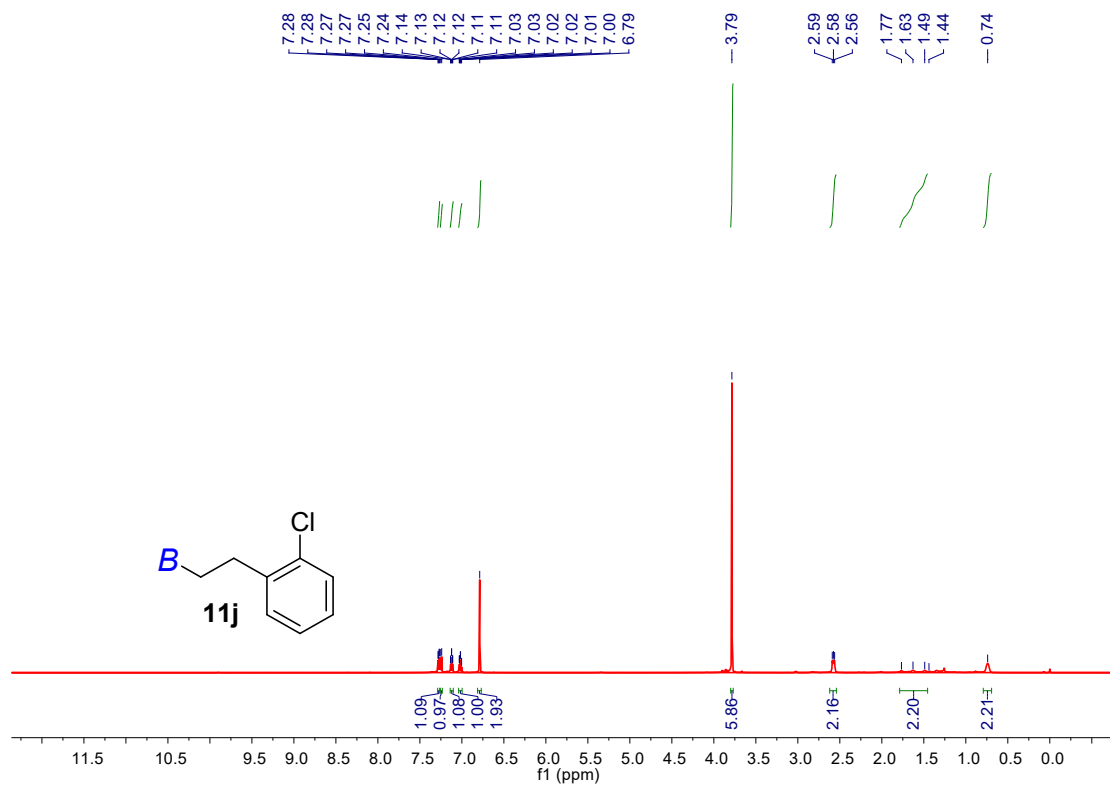
^{11}B NMR (193 MHz, CDCl_3)

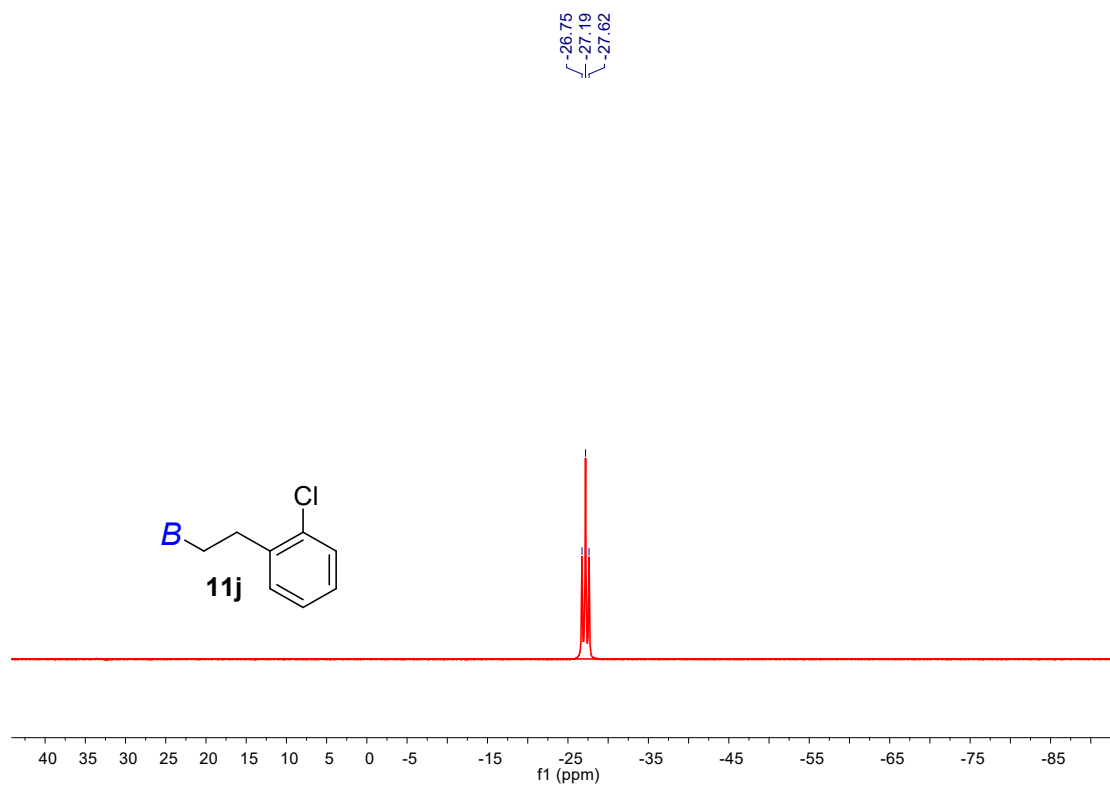
-27.17



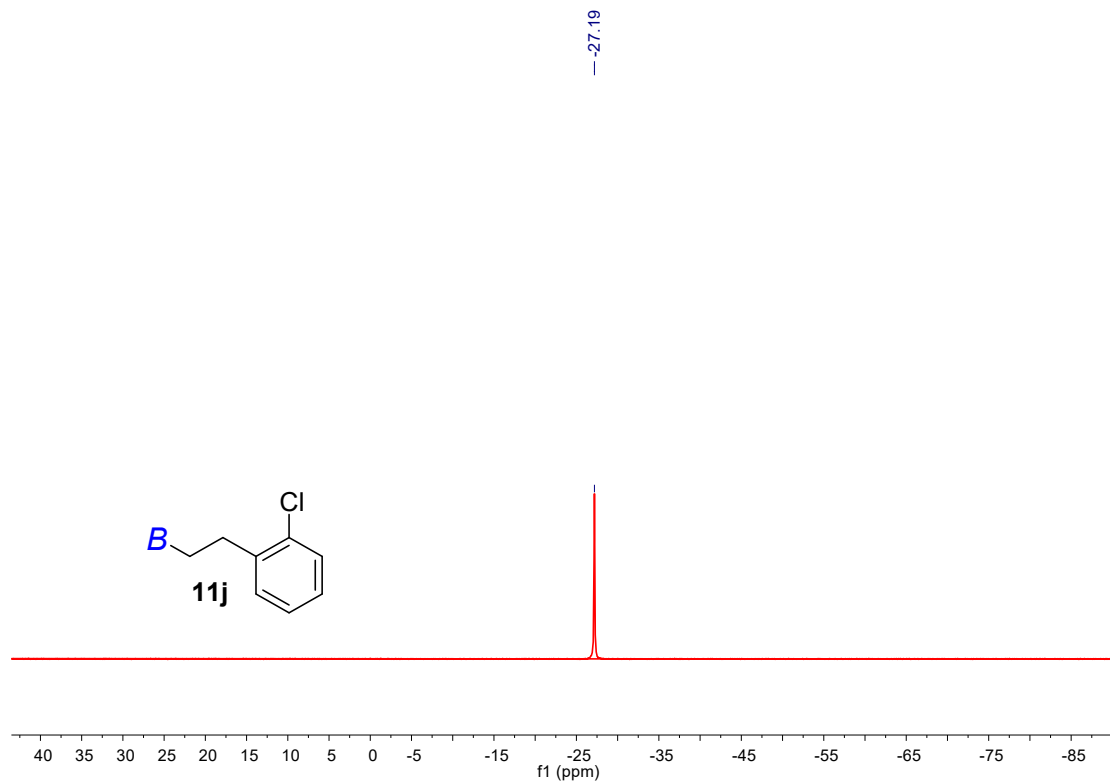
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



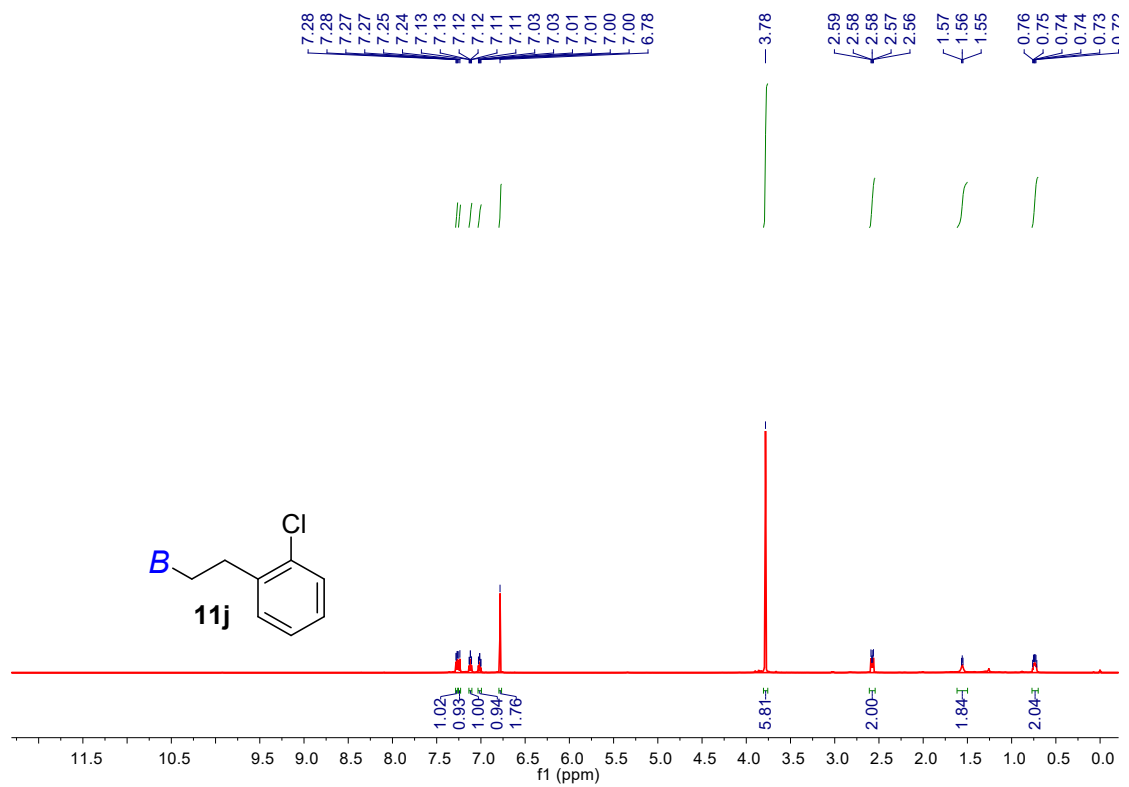


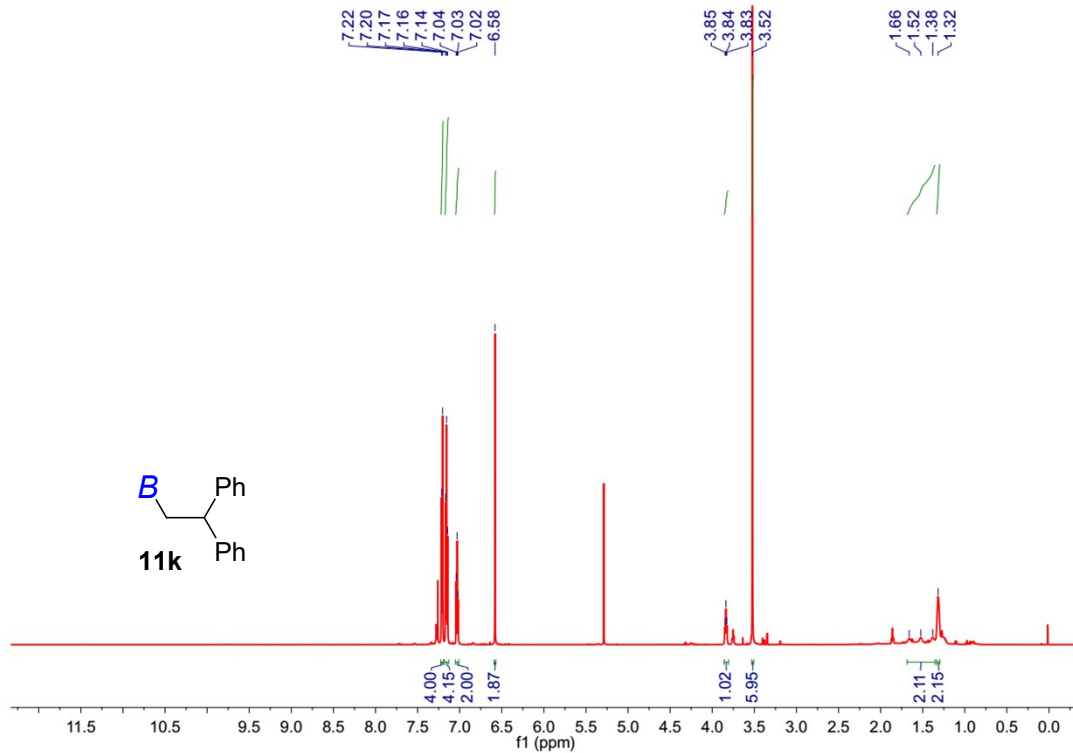


^{11}B NMR (193 MHz, CDCl_3)

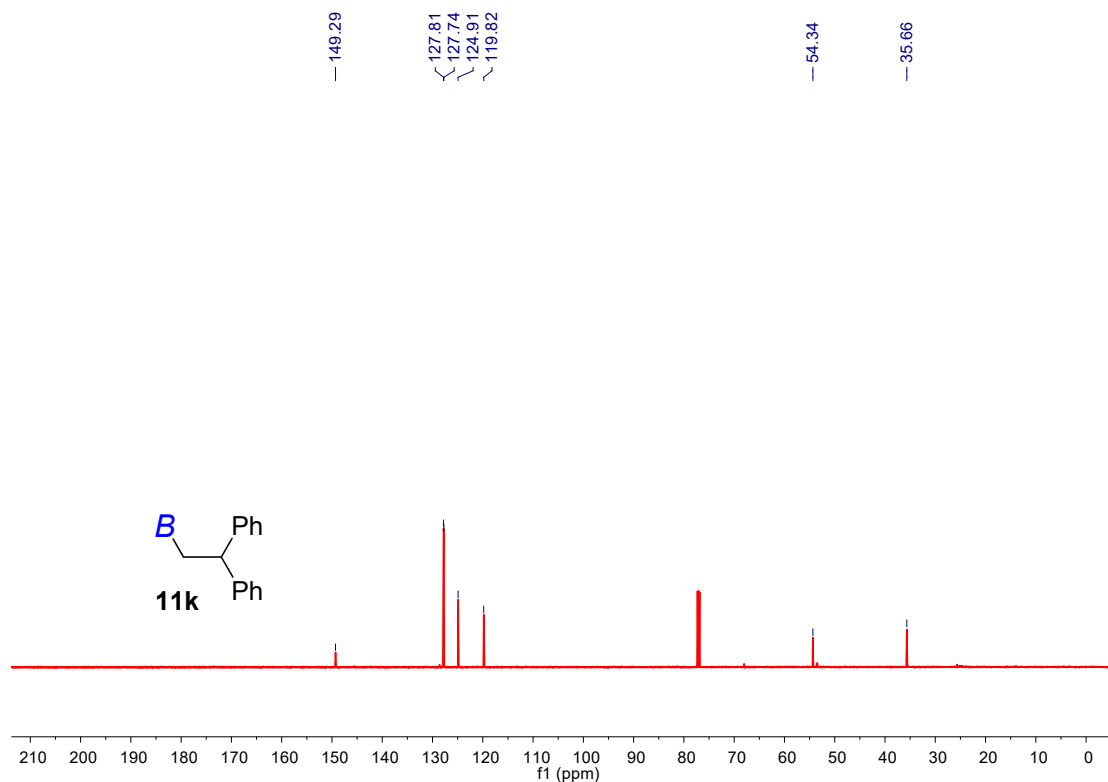


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

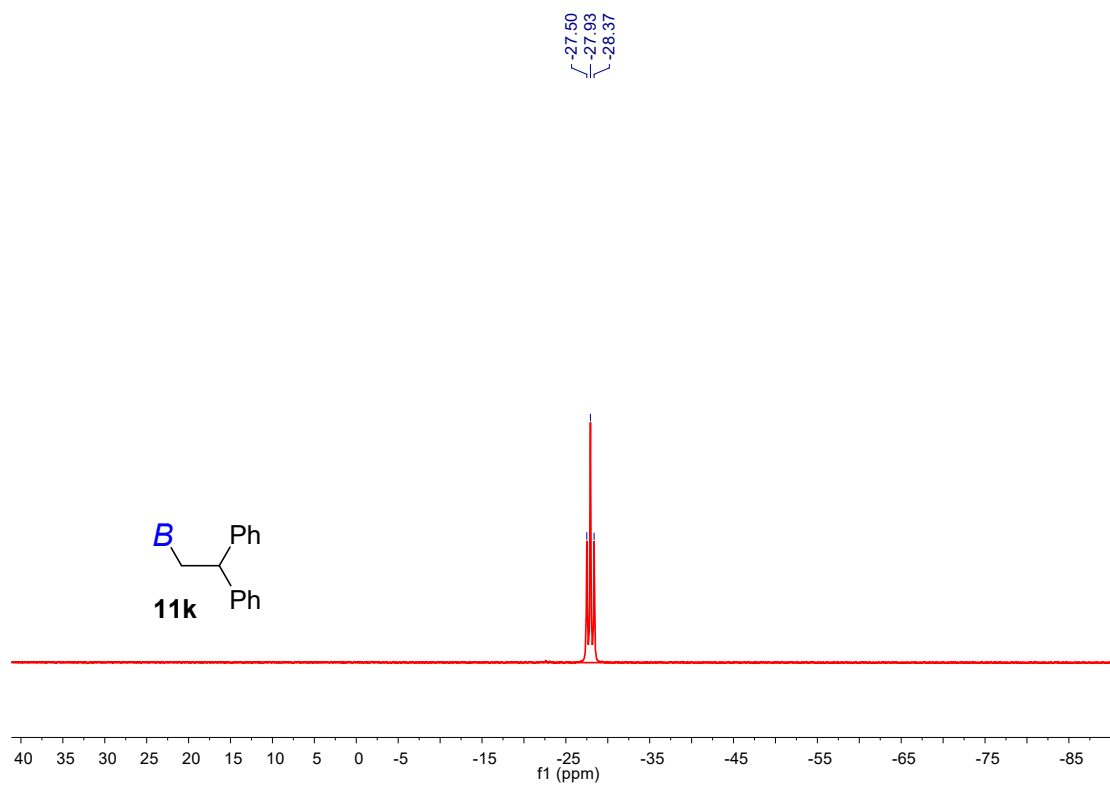




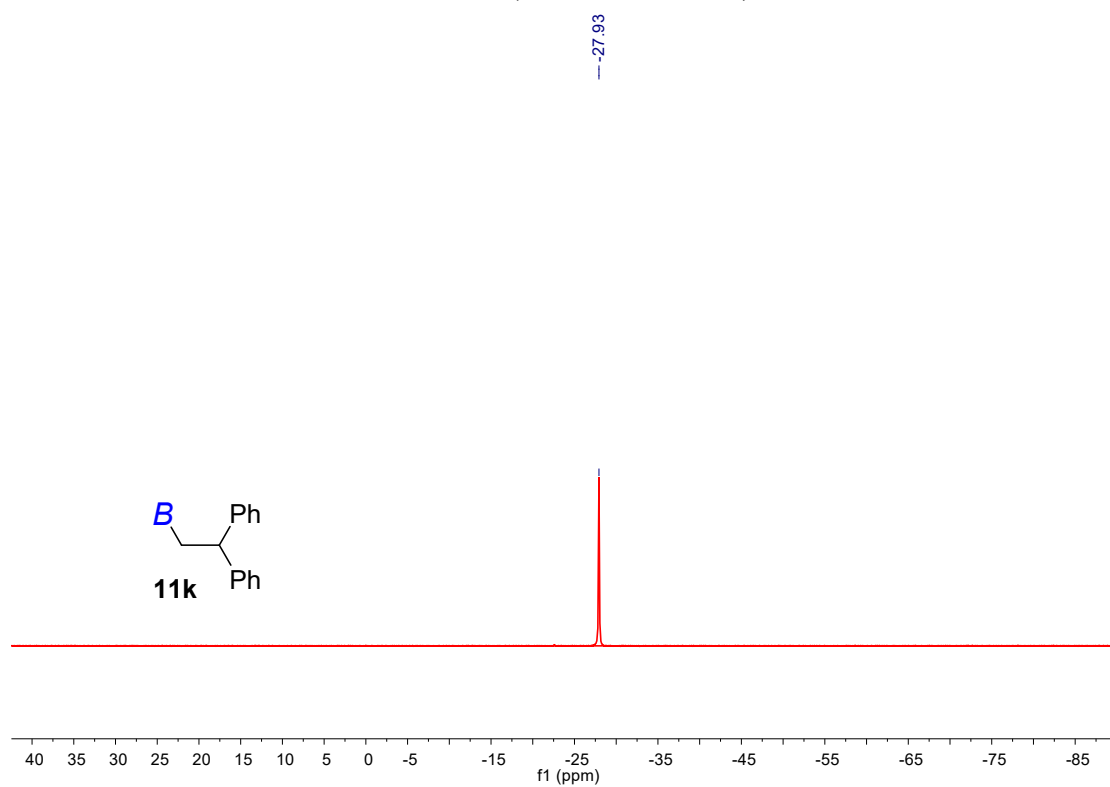
^1H NMR (600 MHz, CDCl_3)



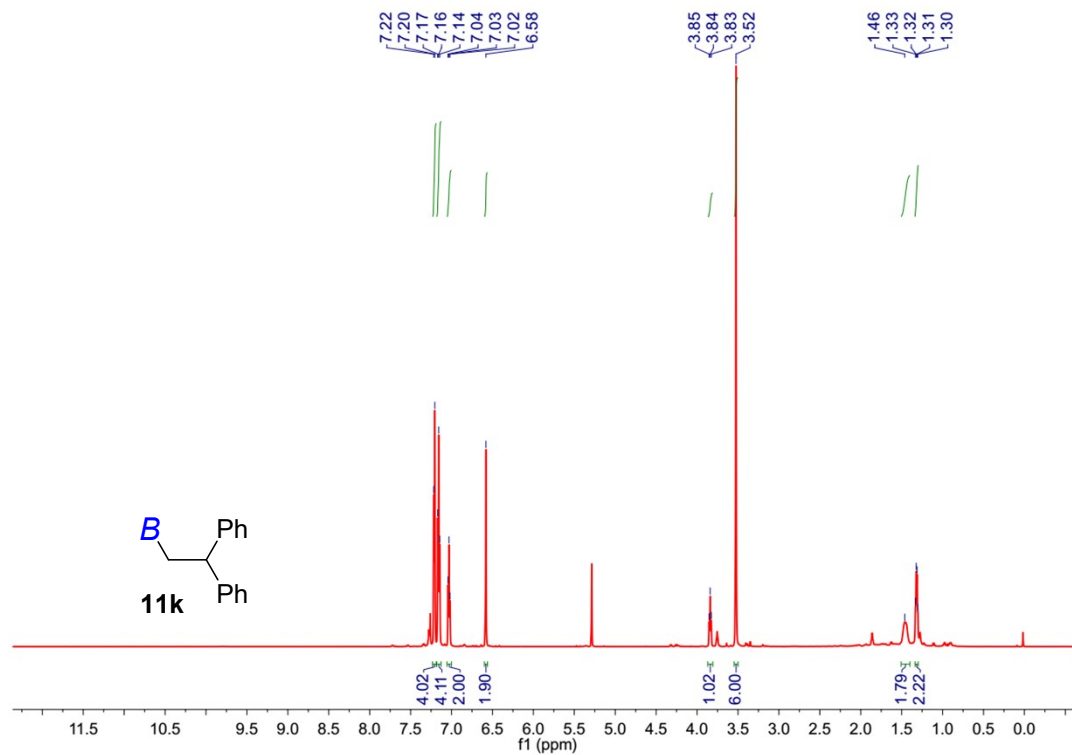
^{13}C NMR (151 MHz, CDCl_3)



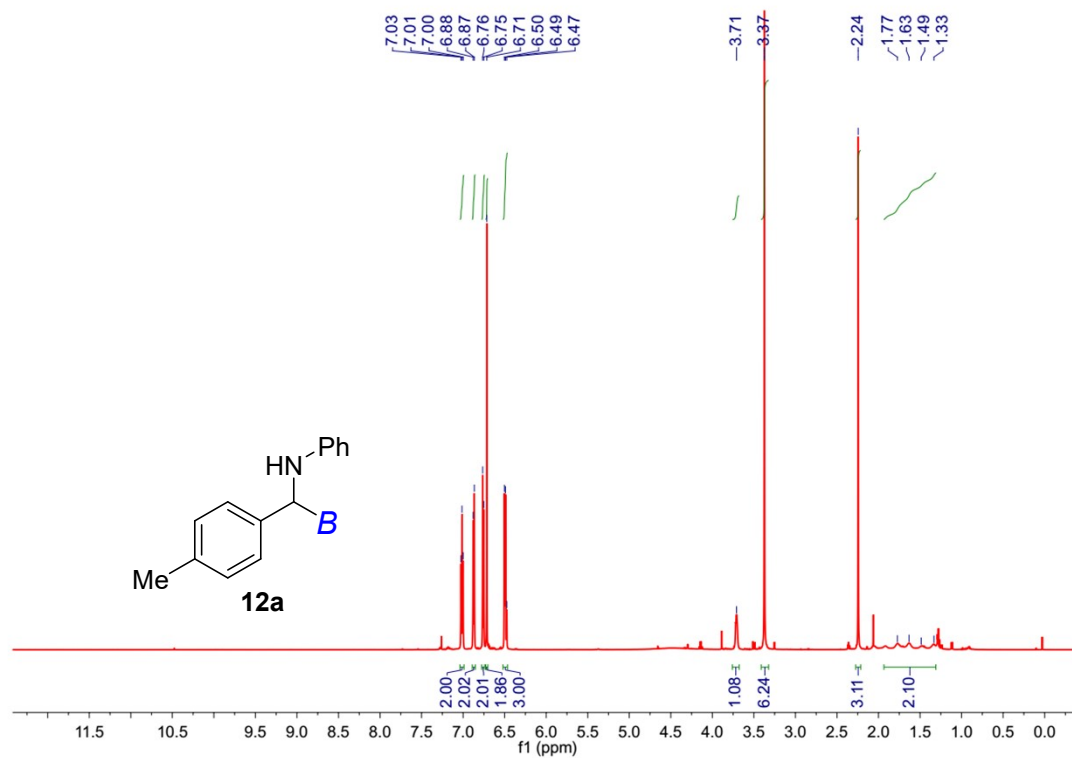
^{11}B NMR (193 MHz, CDCl_3)



$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



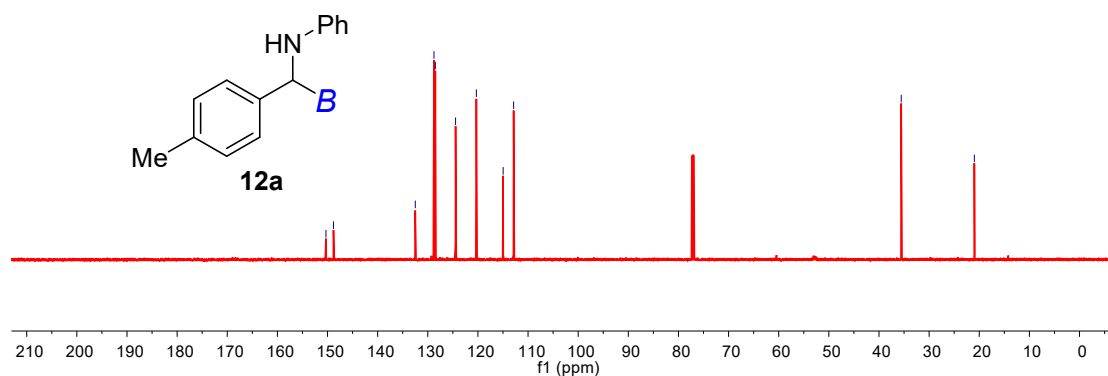
¹H NMR (600 MHz, CDCl₃)

150.31
148.81

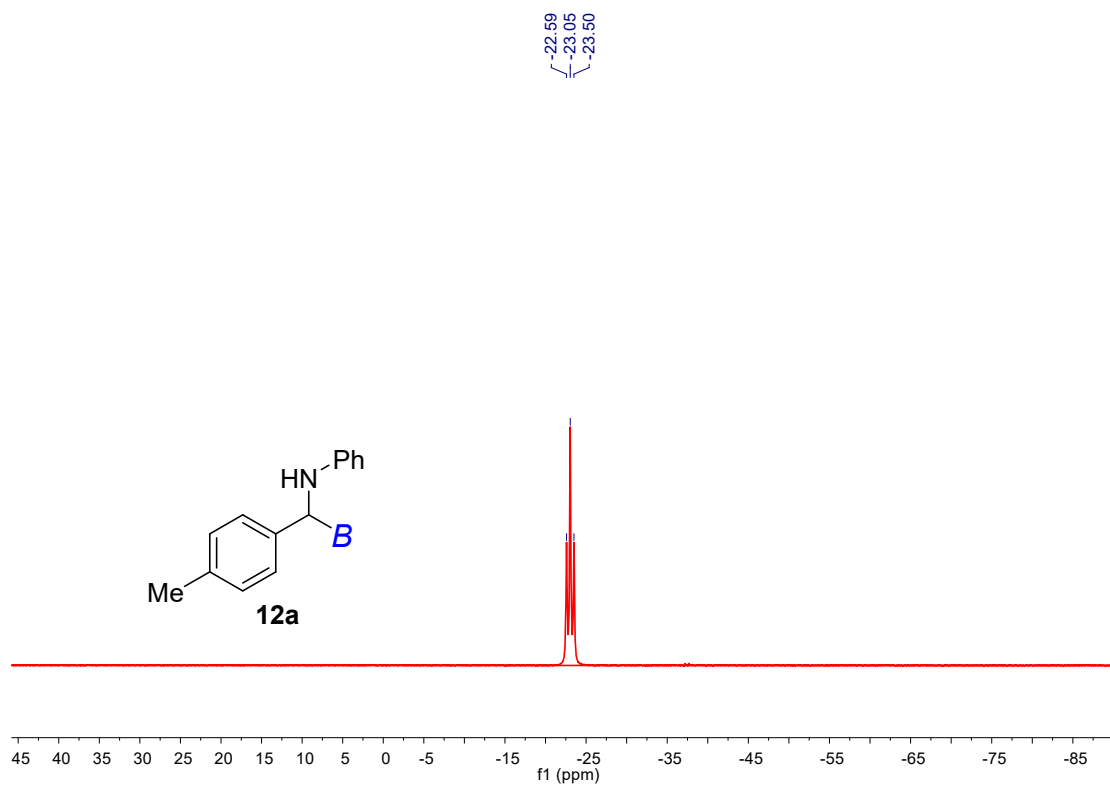
132.50
128.77
128.49
124.46
120.31
114.97
112.88

35.57

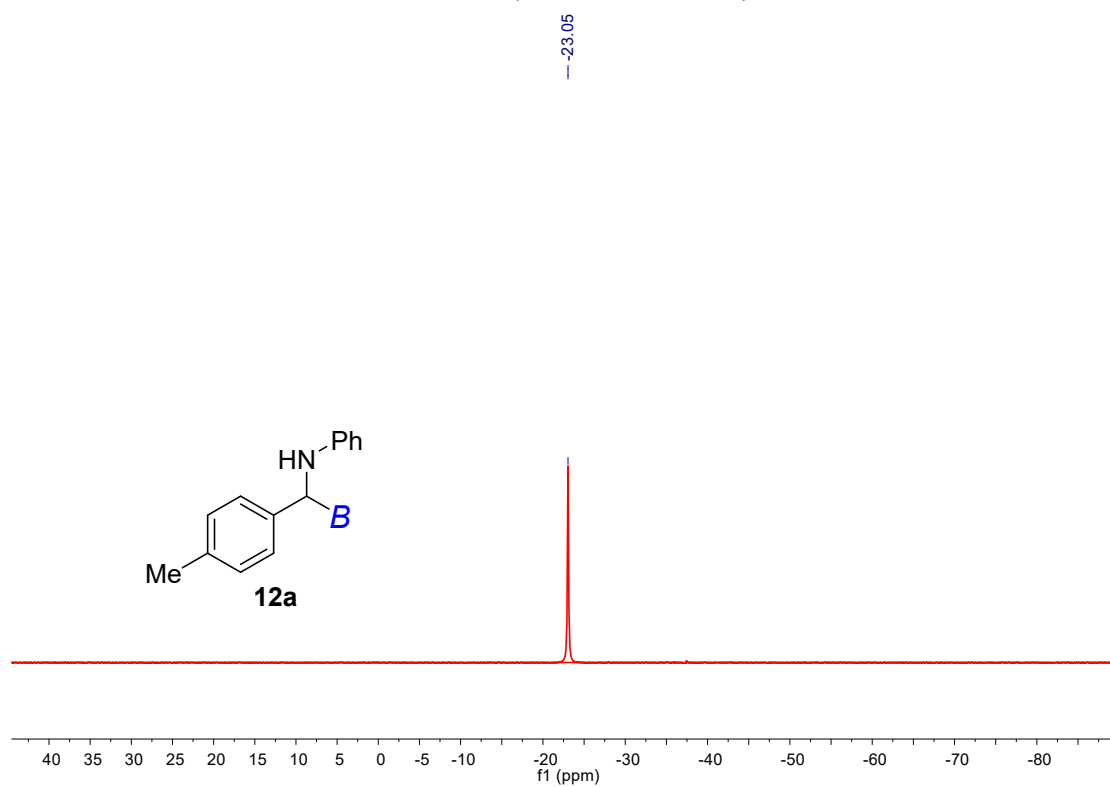
20.98



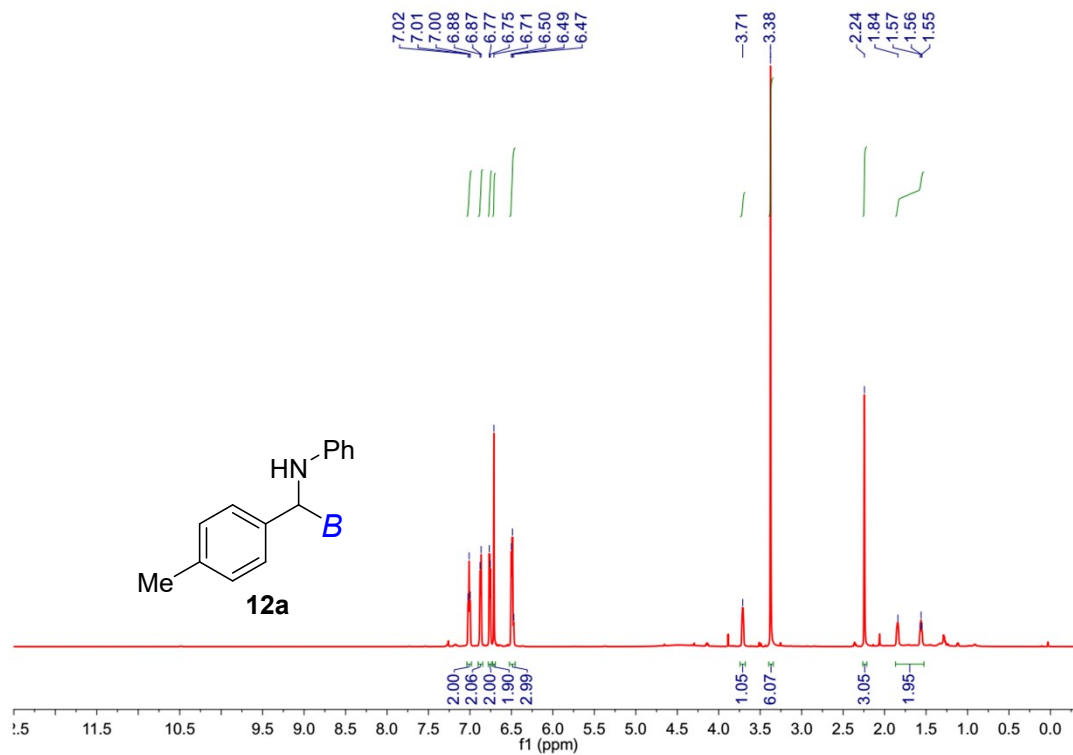
¹³C NMR (151 MHz, CDCl₃)

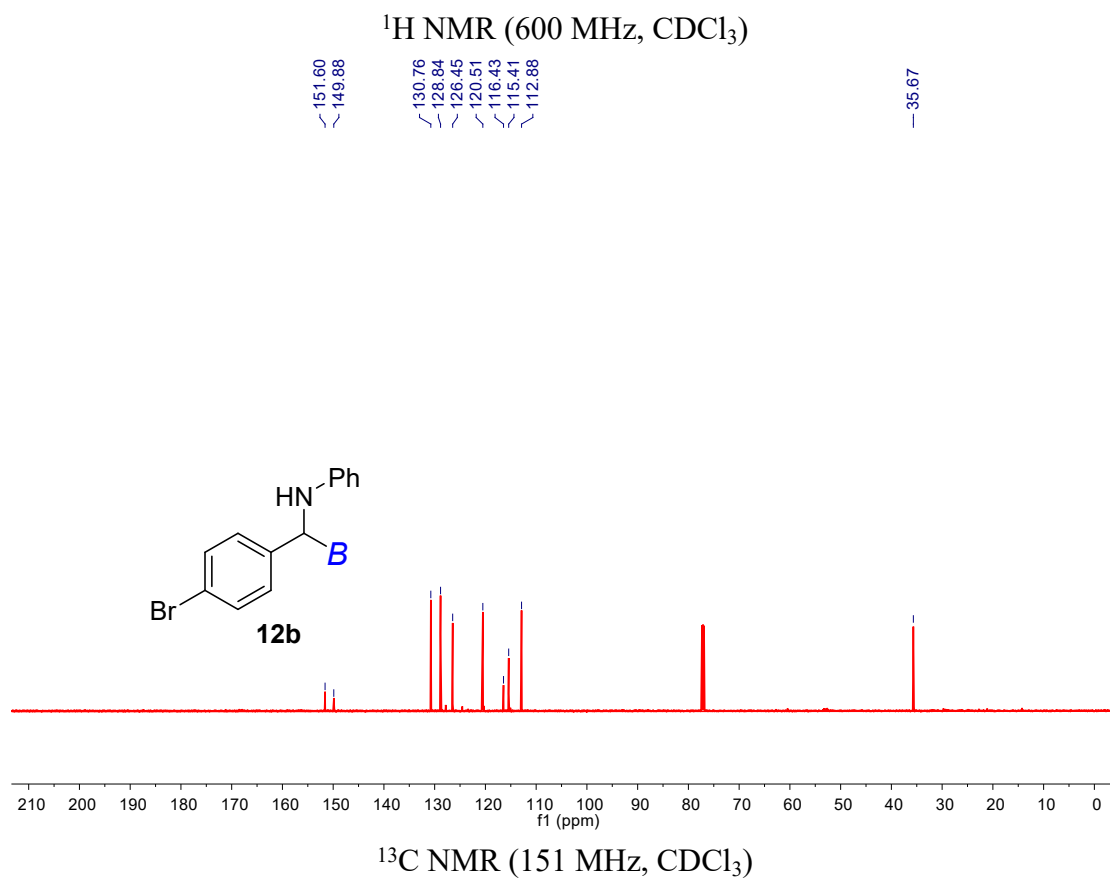
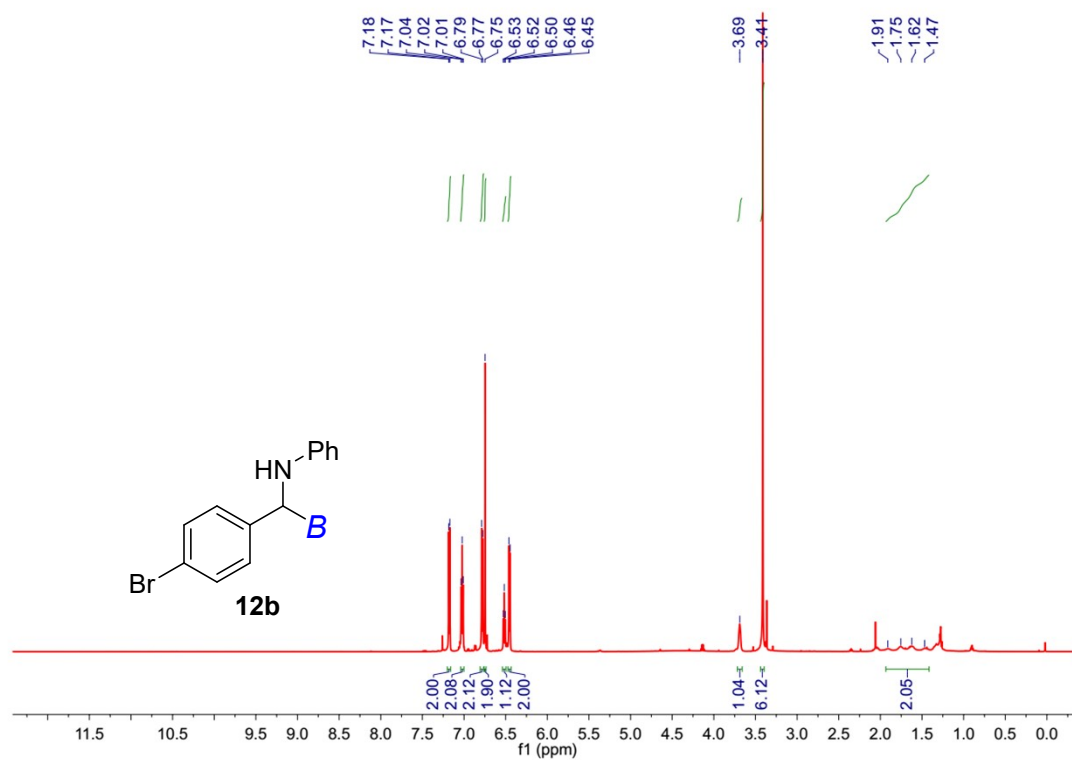


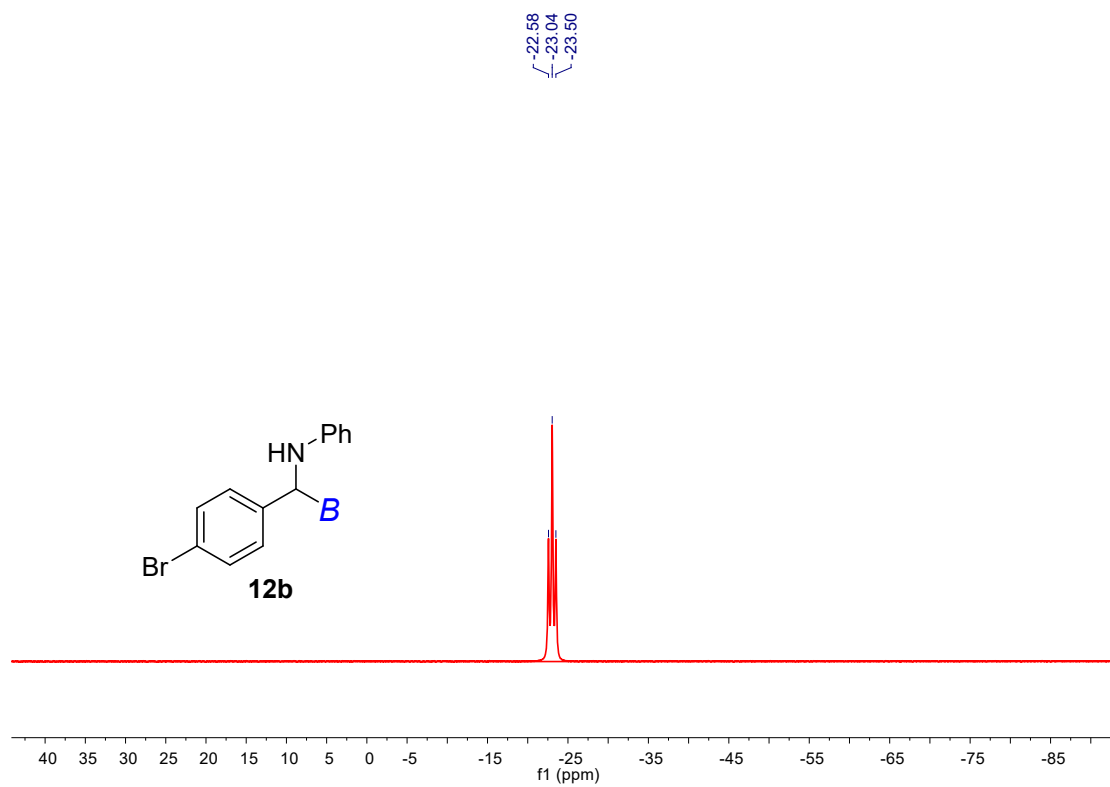
^{11}B NMR (193 MHz, CDCl_3)



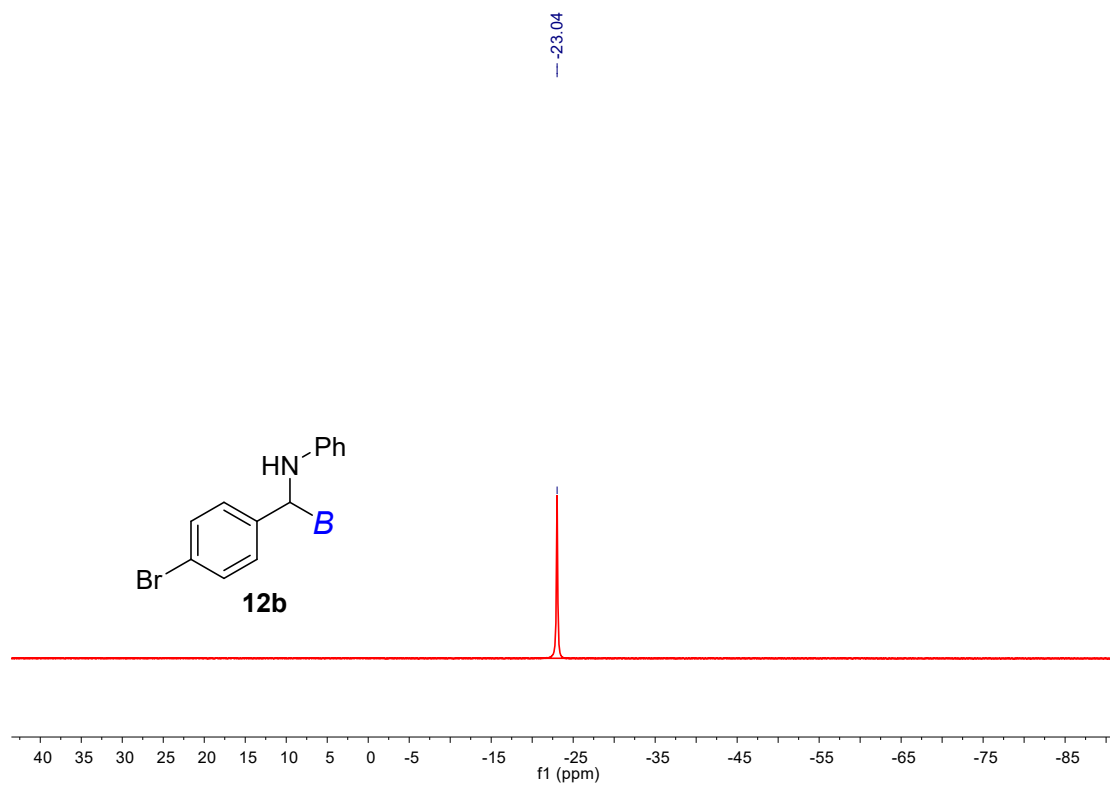
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



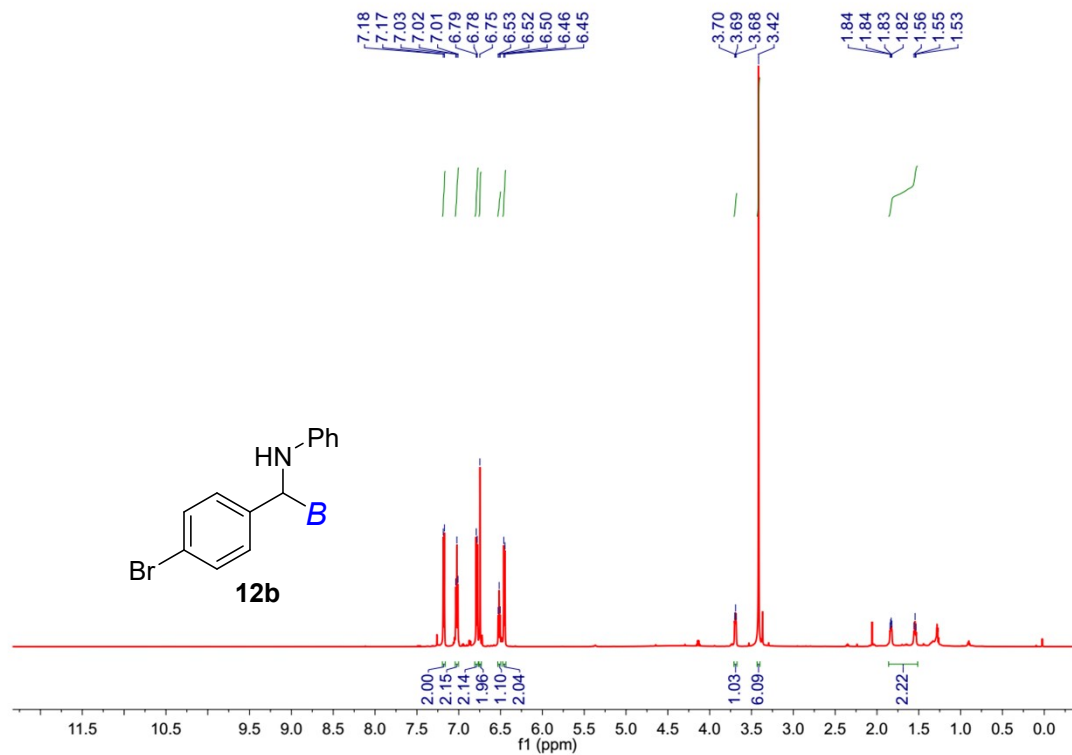




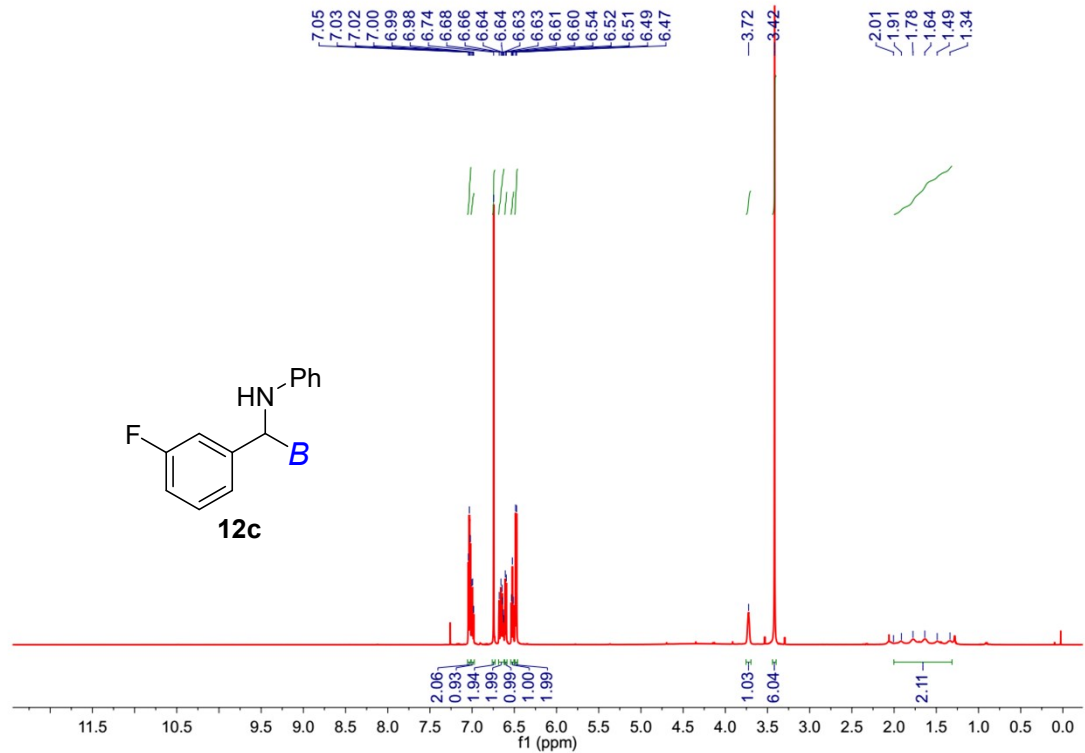
^{11}B NMR (193 MHz, CDCl_3)



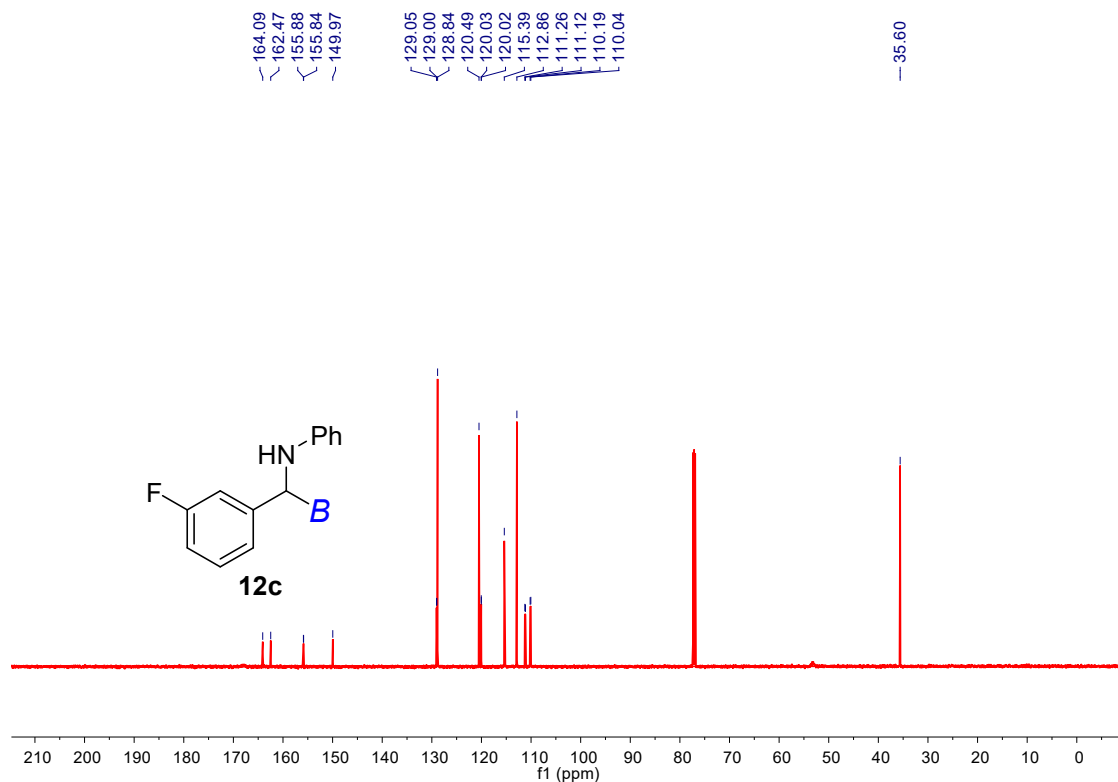
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



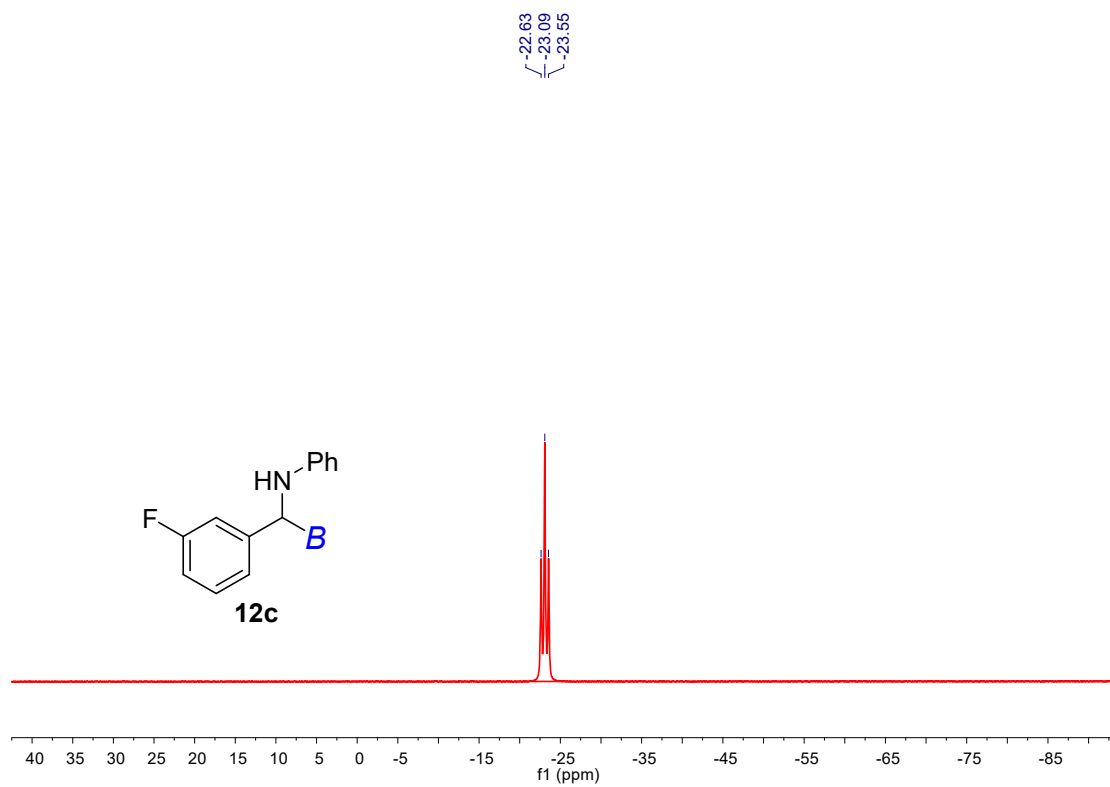
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



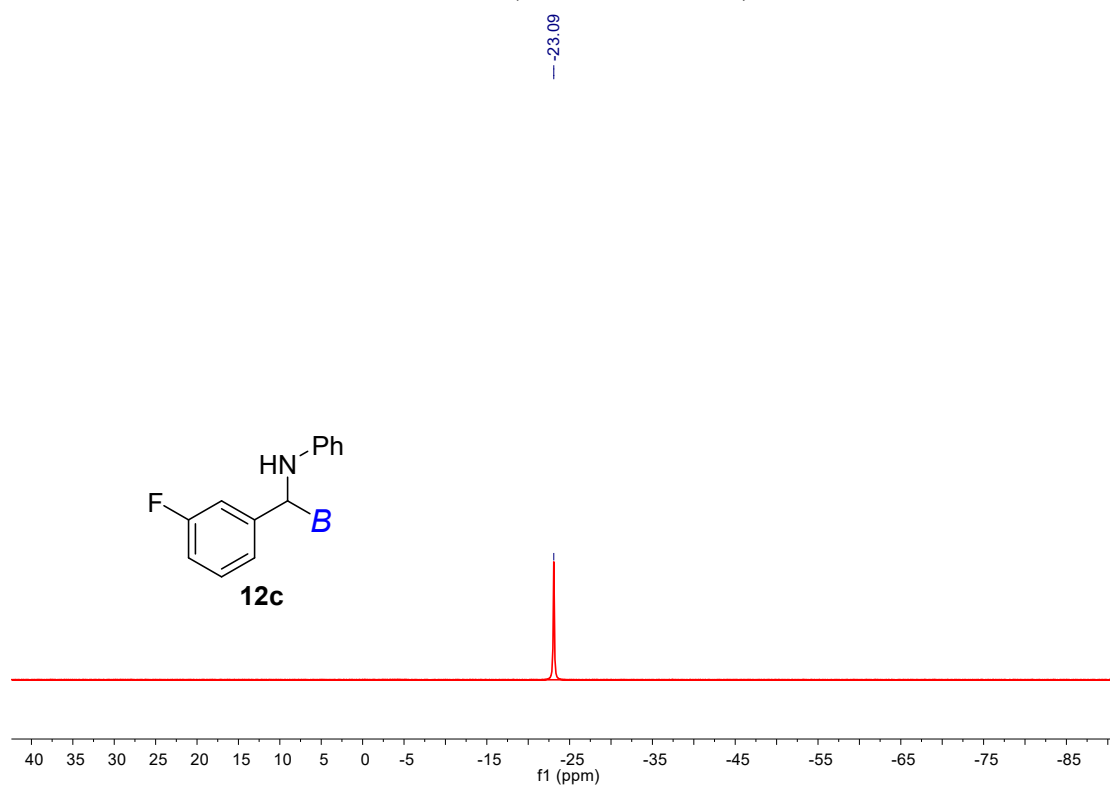
¹H NMR (600 MHz, CDCl₃)



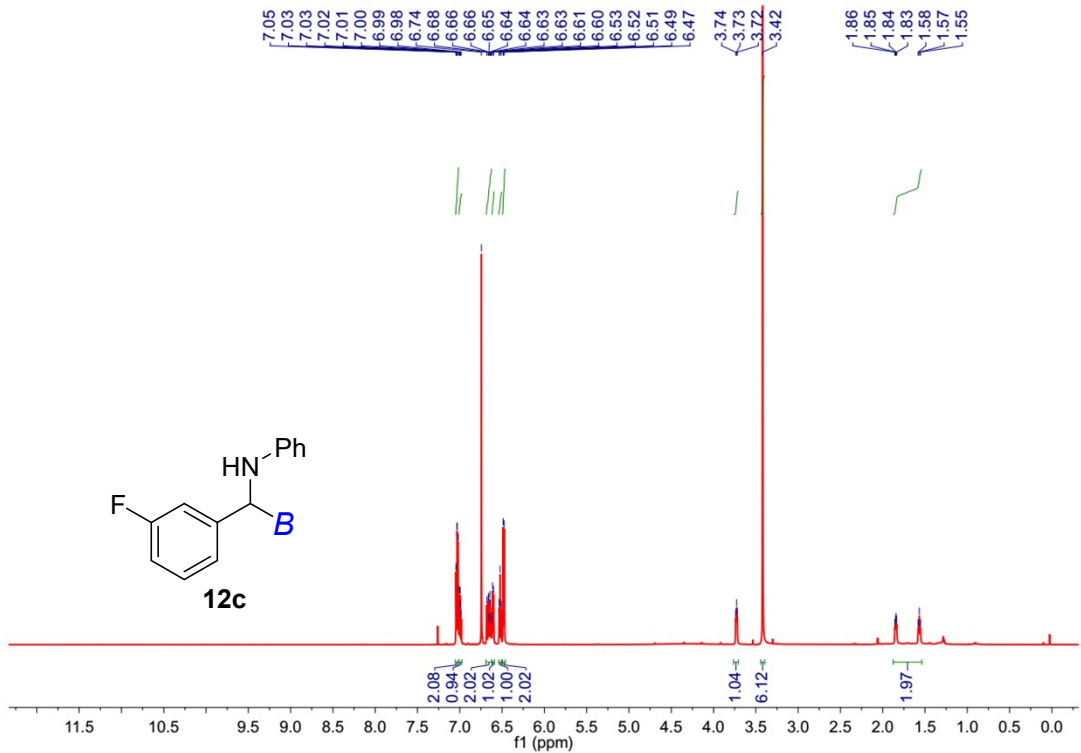
¹³C NMR (151 MHz, CDCl₃)



^{11}B NMR (193 MHz, CDCl_3)

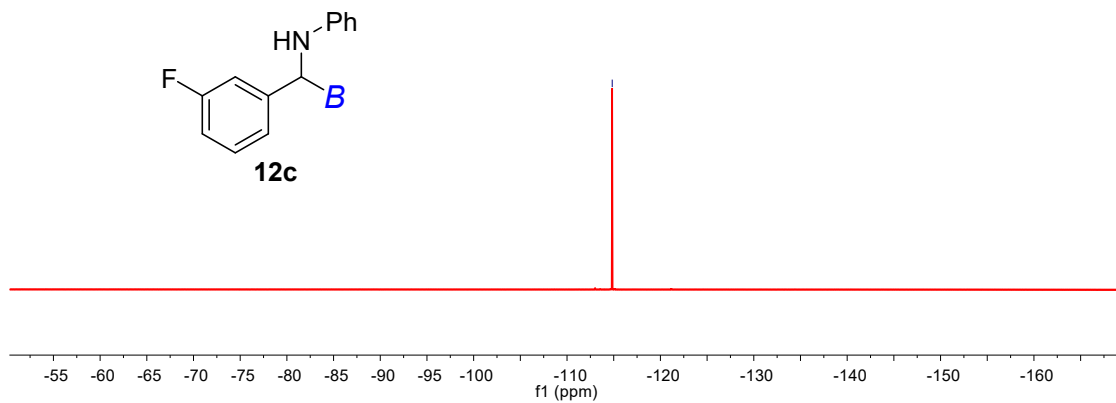


$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

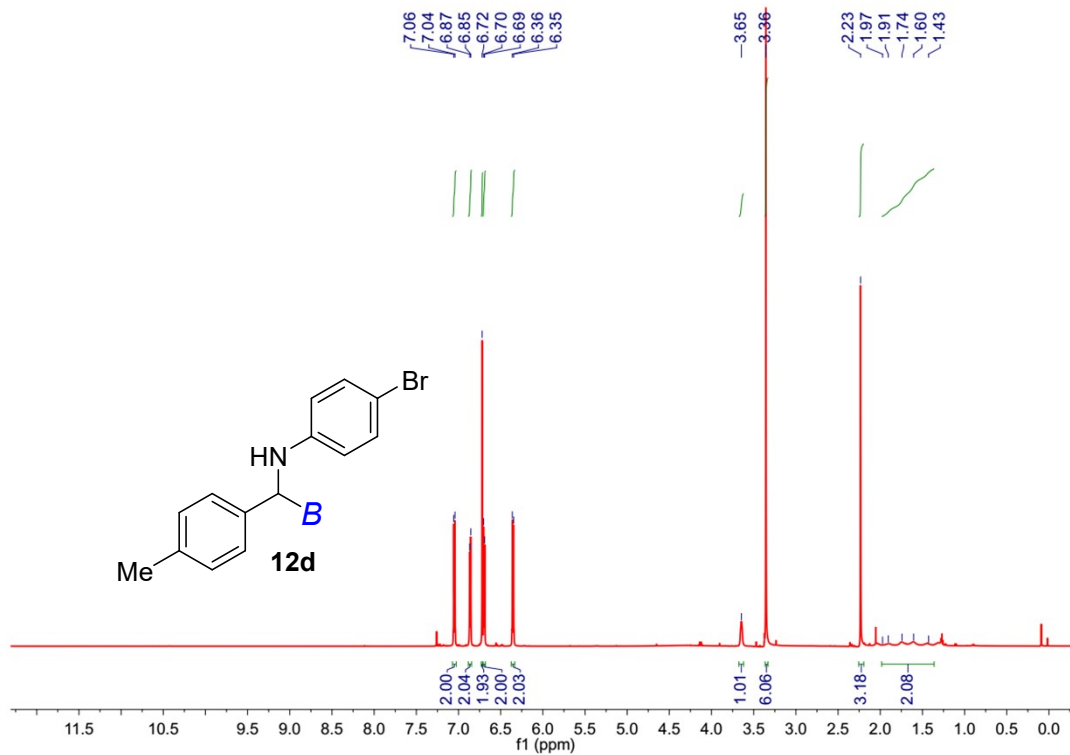


$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

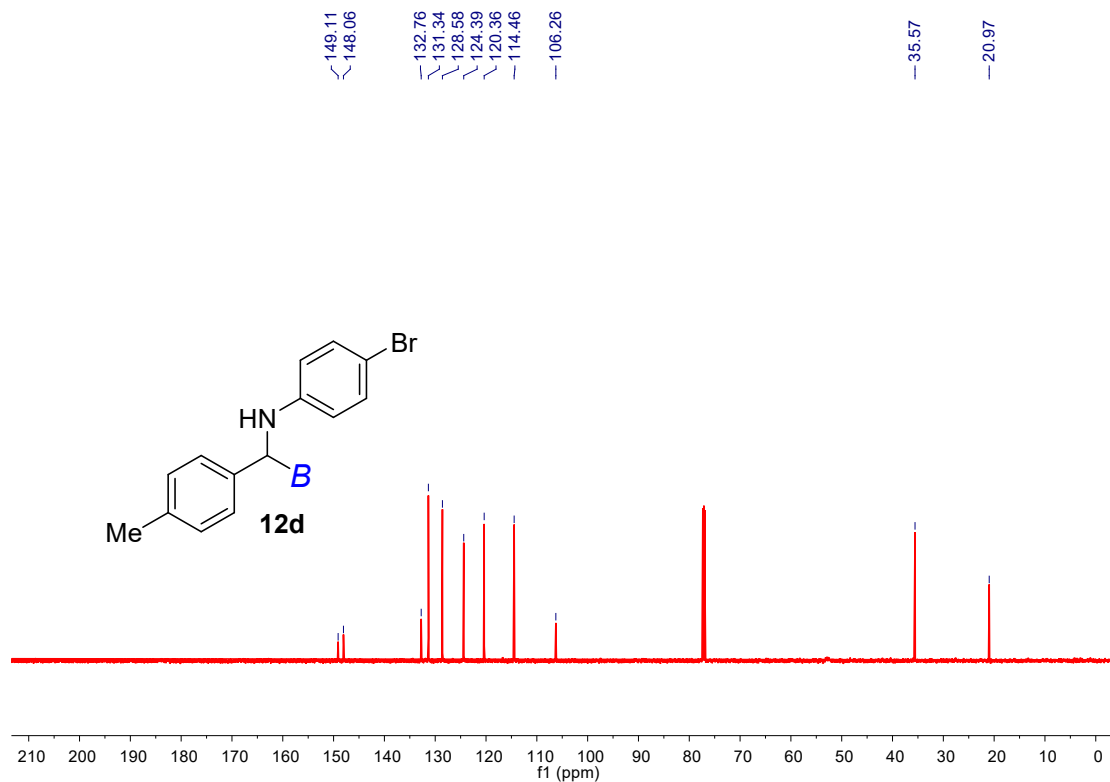
--114.84



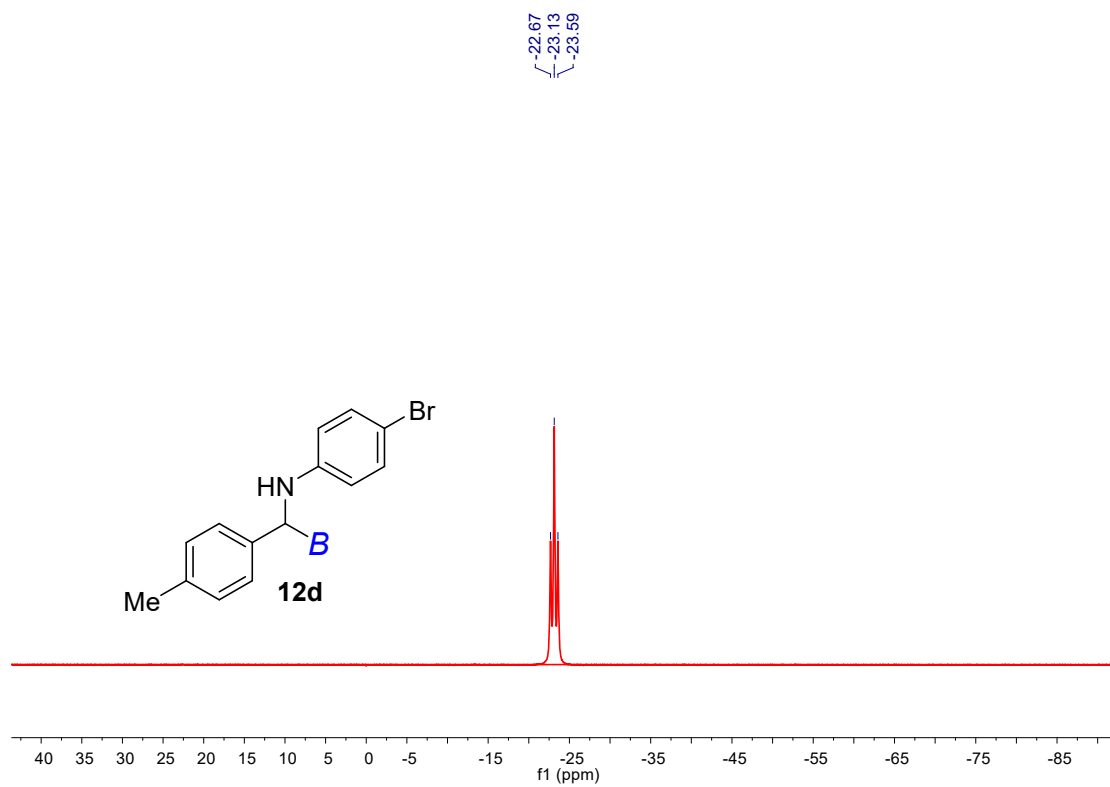
^{19}F NMR (565 MHz, CDCl_3)



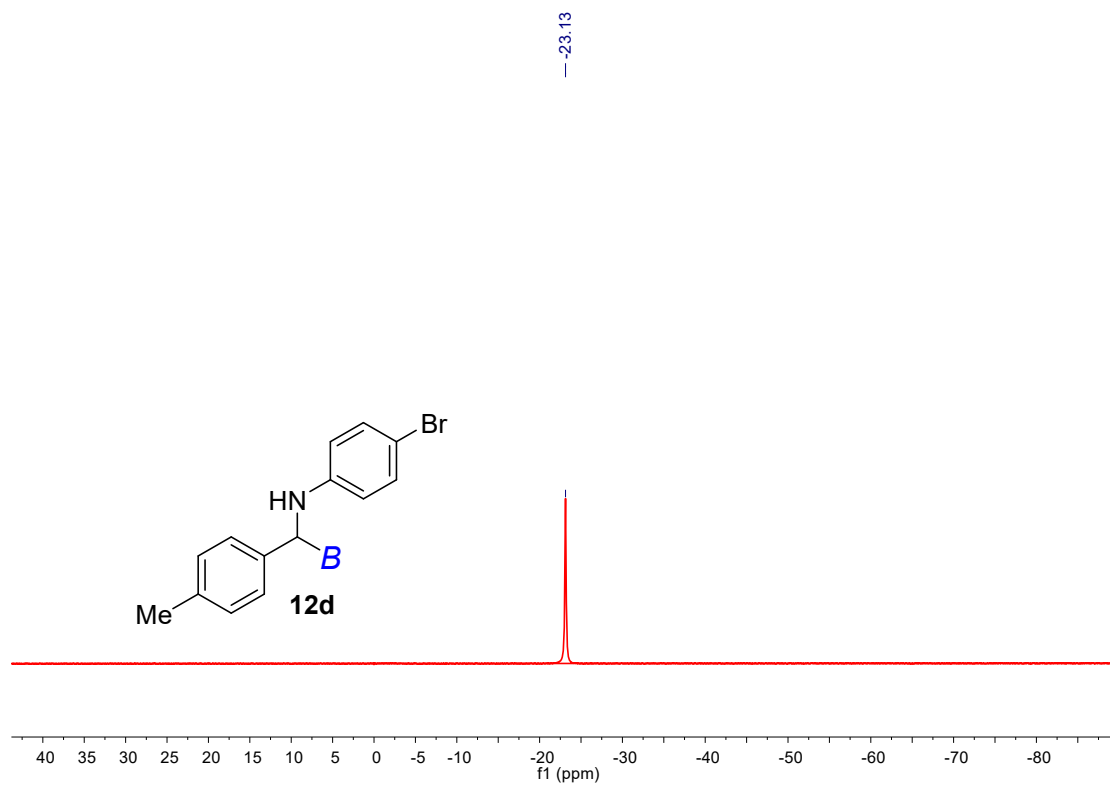
¹H NMR (600 MHz, CDCl₃)



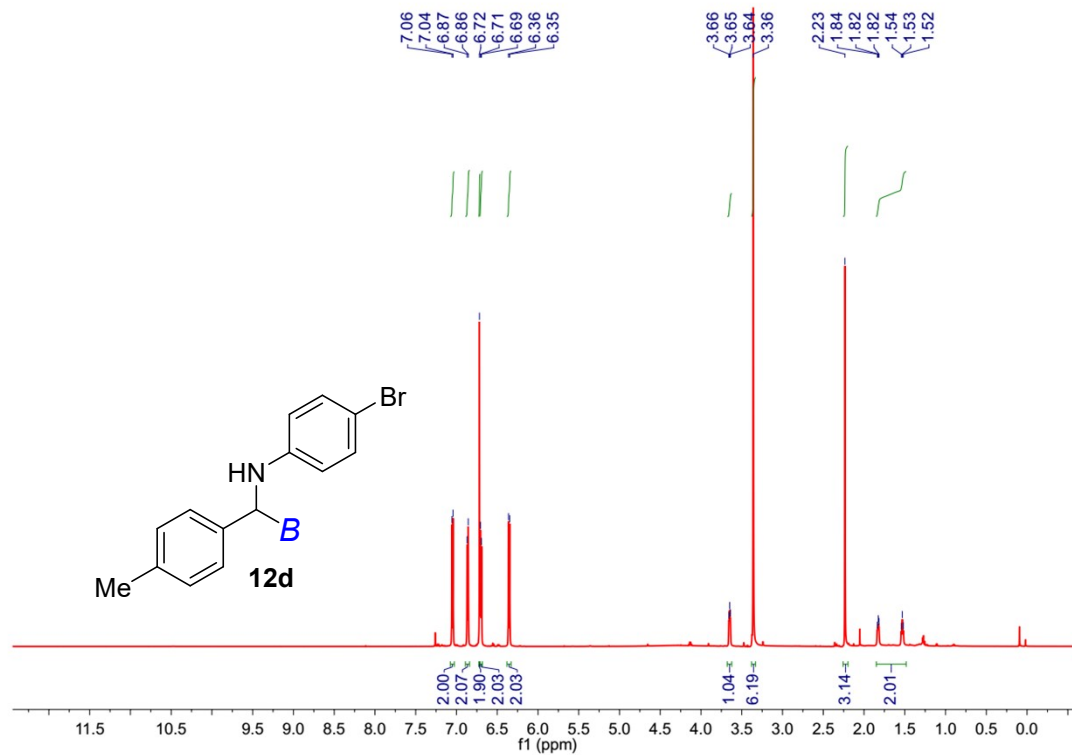
¹³C NMR (151 MHz, CDCl₃)



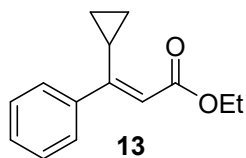
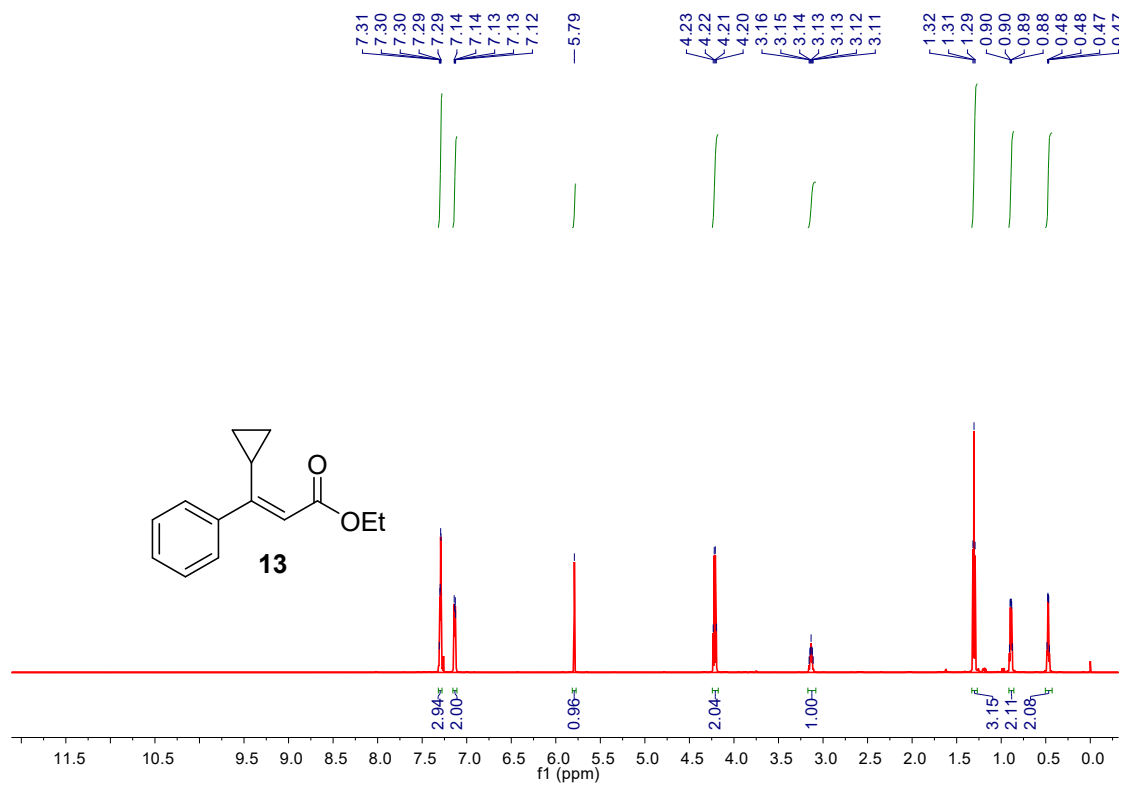
^{11}B NMR (193 MHz, CDCl_3)



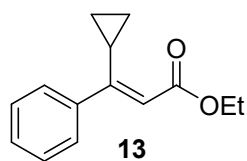
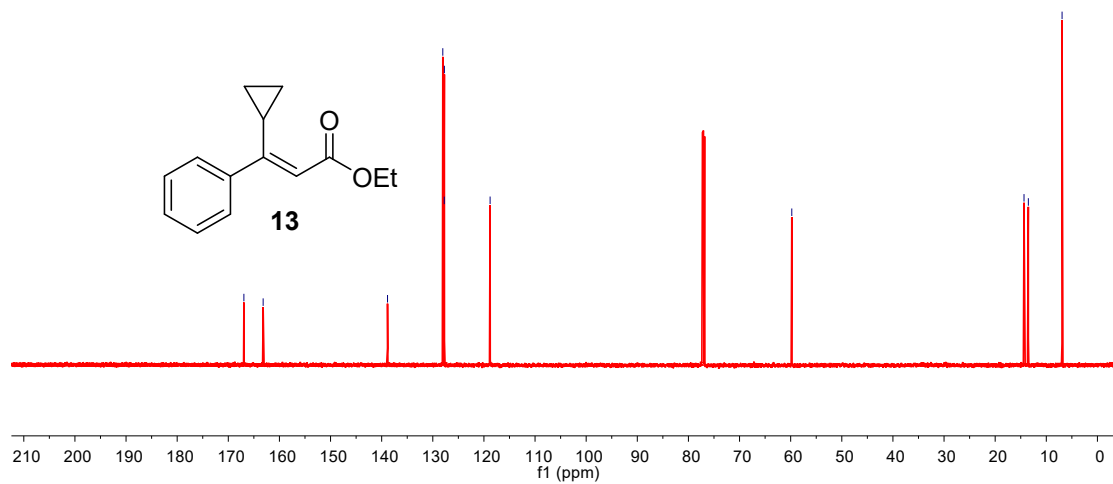
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)

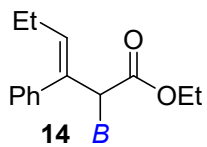
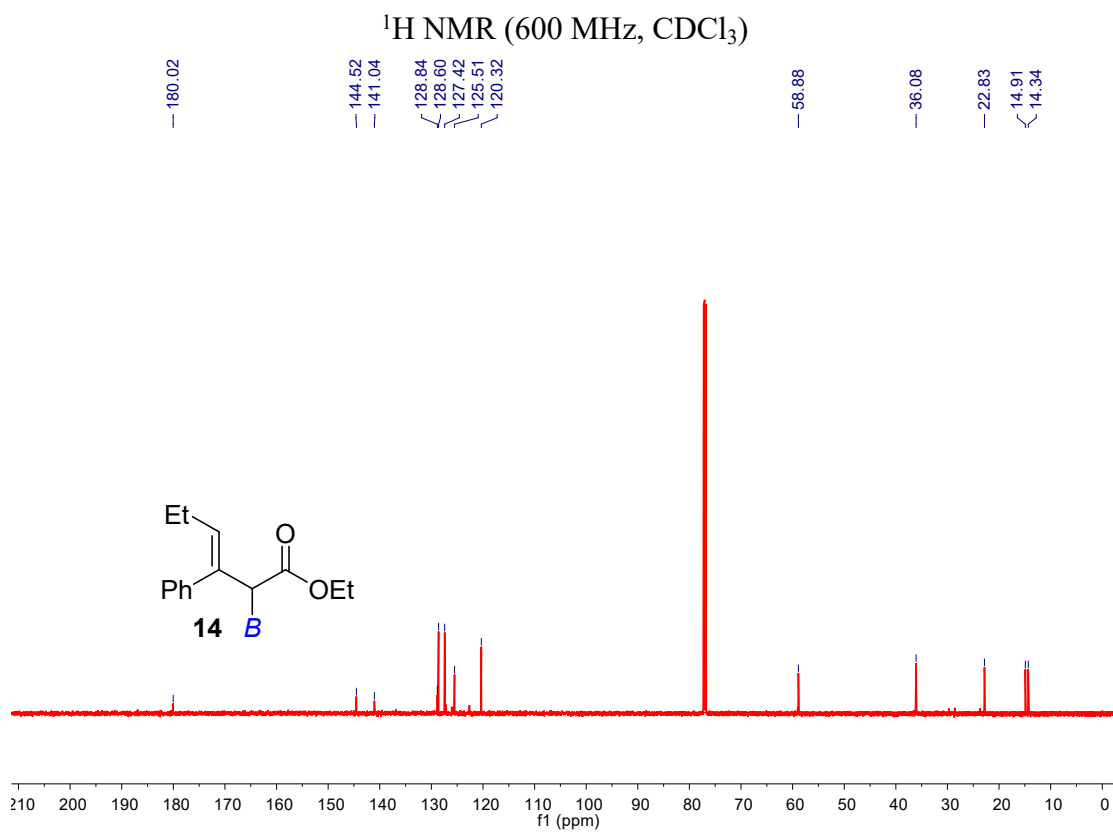
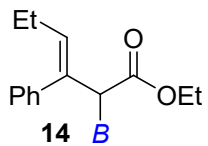
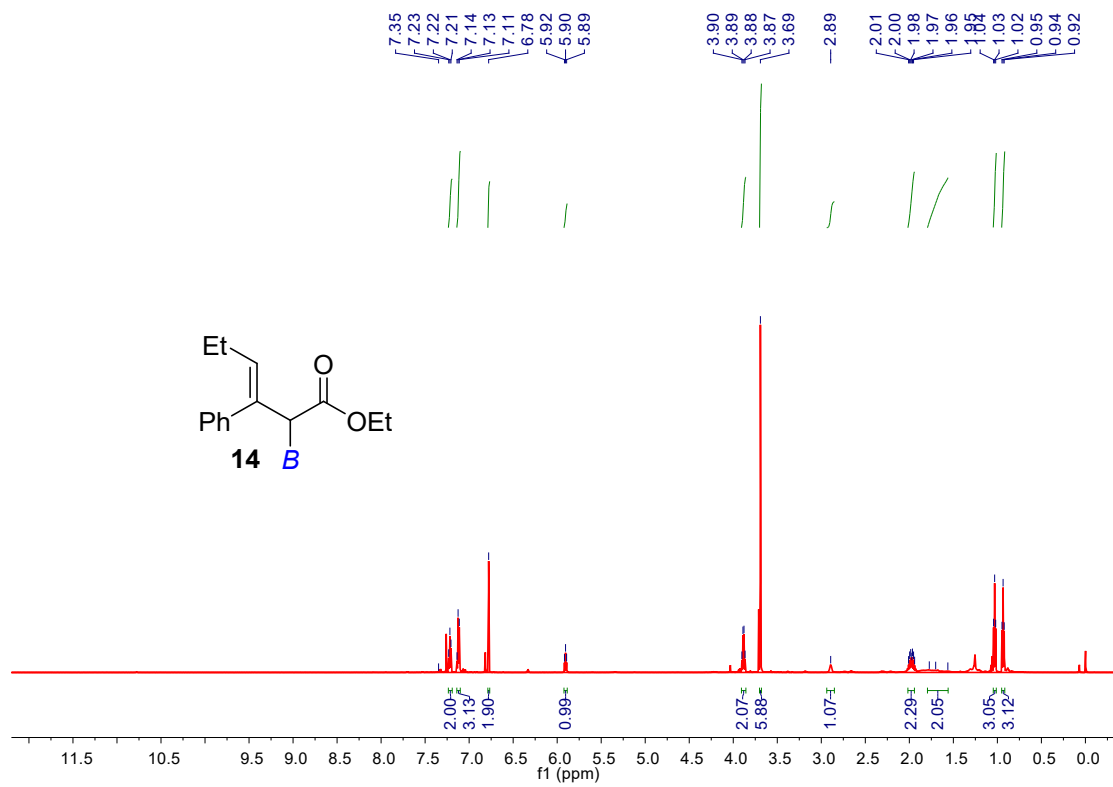


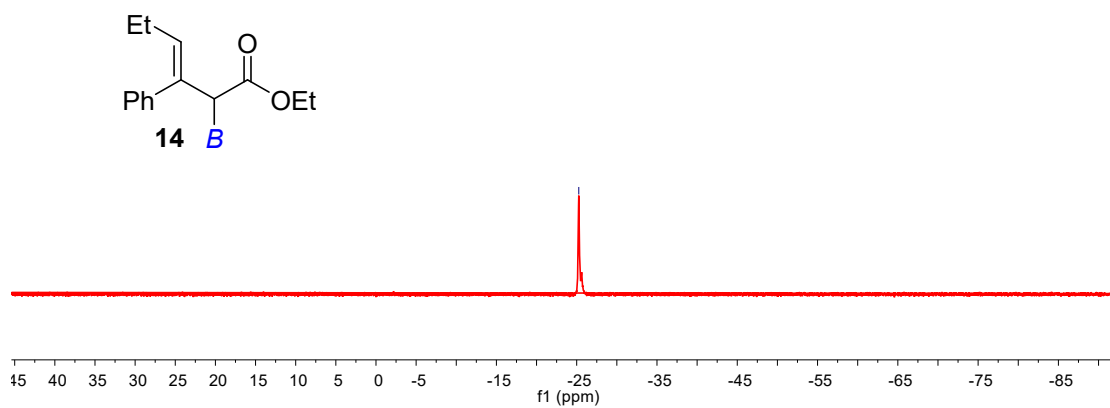
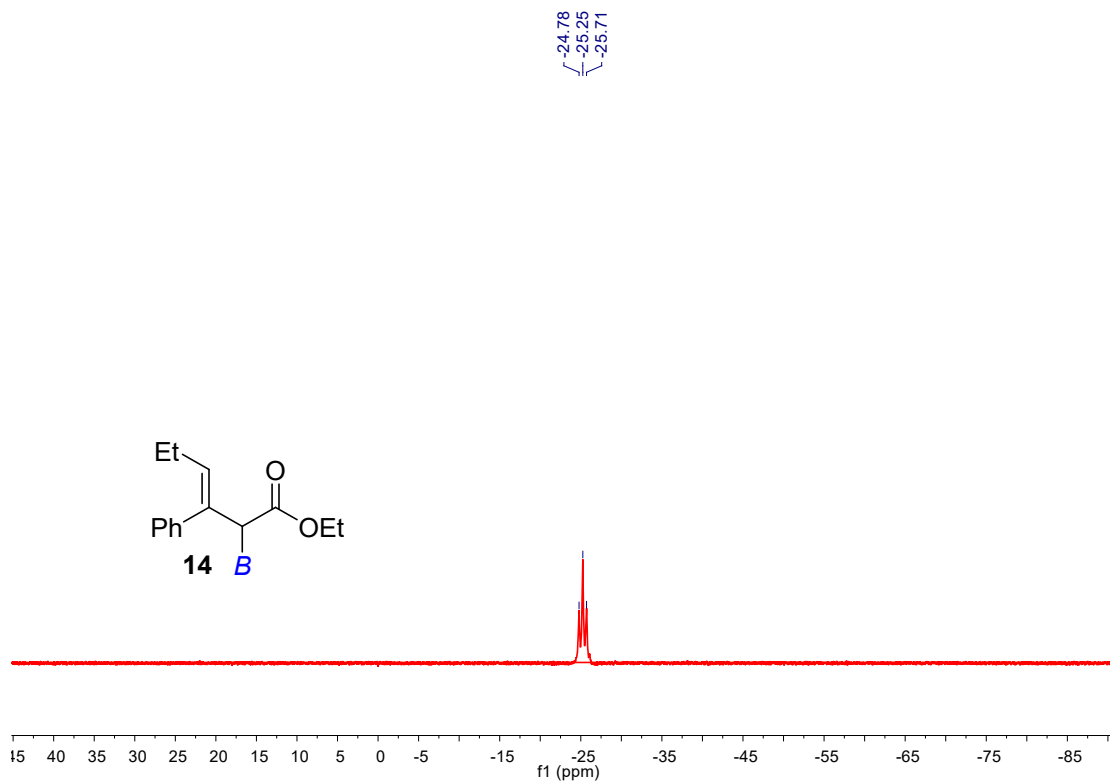
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)

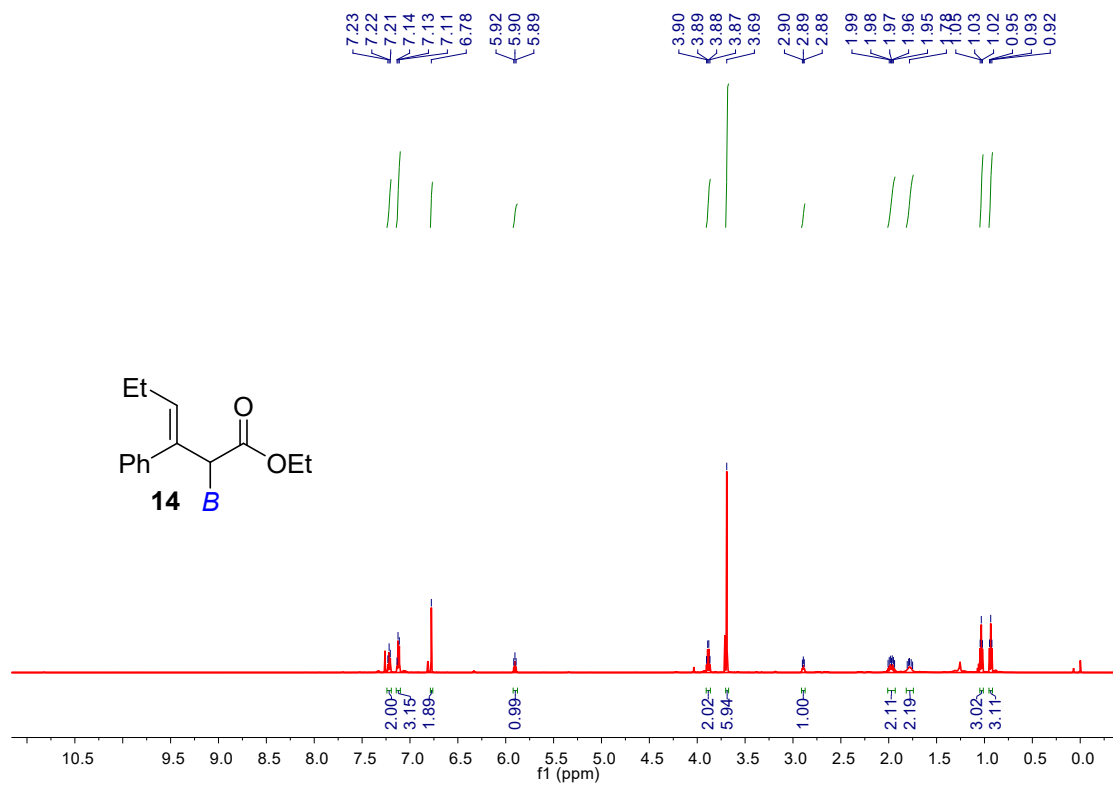


— 166.95
— 163.18
— 138.85
— 128.04
— 127.76
— 127.73
— 118.79
— 59.78
— 14.36
— 13.51
— 6.91

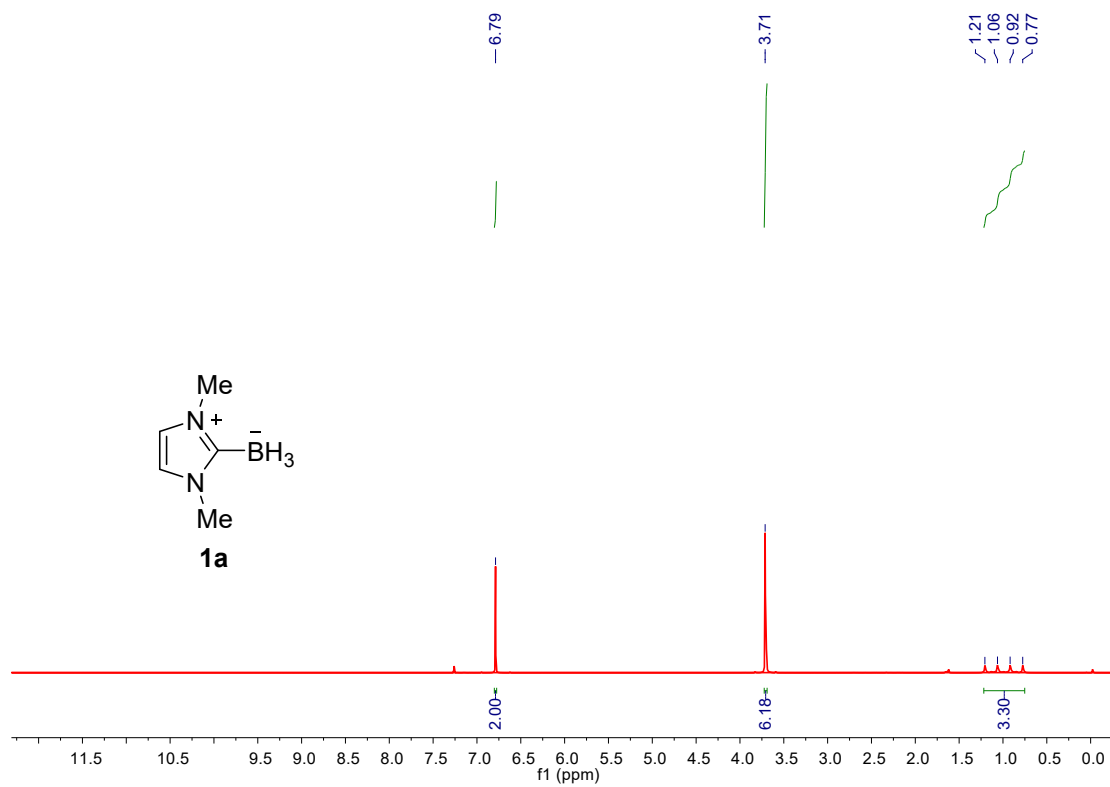




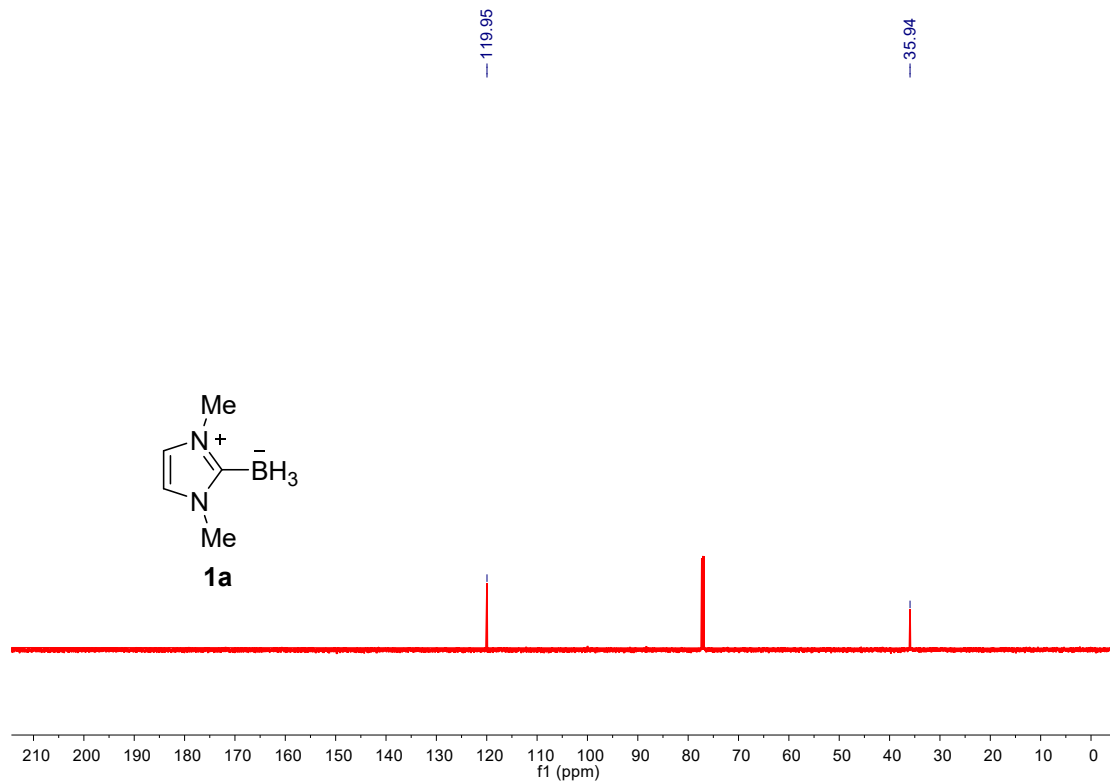




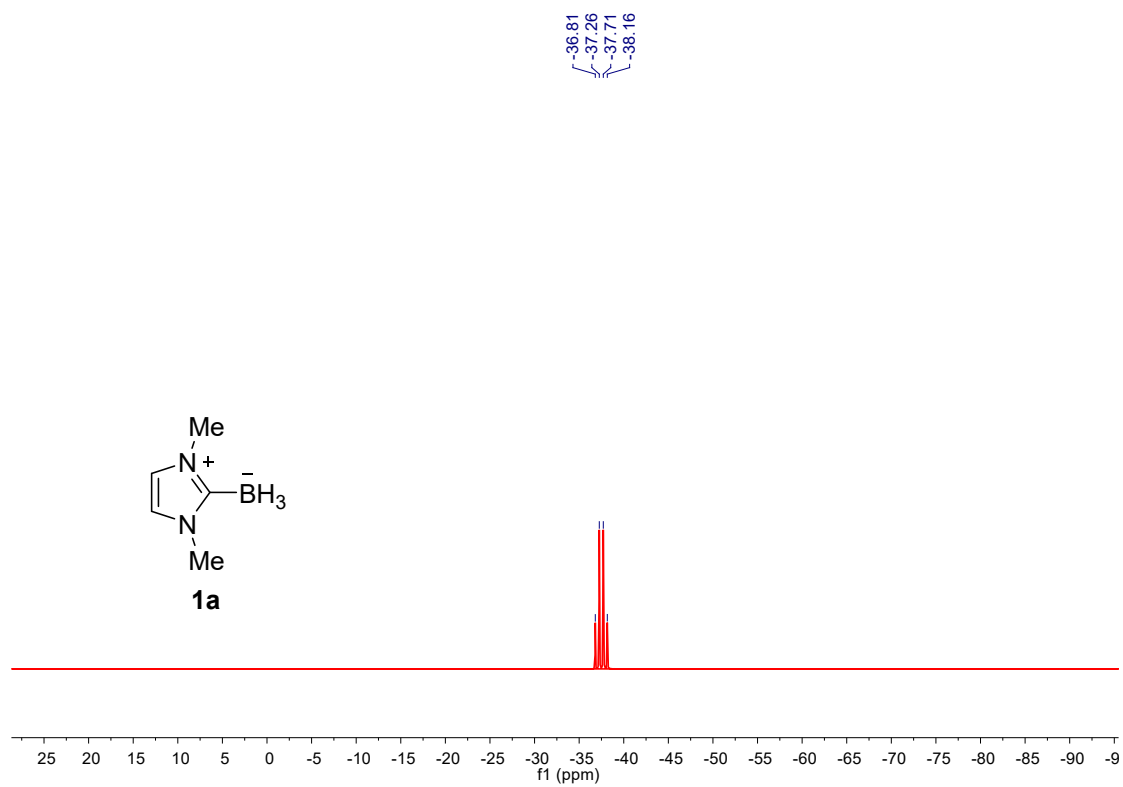
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



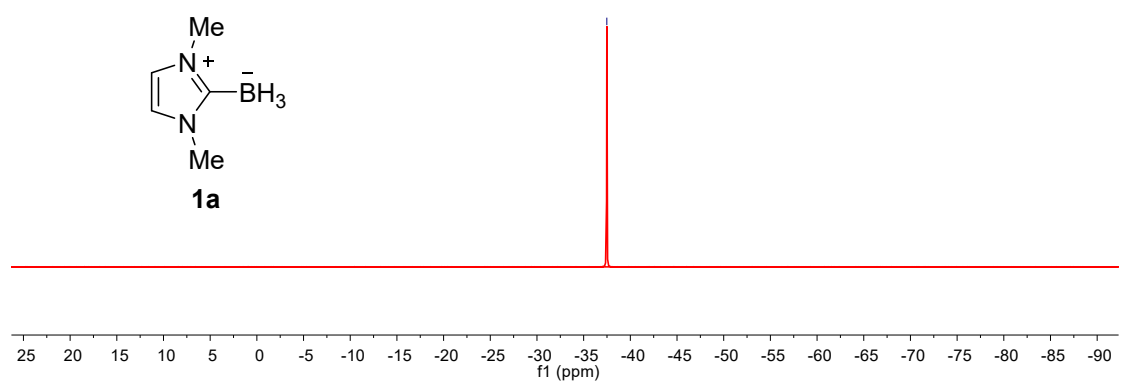
¹H NMR (600 MHz, CDCl₃)



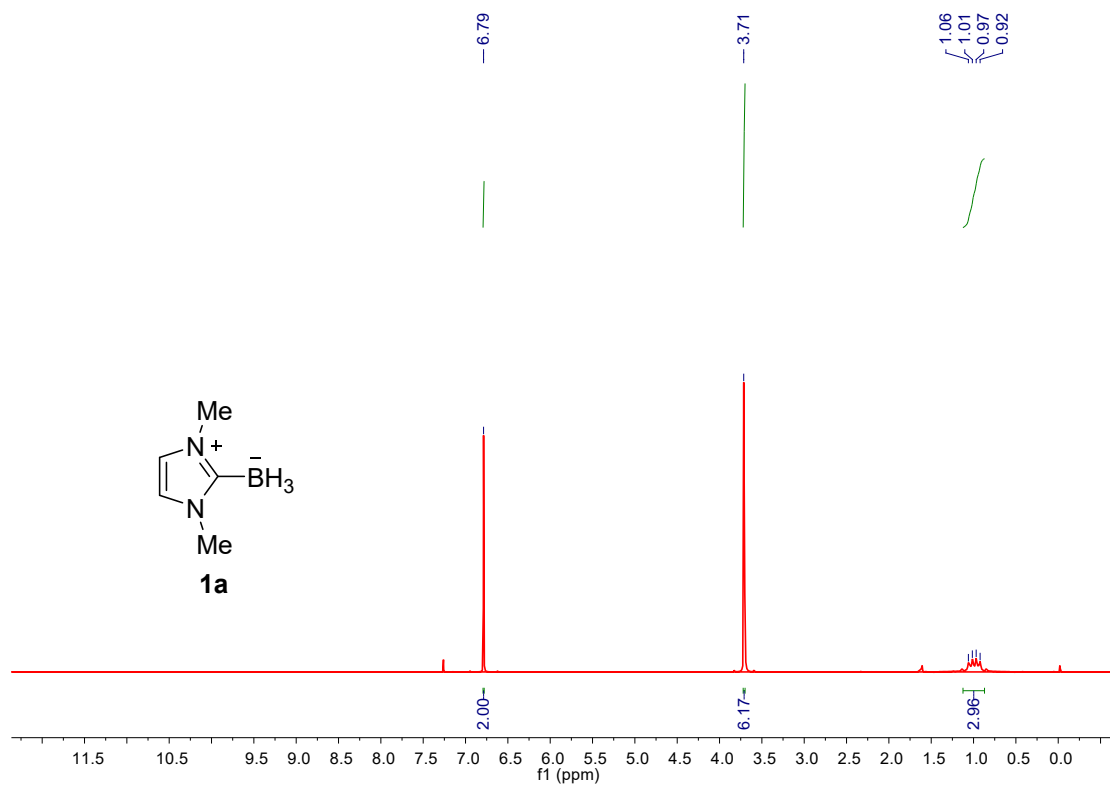
¹³C NMR (151 MHz, CDCl₃)



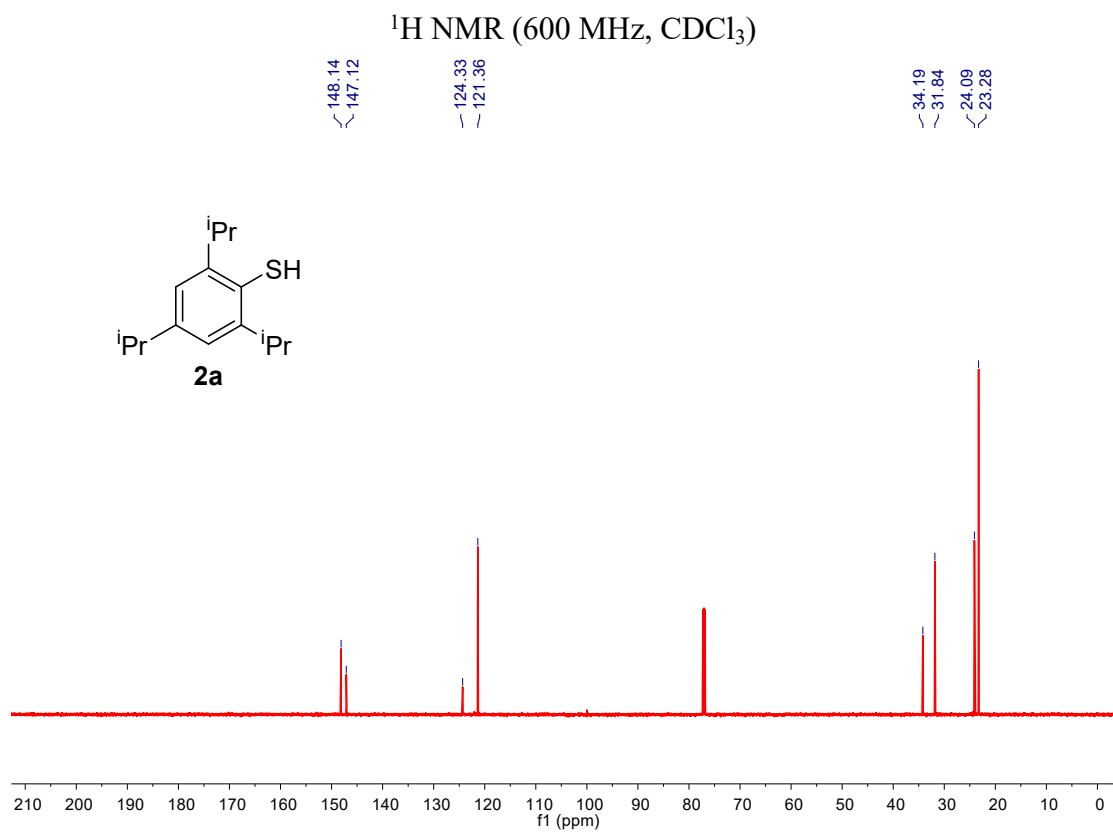
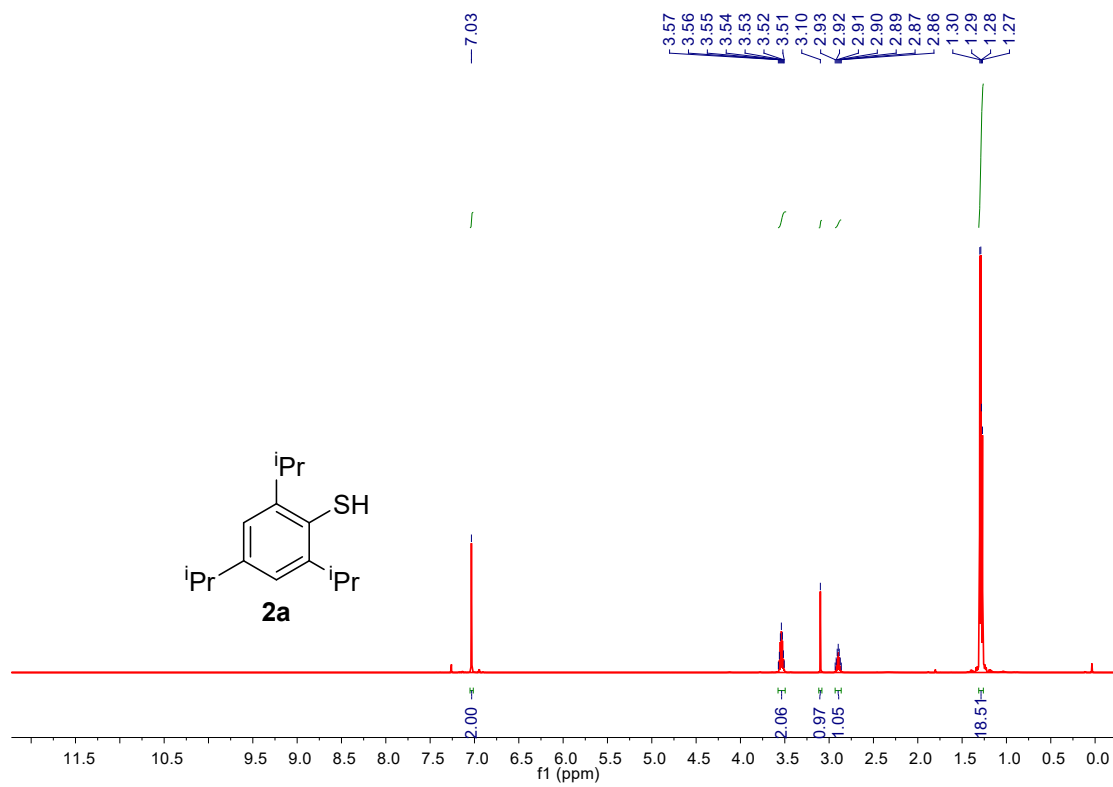
^{11}B NMR (193 MHz, CDCl_3)

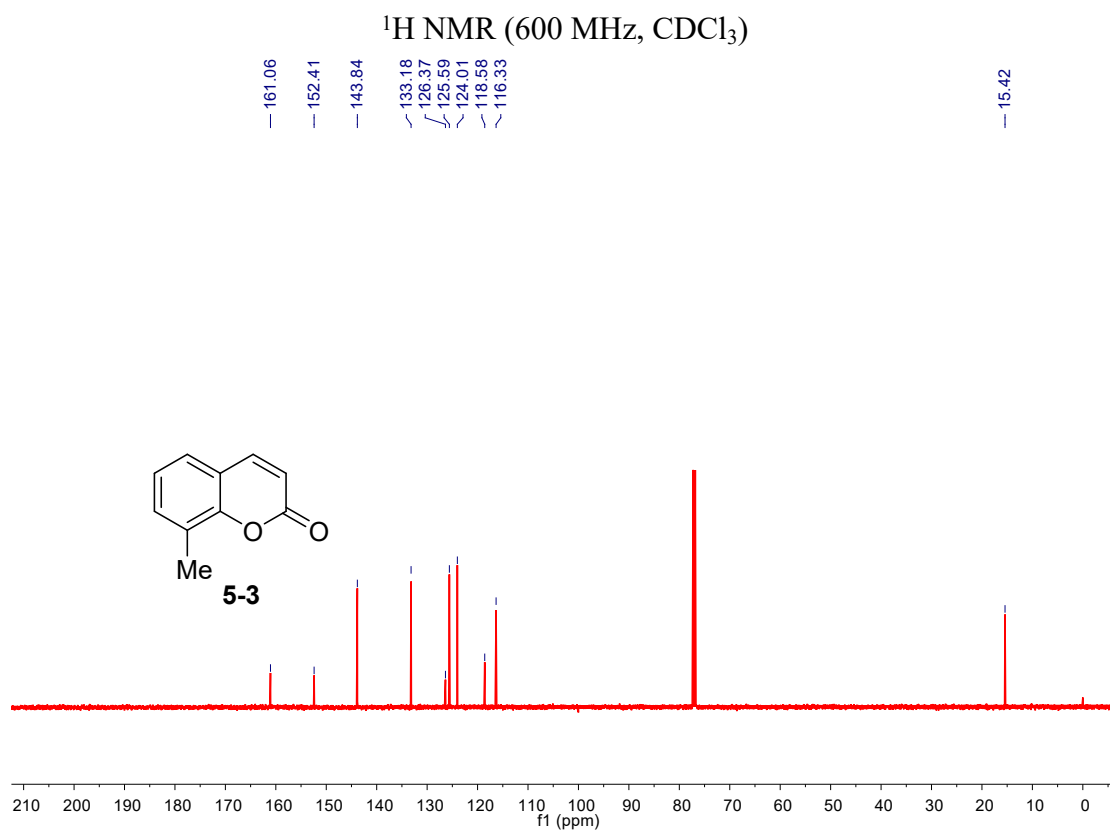
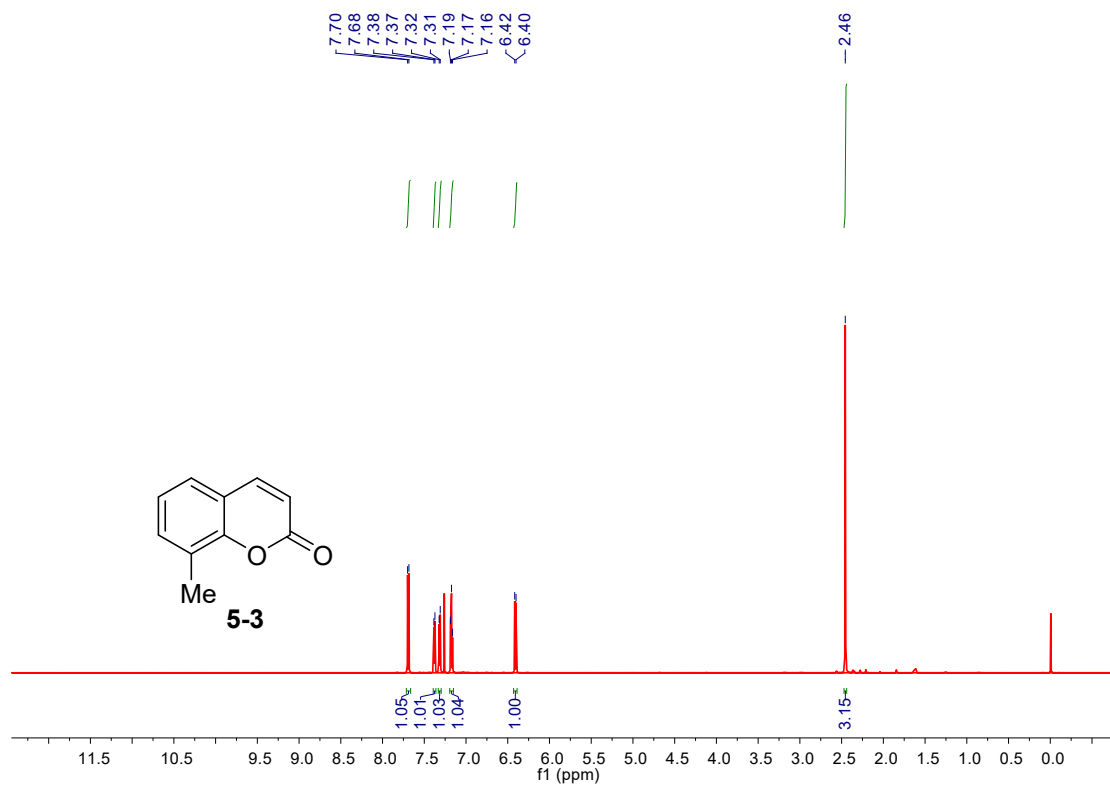


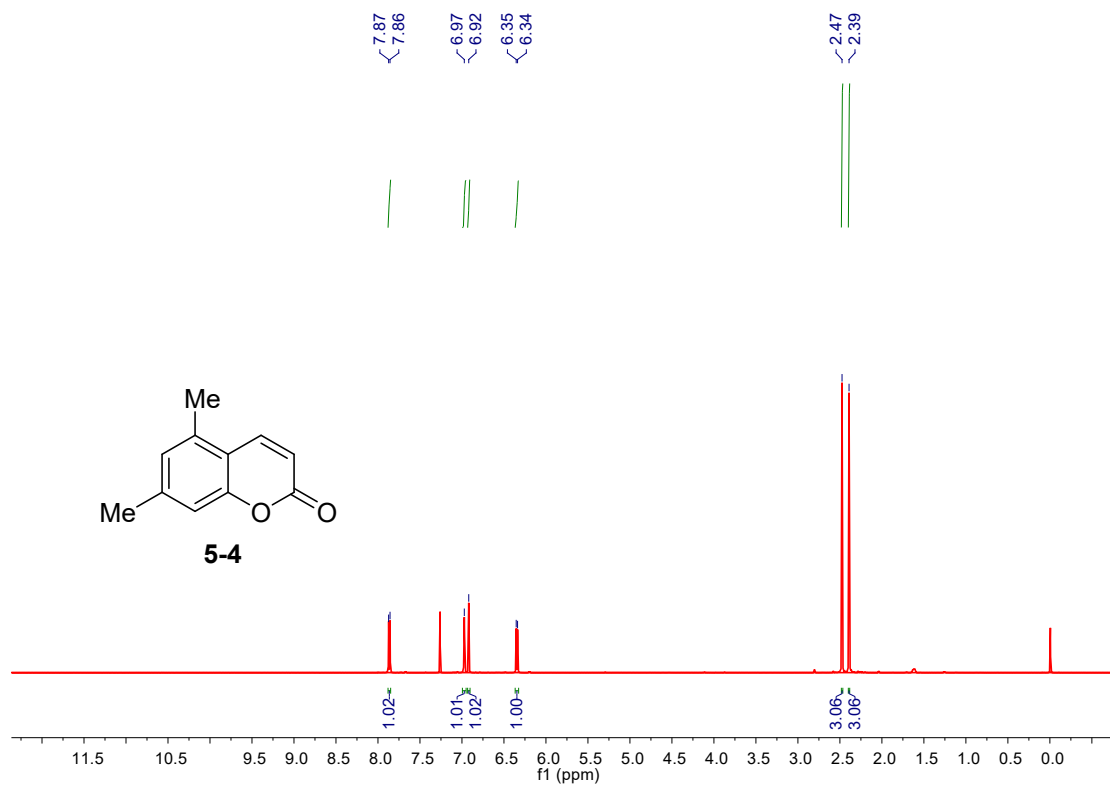
$^{11}\text{B}\{^1\text{H}\}$ NMR (193 MHz, CDCl_3)



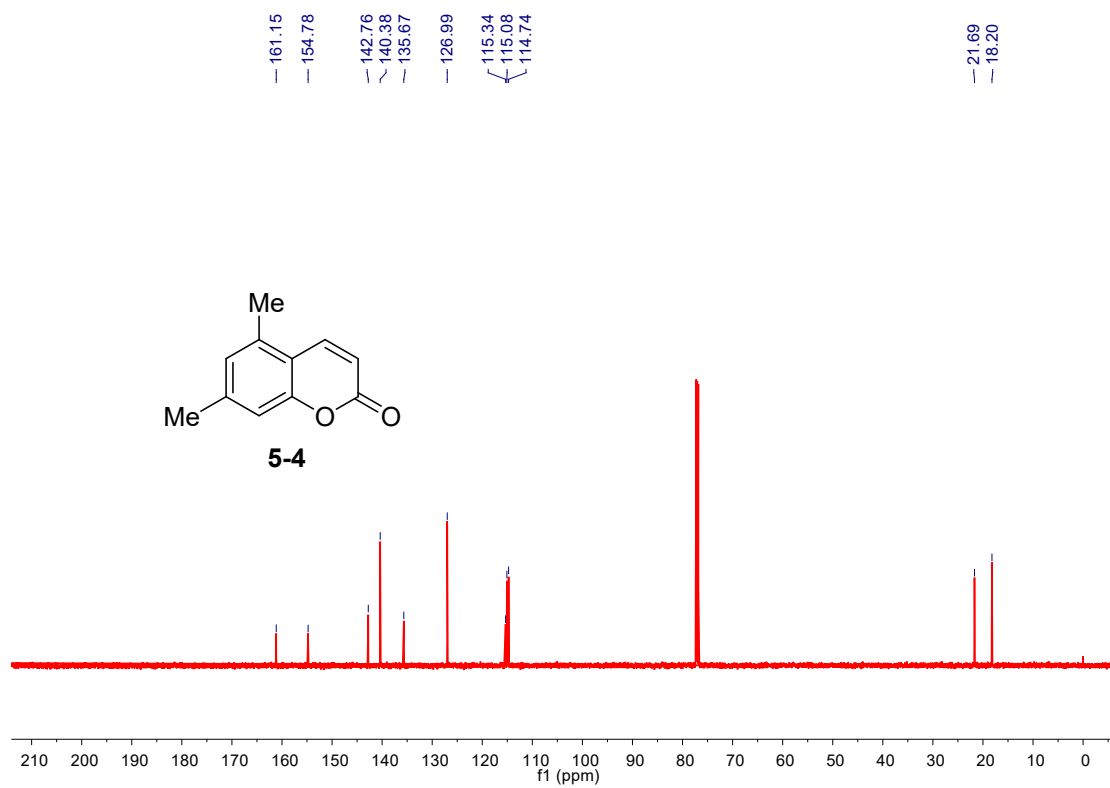
$^1\text{H}\{^{11}\text{B}\}$ NMR (600 MHz, CDCl_3)



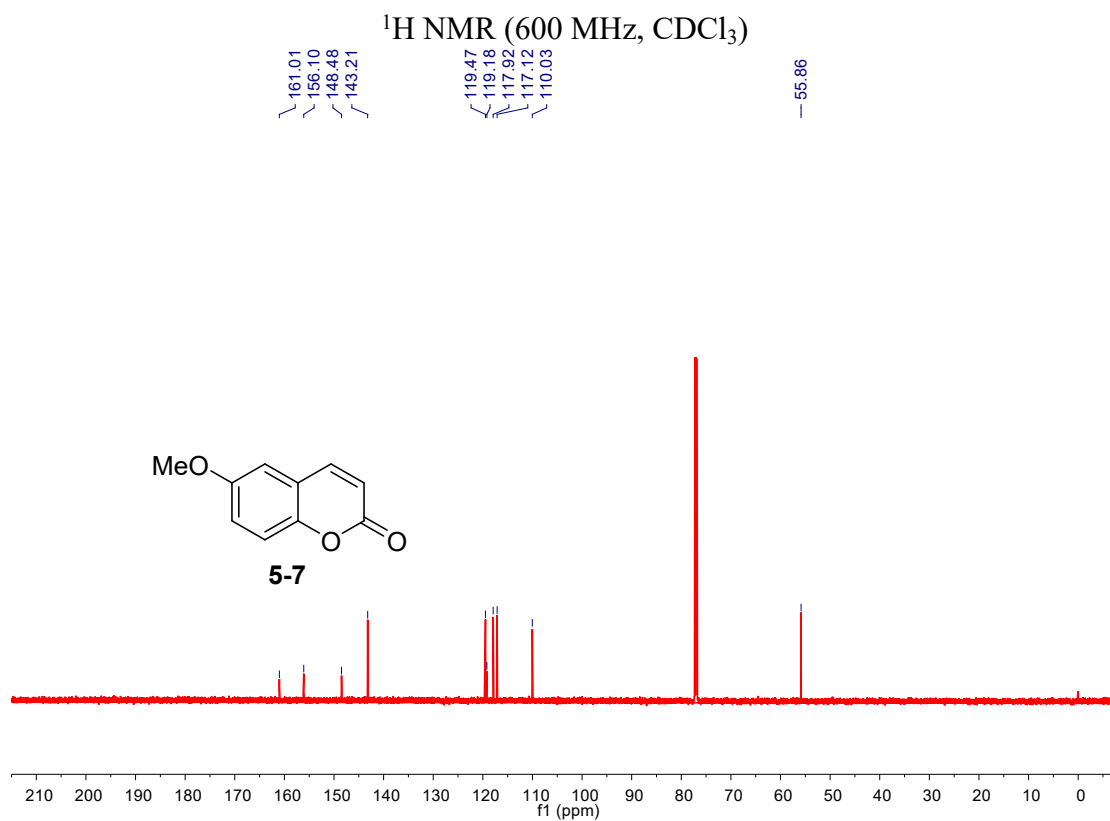
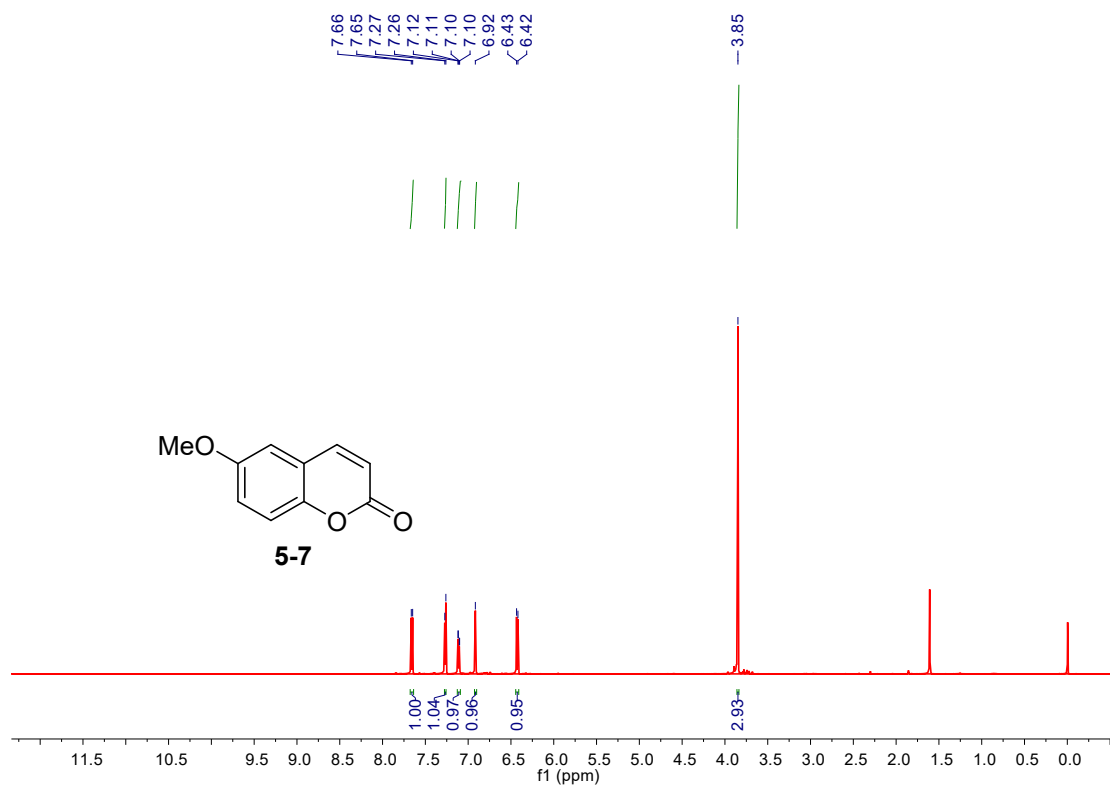


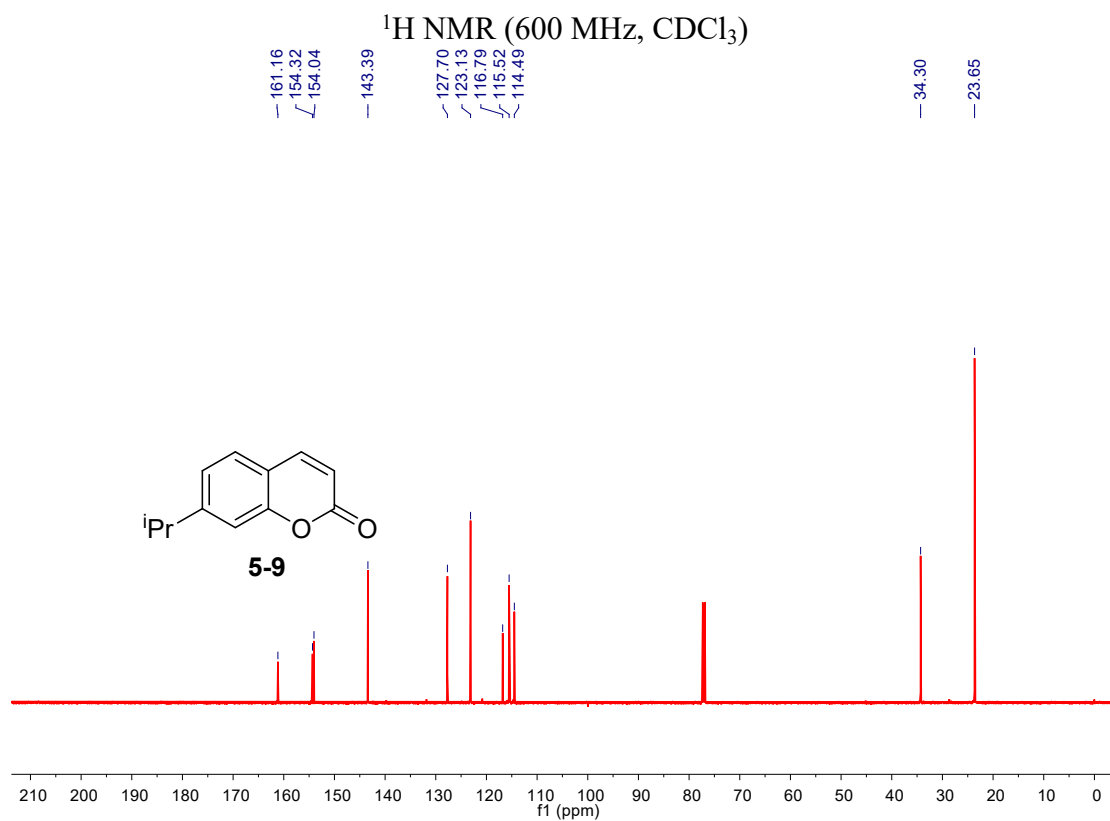
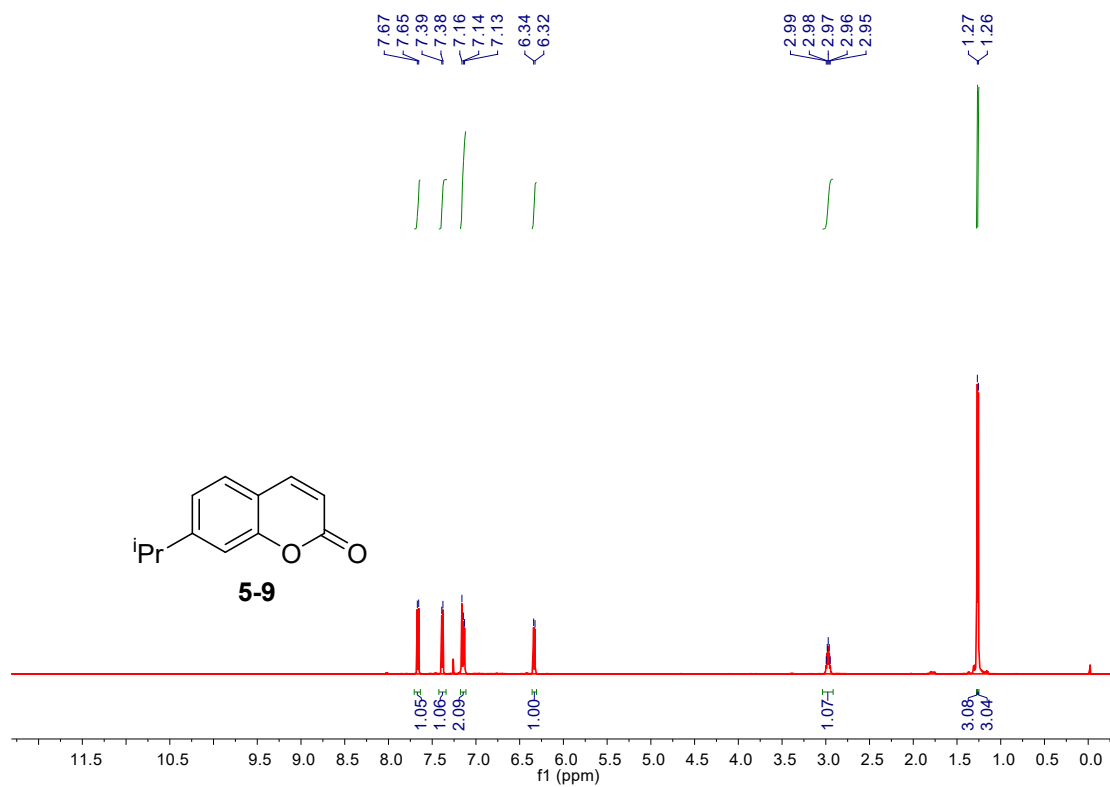


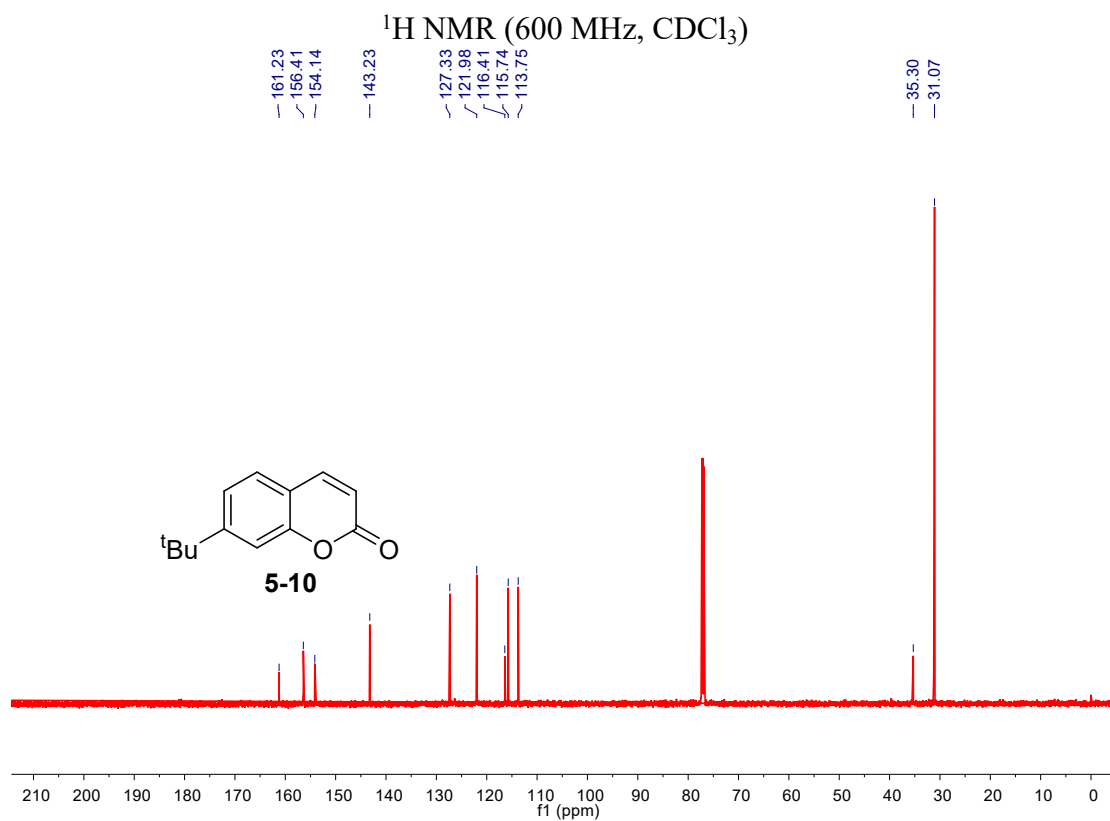
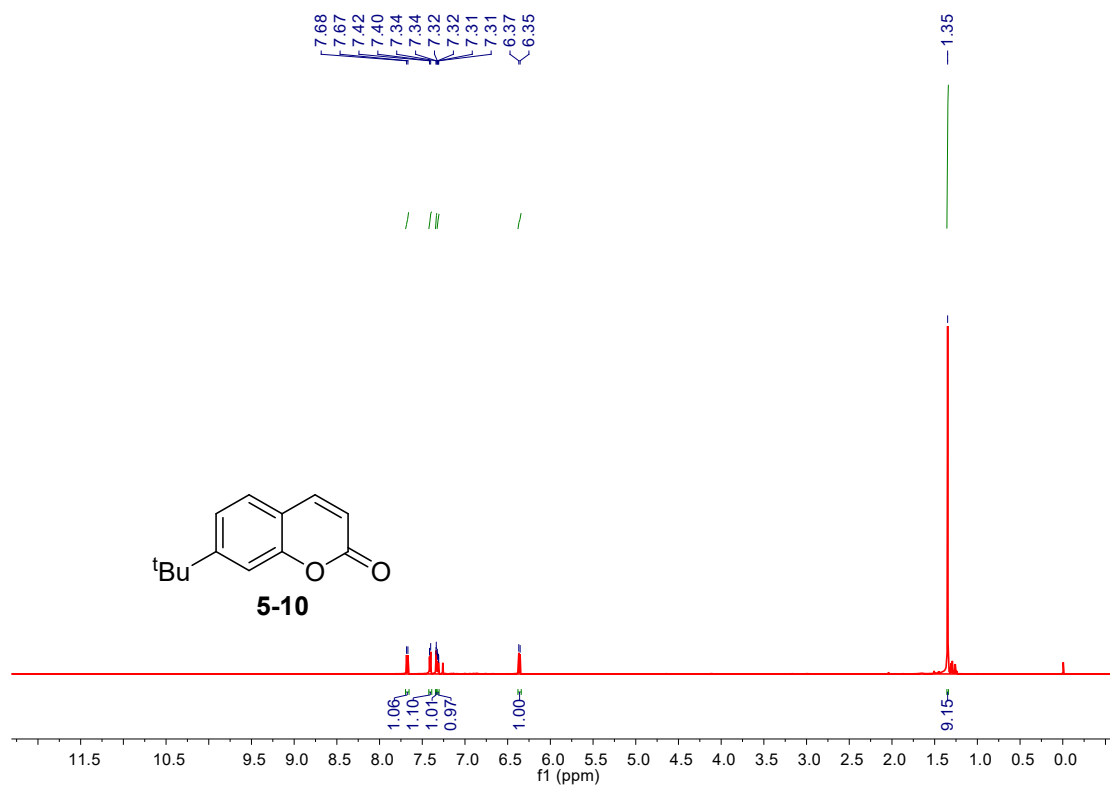
¹H NMR (600 MHz, CDCl₃)

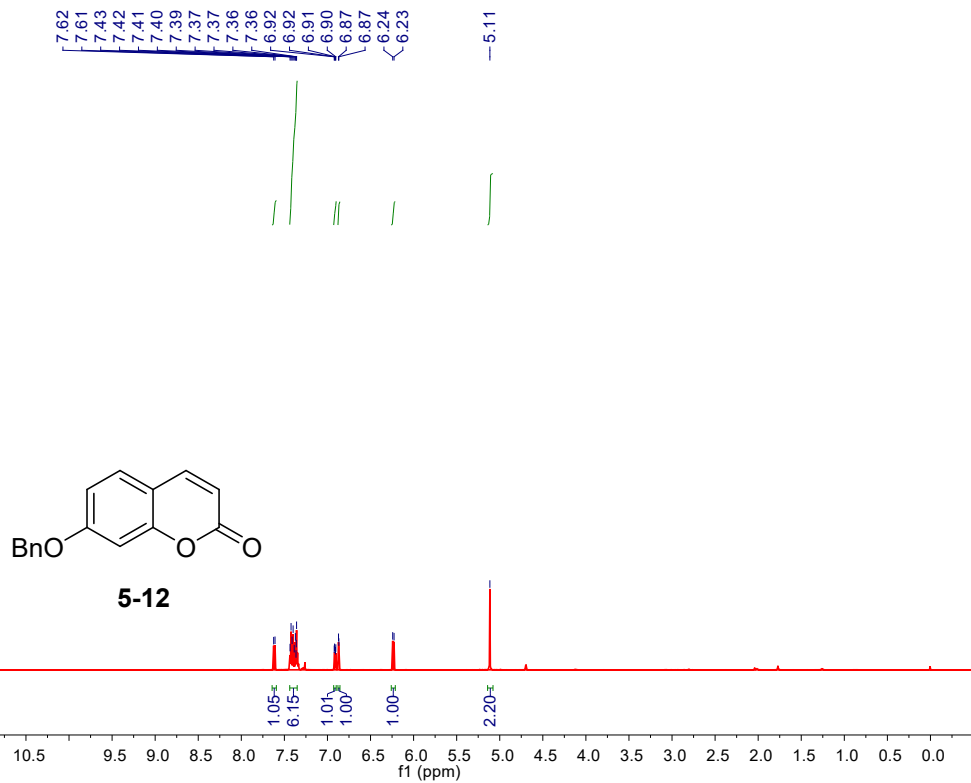


¹³C NMR (151 MHz, CDCl₃)

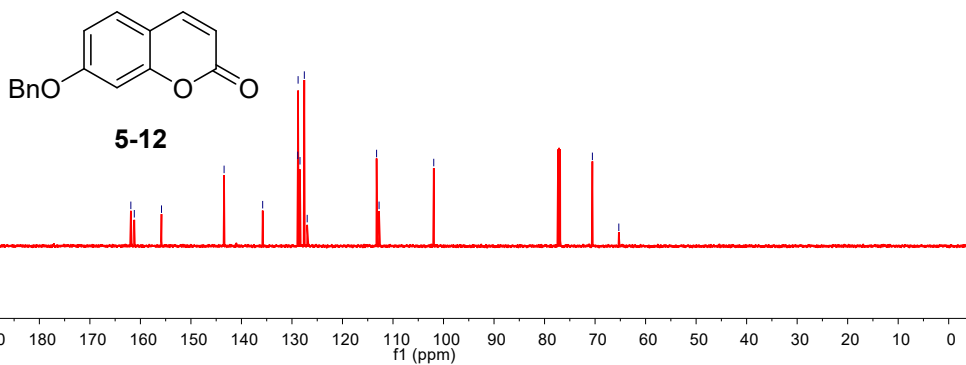
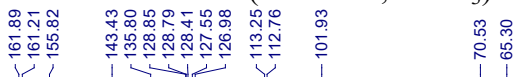




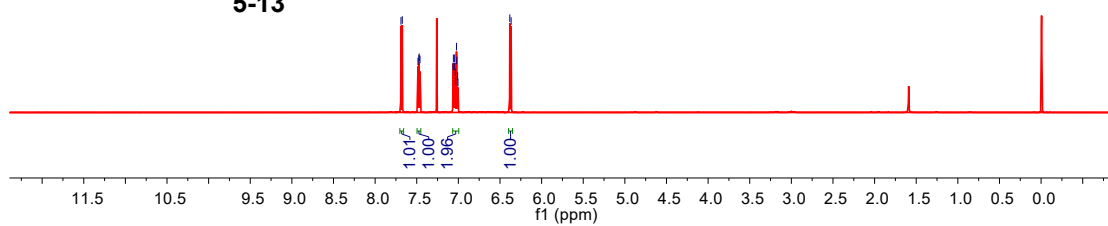
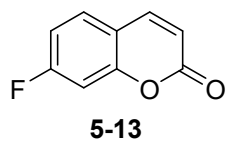
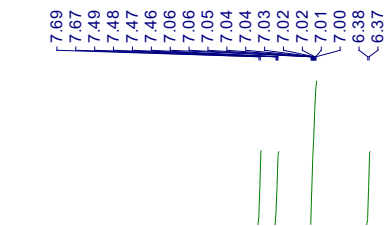




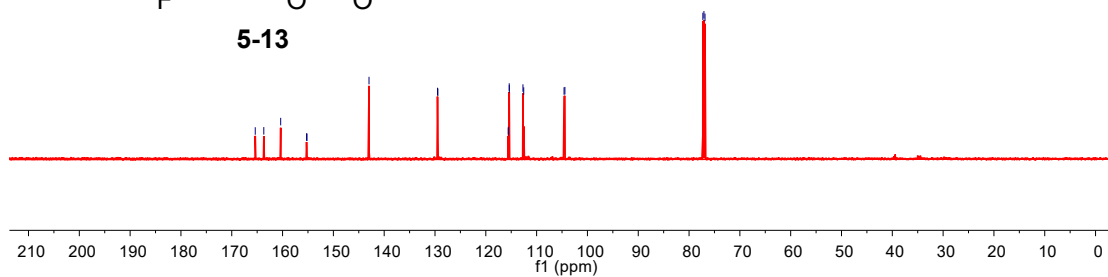
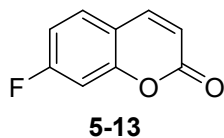
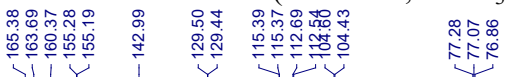
¹H NMR (600 MHz, CDCl₃)



¹³C NMR (151 MHz, CDCl₃)

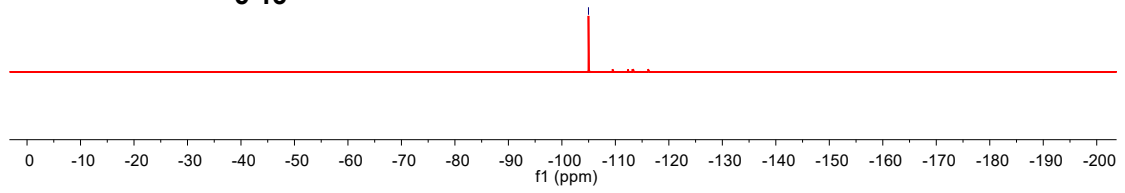
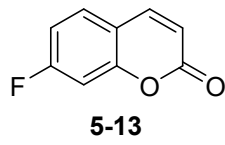


¹H NMR (600 MHz, CDCl₃)

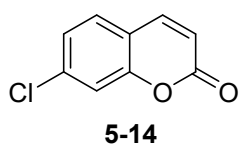
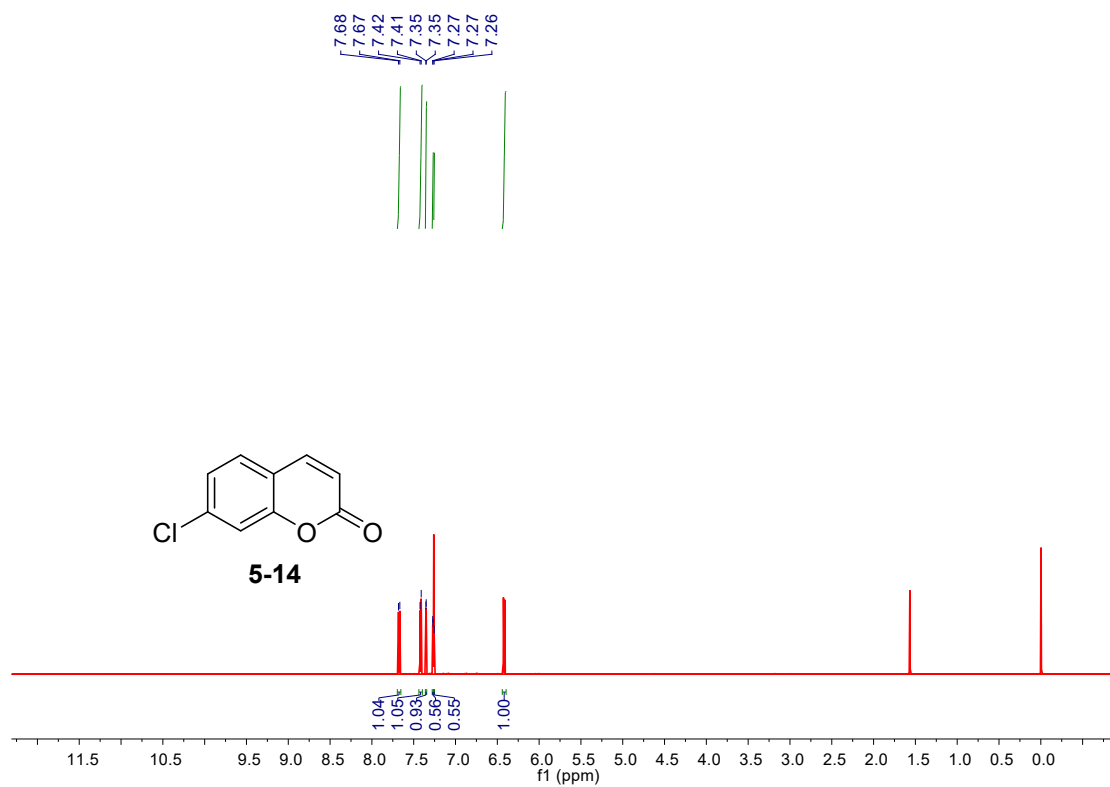


¹³C NMR (151 MHz, CDCl₃)

-104.97

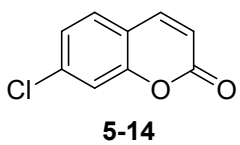
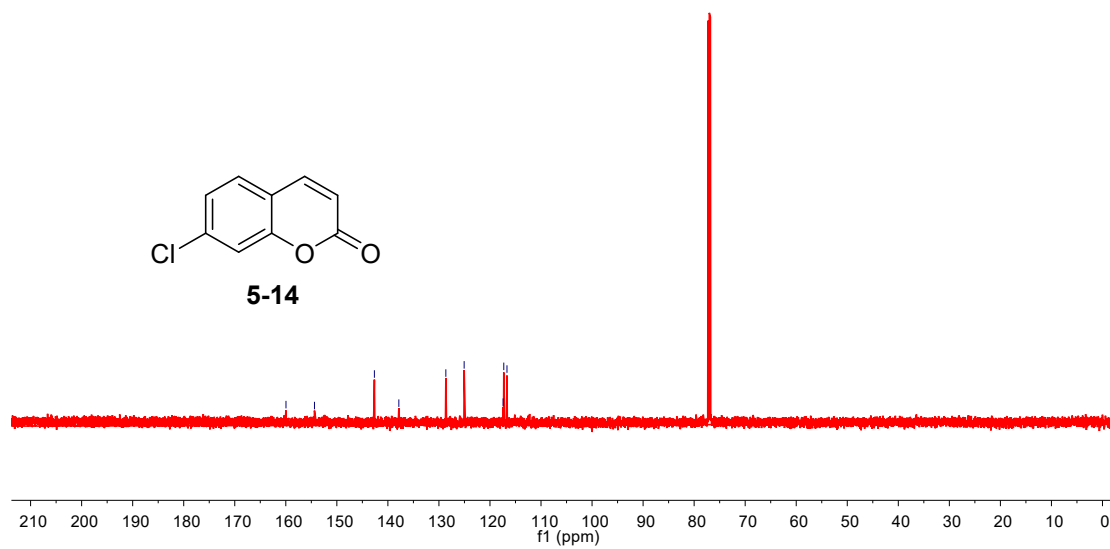


^{19}F NMR (565 MHz, CDCl_3)

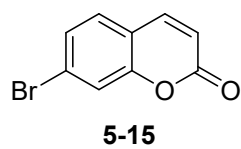
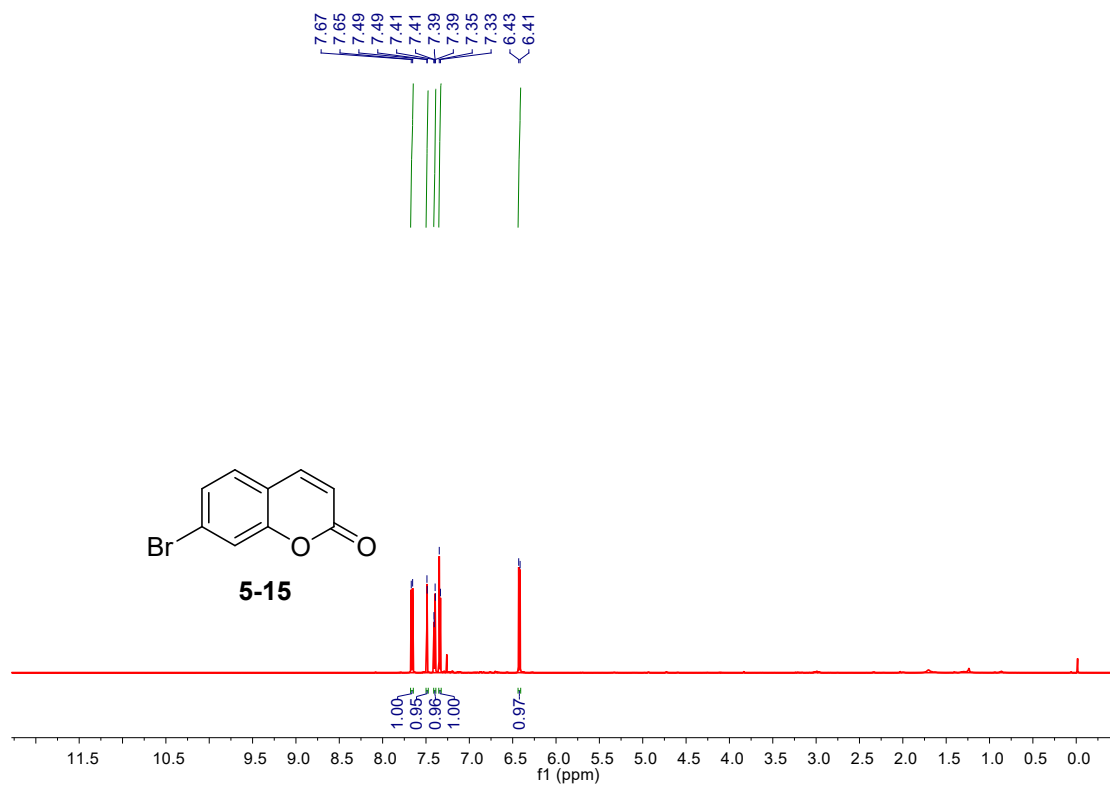


¹H NMR (600 MHz, CDCl₃)

— 159.96
 — 154.38
 — 142.64
 — 137.85
 — 128.64
 — 125.06
 — 117.42
 — 117.29
 — 116.67

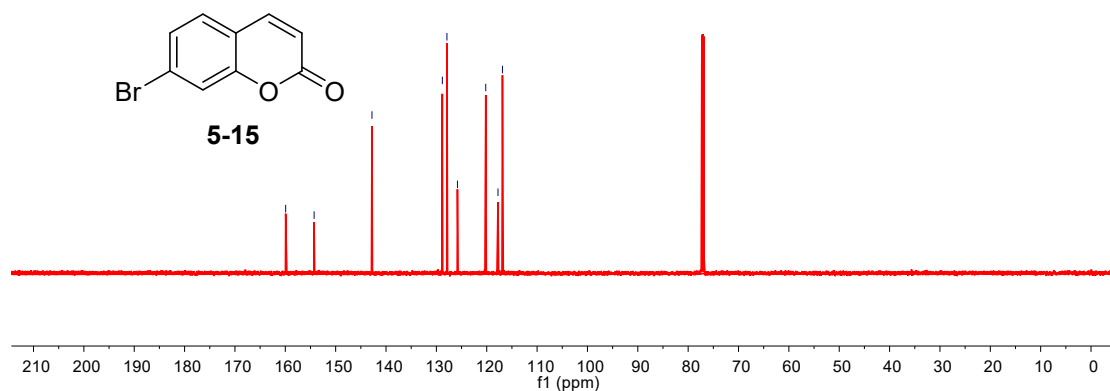


¹³C NMR (151 MHz, CDCl₃)

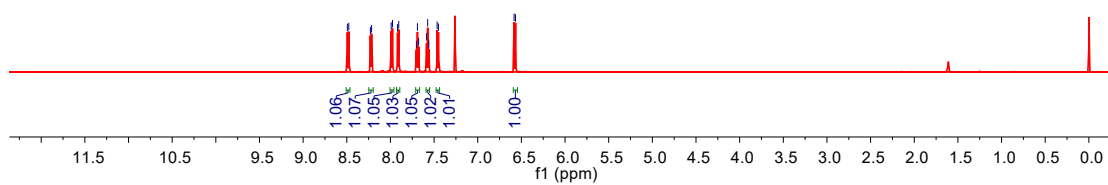
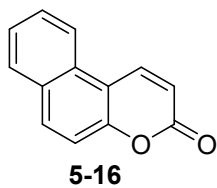
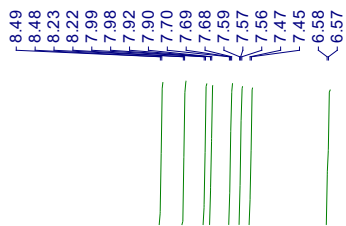


¹H NMR (600 MHz, CDCl₃)

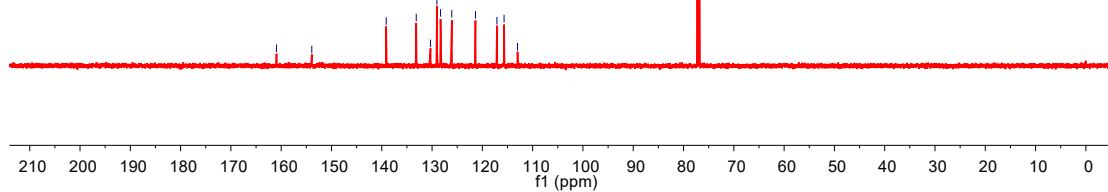
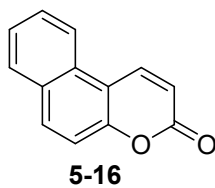
— 159.93
 — 154.28
 — 142.79
 — 128.81
 — 127.91
 — 125.82
 — 120.20
 — 117.76
 — 116.88



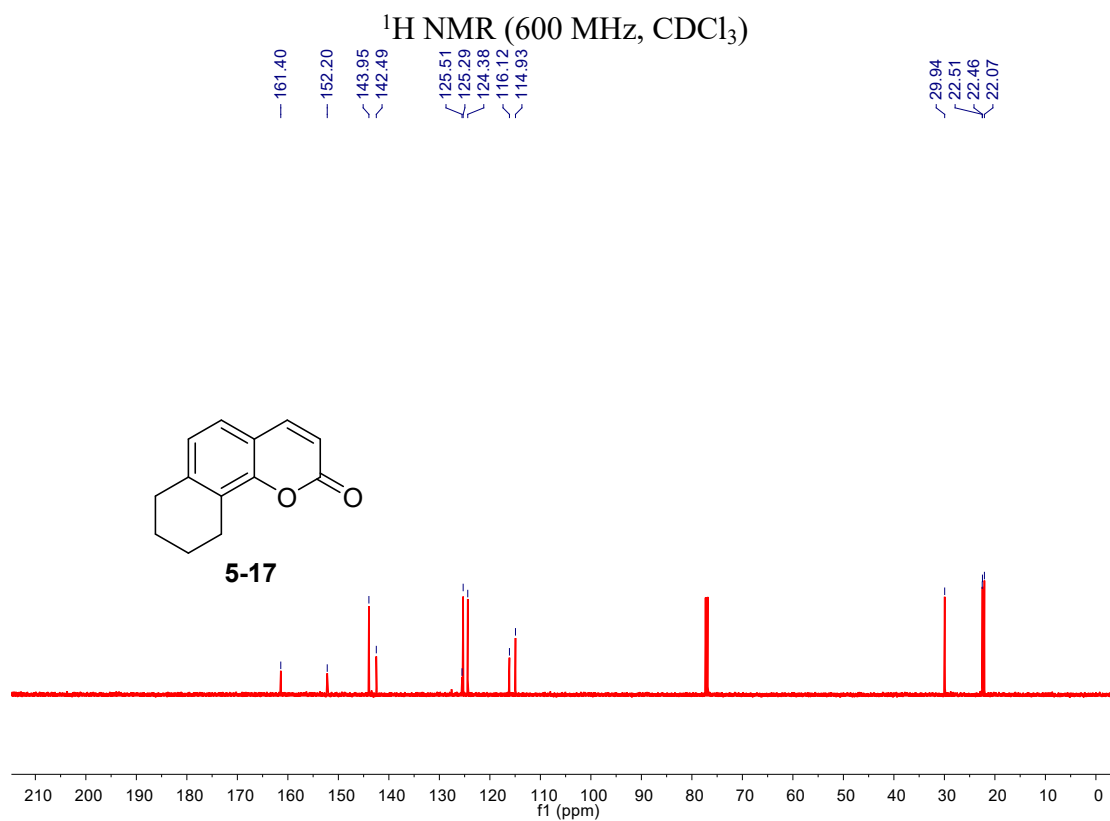
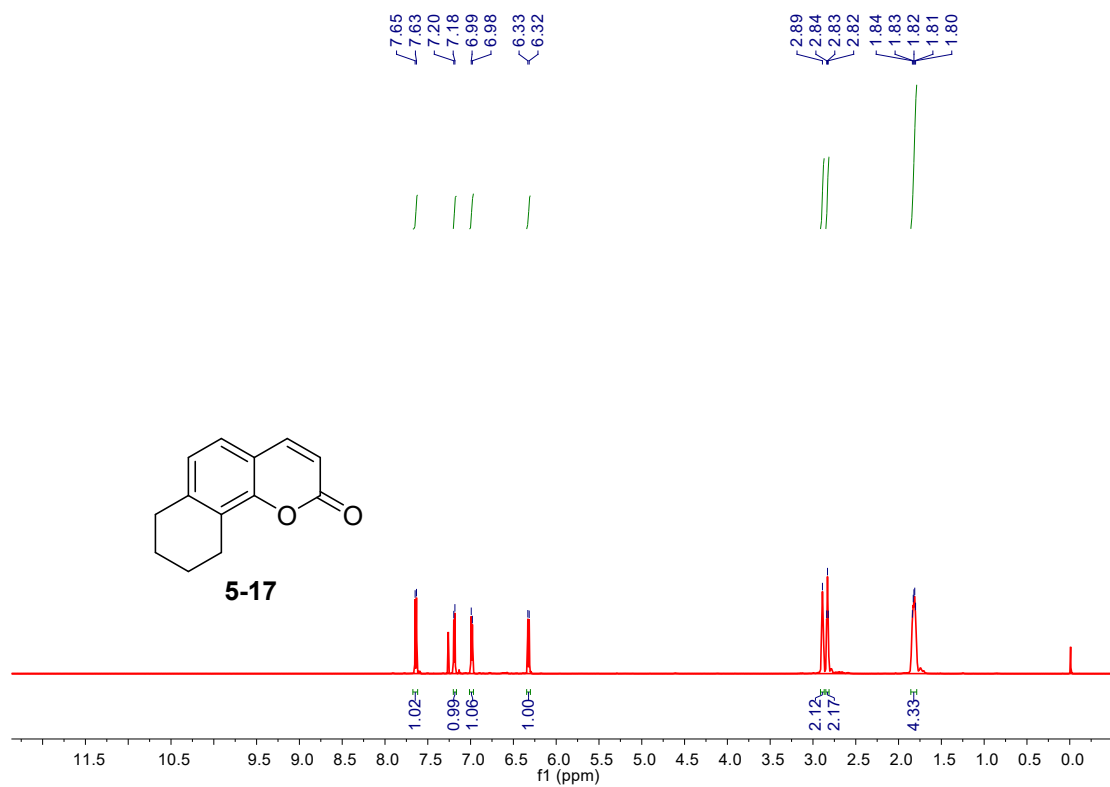
¹³C NMR (151 MHz, CDCl₃)

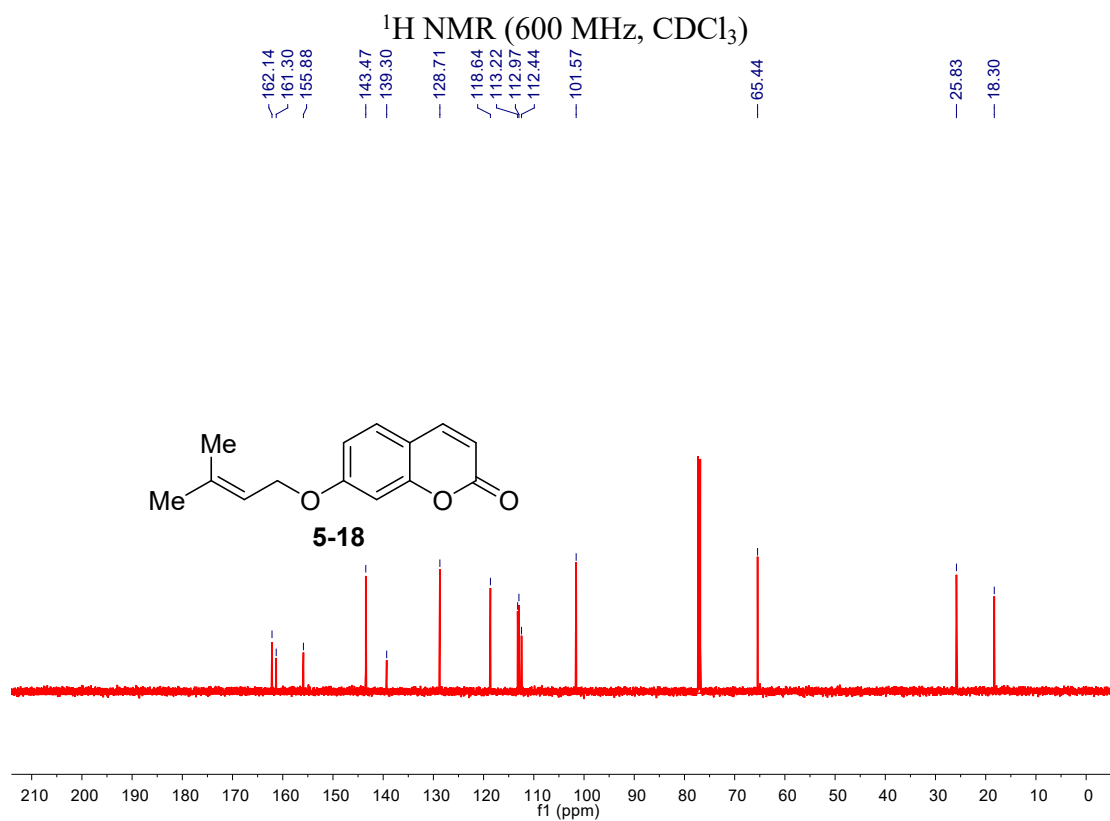
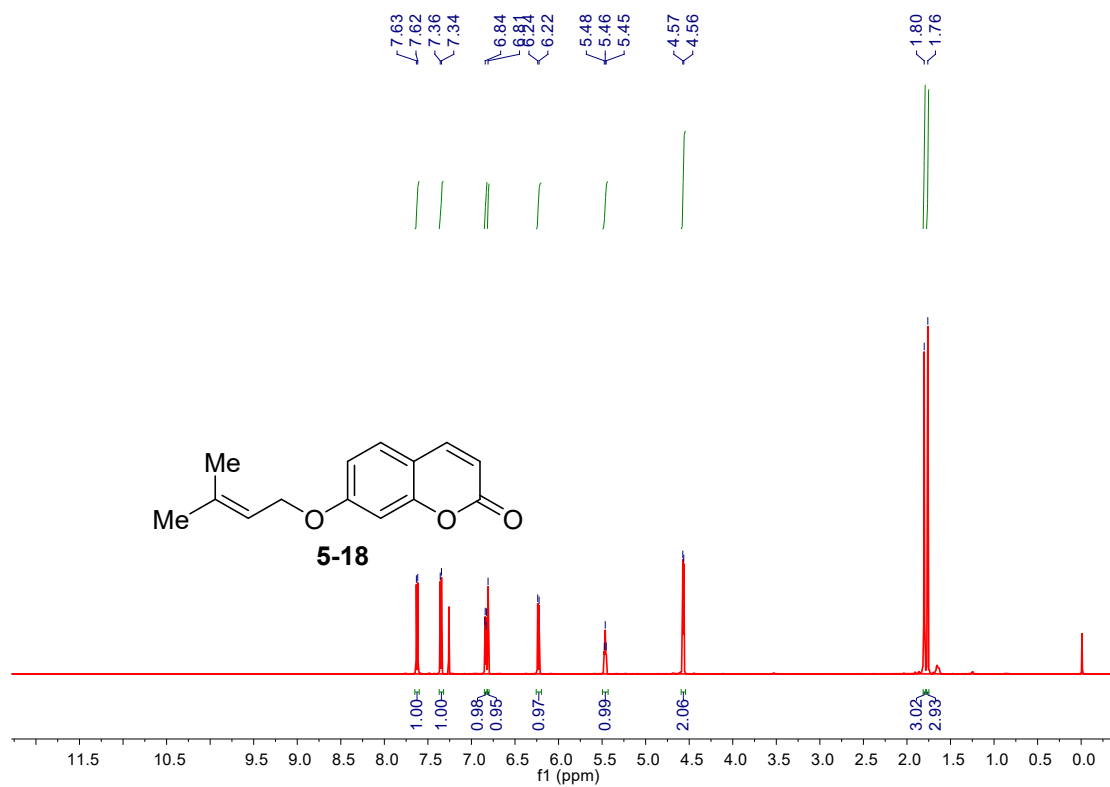


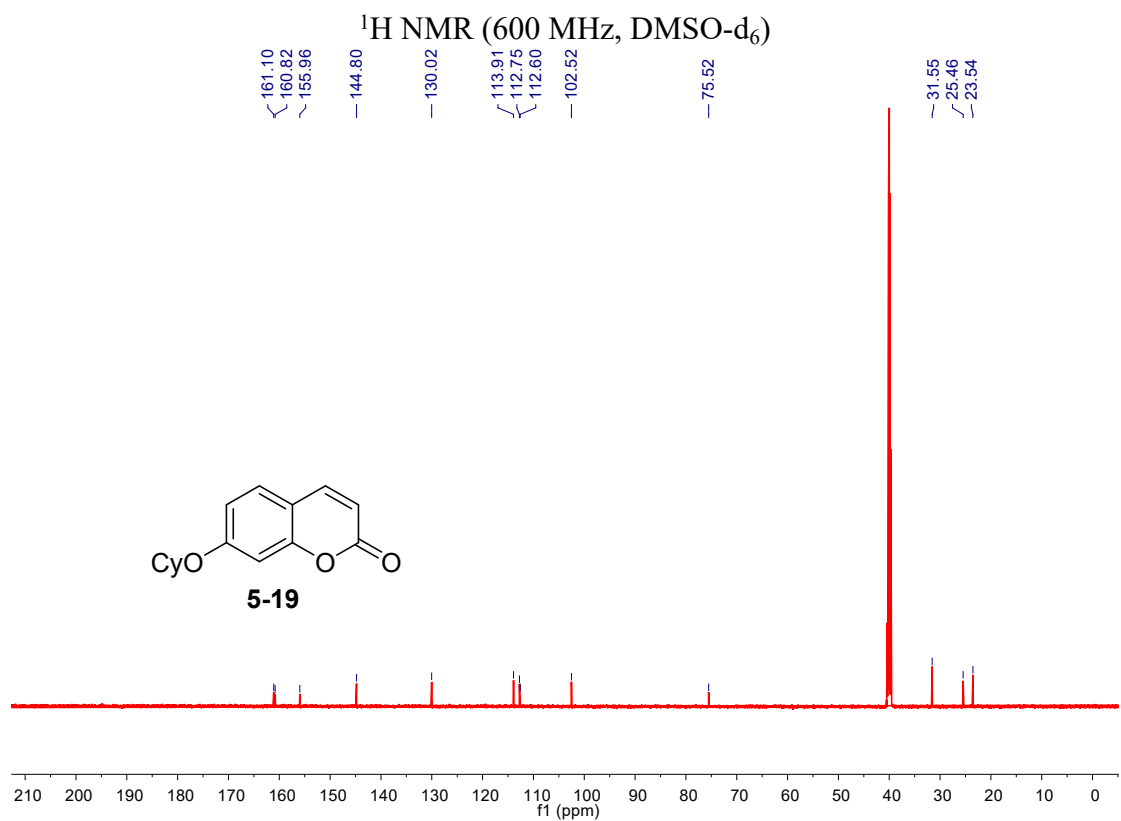
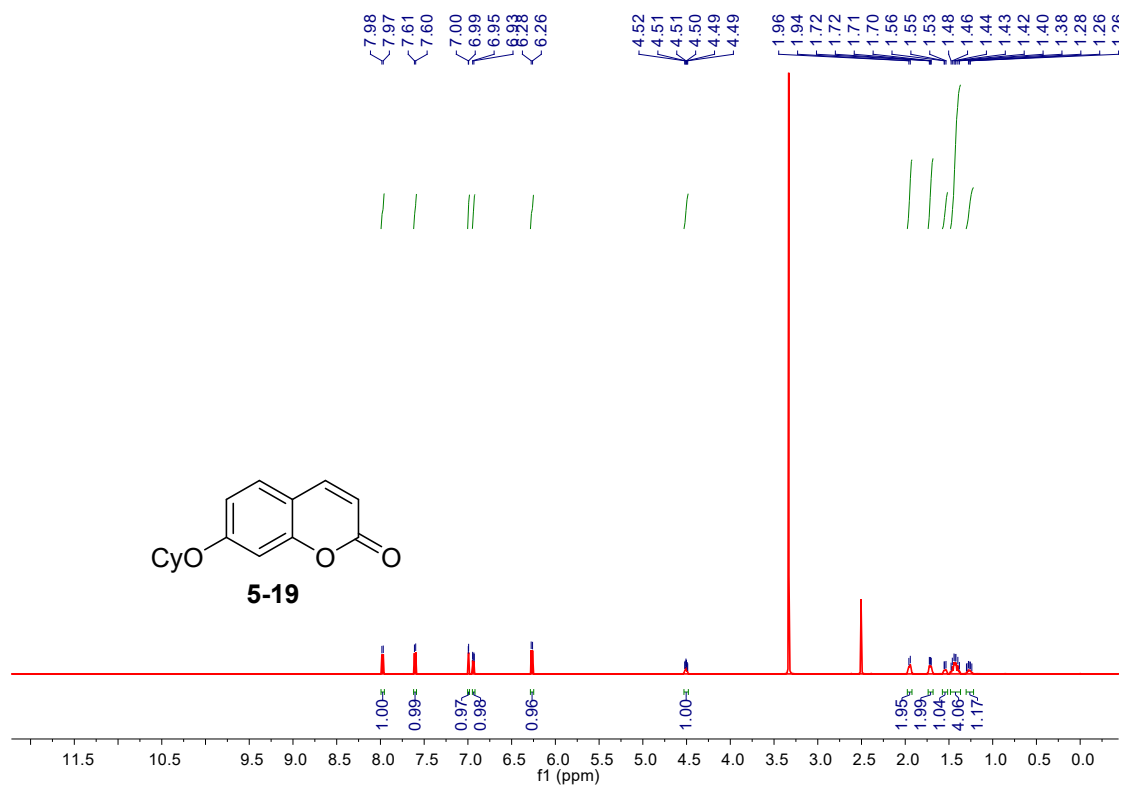
¹H NMR (600 MHz, CDCl₃)

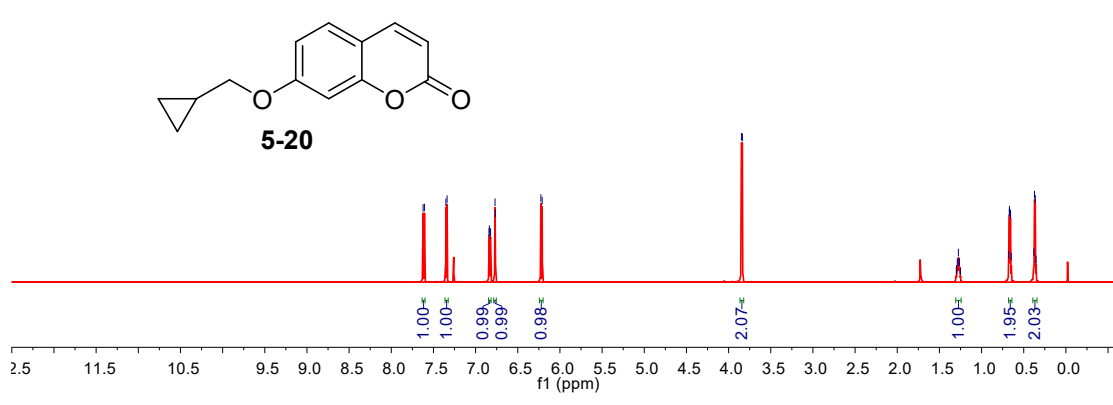
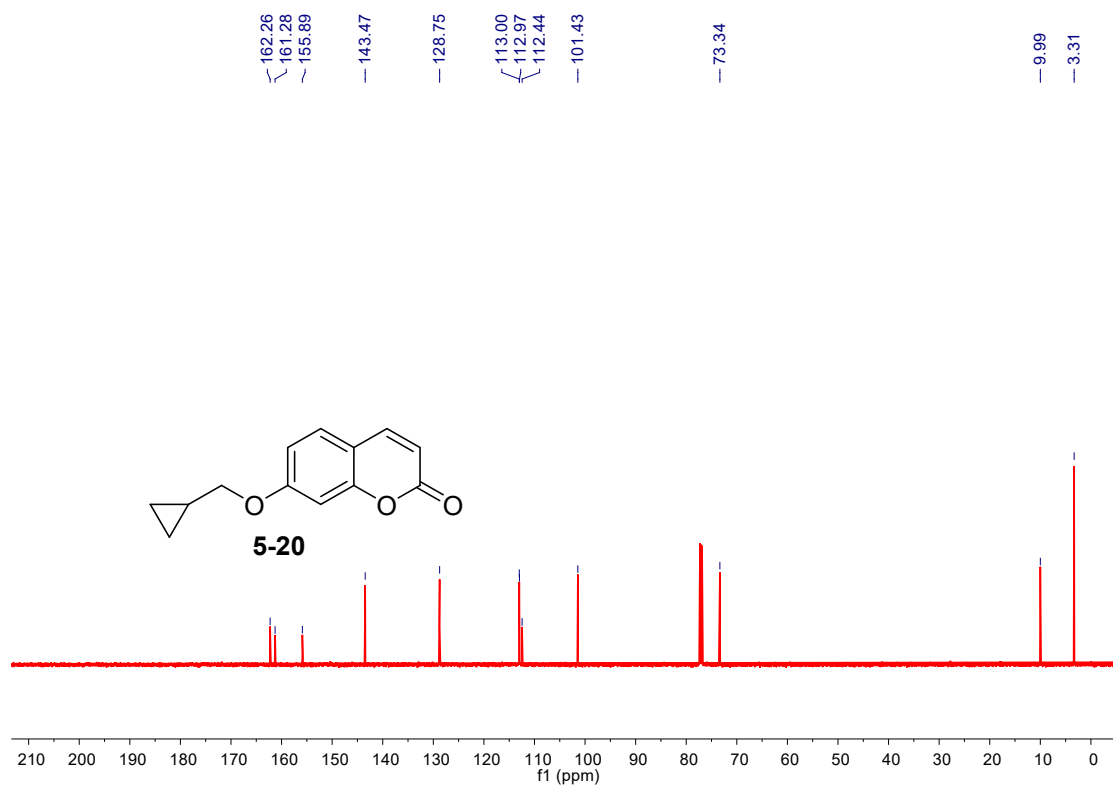


¹³C NMR (151 MHz, CDCl₃)

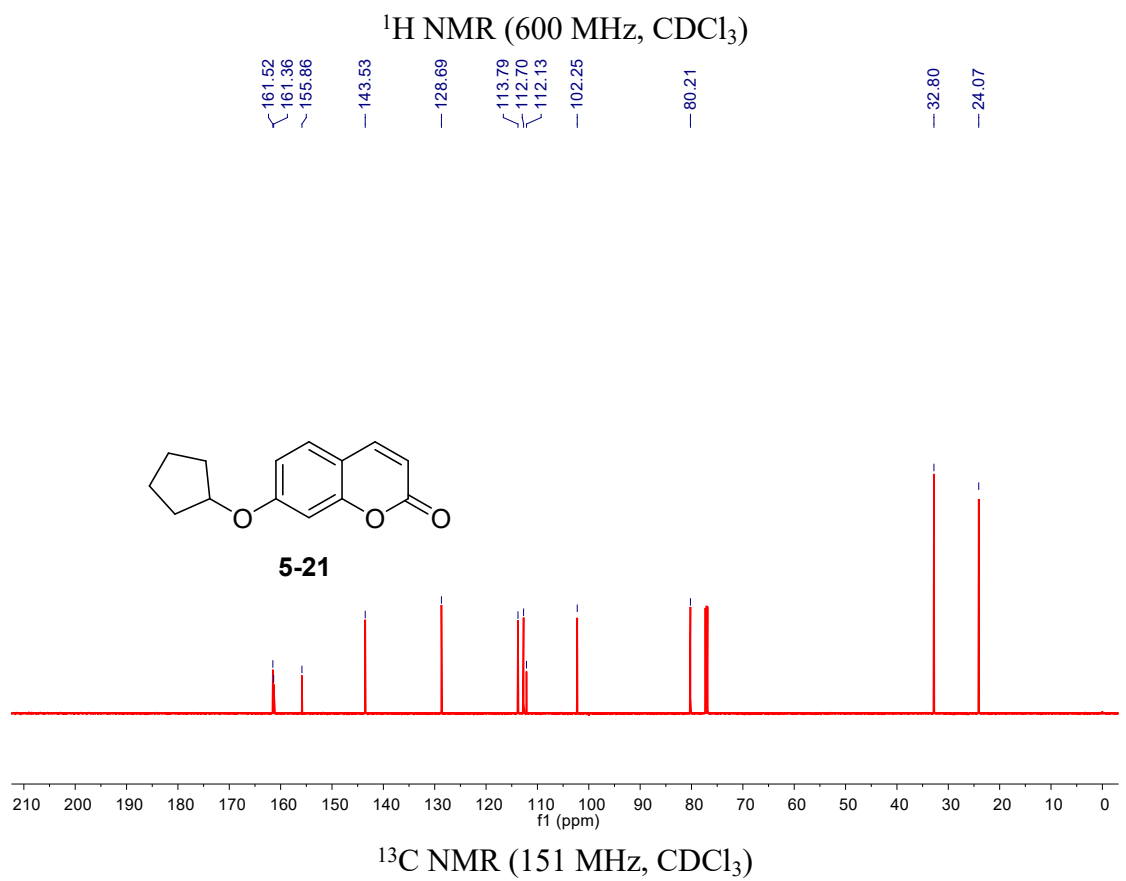
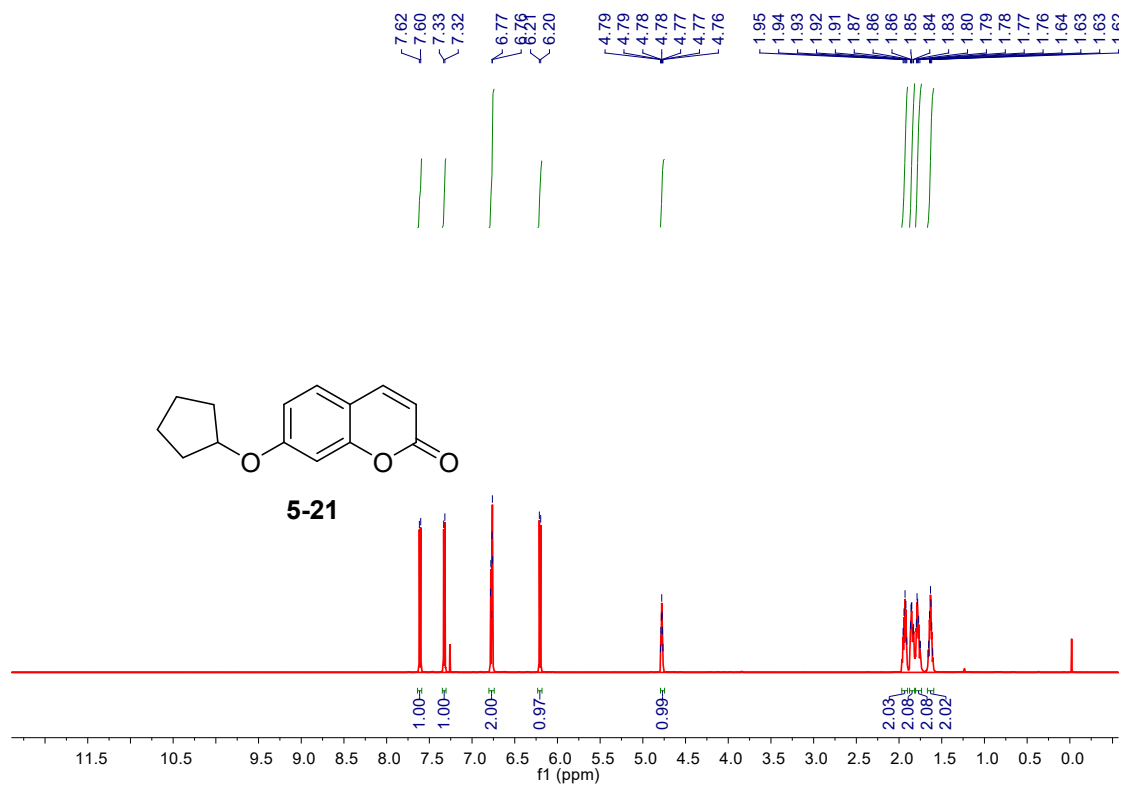


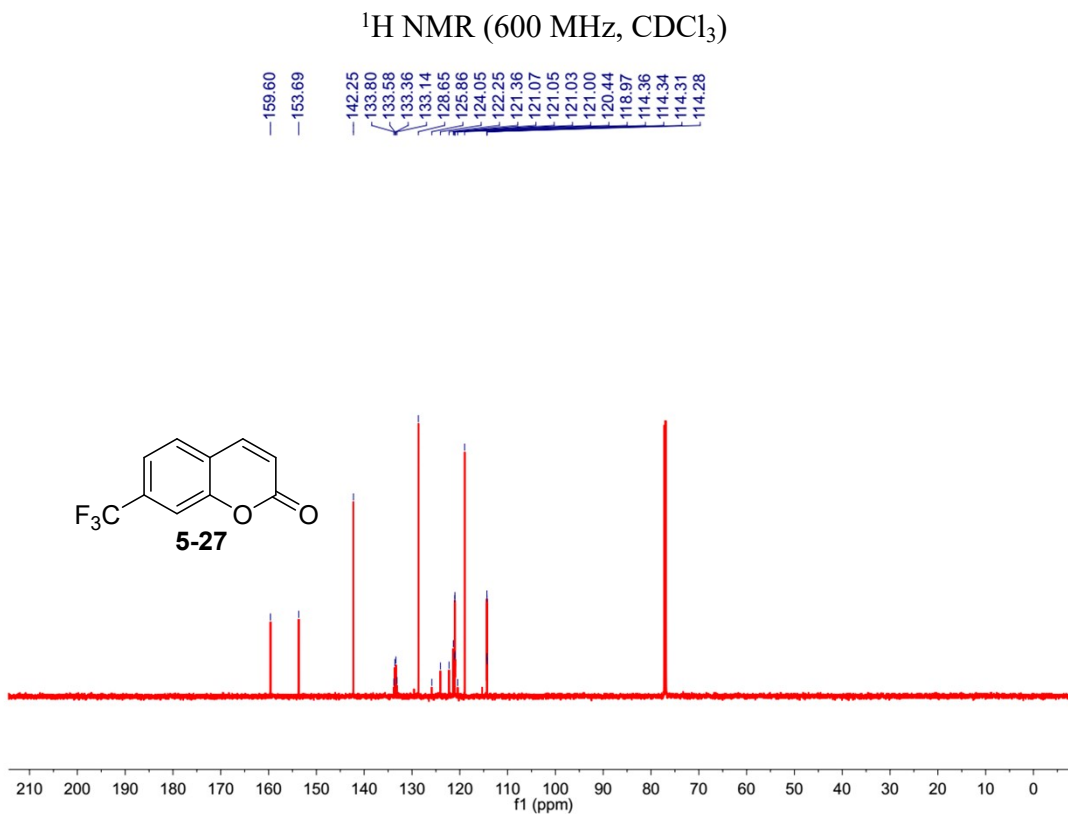
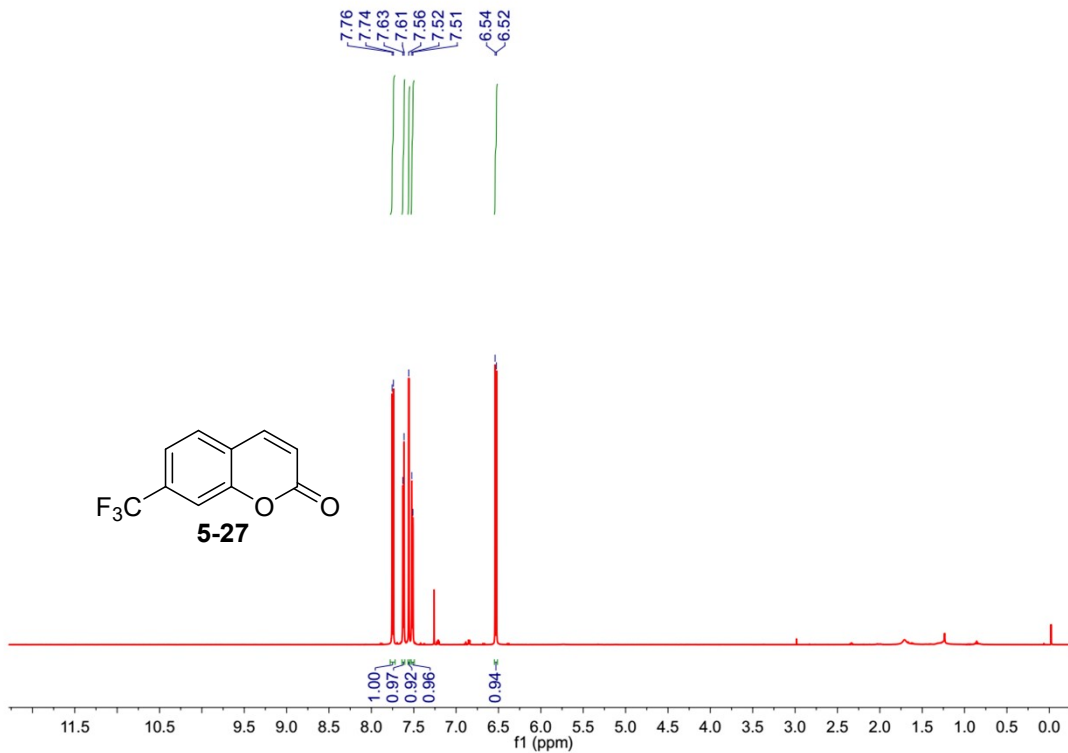


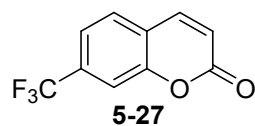
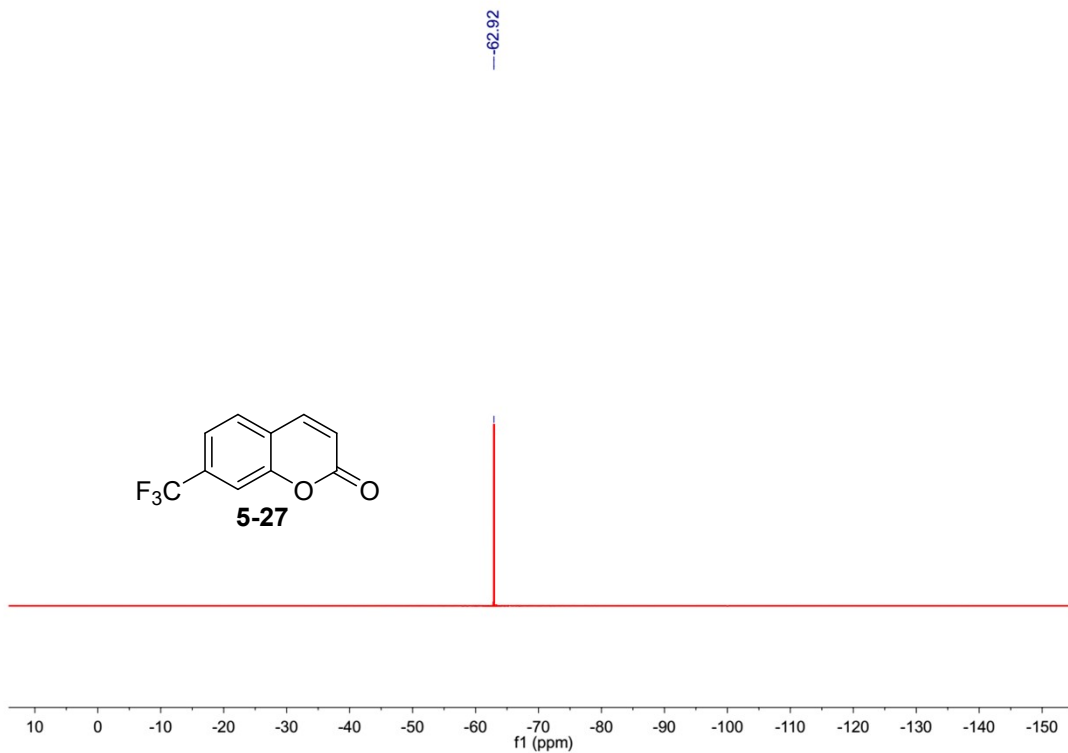




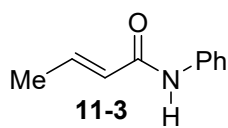
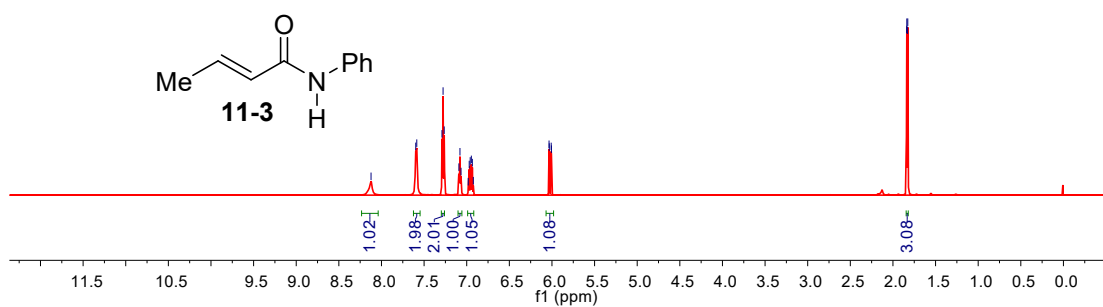
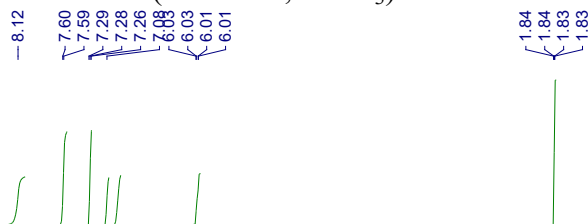
^{13}C NMR (151 MHz, CDCl_3)



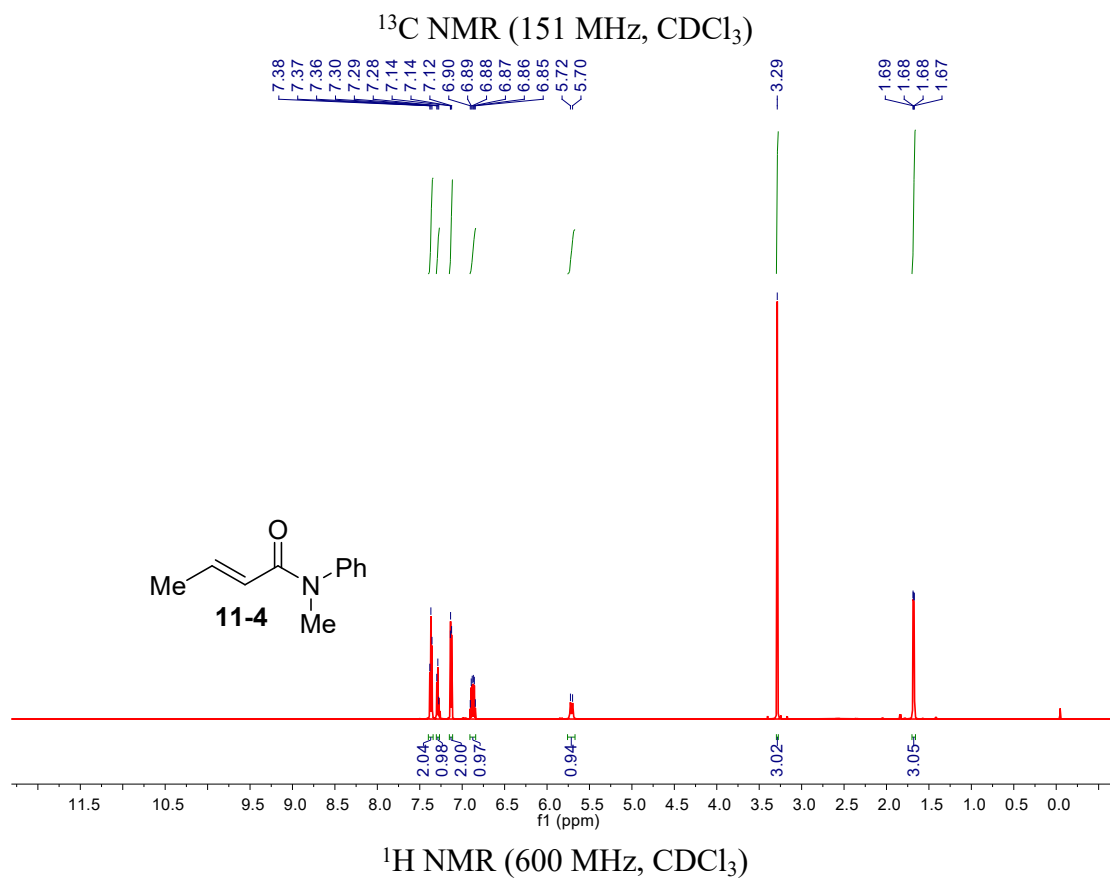
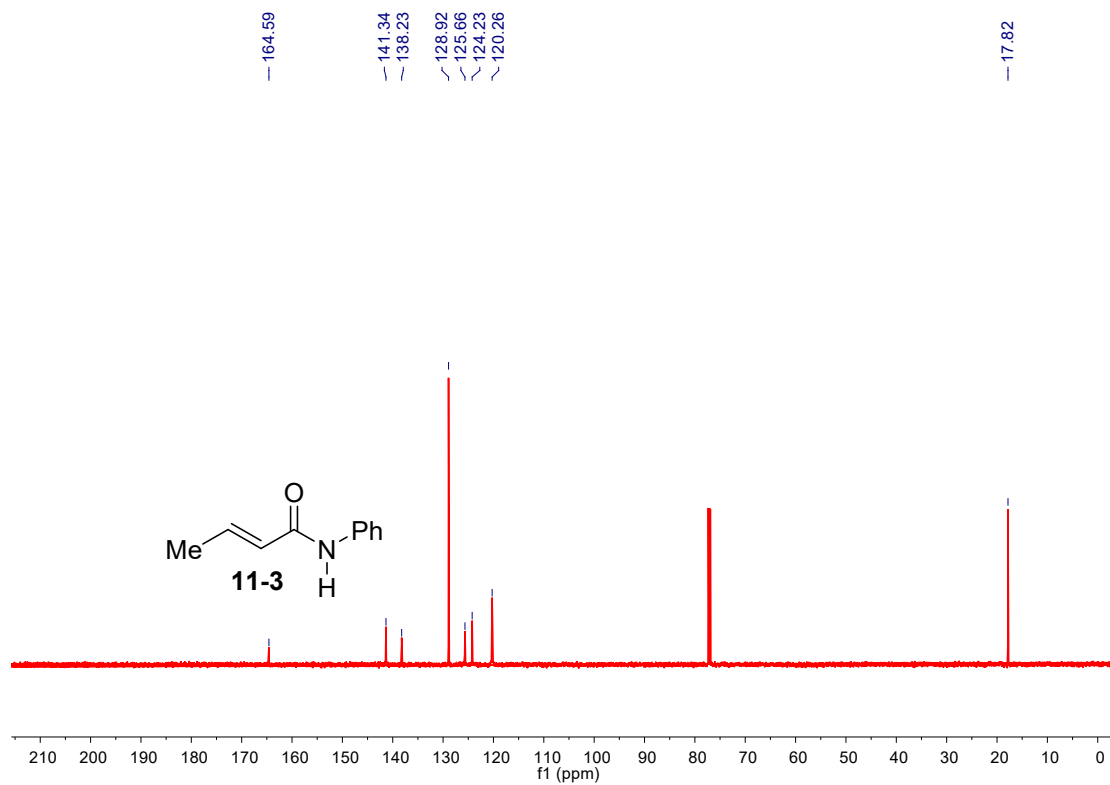


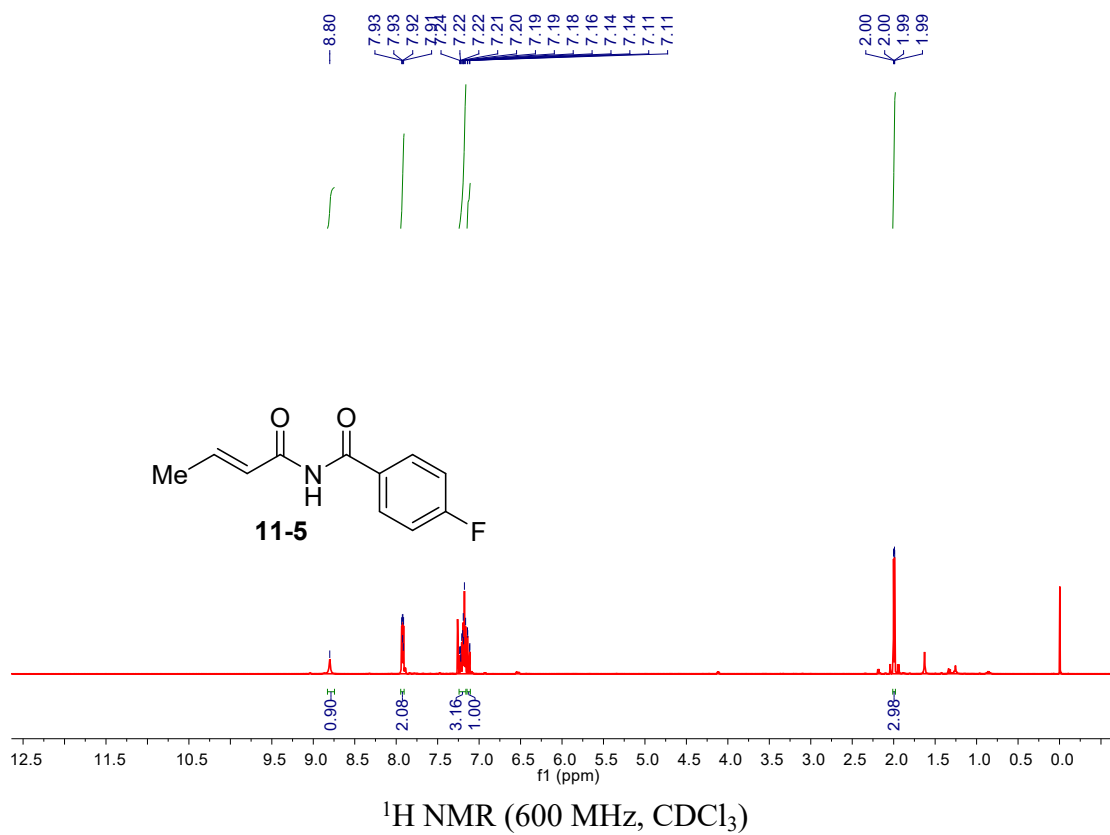
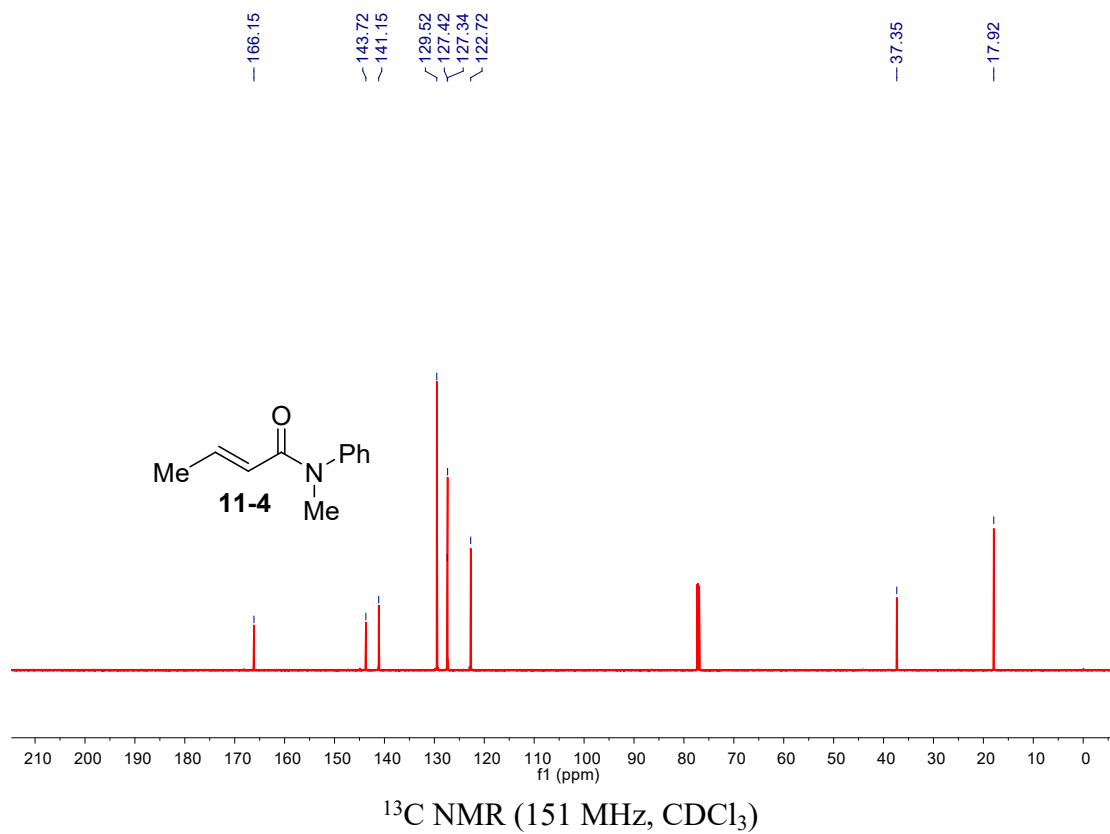


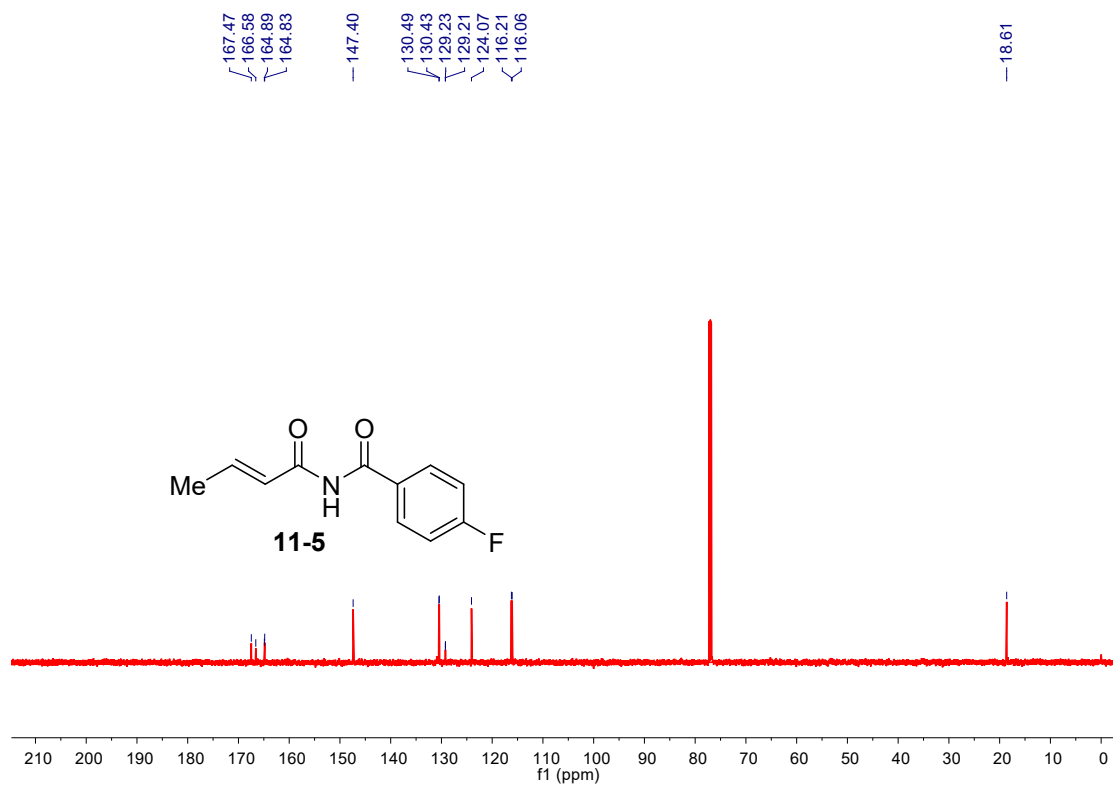
¹⁹F NMR (565 MHz, CDCl₃)



¹H NMR (600 MHz, CDCl₃)

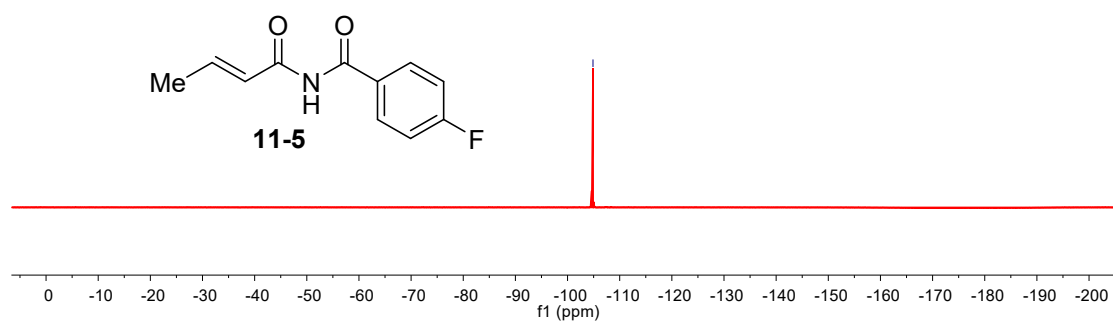




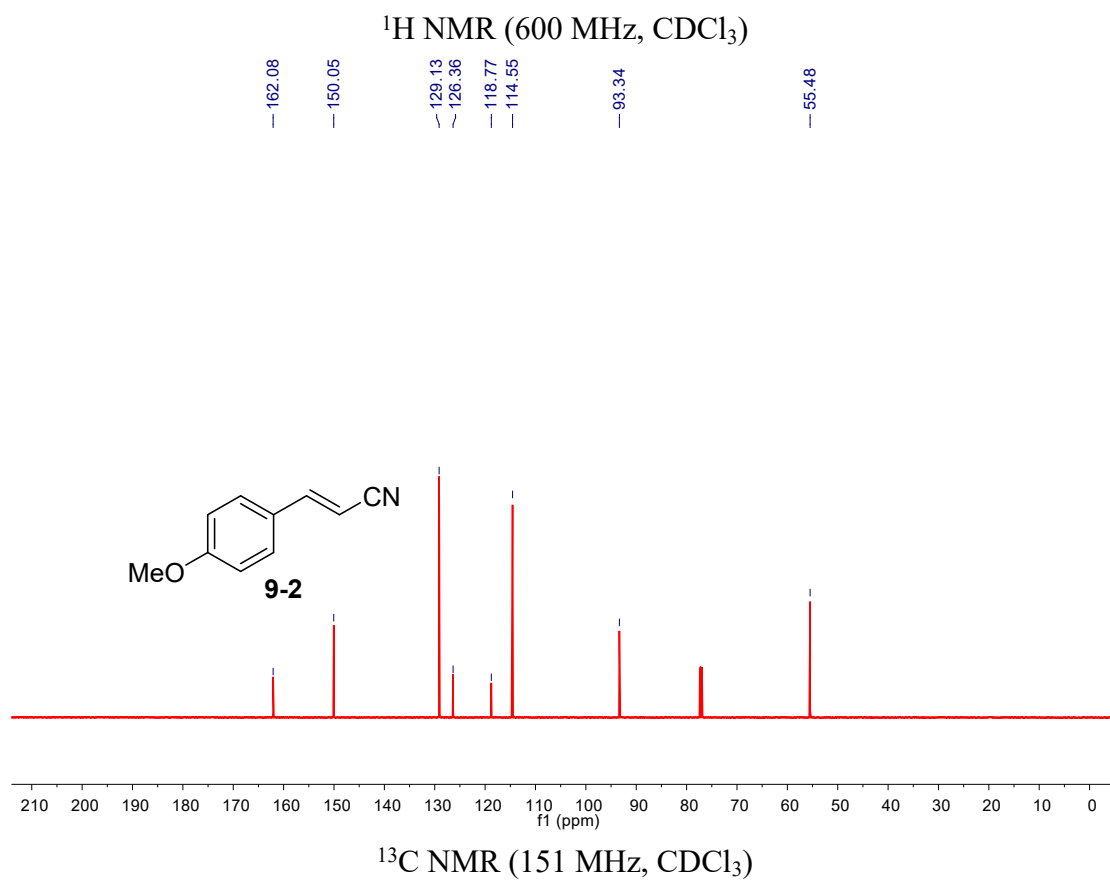
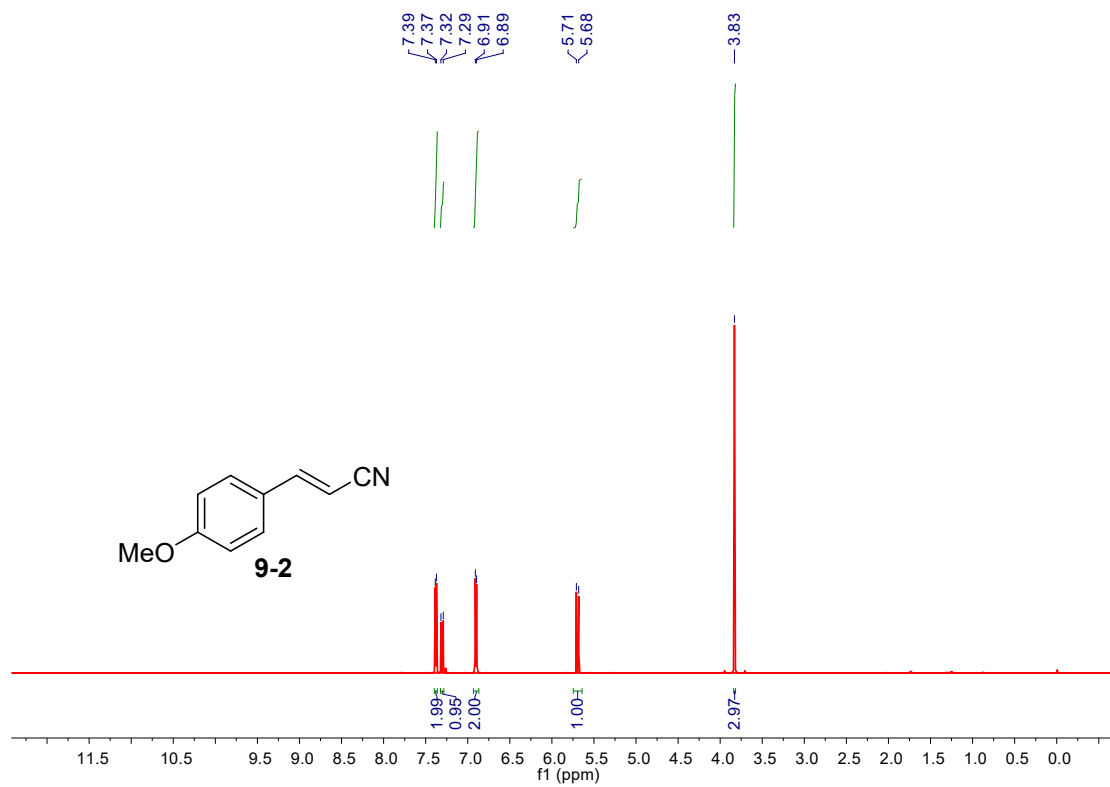


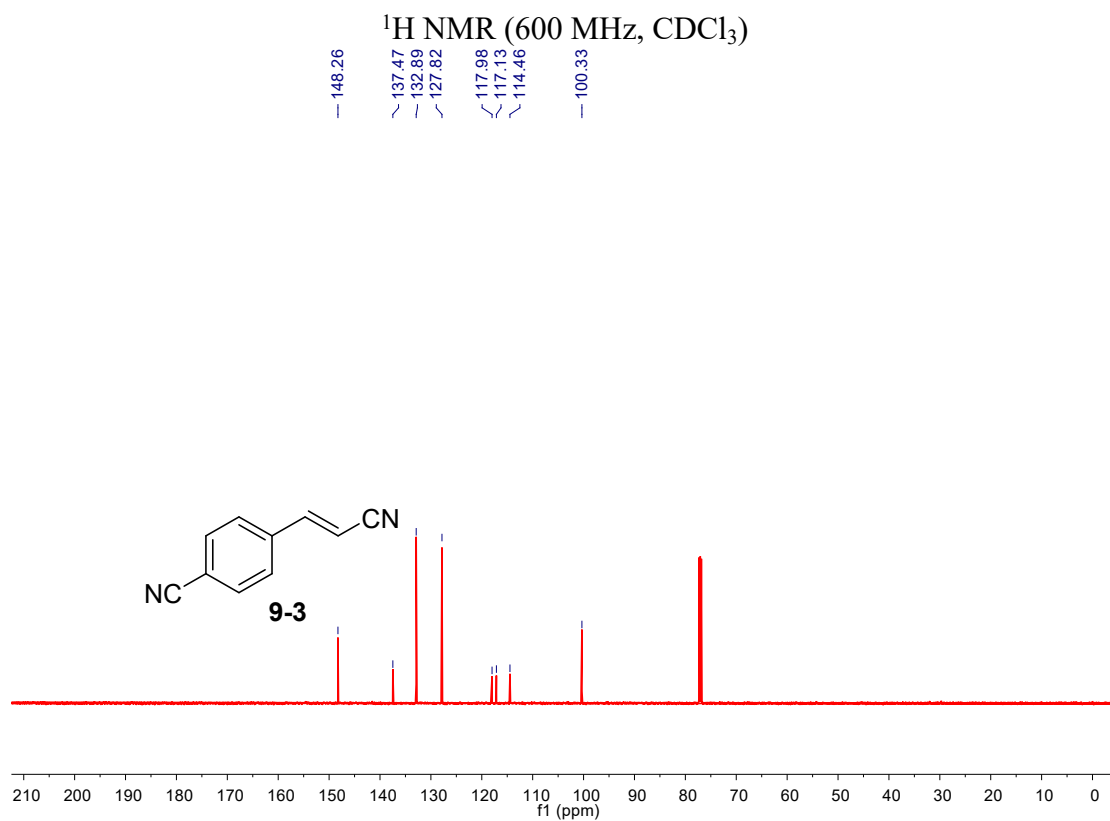
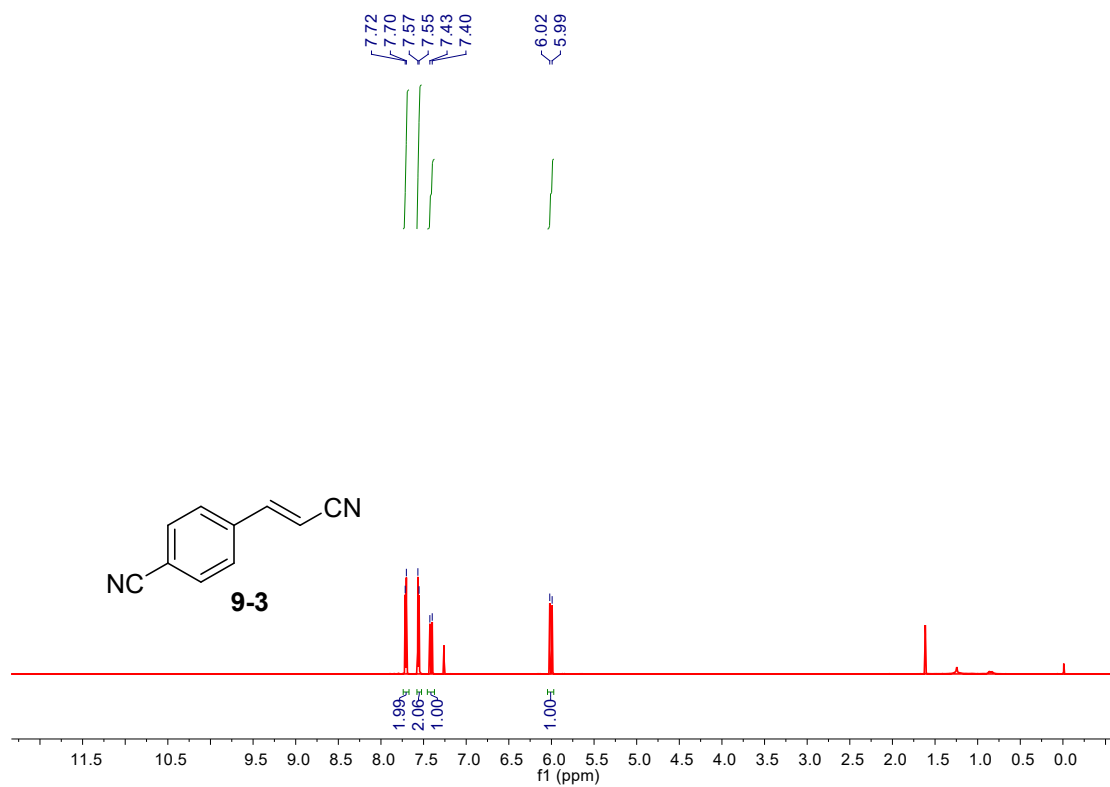
^{13}C NMR (151 MHz, CDCl_3)

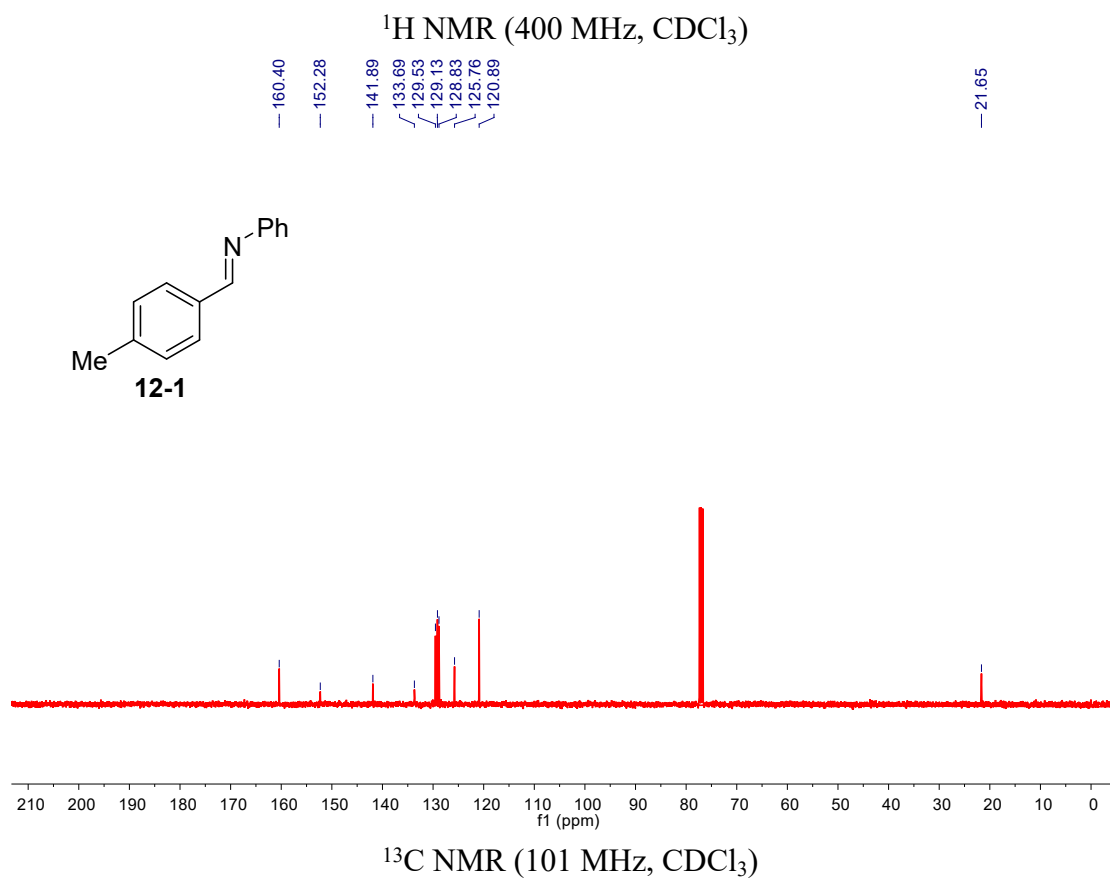
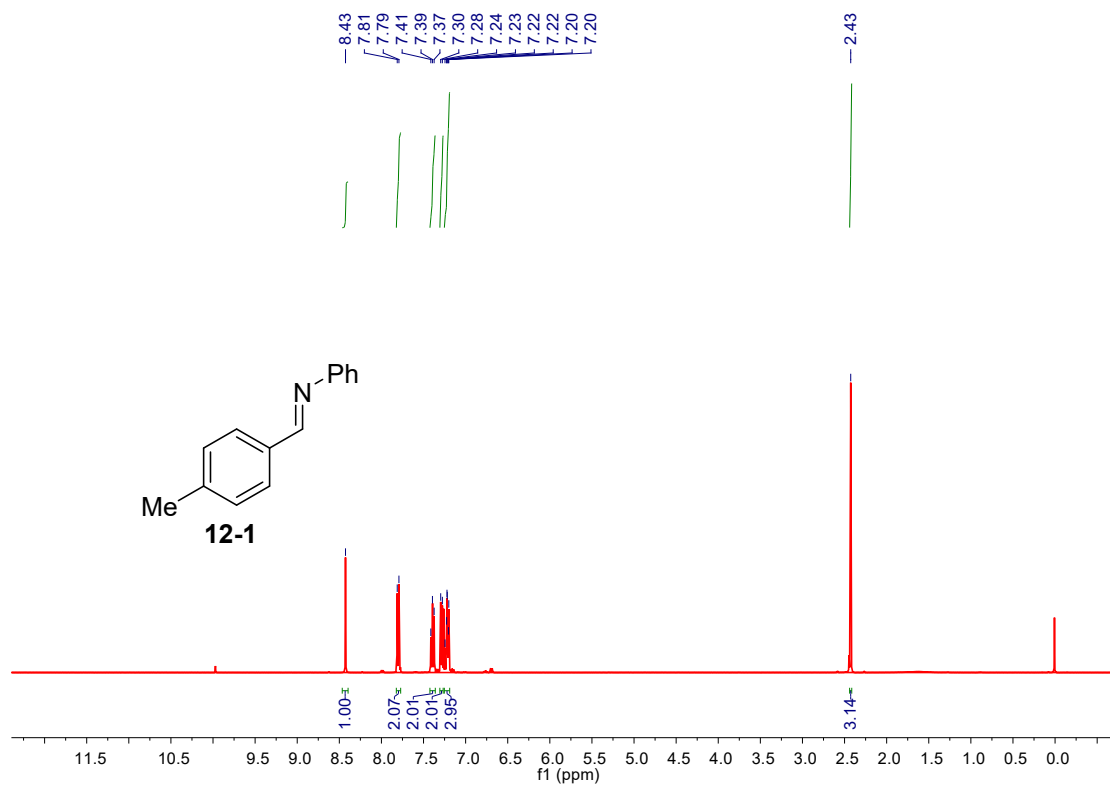
104.88

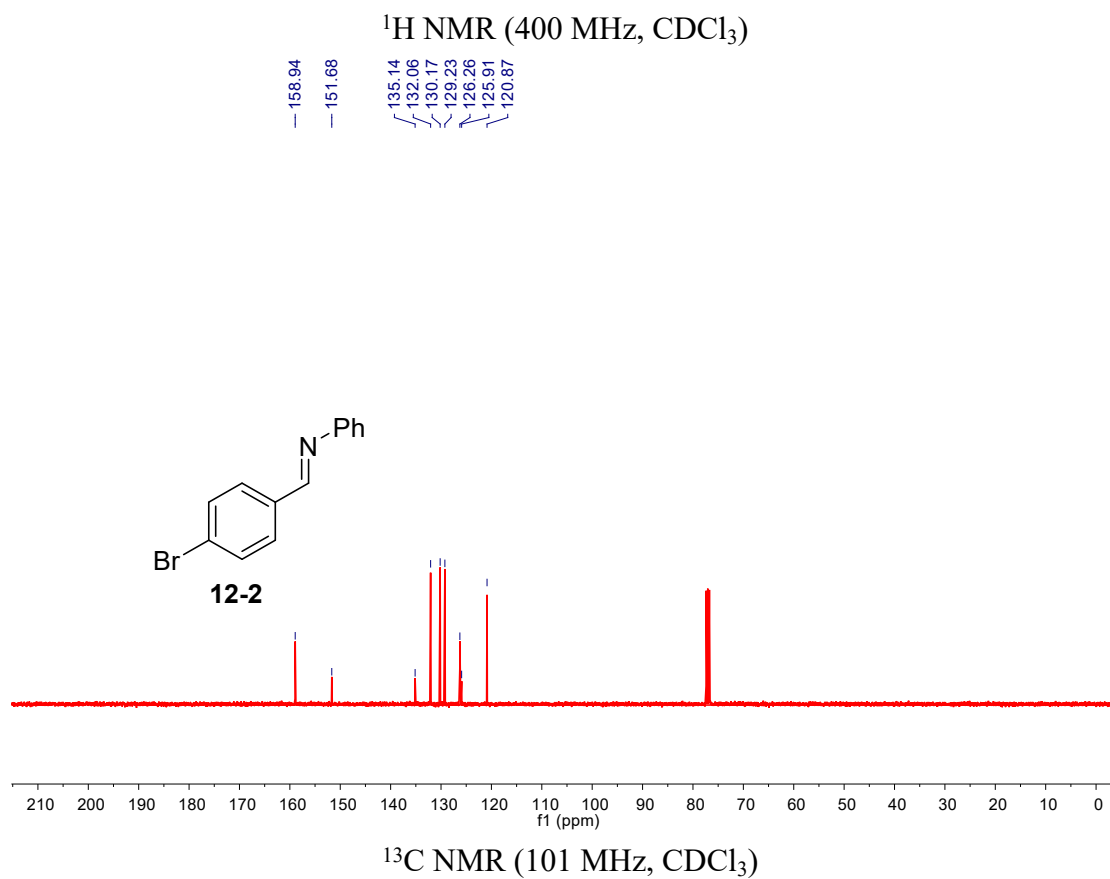
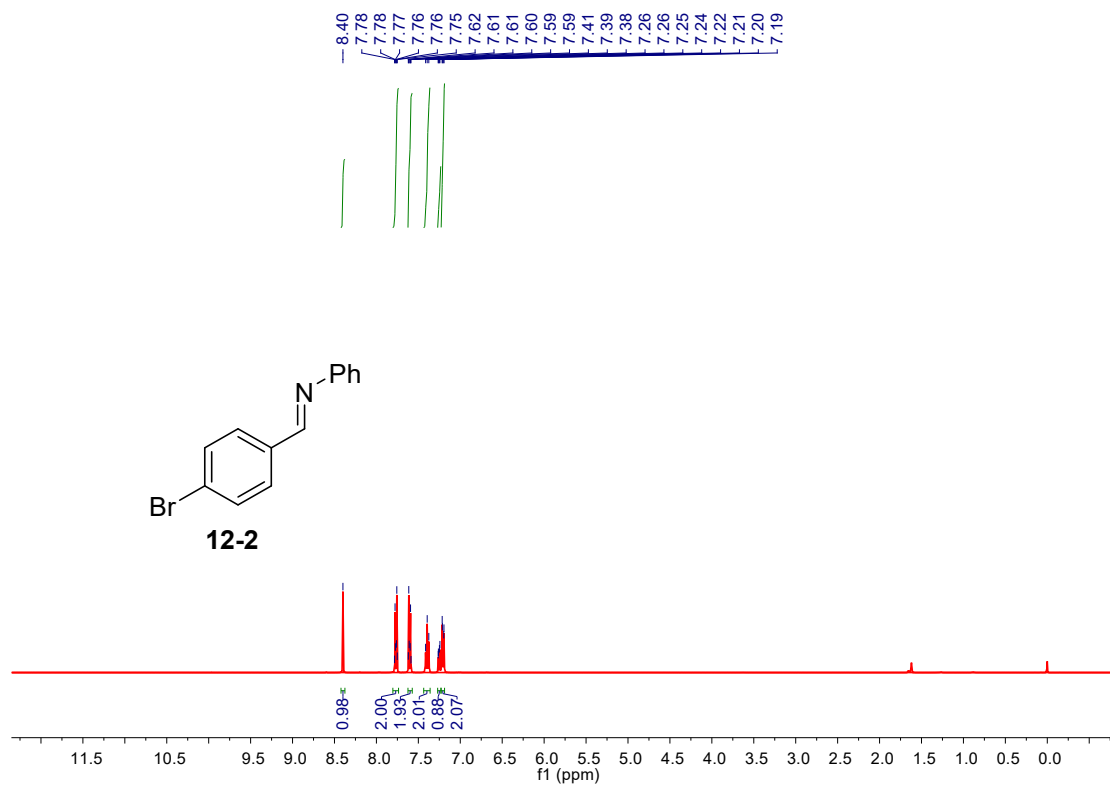


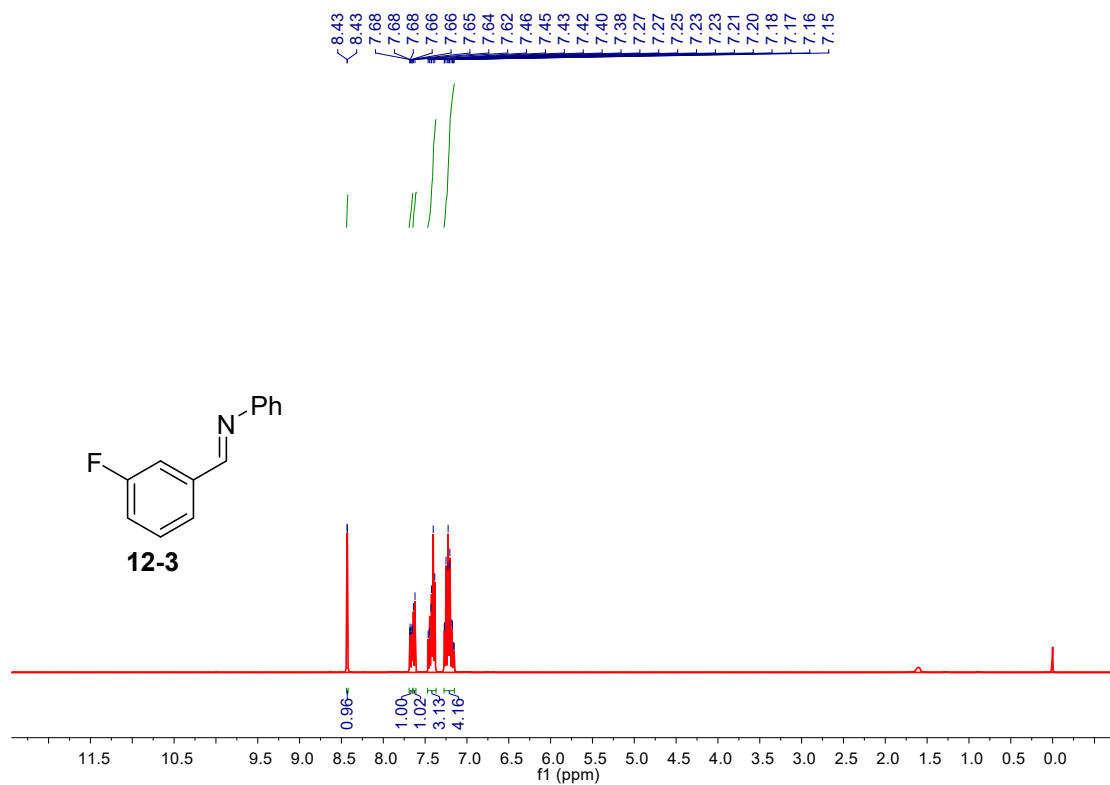
^{19}F NMR (565 MHz, CDCl_3)



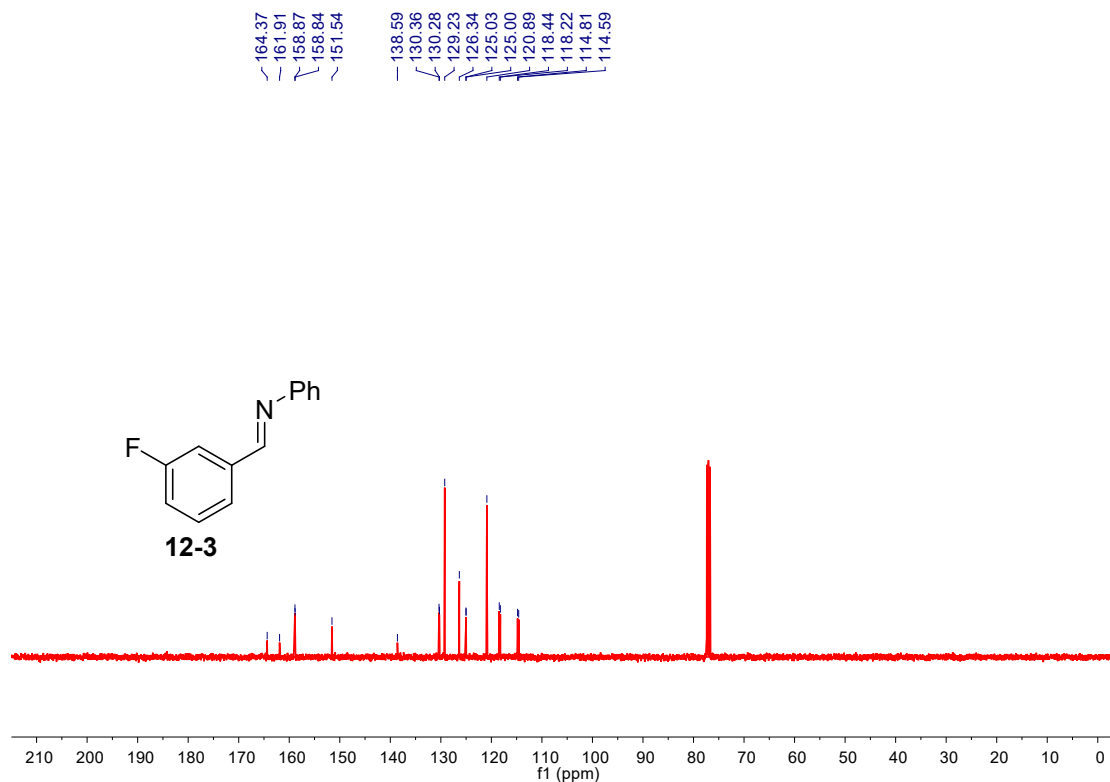






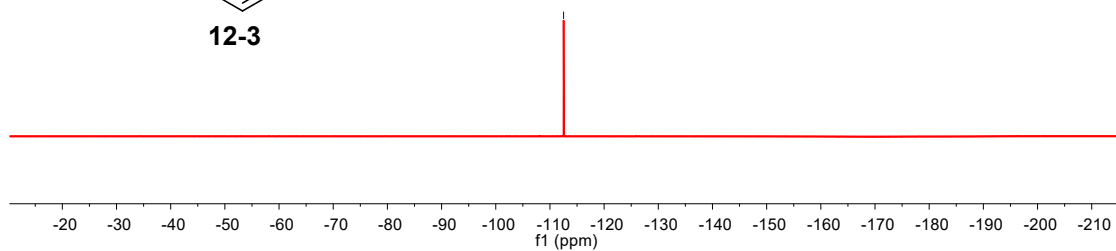
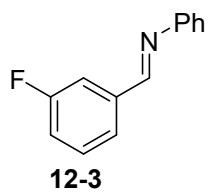


¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)

--112.52



^{19}F NMR (565 MHz, CDCl_3)

