

Supplementary Information
for
Decarboxylation of β -Boryl NHPI Esters Enables Radical 1,2-Boron Shift for the Assembly of Versatile Organoborons

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Table of Contents

1. Supplementary Methods	3
1.1 General information	3
1.2 General process	4
2.1 Optimization studies	15
2.2 Control experiments	27
2.3 Crystal data	31
2.4 Characterization data for Substrates.....	33
2.5 Characterization data for Products	44
2.6 NMR spectroscopic data	90
2.7 References.....	327

1. Supplementary Methods

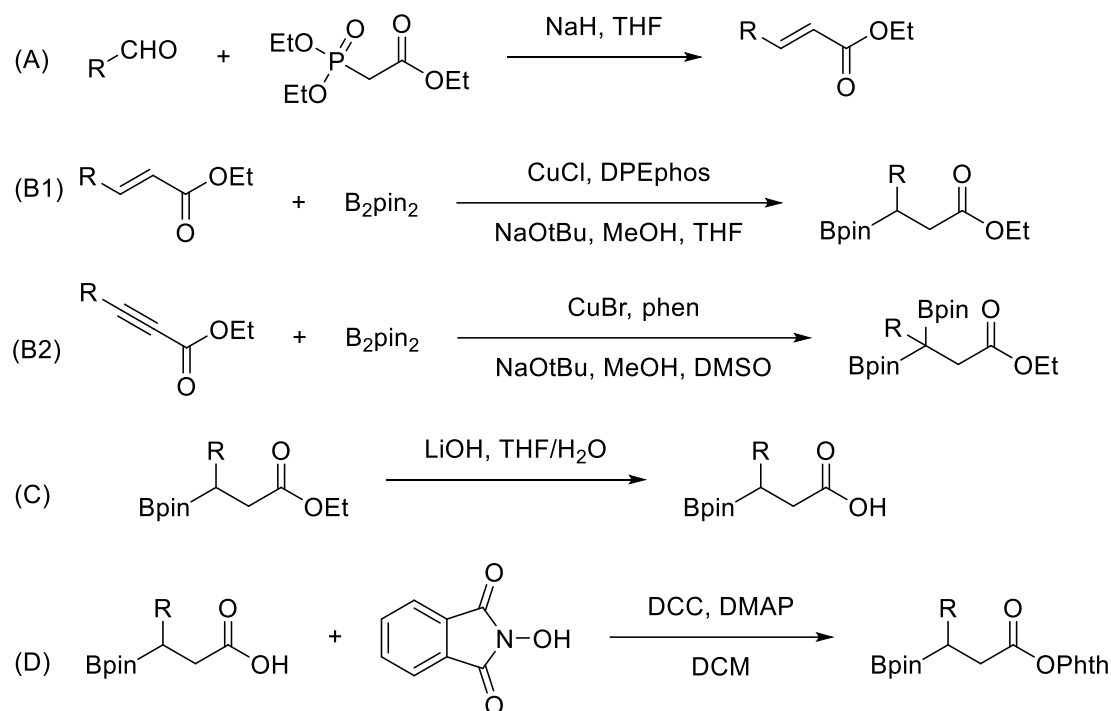
1.1 General information

All chemicals were purchased from Leyan.com (B₂pin₂, Ir(ppy)₃, [Ir(dtbppy)₃]PF₆, Ru(bpy)₃(PF₆)₂, 4CzIPN), Energy chemical company (Ru(bpy)₃Cl₂, Ru(bpy)₃Cl₂·6H₂O, CuCl, Ethyl acrylate, Phenyl Acrylate). Unless otherwise stated, all experiments were conducted in a sealed tube under N₂ atmosphere. Reactions were monitored by TLC or GC-MS analysis. Flash column chromatography was performed over silica gel (200-300 mesh).

¹H-NMR and ¹³C-NMR spectra were recorded in CDCl₃ and DMSO-d₆ on a Bruker Avance 500 spectrometer (500 MHz ¹H, 125 MHz ¹³C (CPD), 470 MHz ¹⁹F) at room temperature. Chemical shifts were reported in ppm on the scale relative to CDCl₃ (δ = 7.26 for ¹H NMR, δ = 77.00 for ¹³C-NMR) as an internal reference. Coupling constants (*J*) were reported in Hertz (Hz).

1.2 General process

General process 1: Preparation of propargyl β -boryl NHPI esters 1.



General process 1A¹: Add triethyl phosphonoacetate (1.5 equiv) dropwise to a stirred suspension of NaH (60% on mineral oil; 1.5 equiv) in dry THF (120 mL) under nitrogen over a period of 5 minutes. Stir the resulting mixture for another 0.5 hours at 0 °C. Add a solution of aldehyde (1 equiv) in dry THF (30 mL) slowly to the resulting mixture. The reaction mixture was stirred at room temperature for 6 h. Then quench the reaction with a saturated aqueous solution of NH₄Cl. Extract the reaction mixture with Et₂O (3 × 100 mL). Wash the combined organic layer with brine. Dry the combined organic layer over NaSO₄. After concentration under vacuum, purify the residue by flash chromatography on silica gel (eluent: petroleum ether: EtOAc =40:1, v/v).

General process 1B1^{2a}: In an oven-dried Schlenk tube were placed CuCl (0.03 equiv), NaO^tBu (0.09 equiv) and DPEphos ligand (0.03 equiv). THF (1 mL) was added under argon. The reaction mixture was stirred for 30 min at room temperature, and then bis(pinacolato)diboron (1.1 equiv) in THF (1 mL) was added. The reaction mixture was stirred for 30 min and then α,β -unsaturated ester (1 equiv) in THF (1 mL) was added, followed by MeOH (2 equiv). The reaction tube was washed with THF (5 mL), sealed, and stirred until no starting material was detected by TLC. The reaction

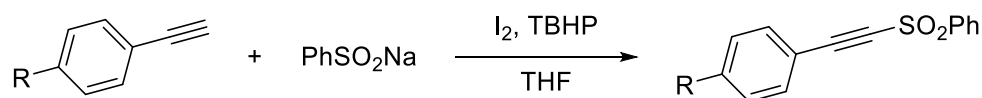
mixture was filtered through a pad of celite and concentrated. The product was purified by silica gel chromatography (eluent: petroleum ether: EtOAc =30:1, v/v).

General process 1B2^{2b}: To a mixture of CuBr (0.86 mg, 2.0 mol%, 6.0 μ mol), phen (2.0 mol%), K_2CO_3 (4.1 mg, 10 mol%, 30 μ mol) and B_2pin_2 (152 mg, 0.60 mmol) were added DMSO (0.6 mL) and then ynoates (0.30 mmol) and methanol (36 μ L, 0.90 mmol) in a pressure vial. After stirring at 30 °C for 20 h, the mixture was filtered through a short plug of silica gel using ethyl acetate as an eluent.

General process 1C³: β -boryl esters was dissolved in 2 ml THF and hydrolyzed with 4 equiv of LiOH dissolved in 2 ml H_2O at room temperature for 1 h. The reaction mixture was diluted with water (100 mL). The aqueous phase was washed with EA (3 x 50 mL) and was acidified with conc. aq. HCl until pH 1. The aqueous phase was extracted with EA (3 x 50 mL). The combined organic phases mixture was washed with water, brine, dried with Na_2SO_4 , filtered and evaporated under reduced pressure, β -boryl acid was obtained without further purification.

General process 1D⁴: The corresponding alkyl β -boryl acid (1.2 equiv.), *N*-hydroxyphthalimide (1.0 equiv.), and 4-dimethylaminopyridine (5 mol %) were mixed in a flask with a magnetic stirring bar. dry THF or CH_2Cl_2 (40 mL) (according to the solubility of the carboxylic acids) was added. Then a solution of *N,N*-dicyclohexylcarbodiimide (1.2 equiv.) in THF or CH_2Cl_2 (15 mL) was added slowly at room temperature. The reaction mixture was stirred at room temperature for 6 h. After *N*-hydroxyphthalimide was completely converted, the white precipitate was filtered off and the solution was concentrated under vacuum. Corresponding redox active esters were purified by column chromatography on silica gel (CH_2Cl_2 or petroleum ether/ethyl acetate as eluent).

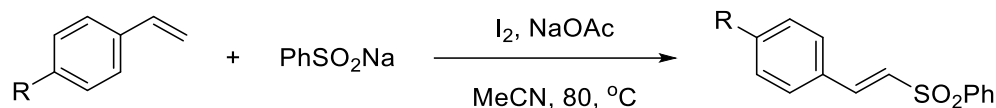
General process 2: Preparation of propargyl alkynyl sulfones 2⁵.



Add arylacetylene (1 equiv, 5 mmol) and tert-butyl hydroperoxide (TBHP) (3 equiv, 15 mmol) to a solution of sodium benzenesulfinate (2 equiv, 10 mmol) and iodine (I₂) (0.5 equiv, 2.5 mmol) in THF (20 mL). Stir the solution at room temperature for 8-12 hours. Monitor the reaction by TLC. Add H_2O (10 mL) to the solution to quench the reaction. Add ($Na_2S_2O_3$) solution dropwise to the

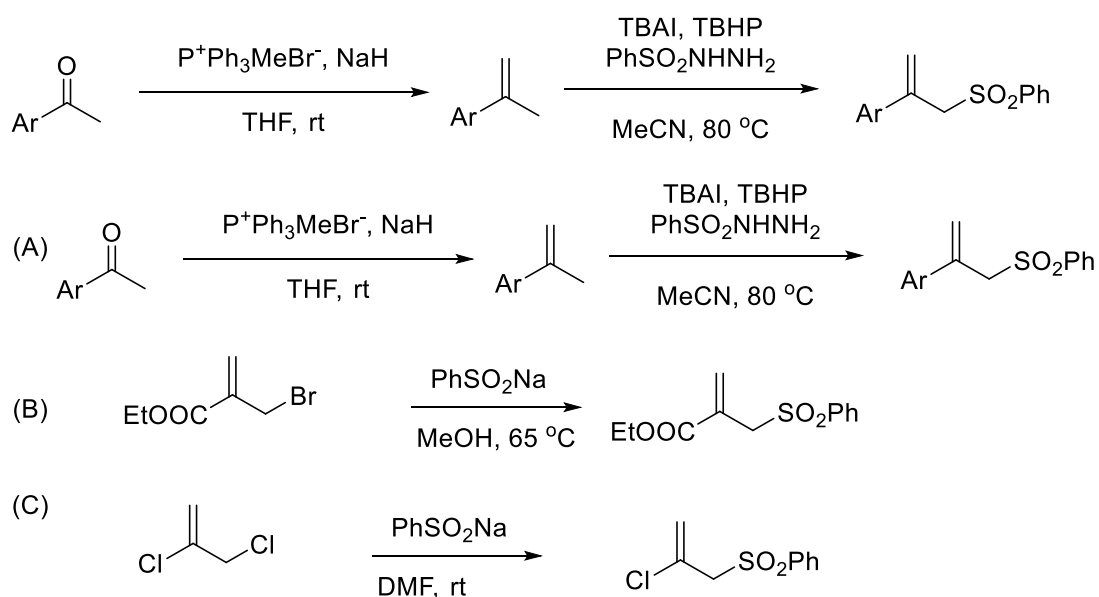
mixture until the solution changes from brown to clear and transparent. Extract the separated aqueous phase with ethyl acetate (3 × 40 mL). Dry the combined organic layers over NaSO₄. Concentrate the combined organic layers in vacuo. Purify the residue by column chromatography using ethyl acetate/petroleum ether as eluent (eluent: petroleum ether: EtOAc = 10:1, v/v).

General process 3: Preparation of propargyl alkenyl sulfones 2⁶.



I₂ (762 mg, 3.0 mmol) was added to a suspension mixture of styrene derivative (2.0 mmol), sulfonic acid sodium (6.0 mmol) and NaOAc (246 mg, 3.0 mmol) in MeCN (10 mL), and the reaction mixture was vigorously stirred at refluxing temperature for 2 hours. Upon completion of the reaction, the reaction mixture was quenched by the addition of saturated aqueous sodium thiosulfate (Na₂S₂O₃) (5 mL), and basified with saturated aqueous sodium hydrogen carbonate (NaHCO₃) (5 mL). Further stirring was followed by extraction with ethyl acetate (3×15 mL). The combined organic extracts were washed with water (20 mL), brine (20 mL), dried over anhydrous Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography (silica gel) to furnish the product (silica gel, petroleum ether: EtOAc = 10:1, v/v).

General process 4: Preparation of propargyl alkenyl sulfones 4⁷.



General process 4A: To a solution of P⁺Ph₃MeBr⁻ (11 mmol, 1.1 equiv) in THF (20 mL) was added

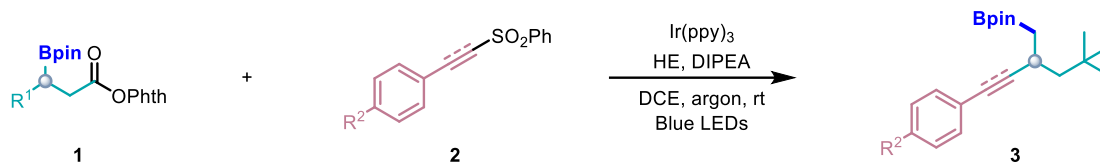
NaH (60%, 1.2 equiv.), the reaction mixture was reacted for 2 h at room temperature. Then the corresponding ketones (10 mmol) in THF (10 mL) were added dropwise at 0 °C. The mixture was reacted overnight at room temperature. When the starting material was consumed (monitored by TLC), the reaction mixture was diluted by petroleum ether and filtered through a pad of silica gel. The filtrate was concentrated to give a crude product which was distilled or purified through flash column chromatography to obtain the desired product. The known products were identical to the literature.

Alkenes (5 mmol) and TBHP (10 mmol) was added to a solution of sulfonylhydrazides (6 mmol) and tetrabutylammonium iodide (1 mmol) in 20 mL of MeCN. After the reaction mixture was stirred at 80 °C in an oil bath for 20 h, the solvent was removed under reduced pressure, then add saturated aqueous Na₂S₂O₃ (20 mL). Further stirring was followed by extraction with EtOAc (2 × 30 mL). The combined organic extracts were washed with H₂O (20 mL) and brine (20 mL), dried with Na₂SO₄, filtered and the filtrate was evaporated and purified by flash chromatography afforded the corresponding product.

General process 4B: To a solution of alkyl bromide (10 mmol) in dry methanol was added sodium phenylsulfinate (15 mmol). After 2.5 h of reflux in an oil bath, the mixture was concentrated under reduced pressure, the obtained residue was dissolved in EtOAc and the mixture was washed with water, brine, dried with Na₂SO₄, filtered and the filtrate was evaporated and purified by flash chromatography afforded product.

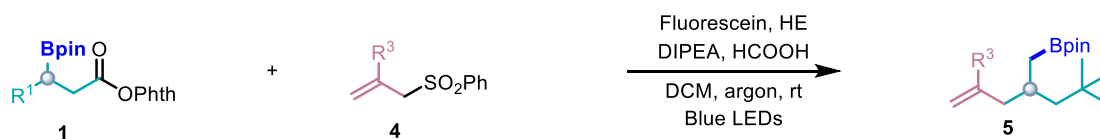
General process 4C: To a stirred solution containing 1.64 g (10 mmol) of benzenesulfinic acid sodium salt in 8 mL of DMF was slowly added 0.98 mL (10 mmol) of 3-chloro-2-chloropropene at room temperature. After being stirred for 5 h at the same temperature, the reaction mixture was diluted with 25 mL of saturated aqueous NH₄Cl and extracted with EtOAc. The mixture was washed with water, brine, dried with Na₂SO₄, filtered and evaporated under reduced pressure. ((2-chloroallyl)sulfonyl)benzene (1.92 g, 98%) was obtained without further purification.

General process 5: For synthesis of 3



A mixture of **1** (0.2 mmol), **2** (0.3 mmol), Ir(ppy)₃ (2% mmol) and HE (0.3 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and DIPEA (0.2 mmol), DCE (2 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (50:1) to afford the corresponding product.

General process 6: For synthesis of 5



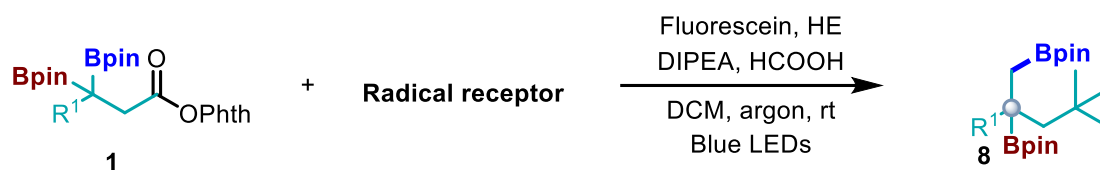
A mixture of **1** (0.2 mmol), Fluorescein (2% mmol) and HE (0.4 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and **4** (0.3 mmol), DIPEA (0.4 mmol), HCOOH (0.2 mmol), DCM (2 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (50:1) to afford the corresponding product.

General process 7: For synthesis of 7



A mixture of **1** (0.2 mmol), Ru(bpy)₃Cl₂ (2% mmol) and HE (0.3 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and **6** (0.3 mmol), THF (2 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (30:1) to afford the corresponding product.

General process 8: For synthesis of 8



1) A mixture of **1** (0.2 mmol), Fluorescein (2% mmol) and HE (0.4 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and **4** (0.3 mmol), DIPEA (0.4 mmol), HCOOH (0.2 mmol), DCM (2 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (20:1) to afford the corresponding product.

2) A mixture of **1** (0.2 mmol), **2** (0.3 mmol), Ir(ppy)₃ (2% mmol) and HE (0.3 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and DIPEA (0.2 mmol), DCE (2 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. The crude material was dissolved in THF (2 mL, 0.1 M) and cooled to 0 °C. An aqueous solution of sodium hydroxide (3 M, 5 mL) and aqueous hydrogen peroxide (30%, 2.5 mL) were subsequently added dropwise. After 10 min, the vigorously stirred biphasic reaction mixture was allowed to reach ambient temperature and stirred for another 2 h, at which point TLC analysis showed the disappearance of the boronic ester. The mixture was diluted with water (5 mL) and ethyl acetate (10 mL) and the phases were separated. The aqueous phase was washed twice with ethyl acetate (10 mL each), and the organic phases were combined and subsequently washed with brine (15 mL). The washed organic solution was then dried over anhydrous sodium sulfate, the dried solution was filtered and the filtrate was concentrated under reduced pressure. The resulting crude material was purified by flash column chromatography on silica gel to afford analytically pure products. The residue was purified by silica gel chromatography using PE/EA (5:1) to afford the corresponding product.

General process 9: Gram-scale synthesis of 3a

A mixture of **1a** (3 mmol), **2a** (4.5 mmol), Ir(ppy)₃ (5% mmol) and HE (4.5 mmol) were charged

into a Schleck tube, then the air was removed, argon was filled of Schleck tube and DIPEA (6 mmol), DCE (30 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (50:1) to afford the corresponding product **3a** (67%).

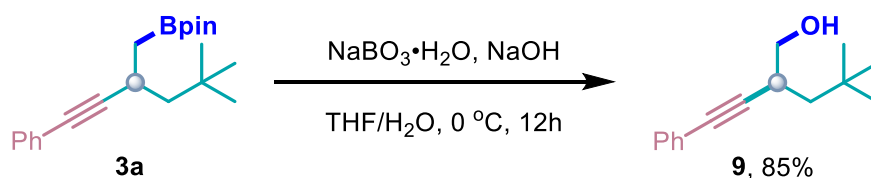
General process 10: Gram-scale synthesis of **5a**

A mixture of **1a** (2 mmol), Fluorescein (5% mmol) and HE (4 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and **4a** (3 mmol), DIPEA (4 mmol), HCOOH (2 mmol), DCM (20 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (50:1) to afford the corresponding product **5a** (71%).

General process 11: Gram-scale synthesis of **7a**

A mixture of **1a** (2 mmol), Ru(bpy)₃Cl₂ (5% mmol) and HE (3 mmol) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and **6a** (3 mmol), THF (20 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (30:1) to afford the corresponding product **7a** (57%).

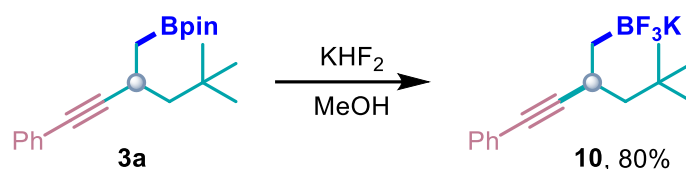
General process 12: For synthesis of **9**⁸.



To a 25 mL Schlenk tube, was added **3a** (1 equiv, 0.2 mmol) followed by 0.3 mmol of sodium perborate monohydrate (1.5 equiv) under air. Then THF (0.8 mL) and H₂O (0.8 mL) was added in ice-bath, with that the NaOH (aq.) was added by drop wise. The reaction mixture was stirred at 0-rt until full conversion by TLC. Then, the reaction mixture was extracted with EA/H₂O.

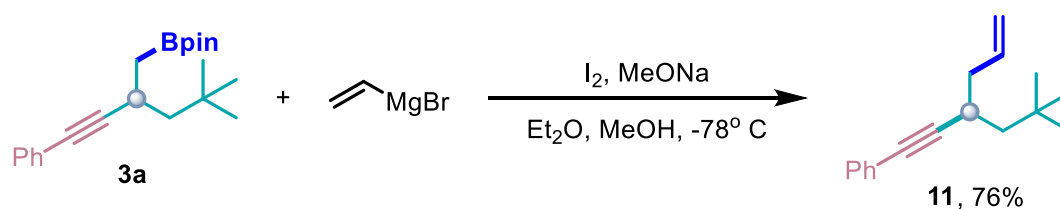
Subsequently, the organic layers were dried with Na₂SO₄, and concentrated to dryness. The crude product was purified by silica gel chromatography (silica gel, PE: EA =100:1, v/v) to afford the product with the yield of 85%.

General process 13: For synthesis of **10**⁹.



In air, to a 25 mL schlenk tube equipped with a magnetic stir bar were added the **3a** (0.2 mmol, 1 equiv) and 5 mL MeOH. To this solution sat. KHF₂ (2.4 mmol, 12 equiv, in water) was added dropwise under room temperature, and the mixture was allowed to stir at ambient temperature for 3 h. The resultant suspension was concentrated under reduced pressure. Then hot acetone (2 mL × 3) was added and filtered. The filtrate was concentrated to near dryness and Et₂O (5 mL) was added to yield a white precipitate. The precipitate was isolated by filtration, washed by CH₂Cl₂ (0.5 mL), to afford the product (80% yield) as a white solid.

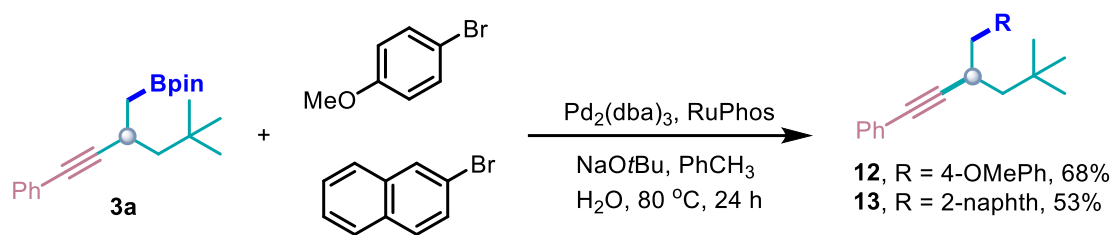
General process 14: For synthesis of **11**¹⁰.



An oven-dried 10 mL Schlenk tube equipped with a Teflon stir bar was added with a solution of **3a** (0.10 mmol) and hydrous THF (1.0 mL) under argon atmosphere via syringe. Then a solution of vinyl magnesium bromide (0.4 mmol, 0.8 mL, 0.5 M in THF) was added dropwise, and the mixture was stirred for 30 min at room temperature. Then the tube was cooled to -78 °C and a solution of I₂ (0.4 mmol in 1.0 mL MeOH) was added dropwise. The solution was stirred for another 30 min at this temperature, followed by addition of a solution of NaOMe (0.8 mmol in 1.0 mL MeOH). The reaction mixture was allowed to warm to room temperature and stirred for another 2 hours. Once

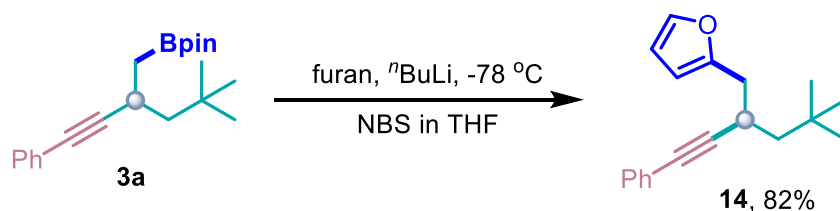
finished, the reaction was quenched with Na₂S₂O₃(saturated aqueous solution, 3.0 mL). The aqueous solution was extracted with Et₂O (3 × 5.0 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated, the crude product was purified carefully by column chromatography on silica gel affording **10** (76% yield, colorless oil).

General process 15: For synthesis of **12** and **13**¹¹.



In air, Pd₂(dba)₃ (4.6 mg, 0.005 mmol, 5.0 mol%), NaOtBu (28.8 mg, 0.3 mmol, 3.0 equiv), Ruphos (4.7 mg, 0.01 mmol, 10.0 mol%) and substrate **3a** (0.1 mmol, 1.0 equiv) were added to a Schlenk tube equipped with a stir bar. The vessel was evacuated and filled with argon (three cycles). Then PhCH₃ (1.0 mL), 4-OMePhBr (0.1 mmol, 1.0 equiv) or 2-bromonaphthalene (0.1 mmol) and H₂O (0.1 mL) was added under argon atmosphere. The mixture was stirred at 80 °C for 24 hours. Then, the mixture was cooled to room temperature and diluted with DCM (20 mL). The organic layer was separated and the aqueous phase was extracted with DCM (20 mL x 2). The combined organic layer was washed with H₂O (30 mL) and brine (30 mL). The combined organic layers were dried over Na₂SO₄ and concentrated in vacuo. The crude product was purified by silica gel chromatography (silica gel, PE: EA =50:1, v/v) to afford the product with the yield of 68% and 53%.

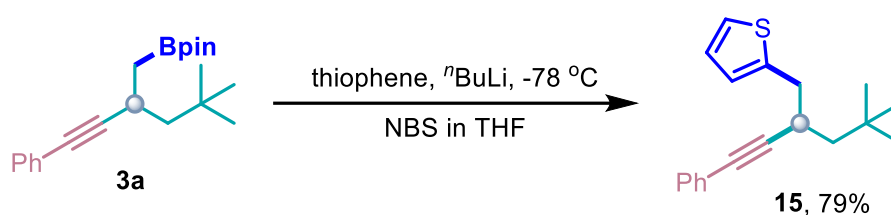
General process 16: For synthesis of **14**¹².



A solution of furan (0.5 mmol, 2.5 equiv) in THF (2.0 mL) was cooled to -78 °C and treated with ⁿBuLi (1.6 M in hexanes, 0.6 mmol, 3 equiv). The cooling bath was removed and the mixture was stirred at room temperature for 1 h. The mixture was cooled to -78 °C and a solution of **3a** (0.2

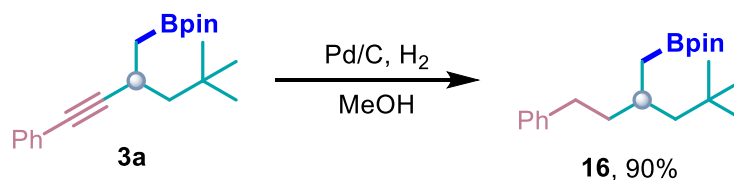
mmol, 1.0 equiv) in THF (1 mL) was added. The mixture was stirred at -78 °C for 1 h, and then a solution of NBS (0.5 mmol, 2.5 equiv) in THF (2.0 mL) was added. After 10 h at room temperature, sat. Na₂S₂O₃ (aq.) (4 mL) was added. The reaction mixture was diluted with water and extracted with ethyl acetate. The combined organic layers were dried over Na₂SO₄ and concentrated in vacuo. The crude product was purified by silica gel chromatography (silica gel, PE: EA=50:1, v/v) to afford the product with the yield of 82%.

General process 17: For synthesis of 15¹².



A solution of thiophene (1 mmol, 5.0 equiv) in THF (2.0 mL) was cooled to -78 °C and treated with ⁿBuLi (1.6 M in hexanes, 1.2 mmol, 6.0 equiv). The cooling bath was removed and the mixture was stirred at room temperature for 1 h. The mixture was cooled to -78 °C and a solution of substrate **3a** (0.2 mmol, 1.0 equiv) in THF (1 mL) was added. The mixture was stirred at -78 °C for 1 h, and then a solution of NBS (1 mmol, 5.0 equiv) in THF (2.0 mL) was added. After 10 h at room temperature, sat. Na₂S₂O₃ (aq.) (4 mL) was added. The reaction mixture was diluted with water and extracted with ethyl acetate. The combined organic layers were dried over Na₂SO₄ and concentrated in vacuo. The crude product was purified by silica gel chromatography (silica gel, PE: EA=50:1, v/v) to afford the product with the yield of 79%.

General process 18: For synthesis of 16¹³.



To a Schlenk tube containing of the **3a** (0.2 mmol) and 2 mL of MeOH, Pd/C (10% w) was added. The tube was evacuated and refilled with H₂ with three times. The reaction was refilled with

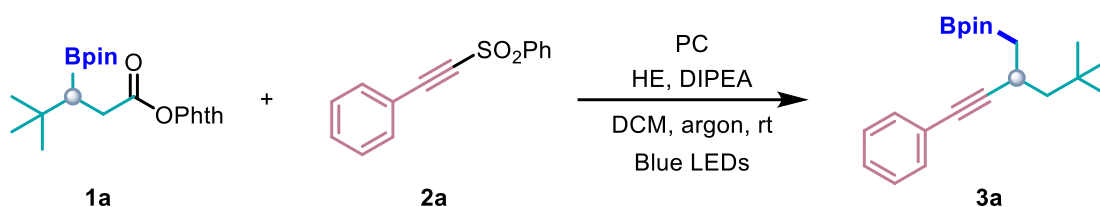
hydrogen through a balloon. The resulting reaction mixture was stirred under the hydrogen atmosphere at room temperature for 12 h. After the reaction was completed, which was determined by TLC analysis. The resulting suspension was filtered through a plug of celite and the filter cake washed with DCM. The mixture was concentrated in vacuum and the residue was purified by silica gel chromatography using petroleum as the eluent to afford the **16** as a colorless oil (90%).

2. Supplementary Discussion

2.1 Optimization studies

2.1.1 The condition screening for the 3a

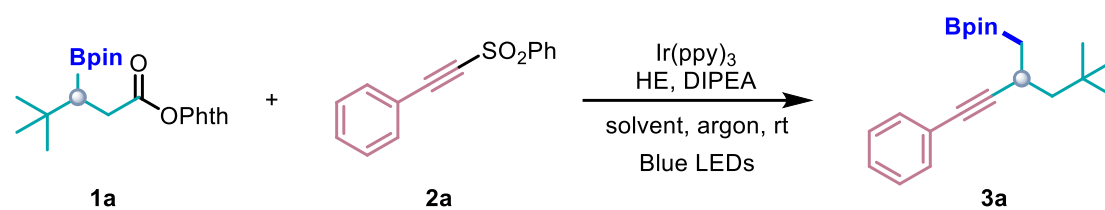
Table S1: Screening of photocatalysts.^a



entry	PC	yield(%) ^b
1	Ru(bpy) ₂ Cl ₂ ·6H ₂ O	26%
2	Ru(bpy) ₃ (PF ₆) ₂	24%
3	Eosin Y	36%
4	Ir(ppy) ₃	52%
5	Ir(dtbpy)(bpy) ₂ PF ₆	48%
6	Ir(dCF ₃ bpy)(bpy) ₂ PF ₆	40%
7	Acid red 94	38%
8	4CzIPN	41%
9	4',5'-dibromofluorescein	40%
10	Rhodamine B	32%
11	Benzophenone	22%
12	Eosin B	34%
13	Fluorescein	33%
14	Ru(bpy) ₃ Cl ₂	40%

^a Reaction conditions: **1a** (0.2 mmol), **2a** (1.5 equiv, 0.3 mmol), photocatalyst (1 mol%, 0.002 mmol), HE (1.5 equiv, 0.3 mmol), DIPEA (2 equiv, 0.4 mmol), DCM (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

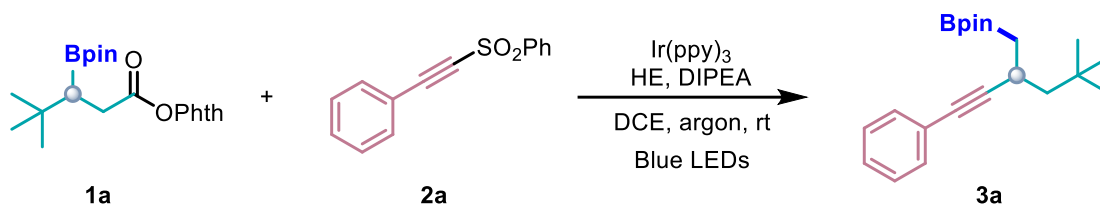
Table S2: Screening of different solvents.^a



entry	solvent	yield (%) ^b
1	THF	45%
2	EA	43%
3	toluene	29%
4	CH_3CN	39%
5	DCE	74%
6	DMSO	nr
7	DMA	38%
8	DMF	48%
9	dioxane	47%
10	CHCl_3	45%
11	MeOH	nr
12	acetone	52%

^a Reaction conditions: **1a** (0.2 mmol), **2a** (1.5 equiv, 0.3 mmol), Ir(ppy)_3 (1 mol%, 0.002 mmol), HE (1.5 equiv, 0.3 mmol), DIPEA (2 equiv, 0.4 mmol), solvent (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

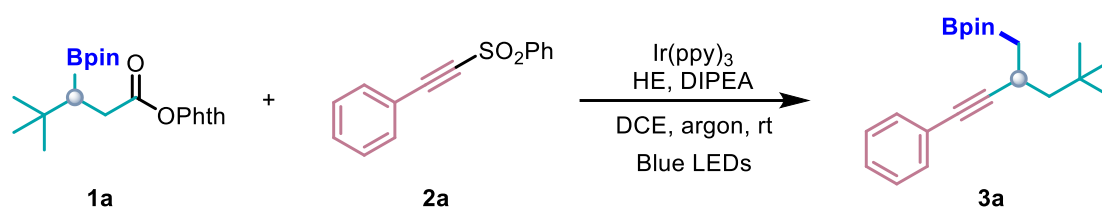
Table S3: The effects of equivalent of 2a, HE and DIPEA.



entry	2a (x equiv)	HE (y equiv)	DIPEA (z equiv)	yield(%) ^b
1	1.5	1	2	61%
2	1.5	2	2	56%
3	1.5	2.5	2	37%
4	1.5	3	2	45%
5	1.5	1.5	1	48%
6	1.5	1.5	3	52%
7	2	1.5	2	39%

^a Reaction conditions: **1a** (0.2 mmol), **2a** (x equiv), Ir(ppy)₃ (1 mol%, 0.002 mmol), HE (y equivl), DIPEA (z equiv), DCE (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

Table S4: Control Experiments.^a

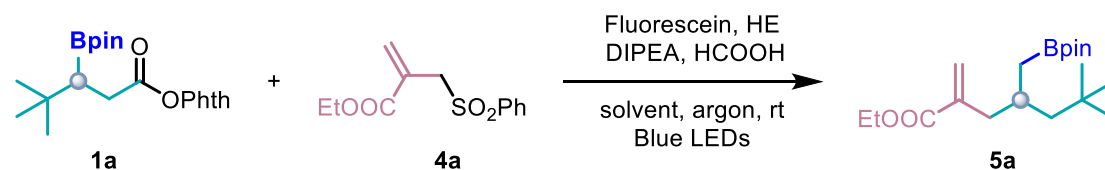


entry	PC	HE	DIPEA	yield(%) ^b
1	-	1.5	2	trace
2	Ir(ppy) ₃	-	2	trace
3	Ir(ppy) ₃	1.5	-	38%
4 ^c	Ir(ppy) ₃	1.5	2	NR

^a Reaction conditions: **1a** (0.2 mmol), **2a** (1.5 equiv, 0.3 mmol), Ir(ppy)₃ (1 mol%, 0.002 mmol), HE (1.5 equiv, 0.3 mmol), DIPEA (2 equiv, 0.4 mmol), DCE (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield. ^c Without light.

2.1.2 The condition screening for the 5a

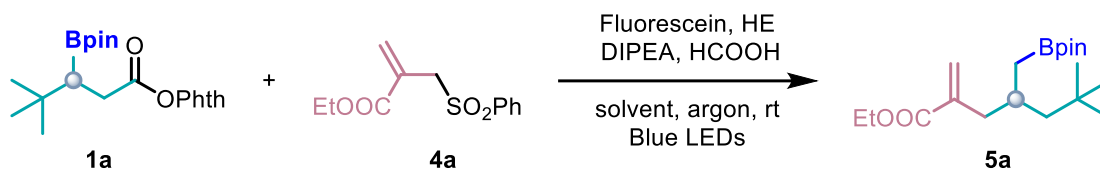
Table S5: Screening of photocatalysts.^a



entry	PC	yield(%) ^b
1	Ru(bpy) ₂ Cl ₂ ·6H ₂ O	45%
2	Ru(bpy) ₃ (PF ₆) ₂	45%
3	Eosin Y	63%
4	Ir(ppy) ₃	65%
5	Ir(dtbpy)(bpy) ₂ PF ₆	39%
6	Ir(dCF ₃ bpy)(bpy) ₂ PF ₆	34%
7	Acid red 94	38%
8	4CzIPN	21%
9	4',5'-dibromofluorescein	31%
10	Rhodamine B	38%
11	Benzophenone	62%
12	Eosin B	70%
13	Fluorescein	83%
14	Ru(bpy) ₃ Cl ₂	54%

^a Reaction conditions: **1a** (0.2 mmol), **4a** (1.5 equiv, 0.3 mmol), PC (1 mol%, 0.002 mmol), HE (2 equiv, 0.4 mmol), DIPEA (1 equiv, 0.2 mmol), HCOOH (1 equiv, 0.2 mmol), DCM (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

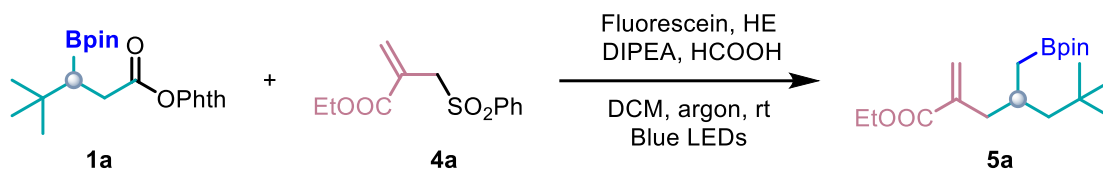
Table S6: Screening of different solvents.^a



entry	solvent	yield(%) ^b
1	THF	58%
2	toluene	31%
3	EA	33%
4	acetone	49%
5	CHCl ₃	44%
6	DCE	65%
7	DMF	38%
8	DMA	48%
9	DMSO	30%
10	dioxane	60%
11	DME	30%
12	CH ₃ CN	70%

^a Reaction conditions: **1a** (0.2 mmol), **4a** (1.5 equiv, 0.3 mmol), Fluorescein (1 mol%, 0.002 mmol), HE (2 equiv, 0.4 mmol), DIPEA (1 equiv, 0.2 mmol), HCOOH (1 equiv, 0.2 mmol), solvent (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

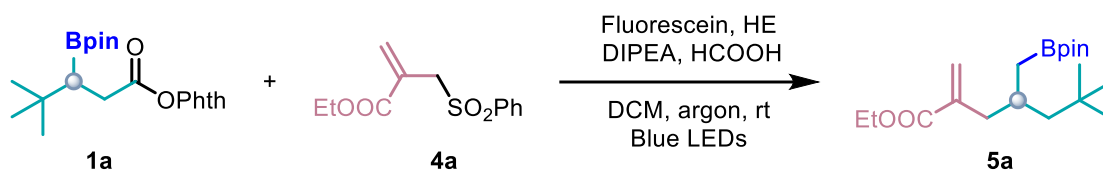
Table S7: The effects of equivalent of 4a, HE and DIPEA.



entry	4a (x equiv)	HE (y equiv)	DIPEA (z equiv)	yield(%) ^b
1	1.5	1	1	60%
2	1.5	1.5	1	68%
3	1.5	3	1	56%
4	2	2	1	70%
5	1.5	2	2	59%
6	1.5	2	3	54%

^a Reaction conditions: **1a** (0.2 mmol), **4a** (x equiv), Fluorescein (1 mol%, 0.002 mmol), HE (y equiv), DIPEA (z equiv), HCOOH (1 equiv, 0.2 mmol), DCM (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

Table S8: Control Experiments.^a



entry	PC	HE	DIPEA	HCOOH	yield(%) ^b
1	-	2	1	1	trace
2	Fluorescein	-	1	1	trace
3	Fluorescein	2	-	1	38%
4	Fluorescein	2	1	-	52%
5 ^c	Fluorescein	2	1	1	NR

^a Reaction conditions: **1a** (0.2 mmol), **4a** (1.5 equiv, 0.3 mmol), Fluorescein (1 mol%, 0.002 mmol), HE (2 equiv, 0.4 mmol), DIPEA (1 equiv, 0.2 mmol), HCOOH (1 equiv, 0.2 mmol), DCM (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield. ^c Without light.

2.1.3 The condition screening for the 7a

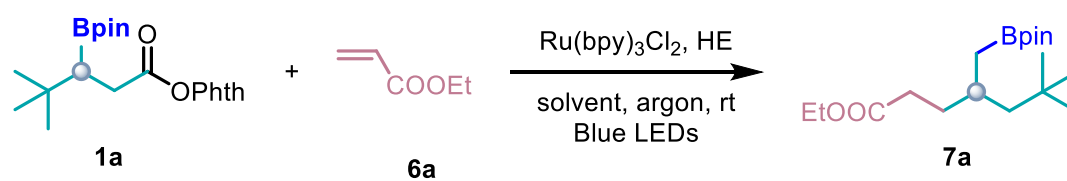
Table S9: Screening of photocatalysts.^a



entry	PC	yield(%) ^b
1	Ru(bpy) ₂ Cl ₂ •6H ₂ O	42%
2	Ru(bpy) ₃ (PF ₆) ₂	45%
3	Eosin Y	43%
4	Ir(ppy) ₃	45%
5	Ir(dtbpy)(bpy) ₂ PF ₆	39%
6	Ir(dCF ₃ bpy)(bpy) ₂ PF ₆	34%
7	Acid red 94	38%
8	4CzIPN	21%
9	Rhodamine B	31%
10	Eosin B	38%
11	Fluorescein	32%
12	Ru(bpy) ₃ Cl ₂	51%

^a Reaction conditions: **1a** (0.2 mmol), **6a** (1.5 equiv, 0.3 mmol), PC (1 mol%, 0.002 mmol), HE (1.5 equiv, 0.3 mmol), DCM (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

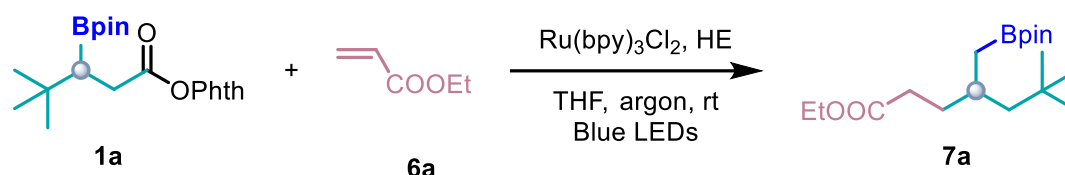
Table S10: Screening of different solvents.^a



entry	solvent	yield(%) ^b
1	CH ₃ CN	14%
2	dioxane	trace
3	THF	71%
4	EA	51%
5	DMF	trace
6	DCE	48%
7	DMSO	NR
8	2-MeTHF	31%
9	EtOH	trace
10	MeOH	43%
11	Et ₂ O	25%
12	toluene	trace
13	CH ₃ Cl	35%
14	DME	50%

^a Reaction conditions: **1a** (0.2 mmol), **6a** (1.5 equiv, 0.3 mmol), $\text{Ru}(\text{bpy})_3\text{Cl}_2$ (1 mol%, 0.002 mmol), HE (1.5 equiv, 0.3 mmol), solvent (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

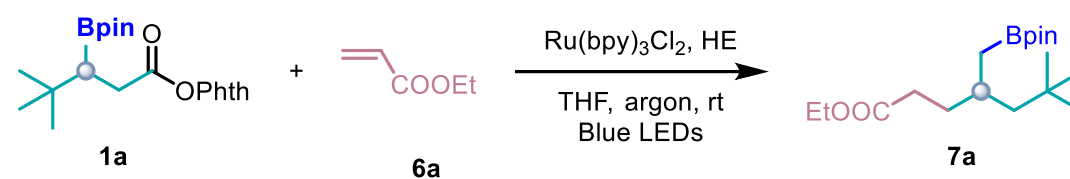
Table S11: The effects of equivalent of **6a and HE.**



entry	6a (x equiv)	HE (y equiv)	yield(%) ^b
1	1.5	1	52%
2	1.5	2	57%
3	1.5	3	57%
4	2	1.5	51%
5	3	1.5	42%

^a Reaction conditions: **1a** (0.2 mmol), **6a** (x equiv), $\text{Ru(bpy)}_3\text{Cl}_2$ (1 mol%, 0.002 mmol), HE (y equiv), THF (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield.

Table S12: Control Experiments.^a

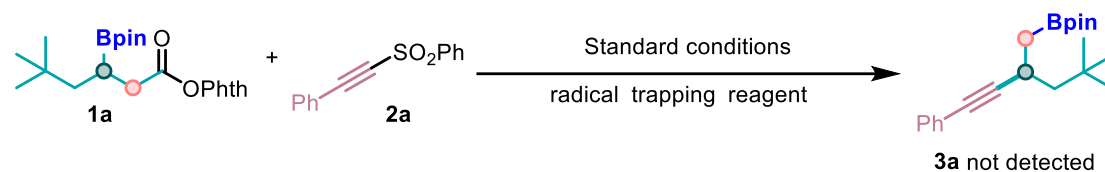


entry	PC	HE	yield(%) ^b
1	-	1.5	trace
2	$\text{Ru(bpy)}_3\text{Cl}_2$	-	trace
3 ^c	$\text{Ru(bpy)}_3\text{Cl}_2$	1.5	NR

^a Reaction conditions: **1a** (0.2 mmol), **6a** (1.5 equiv, 0.3 mmol), $\text{Ru(bpy)}_3\text{Cl}_2$ (1 mol%, 0.002 mmol), HE (1.5 equiv, 0.3 mmol), THF (2 mL) at room temperature, 40 W blue LEDs, 12 h in argon. ^b Isolated yield. ^c Without light.

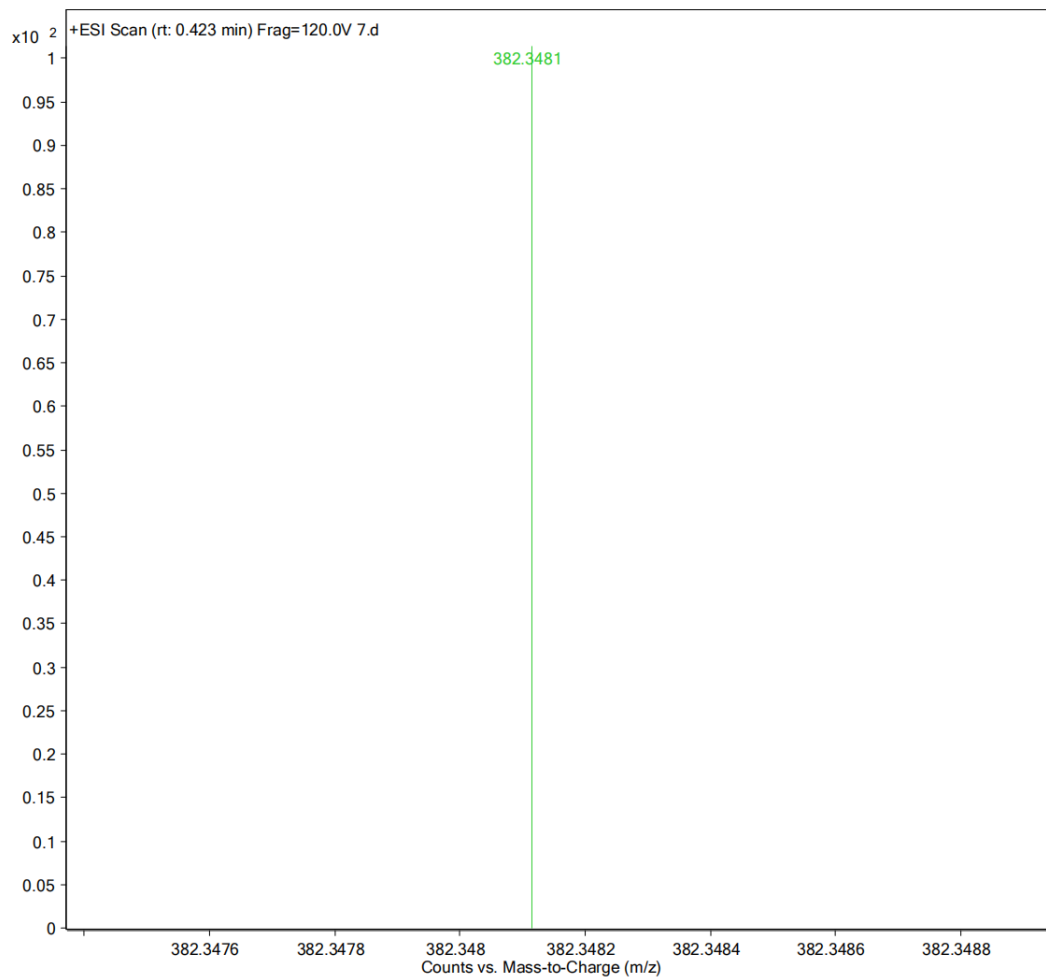
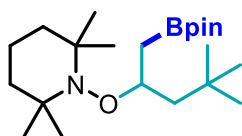
2.2 Control experiments

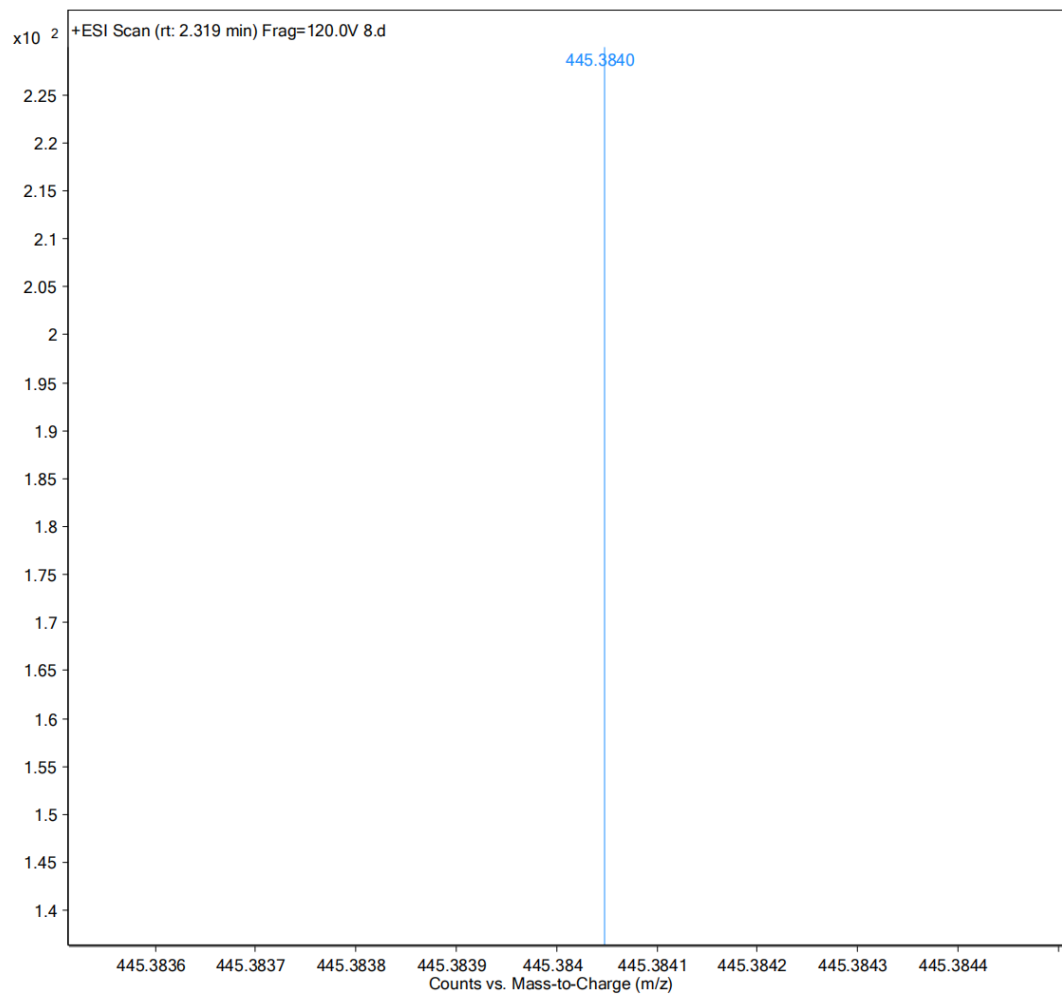
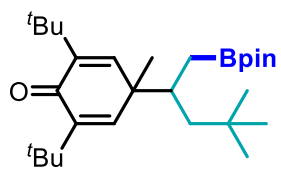
Radical capture experiment with HRMS analysis

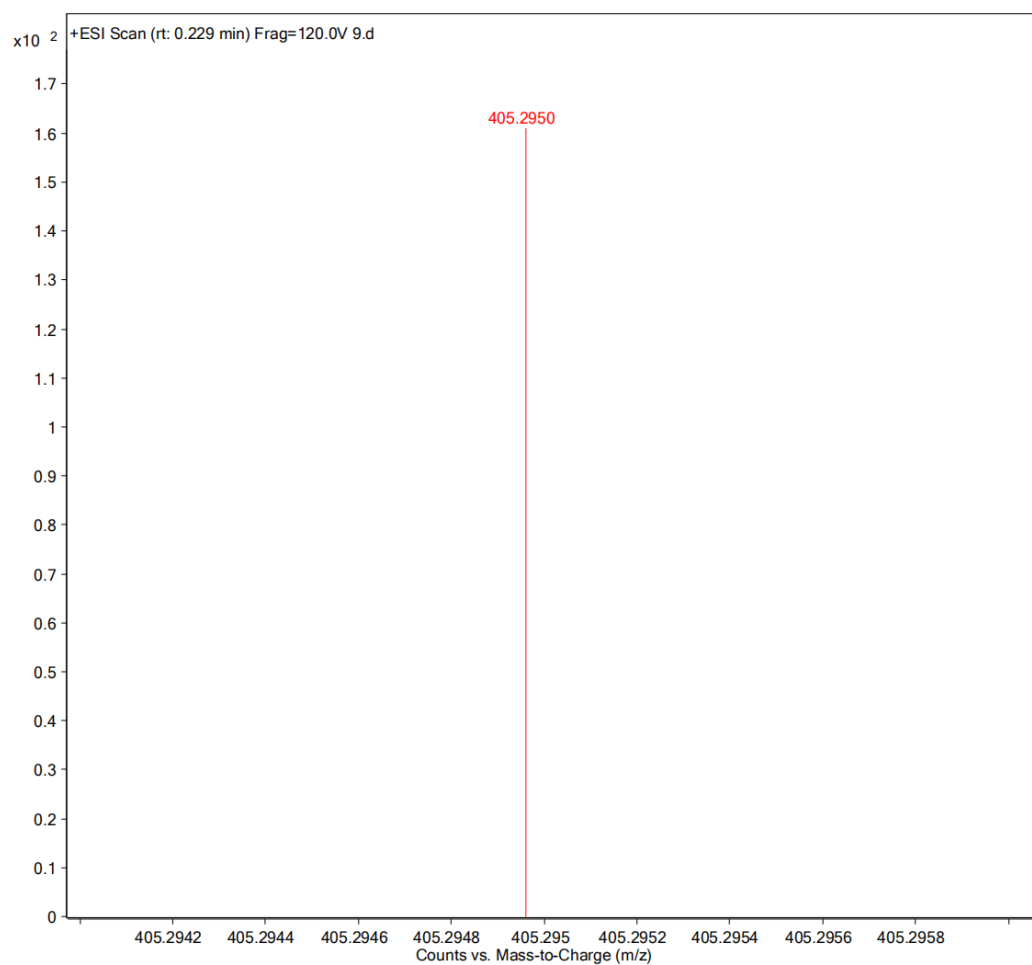
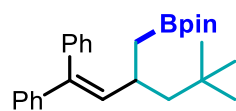


TMPEO (3 equiv)	BHT (3 equiv)	1,1-diphenylethylene (3 equiv)
17, HRMS: $[M+H]^+$ Calcd: 382.3487 Found: 382.3481	18, HRMS: $[M+H]^+$ Calcd: 445.3848 Found: 445.3840	19, HRMS: $[M+H]^+$ Calcd: 405.2959 Found: 405.2950

A mixture of **1** (0.2 mmol), **2** (0.3 mmol), Ir(ppy)₃ (2% mmol), HE (0.3 mmol) and radical trapping reagent (3 equiv, if solid) were charged into a Schleck tube, then the air was removed, argon was filled of Schleck tube and DIPEA (0.2 mmol), radical trapping reagent (3 equiv, if liquid), DCE (2 mL) is added the mixture. The mixture was stirred under irradiation from 40W Blue LEDs. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using PE/EA (50:1) to afford the corresponding product.

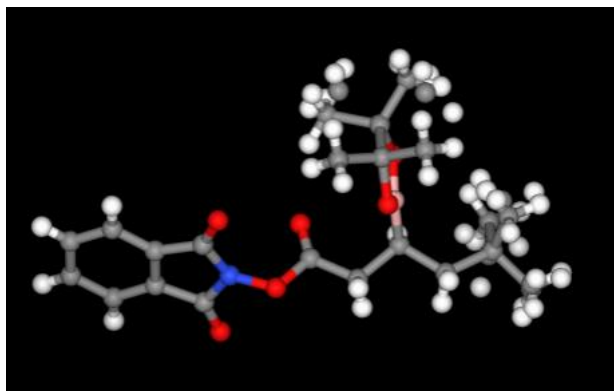






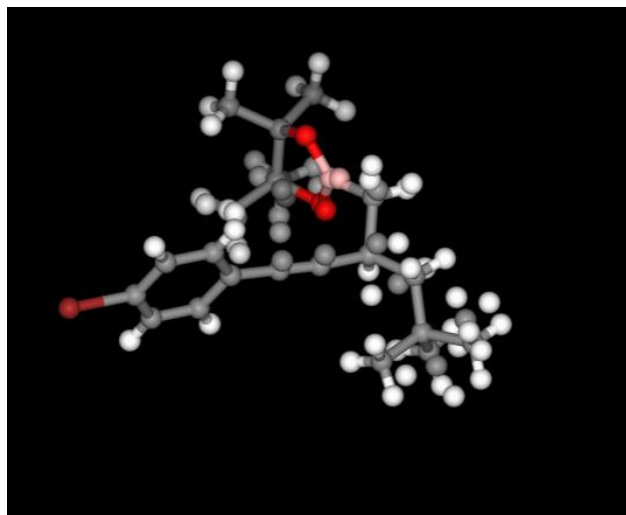
2.3 Crystal data

Crystallographic data for compound **1a** (CCDC 2247689) has been deposited with the Cambridge Crystallographic Data Centre. Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk).



Bond precision:	C-C = 0.0034 Å	Wavelength=0.71073
Cell:	a=9.751(1)	b=10.9070(11) c=12.1691(15)
	alpha=93.921(9)	beta=110.358(10) gamma=104.886(9)
Temperature:	293 K	
Volume	Calculated 1154.8(2)	Reported 1154.8(2)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C22 H30 B N O6	C22 H30 B N O6
Sum formula	C22 H30 B N O6	C22 H30 B N O6
Mr	415.28	415.28
Dx, g cm-3	1.194	1.194
Z	2	2
Mu (mm-1)	0.085	0.085
F000	444.0	444.0
F000'	444.23	
h,k,lmax	12,13,15	12,13,15
Nref	4777	4754
Tmin, Tmax		0.563, 1.000
Tmin'		
Correction method= # Reported T Limits: Tmin=0.563 Tmax=1.000		
AbsCorr = MULTI-SCAN		
Data completeness=	0.995	Theta (max)= 26.499
R(reflections)=	0.0663(2670)	wR2(reflections)=
S =	1.032	0.1867(4754)
Npar=	318	

Crystallographic data for compound **3e** (CCDC 2242317) has been deposited with the Cambridge Crystallographic Data Centre. Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk).



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Bond precision:   C-C = 0.0031 Å                Wavelength=1.54184

Cell:             a=10.1974(6)                   b=10.2772(6)                   c=11.2980(6)
                  alpha=100.4422(19)             beta=104.9074(18)             gamma=107.0325(18)
Temperature:      100 K

                  Calculated                      Reported
Volume            1050.97(11)                    1050.97(10)
Space group       P -1                           P -1
Hall group        -P 1                           -P 1
Moiety formula    C21 H30 B Br O2                C21 H30 B Br O2
Sum formula       C21 H30 B Br O2                C21 H30 B Br O2
Mr               405.16                          405.17
Dx,g cm-3        1.280                          1.280
Z                2                               2
Mu (mm-1)        2.732                          2.732
F000             424.0                          424.0
F000'            423.58
h,k,lmax         12,12,13                       12,12,13
Nref             3862                           3832
Tmin,Tmax        0.442,0.465                    0.513,0.753
Tmin'            0.334

Correction method= # Reported T Limits: Tmin=0.513 Tmax=0.753
AbsCorr = MULTI-SCAN

Data completeness= 0.992                        Theta(max)= 68.327

R(reflections)= 0.0304( 3805)                   wR2(reflections)=
                                                    0.0762( 3832)

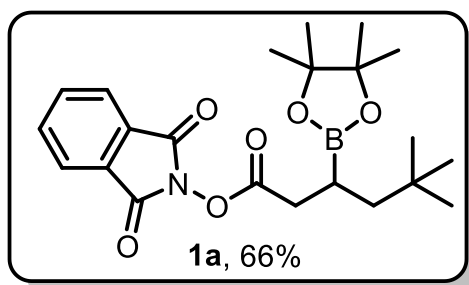
S = 1.192                                         Npar= 274

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2.4 Characterization data for Substrates

1,3-dioxoisindolin-2-yl

5,5-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1a)



Following the **general procedure 1** on 50 mmol scale, **1a** was obtained in 66% yield (13.7 g) as a white solid (m.p. : 86-88 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

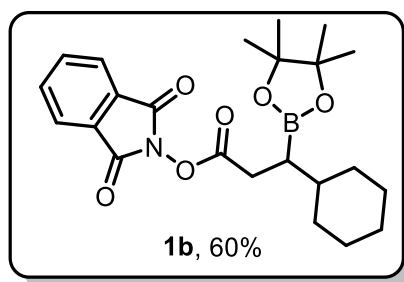
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, J = 5.4, 3.1 Hz, 2H), 7.77 (dd, J = 5.4, 3.1 Hz, 2H), 2.78 (dd, J = 16.8, 7.3 Hz, 1H), 2.69 (dd, J = 16.9, 7.5 Hz, 1H), 1.56 – 1.48 (m, 1H), 1.23 (d, J = 1.9 Hz, 13H), 0.92 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.8, 161.9, 134.6, 129.0, 123.9, 83.5, 44.5, 34.3, 31.1, 29.6, 24.7.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.58.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{22}\text{H}_{30}\text{BNO}_6$ 416.2239; Found: 416.2236.

1,3-dioxoisindolin-2-yl 3-cyclohexyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate (1b)



Following the **general procedure 1** on 20 mmol scale, **1b** was obtained in 60% yield (5.1 g) as a white solid (m.p. : 108-110 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

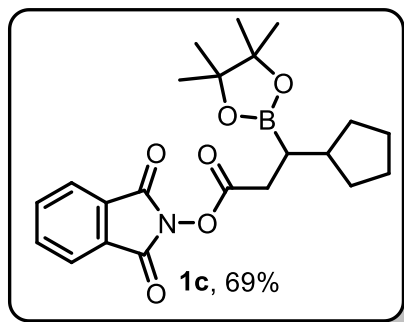
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, J = 5.5, 3.1 Hz, 2H), 7.77 (dd, J = 5.5, 3.1 Hz, 2H), 2.85 (dd, J = 17.4, 10.1 Hz, 1H), 2.72 (dd, J = 17.4, 5.5 Hz, 1H), 1.77 – 1.69 (m, 4H), 1.68 – 1.64 (m, 1H), 1.53 – 1.40 (m, 2H), 1.24 (d, J = 9.0 Hz, 14H), 1.16 – 1.02 (m, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6, 134.6, 129.0, 123.9, 83.5, 39.1, 32.4, 31.9, 30.1, 26.7, 26.6, 26.4, 24.9, 24.6.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.97.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{23}\text{H}_{31}\text{BNO}_6$ 429.2239; Found: 429.2236.

1,3-dioxoisindolin-2-yl 3-cyclopentyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate (1c)



Following the **general procedure 1** on 20 mmol scale, **1c** was obtained in 69% yield (5.7 g) as a white solid (m.p. : 107-109 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

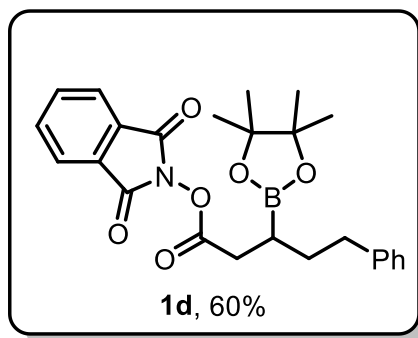
^1H NMR (500 MHz, Chloroform-*d*) δ 7.79 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.70 (dd, $J = 5.5, 3.1$ Hz, 2H), 2.80 – 2.66 (m, 2H), 1.83 – 1.70 (m, 3H), 1.55 (m, 2H), 1.50 – 1.42 (m, 2H), 1.36 (m, 1H), 1.16 (d, $J = 6.9$ Hz, 14H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 161.9, 134.6, 129.0, 123.9, 83.5, 41.1, 3.18, 31.9, 31.6, 25.3, 25.1, 24.9, 24.6.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.89.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{22}\text{H}_{29}\text{BNO}_6$ 414.2082; Found: 414.2079.

1,3-dioxoisindolin-2-yl 5-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1d)



Following the **general procedure 1** on 20 mmol scale, **1d** was obtained in 60% yield (5.4 g) as a white solid (m.p. : 123-125°C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

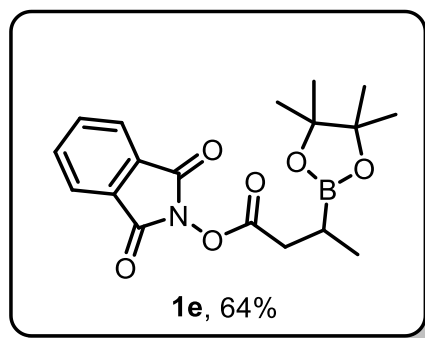
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.77 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.28 (m, 2H), 7.23 – 7.15 (m, 3H), 2.88 (dd, $J = 17.1, 8.4$ Hz, 1H), 2.79 (dd, $J = 17.2, 6.4$ Hz, 1H), 2.69 (m, 2H), 1.94 – 1.73 (m, 3H), 1.61 – 1.52 (m, 1H), 1.26 (d, $J = 4.2$ Hz, 12H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.0, 161.9, 142.2, 134.7, 129.0, 128.5, 128.4, 125.8, 123.9, 83.7, 34.9, 32.2, 32.2, 24.8, 24.6.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.27.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{25}\text{H}_{29}\text{BNO}_6$ 450.2082; Found: 450.2086.

1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butanoate (1e)



Following the **general procedure 1** on 20 mmol scale, **1e** was obtained in 64% yield (4.6 g) as a white solid (m.p. : 47-49°C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

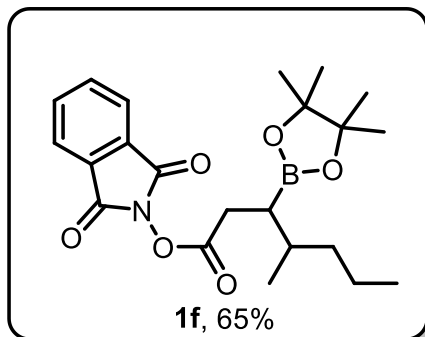
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.77 (dd, $J = 5.5, 3.1$ Hz, 2H), 2.83 (dd, $J = 16.9, 7.0$ Hz, 1H), 2.67 (dd, $J = 16.9, 7.4$ Hz, 1H), 1.55 (m, 1H), 1.23 (d, $J = 2.6$ Hz, 11H), 1.12 (d, $J = 7.5$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.0, 161.9, 134.6, 129.0, 123.9, 83.6, 34.1, 24.7, 24.6, 14.8.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.20.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{18}\text{H}_{23}\text{BNO}_6$ 360.1613; Found: 360.1609.

1,3-dioxoisindolin-2-yl 4-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptanoate (1f)



Following the **general procedure 1** on 20 mmol scale, **1f** was obtained in 65% yield (5.4 g) as a white solid (m.p. : 70-72°C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

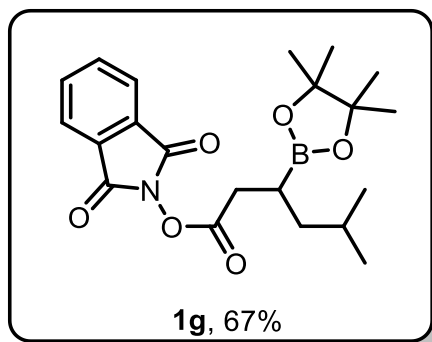
^1H NMR (500 MHz, Chloroform-*d*) δ 7.86 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.76 (dd, $J = 5.5, 3.0$ Hz, 2H), 2.85 (m, 1H), 2.66 (m, 1H), 1.78 – 1.65 (m, 1H), 1.60 – 1.51 (m, 1H), 1.37 (m, 2H), 1.30 – 1.17 (m, 14H), 0.94 (dd, $J = 6.8, 3.4$ Hz, 3H), 0.92 – 0.86 (m, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.7, 170.5, 161.9, 134.6, 129.0, 123.9, 83.6, 83.5, 38.5, 37.6, 33.9, 33.4, 30.5, 29.1, 25.0, 24.9, 24.6, 24.5, 20.6, 20.5, 18.6, 18.2, 14.3, 14.2.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.98.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{22}\text{H}_{30}\text{BNO}_6$ 416.2239; Found: 416.2233.

1,3-dioxoisindolin-2-yl 5-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1g)



Following the **general procedure 1** on 20 mmol scale, **1g** was obtained in 67% yield (5.4 g) as a white solid (m.p. : 68-70 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

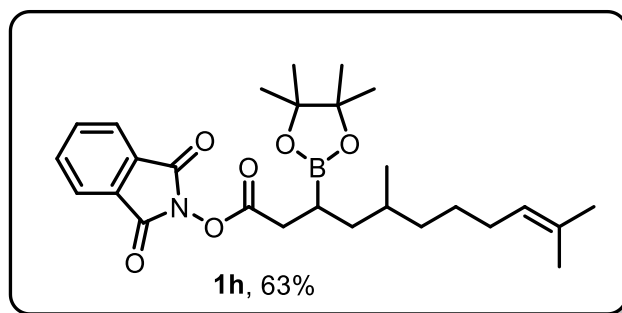
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, $J = 5.4, 3.1$ Hz, 2H), 7.77 (dd, $J = 5.5, 3.1$ Hz, 2H), 2.83 – 2.67 (m, 2H), 1.59 – 1.53 (m, 1H), 1.46 – 1.41 (m, 1H), 1.34 – 1.28 (m, 2H), 1.23 (d, $J = 5.2$ Hz, 14H), 0.91 (dd, $J = 11.9, 6.6$ Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.1, 161.9, 134.6, 129.0, 123.9, 83.5, 39.3, 32.3, 26.6, 24.8, 24.6, 22.8, 22.7, 22.6.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.38.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{21}\text{H}_{29}\text{BNO}_6$ 402.2082; Found: 402.2080.

1,3-dioxoisindolin-2-yl 5,10-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)undec-9-enoate (1h)



Following the **general procedure 1** on 20 mmol scale, **1h** was obtained in 63%% yield (6.1 g) as a white solid (m.p. : 70-71 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

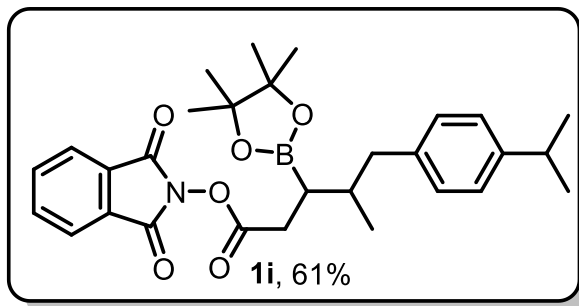
^1H NMR (500 MHz, Chloroform-*d*) δ 7.86 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.76 (dd, $J = 5.5, 3.1$ Hz, 2H), 5.14 – 5.03 (m, 1H), 2.84 – 2.62 (m, 2H), 1.97 (m, 2H), 1.66 (s, 3H), 1.59 (s, 4H), 1.55 – 1.45 (m, 2H), 1.42 – 1.29 (m, 2H), 1.22 (d, $J = 5.2$ Hz, 12H), 1.17 – 1.10 (m, 1H), 0.89 (dd, $J = 13.4, 6.5$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.0, 161.9, 134.7, 131.1, 129.0, 124.9, 123.9, 83.5, 83.5, 38.0, 37.4, 32.9, 31.3, 25.7, 25.5, 24.8, 24.6, 24.6, 19.5, 17.7.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.28.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{27}\text{H}_{39}\text{BNO}_6$ 484.2865; Found: 484.2858.

1,3-dioxoisindolin-2-yl 5-(4-isopropylphenyl)-4-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1i)



Following the **general procedure 1** on 20 mmol scale, **1i** was obtained in 61% yield (6.2 g) as a white solid (mp: 81-83 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

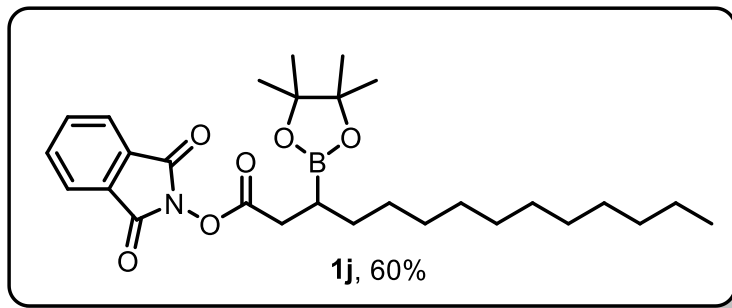
^1H NMR (500 MHz, Chloroform-*d*) δ 7.88 (m, 2H), 7.77 (m, 2H), 7.16 – 7.06 (m, 4H), 3.00 – 2.84 (m, 2H), 2.83 – 2.67 (m, 2H), 2.37 (m, 1H), 2.09 – 1.94 (m, 1H), 1.64 (m, 1H), 1.32 – 1.20 (m, 18H), 0.91 (dd, $J = 8.6, 6.8$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.4, 161.9, 146.3, 146.2, 138.4, 138.3, 134.7, 129.1, 129.1, 129.0, 126.3, 126.2, 123.9, 83.7, 83.7, 42.3, 41.6, 36.7, 35.9, 33.7, 30.8, 29.4, 25.1, 24.9, 24.6, 24.1, 18.1, 17.8.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.21.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{29}\text{H}_{37}\text{BNO}_6$ 506.2708; Found: 506.2706.

1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentadecanoate (1j)



Following the **general procedure 1** on 20 mmol scale, **1j** was obtained in 60% yield (6.0 g) as a white solid (mp: 65-67 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

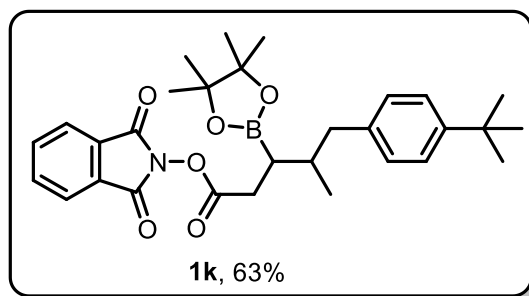
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.77 (dd, $J = 5.5, 3.1$ Hz, 2H), 2.81 (dd, $J = 17.0, 8.0$ Hz, 1H), 2.71 (dd, $J = 17.1, 5.9$ Hz, 1H), 1.53 – 1.47 (m, 2H), 1.33 – 1.21 (m, 31H), 0.87 (t, $J = 6.9$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.1, 161.9, 134.6, 129.0, 123.9, 83.5, 32.2, 31.9, 30.2, 29.7, 29.7, 29.6, 29.5, 29.4, 28.6, 24.8, 24.6, 22.7, 14.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.30.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{28}\text{H}_{42}\text{BNO}_6$ 500.3178; Found: 500.3173.

1,3-dioxoisindolin-2-yl 5-(4-(tert-butyl)phenyl)-4-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1k)



Following the **general procedure 1** on 20 mmol scale, **1k** was obtained in 63% yield (6.5 g) as a white solid (mp: 87-89 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

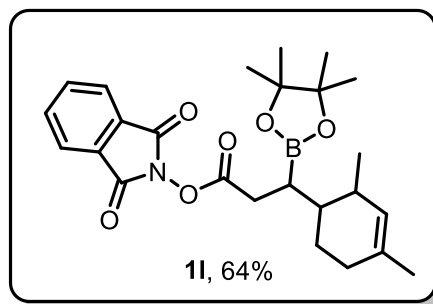
^1H NMR (500 MHz, Chloroform-*d*) δ 7.88 (m, 2H), 7.78 (m, 2H), 7.32 – 7.28 (m, 2H), 7.17 – 7.09 (m, 2H), 2.94 (m, 1H), 2.85 – 2.67 (m, 2H), 2.02 (m, 1H), 1.71 – 1.59 (m, 1H), 1.31 (d, $J = 1.8$ Hz, 9H), 1.26 (dd, $J = 10.5, 5.1$ Hz, 12H), 0.91 (dd, $J = 9.6, 6.8$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.4, 161.9, 148.6, 148.5, 138.0, 138.0, 134.7, 129.0, 128.9, 128.8, 125.2, 125.1, 123.9, 83.7, 83.7, 42.1, 41.5, 36.6, 35.9, 34.4, 31.5, 30.8, 29.4, 25.1, 24.9, 24.6, 18.1, 17.9.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.09.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{30}\text{H}_{38}\text{BNO}_6$ 520.2865; Found: 520.2863.

1,3-dioxoisindolin-2-yl 3-(2,4-dimethylcyclohex-3-en-1-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate (1l)



Following the **general procedure 1** on 20 mmol scale, **1l** was obtained in 64%% yield (5.8 g) as a white solid (mp: 82-85 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

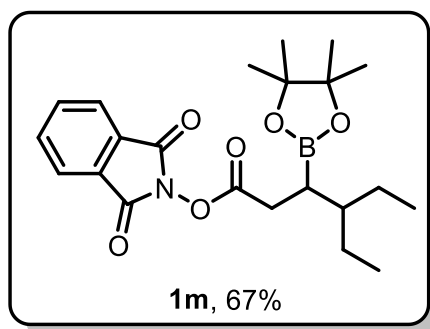
^1H NMR (500 MHz, Chloroform-*d*) δ 7.89 – 7.85 (m, 2H), 7.77 (dd, $J = 5.5, 3.1$ Hz, 2H), 5.35 (m, 1H), 2.90 – 2.77 (m, 2H), 2.23 (m, 1H), 2.01 – 1.90 (m, 2H), 1.80 – 1.72 (m, 1H), 1.66 – 1.59 (m, 4H), 1.49 – 1.38 (m, 2H), 1.22 (d, $J = 7.0$ Hz, 12H), 0.90 (d, $J = 7.0$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 161.9, 134.7, 132.6, 129.0, 127.9, 123.9, 83.5, 38.7, 32.8, 31.2, 31.1, 24.9, 24.9, 24.6, 23.4, 22.4, 15.5.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.25.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{25}\text{H}_{32}\text{BNO}_6$ 454.2395; Found: 454.2394.

1,3-dioxoisindolin-2-yl 4-ethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1m)



Following the **general procedure 1** on 20 mmol scale, **1m** was obtained in 67% yield (5.6 g) as a white solid (mp: 75-77 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

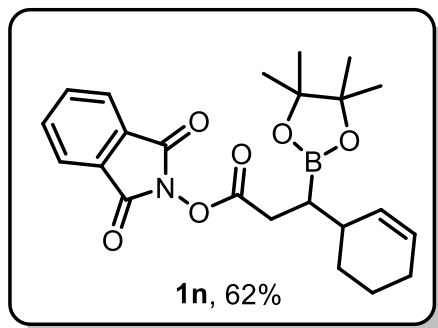
^1H NMR (500 MHz, Chloroform-*d*) δ 7.41 – 7.37 (m, 2H), 7.27 (m, 3H), 2.97 (m, 1H), 1.59 (m, 1H), 1.55 – 1.46 (m, 2H), 1.44 – 1.38 (m, 1H), 1.28 (s, 13H), 1.20 (dd, $J = 15.1, 10.2$ Hz, 1H), 1.06 (dd, $J = 15.2, 5.9$ Hz, 1H), 0.96 (m, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.5, 128.1, 127.2, 124.5, 93.9, 83.2, 81.5, 46.8, 30.4, 25.0, 24.8, 24.7, 23.9, 22.8, 12.0, 11.8.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.89.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{22}\text{H}_{30}\text{BNO}_6$ 416.2239; Found: 416.2235.

1,3-dioxoisindolin-2-yl 3-(cyclohex-2-en-1-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate (1n)



Following the **general procedure 1** on 20 mmol scale, **1n** was obtained in 62% yield (5.3 g) as a white solid (mp: 95-97 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

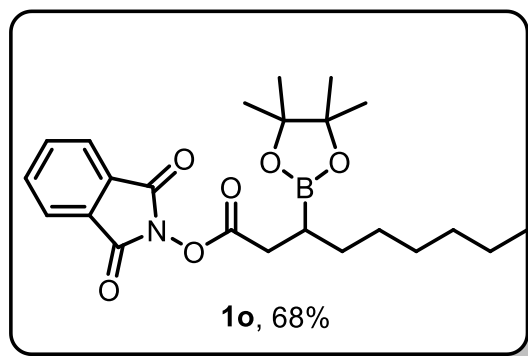
^1H NMR (500 MHz, Chloroform-*d*) δ 7.37 (m, 2H), 7.26 – 7.24 (m, 2H), 5.73 – 5.64 (m, 2H), 2.83 – 2.70 (m, 1H), 2.26 – 1.91 (m, 5H), 1.72 – 1.61 (m, 1H), 1.46 – 1.37 (m, 1H), 1.26 (s, 12H), 1.21 – 1.05 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.6, 128.1, 127.3, 127.1, 126.8, 126.7, 126.5, 124.3, 93.4, 93.1, 83.3, 82.1, 81.9, 39.6, 39.4, 33.7, 33.4, 30.1, 27.9, 27.5, 25.7, 25.3, 25.0, 25.0, 24.7.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.96.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{23}\text{H}_{28}\text{BNO}_6$ 426.2082; Found: 426.2080.

1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)nonanoate (1o)



Following the **general procedure 1** on 20 mmol scale, **1o** was obtained in 68% yield (5.8 g) as a white solid (mp: 91-93 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

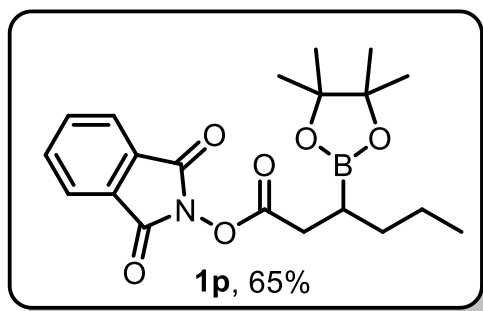
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, J = 5.5, 3.1 Hz, 2H), 7.79 – 7.74 (m, 2H), 2.81 (dd, J = 17.0, 8.0 Hz, 1H), 2.71 (dd, J = 17.1, 6.0 Hz, 1H), 1.57 – 1.44 (m, 3H), 1.40 – 1.27 (m, 7H), 1.23 (d, J = 5.7 Hz, 13H), 0.88 (t, J = 6.9 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.1, 161.9, 134.6, 129.0, 123.9, 83.5, 32.1, 31.9, 30.1, 28.2, 24.8, 24.5, 22.5, 14.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.30.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{23}\text{H}_{32}\text{BNO}_6$ 430.2395; Found: 430.2393.

1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1p)



Following the **general procedure 1** on 20 mmol scale, **1p** was obtained in 65% yield (5.0 g) as a white solid (mp: 73-75 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

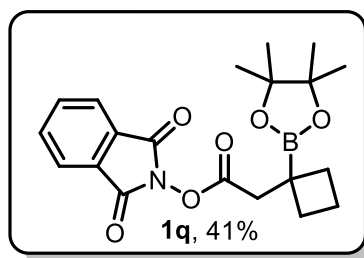
^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 (dd, J = 5.5, 3.1 Hz, 2H), 7.77 (dd, J = 5.5, 3.1 Hz, 2H), 2.81 (dd, J = 17.0, 7.9 Hz, 1H), 2.71 (dd, J = 17.0, 5.9 Hz, 1H), 1.56 – 1.44 (m, 3H), 1.37 – 1.27 (m, 6H), 1.23 (d, J = 5.6 Hz, 12H), 0.88 (t, J = 6.8 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.1, 161.9, 134.7, 129.0, 123.9, 83.5, 32.2, 31.9, 30.2, 28.2, 24.8, 24.6, 22.5, 14.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.34.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{20}\text{H}_{27}\text{BNO}_6$ 388.1926; Found: 388.1923.

1,3-dioxoisindolin-2-yl 2-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)cyclobutyl)acetate (1q)



Following the **general procedure 1** on 20 mmol scale, **1q** was obtained in 41% yield (3.16 g) as a white solid (mp: 66-68 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

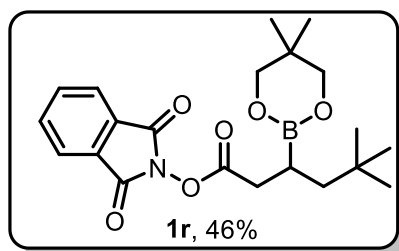
^1H NMR (500 MHz, Chloroform-*d*) δ 7.80 (dd, $J = 5.5, 3.1$ Hz, 2H), 7.70 (dd, $J = 5.5, 3.1$ Hz, 2H), 2.88 (s, 2H), 2.22 – 2.15 (m, 2H), 2.03 – 1.88 (m, 2H), 1.85 – 1.77 (m, 2H), 1.18 (s, 12H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.2, 161.9, 134.6, 129.0, 123.9, 83.6, 39.9, 29.4, 24.5, 18.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.23.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{20}\text{H}_{24}\text{BNO}_6$ 386.1769; Found: 386.1765.

1,3-dioxoisindolin-2-yl 3-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-5,5-dimethylhexanoate (1r)



Following the **general procedure 1** on 20 mmol scale, **1r** was obtained in 46% yield (3.69 g) as a white solid (mp: 113-115 °C), $R_f = 0.4$ (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

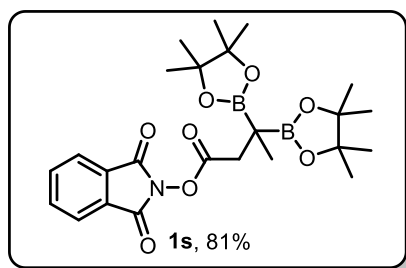
^1H NMR (500 MHz, Chloroform-*d*) δ 7.89 – 7.84 (m, 2H), 7.79 – 7.76 (m, 2H), 3.60 (d, $J = 0.8$ Hz, 4H), 2.75 (dd, $J = 16.6, 8.2$ Hz, 1H), 2.66 (dd, $J = 16.6, 6.9$ Hz, 1H), 1.61 (t, $J = 4.2$ Hz, 1H), 1.45 – 1.39 (m, 1H), 1.23 (dd, $J = 13.7, 4.4$ Hz, 1H), 0.93 (dd, $J = 8.7, 0.9$ Hz, 15H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 162.0, 134.6, 129.1, 123.9, 72.0, 44.8, 34.4, 31.7, 31.2, 29.6, 22.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.72.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{21}\text{H}_{28}\text{BNO}_6$ 402.2082; Found: 402.2080.

1,3-dioxoisindolin-2-yl 3,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butanoate (1s)



Following the **general procedure 1** on 10 mmol scale, **1s** was obtained in 81% yield (3.9 g) as a white solid (mp: 103-105 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

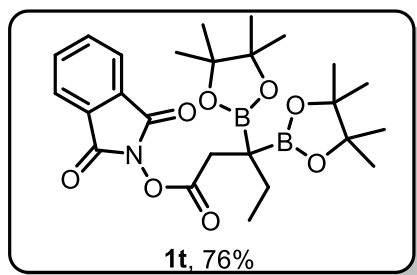
^1H NMR (500 MHz, Chloroform-*d*) δ 7.86 (dd, J = 5.5, 3.1 Hz, 2H), 7.76 (dd, J = 5.5, 3.1 Hz, 2H), 2.94 (s, 2H), 1.21 (d, J = 4.5 Hz, 27H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 161.9, 134.6, 129.1, 124.0, 123.8, 83.6, 35.5, 24.6, 24.6, 16.2.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.37, 32.90.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{24}\text{H}_{33}\text{B}_2\text{NO}_8$ 486.2465; Found: 486.2463.

1,3-dioxoisindolin-2-yl 3,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1t**)**



Following the **general procedure 1** on 10 mmol scale, **1t** was obtained in 76% yield (3.8 g) as a white solid (mp: 113-115 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

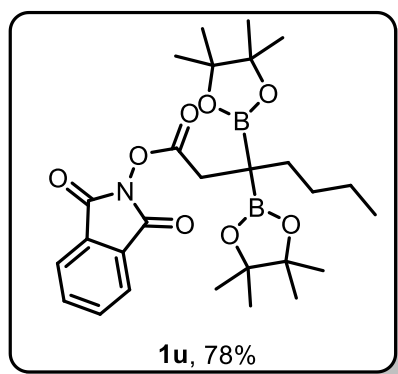
^1H NMR (500 MHz, Chloroform-*d*) δ 7.84 (dd, J = 5.5, 3.1 Hz, 2H), 7.75 (dd, J = 5.5, 3.1 Hz, 2H), 3.01 (s, 2H), 1.79 (d, J = 7.5 Hz, 2H), 1.20 (d, J = 1.9 Hz, 24H), 0.92 (t, J = 7.5 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 161.9, 134.6, 129.1, 123.8, 83.6, 31.1, 24.7, 22.4, 11.5.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 35.04, 34.43.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for: $\text{C}_{25}\text{H}_{35}\text{B}_2\text{NO}_8$ 500.2622; Found: 500.2620.

1,3-dioxoisindolin-2-yl 3,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptanoate (1u**)**



Following the **general procedure 1** on 10 mmol scale, **1u** was obtained in 78% yield (4.0 g) as a white solid (mp: 142-144 °C), R_f = 0.4 (silica gel, PE: EA = 5:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.86 (dd, J = 5.5, 3.1 Hz, 2H), 7.75 (dd, J = 5.5, 3.1 Hz, 2H), 3.01 (s, 2H), 1.76 – 1.71 (m, 2H), 1.29 – 1.27 (m, 4H), 1.21 (s, 24H), 0.90 – 0.84 (m, 3H).

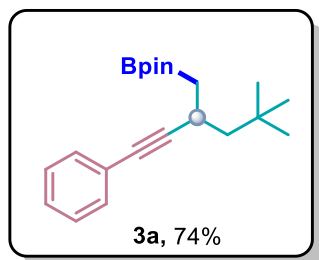
¹³C NMR (126 MHz, Chloroform-*d*) δ 170.4, 161.9, 134.5, 129.1, 123.8, 83.6, 31.8, 31.5, 29.8, 29.5, 27.4, 24.7, 24.6, 22.6, 14.1.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.42, 32.77.

HRMS (ESI) m/z: [M+H]⁺ Calcd. for: C₂₇H₄₀B₂NO₈ 528.2935; Found: 528.2938.

2.5 Characterization data for Products

2-(4,4-dimethyl-2-(phenylethynyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 74% (48.3 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

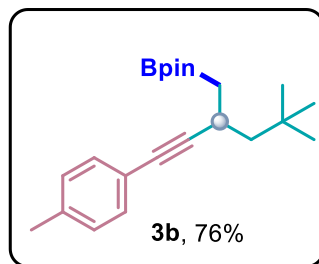
^1H NMR (500 MHz, Chloroform-*d*) δ 7.37 – 7.33 (m, 2H), 7.27 – 7.22 (m, 3H), 2.83 (m, 1H), 1.37 (d, J = 2.5 Hz, 1H), 1.34 (dd, J = 6.0, 2.4 Hz, 1H), 1.26 (d, J = 1.6 Hz, 12H), 1.19 (dd, J = 15.3, 8.4 Hz, 1H), 1.08 (dd, J = 15.3, 6.9 Hz, 1H), 1.02 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.23, 128.04, 127.15, 124.52, 96.58, 83.17, 51.79, 31.13, 29.95, 24.87, 24.79, 24.07.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.41.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{31}\text{BO}_2$ 327.2490; Found: 327.2487.

2-(4,4-dimethyl-2-(p-tolylethynyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3b)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 76% (51.7 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

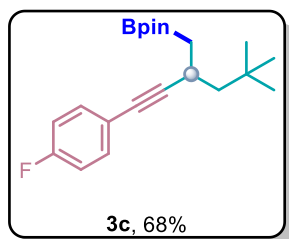
^1H NMR (500 MHz, Chloroform-*d*) δ 7.24 (d, J = 7.8 Hz, 2H), 7.06 (d, J = 7.8 Hz, 2H), 2.83 (m, 1H), 2.31 (s, 3H), 1.68 – 1.61 (m, 1H), 1.37 (d, J = 2.8 Hz, 1H), 1.26 (s, 12H), 1.20 – 1.15 (m, 1H), 1.10 – 1.05 (m, 1H), 1.02 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 137.0, 131.1, 128.8, 121.5, 95.7, 83.1, 51.8, 31.1, 30.0, 24.9, 24.8, 24.1, 21.3.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.03.

HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ Calcd. for $\text{C}_{22}\text{H}_{33}\text{BO}_2$ 363.2466; Found: 363.2461.

2-(2-((4-fluorophenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 68% (44.1 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.34 – 7.28 (m, 2H), 6.97 – 6.91 (m, 2H), 2.86 – 2.76 (m, 1H), 1.62 (dd, J = 13.6, 10.4 Hz, 1H), 1.38 – 1.32 (m, 1H), 1.26 – 1.23 (m, 12H), 1.17 (dd, J = 15.3, 8.3 Hz, 1H), 1.07 (dd, J = 15.3, 6.9 Hz, 1H), 1.00 (s, 9H).

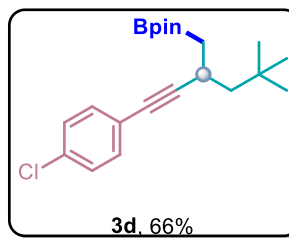
^{13}C NMR (126 MHz, Chloroform-*d*) δ 162.9, 160.9, 133.0, 132.9, 120.6, 120.6, 115.4, 115.2, 96.2, 83.2, 51.8, 31.2, 30.0, 24.9, 24.8, 24.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.66.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -112.88.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{30}\text{BFO}_2$ 352.2545; Found: 325.2545.

2-(2-((4-chlorophenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3d)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 66% (47.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

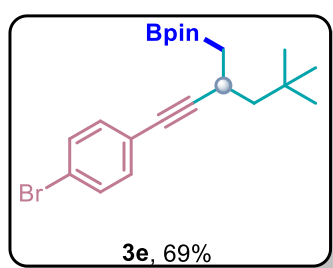
^1H NMR (500 MHz, Chloroform-*d*) δ 7.86 (dd, J = 5.5, 3.1 Hz, 2H), 7.76 (dd, J = 5.5, 3.1 Hz, 2H), 2.73 (m, 2H), 1.65 (m, 1H), 1.56 (m, 1H), 1.43 (m, 1H), 1.33 – 1.27 (m, 1H), 1.22 (d, J = 5.2 Hz, 12H), 0.90 (dd, J = 11.9, 6.6 Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 133.1, 132.5, 128.4, 123.0, 97.7, 83.2, 51.7, 31.2, 30.0, 24.9, 24.8, 24.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.77.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{30}\text{BClO}_2$ 361.2100; Found: 361.2098.

2-(2-((4-bromophenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 69% (55.8 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

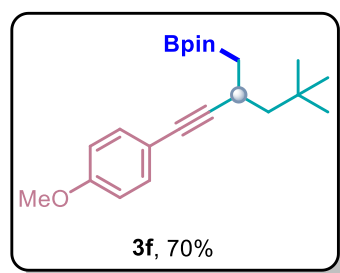
^1H NMR (500 MHz, Chloroform-*d*) δ 7.41 – 7.34 (m, 2H), 7.23 – 7.17 (m, 2H), 2.84 – 2.75 (m, 1H), 1.66 – 1.61 (m, 1H), 1.38 – 1.32 (m, 1H), 1.24 (s, 12H), 1.18 – 1.13 (m, 1H), 1.07 (dd, J = 15.4, 6.8 Hz, 1H), 1.00 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 132.8, 131.3, 123.5, 121.3, 97.9, 83.2, 51.7, 31.2, 30.0, 24.9, 24.82, 2.14, 1.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.64.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{30}\text{BBrO}_2$ 405.1595; Found: 405.1595.

2-((4-methoxyphenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3f)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 70% (49.8 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

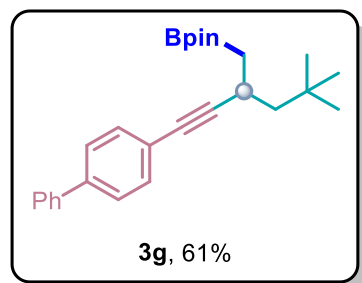
^1H NMR (500 MHz, Chloroform-*d*) δ 7.26 (dd, J = 6.1, 2.6 Hz, 2H), 6.85 – 6.79 (m, 2H), 6.30 (d, J = 15.8 Hz, 1H), 5.88 (dd, J = 15.8, 8.9 Hz, 1H), 3.79 (s, 3H), 2.50 (m, 1H), 1.58 (m, 1H), 1.34 – 1.27 (m, 2H), 1.20 (d, J = 3.3 Hz, 13H), 0.95 (dd, J = 15.1, 6.1 Hz, 1H), 0.86 (dd, J = 6.6, 4.9 Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 134.7, 130.8, 127.7, 127.1, 113.8, 83.0, 55.3, 47.6, 37.1, 25.7, 25.0, 24.8, 23.6, 22.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.91.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{33}\text{BO}_3$ 357.2596; Found: 357.2596.

2-([1,1'-biphenyl]-4-ylethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3g)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 61% (49.0 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

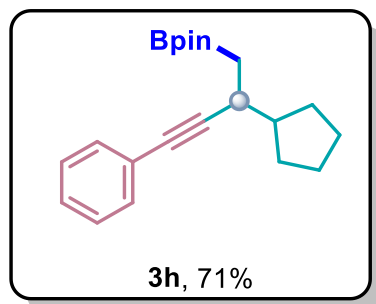
¹H NMR (500 MHz, Chloroform-*d*) δ 7.61 – 7.56 (m, 2H), 7.54 – 7.49 (m, 2H), 7.44 (m, 4H), 7.35 (m, 1H), 2.88 (m, 1H), 1.72 – 1.64 (m, 1H), 1.40 (dd, J = 13.6, 2.9 Hz, 1H), 1.29 (d, J = 1.7 Hz, 12H), 1.24 – 1.20 (m, 1H), 1.05 (s, 10H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 140.6, 140.0, 131.7, 128.8, 127.4, 127.0, 126.8, 123.6, 97.4, 83.3, 51.9, 31.2, 30.0, 30.0, 25.0, 24.9, 24.2.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.58.

HRMS (ESI) m/z : $[M+K]^+$ Calcd. for C₂₇H₃₅BO₂ 441.2367; Found: 441.2365.

2-(2-cyclopentyl-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3h)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 71% (46.0 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

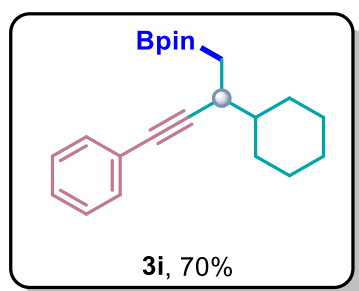
¹H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.35 (m, 2H), 7.27 – 7.23 (m, 3H), 2.77 (dt, J = 9.6, 6.4 Hz, 1H), 2.00 – 1.91 (m, 1H), 1.88 – 1.74 (m, 2H), 1.66 (p, J = 5.2, 4.7 Hz, 2H), 1.58 – 1.47 (m, 3H), 1.40 – 1.33 (m, 1H), 1.26 (s, 12H), 1.15 – 1.07 (m, 2H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 131.54, 128.09, 127.95, 127.25, 124.43, 94.05, 83.23, 46.22, 32.96, 31.07, 29.65, 25.72, 25.61, 25.01, 24.78, 24.68.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.85.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for C₂₁H₂₉BO₂ 325.2333; Found: 325.2337.

2-(2-cyclohexyl-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3i)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 70% (47.3 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

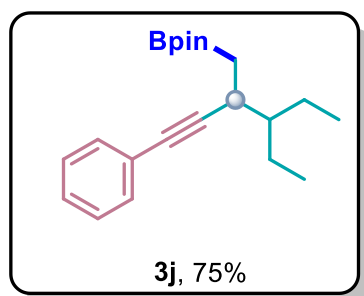
¹H NMR (500 MHz, Chloroform-*d*) δ 7.42 – 7.38 (m, 2H), 7.27 (m, 3H), 2.69 (m, 1H), 1.98 – 1.91 (m, 1H), 1.84 – 1.74 (m, 3H), 1.68 – 1.64 (m, 1H), 1.40 – 1.36 (m, 1H), 1.27 (s, 12H), 1.24 – 1.13 (m, 6H), 1.08 (dd, J = 15.2, 5.7 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 131.5, 128.1, 127.2, 124.5, 93.8, 83.2, 81.9, 43.4, 34.1, 31.4, 29.2, 26.5, 26.4, 25.0, 24.7.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.65.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $C_{22}H_{31}BO_2$ 369.2596; Found: 369.2591.

2-(3-ethyl-2-(phenylethynyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3j)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 75% (48.9 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

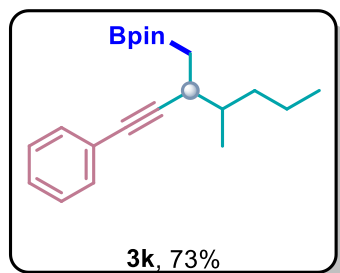
1H NMR (500 MHz, Chloroform- d) δ 7.41 – 7.37 (m, 2H), 7.27 (m, 3H), 2.97 (m, 1H), 1.59 (m, 1H), 1.55 – 1.46 (m, 2H), 1.44 – 1.38 (m, 1H), 1.28 (s, 13H), 1.20 (dd, J = 15.1, 10.2 Hz, 1H), 1.06 (dd, J = 15.2, 5.9 Hz, 1H), 0.96 (m, 6H).

^{13}C NMR (126 MHz, Chloroform- d) δ 131.5, 128.0, 127.2, 124.5, 93.9, 83.2, 81.5, 46.8, 30.4, 25.0, 24.8, 24.7, 23.9, 22.8, 12.0, 11.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.89.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $C_{21}H_{31}BO_2$ 327.2490; Found: 327.2495.

4,4,5,5-tetramethyl-2-(3-methyl-2-(phenylethynyl)hexyl)-1,3,2-dioxaborolane (3k)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 73% (47.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

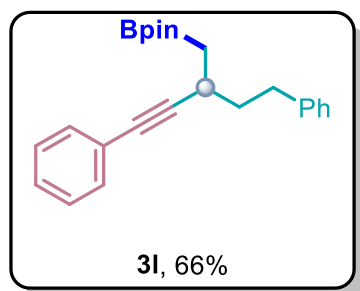
1H NMR (500 MHz, Chloroform- d) δ 7.40 – 7.35 (m, 2H), 7.29 – 7.21 (m, 3H), 2.91 – 2.72 (m, 1H), 1.60 (m, 1H), 1.55 – 1.34 (m, 3H), 1.26 (s, 14H), 1.19 – 1.09 (m, 1H), 1.07 – 0.96 (m, 4H), 0.95 – 0.89 (m, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 131.5, 131.5, 128.0, 127.2, 124.5, 124.5, 94.0, 93.1, 83.2, 82.0, 81.7, 38.1, 38.0, 37.8, 35.6, 33.8, 33.1, 25.0, 25.0, 24.7, 24.7, 20.5, 20.4, 17.3, 15.4, 14.4, 14.3.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.86.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $C_{21}H_{31}BO_2$ 327.2490; Found: 327.2487.

4,4,5,5-tetramethyl-2-(2-phenethyl-4-phenylbut-3-yn-1-yl)-1,3,2-dioxaborolane (3l)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 66% (47.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

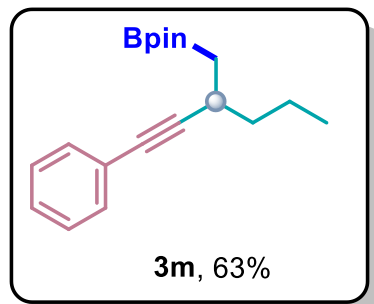
^1H NMR (500 MHz, Chloroform-*d*) δ 7.46 – 7.39 (m, 2H), 7.34 – 7.26 (m, 5H), 7.26 – 7.22 (m, 2H), 7.21 – 7.17 (m, 1H), 2.92 (m, 1H), 2.87 – 2.74 (m, 2H), 1.94 – 1.84 (m, 2H), 1.26 (s, 12H), 1.24 – 1.17 (m, 1H), 1.13 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.3, 131.6, 128.6, 128.4, 128.2, 127.4, 125.8, 124.2, 94.3, 83.3, 39.3, 33.9, 27.7, 24.9, 24.8.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.84.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. For $\text{C}_{24}\text{H}_{29}\text{BO}_2$ 361.2333; Found: 361.2337.

4,4,5,5-tetramethyl-2-(2-(phenylethynyl)pentyl)-1,3,2-dioxaborolane (3m)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 63% (37.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

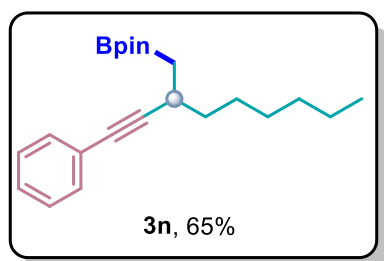
^1H NMR (500 MHz, Chloroform-*d*) δ 7.42 – 7.38 (m, 2H), 7.29 – 7.25 (m, 3H), 2.84 – 2.77 (m, 1H), 1.56 (dd, J = 7.0, 4.4 Hz, 2H), 1.34 (dd, J = 4.9, 2.8 Hz, 2H), 1.28 (d, J = 2.2 Hz, 12H), 1.19 – 1.09 (m, 2H), 0.92 (t, J = 6.8 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.5, 128.1, 128.1, 128.0, 127.3, 124.4, 95.0, 83.2, 37.5, 31.7, 28.0, 27.2, 24.9, 24.8, 24.8, 22.6, 14.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 32.96.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{27}\text{BO}_2$ 299.2177; Found: 299.2173.

4,4,5,5-tetramethyl-2-(2-(phenylethynyl)octyl)-1,3,2-dioxaborolane (3n)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 65% (44.2 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

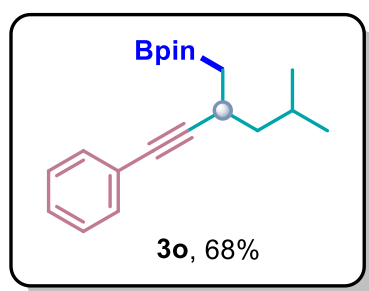
^1H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.35 (m, 2H), 7.28 – 7.23 (m, 3H), 2.82 – 2.73 (m, 1H), 1.55 – 1.51 (m, 2H), 1.48 – 1.40 (m, 1H), 1.34 – 1.28 (m, 6H), 1.25 (d, J = 2.2 Hz, 12H), 1.21 (t, J = 2.9 Hz, 1H), 1.17 – 1.05 (m, 2H), 0.91 – 0.86 (m, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.5, 128.1, 128.1, 128.0, 127.3, 124.4, 95.0, 83.2, 37.6, 31.8, 29.1, 28.0, 27.5, 24.9, 24.8, 22.7, 14.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.73.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $\text{C}_{22}\text{H}_{33}\text{BO}_2$ 371.2752; Found: 371.2752.

4,4,5,5-tetramethyl-2-(4-methyl-2-(phenylethynyl)pentyl)-1,3,2-dioxaborolane (**3o**)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 68% (42.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

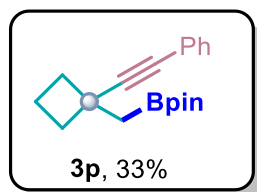
^1H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.35 (m, 2H), 7.28 – 7.21 (m, 3H), 2.84 (m, 1H), 1.89 (m, 1H), 1.54 (m, 1H), 1.26 (s, 13H), 1.16 – 1.04 (m, 2H), 0.94 (dd, J = 6.7, 1.9 Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.5, 128.1, 128.1, 127.3, 124.4, 94.0, 83.2, 5.10, 26.3, 26.1, 24.9, 24.8, 24.8, 23.4, 21.9.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 35.05.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $\text{C}_{20}\text{H}_{29}\text{BO}_2$ 313.2333; Found: 313.2334.

4,4,5,5-tetramethyl-2-((1-(phenylethynyl)cyclobutyl)methyl)-1,3,2-dioxaborolane (**3p**)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 33% (20.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

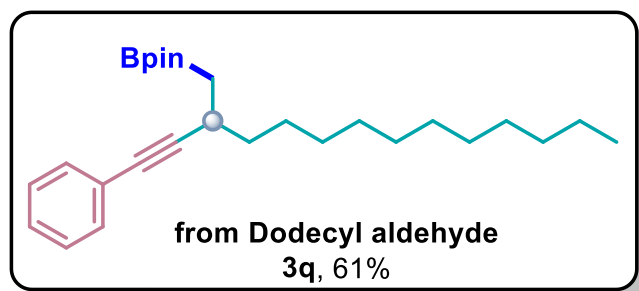
¹H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.37 (m, 2H), 7.28 – 7.23 (m, 3H), 2.45 – 2.39 (m, 2H), 2.20 – 2.09 (m, 2H), 2.04 – 1.94 (m, 2H), 1.36 (d, *J* = 7.6 Hz, 2H), 1.26 (d, *J* = 13.3 Hz, 12H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 131.6, 131.5, 128.0, 127.2, 98.1, 83.0, 80.8, 36.7, 34.3, 31.5, 30.1, 28.9, 24.8, 24.7, 16.2.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.15.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₀H₂₇BO₂ 297.2020; Found: 297.2025.

4,4,5,5-tetramethyl-2-(2-(phenylethynyl)tridecyl)-1,3,2-dioxaborolane (3q)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 61% (50.1 mg), *R*_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

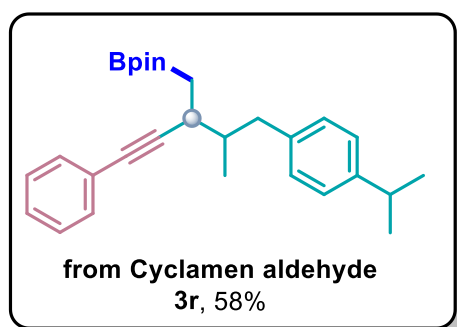
¹H NMR (500 MHz, Chloroform-*d*) δ 7.40 – 7.36 (m, 2H), 7.30 – 7.21 (m, 3H), 2.83 – 2.74 (m, 1H), 1.54 (m, 3H), 1.48 – 1.42 (m, 1H), 1.26 (s, 30H), 1.12 (m, 2H), 0.88 (t, *J* = 6.9 Hz, 3H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 131.5, 128.1, 128.1, 127.3, 124.4, 95.0, 83.2, 37.6, 32.0, 29.7, 29.7, 29.7, 29.6, 29.5, 29.4, 28.0, 27.5, 24.9, 24.8, 24.8, 22.7, 14.2.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.20.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₇H₄₃BO₂ 411.3429; Found: 411.3427.

2-(2-(1-(4-isopropylphenyl)propan-2-yl)-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3r)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 58% (48.3 mg), *R*_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

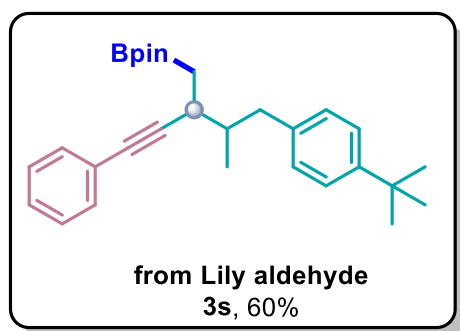
¹H NMR (500 MHz, Chloroform-*d*) δ 7.42 (m, 2H), 7.32 – 7.26 (m, 3H), 7.17 – 7.10 (m, 4H), 3.02 (dd, *J* = 13.4, 3.9 Hz, 0.5H), 2.92 – 2.80 (m, 2.5H), 2.52 (dd, *J* = 13.5, 8.3 Hz, 0.5H), 2.36 (dd, *J* = 13.4, 10.2 Hz, 0.5H), 1.88 (m, 1H), 1.28 (s, 6H), 1.25 (m, 13H), 1.19 – 1.13 (m, 1H), 1.04 (dd, *J* = 15.2, 6.3 Hz, 1H), 0.97 (dd, *J* = 17.3, 6.7 Hz, 3H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 146.2, 146.2, 138.8, 138.6, 131.6, 131.6, 129.2, 129.1, 128.1, 127.4, 126.2, 126.2, 124.4, 124.3, 93.6, 92.6, 83.3, 83.3, 82.5, 82.2, 41.7, 40.9, 40.5, 39.2, 33.9, 33.7, 33.7, 32.7, 25.0, 24.9, 24.7, 24.7, 24.1, 24.1, 17.2, 15.0.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.91.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₈H₃₇BO₂ 417.2959; Found: 417.2959.

2-(2-(1-(4-isopropylphenyl)propan-2-yl)-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3s)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 60% (50.0 mg), *R_f* = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

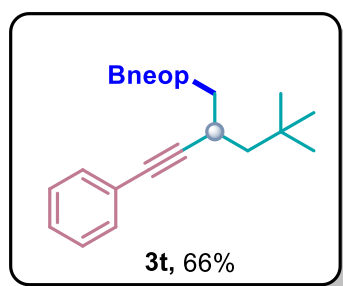
¹H NMR (500 MHz, Chloroform-*d*) δ 7.43 (m, 2H), 7.30 (m, 5H), 7.14 (t, *J* = 7.9 Hz, 2H), 3.02 (dd, *J* = 13.4, 3.9 Hz, 0.5H), 2.95 – 2.82 (m, 1.5H), .53 (dd, *J* = 13.5, 8.2 Hz, 0.5H), 2.37 (dd, *J* = 13.4, 10.1 Hz, 0.5H), 1.91 (m, 1H), 1.33 – 1.25 (m, 21H), 1.17 (dd, *J* = 15.2, 5.5 Hz, 1H), 1.05 (dd, *J* = 15.3, 6.4 Hz, 1H), 0.98 (dd, *J* = 17.5, 6.6 Hz, 3H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 148.5, 148.4, 138.4, 138.2, 131.6, 131.6, 128.9, 128.9, 128.2, 127.4, 125.1, 125.1, 124.9, 124.4, 124.3, 93.6, 92.6, 83.3, 83.3, 82.5, 82.2, 41.6, 40.9, 40.5, 39.3, 34.4, 33.9, 32.7, 31.5, 25.1, 25.0, 24.9, 24.8, 24.7, 17.3, 15.0.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.36.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₈H₃₇BO₂ 431.3116; Found: 431.3115.

2-(4,4-dimethyl-2-(phenylethynyl)pentyl)-5,5-dimethyl-1,3,2-dioxaborinane (3t)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 66% (41.2 mg), *R_f* = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

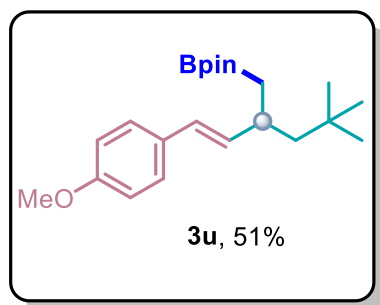
¹H NMR (500 MHz, Chloroform-*d*) δ 7.38 – 7.34 (m, 2H), 7.28 – 7.22 (m, 3H), 3.63 (s, 4H), 2.87 – 2.78 (m, 1H), 1.65 (dd, *J* = 13.6, 10.2 Hz, 1H), 1.37 (dd, *J* = 13.7, 2.8 Hz, 1H), 1.13 (dd, *J* = 15.2, 8.3 Hz, 1H), 1.03 (s, 9H), 0.98 (s, 6H), 0.96 – 0.87 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.3, 128.1, 127.1, 124.7, 97.3, 80.6, 72.1, 51.9, 31.7, 31.2, 30.0, 24.0, 21.9.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.51.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{28}\text{H}_{37}\text{BO}_2$ 313.2333; Found: 313.2329.

(E)-2-(2-(4-methoxystyryl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3u)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 51% (36.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

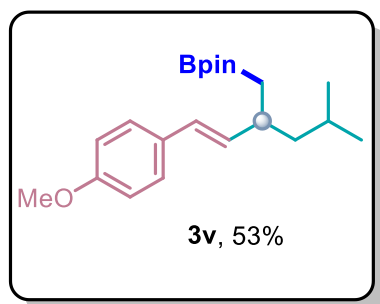
^1H NMR (500 MHz, Chloroform-*d*) δ 7.33 (d, J = 8.6 Hz, 2H), 6.89 – 6.83 (m, 2H), 6.14 (d, J = 11.7 Hz, 1H), 5.45 (dd, J = 11.8, 10.6 Hz, 1H), 3.81 (s, 3H), 3.11 (dt, J = 11.1, 6.3 Hz, 1H), 1.31 – 1.29 (m, 2H), 1.22 (d, J = 4.3 Hz, 12H), 1.01 – 0.96 (m, 1H), 0.92 (s, 1H), 0.76 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 157.9, 140.3, 130.8, 129.9, 125.2, 113.5, 83.1, 55.2, 52.8, 31.2, 30.4, 30.8, 25.1, 24.7.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.89.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{35}\text{BO}_3$ 359.2752; Found: 359.2750.

(E)-2-(2-(4-methoxystyryl)-4-methylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3v)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 53% (36.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

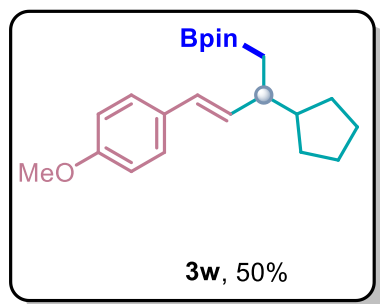
^1H NMR (500 MHz, Chloroform-*d*) δ 7.26 (dd, J = 6.1, 2.6 Hz, 2H), 6.85 – 6.79 (m, 2H), 6.30 (d, J = 15.8 Hz, 1H), 5.88 (dd, J = 15.8, 8.9 Hz, 1H), 3.79 (s, 3H), 2.50 (m, 1H), 1.58 (m, 1H), 1.34 – 1.27 (m, 2H), 1.20 (d, J = 3.3 Hz, 13H), 0.95 (dd, J = 15.1, 6.1 Hz, 1H), 0.86 (dd, J = 6.6, 4.9 Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 134.70, 130.8, 127.7, 127.1, 113.8, 83.0, 55.3, 47.6, 37.1, 25.7, 25.0, 24.8, 23.6, 22.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.91.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{33}\text{BO}_3$ 345.2596; Found: 345.2590.

(E)-2-(2-cyclopentyl-4-(4-methoxyphenyl)but-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(3w)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 50% (35.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

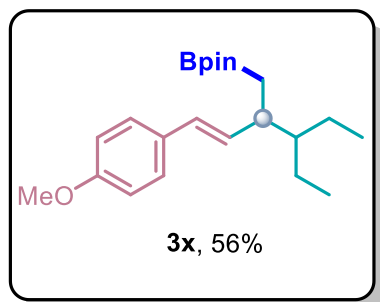
^1H NMR (500 MHz, Chloroform- d) δ 7.25 (d, J = 8.3 Hz, 2H), 6.85 – 6.78 (m, 2H), 6.29 (d, J = 15.8 Hz, 1H), 5.96 (dd, J = 15.8, 8.9 Hz, 1H), 3.79 (s, 3H), 2.31 – 2.21 (m, 1H), 1.75 (m 2H), 1.68 – 1.62 (m, 1H), 1.59 – 1.46 (m, 4H), 1.17 (s, 14H), 1.08 (dd, J = 15.1, 4.6 Hz, 1H), 0.88 (dd, J = 15.1, 10.3 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 158.5 133.6 130.9, 128.1 127.1 113.8, 830 55.3 47.1, 44.5 30.8 30.49, 25.5 25.4, 25.1 24.8, 24.7.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.16.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{33}\text{BO}_3$ 357.2596; Found: 357.2595.

(E)-2-(3-ethyl-2-(4-methoxystyryl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3x)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 56% (40.1 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

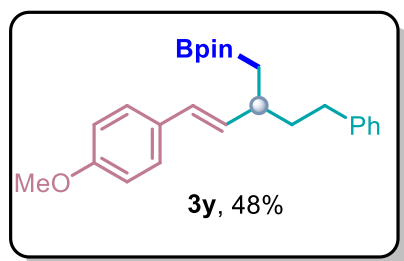
^1H NMR (500 MHz, Chloroform- d) δ 7.26 – 7.24 (m, 2H), 6.86 – 6.78 (m, 2H), 6.28 (d, J = 15.8 Hz, 1H), 5.95 (dd, J = 15.8, 8.8 Hz, 1H), 3.79 (s, 3H), 2.53 (tt, J = 9.7, 5.0 Hz, 1H), 1.45 (dddd, J = 11.4, 9.5, 7.5, 4.1 Hz, 2H), 1.17 (s, 12H), 0.90 – 0.80 (m, 10H).

^{13}C NMR (126 MHz, Chloroform- d) δ 158.48, 132.79, 130.95, 128.51, 127.05, 113.81, 82.93, 55.28, 47.35, 40.75, 25.04, 24.90, 24.83, 24.80, 24.77, 23.00, 22.55, 12.14, 12.06, 11.75, 10.94.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.96.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{35}\text{O}_3$ 359.2752; Found: 359.2756.

(E)-2-(4-(4-methoxyphenyl)-2-phenethylbut-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3y)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 48% (37.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

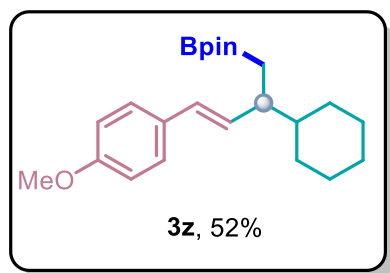
^1H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.25 (m, 4H), 7.20 – 7.16 (m, 3H), 6.89 – 6.81 (m, 2H), 6.35 (d, J = 15.7 Hz, 1H), 5.97 (dd, J = 15.8, 8.7 Hz, 1H), 3.81 (s, 3H), 2.70 – 2.62 (m, 1H), 2.62 – 2.54 (m, 1H), 2.49 (m, 1H), 1.85 – 1.75 (m, 1H), 1.69 (m, 1H), 1.20 (d, J = 1.9 Hz, 12H), 1.04 (dd, J = 15.2, 6.1 Hz, 1H), 0.96 (dd, J = 15.2, 8.6 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.6, 142.9, 134.0, 130.7, 128.5, 128.4, 128.4, 128.3, 128.2, 127.1, 125.6, 113.9, 83.1, 55.3, 39.7, 39.0, 33.9, 25.0, 24.9, 24.8.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.41.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{25}\text{H}_{33}\text{BO}_3$ 393.2596; Found: 393.2597.

(E)-2-(2-cyclohexyl-4-(4-methoxyphenyl)but-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3z)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 52% (38.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

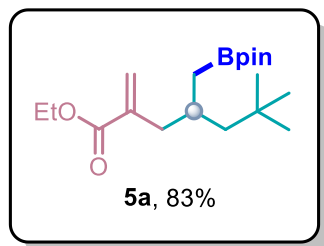
^1H NMR (500 MHz, Chloroform-*d*) δ 7.26 (dd, J = 6.3, 2.5 Hz, 3H), 6.83 – 6.79 (m, 2H), 6.26 (d, J = 15.7 Hz, 1H), 5.94 (dd, J = 15.8, 9.0 Hz, 1H), 3.79 (s, 3H), 2.24 (m, 1H), 1.79 – 1.66 (m, 5H), 1.17 (s, 13H), 1.13 – 1.00 (m, 3H), 1.00 – 0.84 (m, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.5, 133.0, 130.9, 128.6, 127.1, 113.8, 83.0, 55.3, 44.9, 44.1, 33.0, 30.9, 30.1, 26.7, 26.7, 25.0, 24.8, 24.7.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.41.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{23}\text{H}_{35}\text{BO}_3$ 371.2752; Found: 371.2750.

ethyl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5a)



Following the **general procedure 5** on 0.2 mmol scale, colorless oil, yield: 83% (56.1mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

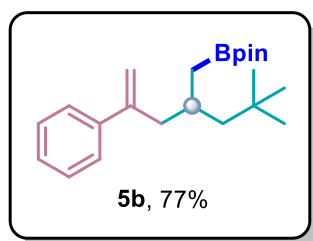
^1H NMR (500 MHz, Chloroform- d) δ 6.15 (d, J = 1.9 Hz, 1H), 5.49 (m, 1H), 4.18 (m, 2H), 2.47 (m, 1H), 2.07 (m, 1H), 1.90 (m, 1H), 1.28 (t, J = 7.2 Hz, 3H), 1.21 (d, J = 2.3 Hz, 12H), 1.18 (dd, J = 9.0, 5.1 Hz, 2H), 0.89 (s, 9H), 0.84 – 0.69 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 167.4, 140.1, 126.4, 82.7, 60.4, 50.7, 41.4, 31.1, 30.1, 29.3, 24.9, 24.7, 14.2.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.78.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{35}\text{BO}_4$ 339.2701; Found: 339.2701.

4,4,5,5-tetramethyl-2-(2-neopentyl-4-phenylpent-4-en-1-yl)-1,3,2-dioxaborolane (**5b**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 77% (52.7mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

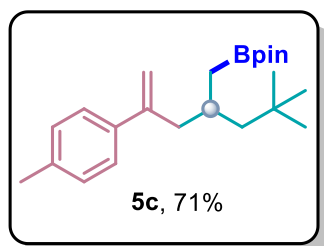
^1H NMR (500 MHz, Chloroform- d) δ 7.48 – 7.44 (m, 2H), 7.31 (dd, J = 8.3, 6.7 Hz, 2H), 7.25 (d, J = 6.8 Hz, 1H), 5.30 (d, J = 1.9 Hz, 1H), 5.05 (d, J = 1.7 Hz, 1H), 2.61 (m, 1H), 2.41 (dd, J = 13.9, 7.7 Hz, 1H), 1.73 (m, 1H), 1.36 – 1.33 (m, 1H), 1.26 (s, 12H), 1.11 (dd, J = 14.0, 6.4 Hz, 1H), 0.92 (dd, J = 15.9, 4.9 Hz, 1H), 0.81 (d, J = 7.6 Hz, 1H), 0.78 (s, 9H).

^{13}C NMR (126 MHz, Chloroform- d) δ 148.0, 141.2, 128.0, 127.1, 126.5, 114.1, 82.7, 49.9, 44.5, 30.9, 30.1, 28.8, 24.9, 24.9.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.01.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{35}\text{BO}_2$ 343.2803; Found: 343.2810.

4,4,5,5-tetramethyl-2-(2-neopentyl-4-(p-tolyl)pent-4-en-1-yl)-1,3,2-dioxaborolane (**5c**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 71% (50.7mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

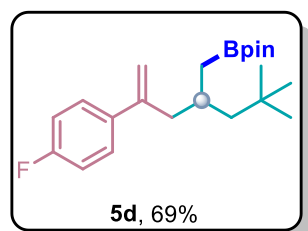
^1H NMR (500 MHz, Chloroform- d) δ 7.38 – 7.33 (m, 2H), 7.11 (d, J = 7.9 Hz, 2H), 5.27 (d, J = 2.0 Hz, 1H), 4.99 (d, J = 1.7 Hz, 1H), 2.58 (m, 1H), 2.41 – 2.33 (m, 4H), 1.73 (m, 1H), 1.34 (dd, J = 13.9, 3.7 Hz, 1H), 1.26 (s, 13H), 1.10 (dd, J = 14.0, 6.4 Hz, 1H), 0.92 (dd, J = 15.9, 4.9 Hz, 1H), 0.79 (s, 9H).

^{13}C NMR (126 MHz, Chloroform- d) δ 147.8, 138.3, 136.7, 128.7, 126.4, 113.3, 82.7, 49.9, 44.5, 31.0, 30.2, 28.8, 24.9, 24.9, 21.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.01.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{23}\text{H}_{37}\text{BO}_2$ 357.2959; Found: 357.2960.

2-(4-(4-fluorophenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5d)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 69% (49.7 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform- d) δ 7.42 (m, 2H), 6.98 (t, J = 8.7 Hz, 2H), 5.23 (d, J = 1.7 Hz, 1H), 5.01 (d, J = 1.6 Hz, 1H), 2.58 (m, 1H), 2.33 (dd, J = 13.9, 7.9 Hz, 1H), 1.71 – 1.64 (m, 1H), 1.32 (dd, J = 14.2, 3.7 Hz, 2H), 1.25 (s, 12H), 1.08 (dd, J = 14.0, 6.5 Hz, 1H), 0.91 (dd, J = 16.1, 4.8 Hz, 1H), 0.76 (s, 9H).

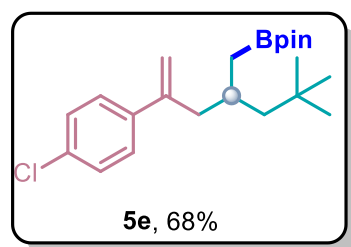
^{13}C NMR (126 MHz, Chloroform- d) δ 163.1, 161.2, 147.0, 137.2, 137.2, 128.1, 128.1, 114.9, 114.7, 114.0, 82.8, 82.8, 49.8, 47.4, 44.6, 30.9, 30.1, 29.4, 28.6, 24.9, 24.9, 24.8, 19.0.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.04.

^{19}F NMR (471 MHz, Chloroform- d) δ -115.84.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{34}\text{BFO}_2$ 361.2709; Found: 361.2707.

2-(4-(4-chlorophenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5e)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 68% (51.1 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

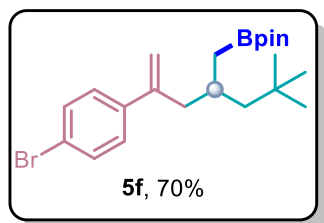
^1H NMR (500 MHz, Chloroform- d) δ 7.40 (d, J = 8.6 Hz, 2H), 7.28 – 7.25 (m, 2H), 5.28 (d, J = 1.7 Hz, 1H), 5.04 (d, J = 1.5 Hz, 1H), 2.58 (m, 1H), 2.32 (dd, J = 13.9, 7.9 Hz, 1H), 1.71 – 1.64 (m, 1H), 1.31 (dd, J = 14.0, 3.5 Hz, 2H), 1.25 (s, 12H), 1.08 (dd, J = 14.0, 6.5 Hz, 1H), 0.91 (dd, J = 16.1, 4.8 Hz, 2H), 0.76 (s, 9H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 146.8, 139.6, 132.8, 128.2, 127.9, 114.6, 82.8, 49.8, 44.4, 30.9, 30.1, 29.4, 28.7, 24.9, 24.8.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.00.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₂H₃₄BClO₂ 377.2413; Found: 377.2413.

2-(4-(4-bromophenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5f)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 70% (58.8 mg), *R_f* = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

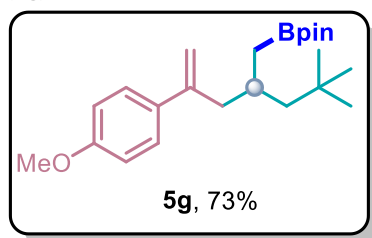
¹H NMR (500 MHz, Chloroform-*d*) δ 7.45 – 7.40 (m, 2H), 7.36 – 7.31 (m, 2H), 5.28 (d, *J* = 1.7 Hz, 1H), 5.05 (d, *J* = 1.6 Hz, 1H), 2.58 (m, 1H), 2.38 – 2.29 (m, 1H), 1.70 – 1.65 (m, 1H), 1.31 (dd, *J* = 14.0, 3.5 Hz, 2H), 1.25 (s, 12H), 1.08 (dd, *J* = 14.0, 6.5 Hz, 1H), 0.91 (dd, *J* = 16.0, 4.7 Hz, 1H), 0.76 (s, 9H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 146.9, 140.0, 131.1, 128.2, 121.0, 114.7, 82.8, 49.8, 44.3, 30.9, 30.1, 28.7, 24.9, 24.8.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.12.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₂H₃₄BBrO₂ 421.1908; Found: 421.1911.

2-(4-(4-methoxyphenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5g)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 73% (54.3 mg), *R_f* = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

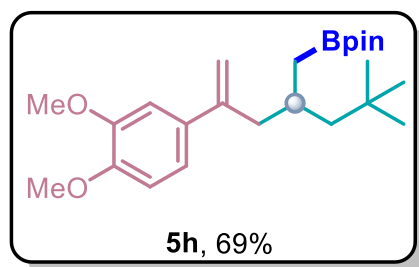
¹H NMR (500 MHz, Chloroform-*d*) δ 7.42 – 7.37 (m, 2H), 6.87 – 6.79 (m, 2H), 5.22 (d, *J* = 1.9 Hz, 1H), 4.95 (d, *J* = 1.9 Hz, 1H), 3.81 (s, 3H), 2.57 (dd, *J* = 13.8, 6.6 Hz, 1H), 2.35 (dd, *J* = 13.8, 7.8 Hz, 1H), 1.71 (m, 1H), 1.37 – 1.32 (m, 1H), 1.25 (d, *J* = 8.3 Hz, 12H), 1.09 (dd, *J* = 14.0, 6.4 Hz, 1H), 0.92 (dd, *J* = 15.9, 4.8 Hz, 1H), 0.77 (s, 9H), 0.66 (t, *J* = 7.4 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 158.8, 147.2, 133.6, 127.6, 127.6, 113.4, 112.6, 82.7, 55.2, 49.8, 44.6, 30.9, 30.1, 28.7, 24.9, 24.9, 24.9, 24.8.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.70.

HRMS (ESI) *m/z*: [M+Na]⁺ Calcd. for C₂₃H₃₇BO₃ 395.2728; Found: 395.2726.

2-(4-(3,4-dimethoxyphenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5h)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 69% (55.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

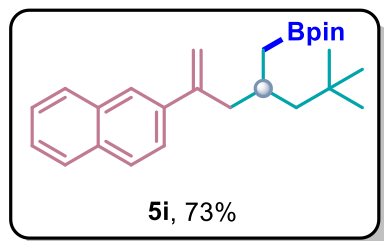
^1H NMR (500 MHz, Chloroform-*d*) δ 7.01 (dd, J = 8.3, 2.1 Hz, 1H), 6.96 (d, J = 2.0 Hz, 1H), 6.81 (d, J = 8.3 Hz, 1H), 5.21 (d, J = 1.9 Hz, 1H), 4.97 (d, J = 1.8 Hz, 1H), 3.89 (d, J = 3.5 Hz, 6H), 2.53 (dd, J = 13.9, 7.3 Hz, 1H), 2.42 (td, J = 15.2, 13.9, 7.5 Hz, 1H), 1.78 – 1.69 (m, 1H), 1.35 – 1.28 (m, 2H), 1.24 (s, 12H), 1.10 (dd, J = 14.1, 6.1 Hz, 1H), 0.90 (dd, J = 15.9, 5.3 Hz, 1H), 0.78 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 148.5, 148.4, 147.6, 134.2, 118.9, 112.9, 110.7, 110.0, 82.7, 55.9, 55.8, 49.9, 44.6, 31.0, 30.2, 28.6, 24.9, 24.9.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.82.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{39}\text{BO}_4$ 403.3014; Found: 403.3010.

4,4,5,5-tetramethyl-2-(4-(naphthalen-2-yl)-2-neopentylpent-4-en-1-yl)-1,3,2-dioxaborolane (5i)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 73% (57.2 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

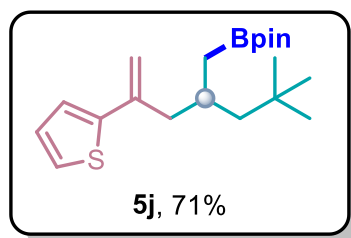
^1H NMR (500 MHz, Chloroform-*d*) δ 7.93 (d, J = 1.8 Hz, 1H), 7.87 – 7.77 (m, 3H), 7.64 (d, J = 1.8 Hz, 1H), 7.46 (pd, J = 6.9, 1.6 Hz, 2H), 5.46 (d, J = 1.8 Hz, 1H), 5.17 (d, J = 1.7 Hz, 1H), 2.79 – 2.71 (m, 1H), 2.54 (dd, J = 13.9, 7.6 Hz, 1H), 1.82 (qt, J = 7.0, 3.0 Hz, 1H), 1.41 (dd, J = 14.0, 3.7 Hz, 1H), 1.29 (d, J = 1.7 Hz, 12H), 1.15 (dd, J = 14.0, 6.3 Hz, 1H), 0.98 (dd, J = 16.0, 5.0 Hz, 1H), 0.84 (dd, J = 16.0, 7.5 Hz, 2H), 0.79 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 147.81, 138.49, 133.41, 132.73, 128.13, 127.51, 127.47, 125.84, 125.55, 125.15, 125.12, 114.79, 82.76, 49.92, 44.53, 30.96, 30.15, 28.79, 24.96, 24.90, 24.80.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.24.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{26}\text{H}_{37}\text{BO}_2$ 393.2959; Found: 393.2955.

4,4,5,5-tetramethyl-2-(2-neopentyl-4-(thiophen-2-yl)pent-4-en-1-yl)-1,3,2-dioxaborolane (5j)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 71% (49.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

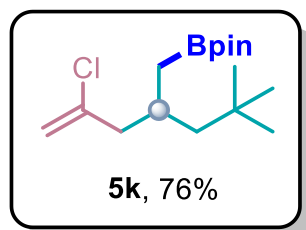
^1H NMR (500 MHz, Chloroform-*d*) δ 7.19 (dd, J = 3.7, 1.1 Hz, 1H), 7.13 (dd, J = 5.1, 1.1 Hz, 1H), 6.96 (dd, J = 5.1, 3.6 Hz, 1H), 5.41 (d, J = 1.2 Hz, 1H), 4.93 (d, J = 1.3 Hz, 1H), 2.52 (m, 1H), 2.37 – 2.30 (m, 1H), 1.95 (m, 1H), 1.37 (dd, J = 13.9, 3.7 Hz, 1H), 1.26 (d, J = 1.8 Hz, 12H), 1.13 (dd, J = 14.0, 6.3 Hz, 1H), 0.98 – 0.93 (m, 1H), 0.83 (s, 10H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 145.6, 141.1, 127.2, 123.9, 123.9, 112.8, 82.9, 50.2, 45.0, 31.0, 30.1, 29.2, 25.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.22.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{20}\text{H}_{33}\text{BO}_2\text{S}$ 349.2367; Found: 349.2368.

2-(4-chloro-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5k)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 76% (45.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

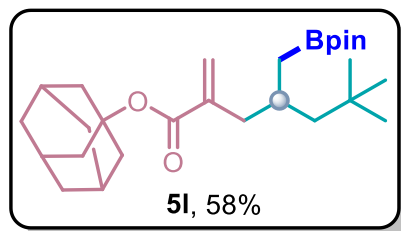
^1H NMR (500 MHz, Chloroform-*d*) δ 5.14 (d, J = 1.0 Hz, 1H), 5.11 – 5.09 (m, 1H), 2.34 (m, 1H), 2.27 (dd, J = 14.0, 8.0 Hz, 1H), 2.04 (m, 1H), 1.23 (d, J = 1.4 Hz, 12H), 1.21 – 1.14 (m, 2H), 0.91 (d, J = 1.5 Hz, 9H), 0.88 – 0.85 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.6, 113.3, 82.8, 49.7, 48.0, 31.1, 30.1, 29.4, 28.4, 24.9, 24.8, 24.8.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.81.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{16}\text{H}_{30}\text{BClO}_2$ 301.2100; Found: 301.2107.

adamantan-1-yl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5l)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 58% (51.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

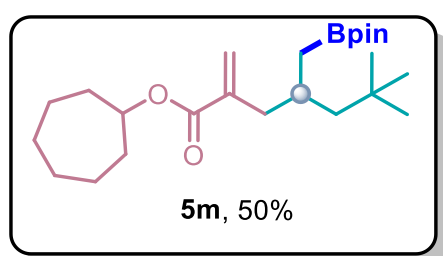
^1H NMR (500 MHz, Chloroform- d) δ 6.20 (t, J = 1.8 Hz, 1H), 5.51 (t, J = 1.7 Hz, 1H), 4.98 (d, J = 3.2 Hz, 1H), 2.47 (m, 1H), 2.16 (dd, J = 13.5, 8.4 Hz, 1H), 2.04 (d, J = 10.6 Hz, 4H), 1.95 (m, 1H), 1.84 (m, 4H), 1.78 (d, J = 11.5 Hz, 2H), 1.73 (s, 2H), 1.59 – 1.53 (m, 2H), 1.22 (s, 12H), 0.89 (s, 9H), 0.86 – 0.79 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 166.7, 140.7, 126.0, 82.7, 50.4, 41.3, 37.4, 36.3, 36.3, 31.9, 31.9, 31.8, 31.1, 30.2, 29.5, 27.3, 27.0, 24.9, 24.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.91.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{27}\text{H}_{45}\text{BO}_4$ 445.3484; Found: 445.3479.

cycloheptyl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5m)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 50% (40.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

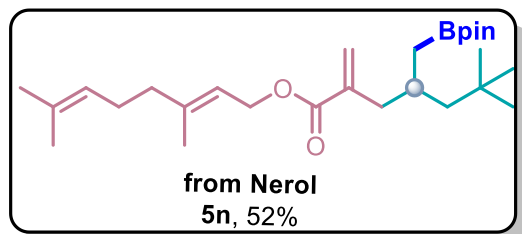
^1H NMR (500 MHz, Chloroform- d) δ 6.13 (d, J = 2.0 Hz, 1H), 5.46 (d, J = 1.9 Hz, 1H), 4.99 (m, 1H), 2.46 (m, 1H), 2.09 (dd, J = 13.4, 8.8 Hz, 1H), 1.98 – 1.86 (m, 3H), 1.73 – 1.63 (m, 4H), 1.59 – 1.53 (m, 4H), 1.50 – 1.40 (m, 2H), 1.22 (d, J = 2.1 Hz, 12H), 1.20 – 1.14 (m, 2H), 0.90 (s, 9H), 0.78 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 166.8, 140.7, 126.2, 82.8, 50.7, 41.5, 33.8, 33.8, 31.2, 30.2, 29.4, 28.3, 28.3, 25.0, 24.8, 23.0, 23.0.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.01.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{43}\text{BO}_4$ 407.3327; Found: 407.3319.

(E)-3,7-dimethylocta-2,6-dien-1-yl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5n)

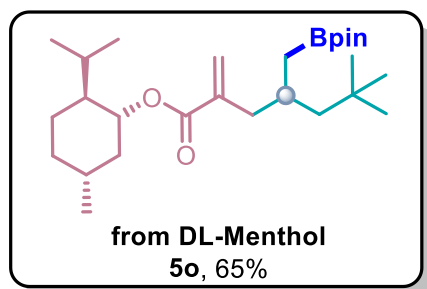


Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 52% (46.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform- d) δ 6.14 (d, J = 1.9 Hz, 1H), 5.50 (d, J = 1.8 Hz, 1H), 5.07 (m, 1H), 4.22 – 4.08 (m, 2H), 2.48 (m, 1H), 2.08 (dd, J = 13.5, 8.9 Hz, 1H), 2.02 – 1.85 (m, 3H), 1.75

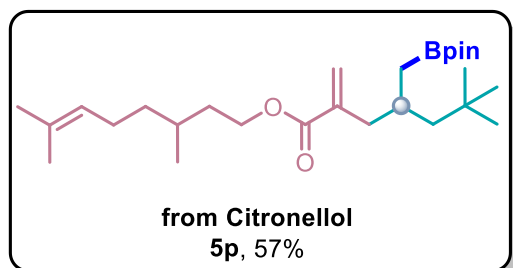
– 1.66 (m, 4H), 1.61 – 1.53 (m, 4H), 1.51 – 1.45 (m, 1H), 1.38 – 1.32 (m, 1H), 1.24 – 1.16 (m, 15H), 0.91 (d, $J = 10.6$ Hz, 12H), 0.84 – 0.79 (m, 1H), 0.74 (m, 1H).
 ^{13}C NMR (126 MHz, Chloroform- d) δ 167.5, 140.2, 131.3, 126.4, 124.6, 82.8, 63.1, 50.7, 50.7, 41.4, 37.0, 35.5, 31.2, 30.2, 29.5, 29.4, 25.7, 25.4, 25.0, 24.8, 19.4, 19.4, 17.7.
 ^{11}B NMR (160 MHz, Chloroform- d) δ 34.10.
 HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{27}\text{H}_{47}\text{BO}_4$ 447.3640; Found: 447.3637.

(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5o**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 65% (58.4 mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).
 ^1H NMR (500 MHz, Chloroform- d) δ 6.13 (dd, $J = 9.8, 2.1$ Hz, 1H), 5.47 (dd, $J = 6.9, 1.9$ Hz, 1H), 4.74 (m, 1H), 2.47 (m, 1H), 2.13 – 1.97 (m, 2H), 1.90 (m, 2H), 1.67 (m, 2H), 1.55 – 1.38 (m, 2H), 1.21 (d, $J = 2.1$ Hz, 15H), 1.10 – 0.97 (m, 2H), 0.91 – 0.86 (m, 15H), 0.84 – 0.78 (m, 1H), 0.77 – 0.70 (m, 4H).
 ^{13}C NMR (126 MHz, Chloroform- d) δ 167.0, 140.6, 140.5, 126.4, 126.14 82.7, 74.3, 50.6, 50.8, 47.1, 47.1, 41.5, 41.4, 40.9, 40.8, 34.4, 34.3, 31.42 31.4, 31.2, 31.1, 30.9, 30.2, 29.5, 26.3, 26.1, 25.0, 24.9, 24.9, 24.8, 23.5, 23.3, 22.1, 20.9, 20.8, 16.4, 16.2.
 ^{11}B NMR (160 MHz, Chloroform- d) δ 34.12.
 HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{27}\text{H}_{49}\text{BO}_4$ 449.3797; Found: 449.3792.

3,7-dimethyloct-6-en-1-yl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5p**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 75% (48.9mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).
 ^1H NMR (500 MHz, Chloroform- d) δ 6.15 (d, $J = 2.0$ Hz, 1H), 5.49 (d, $J = 1.8$ Hz, 1H), 5.38 (td, $J = 7.2, 1.6$ Hz, 1H), 5.12 – 5.05 (m, 1H), 4.62 (qdd, $J = 12.6, 7.3, 1.1$ Hz, 2H), 2.49 (ddd, $J = 13.6, 5.3, 1.2$ Hz, 1H), 2.14 – 2.03 (m, 5H), 1.94 – 1.87 (m, 1H), 1.75 (d, $J = 1.4$ Hz, 3H), 1.67 (d,

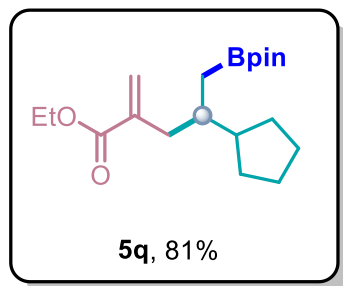
$J = 1.5$ Hz, 3H), 1.59 (d, $J = 1.3$ Hz, 3H), 1.22 (d, $J = 2.3$ Hz, 12H), 1.20 – 1.14 (m, 2H), 0.89 (s, 9H), 0.81 (dd, $J = 15.7, 6.2$ Hz, 1H), 0.73 (dd, $J = 15.7, 6.3$ Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 167.43, 142.08, 140.14, 132.09, 126.49, 123.65, 119.45, 82.77, 61.19, 50.71, 41.50, 32.19, 31.18, 30.11, 29.33, 26.66, 25.71, 24.97, 24.79, 23.49, 17.68.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.90.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{27}\text{H}_{49}\text{BO}_4$ 449.3797; Found: 449.3795.

ethyl 4-cyclopentyl-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (5q)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 81% (54.5 mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

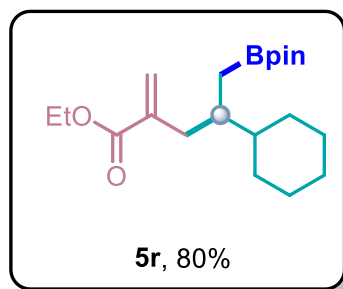
^1H NMR (500 MHz, Chloroform- d) δ 6.12 (d, $J = 1.9$ Hz, 1H), 5.51 (d, $J = 1.7$ Hz, 1H), 4.21 – 4.11 (m, 2H), 2.54 (ddd, $J = 13.7, 4.0, 1.3$ Hz, 1H), 2.03 (dd, $J = 13.6, 9.0$ Hz, 1H), 1.79 – 1.64 (m, 4H), 1.59 – 1.51 (m, 2H), 1.46 (dtd, $J = 15.3, 7.7, 7.2, 4.0$ Hz, 2H), 1.27 (t, $J = 7.1$ Hz, 3H), 1.19 (s, 14H), 0.75 – 0.68 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 167.43, 140.08, 126.03, 82.75, 60.42, 46.09, 38.13, 38.12, 30.44, 30.18, 25.54, 25.49, 24.90, 24.84, 24.71, 14.19.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.08.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{33}\text{BO}_4$ 337.2545; Found: 337.2543.

ethyl 4-cyclohexyl-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (5r)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 80% (56.0 mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

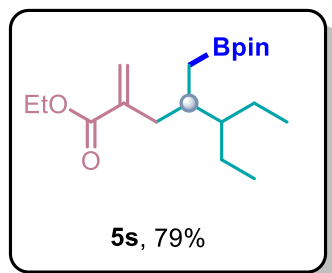
^1H NMR (500 MHz, Chloroform- d) δ 6.14 (d, $J = 1.8$ Hz, 1H), 5.49 (q, $J = 1.3$ Hz, 1H), 4.23 – 4.12 (m, 2H), 2.50 (ddd, $J = 13.6, 5.2, 1.3$ Hz, 1H), 1.99 (dd, $J = 13.6, 9.0$ Hz, 1H), 1.75 – 1.69 (m, 3H), 1.62 (tt, $J = 7.9, 4.2$ Hz, 3H), 1.28 (t, $J = 7.1$ Hz, 3H), 1.21 (s, 15H), 1.12 – 0.98 (m, 3H), 0.70 (dd, $J = 7.0, 3.5$ Hz, 2H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 167.41, 140.31, 125.92, 82.77, 60.42, 42.54, 38.39, 36.20, 29.73, 29.38, 26.84, 26.79, 24.85, 24.68, 14.17.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.48.

HRMS (ESI) m/z: [M+H]⁺ Calcd. for C₂₀H₃₅BO₄ 351.2701; Found: 351.2702.

ethyl 5-ethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5s)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 79% (53.4 mg), *R*_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

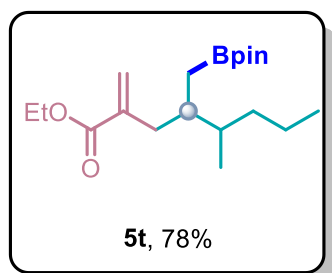
¹H NMR (500 MHz, Chloroform-*d*) δ 6.12 (d, *J* = 1.8 Hz, 1H), 5.46 (d, *J* = 1.8 Hz, 1H), 4.16 (m, 2H), 2.46 – 2.38 (m, 1H), 2.03 – 1.93 (m, 2H), 1.28 (m, 5H), 1.22 – 1.11 (m, 15H), 1.03 (m, 1H), 0.85 (m, 6H), 0.65 (d, *J* = 6.6 Hz, 2H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 167.4, 140.3, 140.3, 126.0, 82.8, 60.4, 46.1, 35.8, 34.1, 34.1, 25.0, 24.7, 22.8, 22.5, 14.2, 12.6, 12.5.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.05.

HRMS (ESI) m/z: [M+H]⁺ Calcd. for C₁₉H₃₅BO₄ 339.2701; Found: 339.2768.

ethyl 5-methyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)octanoate (5t)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 78% (52.7 mg), *R*_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

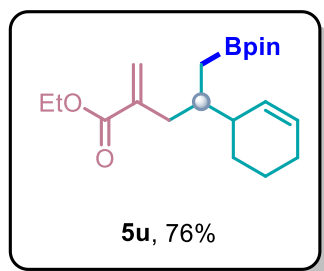
¹H NMR (500 MHz, Chloroform-*d*) δ 6.14 (dd, *J* = 4.3, 1.8 Hz, 1H), 5.49 (dd, *J* = 3.6, 2.1 Hz, 1H), 4.17 (m, 2H), 2.38 (m, 1H), 2.08 (m, 1H), 1.91 – 1.80 (m, 1H), 1.46 (m, 1H), 1.38 – 1.32 (m, 1H), 1.28 (t, *J* = 7.1 Hz, 4H), 1.20 (d, *J* = 5.5 Hz, 13H), 1.15 – 1.01 (m, 1H), 0.86 (m, 3H), 0.79 (t, *J* = 6.5 Hz, 3H), 0.73 – 0.66 (m, 1H), 0.59 (dd, *J* = 15.6, 8.1 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 167.4, 167.4, 140.2, 126.1, 125.9, 82.8, 82.8, 60.5, 60.4, 37.4, 37.4, 36.9, 36.8, 36.3, 36.2, 36.0, 34.9, 25.0, 24.9, 24.8, 24.7, 20.8, 20.7, 15.3, 15.0, 14.4, 14.2, 14.2.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.16.

HRMS (ESI) m/z: $[M+H]^+$ Calcd. for $C_{19}H_{35}BO_4$ 339.2701; Found: 339.2700.

ethyl 4-(cyclohex-2-en-1-yl)-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**5u**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 76% (52.9 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

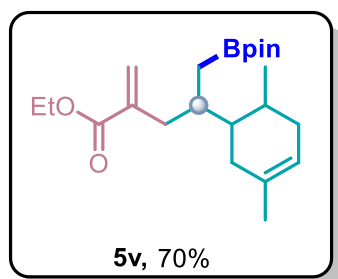
1H NMR (500 MHz, Chloroform-*d*) δ 6.15 (d, J = 1.8 Hz, 1H), 5.68 – 5.59 (m, 2H), 5.51 (m, 1H), 4.22 – 4.11 (m, 2H), 2.57 – 2.47 (m, 1H), 2.07 – 1.96 (m, 3H), 1.87 – 1.77 (m, 2H), 1.70 (m, 1H), 1.61 – 1.51 (m, 1H), 1.28 (t, J = 7.1 Hz, 4H), 1.20 (s, 13H), 0.73 (d, J = 1.8 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.3, 140.2, 140.1, 127.1, 127.1, 127.0, 126.9, 126.2, 126.1, 82.9, 60.5, 38.4, 38.2, 37.9, 37.6, 36.4, 36.0, 28.8, 28.0, 26.1, 25.9, 25.5, 24.9, 24.7, 14.21.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.44.

HRMS (ESI) m/z: $[M+H]^+$ Calcd. for $C_{20}H_{33}BO_4$ 349.2545; Found: 349.2543.

ethyl 4-(3,6-dimethylcyclohex-3-en-1-yl)-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**5v**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 70% (52.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

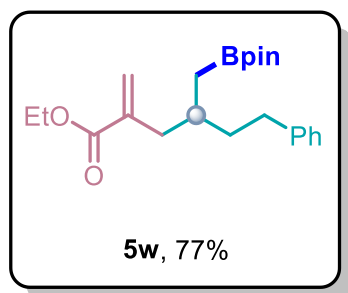
1H NMR (500 MHz, Chloroform-*d*) δ 6.15 (dd, J = 8.1, 1.9 Hz, 1H), 5.54 (s, 1H), 5.38 – 5.29 (m, 1H), 4.16 (m, 2H), 2.72 (m, 1H), 2.27 (m, 1H), 2.03 – 1.85 (m, 3H), 1.82 – 1.69 (m, 1H), 1.59 (d, J = 3.5 Hz, 4H), 1.35 (m, 1H), 1.31 – 1.25 (m, 4H), 1.18 (d, J = 4.7 Hz, 13H), 0.90 (d, J = 6.9 Hz, 2H), 0.82 (d, J = 6.9 Hz, 1H), 0.75 (t, J = 5.1 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.4, 167.4, 140.3, 140.2, 132.5, 132.4, 128.3, 128.3, 126.6, 126.3, 82.7, 82.7, 60.5, 60.4, 42.7, 42.3, 36.7, 36.5, 34.5, 33.9, 31.7, 31.6, 31.2, 25.0, 24.9, 24.7, 24.6, 23.4, 21.4, 20.8, 15.1, 14.9, 14.2, 14.2.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.32.

HRMS (ESI) m/z: $[M+H]^+$ Calcd. for $C_{22}H_{37}BO_4$ 377.2858; Found: 377.2853.

ethyl 2-methylene-6-phenyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexanoate (**5w**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 77% (57.2 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

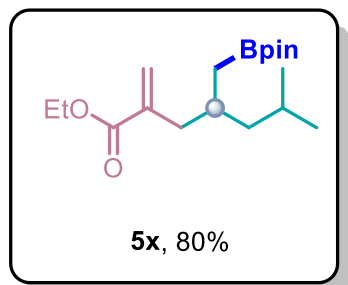
^1H NMR (500 MHz, Chloroform-*d*) δ 7.26 (t, J = 7.5 Hz, 2H), 7.20 – 7.14 (m, 3H), 6.18 (d, J = 1.8 Hz, 1H), 5.52 (d, J = 1.7 Hz, 1H), 4.19 (m, 2H), 2.63 (m, 2H), 2.48 (dd, J = 13.6, 6.1 Hz, 1H), 2.20 (dd, J = 13.7, 7.9 Hz, 1H), 1.93 (m, 1H), 1.60 (m, 2H), 1.28 (d, J = 4.4 Hz, 3H), 1.24 (d, J = 1.9 Hz, 12H), 0.84 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.4, 143.1, 139.7, 128.4, 128.4, 128.2, 126.2, 125.5, 83.0, 60.6, 39.2, 38.4, 33.3, 33.2, 25.0, 24.8, 14.2.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.41.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $\text{C}_{22}\text{H}_{33}\text{BO}_4$ 373.2545; Found: 373.2546.

ethyl 6-methyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5x**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 80% (51.8 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

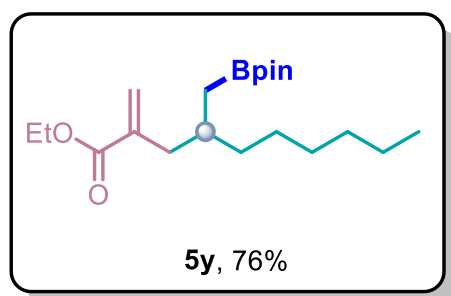
^1H NMR (500 MHz, Chloroform-*d*) δ 6.14 (d, J = 1.9 Hz, 1H), 5.48 (s, 1H), 4.17 (m, 2H), 2.42 (dd, J = 13.5, 5.2 Hz, 1H), 2.02 (dd, J = 13.5, 8.6 Hz, 1H), 1.93 – 1.83 (m, 1H), 1.66 (m, 1H), 1.27 (t, J = 7.1 Hz, 3H), 1.20 (d, J = 2.9 Hz, 12H), 1.09 (m, 2H), 0.84 (dd, J = 20.5, 6.6 Hz, 6H), 0.75 (dd, J = 15.5, 5.9 Hz, 1H), 0.63 (dd, J = 15.5, 6.9 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.5, 139.9, 126.1, 82.8, 60.4, 46.5, 39.5, 30.9, 25.3, 25.0, 24.8, 24.7, 22.9, 22.8, 14.2.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.04.

HRMS (ESI) m/z : $[M+H]^+$ Calcd. for $\text{C}_{18}\text{H}_{33}\text{BO}_4$ 325.2545; Found: 325.2545.

ethyl 2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)decanoate (**5y**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 76% (53.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

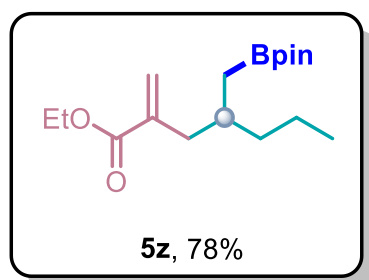
^1H NMR (500 MHz, Chloroform-*d*) δ 6.15 (d, J = 1.8 Hz, 1H), 5.49 (d, J = 1.8 Hz, 1H), 4.18 (m, 2H), 2.40 (m, 1H), 2.09 (dd, J = 13.6, 8.0 Hz, 1H), 1.84 – 1.75 (m, 1H), 1.29 – 1.24 (m, 13H), 1.22 (d, J = 1.6 Hz, 12H), 0.86 (t, J = 6.8 Hz, 3H), 0.77 (dd, J = 15.5, 6.1 Hz, 1H), 0.69 (dd, J = 15.6, 7.3 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.5, 140.0, 126.0, 82.8, 60.5, 39.3, 36.5, 33.3, 31.9, 29.6, 26.8, 24.9, 24.8, 24.8, 24.7, 22.7, 14.2, 14.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.33.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{20}\text{H}_{37}\text{BO}_4^+$ 353.2858; Found: 353.2855.

ethyl 2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5z**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 78% (48.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

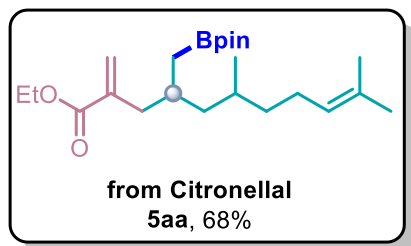
^1H NMR (500 MHz, Chloroform-*d*) δ 6.14 (d, J = 1.8 Hz, 1H), 5.48 (d, J = 1.9 Hz, 1H), 4.17 (m, 2H), 2.40 (m, 1H), 2.09 (dd, J = 13.6, 8.1 Hz, 1H), 1.80 (m, 1H), 1.28 (t, J = 7.1 Hz, 7H), 1.21 (d, J = 1.6 Hz, 12H), 0.85 (t, J = 7.1 Hz, 3H), 0.76 (dd, J = 15.6, 6.2 Hz, 1H), 0.68 (dd, J = 15.6, 7.3 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.4, 140.0, 125.9, 82.8, 60.4, 39.2, 36.4, 33.2, 32.1, 32.1, 26.4, 24.9, 24.8, 24.8, 24.7, 22.6, 14.2, 14.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.95.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{17}\text{H}_{31}\text{BO}_4$ 311.2388; Found: 311.2387.

ethyl 6,10-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)undec-9-enoate (**5aa**)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 68% (53.3 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

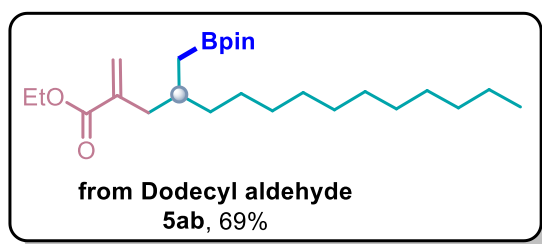
^1H NMR (500 MHz, Chloroform- d) δ 6.12 (t, J = 2.1 Hz, 1H), 5.46 (d, J = 2.2 Hz, 1H), 5.05 (m, 1H), 4.24 – 4.03 (m, 2H), 2.33 (dd, J = 13.7, 5.9 Hz, 1H), 1.99 – 1.83 (m, 4H), 1.63 (s, 3H), 1.55 (s, 3H), 1.49 (m, 1H), 1.27 – 1.23 (m, 3H), 1.18 (d, J = 3.4 Hz, 15H), 1.11 – 0.99 (m, 2H), 0.86 – 0.78 (m, 3H), 0.74 – 0.65 (m, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 167.4, 167.3, 139.9, 139.9, 130.8, 130.8, 126.2, 123.0, 125.1, 125.0, 82.7, 60.4, 60.4, 44.7, 44.4, 39.8, 39.1, 37.5, 37.5, 30.7, 30.7, 29.8, 29.8, 25.7, 25.4, 25.4, 25.0, 24.9, 24.9, 24.8, 24.8, 24.8, 24.7, 24.7, 19.7, 19.6, 17.6, 14.2, 14.2.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.21.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{23}\text{H}_{41}\text{BO}_4$ 393.3171; Found: 393.3169.

ethyl 2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)pentadecanoate
(5ab)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 69% (58.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

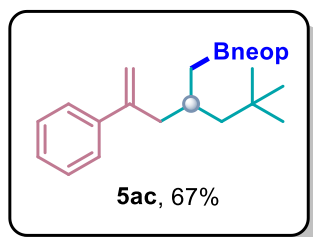
^1H NMR (500 MHz, Chloroform- d) δ 6.14 (d, J = 1.8 Hz, 1H), 5.48 (d, J = 1.8 Hz, 1H), 4.17 (m, 2H), 2.39 (dd, J = 13.6, 5.9 Hz, 1H), 2.08 (dd, J = 13.6, 8.1 Hz, 1H), 1.29 – 1.20 (m, 36H), 0.85 (t, J = 6.9 Hz, 3H), 0.79 – 0.63 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 140.0, 125.9, 82.9, 82.8, 39.3, 36.5, 33.3, 31.9, 29.9, 29.7, 29.7, 29.6, 29.4, 26.8, 24.9, 24.7, 22.7, 14.2, 14.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.28.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{25}\text{H}_{47}\text{BO}_4$ 423.3640; Found: 423.3643.

5,5-dimethyl-2-(2-neopentyl-4-phenylpent-4-en-1-yl)-1,3,2-dioxaborinane (5ac)



Following the **general procedure 6** on 0.2 mmol scale, colorless oil, yield: 67% (44.0 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

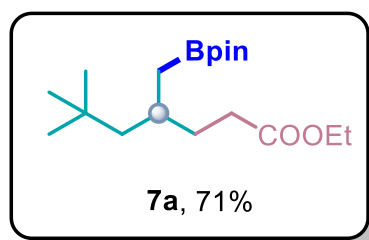
^1H NMR (500 MHz, Chloroform- d) δ 7.47 – 7.44 (m, 2H), 7.34 – 7.31 (m, 2H), 7.29 – 7.23 (m, 1H), 5.31 (t, J = 1.4 Hz, 1H), 5.06 (t, J = 1.5 Hz, 1H), 3.59 (d, J = 1.1 Hz, 4H), 2.60 – 2.55 (m, 1H), 2.42 (dd, J = 13.9, 7.5 Hz, 1H), 1.80 – 1.71 (m, 1H), 1.35 (dd, J = 13.9, 3.7 Hz, 1H), 1.13 (dd, J = 14.0, 6.1 Hz, 1H), 0.98 (d, J = 1.1 Hz, 6H), 0.89 – 0.85 (m, 1H), 0.81 (d, J = 1.3 Hz, 9H), 0.75 (dd, J = 15.8, 7.8 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 148.3, 141.5, 128.0, 127.1, 126.6, 113.9, 71.8, 50.2, 45.0, 31.6, 31.1, 30.2, 28.8, 22.0.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.98.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{25}\text{H}_{47}\text{BO}_4$ 329.2646; Found: 329.2640.

ethyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7a)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 71% (46.2 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

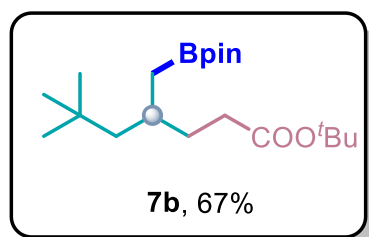
^1H NMR (500 MHz, Chloroform- d) δ 4.10 (m, 2H), 2.28 (m, 2H), 1.68 (m, 2H), 1.61 – 1.50 (m, 1H), 1.23 (d, J = 4.2 Hz, 15H), 1.20 – 1.09 (m, 2H), 0.88 (s, 9H), 0.84 – 0.76 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 174.2, 82.9, 60.1, 50.5, 33.3, 32.1, 31.1, 30.1, 30.1, 24.8, 24.8, 14.3.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.86.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{18}\text{H}_{35}\text{BO}_4$ 327.2701; Found: 327.2707.

tert-butyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7b)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 67% (47.4 mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

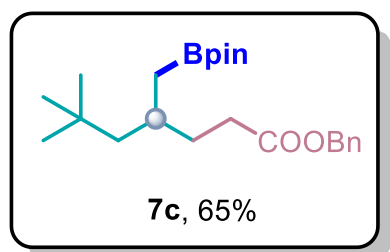
^1H NMR (500 MHz, Chloroform- d) δ 2.23 – 2.16 (m, 2H), 1.70 – 1.65 (m, 2H), 1.53 – 1.48 (m, 1H), 1.43 (s, 9H), 1.23 (s, 12H), 1.20 – 1.09 (m, 2H), 0.89 (s, 9H), 0.81 (dd, $J = 7.5, 6.3$ Hz, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 173.6, 82.8, 50.5, 33.6, 33.4, 31.1, 30.1, 30.07, 28.1, 24.9, 24.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.99.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}^{20}\text{H}^{39}\text{BO}_4$ 355.3014; Found: 355.3012.

benzyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (4c)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 65% (50.4 mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

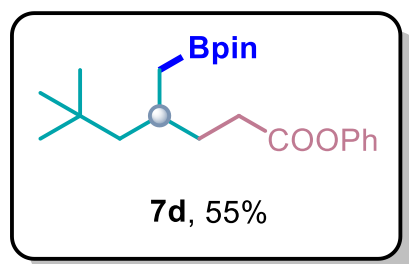
^1H NMR (500 MHz, Chloroform- d) δ 7.35 (d, $J = 4.0$ Hz, 4H), 7.33 – 7.29 (m, 1H), 5.10 (s, 2H), 2.36 (m, 2H), 1.72 – 1.66 (m, 1H), 1.60 (m, 1H), 1.22 (d, $J = 1.5$ Hz, 12H), 1.20 – 1.12 (m, 2H), 0.88 (d, $J = 1.5$ Hz, 9H), 0.82 (dd, $J = 10.2, 6.2$ Hz, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 173.9, 136.1, 128.5, 128.1, 128.1, 82.9, 66.0, 50.4, 33.3, 32.0, 31.1, 30.1, 24.8, 24.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.13.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{23}\text{H}_{37}\text{BO}_4$ 389.2858; Found: 389.2858.

phenyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7d)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 55% (41.4 mg), $R_f = 0.4$ (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

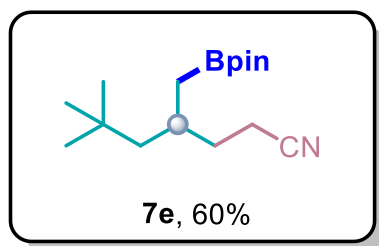
^1H NMR (500 MHz, Chloroform- d) δ 7.39 – 7.34 (m, 2H), 7.24 – 7.18 (m, 1H), 7.10 – 7.05 (m, 2H), 2.56 (m, 2H), 1.88 – 1.75 (m, 2H), 1.70 (m, 1H), 1.24 (s, 13H), 1.20 (dd, $J = 14.1, 5.0$ Hz, 1H), 0.93 (s, 9H), 0.91 – 0.85 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 172.6, 150.8, 129.3, 125.6, 121.6, 82.9, 50.5, 33.3, 32.1, 31.1, 30.1, 30.0, 24.9, 24.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.93.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{35}\text{BO}_4$ 375.2701; Found: 375.2698.

6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanenitrile (7e)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 60% (33.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

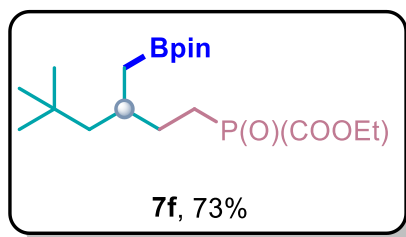
^1H NMR (500 MHz, Chloroform-*d*) δ 2.32 (m, 2H), 1.78 – 1.69 (m, 2H), 1.66 – 1.58 (m, 1H), 1.23 (s, 12H), 1.17 (dd, J = 4.9, 2.9 Hz, 2H), 0.90 (s, 9H), 0.83 (dd, J = 8.3, 6.1 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 120.2, 83.1, 50.2, 33.7, 31.1, 30.0, 29.9, 24.9, 24.8, 14.9.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.79.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{16}\text{H}_{30}\text{BNO}_2$ 280.2442; Found: 280.2445.

ethyl (5,5-dimethyl-3-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexyl)(oxo)-14-phosphanecarboxylate (7f)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 78% (48.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 4.09 – 3.99 (m, 4H), 1.74 – 1.58 (m, 4H), 1.53 – 1.45 (m, 1H), 1.27 (t, J = 7.1 Hz, 6H), 1.20 (s, 12H), 1.17 – 1.08 (m, 2H), 0.86 (d, J = 3.9 Hz, 9H), 0.82 – 0.72 (m, 2H).

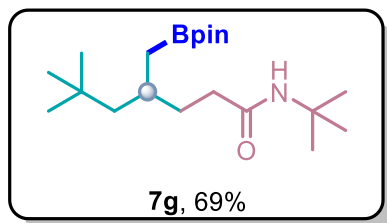
^{13}C NMR (126 MHz, Chloroform-*d*) δ 82.9, 61.38, 61.3, 50.2, 31.3, 31.1, 31.1, 30.5, 30.4, 30.1, 30.0, 24.9, 24.8, 23.7, 22.6, 16.5, 16.4.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.72.

^{31}P NMR (202 MHz, Chloroform-*d*) δ 33.44.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{18}\text{H}_{35}\text{BO}_5\text{P}$ 374.2388; Found: 374.2387.

N-(tert-butyl)-6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanamide (7g)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 69% (48.7 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

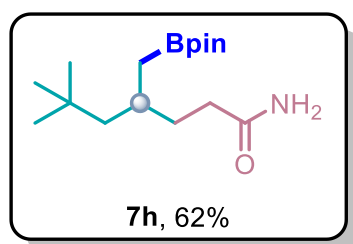
^1H NMR (500 MHz, Chloroform- d) δ 5.36 (s, 1H), 2.17 – 2.00 (m, 2H), 1.70 (m, 2H), 1.56 – 1.40 (m, 2H), 1.35 (d, J = 1.6 Hz, 9H), 1.25 (s, 12H), 1.23 – 1.12 (m, 2H), 0.94 – 0.82 (m, 10H).

^{13}C NMR (126 MHz, Chloroform- d) δ 172.9, 82.9, 51.0, 50.7, 35.5, 34.6, 31.2, 30.1, 30.1, 28.9, 24.9, 24.9.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.12.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{20}\text{H}_{40}\text{BNO}_3$ 354.3174; Found: 354.3179.

6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanamide (7h)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 62% (36.8 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

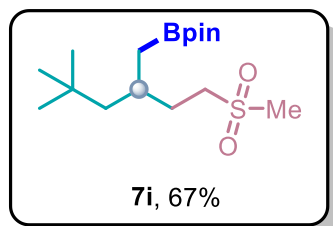
^1H NMR (500 MHz, Chloroform- d) δ 5.92 (s, 1H), 5.70 (s, 1H), 2.22 (m, 2H), 1.72 – 1.51 (m, 3H), 1.23 (s, 14H), 1.12 (dd, J = 14.1, 5.3 Hz, 1H), 0.87 (s, 9H), 0.77 (dd, J = 15.8, 7.3 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 176.2, 83.0, 50.7, 34.1, 33.6, 31.1, 30.0, 29.9, 24.8, 24.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.04.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{16}\text{H}_{32}\text{BNO}_3$ 298.2548; Found: 298.2457.

2-(4,4-dimethyl-2-(2-(methylsulfonyl)ethyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (7i)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 67% (44.5 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

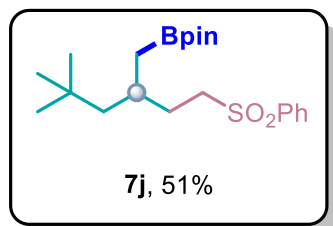
^1H NMR (500 MHz, Chloroform- d) δ 3.08 – 2.91 (m, 2H), 2.86 (s, 3H), 1.91 – 1.83 (m, 1H), 1.83 – 1.72 (m, 2H), 1.21 (d, J = 3.2 Hz, 15H), 1.18 (d, J = 4.7 Hz, 1H), 0.88 (s, 9H), 0.78 (dd, J = 15.7, 7.0 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 83.2, 52.9, 50.5, 40.2, 40.2, 31.1, 30.5, 30.0, 29.7, 29.4, 24.9, 24.9, 24.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.76.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{16}\text{H}_{33}\text{BO}_4\text{S}$ 333.2265; Found: 333.2261.

2-(4,4-dimethyl-2-(2-(phenylsulfonyl)ethyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (7j)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 78% (48.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

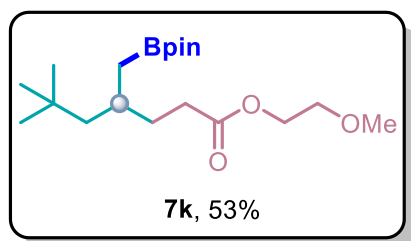
^1H NMR (500 MHz, Chloroform-*d*) δ 7.92 – 7.86 (m, 2H), 7.65 – 7.60 (m, 1H), 7.57 – 7.51 (m, 2H), 3.16 – 3.01 (m, 2H), 1.79 – 1.66 (m, 3H), 1.65 – 1.55 (m, 1H), 1.15 (d, J = 5.6 Hz, 12H), 1.10 (d, J = 4.9 Hz, 2H), 0.82 (d, J = 1.5 Hz, 9H), 0.79 – 0.65 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 139.2, 133.4, 129.1, 128.0, 83.0, 54.3, 50.3, 31.0, 30.6, 29.9, 29.6, 24.8, 24.7.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.79.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{35}\text{BO}_4\text{S}$ 395.2422; Found: 395.2428.

2-methoxyethyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7k)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 53% (37.3 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

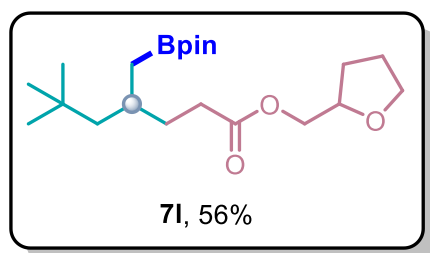
^1H NMR (500 MHz, Chloroform-*d*) δ 4.24 – 4.17 (m, 2H), 3.61 – 3.55 (m, 2H), 3.38 (d, J = 1.2 Hz, 3H), 2.33 (m, 2H), 1.68 – 1.63 (m, 1H), 1.56 (m, 1H), 1.22 (s, 14H), 1.20 – 1.09 (m, 2H), 0.91 – 0.78 (m, 10H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.2, 82.9, 82.9, 70.5, 63.2, 59.0, 50.4, 33.3, 31.9, 31.1, 30.1, 30.0, 24.9, 24.8, 12.5.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.93.

HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ Calcd. for $\text{C}_{19}\text{H}_{37}\text{BO}_5$ 379.2626; Found: 379.2624.

(tetrahydrofuran-2-yl)methyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7l)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 56% (42.8 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

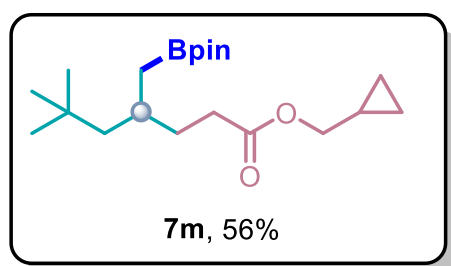
^1H NMR (500 MHz, Chloroform-*d*) δ 4.14 – 4.06 (m, 2H), 3.97 (m, 1H), 3.87 (dt, J = 8.4, 6.7 Hz, 1H), 3.77 (m, 1H), 2.41 – 2.25 (m, 2H), 2.03 – 1.93 (m, 1H), 1.93 – 1.83 (m, 2H), 1.74 – 1.63 (m, 2H), 1.57 (m, 2H), 1.22 (s, 13H), 1.19 – 1.10 (m, 2H), 0.90 – 0.78 (m, 10H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.1, 82.8, 68.4, 66.23, 66.2, 50.4, 45.7, 33.3, 32.7, 31.9, 31.1, 30.1, 28.2, 28.0, 25.6, 24.8, 24.8, 22.7, 22.6, 12.5.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 33.98.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{39}\text{BO}_5$ 383.2963; Found: 383.2967.

cyclopropylmethyl **6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7m)**



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 56% (39.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

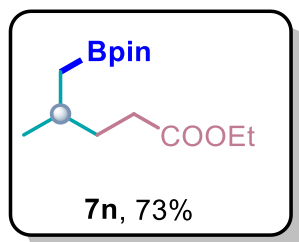
^1H NMR (500 MHz, Chloroform-*d*) δ 4.44 – 4.29 (m, 1H), 4.01 – 3.86 (m, 1H), 3.20 (m, 1H), 2.87 – 2.79 (m, 1H), 2.68 – 2.61 (m, 1H), 2.54 (m, 1H), 1.77 – 1.69 (m, 1H), 1.62 (m, 1H), 1.22 (d, J = 4.2 Hz, 12H), 1.20 – 1.17 (m, 1H), 1.15 (m, 4H), 0.89 (d, J = 2.2 Hz, 9H), 0.87 – 0.79 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 177.0, 176.9, 82.9, 82.8, 64.8, 65.0, 64.6, 50.9, 50.8, 49.4, 49.4, 44.7, 44.7, 44.7, 43.0, 42.9, 42.8, 42.7, 37.4, 37.4, 37.3, 37.3, 31.2, 31.1, 30.2, 30.2, 28.6, 28.2, 28.2, 25.0, 24.9, 24.9, 17.8, 17.8, 17.0, 17.0.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.10.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{20}\text{H}_{37}\text{BO}_4$ 353.2858; Found: 353.2854.

ethyl 4-methyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (7n)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 73% (39.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

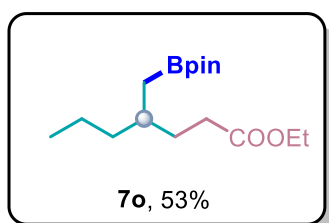
^1H NMR (500 MHz, Chloroform- d) δ 4.10 (m, 2H), 2.28 (m, 2H), 1.61 (m, 1H), 1.51 (m, 1H), 1.26 – 1.21 (m, 15H), 0.91 (d, J = 6.6 Hz, 3H), 0.83 (dd, J = 15.4, 5.5 Hz, 1H), 0.66 (dd, J = 15.4, 8.5 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 174.1, 82.9, 60.1, 34.4, 32.4, 29.2, 24.9, 24.7, 21.9, 14.2.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.99.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{14}\text{H}_{27}\text{BO}_4$ 271.2075; Found: 271.2078.

ethyl 4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7o)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 53% (31.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

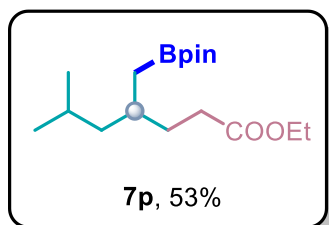
^1H NMR (500 MHz, Chloroform- d) δ 4.12 (m, 2H), 2.33 – 2.24 (m, 2H), 1.71 – 1.51 (m, 3H), 1.29 – 1.24 (m, 19H), 0.88 (t, J = 7.1 Hz, 3H), 0.77 (d, J = 6.6 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 174.2, 82.9, 60.1, 36.1, 33.8, 32.2, 32.0, 31.5, 26.4, 24.8, 24.8, 22.6, 14.2, 14.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.95.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{16}\text{H}_{31}\text{BO}_4$ 299.2388; Found: 299.2390.

ethyl 6-methyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7p)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 53% (33.1 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

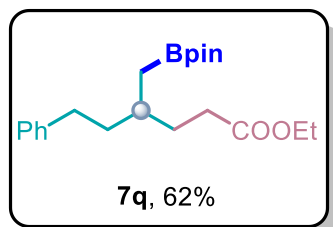
^1H NMR (500 MHz, Chloroform- d) δ 4.07 (m, 2H), 2.25 (m, 2H), 1.69 – 1.56 (m, 3H), 1.48 (m, 1H), 1.23 – 1.18 (m, 15H), 1.09 (m, 1H), 1.02 (m, 1H), 0.81 (dd, J = 8.8, 6.5 Hz, 6H), 0.70 (dd, J = 6.4, 3.6 Hz, 2H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 174.2, 82.9, 60.1, 46.1, 31.8, 31.5, 31.4, 25.2, 24.8, 24.8, 22.9, 22.8, 14.2.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.87.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₁₇H₃₃BO₄ 313.2545; Found: 313.2548.

ethyl 6-phenyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexanoate (7q)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 62% (44.6 mg), *R_f* = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

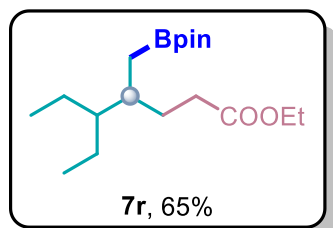
¹H NMR (500 MHz, Chloroform-*d*) δ 7.26 (t, *J* = 7.5 Hz, 2H), 7.19 – 7.14 (m, 3H), 4.11 (q, *J* = 7.1 Hz, 2H), 2.61 (m, 2H), 2.31 (m, 2H), 1.73 (m, 2H), 1.60 (m, 3H), 1.24 (s, 15H), 0.85 (d, *J* = 6.4 Hz, 2H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 174.1, 142.9, 128.4, 128.4, 128.3, 125.6, 83.0, 60.2, 38.2, 33.7, 33.2, 21.0, 31.3, 24.9, 24.9, 14.3.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.29.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₁H₃₃BO₄ 361.2545; Found: 361.2547.

ethyl 5-ethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7r)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 65% (42.4 mg), *R_f* = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

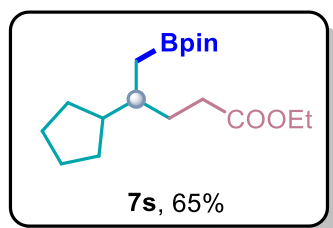
¹H NMR (500 MHz, Chloroform-*d*) δ 4.08 (m, 2H), 2.25 (m, 2H), 1.75 – 1.68 (m, 1H), 1.67 – 1.59 (m, 1H), 1.48 – 1.39 (m, 1H), 1.29 – 1.13 (m, 19H), 1.02 (m, 1H), 0.85 (dd, *J* = 7.4, 5.1 Hz, 6H), 0.73 (dd, *J* = 15.5, 6.9 Hz, 1H), 0.64 (dd, *J* = 15.5, 7.4 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 174.2, 82.9, 60.1, 45.8, 35.1, 32.9, 28.3, 24.8, 22.7, 22.6, 14.2, 12.5, 12.5.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.20.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₁₈H₃₅BO₄ 327.2701; Found: 327.2775.

ethyl 4-cyclopentyl-5-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)pentanoate (7s)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 65% (42.1 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

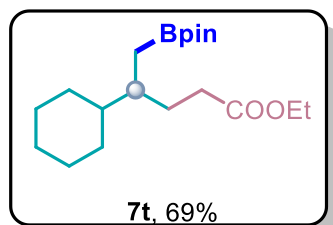
^1H NMR (500 MHz, Chloroform-*d*) δ 4.10 (m, 2H), 2.37 – 2.21 (m, 2H), 1.76 – 1.66 (m, 4H), 1.60 – 1.46 (m, 6H), 1.23 (d, J = 2.6 Hz, 15H), 1.11 (m, 2H), 0.88 – 0.67 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.4, 82.9, 60.1, 45.8, 38.9, 31.7, 30.6, 30.4, 30.3, 25.4, 25.3, 24.9, 24.8, 14.3.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.31.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{18}\text{H}_{33}\text{BO}_4$ 325.2545; Found: 325.2540.

ethyl 4-cyclohexyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (7t)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 69% (46.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

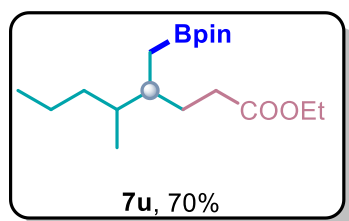
^1H NMR (500 MHz, Chloroform-*d*) δ 4.10 (m, 2H), 2.35 – 2.19 (m, 2H), 1.76 – 1.68 (m, 3H), 1.64 – 1.56 (m, 3H), 1.49 (m, 2H), 1.23 (d, J = 2.8 Hz, 18H), 1.12 – 1.06 (m, 1H), 1.04 – 0.93 (m, 2H), 0.82 – 0.75 (m, 1H), 0.66 (dd, J = 15.6, 6.9 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.2, 82.9, 60.1, 42.2, 39.2, 32.6, 30.0, 29.2, 28.7, 26.8, 26.8, 26.7, 24.8, 24.8, 24.8, 14.2.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.11.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{35}\text{BO}_4$ 339.2701; Found: 339.2705.

ethyl 5-methyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)octanoate (7u)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 70% (45.6mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

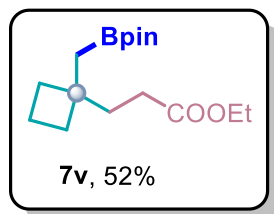
^1H NMR (500 MHz, Chloroform-*d*) δ 4.09 (m, 2H), 2.36 – 2.18 (m, 2H), 1.74 – 1.47 (m, 3H), 1.46 – 1.31 (m, 3H), 1.32 – 1.12 (m, 19H), 1.06 (m, 1H), 0.85 (m, 3H), 0.77 (dd, J = 19.0, 6.8 Hz, 3H), 0.72 (dd, J = 7.0, 3.8 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 174.2, 174.2, 82.9, 60.1, 60.1, 38.5, 38.3, 36.8, 36.5, 35.9, 35.7, 32.9, 32.8, 29.3, 27.6, 24.9, 24.8, 24.8, 20.8, 20.8, 15.6, 14.7, 14.4, 14.3, 14.25.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.29.

HRMS (ESI) m/z: [M+H]⁺ Calcd. for C₁₈H₃₅BO₄ 327.2701; Found: 327.2768.

ethyl 3-(1-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)cyclobutyl)propanoate (7v)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 52% (30.8 mg), *R*_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

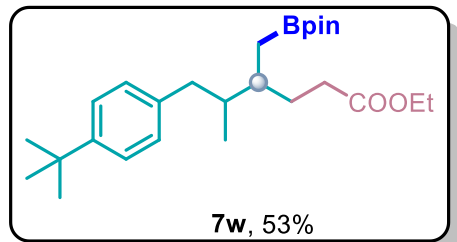
¹H NMR (500 MHz, Chloroform-*d*) δ 4.11 (q, *J* = 7.1 Hz, 2H), 2.26 – 2.19 (m, 2H), 1.86 – 1.77 (m, 8H), 1.26 – 1.25 (m, 3H), 1.23 (s, 12H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 174.5, 82.9, 60.2, 39.5, 36.5, 33.4, 30.1, 24.9, 24.7, 14.9, 14.2.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.58.

HRMS (ESI) m/z: [M+H]⁺ Calcd. for C₁₆H₂₉BO₄ 297.2232; Found: 297.2235.

ethyl 6-(4-(tert-butyl)phenyl)-5-methyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexanoate (7w)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 53% (45.6 mg), *R*_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

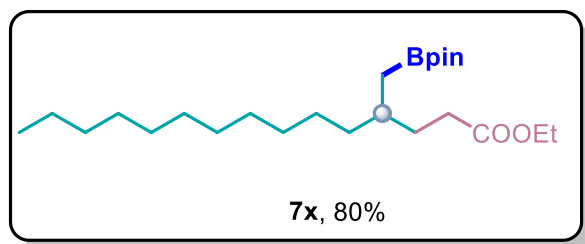
¹H NMR (500 MHz, Chloroform-*d*) δ 7.29 – 7.25 (m, 2H), 7.07 (m, 2H), 4.11 (m, 2H), 2.68 (m, 1H), 2.38 (m, 1H), 2.32 – 2.24 (m, 2H), 1.86 – 1.75 (m, 2H), 1.72 – 1.56 (m, 2H), 1.49 (m, 1H), 1.30 (d, *J* = 1.3 Hz, 9H), 1.26 – 1.22 (m, 14H), 0.91 – 0.69 (m, 5H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 174.2, 148.3, 138.8, 128.8, 128.8, 125.0, 83.0, 83.0, 60.2, 60.2, 40.1, 39.6, 39.5, 38.7, 38.5, 38.4, 34.3, 32.9, 32.8, 31.5, 29.3, 27.8, 24.9, 24.9, 24.9, 24.8, 15.1, 14.4, 14.3.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.68.

HRMS (ESI) m/z: [M+H]⁺ Calcd. for C₂₆H₄₃BO₄ 431.3327; Found: 431.3324.

ethyl 4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)pentadecanoate (7x)



Following the **general procedure 7** on 0.2 mmol scale, colorless oil, yield: 80% (65.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

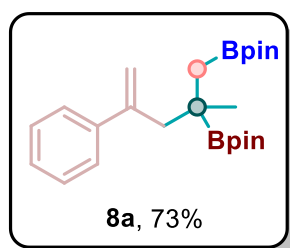
^1H NMR (500 MHz, Chloroform-*d*) δ 4.09 (m, 2H), 2.26 (m, 2H), 1.69 – 1.50 (m, 3H), 1.28 – 1.19 (m, 35H), 0.86 (t, J = 6.9 Hz, 3H), 0.74 (d, J = 6.6 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 174.2, 82.9, 60.1, 36.1, 33.8, 32.0, 31.9, 31.5, 30.0, 29.7, 29.7, 29.6, 29.6, 29.4, 26.7, 24.8, 24.8, 22.7, 14.2, 14.1.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.24.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{47}\text{BO}_4$ 411.3640; Found: 411.3637.

2,2'-(2-methyl-4-phenylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8a**)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 73% (60.2 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

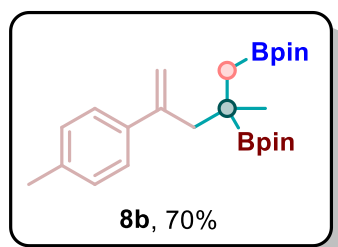
^1H NMR (500 MHz, Chloroform-*d*) δ 7.41 – 7.37 (m, 2H), 7.26 (m, 2H), 7.22 – 7.17 (m, 1H), 5.20 (d, J = 2.0 Hz, 1H), 5.07 (d, J = 2.1 Hz, 1H), 2.73 (d, J = 13.6 Hz, 1H), 2.57 (d, J = 13.6 Hz, 1H), 1.21 (d, J = 4.0 Hz, 12H), 1.14 (s, 12H), 0.95 (d, J = 15.7 Hz, 1H), 0.87 (s, 3H), 0.72 (d, J = 15.7 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 147.6, 143.7, 128.0, 126.9, 126.7, 116.0, 83.0, 82.8, 45.6, 25.0, 24.9, 24.8, 24.8, 24.4.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.60, 34.10.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{38}\text{B}_2\text{O}_4$ 413.3029; Found: 413.3027.

2,2'-(2-methyl-4-(*p*-tolyl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8b**)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 70% (60.2 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

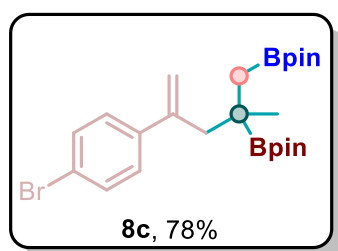
^1H NMR (500 MHz, Chloroform- d) δ 7.30 – 7.27 (m, 2H), 7.07 (d, J = 7.9 Hz, 2H), 5.18 (d, J = 2.0 Hz, 1H), 5.03 (d, J = 2.0 Hz, 1H), 2.70 (d, J = 13.6 Hz, 1H), 2.56 (d, J = 13.6 Hz, 1H), 2.31 (s, 3H), 1.21 (d, J = 4.0 Hz, 12H), 1.15 (s, 12H), 0.95 (d, J = 15.7 Hz, 1H), 0.87 (s, 3H), 0.71 (d, J = 15.7 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 147.4, 140.8, 136.49, 128.7, 126.59, 115.2, 83.0, 82.7, 45.5, 25.0, 24.9, 24.8, 24.8, 24.3, 21.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.38, 34.10.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{25}\text{H}_{41}\text{B}_2\text{O}_4$ 427.3185; Found: 427.3183.

2,2'-(4-(4-bromophenyl)-2-methylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8c)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 78% (76.4 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

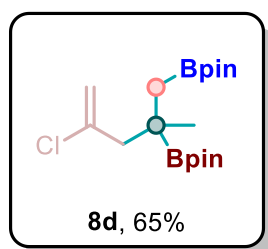
^1H NMR (500 MHz, Chloroform- d) δ 7.38 – 7.35 (m, 2H), 7.27 – 7.23 (m, 2H), 5.18 (d, J = 1.8 Hz, 1H), 5.07 (d, J = 1.7 Hz, 1H), 2.66 (d, J = 13.7 Hz, 1H), 2.53 (d, J = 13.6 Hz, 1H), 1.20 (d, J = 3.6 Hz, 12H), 1.14 (s, 12H), 0.90 (d, J = 15.6 Hz, 1H), 0.84 (s, 3H), 0.68 (d, J = 15.6 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 146.6, 142.7, 131.1, 128.5, 120.8, 116.6, 83.1, 82.8, 45.3, 25.0, 24.9, 24.7, 24.8, 24.4.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.91, 34.42.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{24}\text{H}_{37}\text{B}_2\text{BrO}_4$ 491.2134; Found: 491.2140.

2,2'-(4-chloro-2-methylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8d)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 65% (48.1 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

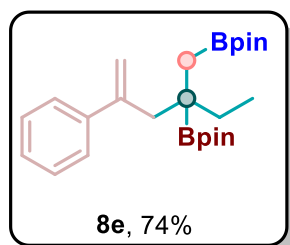
^1H NMR (500 MHz, Chloroform- d) δ 5.49 (s, 1H), 5.43 (s, 1H), 2.86 (d, J = 14.3 Hz, 1H), 2.77 (d, J = 14.3 Hz, 1H), 1.56 (d, J = 8.0 Hz, 24H), 1.35 (d, J = 20.9 Hz, 4H), 1.12 (d, J = 15.7 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 141.37, 114.24, 83.23, 82.84, 49.25, 24.94, 24.90, 24.86, 24.80, 23.63.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 33.90, 33.87.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₁₈H₃₃B₂ClO₄ 371.2326; Found: 371.2322.

2,2'-(2-ethyl-4-phenylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8e)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 74% (63.2 mg), *R_f* = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

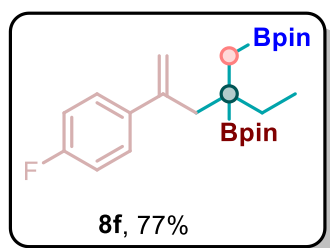
¹H NMR (500 MHz, Chloroform-*d*) δ 7.40 – 7.36 (m, 2H), 7.28 – 7.23 (m, 2H), 7.21 – 7.17 (m, 1H), 5.16 (d, *J* = 2.1 Hz, 1H), 5.10 (d, *J* = 2.1 Hz, 1H), 2.77 (d, *J* = 13.9 Hz, 1H), 2.69 (d, *J* = 13.9 Hz, 1H), 1.39 (dd, *J* = 13.8, 7.5 Hz, 1H), 1.32 – 1.27 (m, 1H), 1.21 (d, *J* = 1.9 Hz, 12H), 1.15 (d, *J* = 3.6 Hz, 12H), 0.84 (d, *J* = 3.7 Hz, 2H), 0.72 (s, 3H).

¹³C NMR (126 MHz, Chloroform-*d*) δ 148.0, 144.3, 127.9, 126.8, 126.8, 116.3, 83.0, 82.6, 41.5, 30.1, 25.1, 24.98, 25.0, 24.9, 9.7.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 35.76, 34.19.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₅H₄₀B₂O₄ 427.3185; Found: 427.3174.

2,2'-(2-ethyl-4-(4-fluorophenyl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8f)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 77% (68.4 mg), *R_f* = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

¹H NMR (500 MHz, Chloroform-*d*) δ 7.36 – 7.31 (m, 2H), 6.96 – 6.90 (m, 2H), 5.11 (d, *J* = 2.0 Hz, 1H), 5.07 (d, *J* = 2.0 Hz, 1H), 2.72 (d, *J* = 13.9 Hz, 1H), 2.64 (d, *J* = 13.8 Hz, 1H), 1.37 (dd, *J* = 13.8, 7.5 Hz, 1H), 1.31 – 1.26 (m, 1H), 1.20 (d, *J* = 1.4 Hz, 12H), 1.14 (d, *J* = 3.2 Hz, 12H), 0.86 – 0.77 (m, 2H), 0.72 (t, *J* = 7.5 Hz, 3H).

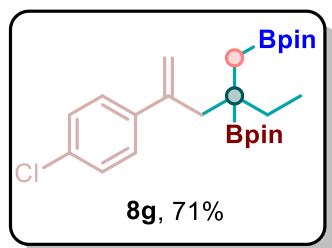
¹³C NMR (126 MHz, Chloroform-*d*) δ 163.0, 161.1, 147.0, 140.3, 140.3, 128.3, 128.3, 116.3, 114.7, 114.6, 83.0, 82.6, 41.7, 30.2, 25.1, 25.0, 24.9, 24.8, 9.7.

¹⁹F NMR (471 MHz, Chloroform-*d*) δ -116.48.

¹¹B NMR (160 MHz, Chloroform-*d*) δ 34.63, 34.22.

HRMS (ESI) *m/z*: [M+H]⁺ Calcd. for C₂₅H₃₉B₂FO₄ 445.3091; Found: 445.3088.

2,2'-(4-(4-chlorophenyl)-2-ethylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8g)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 71% (65.3 mg), $R_f = 0.4$ (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

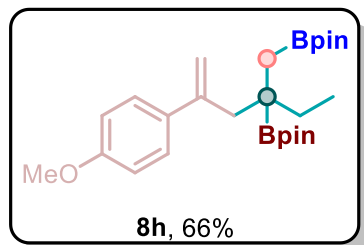
^1H NMR (500 MHz, Chloroform- d) δ 7.32 – 7.29 (m, 2H), 7.23 – 7.19 (m, 2H), 5.14 (d, $J = 1.9$ Hz, 1H), 5.10 (d, $J = 1.9$ Hz, 1H), 2.71 (d, $J = 13.9$ Hz, 1H), 2.64 (d, $J = 13.9$ Hz, 1H), 1.36 (dd, $J = 13.8, 7.4$ Hz, 1H), 1.28 – 1.24 (m, 1H), 1.20 (d, $J = 1.6$ Hz, 12H), 1.14 (d, $J = 2.9$ Hz, 12H), 0.86 – 0.76 (m, 2H), 0.72 (t, $J = 7.5$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 146.9, 142.7, 132.5, 128.2, 128.0, 116.8, 83.0, 82.6, 41.4, 30.3, 25.1, 25.0, 24.9, 24.8, 9.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.72, 34.65.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{25}\text{H}_{39}\text{B}_2\text{ClO}_4$ 461.2796; Found: 461.2798.

2,2'-(2-ethyl-4-(4-methoxyphenyl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8h)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 66% (60.2 mg), $R_f = 0.4$ (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

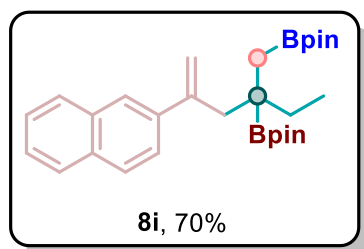
^1H NMR (500 MHz, Chloroform- d) δ 7.34 – 7.29 (m, 2H), 6.82 – 6.76 (m, 2H), 5.10 (d, $J = 2.1$ Hz, 1H), 5.02 (d, $J = 2.1$ Hz, 1H), 3.77 (s, 3H), 2.72 (d, $J = 13.9$ Hz, 1H), 2.64 (d, $J = 13.7$ Hz, 1H), 1.42 – 1.35 (m, 1H), 1.32 – 1.27 (m, 1H), 1.21 (d, $J = 1.8$ Hz, 12H), 1.15 (d, $J = 3.7$ Hz, 12H), 0.83 (d, $J = 1.8$ Hz, 2H), 0.73 (t, $J = 7.5$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 158.7, 147.3, 136.8, 127.8, 115.0, 113.3, 83.0, 82.6, 55.2, 41.6, 30.1, 25.1, 25.0, 24.9, 9.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 35.04, 34.72.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{26}\text{H}_{42}\text{B}_2\text{O}_5$ 457.3291; Found: 457.3288.

2,2'-(2-ethyl-4-(naphthalen-2-yl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8i)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 70% (66.6 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

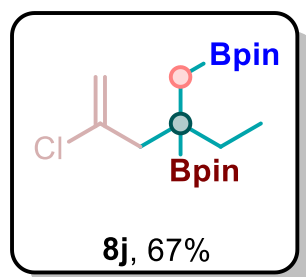
^1H NMR (500 MHz, Chloroform- d) δ 7.84 (d, J = 1.7 Hz, 1H), 7.79 (m, 2H), 7.74 (d, J = 8.5 Hz, 1H), 7.56 (dd, J = 8.5, 1.8 Hz, 1H), 7.42 (m, 2H), 5.31 (d, J = 2.0 Hz, 1H), 5.22 (d, J = 1.9 Hz, 1H), 2.91 (d, J = 14.1 Hz, 1H), 2.85 – 2.77 (m, 1H), 1.44 (dd, J = 13.8, 7.5 Hz, 1H), 1.36 – 1.31 (m, 1H), 1.22 (d, J = 4.4 Hz, 12H), 1.11 (d, J = 3.5 Hz, 12H), 0.92 – 0.89 (m, 2H), 0.75 (t, J = 7.4 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 147.9, 141.6, 133.4, 132.7, 128.1, 127.5, 127.4, 125.8, 125.8, 125.4, 125.2, 117.0, 83.0, 82.6, 41.7, 30.3, 25.0, 25.0, 24.9, 24.9, 9.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 35.13, 34.10.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{29}\text{H}_{42}\text{B}_2\text{O}_4$ 477.3342; Found: 477.3345.

2,2'-(4-chloro-2-ethylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8j)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 67% (51.5 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

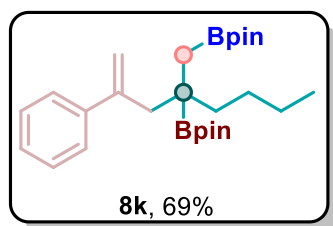
^1H NMR (500 MHz, Chloroform- d) δ 5.16 (d, J = 0.8 Hz, 1H), 5.11 (d, J = 0.8 Hz, 1H), 2.57 (t, J = 1.0 Hz, 2H), 1.55 (dd, J = 13.8, 7.5 Hz, 1H), 1.51 – 1.43 (m, 1H), 1.23 (s, 12H), 1.20 (s, 12H), 1.00 – 0.88 (m, 2H), 0.84 (t, J = 7.5 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 141.3, 114.5, 83.2, 82.7, 45.1, 29.8, 25.1, 25.0, 24.9, 24.8, 9.8.

^{11}B NMR (160 MHz, Chloroform- d) δ 33.92, 32.96.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{35}\text{B}_2\text{ClO}_4$ 385.2483; Found: 385.2480.

2,2'-(2-(2-phenylallyl)hexane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8k)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 69% (65.7 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

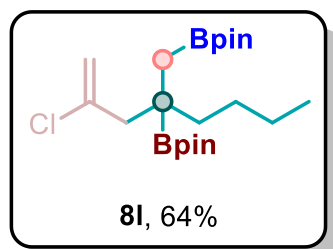
^1H NMR (500 MHz, Chloroform- d) δ 7.39 – 7.35 (m, 2H), 7.27 – 7.22 (m, 2H), 7.21 – 7.16 (m, 1H), 5.15 (d, J = 2.1 Hz, 1H), 5.10 (d, J = 2.0 Hz, 1H), 2.77 (d, J = 13.9 Hz, 1H), 2.67 (d, J = 13.8 Hz, 1H), 1.29 – 1.24 (m, 2H), 1.21 (s, 12H), 1.15 (d, J = 4.9 Hz, 12H), 1.10 (dd, J = 7.0, 2.6 Hz, 2H), 0.98 (p, J = 7.0 Hz, 2H), 0.86 (d, J = 4.5 Hz, 2H), 0.82 (t, J = 7.2 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 148.1, 144.3, 127.9, 126.8, 126.8, 116.4, 83.0, 82.6, 41.6, 37.7, 31.8, 30.0, 25.1, 25.0, 24.8, 22.6, 14.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.50, 33.86.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{27}\text{H}_{44}\text{B}_2\text{O}_4$ 455.3498; Found: 455.3499.

2,2'-(2-(2-chloroallyl)hexane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8l)



Following the **general procedure 8.1** on 0.2 mmol scale, colorless oil, yield: 64% (52.7 mg), R_f = 0.4 (silica gel, PE: EA = 20:1, v/v), column chromatography (silica gel, PE: EA = 20:1, v/v).

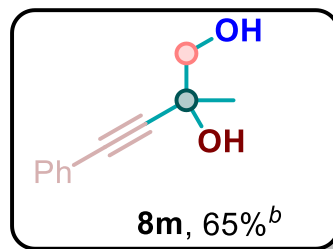
^1H NMR (500 MHz, Chloroform- d) δ 5.16 (d, J = 0.8 Hz, 1H), 5.10 (d, J = 0.8 Hz, 1H), 2.57 (s, 2H), 1.51 – 1.44 (m, 1H), 1.42 – 1.33 (m, 1H), 1.23 (s, 16H), 1.20 (s, 12H), 1.02 – 0.90 (m, 2H), 0.84 (t, J = 6.7 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform- d) δ 141.4, 114.5, 83.2, 82.7, 45.5, 37.5, 31.8, 30.1, 25.2, 25.1, 25.0, 25.0, 24.8, 22.6, 14.1.

^{11}B NMR (160 MHz, Chloroform- d) δ 34.40, 33.78.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{39}\text{B}_2\text{ClO}_4$ 413.2796; Found: 413.2798.

2-methyl-4-phenylbut-3-yne-1,2-diol (8m)



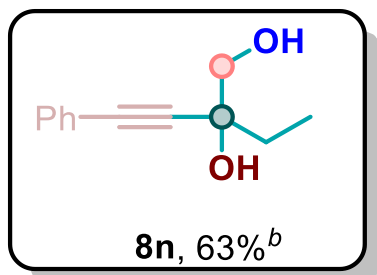
Following the **general procedure 8.2** on 0.2 mmol scale, colorless oil, yield: 65% (22.9 mg), $R_f = 0.4$ (silica gel, PE: EA = 2:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.46 – 7.40 (m, 2H), 7.31 (m, 3H), 3.76 (d, $J = 11.1$ Hz, 1H), 3.58 (d, $J = 11.1$ Hz, 1H), 1.55 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.78, 128.62, 128.33, 122.17, 90.36, 84.51, 70.77, 69.05, 25.38.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{11}\text{H}_{12}\text{O}_2$ 177.0910; Found: 177.0916.

2-ethyl-4-phenylbut-3-yne-1,2-diol (**8n**)



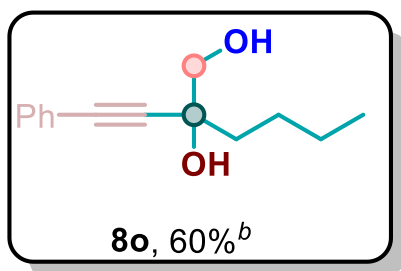
Following the **general procedure 8.2** on 0.2 mmol scale, colorless oil, yield: 63% (24.0 mg), $R_f = 0.4$ (silica gel, PE: EA = 2:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.47 – 7.40 (m, 2H), 7.36 – 7.28 (m, 3H), 3.78 (m, 1H), 3.61 (m, 1H), 2.80 (s, 1H), 2.28 (m, 1H), 1.78 (m, 2H), 1.13 (t, $J = 7.5$ Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.8, 128.6, 128.3, 122.3, 89.3, 85.7, 72.8, 69.4, 30.9, 8.6.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{12}\text{H}_{14}\text{O}_2$ 191.1067; Found: 191.1063.

2-(phenylethynyl)hexane-1,2-diol (**8o**)



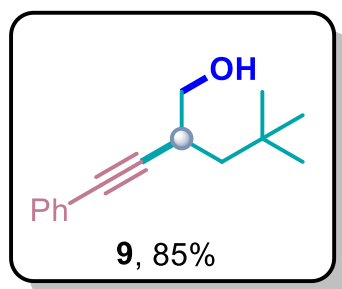
Following the **general procedure 8.2** on 0.2 mmol scale, colorless oil, yield: 60% (26.2 mg), $R_f = 0.4$ (silica gel, PE: EA = 2:1, v/v), column chromatography (silica gel, PE: EA = 5:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.48 – 7.40 (m, 2H), 7.31 (d, $J = 6.5$ Hz, 3H), 3.77 (dd, $J = 11.1, 4.2$ Hz, 1H), 3.60 (dd, $J = 11.0, 8.2$ Hz, 1H), 2.86 (s, 1H), 2.34 (dd, $J = 8.6, 5.0$ Hz, 1H), 1.74 (ddd, $J = 15.4, 7.0, 4.3$ Hz, 2H), 1.65 – 1.48 (m, 2H), 1.42 – 1.23 (m, 6H), 0.94 – 0.83 (m, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 131.80, 128.58, 128.32, 122.29, 89.62, 85.58, 72.37, 69.77, 37.96, 31.76, 29.49, 24.18, 22.62, 14.11.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{14}\text{H}_{18}\text{O}_2$ 219.1380; Found: 219.1382.

4,4-dimethyl-2-(phenylethynyl)pentan-1-ol (9)



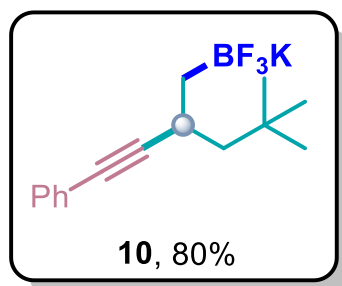
Following the **general procedure 12** on 0.2 mmol scale, colorless oil, yield: 85% (45.9 mg), $R_f = 0.4$ (silica gel, PE: EA = 10:1, v/v), column chromatography (silica gel, PE: EA = 10:1, v/v).

^1H NMR (500 MHz, Chloroform- d) δ 7.43 – 7.37 (m, 2H), 7.29 (m, 3H), 3.61 (m, 2H), 2.82 (m, 1H), 2.01 (s, 1H), 1.58 (dd, $J = 13.7, 10.0$ Hz, 1H), 1.38 (dd, $J = 13.6, 2.8$ Hz, 1H), 1.03 (s, 9H).

^{13}C NMR (126 MHz, Chloroform- d) δ 131.5, 128.3, 127.9, 123.5, 91.8, 83.4, 66.9, 45.1, 32.2, 30.8, 29.9.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{15}\text{H}_{20}\text{O}$ 217.1587; Found: 217.1586.

(4,4-dimethyl-2-(phenylethynyl)pentyl)trifluoro-borane, potassium salt (10)



Following the **general procedure 13** on 0.2 mmol scale, white solid, yield: 80% (49.0 mg).

^1H NMR (500 MHz, Acetone- d_6) δ 7.32 – 7.29 (m, 2H), 7.28 – 7.24 (m, 2H), 7.24 – 7.21 (m, 1H), 2.65 (m, 1H), 1.76 (dd, $J = 13.7, 2.5$ Hz, 1H), 1.36 (dd, $J = 13.6, 10.6$ Hz, 1H), 0.99 (s, 12H), 0.67 (m, 1H), 0.52 – 0.40 (m, 1H).

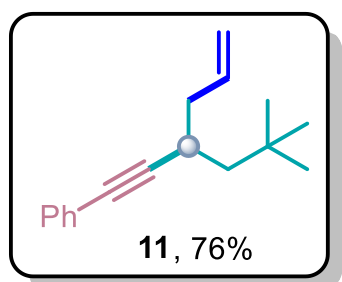
^{13}C NMR (126 MHz, Acetone- d_6) δ 130.9, 128.2, 126.7, 125.7, 101.3, 78.9, 50.4, 30.7, 29.7, 24.3, 24.3, 24.3.

^{11}B NMR (160 MHz, Acetone- d_6) δ 5.08.

^{19}F NMR (471 MHz, Acetone- d_6) δ -138.83 (t, $J = 54.9$ Hz).

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{15}\text{H}_{19}\text{BF}_3\text{K}$: 307.1242; Found: 307.1238.

(3-neopentylhex-5-en-1-yn-1-yl)benzene (11)



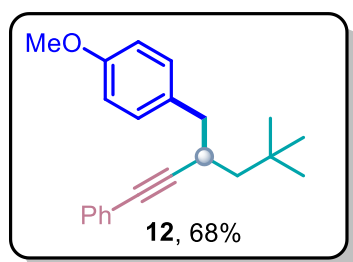
Following the **general procedure 14** on 0.2 mmol scale, colorless oil, yield: 76% (34.4 mg), R_f = 0.4 (silica gel, PE: EA = 100:1, v/v), column chromatography (silica gel, PE: EA = 100:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.42 – 7.37 (m, 2H), 7.31 – 7.24 (m, 3H), 5.97 (m, 1H), 5.19 – 5.06 (m, 2H), 2.67 (m, 1H), 2.32 (m, 2H), 1.61 – 1.55 (m, 1H), 1.41 (dd, J = 13.7, 2.8 Hz, 1H), 1.04 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 136.2, 131.4, 128.2, 127.5, 124.3, 116.7, 94.7, 82.1, 48.5, 41.6, 31.0, 30.0, 28.3.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{17}\text{H}_{22}$ 227.1794; Found: 227.1790.

1-(4,4-dimethyl-2-(phenylethynyl)pentyl)-4-methoxybenzene (12)



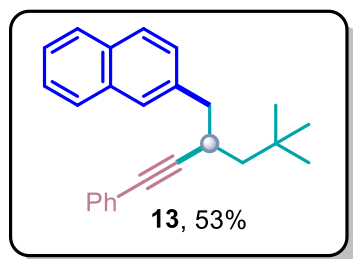
Following the **general procedure 15** on 0.2 mmol scale, colorless oil, yield: 68% (41.6mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.36 – 7.32 (m, 2H), 7.29 – 7.25 (m, 3H), 7.23 – 7.20 (m, 2H), 6.90 – 6.80 (m, 2H), 3.81 (s, 3H), 2.73 (s, 3H), 1.62 – 1.57 (m, 1H), 1.46 – 1.41 (m, 1H), 1.00 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.1, 131.8, 131.3, 130.4, 128.2, 127.4, 124.3, 113.5, 94.9, 82.6, 55.3, 48.4, 42.6, 31.0, 30.8, 29.9.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{22}\text{H}_{26}\text{O}$ 307.2056; Found: 307.2050.

2-(4,4-dimethyl-2-(phenylethynyl)pentyl)naphthalene (13)



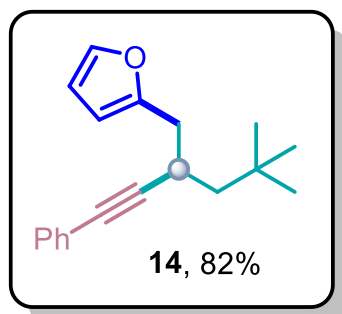
Following the **general procedure 15** on 0.2 mmol scale, colorless oil, yield: 53% (34.6 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.87 – 7.80 (m, 3H), 7.78 – 7.73 (m, 1H), 7.52 – 7.44 (m, 3H), 7.35 – 7.30 (m, 2H), 7.28 (m, 3H), 3.08 (td, J = 9.8, 3.5 Hz, 1H), 3.02 – 2.95 (m, 2H), 1.68 (dd, J = 13.6, 9.6 Hz, 1H), 1.53 (dd, J = 13.7, 2.3 Hz, 1H), 1.03 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 137.3, 133.5, 132.3, 131.3, 128.2, 128.1, 127.9, 127.7, 127.6, 127.5, 125.9, 125.2, 124.2, 94.7, 82.7, 48.5, 43.7, 31.0, 30.5, 29.9.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for : $\text{C}_{25}\text{H}_{26}$ 327.2107; Found: 327.2101.

2-(4,4-dimethyl-2-(phenylethynyl)pentyl)furan (14)



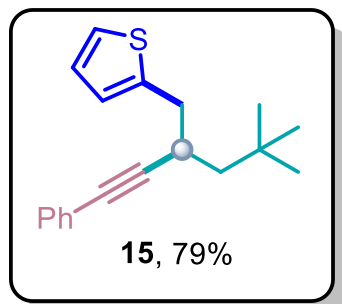
Following the **general procedure 16** on 0.2 mmol scale, colorless oil, yield: 82% (43.6 mg), R_f = 0.4 (silica gel, PE: EA = 100:1, v/v), column chromatography (silica gel, PE: EA = 100:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.34 (ddd, J = 4.3, 3.3, 2.2 Hz, 3H), 7.26 (h, J = 3.0 Hz, 3H), 6.31 (dd, J = 3.2, 1.9 Hz, 1H), 6.16 (dd, J = 3.1, 0.9 Hz, 1H), 2.97 – 2.90 (m, 2H), 2.86 – 2.77 (m, 1H), 1.64 – 1.58 (m, 1H), 1.43 (dd, J = 13.6, 2.4 Hz, 1H), 1.00 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 153.69, 141.14, 131.35, 128.16, 127.50, 124.11, 110.19, 106.76, 94.26, 82.09, 48.28, 35.80, 30.99, 29.83, 28.01.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{22}\text{O}$ 267.1743; Found: 267.1740.

2-(4,4-dimethyl-2-(phenylethynyl)pentyl)thiophene (15)



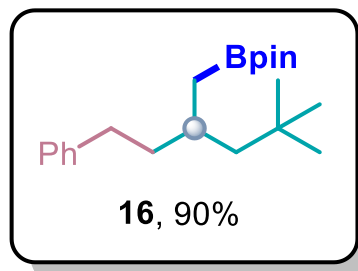
Following the **general procedure 17** on 0.2 mmol scale, colorless oil, yield: 79% (44.6 mg), R_f = 0.4 (silica gel, PE: EA = 100:1, v/v), column chromatography (silica gel, PE: EA = 100:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.44 – 7.39 (m, 2H), 7.33 – 7.28 (m, 3H), 7.19 (dd, J = 5.1, 1.2 Hz, 1H), 6.98 (dd, J = 5.1, 3.4 Hz, 1H), 6.95 (dd, J = 3.4, 1.1 Hz, 1H), 3.09 (m, 2H), 2.94 – 2.85 (m, 1H), 1.64 (dd, J = 13.7, 10.2 Hz, 1H), 1.53 – 1.46 (m, 1H), 1.04 (s, 10H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 142.0, 131.4, 128.2, 127.6, 126.5, 125.9, 124.1, 123.9, 94.4, 82.9, 48.3, 37.7, 31.0, 30.9, 30.0.

HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{19}\text{H}_{22}\text{S}$ 283.1515; Found: 283.1519.

2-(4,4-dimethyl-2-phenethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (16)



Following the **general procedure 18** on 0.2 mmol scale, colorless oil, yield: 90% (59.4 mg), R_f = 0.4 (silica gel, PE: EA = 30:1, v/v), column chromatography (silica gel, PE: EA = 30:1, v/v).

^1H NMR (500 MHz, Chloroform-*d*) δ 7.29 – 7.25 (m, 2H), 7.21 – 7.14 (m, 3H), 2.61 (m, 2H), 1.79 – 1.72 (m, 1H), 1.67 (m, 1H), 1.62 – 1.52 (m, 1H), 1.32 (dd, J = 14.1, 5.0 Hz, 1H), 1.26 (d, J = 1.9 Hz, 14H), 1.20 (dd, J = 14.1, 5.1 Hz, 1H), 0.92 (s, 9H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 143.4, 128.4, 128.2, 125.5, 82.9, 82.8, 50.9, 40.9, 33.5, 31.3, 30.7, 30.2, 29.5, 25.0, 24.9, 24.9.

^{11}B NMR (160 MHz, Chloroform-*d*) δ 34.03.

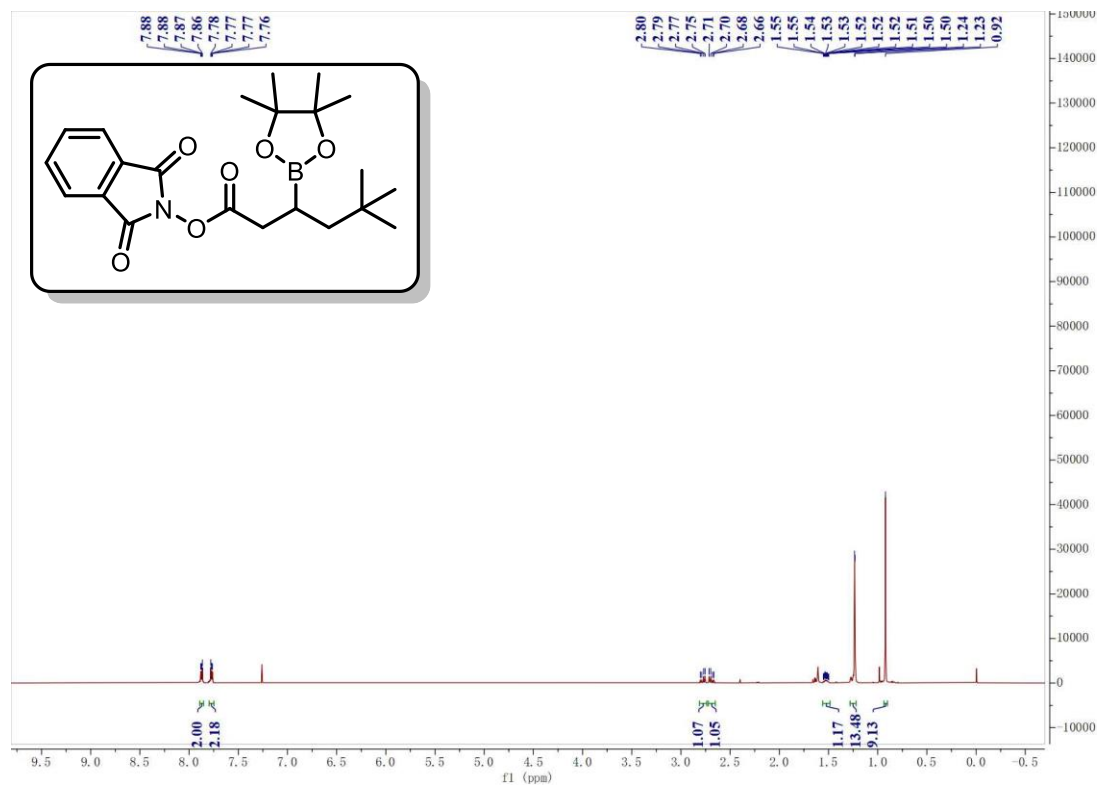
HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd. for $\text{C}_{21}\text{H}_{35}\text{BO}_2$ 331.2803; Found: 331.2795.

2.6 NMR spectroscopic data

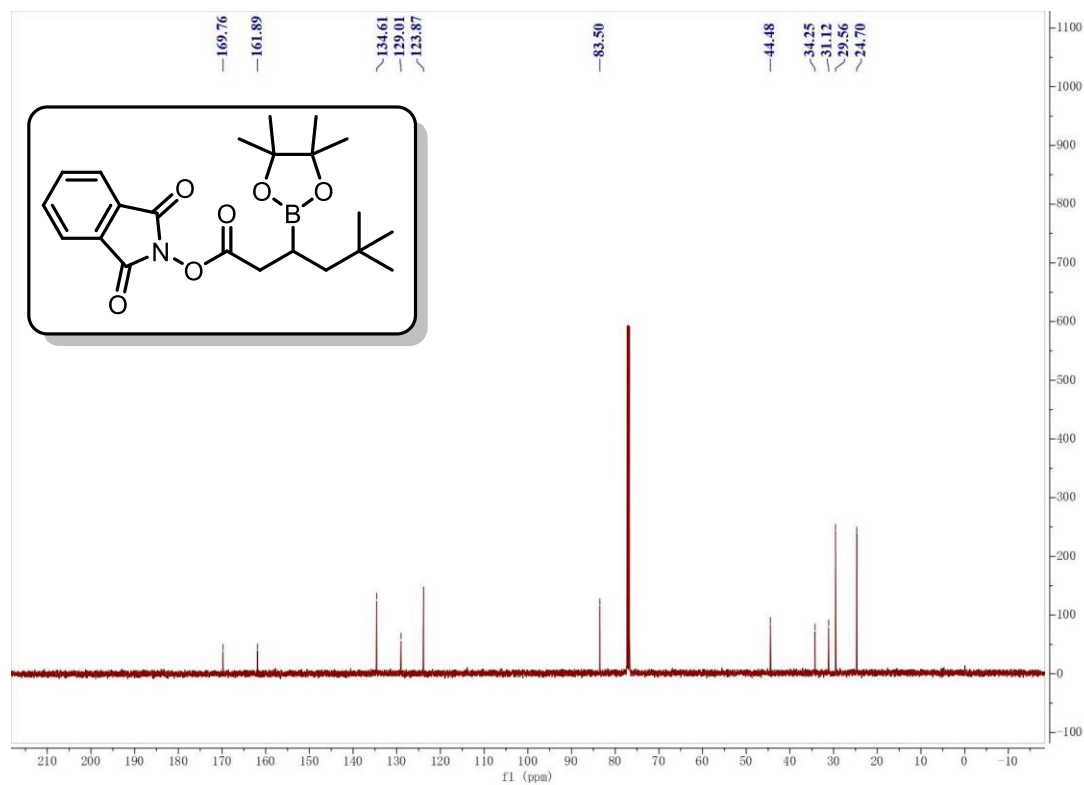
1,3-dioxoisindolin-2-yl

5,5-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1a)

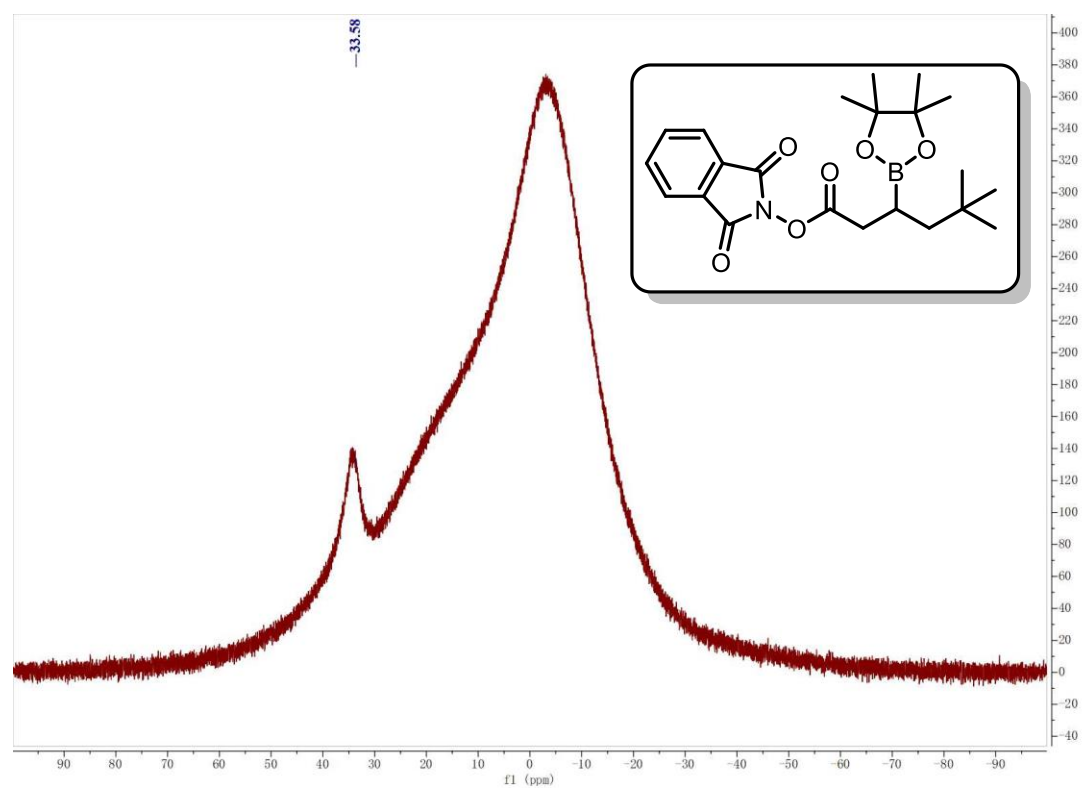
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

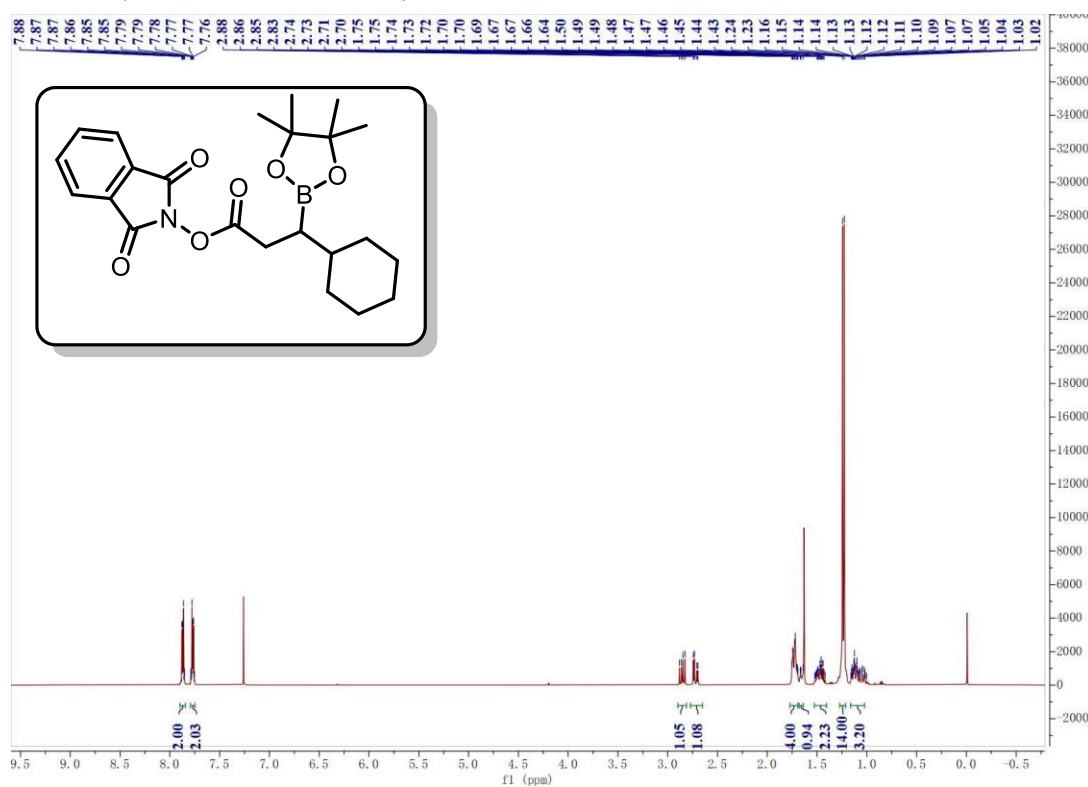


^{11}B NMR (160 MHz, Chloroform-*d*)

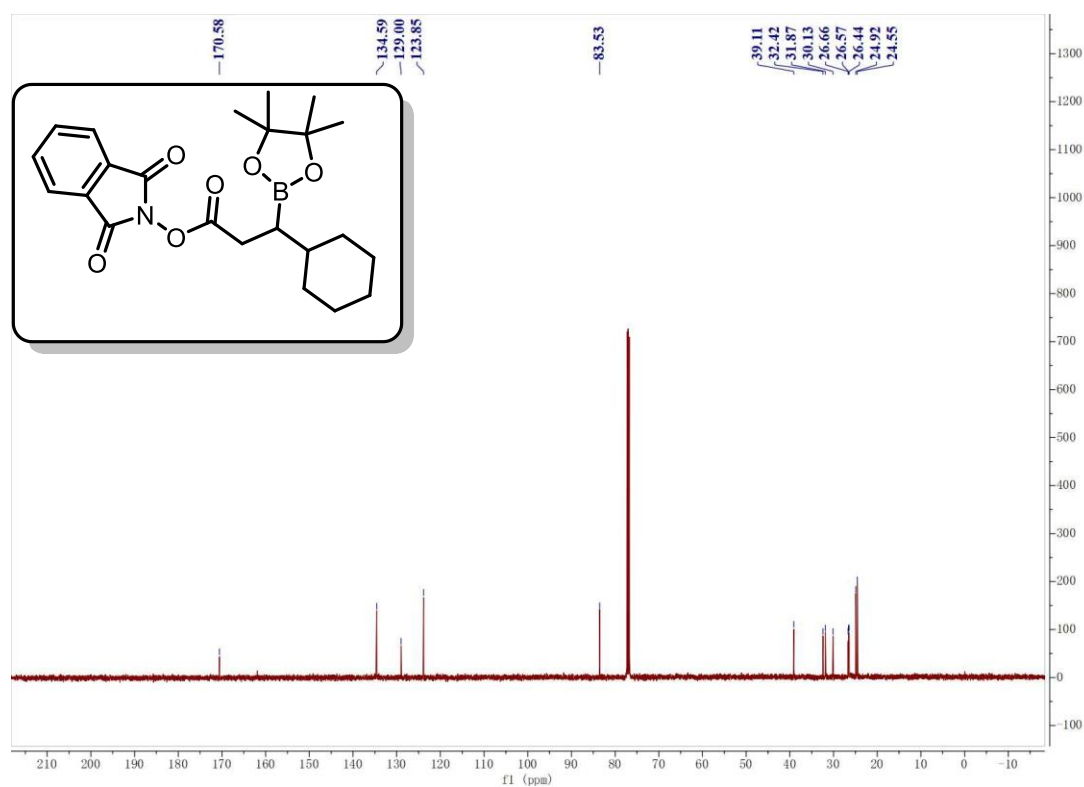


1,3-dioxoisindolin-2-yl 3-cyclohexyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate
(1b)

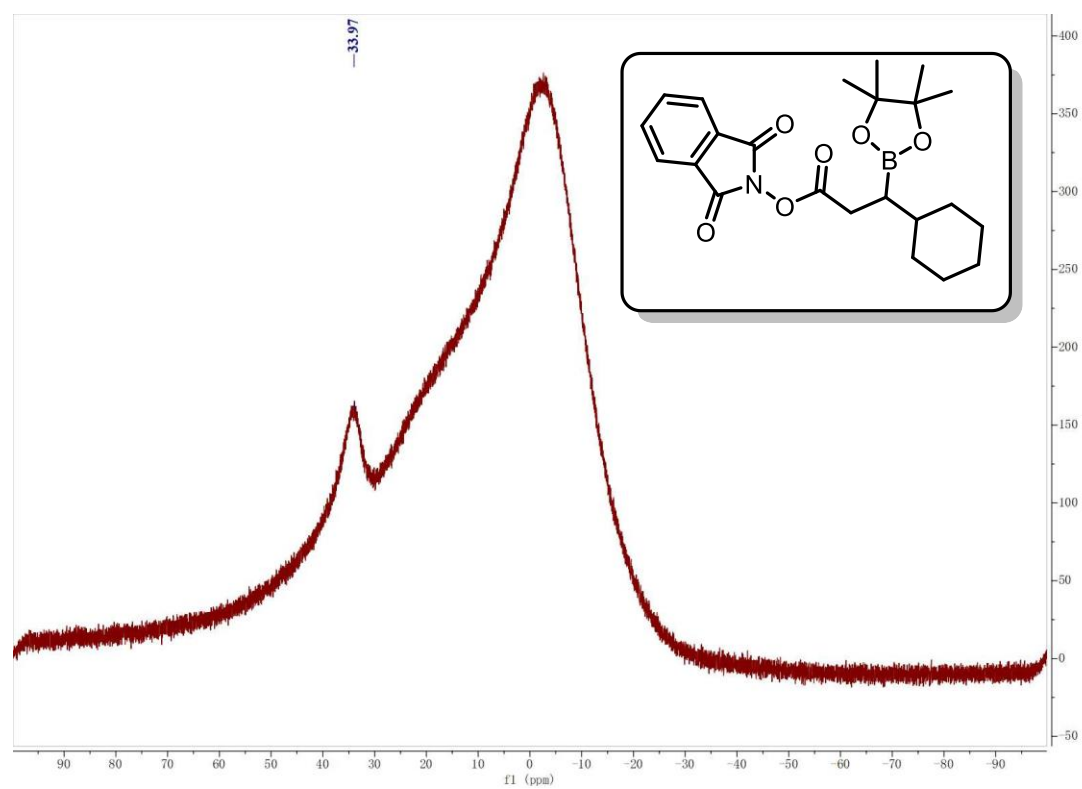
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

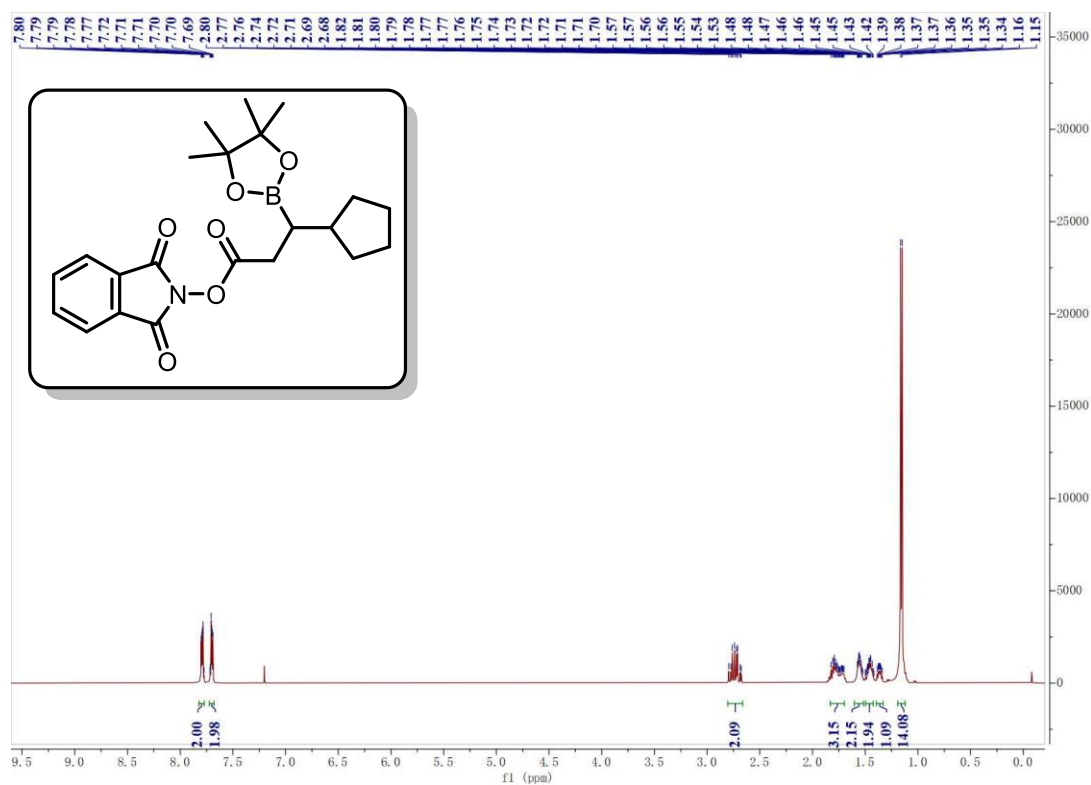


^{11}B NMR (160 MHz, Chloroform-*d*)

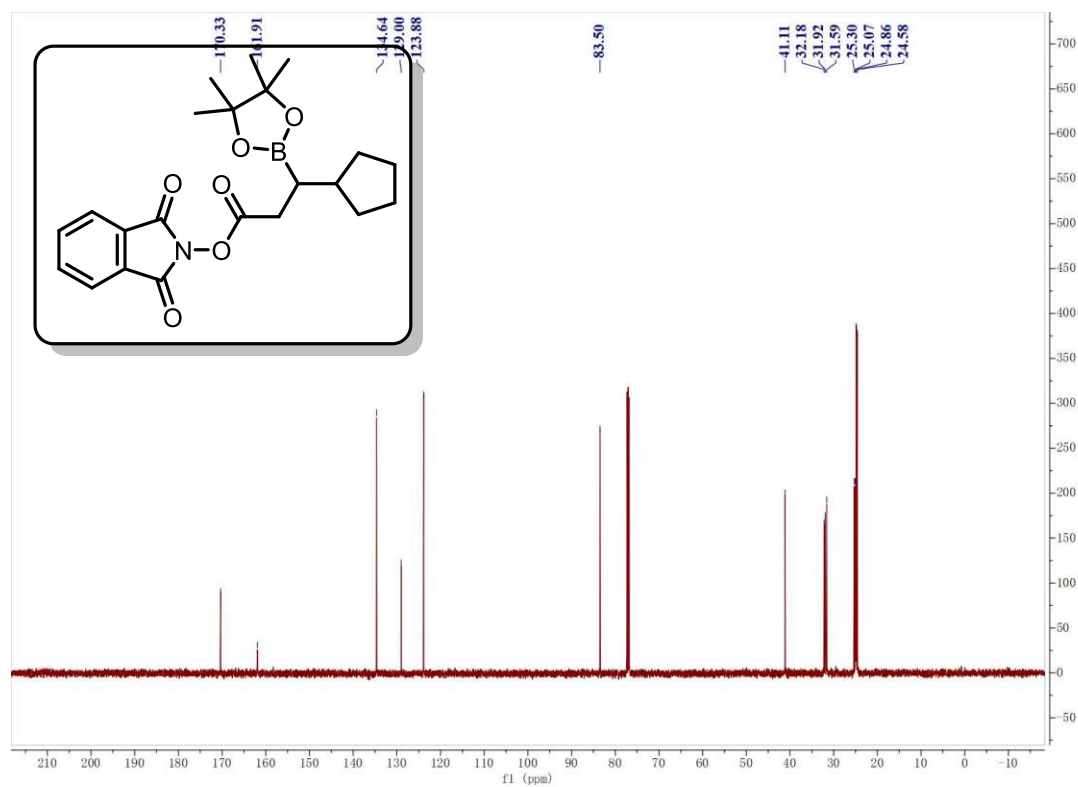


**1,3-dioxoisindolin-2-yl 3-cyclopentyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate
(1c)**

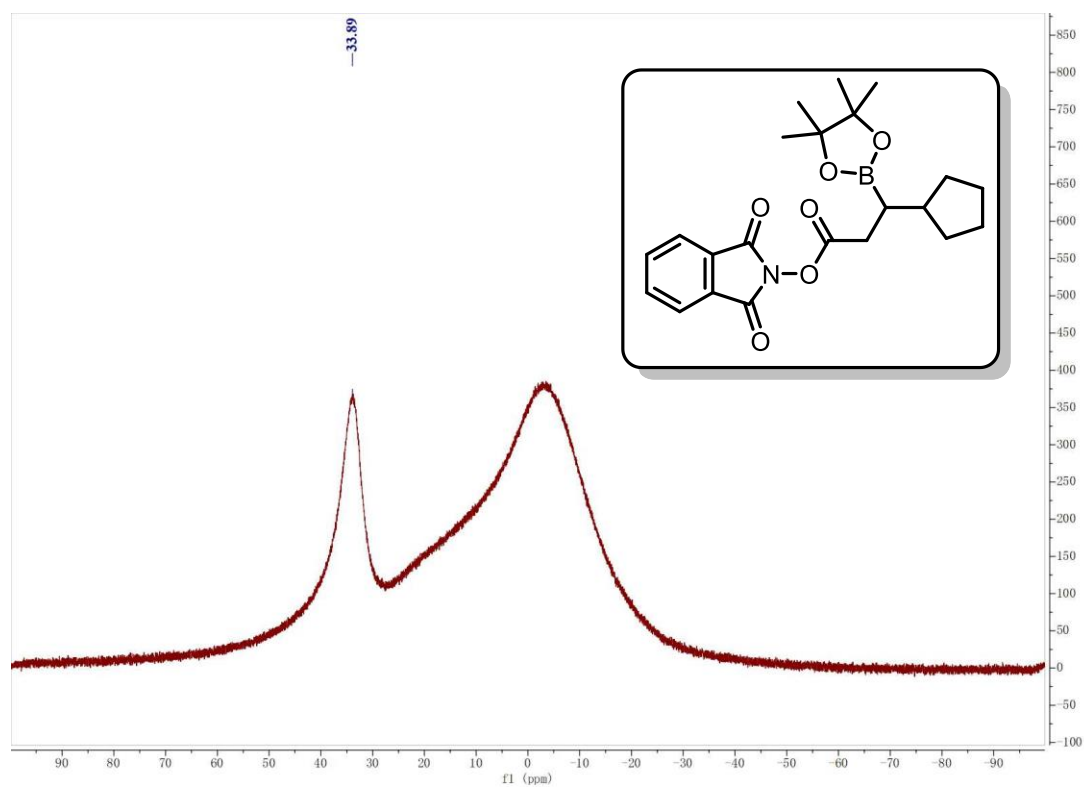
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

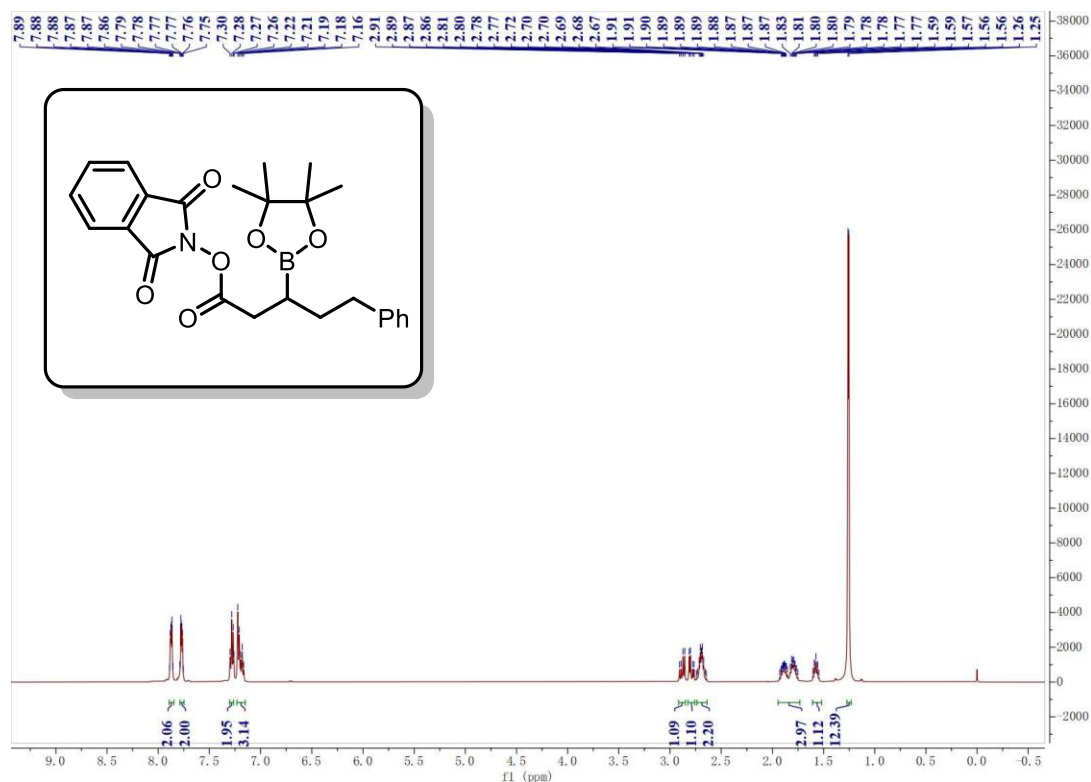


^{11}B NMR (160 MHz, Chloroform-*d*)

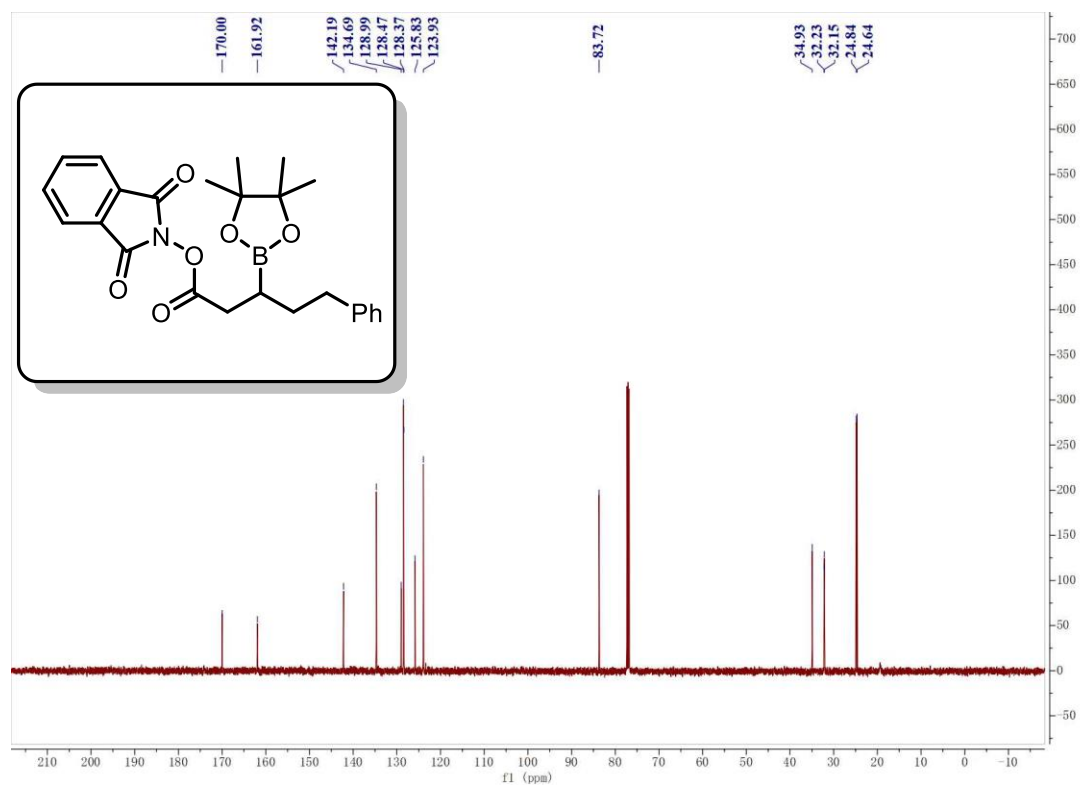


1,3-dioxoisindolin-2-yl 5-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1d)

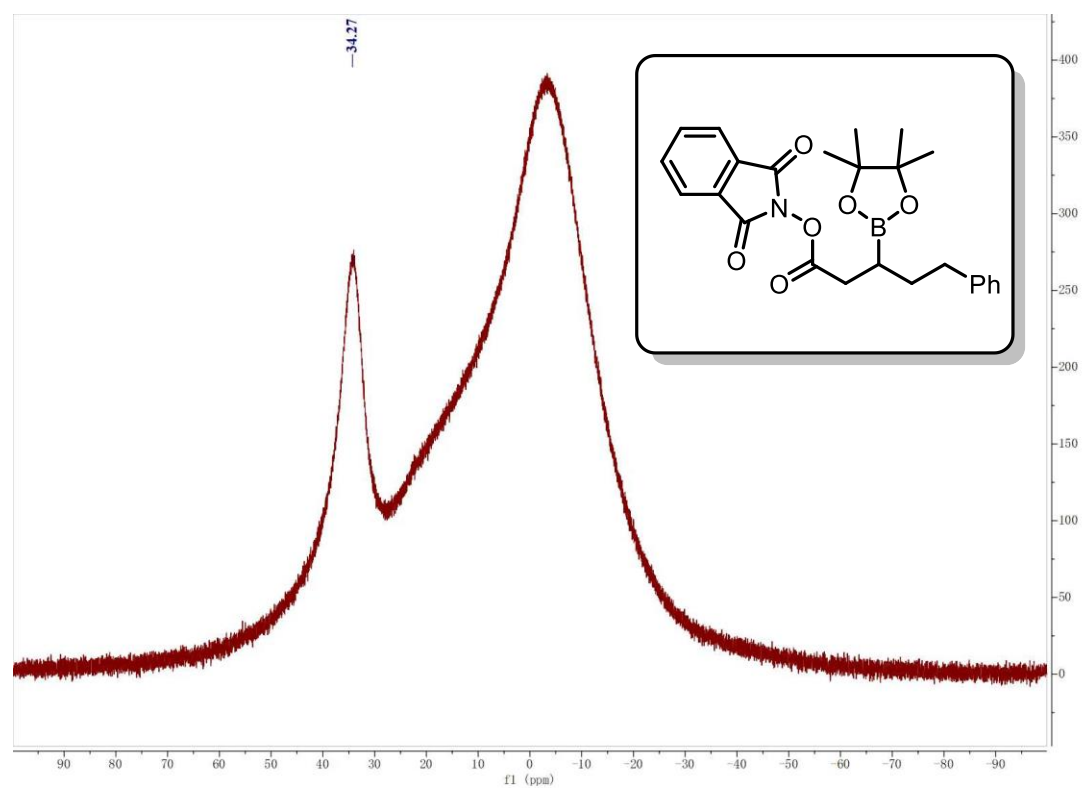
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

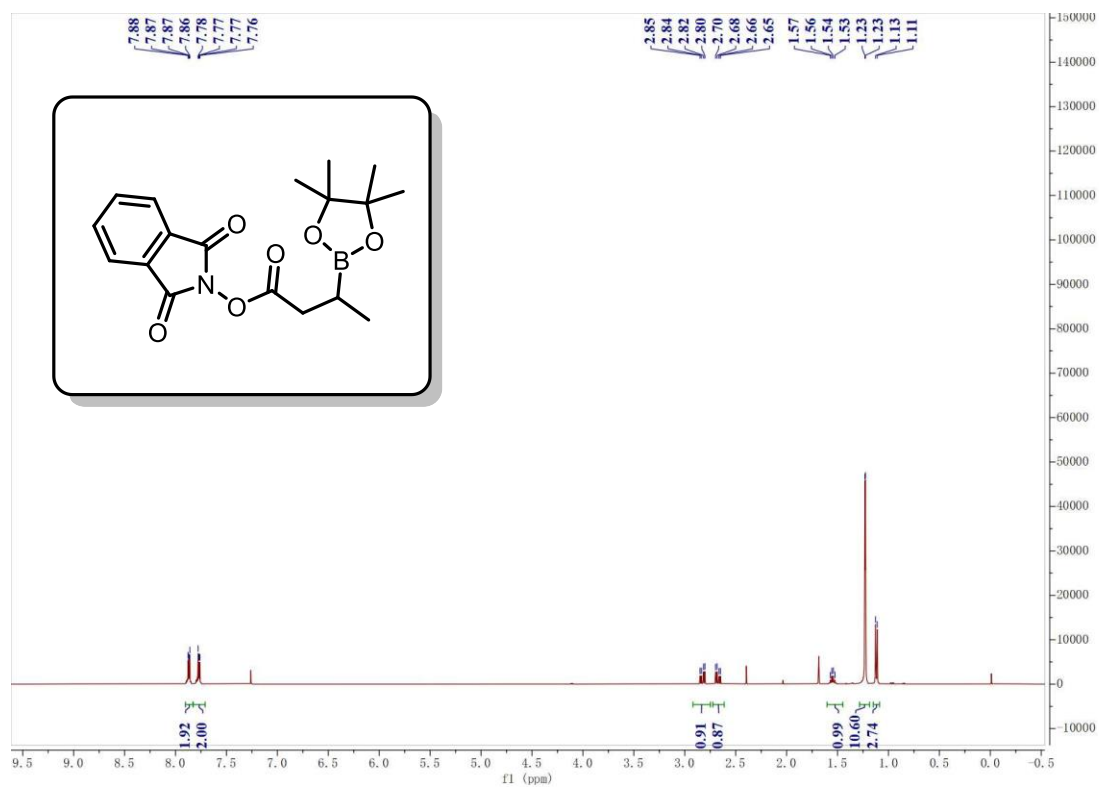


^{11}B NMR (160 MHz, Chloroform-*d*)

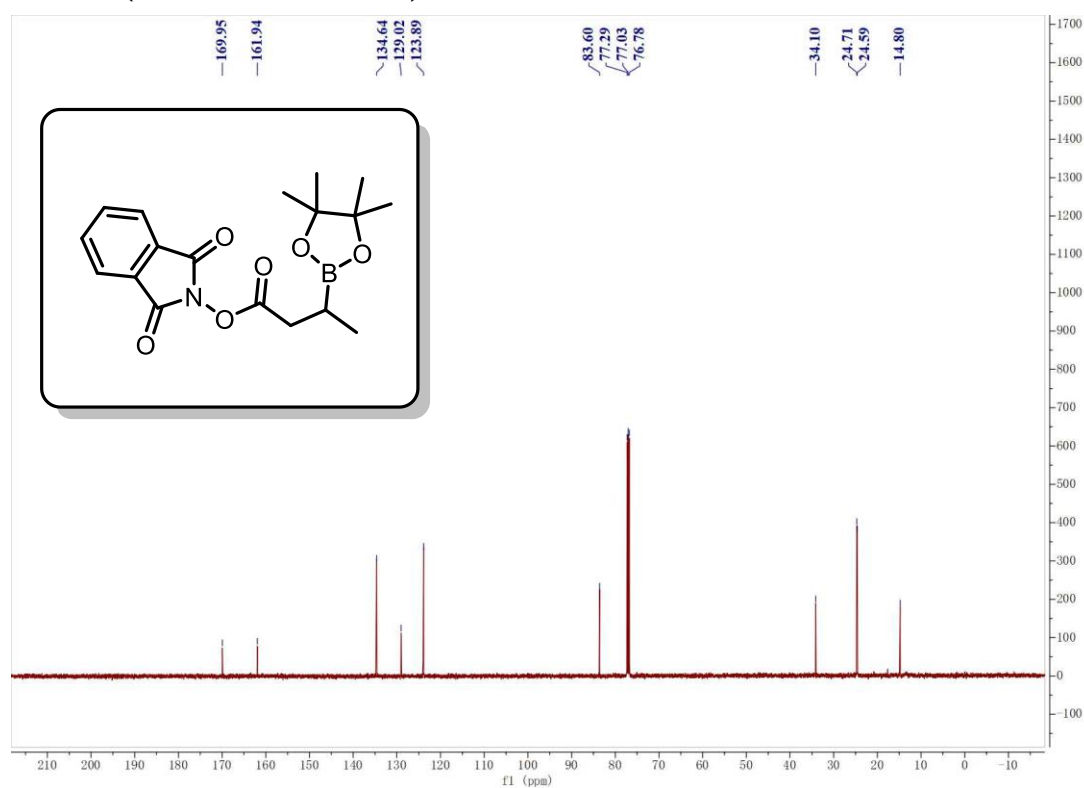


1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butanoate (1e)

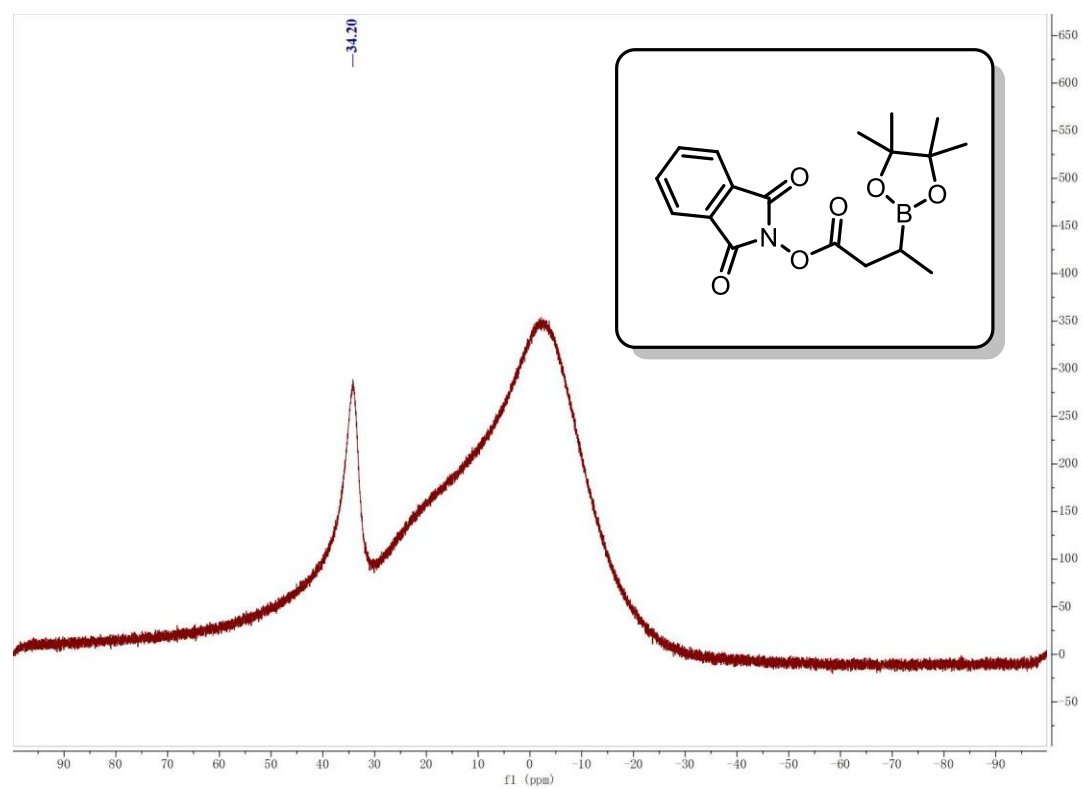
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

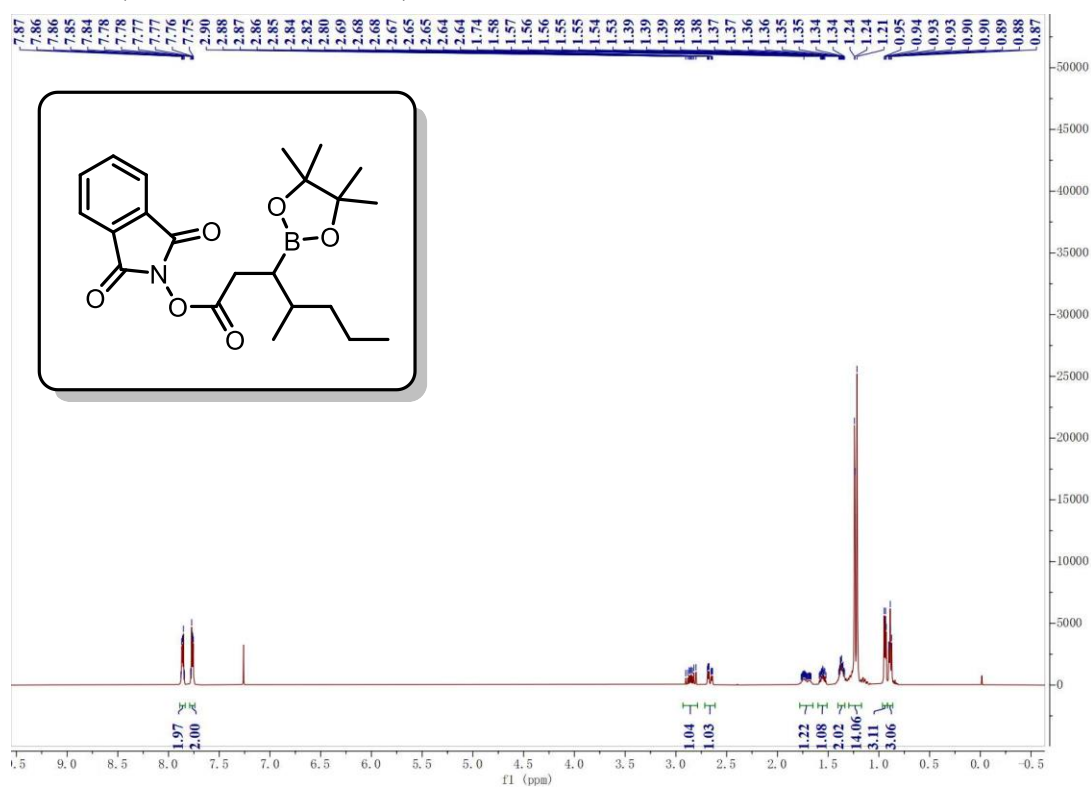


^{11}B NMR (160 MHz, Chloroform-*d*)

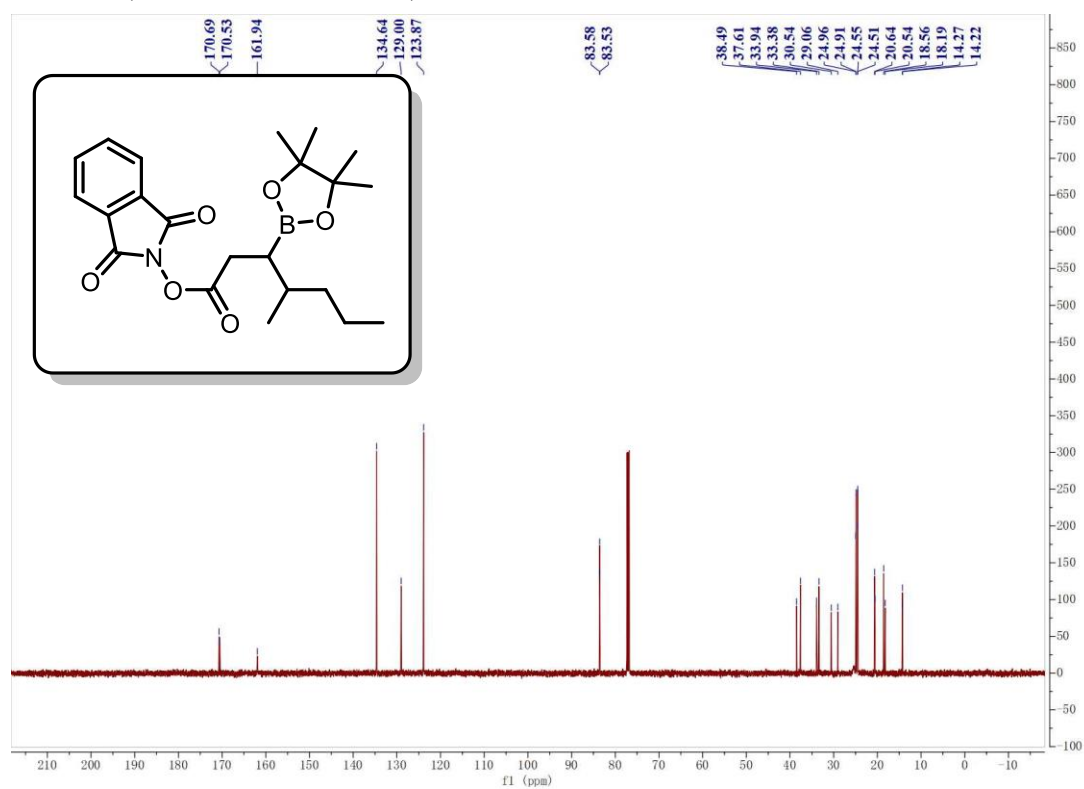


**1,3-dioxoisindolin-2-yl 4-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptanoate
(1f)**

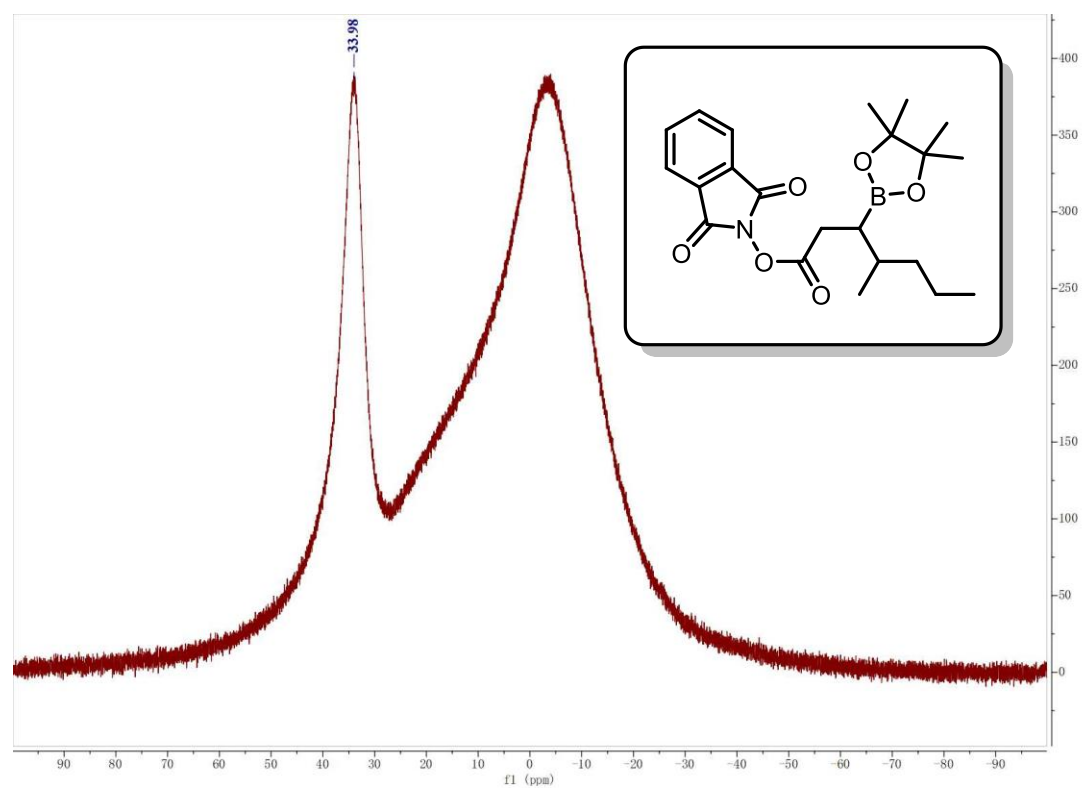
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

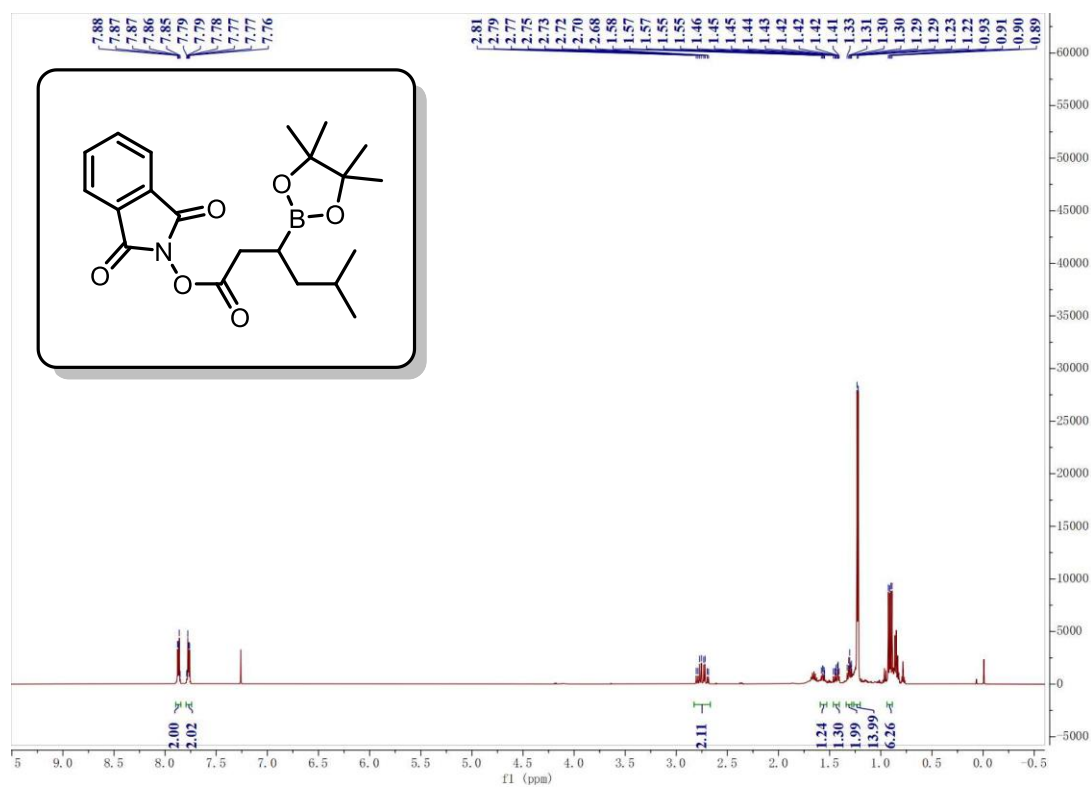


^{11}B NMR (160 MHz, Chloroform-*d*)

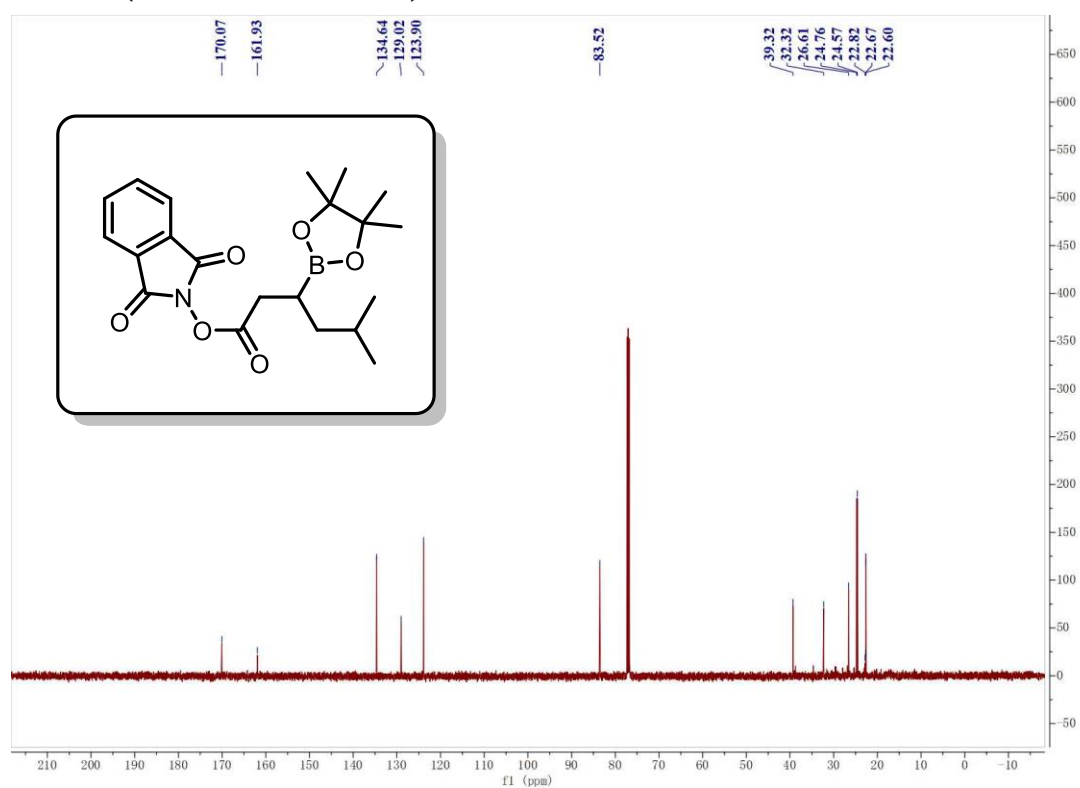


1,3-dioxoisindolin-2-yl 5-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1g)

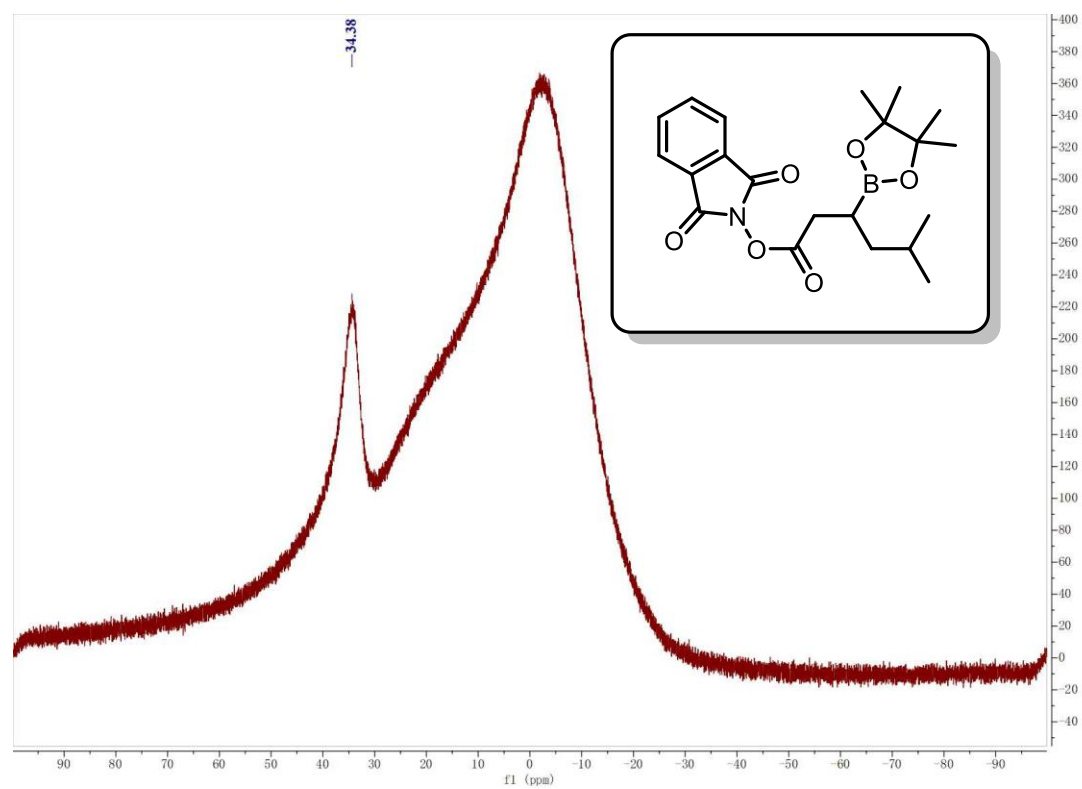
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

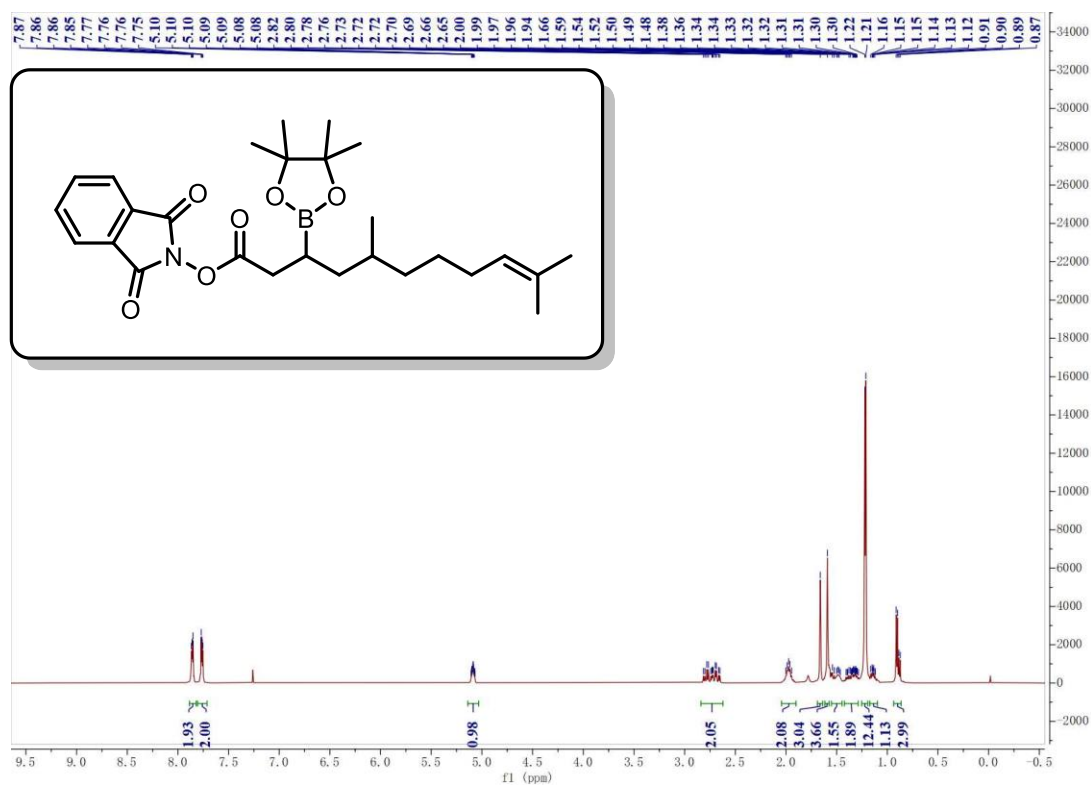


^{11}B NMR (160 MHz, Chloroform-*d*)

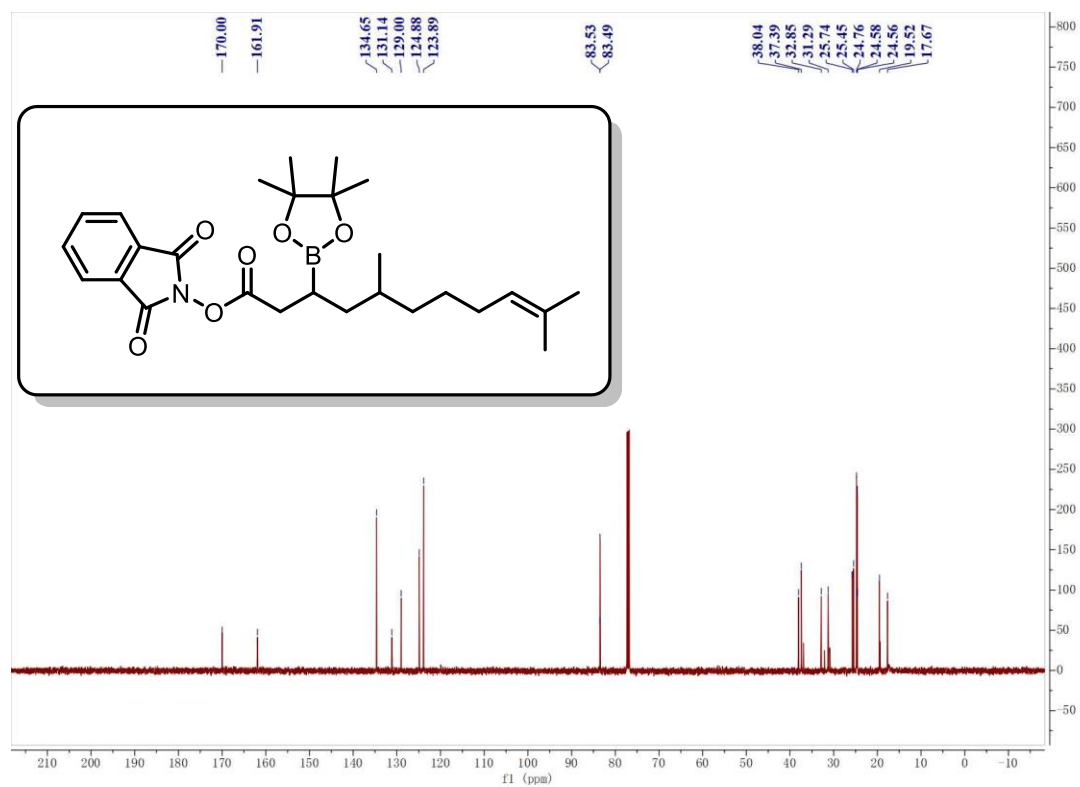


1,3-dioxoisindolin-2-yl 5,10-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)undec-9-enoate (1h)

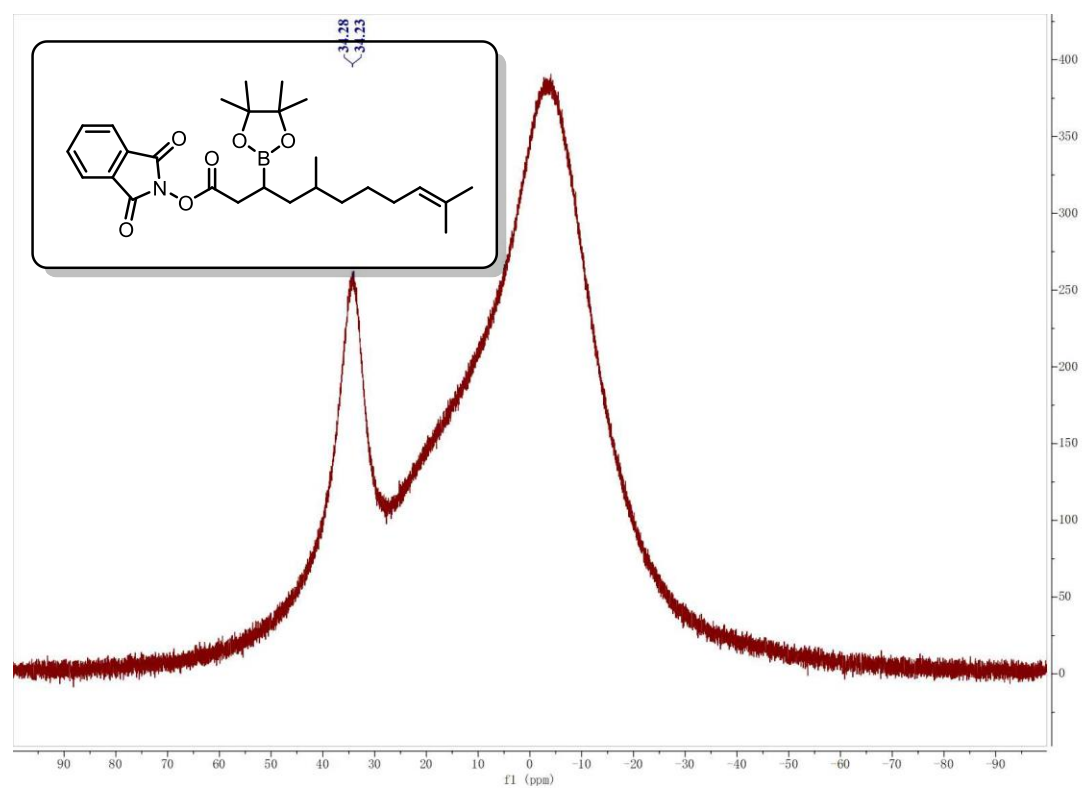
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

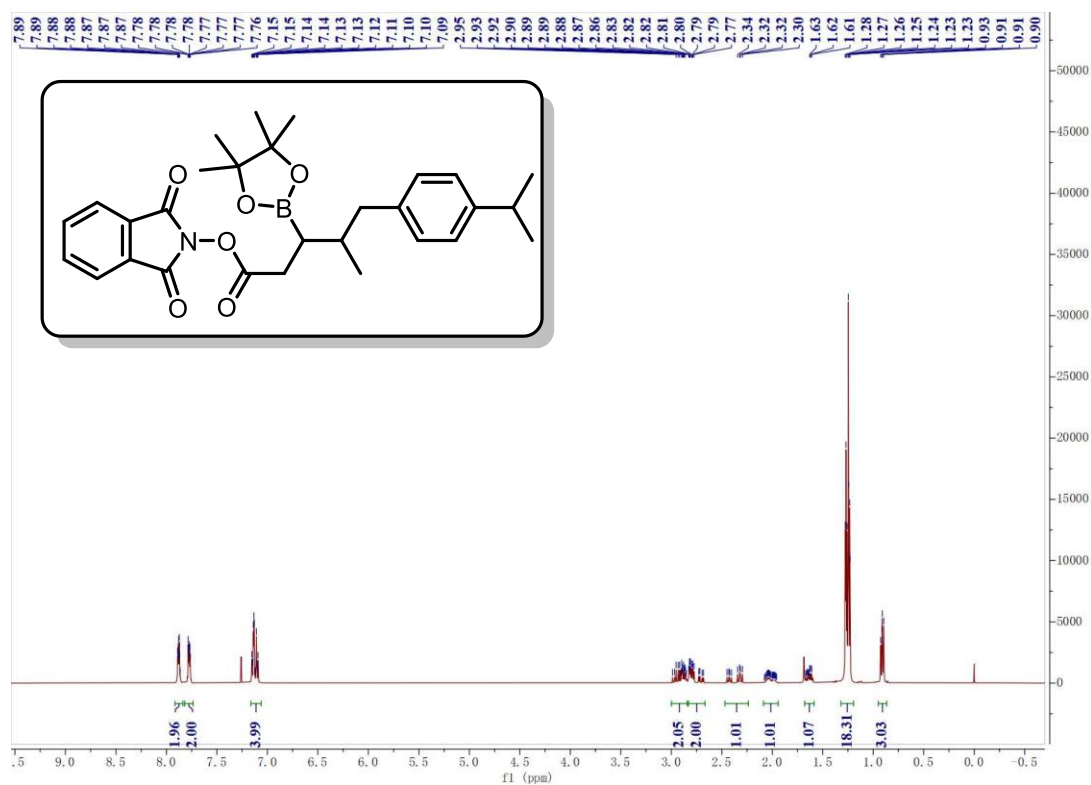


^{11}B NMR (160 MHz, Chloroform-*d*)

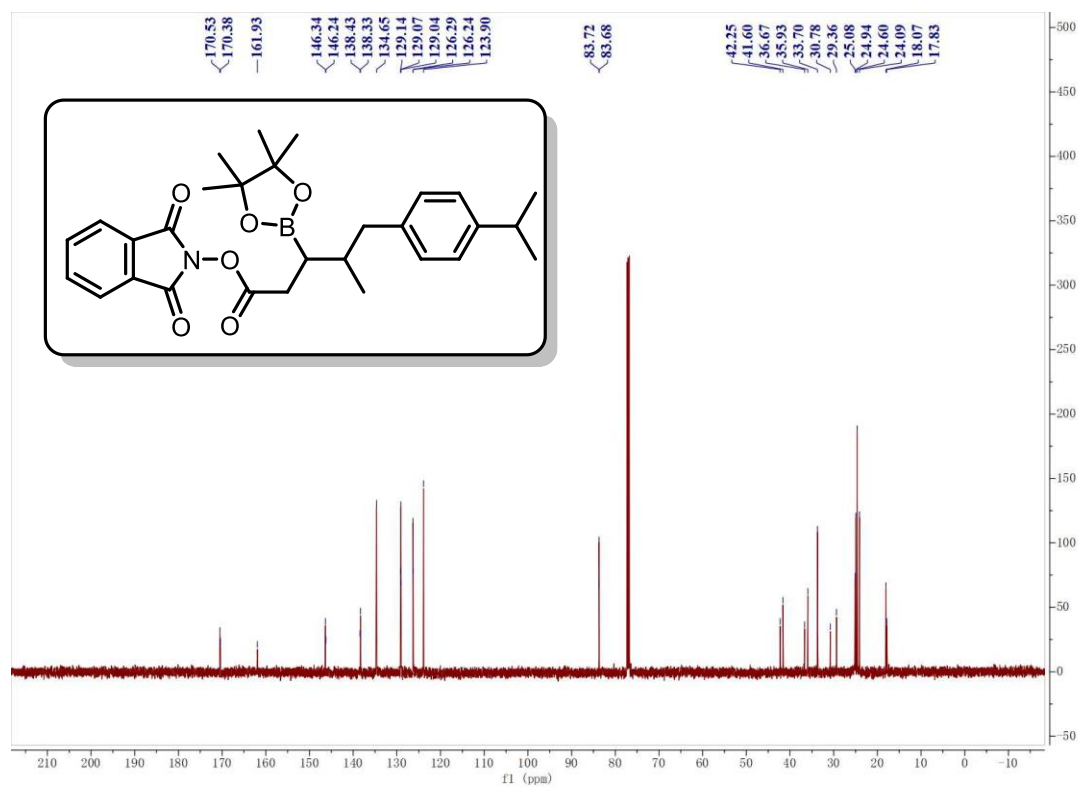


1,3-dioxoisindolin-2-yl 5-(4-isopropylphenyl)-4-methyl-3-(4,4,5,5-tetramethyl-1,3-dioxaborolan-2-yl)pentanoate (1i)

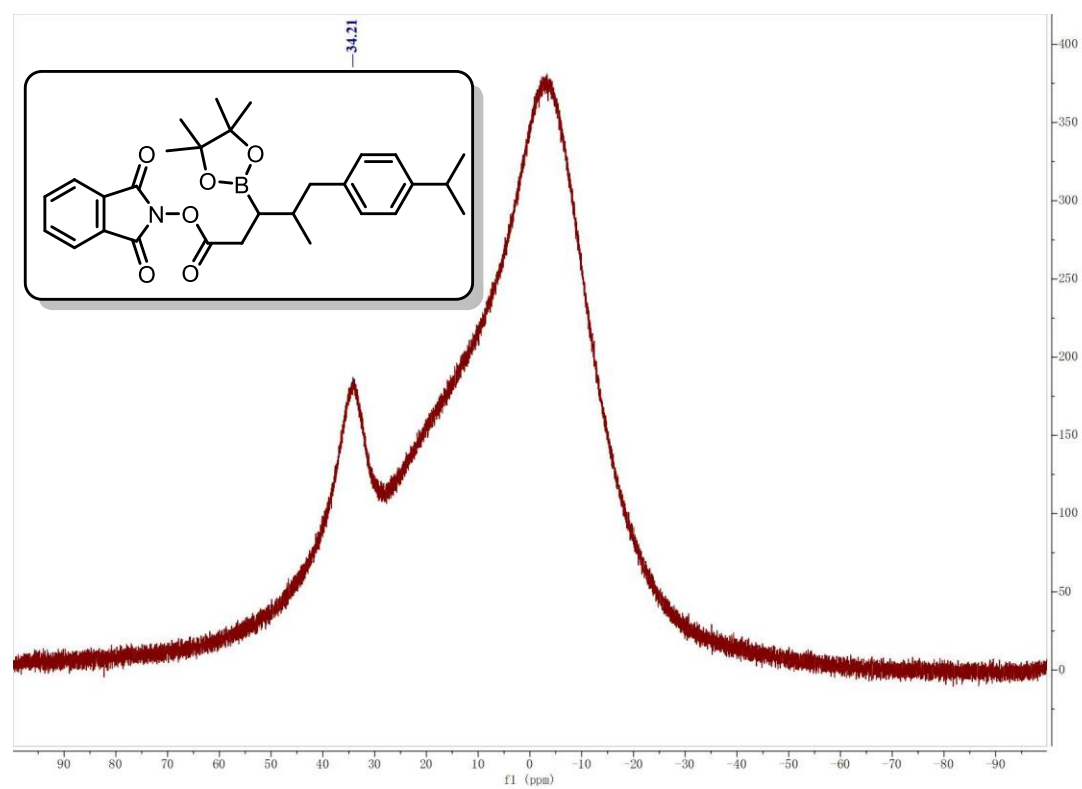
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

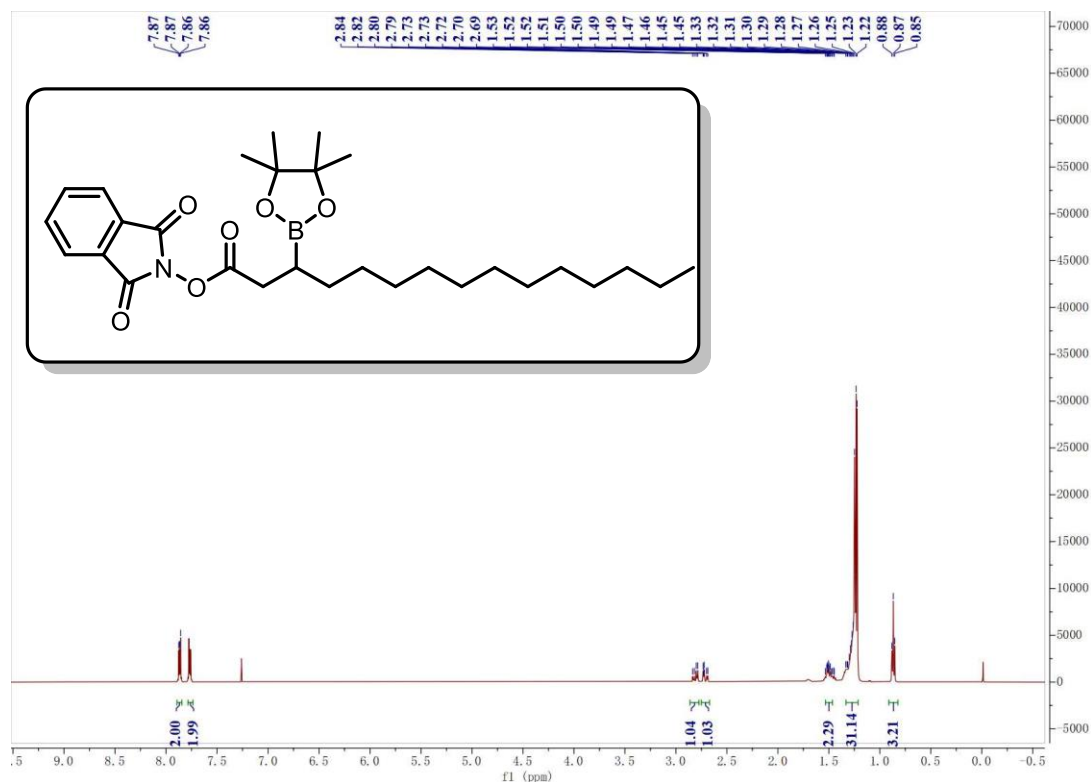


^{11}B NMR (160 MHz, Chloroform-*d*)

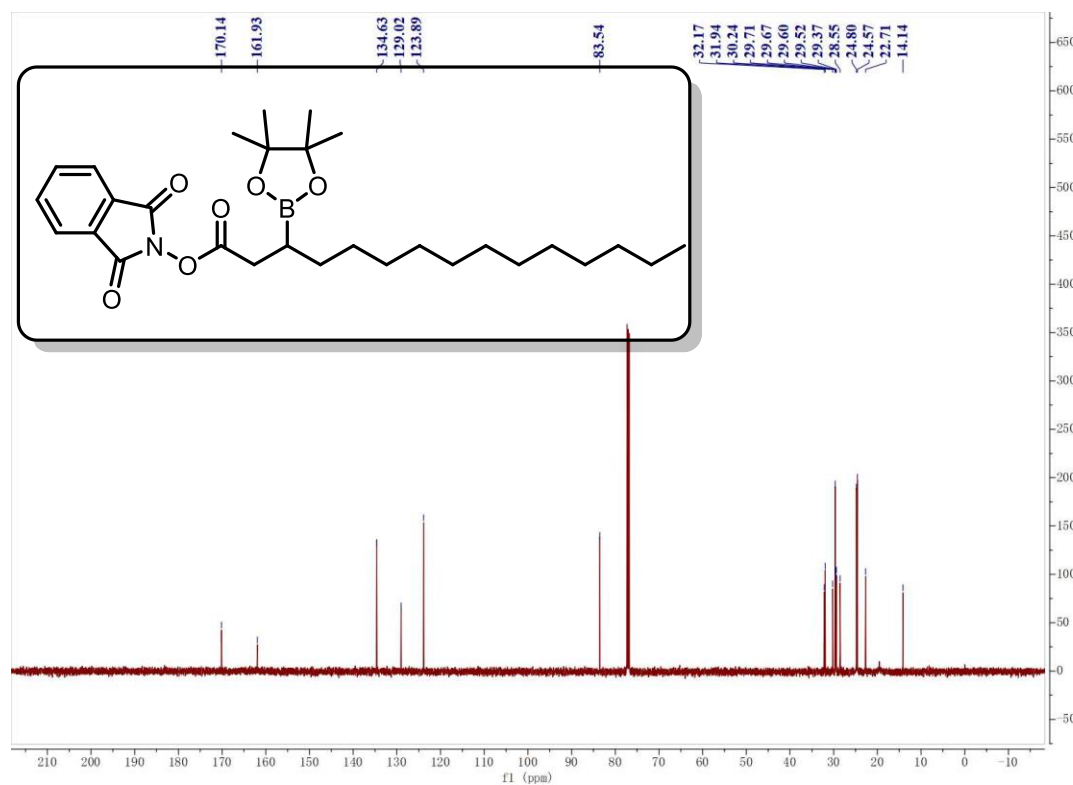


1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentadecanoate (1j)

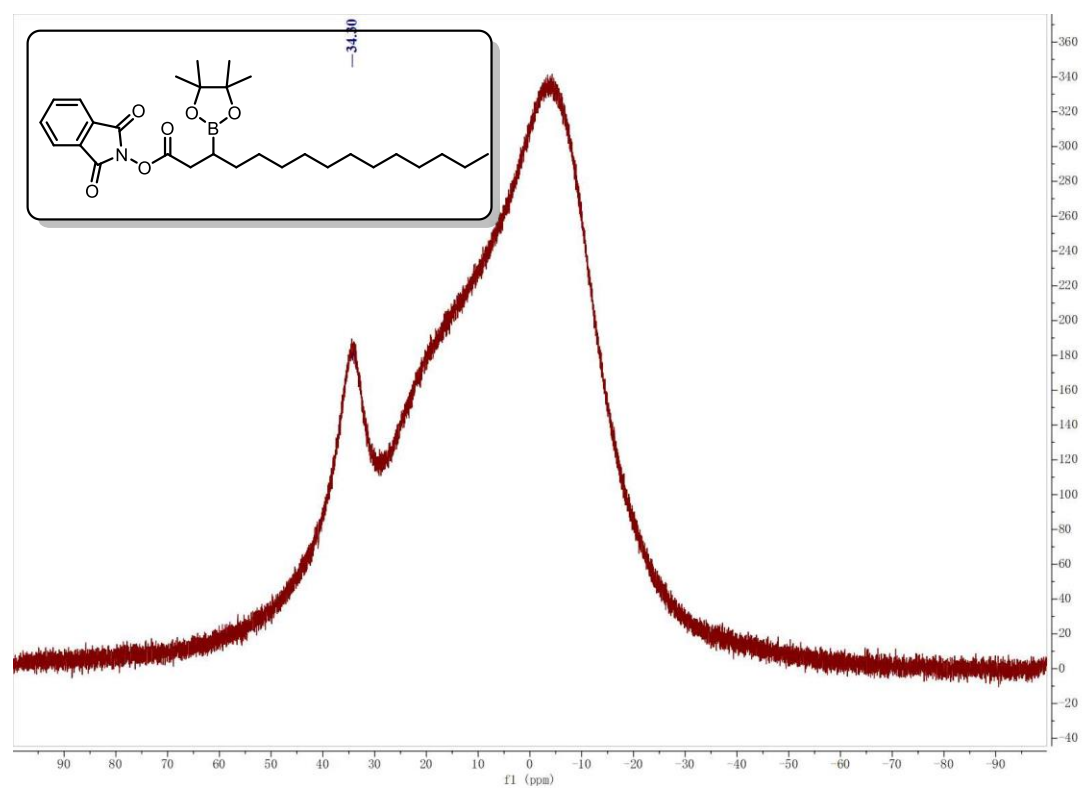
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

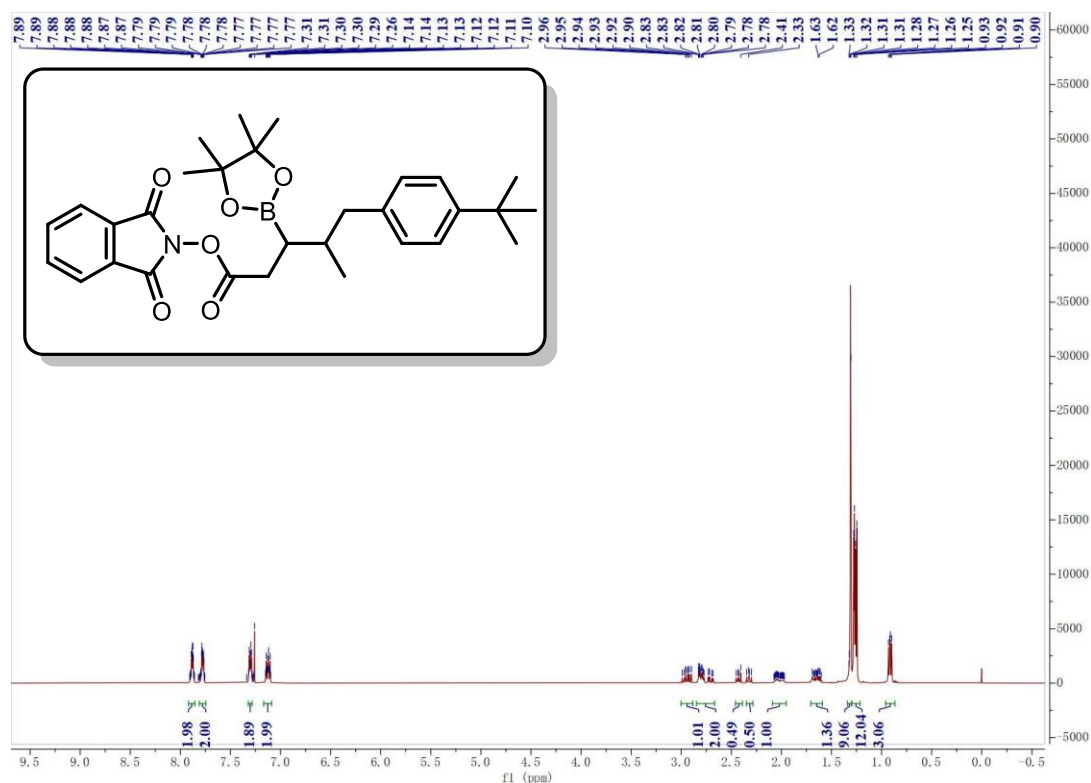


^{11}B NMR (160 MHz, Chloroform-*d*)

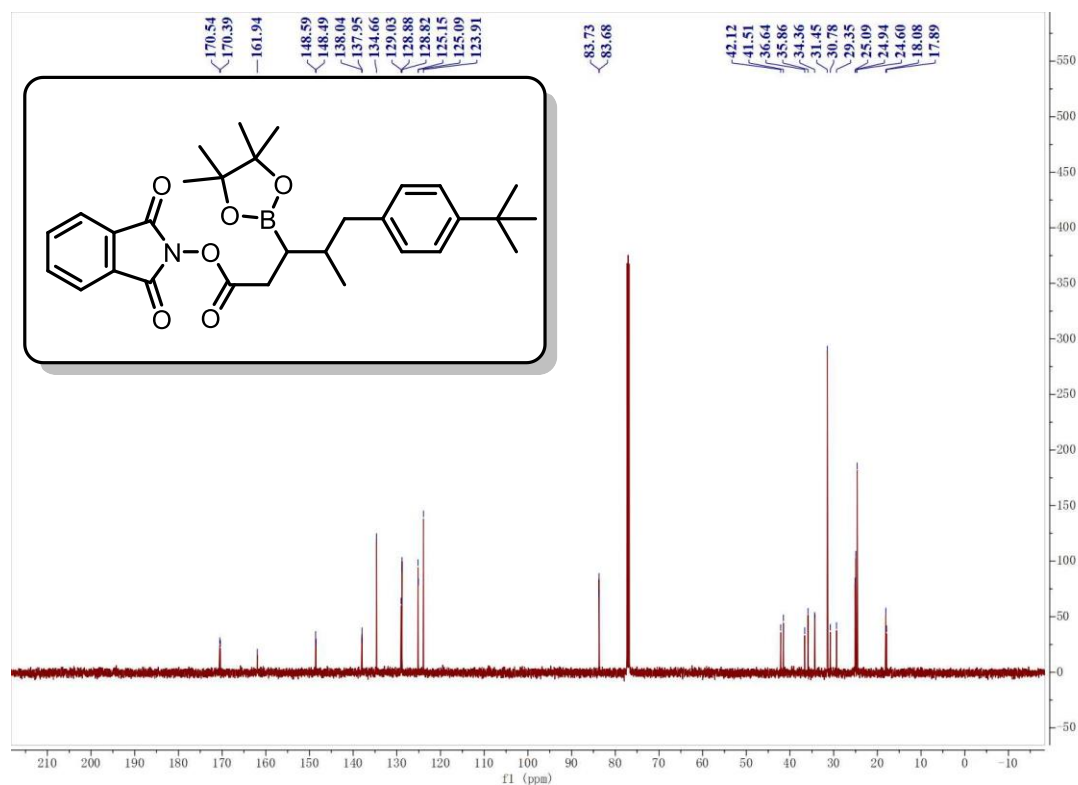


1,3-dioxoisindolin-2-yl 5-(4-(tert-butyl)phenyl)-4-methyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1k)

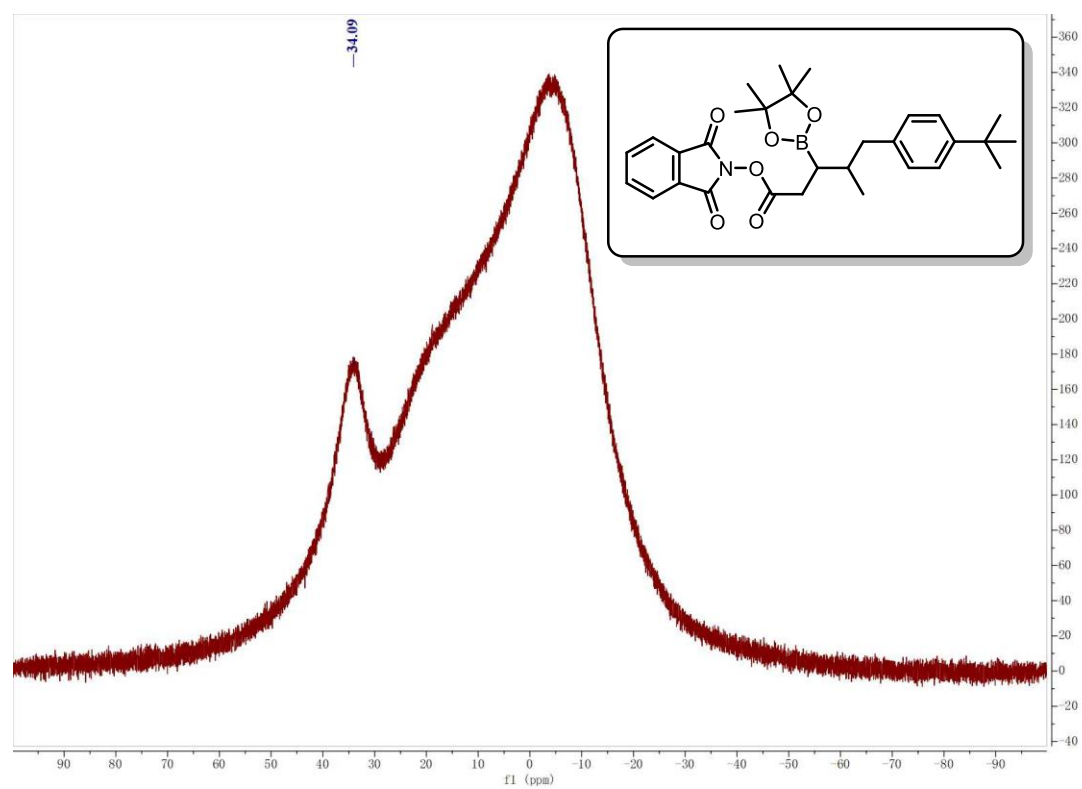
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

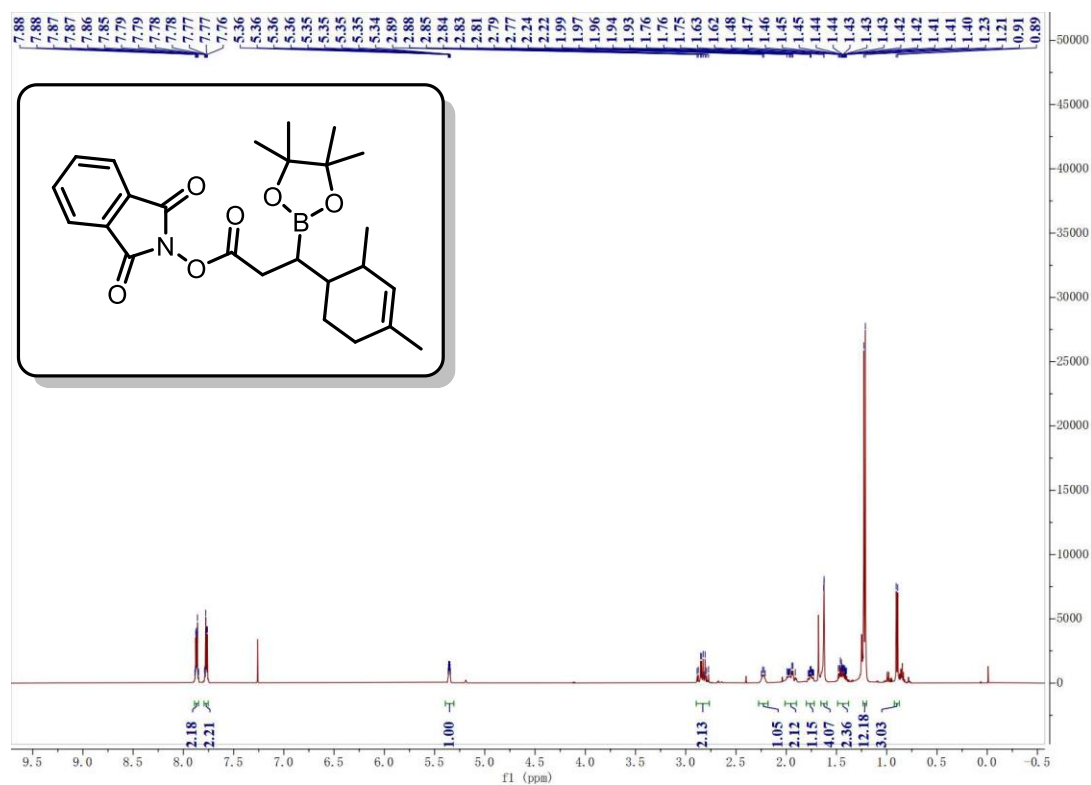


^{11}B NMR (160 MHz, Chloroform-*d*)

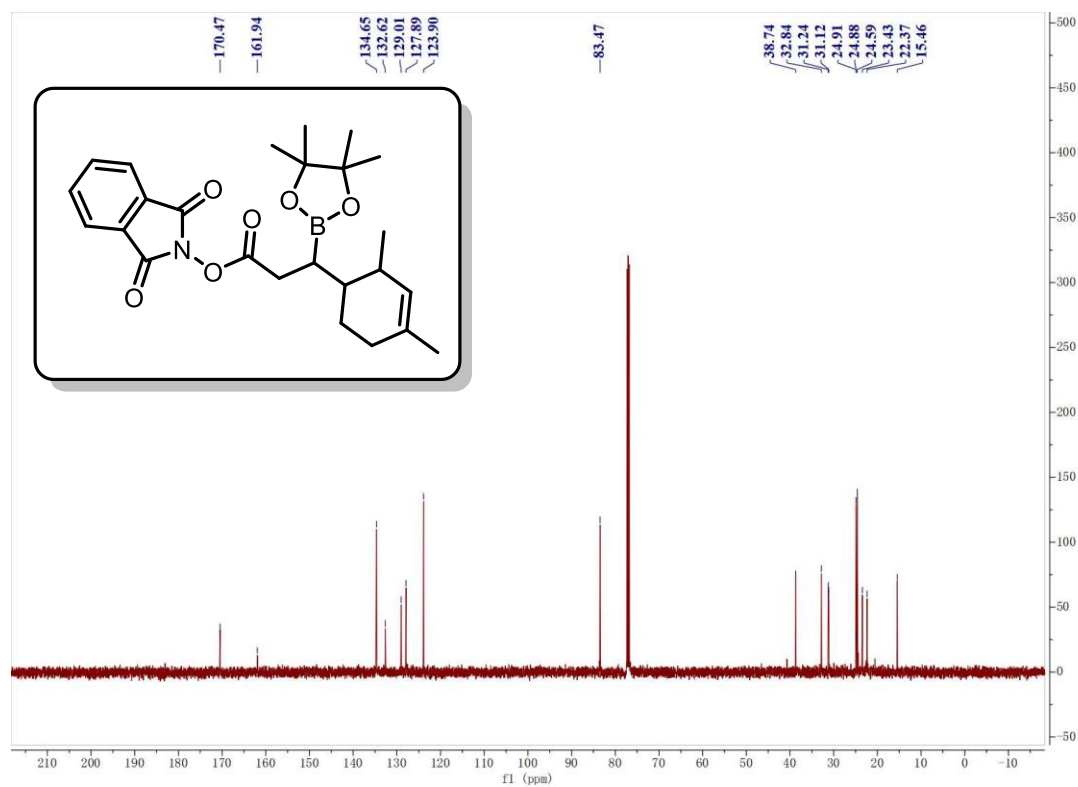


1,3-dioxoisindolin-2-yl 3-(2,4-dimethylcyclohex-3-en-1-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate (11)

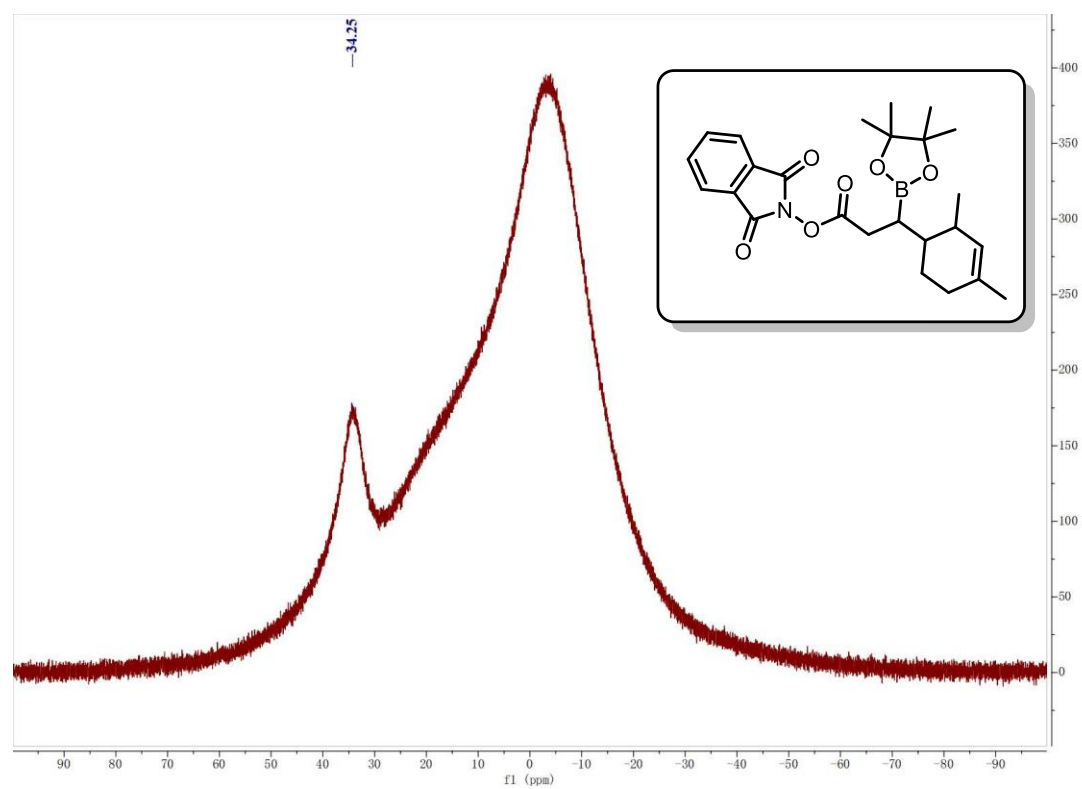
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

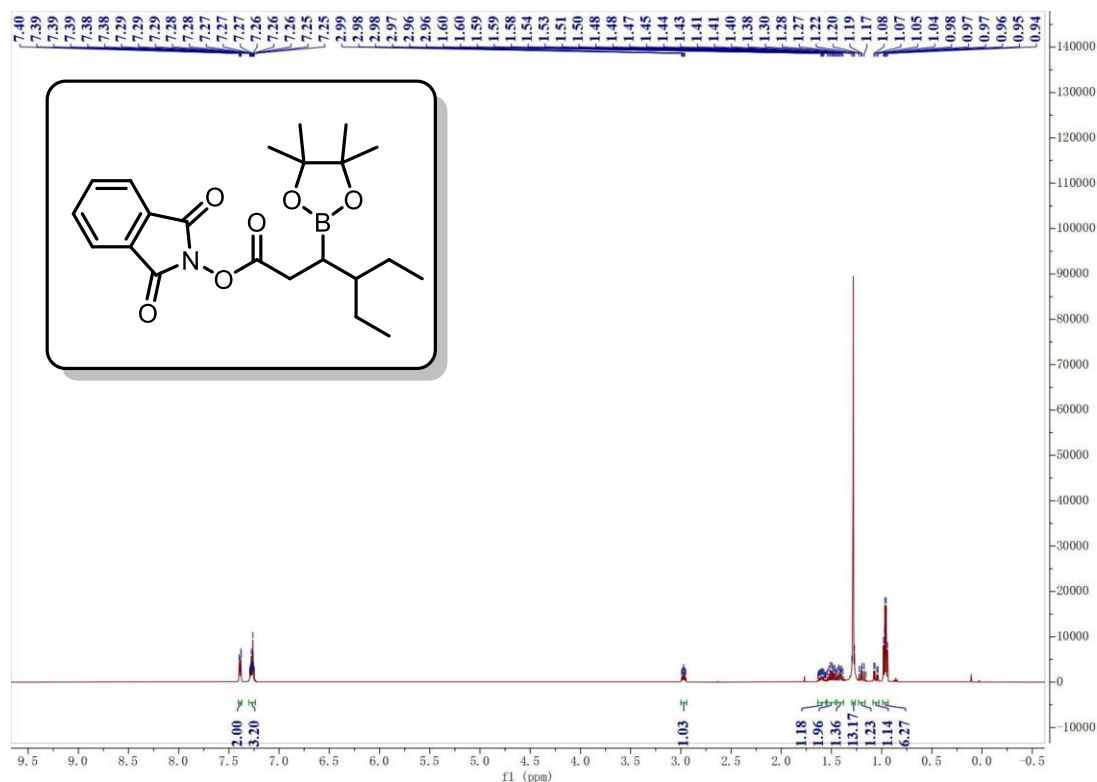


^{11}B NMR (160 MHz, Chloroform-*d*)

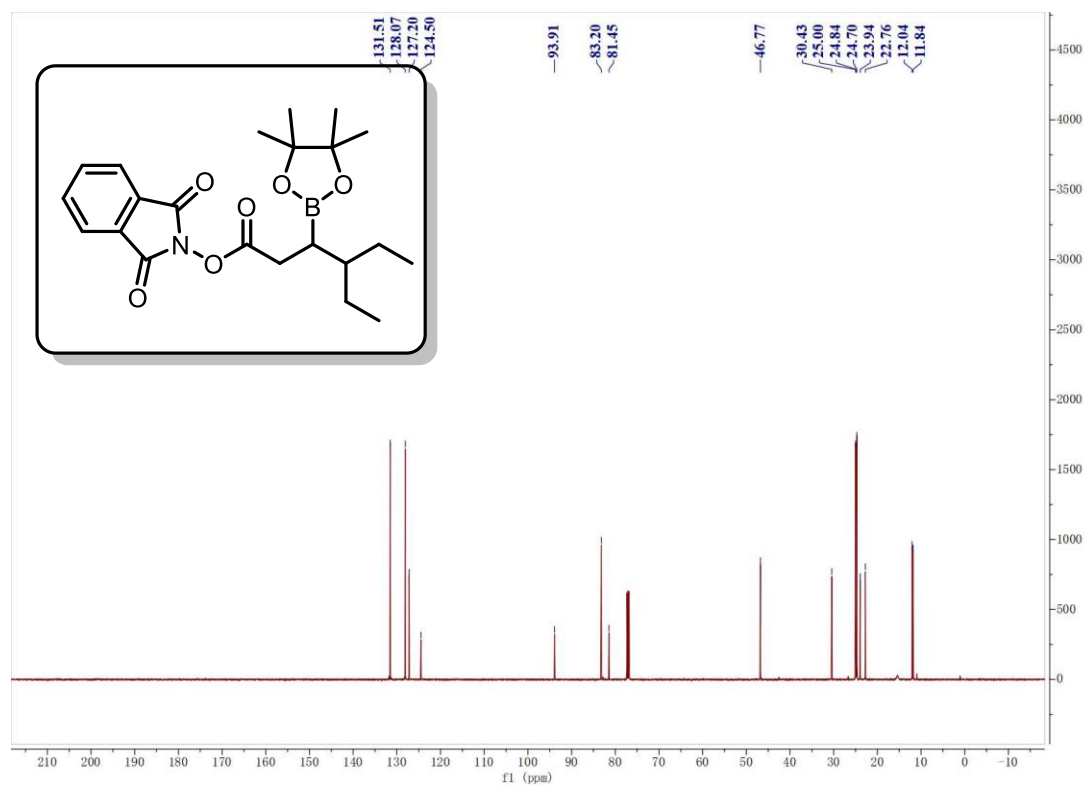


1,3-dioxoisindolin-2-yl 4-ethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1m)

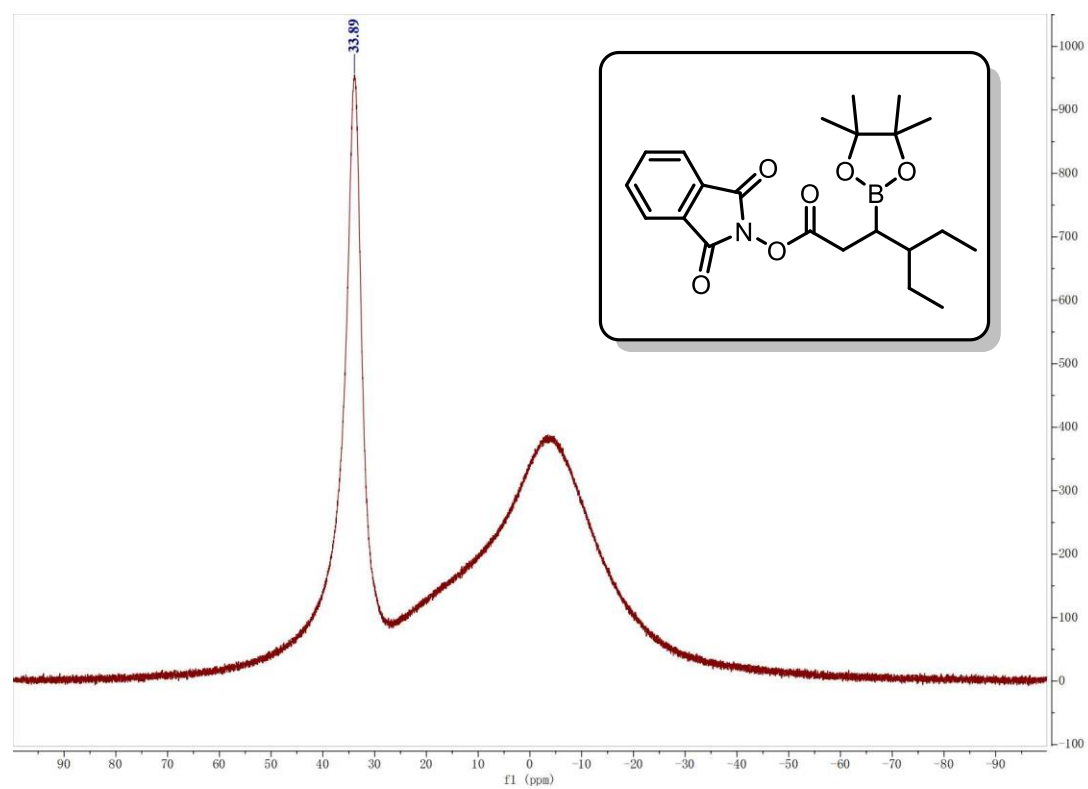
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

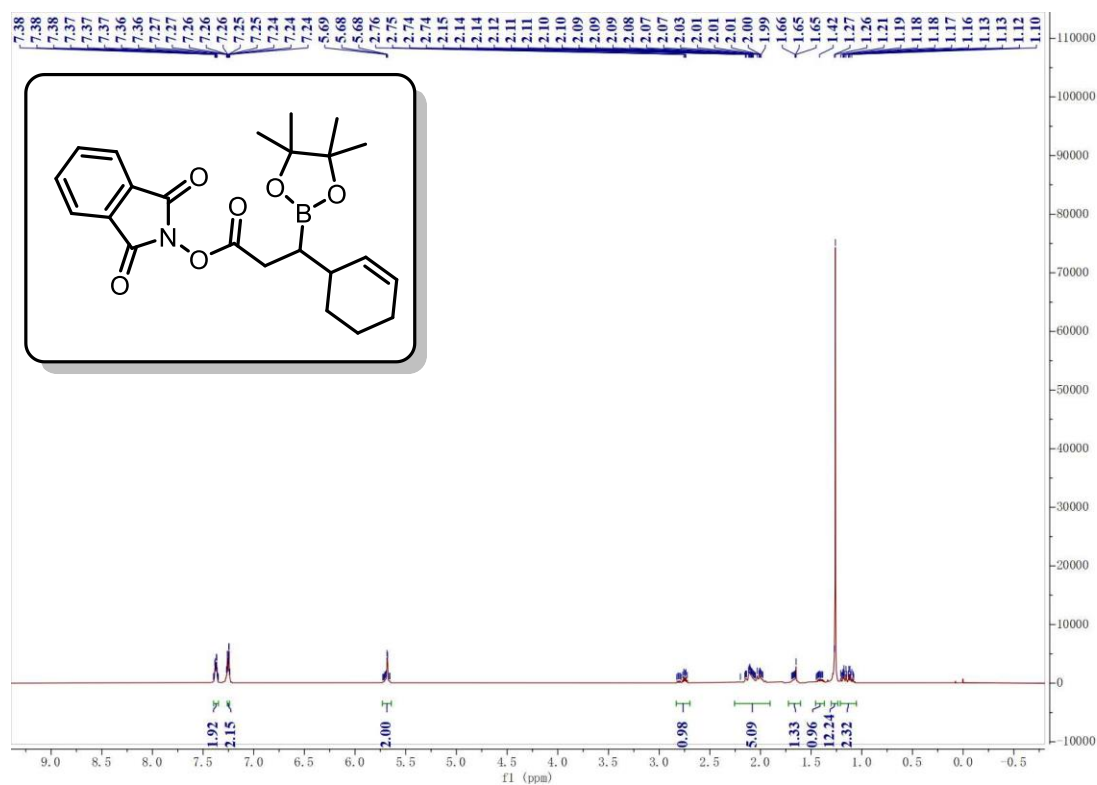


^{11}B NMR (160 MHz, Chloroform-*d*)

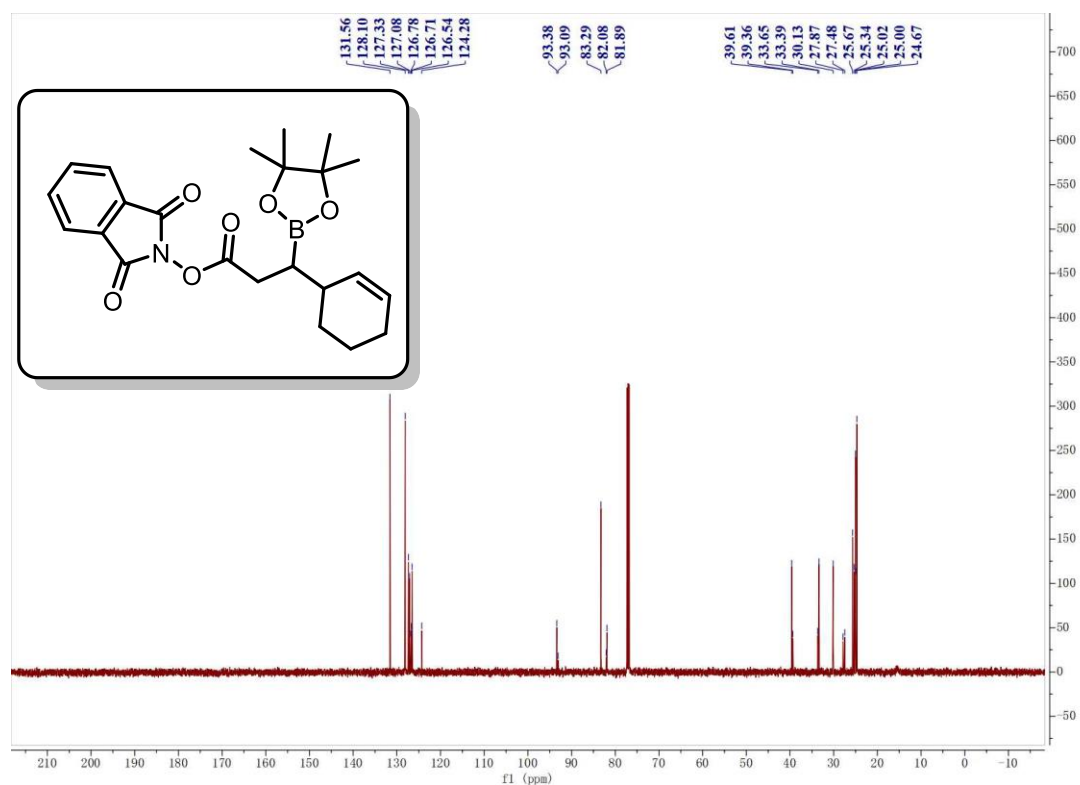


1,3-dioxoisindolin-2-yl 3-(cyclohex-2-en-1-yl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propanoate (1n)

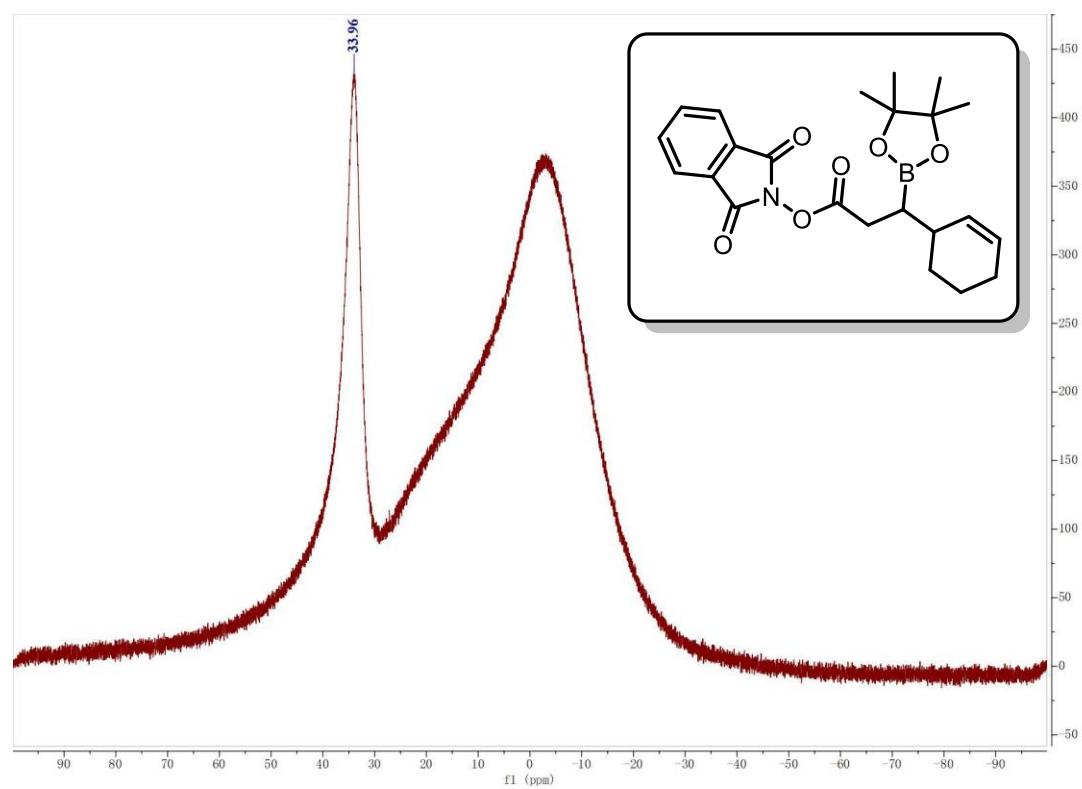
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

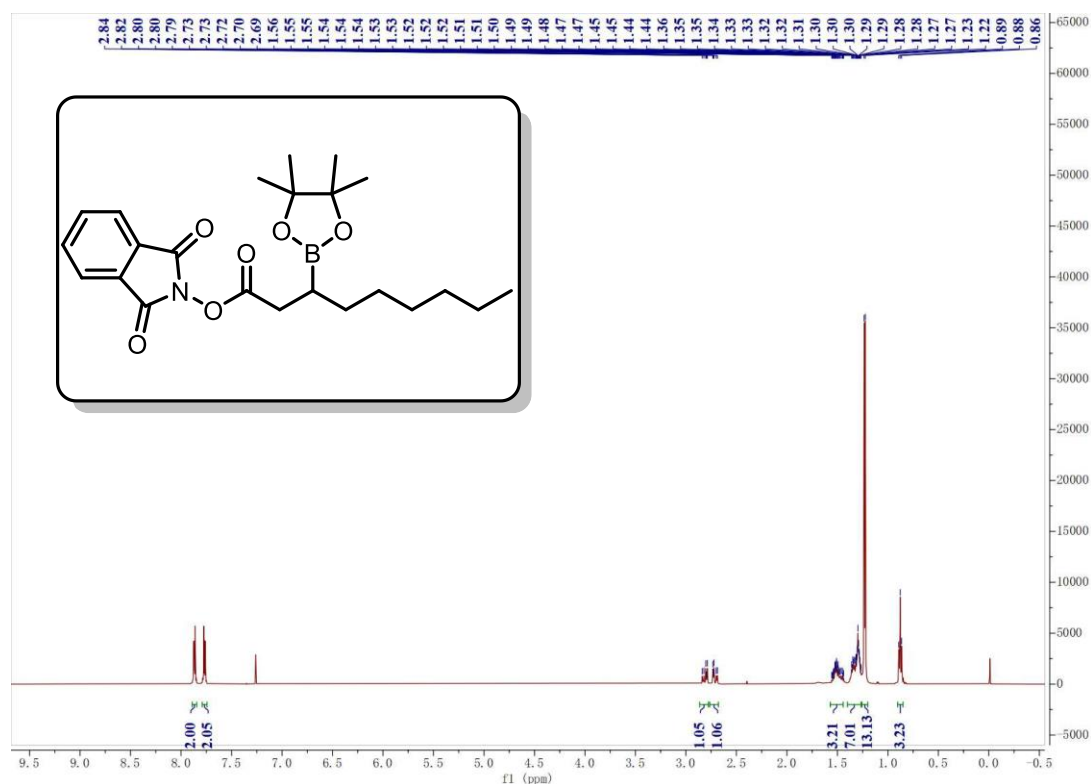


^{11}B NMR (160 MHz, Chloroform-*d*)

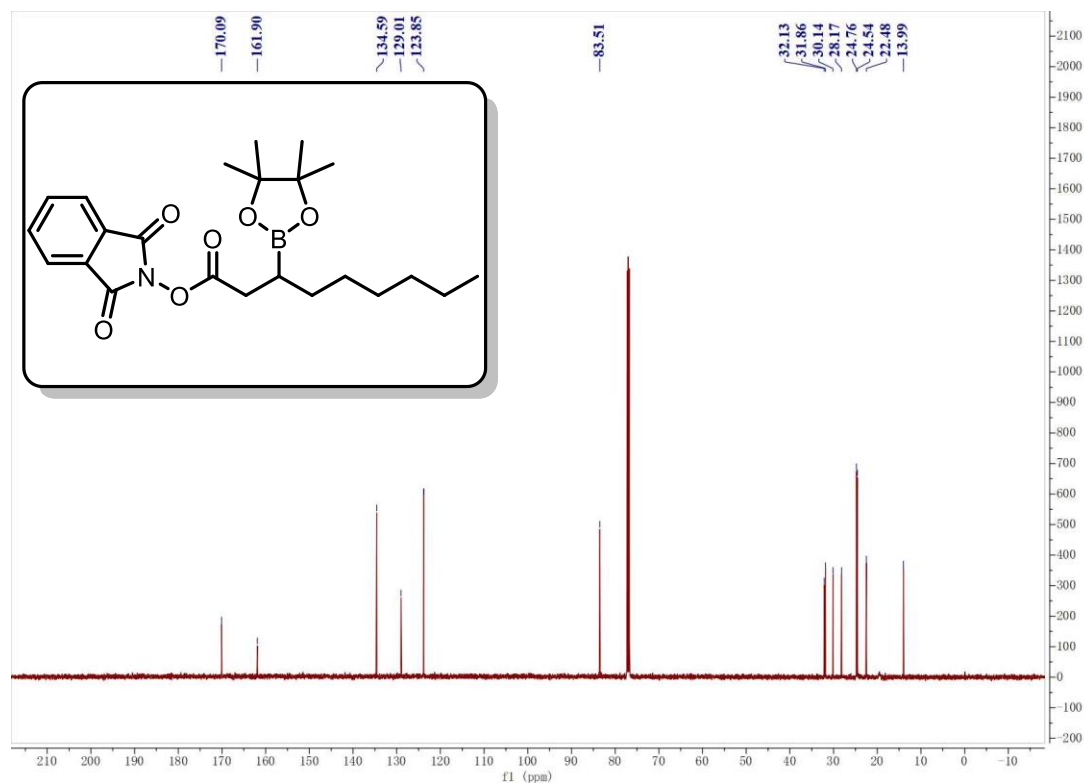


1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)nonanoate (1o)

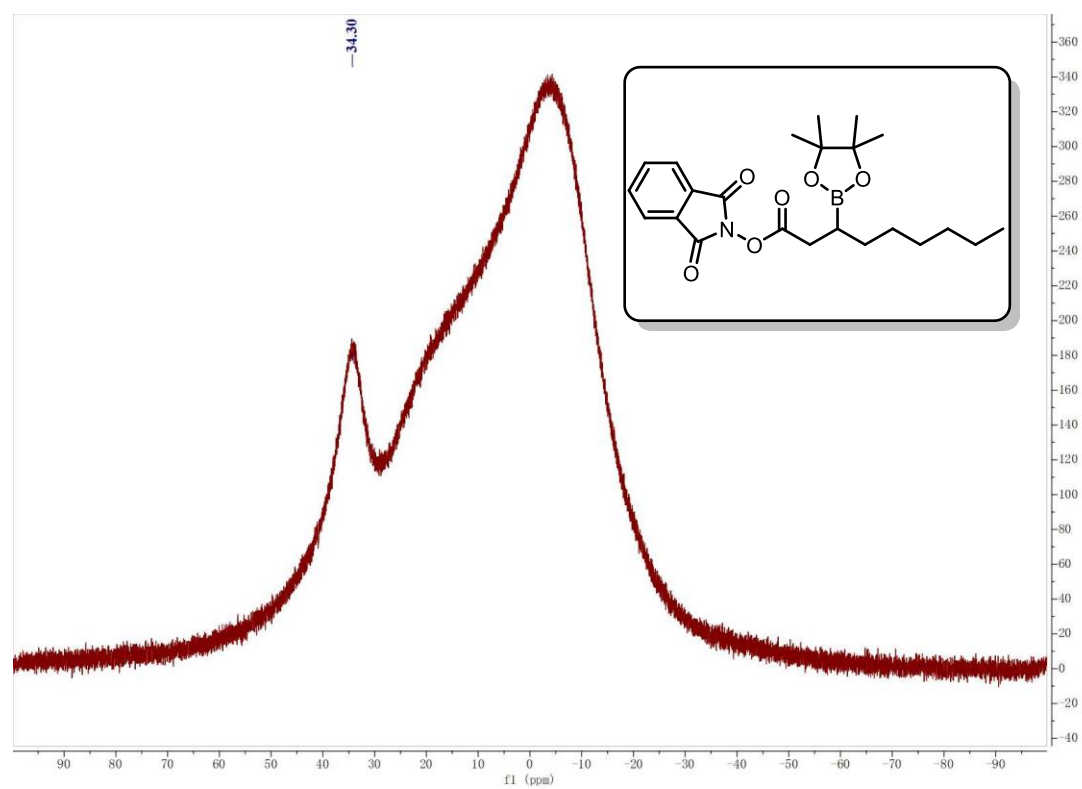
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

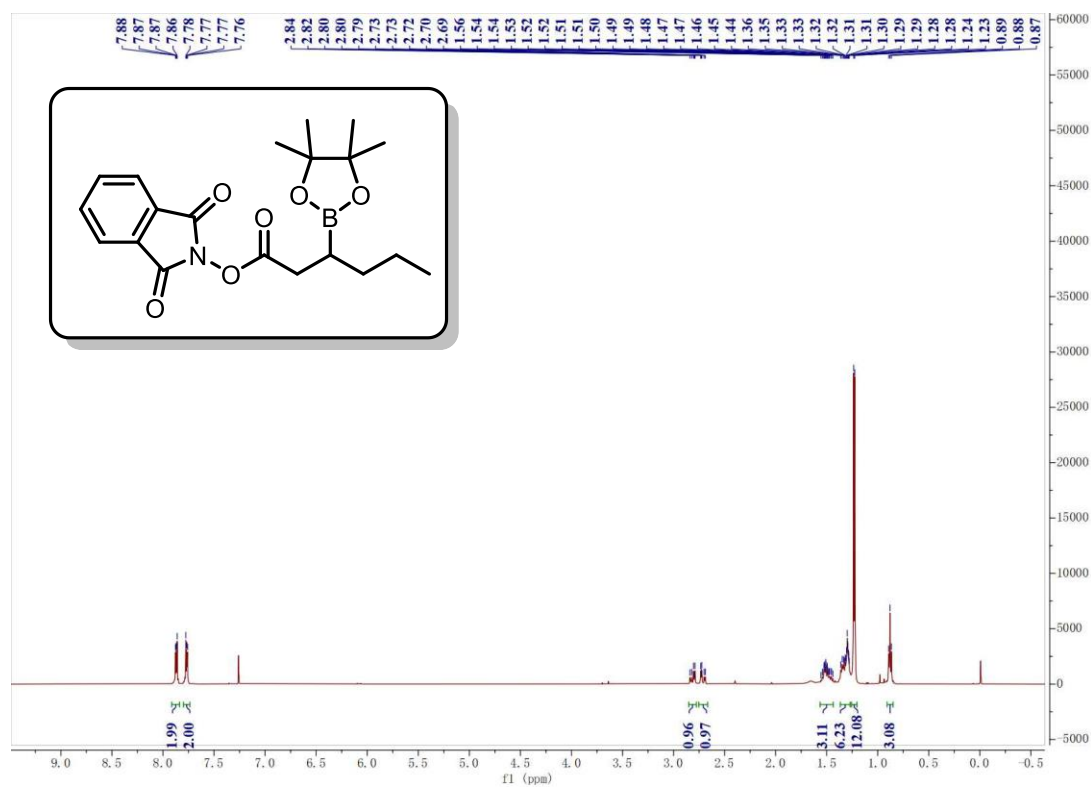


^{11}B NMR (160 MHz, Chloroform-*d*)

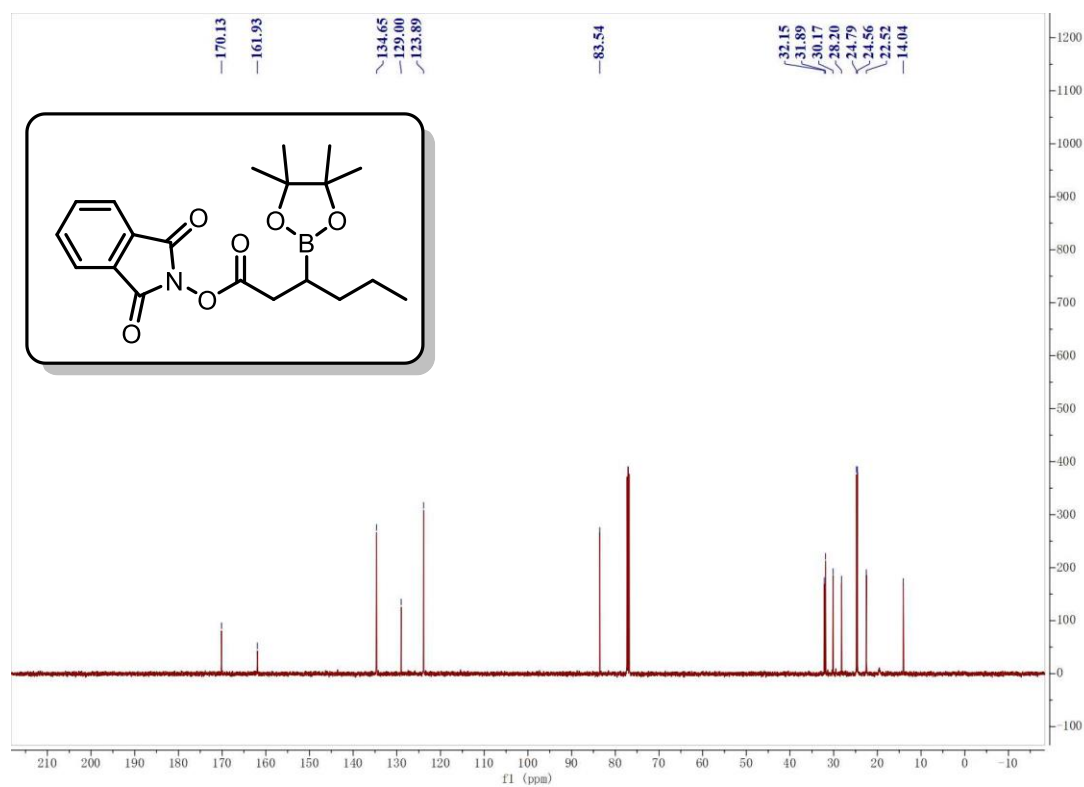


1,3-dioxoisindolin-2-yl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanoate (1p)

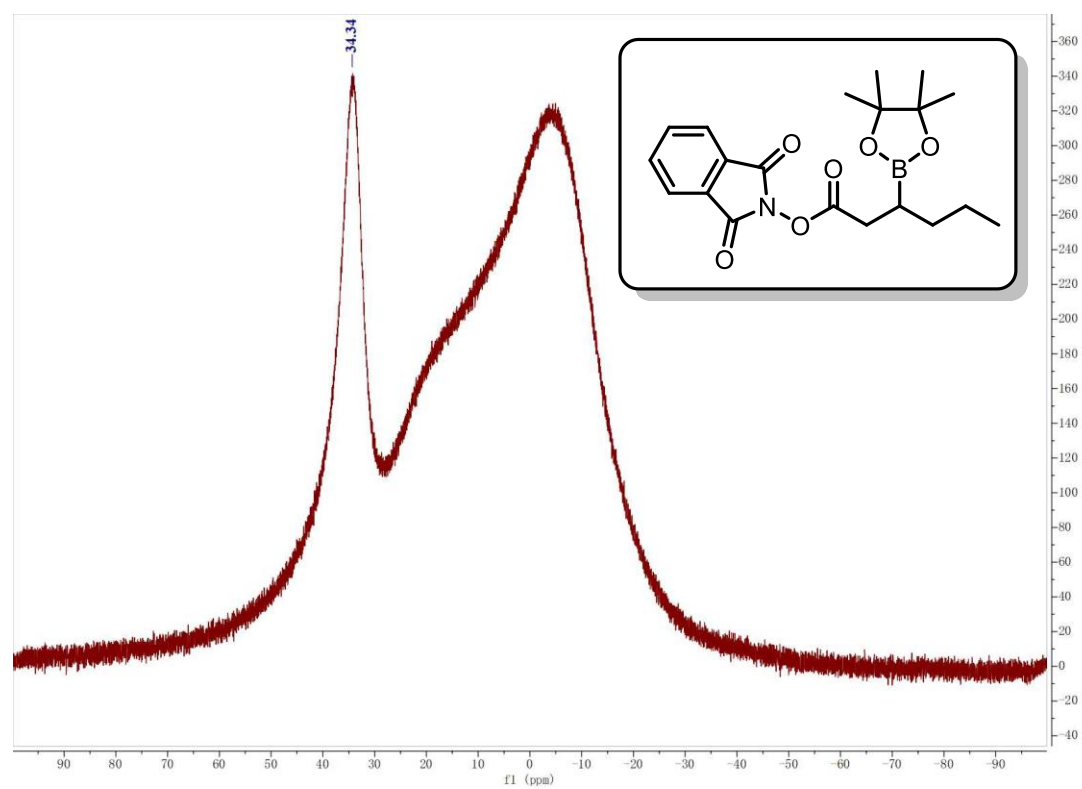
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

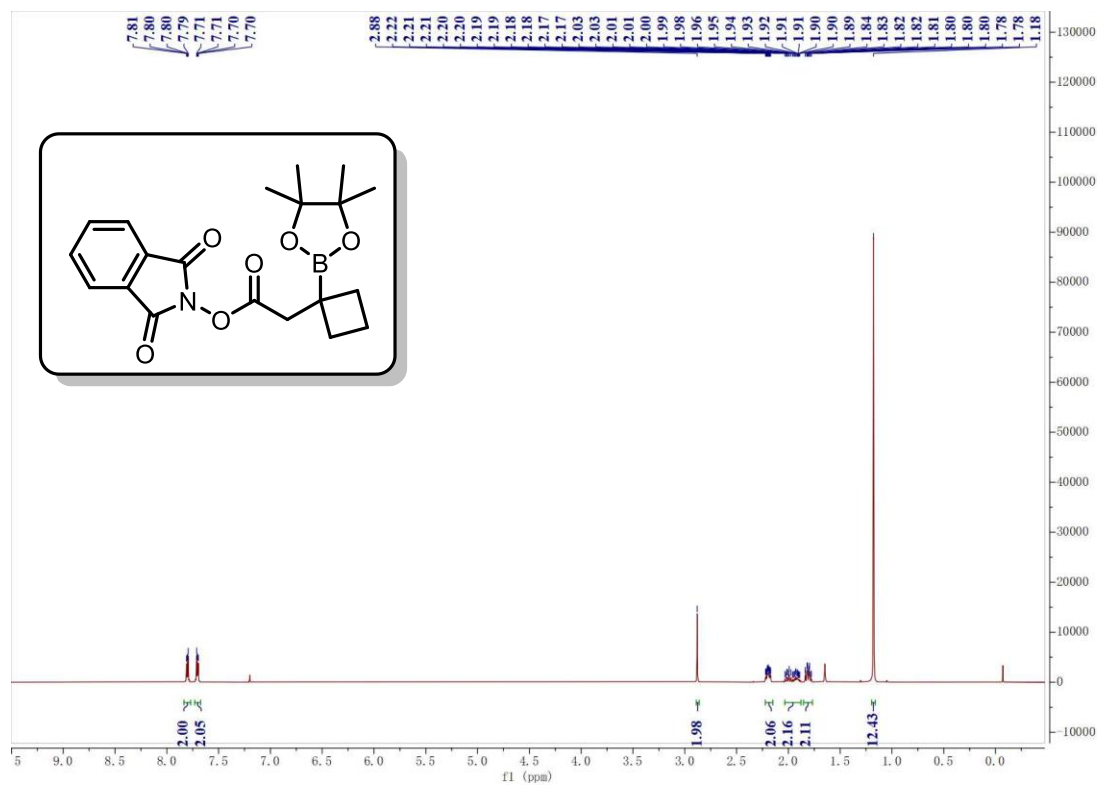


^{11}B NMR (160 MHz, Chloroform-*d*)

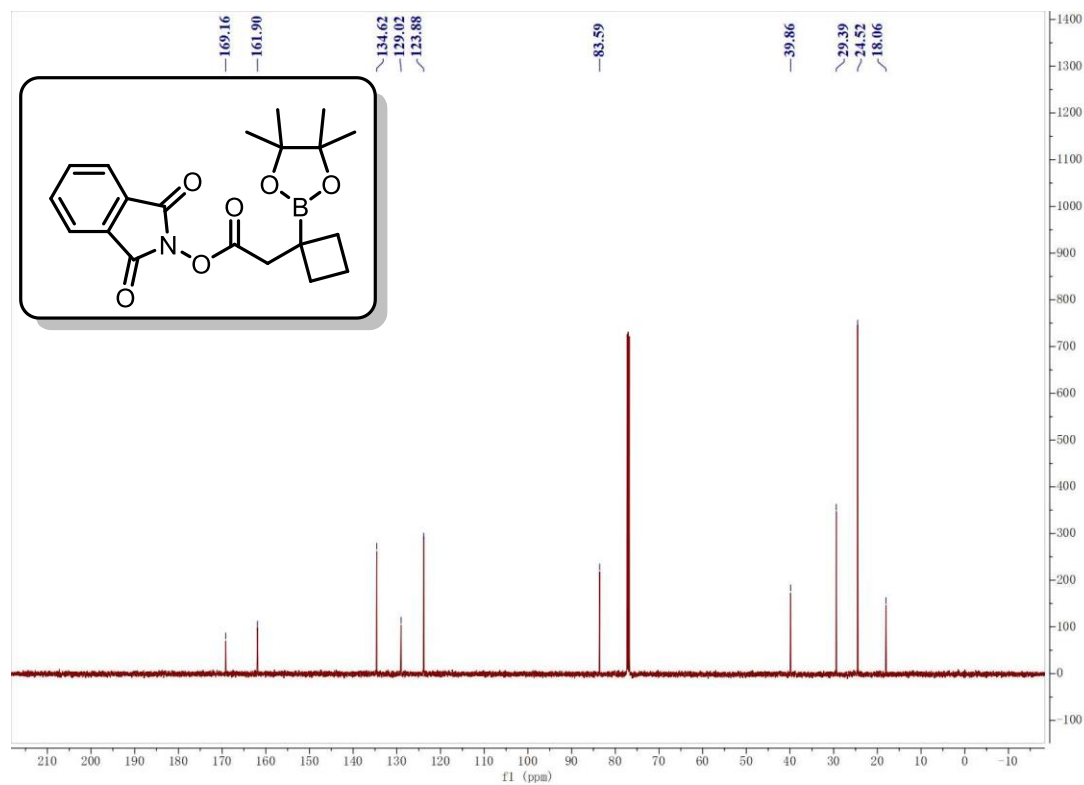


1,3-dioxoisindolin-2-yl 2-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)cyclobutyl)acetate (1q)

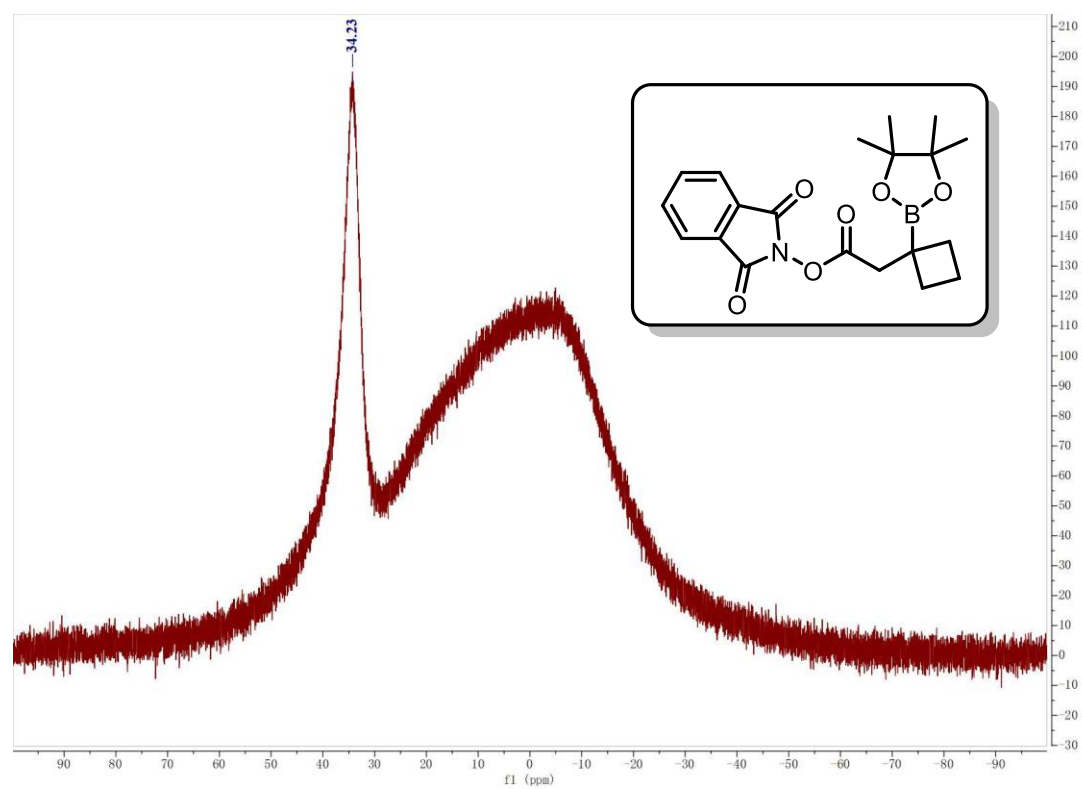
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

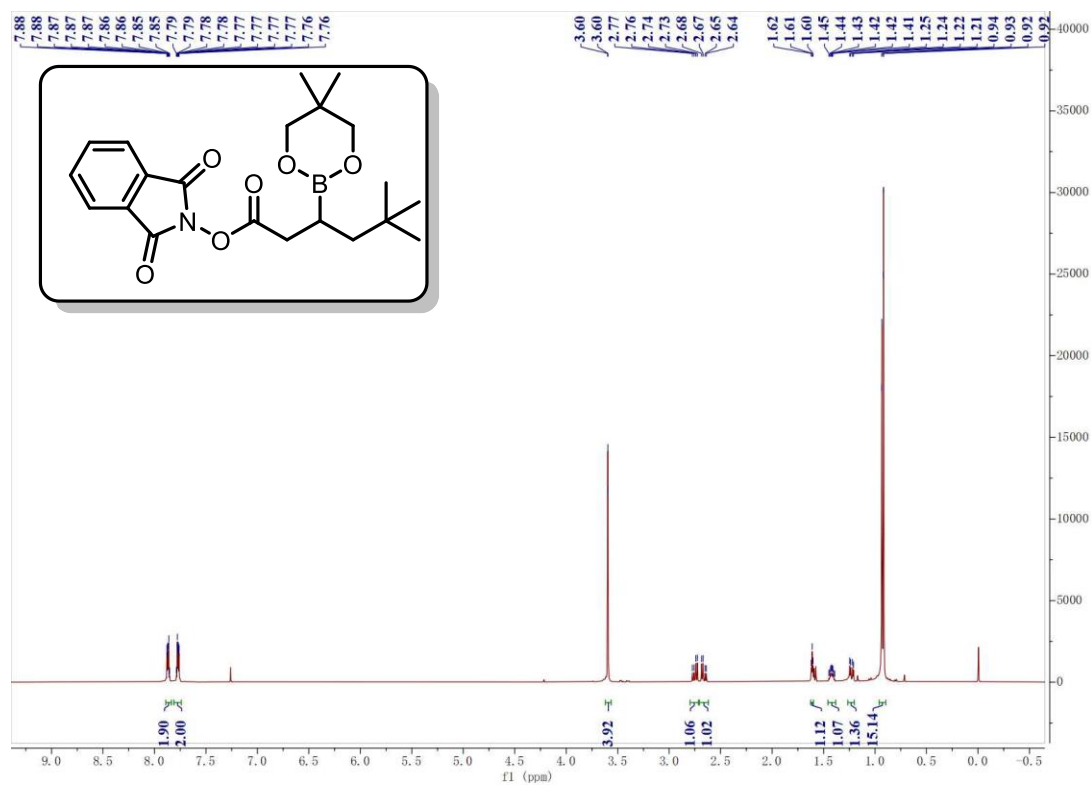


^{11}B NMR (160 MHz, Chloroform-*d*)

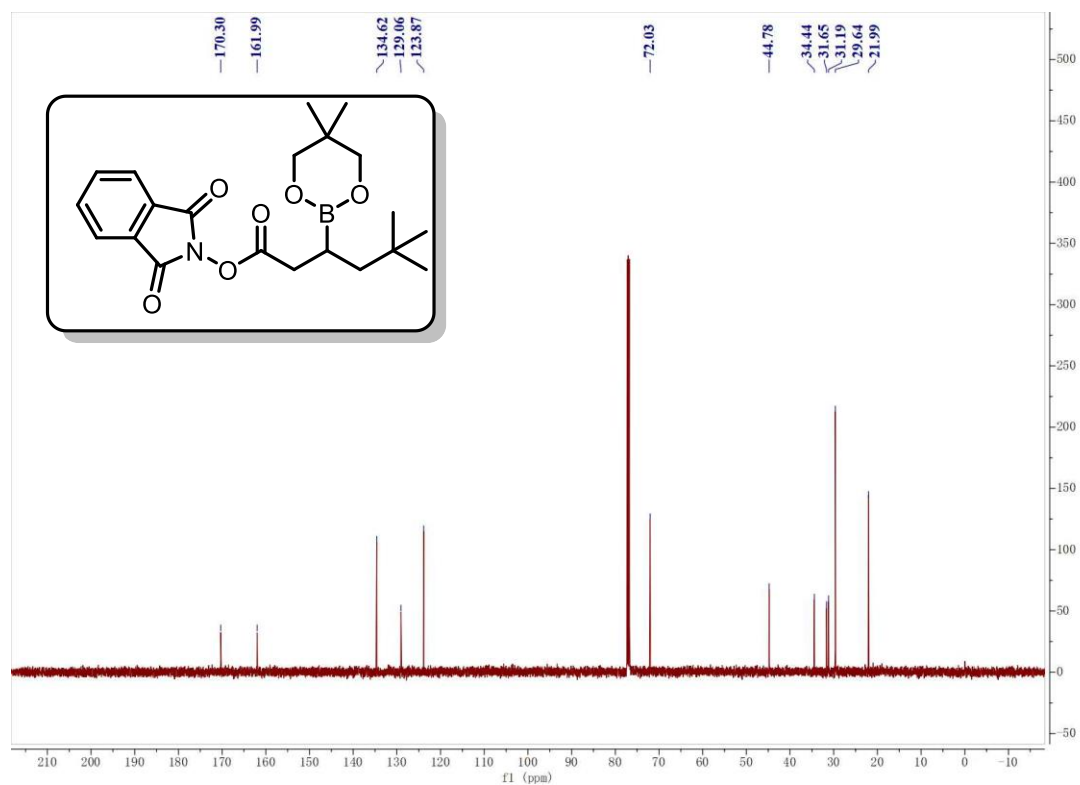


1,3-dioxoisindolin-2-yl 3-(5,5-dimethyl-1,3,2-dioxaborinan-2-yl)-5,5-dimethylhexanoate (1r)

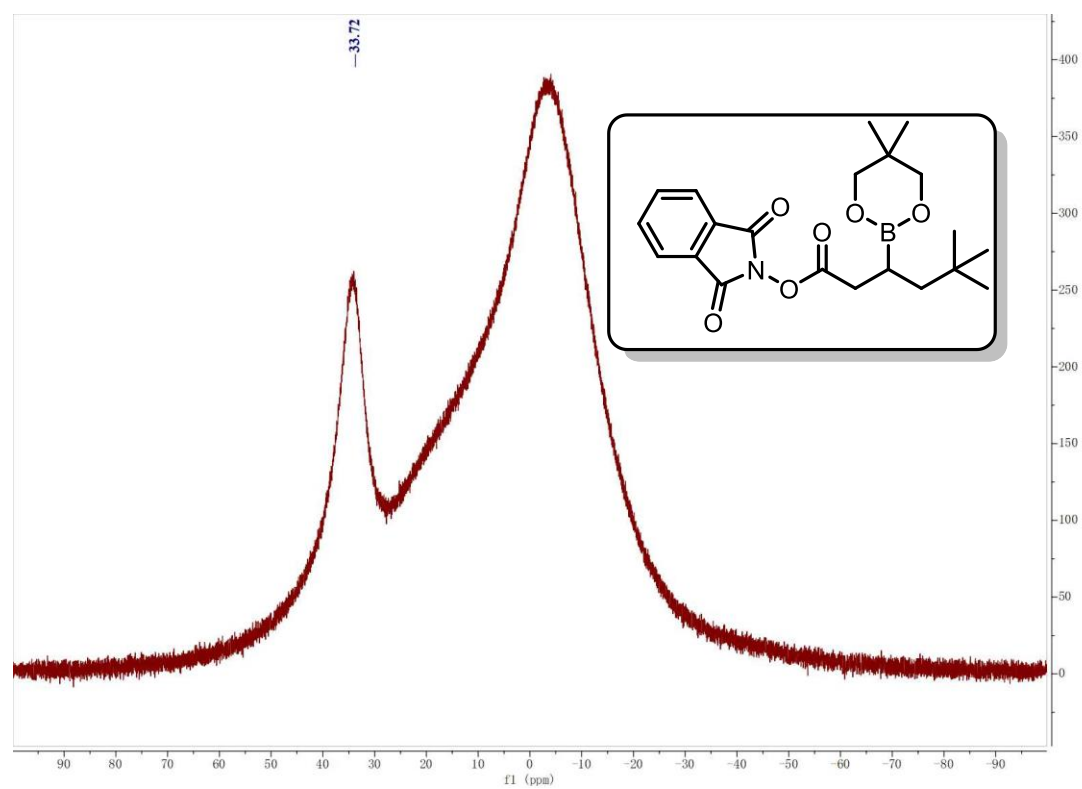
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

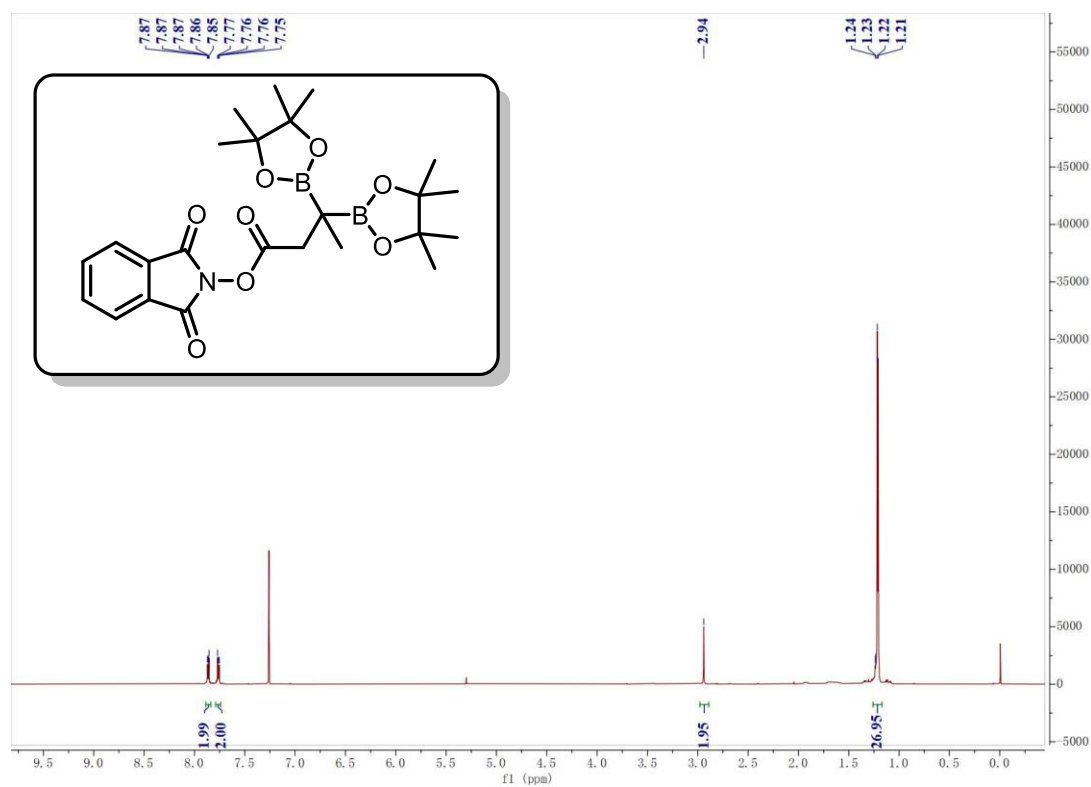


^{11}B NMR (160 MHz, Chloroform-*d*)

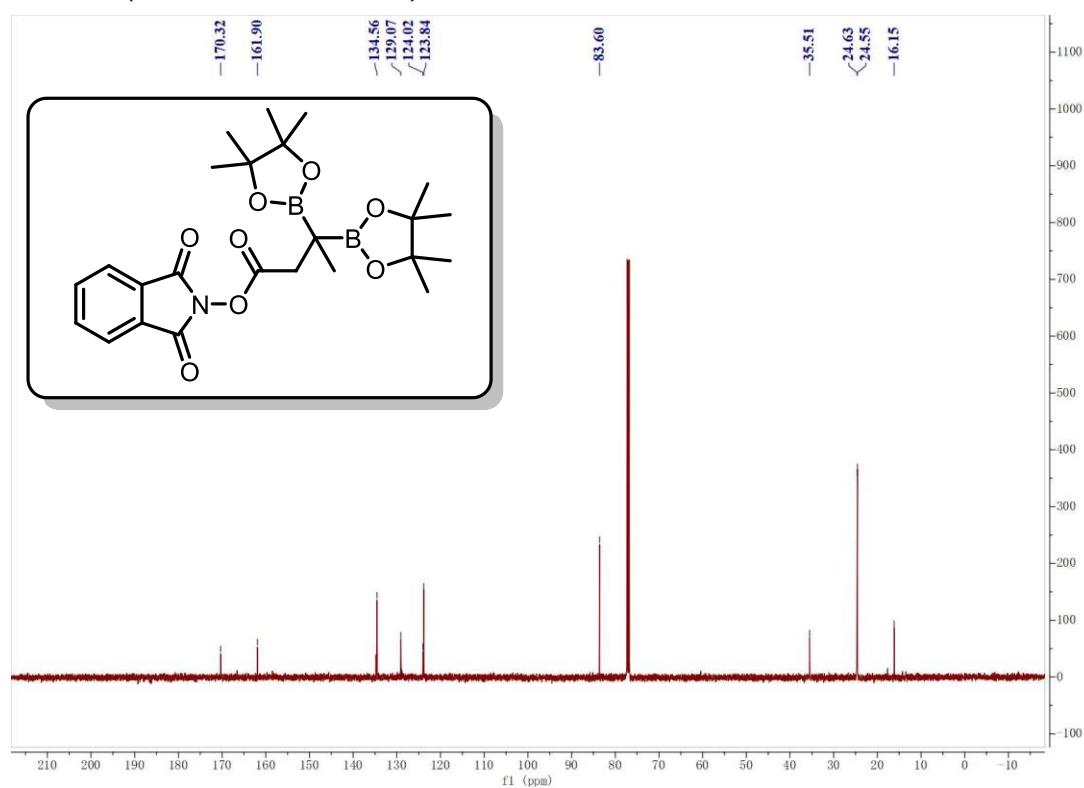


1,3-dioxoisindolin-2-yl 3,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)butanoate (1s)

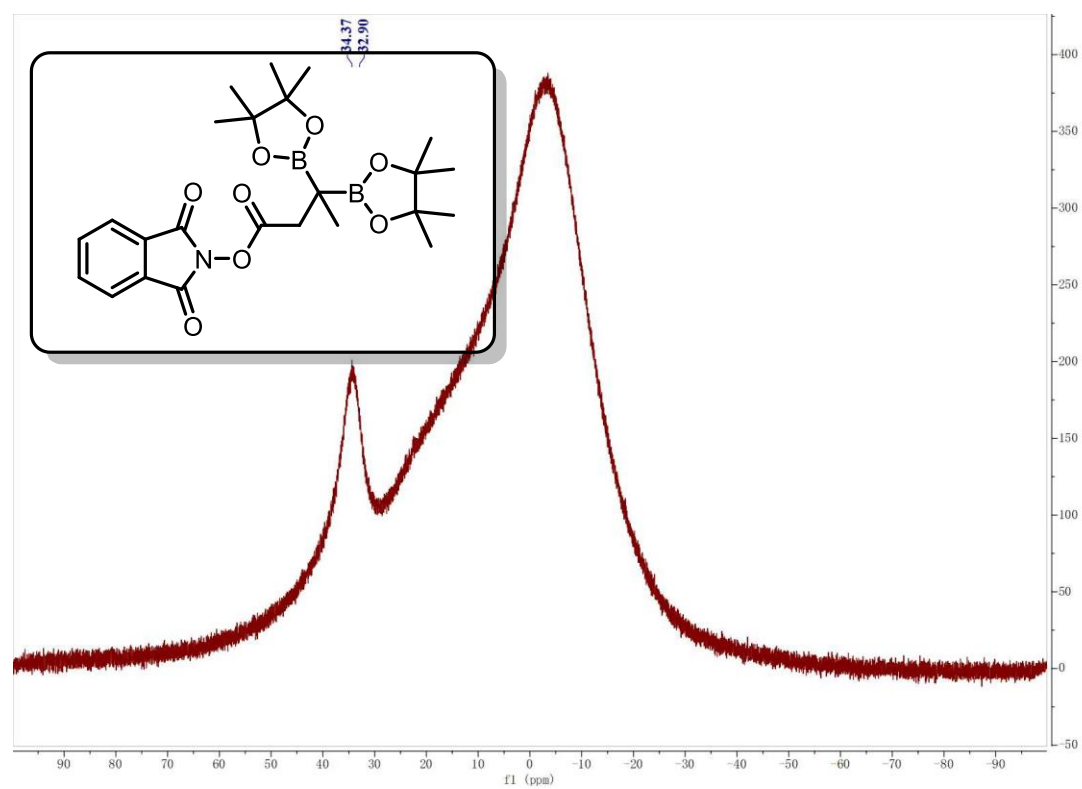
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

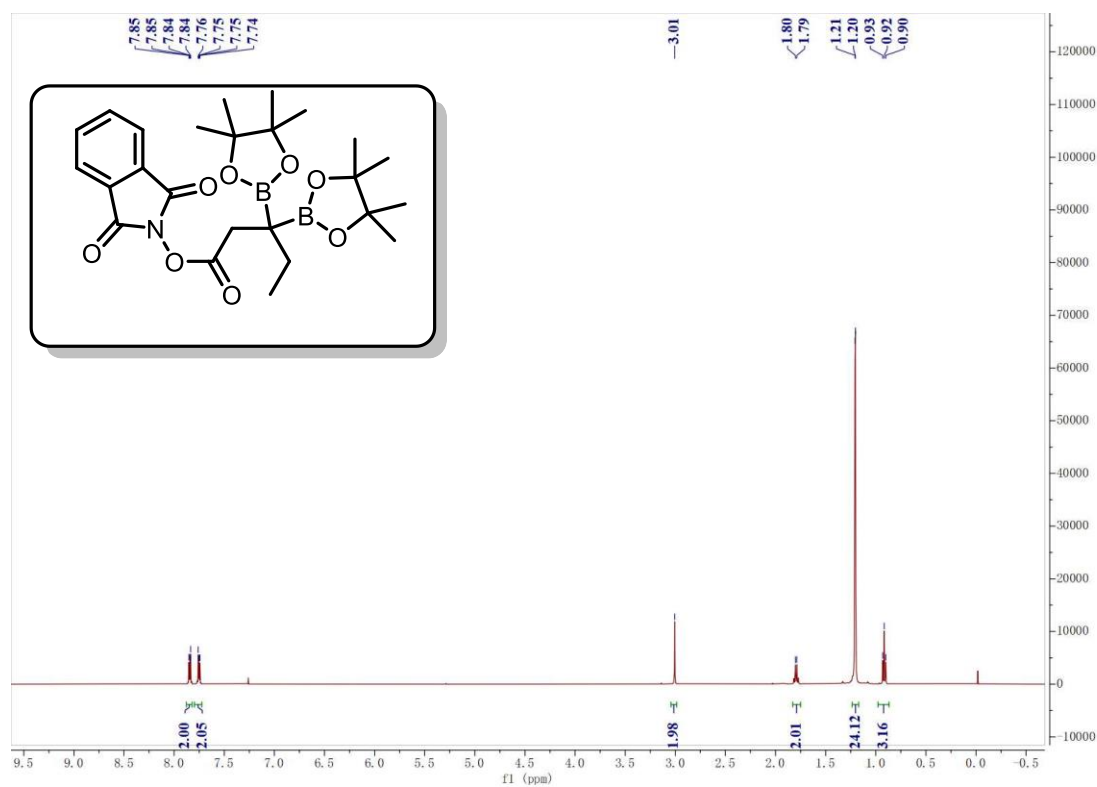


^{11}B NMR (160 MHz, Chloroform-*d*)

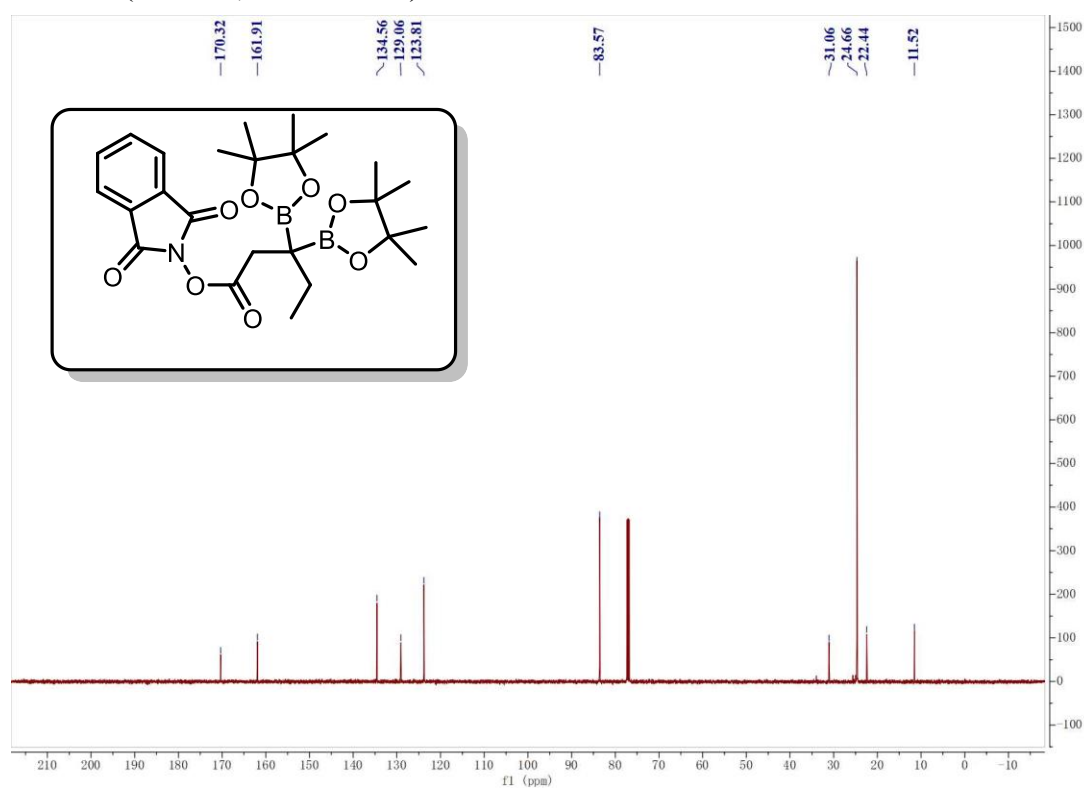


1,3-dioxoisindolin-2-yl 3,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (1t)

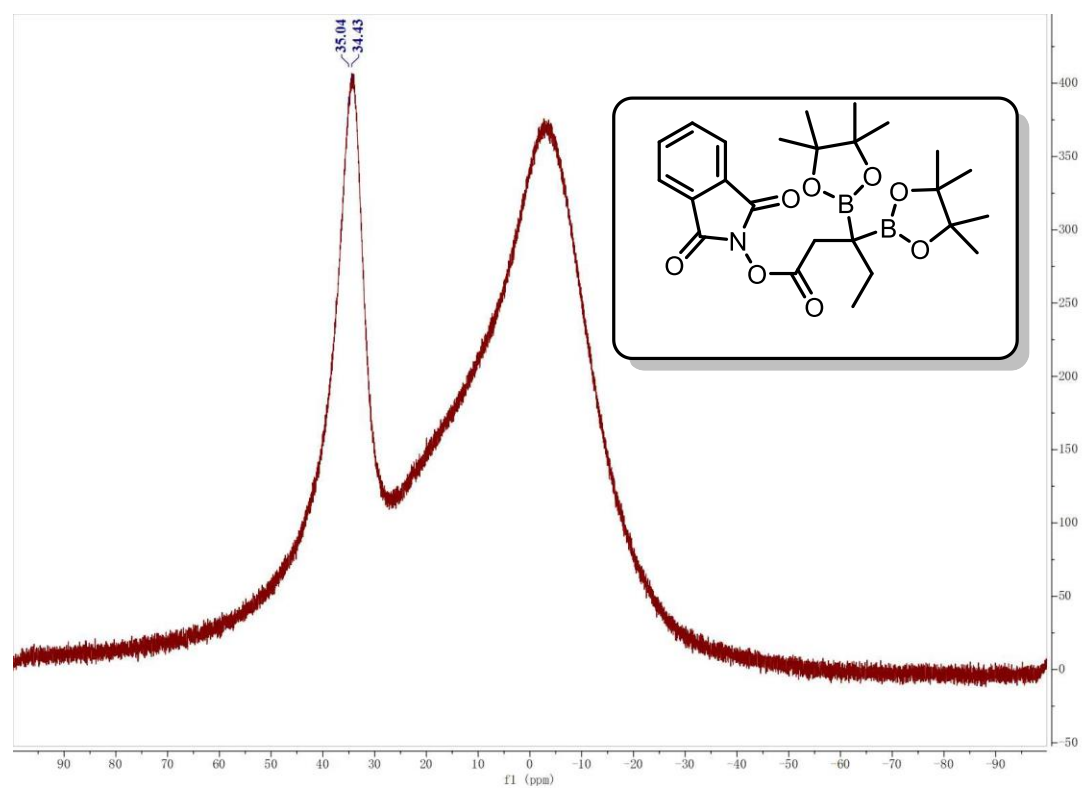
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

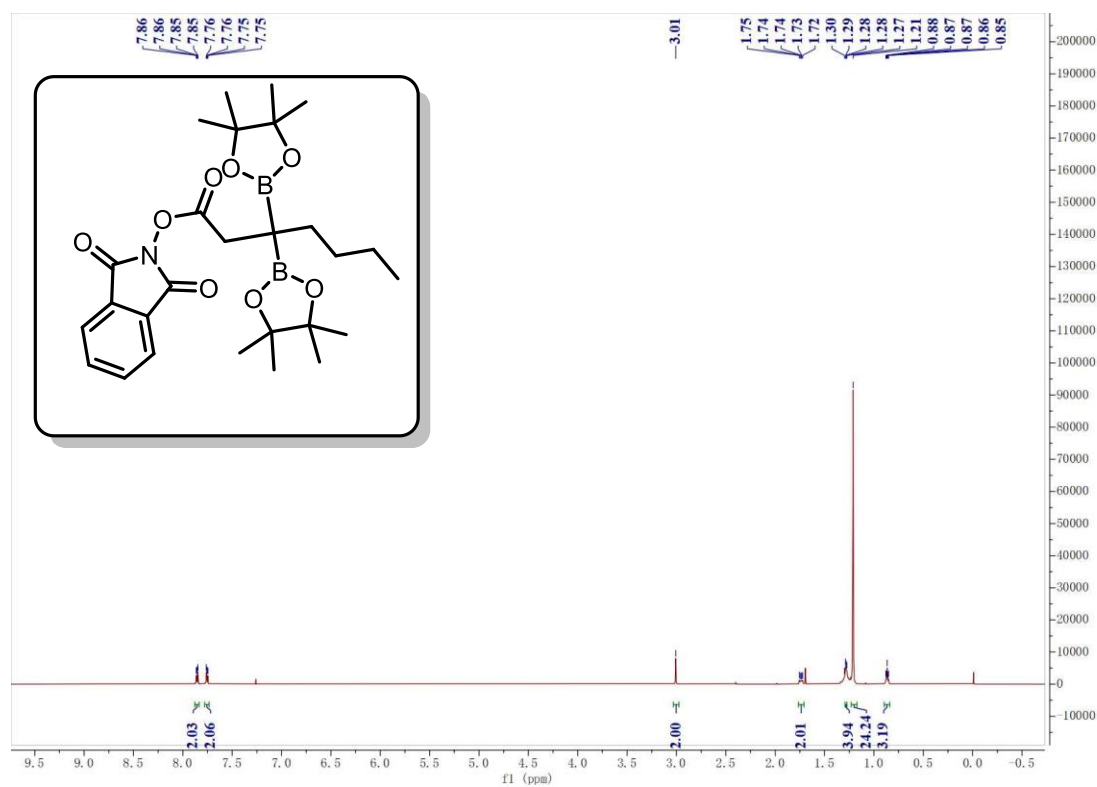


^{11}B NMR (160 MHz, Chloroform-*d*)

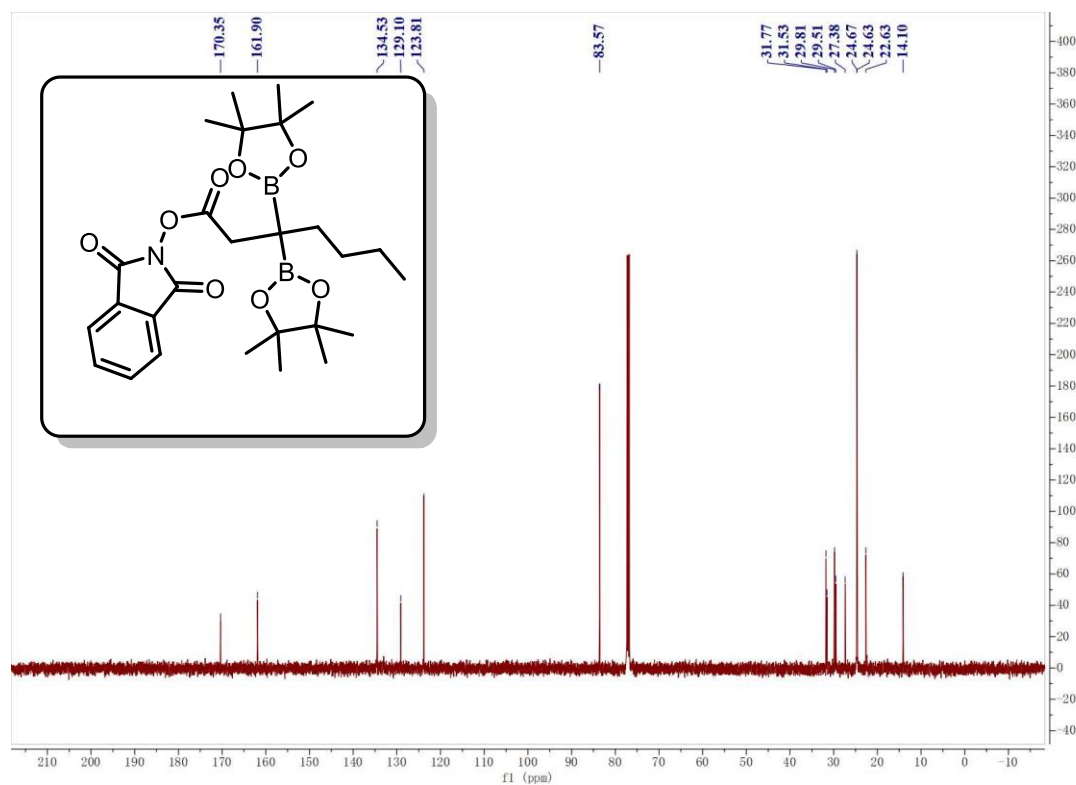


1,3-dioxoisindolin-2-yl 3,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)heptanoate (1u)

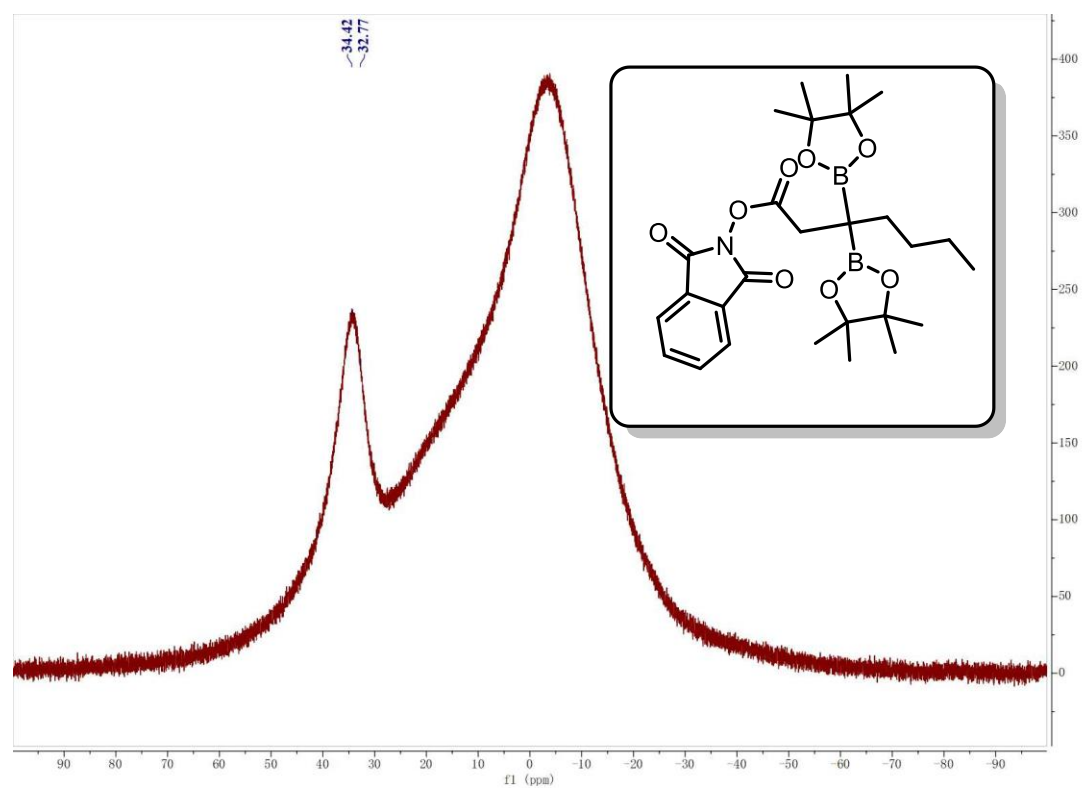
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

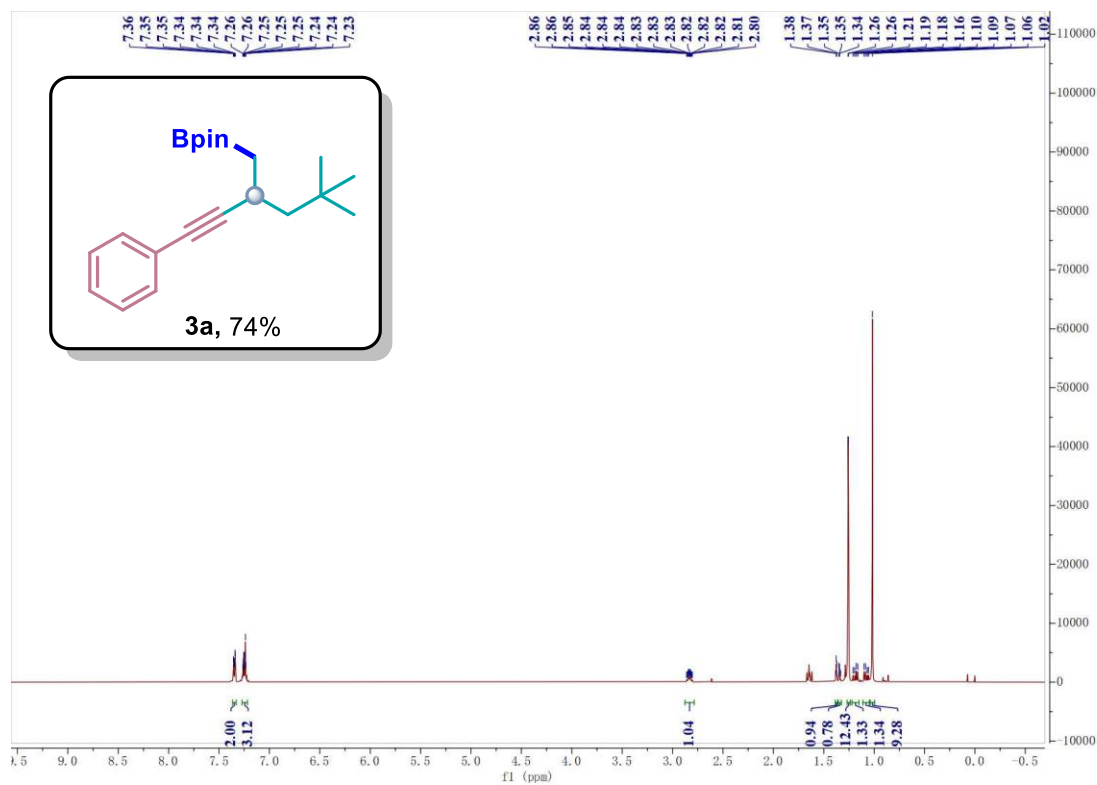


^{11}B NMR (160 MHz, Chloroform-*d*)

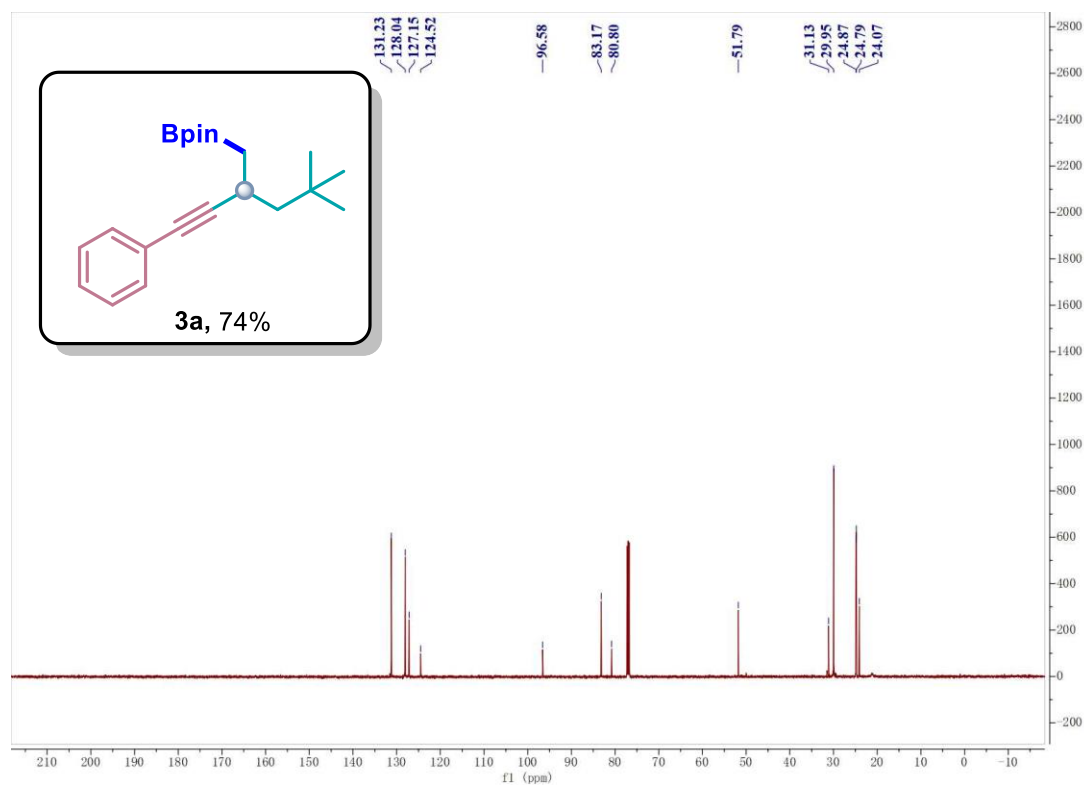


2-(4,4-dimethyl-2-(phenylethynyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a)

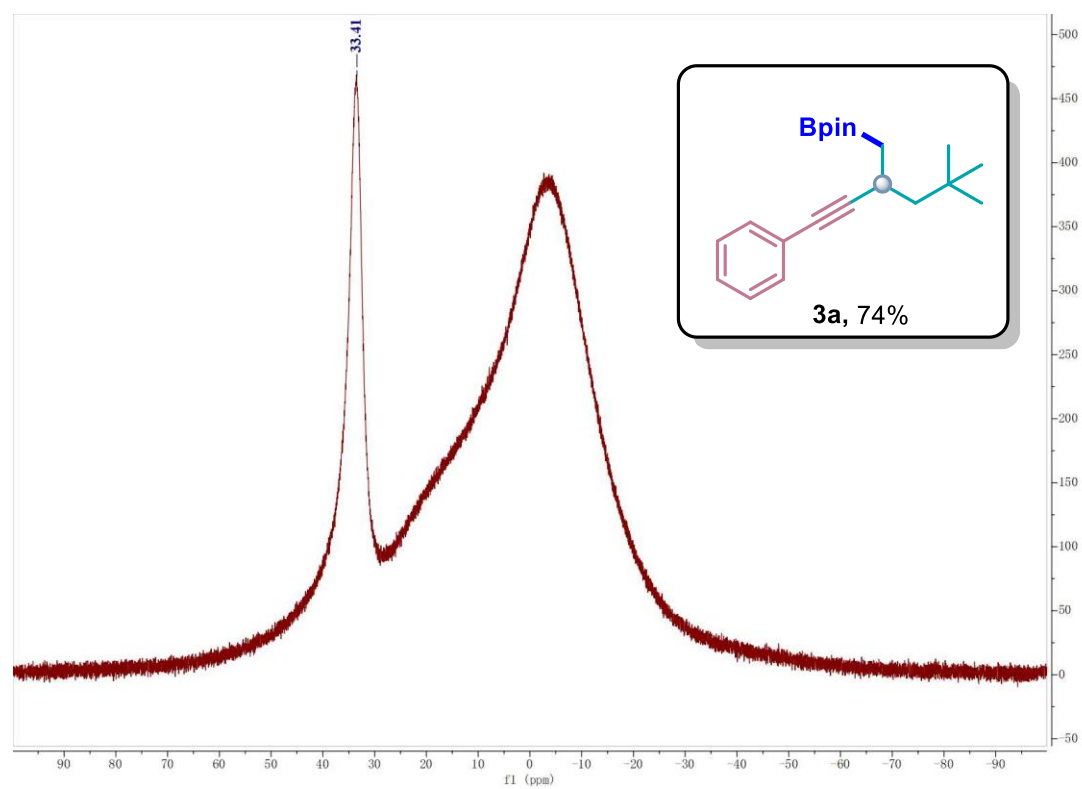
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

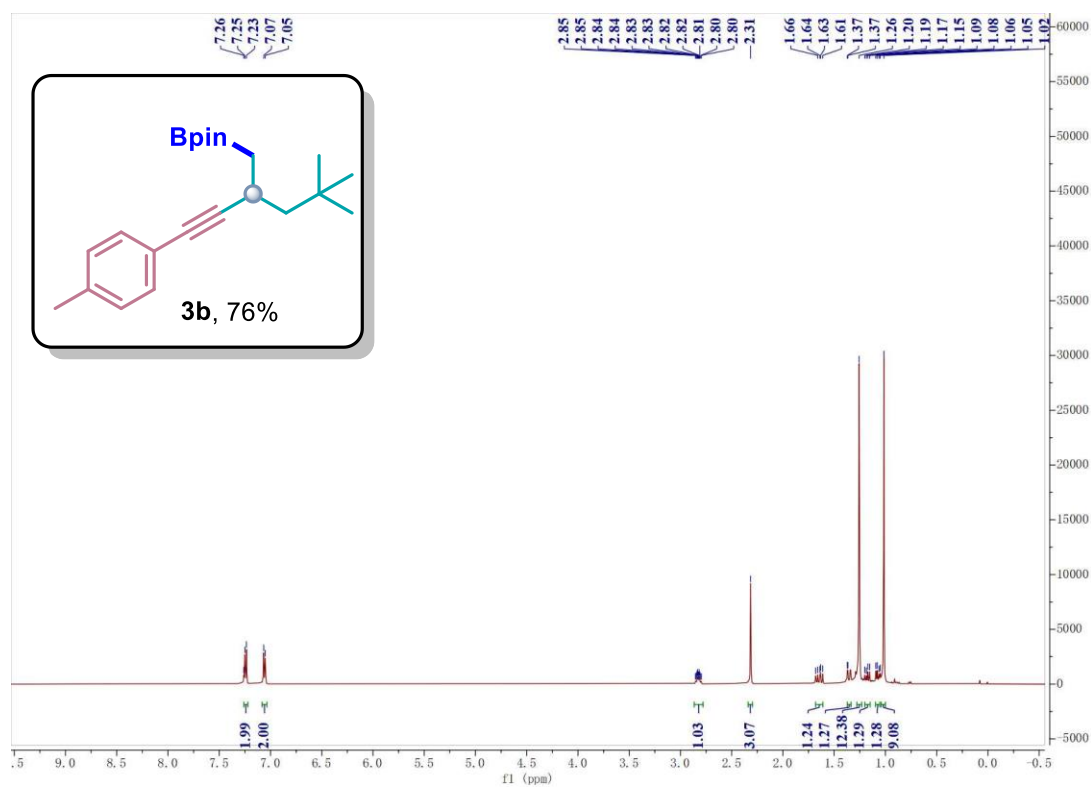


^{11}B NMR (160 MHz, Chloroform-*d*)

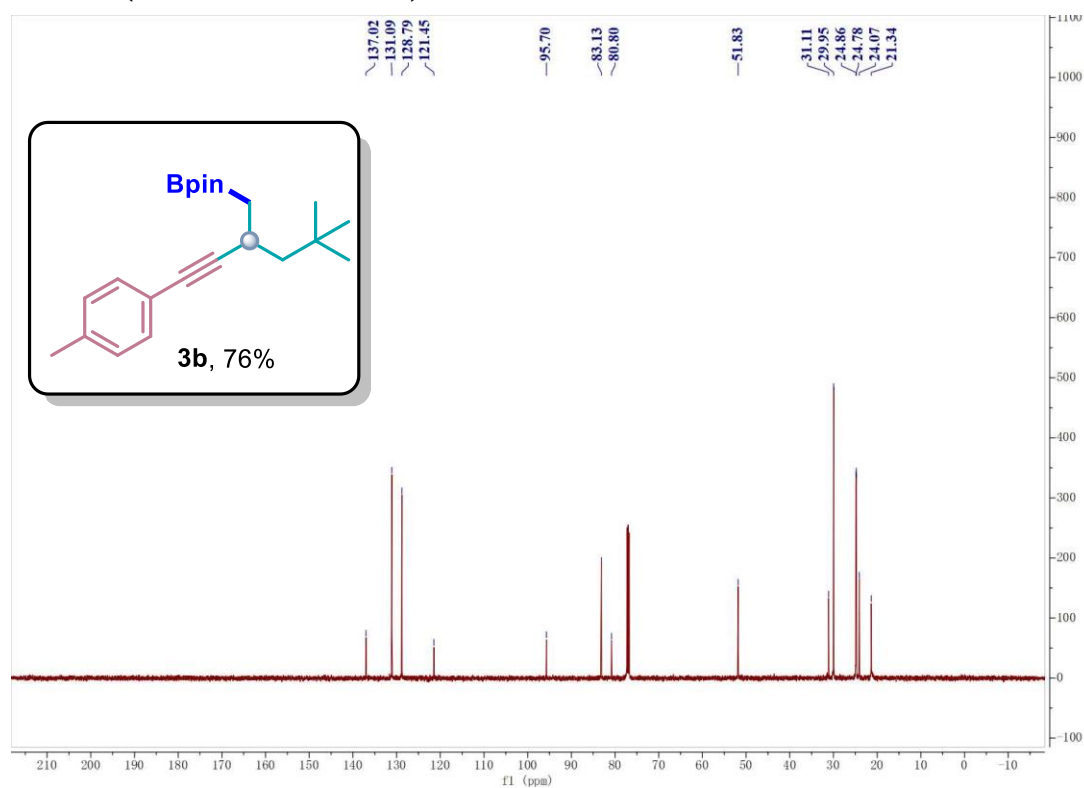


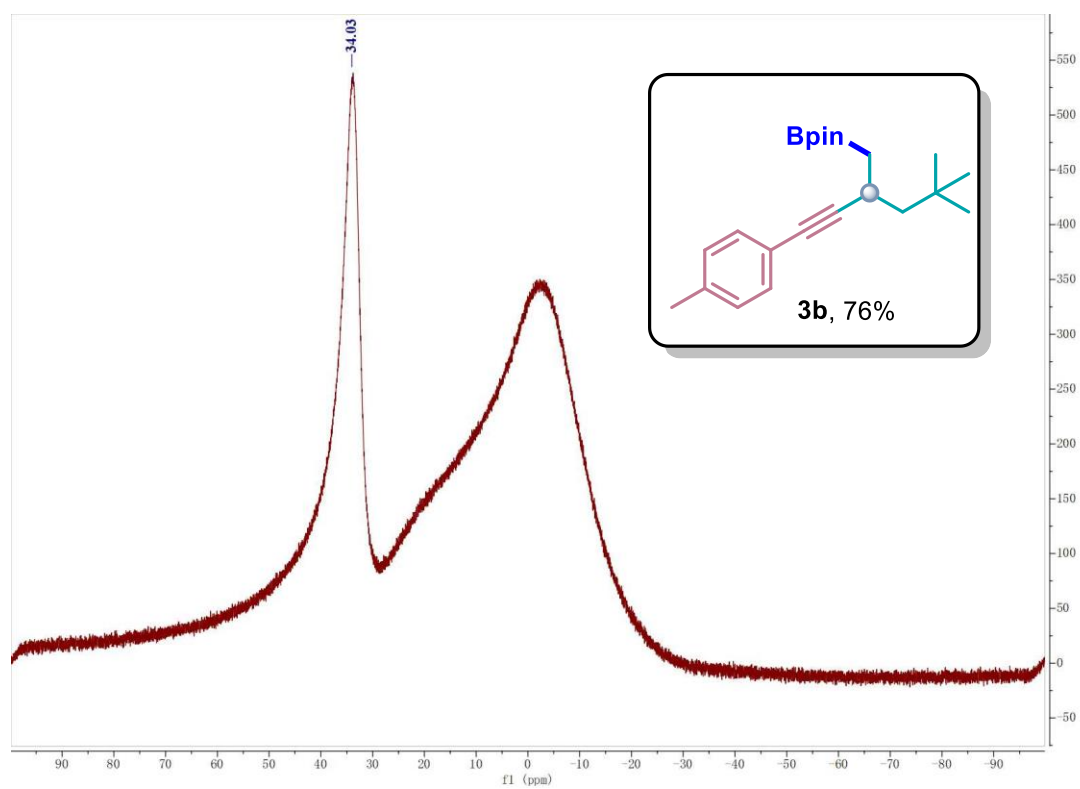
2-(4,4-dimethyl-2-(p-tolylethynyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3b)

¹H NMR (500 MHz, Chloroform-*d*)



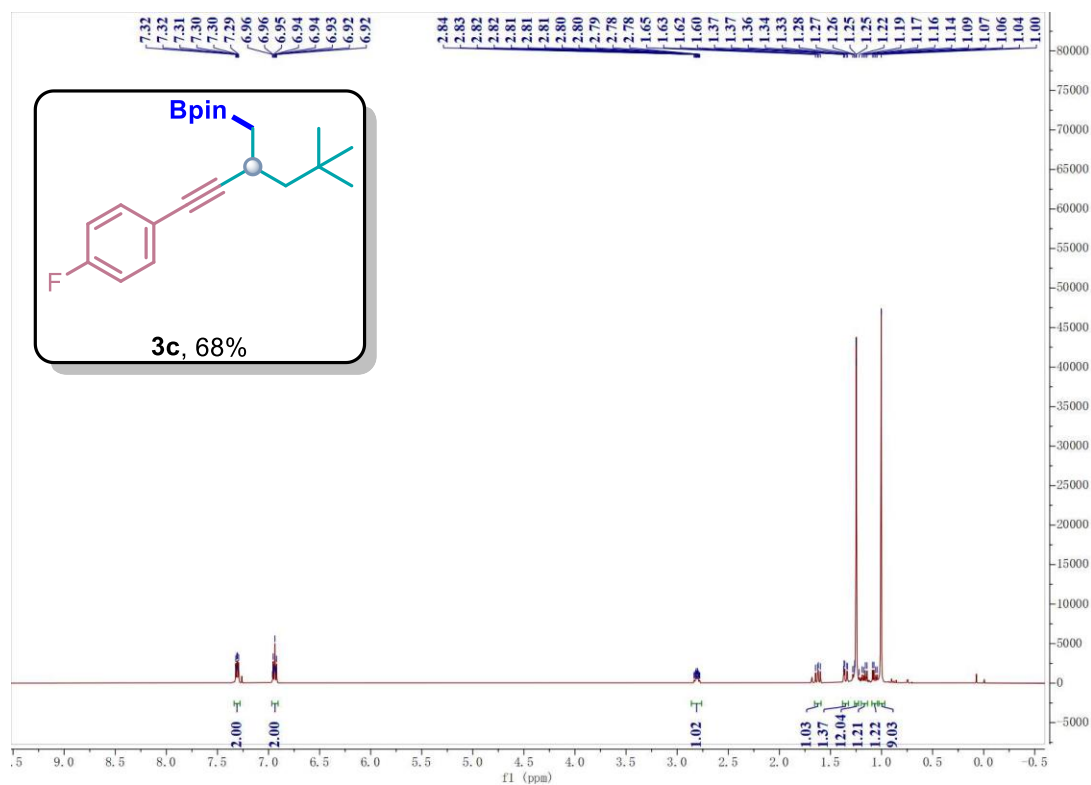
¹³C NMR (126 MHz, Chloroform-*d*)



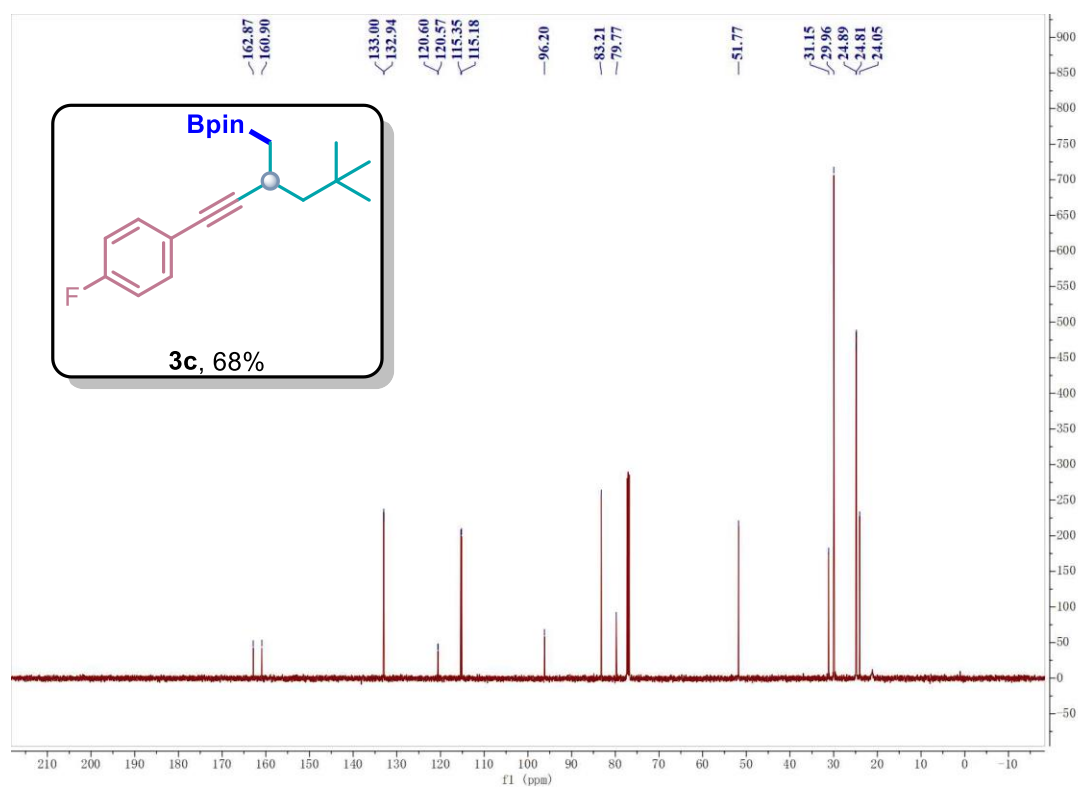
¹¹B NMR (160 MHz, Chloroform-*d*)

2-(2-((4-fluorophenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane
(3c)

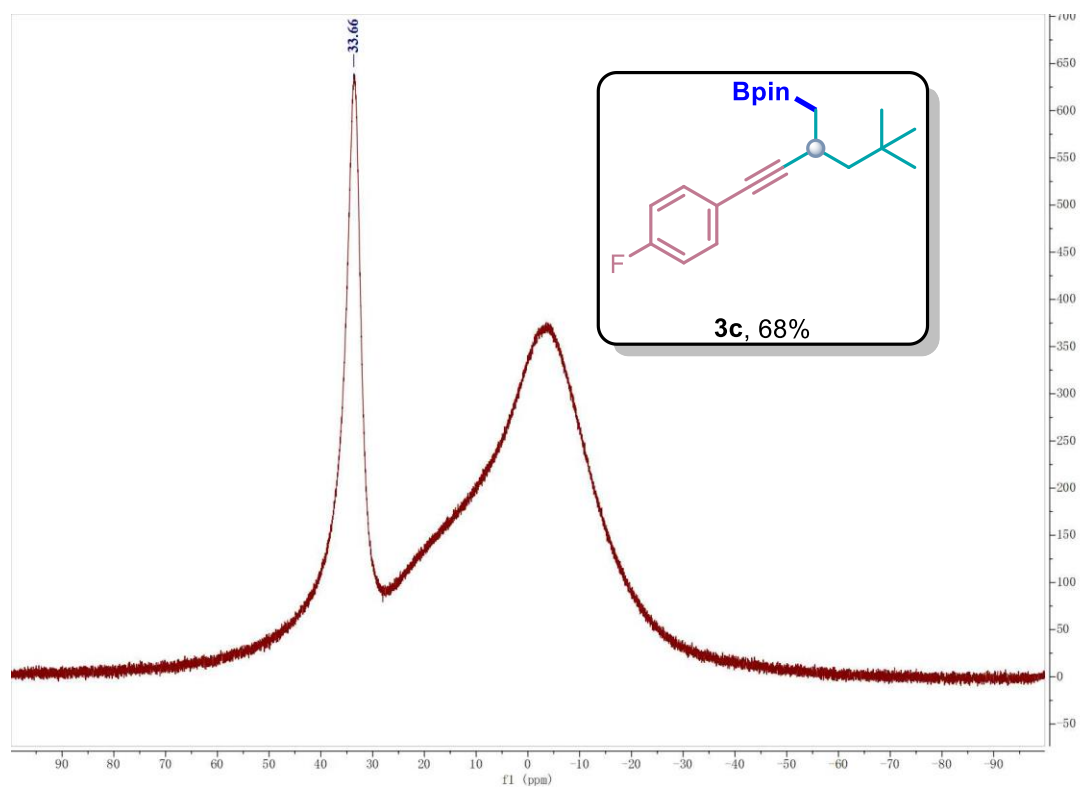
¹H NMR (500 MHz, Chloroform-*d*)



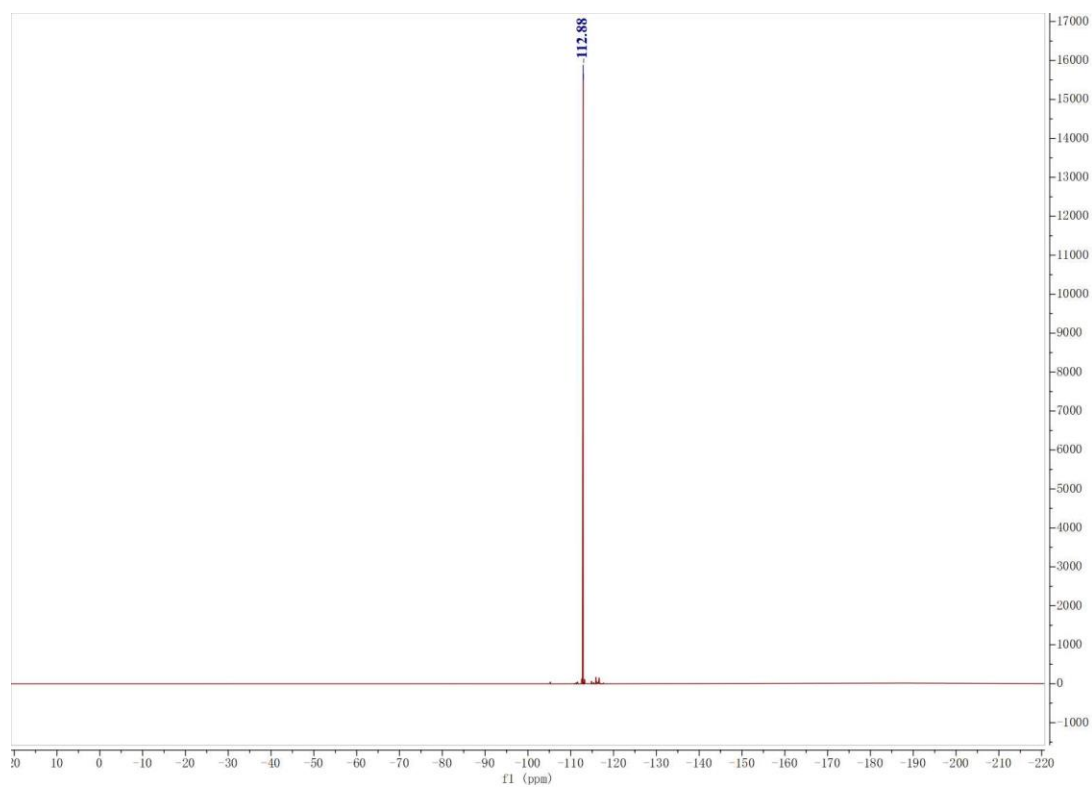
¹³C NMR (126 MHz, Chloroform-*d*)



^{11}B NMR (160 MHz, Chloroform-*d*)

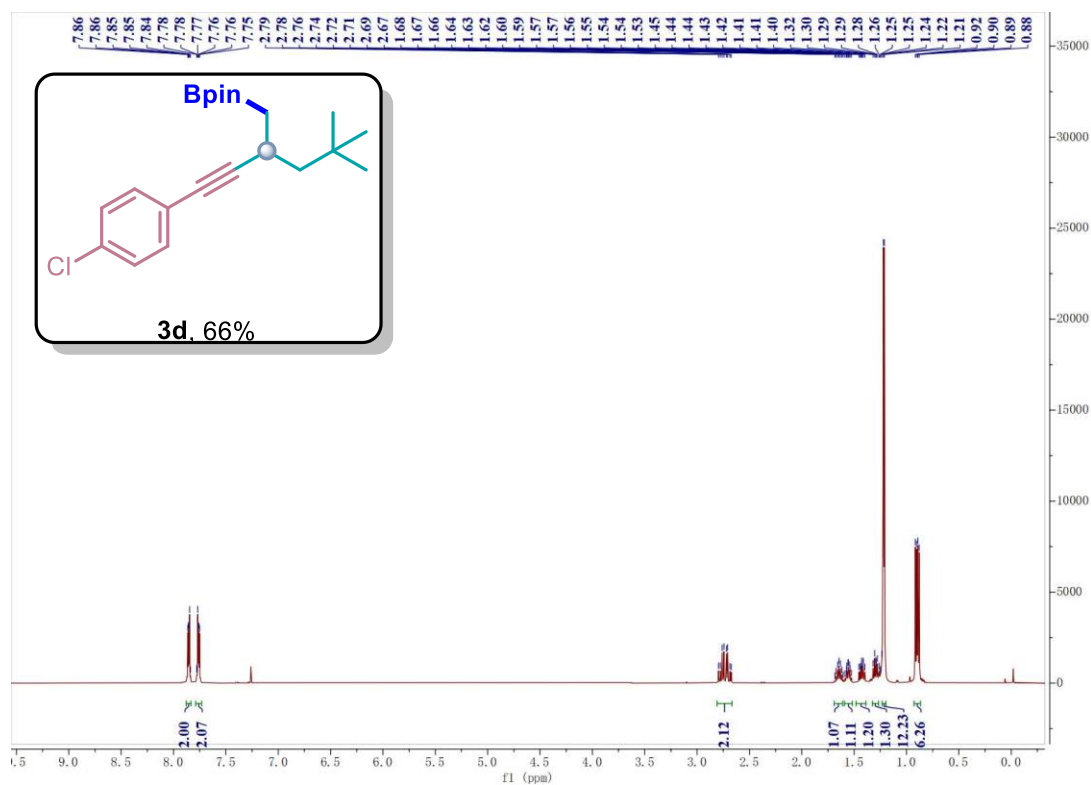


^{19}F NMR (471 MHz, Chloroform-*d*)

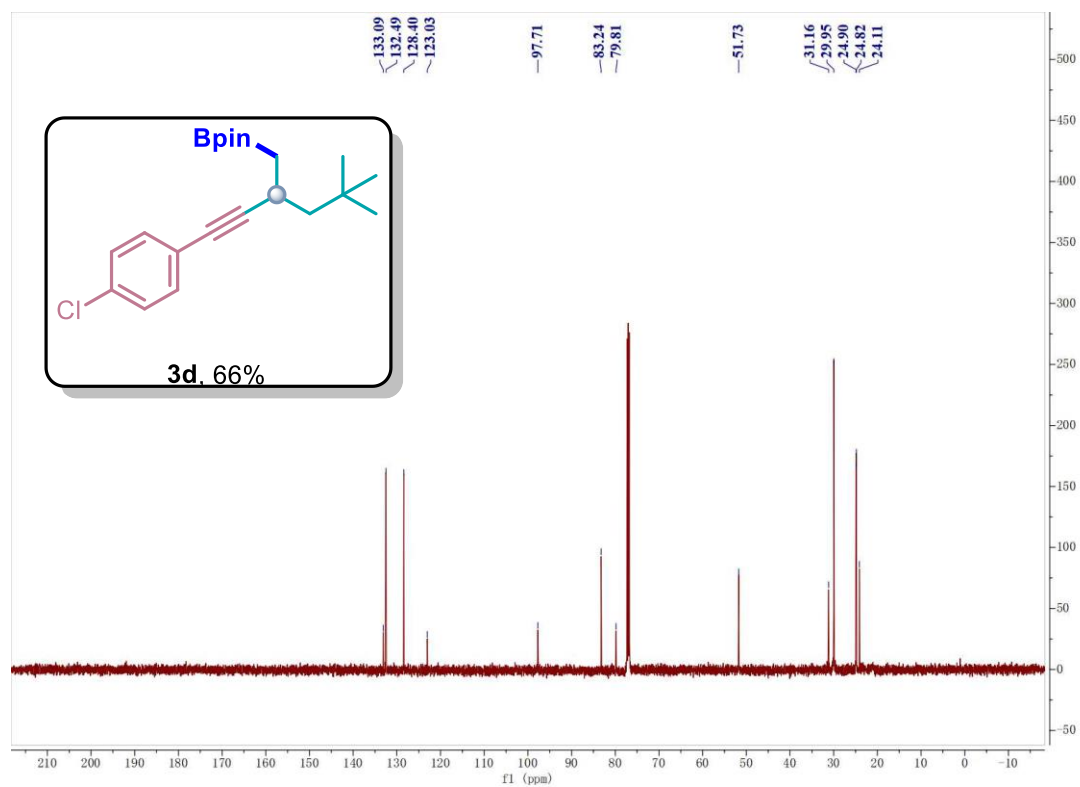


2-(2-((4-chlorophenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3d)

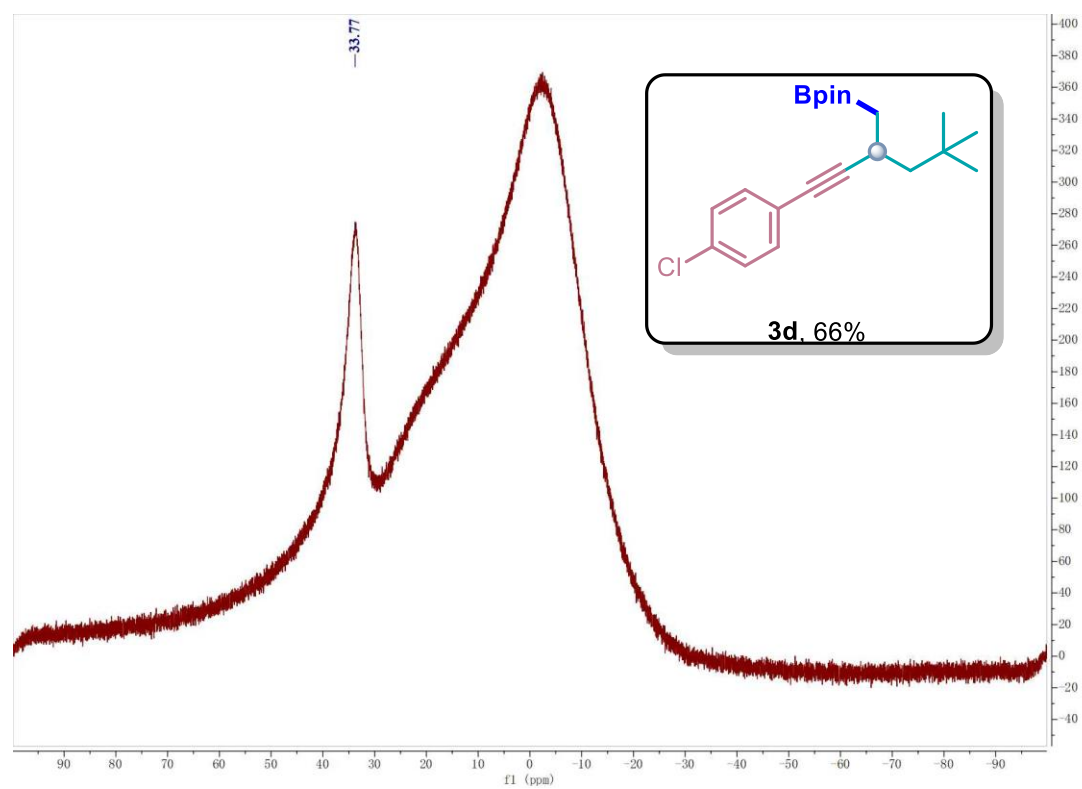
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

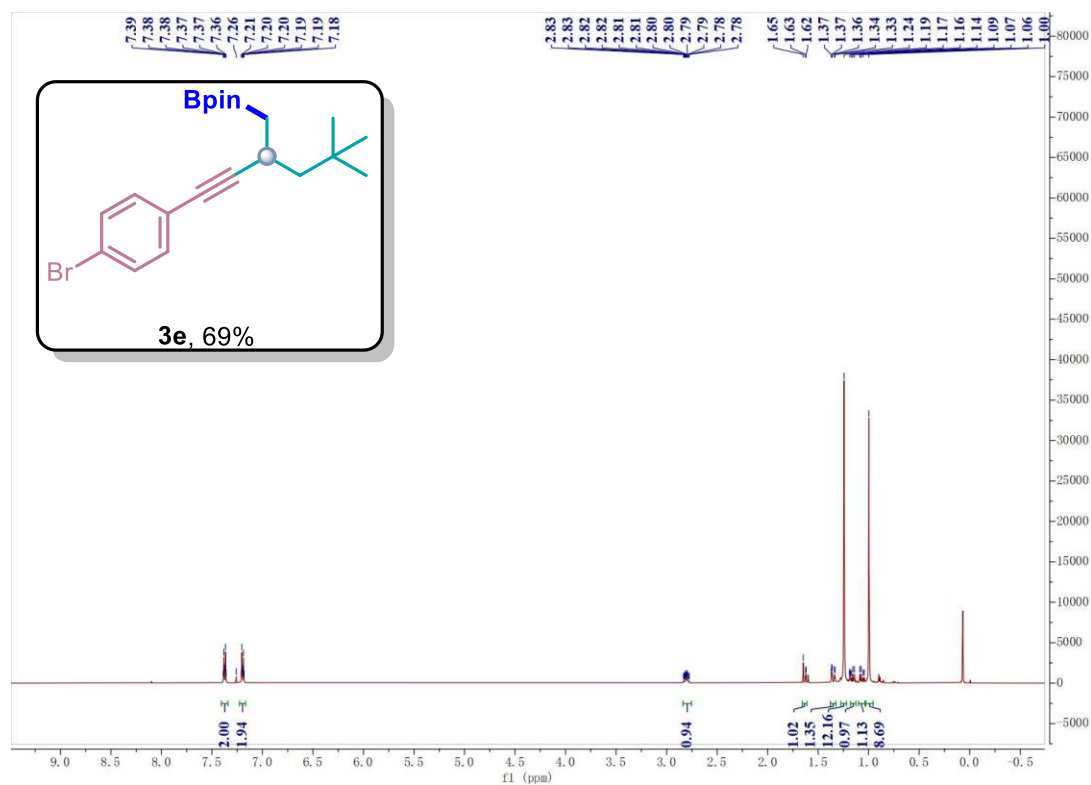


^{11}B NMR (160 MHz, Chloroform-*d*)

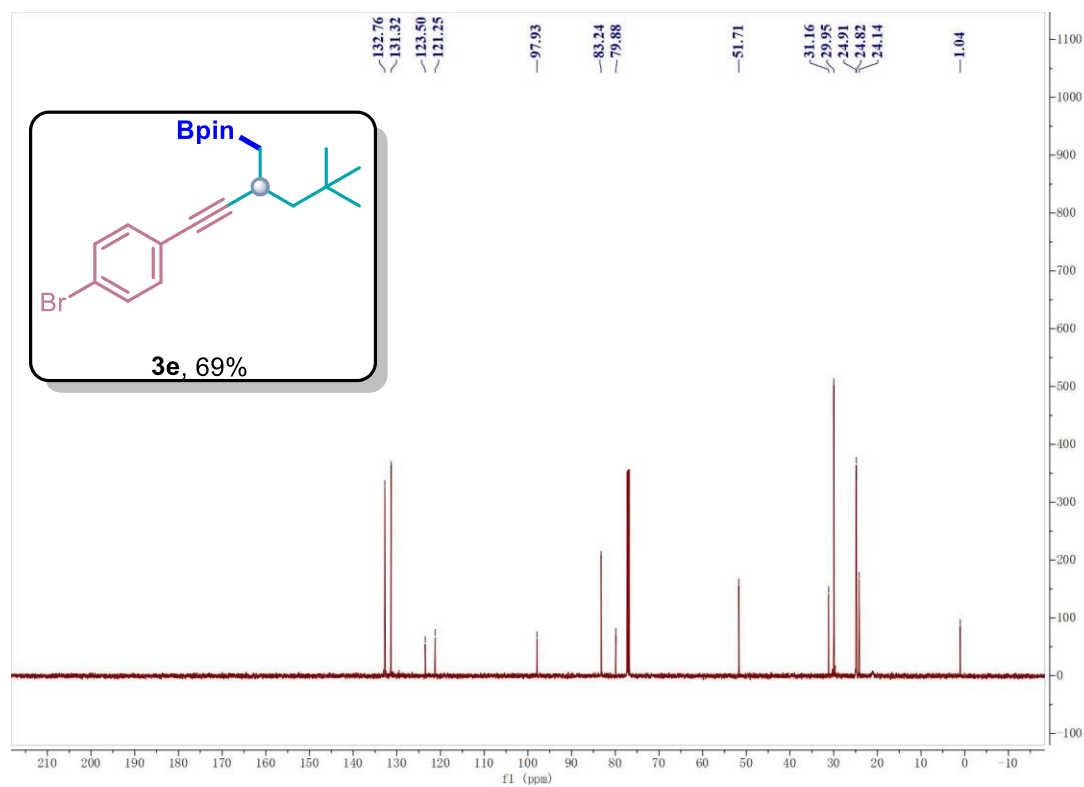


2-((4-bromophenyl)ethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e)

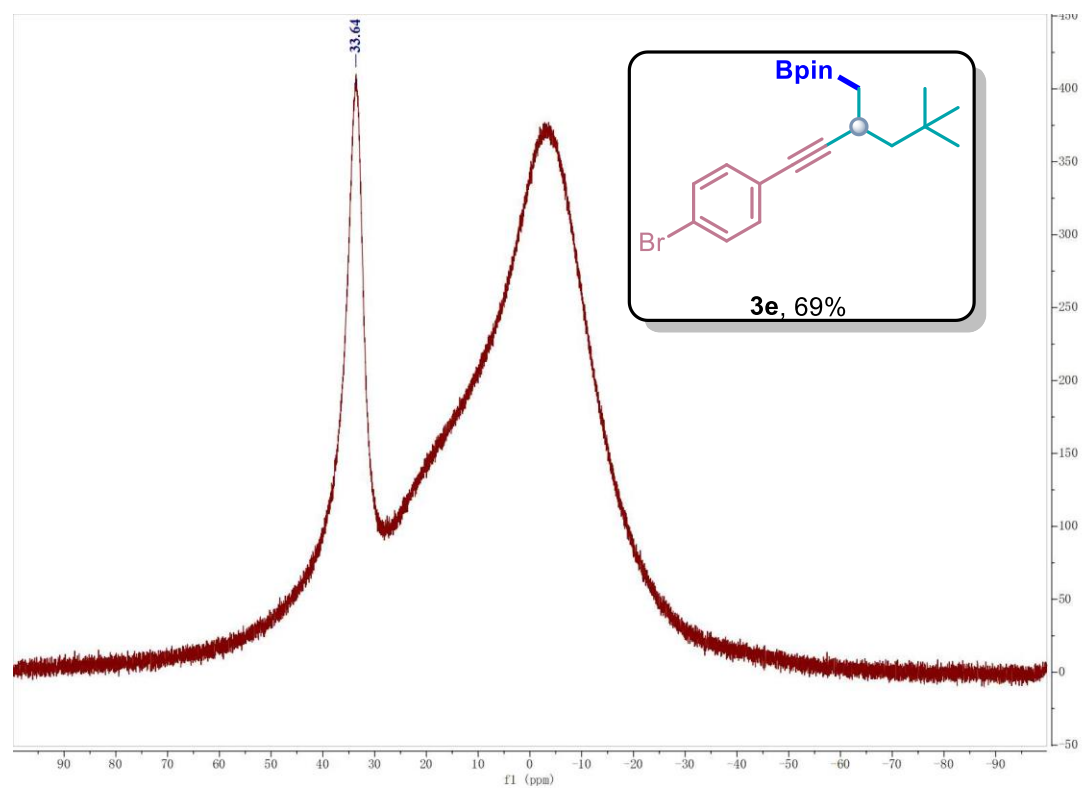
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

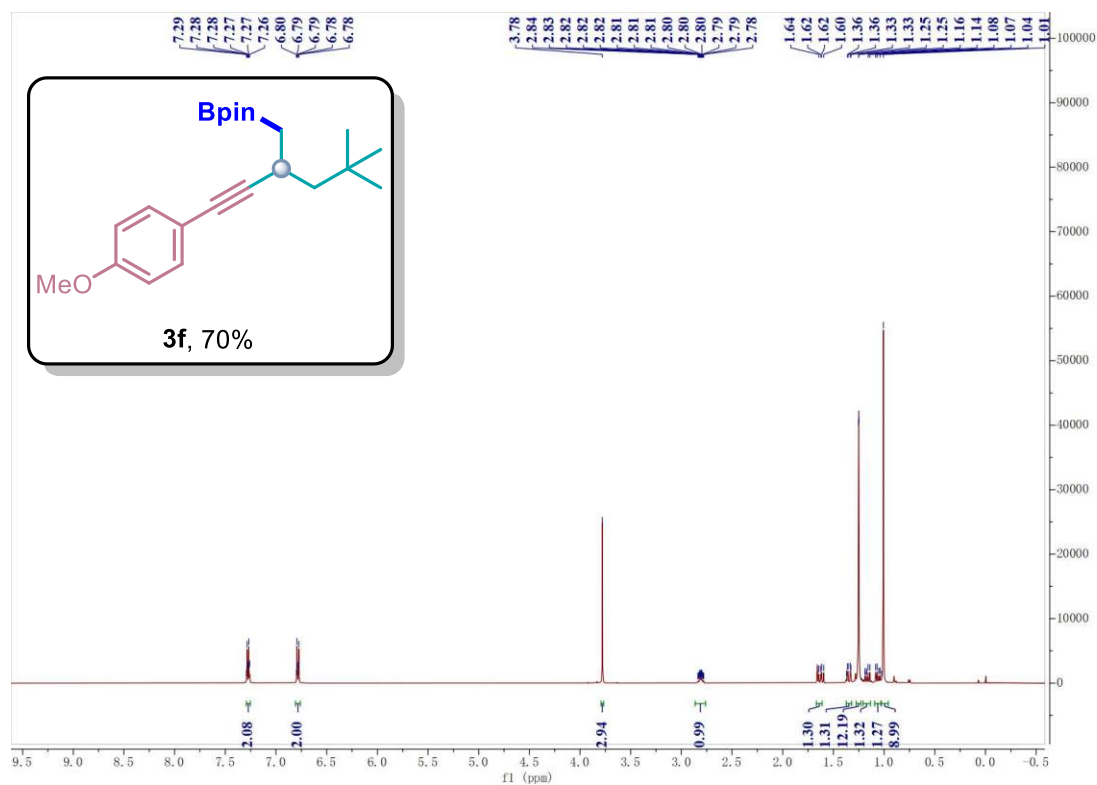


^{11}B NMR (160 MHz, Chloroform-*d*)

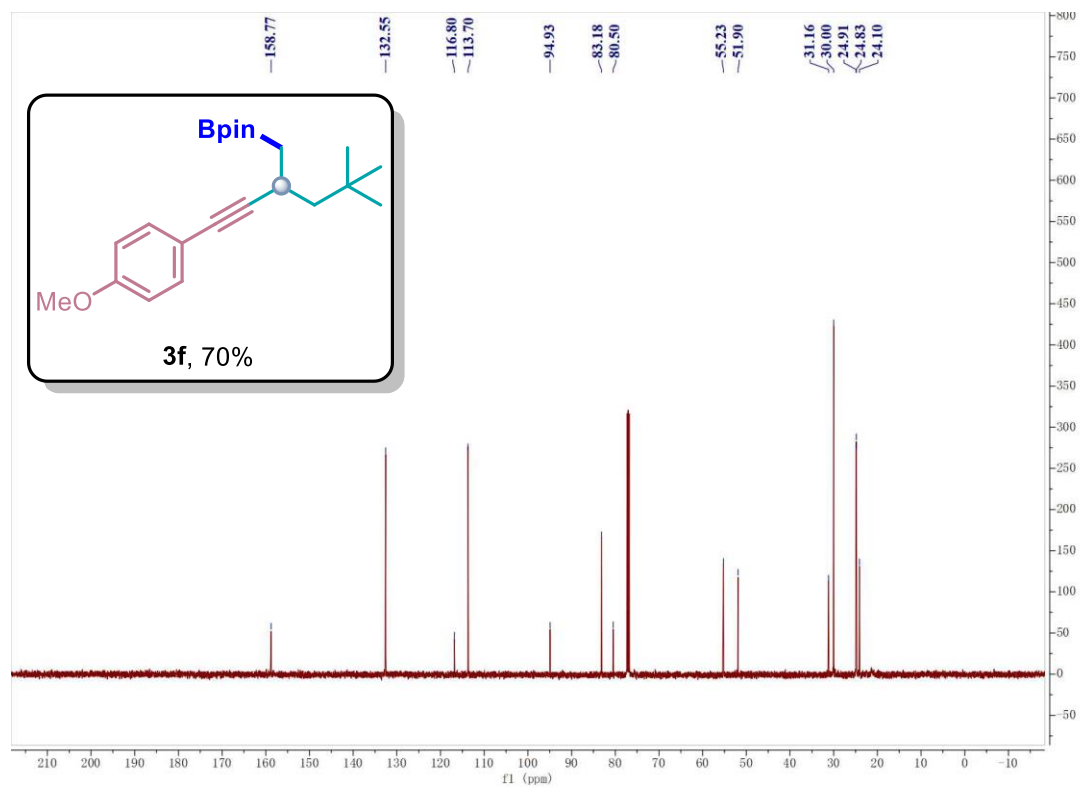


2-((4-methoxyphenyl)ethynyl)-4,4-dimethylpentyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3f)

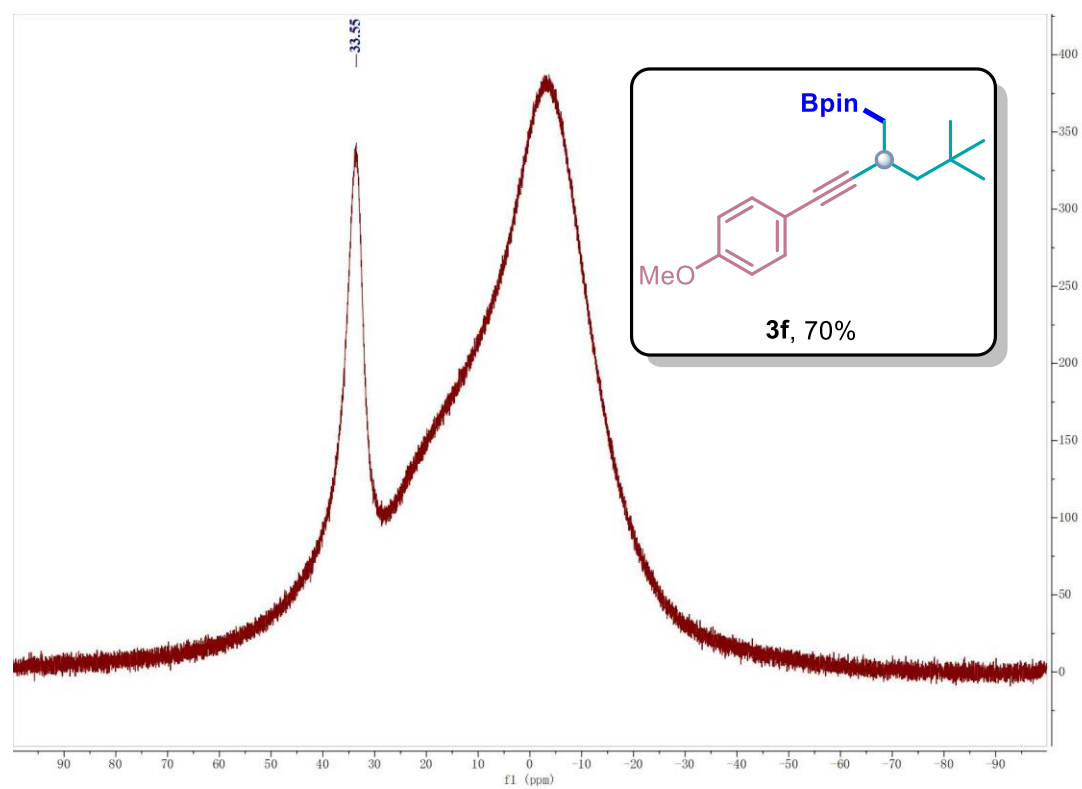
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

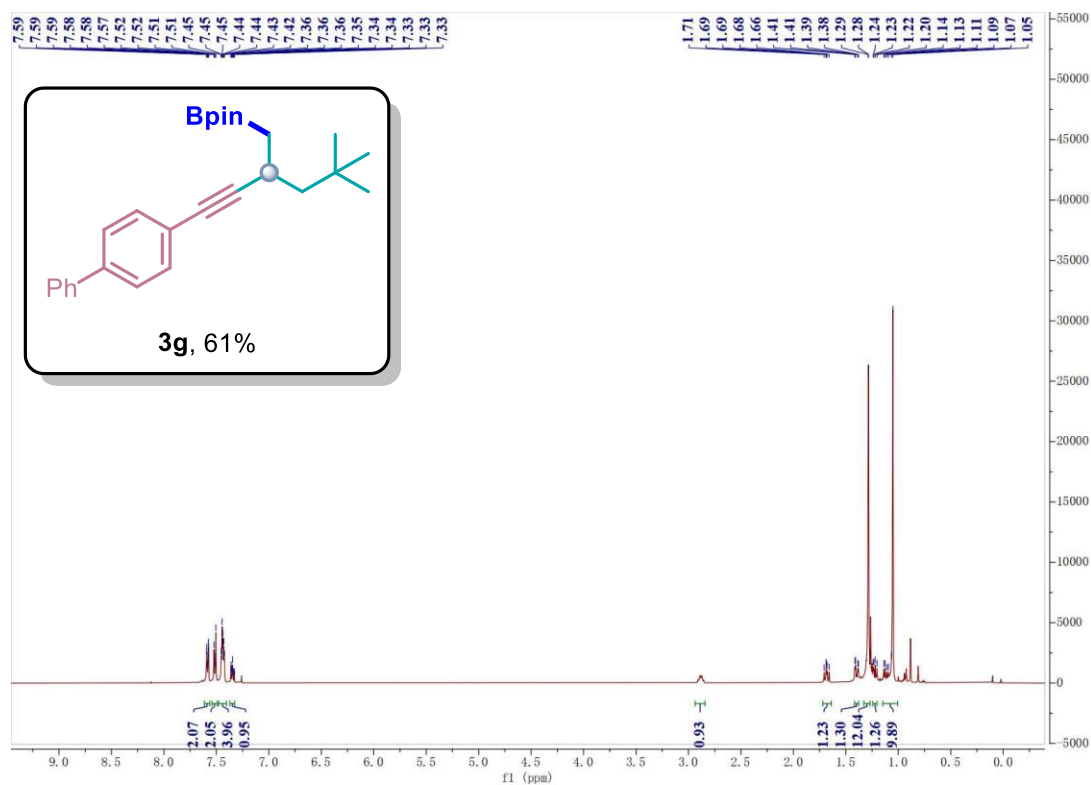


^{11}B NMR (160 MHz, Chloroform-*d*)

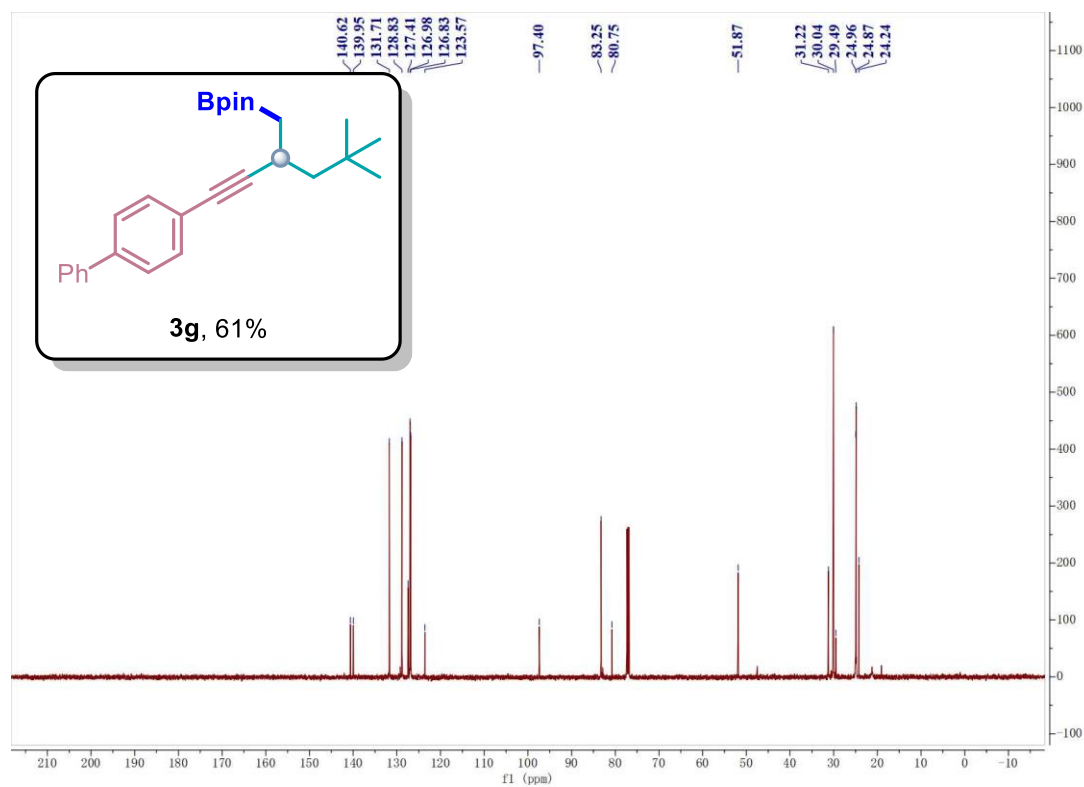


2-(2-([1,1'-biphenyl]-4-ylethynyl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3g)

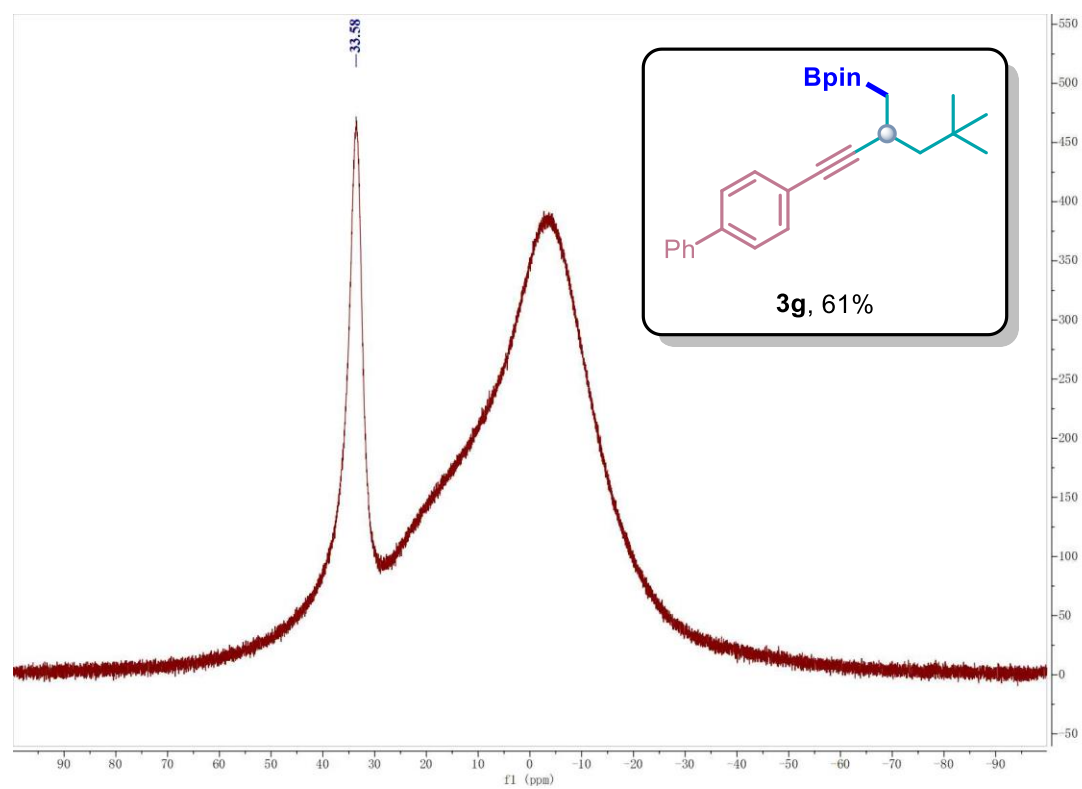
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

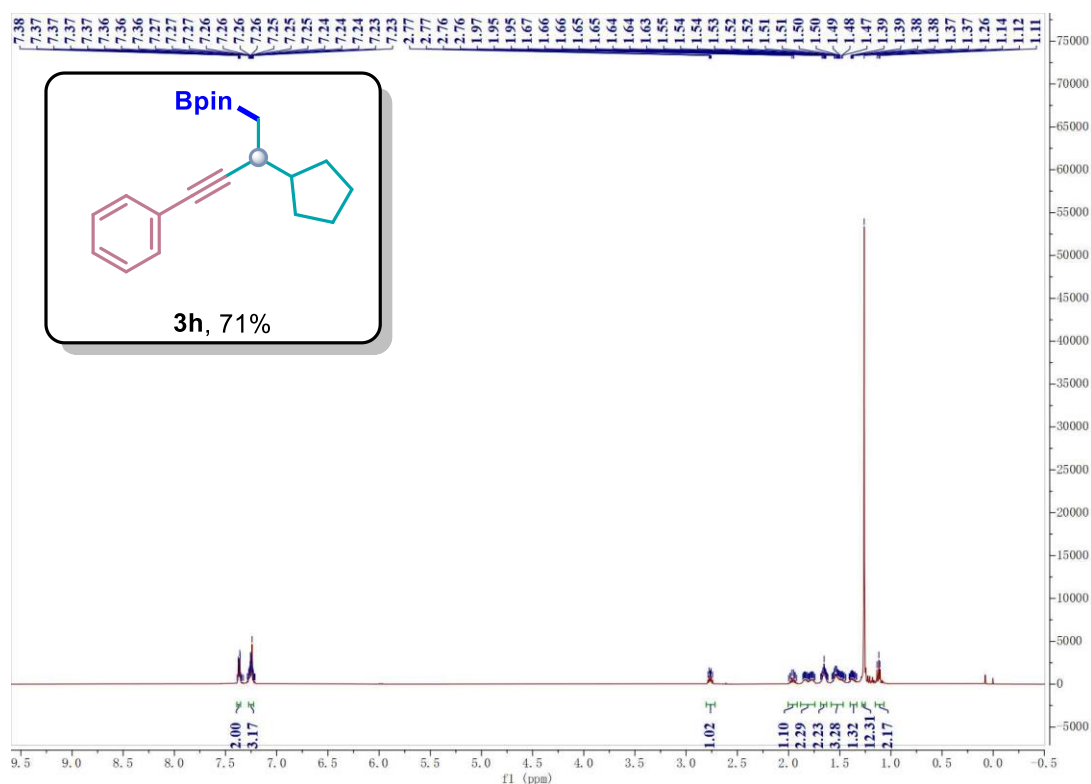


^{11}B NMR (160 MHz, Chloroform-*d*)

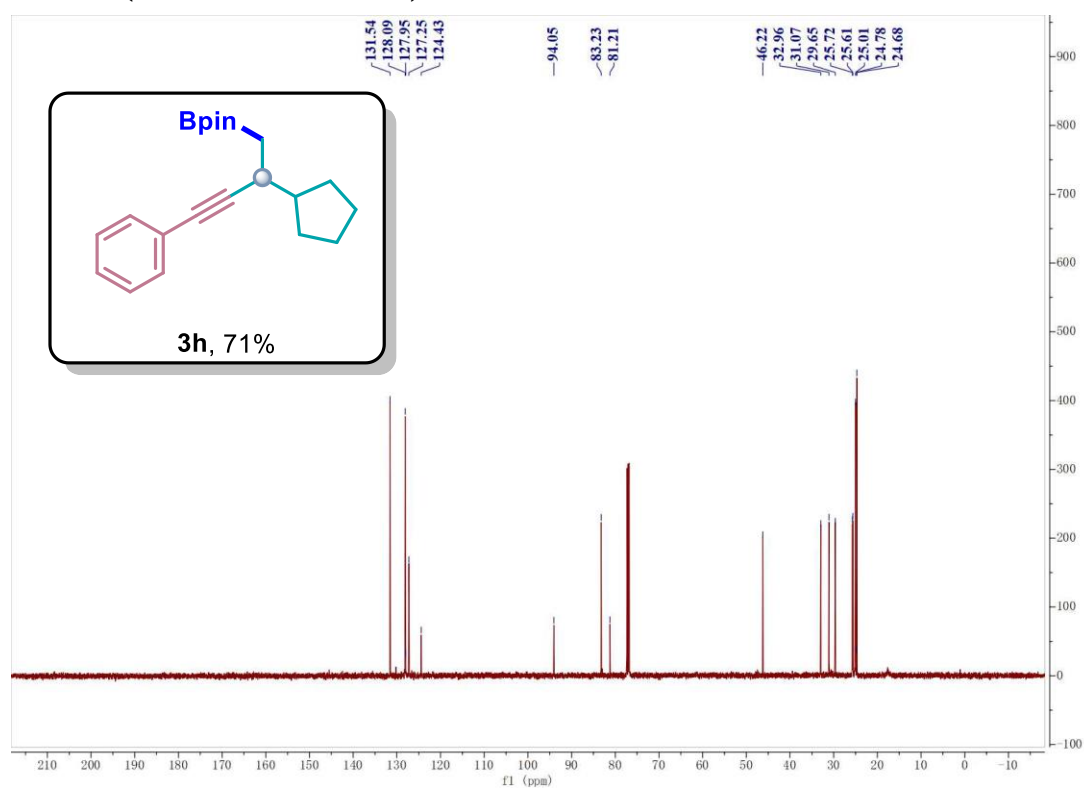


2-(2-cyclopentyl-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3h)

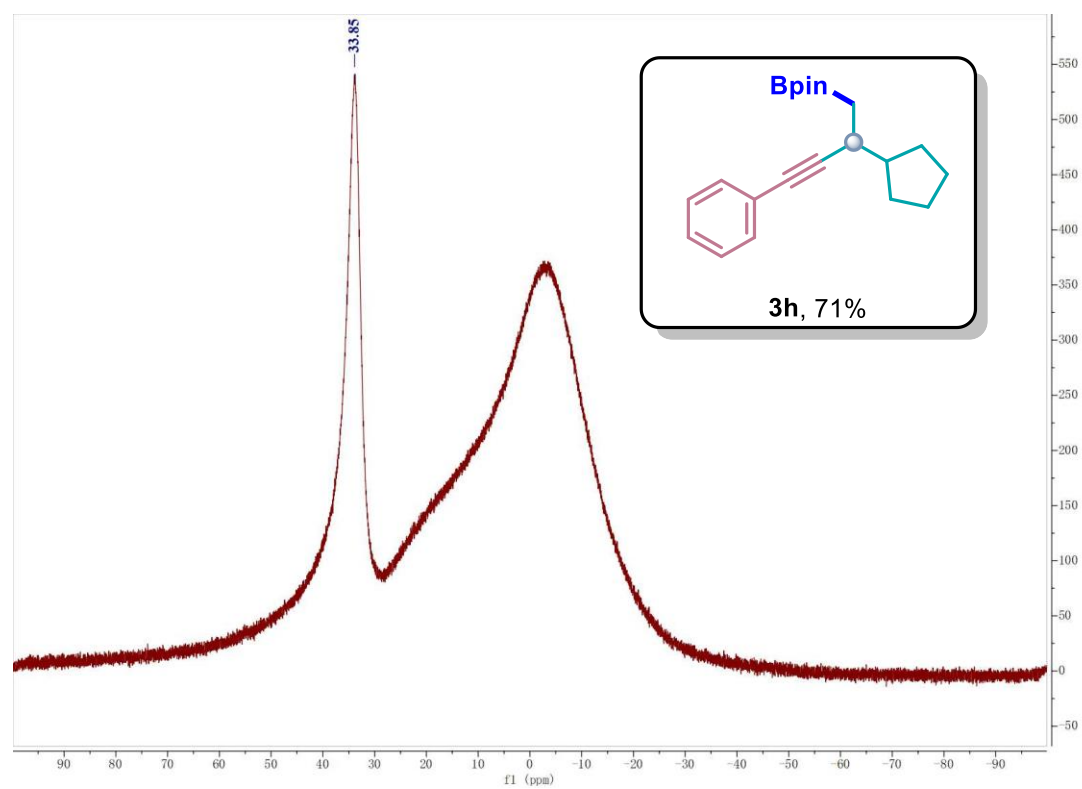
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)



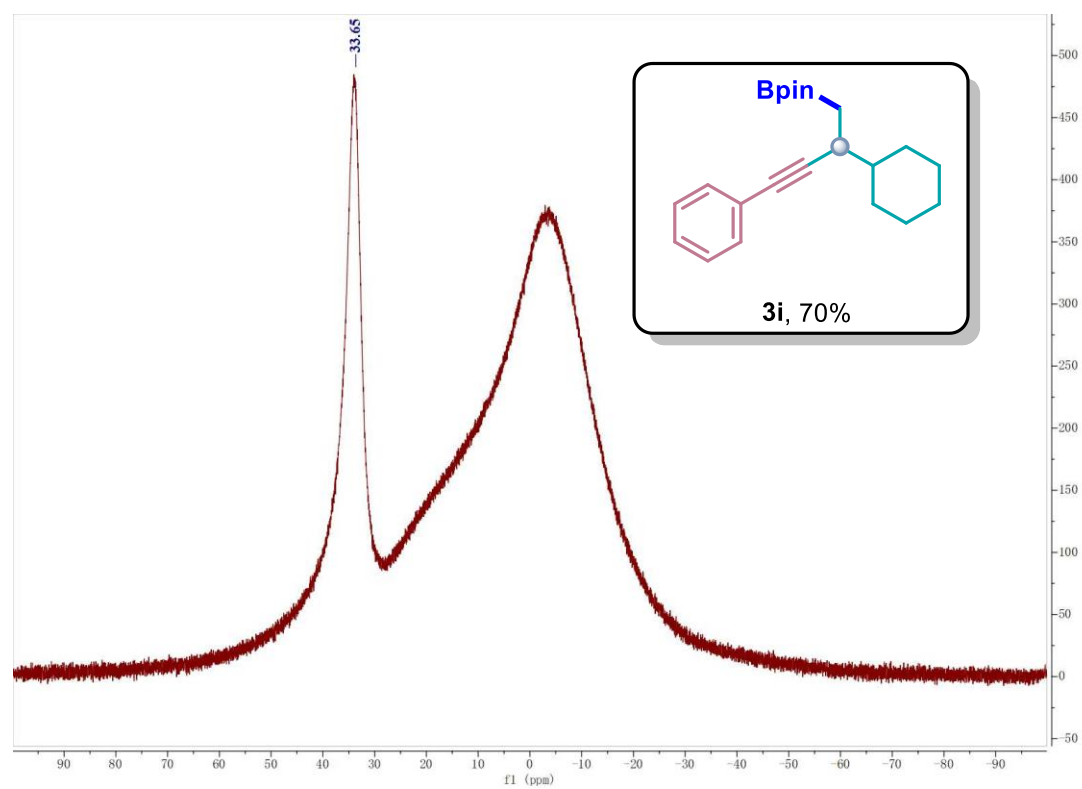
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

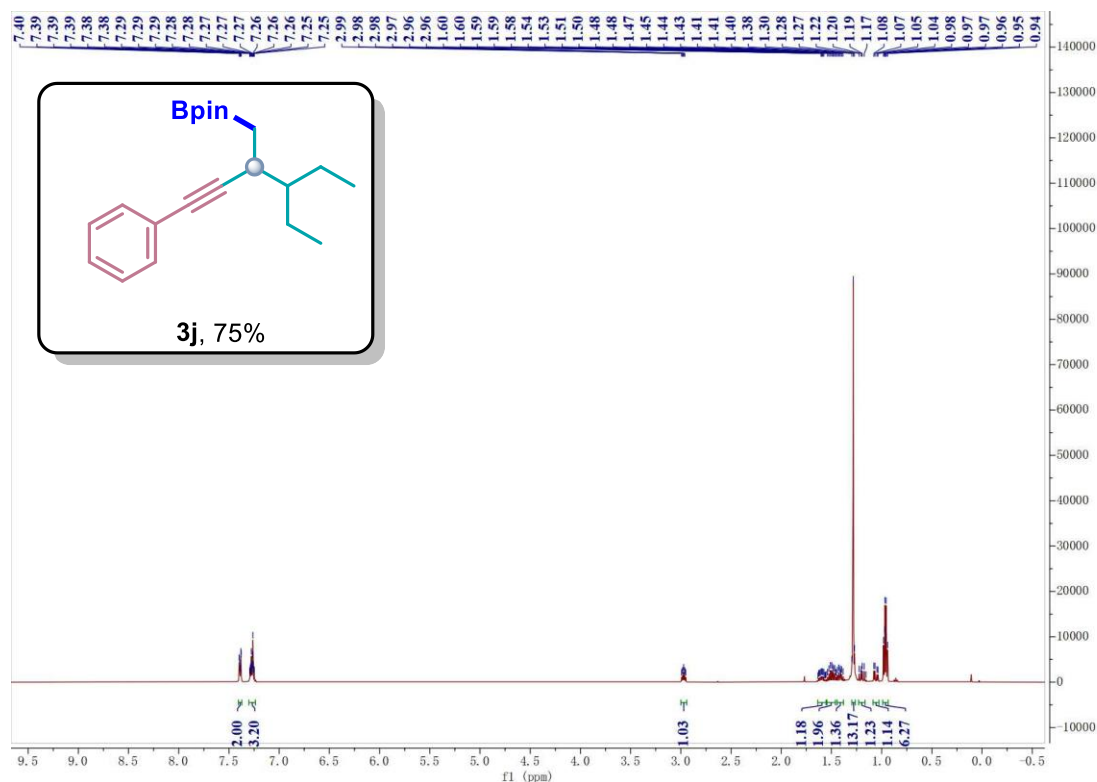


^{11}B NMR (160 MHz, Chloroform-*d*)

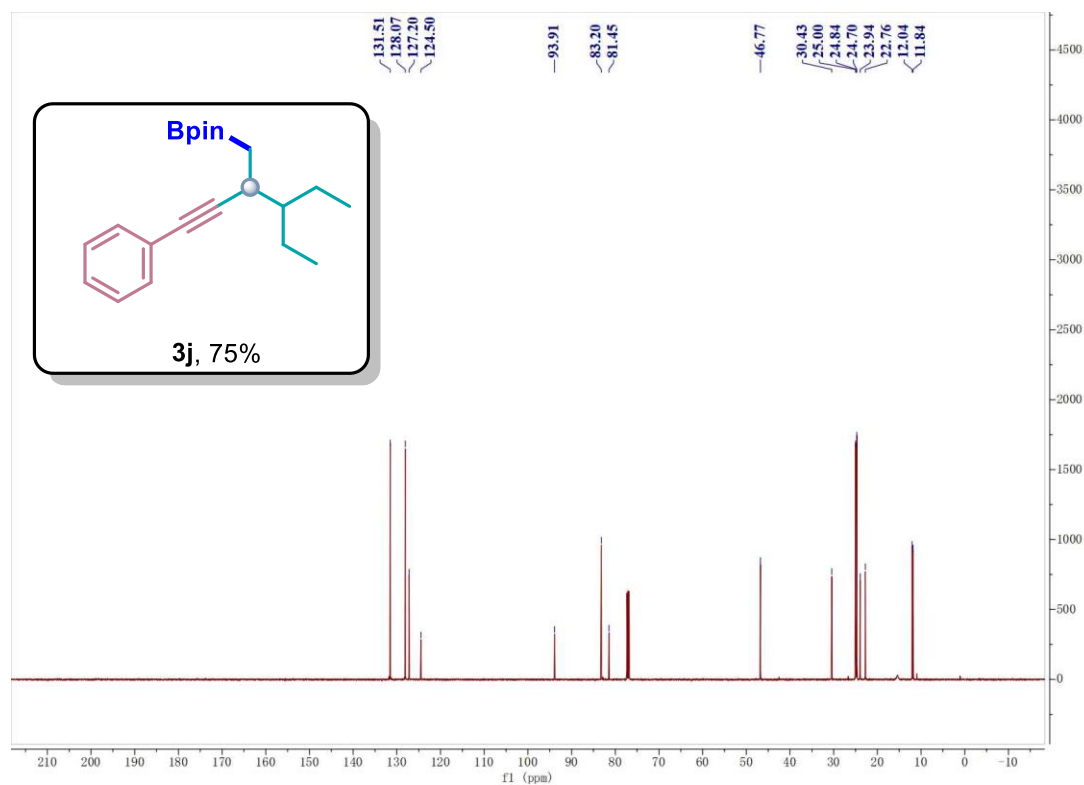


2-(3-ethyl-2-(phenylethynyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3j)

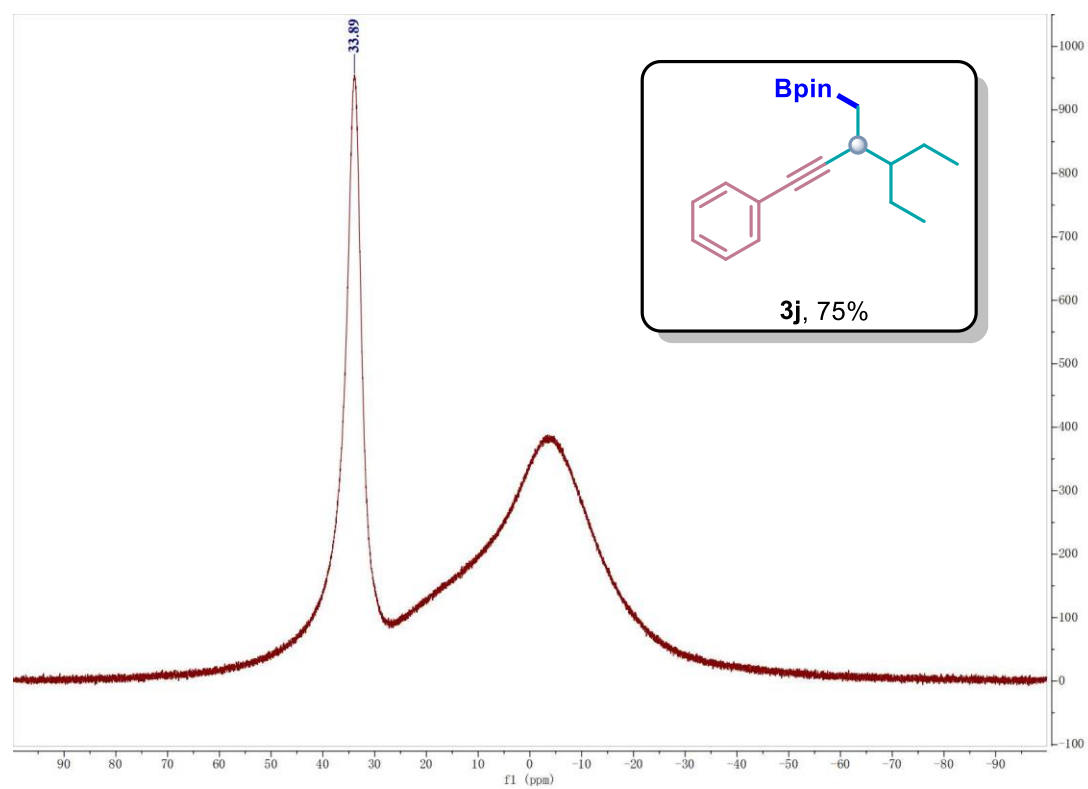
¹H NMR (500 MHz, Chloroform-*d*)



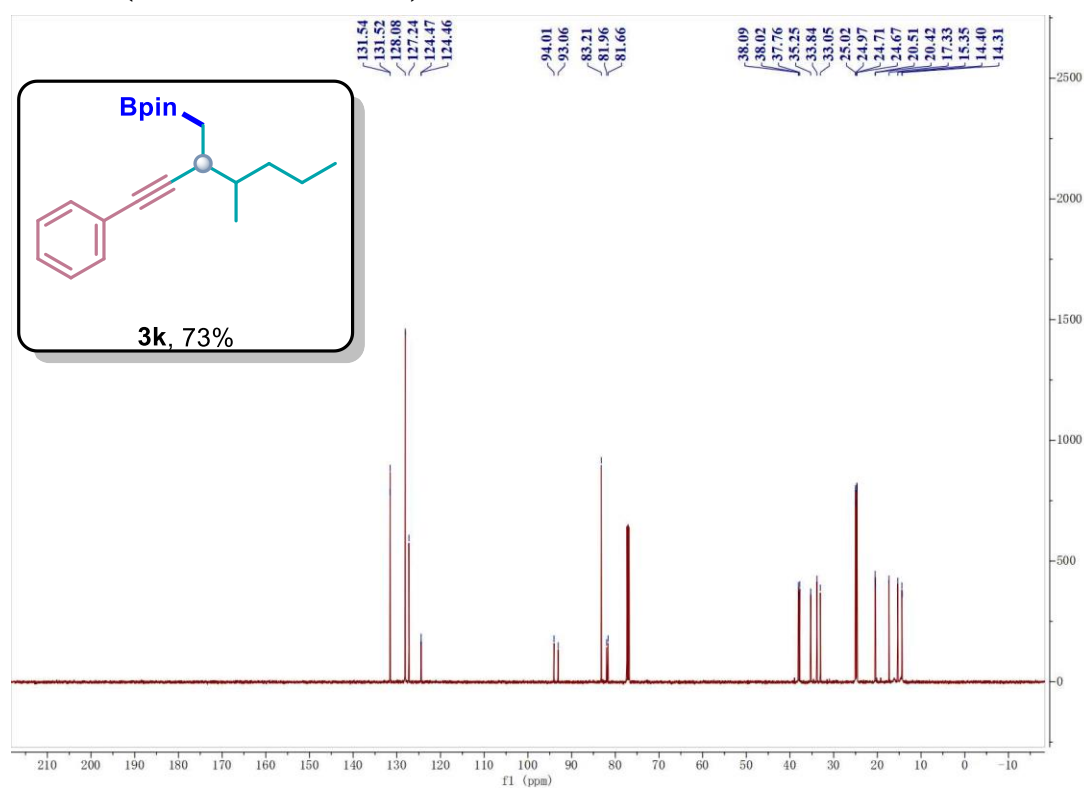
¹³C NMR (126 MHz, Chloroform-*d*)



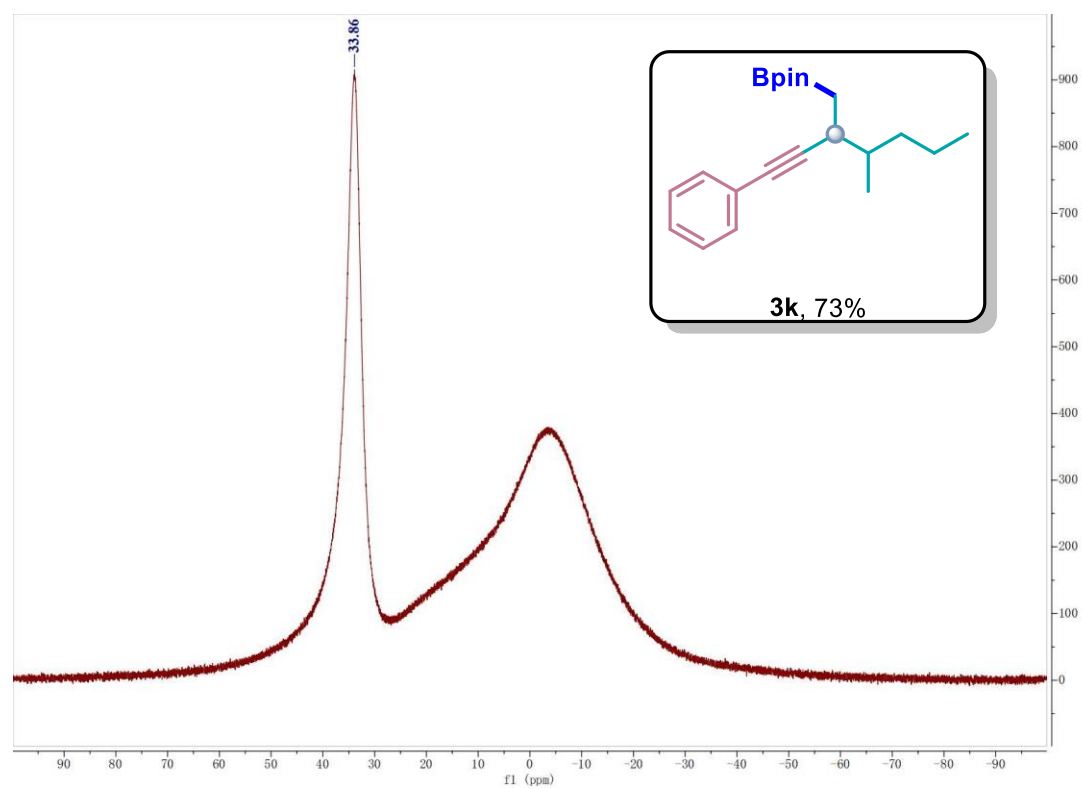
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

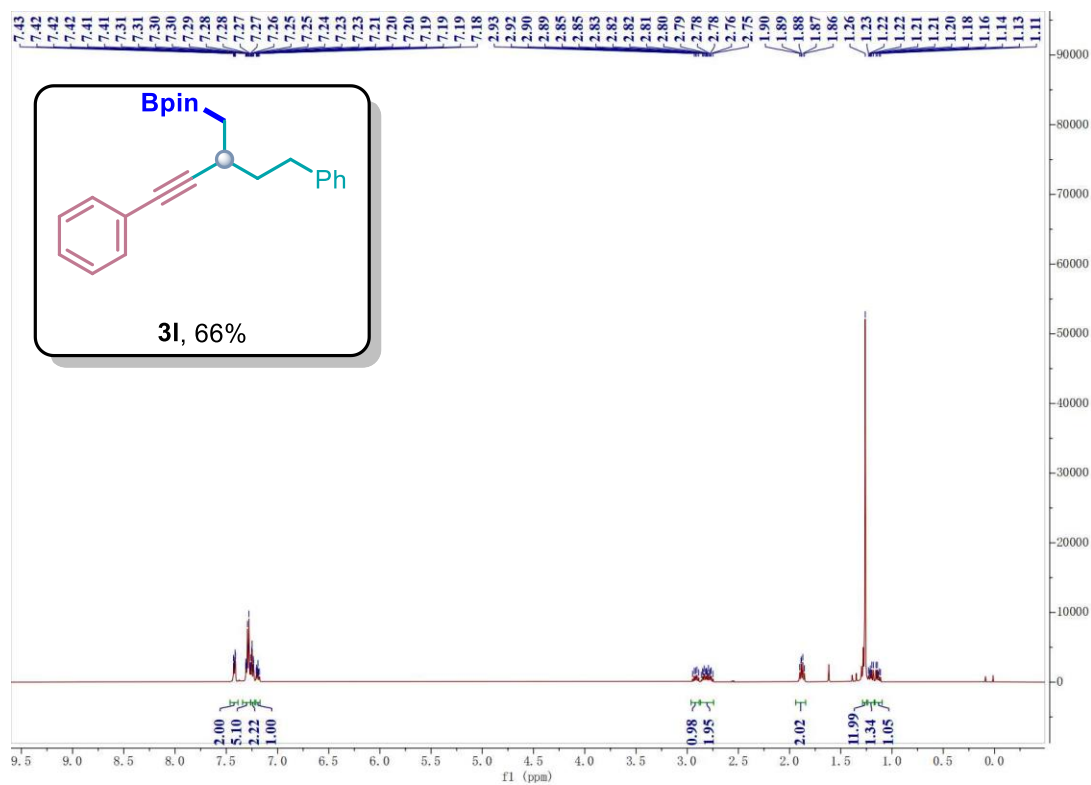


^{11}B NMR (160 MHz, Chloroform-*d*)

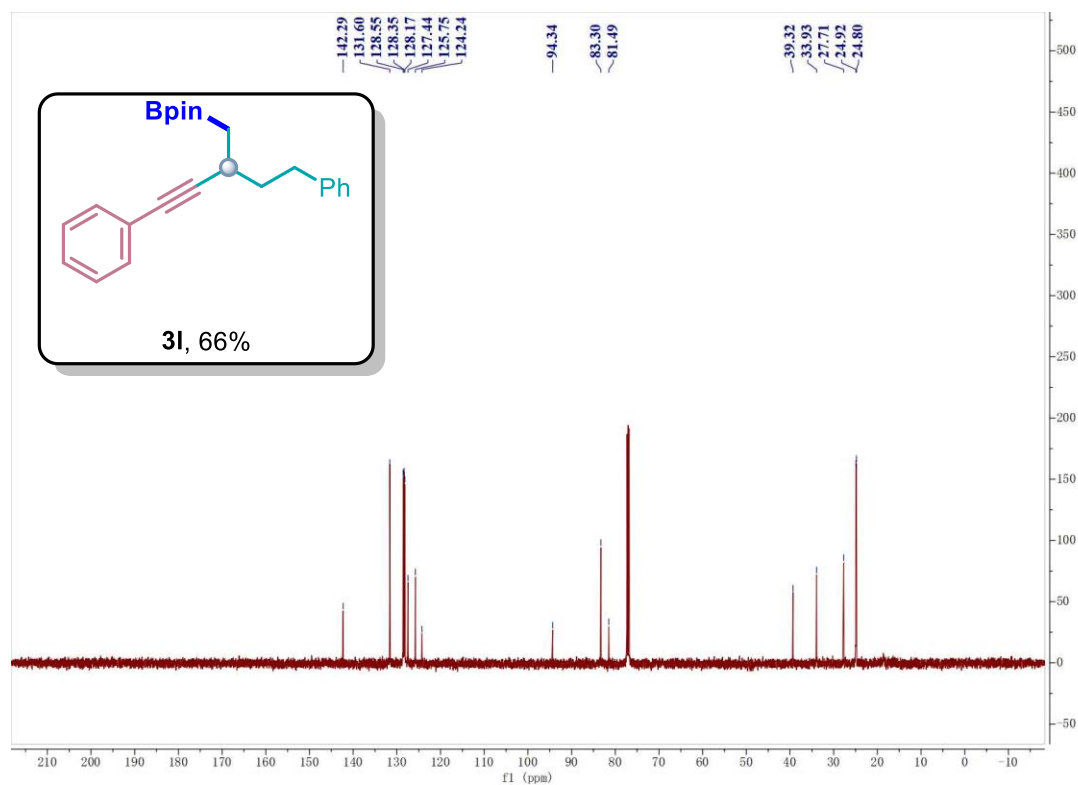


4,4,5,5-tetramethyl-2-(2-phenethyl-4-phenylbut-3-yn-1-yl)-1,3,2-dioxaborolane (3I)

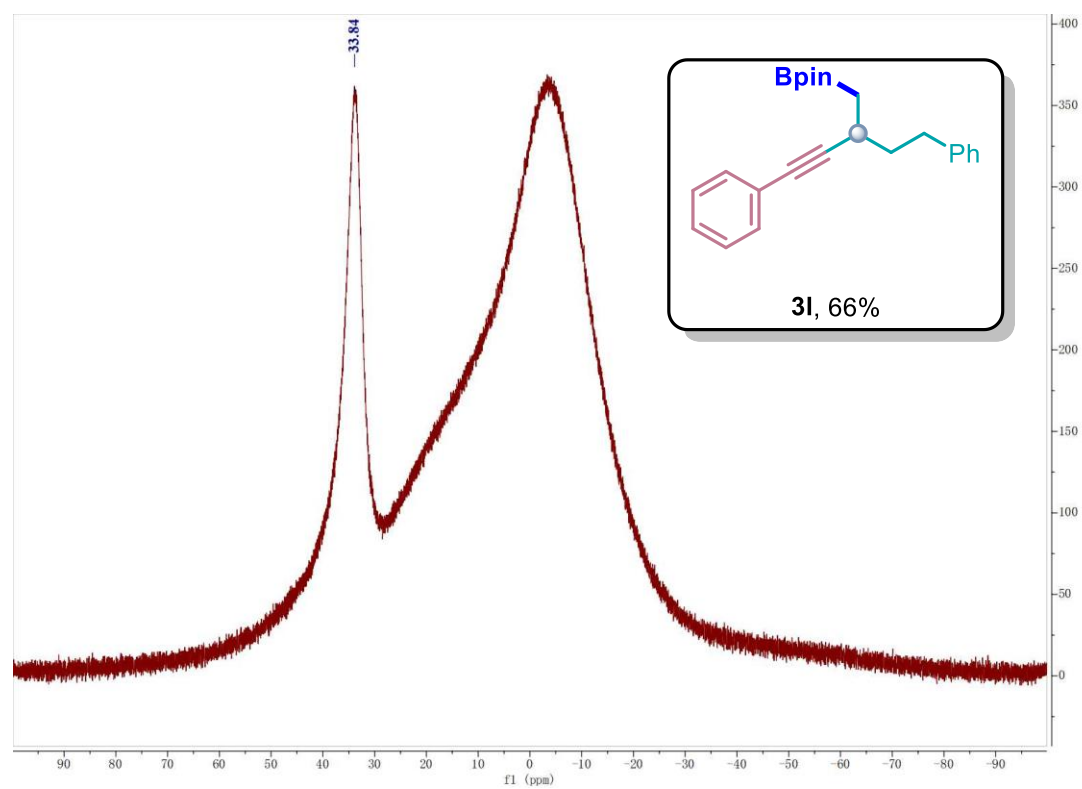
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

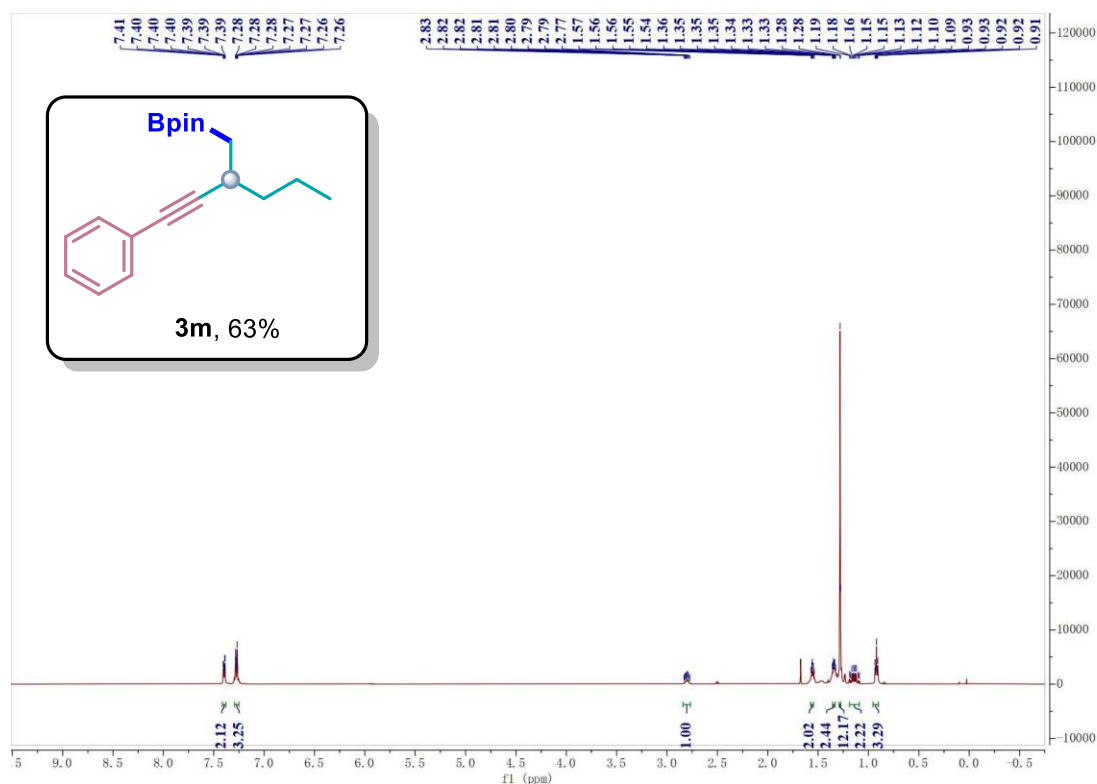


^{11}B NMR (160 MHz, Chloroform-*d*)

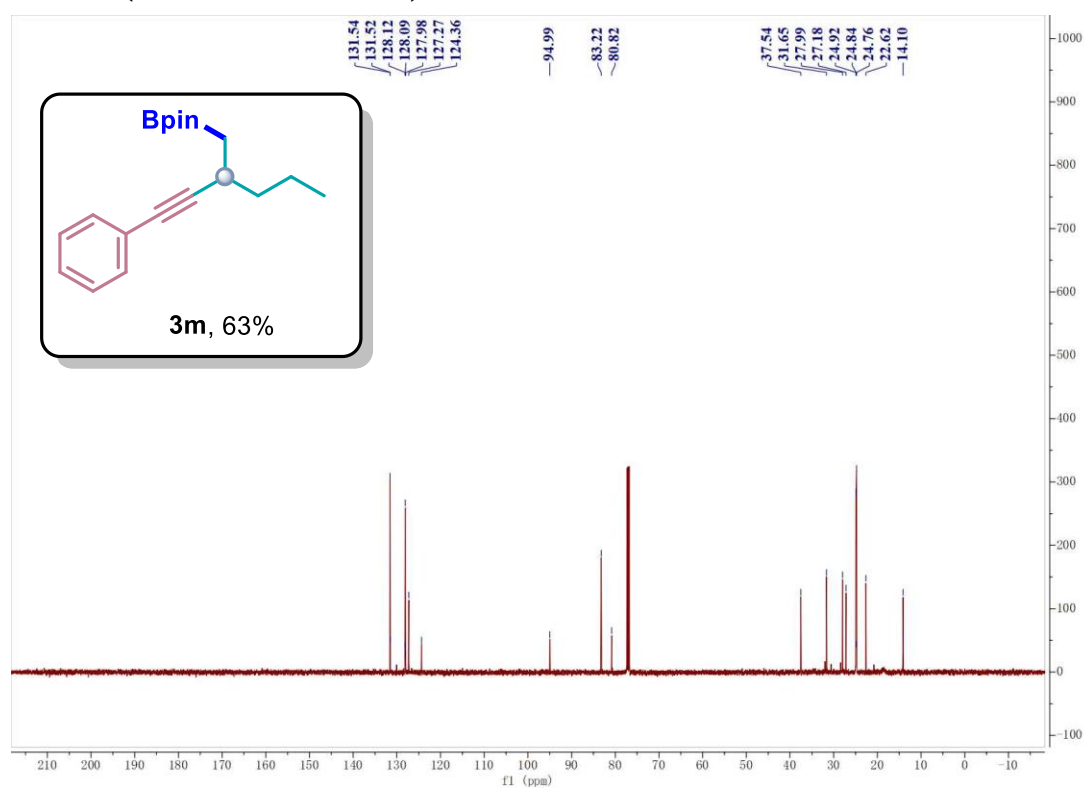


4,4,5,5-tetramethyl-2-(2-(phenylethynyl)pentyl)-1,3,2-dioxaborolane (3m)

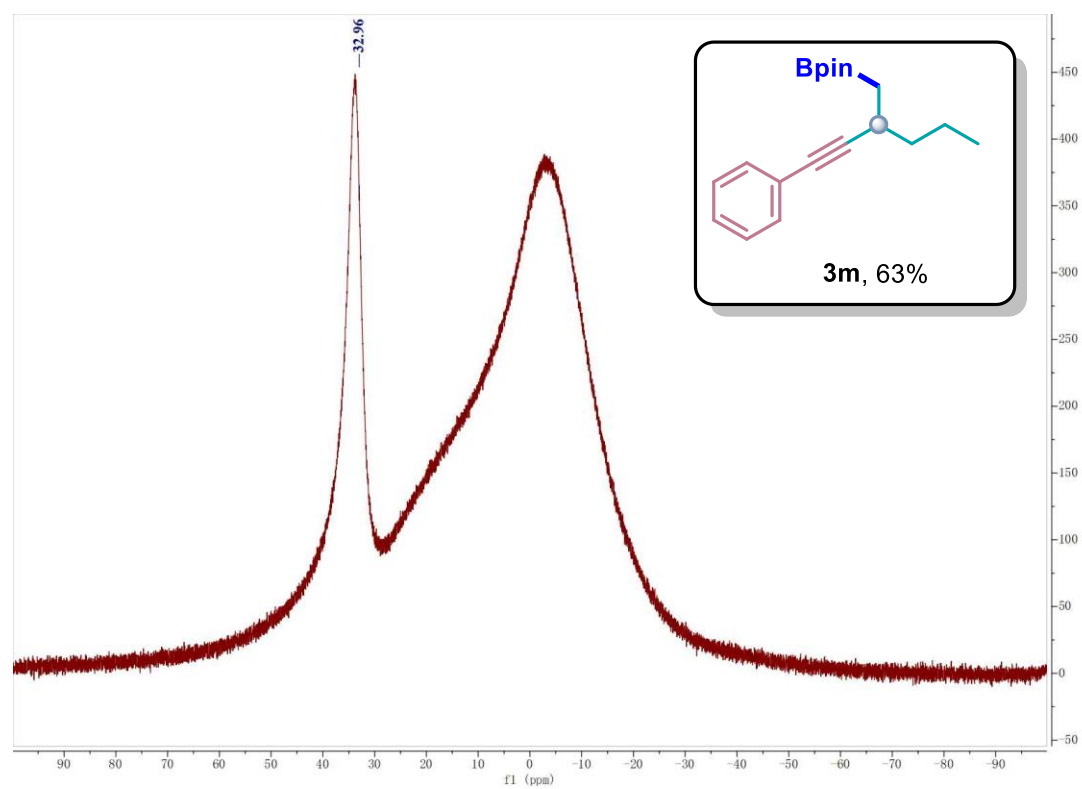
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

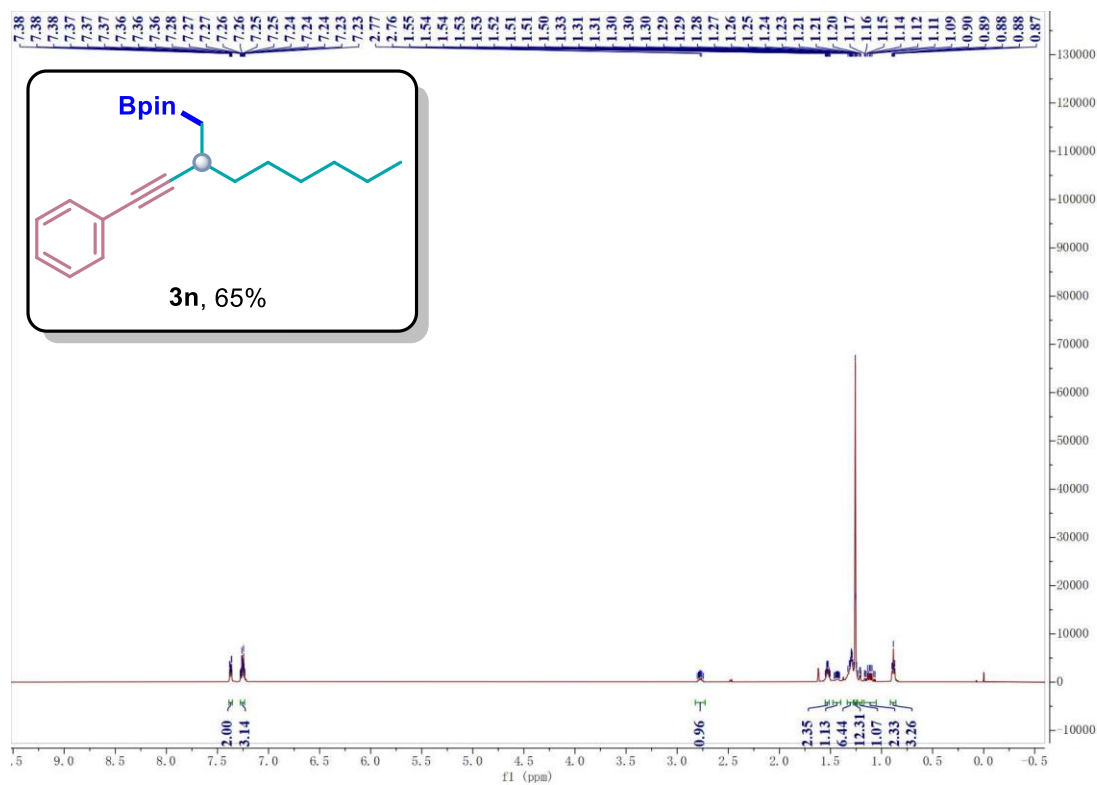


^{11}B NMR (160 MHz, Chloroform-*d*)

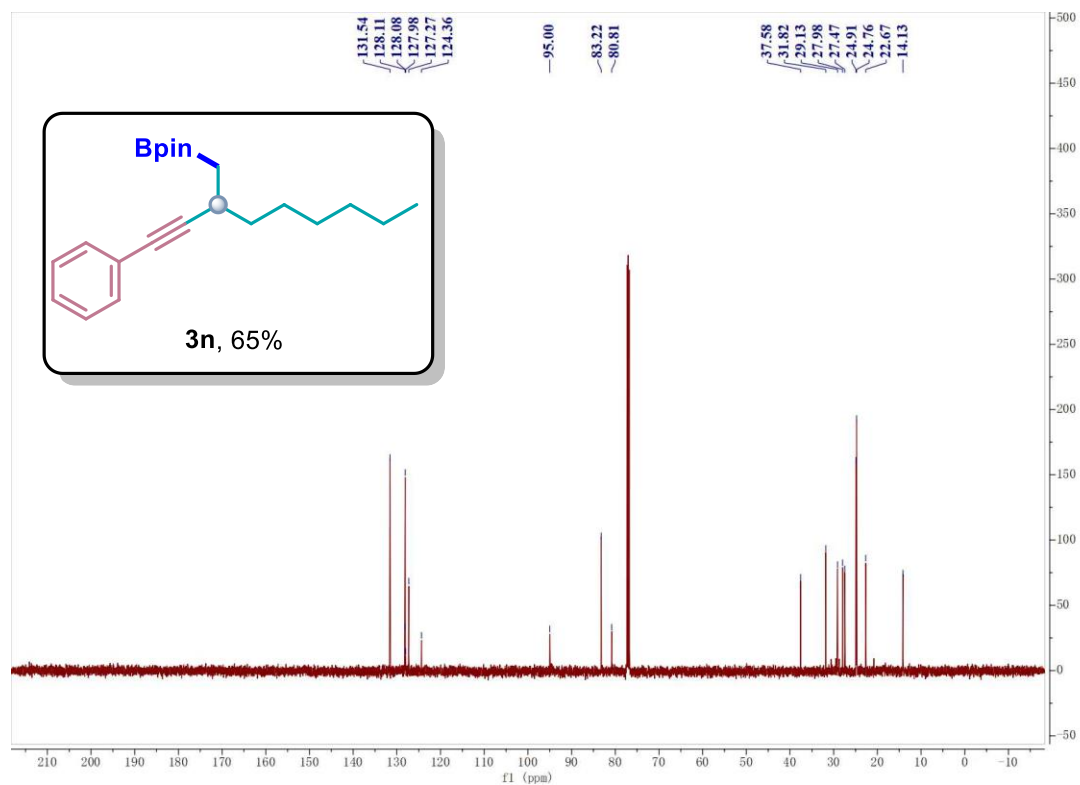


4,4,5,5-tetramethyl-2-(2-(phenylethynyl)octyl)-1,3,2-dioxaborolane (3n)

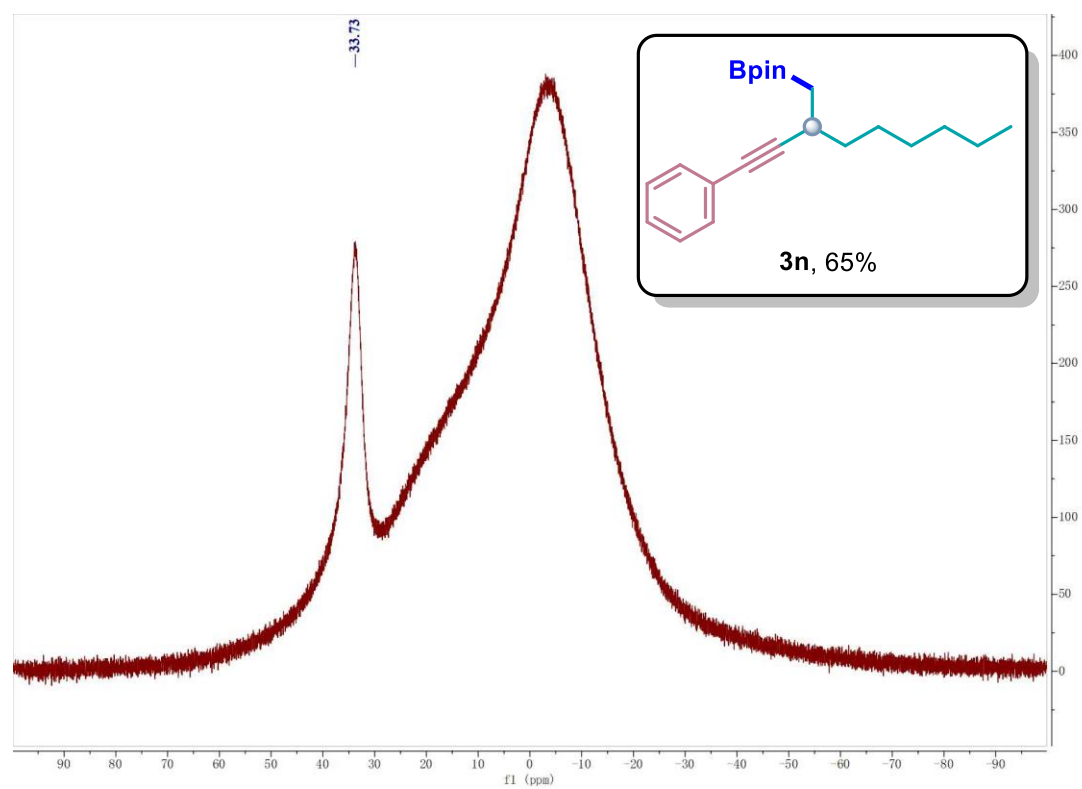
¹H NMR (500 MHz, Chloroform-*d*)



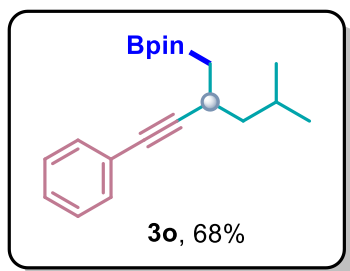
¹³C NMR (126 MHz, Chloroform-*d*)



^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

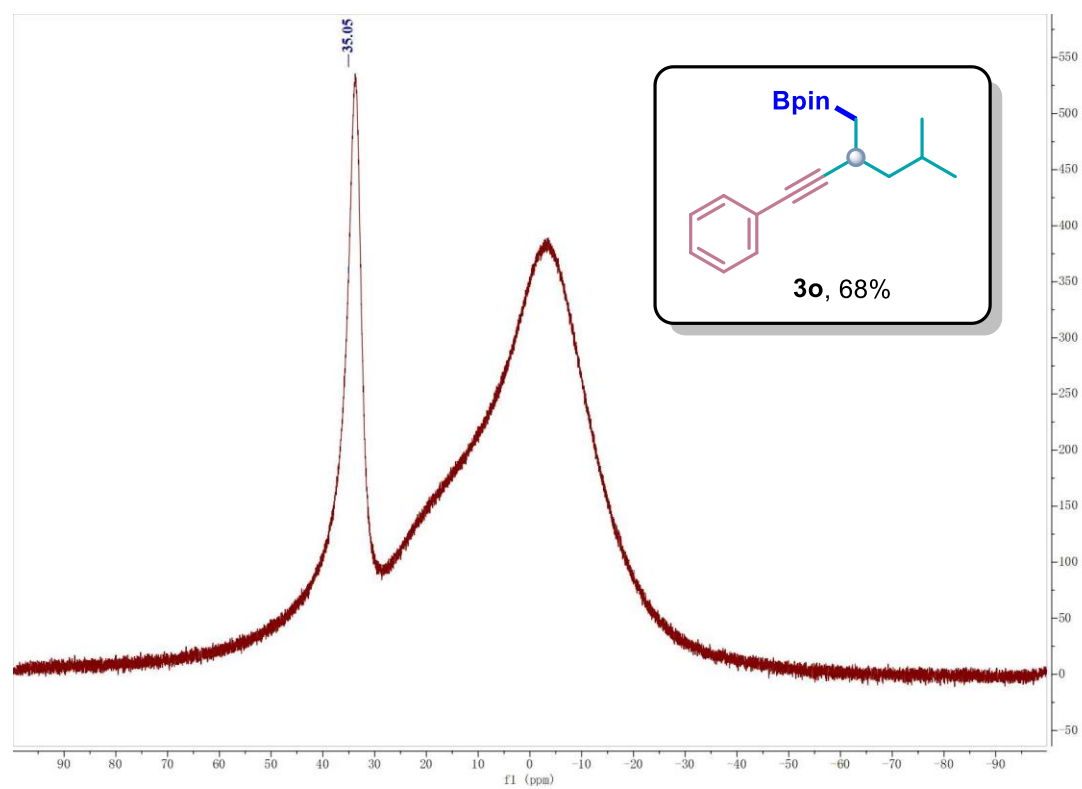


Chemical structure of **3o** is shown in the inset: **3o**, 68%.

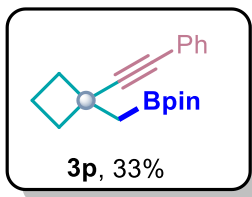
¹H NMR spectrum (CDCl₃) of compound **3o**. The x-axis represents the chemical shift in ppm (f1), ranging from -10 to 210. The y-axis represents the intensity. The spectrum shows several peaks corresponding to the structure of **3o**, including aromatic protons, alkyne protons, and alkenic protons. The integration values are provided below the baseline.

Chemical Shift (ppm)	Integration
7.2-7.4 (multiplet)	1.00
6.8 (doublet)	1.00
5.1 (singlet)	1.00
4.8 (doublet)	1.00
3.0 (singlet)	1.00
2.1-2.5 (multiplet)	1.00

^{11}B NMR (160 MHz, Chloroform-*d*)

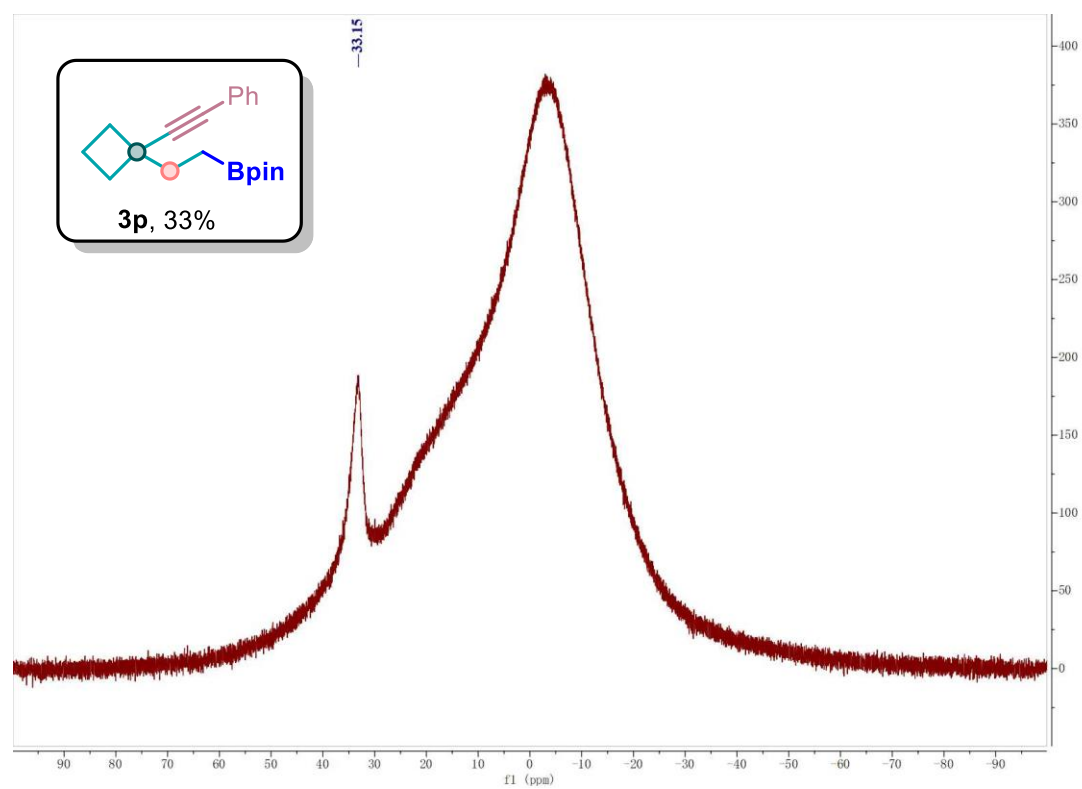


¹H NMR (500 MHz, Chloroform-*d*)



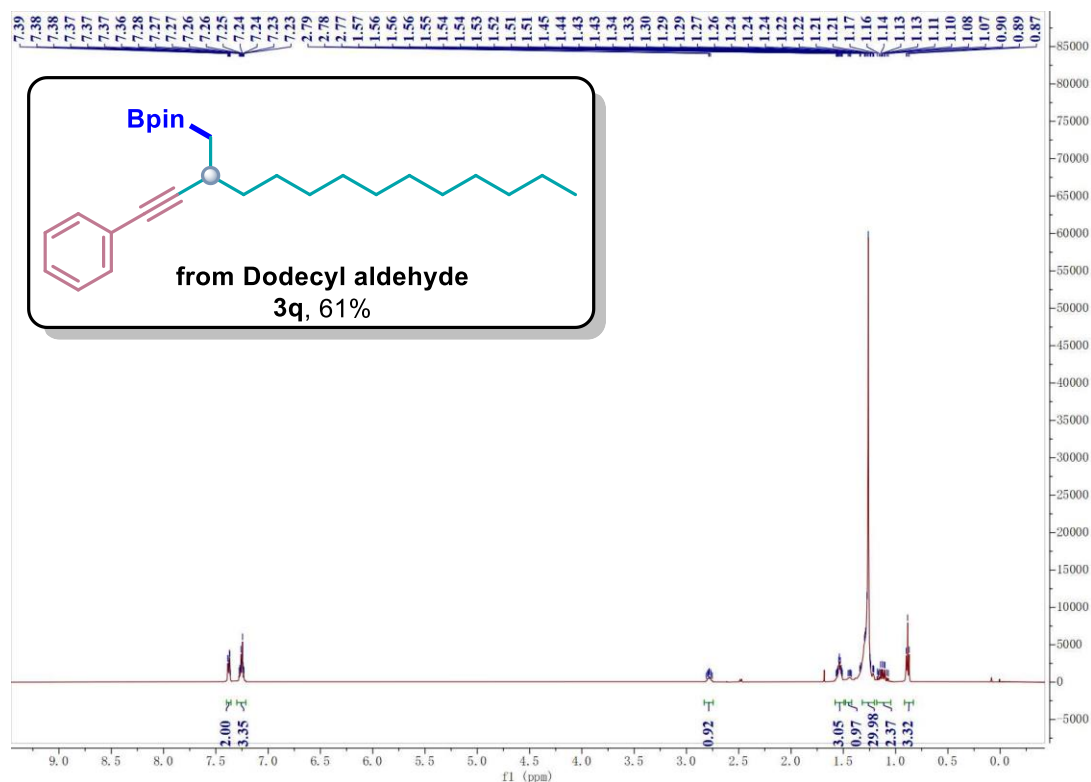
Chemical structure of **3p** is shown in the inset. The structure is a cyclobutane ring with a phenyl group (Ph) and a Bpin group. The spectrum shows peaks at 131.59, 131.48, 128.05, 127.20, 98.09, 83.01, 80.59, 36.74, 34.34, 31.51, 30.15, 28.88, 24.86, 24.67, and 16.24 ppm. The x-axis is labeled 'f1 (ppm)' and ranges from -10 to 210. The y-axis is labeled 'Intensity' and ranges from -50 to 700.

^{11}B NMR (160 MHz, Chloroform-*d*)

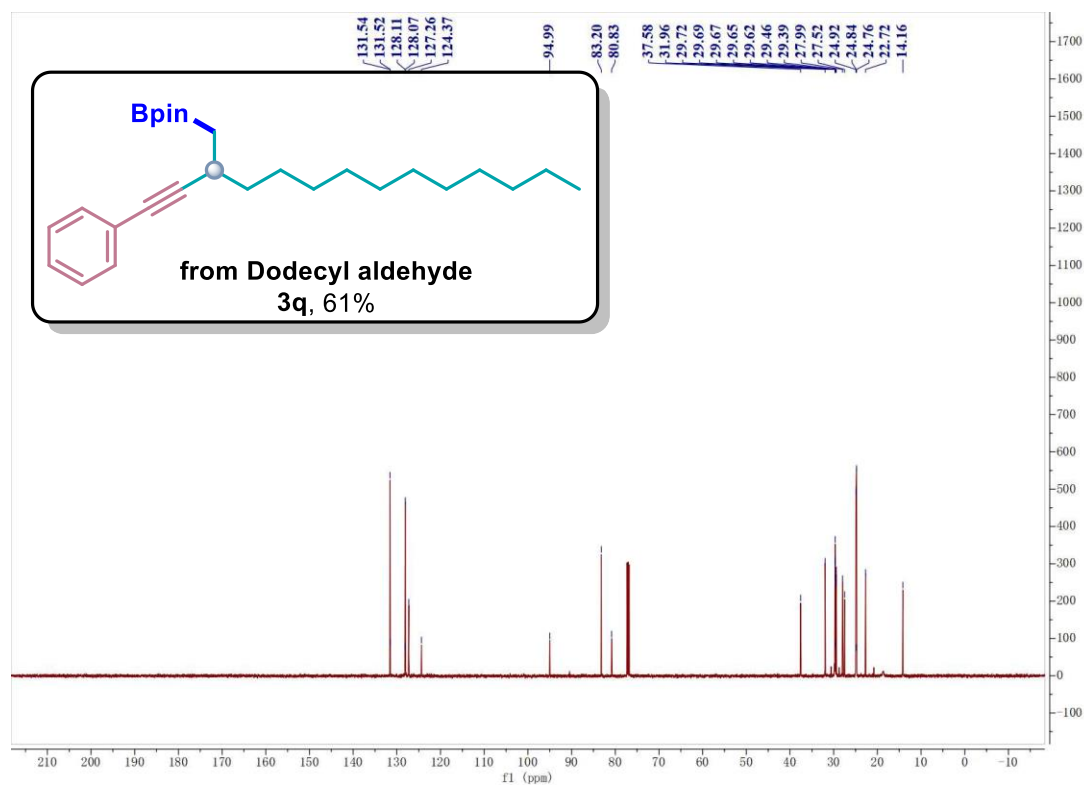


4,4,5,5-tetramethyl-2-(2-(phenylethynyl)tridecyl)-1,3,2-dioxaborolane (3q)

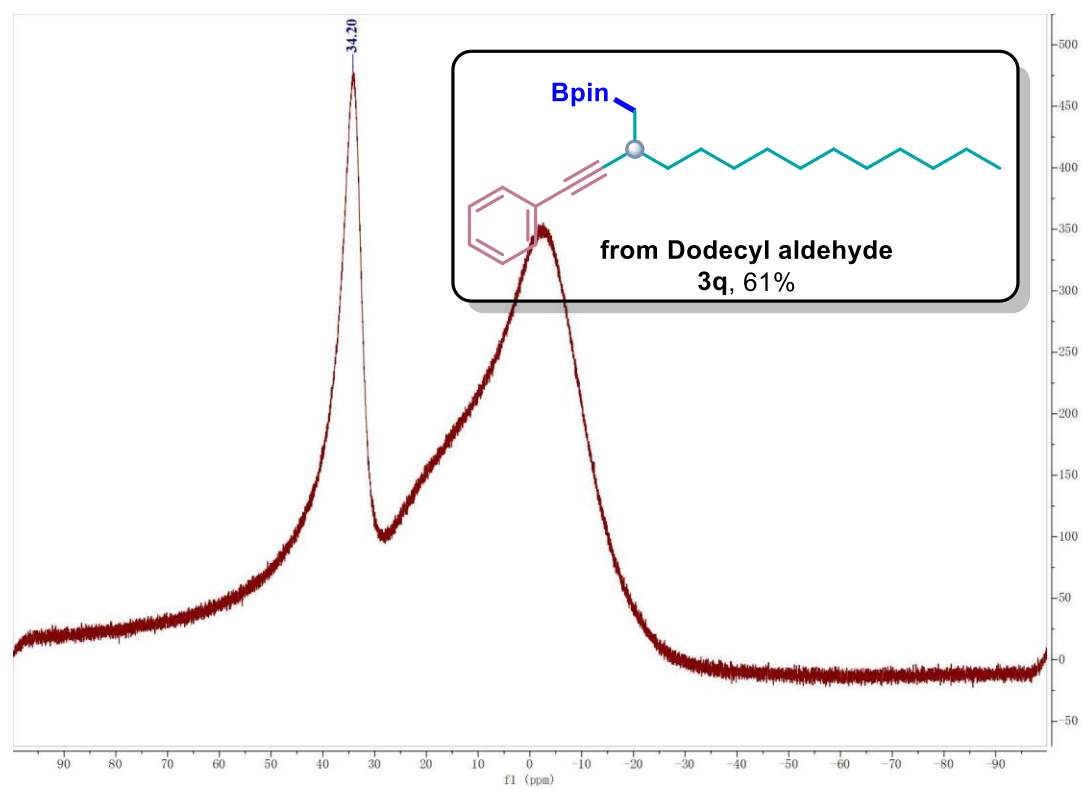
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

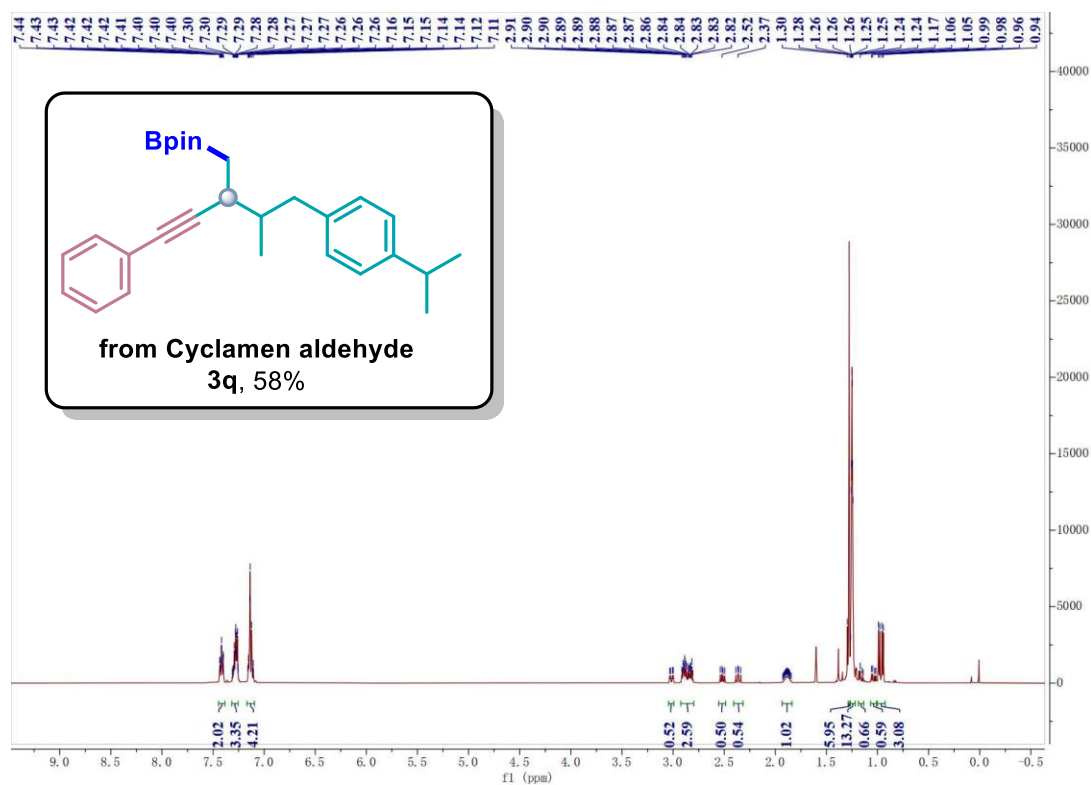


^{11}B NMR (160 MHz, Chloroform-*d*)

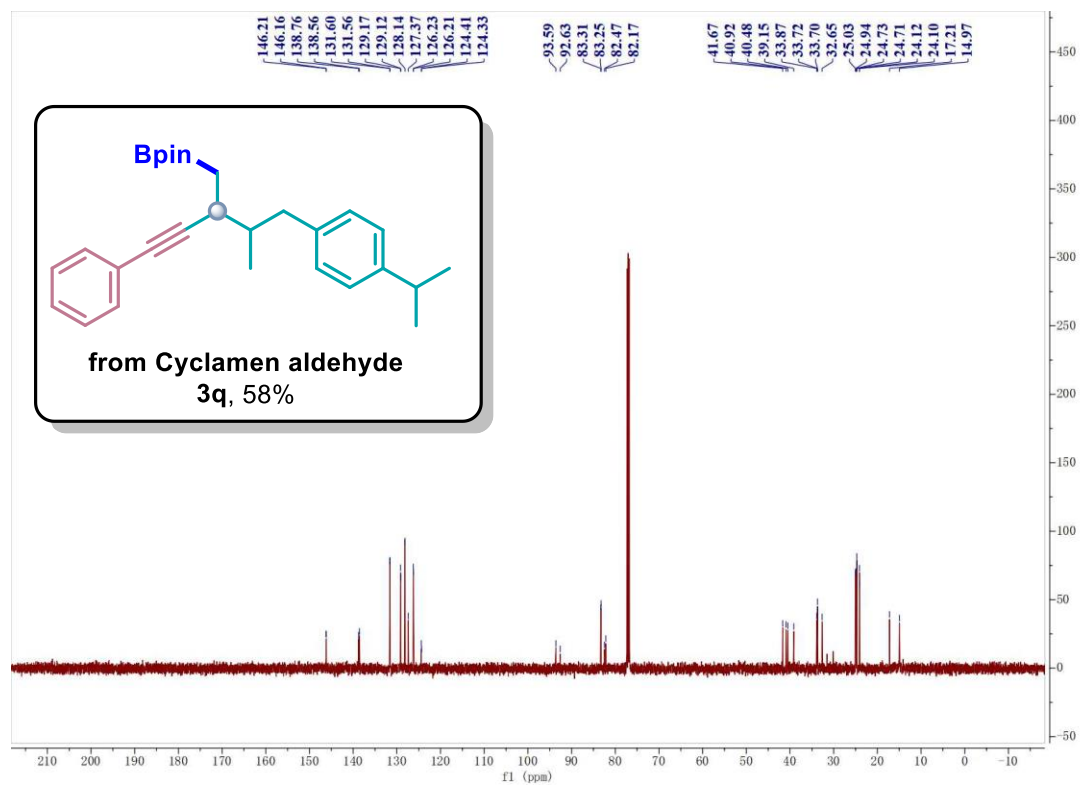


2-(2-(1-(4-isopropylphenyl)propan-2-yl)-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3q)

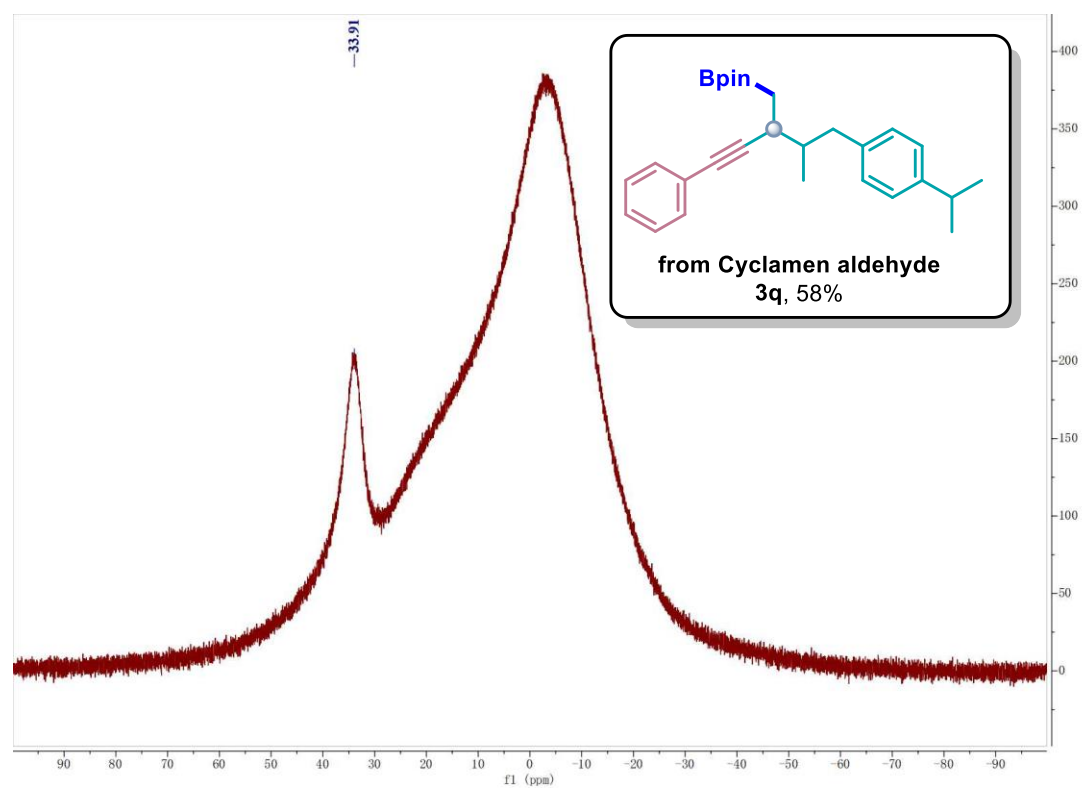
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

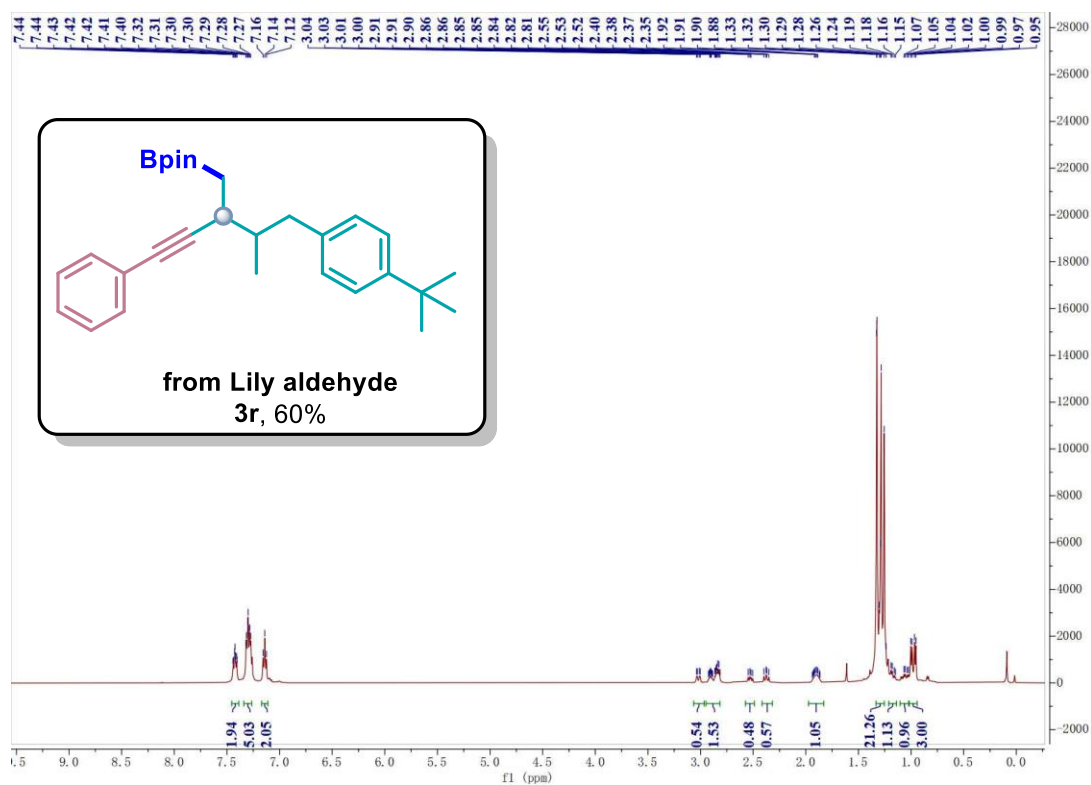


^{11}B NMR (160 MHz, Chloroform-*d*)

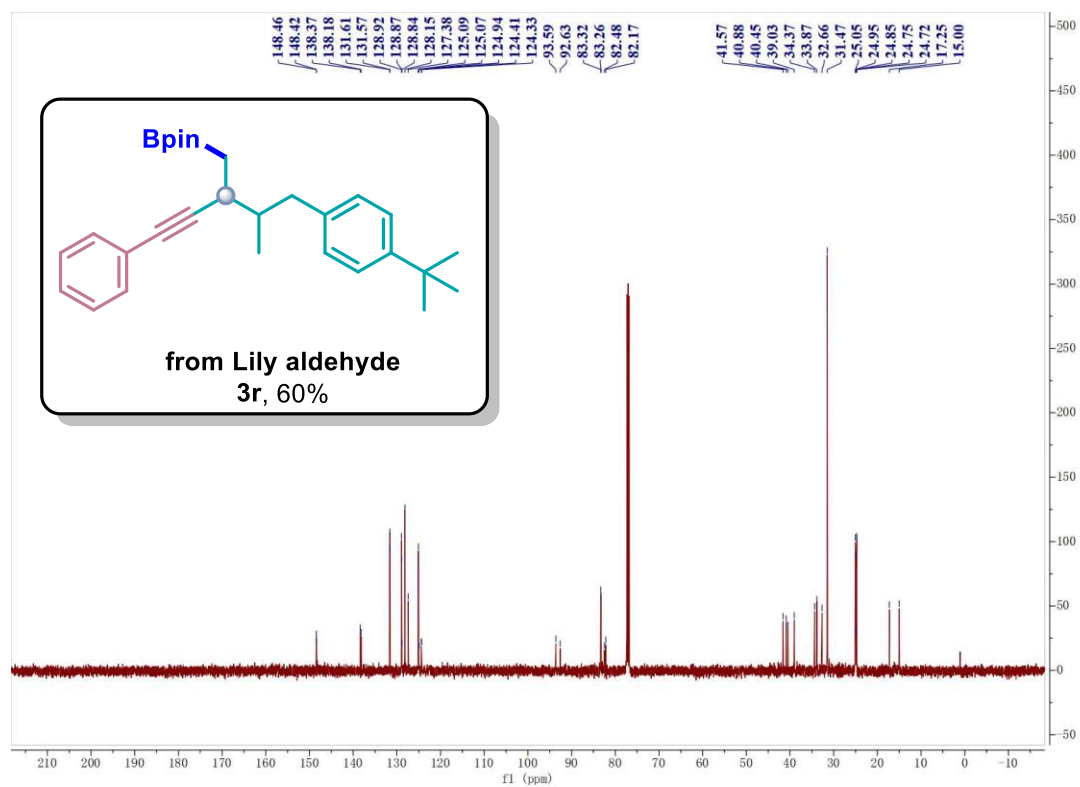


2-(2-(1-(4-isopropylphenyl)propan-2-yl)-4-phenylbut-3-yn-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3r)

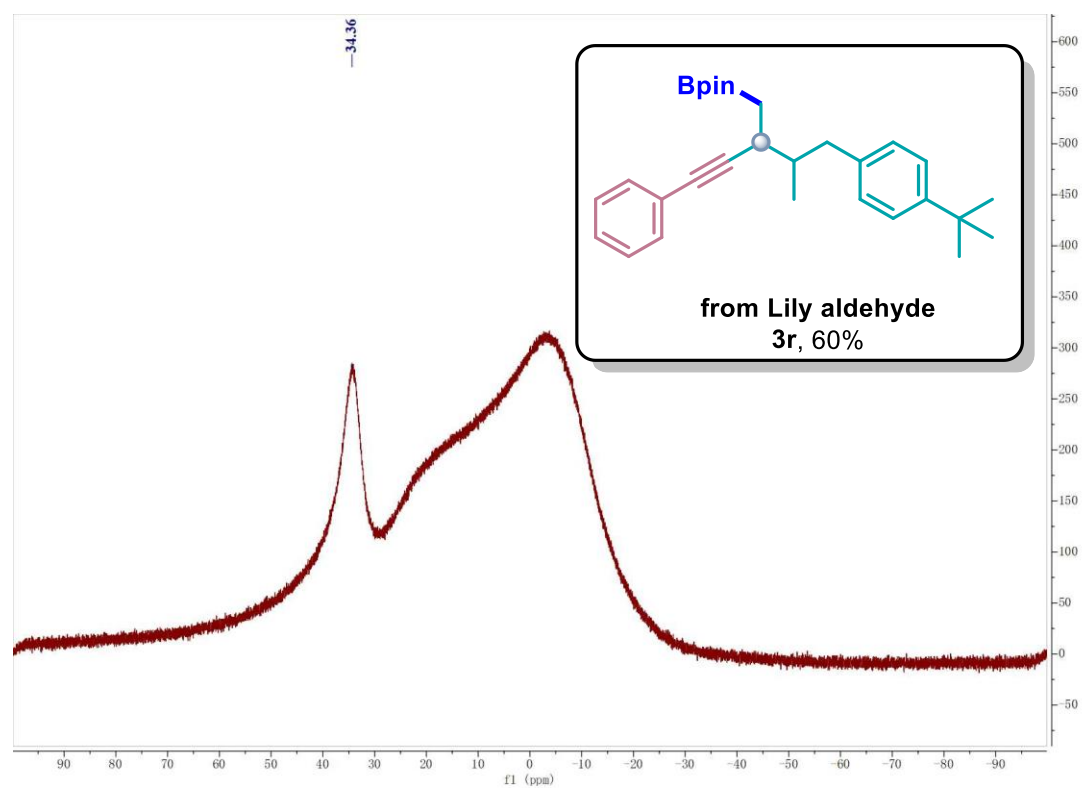
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

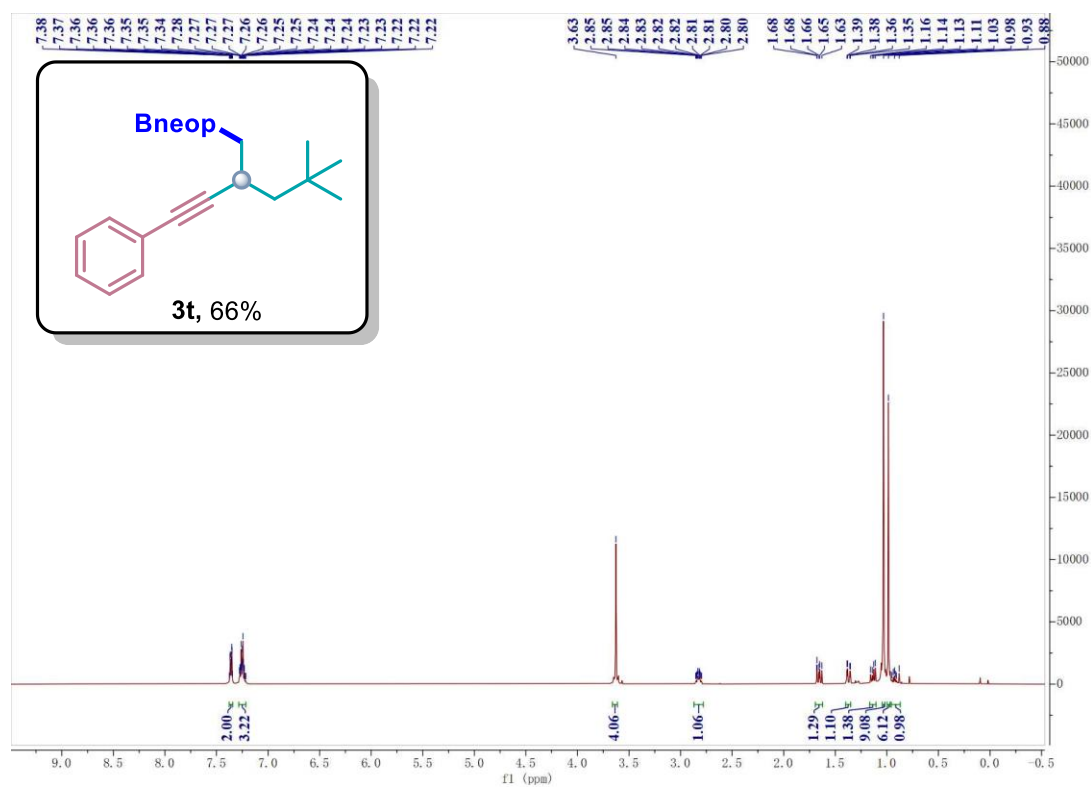


^{11}B NMR (160 MHz, Chloroform-*d*)

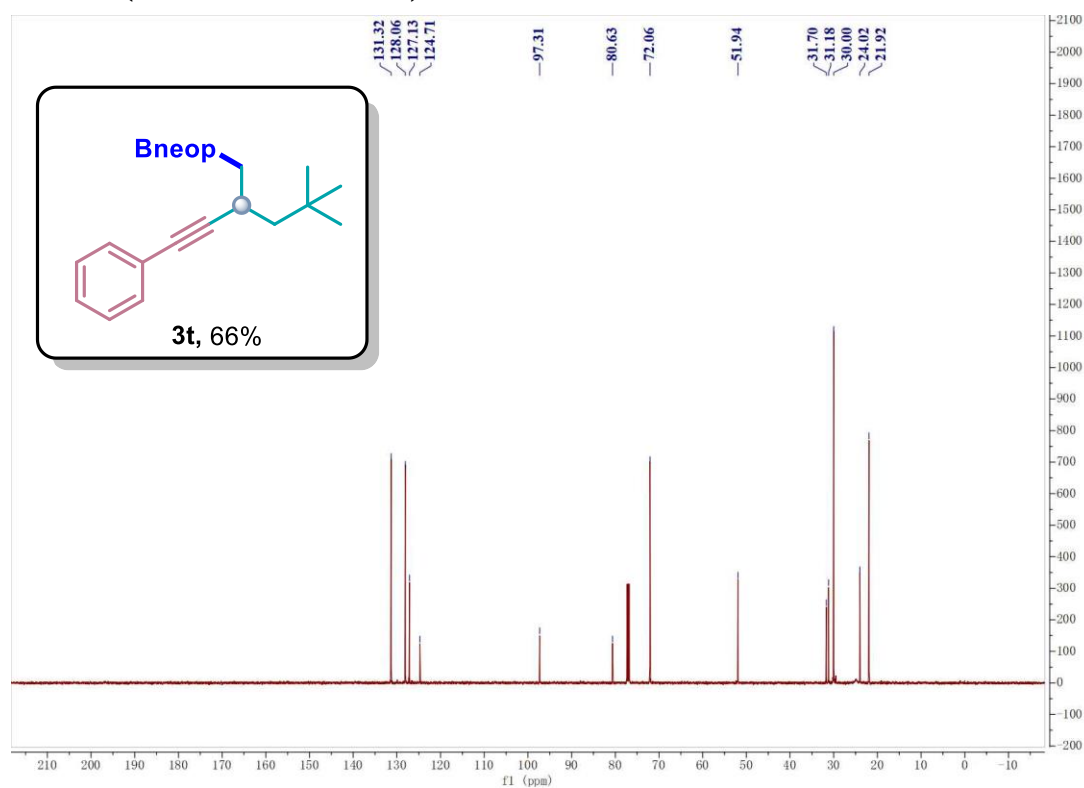


2-(4,4-dimethyl-2-(phenylethynyl)pentyl)-5,5-dimethyl-1,3,2-dioxaborinane (3t)

¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

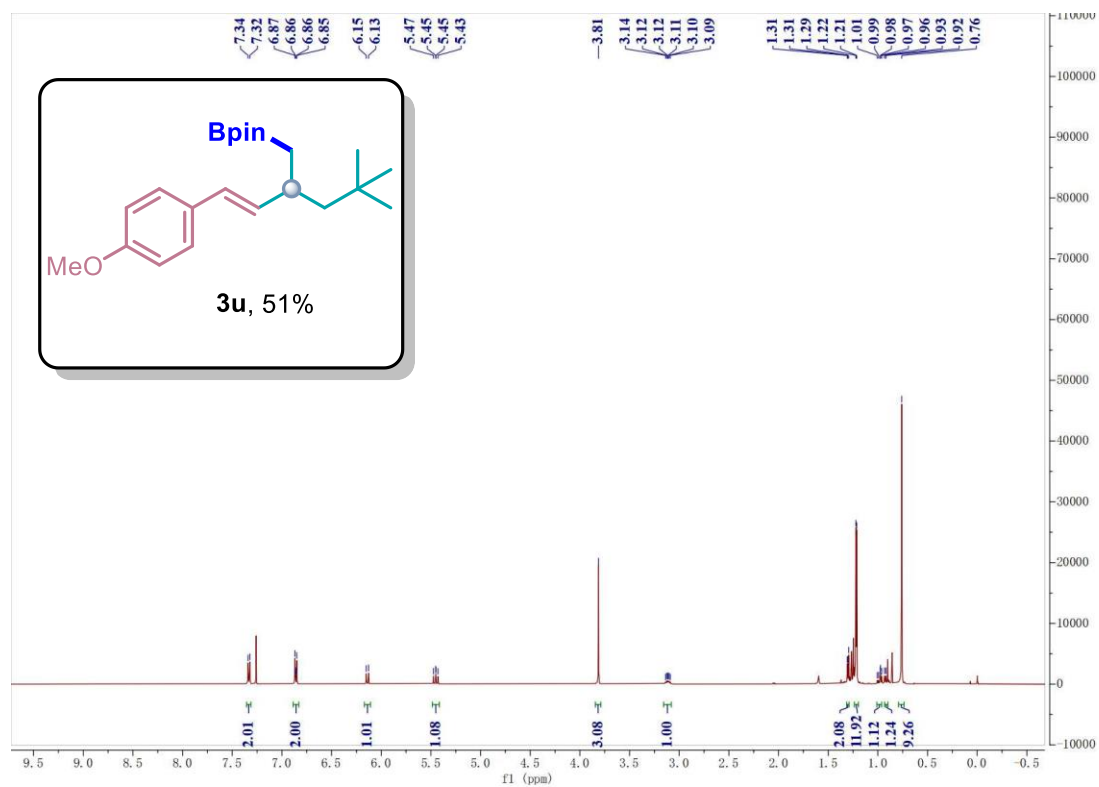


3t, 66%

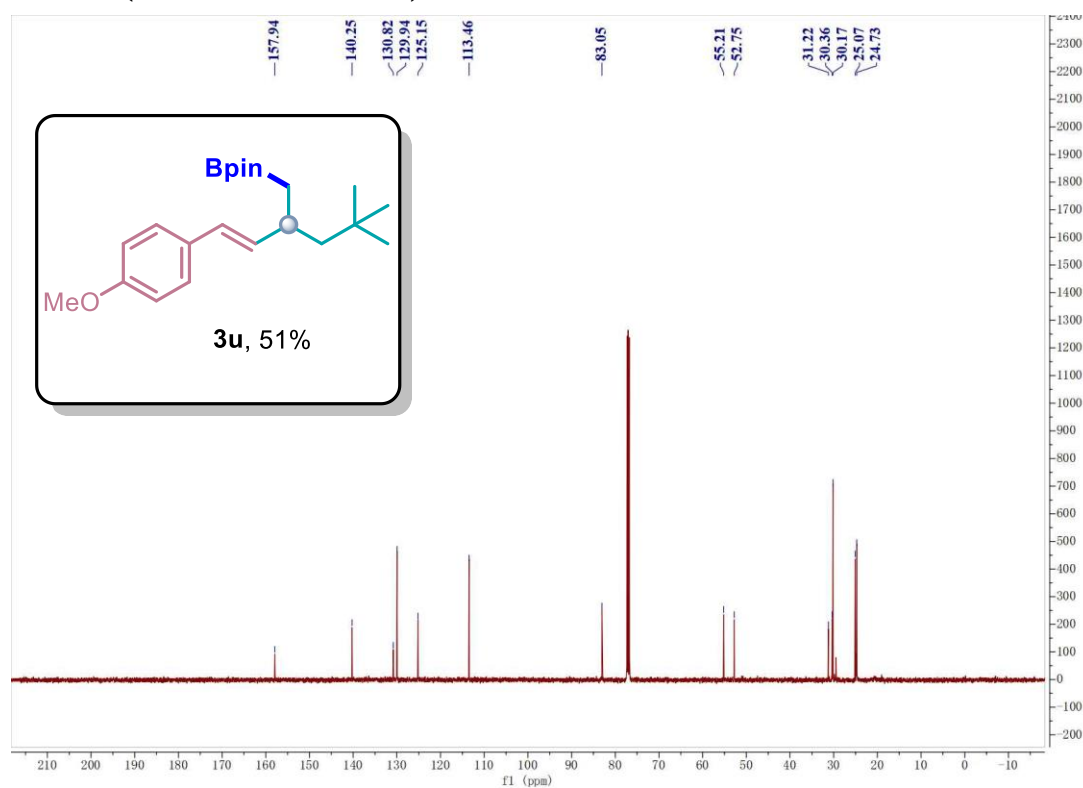
Chemical structure of **3t** is shown in the inset: CC(C)(C)CC#CC(=O)c1ccc(cc1). The structure features a tert-butyl group (blue) and an alkyne (red).

(E)-2-(2-(4-methoxystyryl)-4,4-dimethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (**3u**)

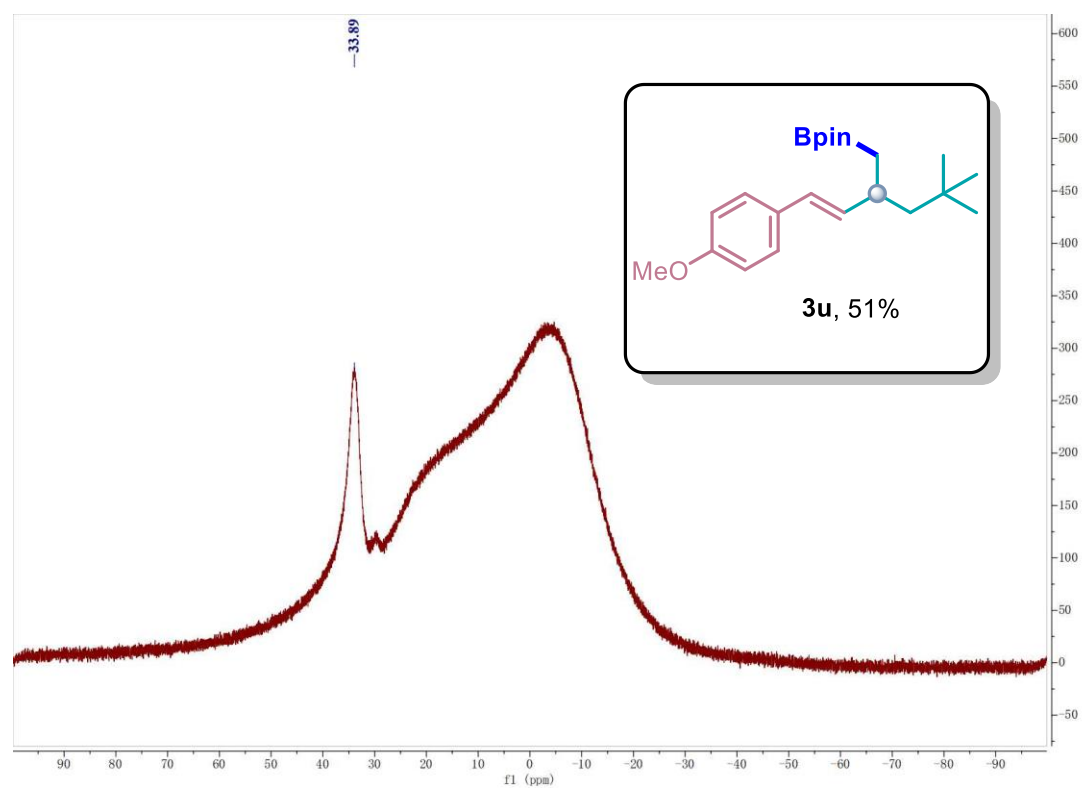
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

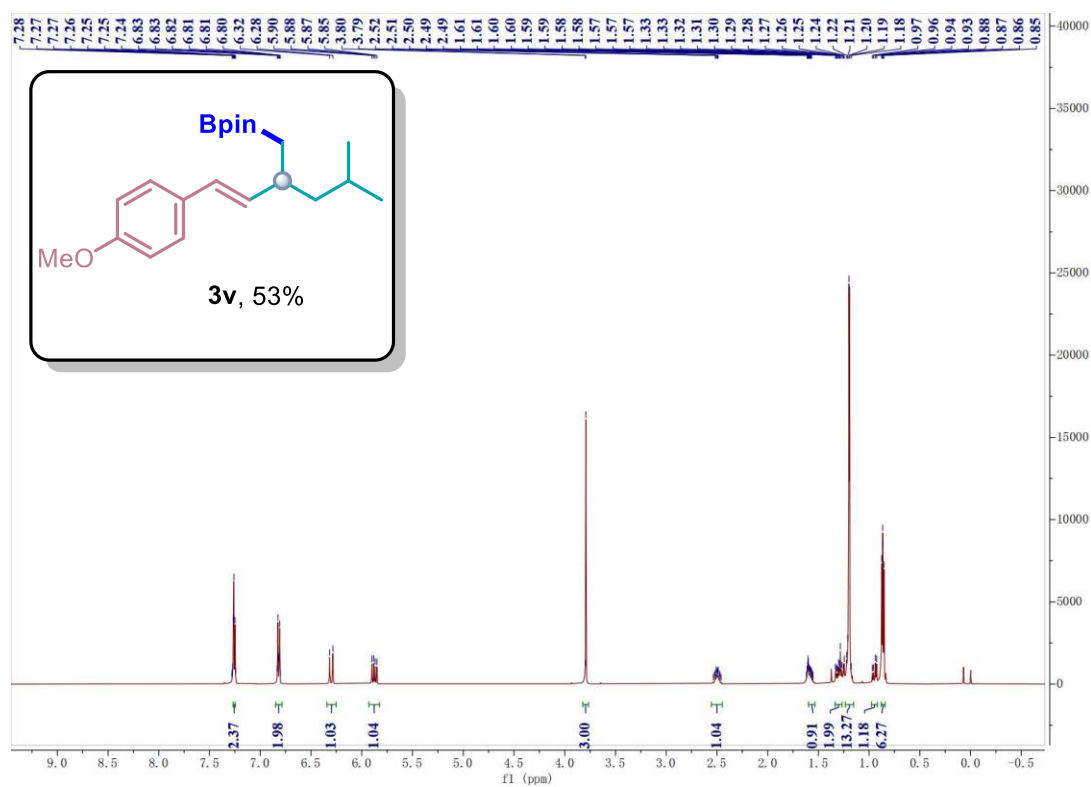


^{11}B NMR (160 MHz, Chloroform-*d*)

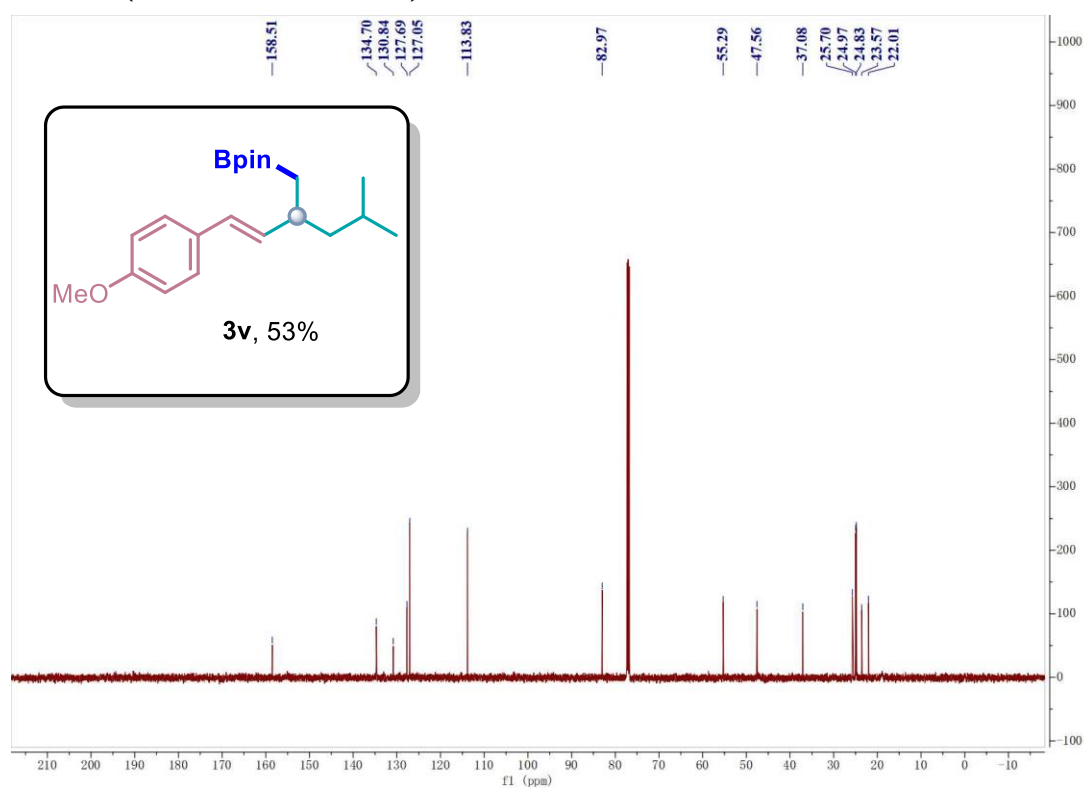


(E)-2-(2-(4-methoxystyryl)-4-methylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3v)

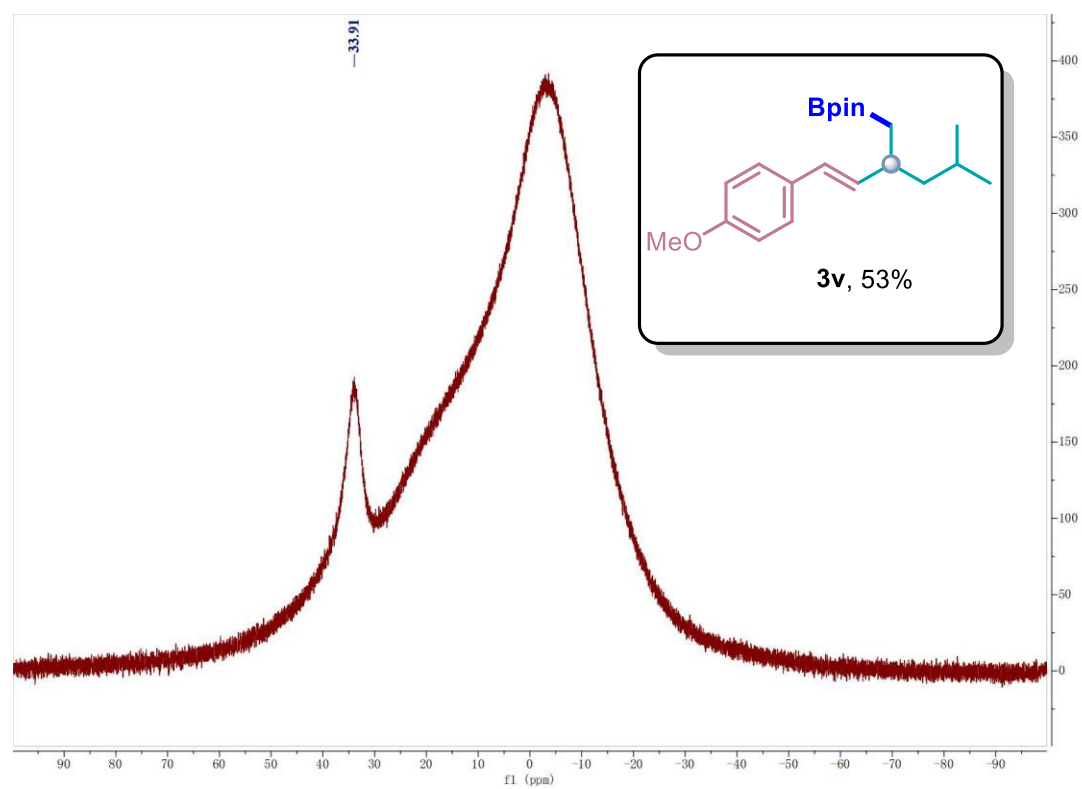
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

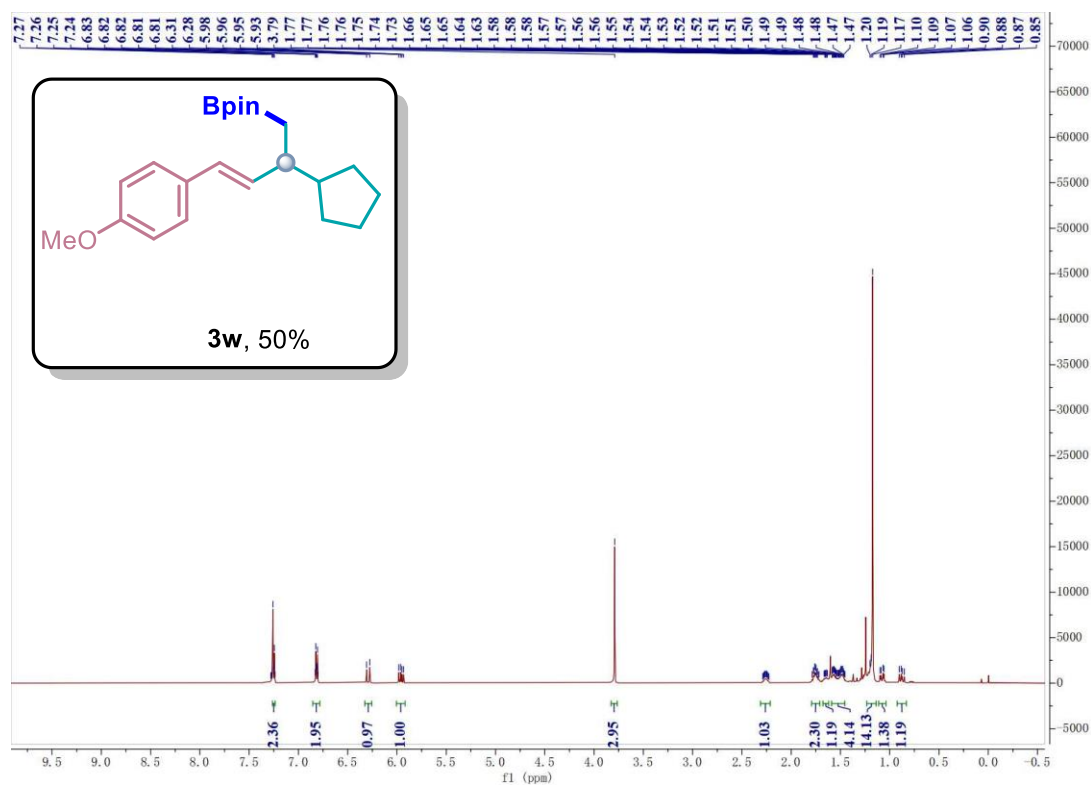


^{11}B NMR (160 MHz, Chloroform-*d*)

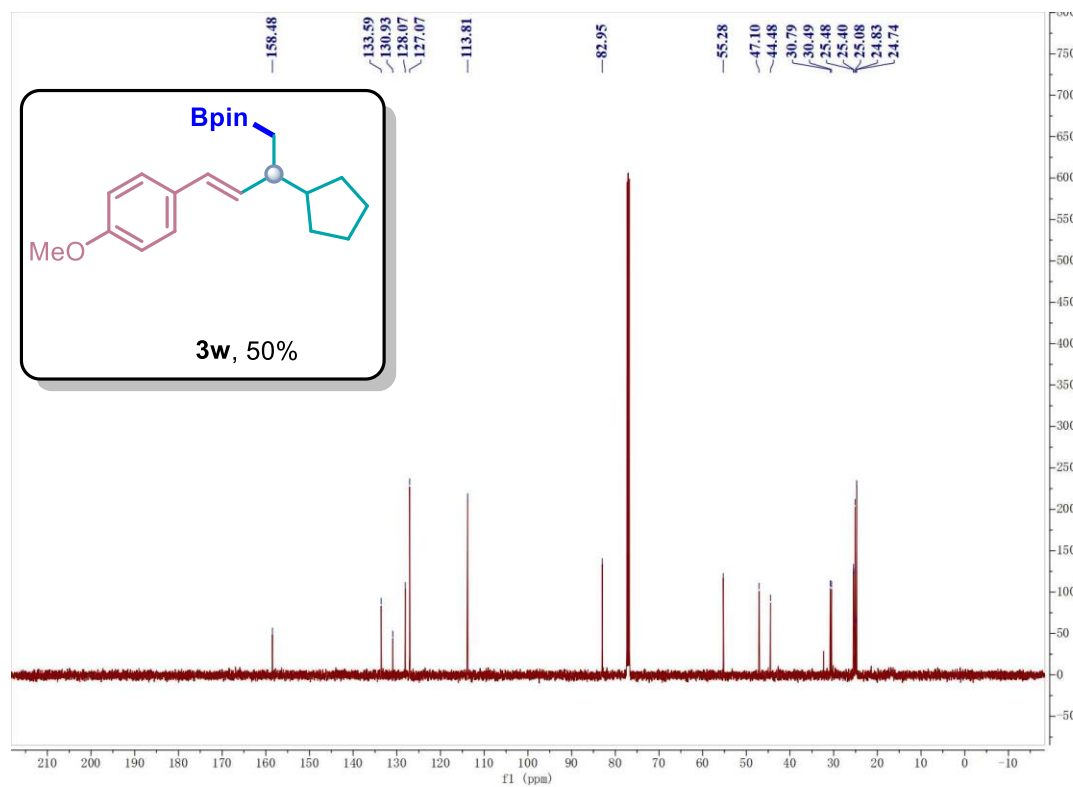


(E)-2-(2-cyclopentyl-4-(4-methoxyphenyl)but-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3w)

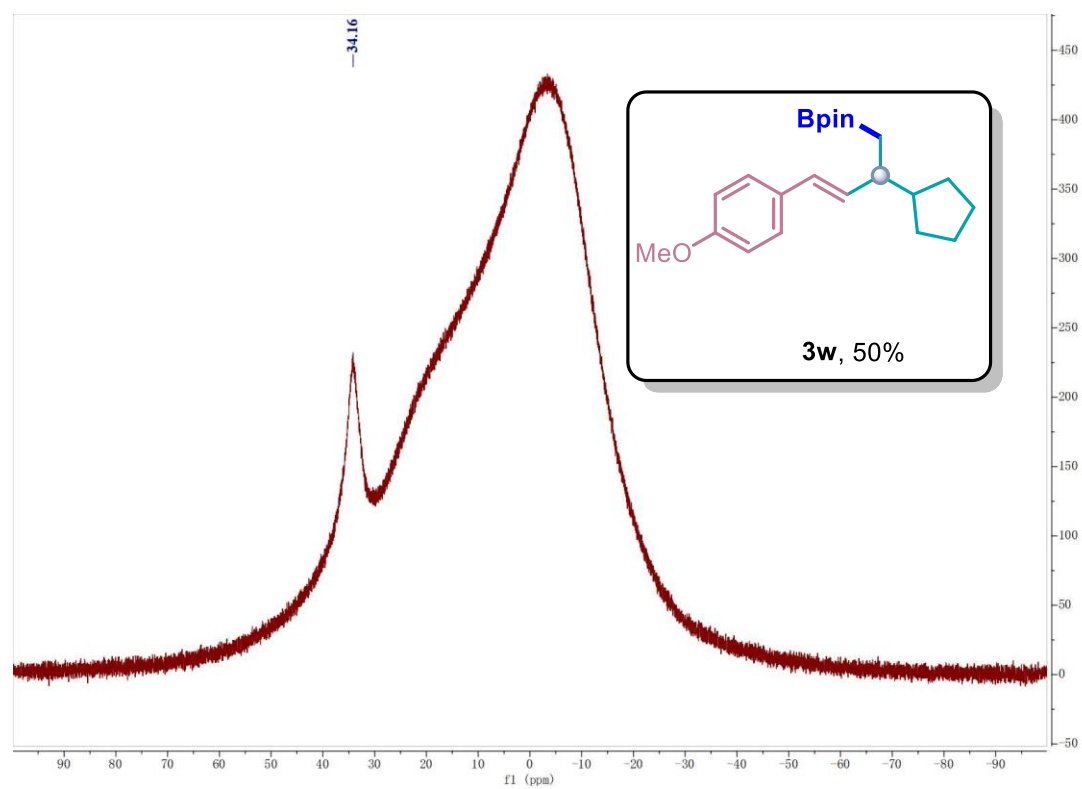
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

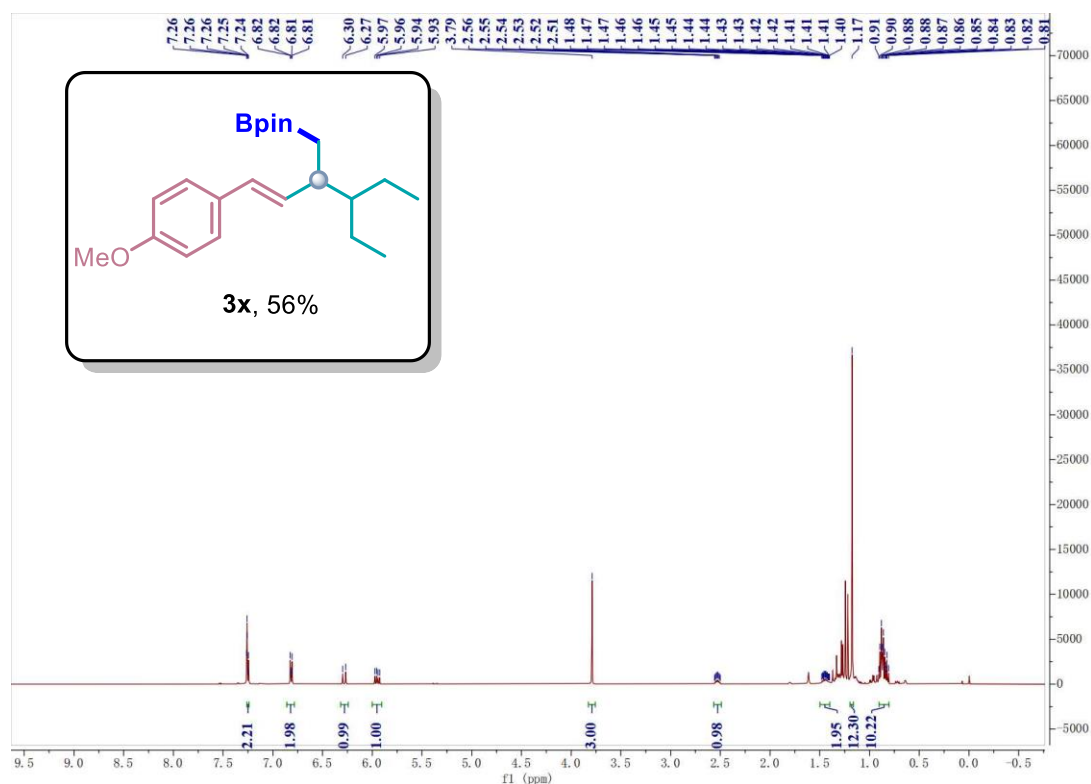


^{11}B NMR (160 MHz, Chloroform-*d*)

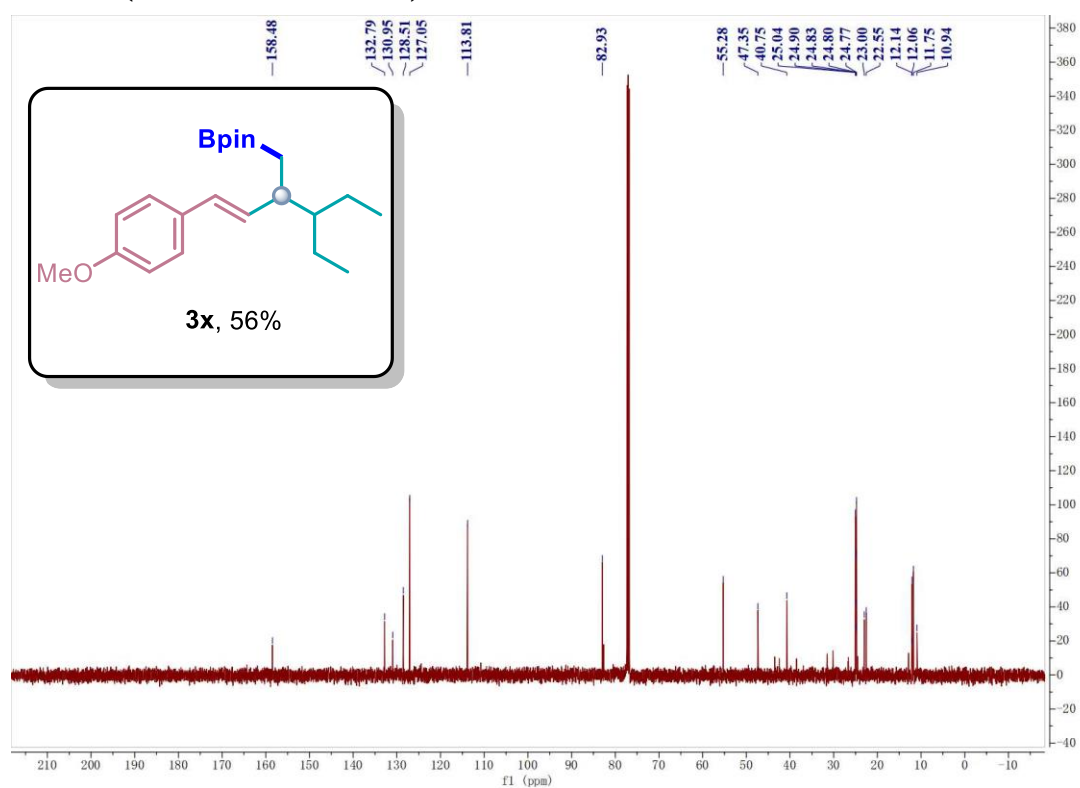


(E)-2-(3-ethyl-2-(4-methoxystyryl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3x)

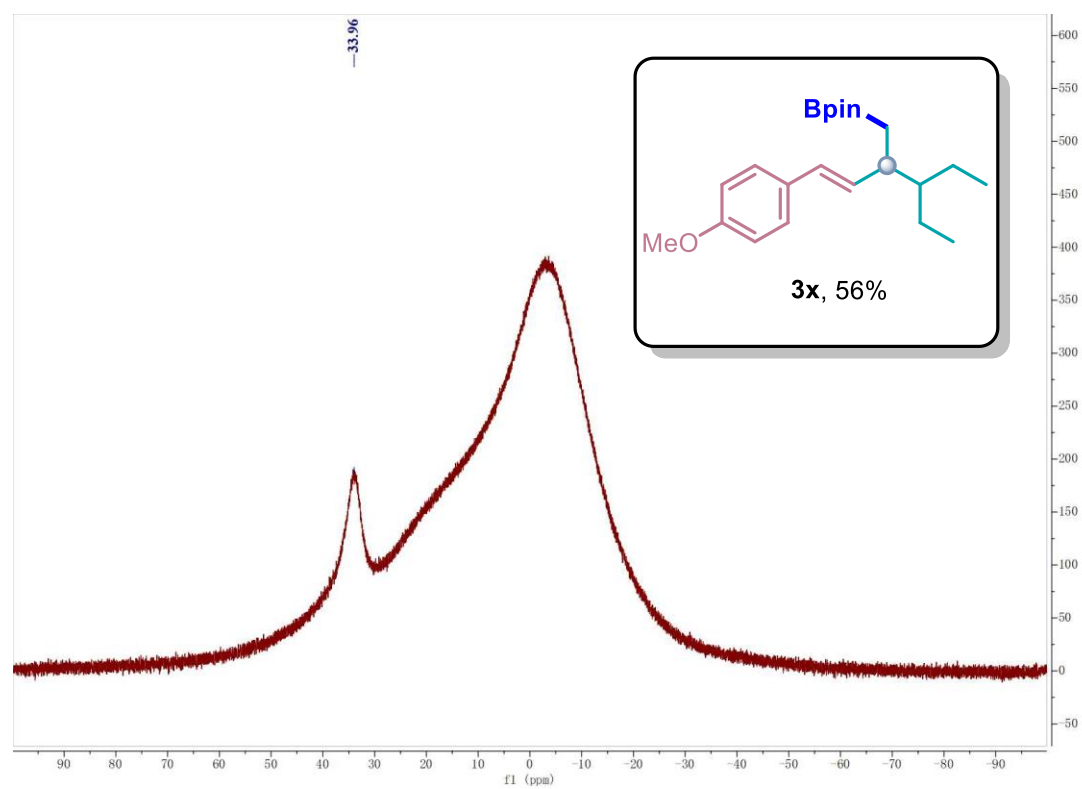
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

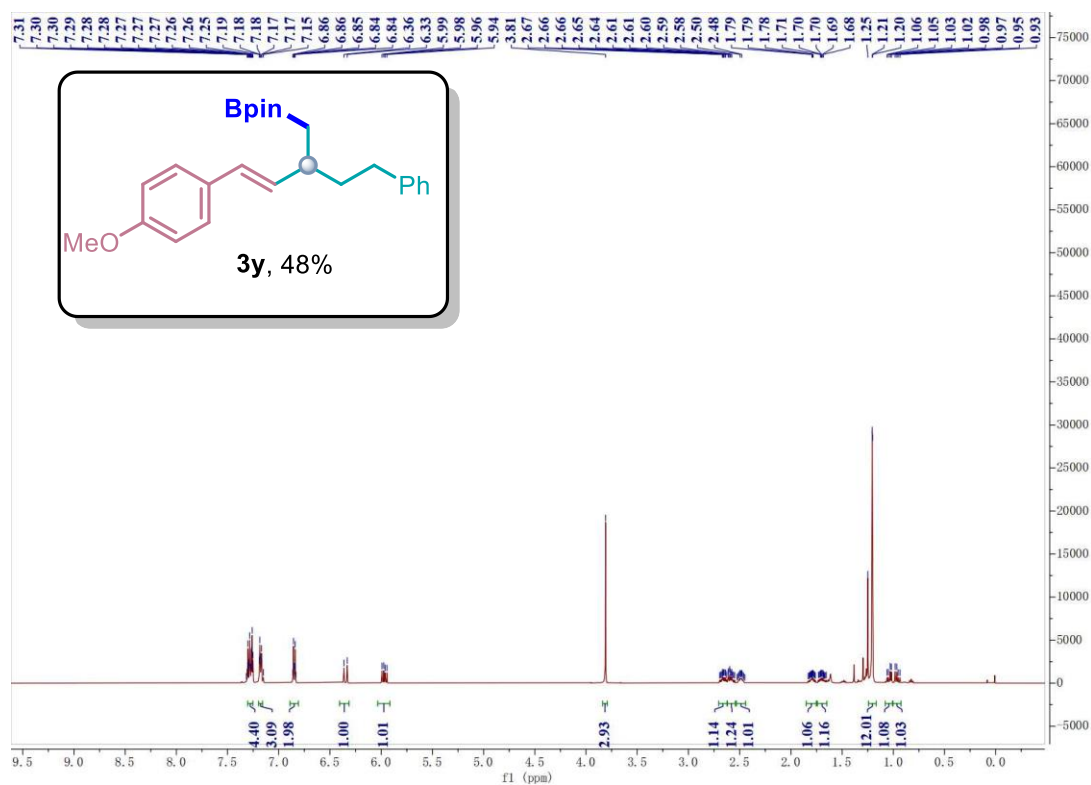


^{11}B NMR (160 MHz, Chloroform-*d*)

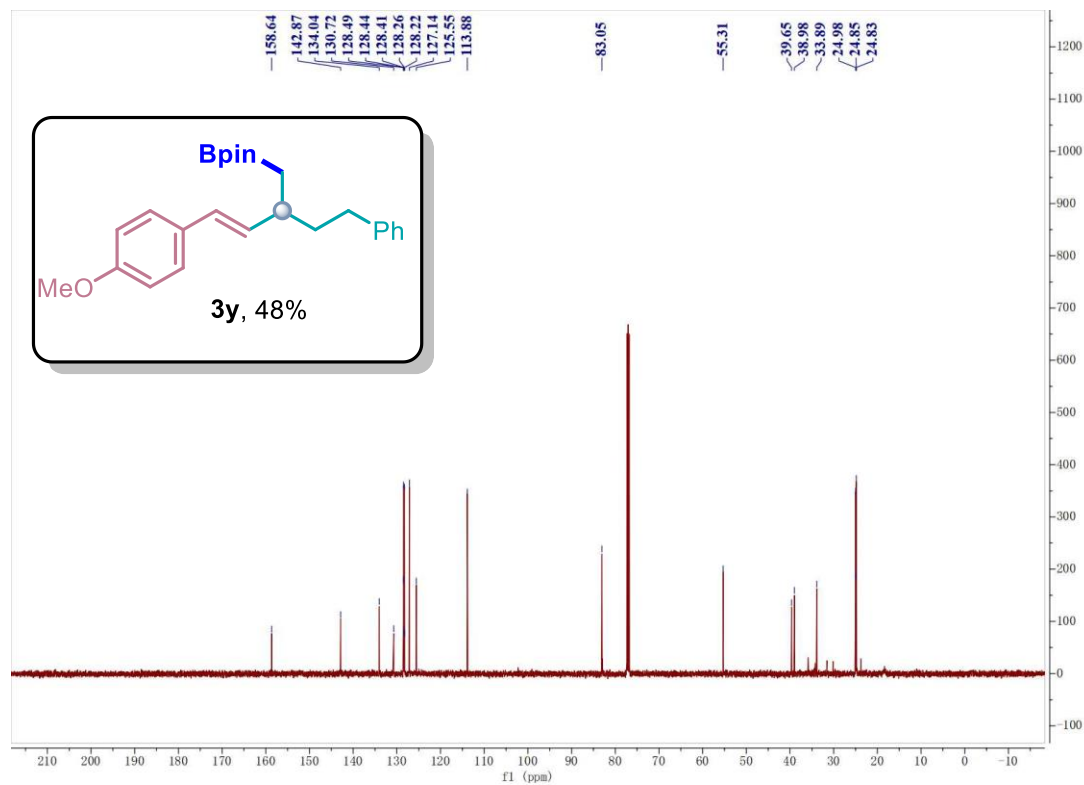


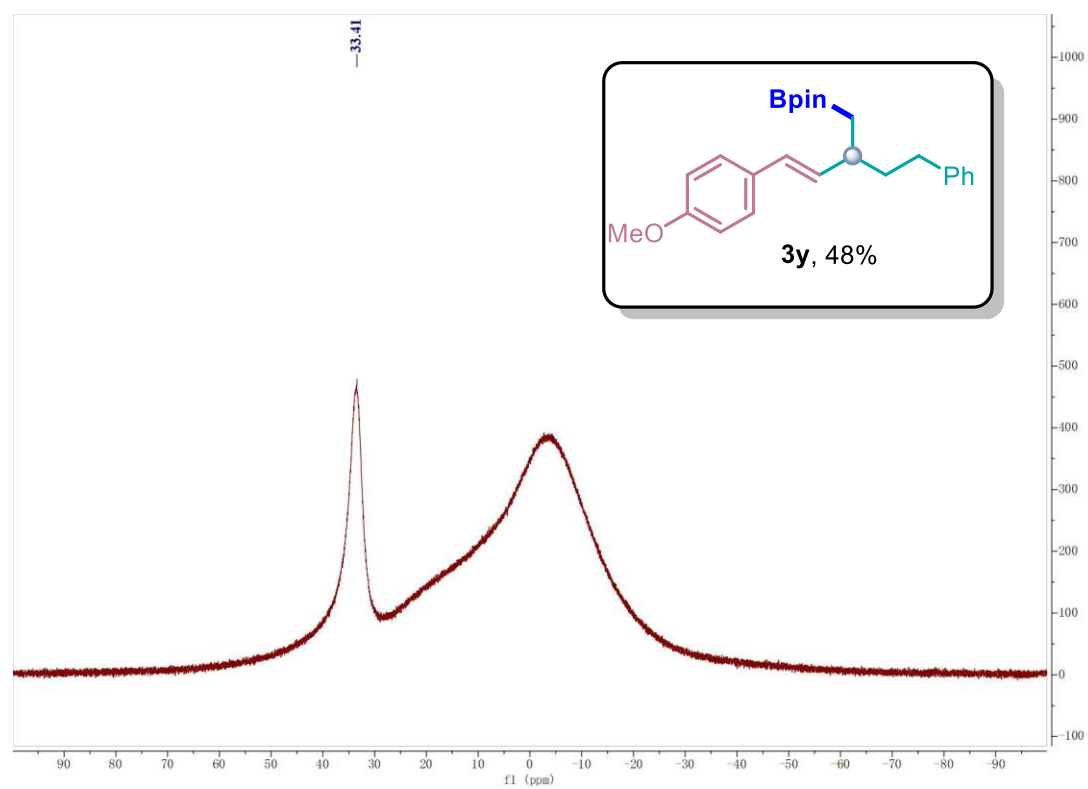
(E)-2-(4-(4-methoxyphenyl)-2-phenethylbut-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3y)

¹H NMR (500 MHz, Chloroform-*d*)



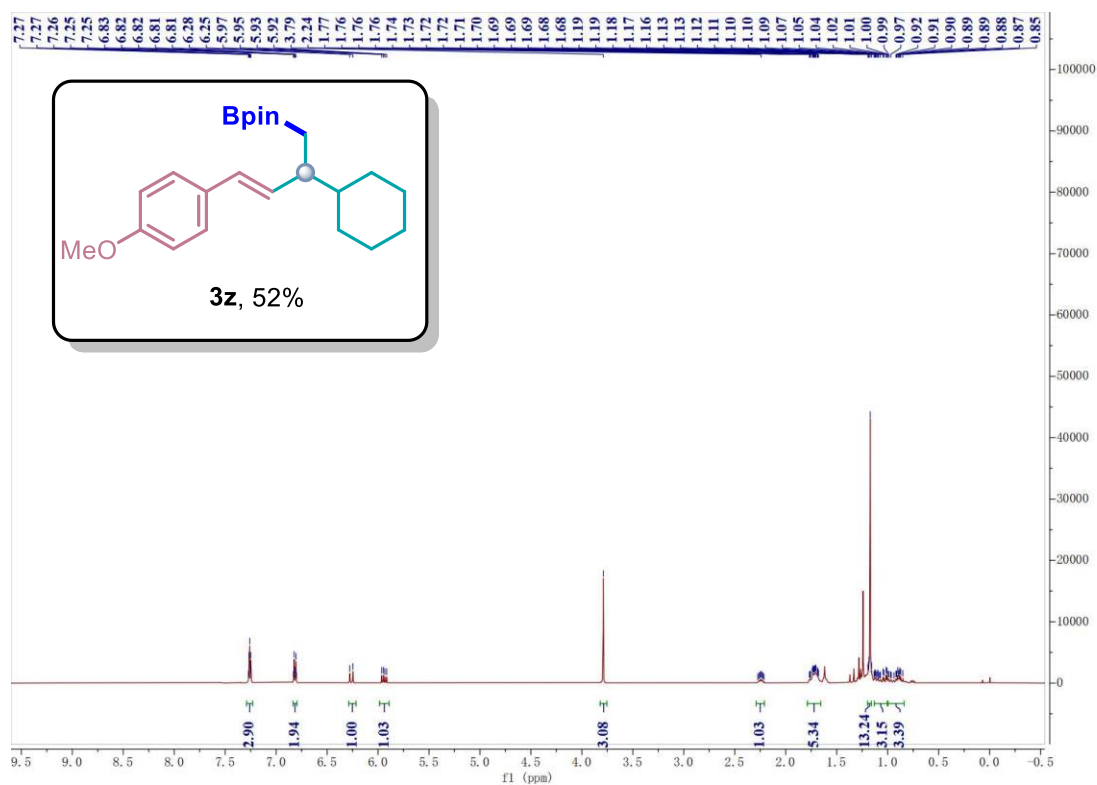
¹³C NMR (126 MHz, Chloroform-*d*)



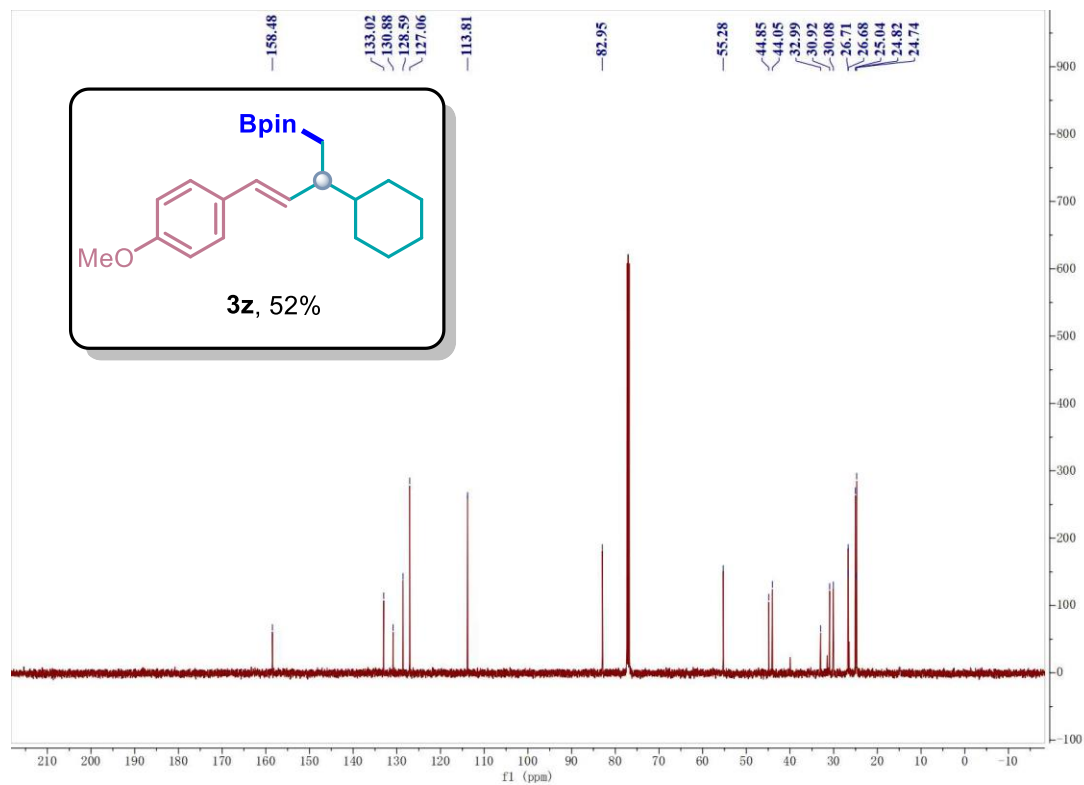
¹¹B NMR (160 MHz, Chloroform-*d*)

(E)-2-(2-cyclohexyl-4-(4-methoxyphenyl)but-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3z)

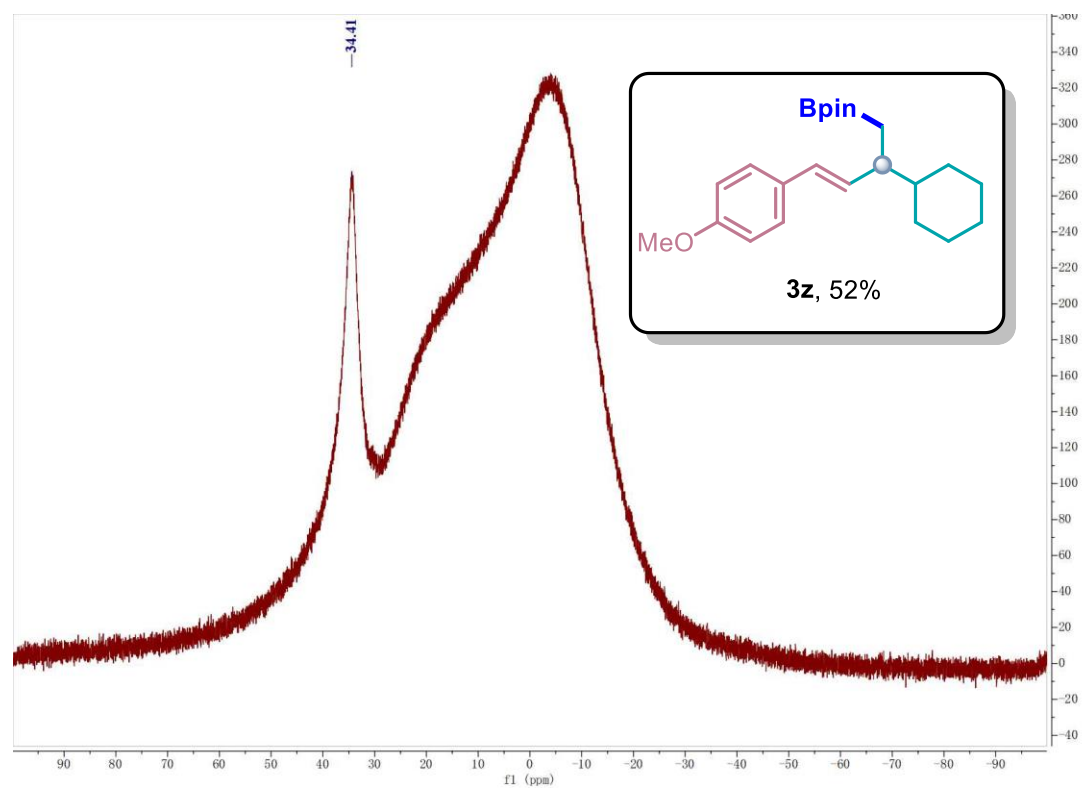
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

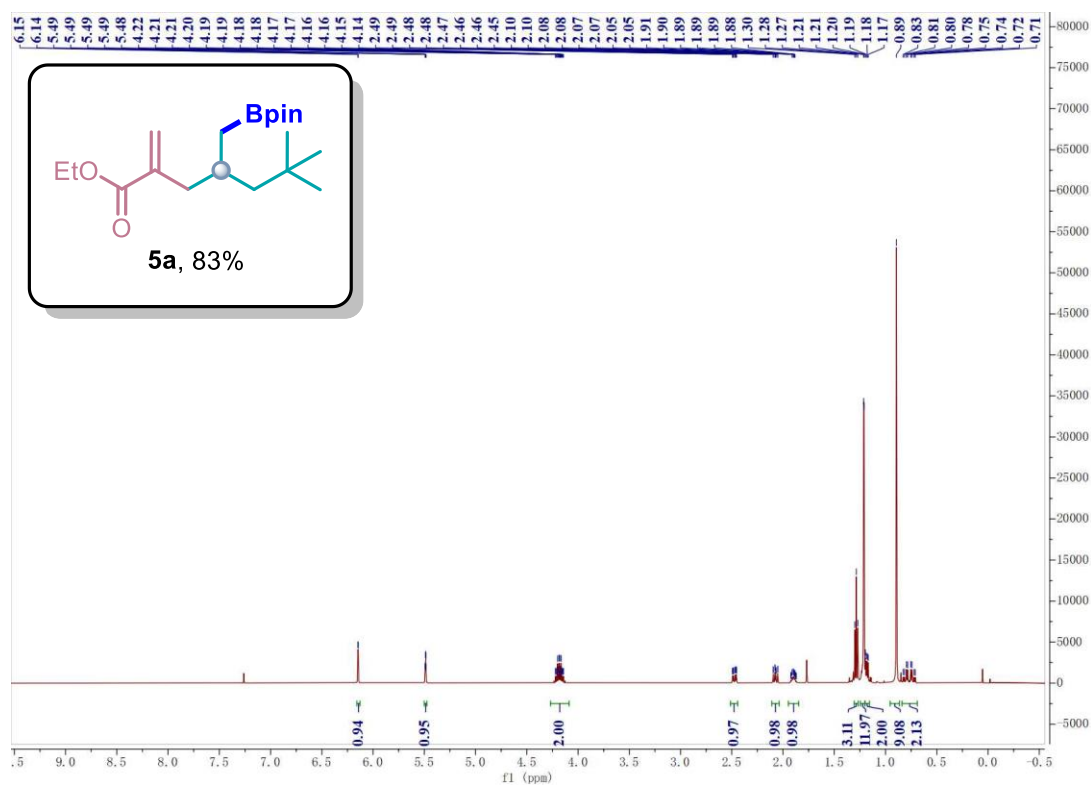


^{11}B NMR (160 MHz, Chloroform-*d*)

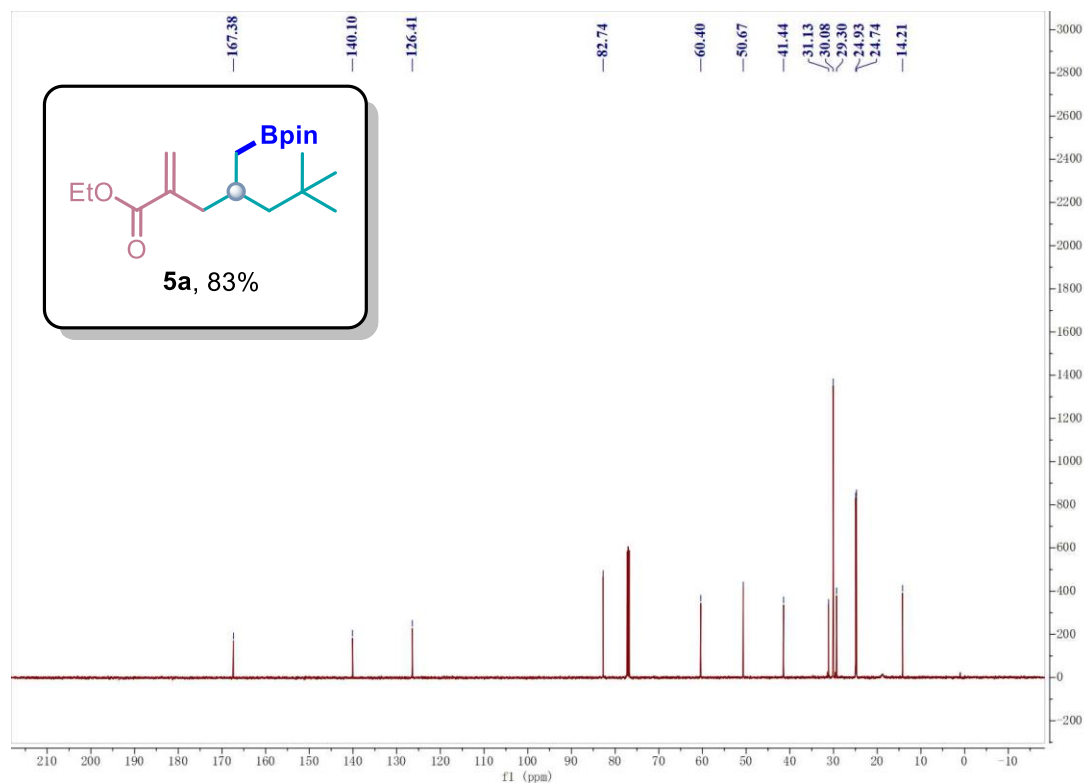


ethyl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5a**)

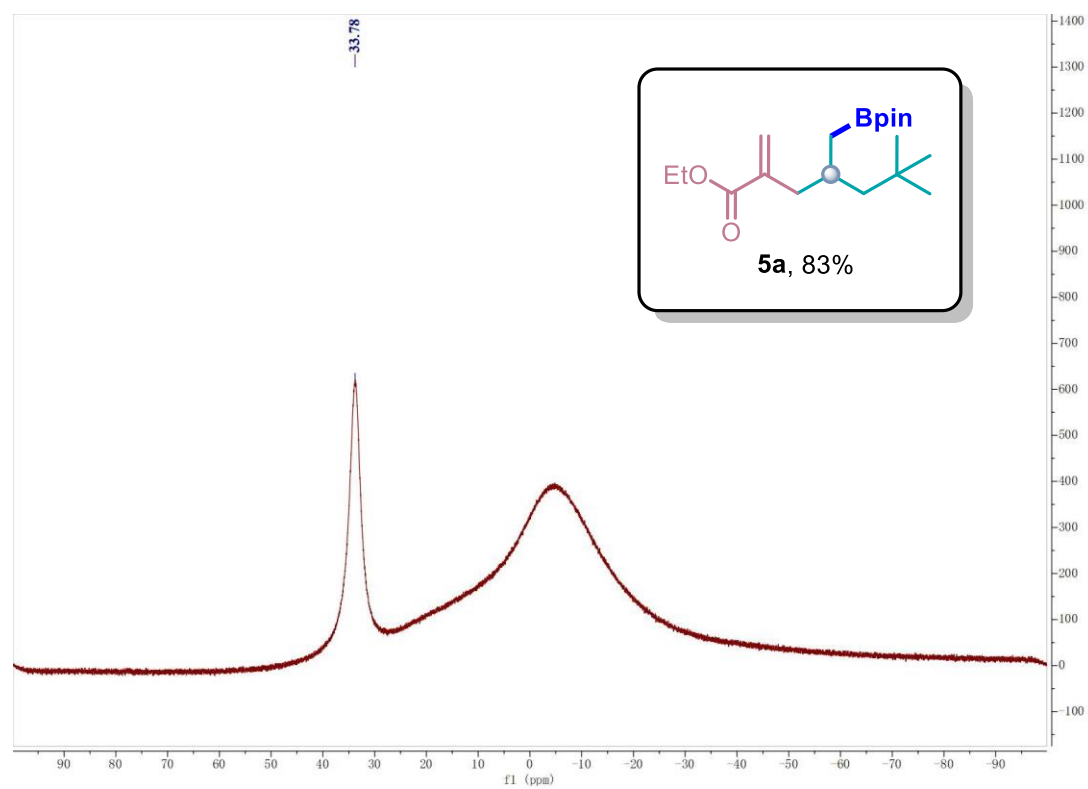
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

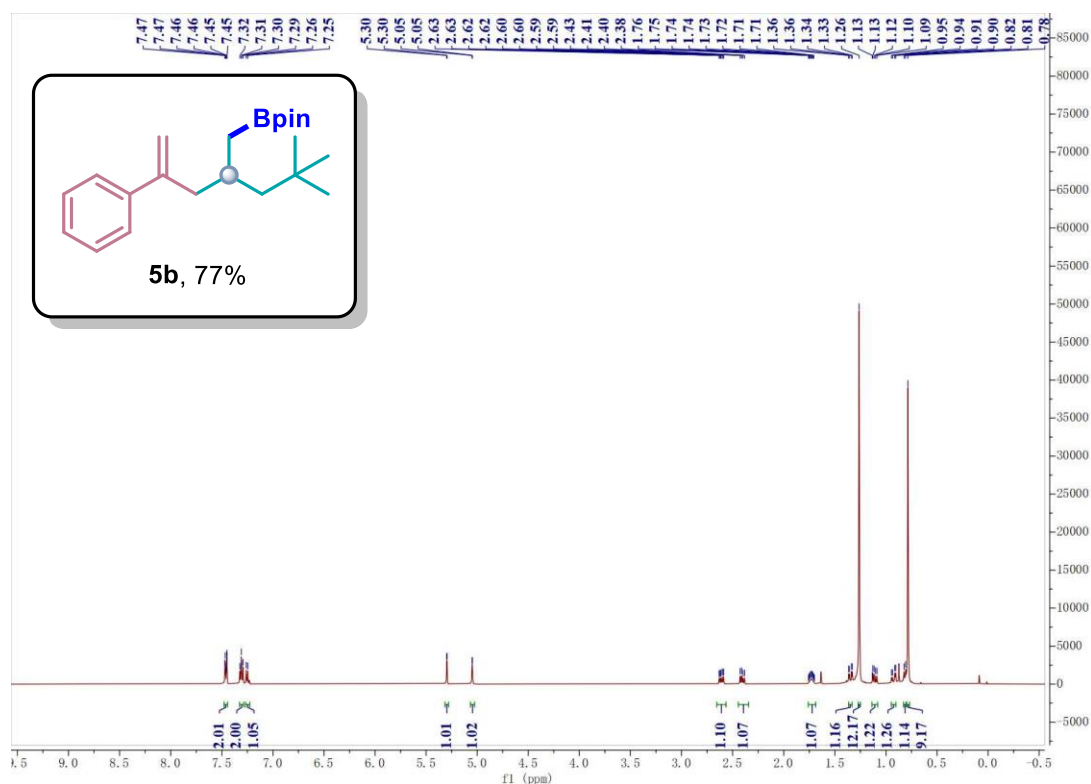


^{11}B NMR (160 MHz, Chloroform-*d*)

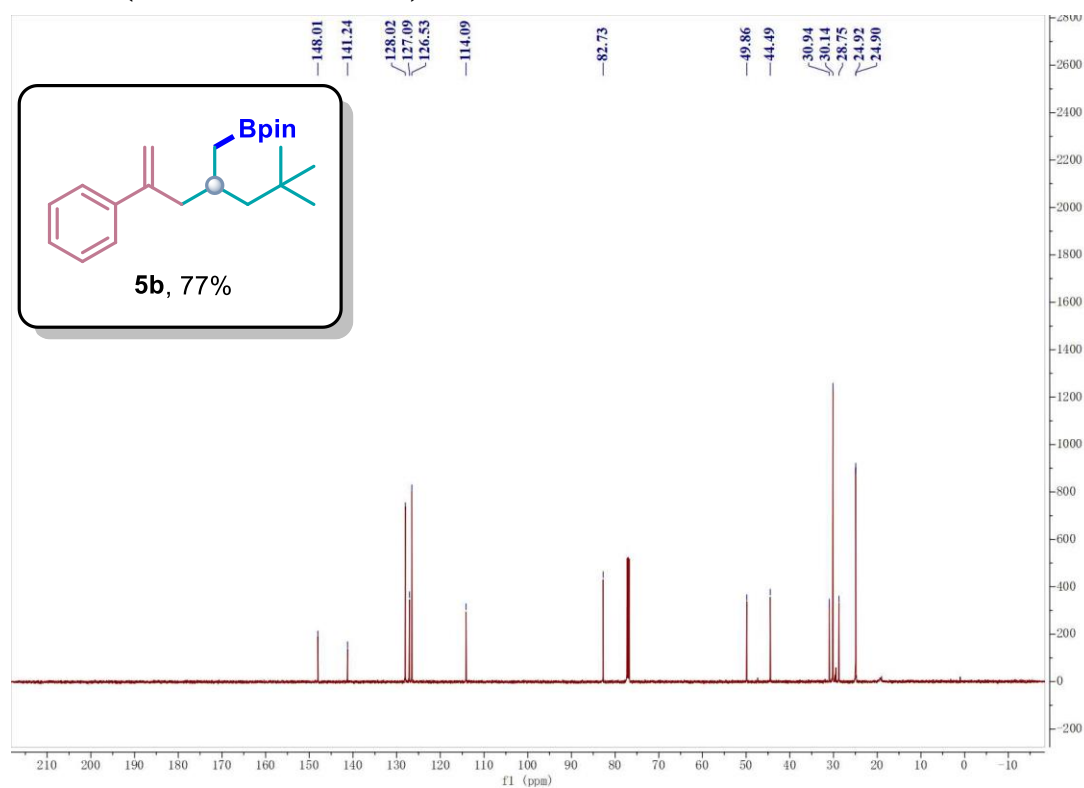


4,4,5,5-tetramethyl-2-(2-neopentyl-4-phenylpent-4-en-1-yl)-1,3,2-dioxaborolane (5b)

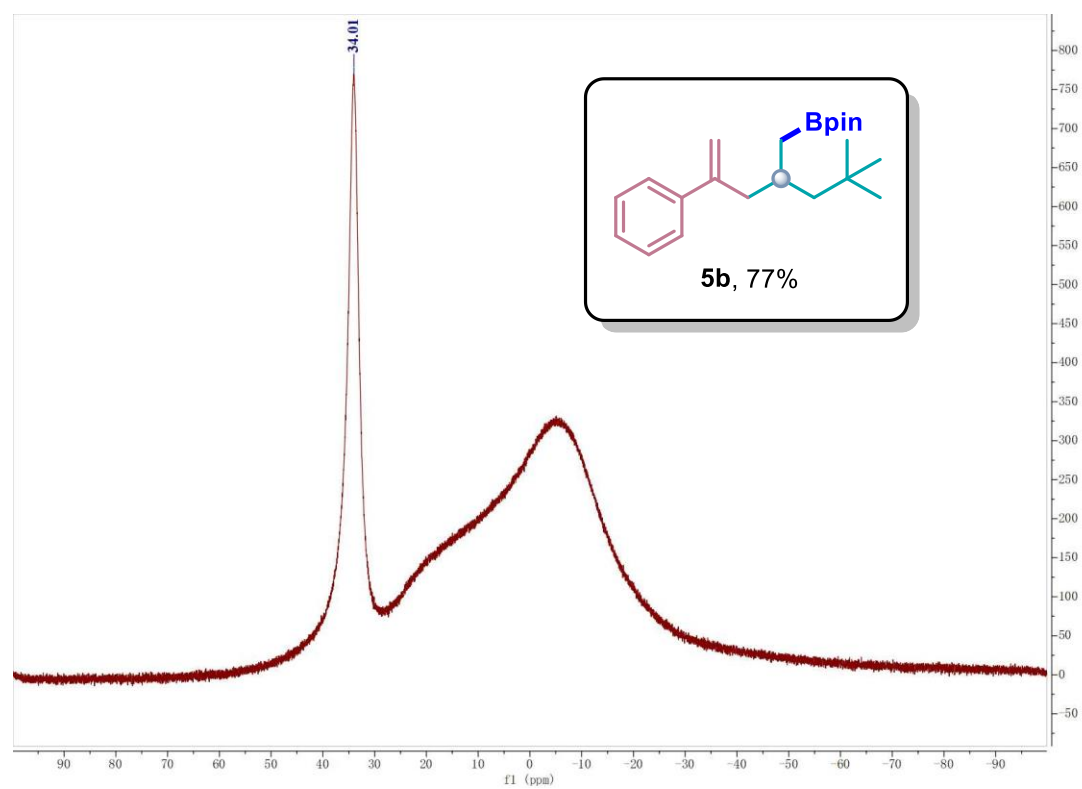
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

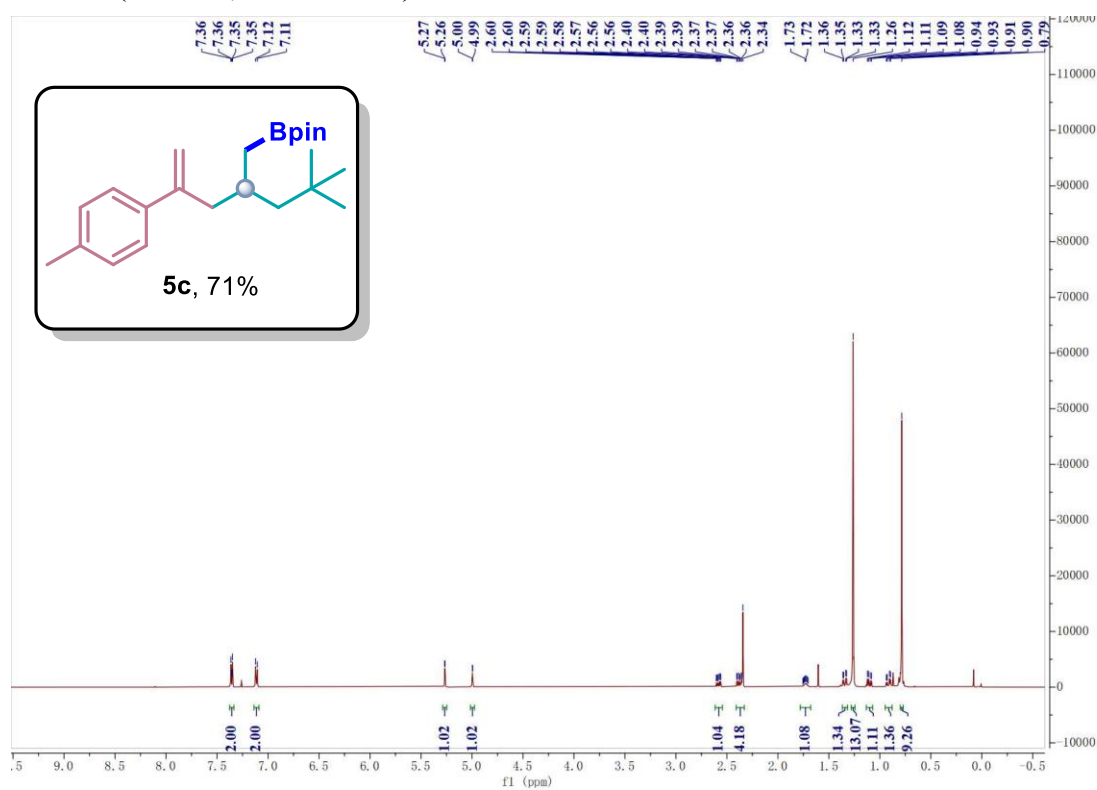


^{11}B NMR (160 MHz, Chloroform-*d*)

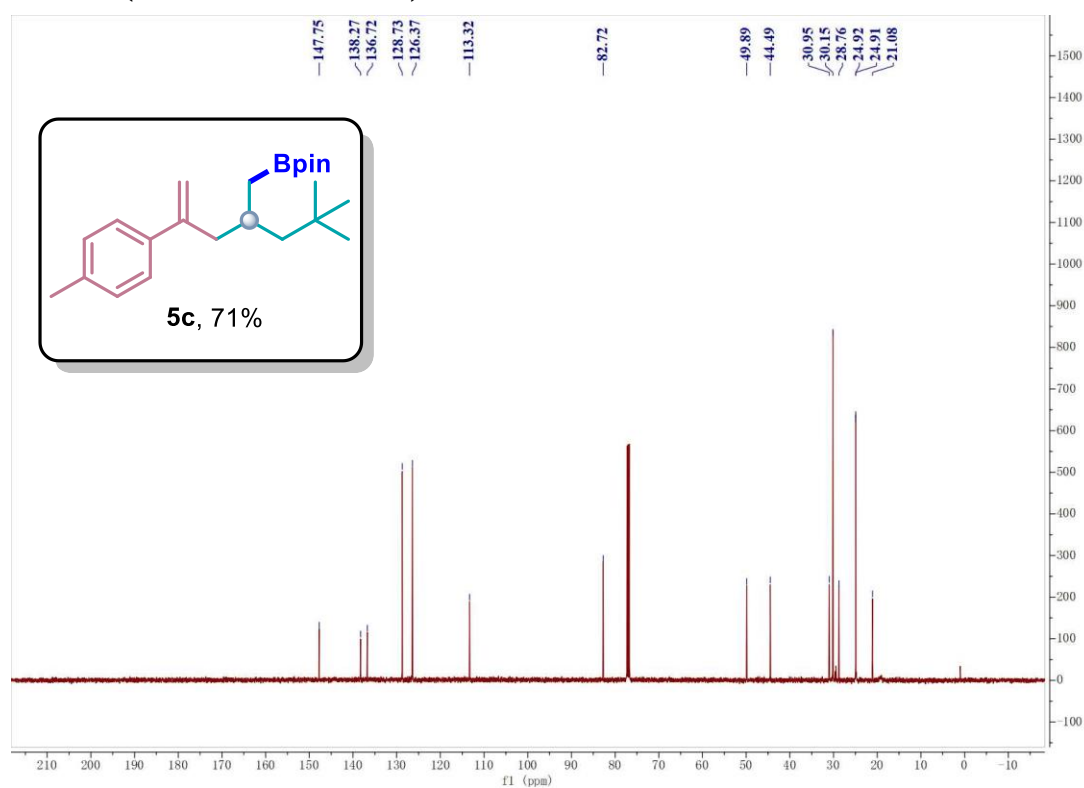


4,4,5,5-tetramethyl-2-(2-neopentyl-4-(p-tolyl)pent-4-en-1-yl)-1,3,2-dioxaborolane (**5c**)

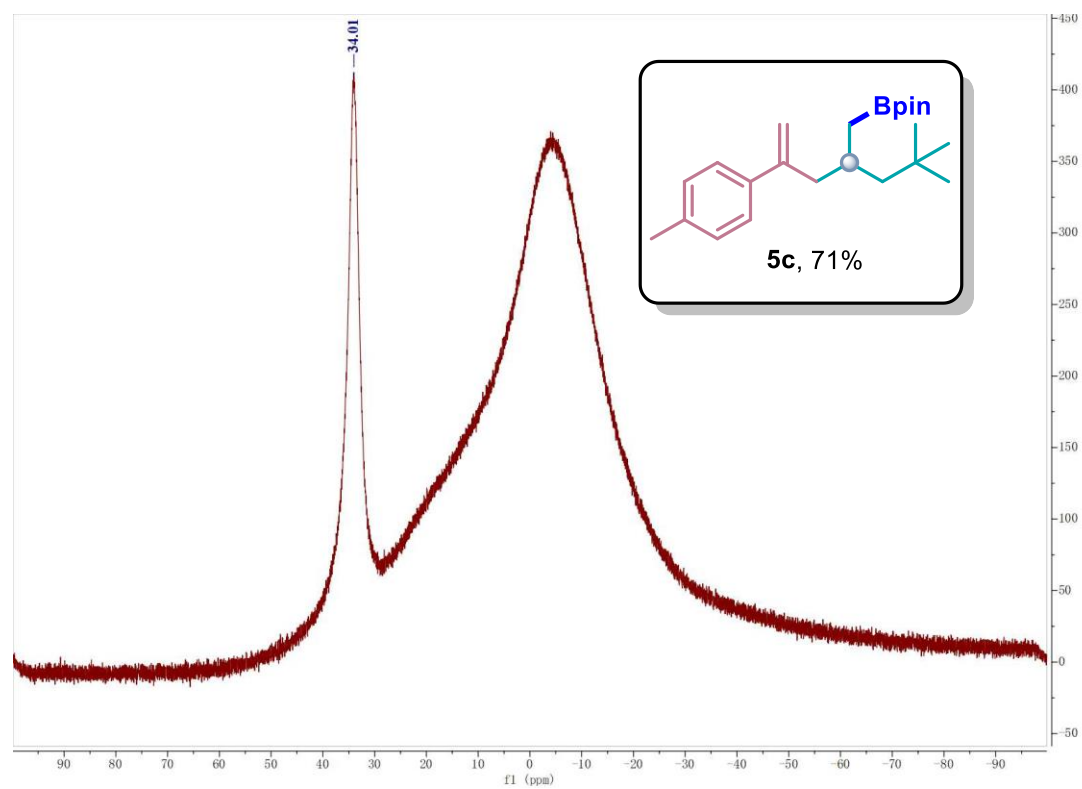
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

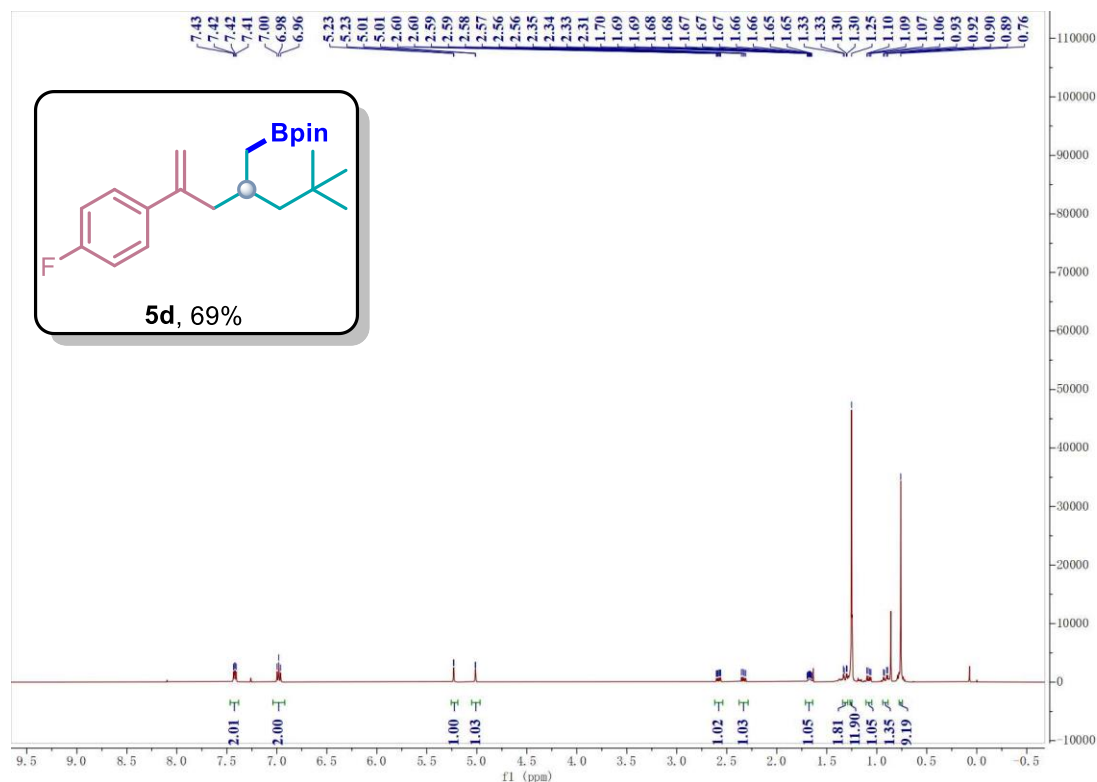


^{11}B NMR (160 MHz, Chloroform-*d*)

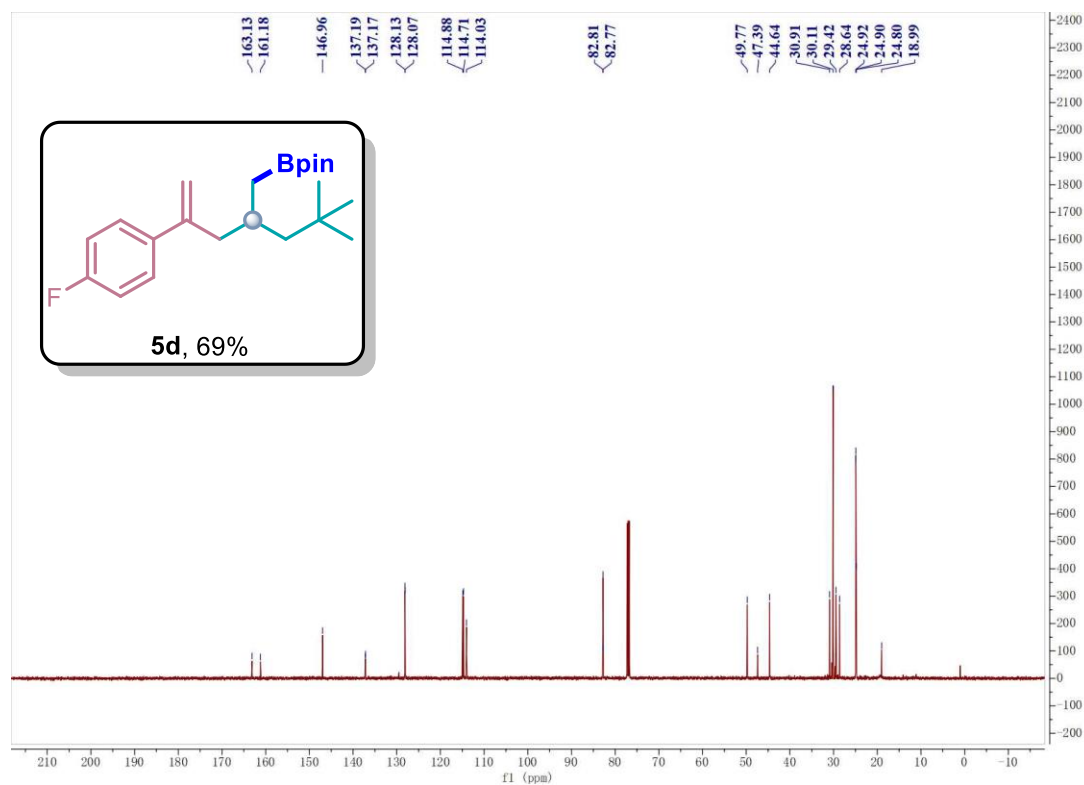


2-(4-(4-fluorophenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5d)

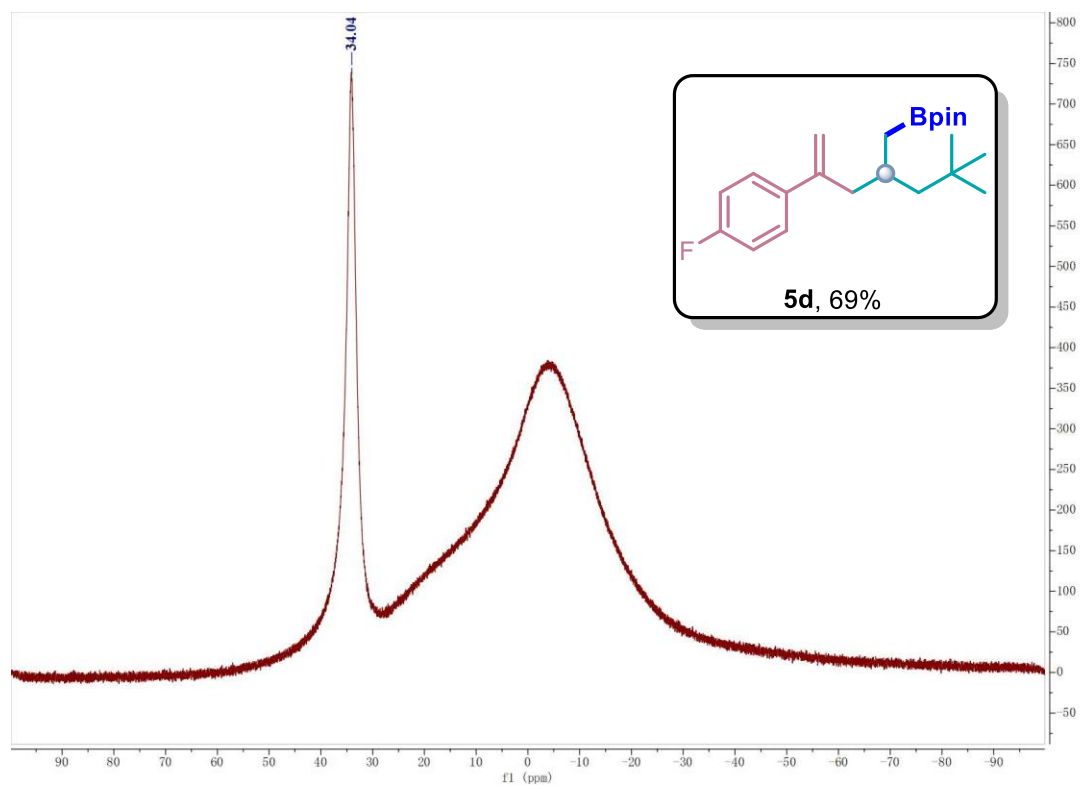
¹H NMR (500 MHz, Chloroform-*d*)



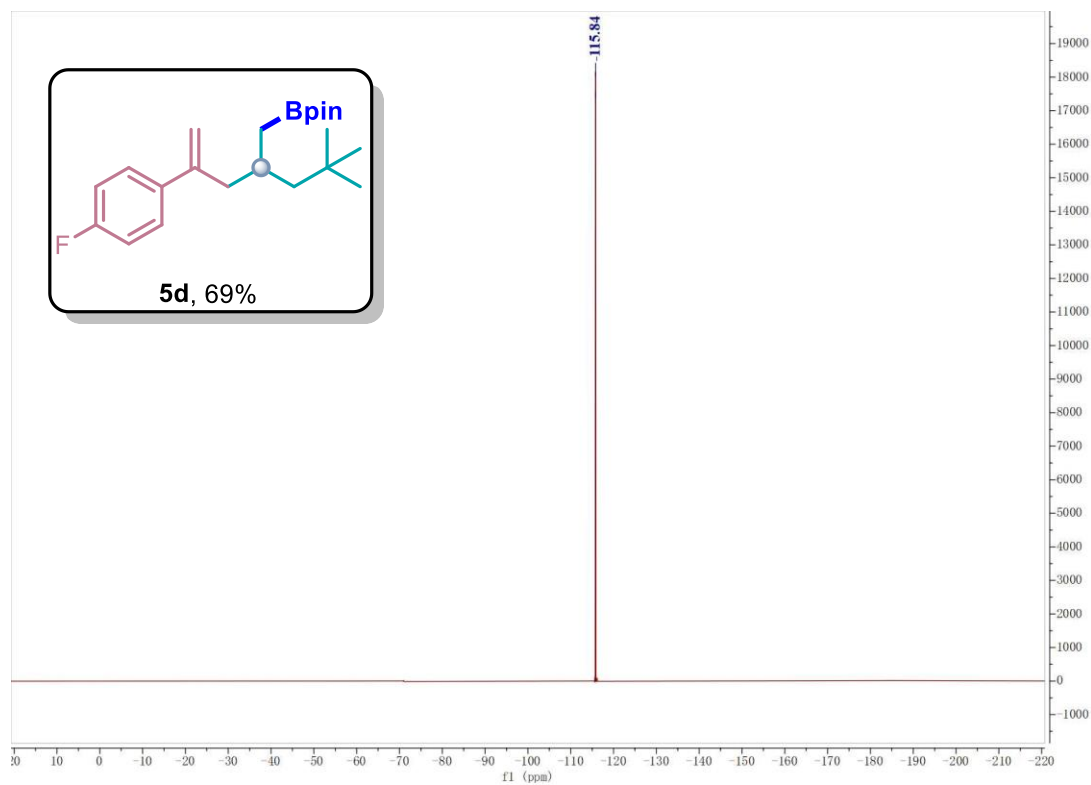
¹³C NMR (126 MHz, Chloroform-*d*)



^{11}B NMR (160 MHz, Chloroform- d)

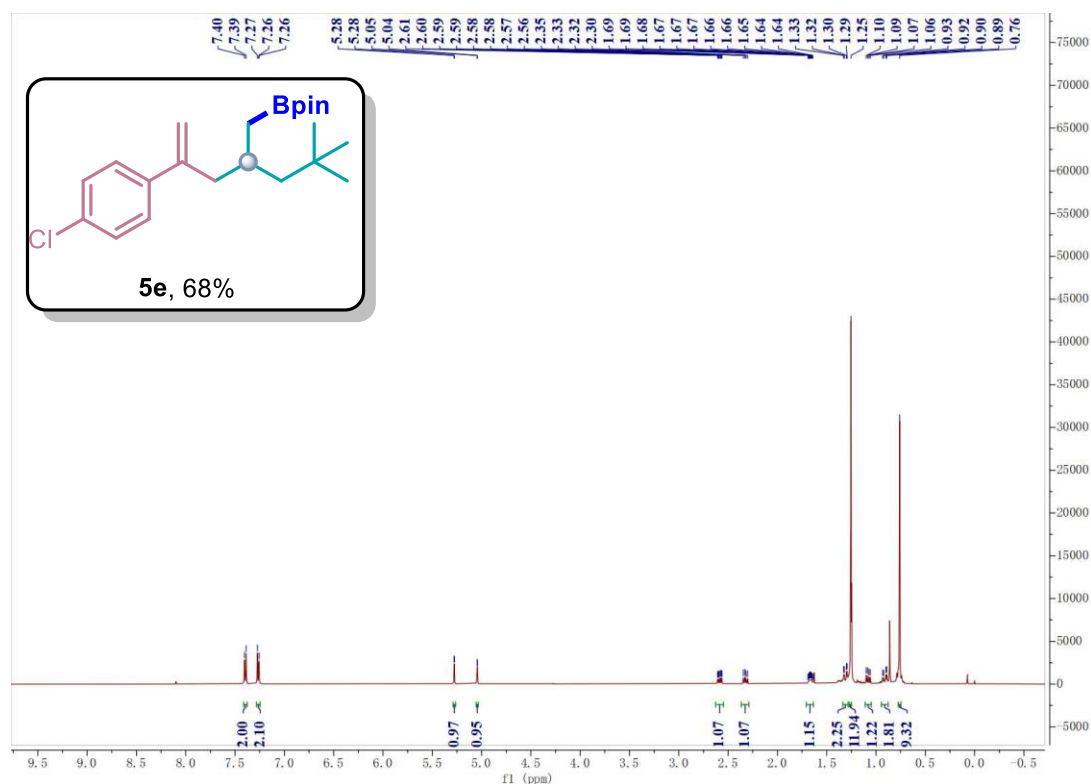


^{19}F NMR (471 MHz, Chloroform- d)

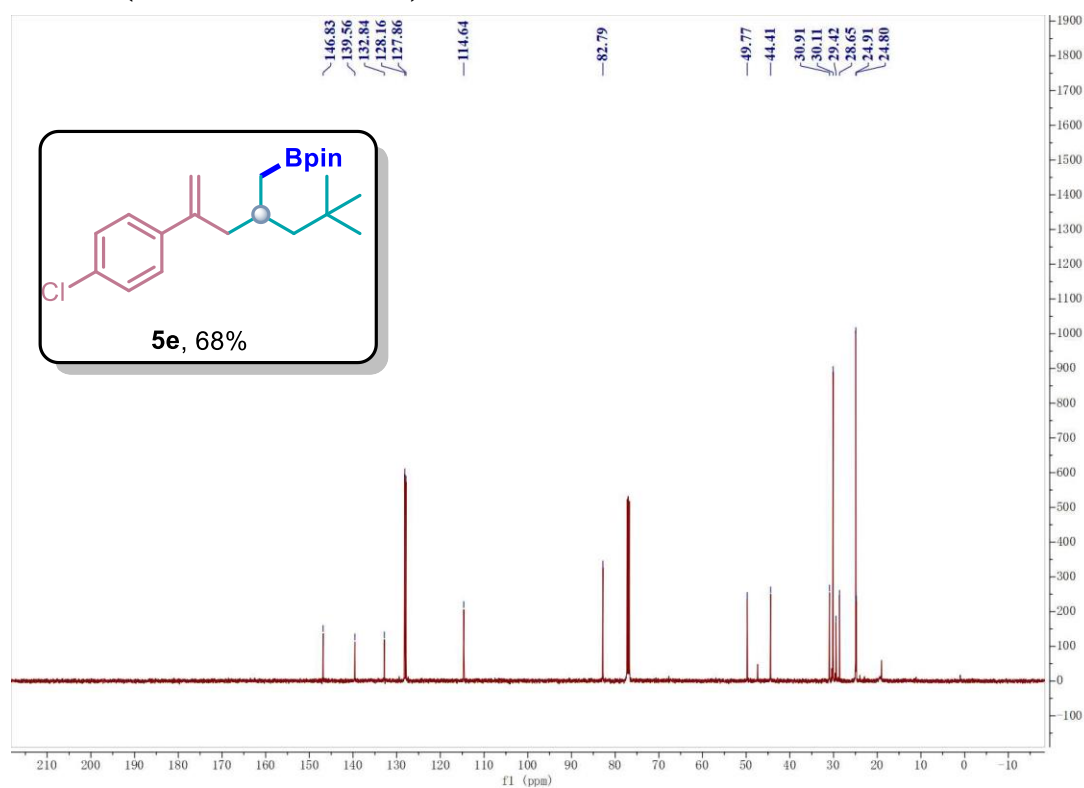


2-(4-(4-chlorophenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5e)

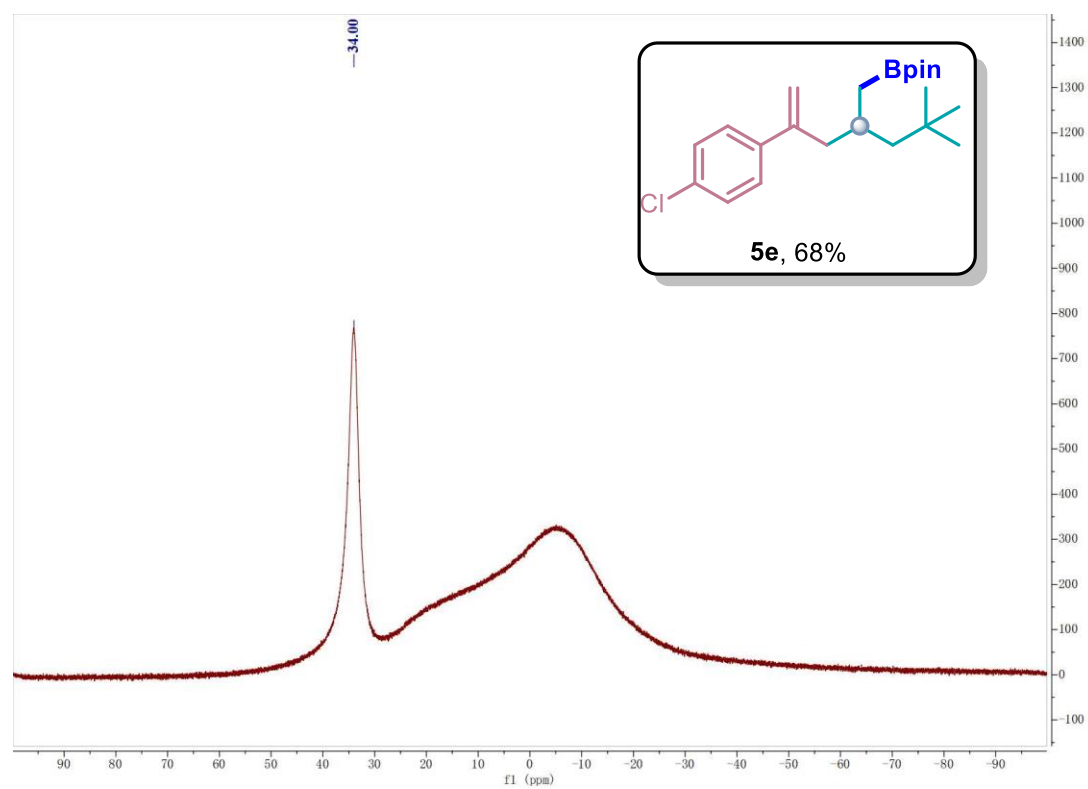
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

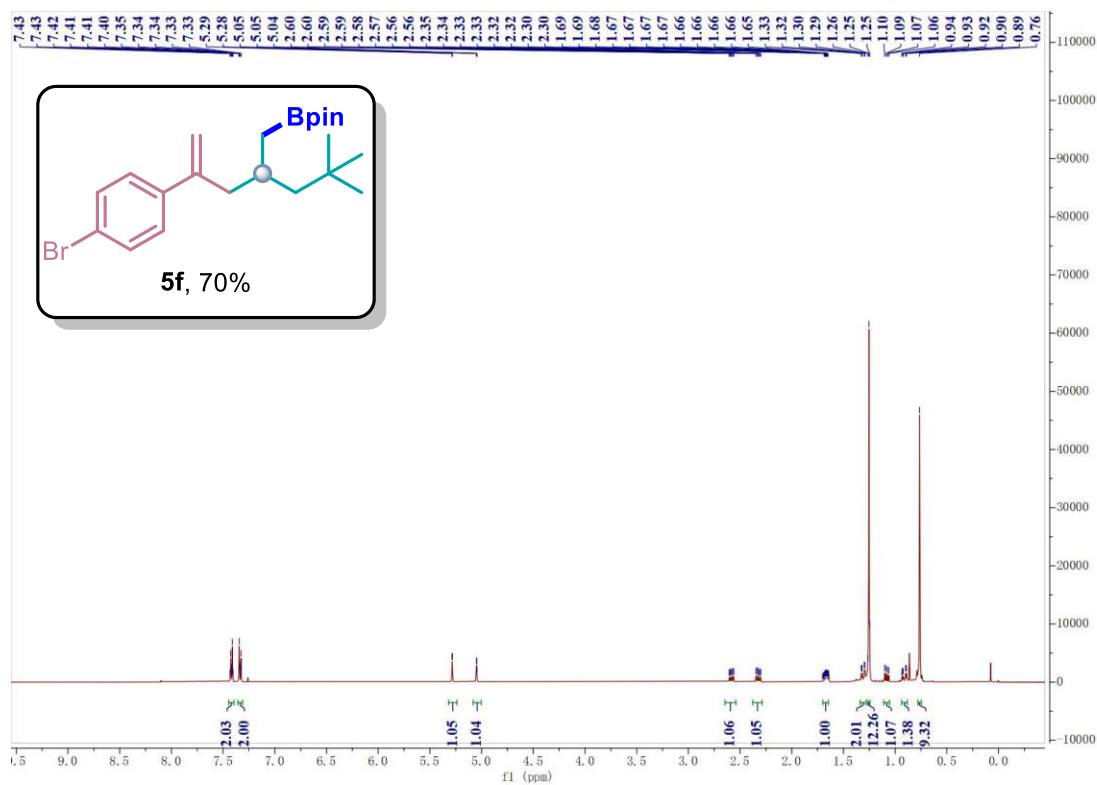


^{11}B NMR (160 MHz, Chloroform-*d*)

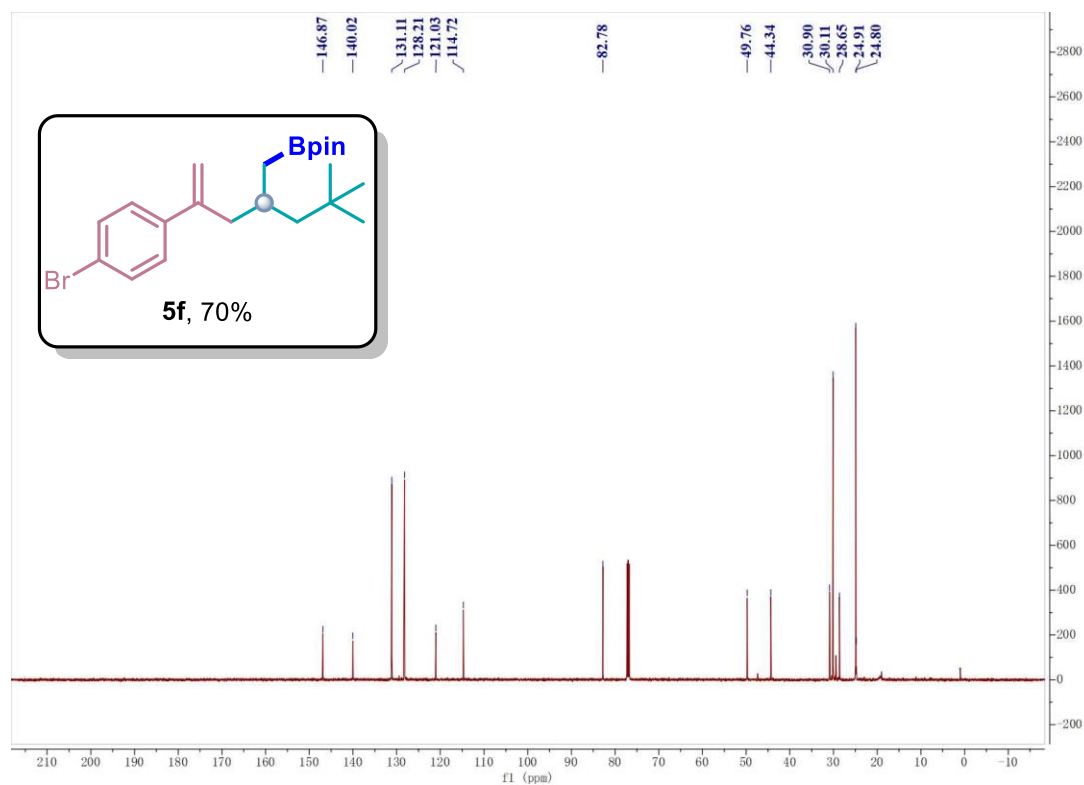


2-(4-(4-bromophenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5f)

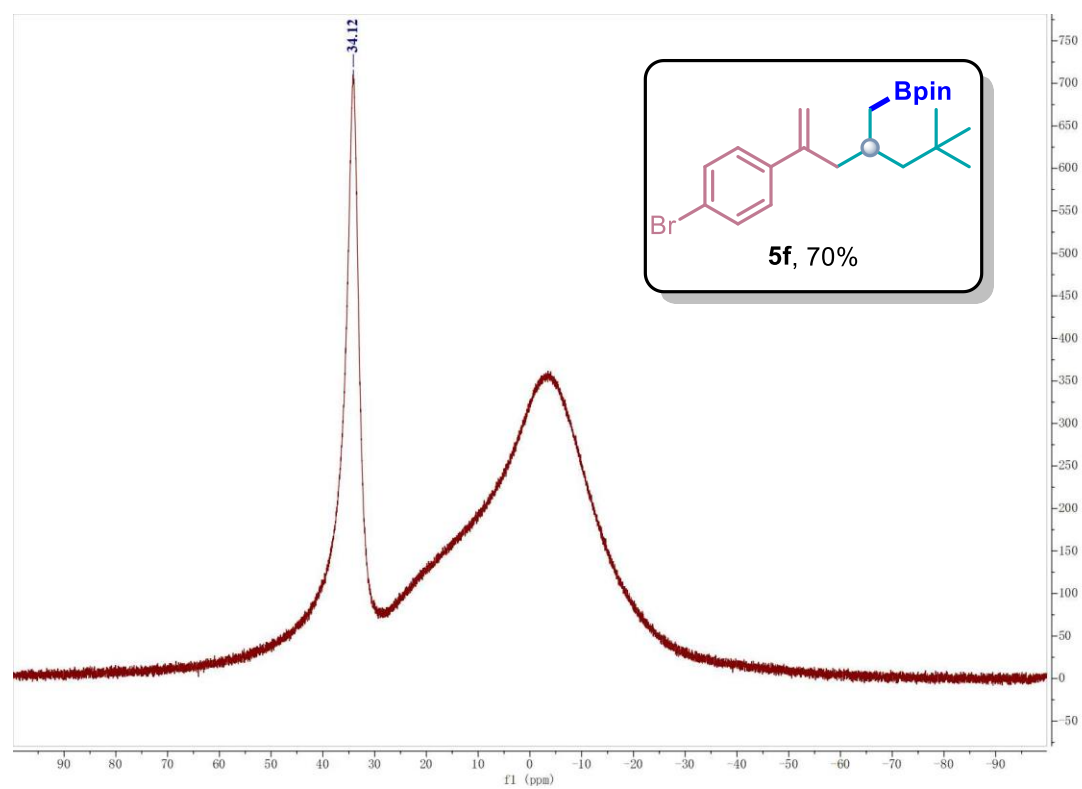
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

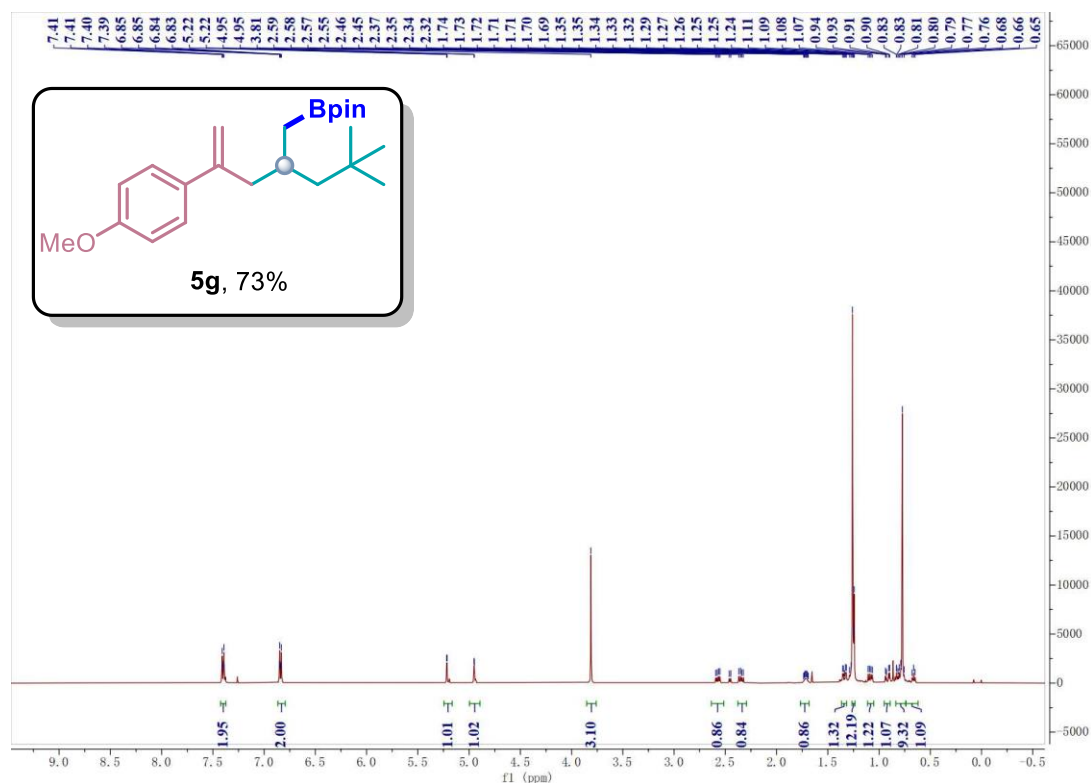


^{11}B NMR (160 MHz, Chloroform-*d*)

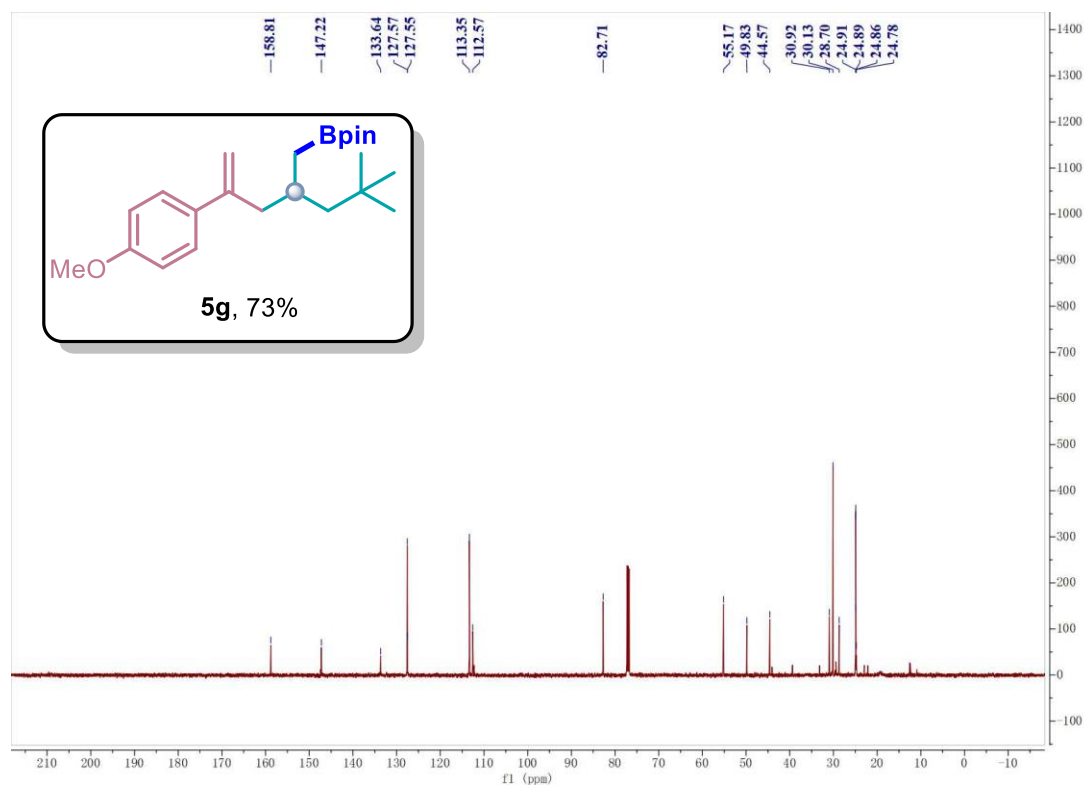


2-(4-(4-methoxyphenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5g)

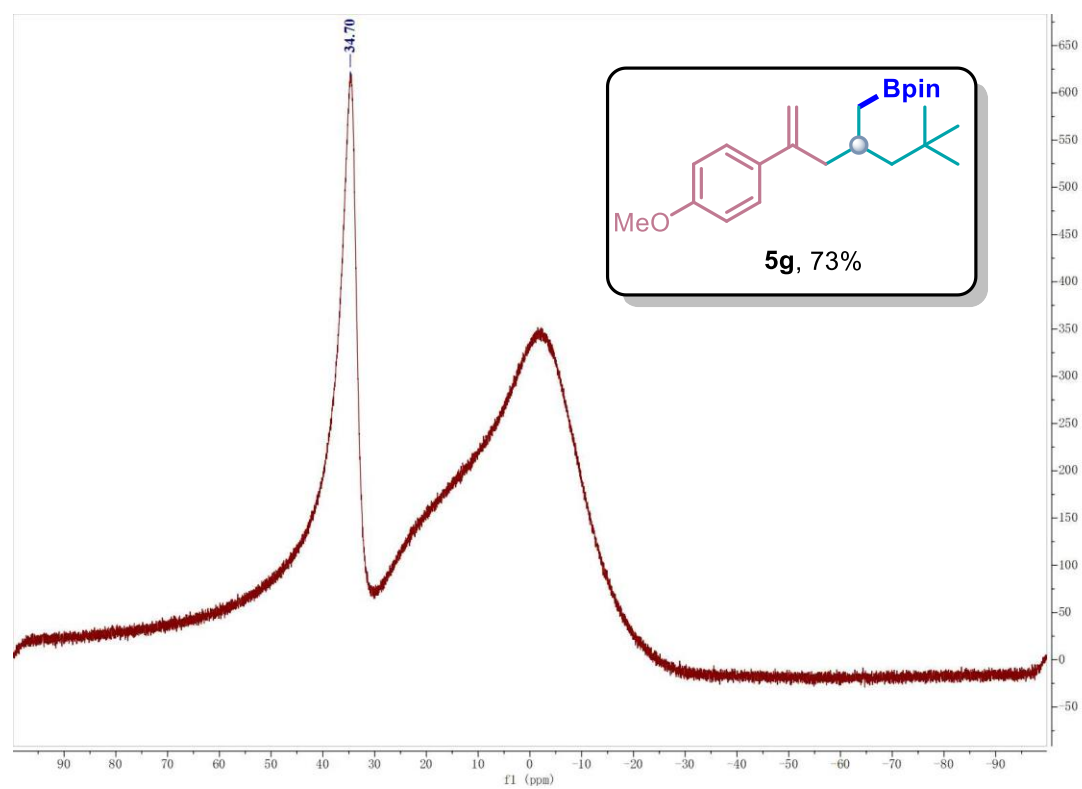
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

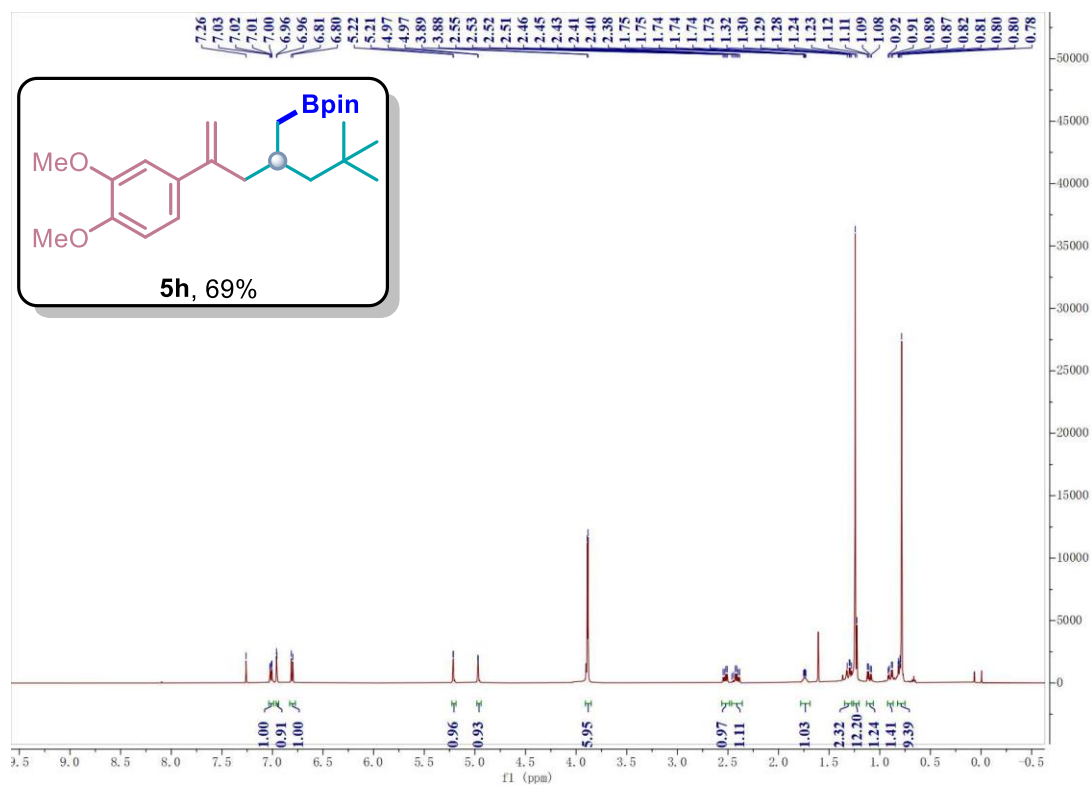


^{11}B NMR (160 MHz, Chloroform-*d*)

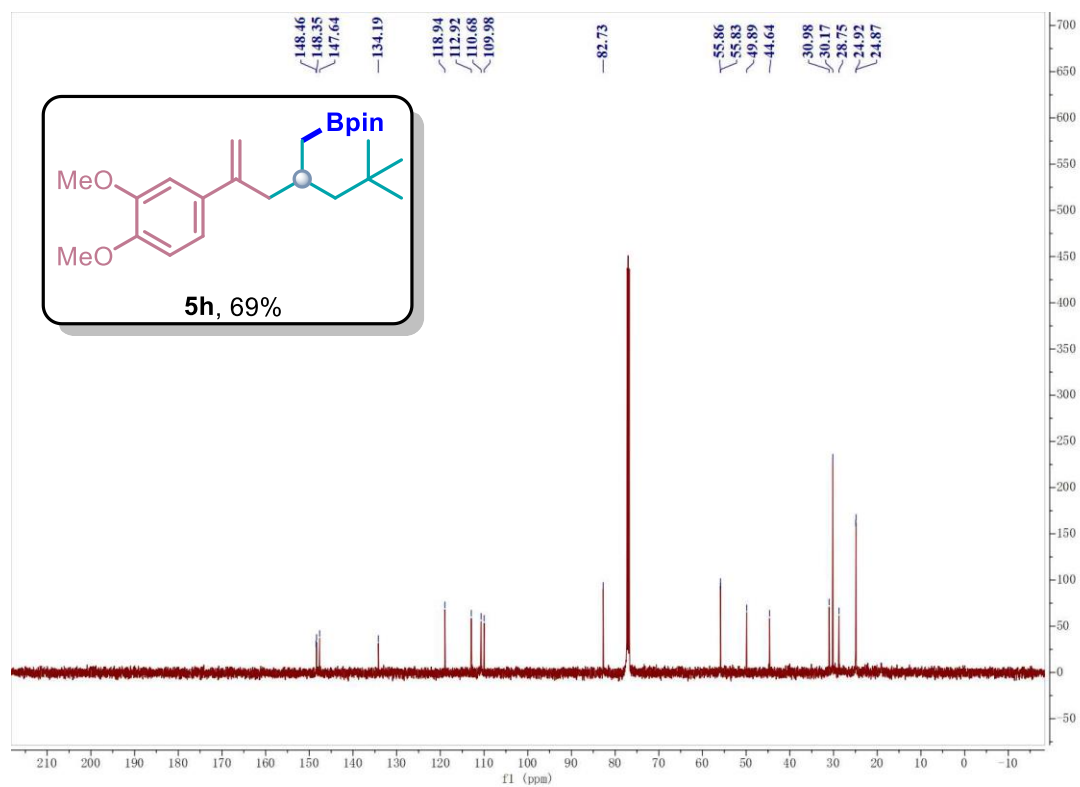


2-(4-(3,4-dimethoxyphenyl)-2-neopentylpent-4-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5h)

¹H NMR (500 MHz, Chloroform-*d*)



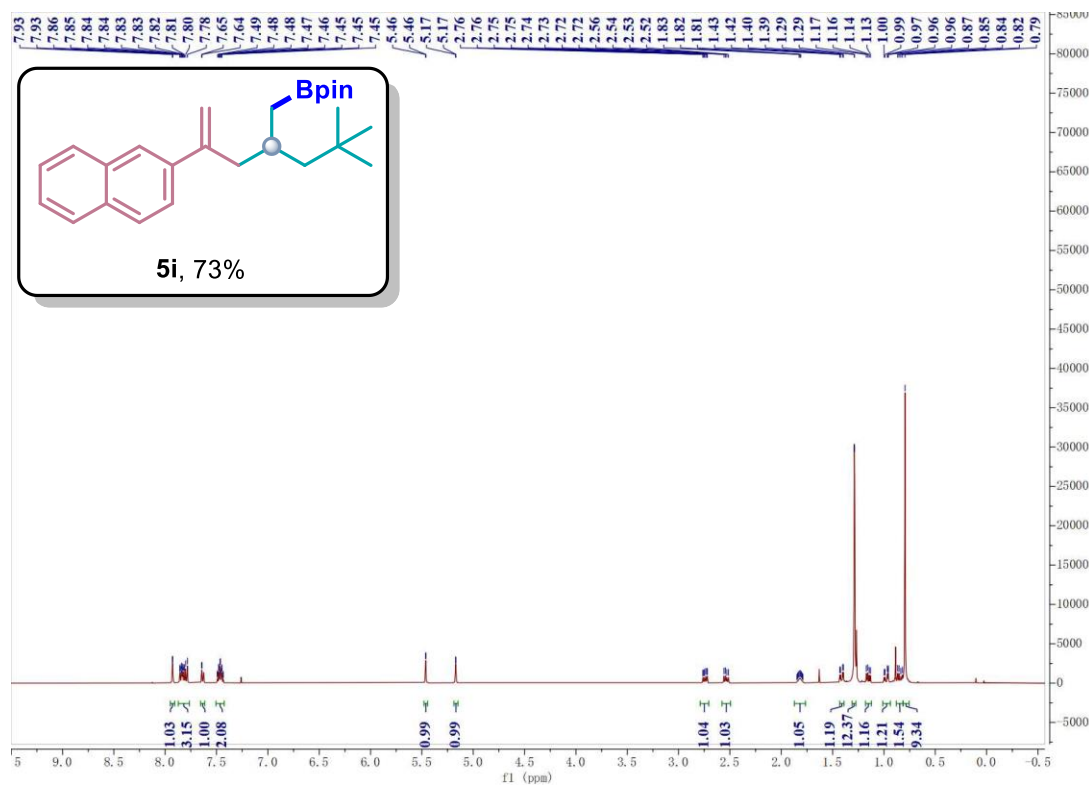
¹³C NMR (126 MHz, Chloroform-*d*)



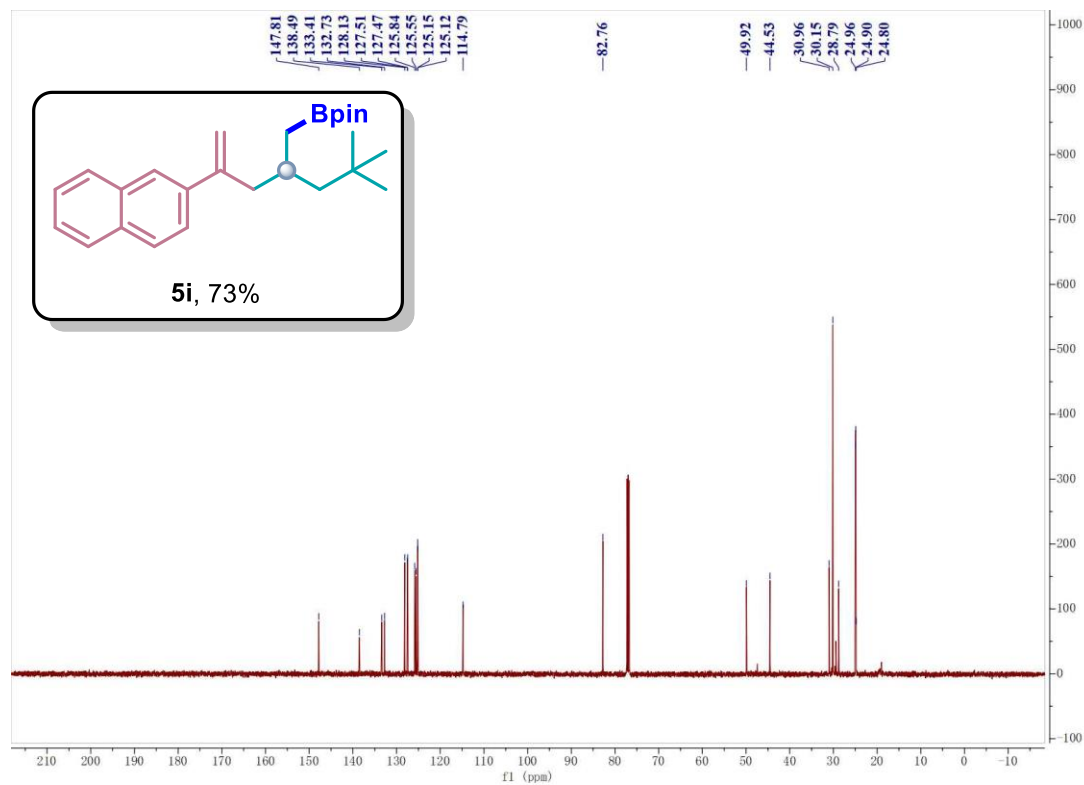
Chemical structure of compound **5h** is shown in the inset, labeled **5h, 69%**. The structure is 2,4-dimethoxy-5-(4-((tert-butyldimethylsilyl)oxy)phenyl)-2-methylbut-3-en-1-ol. The Bpin group is highlighted in blue.

**4,4,5,5-tetramethyl-2-(4-(naphthalen-2-yl)-2-neopentylpent-4-en-1-yl)-1,3,2-dioxaborolane
(5i)**

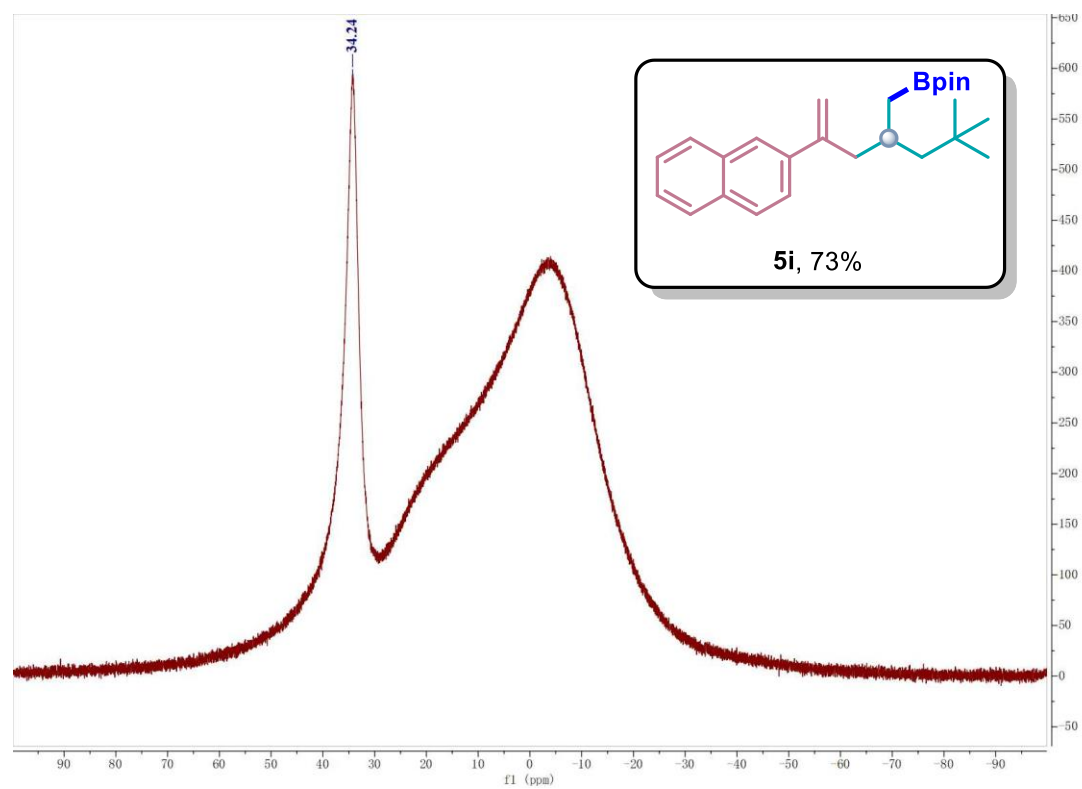
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

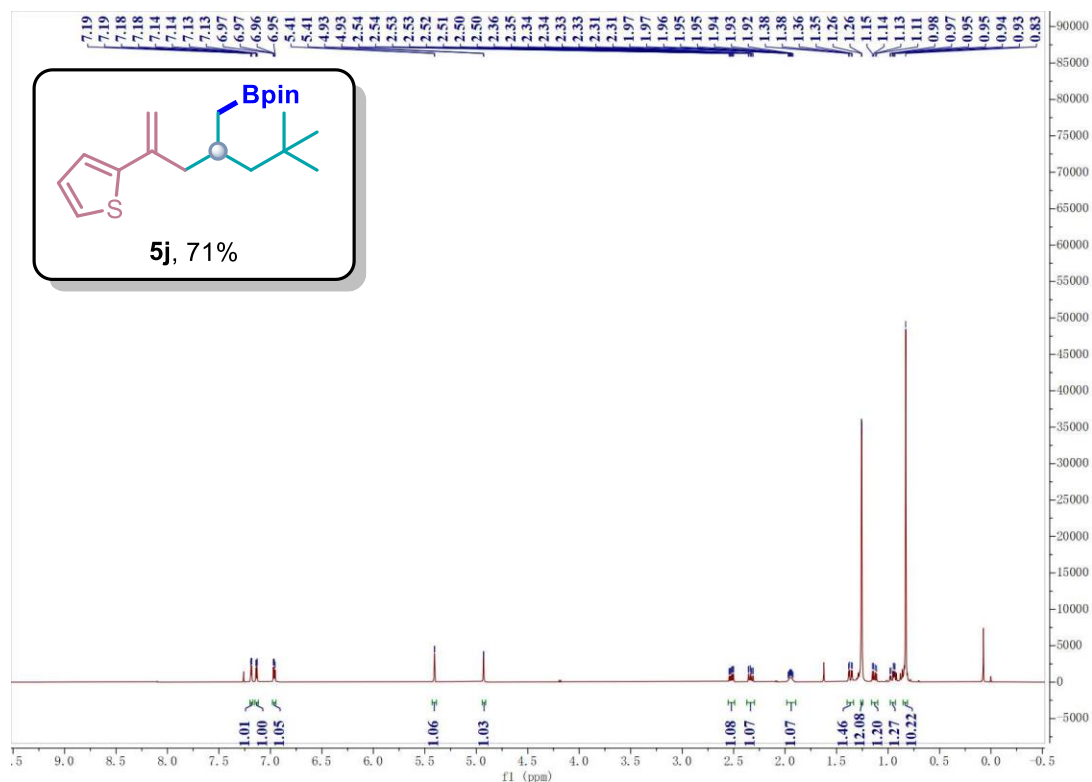


^{11}B NMR (160 MHz, Chloroform-*d*)

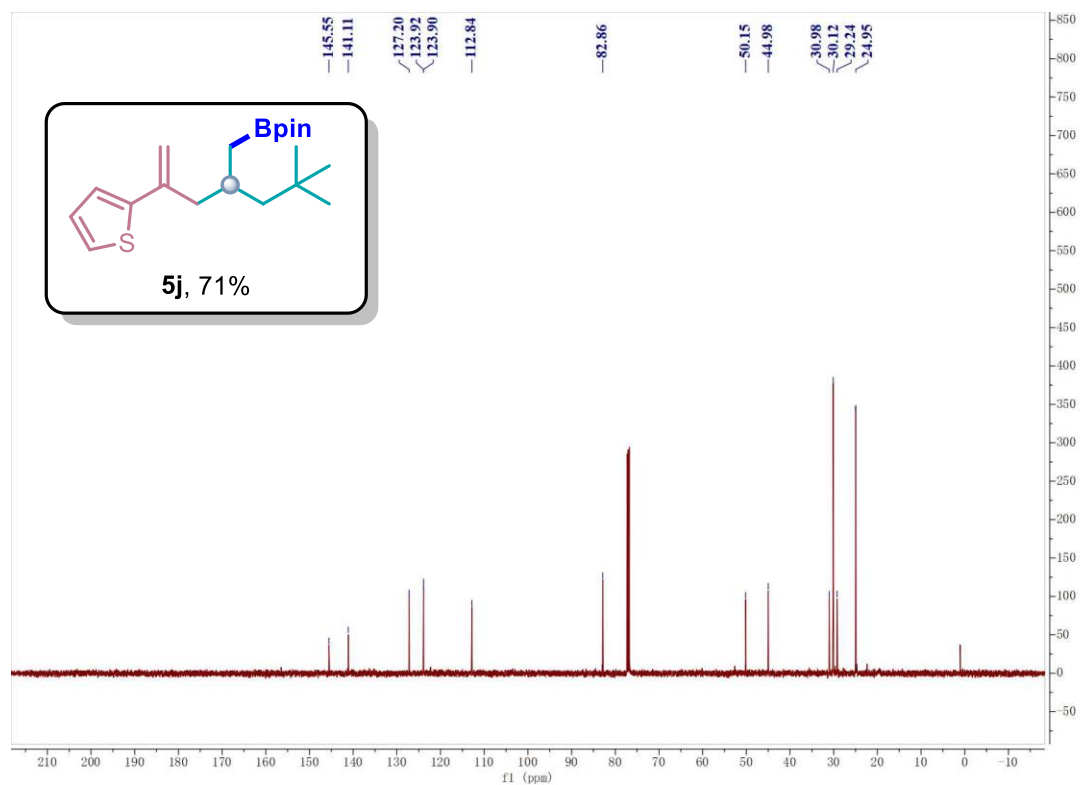


4,4,5,5-tetramethyl-2-(2-neopentyl-4-(thiophen-2-yl)pent-4-en-1-yl)-1,3,2-dioxaborolane (5j)

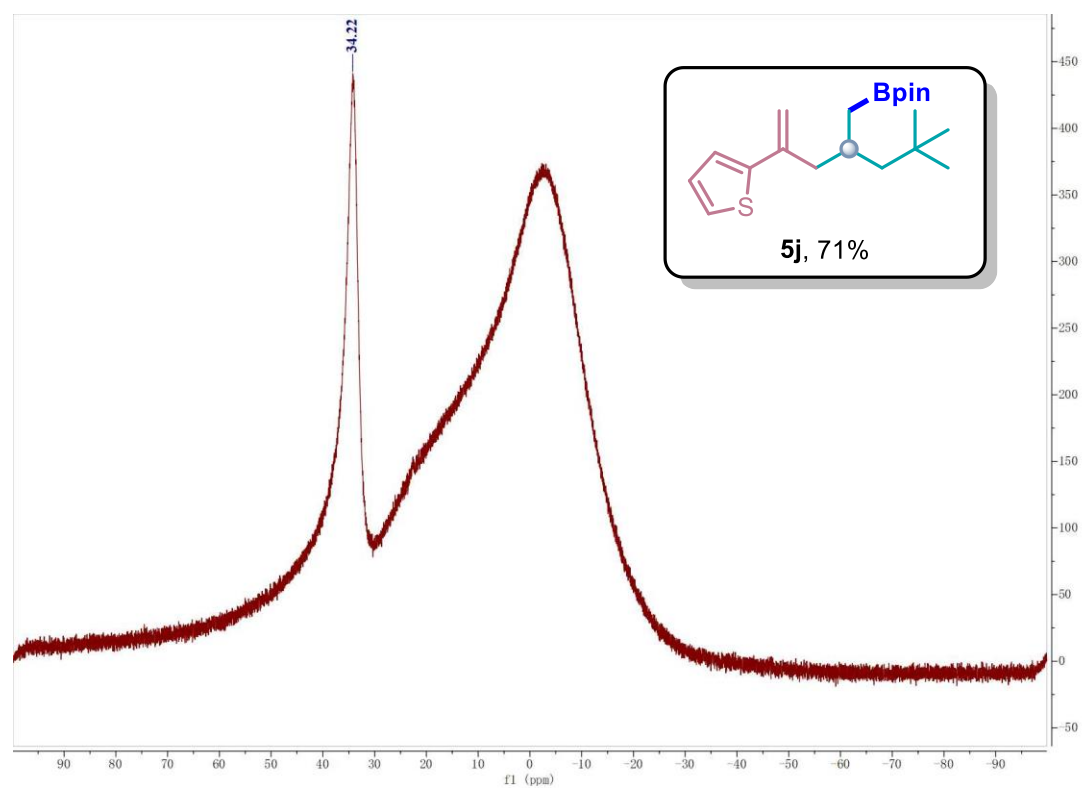
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)



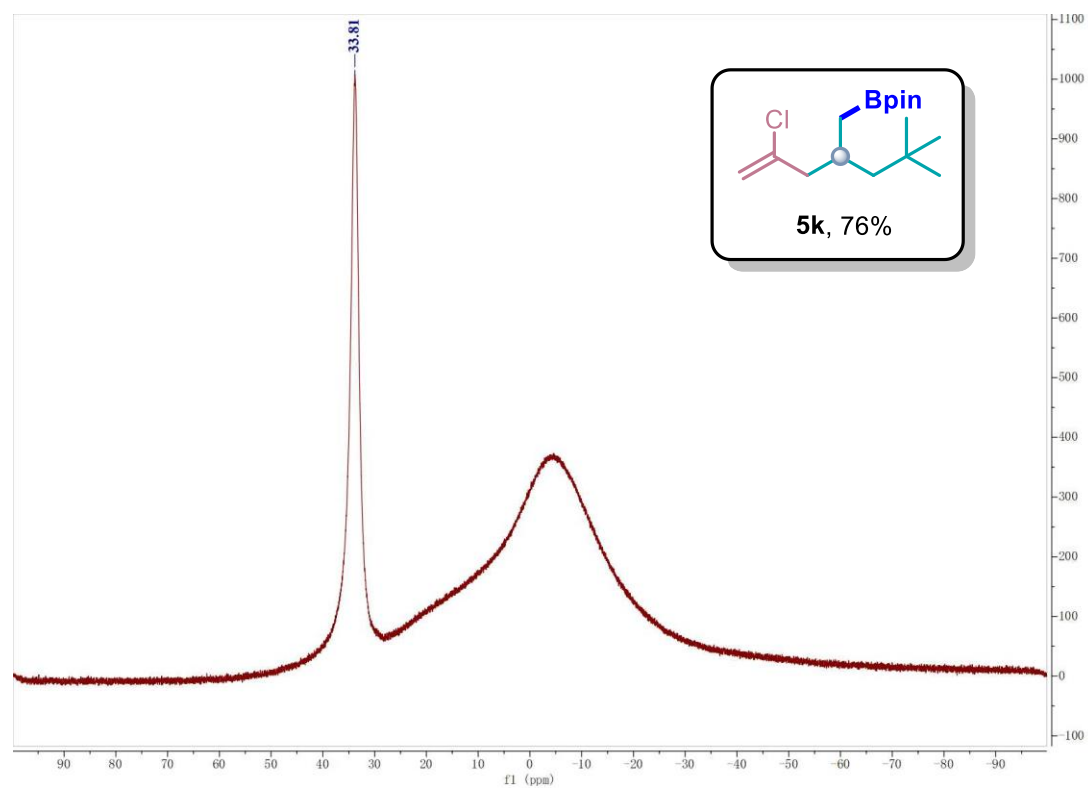
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

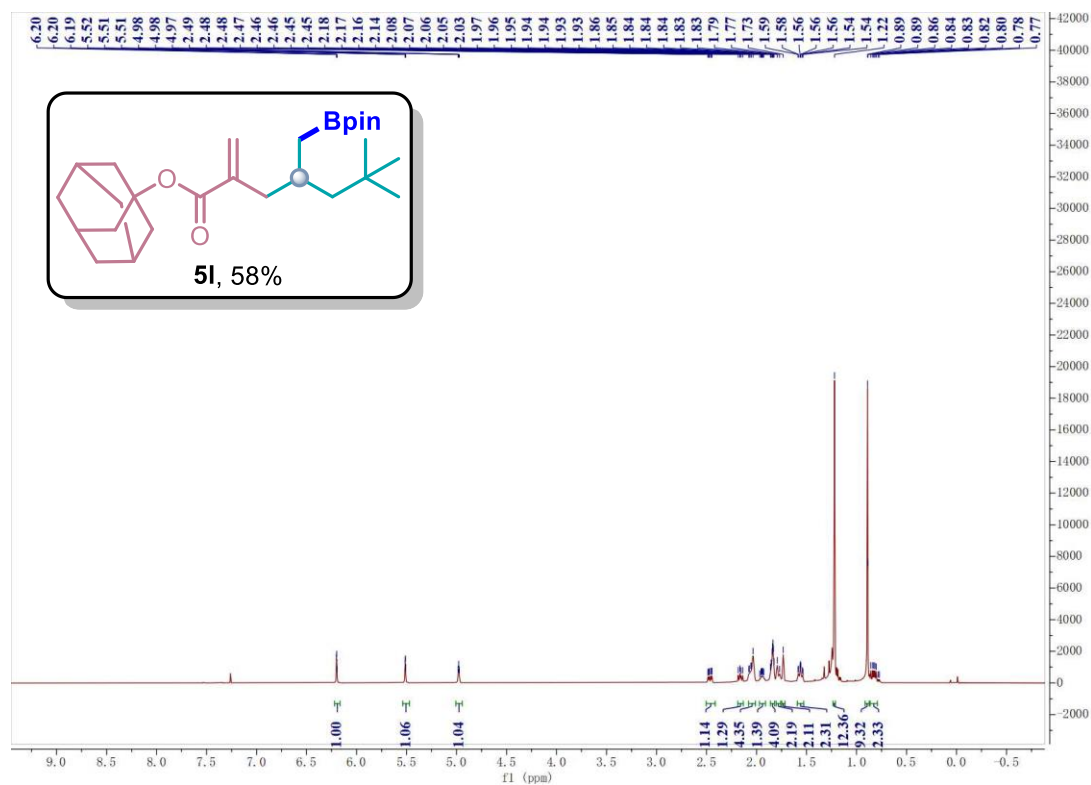


^{11}B NMR (160 MHz, Chloroform-*d*)

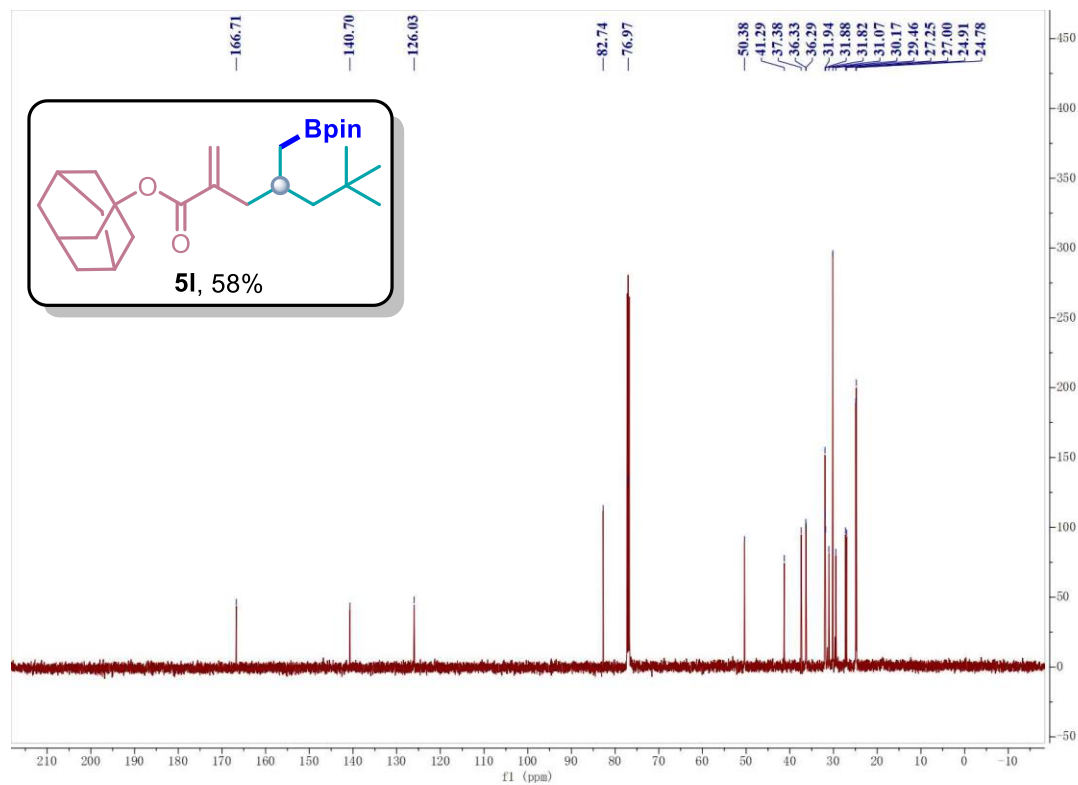


adamantan-1-yl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5I)

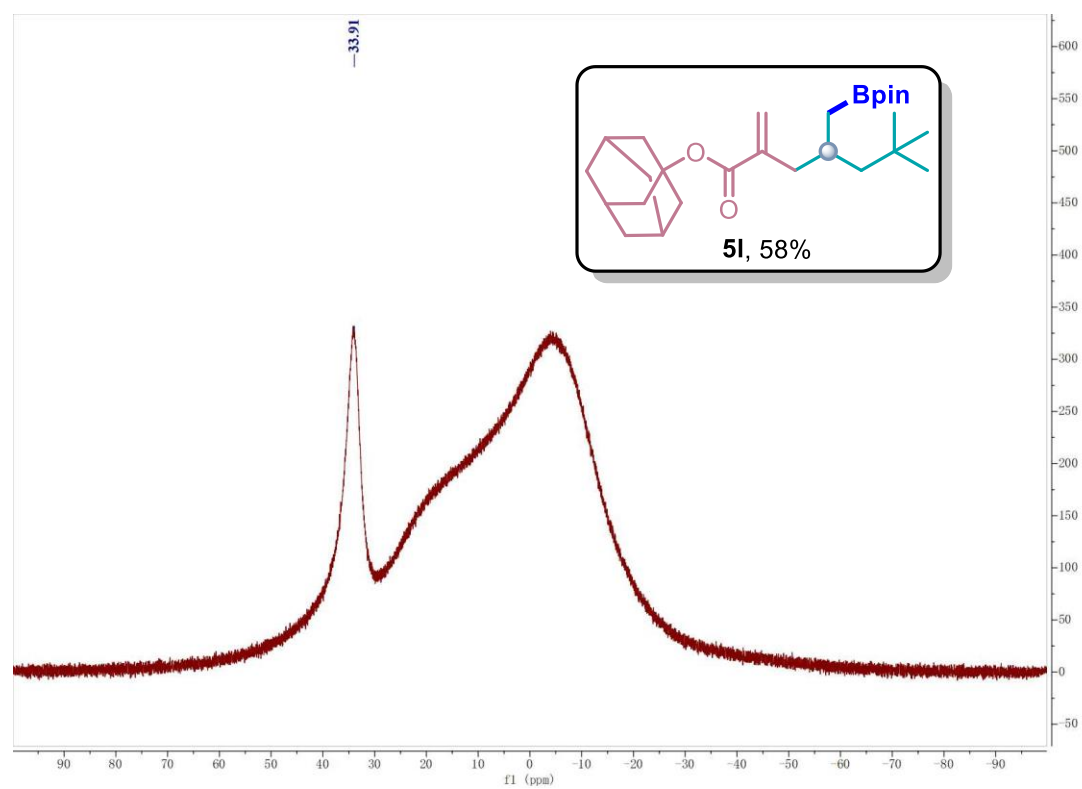
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

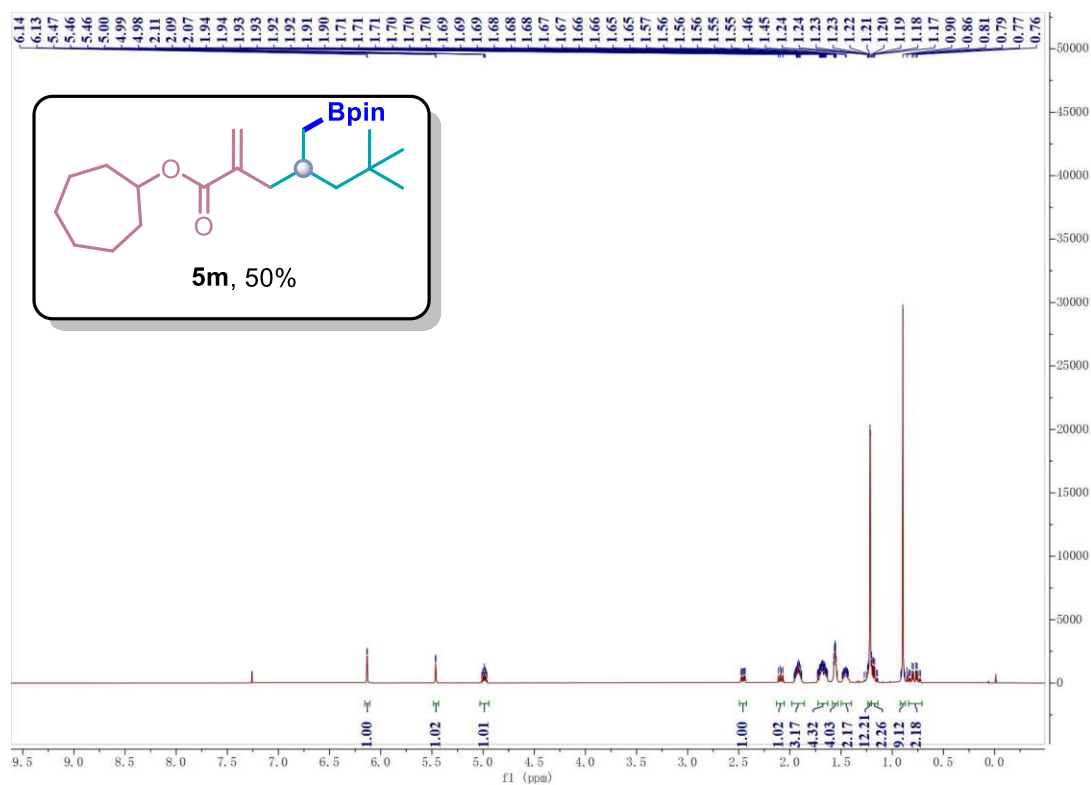


^{11}B NMR (160 MHz, Chloroform-*d*)

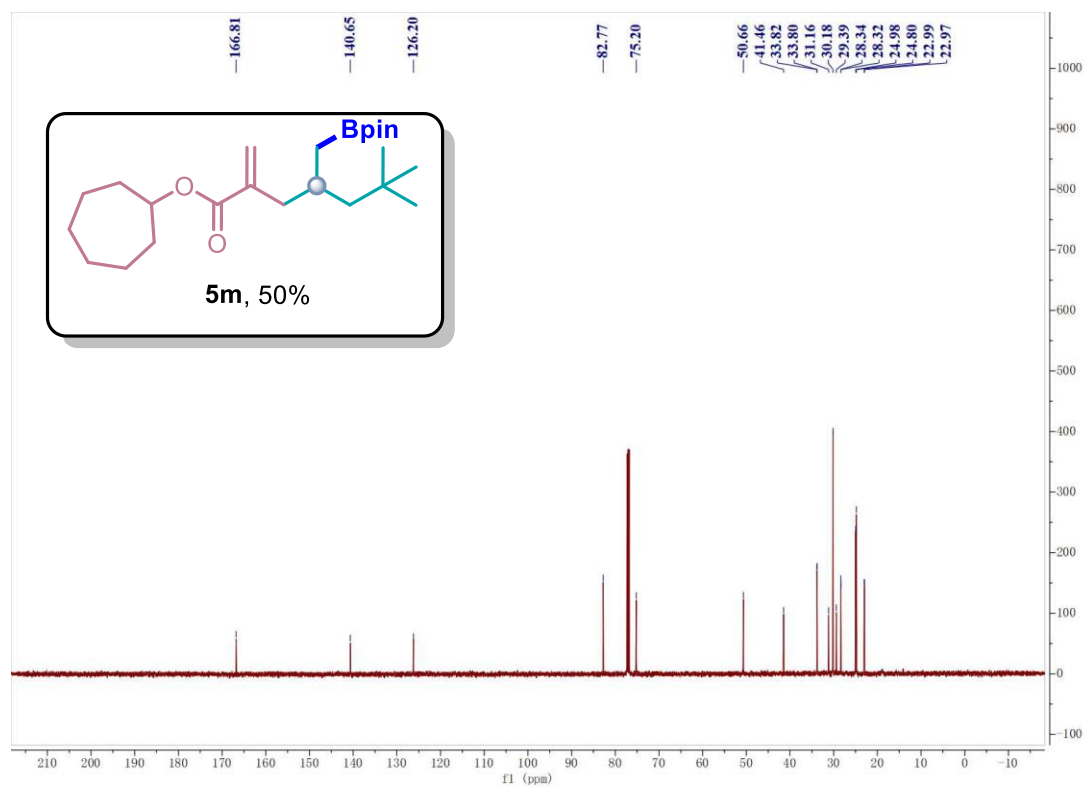


cycloheptyl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5m)

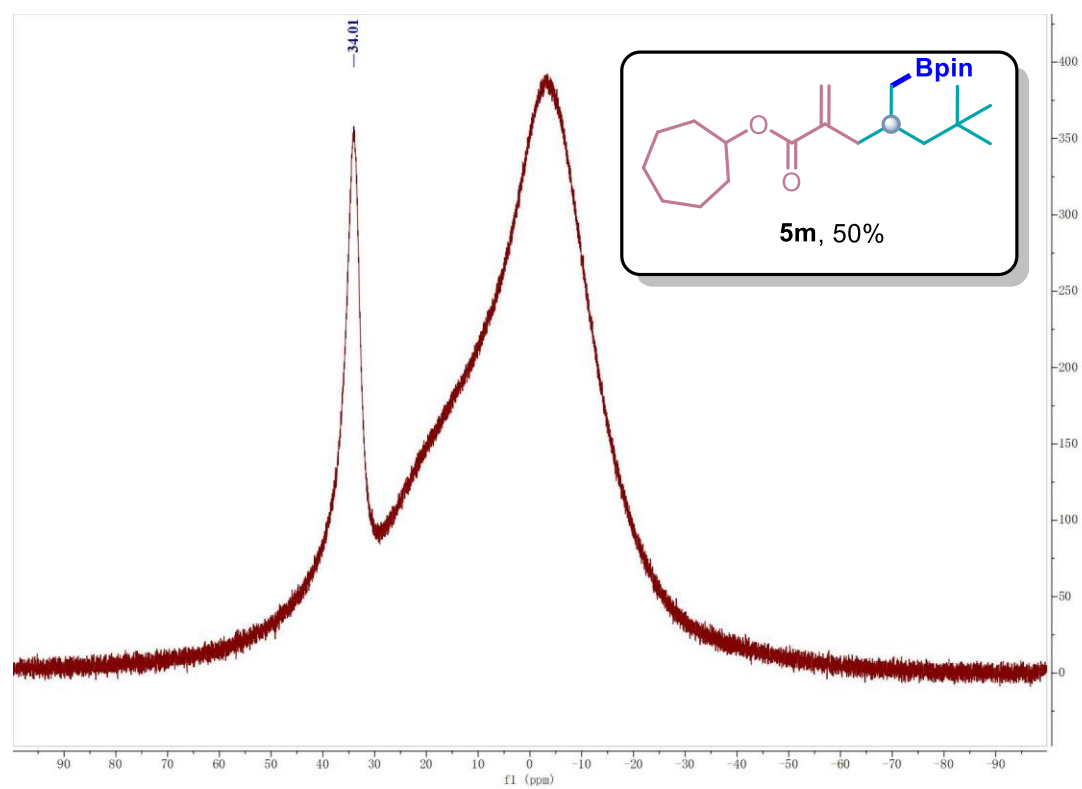
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

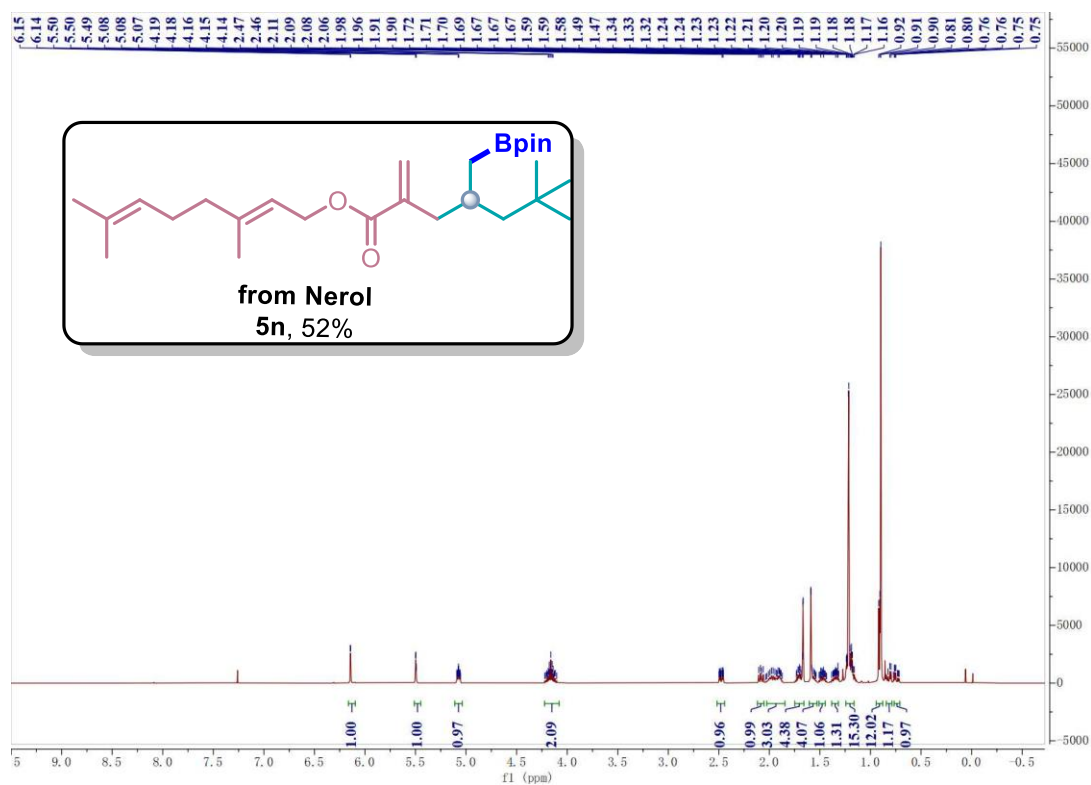


^{11}B NMR (160 MHz, Chloroform-*d*)

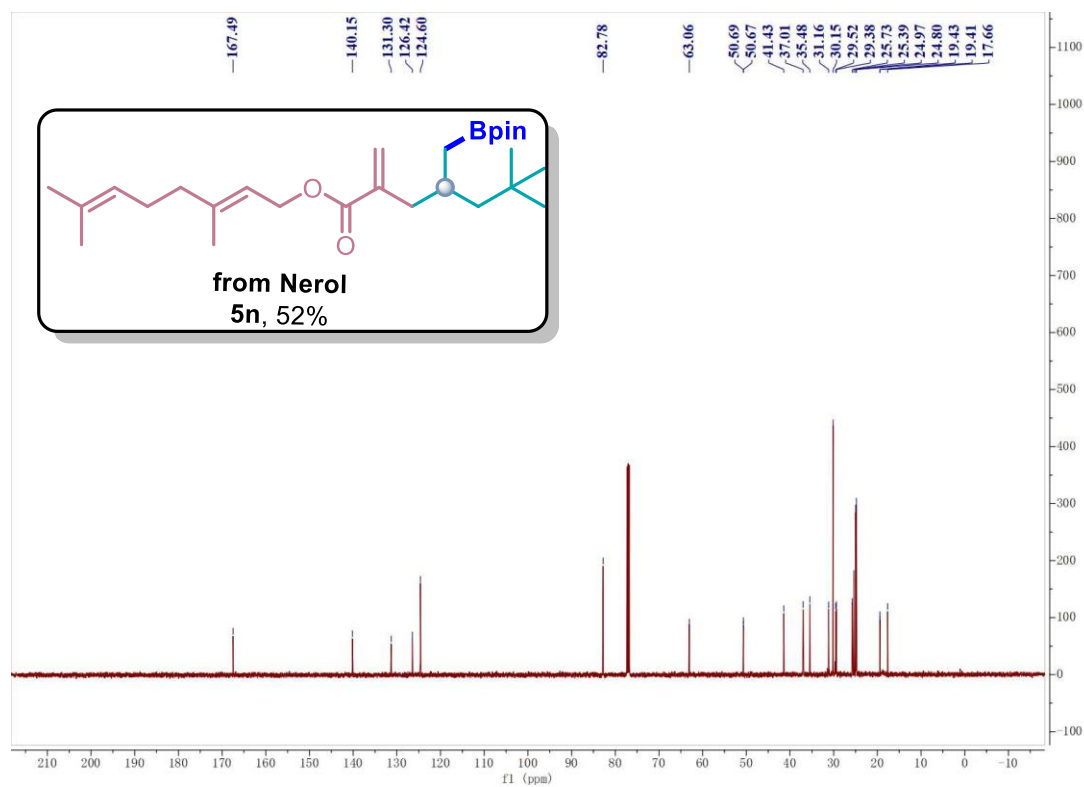


(E)-3,7-dimethylocta-2,6-dien-1-yl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5n)

¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)



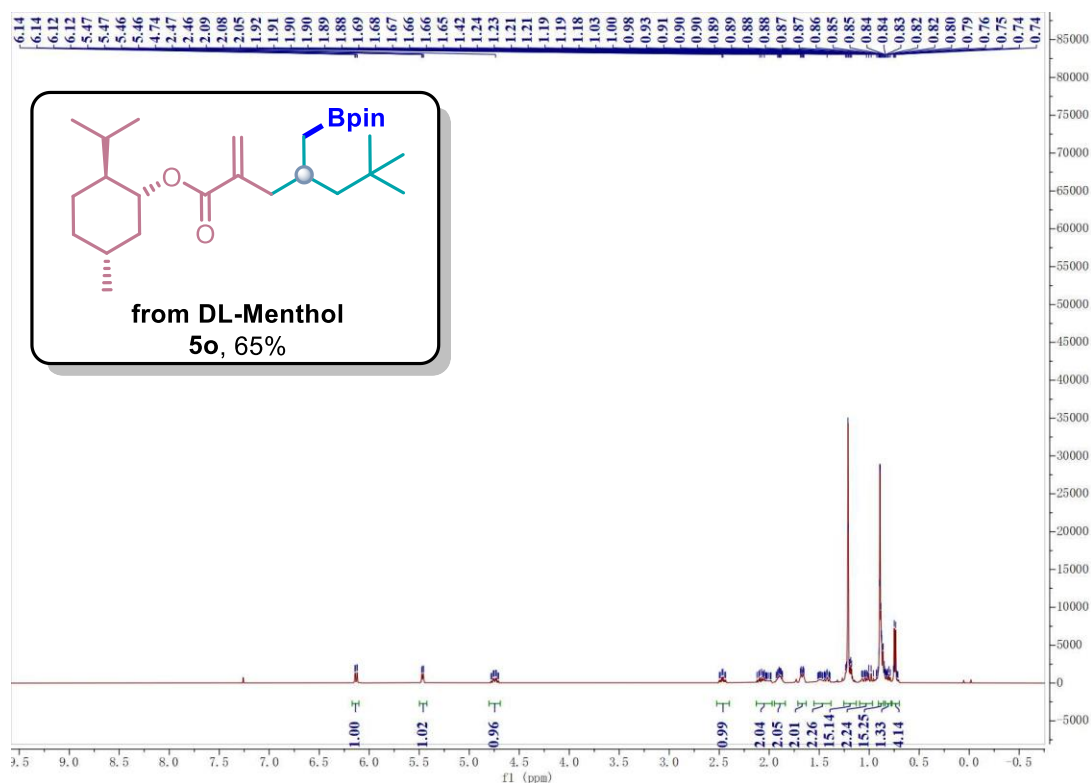
from Nerol
5n, 52%

(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl

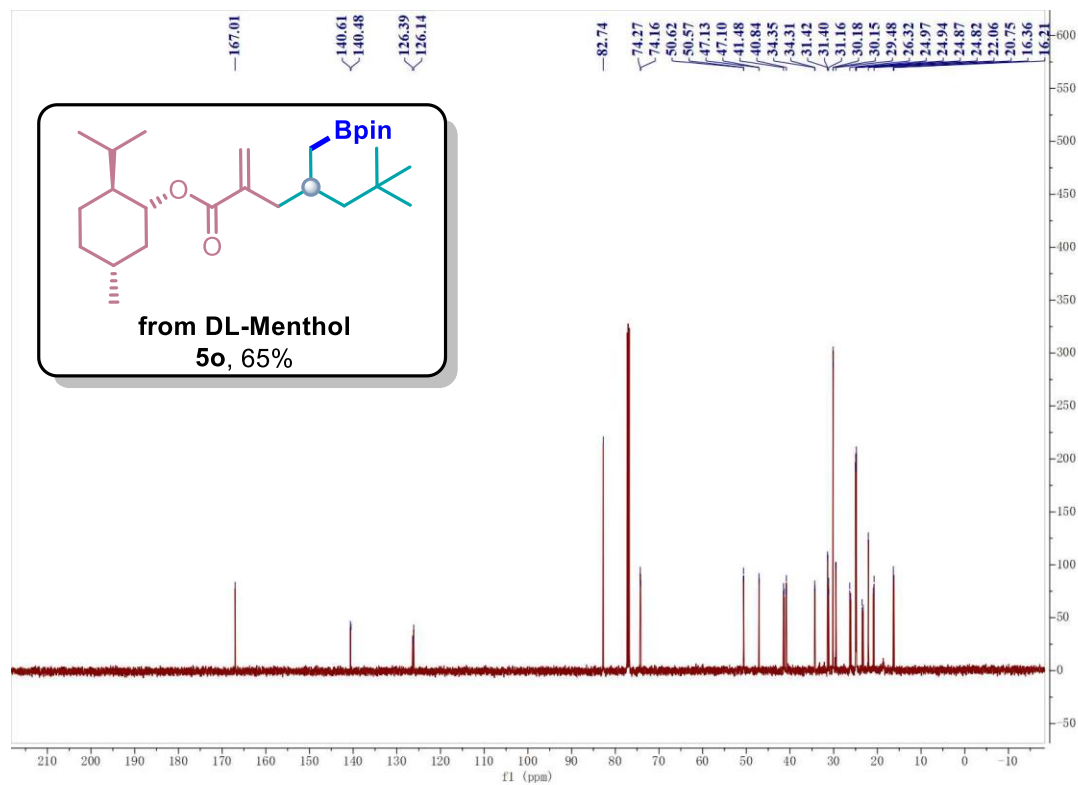
6,6-dimethyl-2-methylene-4-((4,4,5,5-

tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5o)

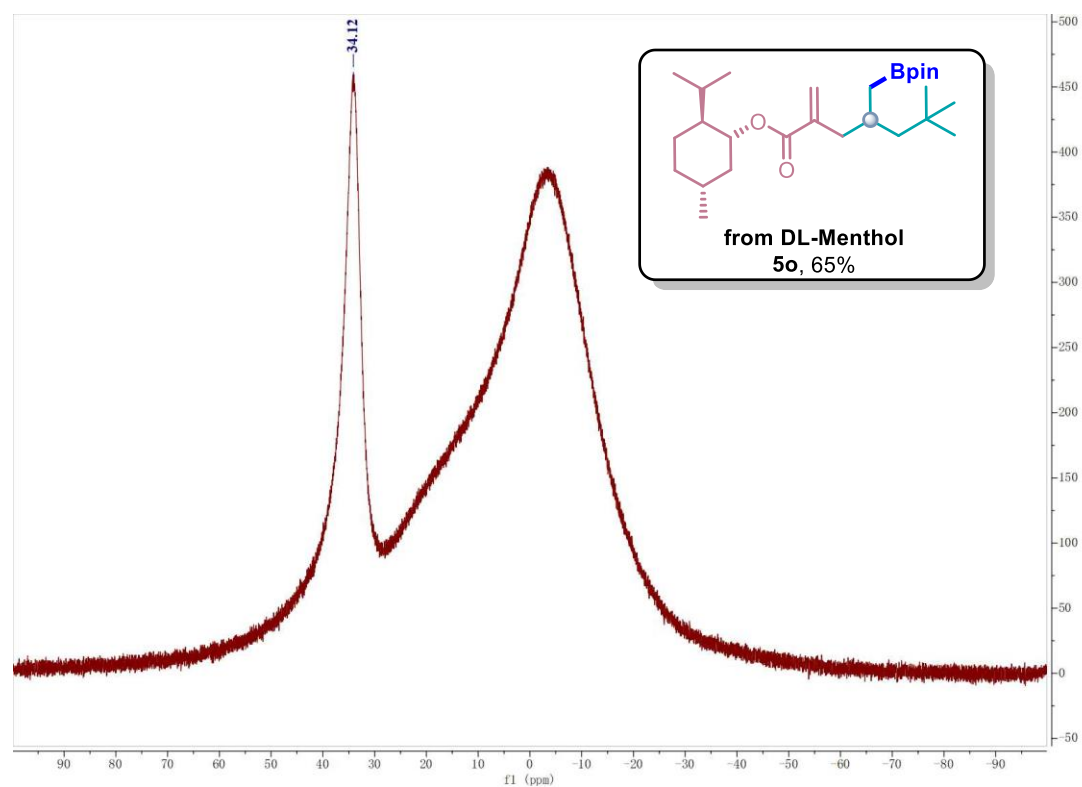
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

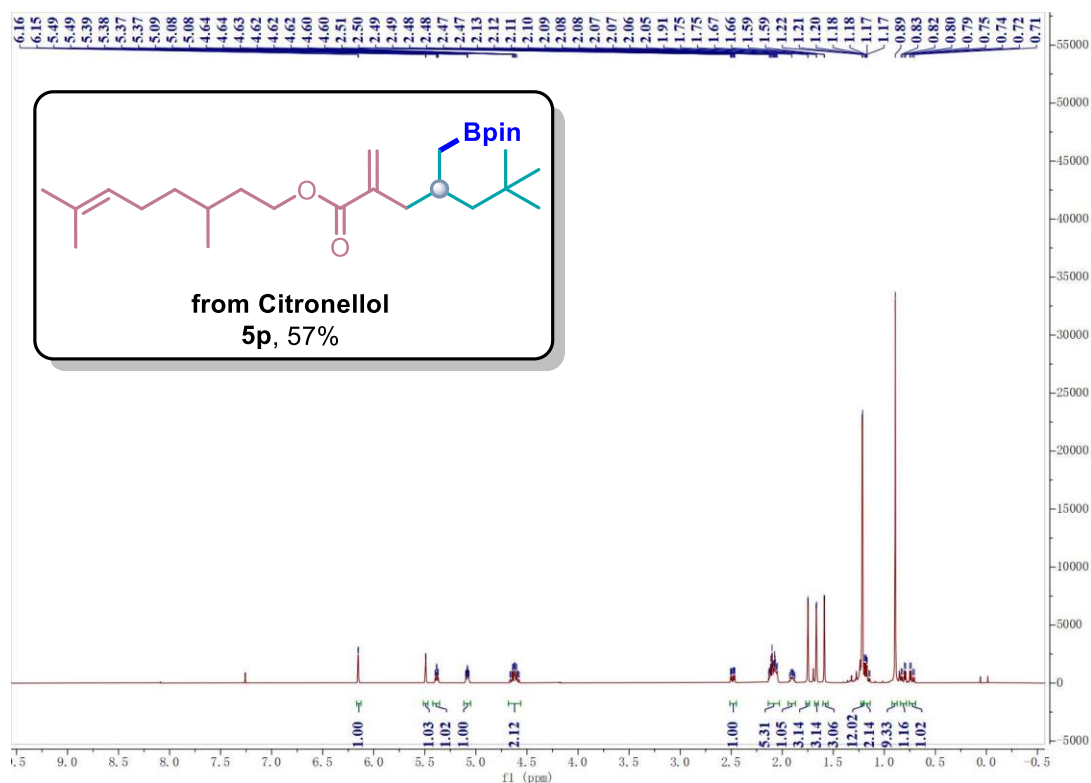


^{11}B NMR (160 MHz, Chloroform-*d*)

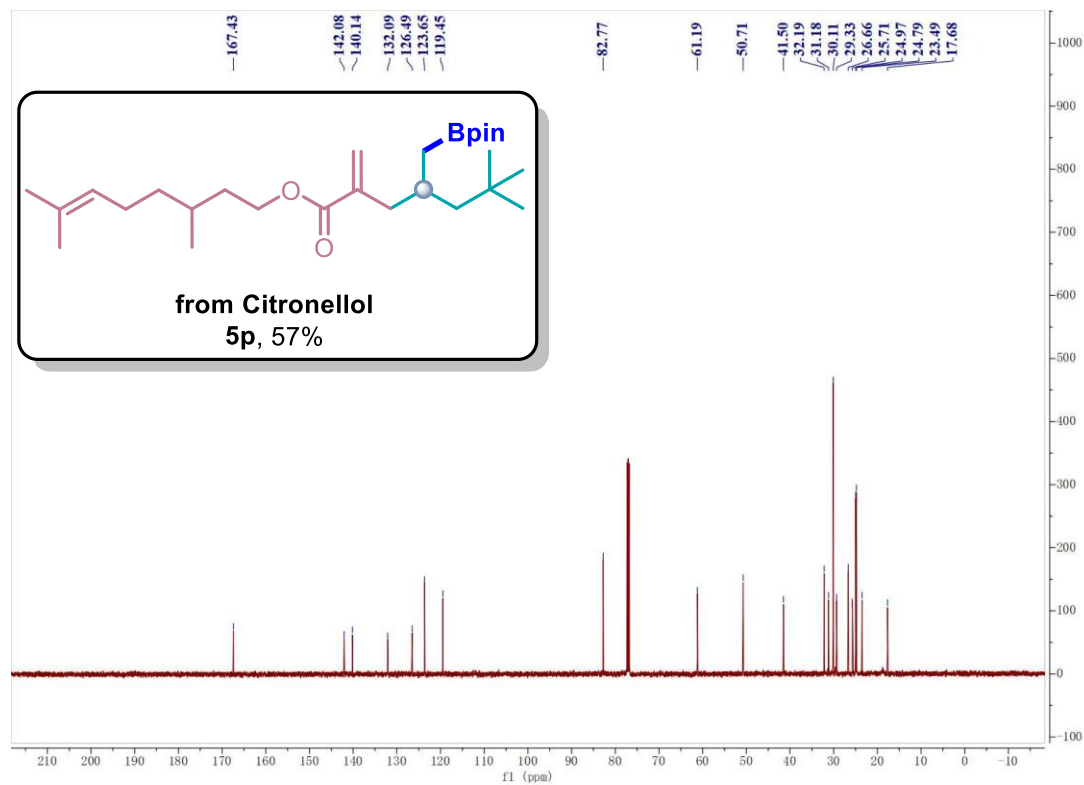


3,7-dimethyloct-6-en-1-yl 6,6-dimethyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (5p)

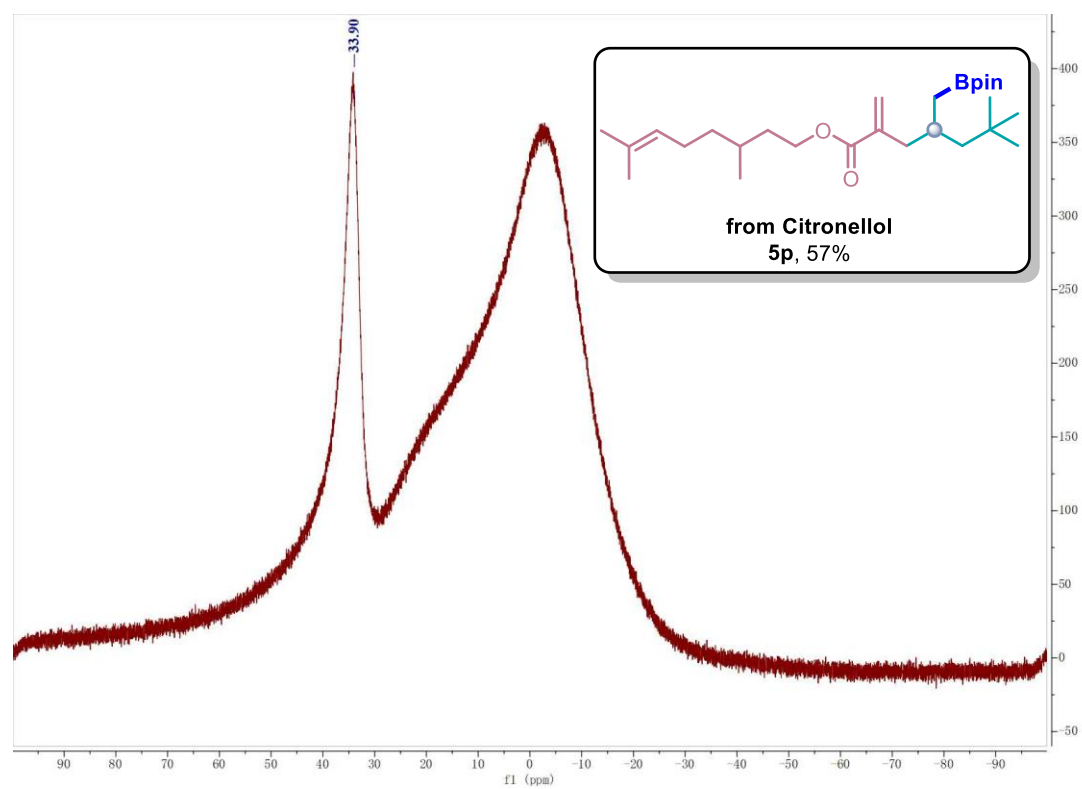
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

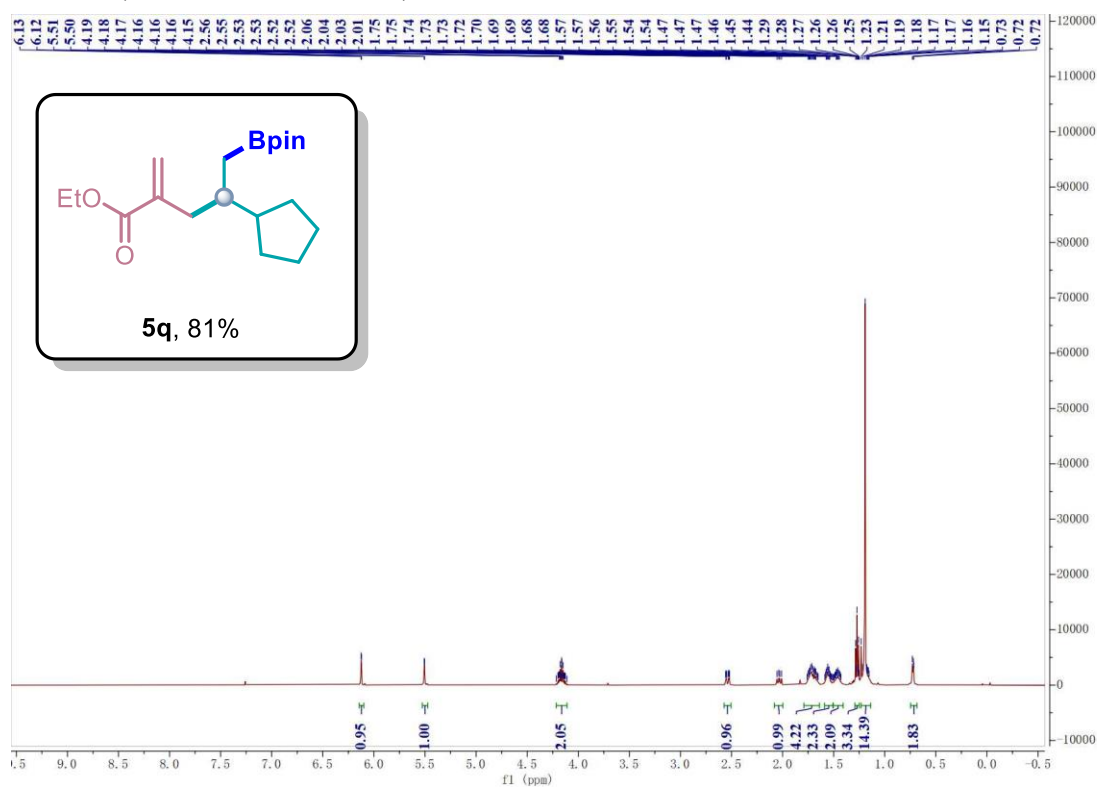


^{11}B NMR (160 MHz, Chloroform-*d*)

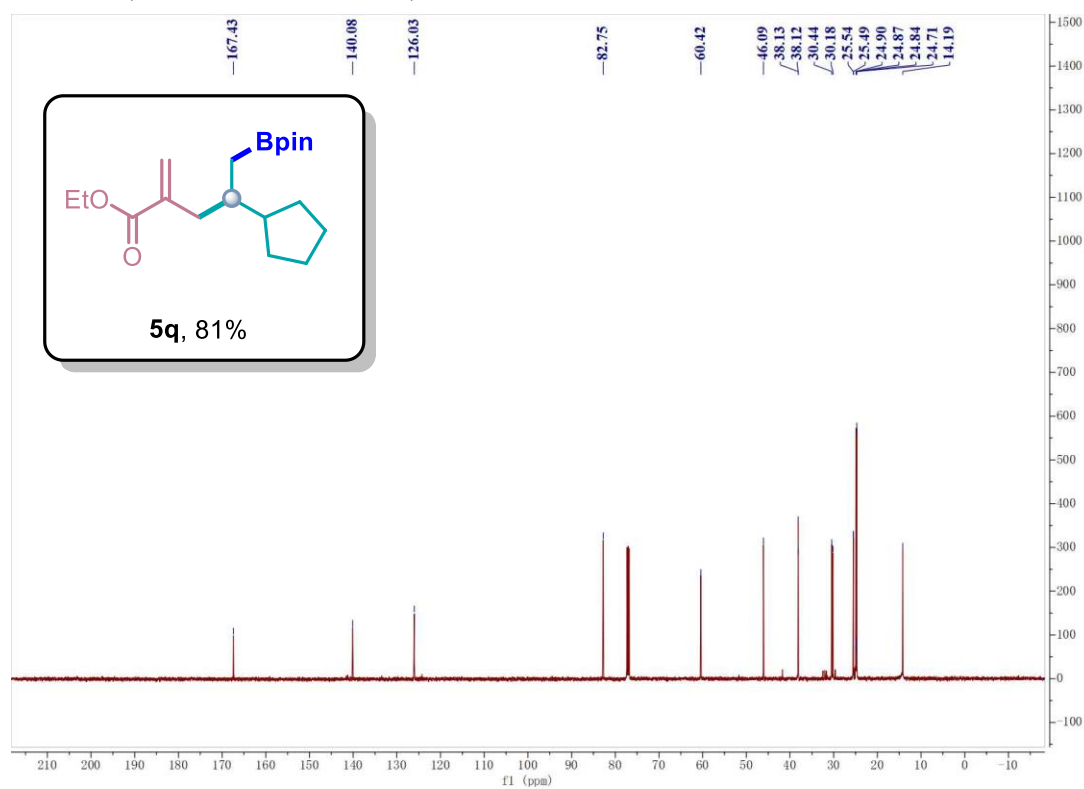


ethyl 4-cyclopentyl-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate
(5q)

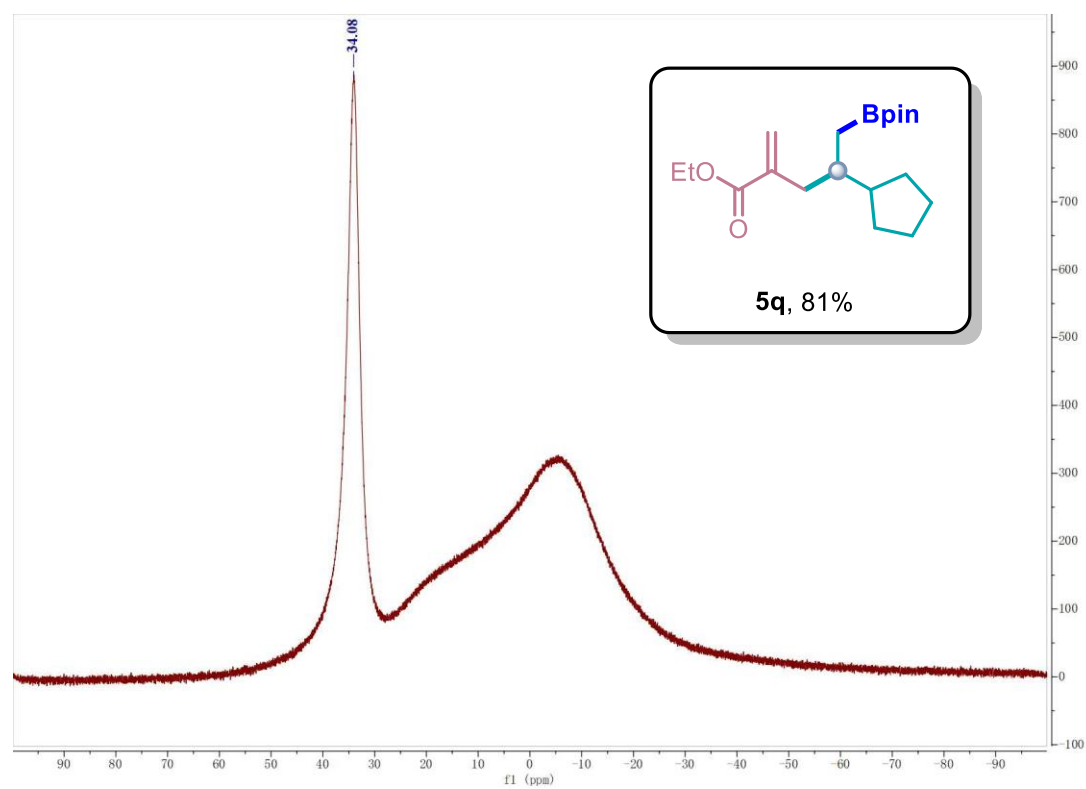
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

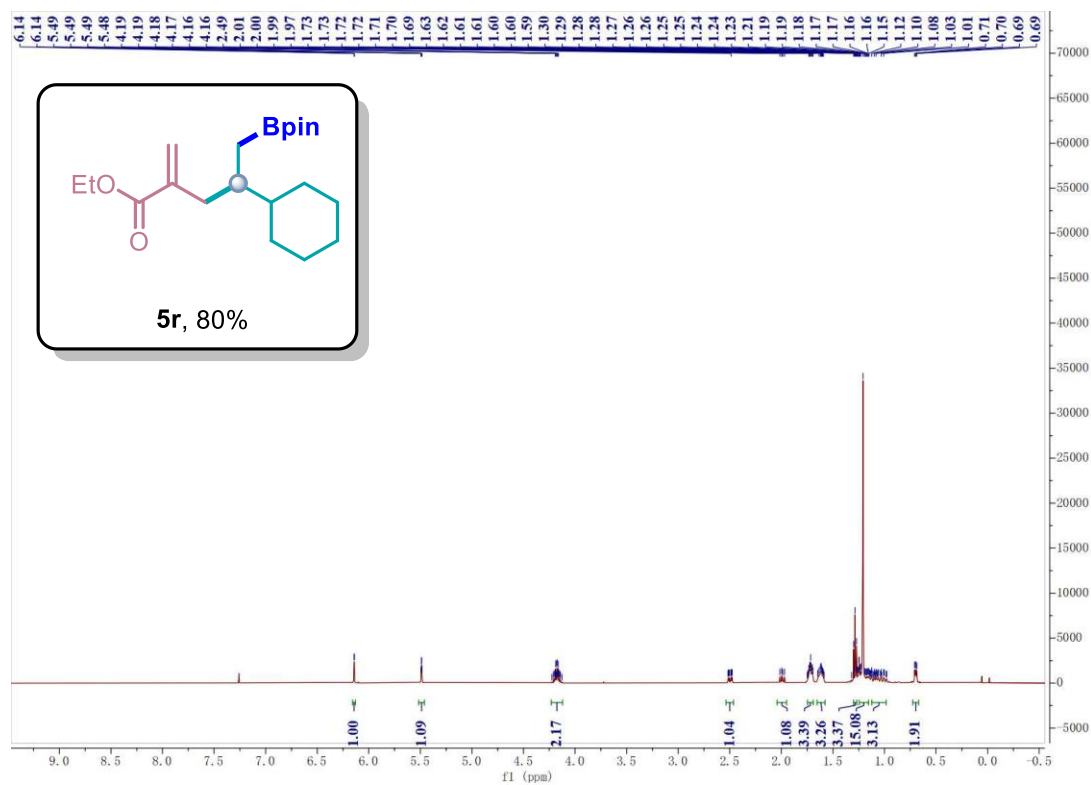


^{11}B NMR (160 MHz, Chloroform-*d*)

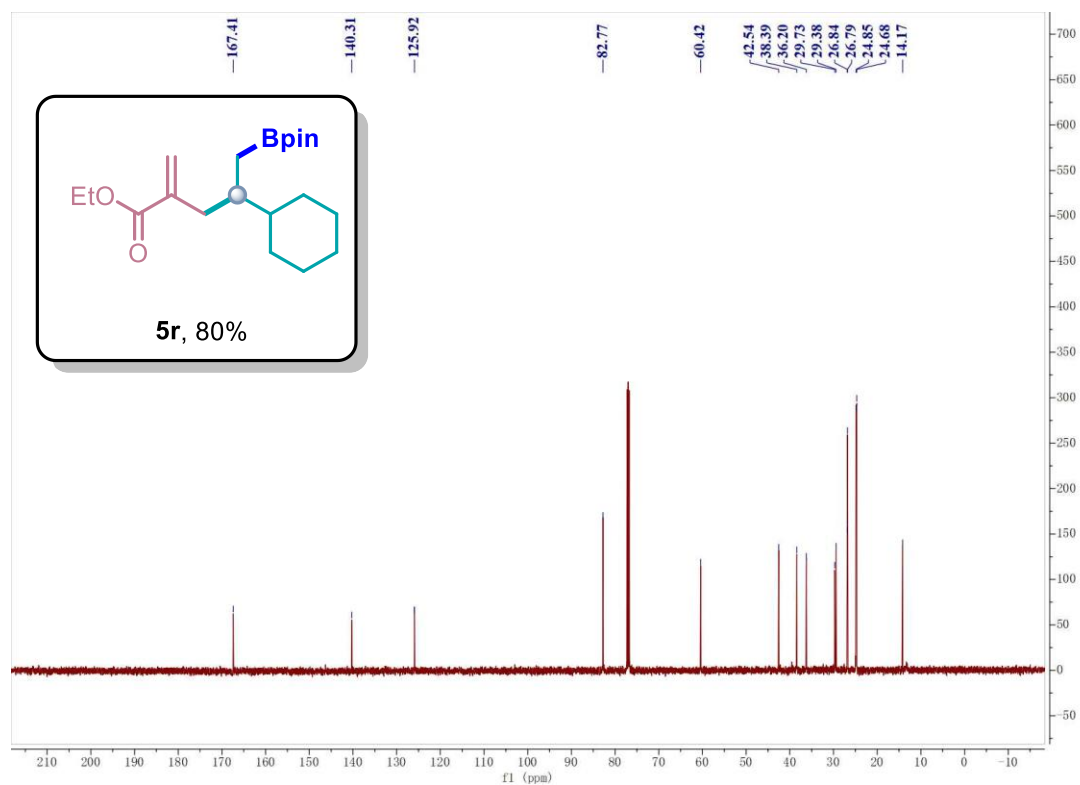


ethyl 4-cyclohexyl-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**5r**)

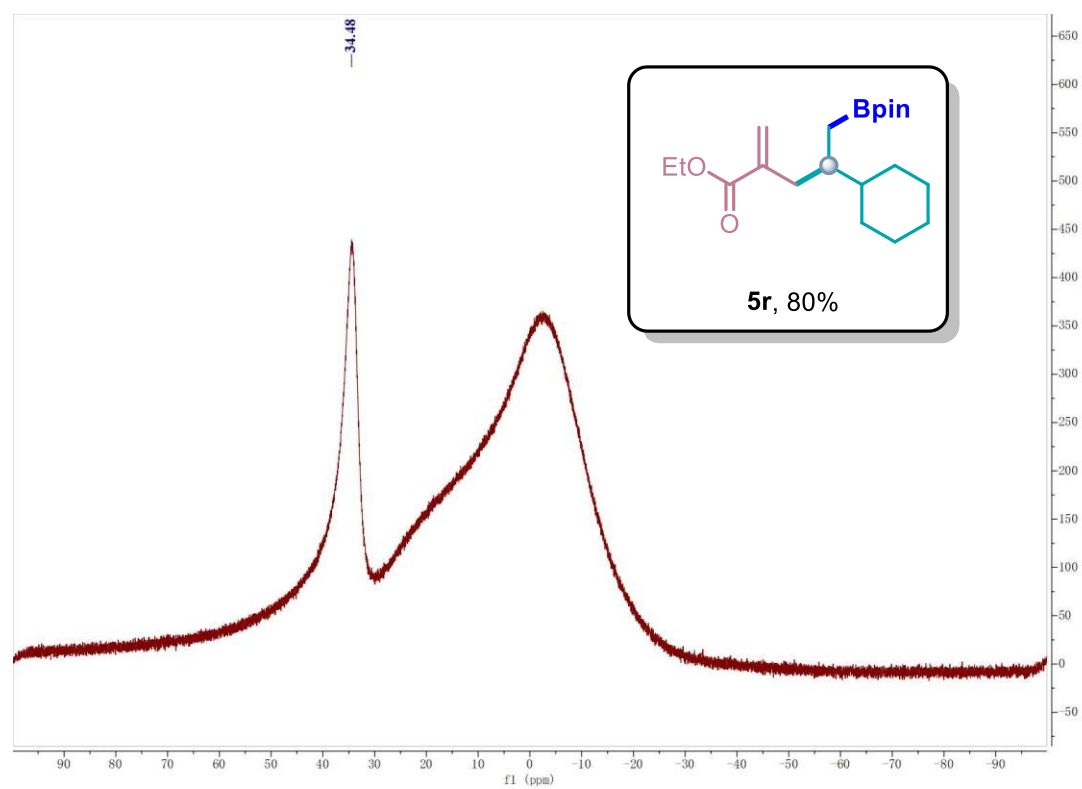
^1H NMR (500 MHz, Chloroform-*d*)



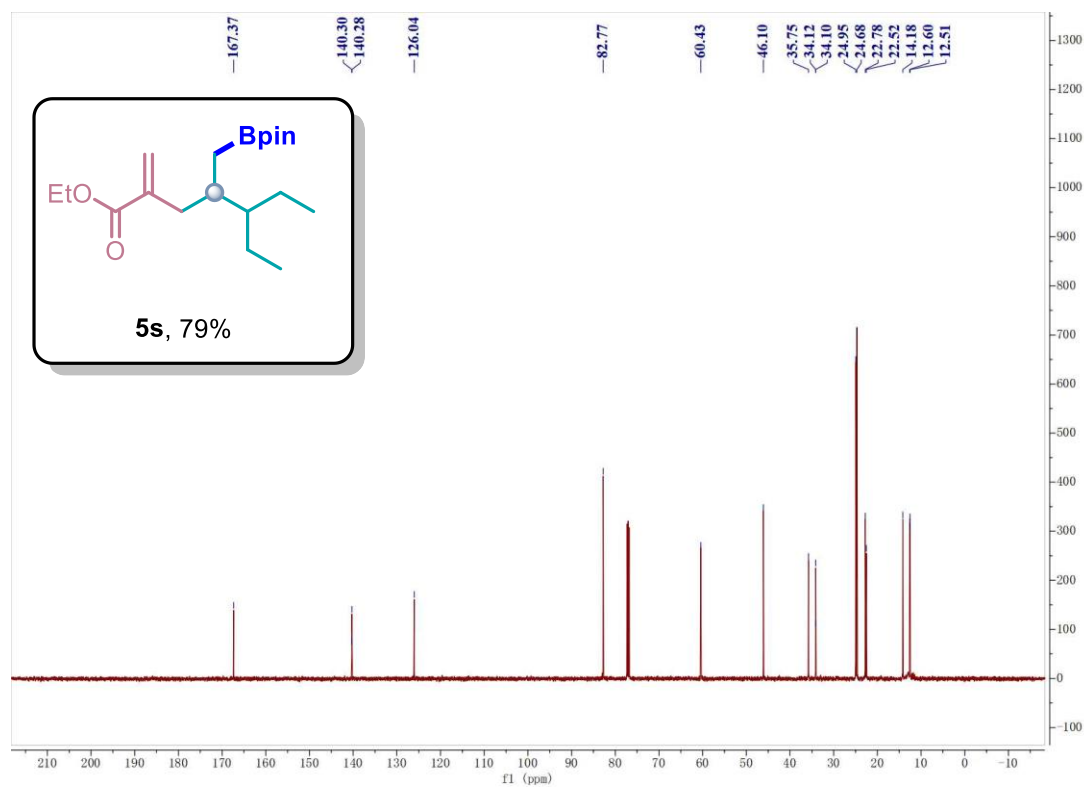
^{13}C NMR (126 MHz, Chloroform-*d*)



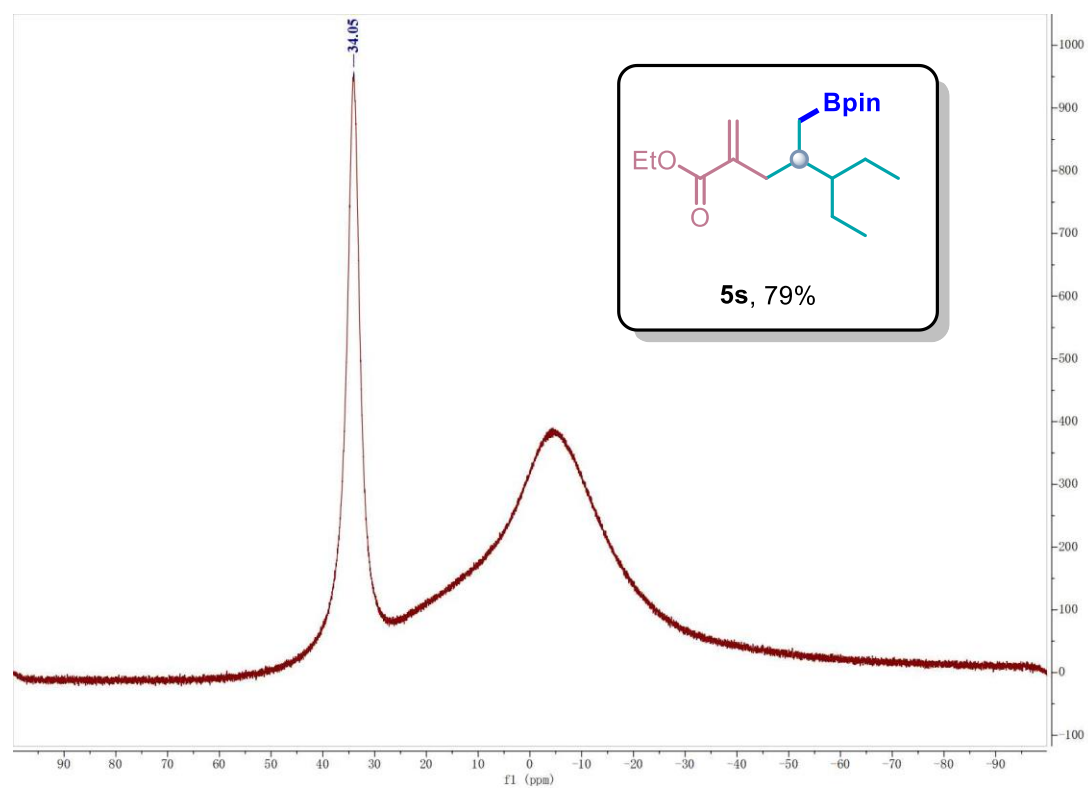
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

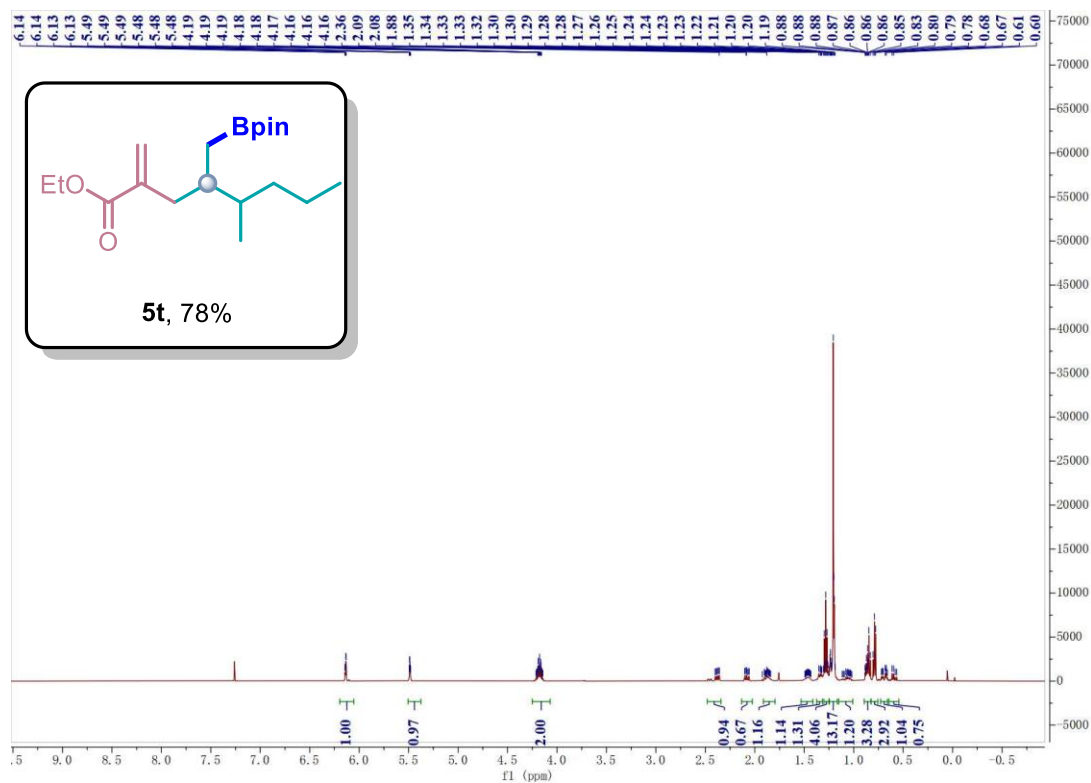


^{11}B NMR (160 MHz, Chloroform-*d*)

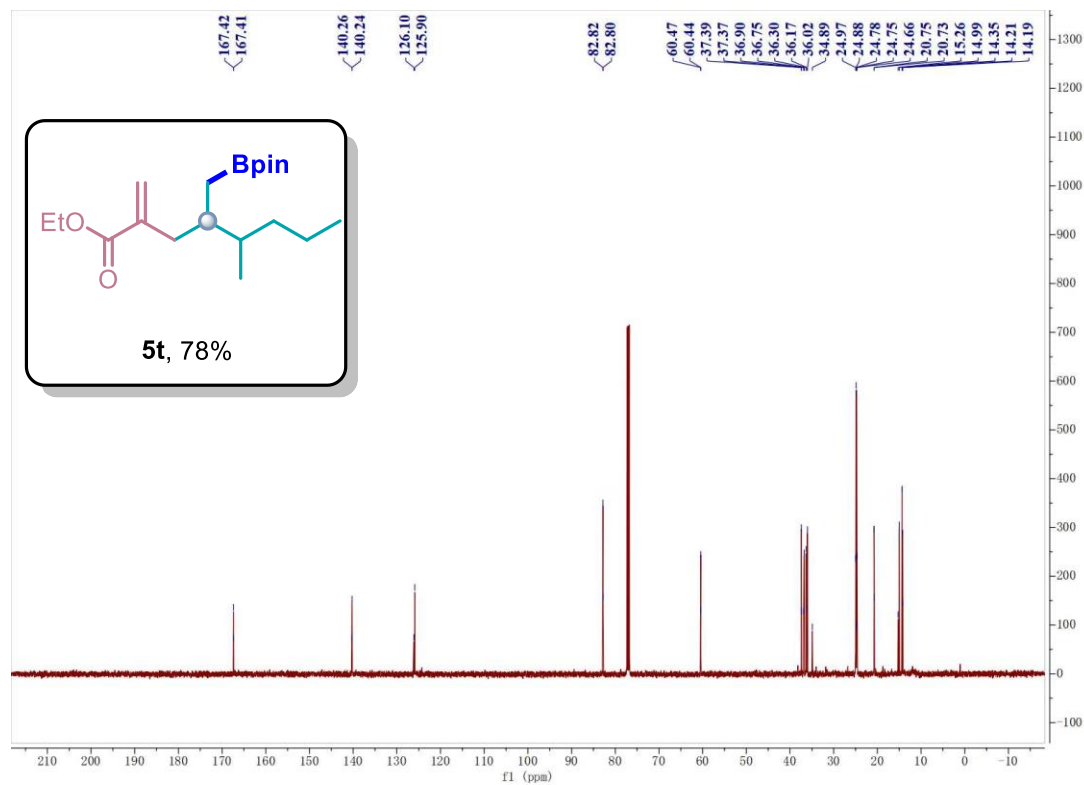


ethyl 5-methyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)octanoate
(5t)

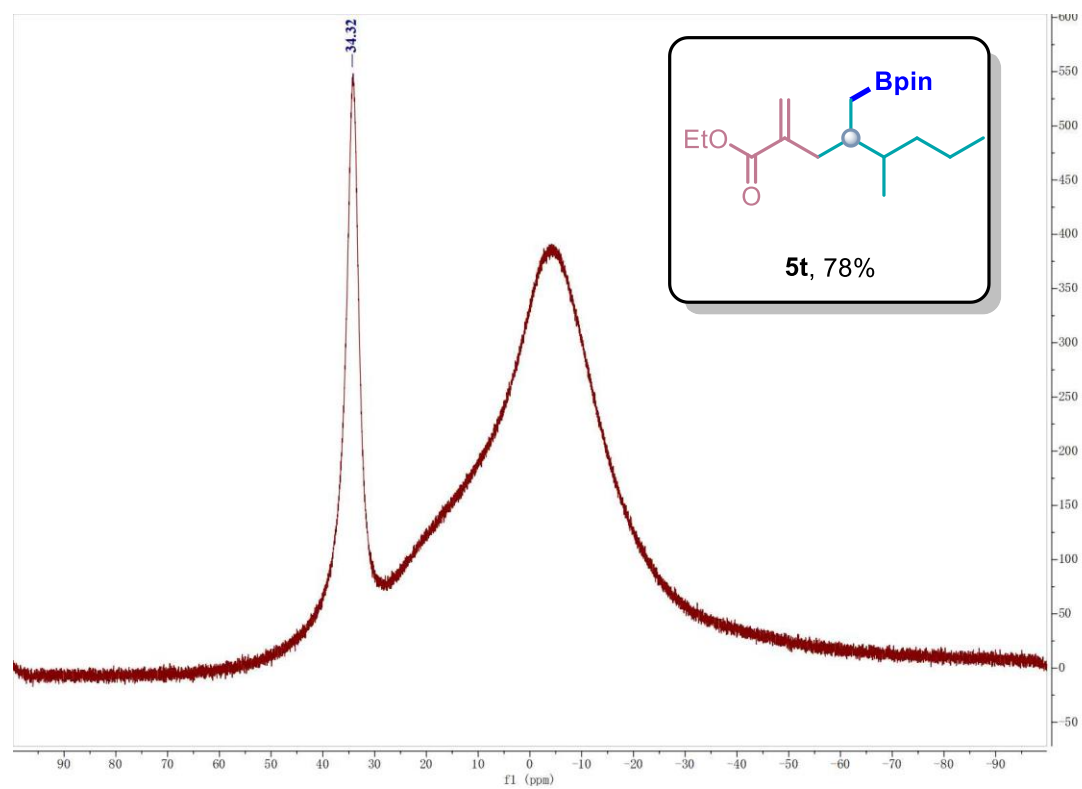
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

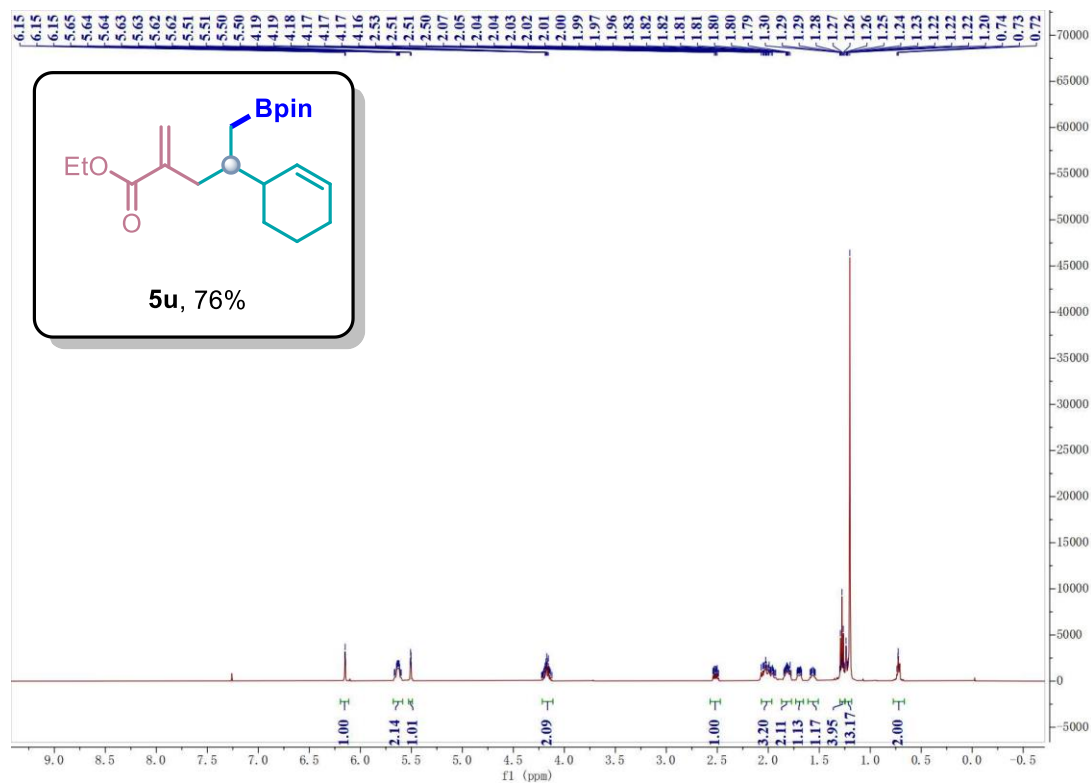


^{11}B NMR (160 MHz, Chloroform-*d*)

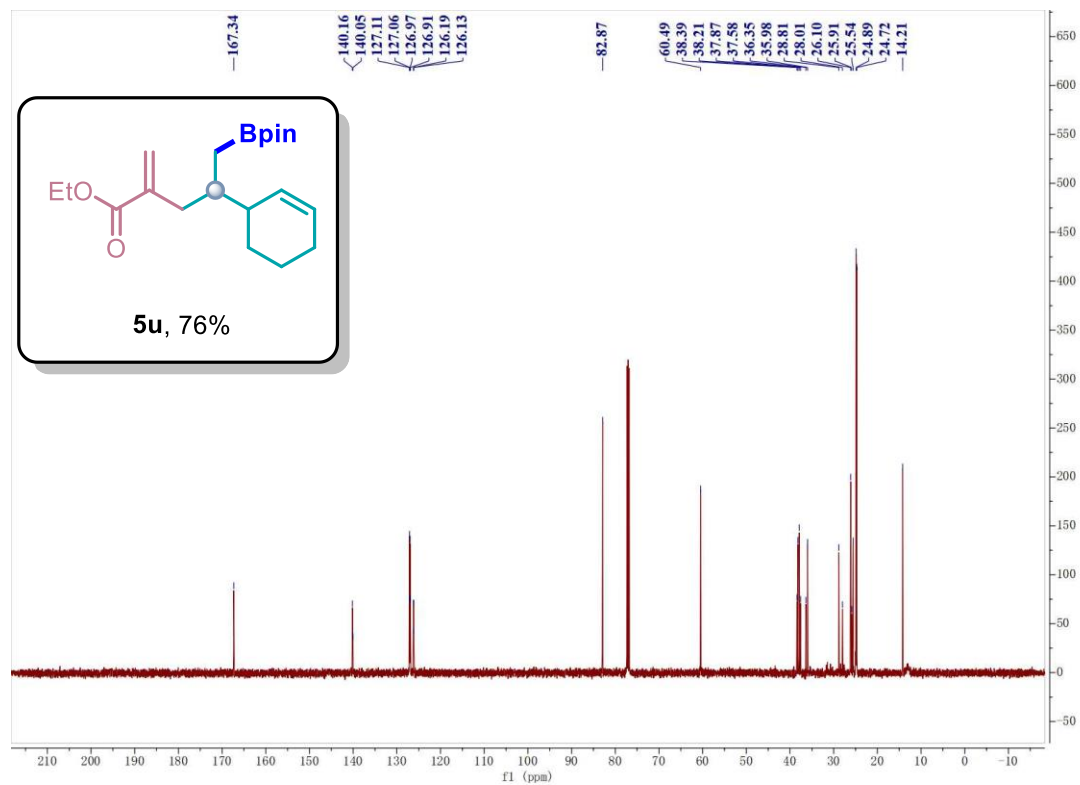


ethyl 4-(cyclohex-2-en-1-yl)-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**5u**)

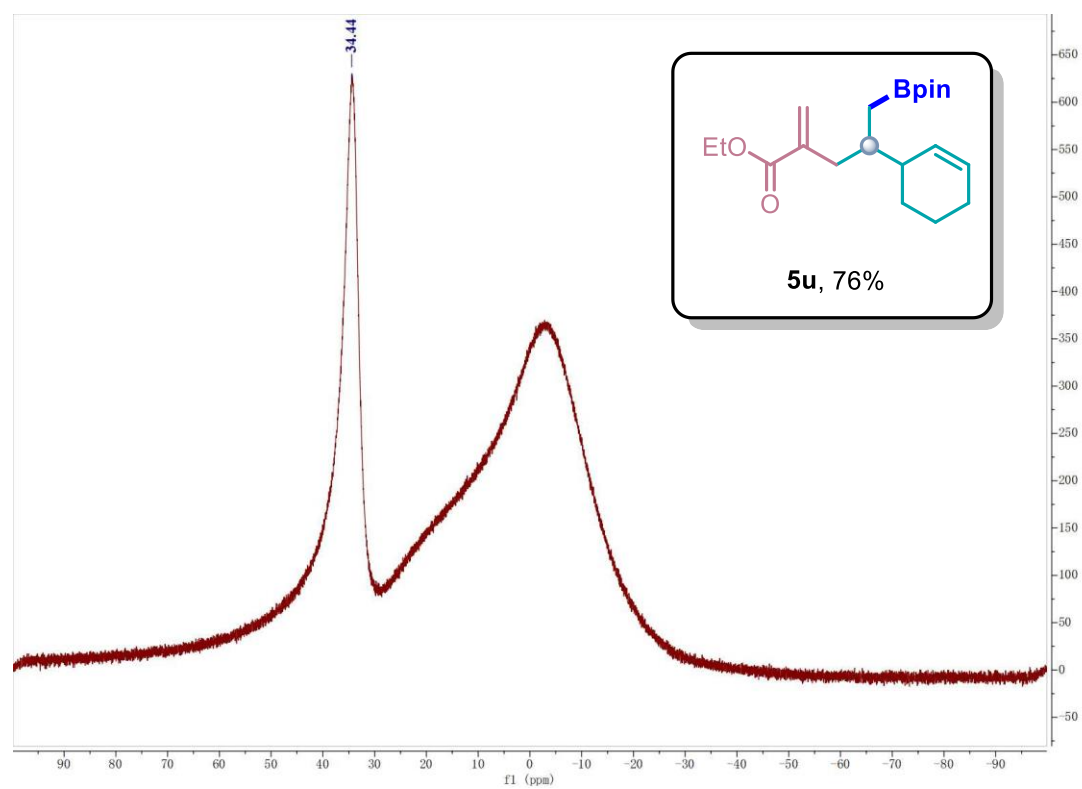
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

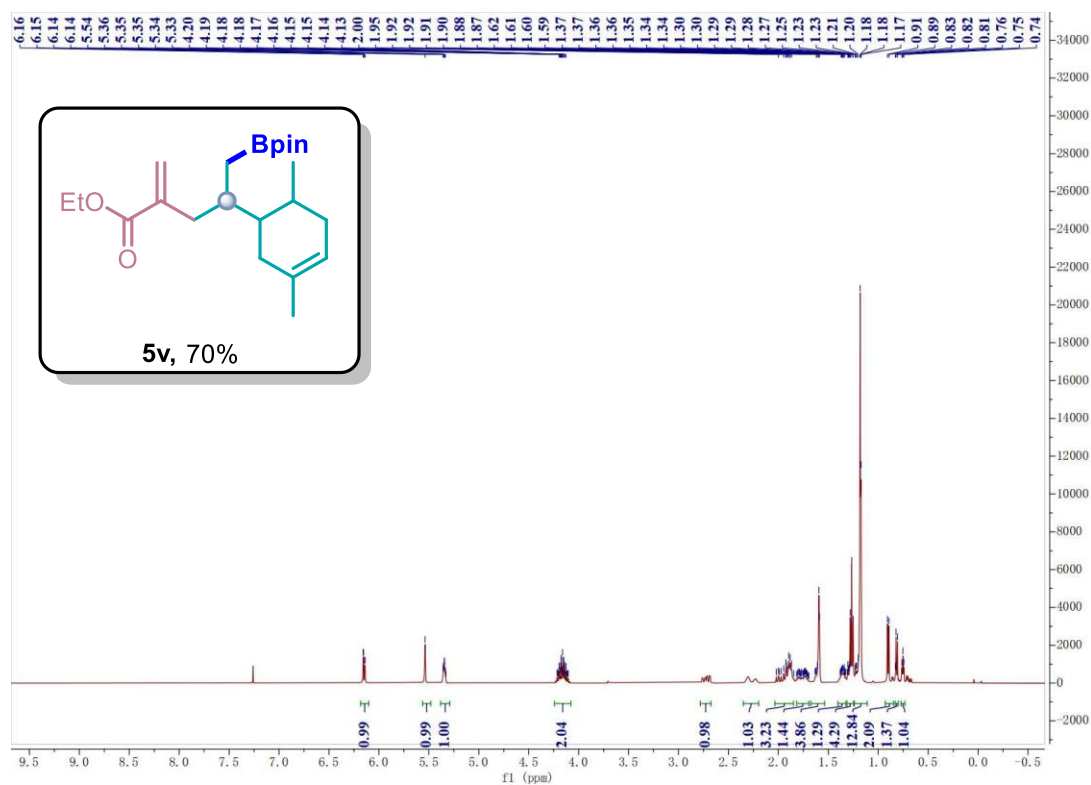


^{11}B NMR (160 MHz, Chloroform-*d*)

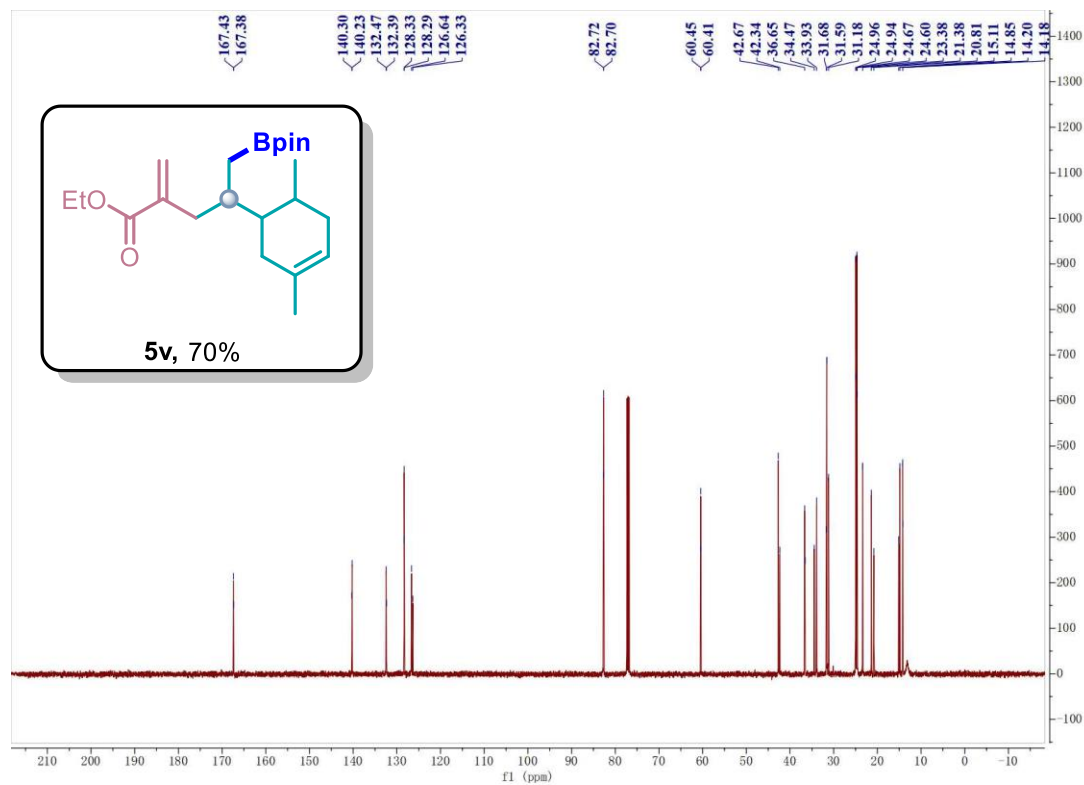


ethyl 4-(3,6-dimethylcyclohex-3-en-1-yl)-2-methylene-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**5v**)

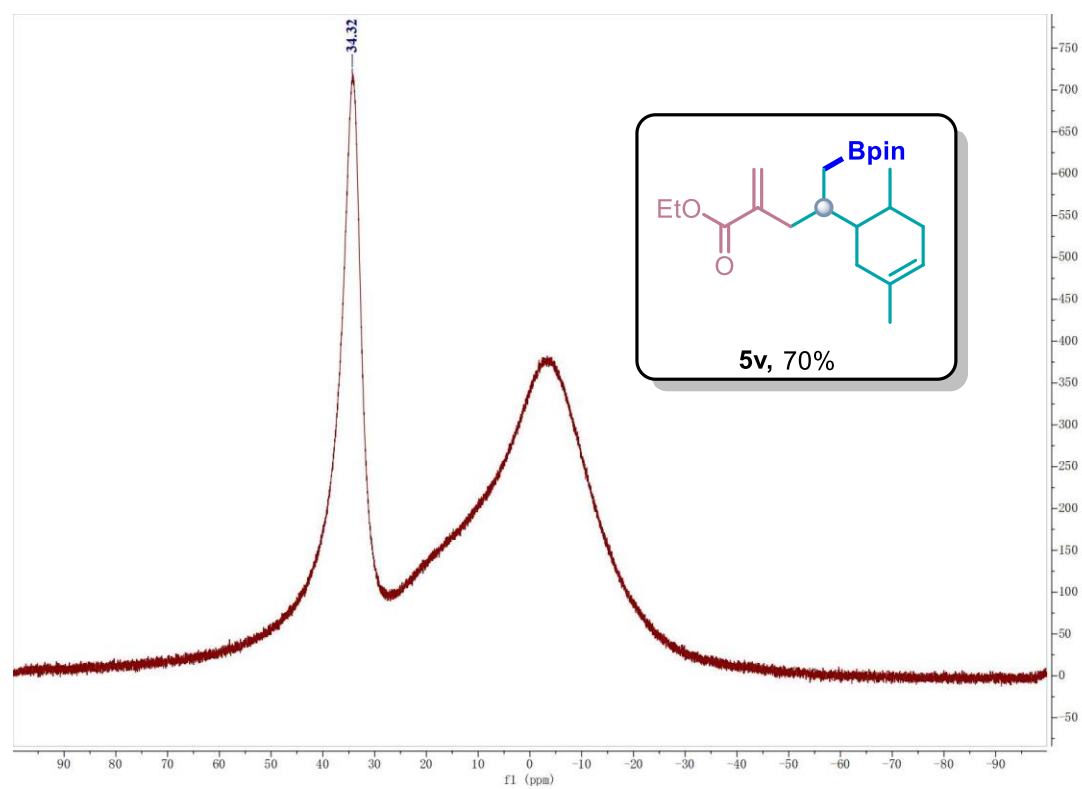
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

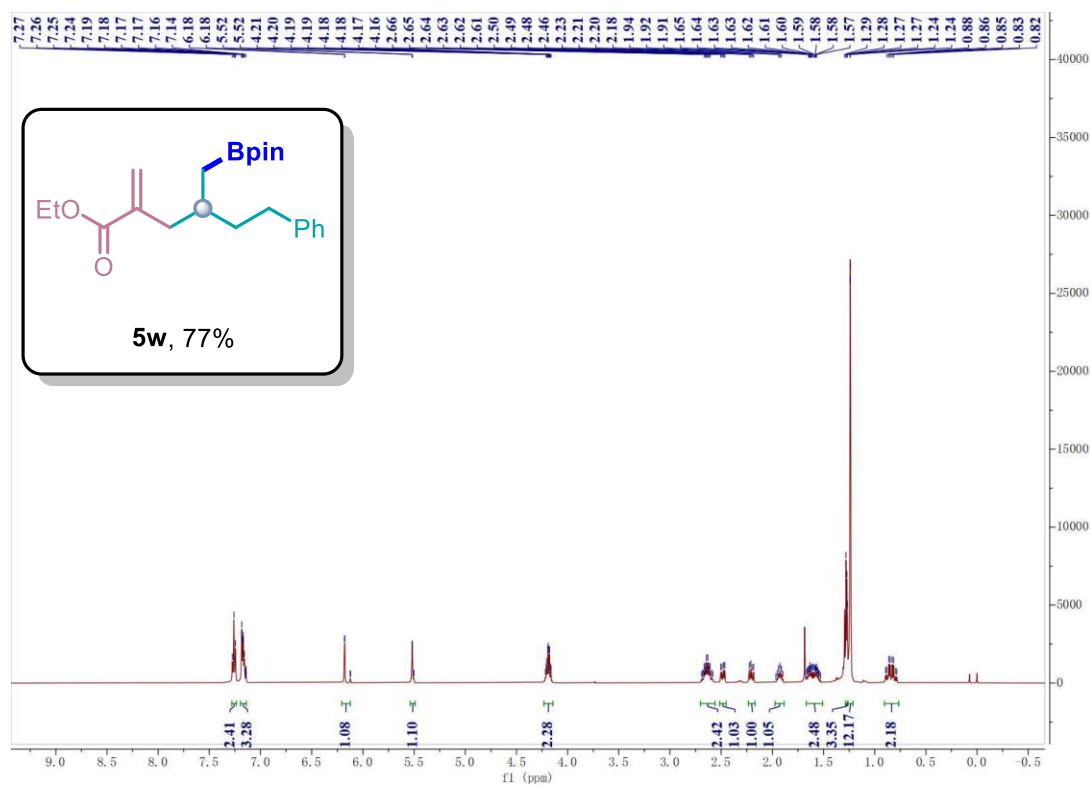


^{11}B NMR (160 MHz, Chloroform-*d*)

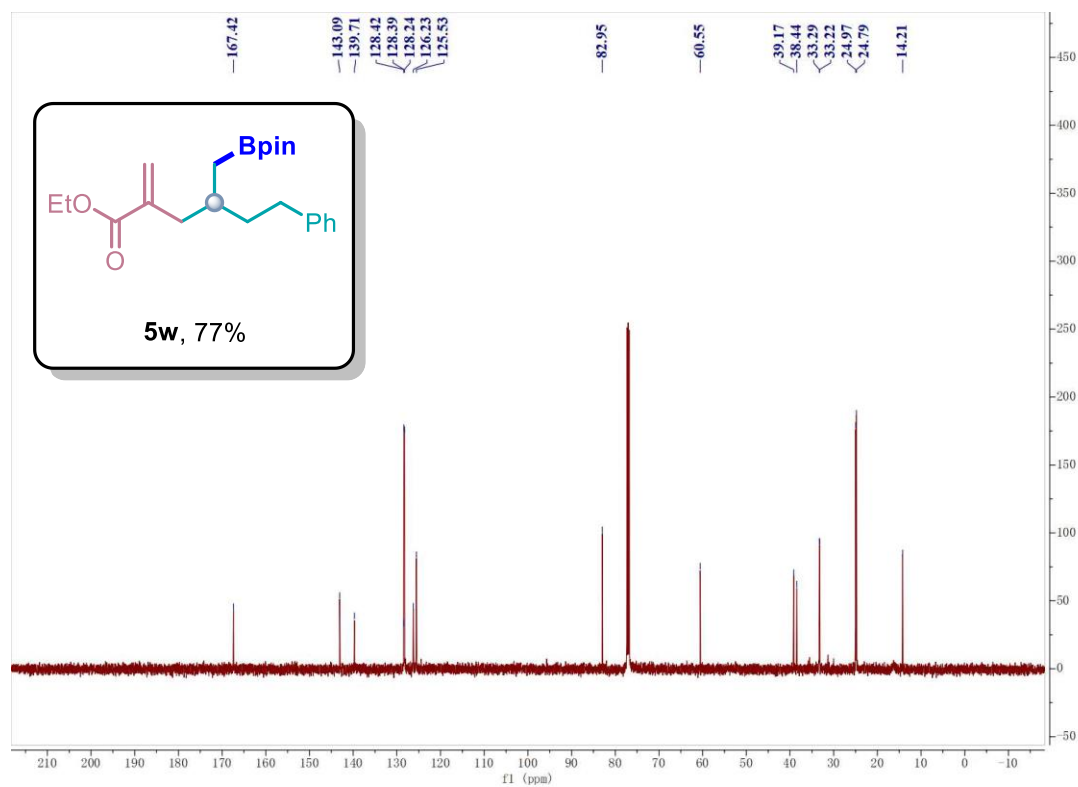


ethyl 2-methylene-6-phenyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexanoate (**5w**)

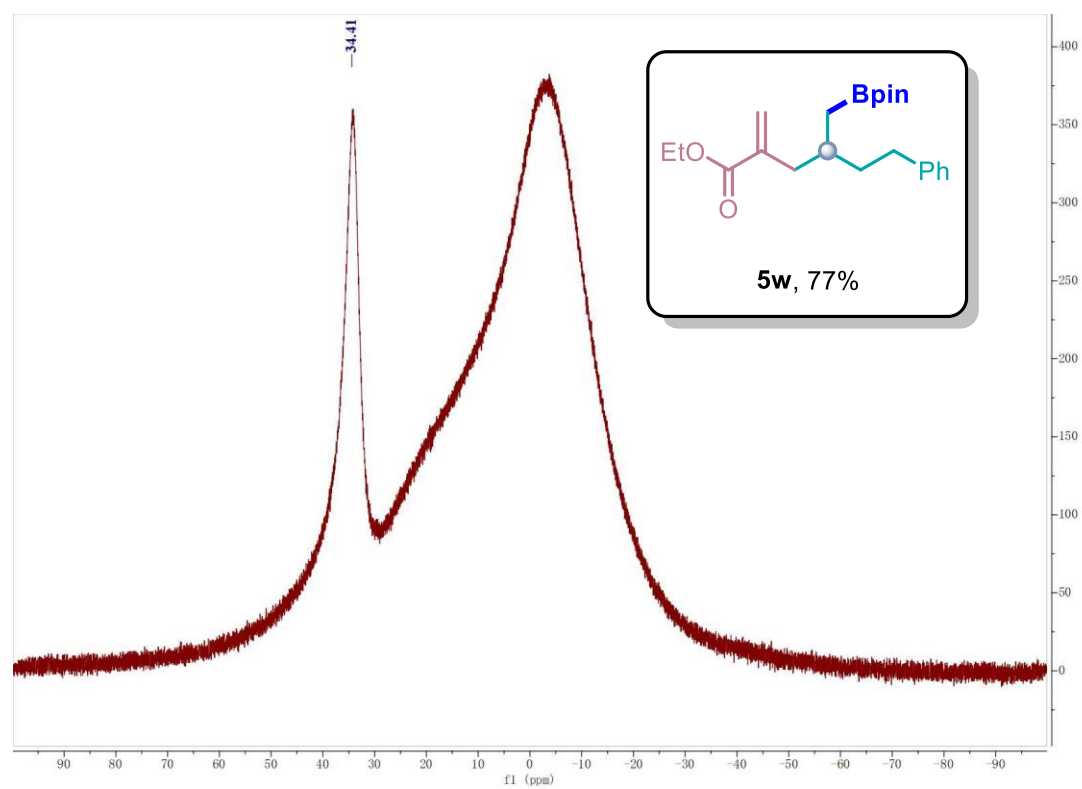
¹H NMR (500 MHz, Chloroform-*d*)



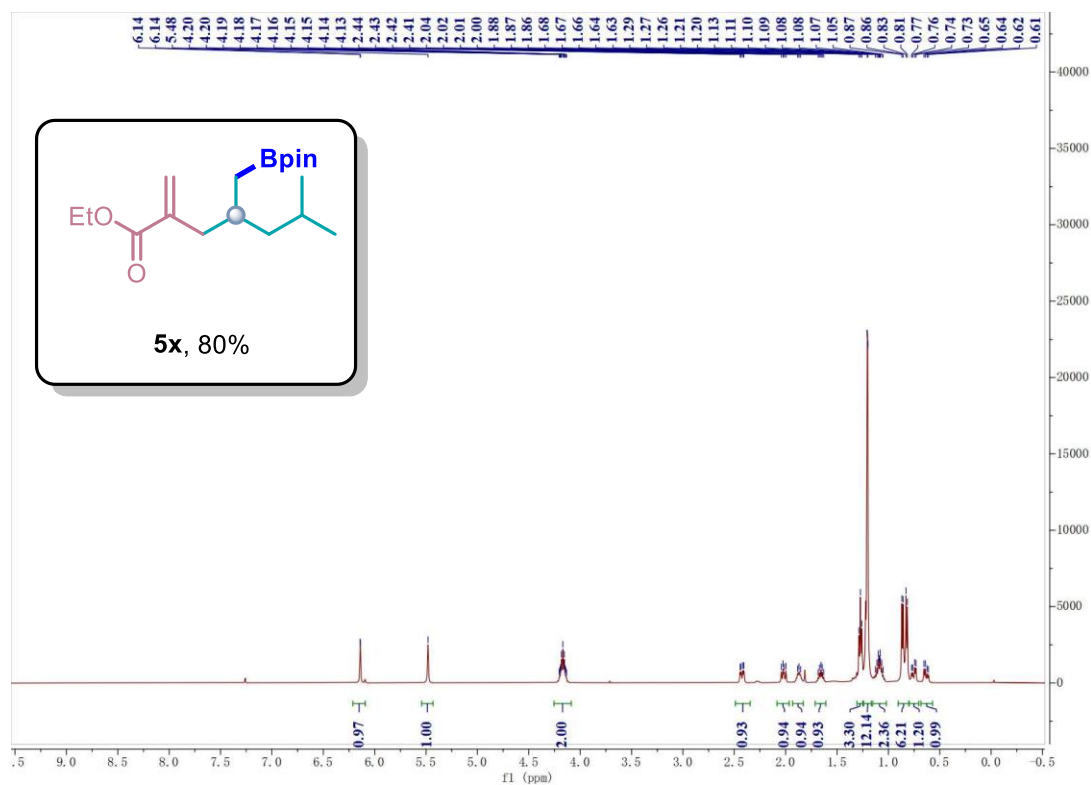
¹³C NMR (126 MHz, Chloroform-*d*)



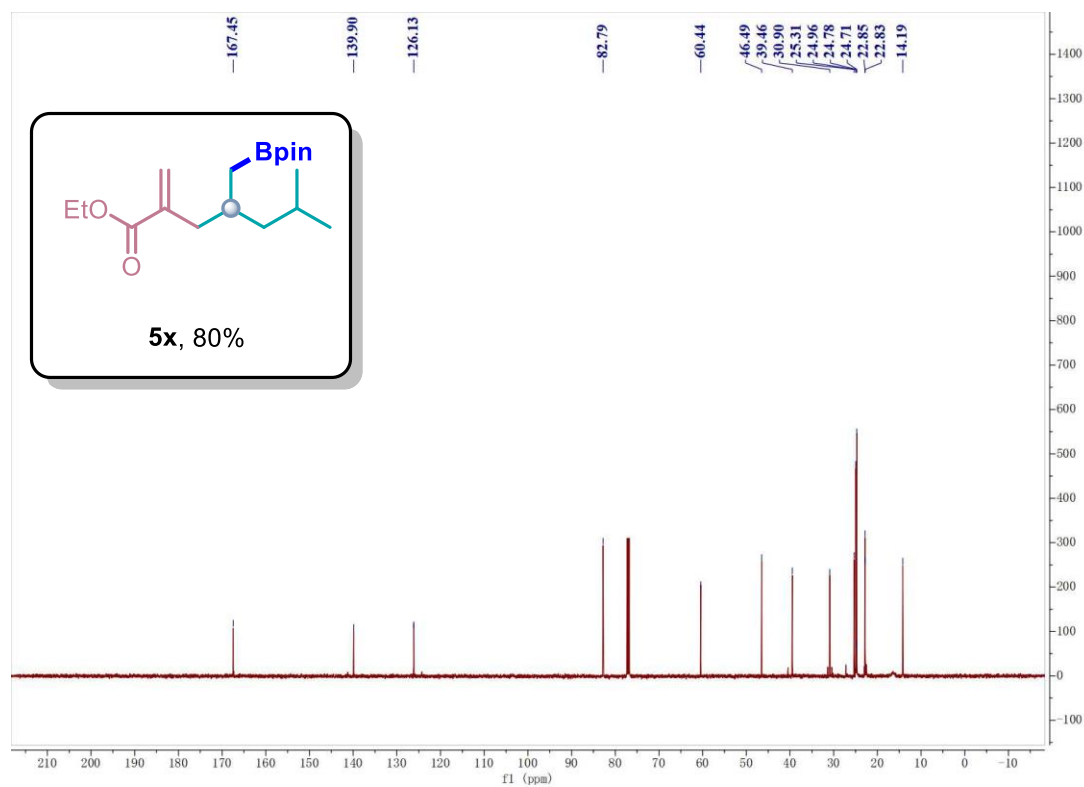
^{11}B NMR (160 MHz, Chloroform-*d*)

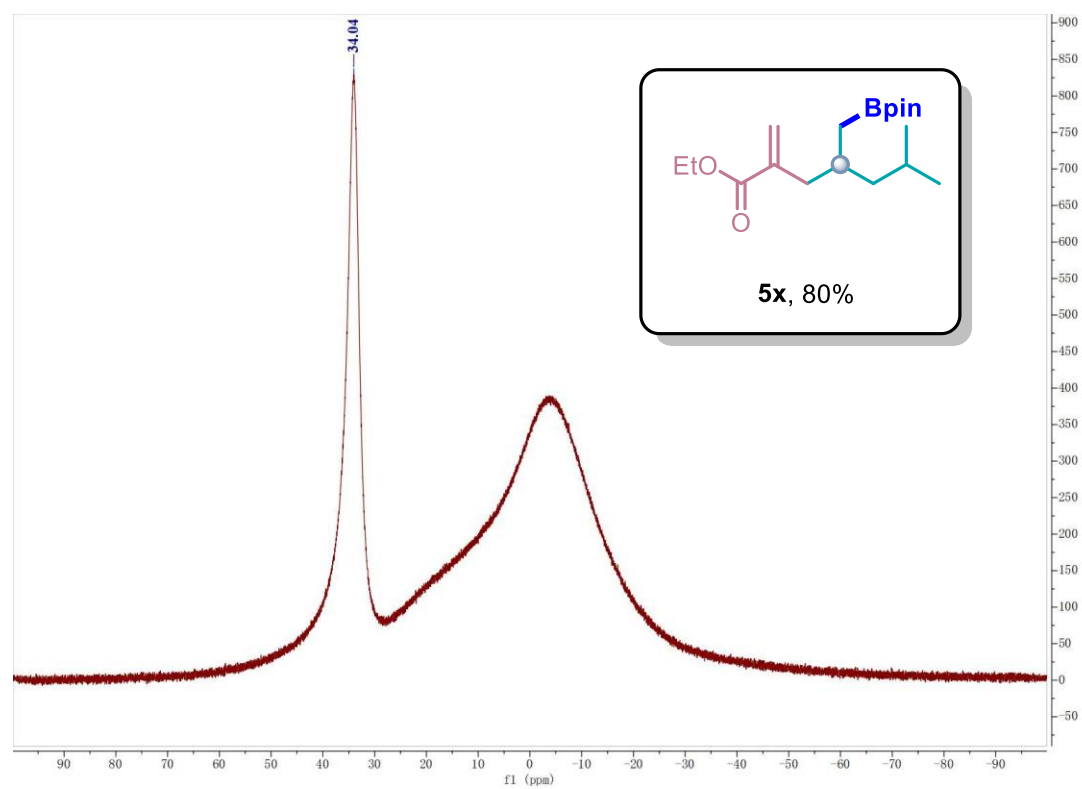


ethyl 6-methyl-2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5x**)
¹H NMR (500 MHz, Chloroform-*d*)



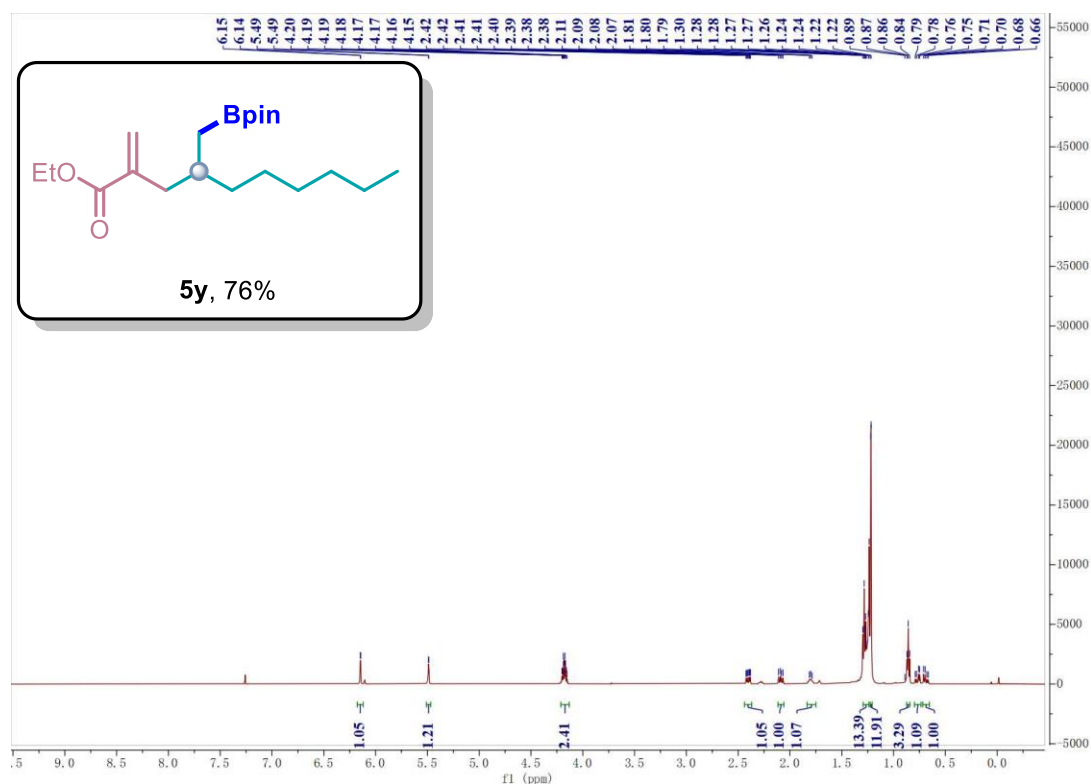
¹³C NMR (126 MHz, Chloroform-*d*)



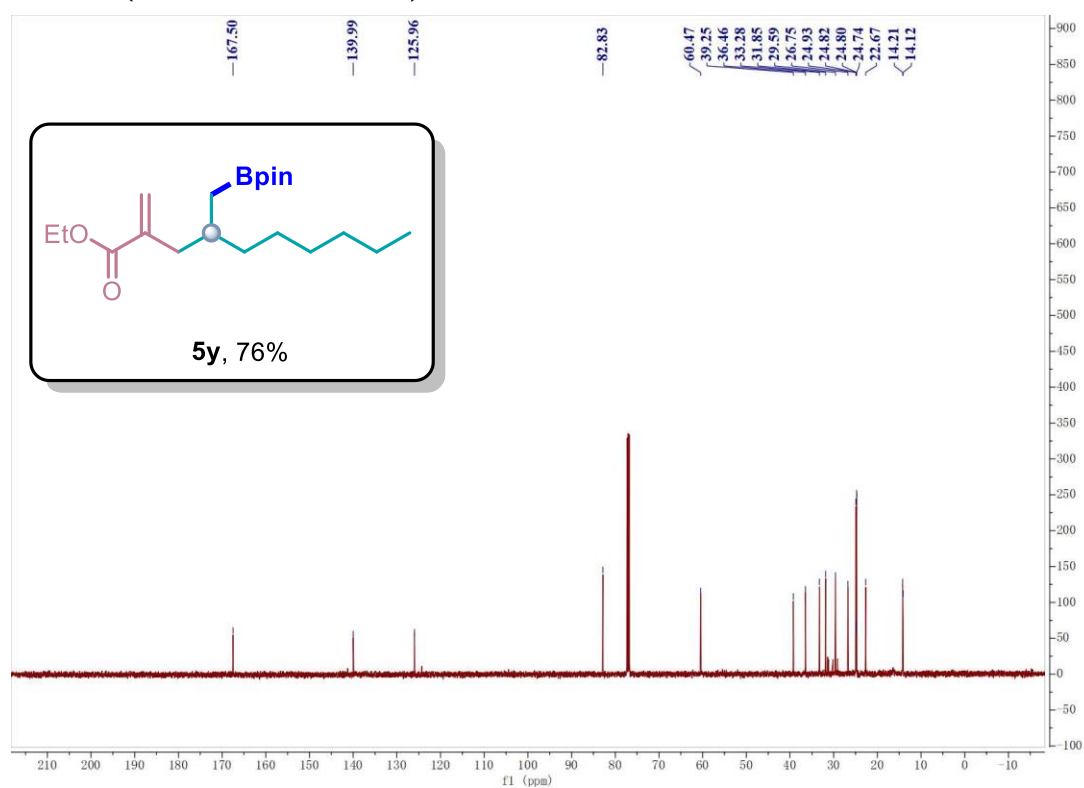
¹¹B NMR (160 MHz, Chloroform-*d*)

ethyl 2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)decanoate (**5y**)

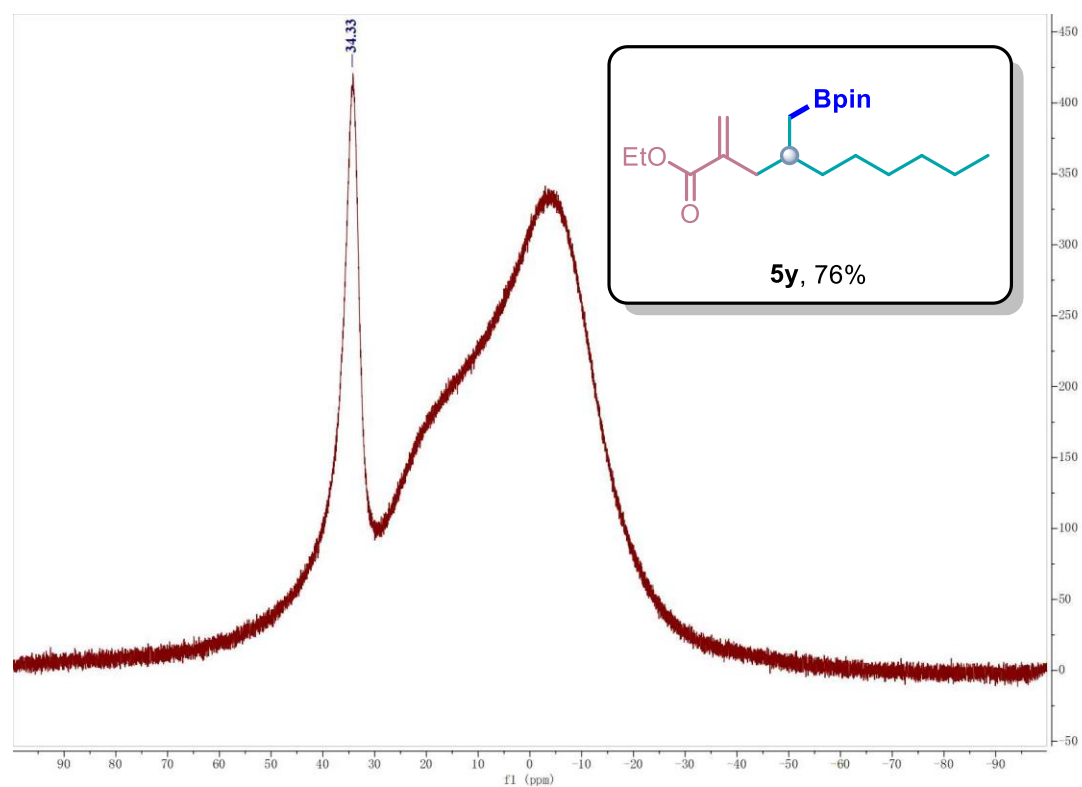
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

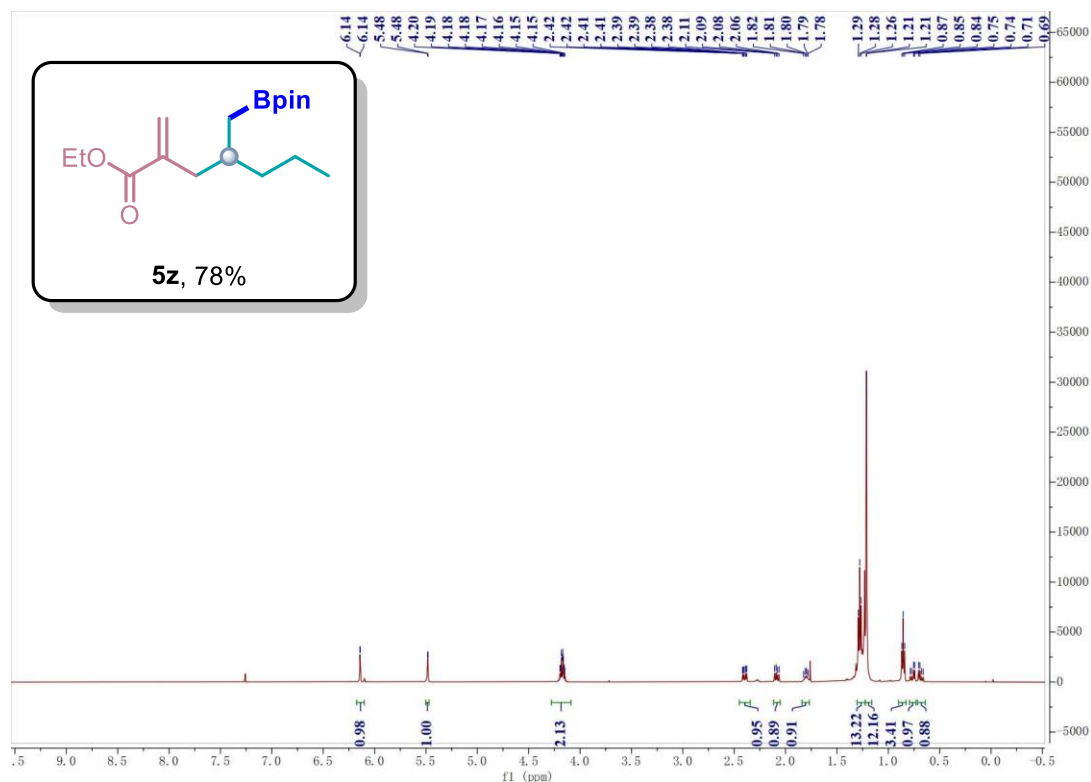


^{11}B NMR (160 MHz, Chloroform-*d*)

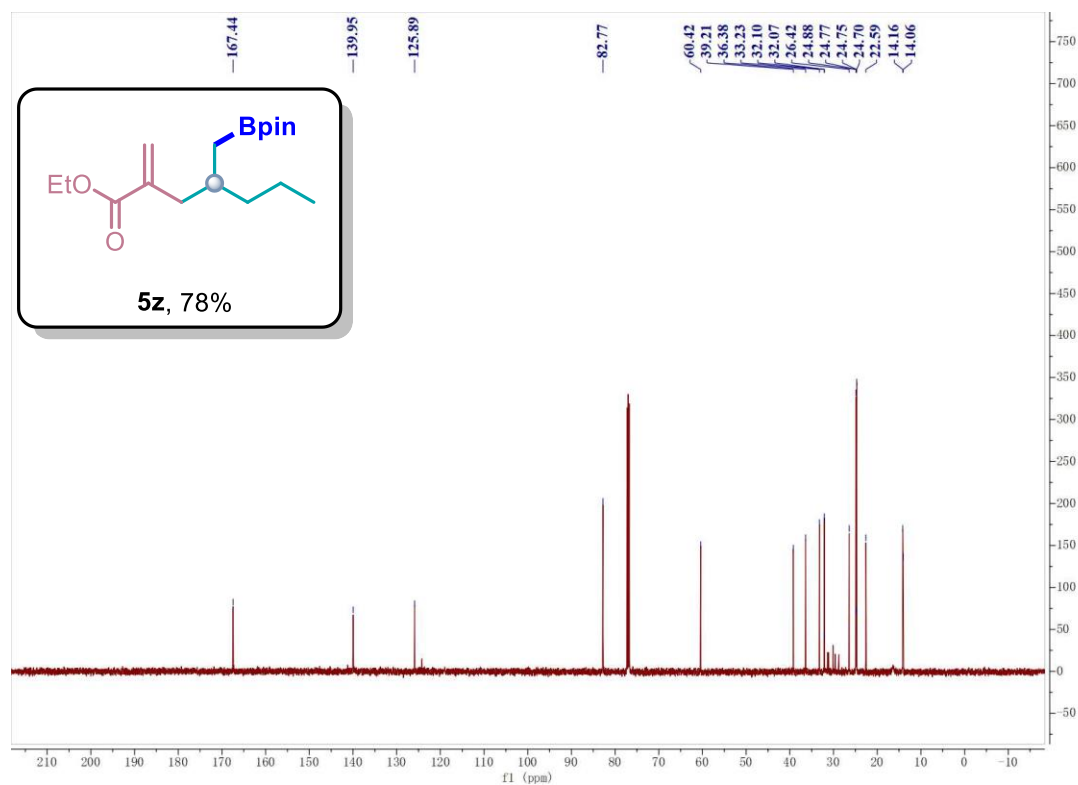


ethyl 2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**5z**)

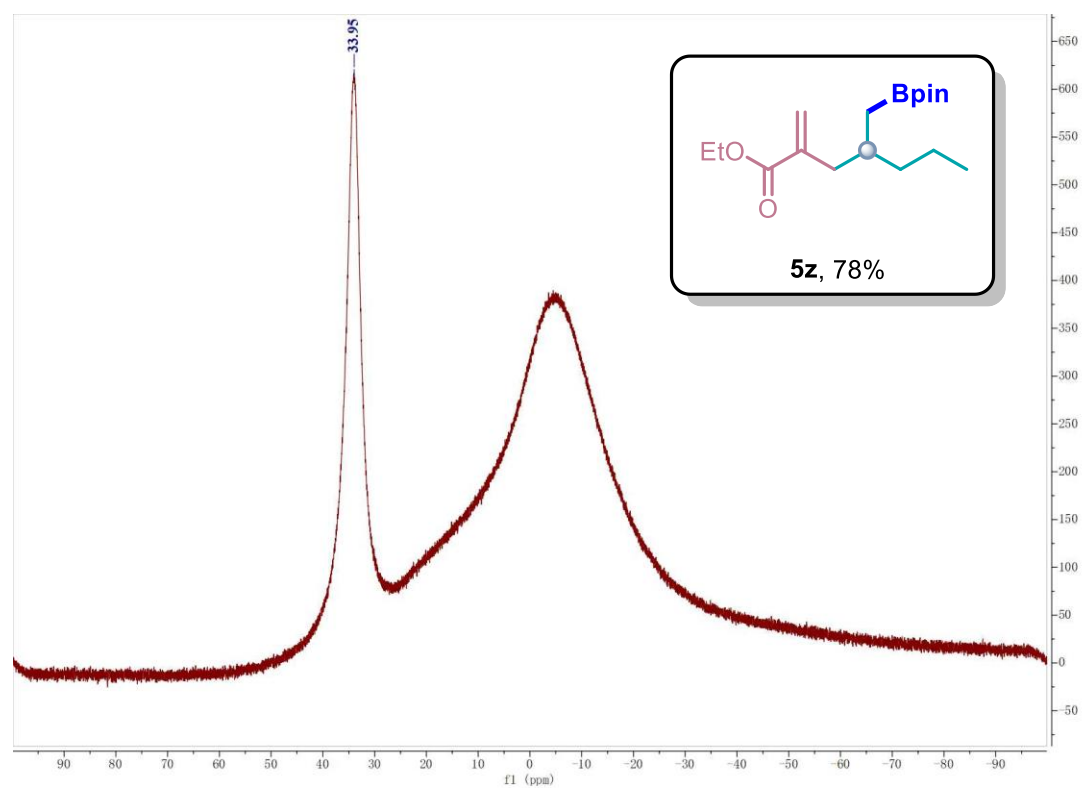
^1H NMR (500 MHz, Chloroform-*d*)



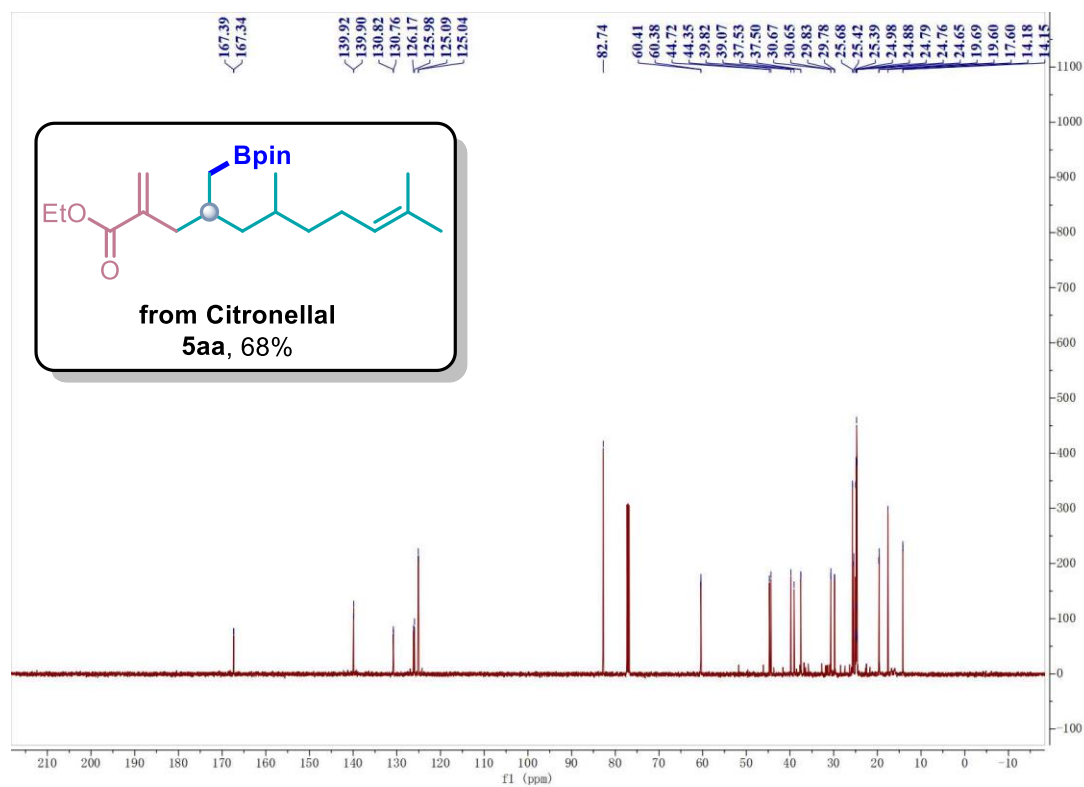
^{13}C NMR (126 MHz, Chloroform-*d*)



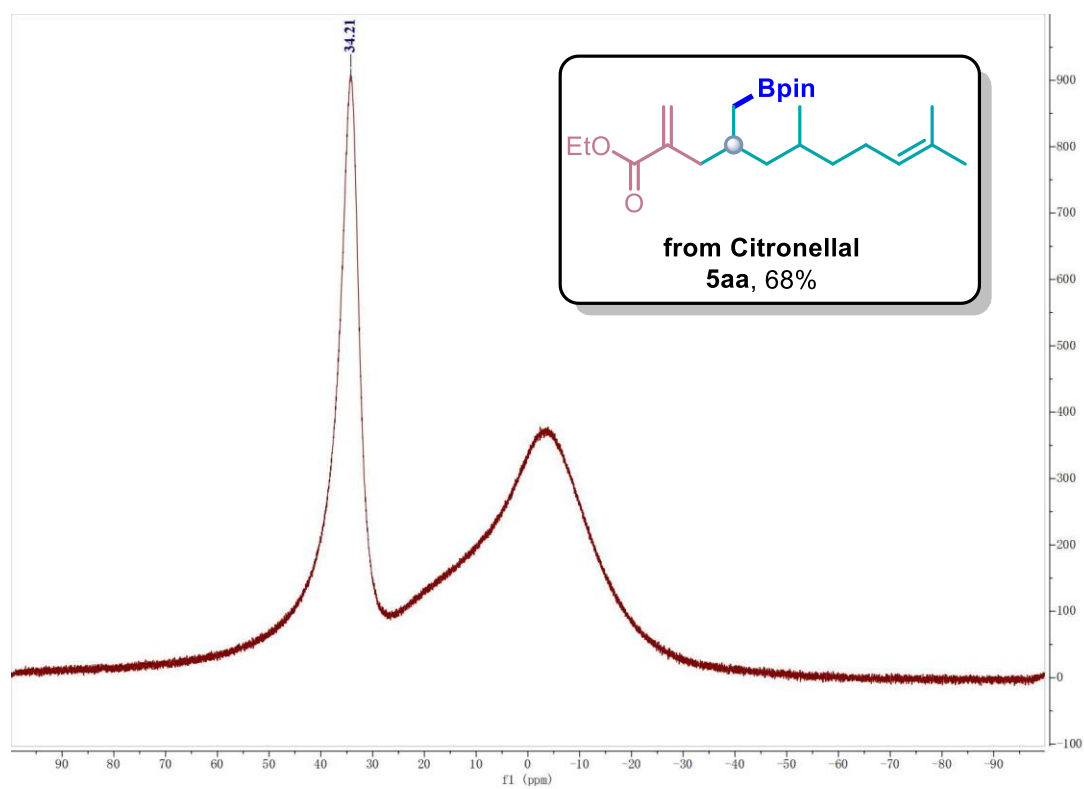
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

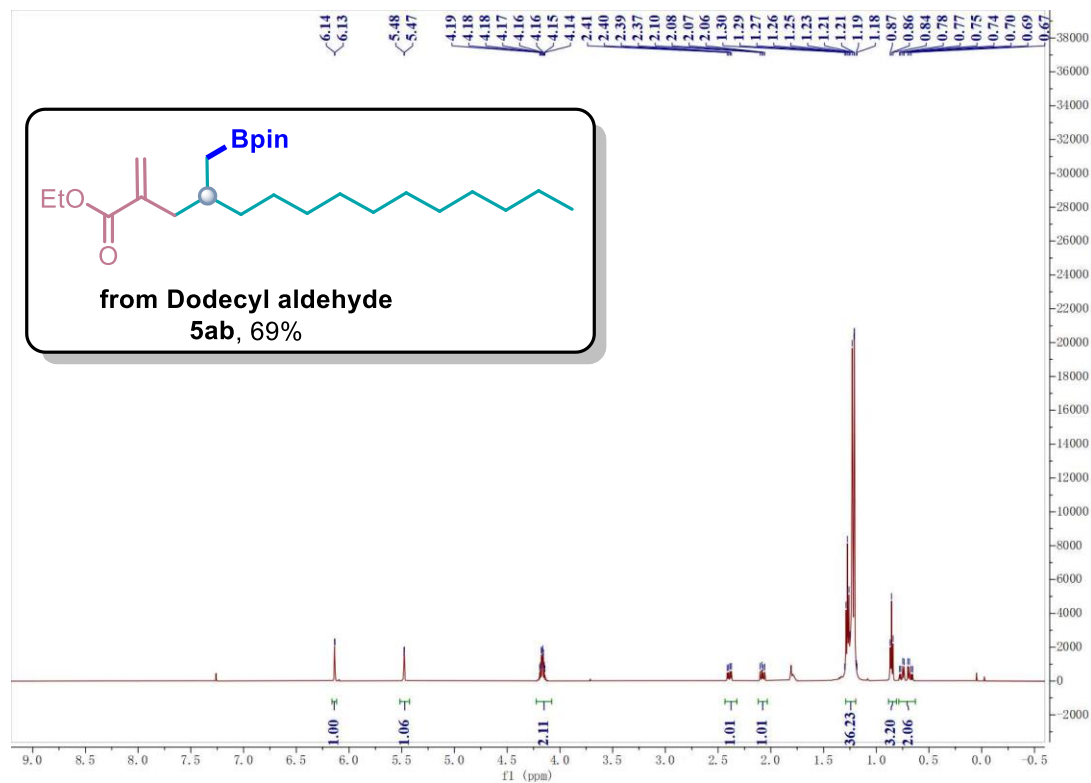


^{11}B NMR (160 MHz, Chloroform-*d*)

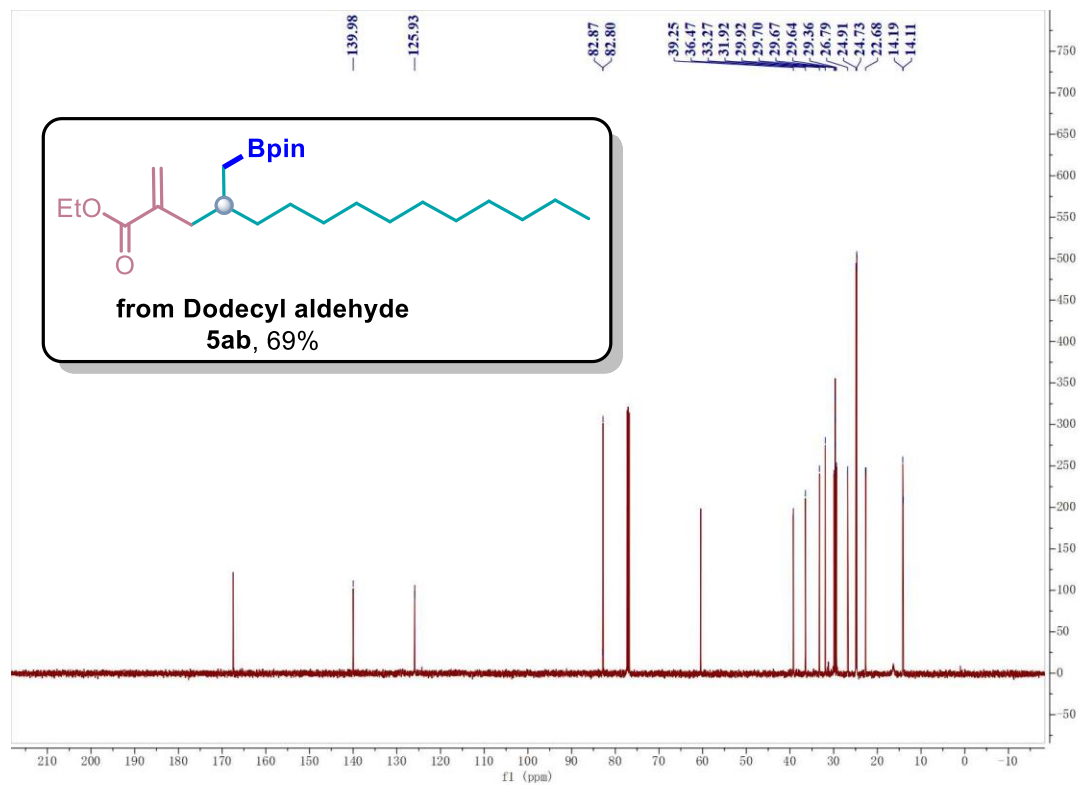


ethyl 2-methylene-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)pentadecanoate
(5ab)

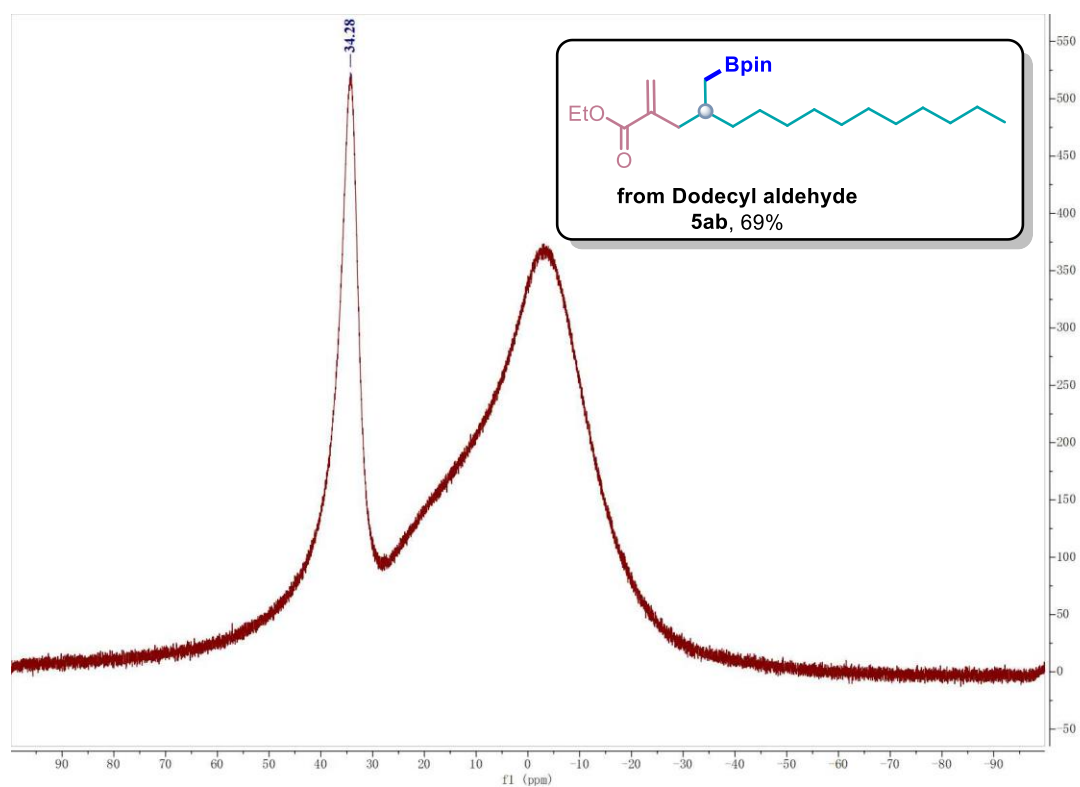
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

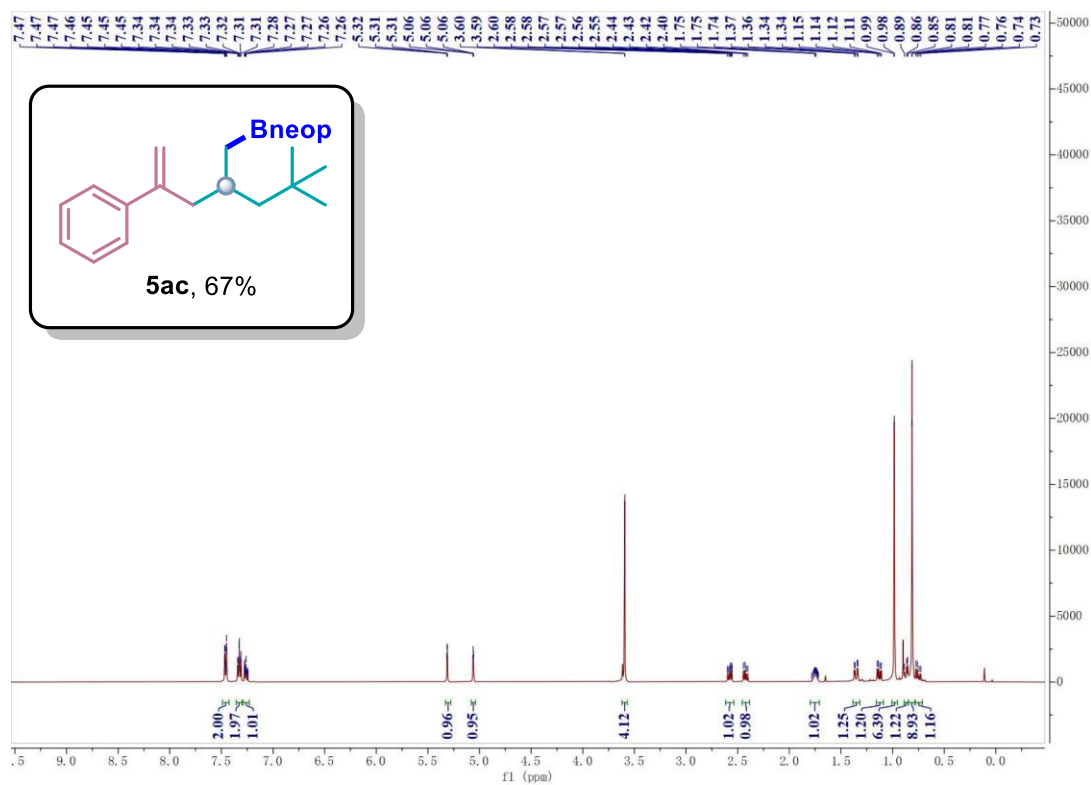


^{11}B NMR (160 MHz, Chloroform-*d*)

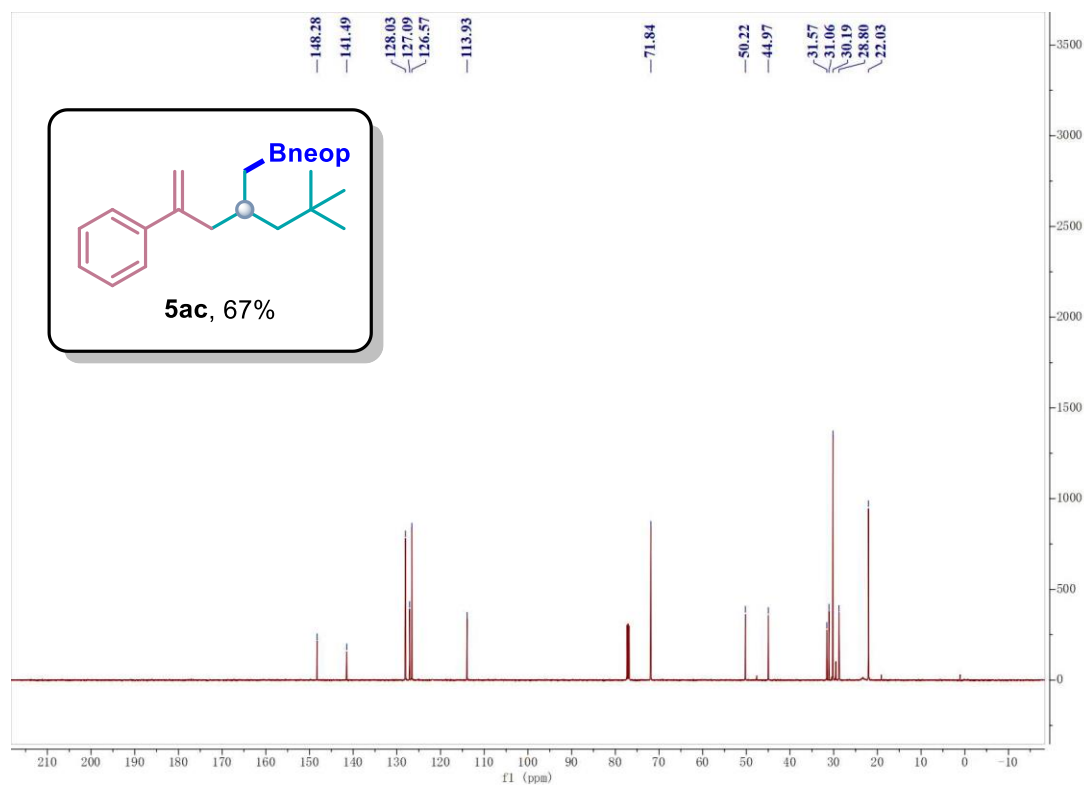


5,5-dimethyl-2-(2-neopentyl-4-phenylpent-4-en-1-yl)-1,3,2-dioxaborinane (5ac)

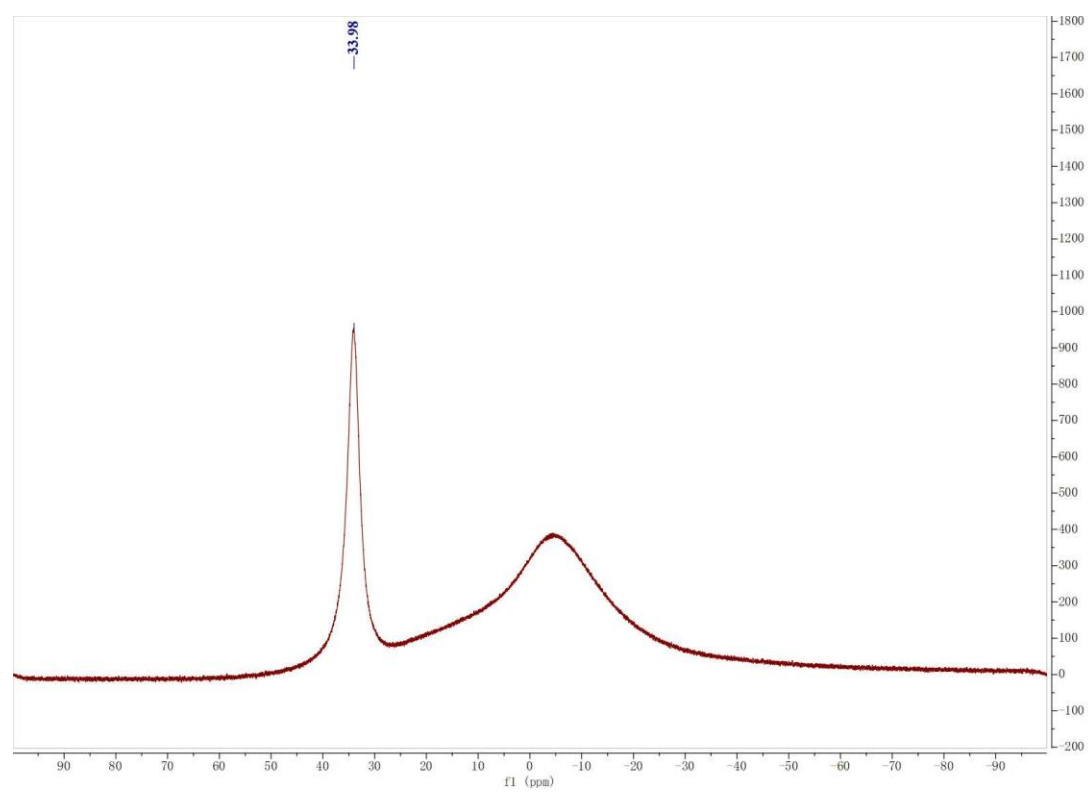
¹H NMR (500 MHz, Chloroform-*d*)



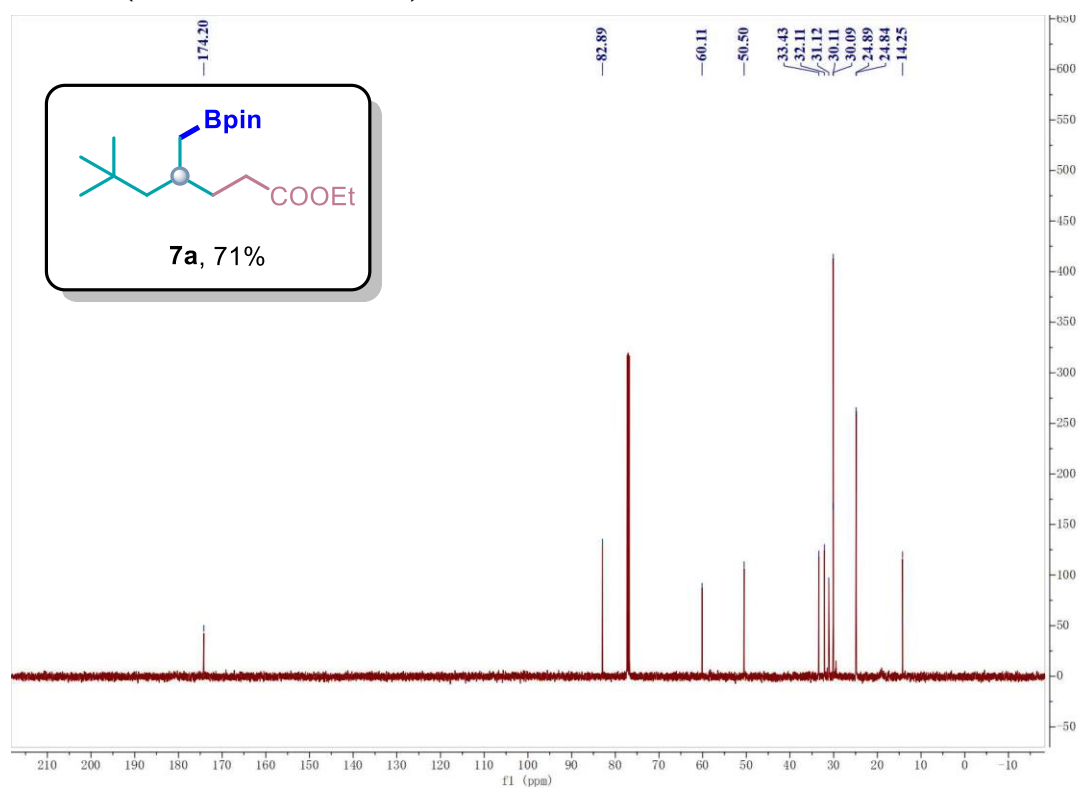
¹³C NMR (126 MHz, Chloroform-*d*)



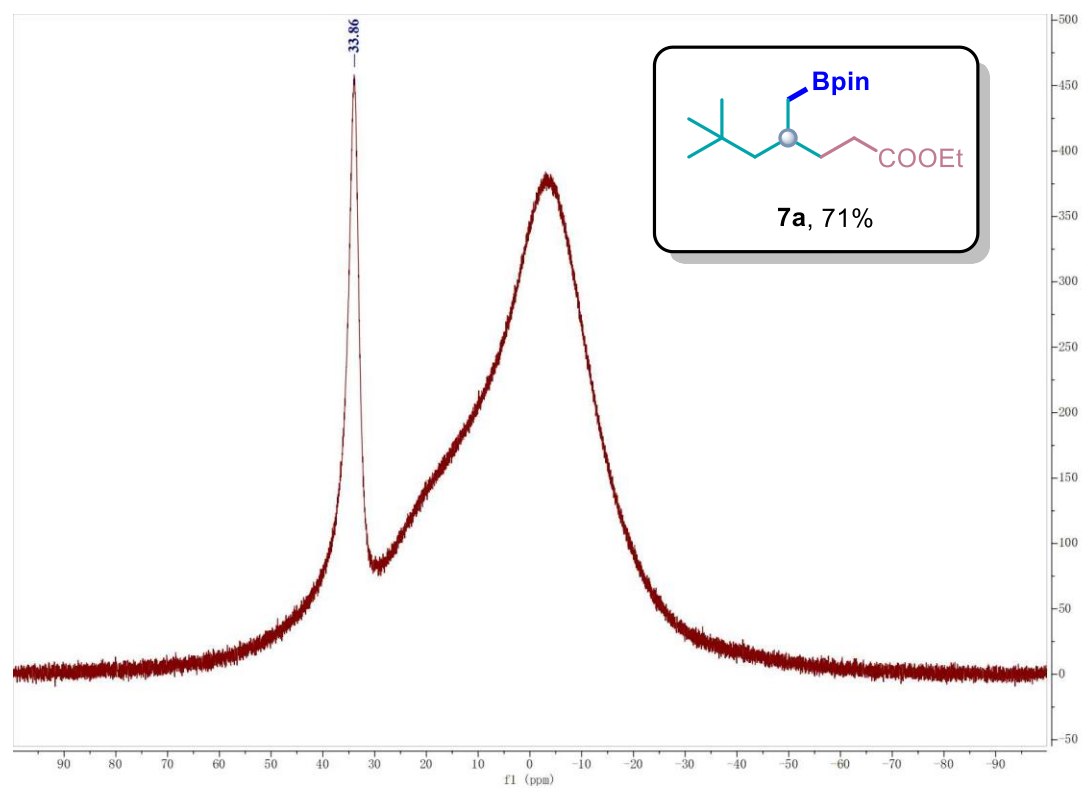
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

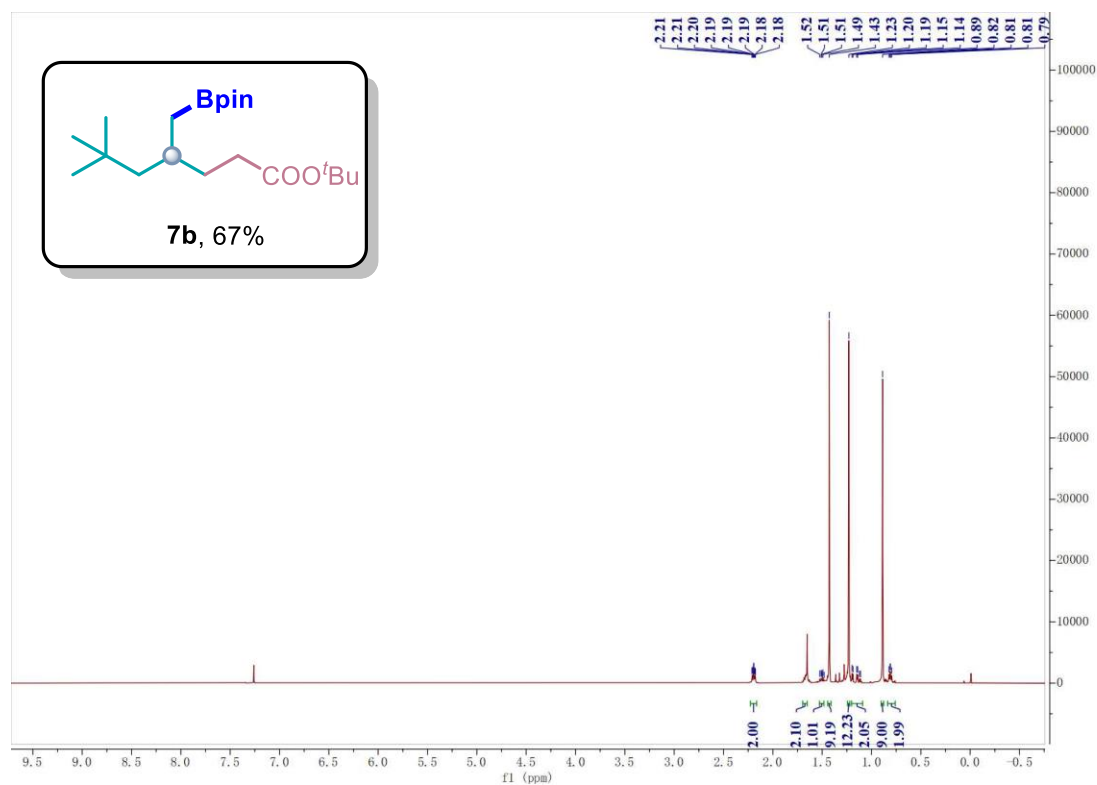


^{11}B NMR (160 MHz, Chloroform-*d*)

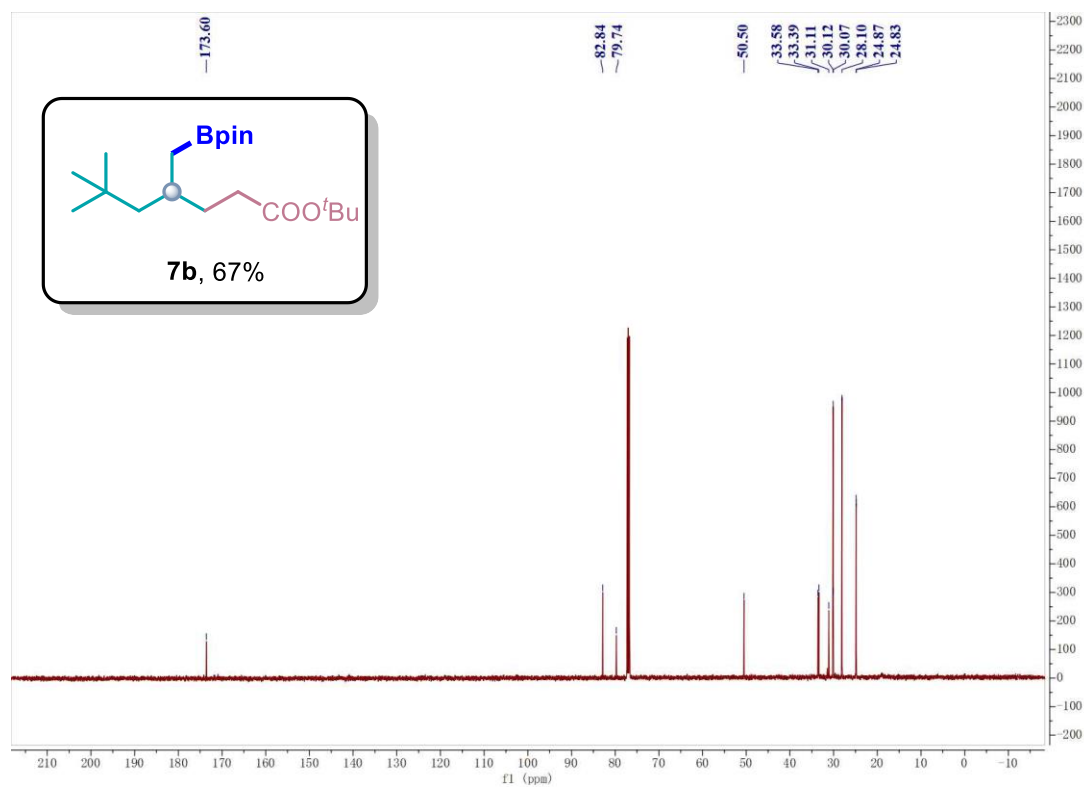


tert-butyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate
(7b)

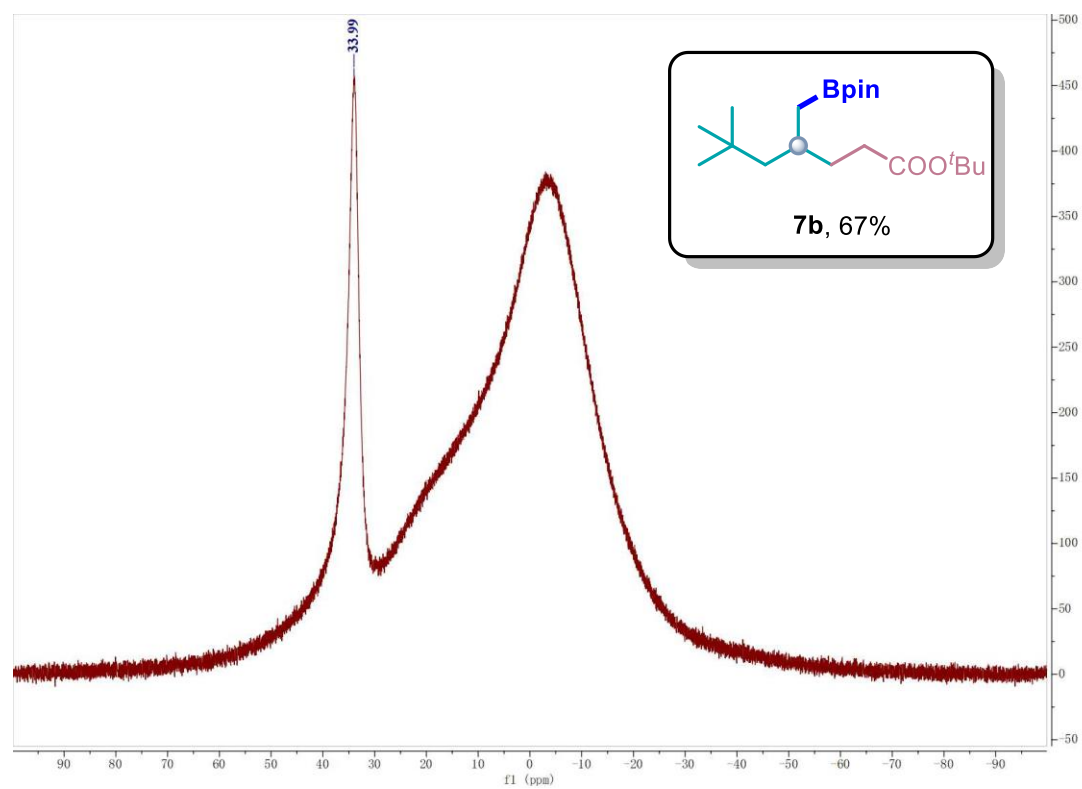
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

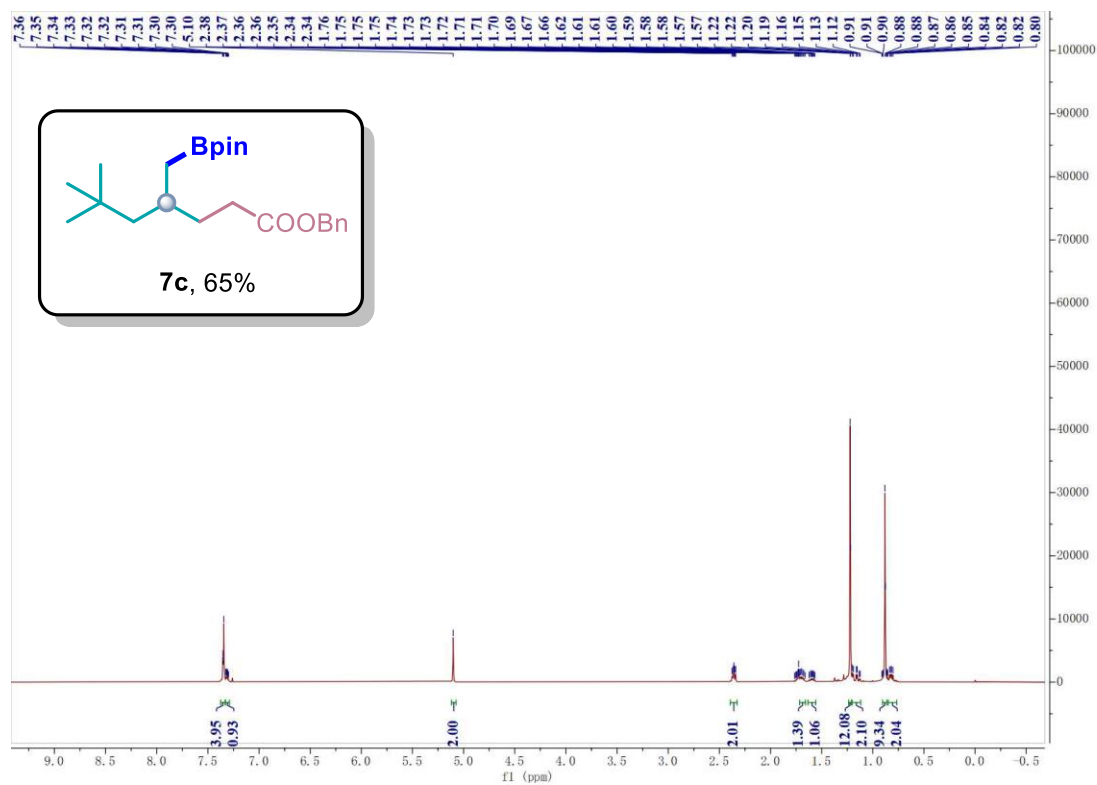


^{11}B NMR (160 MHz, Chloroform-*d*)

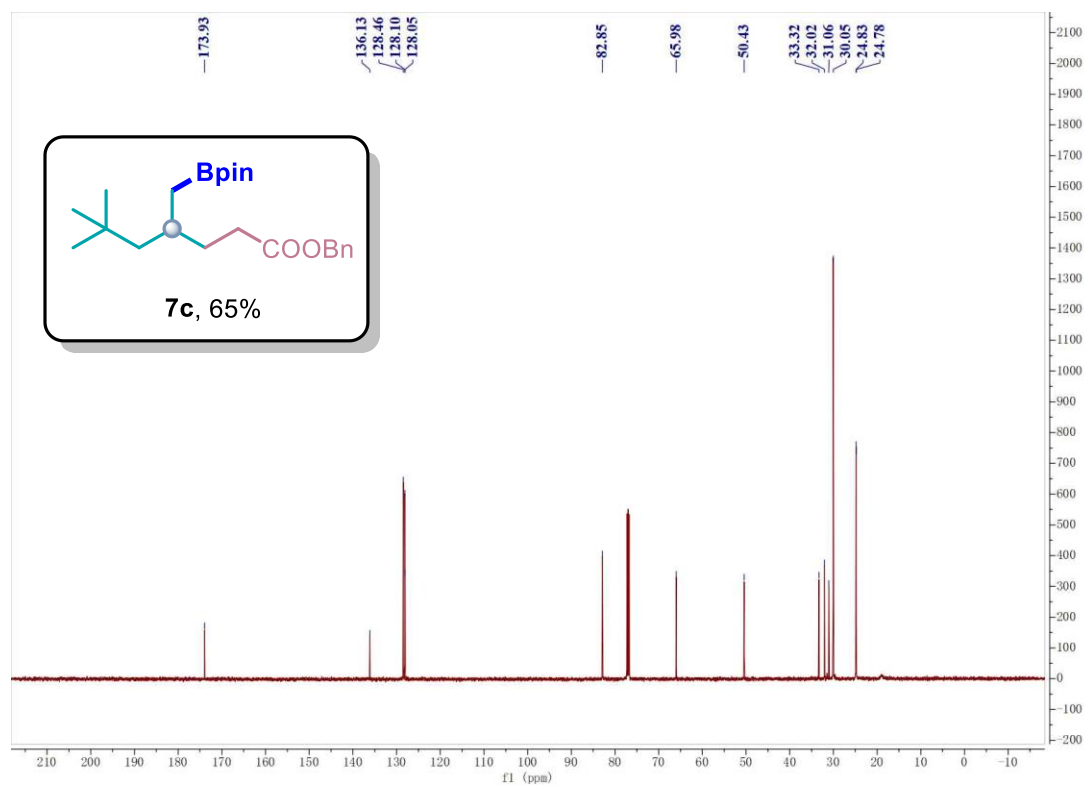


benzyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**7c**)

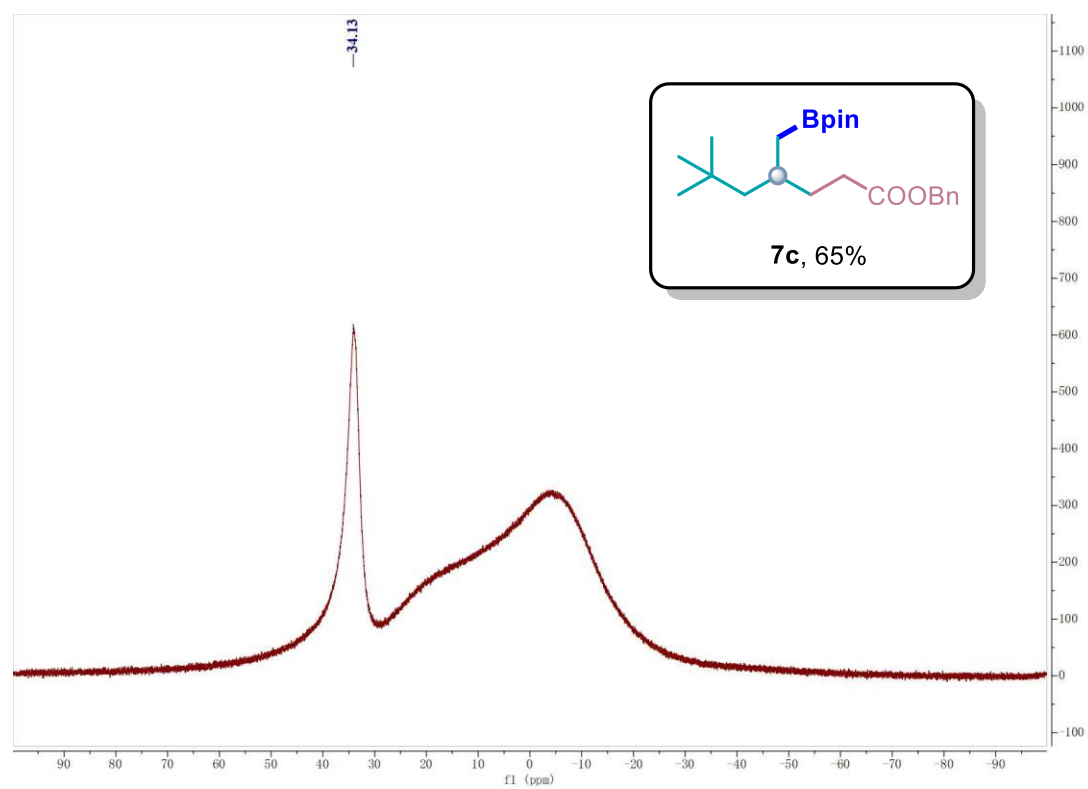
^1H NMR (500 MHz, Chloroform-*d*)



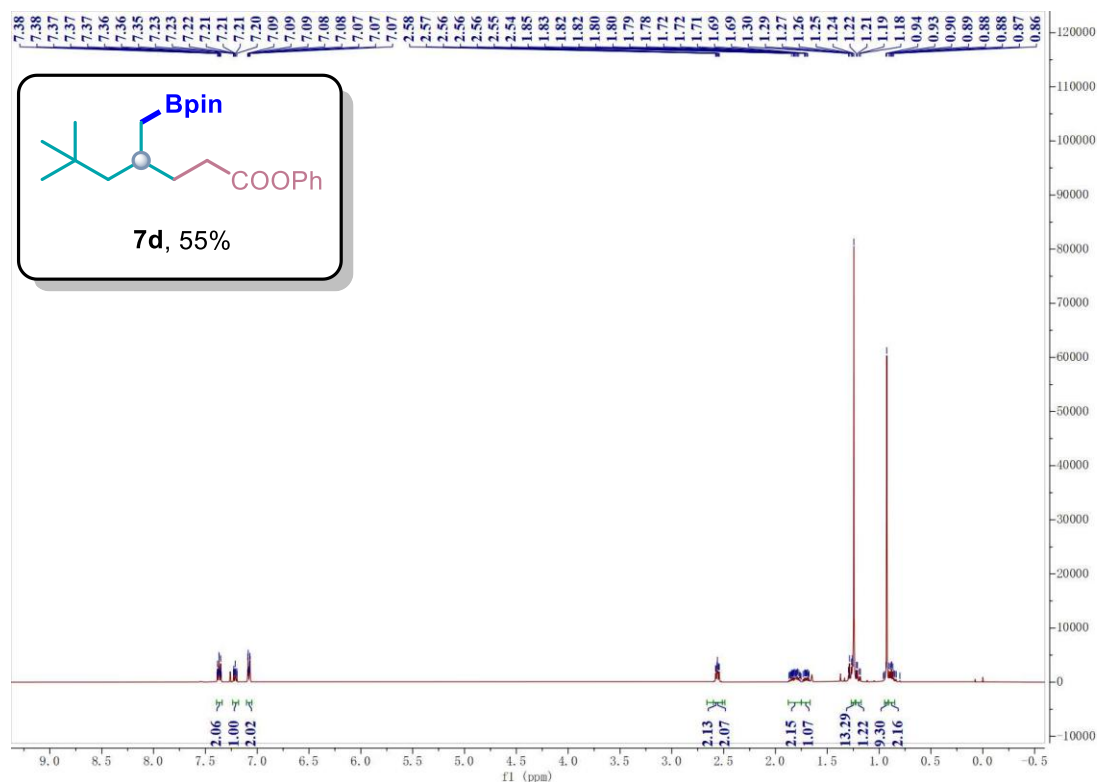
^{13}C NMR (126 MHz, Chloroform-*d*)



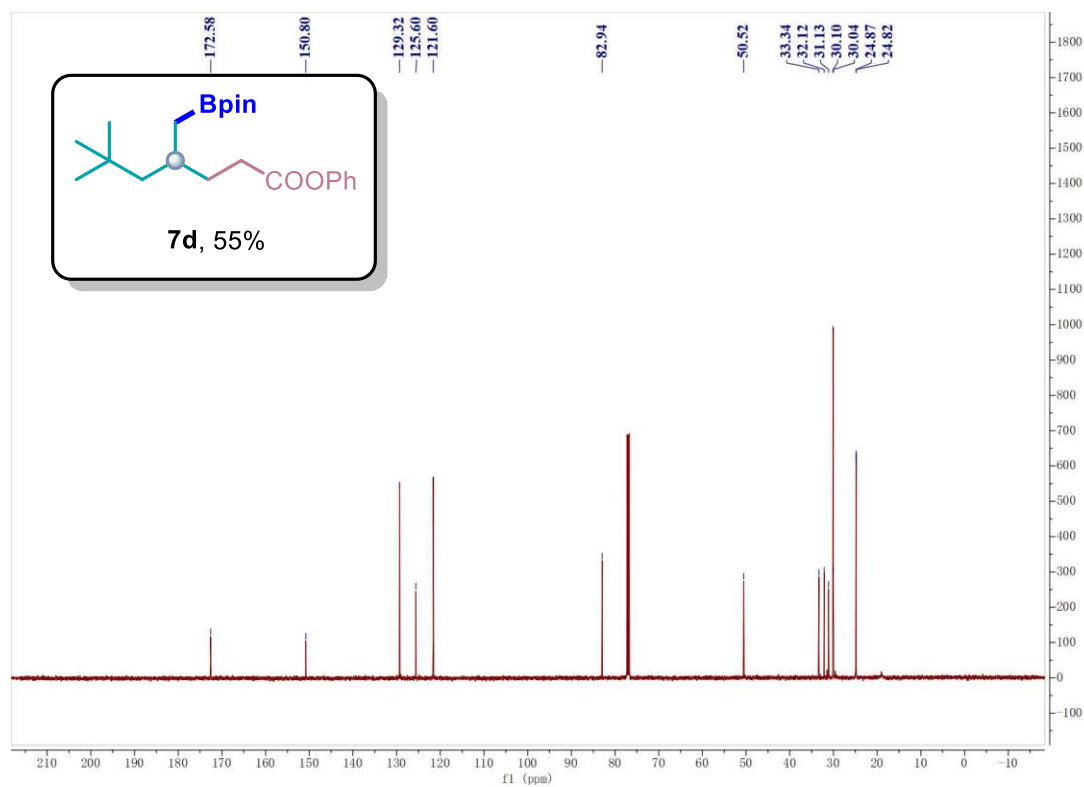
^{11}B NMR (160 MHz, Chloroform-*d*)



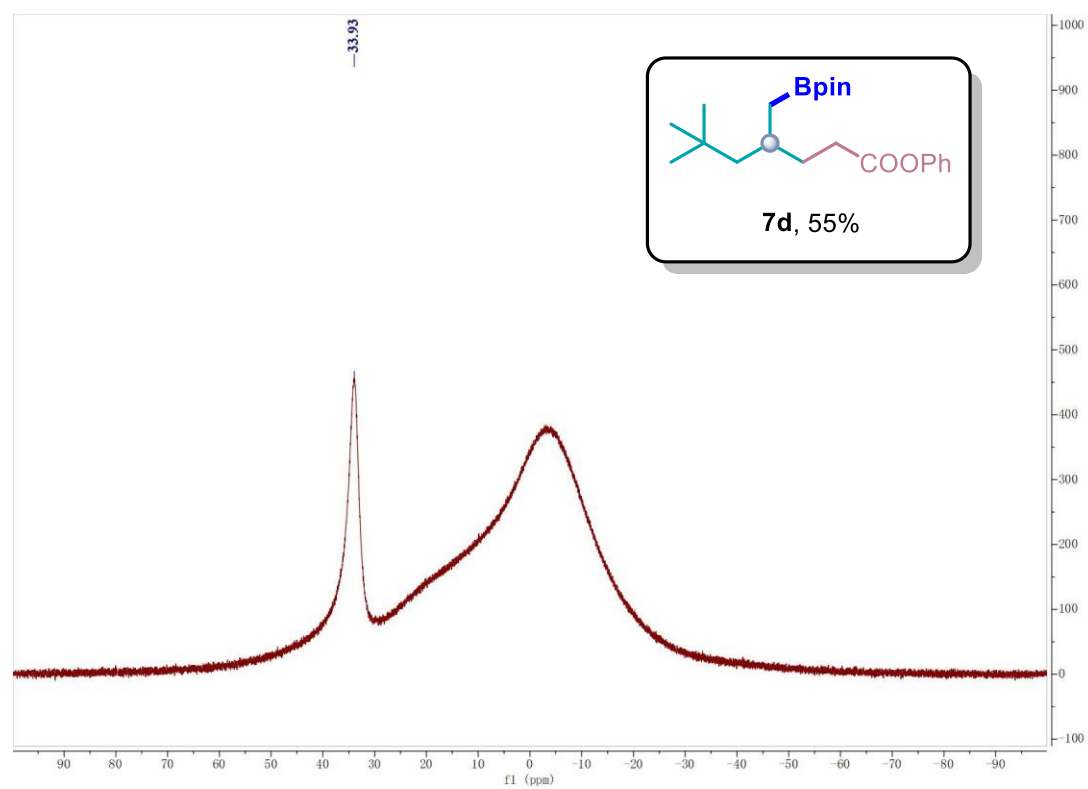
phenyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7d)
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

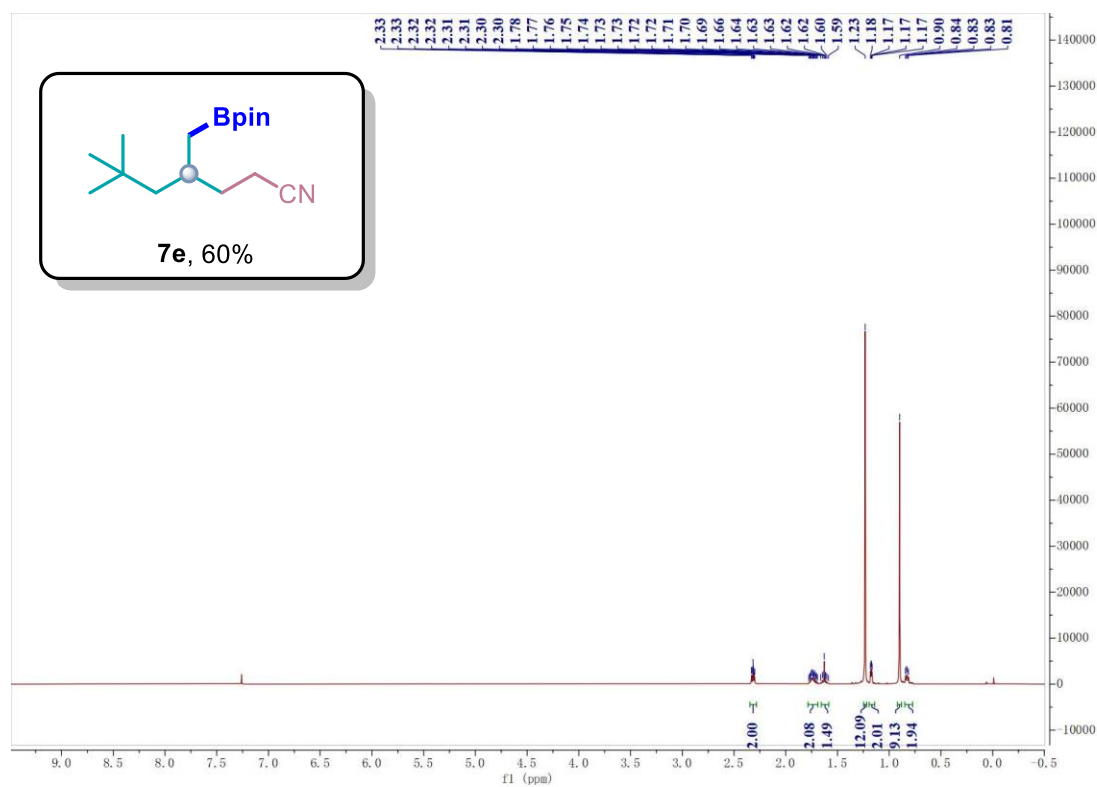


^{11}B NMR (160 MHz, Chloroform-*d*)

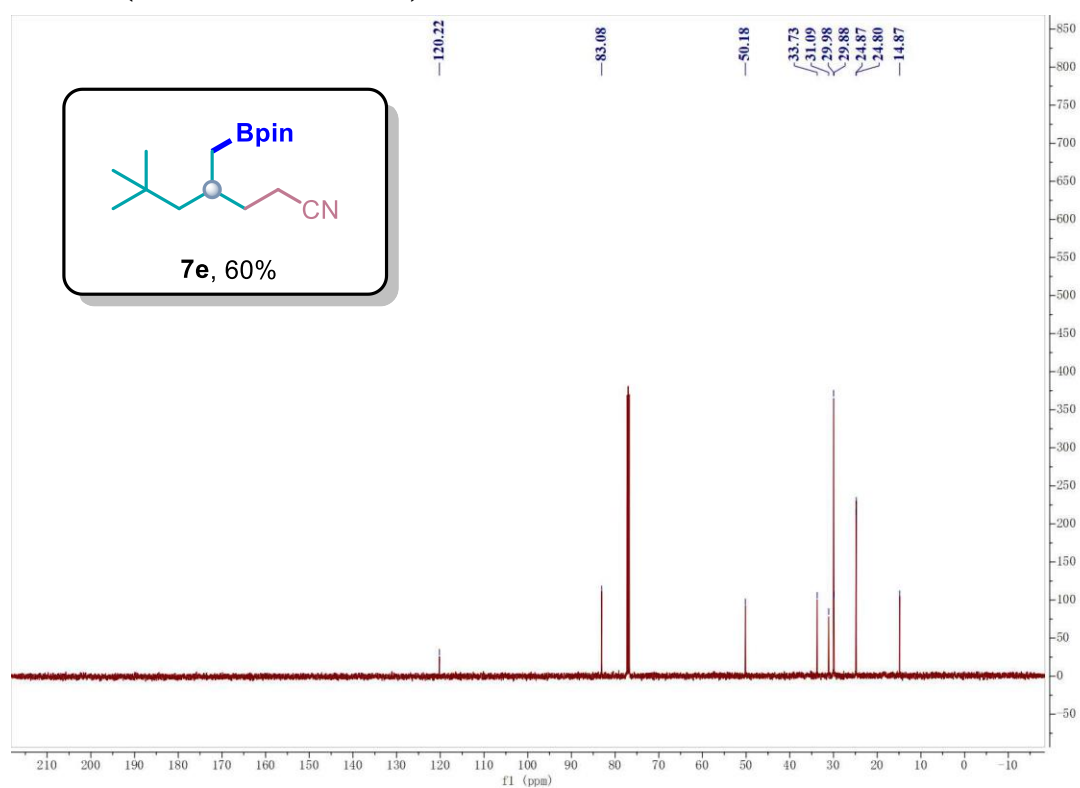


6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanenitrile (**7e**)

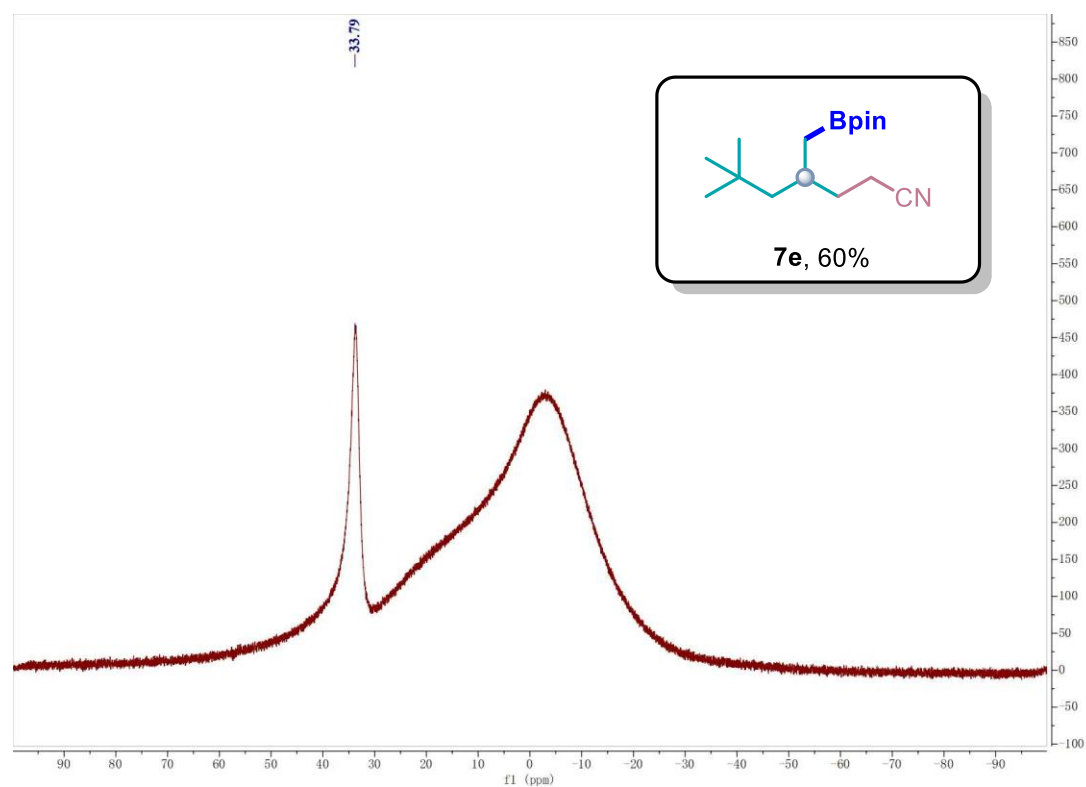
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

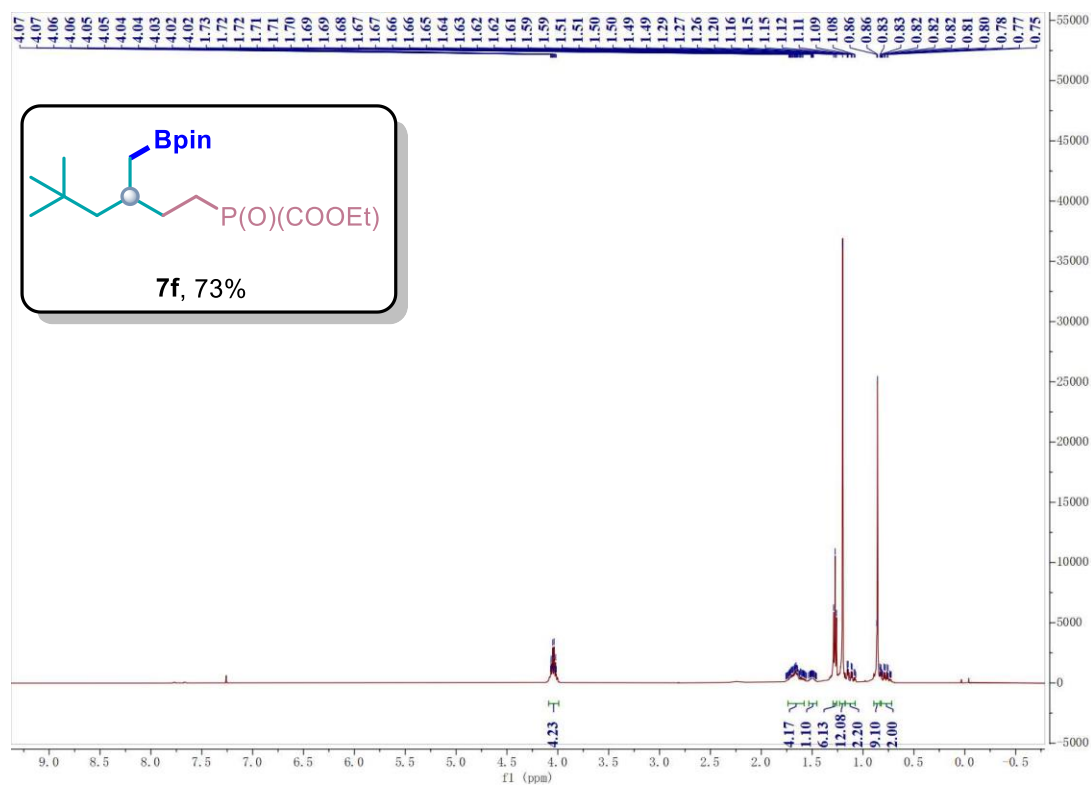


^{11}B NMR (160 MHz, Chloroform-*d*)

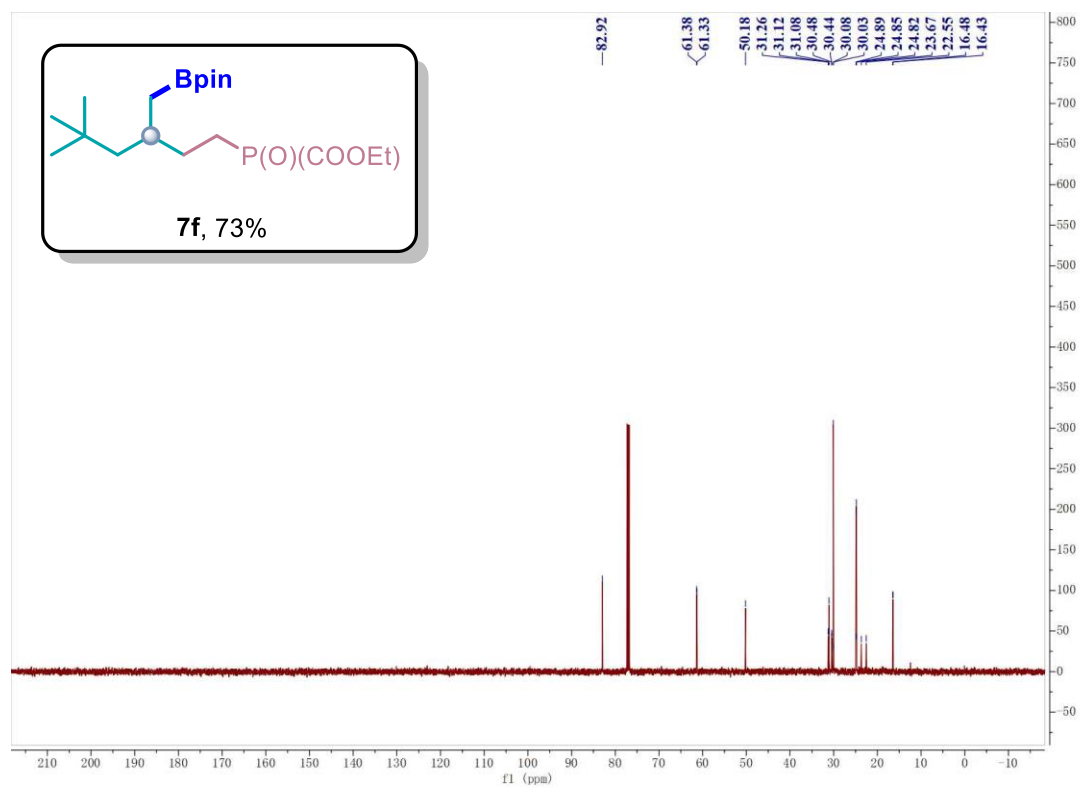


ethyl (5,5-dimethyl-3-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexyl)(oxo)-14-phosphanecarboxylate (**7f**)

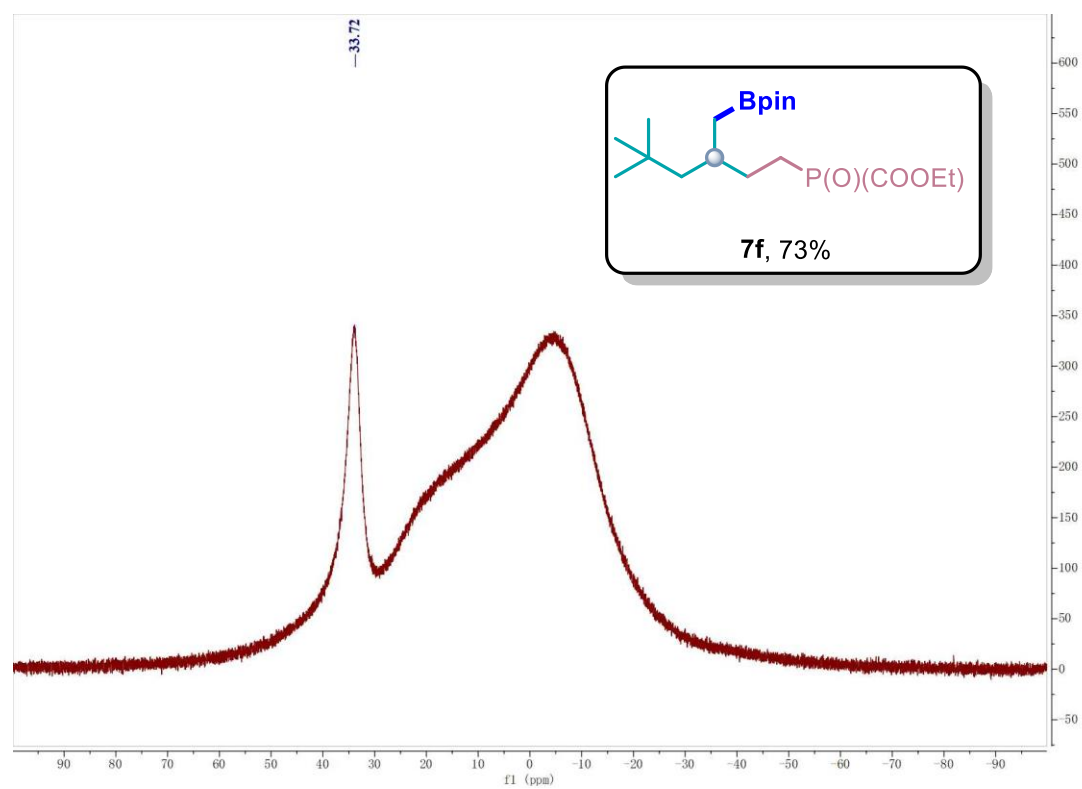
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

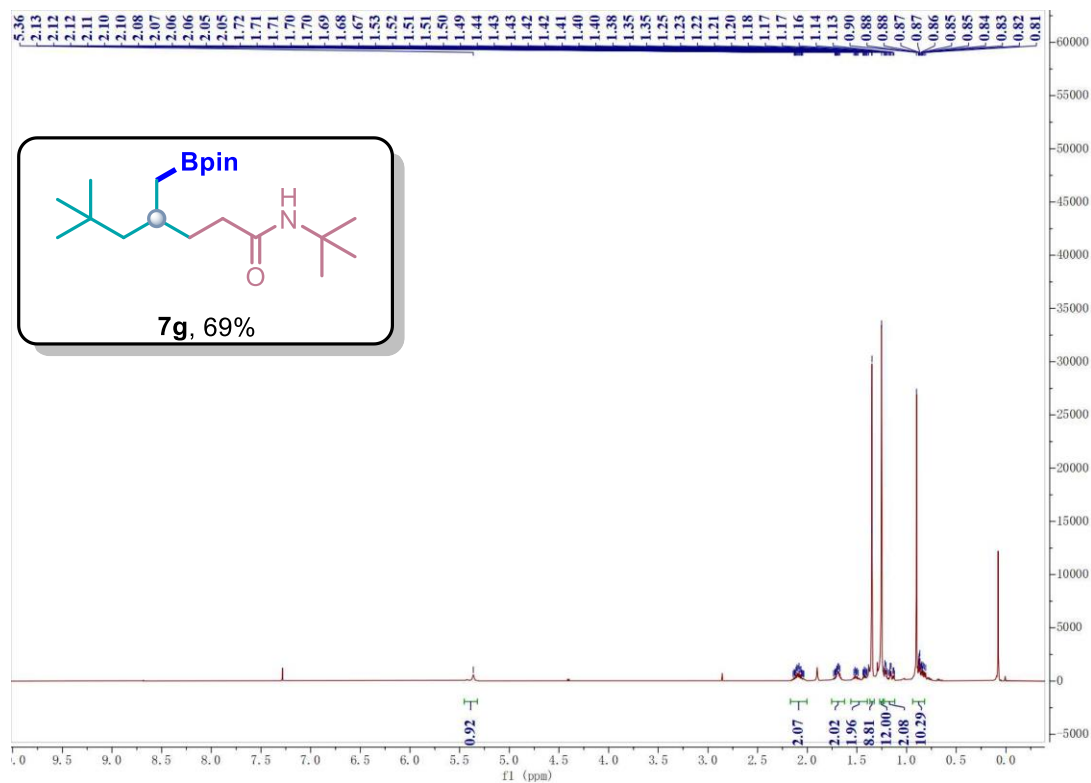


^{11}B NMR (160 MHz, Chloroform-*d*)

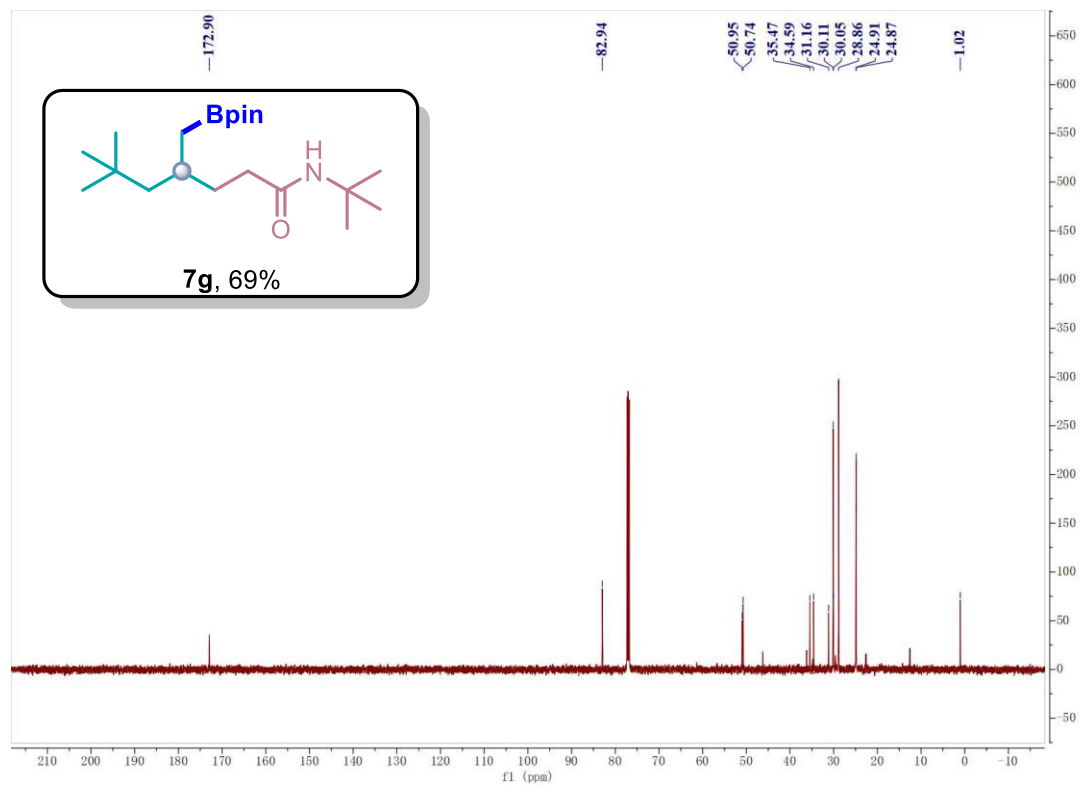


N-(tert-butyl)-6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanamide (7g)

¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

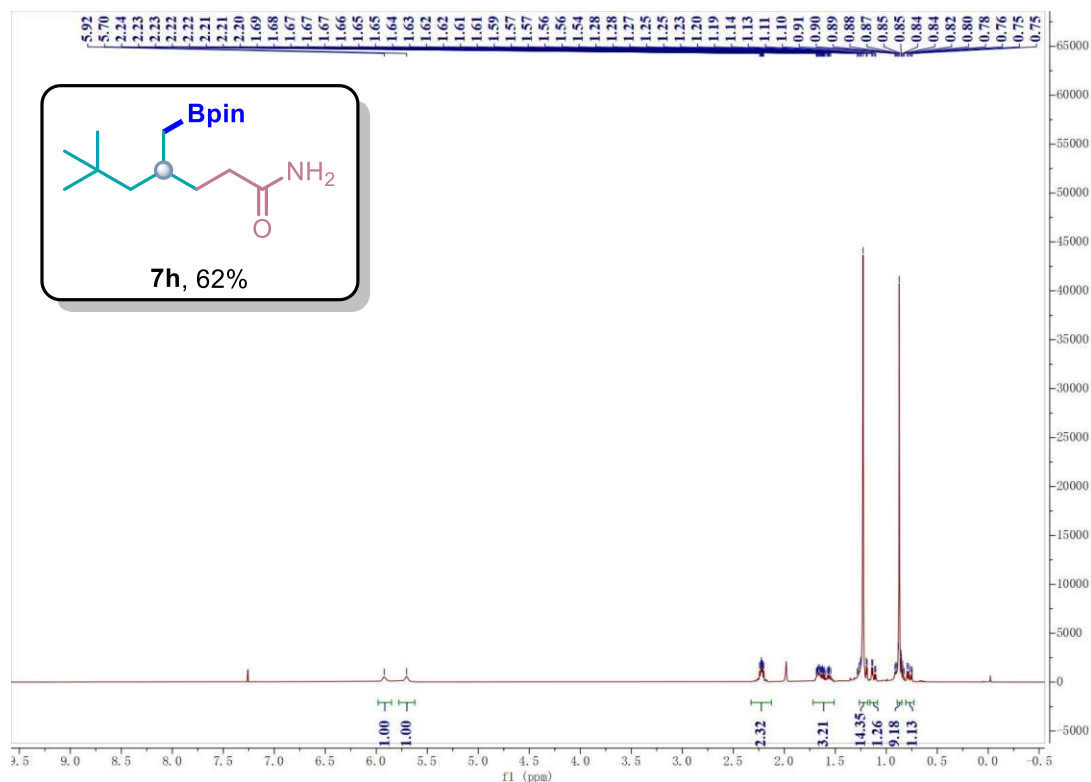


3.412

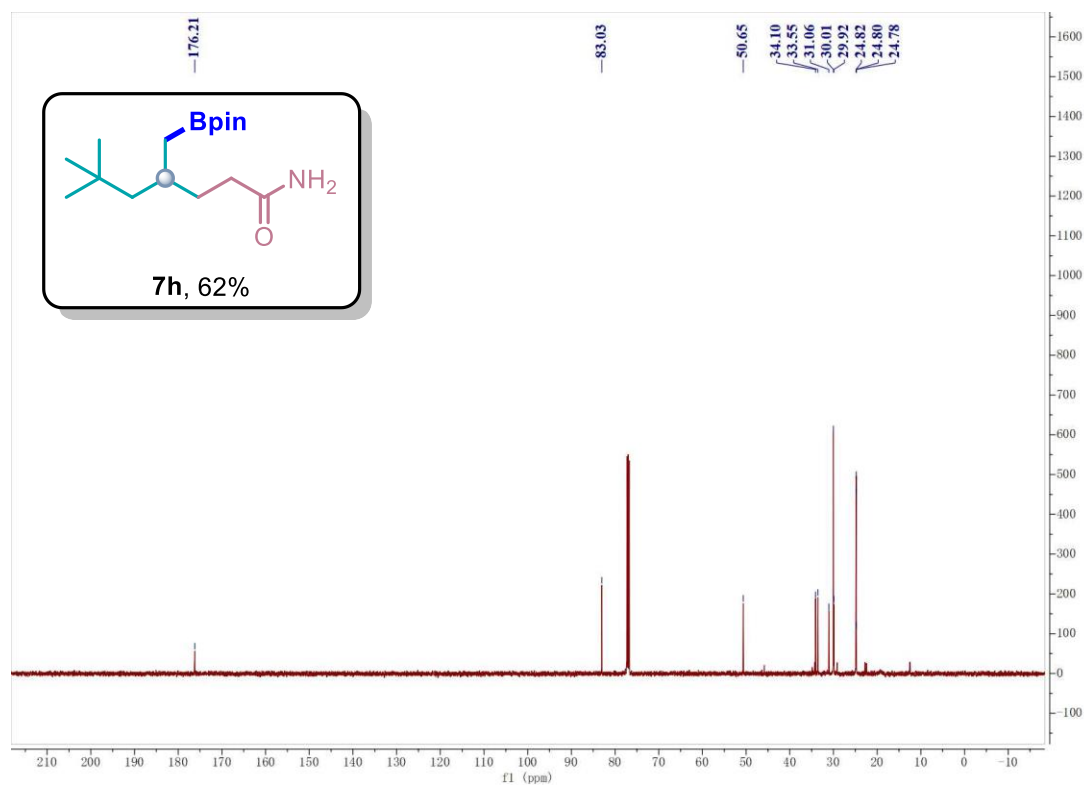
7g, 69%

6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanamide (7h)

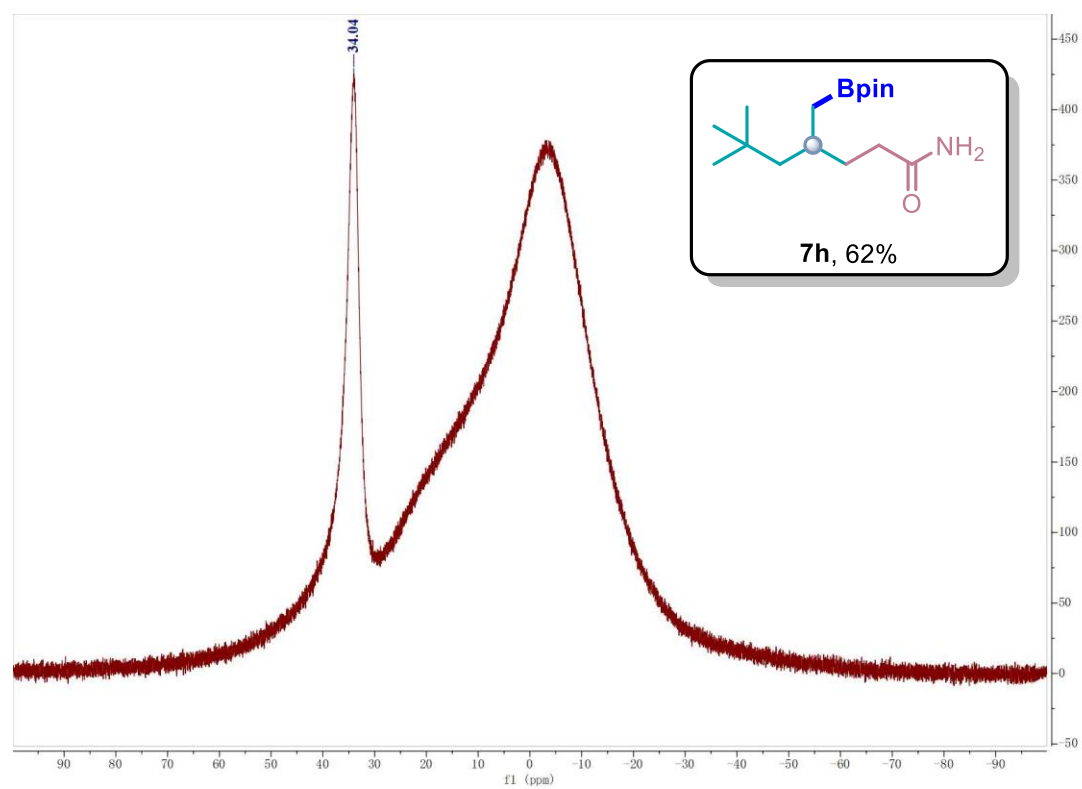
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

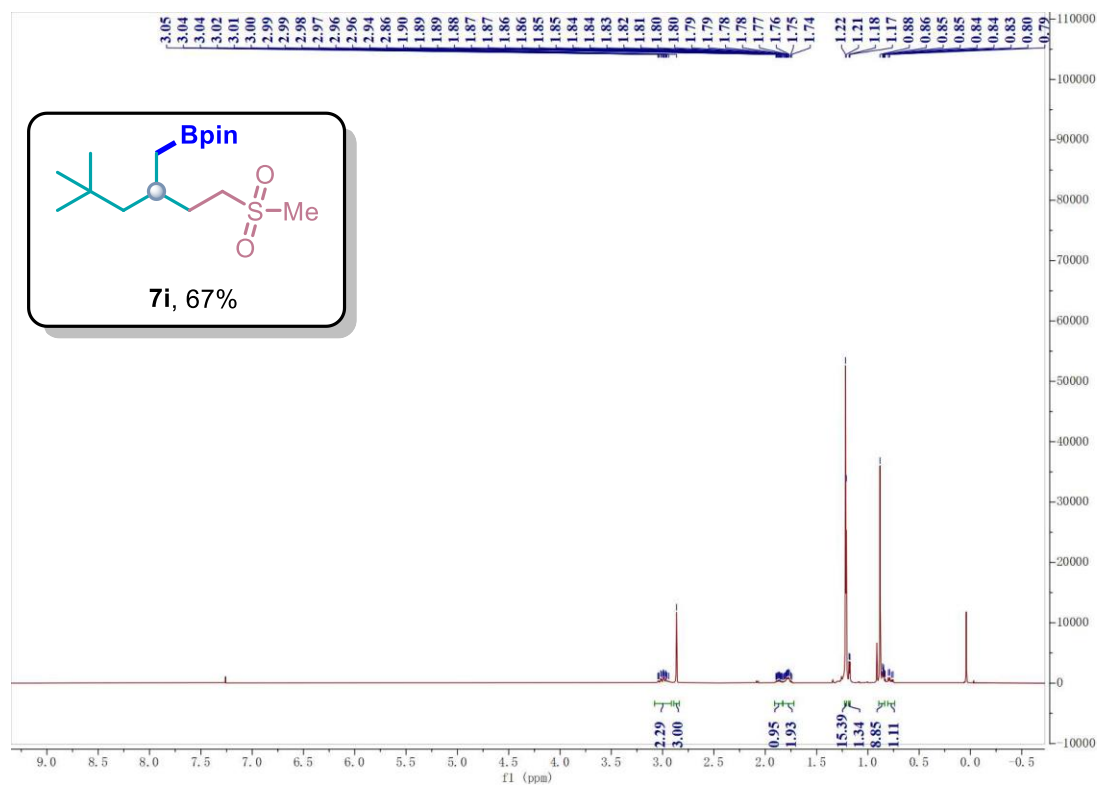


^{11}B NMR (160 MHz, Chloroform-*d*)

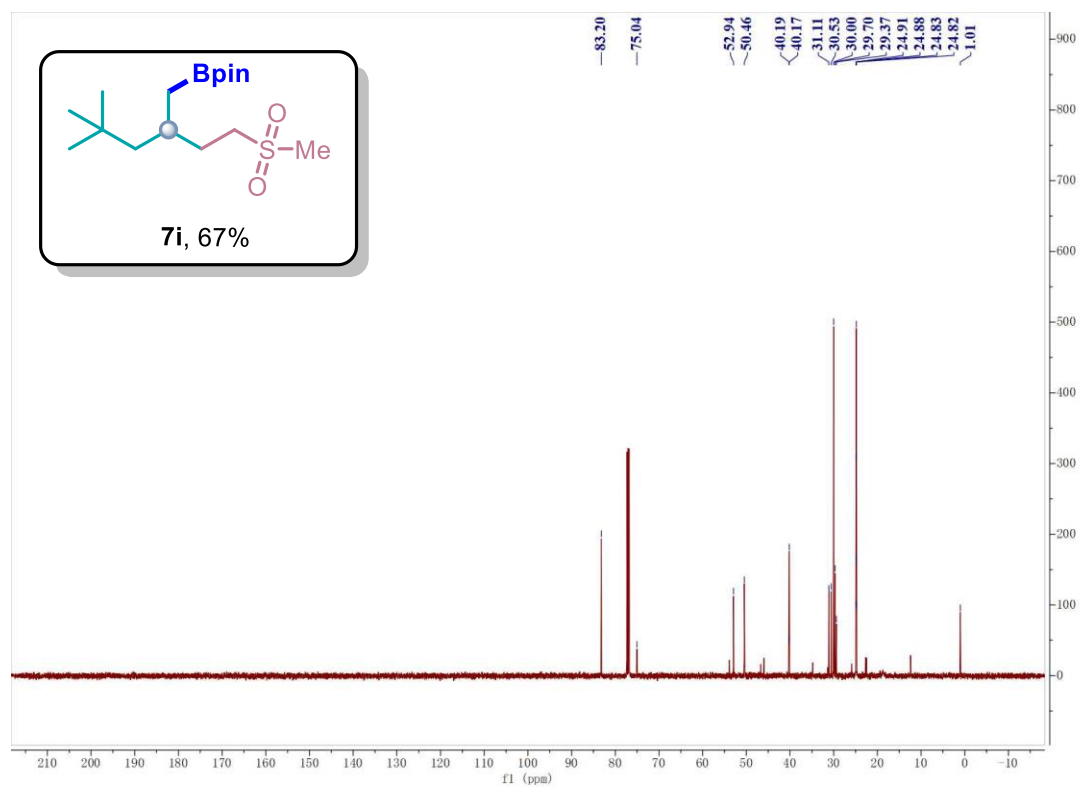


2-(4,4-dimethyl-2-(2-(methylsulfonyl)ethyl)pentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (7i)

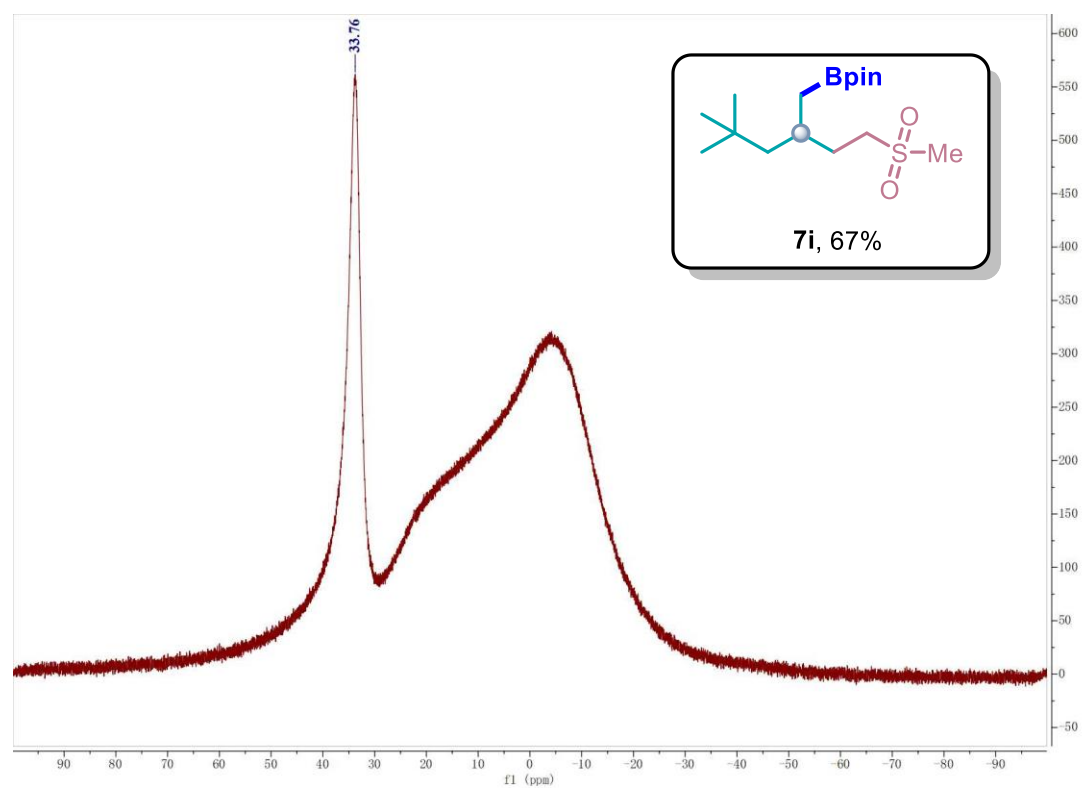
¹H NMR (500 MHz, Chloroform-*d*)



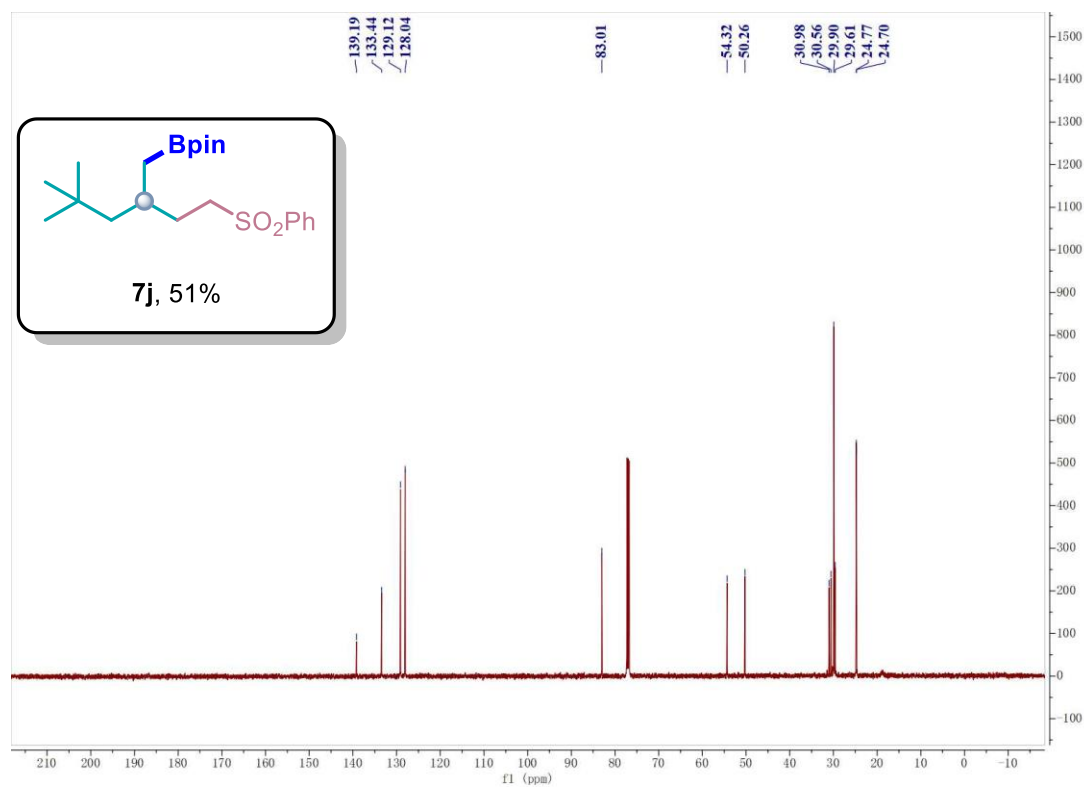
¹³C NMR (126 MHz, Chloroform-*d*)



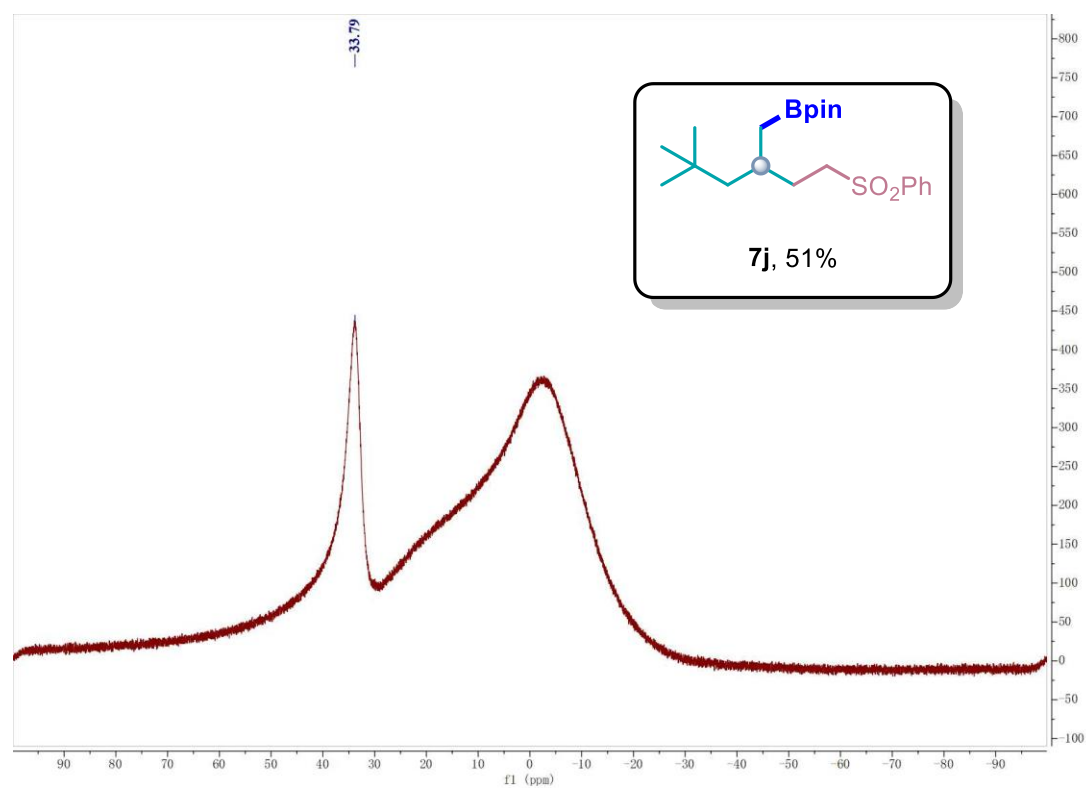
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)



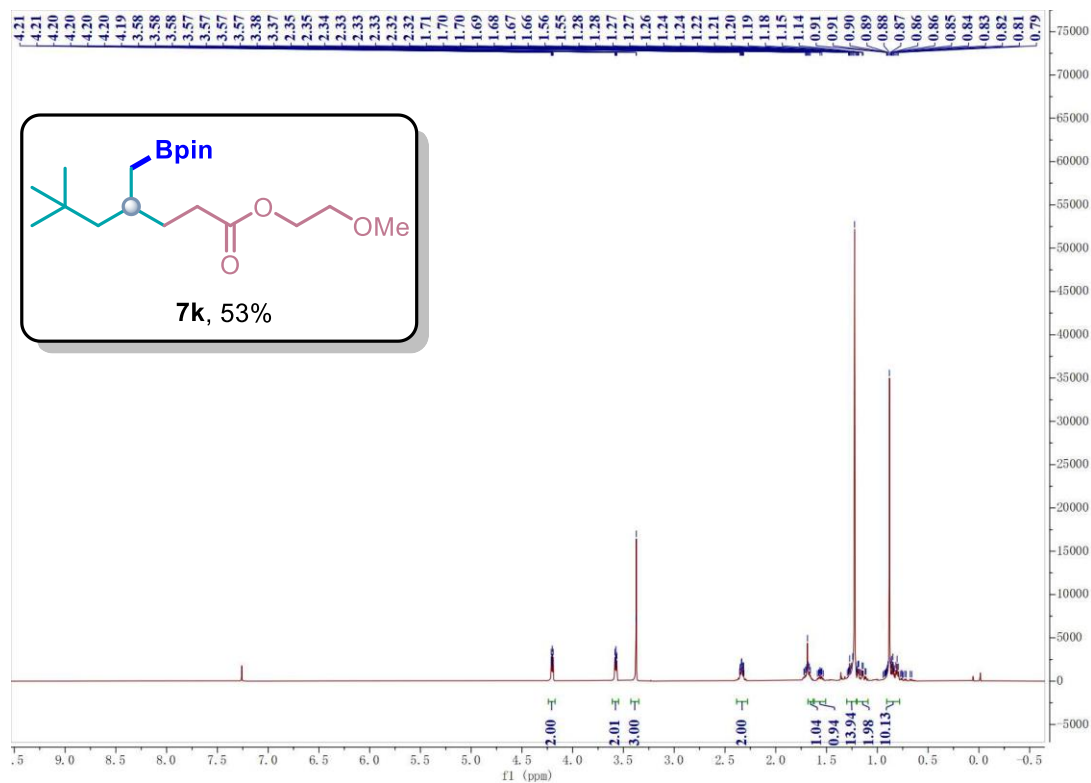
^{11}B NMR (160 MHz, Chloroform-*d*)



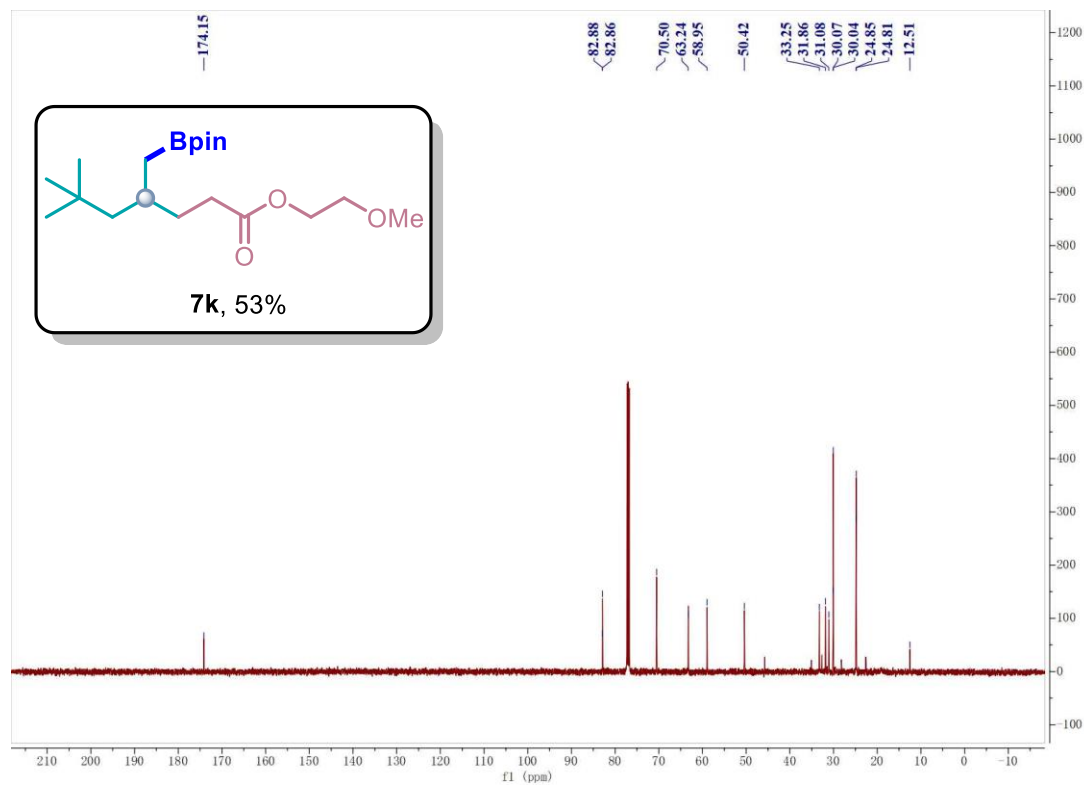
2-methoxyethyl
yl)methyl)heptanoate (7k)

6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-

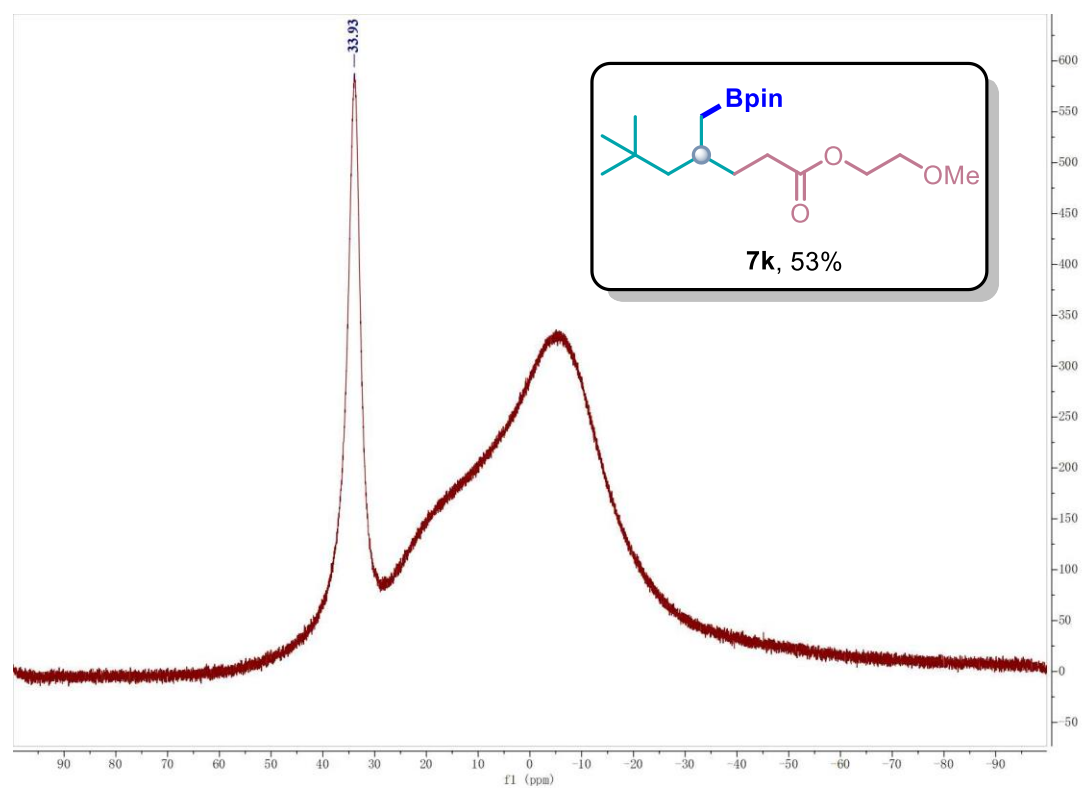
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

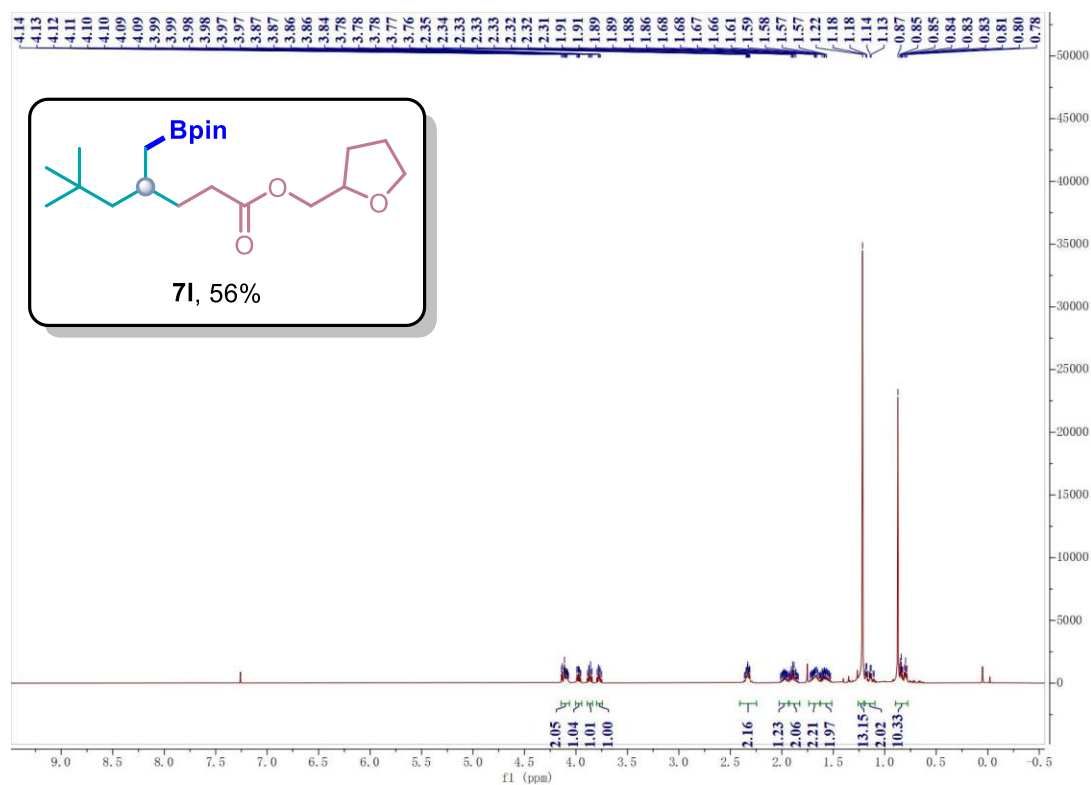


^{11}B NMR (160 MHz, Chloroform-*d*)

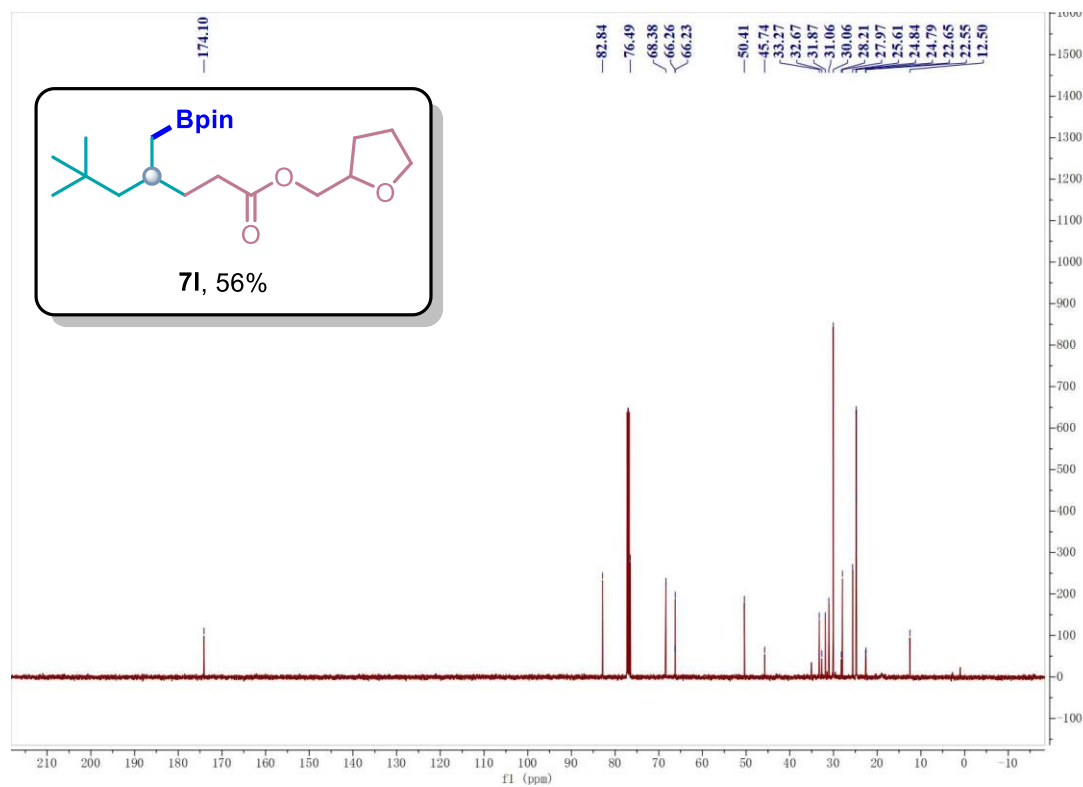


(tetrahydrofuran-2-yl)methyl 6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7l)

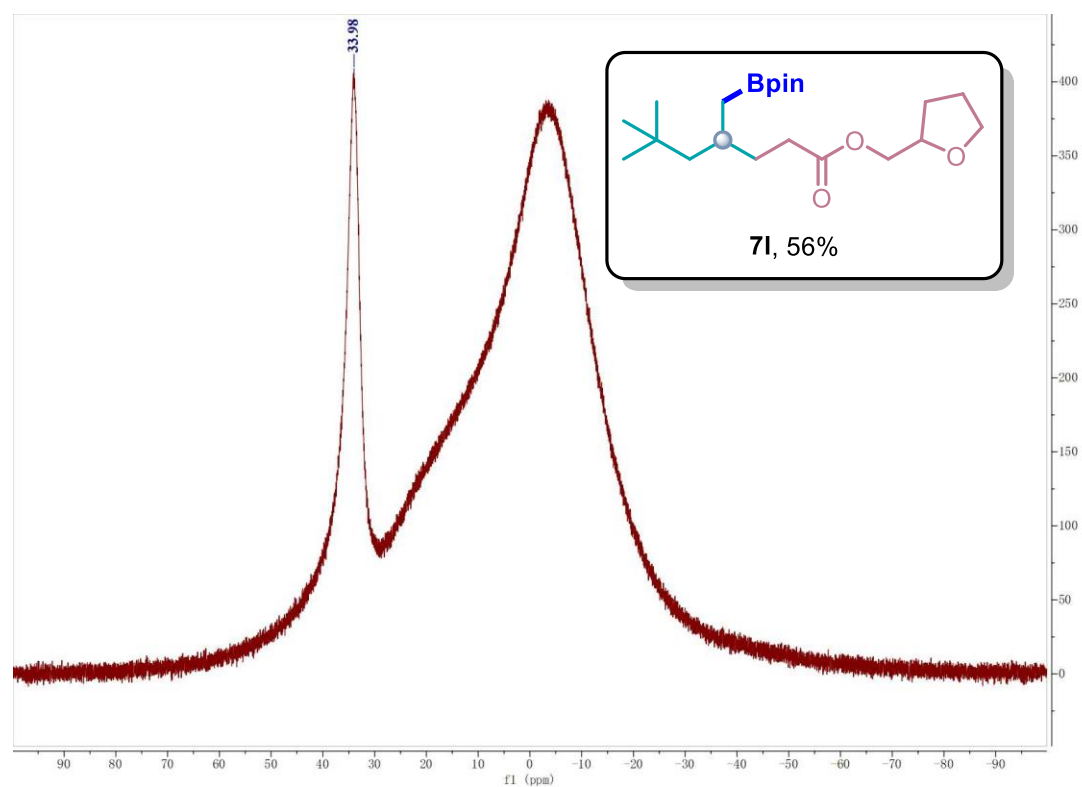
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)



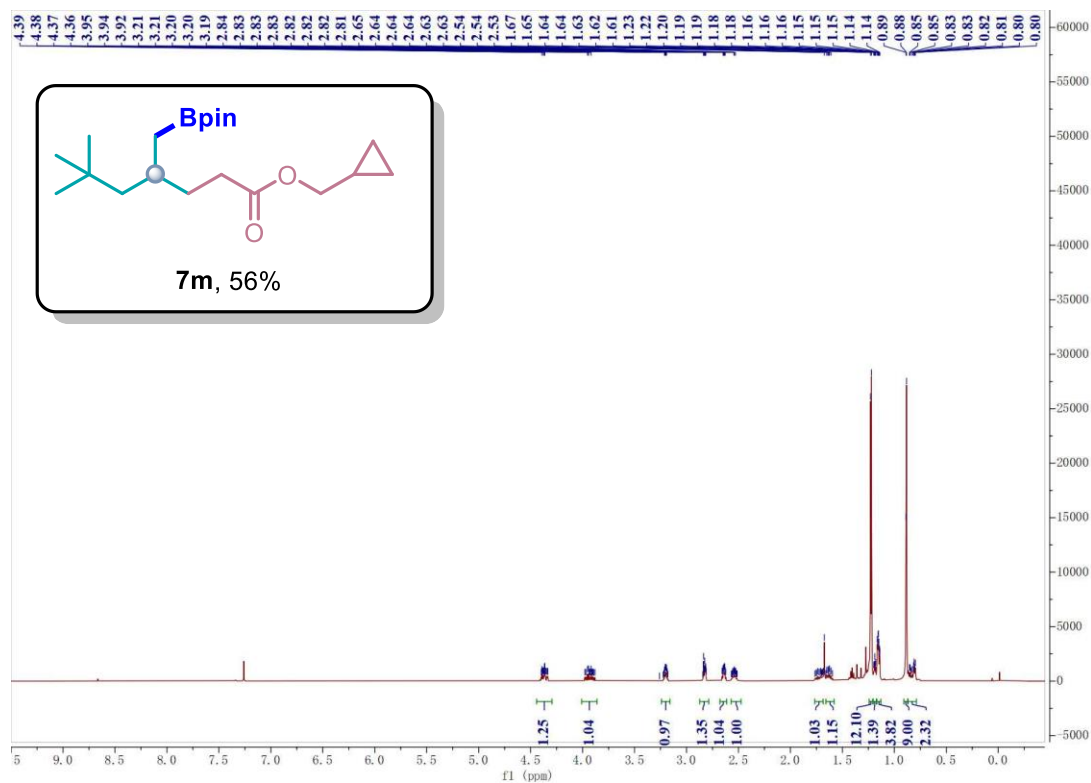
^{11}B NMR (160 MHz, Chloroform-*d*)



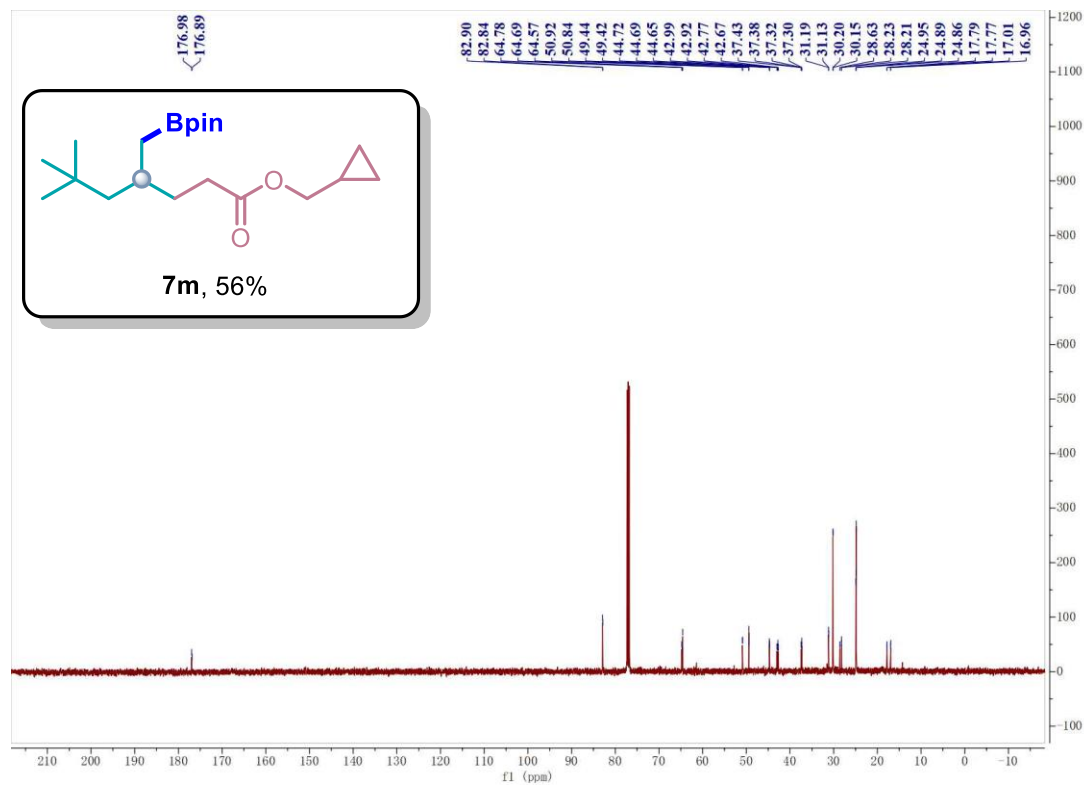
cyclopropylmethyl
yl)methyl)heptanoate (7m)

6,6-dimethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-

¹H NMR (500 MHz, Chloroform-*d*)



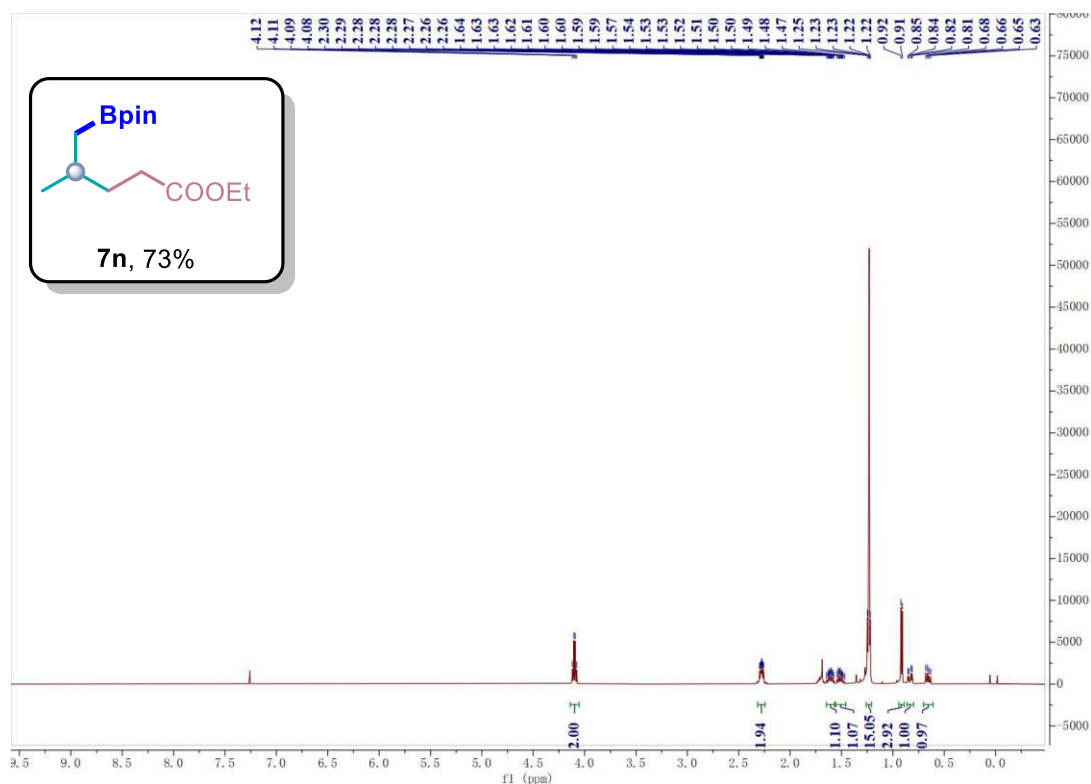
¹³C NMR (126 MHz, Chloroform-*d*)



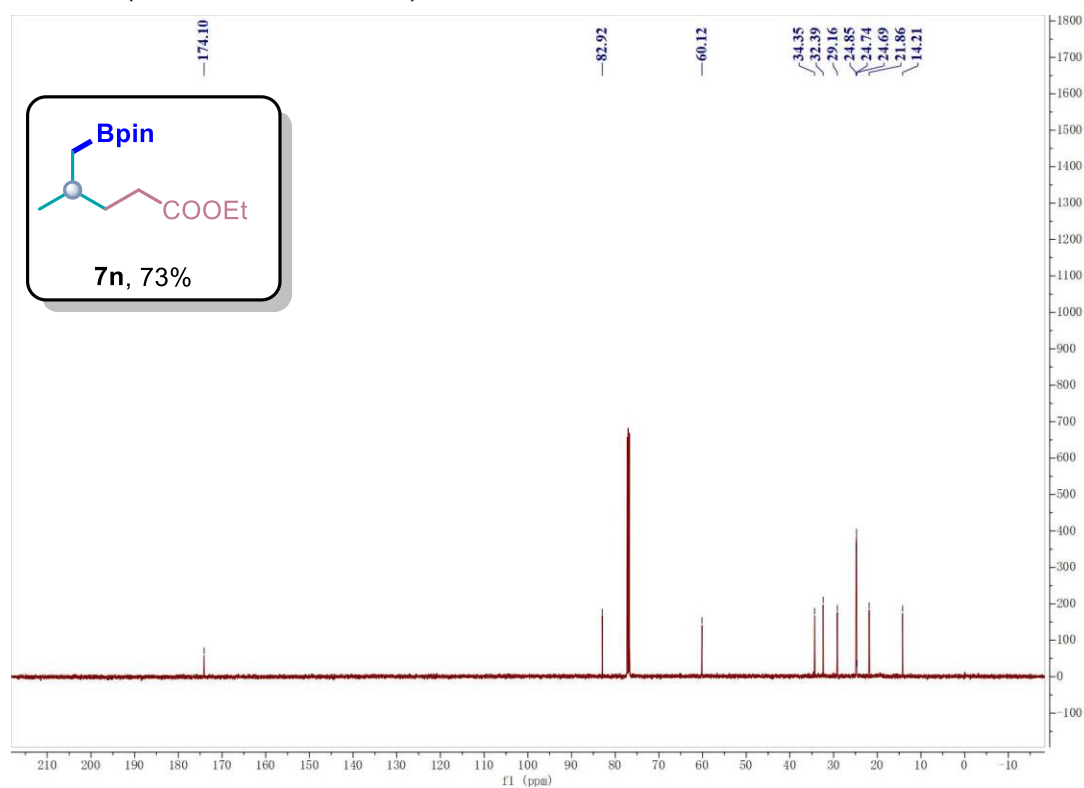
Chemical structure of compound **7m** is shown in the inset, which is a bicyclic ester derivative. The structure is labeled **7m, 56%**.

ethyl 4-methyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (7n)

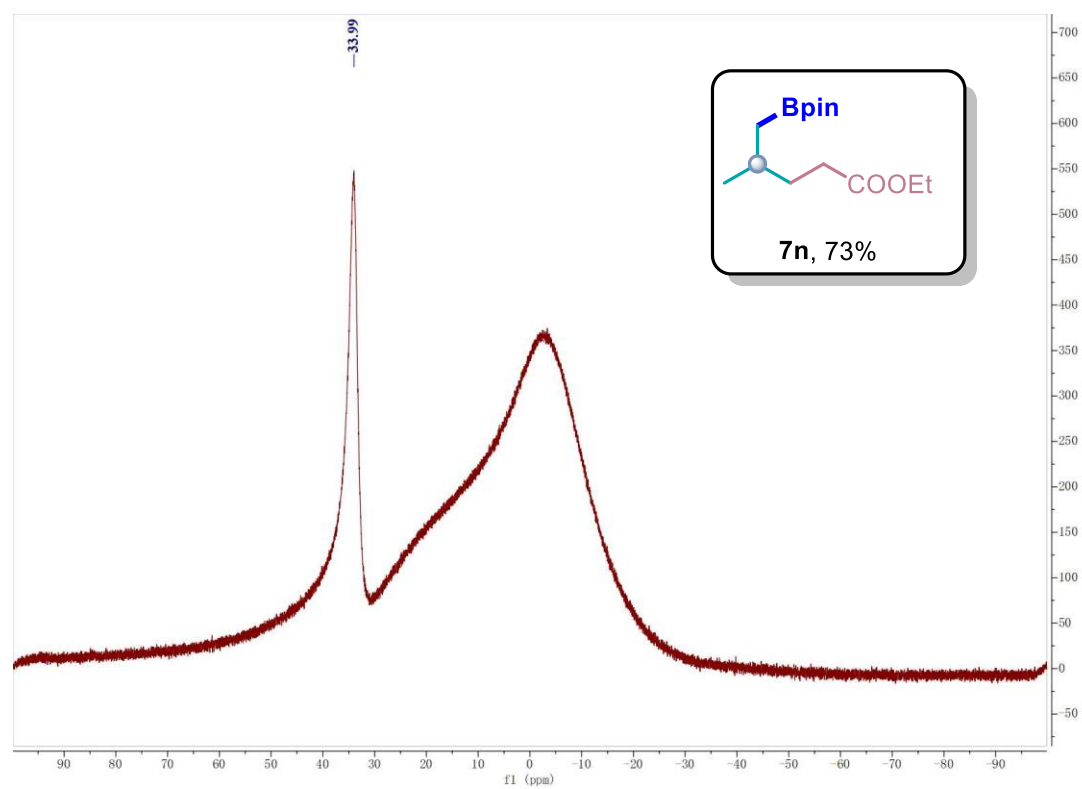
^1H NMR (500 MHz, Chloroform- d)



^{13}C NMR (126 MHz, Chloroform- d)

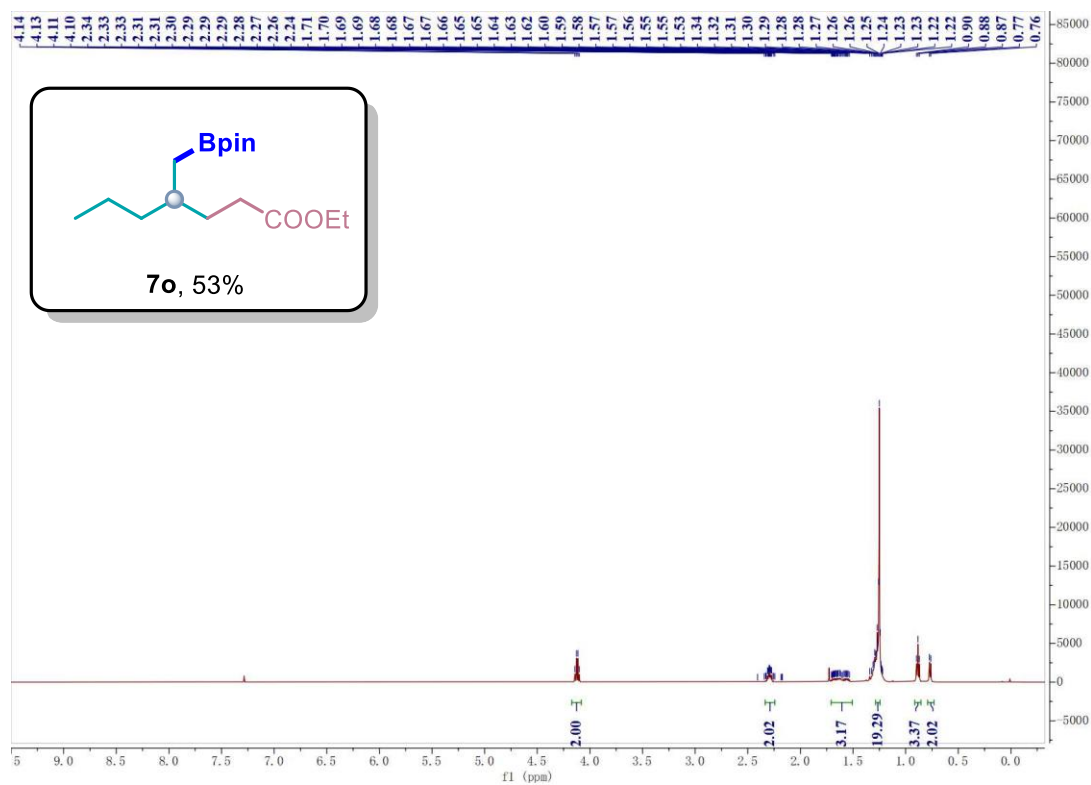


^{11}B NMR (160 MHz, Chloroform-*d*)

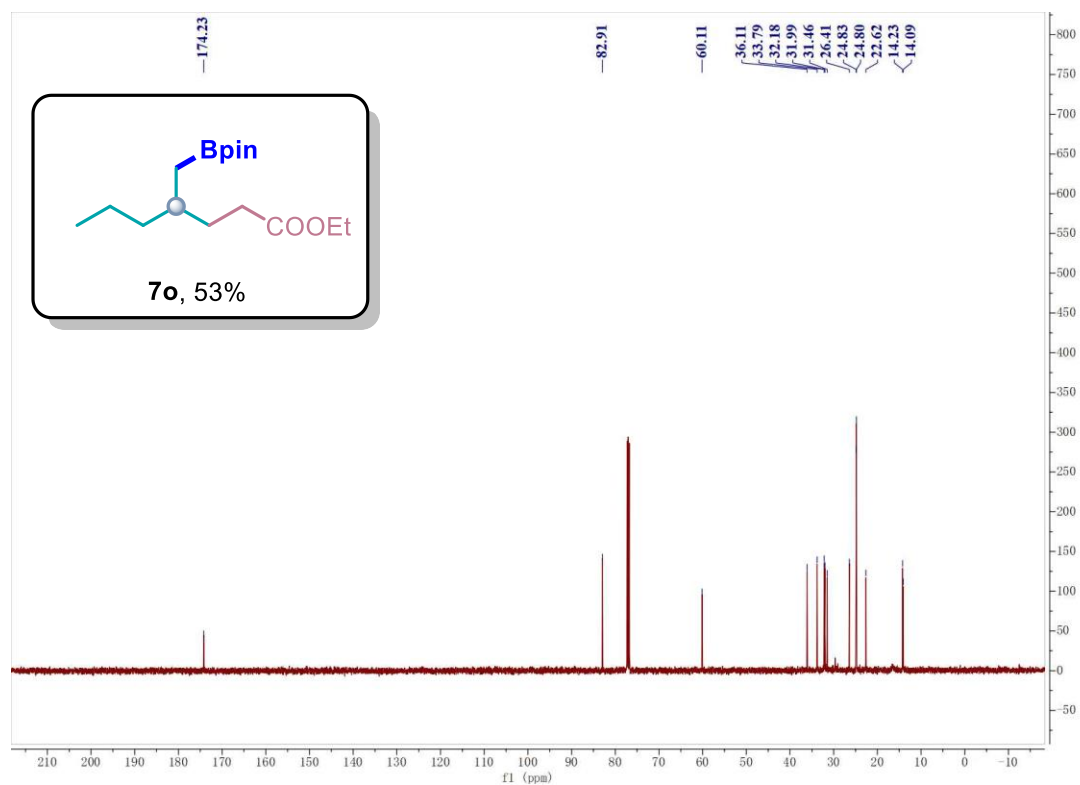


ethyl 4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**7o**)

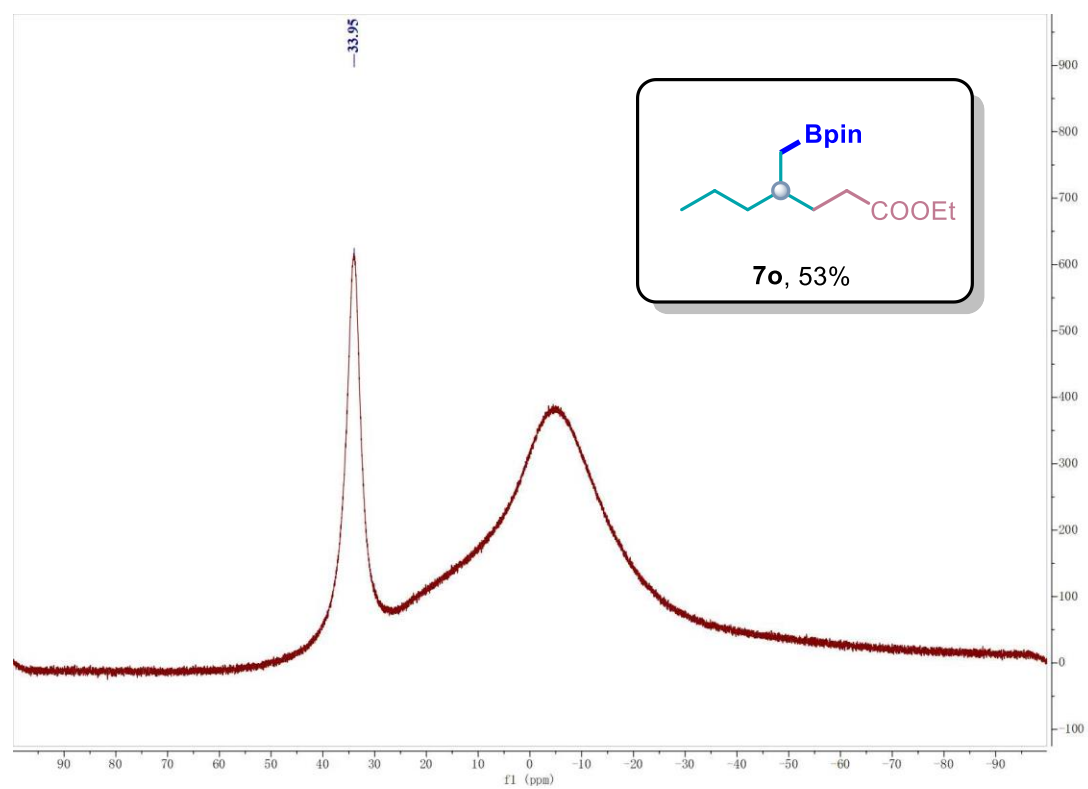
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

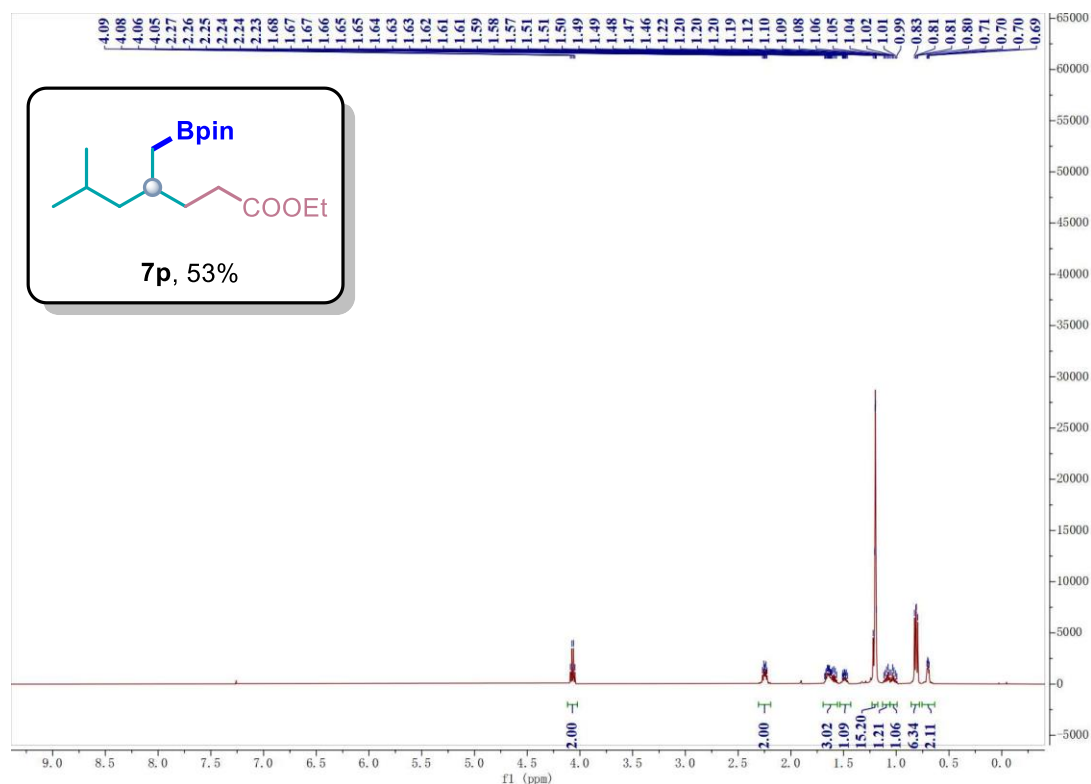


^{11}B NMR (160 MHz, Chloroform-*d*)

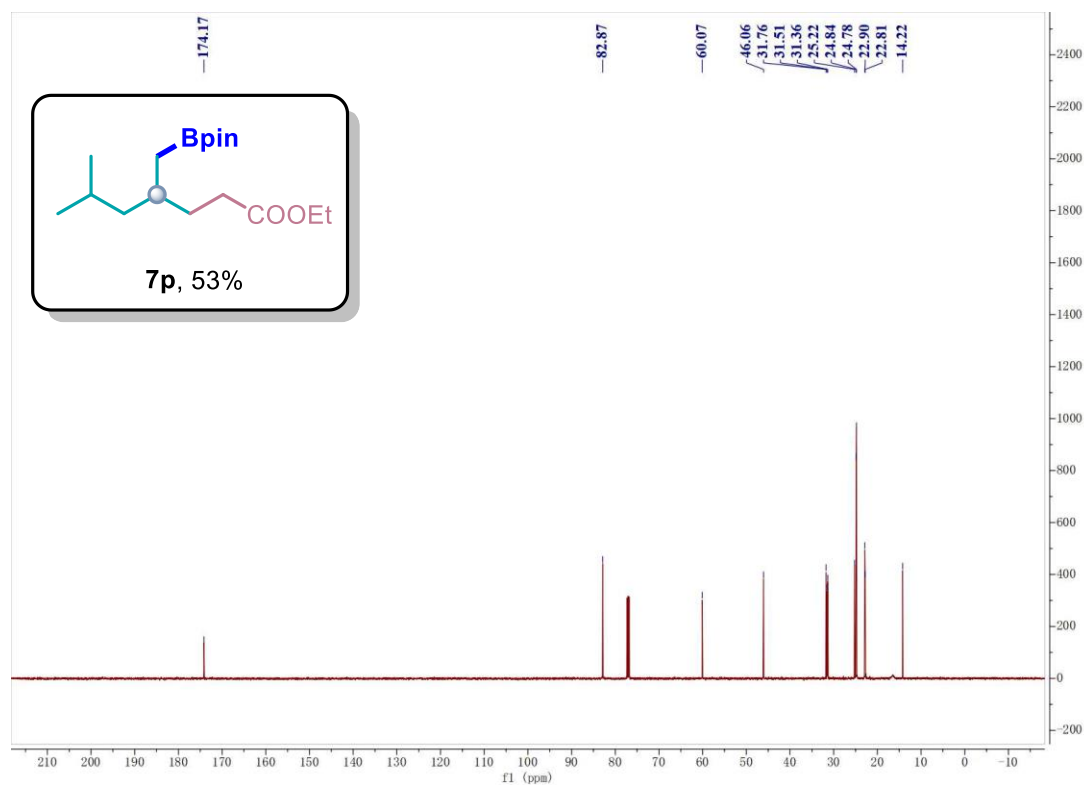


ethyl 6-methyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (**7p**)

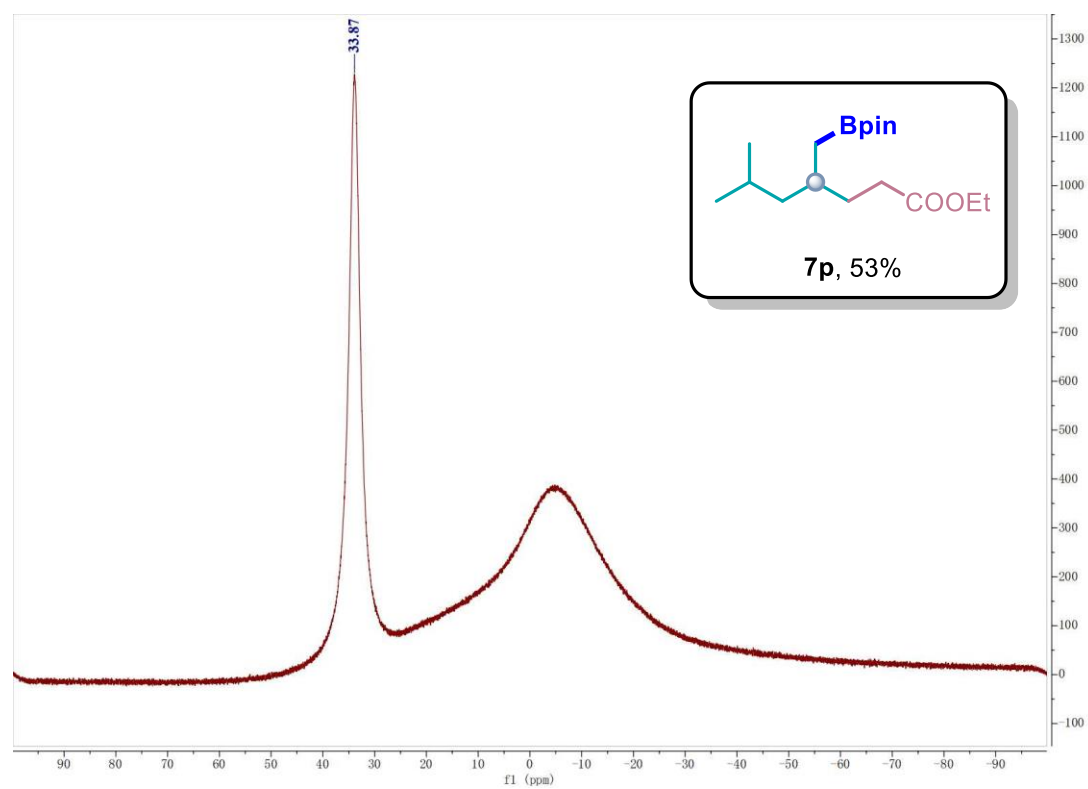
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

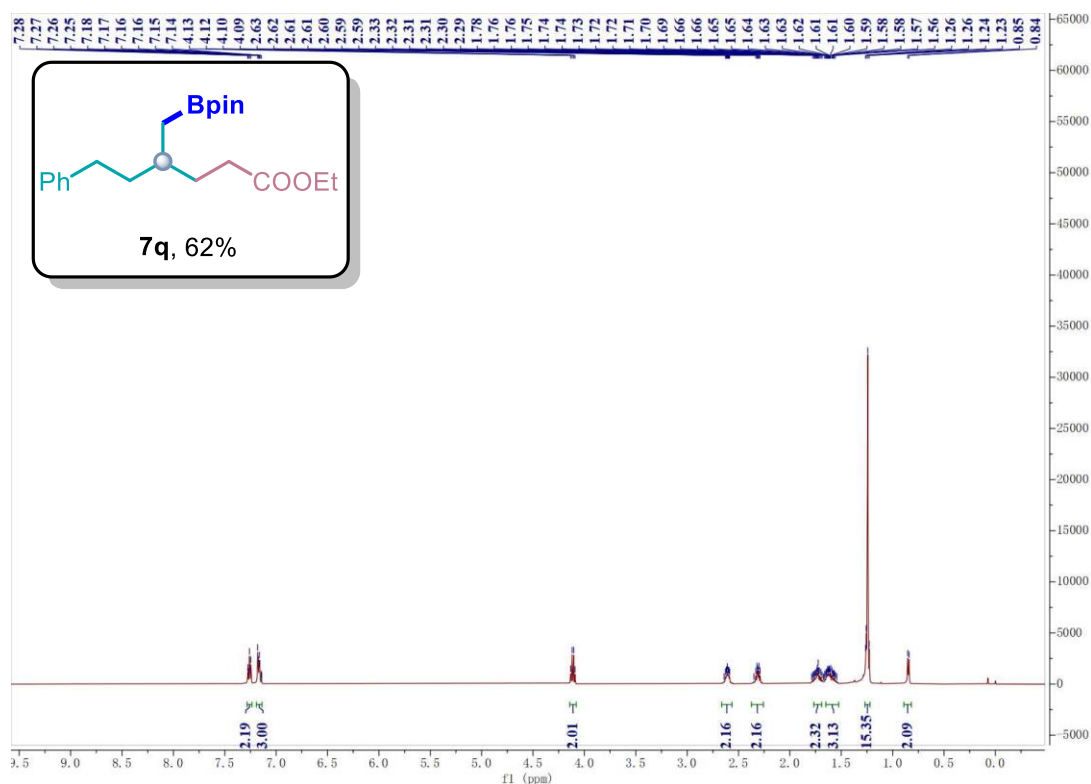


^{11}B NMR (160 MHz, Chloroform-*d*)

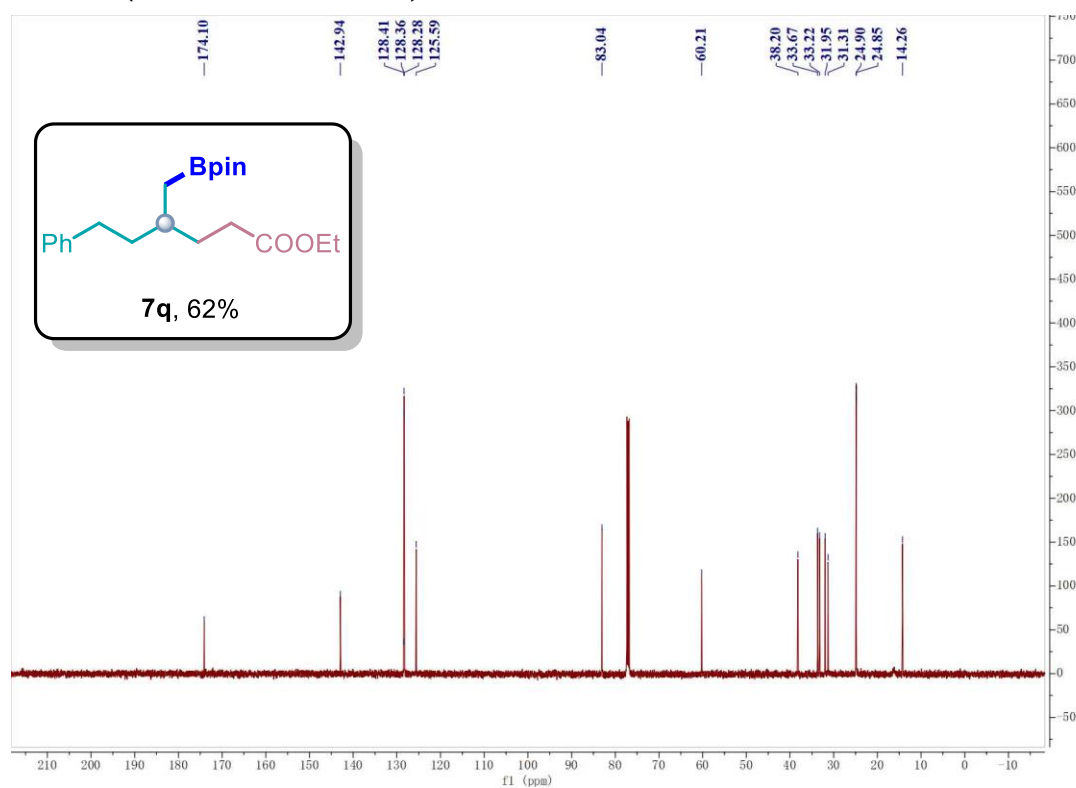


ethyl 6-phenyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)hexanoate (7q)

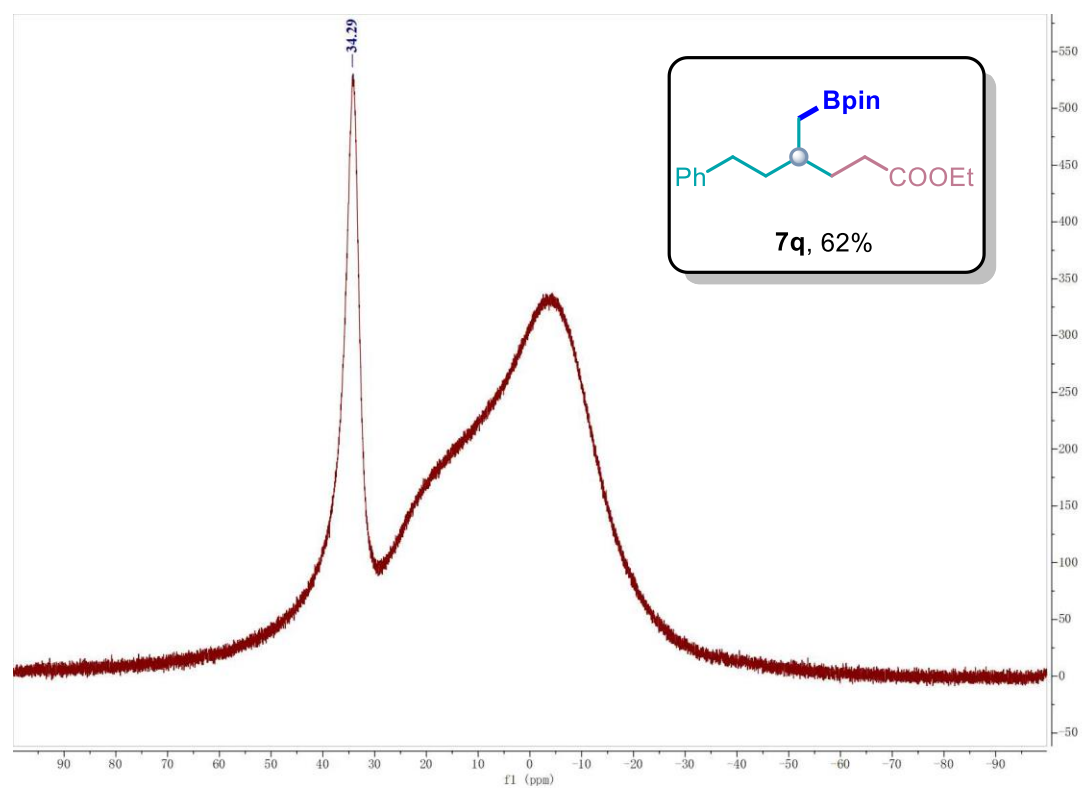
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

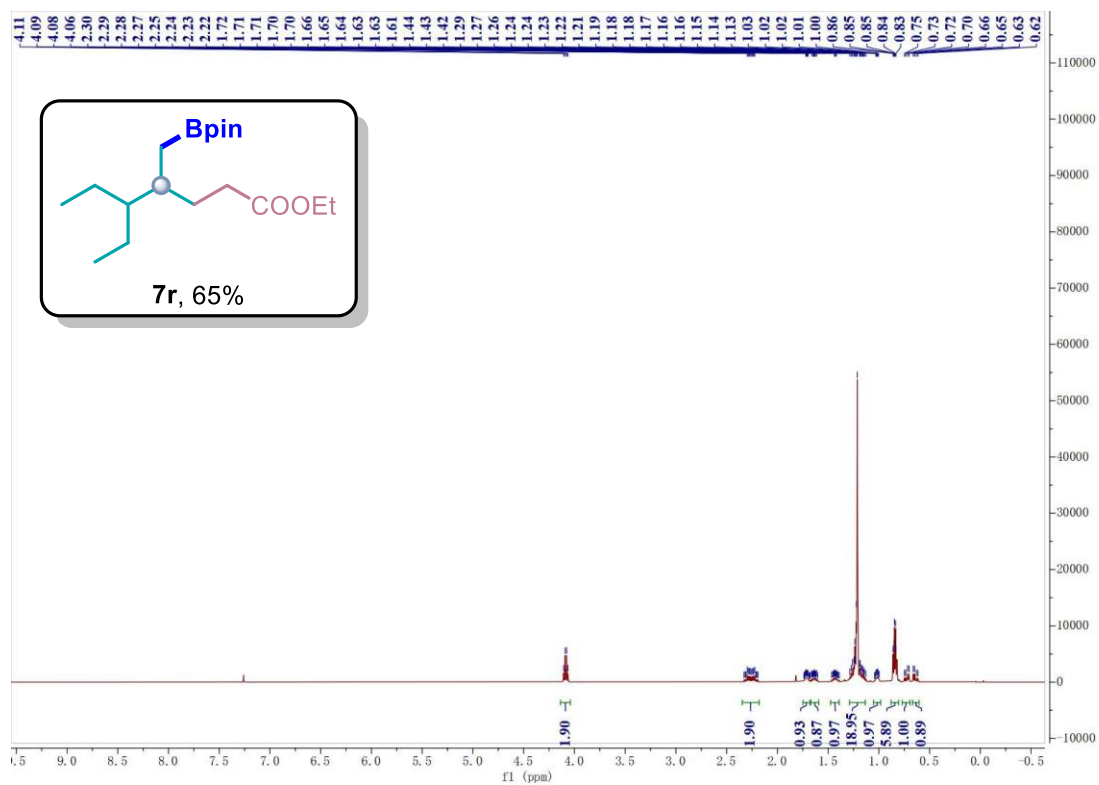


^{11}B NMR (160 MHz, Chloroform-*d*)

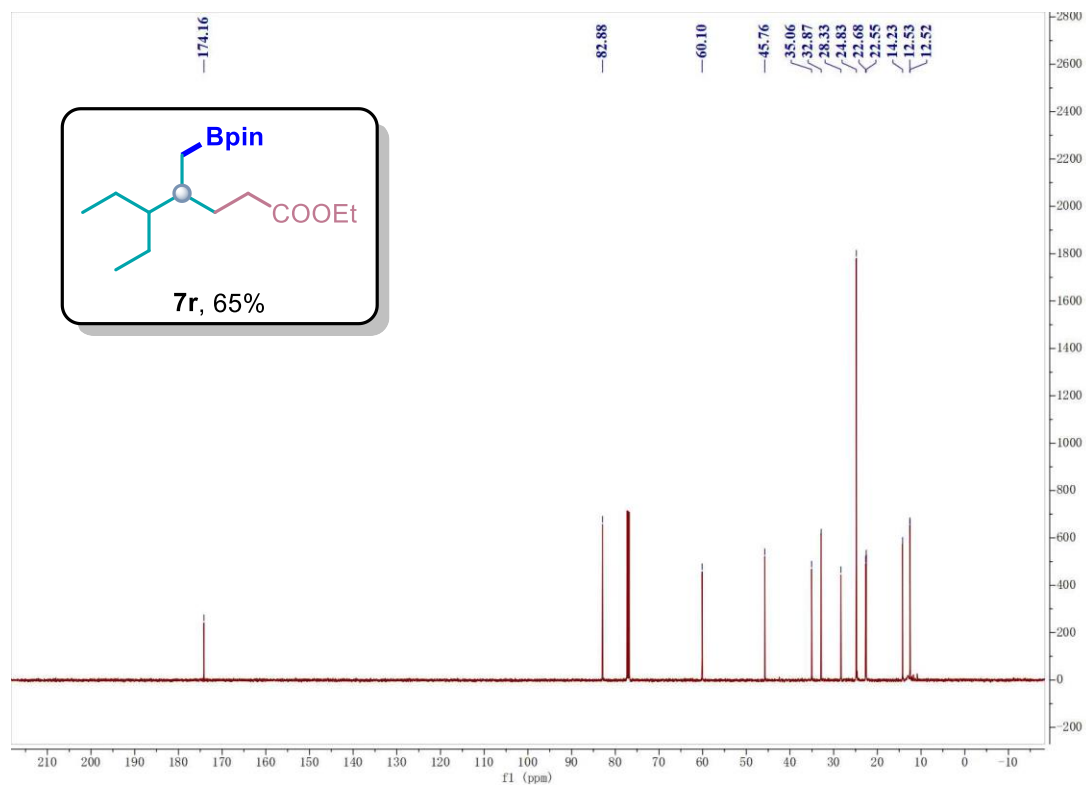


ethyl 5-ethyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)heptanoate (7r)

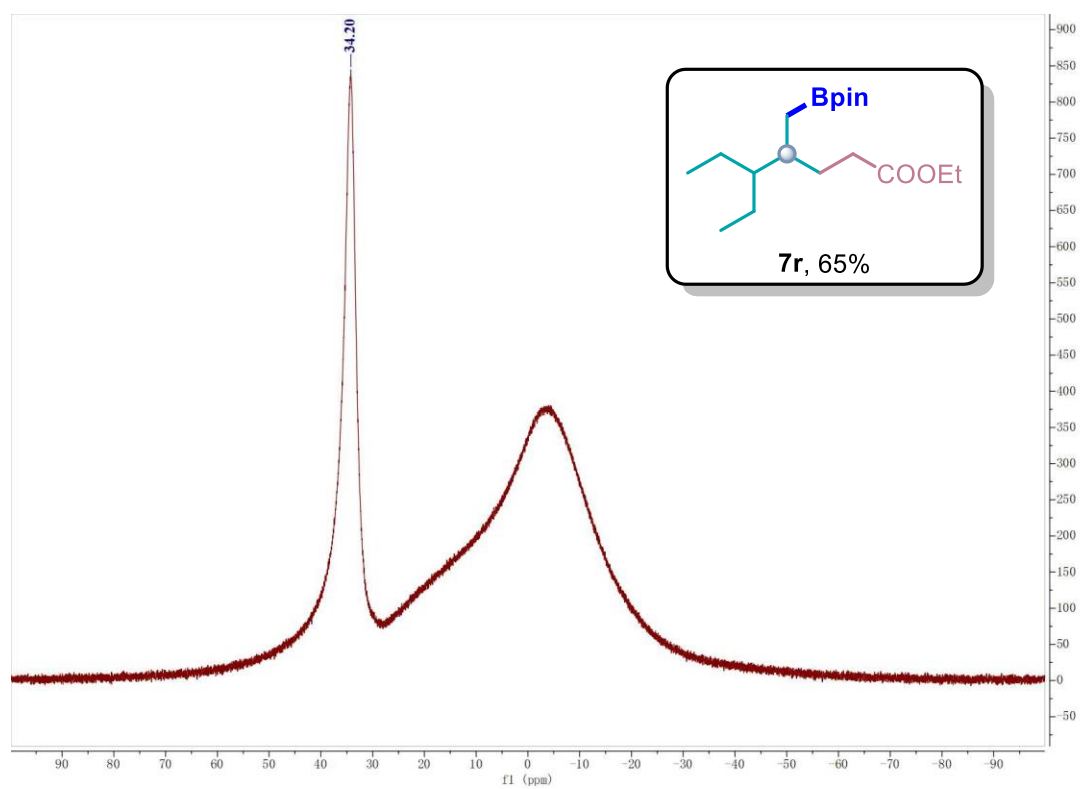
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

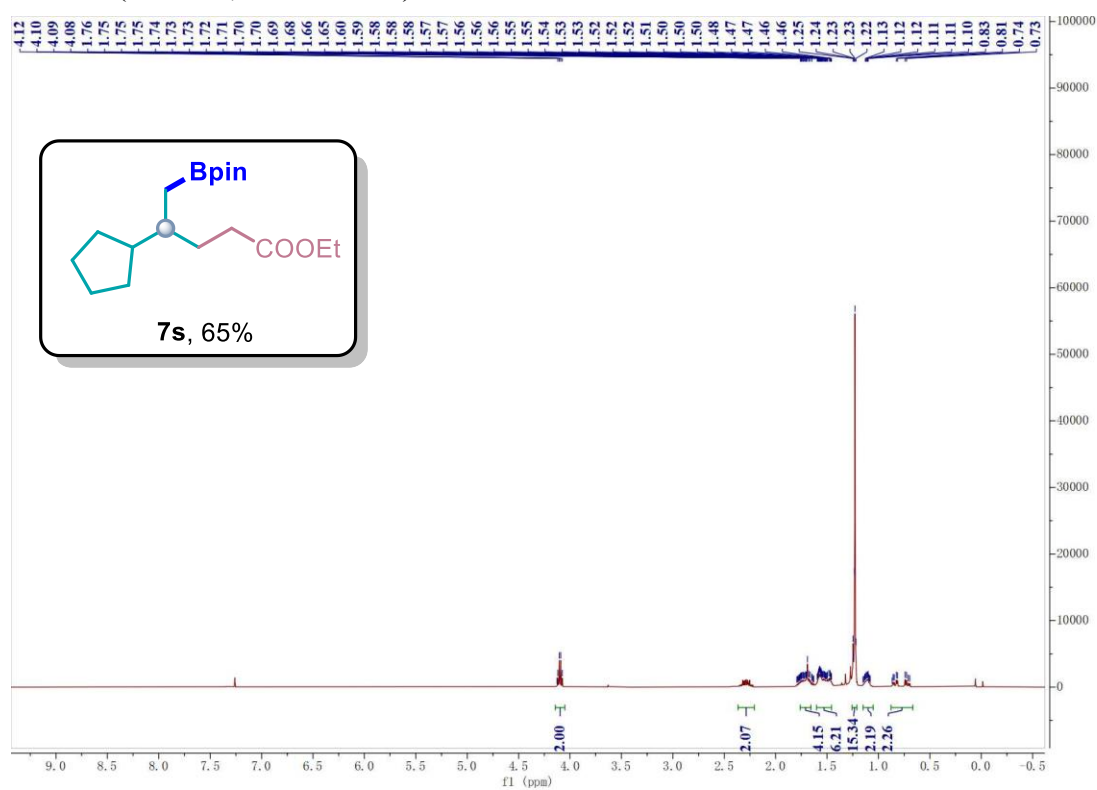


^{11}B NMR (160 MHz, Chloroform-*d*)

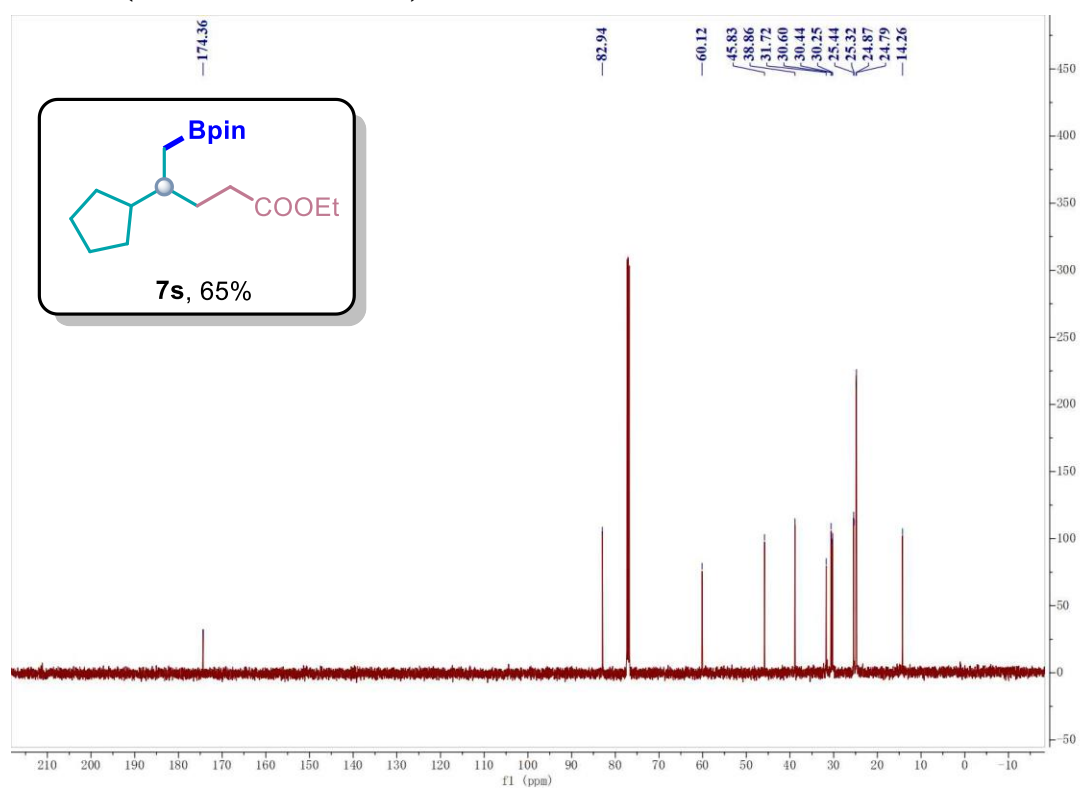


ethyl 4-cyclopentyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (**7s**)

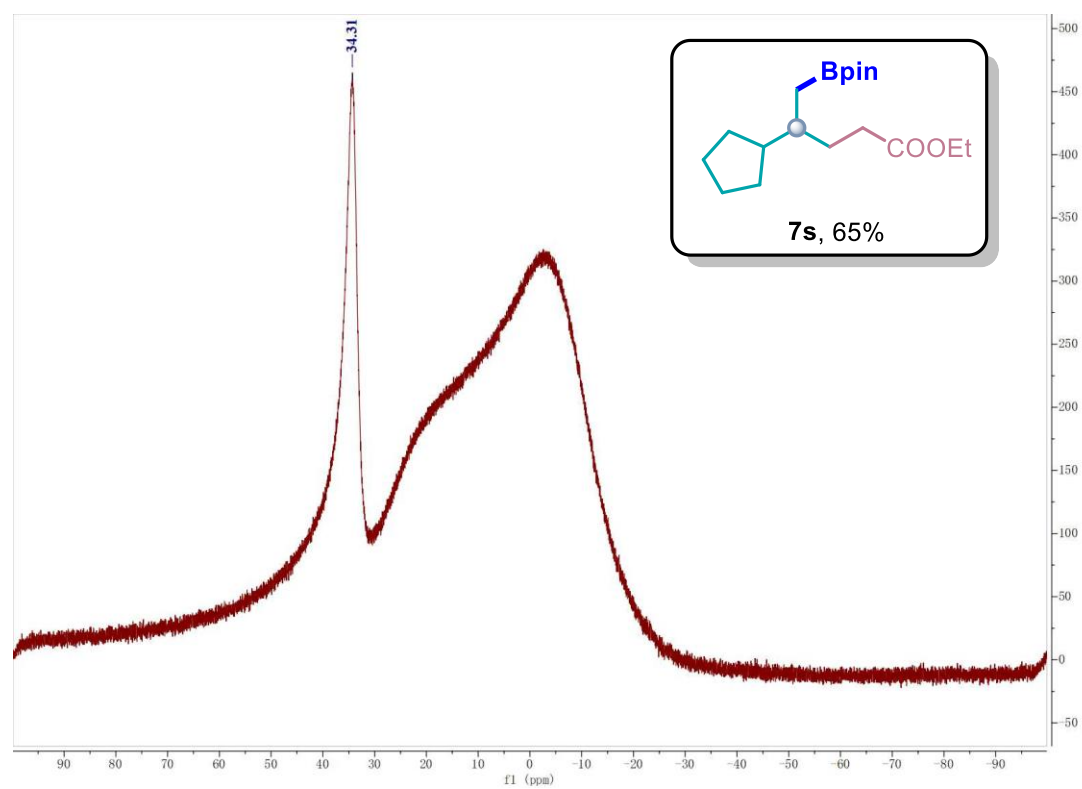
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

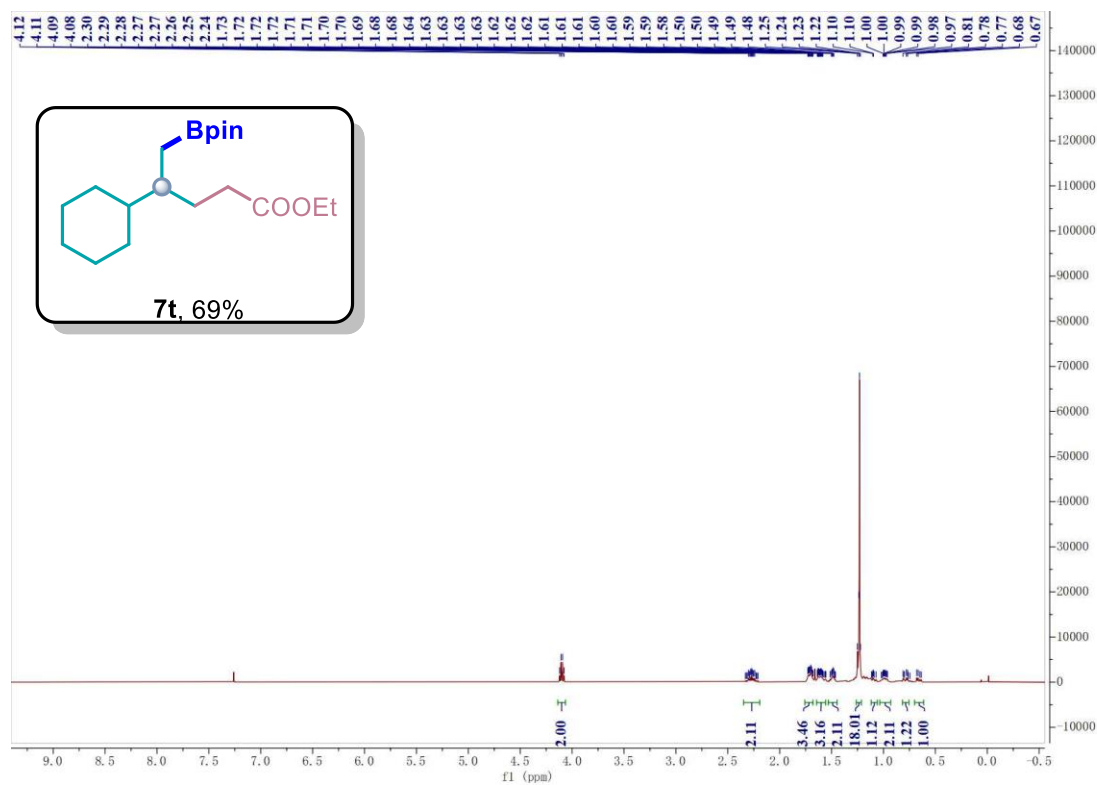


^{11}B NMR (160 MHz, Chloroform-*d*)

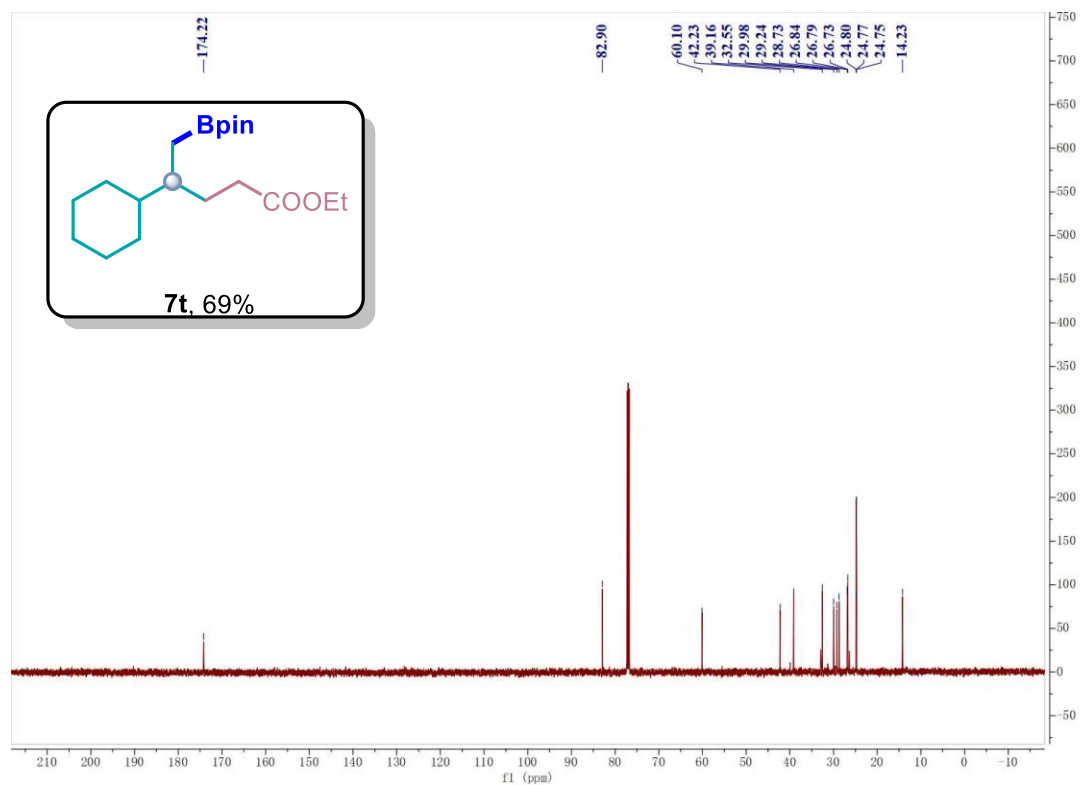


ethyl 4-cyclohexyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanoate (7t)

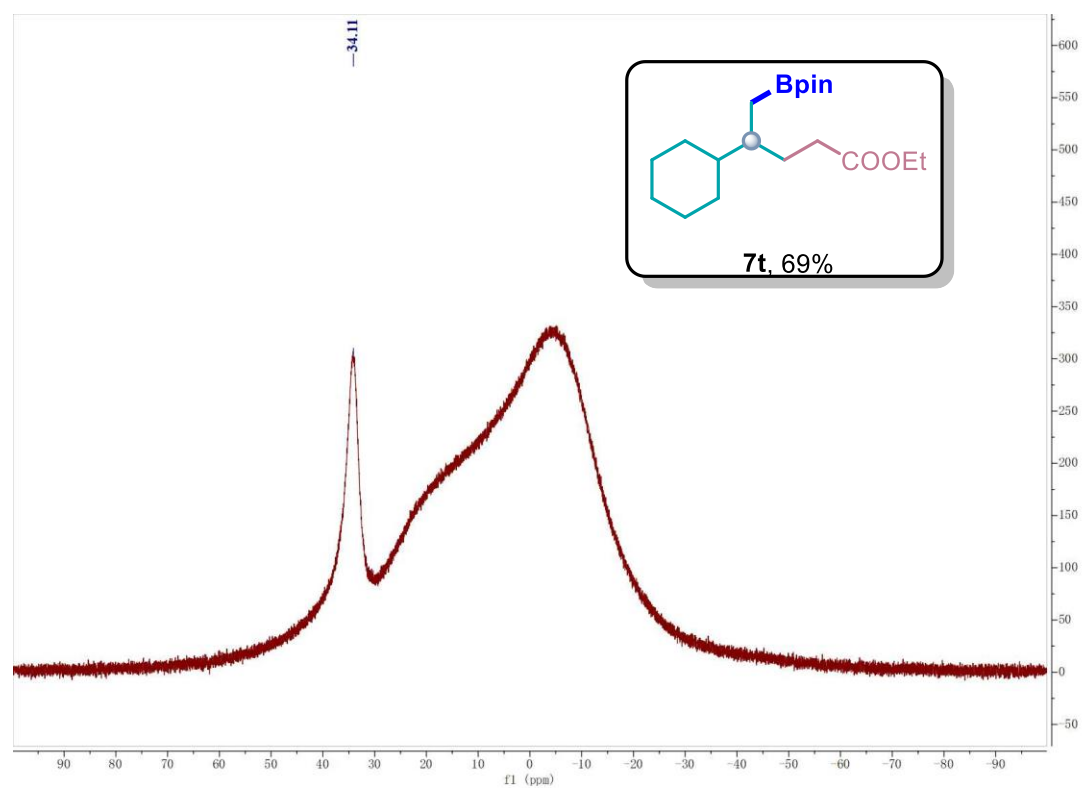
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

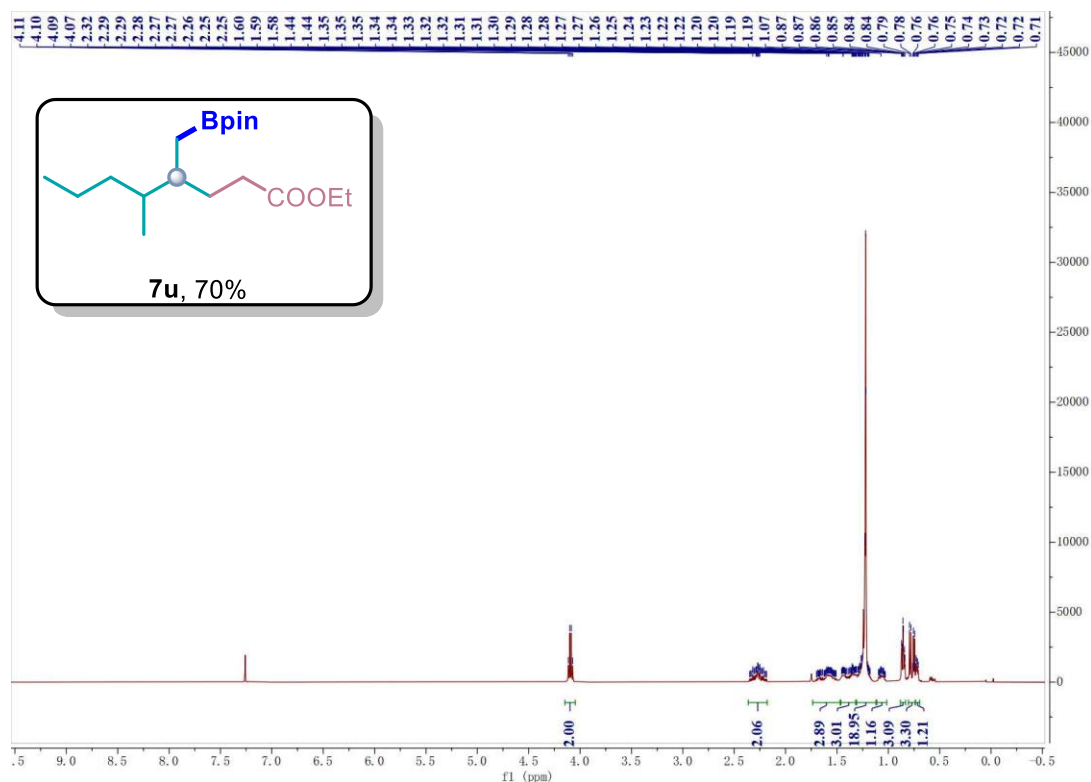


^{11}B NMR (160 MHz, Chloroform-*d*)

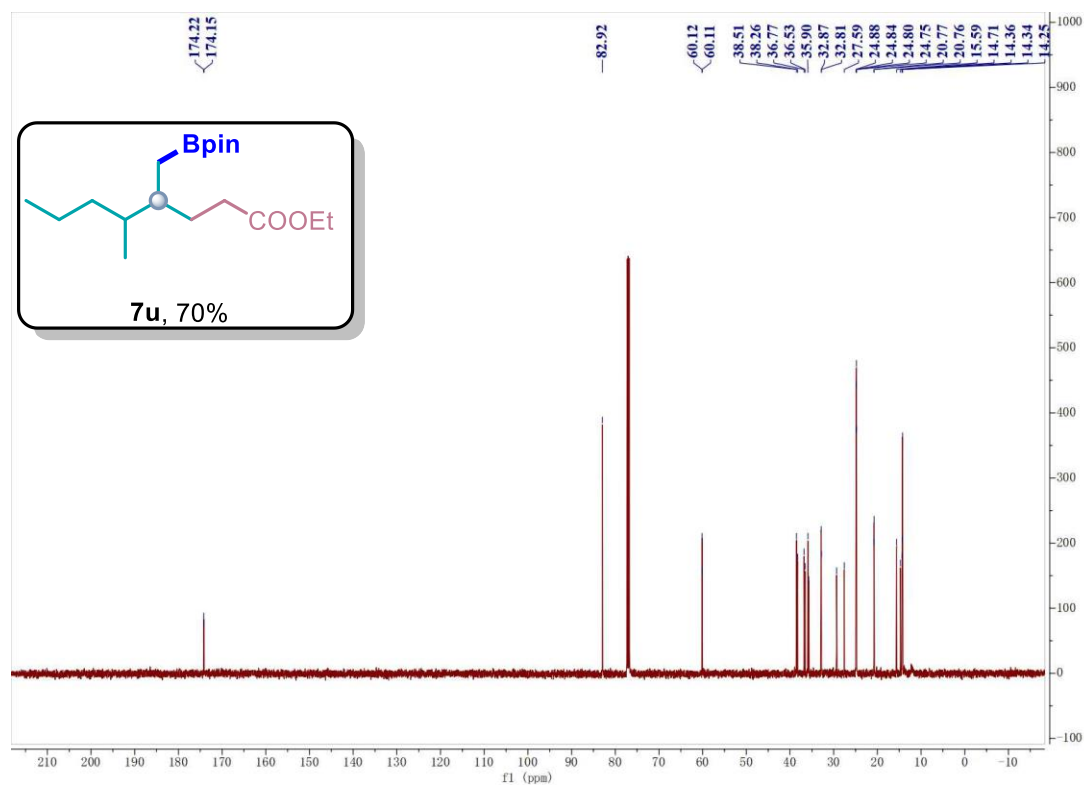


ethyl 5-methyl-4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)octanoate (**7u**)

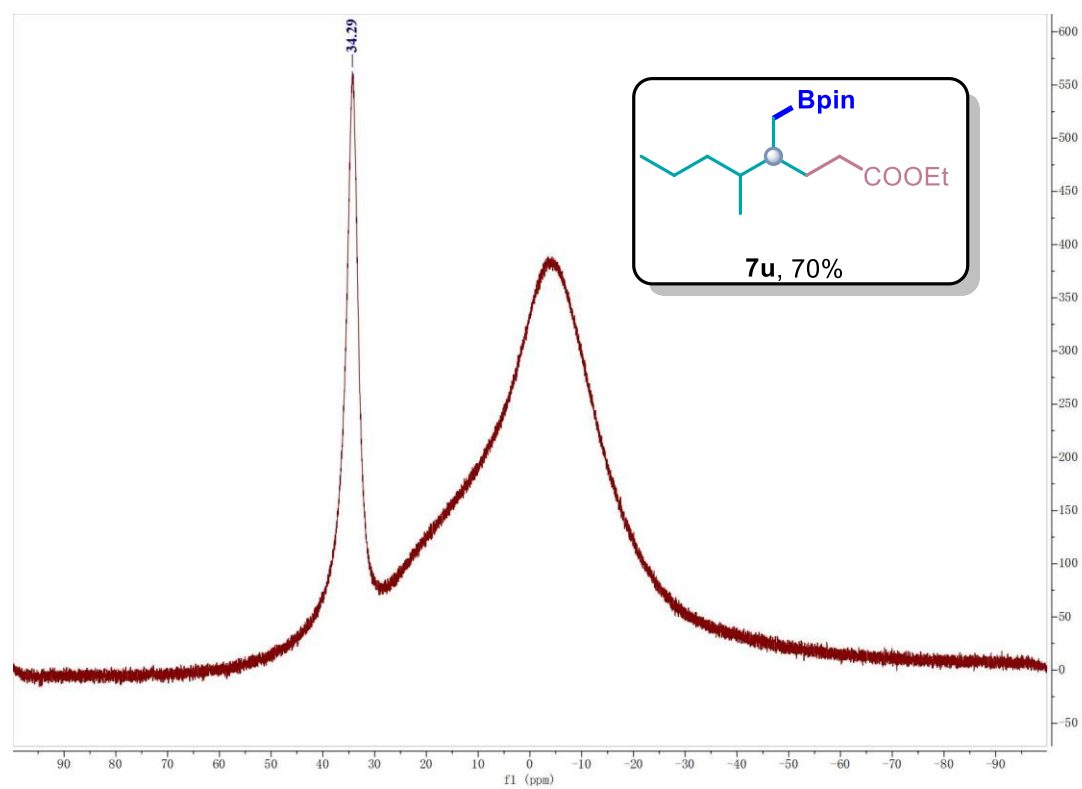
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

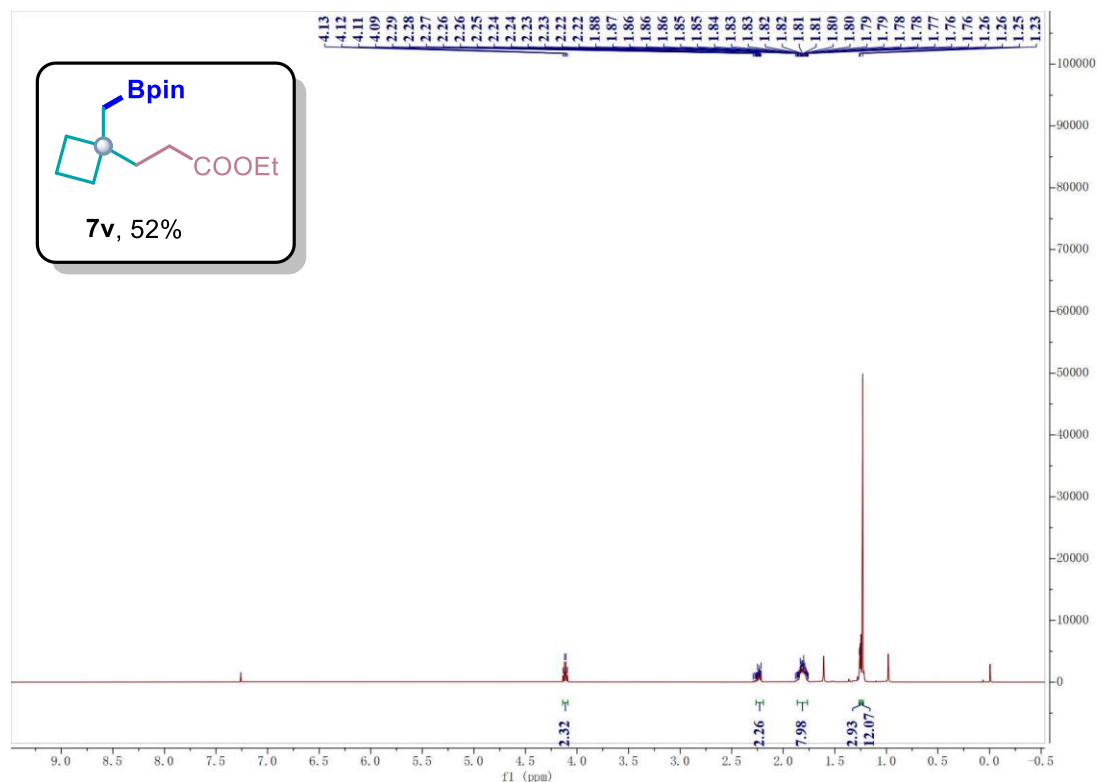


^{11}B NMR (160 MHz, Chloroform-*d*)

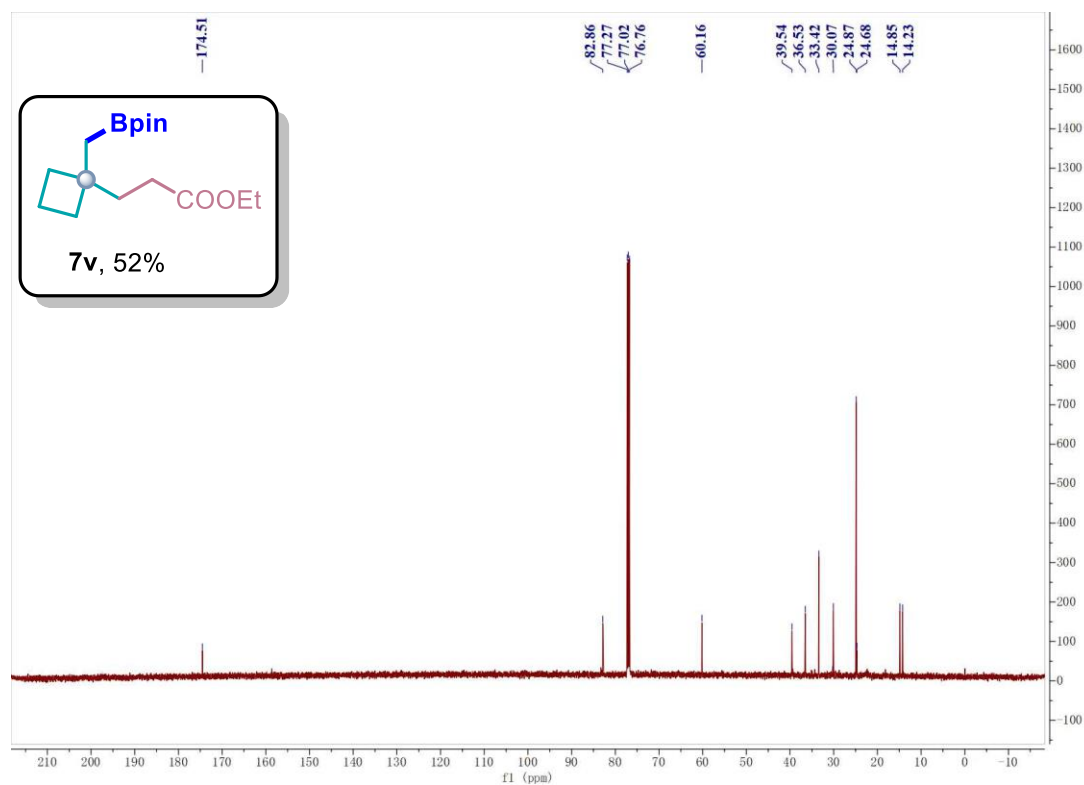


ethyl 3-(1-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)cyclobutyl)propanoate (7v)

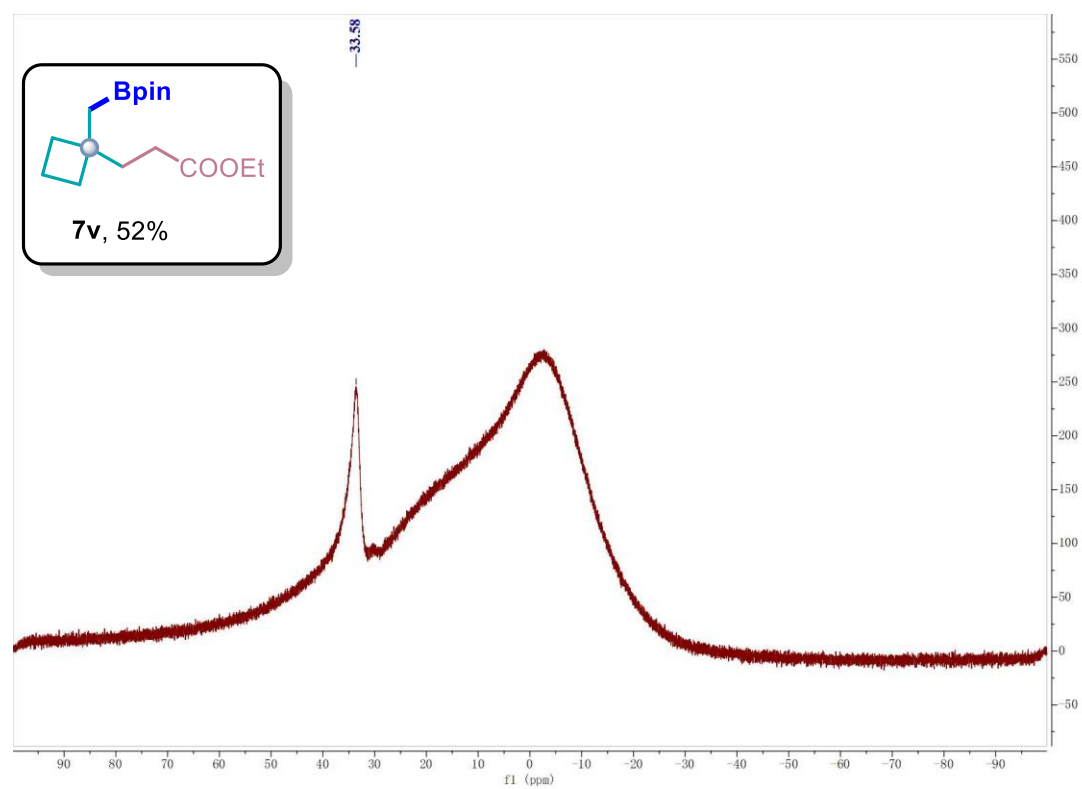
^1H NMR (500 MHz, Chloroform- d)



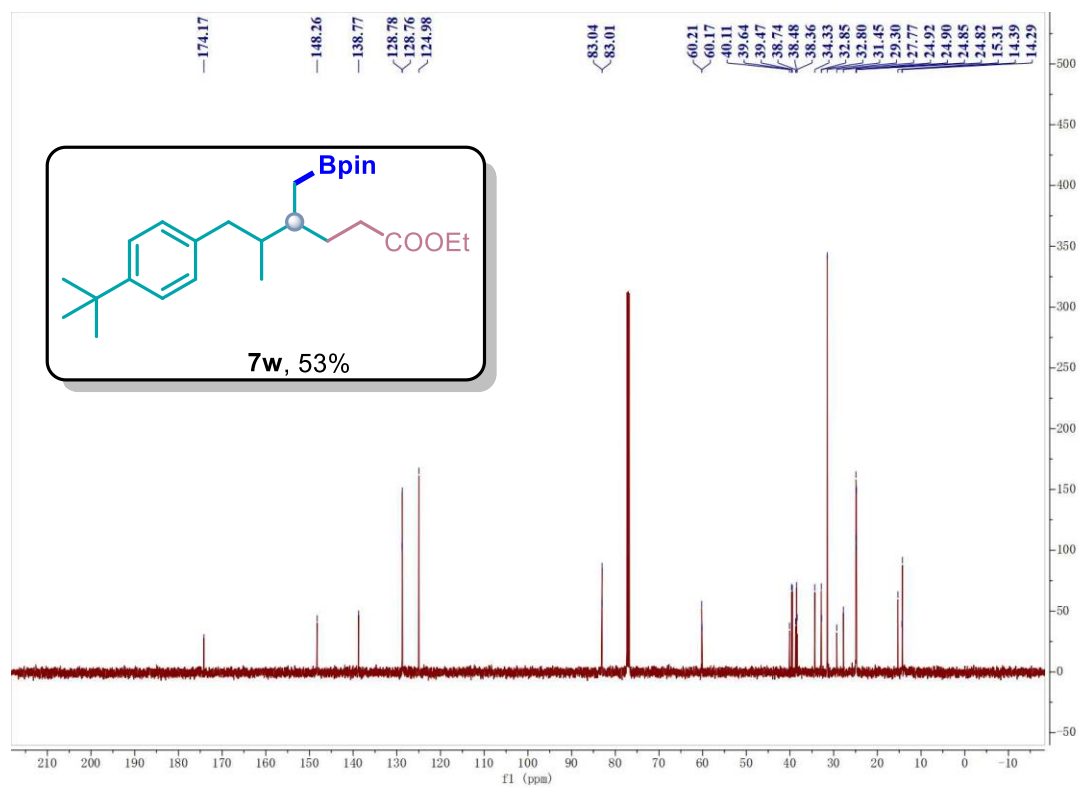
^{13}C NMR (126 MHz, Chloroform- d)



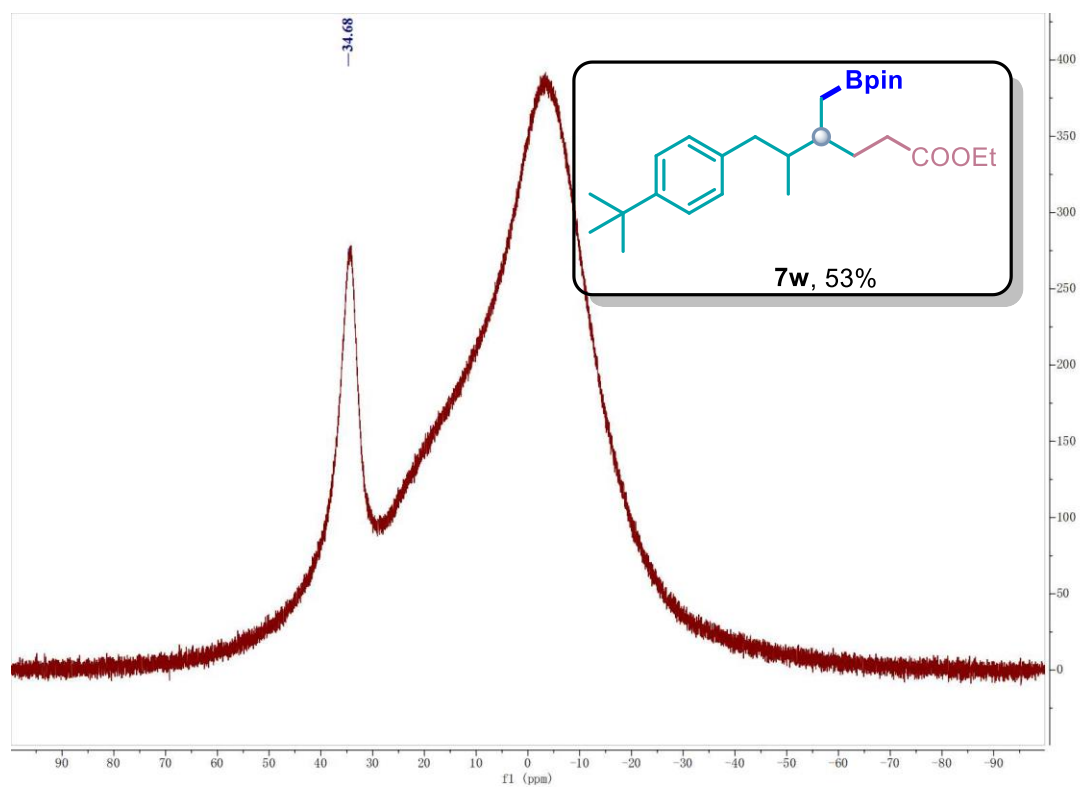
^{11}B NMR (160 MHz, Chloroform-*d*)



¹H NMR (500 MHz, Chloroform-*d*)

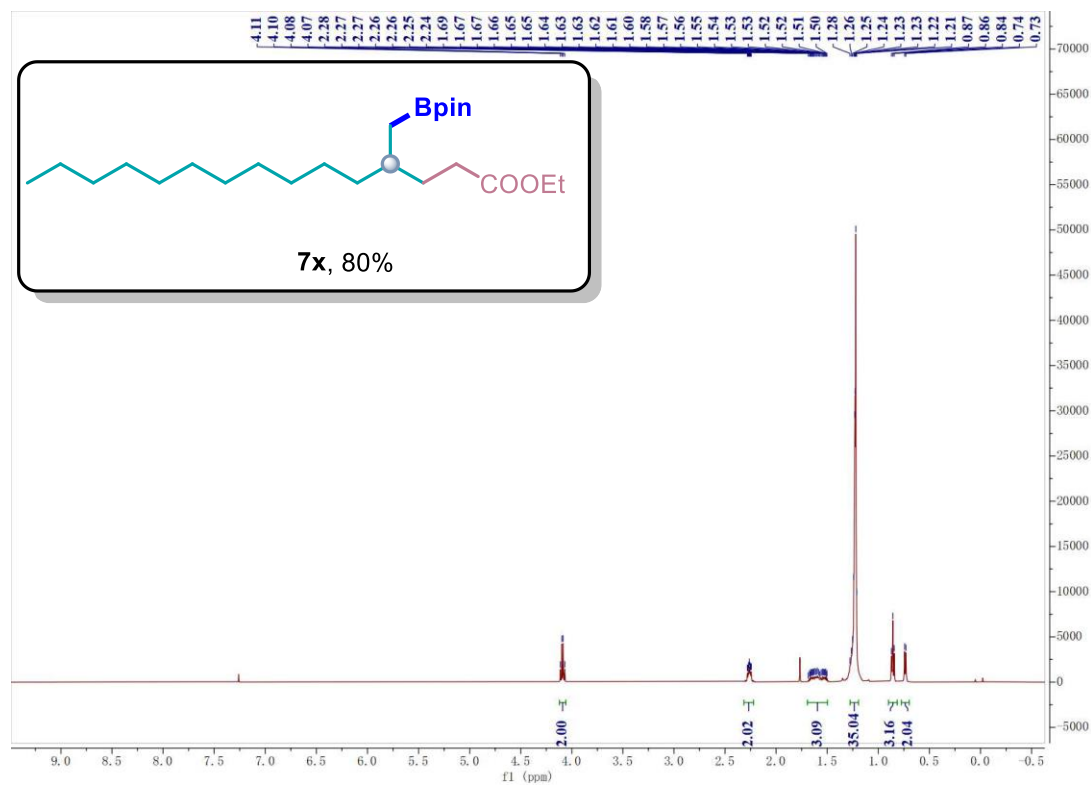


^{11}B NMR (160 MHz, Chloroform-*d*)

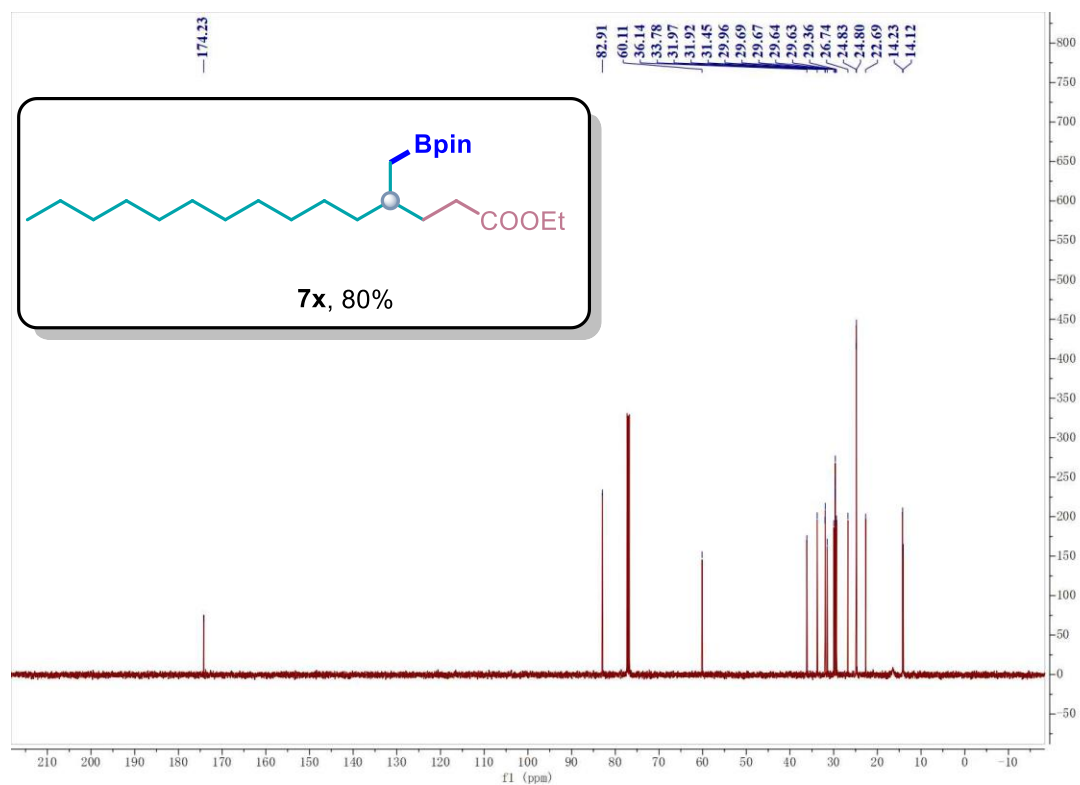


ethyl 4-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)pentadecanoate (7x)

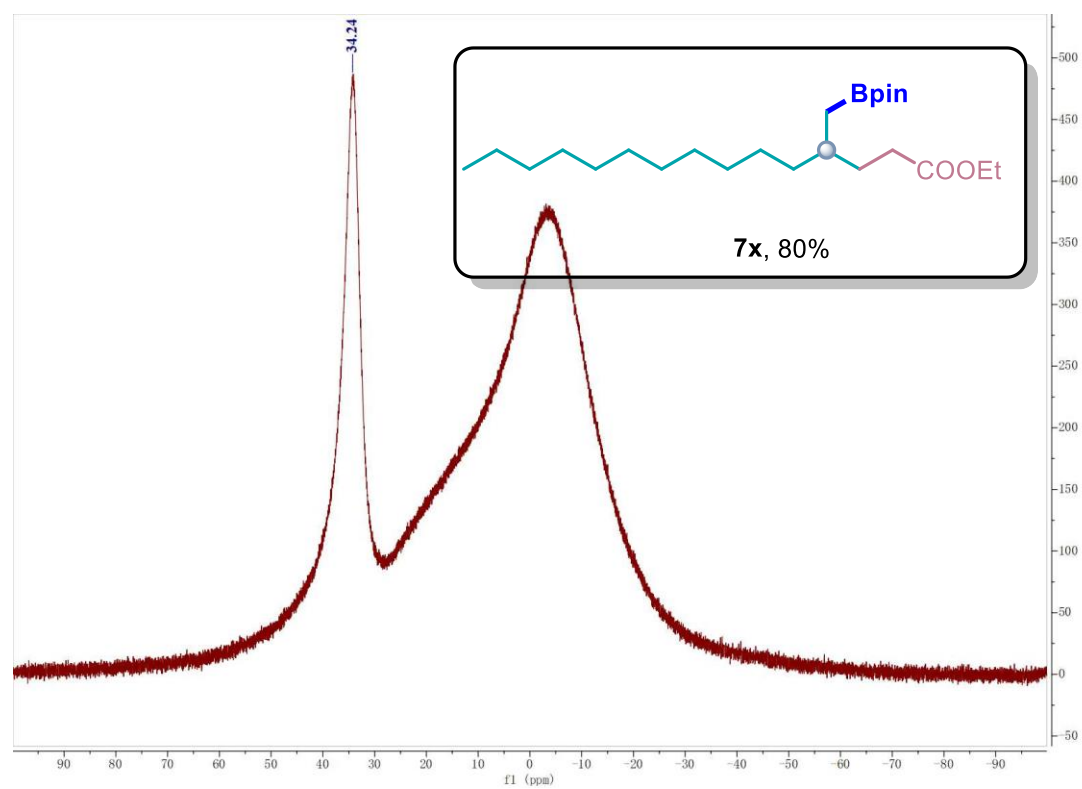
^1H NMR (500 MHz, Chloroform- d)



^{13}C NMR (126 MHz, Chloroform- d)

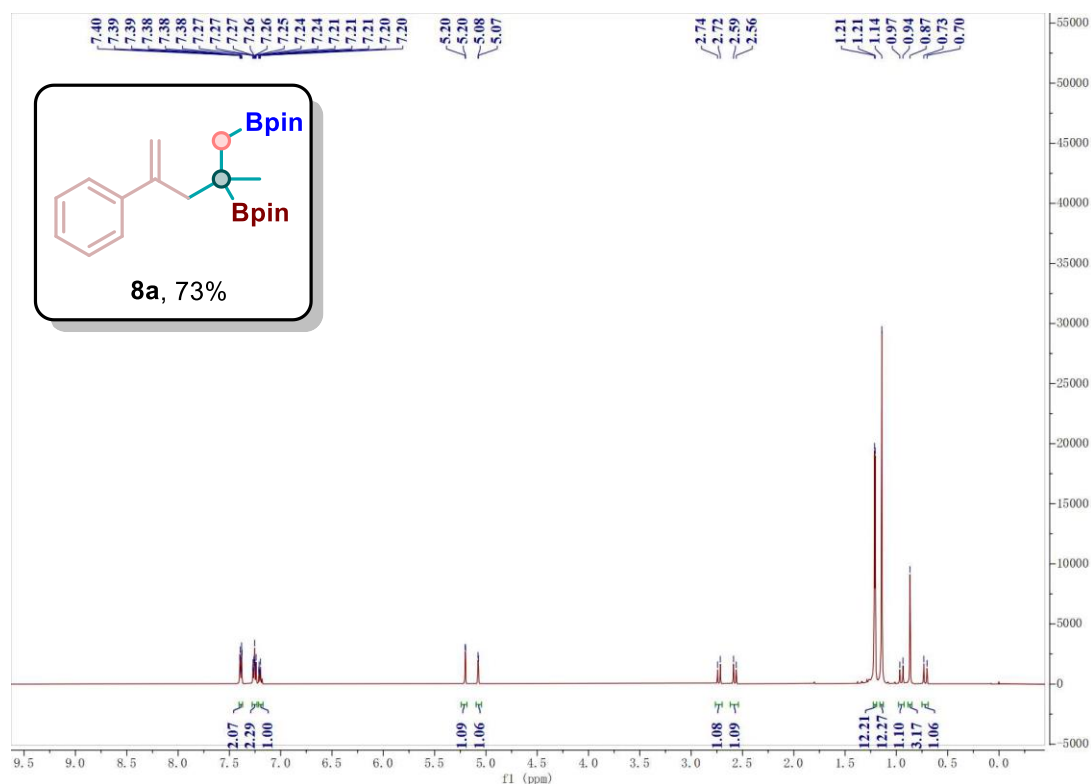


^{11}B NMR (160 MHz, Chloroform-*d*)

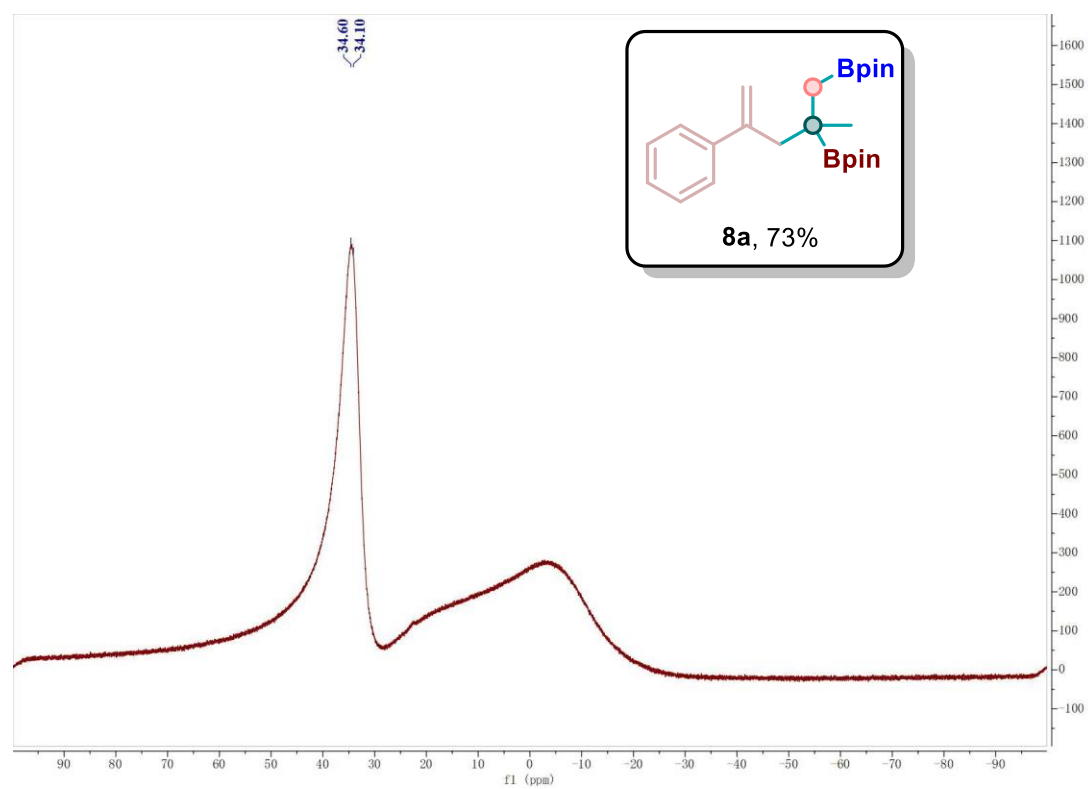


2,2'-(2-methyl-4-phenylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8a**)

^1H NMR (500 MHz, Chloroform-*d*)

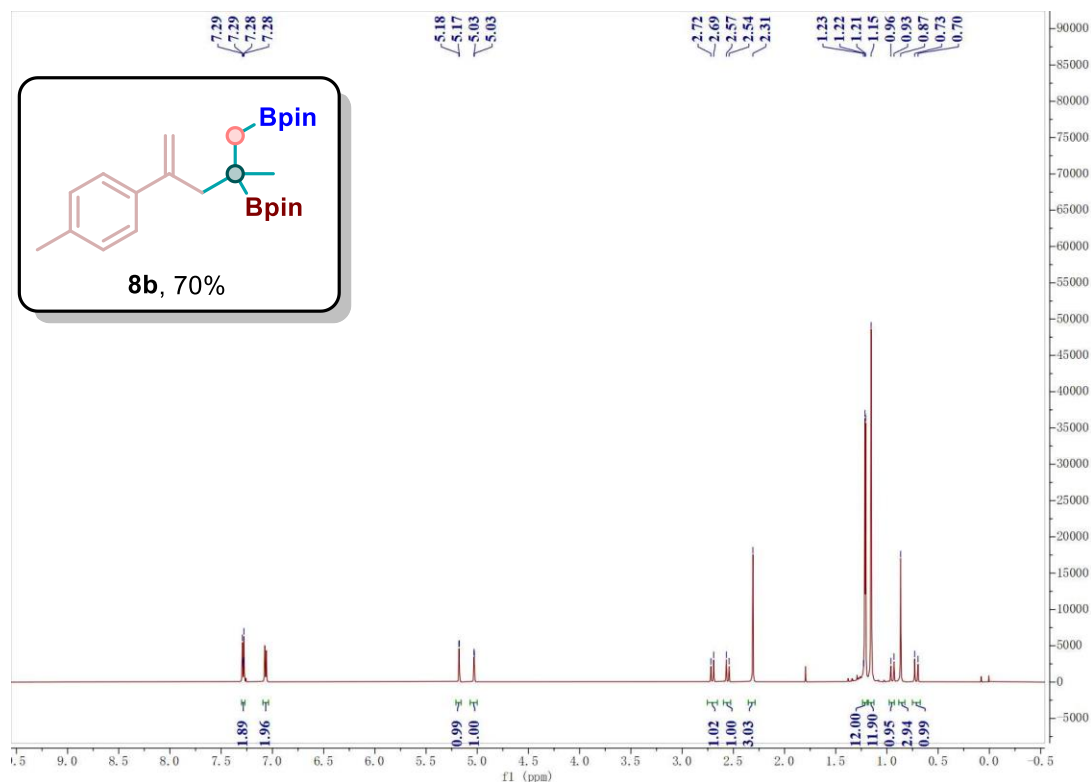


^{11}B NMR (160 MHz, Chloroform-*d*)

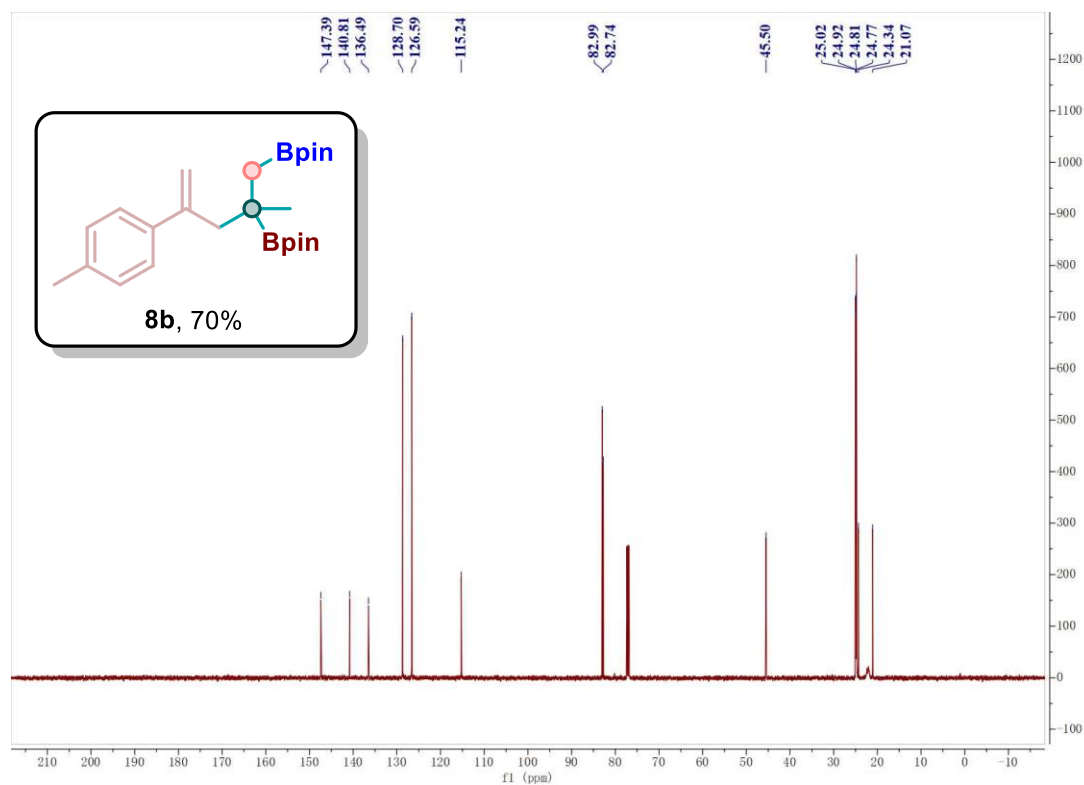


2,2'-(2-methyl-4-(p-tolyl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8b**)

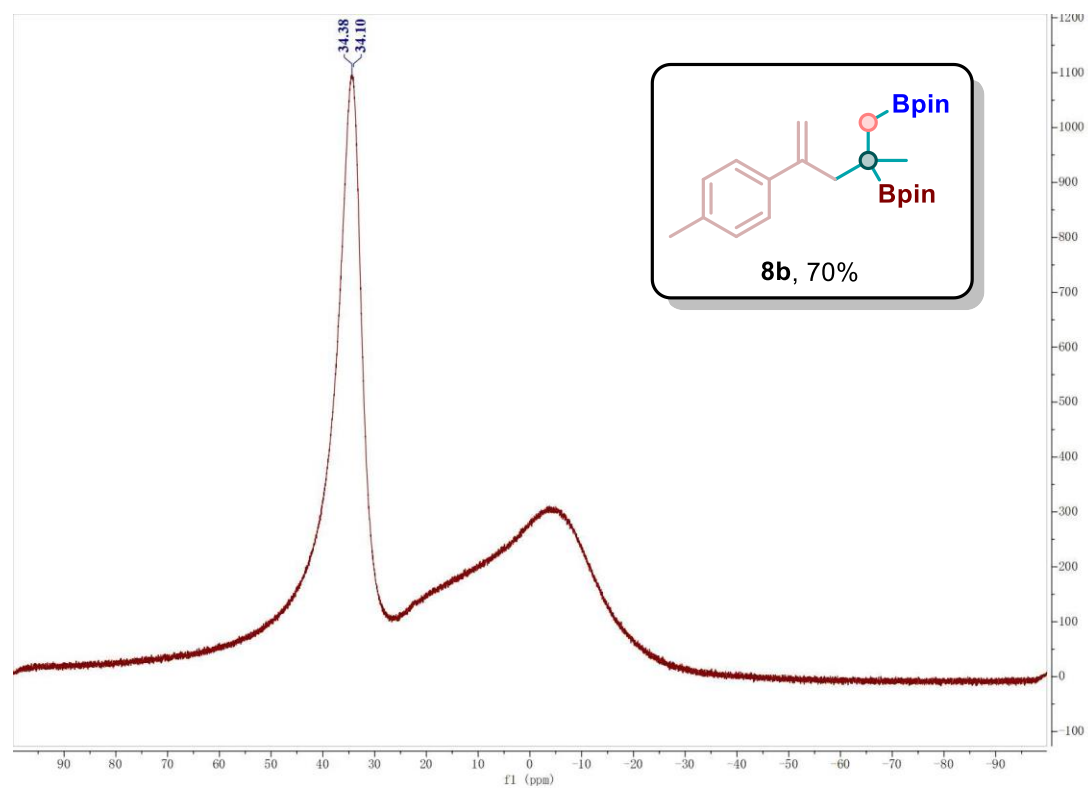
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

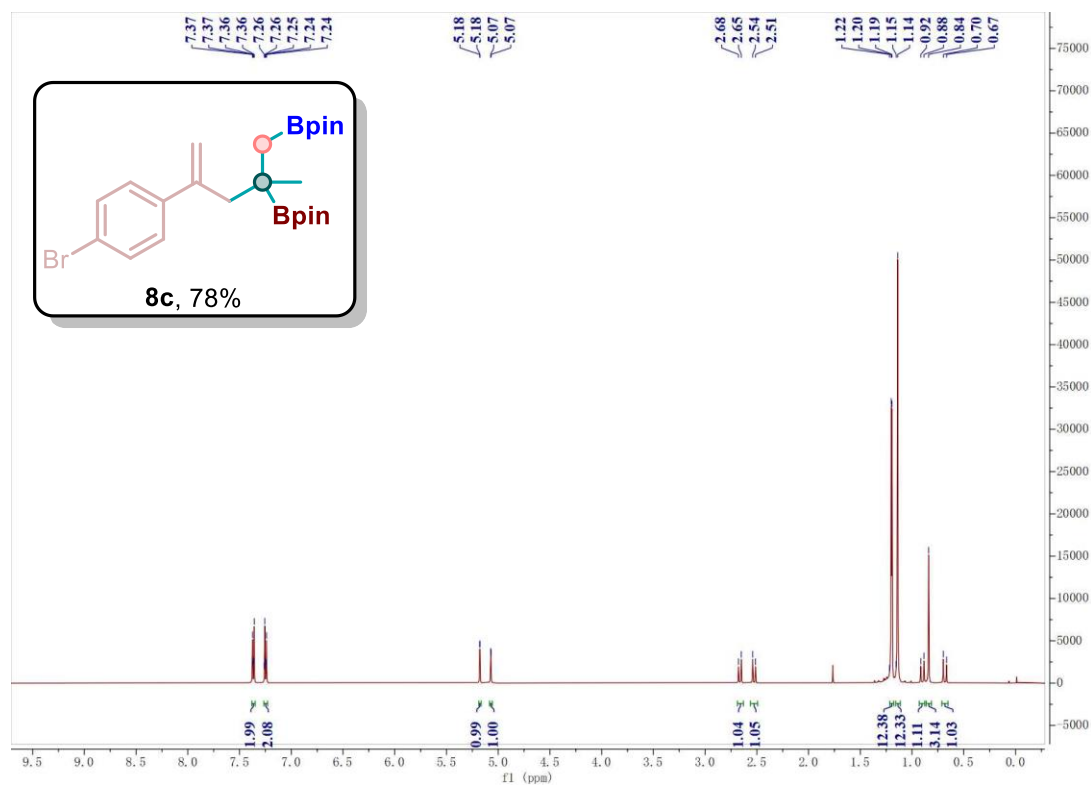


^{11}B NMR (160 MHz, Chloroform-*d*)

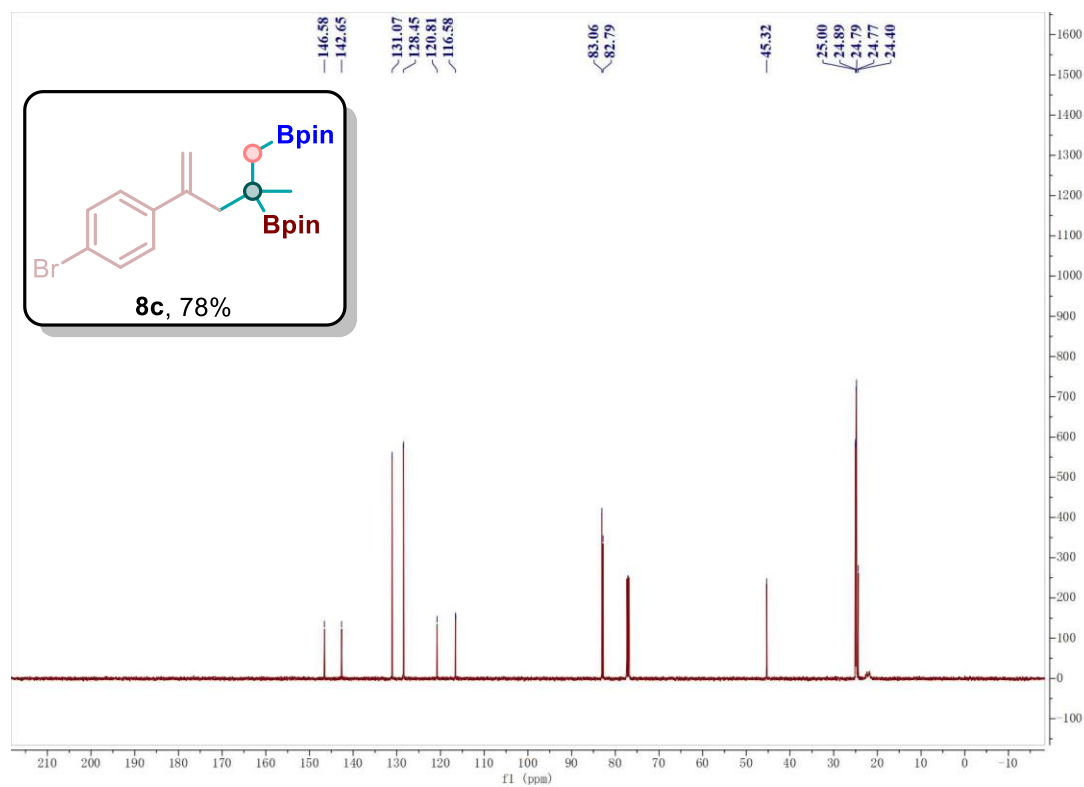


2,2'-(4-(4-bromophenyl)-2-methylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)
(**8c**)

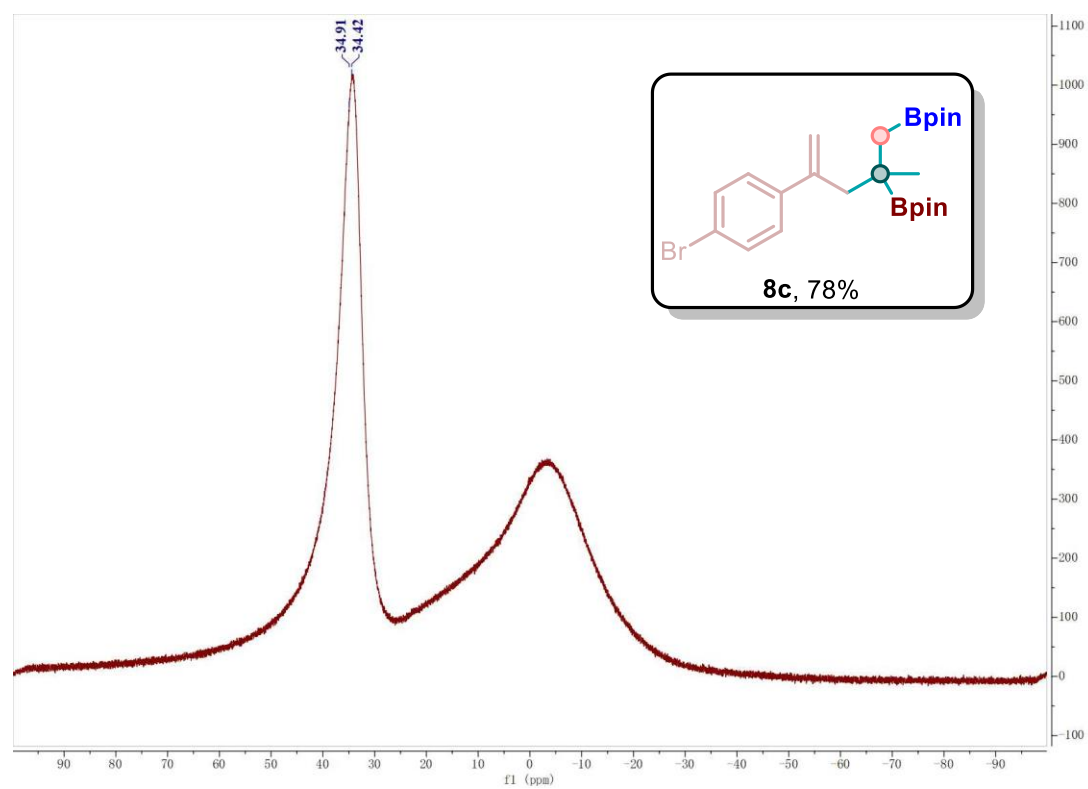
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

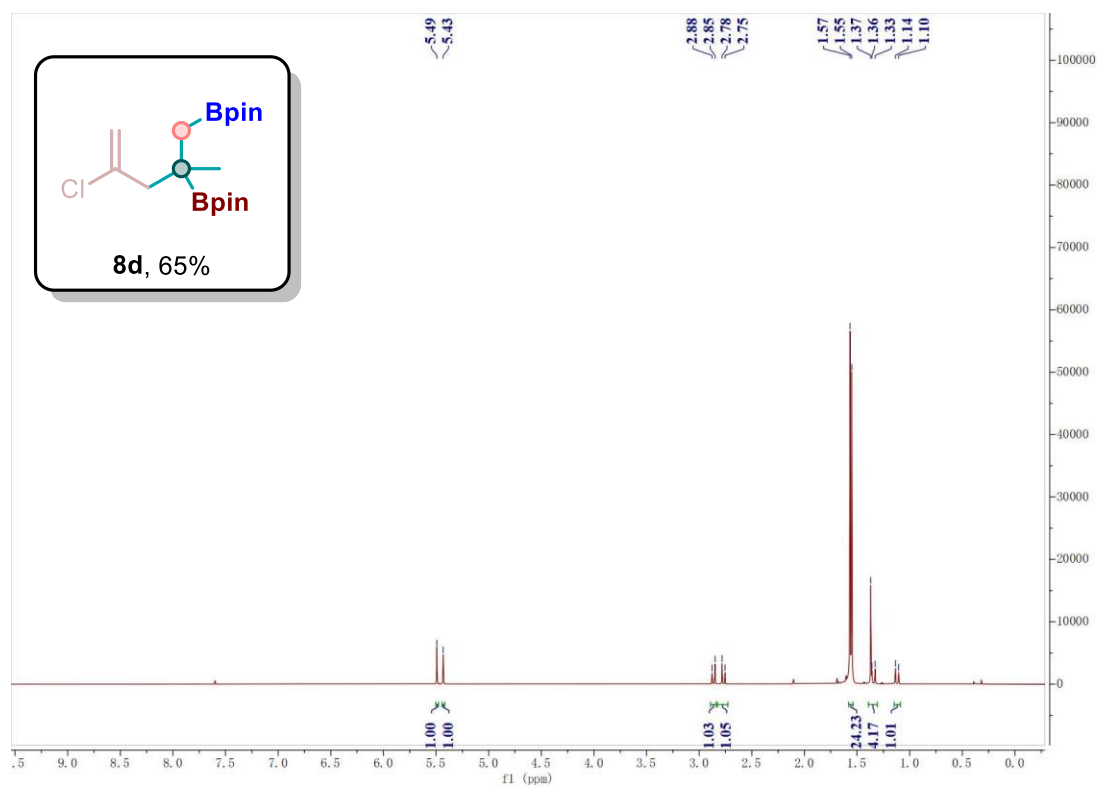


^{11}B NMR (160 MHz, Chloroform-*d*)

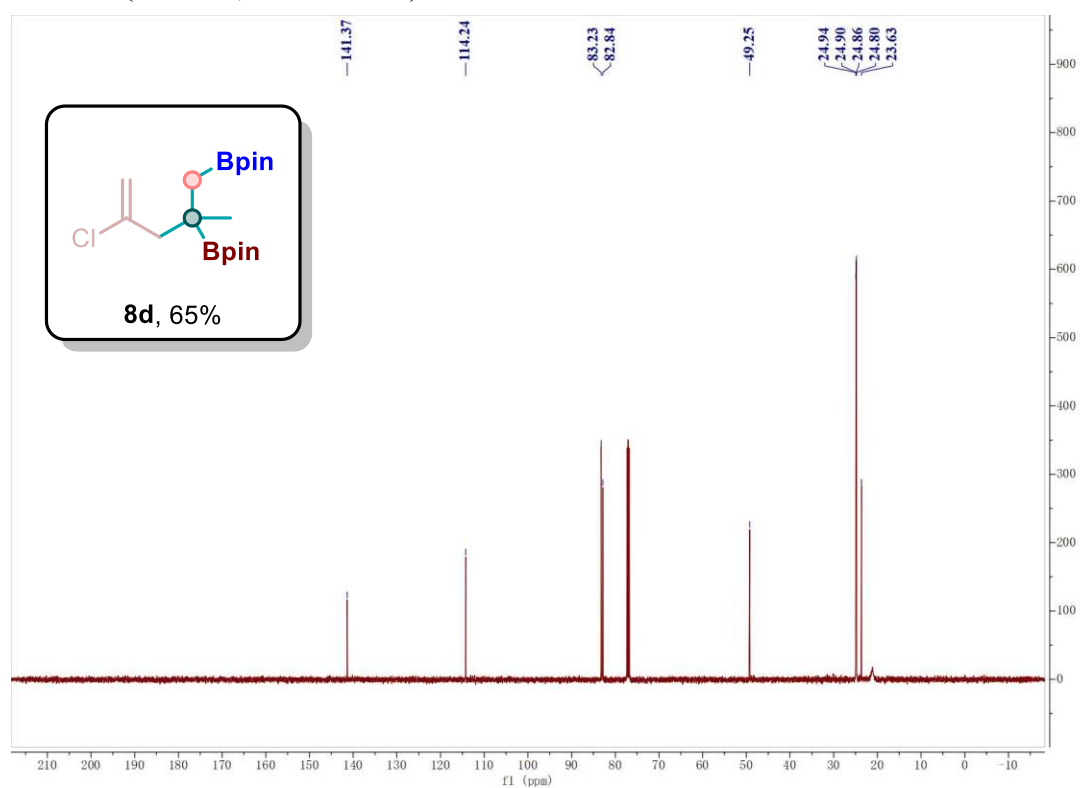


2,2'-(4-chloro-2-methylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8d)

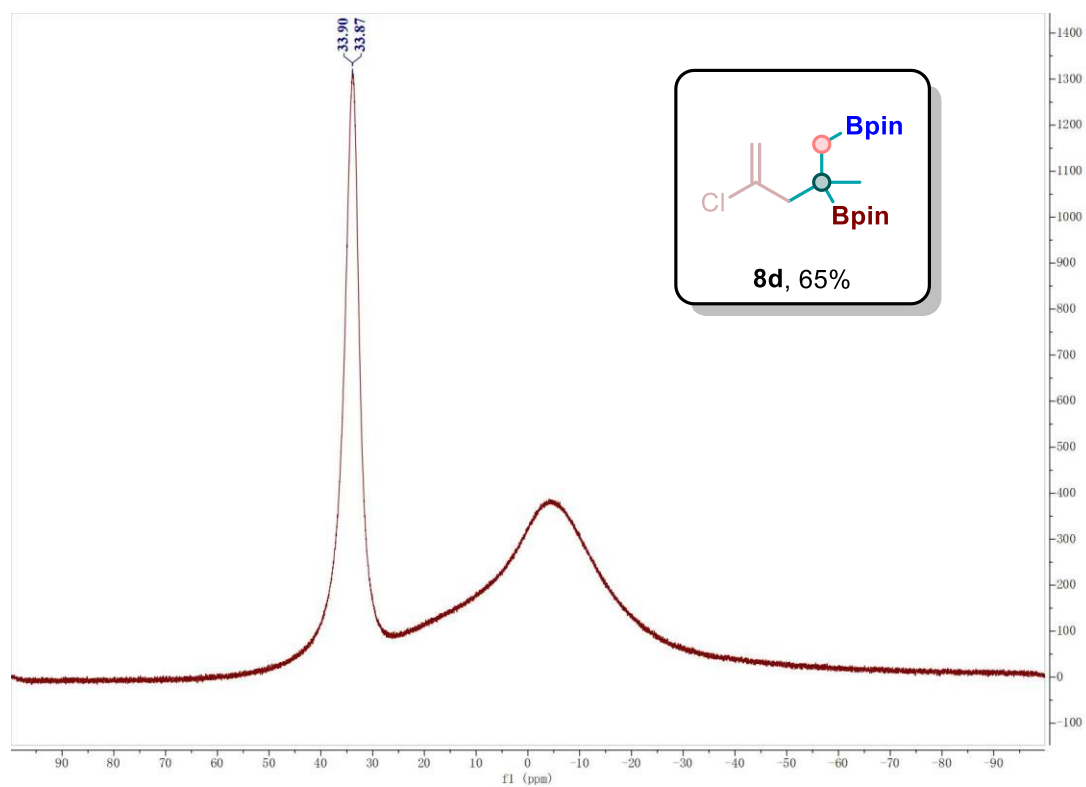
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

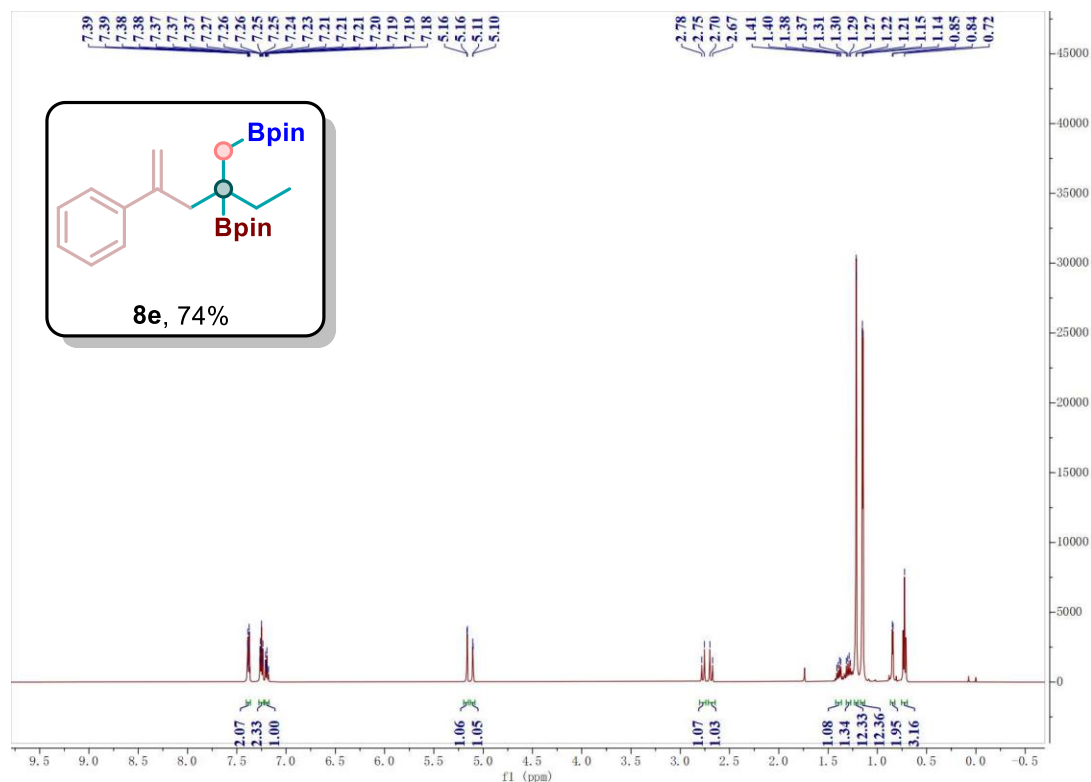


^{11}B NMR (160 MHz, Chloroform-*d*)

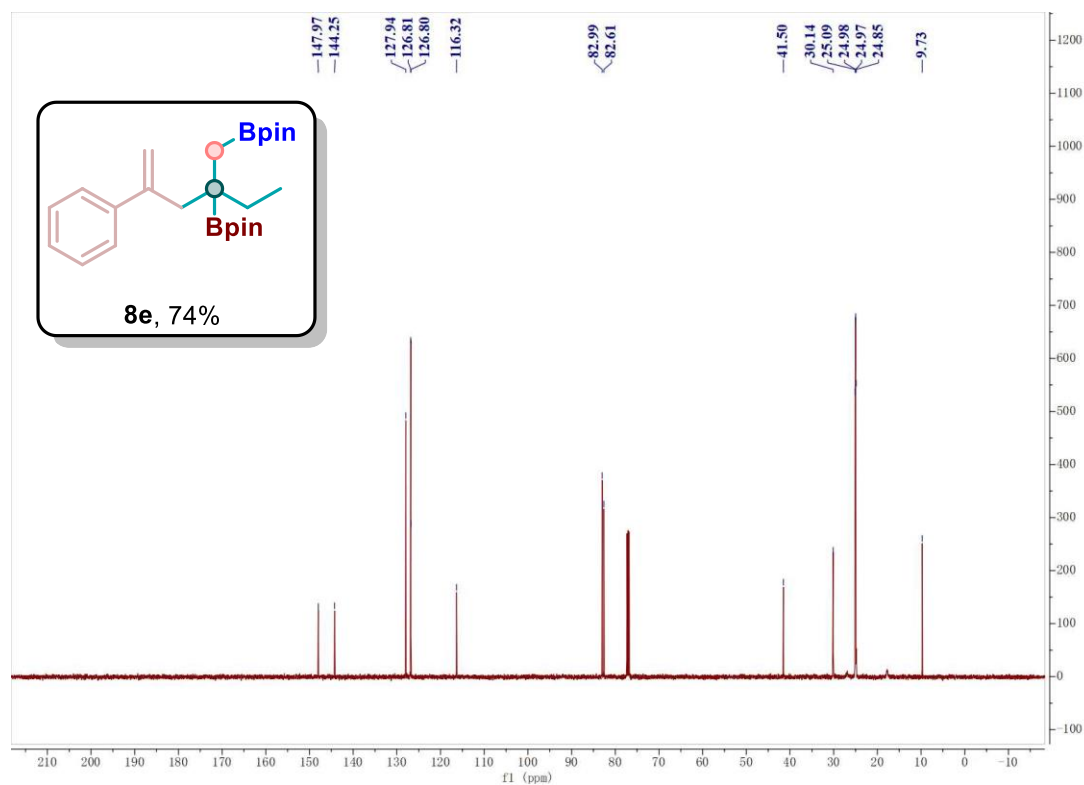


2,2'-(2-ethyl-4-phenylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8e**)

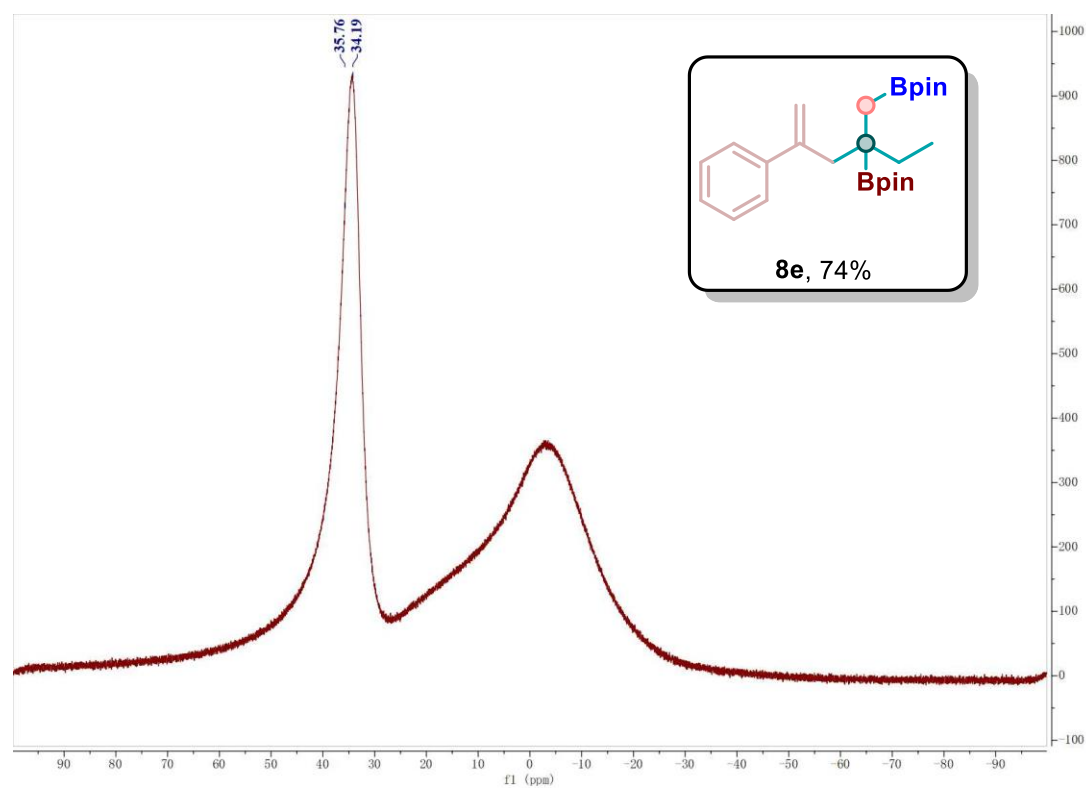
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

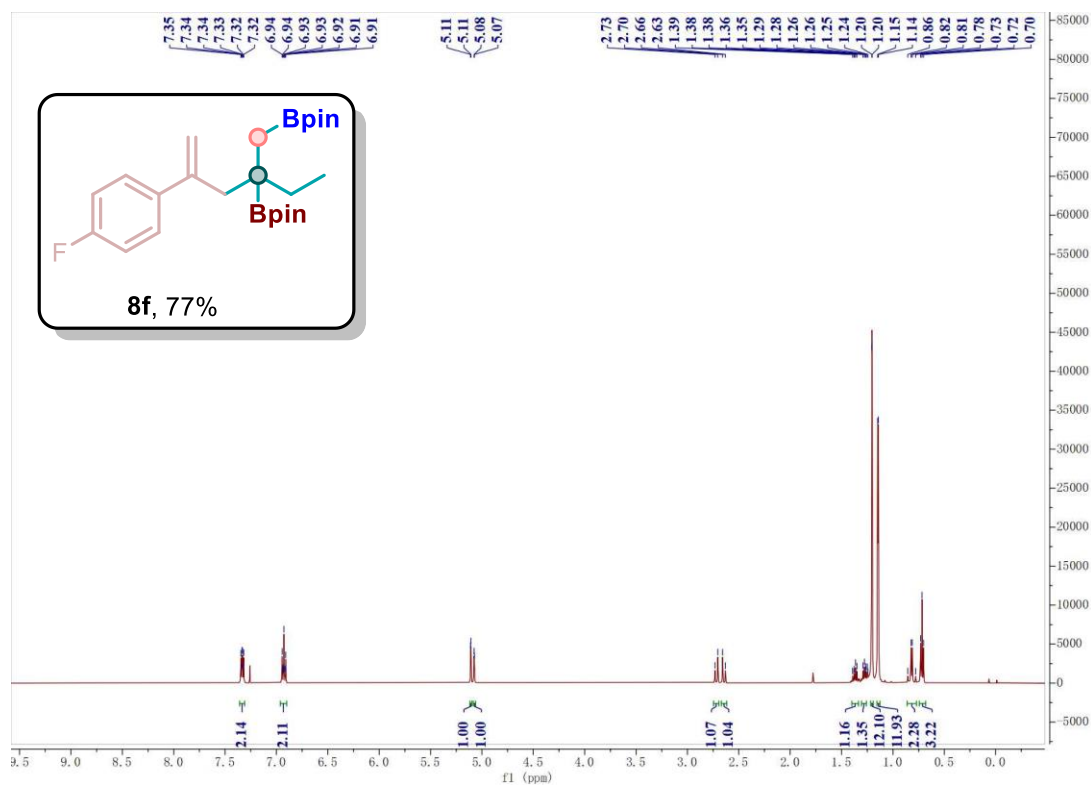


^{11}B NMR (160 MHz, Chloroform-*d*)

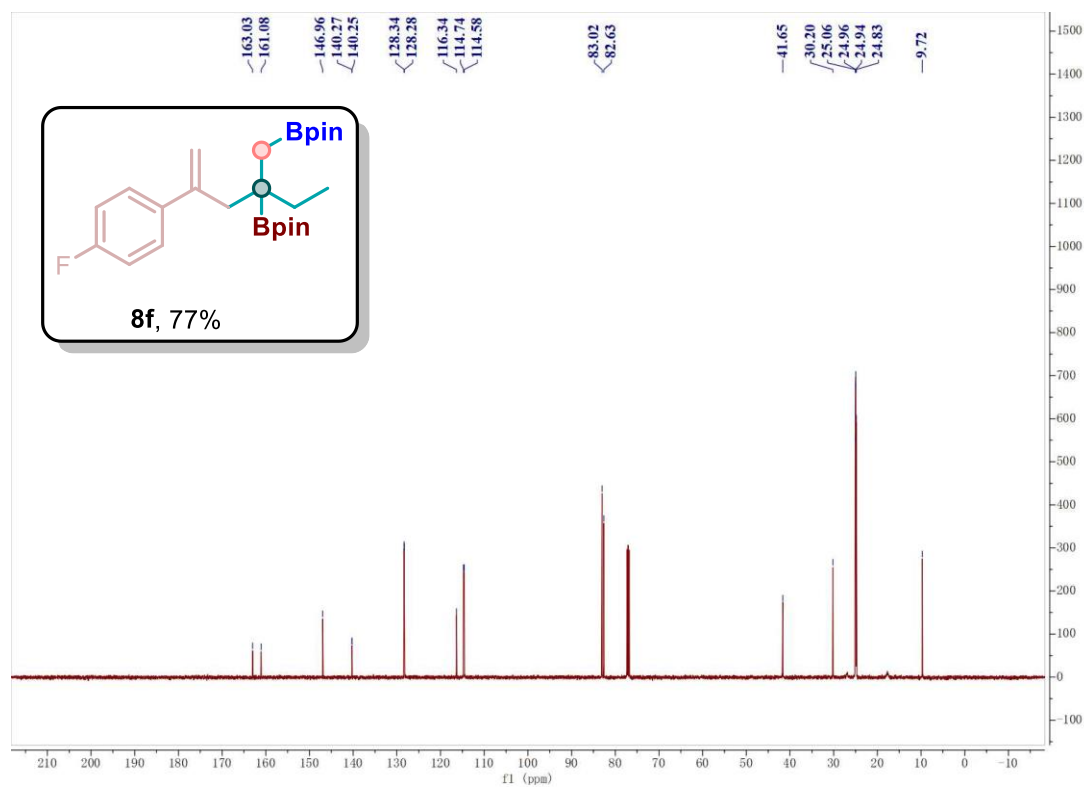


2,2'-(2-ethyl-4-(4-fluorophenyl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)
(8f)

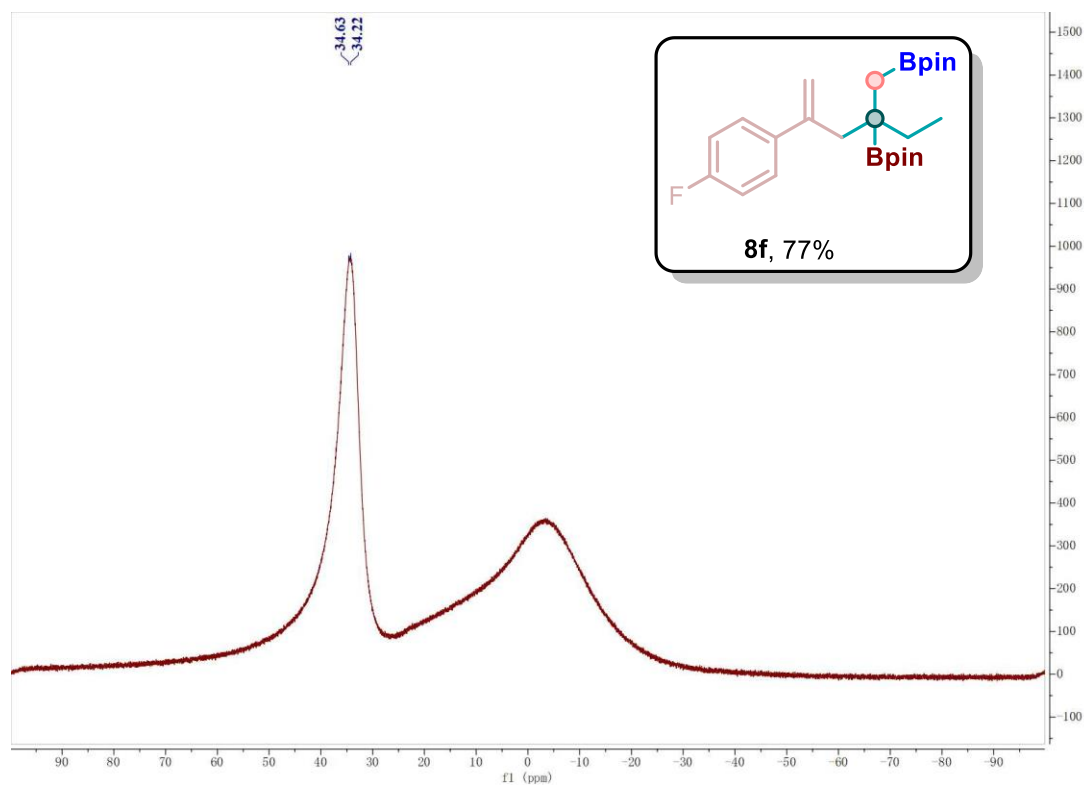
¹H NMR (500 MHz, Chloroform-*d*)



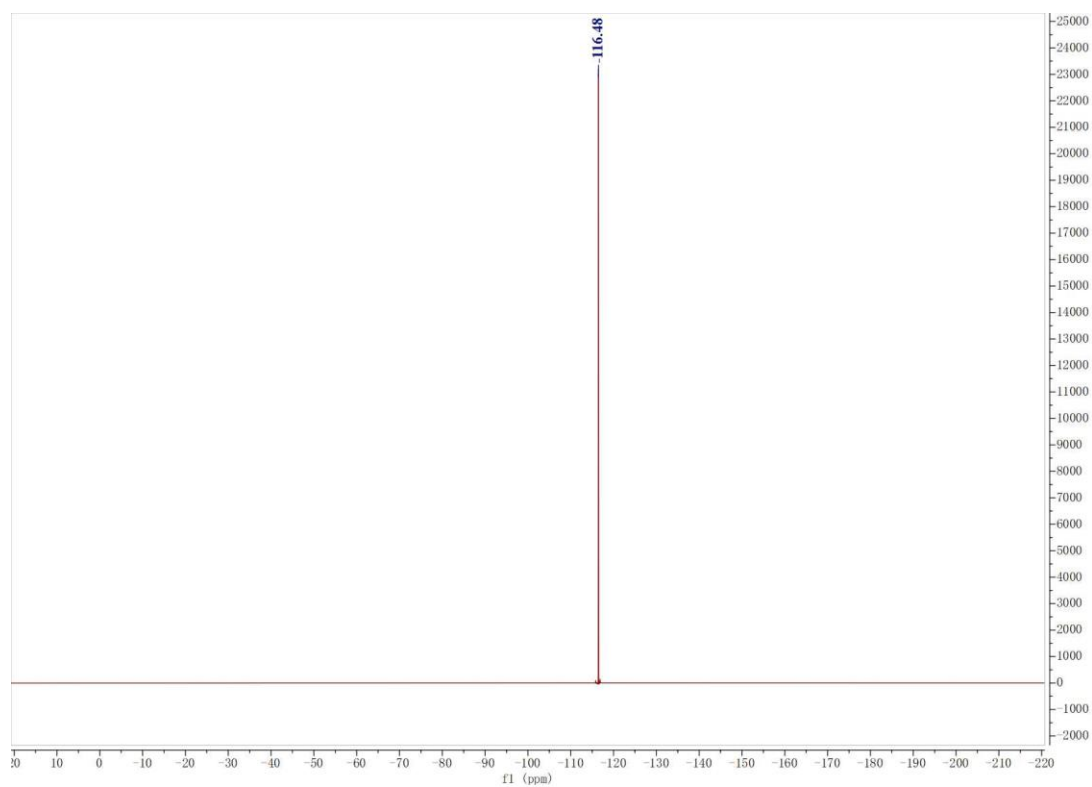
¹³C NMR (126 MHz, Chloroform-*d*)



^{11}B NMR (160 MHz, Chloroform-*d*)

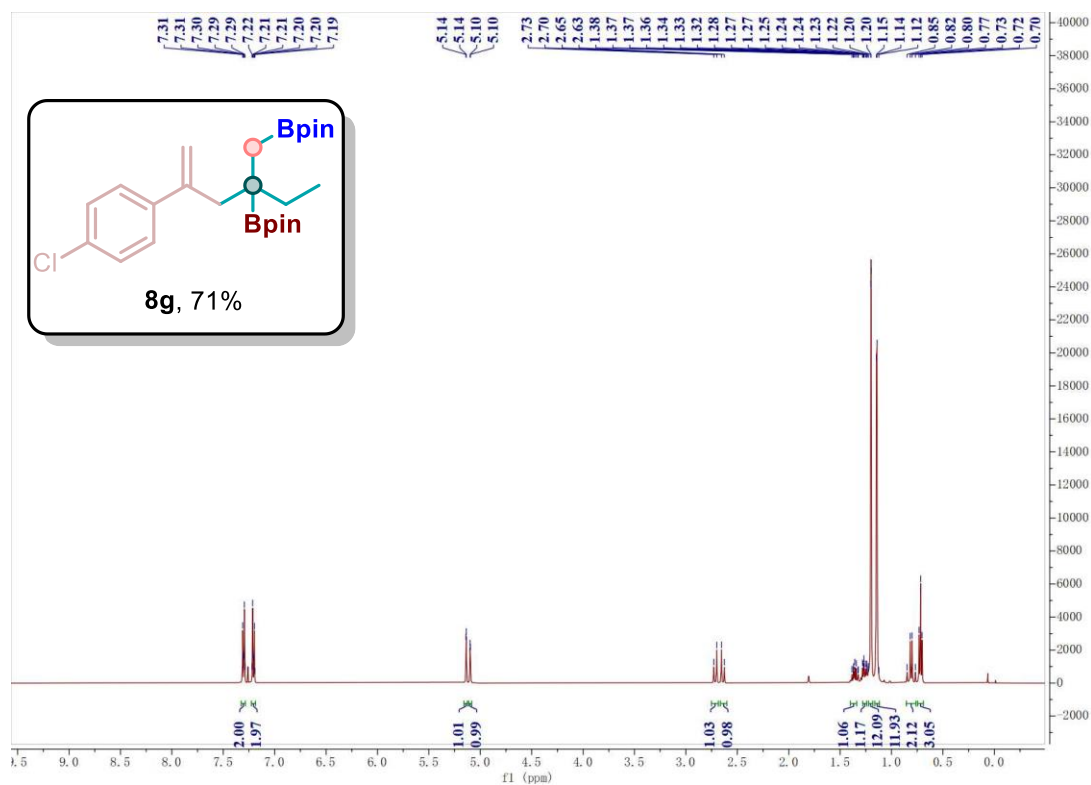


^{19}F NMR (471 MHz, Chloroform-*d*)

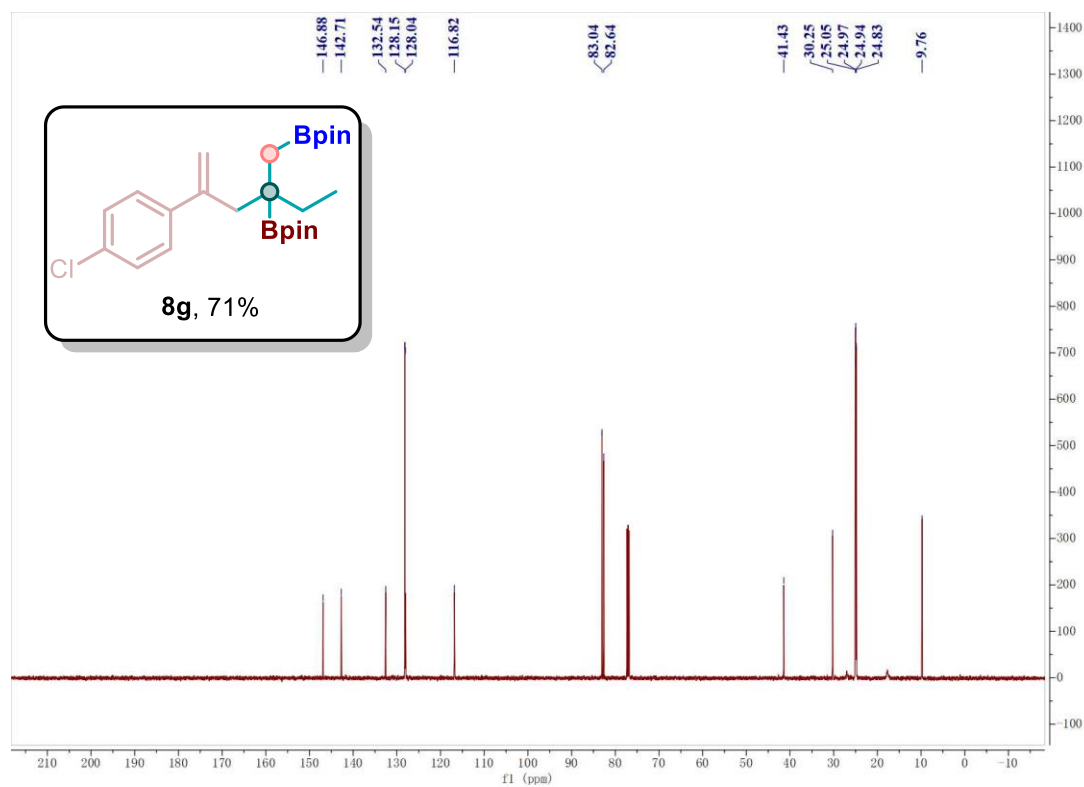


2,2'-(4-(4-chlorophenyl)-2-ethylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)
(8g)

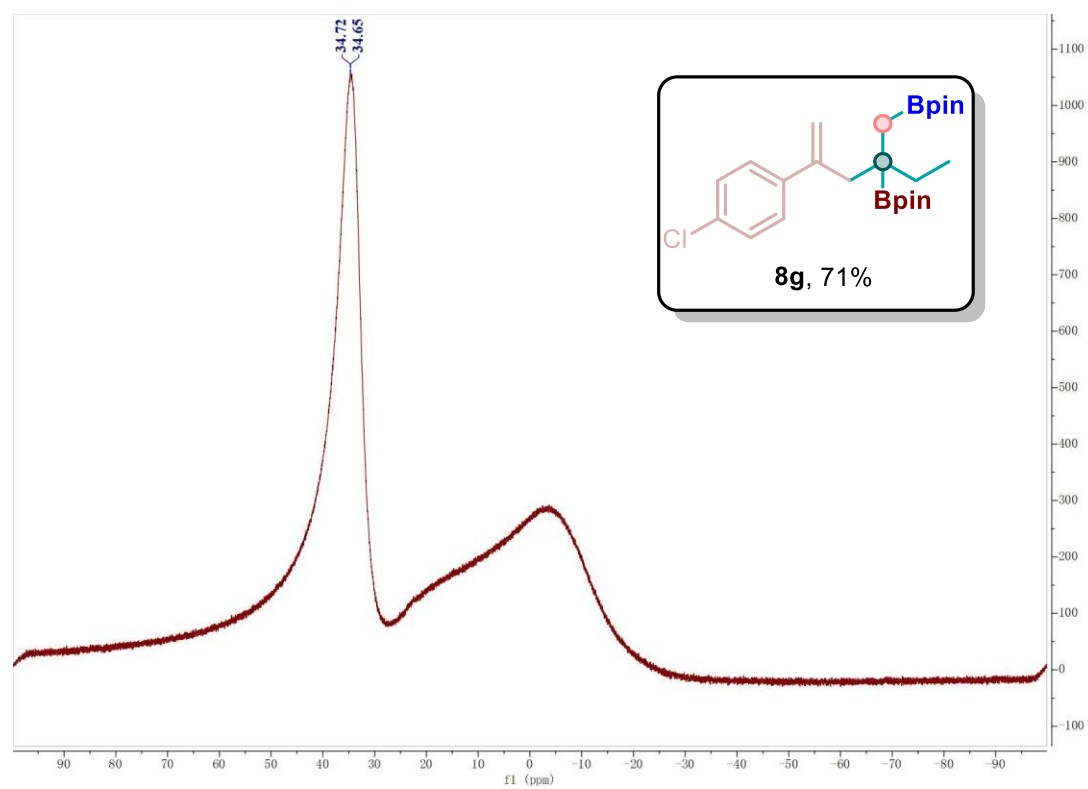
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

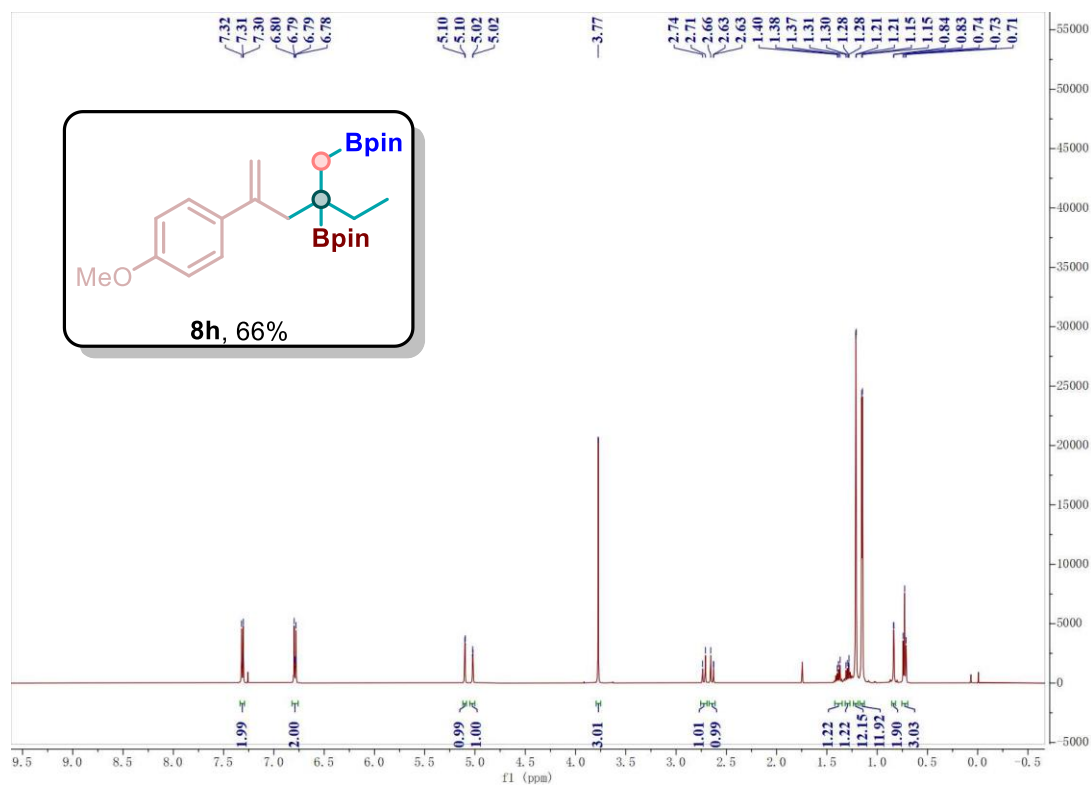


^{11}B NMR (160 MHz, Chloroform-*d*)

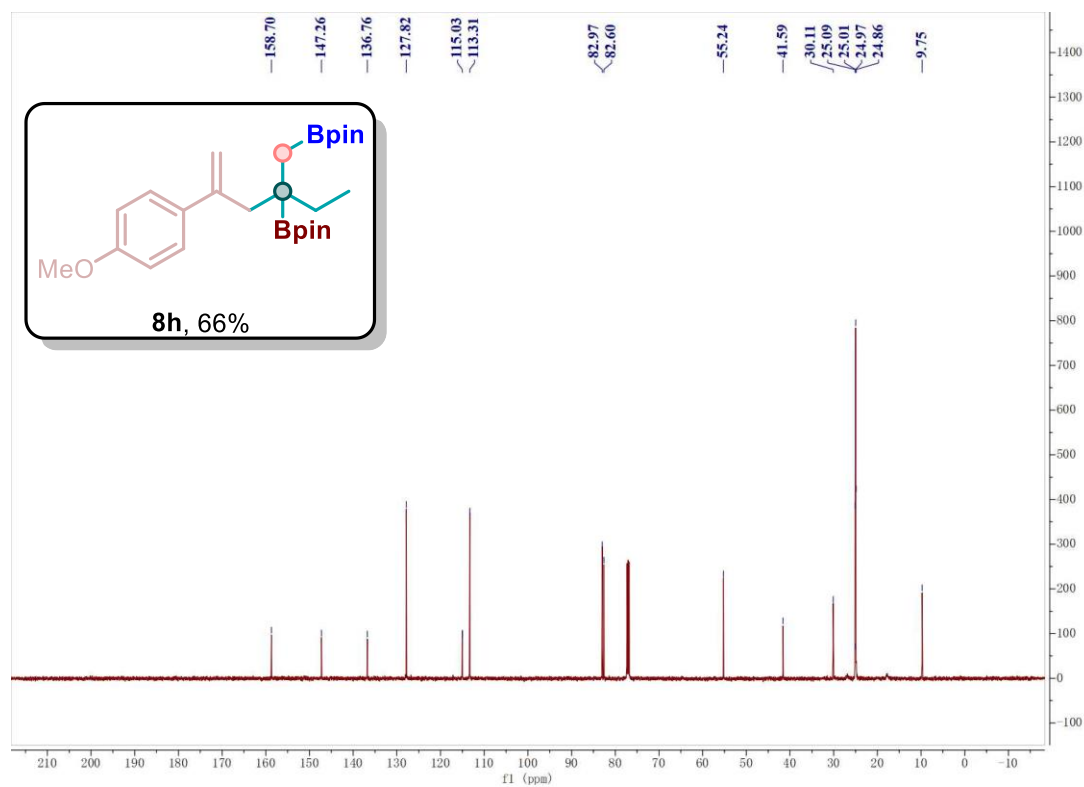


2,2'-(2-ethyl-4-(4-methoxyphenyl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)
(8h)

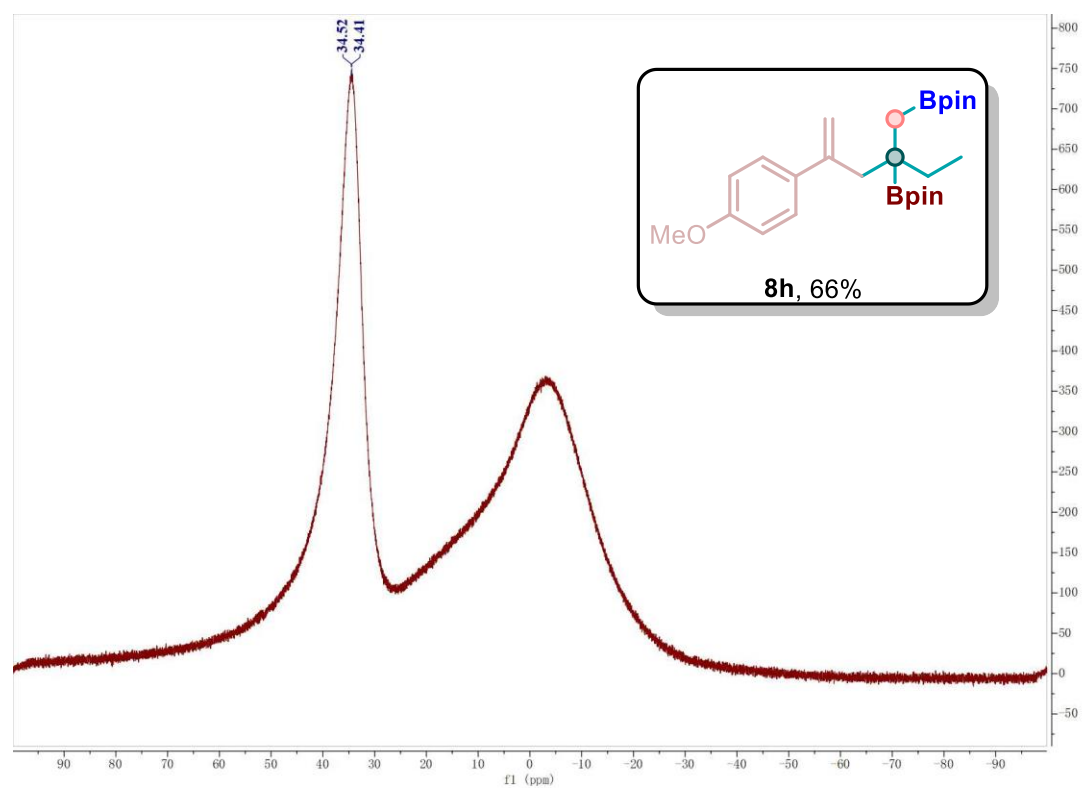
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

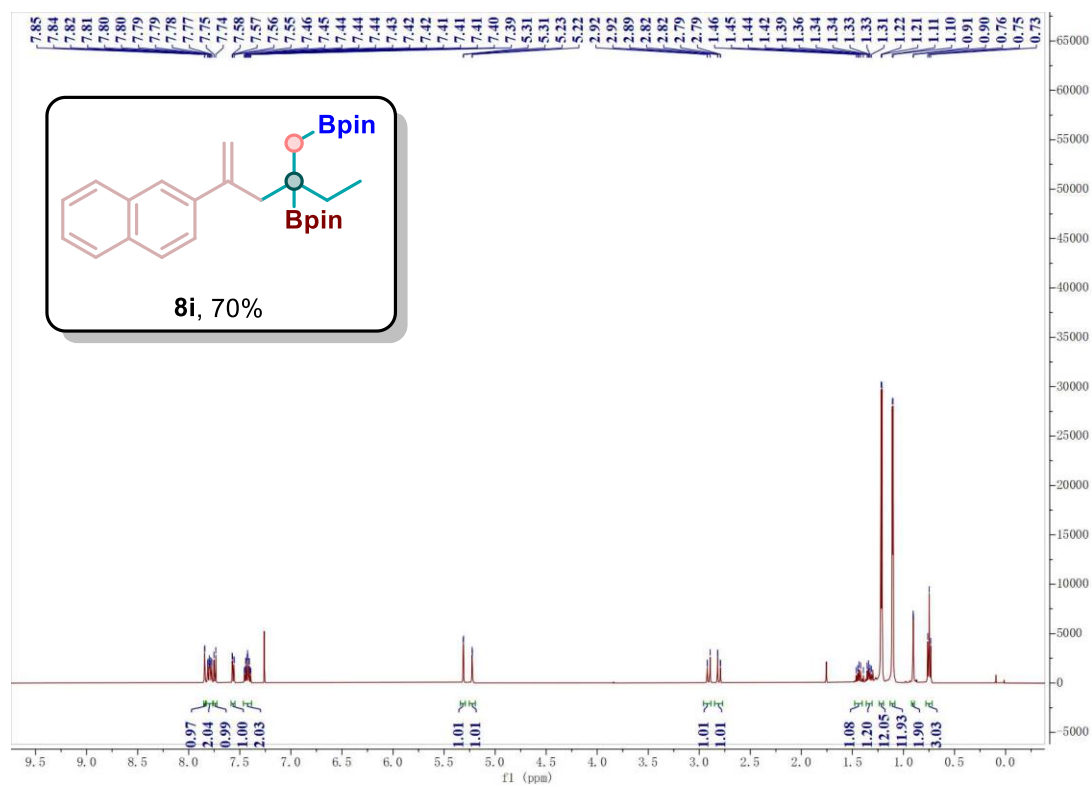


^{11}B NMR (160 MHz, Chloroform-*d*)

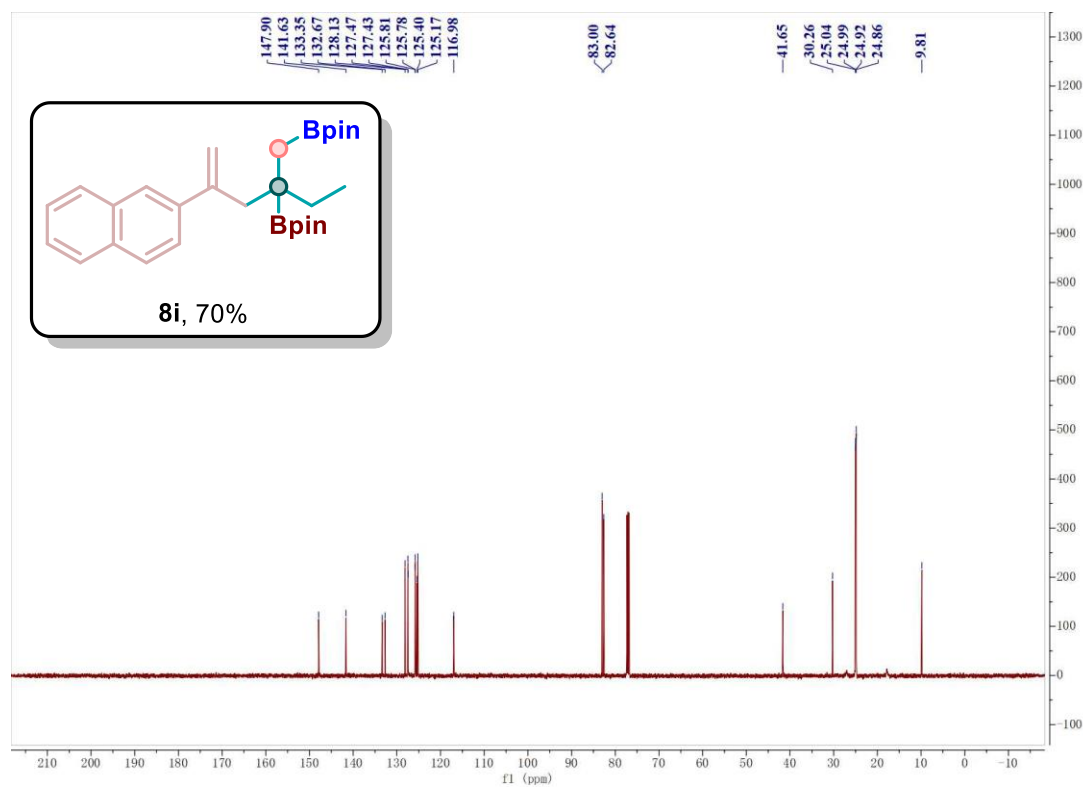


2,2'-(2-ethyl-4-(naphthalen-2-yl)pent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane)
(8i)

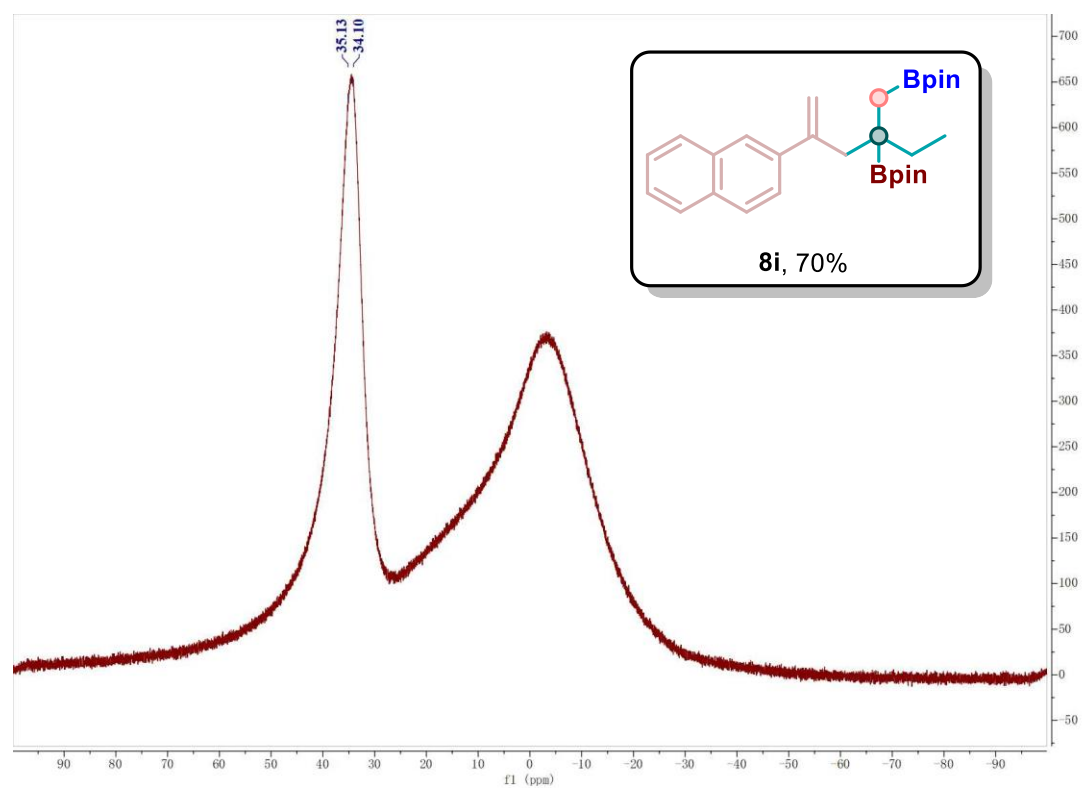
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

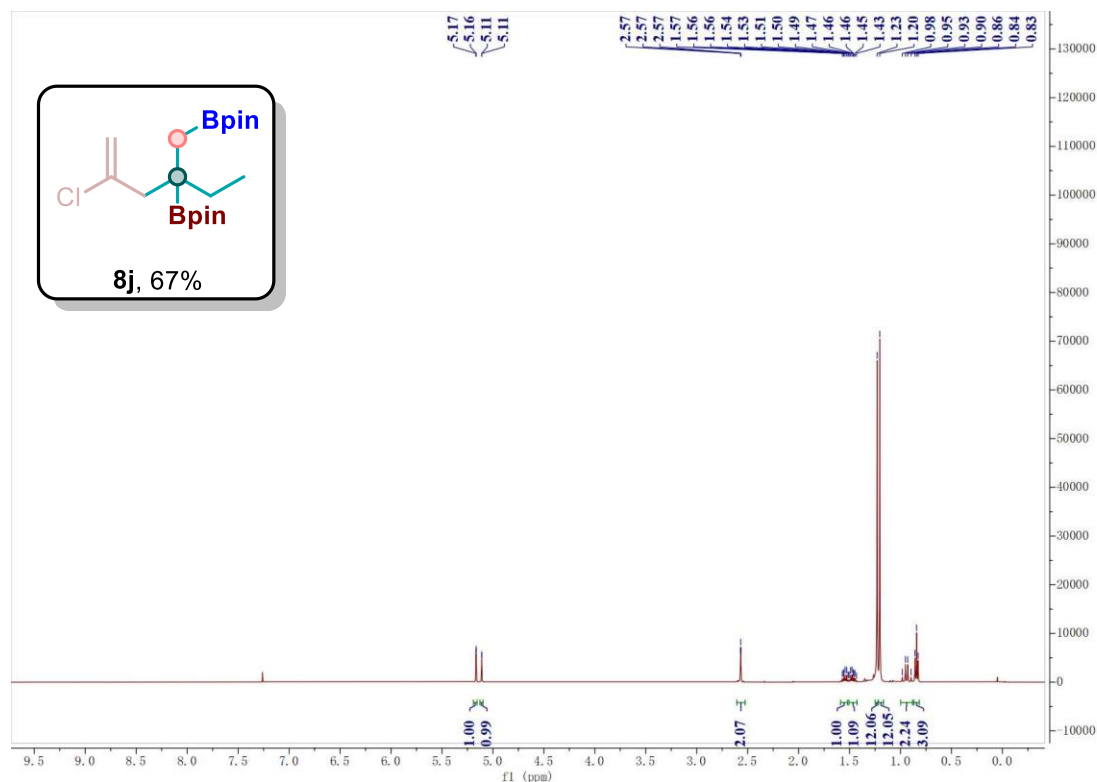


^{11}B NMR (160 MHz, Chloroform-*d*)

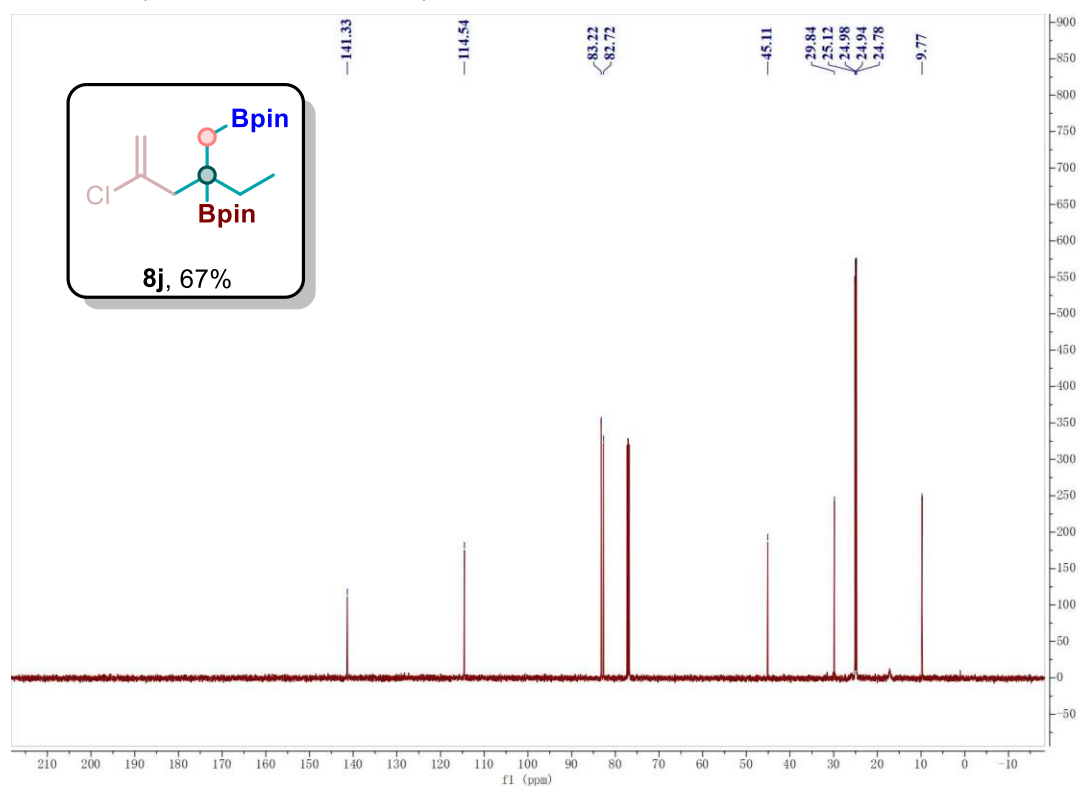


2,2'-(4-chloro-2-ethylpent-4-ene-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8j**)

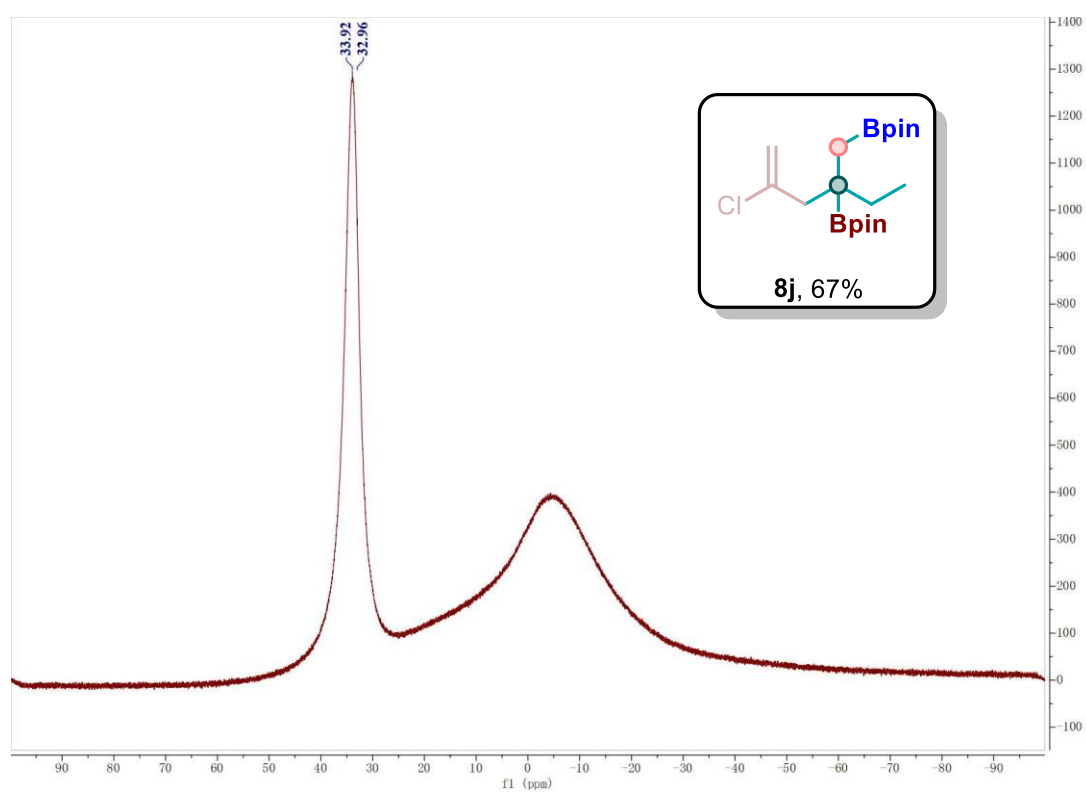
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

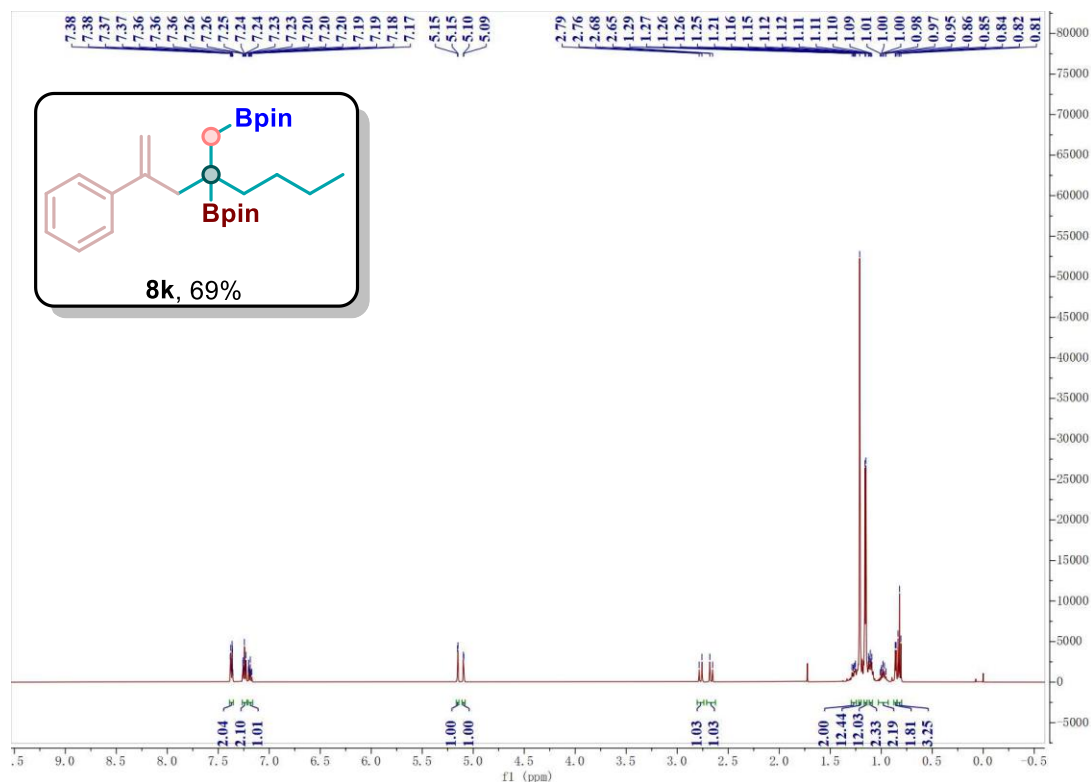


^{11}B NMR (160 MHz, Chloroform-*d*)

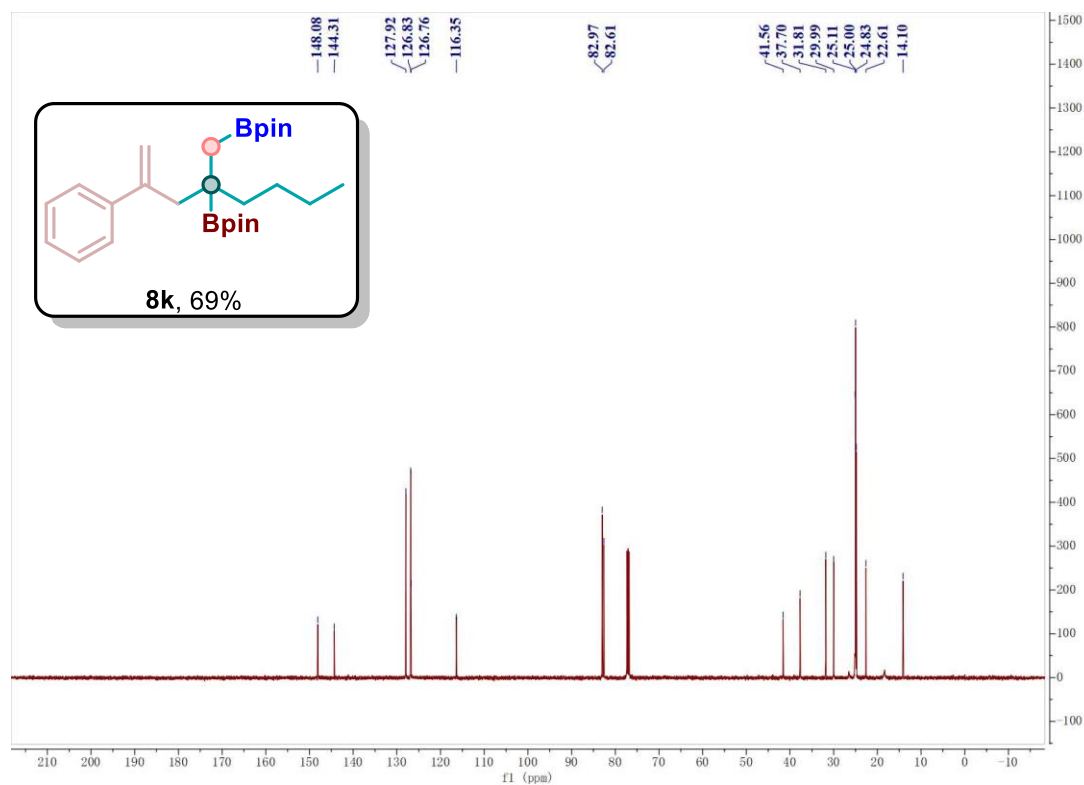


2,2'-(2-(2-phenylallyl)hexane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (8k)

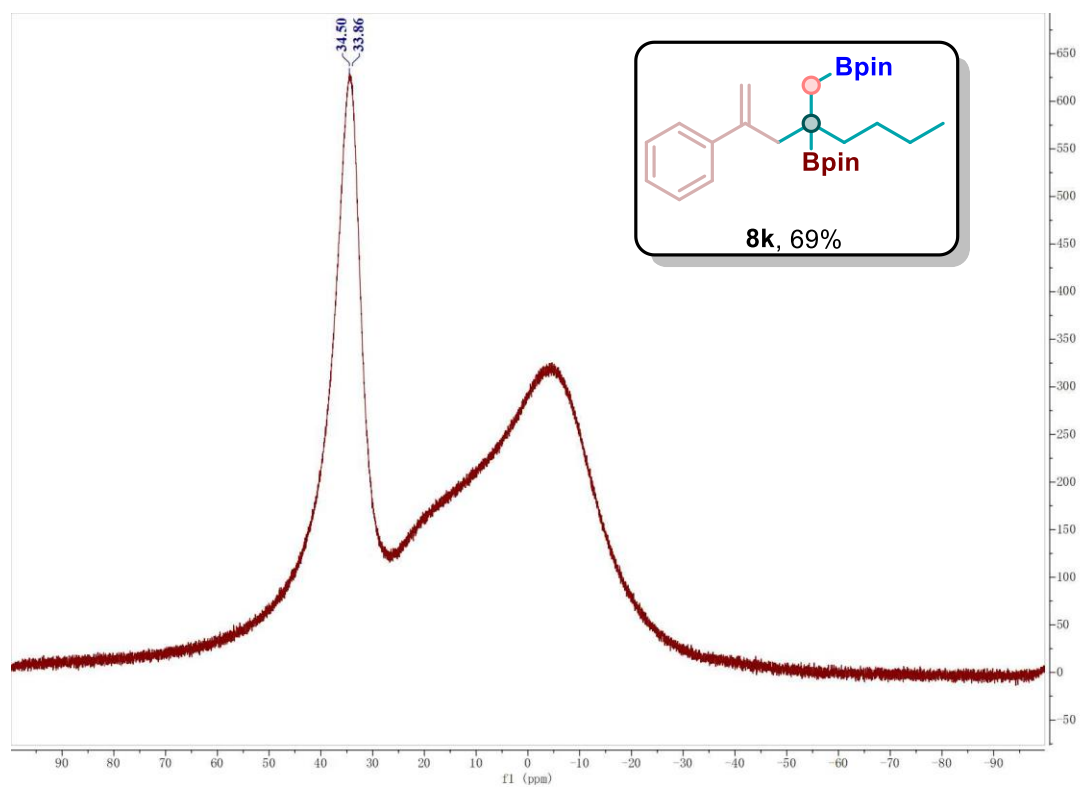
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)

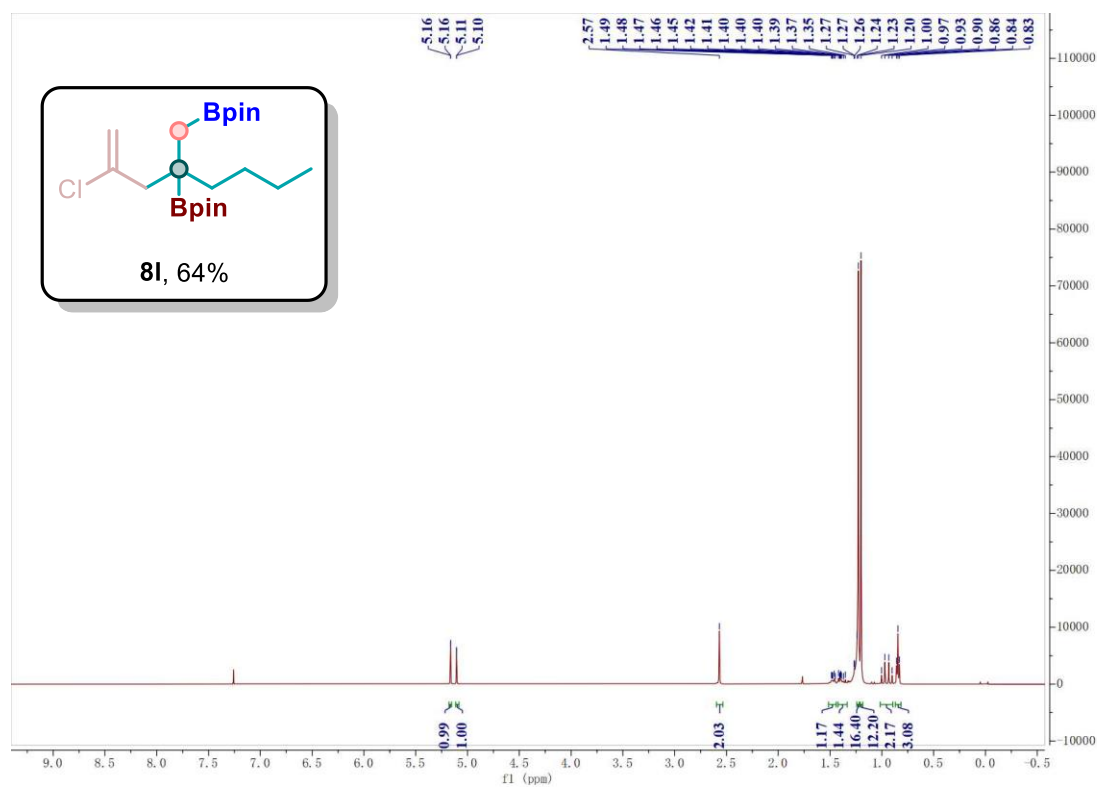


^{11}B NMR (160 MHz, Chloroform-*d*)

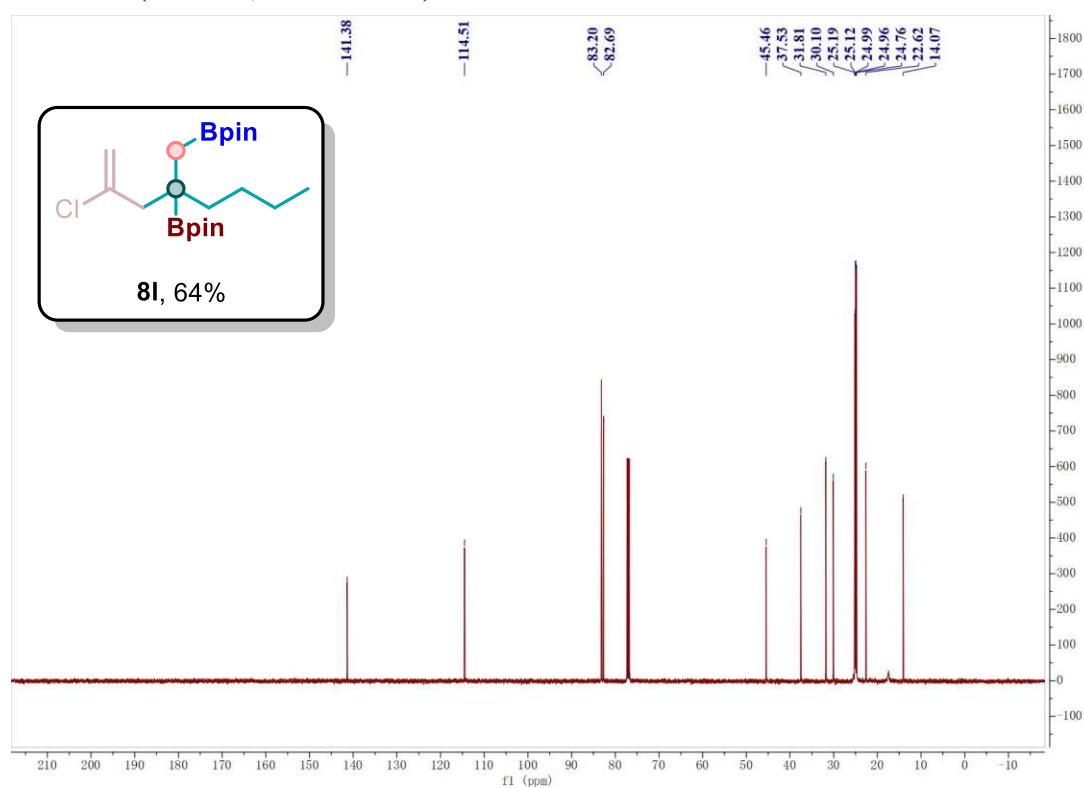


2,2'-(2-(2-chloroallyl)hexane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (**8l**)

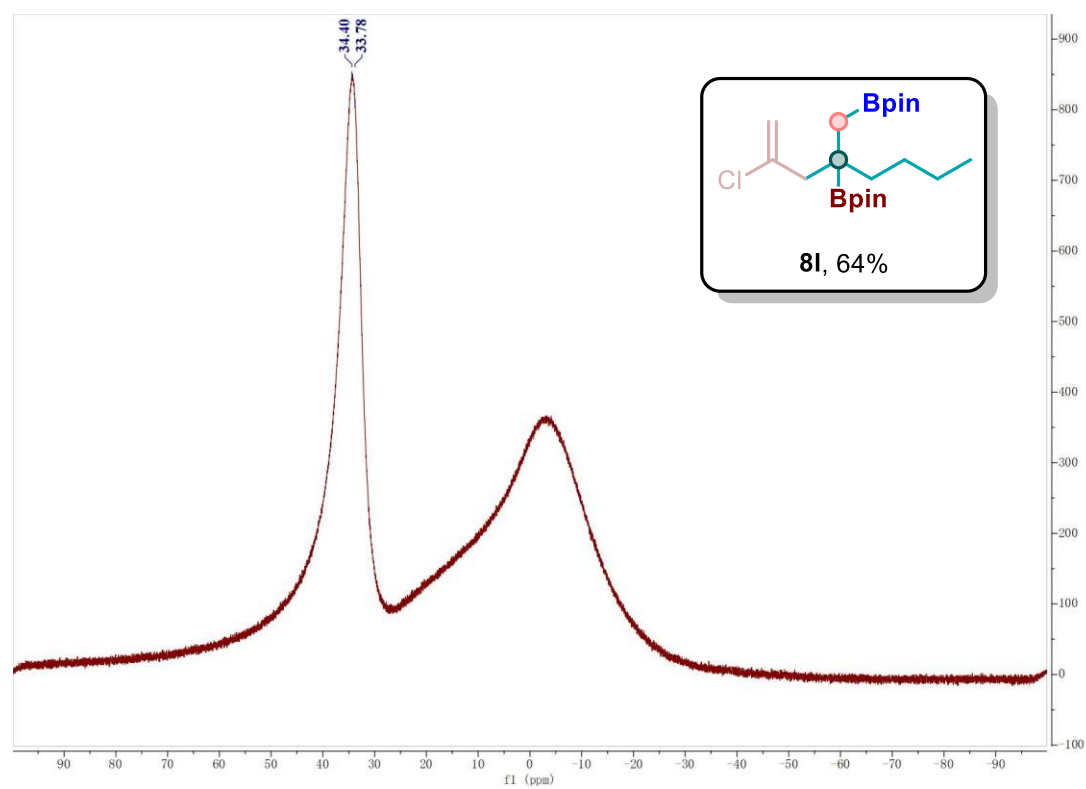
^1H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)

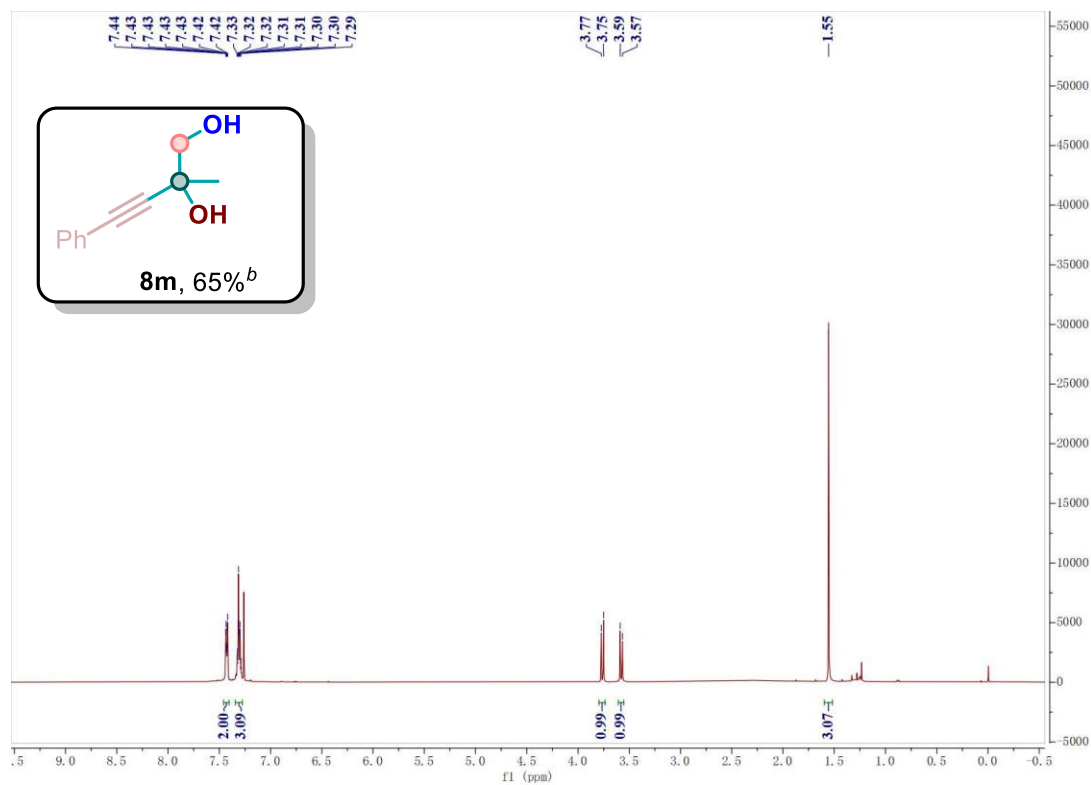


^{11}B NMR (160 MHz, Chloroform-*d*)

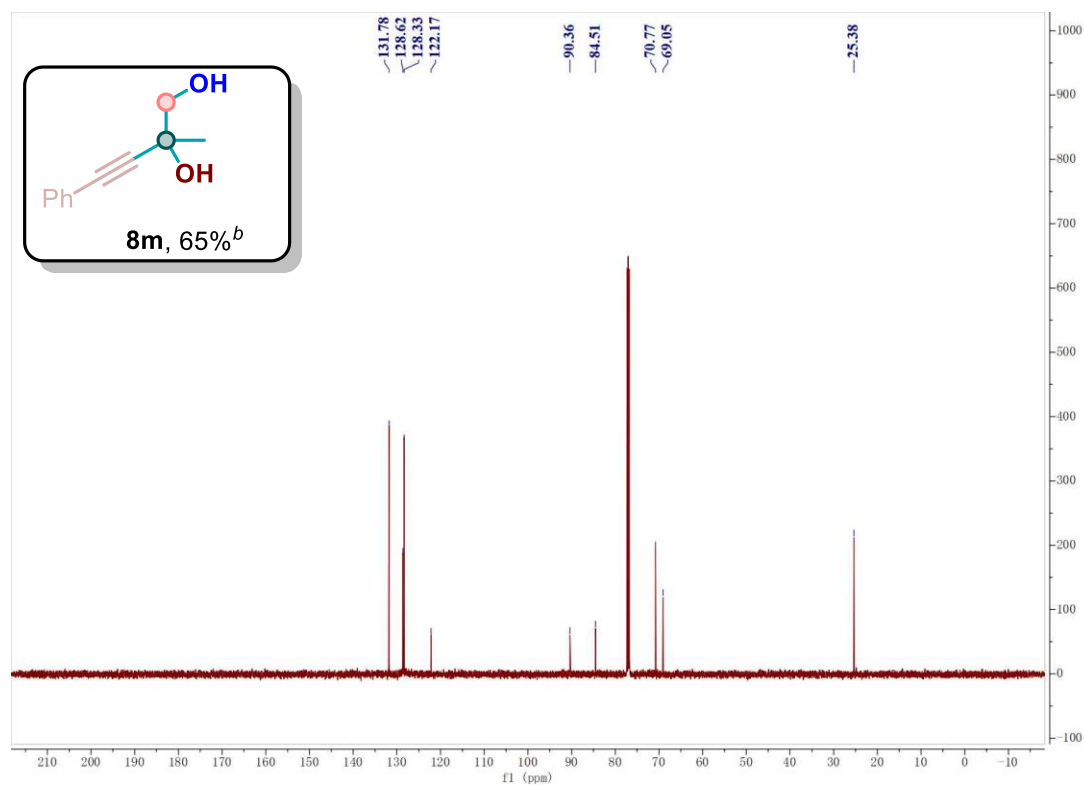


2-methyl-4-phenylbut-3-yn-1,2-diol (8m)

¹H NMR (500 MHz, Chloroform-*d*)

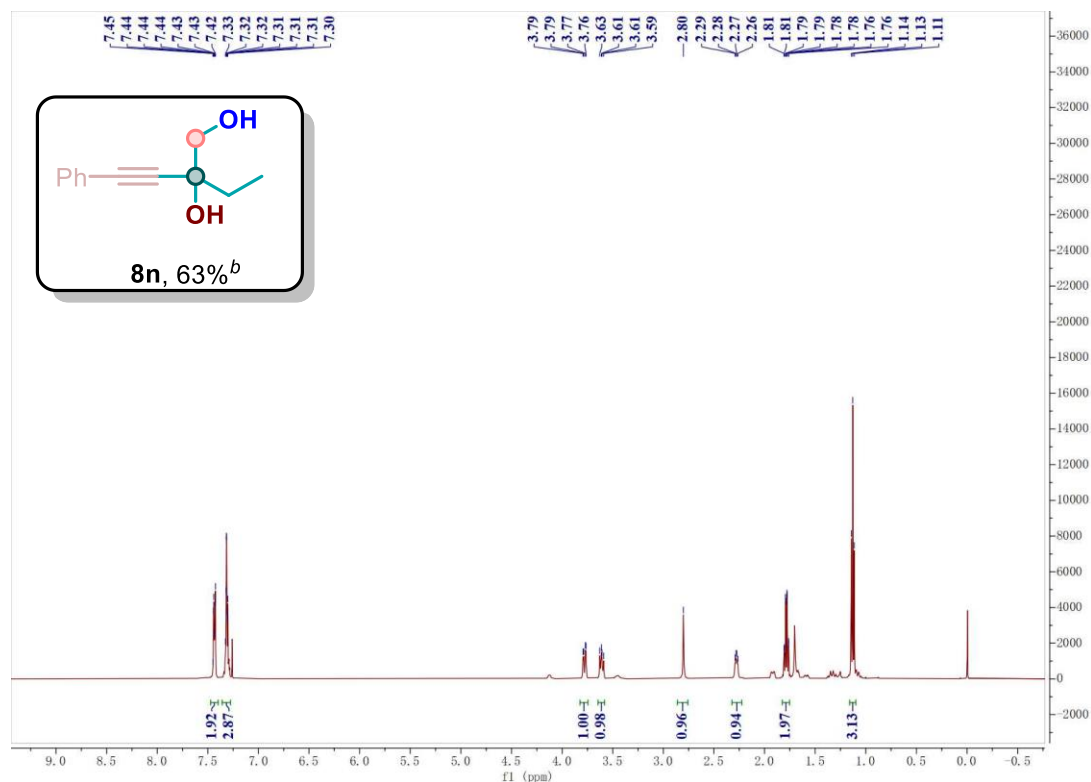


¹³C NMR (126 MHz, Chloroform-*d*)

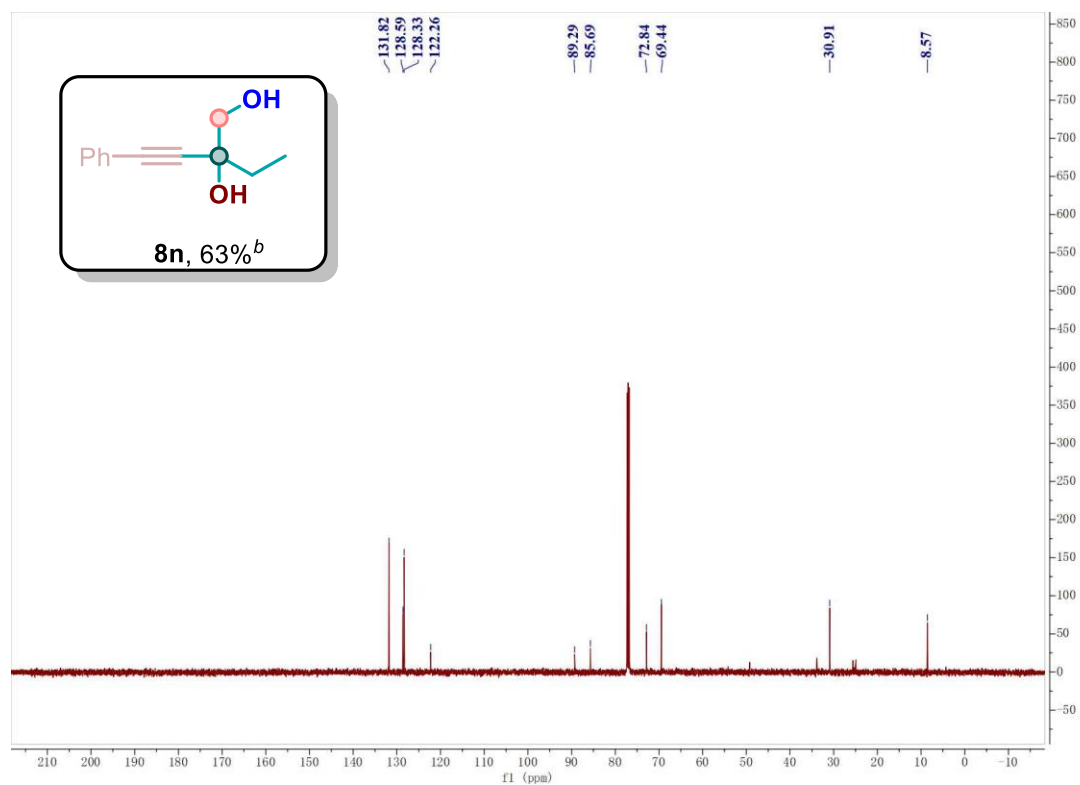


2-ethyl-4-phenylbut-3-yne-1,2-diol (**8n**)

^1H NMR (500 MHz, Chloroform-*d*)

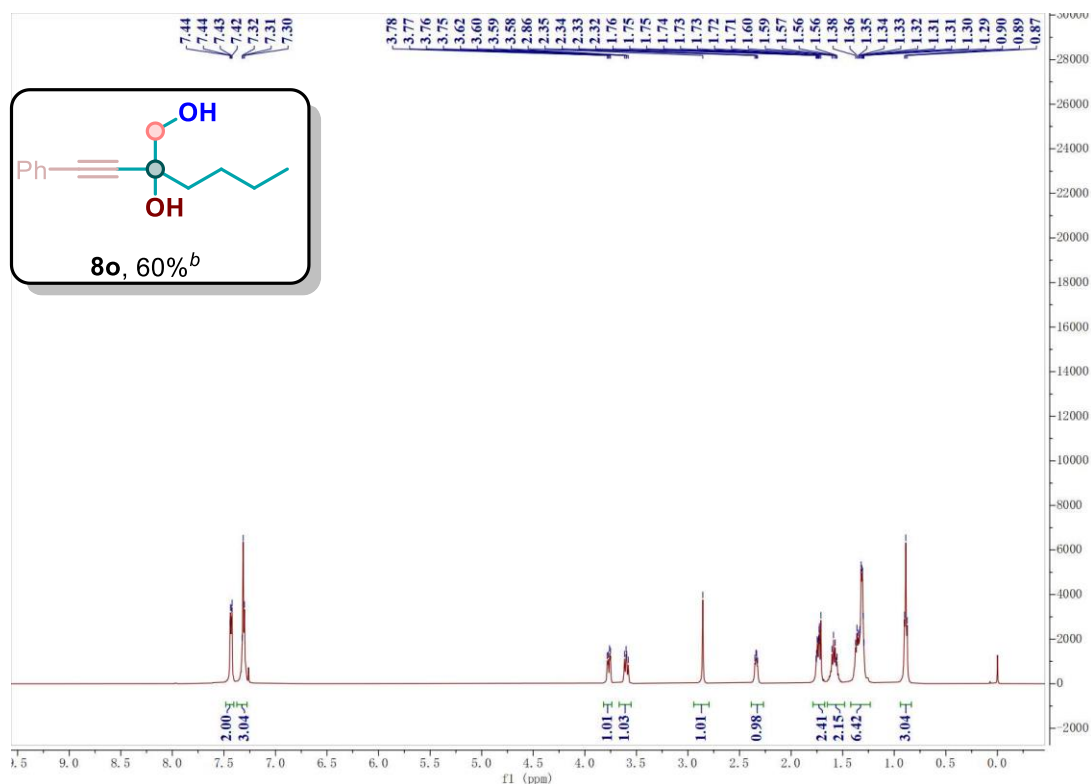


^{13}C NMR (126 MHz, Chloroform-*d*)

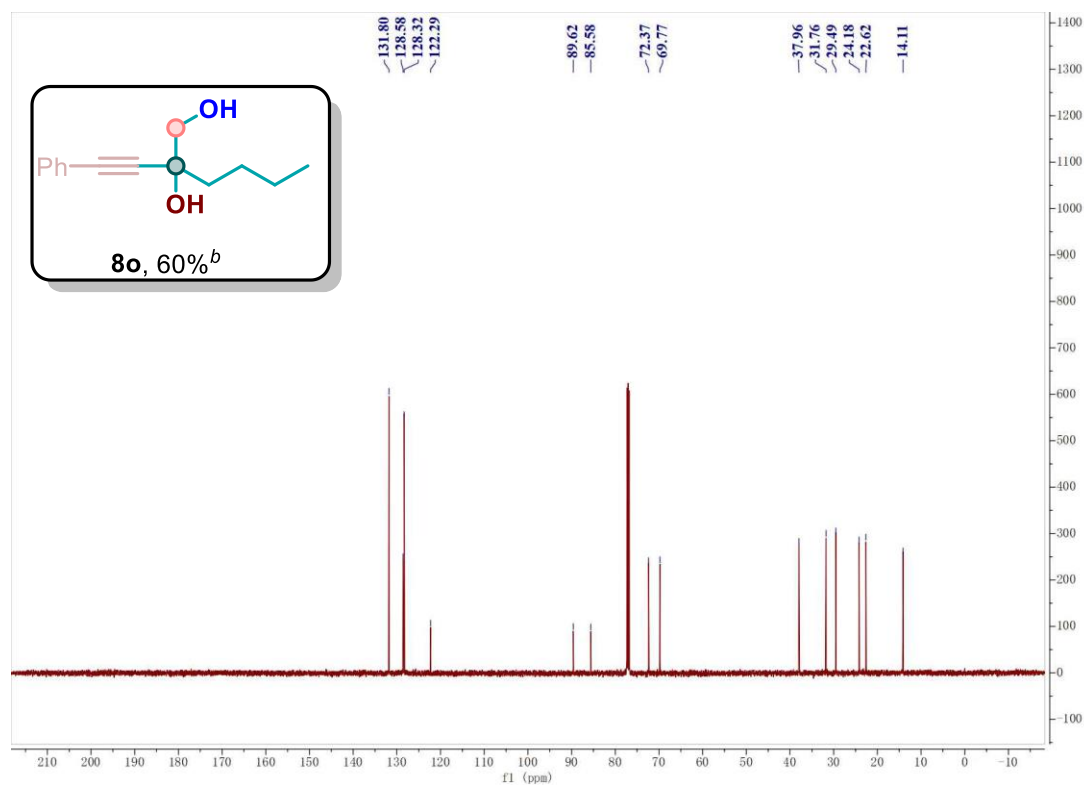


2-(phenylethynyl)hexane-1,2-diol (8o)

¹H NMR (500 MHz, Chloroform-*d*)

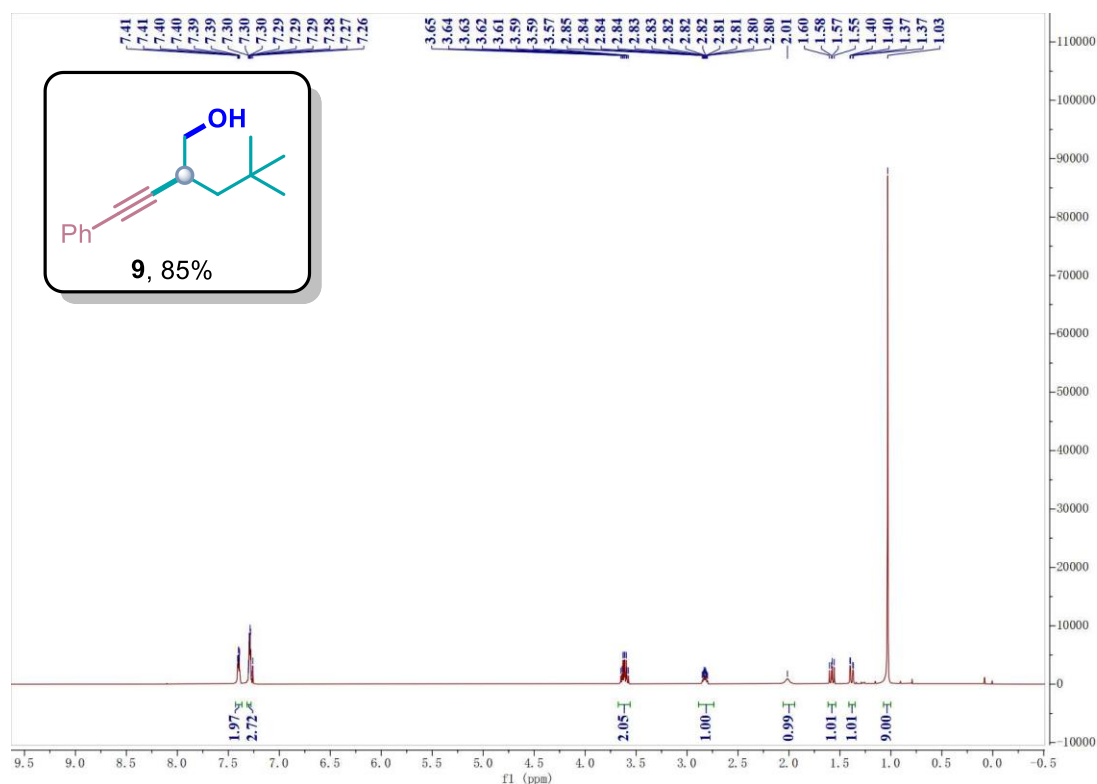


¹³C NMR (126 MHz, Chloroform-*d*)

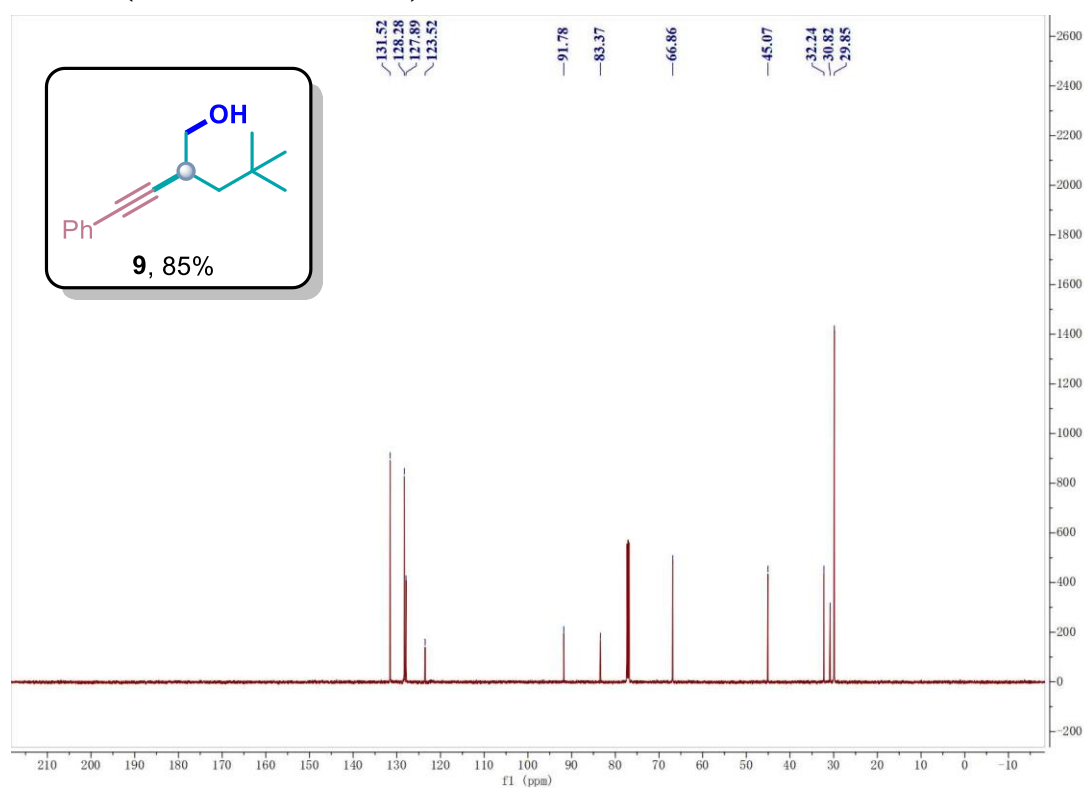


4,4-dimethyl-2-(phenylethynyl)pentan-1-ol (9)

¹H NMR (500 MHz, Chloroform-*d*)

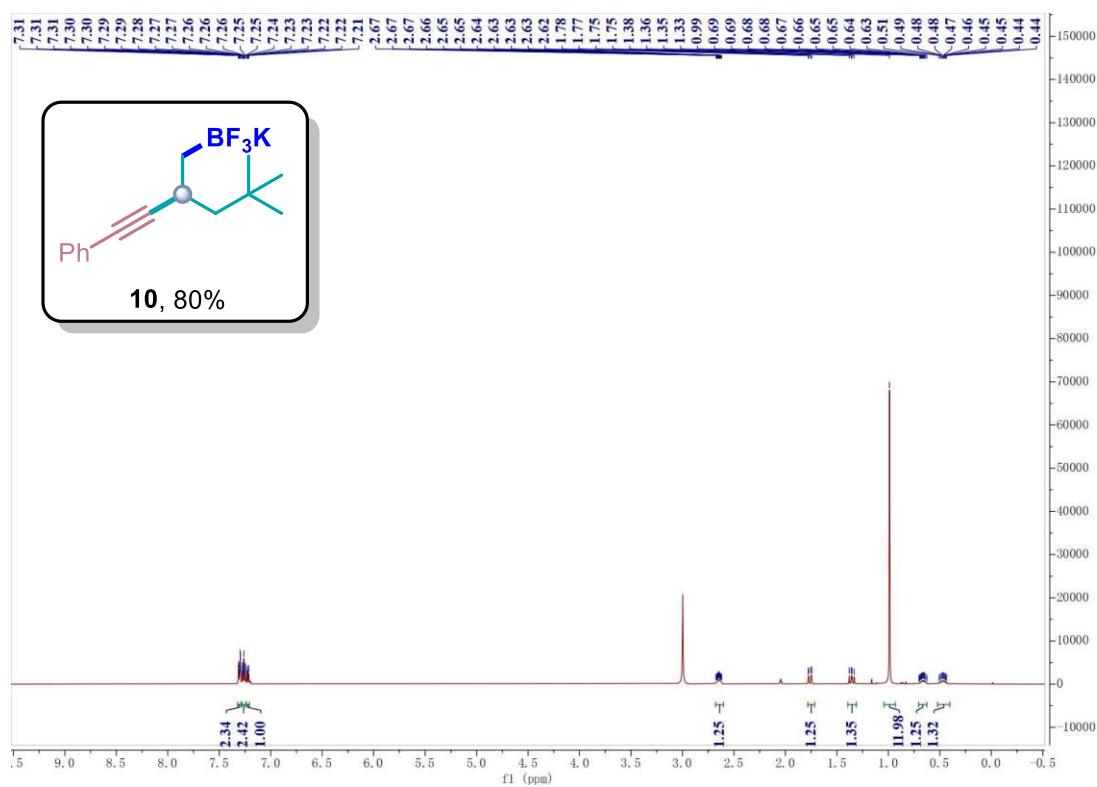


¹³C NMR (126 MHz, Chloroform-*d*)

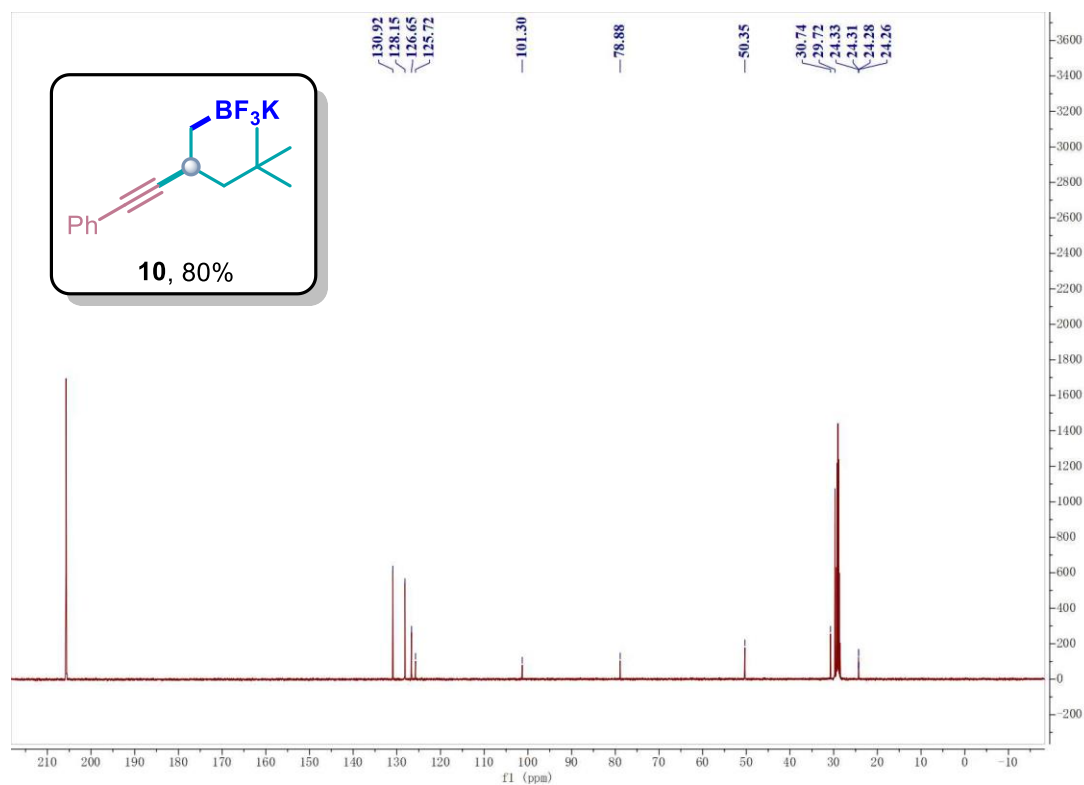


(4,4-dimethyl-2-(phenylethynyl)pentyl)trifluoro-borane, potassium salt (10)

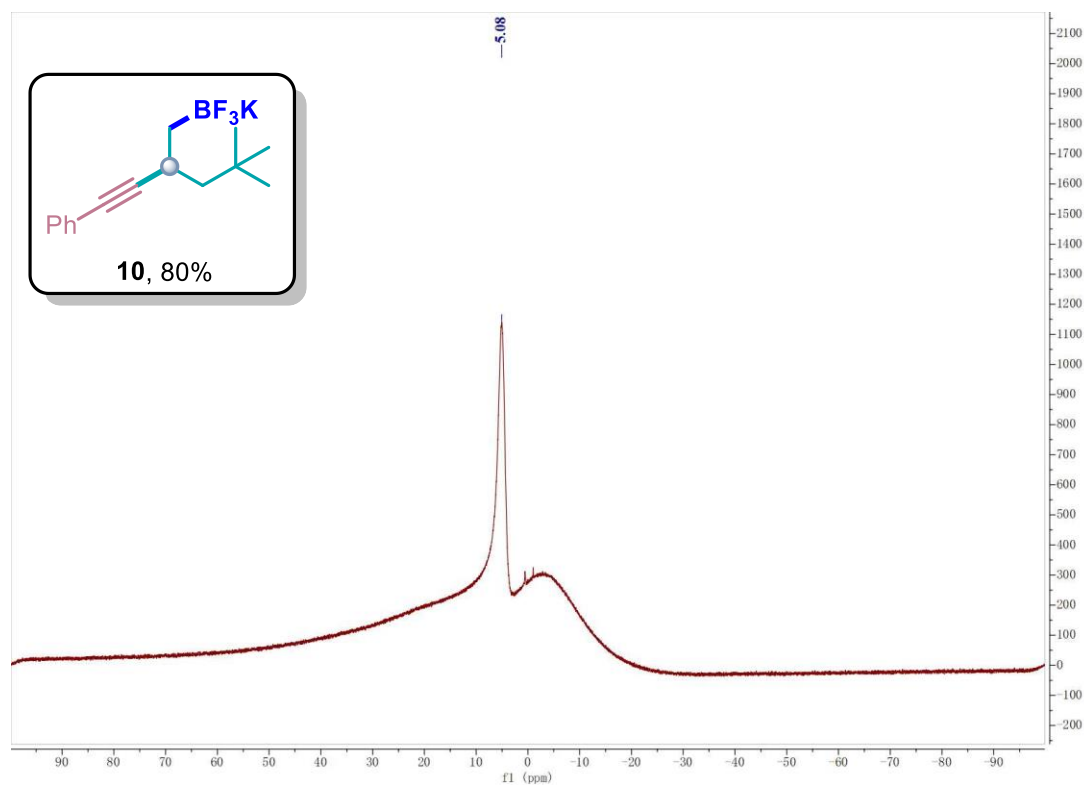
¹H NMR (500 MHz, Acetone-*d*₆)



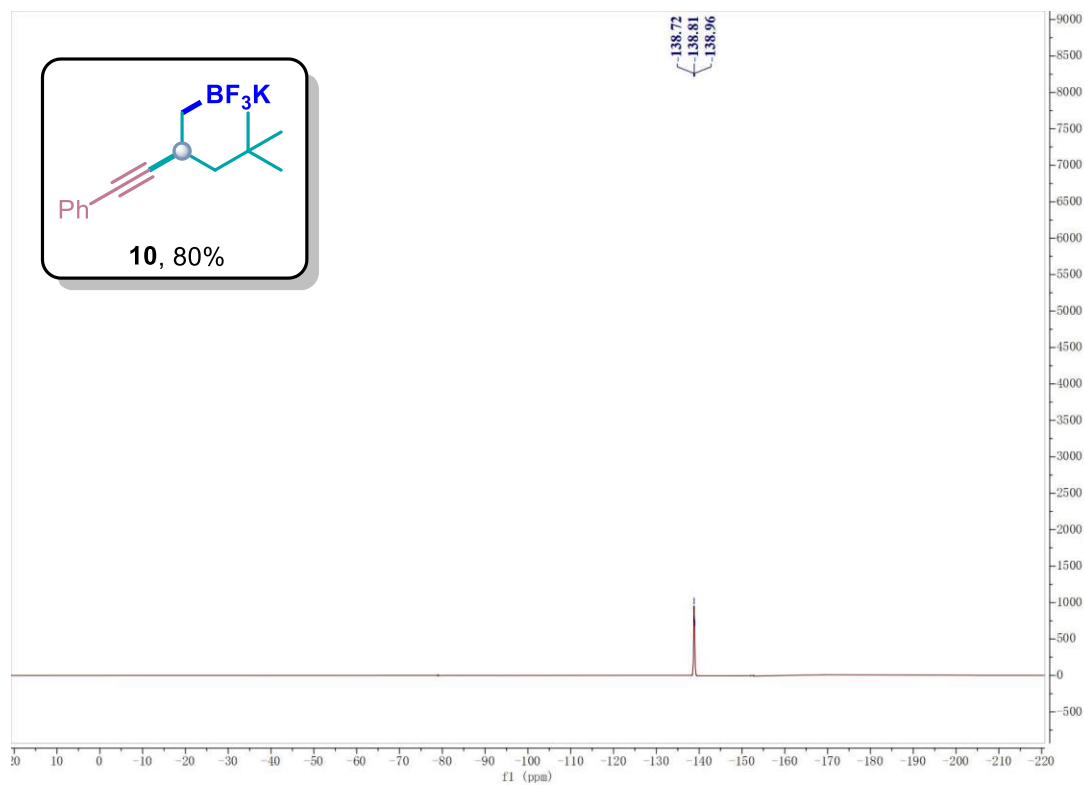
¹³C NMR (126 MHz, Acetone-*d*₆)



^{11}B NMR (160 MHz, Acetone- d_6)

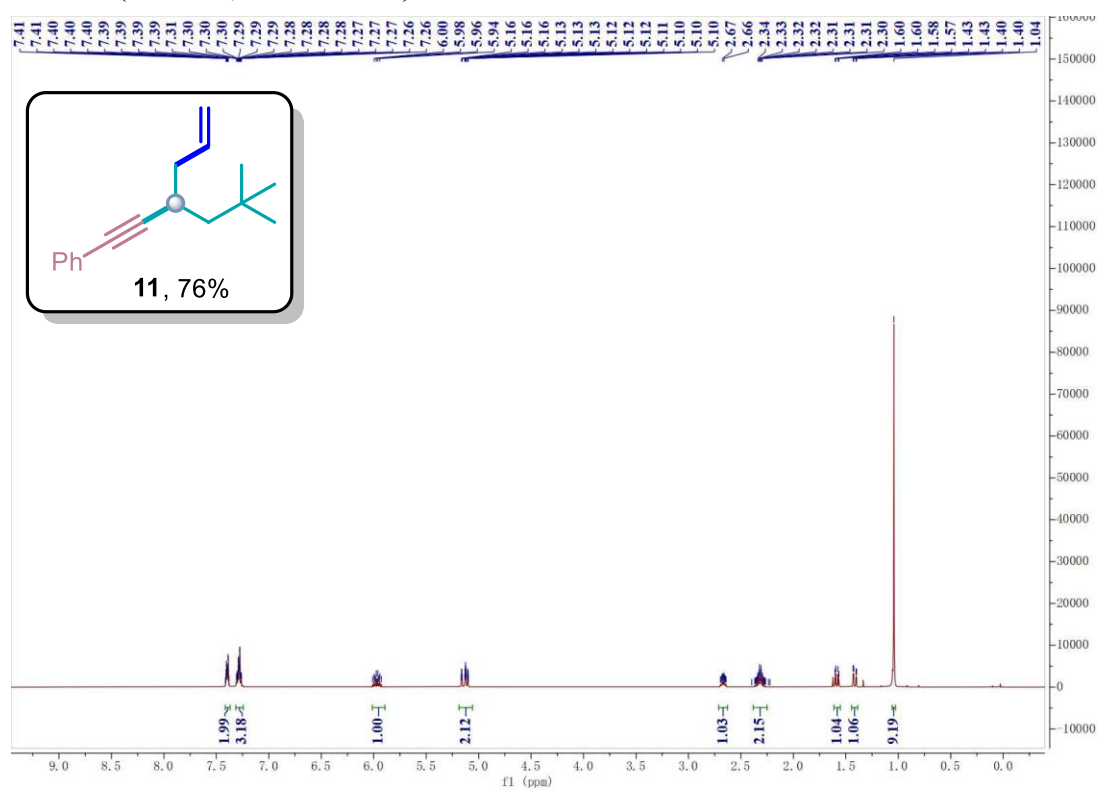


^{19}F NMR (471 MHz, Acetone- d_6)

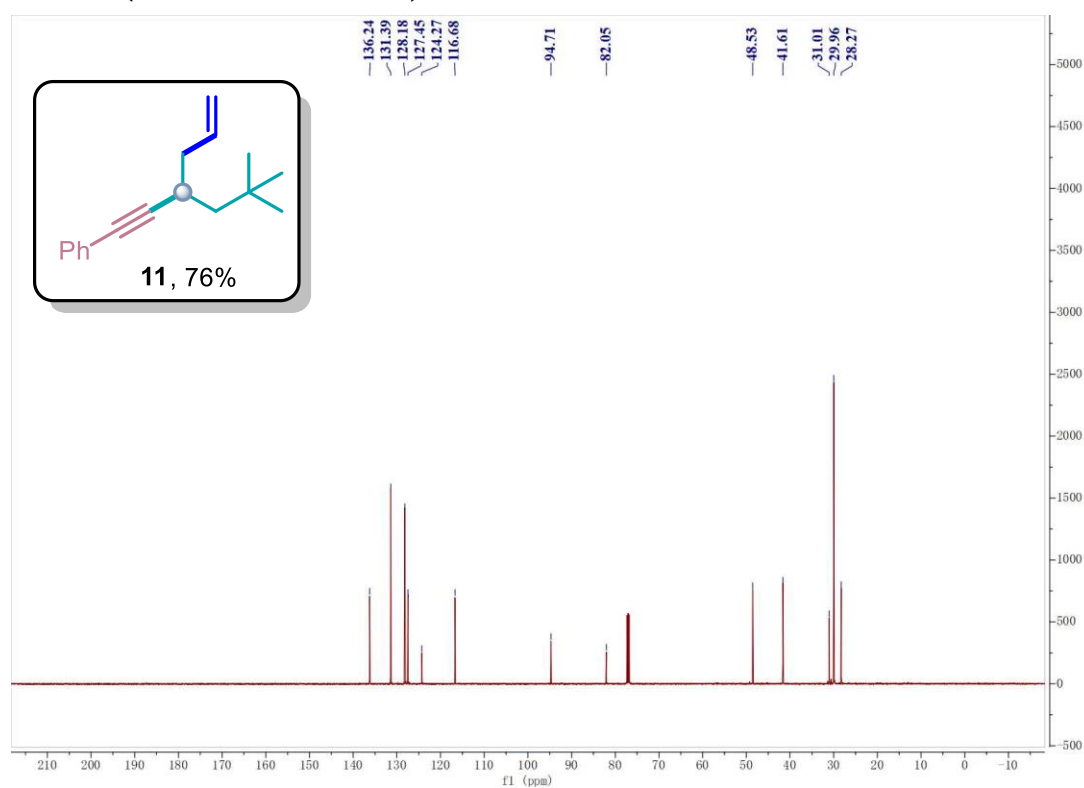


(3-neopentylhex-5-en-1-yn-1-yl)benzene (11)

¹H NMR (500 MHz, Chloroform-*d*)

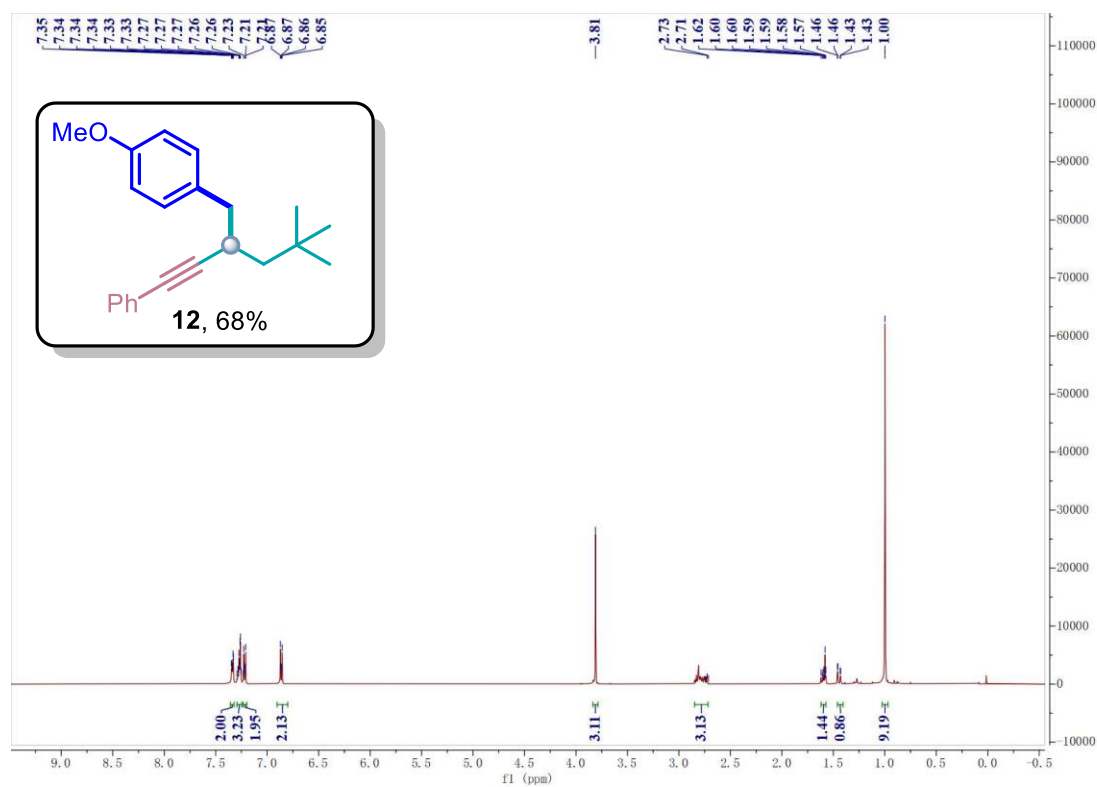


¹³C NMR (126 MHz, Chloroform-*d*)

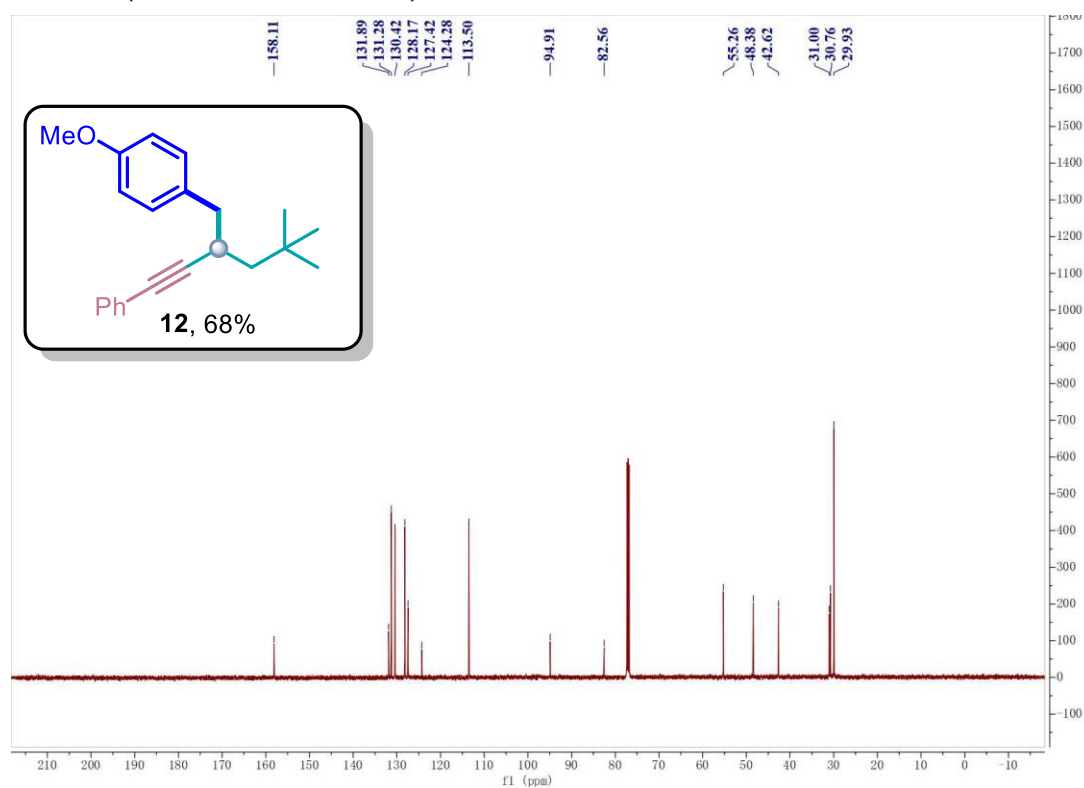


1-(4,4-dimethyl-2-(phenylethynyl)pentyl)-4-methoxybenzene (12)

¹H NMR (500 MHz, Chloroform-*d*)

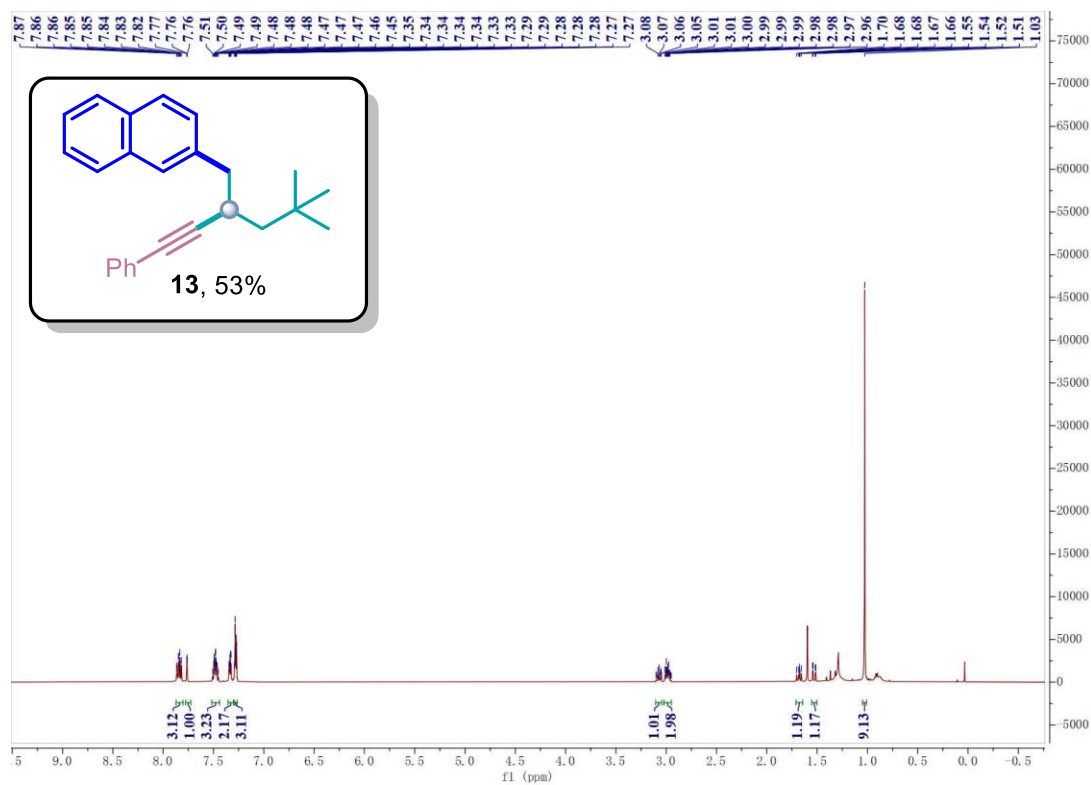


¹³C NMR (126 MHz, Chloroform-*d*)

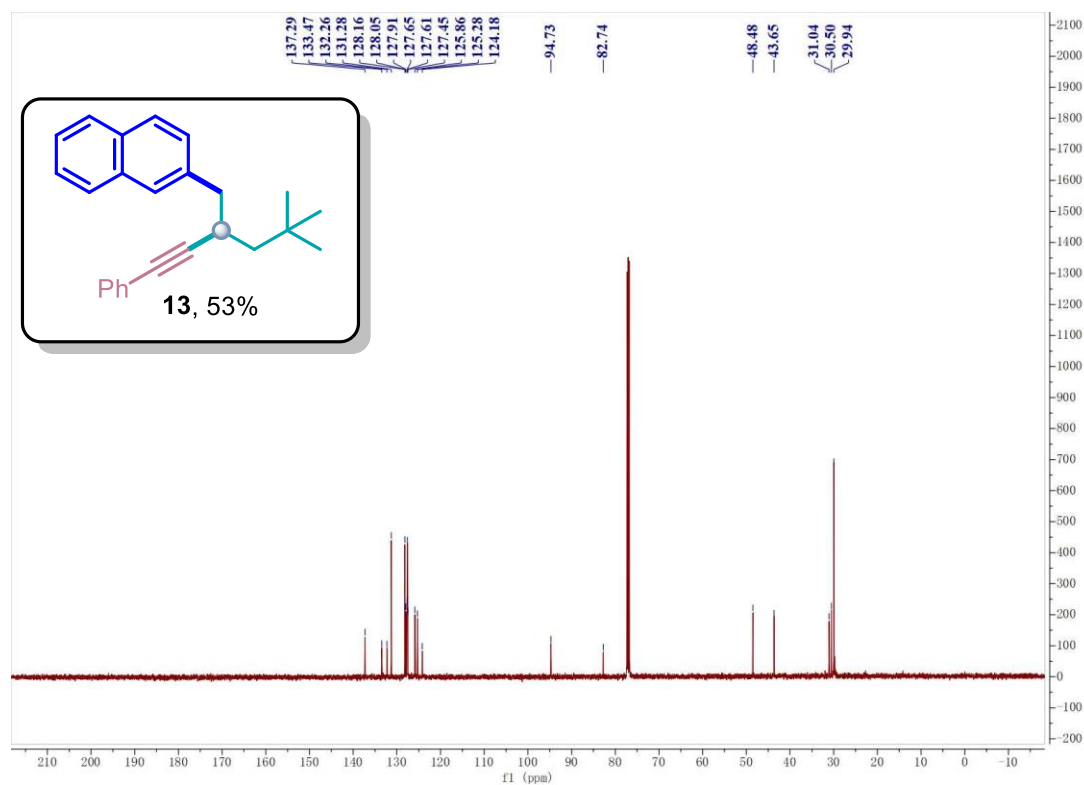


2-(4,4-dimethyl-2-(phenylethynyl)pentyl)naphthalene (13)

¹H NMR (500 MHz, Chloroform-*d*)

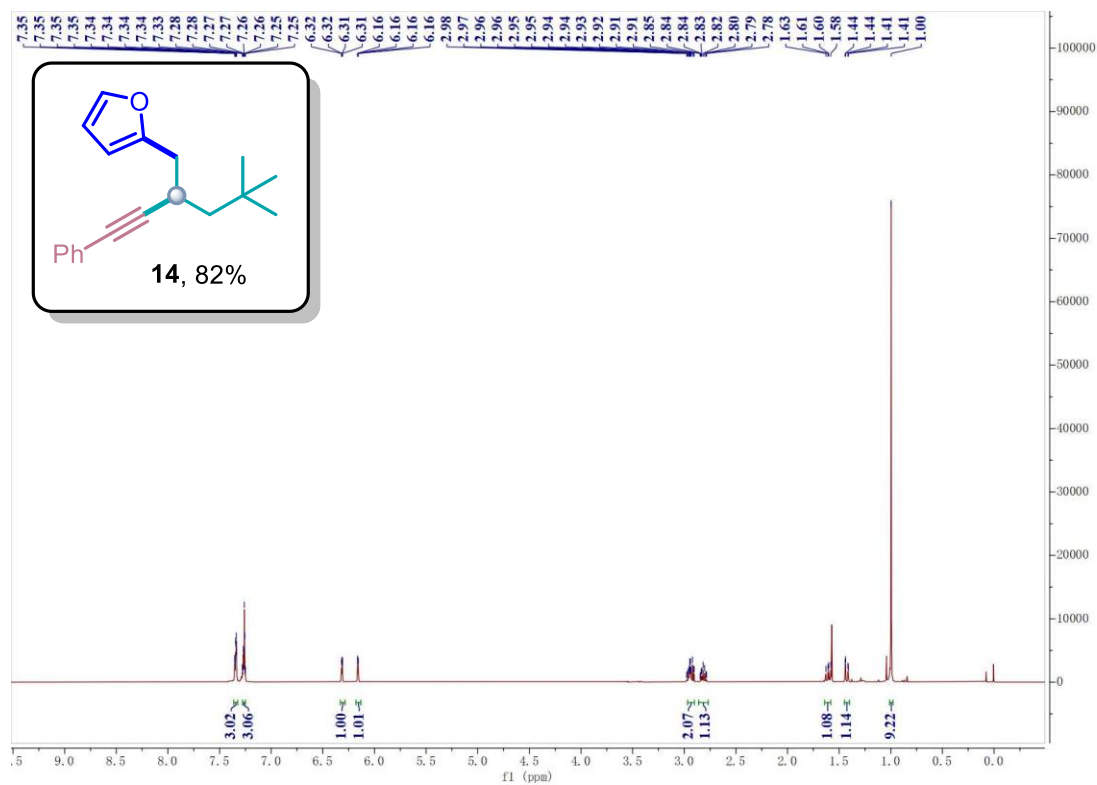


¹³C NMR (126 MHz, Chloroform-*d*)

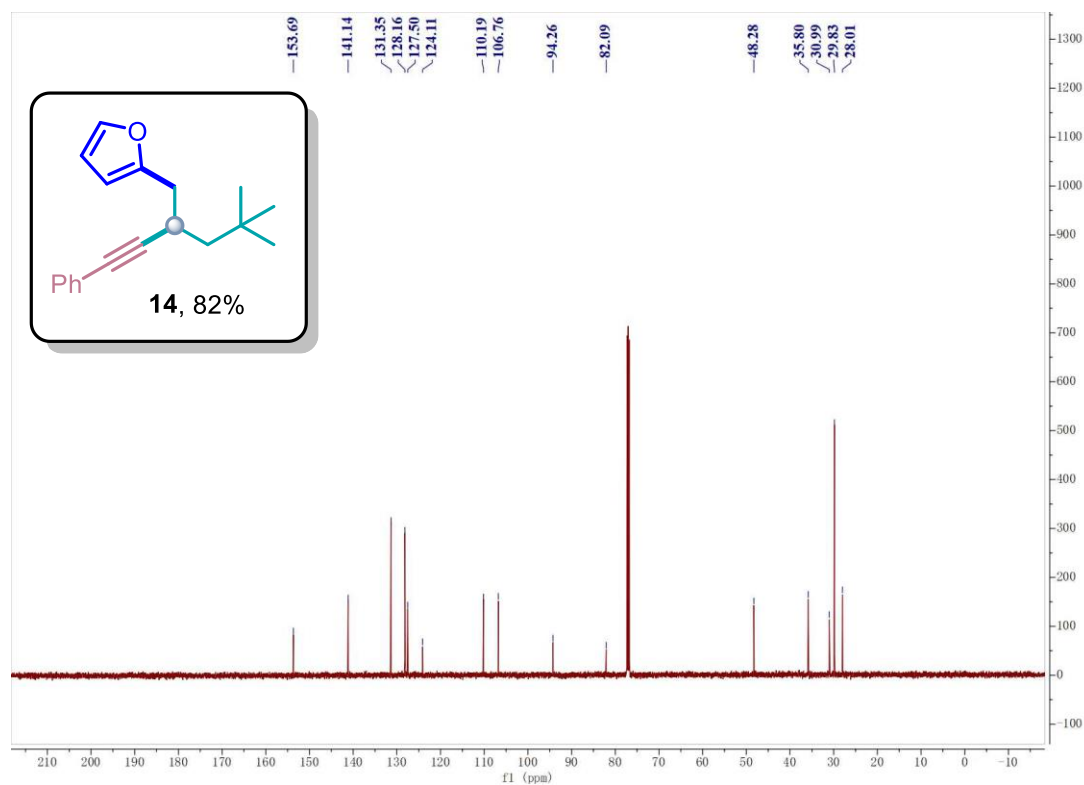


2-(4,4-dimethyl-2-(phenylethynyl)pentyl)furan (14)

¹H NMR (500 MHz, Chloroform-*d*)

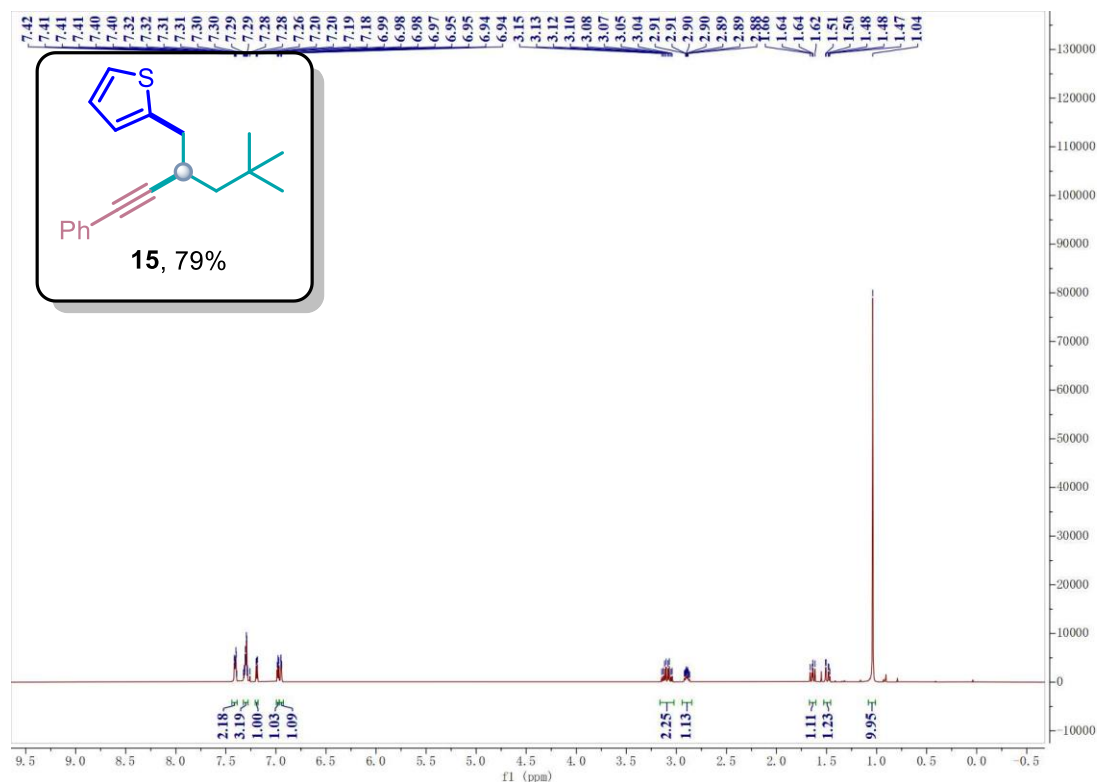


¹³C NMR (126 MHz, Chloroform-*d*)

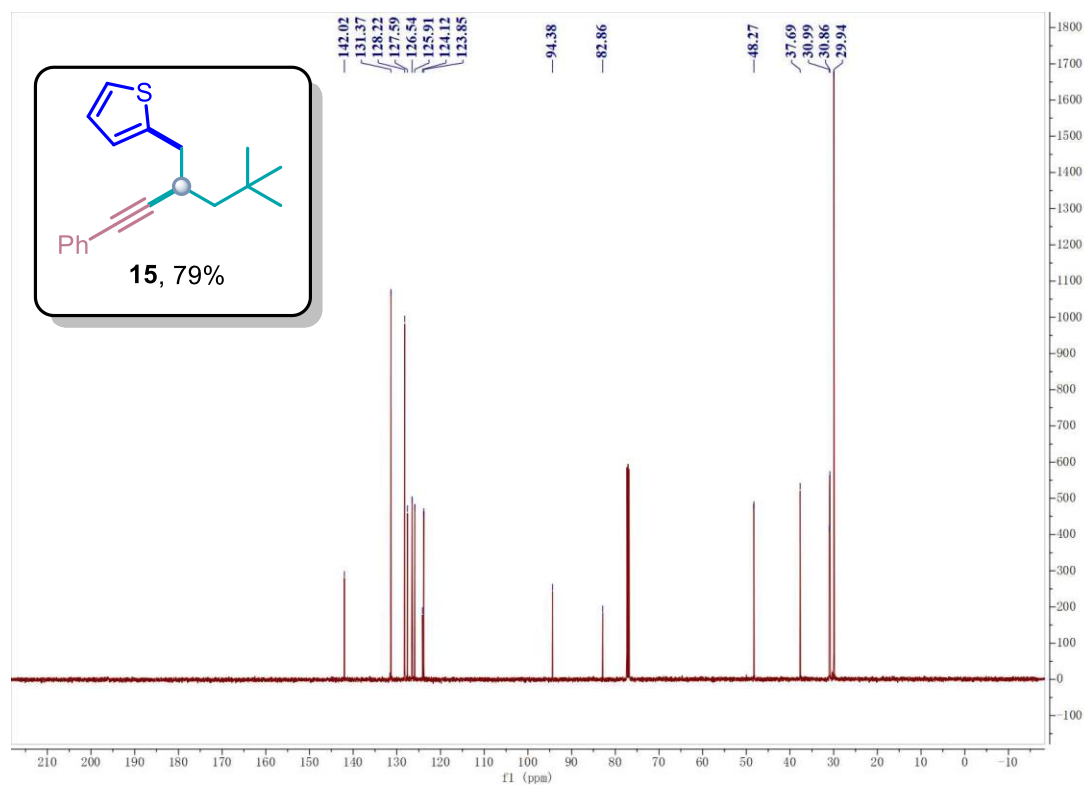


2-(4,4-dimethyl-2-(phenylethynyl)pentyl)thiophene (15)

¹H NMR (500 MHz, Chloroform-*d*)

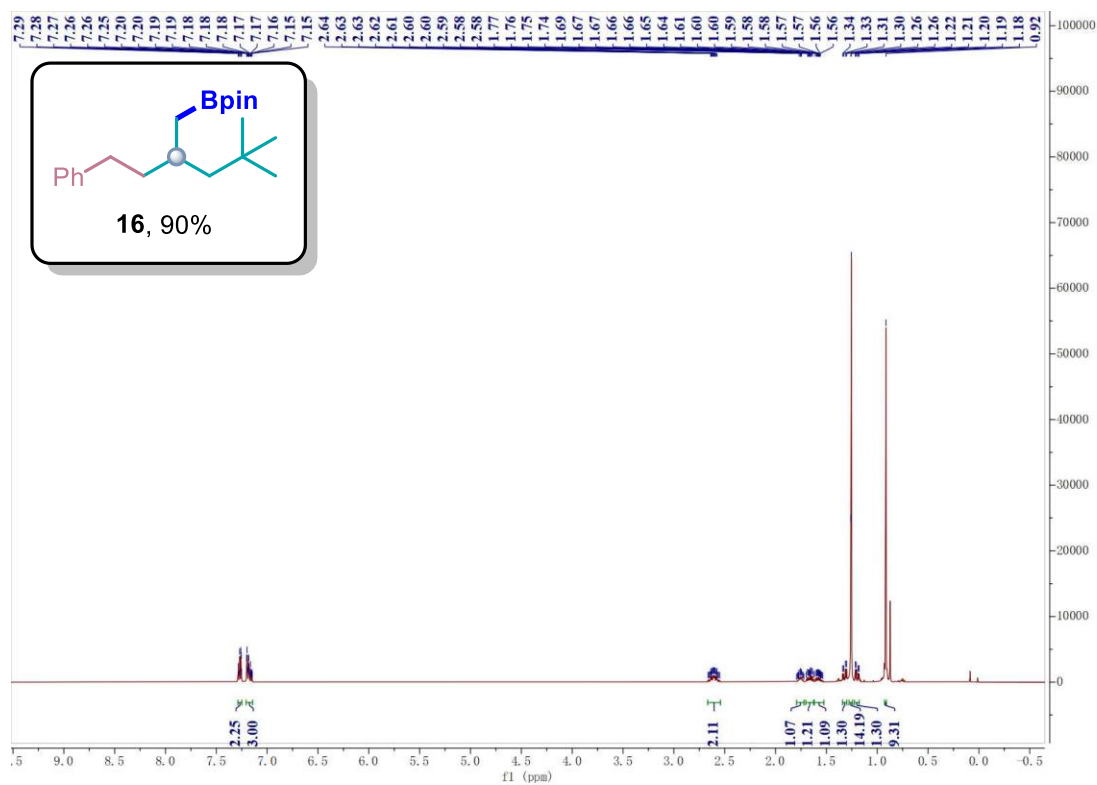


¹³C NMR (126 MHz, Chloroform-*d*)

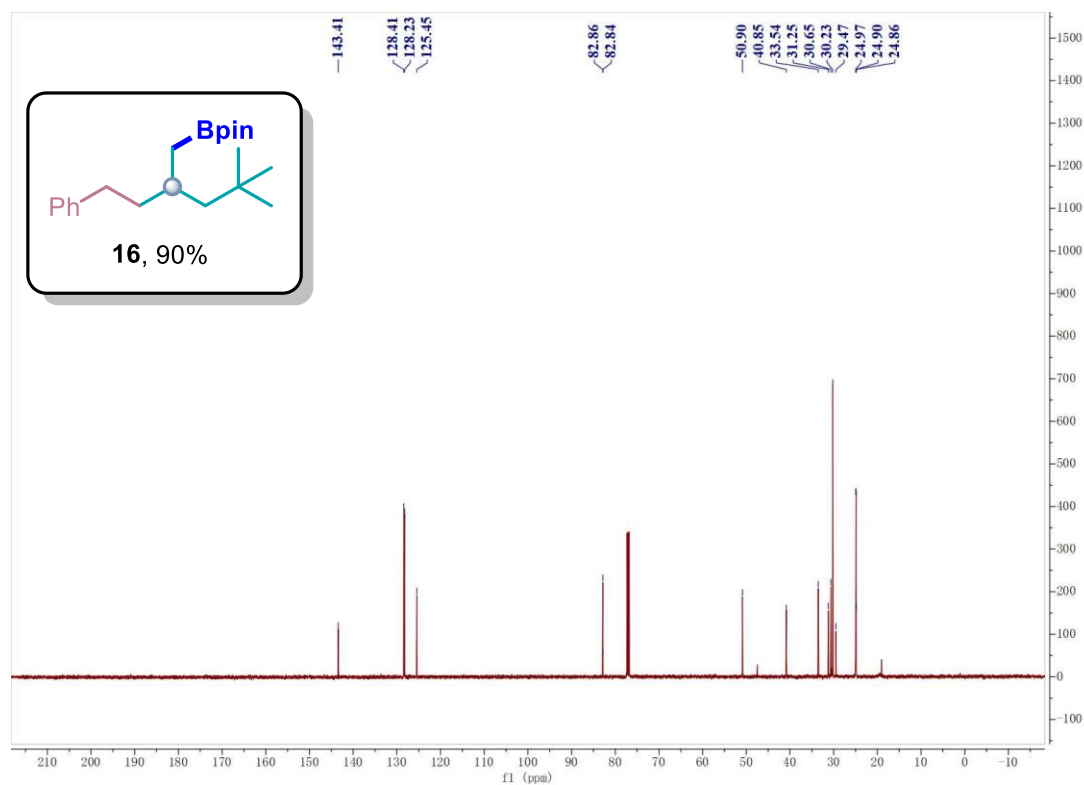


2-(4,4-dimethyl-2-phenethylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (16)

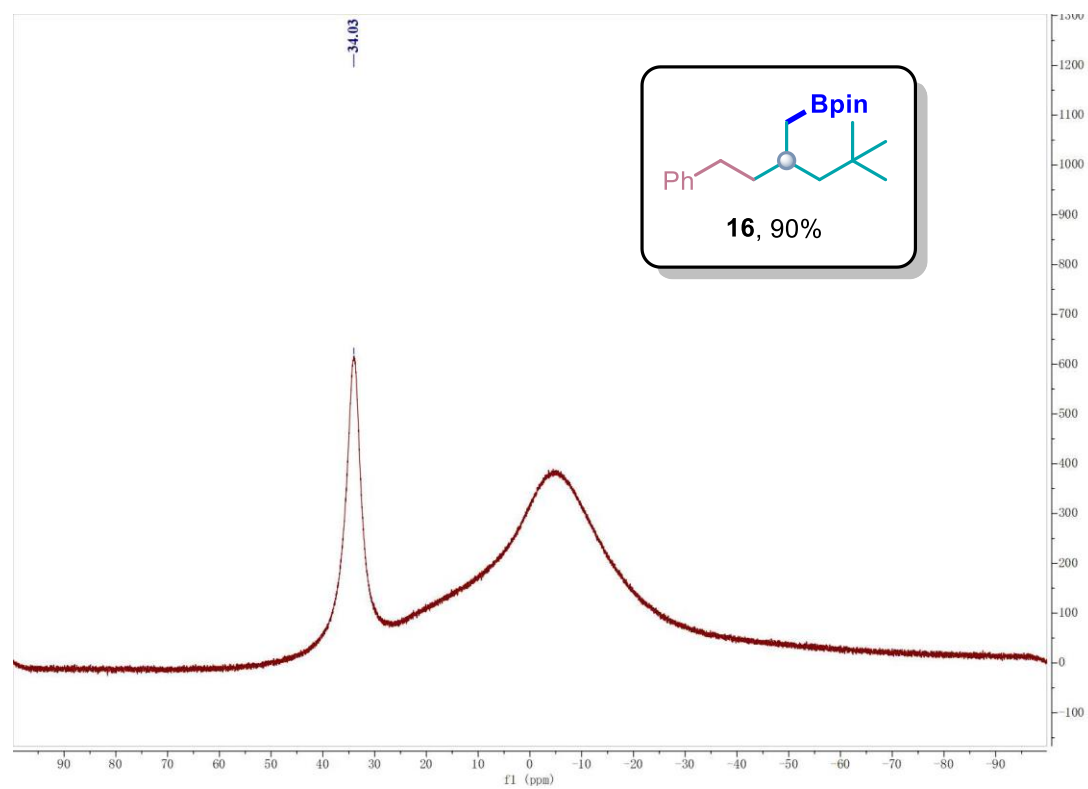
¹H NMR (500 MHz, Chloroform-*d*)



¹³C NMR (126 MHz, Chloroform-*d*)



^{11}B NMR (160 MHz, Chloroform-*d*)



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