

Supporting Information

Copper-Catalyzed Synthesis of Cyclopropyl Bis(boronates) from Aryl Olefins and Carbon Monoxide

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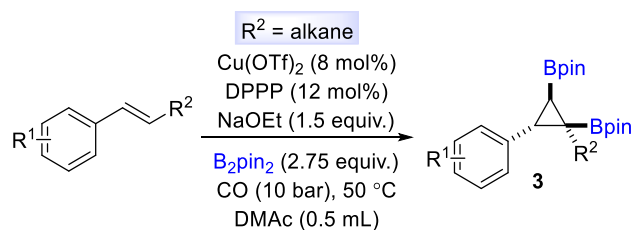
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2. General information

Unless otherwise noted, all commercial reagents were ordered from Sigma-Aldrich, TCI, ABCR, or Across. All solvents (anhydrous and under inert atmosphere) were collected by M BRAUN from the solvent purification system and used under standard Schlenk techniques. Olefins were synthesized based on known literatures. Column chromatography was performed on silica gel (200-300 meshes) using *n*-pentane (bp. 36.1 °C), dichloromethane and ethyl acetate as eluents. All NMR spectra were recorded at ambient temperature using Bruker Advance 300 NMR, Bruker ARX 400 NMR spectrometers. Multiplets were assigned as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (Hz), and integration. All ^{13}C NMR spectra were broad-band ^1H decoupled. Gas chromatography (GC) analyses were performed on an Agilent HP-7890A instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d. 0.25 μm film thickness) using argon as carrier gas. High resolution mass spectra (HRMS) were recorded on an Agilent 6210 system. Because of the high toxicity of carbon monoxide, all reactions should be performed in an autoclave. The laboratory should be well-equipped with a CO detector and alarm system.

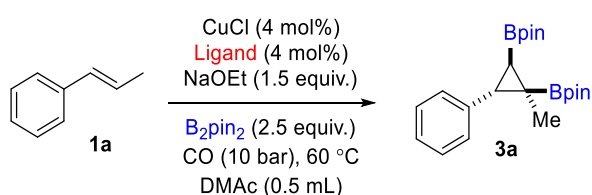
3. Experimental Procedures

3.1 Supplementary Tables



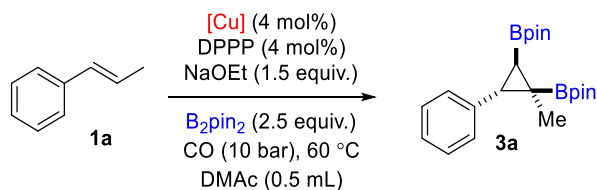
3.1.1 Optimization reaction condition for 3a.

Table S1 Screening of Ligand^a



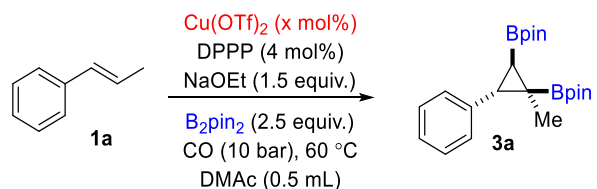
Entry	Ligand	Yield of 3a (%)
1	DPPM	11
2	DPPE	23
3	DPPP	37
4	DPPB	17
5	DPPF	19
6	DCyPE	23
7	DCyPP	20
8	DPEphos	9
9	Xantphos	14
10	(R)-Tol-BINAP	2
11	ChiraPhos	15
12	Ph-BPE	27
13	BuPAd ₂	13
14	BDPP	19
15	DPPBz	13
6	4-OMe-dppp	32
17	4-CF ₃ -dppp	6

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), CuCl (4 mol%), ligand (4 mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S2 Screening of Catalysis^a

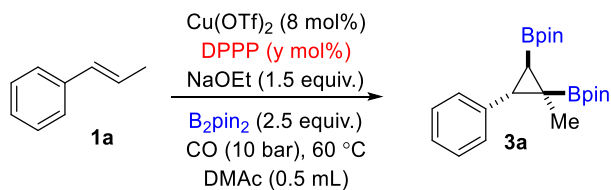
Entry	[Cu]	Yield of 3a (%)
1	CuCl ₂	34
2	CuCN	18
3	Cu(OAc) ₂	39
4	CuI	37
5	CuBr(Me ₂ S)	37
6	LiCuCl ₄	30
7	iPrCuCl	34
8	iMesCuCl	38
9	CuSO ₄	28
10	Cu(OTf) ₂	40
11	Cu(acac) ₂	32
12	Cu(OAc) ₂	35
13	Cu(OTf)•Toluene	34

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), [Cu] (4 mol%), DPPP (4 mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 16 h. Yields are determined by GC with hexadecane as an internal standard.

Table S3 Screening of the amount of Catalysis^a

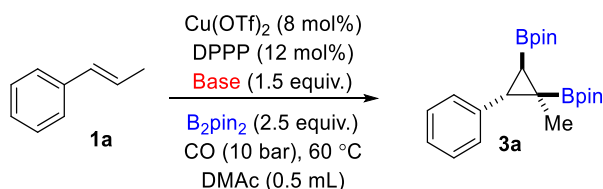
Entry	Cu(OTf) ₂ (mol%)	Yield of 3a (%)
1	4	40
2	8	51
3	10	34

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), Cu(OTf)₂ (x mol%), DPPP (x mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S4 Screening of the amount of Ligand^a

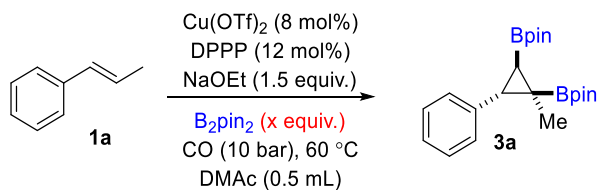
Entry	Dppp (mol%)	Yield of 3a (%)
1	8	51
2	12	55
3	16	47
4	20	41

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), Cu(OTf)₂ (8 mol%), DPPPP (y mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 22 h. Yields are determined by GC with hexadecane as an internal standard.

Table S5 Screening of Base^a

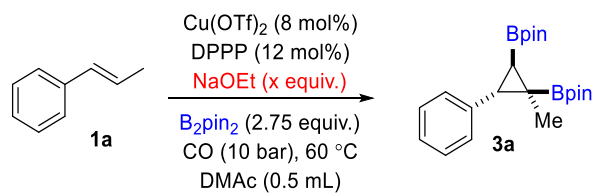
Entry	Solvent	Yield of 3a (%)
1	NaO ^t Bu	14
2	NaOMe	6
3	NaOPh	/
4	KO ^t Bu	10
5	LiO ^t Bu	6
6	NaO ⁱ Pr	/
7	Cs ₂ CO ₃	50
8	Na ₂ CO ₃	/
9	Cs(OAc)	Trace
10	K ₂ CO ₃	5
11	K ₃ PO ₄	25
12	KOH	9
13	NEt ₃	/
14	DIPEA	/
15	NaOEt	55

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), Cu(OTf)₂ (8 mol%), DPPPP (12 mol%), Base (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 22 h. Yields are determined by GC with hexadecane as an internal standard.

Table S6 Screening of the amount of B₂pin₂^a

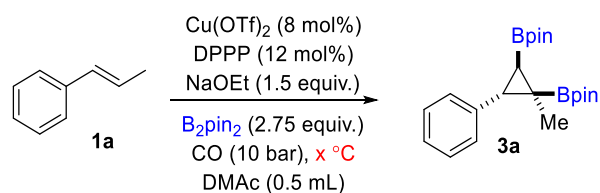
Entry	B ₂ pin ₂	Yield of 3a (%)
1	1.5	33
2	2.0	46
3	2.2	45
4	2.5	55
5	2.75	63
6	3.0	60
7	3.5	60

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), Cu(OTf)₂ (8 mol%), DPPPP (12 mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 22 h. Yields are determined by GC with hexadecane as an internal standard.

Table S7 Screening of the amount of Base^a

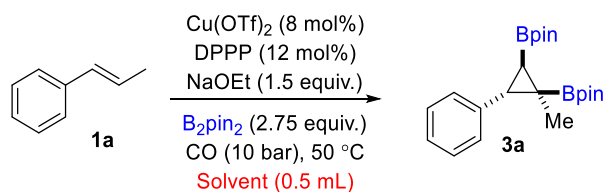
Entry	NaOEt	Yield of 3a (%)
1	1.3	60
2	1.5	63
3	1.7	58
4	2.0	56
5	2.5	53

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B₂pin₂ (2.5 equiv.), Cu(OTf)₂ (8 mol%), DPPPP (12 mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 22 h. Yields are determined by GC with hexadecane as an internal standard.

Table S8 Screening of Temperature^a

Entry	Temperature	Yield of 3a (%)
1	40	45
2	50	74 (61 ^b)
3	60	63
4	70	57

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B_2pin_2 (2.5 equiv.), $\text{Cu}(\text{OTf})_2$ (8 mol%), DPPP (12 mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 22 h. Yields are determined by GC with hexadecane as an internal standard. [b] Isolated yield.

Table S9 Screening of Solvent^a

Entry	Solvent	Yield of 3a (%)
1	DMF	35
2	DMSO	23
3	NMP	34
4	THF	16
5	Toluene	34
6	MeCN	29
7	Cyclohexane	25

[a] Reaction conditions: *trans*- β -methylstyrene (0.2 mmol), B_2pin_2 (2.5 equiv.), $\text{Cu}(\text{OTf})_2$ (8 mol%), DPPP (12 mol%), NaOEt (1.5 equiv.), CO (10 bar), DMAc (0.4 M), stirred at 60 °C for 22 h. Yields are determined by GC with hexadecane as an internal standard.

3.1.2 Optimization reaction condition for 4a.

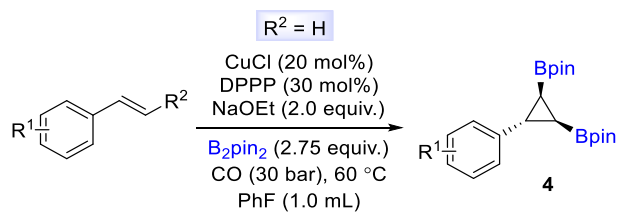
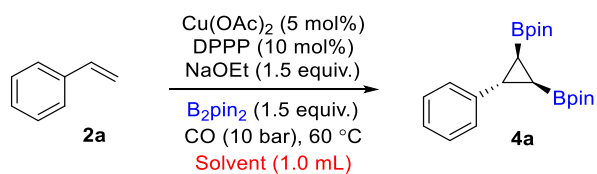
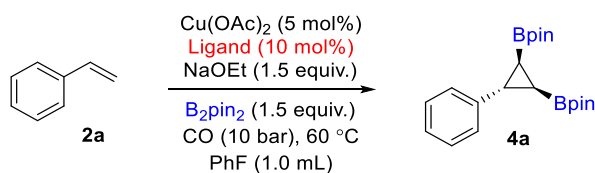


Table S10 Screening of Solvent^a



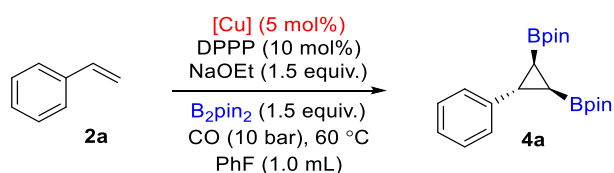
Entry	Solvent	Yield of 4a (%)
1	Cycloheptane	2
2	PhCl	15
3	PhF	18
4	PhOMe	4
5	MeCN	8
6	THF	8
7	1,4-Dioxane	13
8	DMAc	3
9	Toluene	7
10	Et ₂ O	14
11	PhCF ₃	13
12	General procedure A	Trace

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (1.5 equiv.), Cu(OAc)₂ (5 mol%), DPPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), solvent (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S11 Screening of Ligand^a

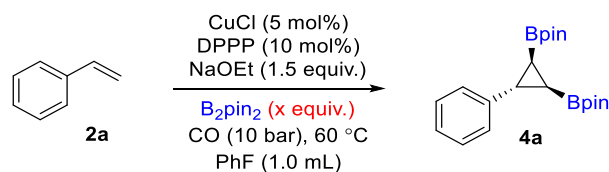
Entry	Ligand	Yield of 4a (%)
1	DPEphos	-
2	Xantphos	-
3	BINAP	4
4	BIPHEphos	5
5	BuPAD ₂	3
6	DPPE	6
7	DPPB	2
8	DPPPE	3
9	DPPBz	2
10	DPPF	2
11	DPPM	-
12	2,2'-Bipyridine	2
13	DPPP	18

[a] Reaction conditions: styrene (0.2 mmol), B_2pin_2 (1.5 equiv.), Cu(OAc)_2 (5 mol%), Ligand (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S12 Screening of Catalysis^a

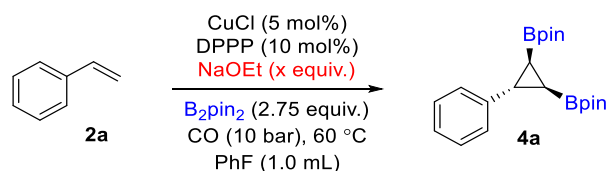
Entry	[Cu]	Yield of 4a (%)
1	Cu(OAc)_2	18
2	CuCl_2	13
3	CuCl	21
4	Cu(acac)_2	12
5	^t MesCuCl	6
6	CuI	10
7	CuBr	8
8	CuCN	-
9	Cu(OTf)_2	4

[a] Reaction conditions: styrene (0.2 mmol), B_2pin_2 (1.5 equiv.), [Cu] (5 mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S13 Screening of the amount of B₂pin₂^a

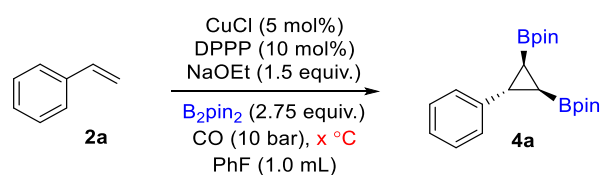
Entry	B ₂ pin ₂ (x equiv.)	Yield of 4a (%)
1	1.5	21
2	2.0	20
3	2.5	23
4	2.75	25
5	3.0	22

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (x equiv.), CuCl (5 mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S14 Screening of the amount of Base^a

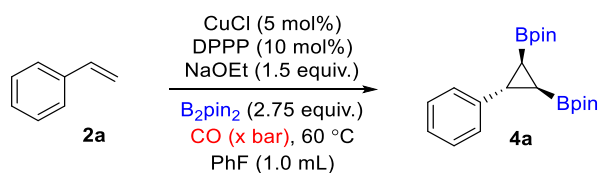
Entry	Base (x equiv.)	Yield of 4a (%)
1	1.5	25
2	2.0	29
3	2.5	27
4	3.0	24

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (x equiv.), CuCl (5 mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S15 Screening of Temperature^a

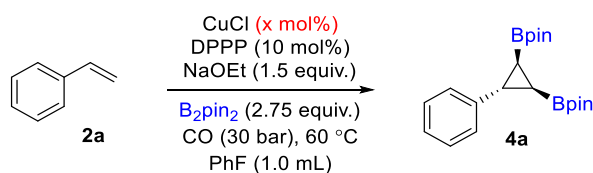
Entry	Temperature (°C)	Yield of 4a (%)
1	40	13
2	50	18
3	60	29
4	80	12
5	100	6

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (1.5 equiv.), [Cu] (5 mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S16 Screening of CO pressure^a

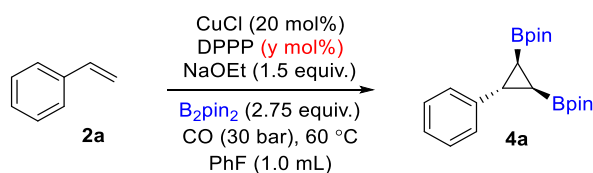
Entry	CO (bar)	Yield of 4a (%)
1	10	29
2	20	38
3	30	49
4	40	44

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (1.5 equiv.), CuCl (10 mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO, PhF (1 mL), stirred at 60 °C for 16 h. Yields are determined by GC with hexadecane as an internal standard.

Table S17 Screening of the amount of Catalysis^a

Entry	CuCl (x mol%)	Yield of 4a (%)
1	10	49
2	20	53
3	30	43
4	40	25

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (1.5 equiv.), CuCl (x mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard.

Table S18 Screening of the amount of Ligand^a

Entry	DPPP (y mol%)	Yield of 4a (%)
1	20	52
2	25	54
3	30	64 (55% ^b)
4	35	57
5	40	39

[a] Reaction conditions: styrene (0.2 mmol), B₂pin₂ (1.5 equiv.), CuCl (x mol%), DPPP (10 mol%), NaOEt (1.5 equiv.), CO (10 bar), PhF (1 mL), stirred at 60 °C for 24 h. Yields are determined by GC with hexadecane as an internal standard. [b] Isolated yield.

3.2 General Procedures for preparing alkenes.

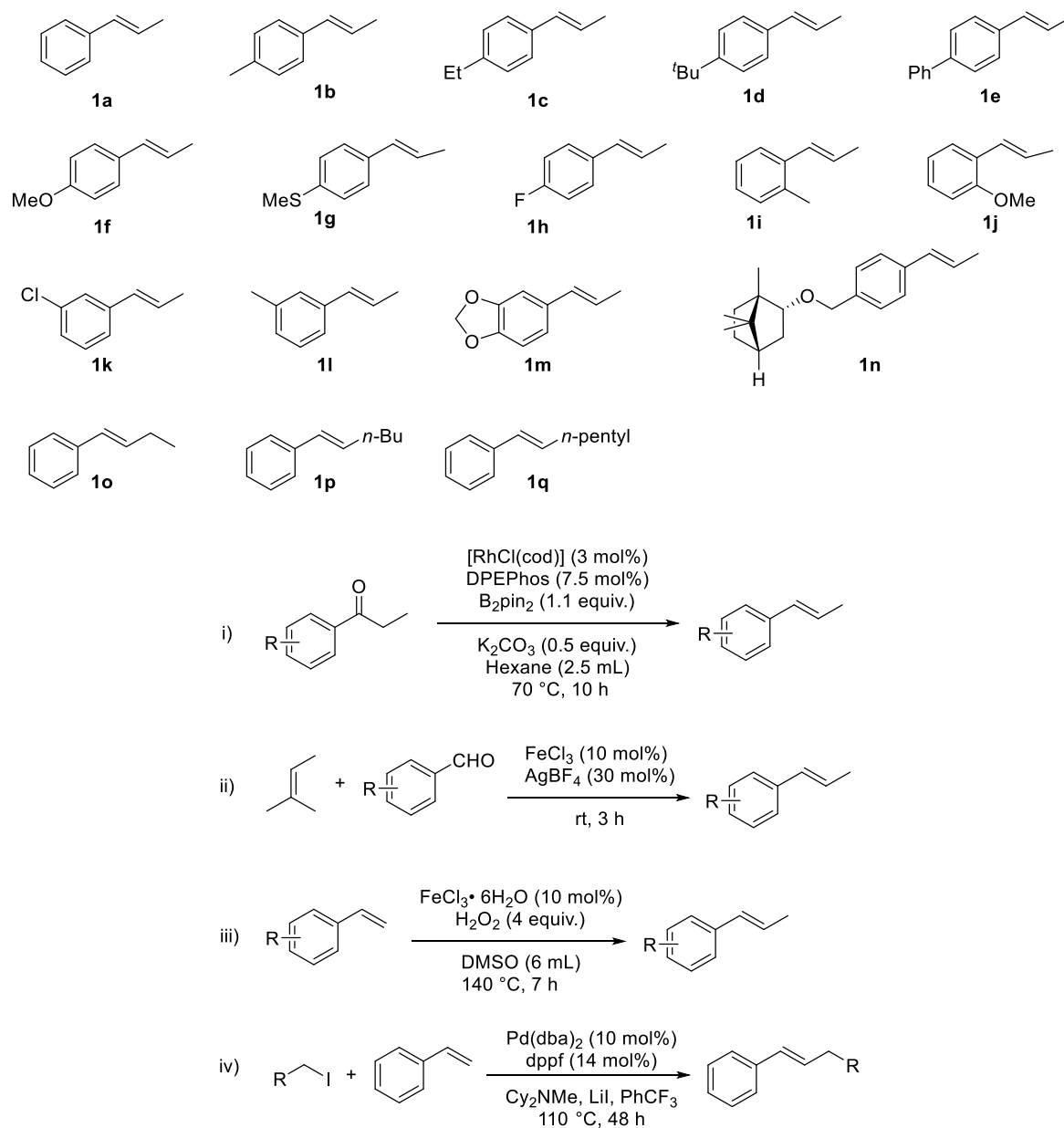


Figure 1. Preparation of substrates

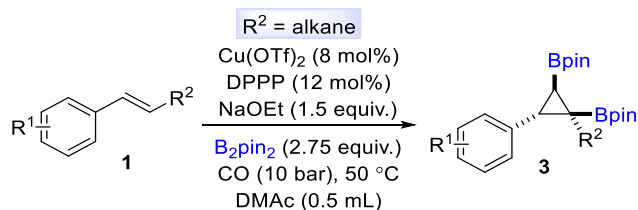
Substrates **1a-1f**, **1h**, **1k**, **1m**, **1o** were prepared according to the reported literature (Figure 1, i).^[1]

Substrates **1g**, **1i**, **1j** and **1l** were prepared according to the reported literature (Figure 1, ii).^[2]

Substrate **1n** was prepared according to the known procedure. (Figure 1, iii).^[3]

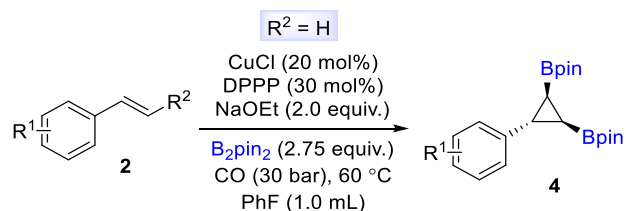
Substrates **1p** and **1q** were prepared according to the reported literature (Figure 1, iv).^[4]

3.3 General Procedures A



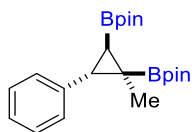
A dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with Cu(OTf)_2 (5.8 mg, 8 mol%), DPPPP (9.9 mg, 12 mol%), B_2pin_2 (140 mg, 2.75 equiv.) and NaOEt (20.4 mg, 1.5 equiv.). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. DMAc (0.5 mL) were added *via* syringe. To this suspension, internal olefins **1** (0.2 mmol) was added *via* Hamilton® syringe. Then the vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (10 bar). The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 24 h at 50 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction purified by gradient column chromatography to yield product **3**.

3.4 General Procedures B



A dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with CuCl (4.0 mg, 20 mol%), DPPPP (24.7 mg, 30 mol%), B_2pin_2 (140 mg, 2.75 equiv.) and NaOEt (20.4 mg, 2.0 equiv.). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. PhF (1.0 mL) were added *via* syringe. To this suspension, terminal olefine **2** (0.2 mmol) was added *via* Hamilton® syringe. Then the vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (30 bar). The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 24 h at 60 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction purified by gradient column chromatography to yield product **4**.

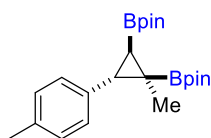
4 NMR Spectra of the Products



2,2'-(1-Methyl-3-phenylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3a)

The title compound was prepared from (*E*)-prop-1-en-1-ylbenzene (23.6 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a white solid (46.8 mg, 61%). mp: 116-118 °C.

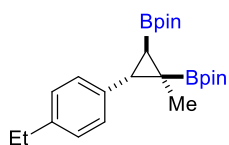
¹H NMR (300 MHz, CDCl₃) δ 7.26 – 7.13 (m, 5H), 2.51 (d, *J* = 7.4 Hz, 1H), 1.26 (d, *J* = 3.2 Hz, 24H), 0.81 (s, 3H), 0.39 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 139.2, 129.4, 127.6, 125.7, 83.2, 83.1, 31.7, 25.0, 24.9, 24.8, 24.6, 17.4. ¹¹B NMR (96 MHz, CDCl₃) δ 32.6. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₂H₃₄¹⁰B¹¹BO₄ 406.2579, found: 406.2584.



2,2'-(1-Methyl-3-(*p*-tolyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3b)

The title compound was prepared from (*E*)-1-methyl-4-(prop-1-en-1-yl)benzene (26.4 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a colorless oil (45.4 mg, 57%).

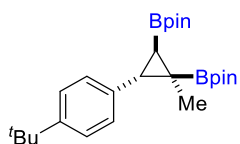
¹H NMR (300 MHz, CDCl₃) δ 7.07 (t, *J* = 3.1 Hz, 4H), 2.47 (d, *J* = 7.4 Hz, 1H), 2.30 (s, 3H), 1.26 (d, *J* = 3.6 Hz, 24H), 0.80 (s, 3H), 0.35 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 136.1, 135.1, 129.3, 128.3, 83.2, 83.1, 31.4, 25.1, 25.0, 24.8, 24.7, 21.0, 17.5. ¹¹B NMR (96 MHz, CDCl₃) δ 32.5. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₃H₃₆¹⁰B¹¹BO₄ 420.2735, found: 420.2740.



2,2'-(3-(4-Ethylphenyl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3c)

The title compound was prepared from (*E*)-1-ethyl-4-(prop-1-en-1-yl)benzene (29.2 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a colorless oil (43 mg, 53%).

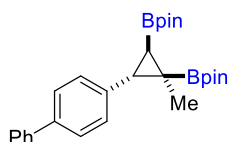
¹H NMR (300 MHz, CDCl₃) δ 7.13 – 7.05 (m, 4H), 2.59 (t, *J* = 7.6 Hz, 2H), 2.48 (d, *J* = 7.4 Hz, 1H), 1.27 – 1.24 (m, 24H), 1.20 (d, *J* = 7.6 Hz, 3H), 0.81 (s, 3H), 0.36 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 141.5, 136.3, 129.3, 127.1, 83.2, 83.1, 31.5, 28.4, 25.0, 24.9, 24.7, 24.6, 17.4, 15.6. ¹¹B NMR (128 MHz, CDCl₃) δ 32.4. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₄H₃₈¹⁰B¹¹BO₄ 434.2892, found: 434.2895.



2,2'-(3-(4-(*tert*-Butyl)phenyl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3d)

The title compound was prepared from (*E*)-1-(*tert*-butyl)-4-(prop-1-en-1-yl)benzene (34.8 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (40.5 mg, 46%).

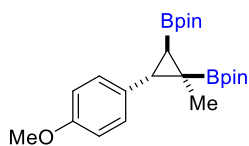
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.21 – 7.17 (m, 2H), 7.05 (d, J = 8.2 Hz, 2H), 2.41 (d, J = 7.4 Hz, 1H), 1.22 (s, 9H), 1.19 (s, 12H), 1.18 (d, J = 3.4 Hz, 12H), 0.77 (s, 3H), 0.30 (d, J = 7.4 Hz, 1H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 148.4, 136.1, 128.9, 124.5, 83.2, 83.1, 34.3, 31.5, 31.4, 25.1, 25.0, 24.8, 24.7, 17.4. $^{11}\text{B NMR}$ (96 MHz, CDCl_3) δ 32.4. **HRMS (ESI-TOF):** calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{26}\text{H}_{42}^{10}\text{B}^{11}\text{BO}_4$ 462.3205, found: 462.3209.



2,2'-(3-([1,1'-Biphenyl]-4-yl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3e)

The title compound was prepared from (*E*)-4-(prop-1-en-1-yl)-1,1'-biphenyl (38.8 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (45.1 mg, 49%).

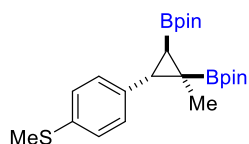
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.60 – 7.56 (m, 2H), 7.52 – 7.47 (m, 2H), 7.44 – 7.39 (m, 2H), 7.33 – 7.27 (m, 2H), 7.25 (d, J = 0.7 Hz, 1H), 2.55 (d, J = 7.4 Hz, 1H), 1.29 – 1.26 (m, 24H), 0.86 (s, 3H), 0.43 (d, J = 7.4 Hz, 1H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 141.1, 138.5, 138.5, 129.8, 128.6, 126.9, 126.9, 126.3, 83.3, 83.2, 31.5, 25.0, 25.0, 24.8, 24.6, 17.5. $^{11}\text{B NMR}$ (96 MHz, CDCl_3) δ 32.9. **HRMS (ESI-TOF):** calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{28}\text{H}_{38}^{10}\text{B}^{11}\text{BO}_4$ 482.2892, found: 482.2894.



2,2'-(3-(4-Methoxyphenyl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3f)

The title compound was prepared from (*E*)-1-methoxy-4-(prop-1-en-1-yl)benzene (29.6 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 20:1, R_f = 0.20) to give the product as a colorless oil (40.5 mg, 49%).

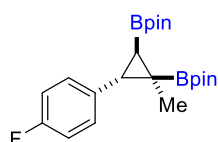
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.13 – 7.09 (m, 2H), 6.81 – 6.76 (m, 2H), 3.78 (s, 3H), 2.45 (d, J = 7.4 Hz, 1H), 1.26 (d, J = 2.4 Hz, 24H), 0.79 (s, 3H), 0.30 (d, J = 7.4 Hz, 1H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 157.7, 131.3, 130.4, 113.1, 83.2, 83.2, 55.2, 31.0, 25.1, 25.0, 24.8, 24.6, 17.5. $^{11}\text{B NMR}$ (96 MHz, CDCl_3) δ 32.5. **HRMS (ESI-TOF):** calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{23}\text{H}_{36}^{10}\text{B}^{11}\text{BO}_5$ 436.2684, found: 436.2691.



2,2'-(1-Methyl-3-(4-(methylthio)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3g)

The title compound was prepared from (*E*)-methyl(4-(prop-1-en-1-yl)phenyl)sulfane (32.8 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 30:1, R_f = 0.20) to give the product as a colorless oil (37.8 mg, 44%).

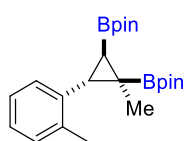
¹H NMR (300 MHz, CDCl₃) δ 7.15 (dd, *J* = 8.7, 6.4 Hz, 4H), 2.45 (d, *J* = 7.4 Hz, 1H), 2.45 (s, 3H), 1.26 – 1.25 (m, 24H), 0.79 (s, 3H), 0.34 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 136.5, 135.1, 130.0, 126.4, 83.3, 83.2, 31.3, 25.0, 25.0, 24.8, 24.6, 17.5, 16.3. ¹¹B NMR (96 MHz, CDCl₃) δ 31.26. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₃H₃₆¹⁰B¹¹BO₄S 452.2456, found: 452.2456.



2,2'-(3-(4-Fluorophenyl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3h)

The title compound was prepared from (*E*)-1-fluoro-4-(prop-1-en-1-yl)benzene (27.2 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (43.4 mg, 54%).

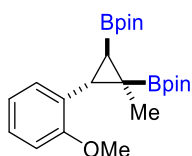
¹H NMR (300 MHz, CDCl₃) δ 7.16 – 7.11 (m, 2H), 6.95 – 6.89 (m, 2H), 2.45 (d, *J* = 7.4 Hz, 1H), 1.26 (d, *J* = 1.4 Hz, 24H), 0.77 (s, 3H), 0.31 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 161.3 (d, *J* = 243.3 Hz), 134.9 (d, *J* = 3.0 Hz), 130.8 (d, *J* = 7.8 Hz), 114.4 (d, *J* = 21.1 Hz), 83.3, 83.2, 30.9, 25.0, 25.0, 24.8, 24.6, 17.5. ¹¹B NMR (96 MHz, CDCl₃) δ 33.4. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₂H₃₃¹⁰B¹¹BO₄F 424.2485, found: 424.2483.



2,2'-(1-Methyl-3-(m-tolyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3i)

The title compound was prepared from (*E*)-1-methyl-3-(prop-1-en-1-yl)benzene (26.4 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a white solid (42.9 mg, 54%). mp: 88-90 °C.

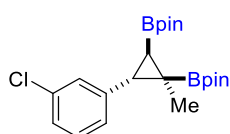
¹H NMR (300 MHz, CDCl₃) δ 7.13 (td, *J* = 7.3, 1.1 Hz, 1H), 7.03 – 6.93 (m, 3H), 2.48 (d, *J* = 7.4 Hz, 1H), 2.31 (s, 3H), 1.28 – 1.24 (m, 24H), 0.81 (s, 3H), 0.38 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 139.1, 137.1, 130.2, 127.5, 126.5, 126.5, 83.2, 83.2, 31.7, 25.1, 25.0, 24.8, 24.7, 21.4, 17.4. ¹¹B NMR (96 MHz, CDCl₃) δ 32.2. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₃H₃₆¹⁰B¹¹BO₄ 420.2735, found: 420.2737.



2,2'-(3-(2-Methoxyphenyl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3j)

The title compound was prepared from (*E*)-1-methoxy-2-(prop-1-en-1-yl)benzene (29.6 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a colorless oil (33.1 mg, 40%).

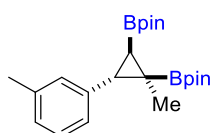
¹H NMR (300 MHz, CDCl₃) δ 7.18 – 7.12 (m, 1H), 7.03 (dd, *J* = 7.4, 0.9 Hz, 1H), 6.86 – 6.78 (m, 2H), 3.79 (s, 3H), 2.46 (d, *J* = 7.9 Hz, 1H), 1.27 – 1.26 (m, 24H), 0.70 (s, 3H), 0.33 (d, *J* = 7.9 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 159.4, 129.6, 128.3, 126.9, 119.6, 109.8, 83.5, 83.1, 55.4, 28.0, 25.0, 25.0, 24.9, 24.4, 17.3. ¹¹B NMR (96 MHz, CDCl₃) δ 30.4. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₃H₃₆¹⁰B¹¹BO₅ 436.2684, found: 436.2692.



2,2'-(3-(3-Chlorophenyl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3k)

The title compound was prepared from (*E*)-1-chloro-3-(prop-1-en-1-yl)benzene (30.4 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a colorless oil (35.1 mg, 42%).

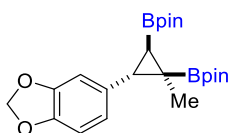
¹H NMR (300 MHz, CDCl₃) δ 7.19 – 7.17 (m, 1H), 7.17 – 7.09 (m, 2H), 7.09 – 7.05 (m, 1H), 2.46 (d, *J* = 7.4 Hz, 1H), 1.26 (d, *J* = 2.5 Hz, 24H), 0.80 (s, 3H), 0.36 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 141.6, 129.5, 128.8, 127.7, 126.0, 125.9, 83.4, 83.3, 31.3, 25.0, 25.0, 24.8, 24.6, 17.5. ¹¹B NMR (96 MHz, CDCl₃) δ 32.8. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₂H₃₃¹⁰B₂ClO₄ 439.2223, found: 439.2216.



2,2'-(1-Methyl-3-(*o*-tolyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3l)

The title compound was prepared from (*E*)-1-methyl-2-(prop-1-en-1-yl)benzene (26.4 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a colorless oil (42.9 mg, 54%).

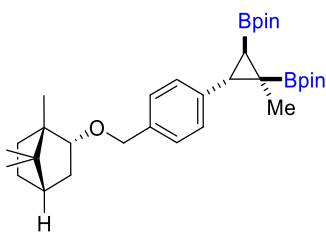
¹H NMR (300 MHz, CDCl₃) δ 7.14 – 7.06 (m, 4H), 2.36 (d, *J* = 7.7 Hz, 1H), 2.31 (s, 3H), 1.27 (s, 12H), 1.26 (d, *J* = 3.1 Hz, 12H), 0.69 (s, 3H), 0.42 (d, *J* = 7.7 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 138.6, 137.8, 129.1, 128.9, 125.9, 125.1, 83.2, 83.1, 31.0, 25.1, 25.0, 24.9, 24.5, 19.5, 17.2. ¹¹B NMR (96 MHz, CDCl₃) δ 32.8. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₃H₃₆¹⁰B¹¹BO₄ 420.2735, found: 420.2733.



2,2'-(3-(Benzo[*d*][1,3]dioxol-5-yl)-1-methylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3m)

The title compound was prepared from (*E*)-5-(prop-1-en-1-yl)benzo[*d*][1,3]dioxole (32.4 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 20:1, *R*_f = 0.20) to give the product as a white solid (46.2 mg, 54%). mp: 120-122 °C.

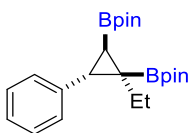
¹H NMR (300 MHz, CDCl₃) δ 6.71 – 6.67 (m, 2H), 6.64 (ddd, *J* = 8.2, 1.5, 0.7 Hz, 1H), 5.92 – 5.88 (m, 2H), 2.42 (d, *J* = 7.4 Hz, 1H), 1.26 (s, 24H), 0.80 (s, 3H), 0.27 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 147.1, 145.6, 133.2, 122.4, 110.1, 107.5, 100.7, 83.2, 83.2, 31.5, 25.0, 25.0, 24.8, 24.6, 17.5. ¹¹B NMR (96 MHz, CDCl₃) δ 32.4. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₃H₃₄¹⁰B¹¹BO₆ 450.2477, found: 450.2483.



2,2'-(1-Methyl-3-(4-(((1S,2R,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)methyl)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3n)

The title compound was prepared from (1*S*,2*R*,4*S*)-1,7,7-trimethyl-2-((4-vinylbenzyl)oxy)bicyclo[2.2.1]heptane (54.0 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a colorless oil (37.4 mg, 34%).

¹H NMR (300 MHz, CDCl₃) δ 7.23 – 7.13 (m, 4H), 4.56 – 4.38 (m, 2H), 3.66 (d, *J* = 9.4 Hz, 1H), 2.50 (d, *J* = 7.4 Hz, 1H), 2.15 – 2.04 (m, 2H), 1.66 (dt, *J* = 21.1, 3.9 Hz, 4H), 1.26 (d, *J* = 3.1 Hz, 24H), 0.87 (d, *J* = 1.0 Hz, 3H), 0.84 – 0.80 (m, 9H), 0.38 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 138.1, 136.9, 129.3, 126.7, 84.1, 83.2, 83.2, 71.5, 49.3, 47.8, 45.1, 36.1, 31.6, 28.3, 26.8, 25.1, 25.0, 24.8, 24.6, 19.8, 18.9, 17.5, 14.0. ¹¹B NMR (96 MHz, CDCl₃) δ 32.7. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₃₃H₅₂¹⁰B¹¹BO₅ 572.3937, found: 572.3947.

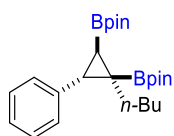


2,2'-(1-Ethyl-3-phenylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3o)

The title compound was prepared from (*E*)-but-1-en-1-ylbenzene (26.4 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, *R*_f = 0.20) to give the product as a white solid (39.0 mg, 49%). mp: 85-87 °C.

¹H NMR (300 MHz, CDCl₃) δ 7.23 (d, *J* = 4.4 Hz, 4H), 7.14 (ddd, *J* = 8.1, 4.9, 3.6 Hz, 1H), 2.56 (d, *J* = 7.4 Hz, 1H), 1.28 (s, 12H), 1.25 (d, *J* = 3.9 Hz, 12H), 1.19 – 1.02 (m, 2H), 0.72 (t, *J* = 7.3 Hz, 3H), 0.43 (d, *J* = 7.4 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 139.3, 129.3, 127.5, 125.6, 83.1, 83.1, 31.9, 25.6, 25.1, 25.0, 24.8, 24.6, 13.1.

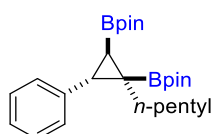
¹¹B NMR (96 MHz, CDCl₃) δ 32.5. **HRMS (ESI-TOF):** calcd for [M+Na]⁺ C₂₃H₃₆¹⁰B¹¹BO₄ 420.2735, found: 420.2731.



2,2'-(1-Butyl-3-phenylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3p)

The title compound was prepared from (*E*)-hex-1-en-1-ylbenzene (32 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (41.7 mg, 49%).

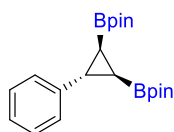
¹H NMR (300 MHz, CDCl₃) δ 7.22 (d, *J* = 4.3 Hz, 4H), 7.17 – 7.12 (m, 1H), 2.53 (d, *J* = 7.4 Hz, 1H), 1.28 (s, 12H), 1.25 (d, *J* = 4.1 Hz, 12H), 1.06 (m, 6H), 0.65 (t, *J* = 7.2 Hz, 3H), 0.43 (d, *J* = 7.4 Hz, 1H). **¹³C NMR (75 MHz, CDCl₃)** δ 139.3, 129.3, 127.5, 125.6, 83.1, 32.2, 31.8, 31.0, 25.2, 25.0, 24.9, 24.6, 22.8, 13.9. **¹¹B NMR (96 MHz, CDCl₃)** δ 33.0.



2,2'-(1-Pentyl-3-phenylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (3q)

The title compound was prepared from (*E*)-hept-1-en-1-ylbenzene (34.8 mg, 0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (36.1 mg, 41%).

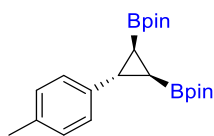
¹H NMR (300 MHz, CDCl₃) δ 7.22 (d, *J* = 4.2 Hz, 4H), 7.17 – 7.11 (m, 1H), 2.53 (d, *J* = 7.4 Hz, 1H), 1.27 (s, 12H), 1.25 (d, *J* = 4.2 Hz, 12H), 1.13 – 0.84 (m, 8H), 0.75 – 0.66 (m, 3H), 0.43 (d, *J* = 7.4 Hz, 1H). **¹³C NMR (75 MHz, CDCl₃)** δ 139.3, 129.3, 127.5, 125.5, 83.1, 83.1, 32.5, 32.0, 31.7, 28.3, 25.2, 25.0, 24.9, 24.6, 22.3, 13.9. **¹¹B NMR (96 MHz, CDCl₃)** δ 33.1. **HRMS (ESI-TOF):** calcd for [M+Na]⁺ C₂₆H₄₂¹⁰B¹¹BO₄ 462.3205, found: 462.3211.



2,2'-(3-Phenylcyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4a)

The title compound was prepared from styrene (20.8 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a white solid (40.7 mg, 55%). mp: 124-126 °C.

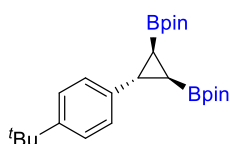
¹H NMR (300 MHz, CDCl₃) δ 7.25 – 7.20 (m, 2H), 7.11 (td, *J* = 6.8, 1.5 Hz, 3H), 2.27 (t, *J* = 6.4 Hz, 1H), 1.26 (s, 24H), 0.58 (d, *J* = 6.4 Hz, 2H). **¹³C NMR (75 MHz, CDCl₃)** δ 143.7, 128.2, 125.5, 125.5, 83.3, 25.9, 25.0, 24.8. **¹¹B NMR (96 MHz, CDCl₃)** δ 31.98. **HRMS (ESI-TOF):** calcd for [M+Na]⁺ C₂₁H₃₂¹⁰B¹¹BO₄ 392.2422, found: 392.2425.



2,2'-(3-(*p*-Tolyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4b)

The title compound was prepared from 1-methyl-4-vinylbenzene (23.6 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (33.0 mg, 43%).

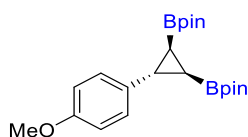
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.05 – 6.98 (m, 4H), 2.28 (s, 3H), 2.24 (t, J = 6.4 Hz, 1H), 1.25 (s, 24H), 0.54 (d, J = 6.4 Hz, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 140.6, 135.0, 128.8, 125.4, 83.2, 25.6, 25.0, 24.7, 20.9. $^{11}\text{B NMR}$ (128 MHz, CDCl_3) δ 32.3. **HRMS (ESI-TOF)**: calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{22}\text{H}_{34}^{10}\text{B}^{11}\text{BO}_4$ 406.2579, found: 406.2576.



2,2'-(3-(4-(*tert*-Butyl)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4c)

The title compound was prepared from 1-(*tert*-butyl)-4-vinylbenzene (32 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (45 mg, 53%).

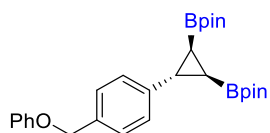
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.26 (s, 2H), 7.04 (d, J = 8.3 Hz, 2H), 2.26 (t, J = 6.4 Hz, 1H), 1.28 (s, 9H), 1.25 (s, 24H), 0.57 (d, J = 6.4 Hz, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 148.4, 140.7, 125.1, 125.0, 83.2, 34.3, 31.4, 25.6, 25.0, 24.7. $^{11}\text{B NMR}$ (128 MHz, CDCl_3) δ 31.9. **HRMS (ESI-TOF)**: calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{25}\text{H}_{40}^{10}\text{B}^{11}\text{BO}_4$ 448.3048, found: 448.3053.



2,2'-(3-(4-Methoxyphenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4d)

The title compound was prepared from 1-methoxy-4-vinylbenzene (26.8 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a colorless oil (40.8 mg, 51%).

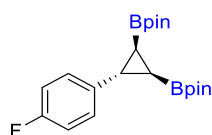
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.02 (d, J = 8.6 Hz, 2H), 6.78 (d, J = 8.8 Hz, 2H), 3.76 (s, 3H), 2.23 (t, J = 6.4 Hz, 1H), 1.25 (s, 24H), 0.50 (d, J = 6.5 Hz, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 157.6, 135.7, 126.5, 113.7, 83.2, 55.3, 25.3, 25.0, 24.7. $^{11}\text{B NMR}$ (128 MHz, CDCl_3) δ 30.3. **HRMS (ESI-TOF)**: calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{22}\text{H}_{34}^{10}\text{B}^{11}\text{BO}_5$ 422.2527, found: 422.2532.



2,2'-(3-(4-(Phoxymethyl)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4e)

The title compound was prepared from 1-(phoxymethyl)-4-vinylbenzene (42 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a white solid (44.7 mg, 47%). mp: 120-122 °C.

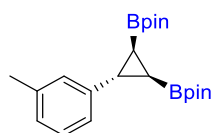
¹H NMR (400 MHz, CDCl₃) δ 7.43 – 7.34 (m, 5H), 7.02 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 5.03 (s, 2H), 2.23 (t, *J* = 6.4 Hz, 1H), 1.25 (s, 24H), 0.50 (d, *J* = 6.4 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 156.8, 137.2, 136.0, 128.5, 127.8, 127.4, 126.5, 114.7, 83.2, 70.1, 25.3, 25.0, 24.7. ¹¹B NMR (128 MHz, CDCl₃) δ 30.5. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₈H₃₈¹⁰B¹¹BO₅ 498.2841, found: 498.2827.



2,2'-(3-(4-Fluorophenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4f)

The title compound was prepared from 1-fluoro-4-vinylbenzene (24.4 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a white solid (44.2 mg, 57%). mp: 108-110 °C.

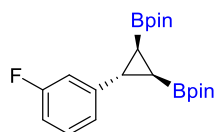
¹H NMR (300 MHz, CDCl₃) δ 7.06 – 7.01 (m, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 2.24 (t, *J* = 6.4 Hz, 1H), 1.26 (s, 24H), 0.51 (d, *J* = 6.4 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 157.8 (d, *J* = 258.1 Hz), 139.2 (d, *J* = 2.7 Hz), 126.9 (d, *J* = 7.9 Hz), 114.9 (d, *J* = 21.3 Hz), 83.3, 25.2, 25.0, 24.7. ¹¹B NMR (96 MHz, CDCl₃) δ 32.1. ¹⁹F NMR (282 MHz, CDCl₃) δ -118.09. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₁H₃₁¹⁰B₂O₄F 409.2362, found: 409.2366.



2,2'-(3-(*m*-Tolyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4g)

The title compound was prepared from 1-methyl-3-vinylbenzene (23.6 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 50:1, R_f = 0.20) to give the product as a white solid (39.2 mg, 51%). mp: 112-114 °C.

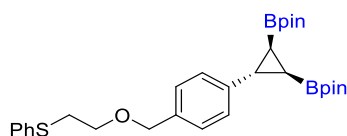
¹H NMR (300 MHz, CDCl₃) δ 7.14 – 7.10 (m, 1H), 7.04 (s, 1H), 6.94 – 6.90 (m, 2H), 2.28 (s, 3H), 2.24 (t, *J* = 6.4 Hz, 1H), 1.25 (s, 24H), 0.57 (d, *J* = 6.4 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 143.7, 137.7, 128.1, 126.5, 126.3, 122.4, 83.3, 25.9, 25.0, 24.8, 21.4. ¹¹B NMR (96 MHz, CDCl₃) δ 32.1. HRMS (ESI-TOF): calcd for [M+Na]⁺ C₂₂H₃₄¹⁰B¹¹BO₄ 406.2579, found: 406.2578.



2,2'-(3-(3-Fluorophenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4h)

The title compound was prepared from 1-fluoro-3-vinylbenzene (24.4 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 20:1, Rf = 0.20) to give the product as a white solid (31.1 mg, 40%). mp: 106-108 °C.

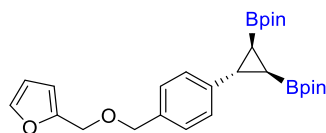
¹H NMR (300 MHz, CDCl₃) δ 7.13 – 7.07 (m, 1H), 6.82 – 6.79 (m, 1H), 6.76 – 6.68 (m, 2H), 2.18 (t, *J* = 6.3 Hz, 1H), 1.19 (s, 24H), 0.49 (d, *J* = 6.4 Hz, 2H). **¹³C NMR (75 MHz, CDCl₃)** δ 163.1 (d, *J* = 244.7 Hz), 129.5 (d, *J* = 8.5 Hz), 121.2 (d, *J* = 2.5 Hz), 112.3 (d, *J* = 23.3 Hz), 83.4, 25.6 (d, *J* = 2.0 Hz), 25.0, 24.7. **¹¹B NMR (96 MHz, CDCl₃)** δ 31.9. **¹⁹F NMR (282 MHz, CDCl₃)** δ -114.21. **HRMS (ESI-TOF):** calcd for [M+Na]⁺ C₂₁H₃₁¹⁰B₂O₄F 409.2362, found: 409.2361.



2,2'-(3-(4-((2-(Phenylthio)ethoxy)methyl)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4i)

The title compound was prepared from phenyl(2-((4-vinylbenzyl)oxy)ethyl)sulfane (54 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 20:1, Rf = 0.20) to give the product as a colorless oil (60 mg, 56%).

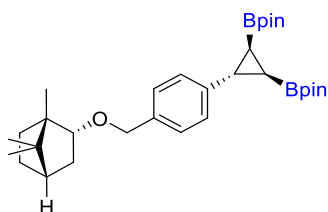
¹H NMR (300 MHz, CDCl₃) δ 7.34 – 7.31 (m, 2H), 7.26 – 7.22 (m, 1H), 7.21 – 7.15 (m, 4H), 7.06 (d, *J* = 8.2 Hz, 2H), 4.46 (s, 2H), 3.61 (t, *J* = 6.9 Hz, 2H), 3.11 (t, *J* = 6.9 Hz, 2H), 2.27 (t, *J* = 6.4 Hz, 1H), 1.26 (s, 24H), 0.56 (d, *J* = 6.4 Hz, 2H). **¹³C NMR (75 MHz, CDCl₃)** δ 143.3, 136.0, 135.1, 129.3, 128.9, 127.8, 126.1, 125.5, 83.3, 72.8, 68.3, 33.3, 25.7, 25.0, 24.7. **¹¹B NMR (96 MHz, CDCl₃)** δ 32.6. **HRMS (ESI-TOF):** calcd for [M+Na]⁺ C₃₀H₄₂¹⁰B₂O₅S 557.2908, found: 557.2911.



2,2'-(3-(4-((Furan-2-ylmethoxy)methyl)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4j)

The title compound was prepared from 2-(((4-vinylbenzyl)oxy)methyl)furan (42.8 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 20:1, Rf = 0.20) to give the product as a colorless oil (44.7 mg, 48%).

¹H NMR (300 MHz, CDCl₃) δ 7.41 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.23 – 7.20 (m, 2H), 7.08 (d, *J* = 8.2 Hz, 2H), 6.34 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.30 (dd, *J* = 3.2, 0.8 Hz, 1H), 4.49 (s, 2H), 4.41 (s, 2H), 2.27 (t, *J* = 6.4 Hz, 1H), 1.26 (s, 24H), 0.57 (d, *J* = 6.4 Hz, 2H). **¹³C NMR (75 MHz, CDCl₃)** δ 151.9, 143.4, 142.8, 135.0, 128.0, 125.6, 110.2, 109.3, 83.3, 71.6, 63.3, 25.7, 25.0, 24.8. **¹¹B NMR (96 MHz, CDCl₃)** δ 32.9. **HRMS (ESI-TOF):** calcd for [M+Na]⁺ C₂₇H₃₈¹⁰B₂O₆ 501.2824, found: 501.2838.

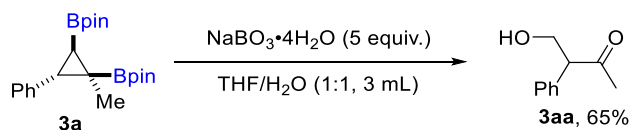


2,2'-(3-(4-(((1*S*,2*R*,4*S*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)oxy)methyl)phenyl)cyclopropane-1,2-diyl)bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolane) (4k)

The title compound was prepared from (1*S*,2*R*,4*S*)-1,7,7-trimethyl-2-((4 vinylbenzyl)oxy)bicyclo[2.2.1]heptane (54 mg, 0.2 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 30:1, R_f = 0.20) to give the product as a colorless oil (43.9 mg, 41%).

$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.19 (d, J = 8.1 Hz, 2H), 7.06 (d, J = 8.2 Hz, 2H), 4.44 (d, J = 28.0 Hz, 2H), 3.67 – 3.63 (m, 1H), 2.27 (t, J = 6.4 Hz, 1H), 2.11 – 2.05 (m, 2H), 1.66 – 1.61 (m, 2H), 1.25 (s, 24H), 1.09 (d, J = 1.9 Hz, 2H), 1.04 (d, J = 3.3 Hz, 1H), 0.87 (s, 3H), 0.84 (s, 3H), 0.81 (s, 3H), 0.57 (d, J = 6.4 Hz, 2H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 142.7, 136.8, 127.3, 125.4, 83.9, 83.3, 71.3, 49.3, 47.8, 45.1, 36.1, 28.3, 26.8, 25.8, 25.0, 24.8, 19.8, 18.9, 14.0. $^{11}\text{B NMR}$ (96 MHz, CDCl_3) δ 32.7. **HRMS (ESI-TOF):** calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{32}\text{H}_{50}^{10}\text{B}^{11}\text{BO}_5$ 558.3781, found: 558.3783.

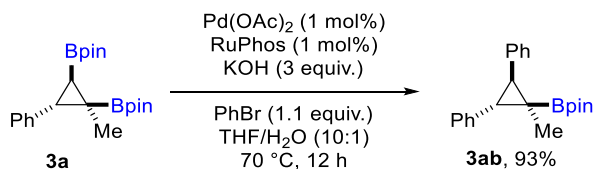
5 Derivatization of 3a



4-Hydroxy-3-phenylbutan-2-one (3aa)

The title compound was synthesized according to the following procedure^[5]: To the boration product **3a** (38.4 mg, 0.1 mmol) in THF (1.5 mL) and water (1.5 mL) was added $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$ (76.5 mg, 5 equiv.). The reaction mixture was stirred vigorously for 0.5 h at room temperature. The reaction mixture was quenched with water and then extracted with EtOAc (5 mL). The combined organic layers were washed with brine (15 mL), dried over Na_2SO_4 and concentrated. The crude product was purified by column chromatography on silica gel to afford the corresponding product **3aa** as colorless oil (10.7 mg, 65%)

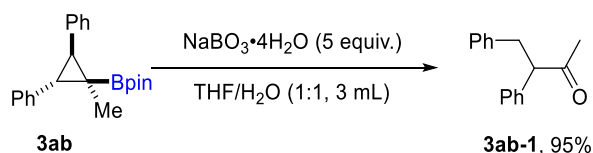
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.40 – 7.27 (m, 3H), 7.25 – 7.14 (m, 2H), 4.15 (dd, $J = 11.3, 8.6$ Hz, 1H), 3.89 (dd, $J = 8.6, 4.7$ Hz, 1H), 3.71 (dd, $J = 11.3, 4.7$ Hz, 1H), 2.09 (d, $J = 0.24$ Hz, 3H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 209.3, 135.5, 129.2, 128.6, 127.9, 64.1, 61.6, 29.5.



4,4,5,5-Tetramethyl-2-(1-methyl-2,3-diphenylcyclopropyl)-1,3,2-dioxaborolane (3ab)

The title compound was synthesized according to the following procedure^[6]: A 4 mL screw-cap vial with a magnetic stir bar was charged with $\text{Pd}(\text{OAc})_2$ (0.23 mg, 1 mol%), RuPhos (0.47 mg, 1 mol%), KOH (16.8 mg, 3 equiv.), and **3a** (38.4 mg, 0.1 mmol). The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. THF (1 mL) was added, followed by H_2O (100 μL), and bromobenzene (15.6 mg, 1 equiv.). The vial was quickly exchanged for a Teflon-lined screw cap and the reaction was stirred at 70°C for 12 h. At this time, the reaction was allowed to cool to room temperature and the reaction was then quenched upon addition of water (5 mL) and the mixture was extracted with EtOAc (3 mL). The combined organic was dried using Na_2SO_4 and then concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford the corresponding product **3ab** as colorless oil (31.1 mg, 93%).

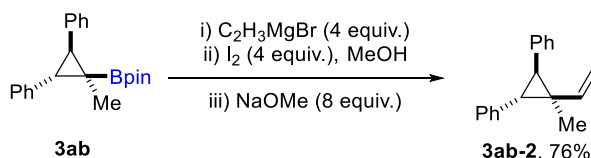
$^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.29 – 7.22 (m, 5H), 7.19 – 7.07 (m, 5H), 3.03 (d, $J = 6.5$ Hz, 1H), 2.41 (d, $J = 6.5$ Hz, 1H), 0.96 (s, 6H), 0.90 (s, 3H), 0.80 (s, 6H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 140.4, 138.9, 129.6, 129.0, 127.9, 127.8, 126.0, 125.9, 83.1, 35.9, 32.0, 24.8, 24.4, 16.8. $^{11}\text{B NMR}$ (96 MHz, CDCl_3) δ 33.3. HRMS (EI) calculated for $[\text{M}] \text{C}_{22}\text{H}_{27}\text{O}_2^{10}\text{B}_1$ 333.2135, found: 333.2134.



3,4-Diphenylbutan-2-one (**3ab-1**)^[9]

The title compound was synthesized according to the following procedure^[5]: To the boration product **3ab** (33.4 mg, 0.1 mmol) in THF (1.5 mL) and water (1.5 mL) was added NaBO₃•4H₂O (76.5 mg, 5 equiv.). The reaction mixture was stirred vigorously for 0.5 h at room temperature. The reaction mixture was quenched with water and then extracted with EtOAc (5 mL). The combined organic layers were washed with brine (15 mL), dried over Na₂SO₄ and concentrated. The crude product was purified by column chromatography on silica gel to afford the corresponding product **3ab-1** as colorless oil (21.3 mg, 95%)

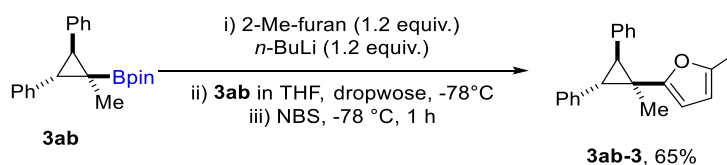
¹H NMR (300 MHz, CDCl₃) δ 7.26 – 7.17 (m, 3H), 7.16 – 7.04 (m, 5H), 7.00 – 6.85 (m, 2H), 3.84 (t, *J* = 7.4 Hz, 1H), 3.35 (dd, *J* = 13.8, 7.5 Hz, 1H), 2.83 (dd, *J* = 13.8, 7.2 Hz, 1H), 1.95 (d, *J* = 0.3 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 207.7, 139.7, 138.5, 129.0, 128.9, 128.4, 128.3, 127.4, 126.1, 61.6, 38.4, 29.6.



(3-Methyl-3-vinylcyclopropane-1,2-diyl)dibenzene (**3ab-2**)

The title compound was synthesized according to the following procedure^[7]: to an oven-dried round bottom flask containing a stirring bar was added a solution of **3ab** (33.4 mg, 0.1 mmol) in THF (2 mL) and subsequently vinylmagnesium bromide (1 M, 0.4 mL, 4.0 equiv.) was added dropwise. The mixture was stirred at room temperature for 30 min. To the above solution at -78 °C, I₂ (102 mg, 2.0 equiv.) in methanol (3.0 mL) was added dropwise. The reaction mixture was allowed to stir 30 min at the same temperature followed by dropwise addition of a solution of NaOMe (44 mg, 8.0 equiv.) in methanol (3 mL). After warming to room temperature, the mixture was stirred for another 1.5 h, diluted with EtOAc (10 mL) and washed sequentially with 10% aqueous solution of Na₂S₂O₃ (5 mL). Then, the mixture was extracted with EtOAc (10 mL), dried over Na₂SO₄, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel to afford the corresponding product **3ab-2** as colorless oil (17.8 mg, 76%).

¹H NMR (300 MHz, CDCl₃) δ 7.32 – 7.19 (m, 8H), 7.19 – 7.07 (m, 2H), 5.29 (dd, *J* = 17.2, 10.6 Hz, 1H), 4.99 (dd, *J* = 17.2, 1.5 Hz, 1H), 4.85 (dd, *J* = 10.6, 1.5 Hz, 1H), 2.70 (d, *J* = 6.9 Hz, 1H), 2.62 (d, *J* = 6.9 Hz, 1H), 1.01 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 142.8, 138.8, 138.3, 129.2, 129.2, 128.2, 126.3, 126.3, 111.6, 36.0, 35.5, 32.1, 17.6. HRMS (EI) calculated for [M] C₁₈H₁₈ 234.1403, found: 234.1398.

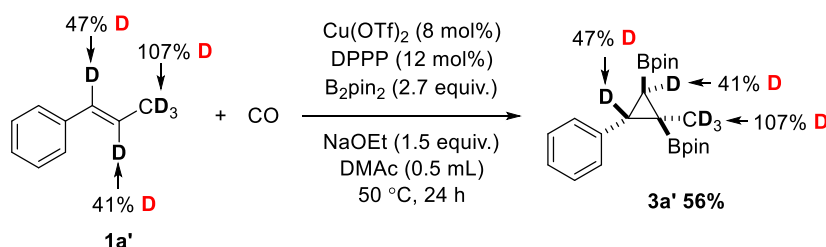


2-Methyl-5-(1-methyl-2,3-diphenylcyclopropyl)furan (**3ab-3**)

The title compound was synthesized according to the following procedure^[8]: A solution of 2-Mefuran (9.8 mg, 1.2 eq.) in THF (0.3 M) was cooled to -78 °C and treated with *n*-BuLi (1.2 eq., 2.5 M in hexanes). The cooling

bath was removed, and the mixture was stirred at room temperature for 1 h. The mixture was cooled to $-78\text{ }^{\circ}\text{C}$ and the **3ab** (33.4 mg, 0.1 mmol.) was added dropwise as a solution in THF (0.5 M). The mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for 1 h. A solution of *N*-bromosuccinimide (NBS, 21.1 mg, 1.2 eq.) in MeOH (0.2 M) was added dropwise. After 1 h at $-78\text{ }^{\circ}\text{C}$, a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ was added and the reaction mixture was allowed to warm to room temperature. The reaction mixture was diluted with Et_2O and water. The layers were separated, and the aqueous layer was extracted with Et_2O . The combined organic layers were dried over Na_2SO_4 , filtered and concentrated under vacuum. Purification by column chromatography on silica gel, gave the desired product **3ab-3** (18.7 mg, 65%).

^1H NMR (300 MHz, CDCl_3) δ 7.40 – 7.32 (m, 4H), 7.29 – 7.26 (m, 1H), 7.23 – 7.11 (m, 5H), 5.85 – 5.77 (m, 1H), 5.72 (dq, $J = 3.0, 1.0$ Hz, 1H), 3.24 (d, $J = 6.9$ Hz, 1H), 2.68 (d, $J = 6.9$ Hz, 1H), 2.09 (d, $J = 0.8$ Hz, 3H), 1.28 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 154.9, 150.2, 138.6, 138.0, 129.3, 128.6, 128.2, 127.6, 126.4, 125.9, 106.3, 105.6, 36.1, 33.6, 29.5, 19.8, 13.4. HRMS (EI) calculated for [M] $\text{C}_{21}\text{H}_{20}\text{O}$ 288.1509, found: 288.1502.



The title compound **3a'** was prepared from **1a'** (0.2 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EA = 50:1, $R_f = 0.20$) to give the product **3a'** as a white solid (43.1 mg, 56%).

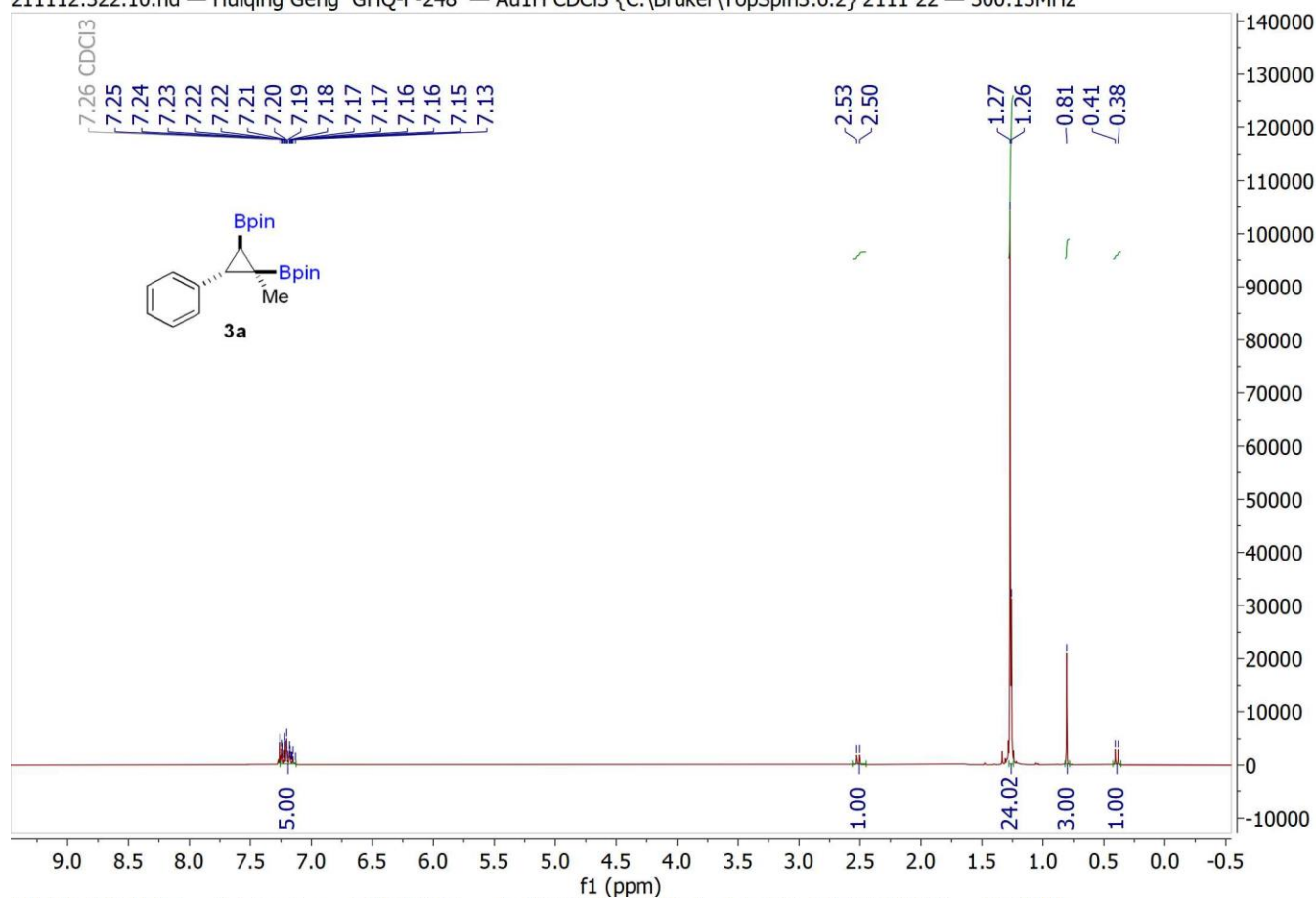
^1H NMR (300 MHz, CDCl_3) δ 7.26 – 7.05 (m, 5H), 2.62 – 2.41 (m, 1H), 1.26 (d, $J = 3.3$ Hz, 24H), 0.82 – 0.77 (m, 2H), 0.41 – 0.37 (m, 1H).

6. Reference

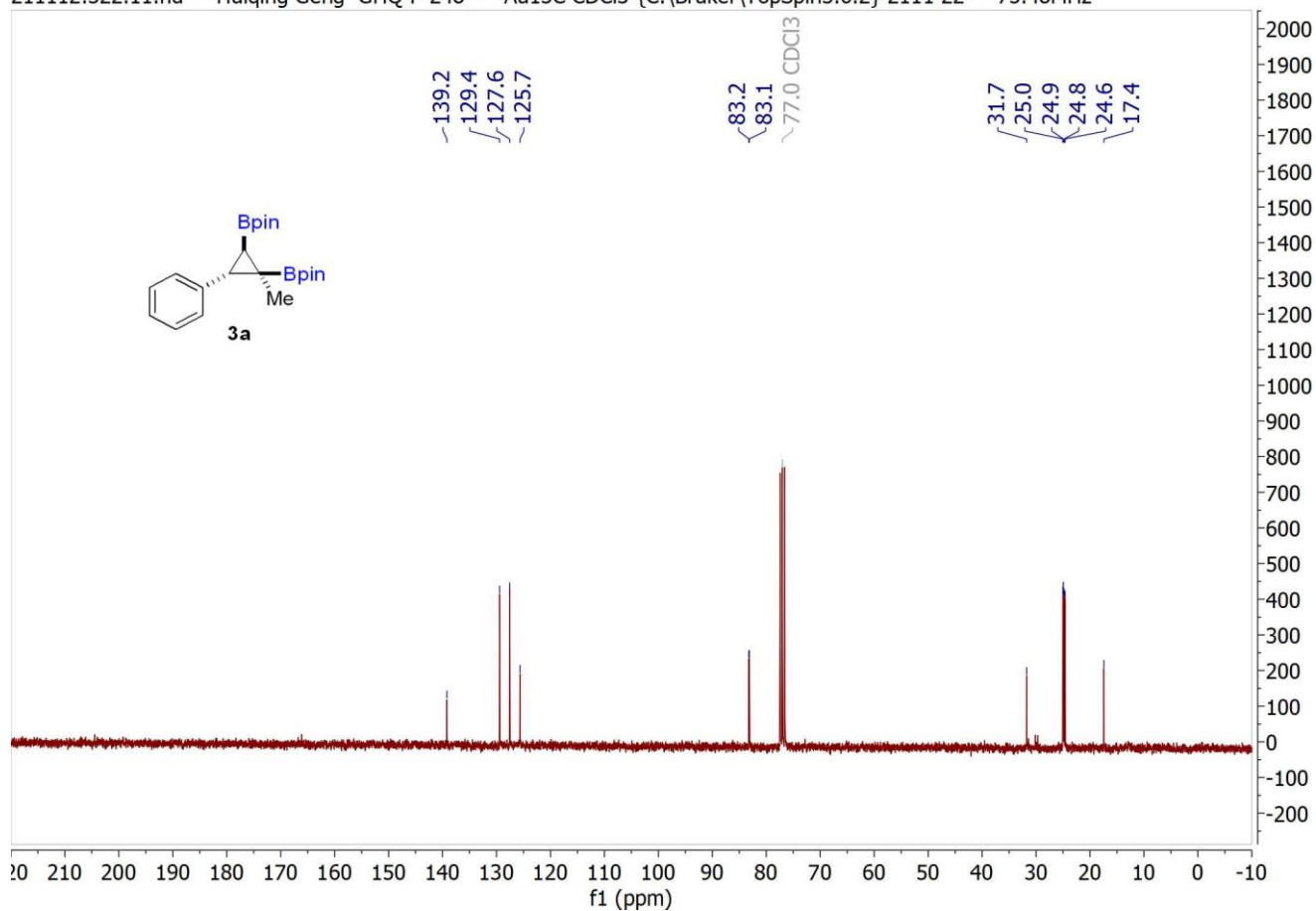
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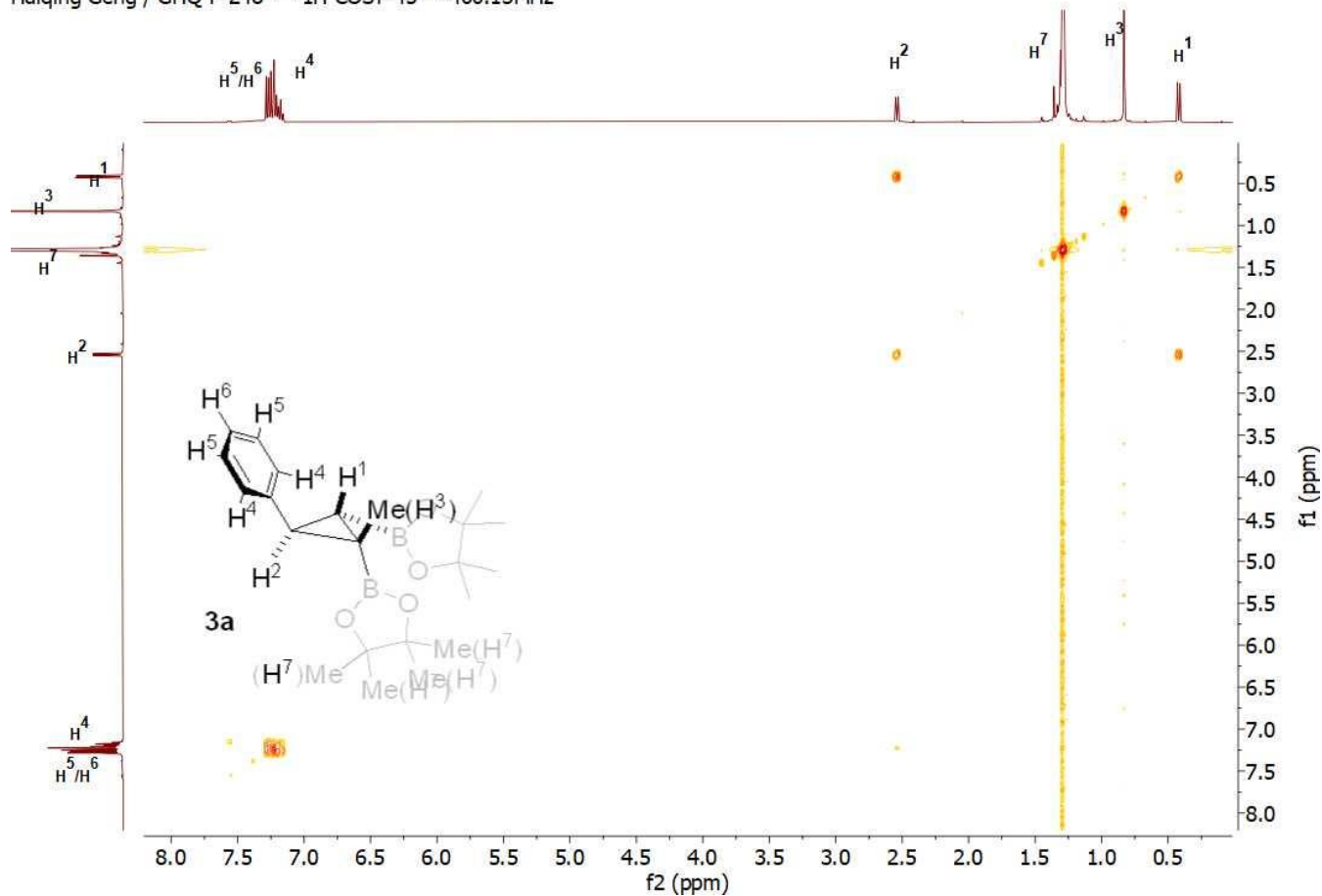
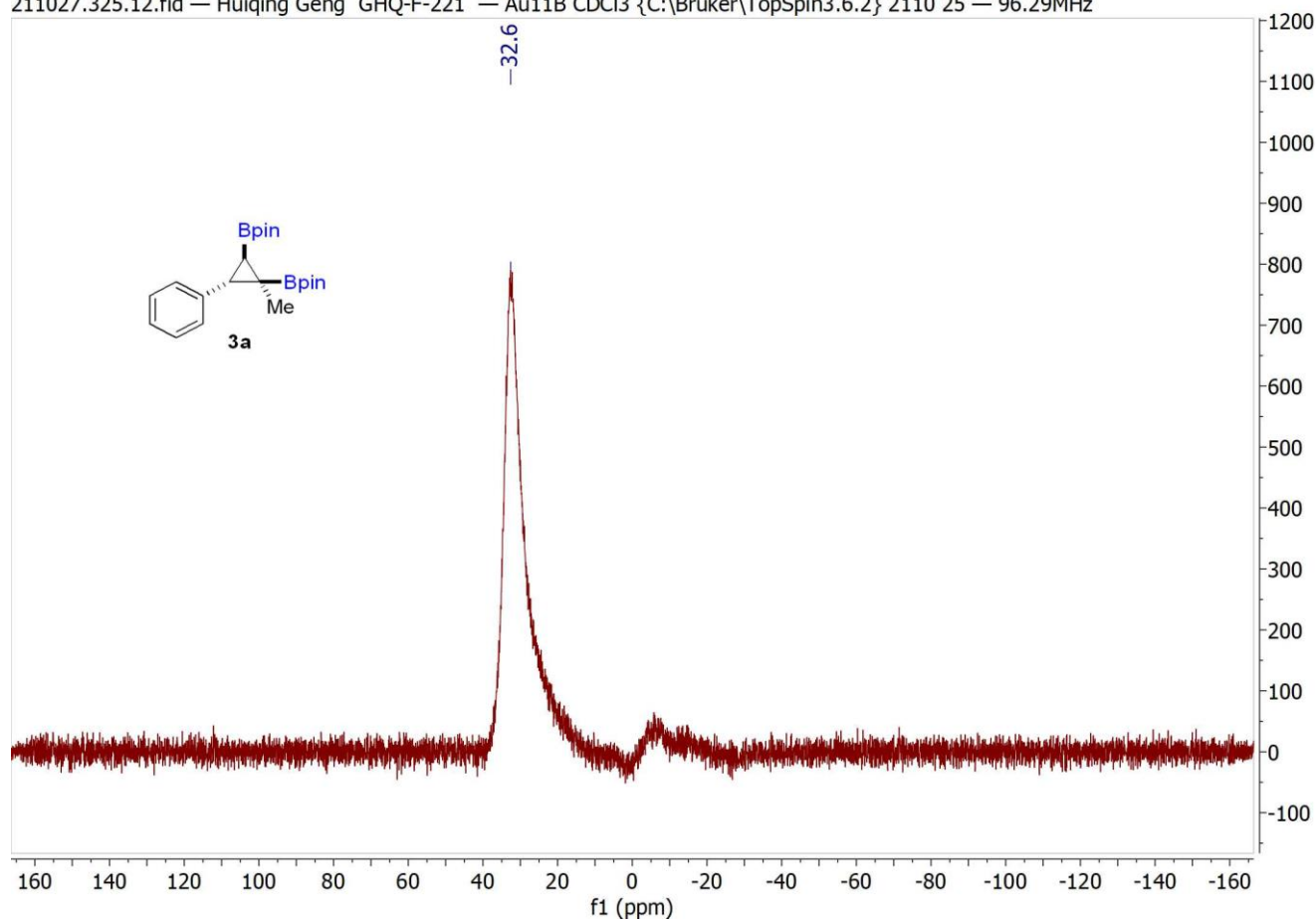
7. Spectra of Products

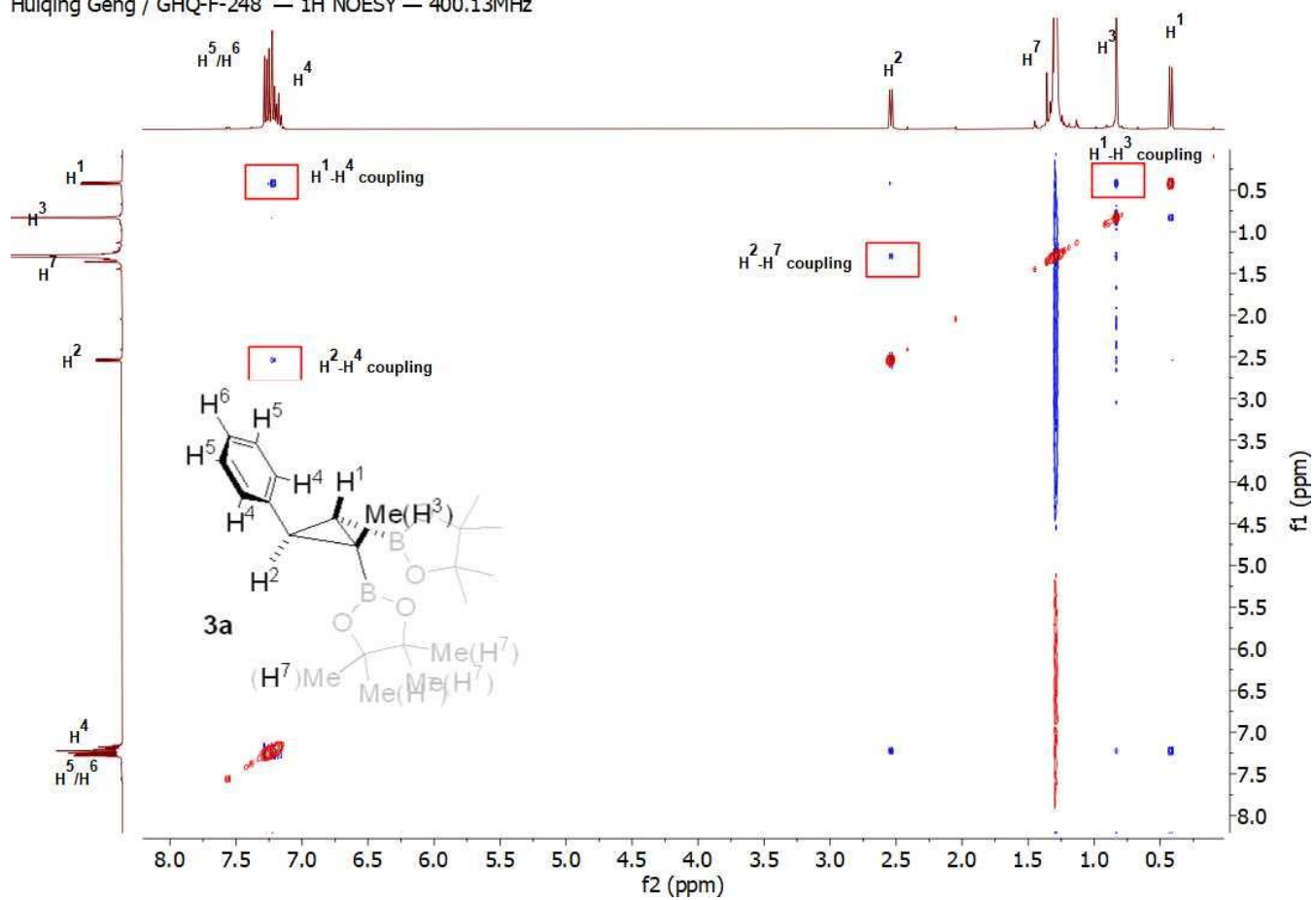
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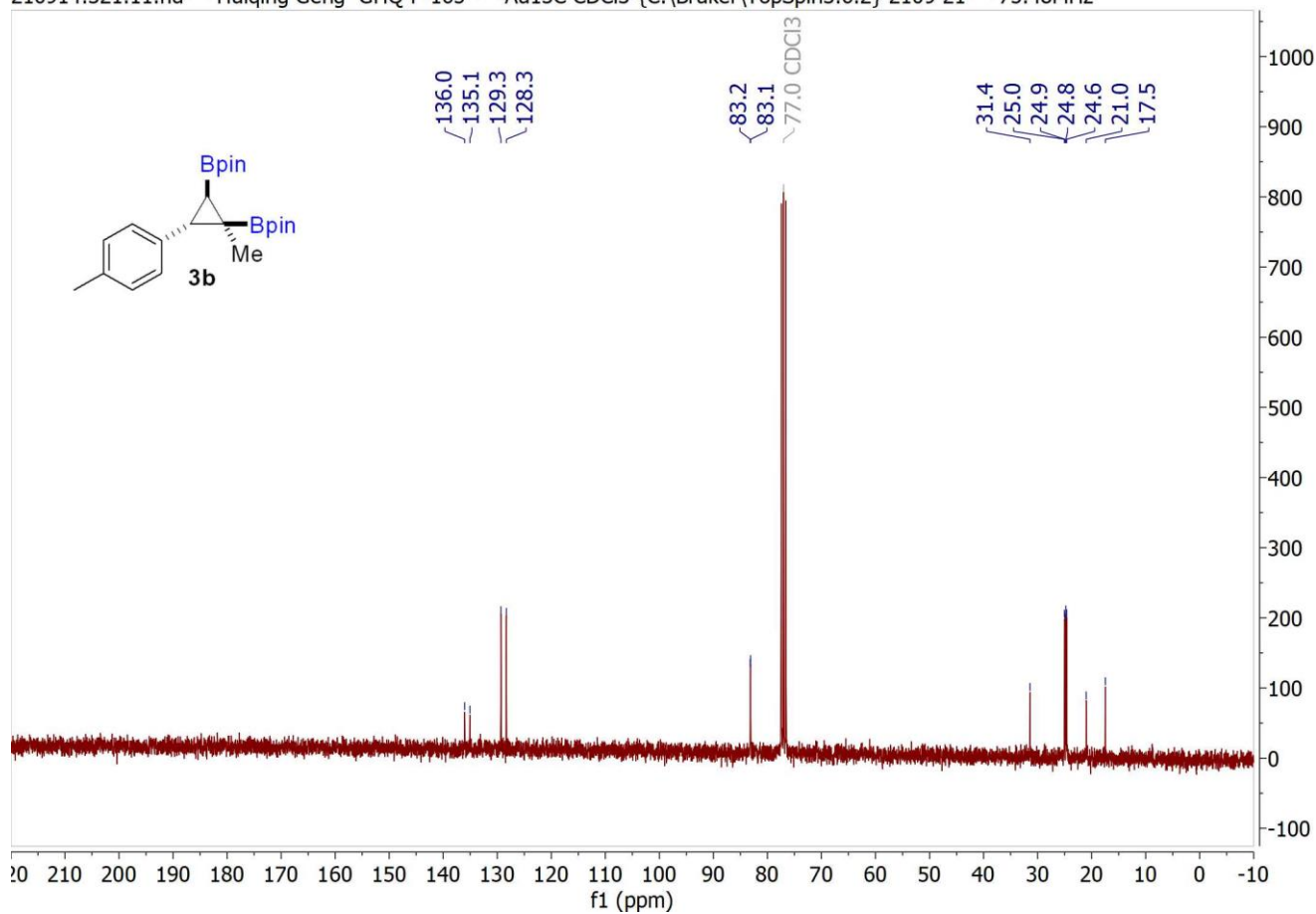
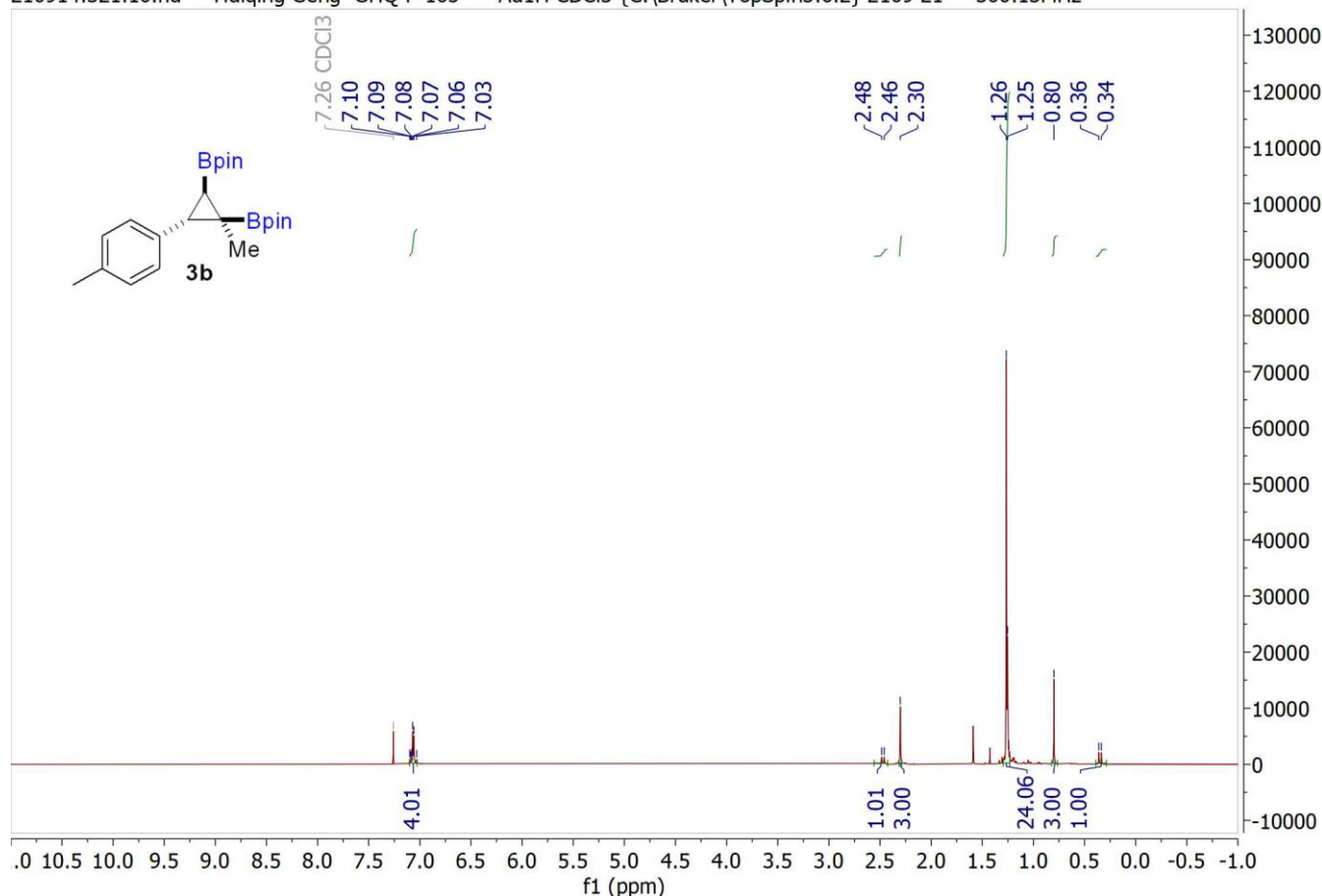


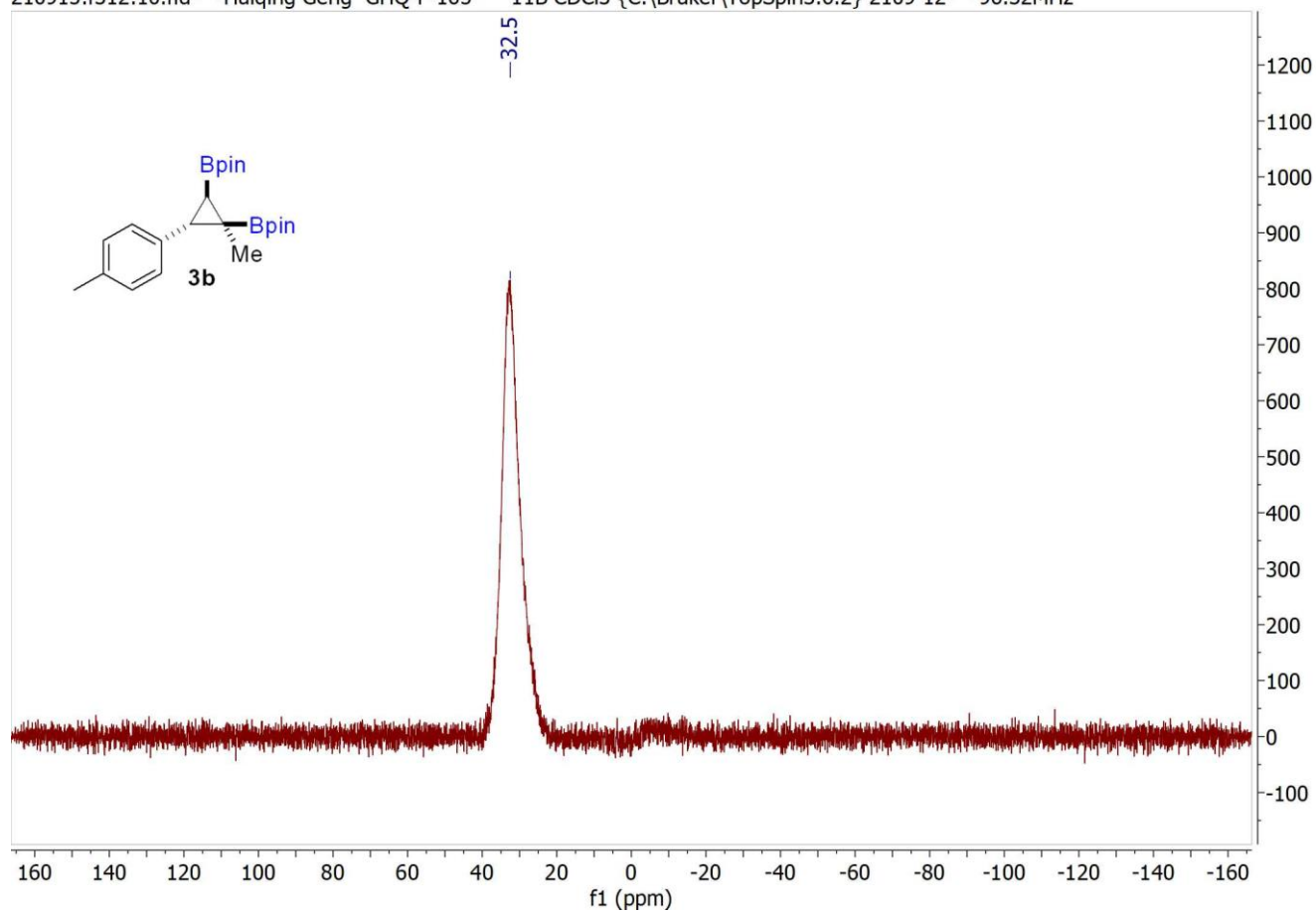
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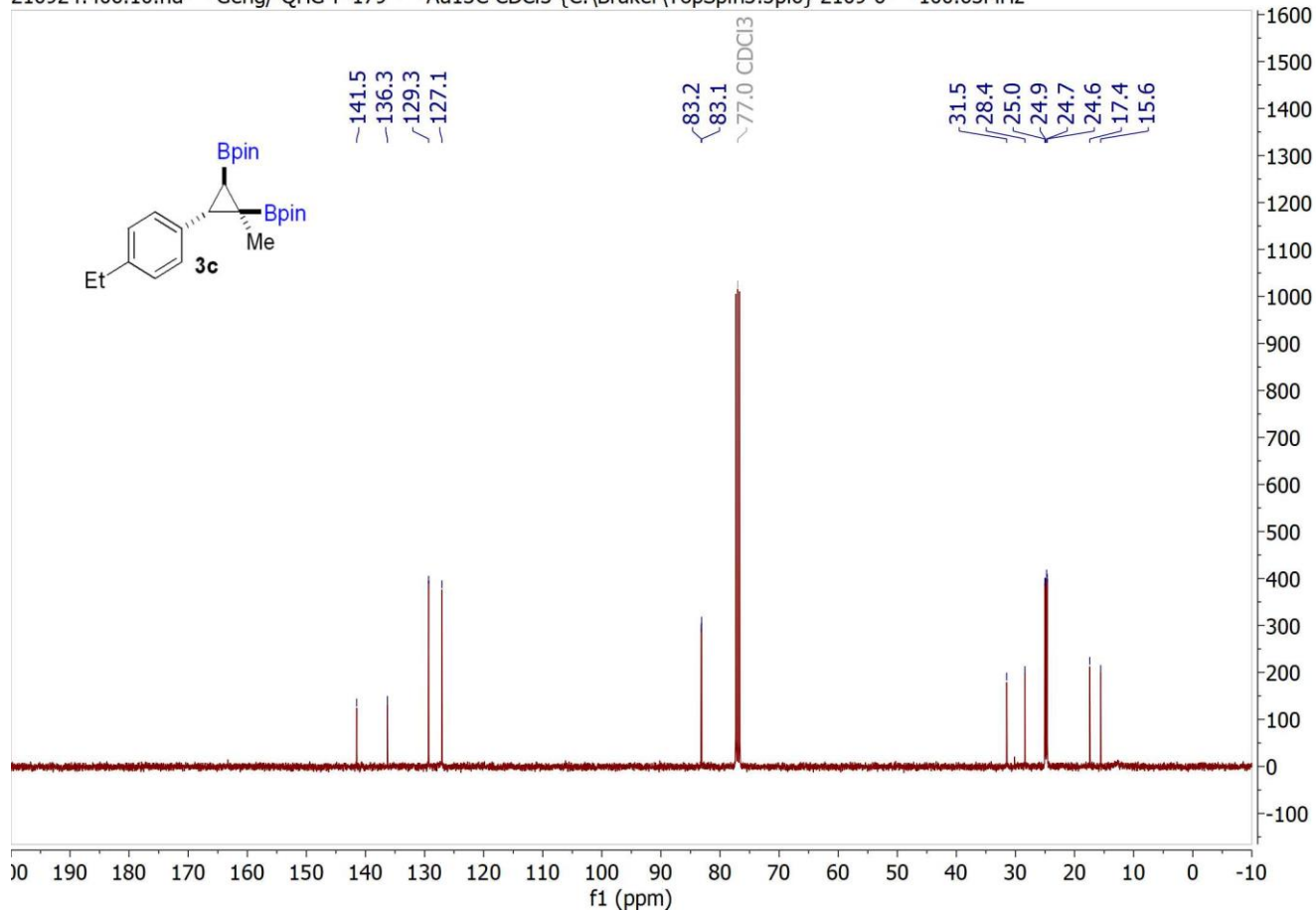
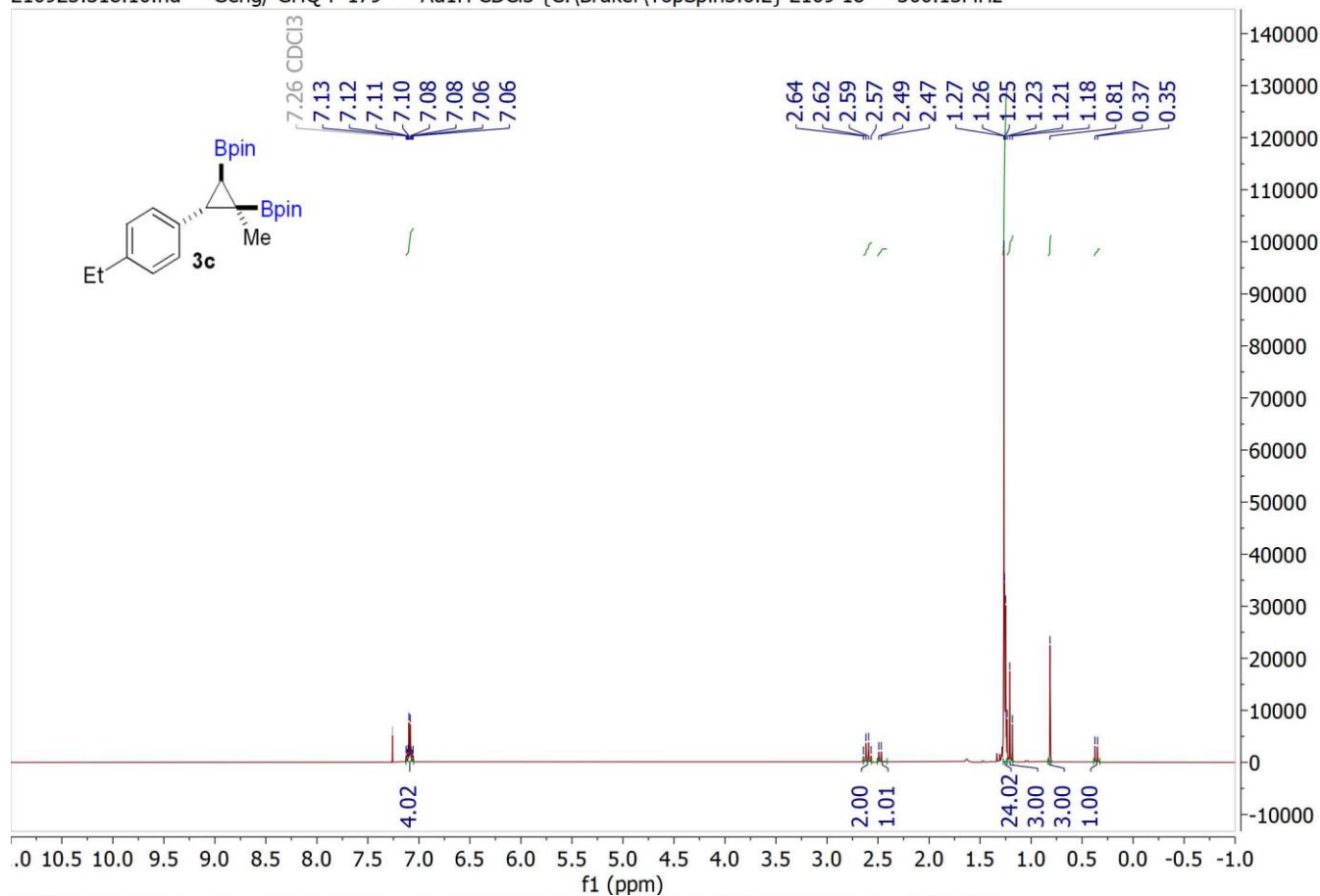


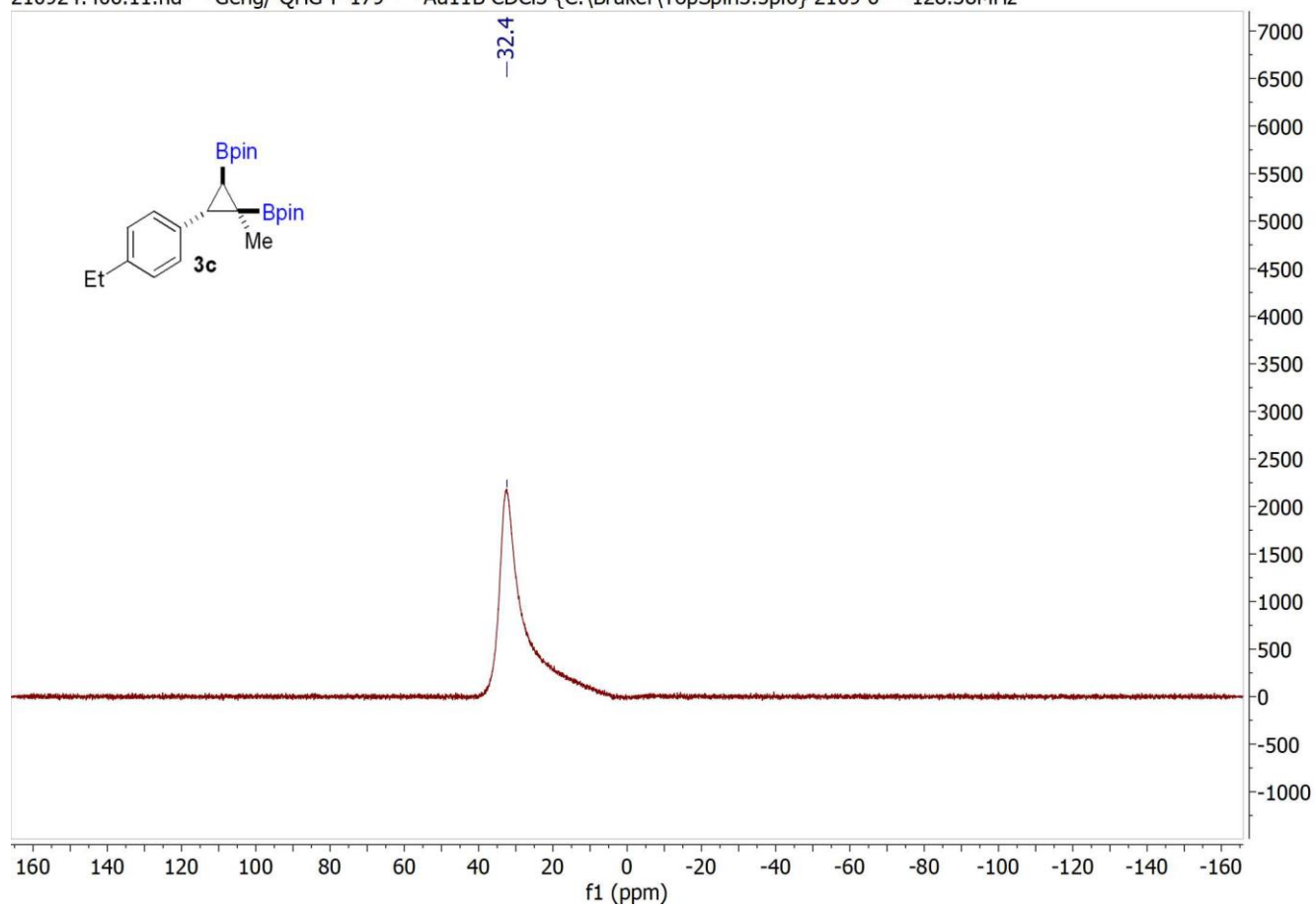




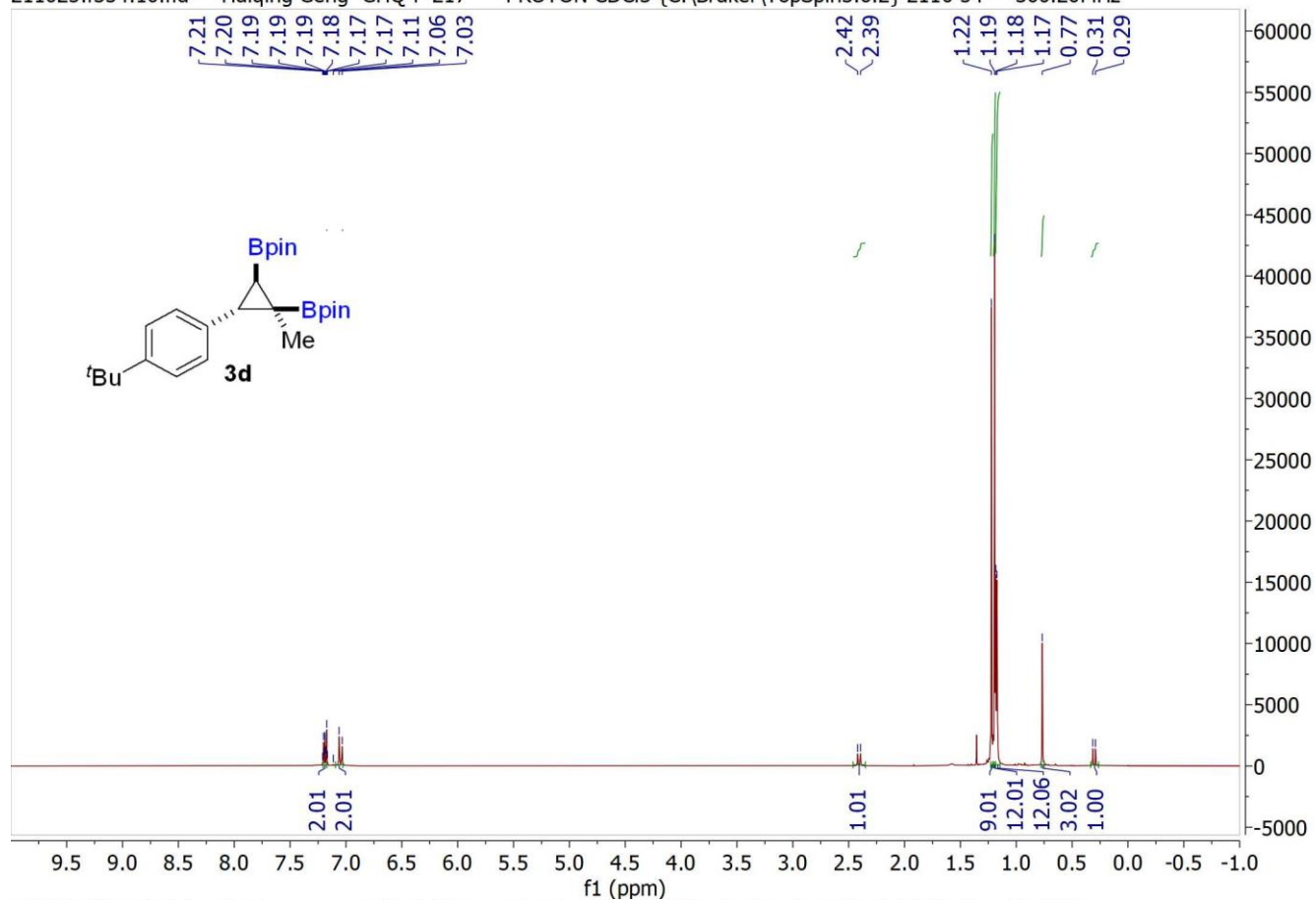




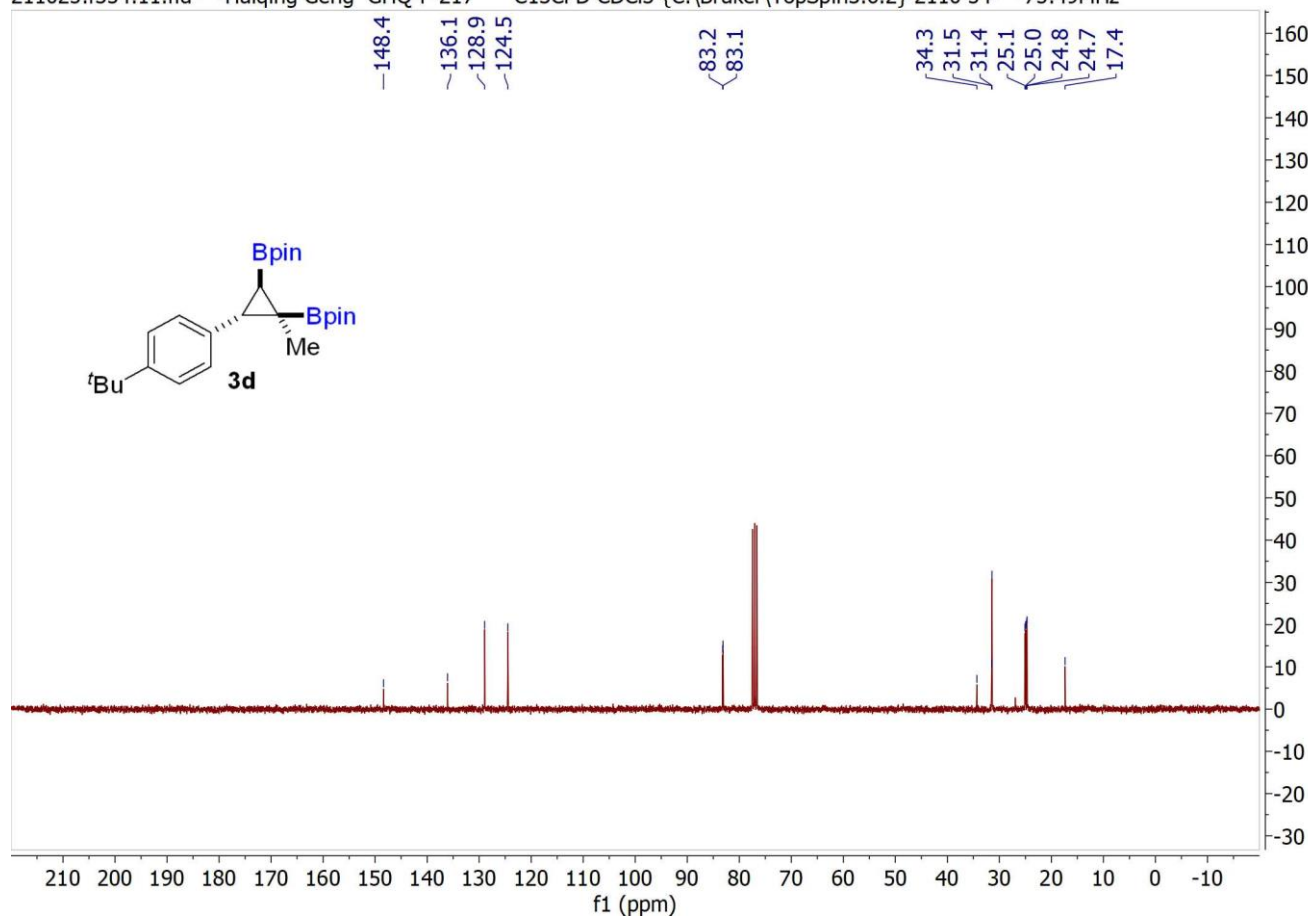


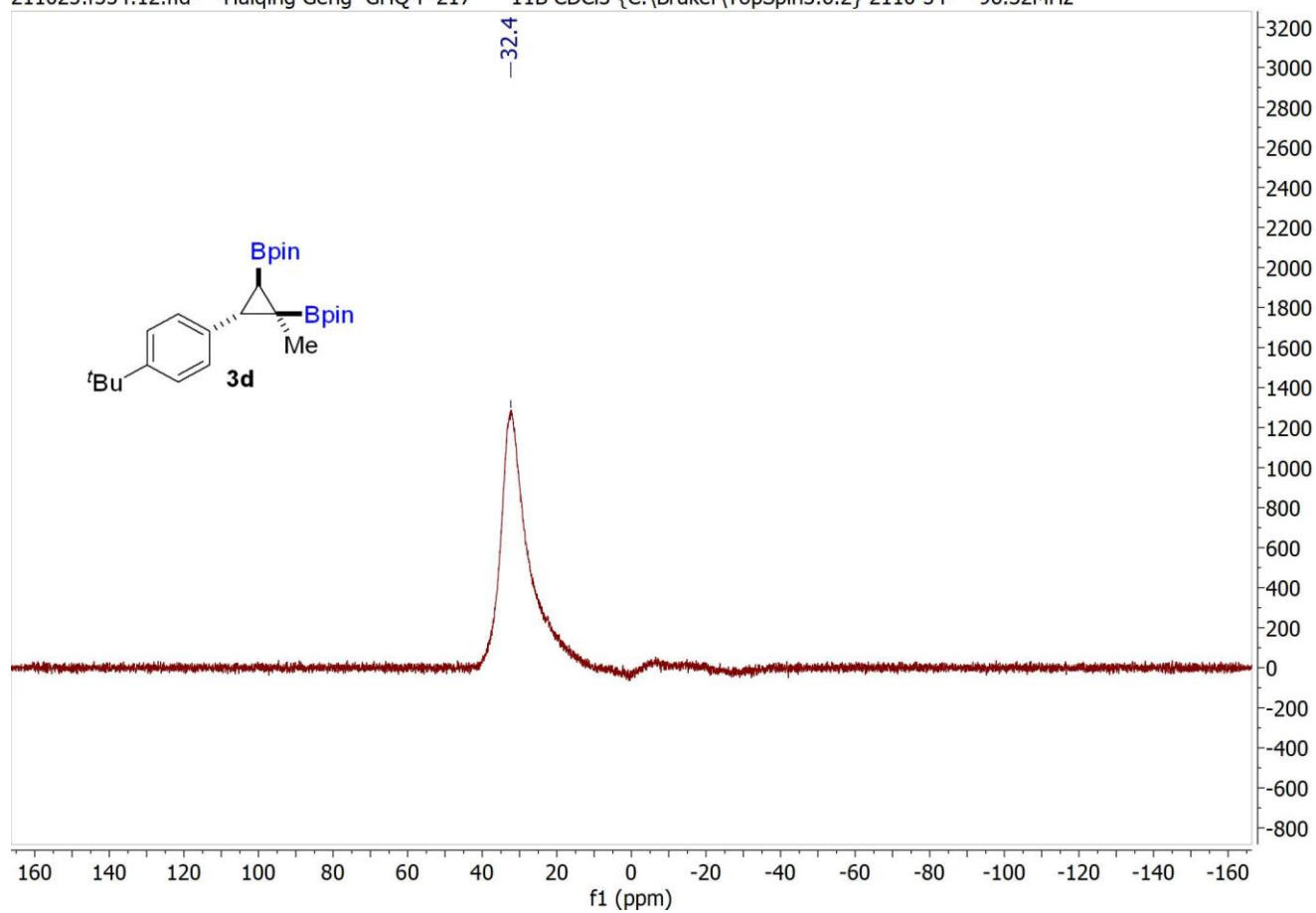


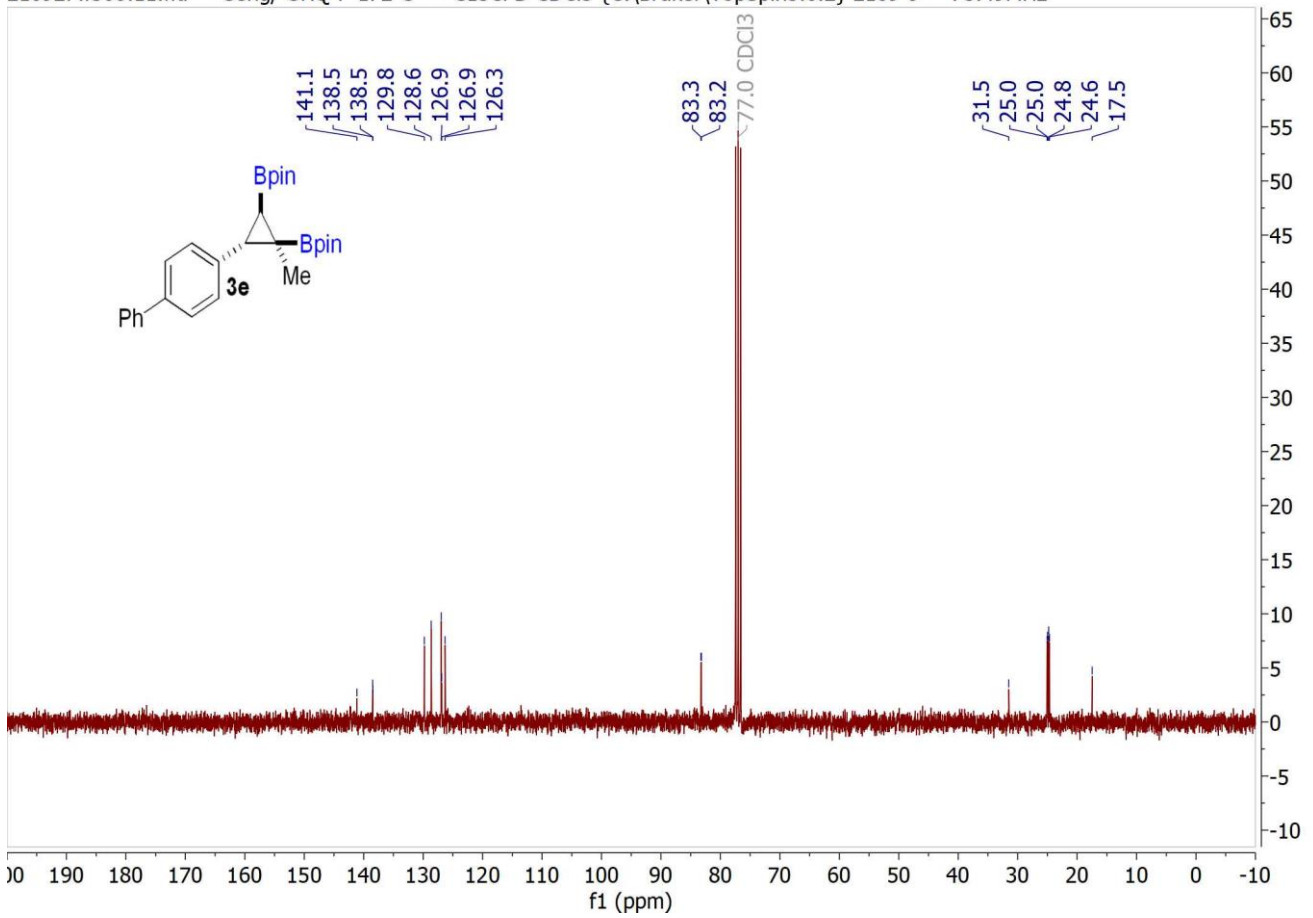
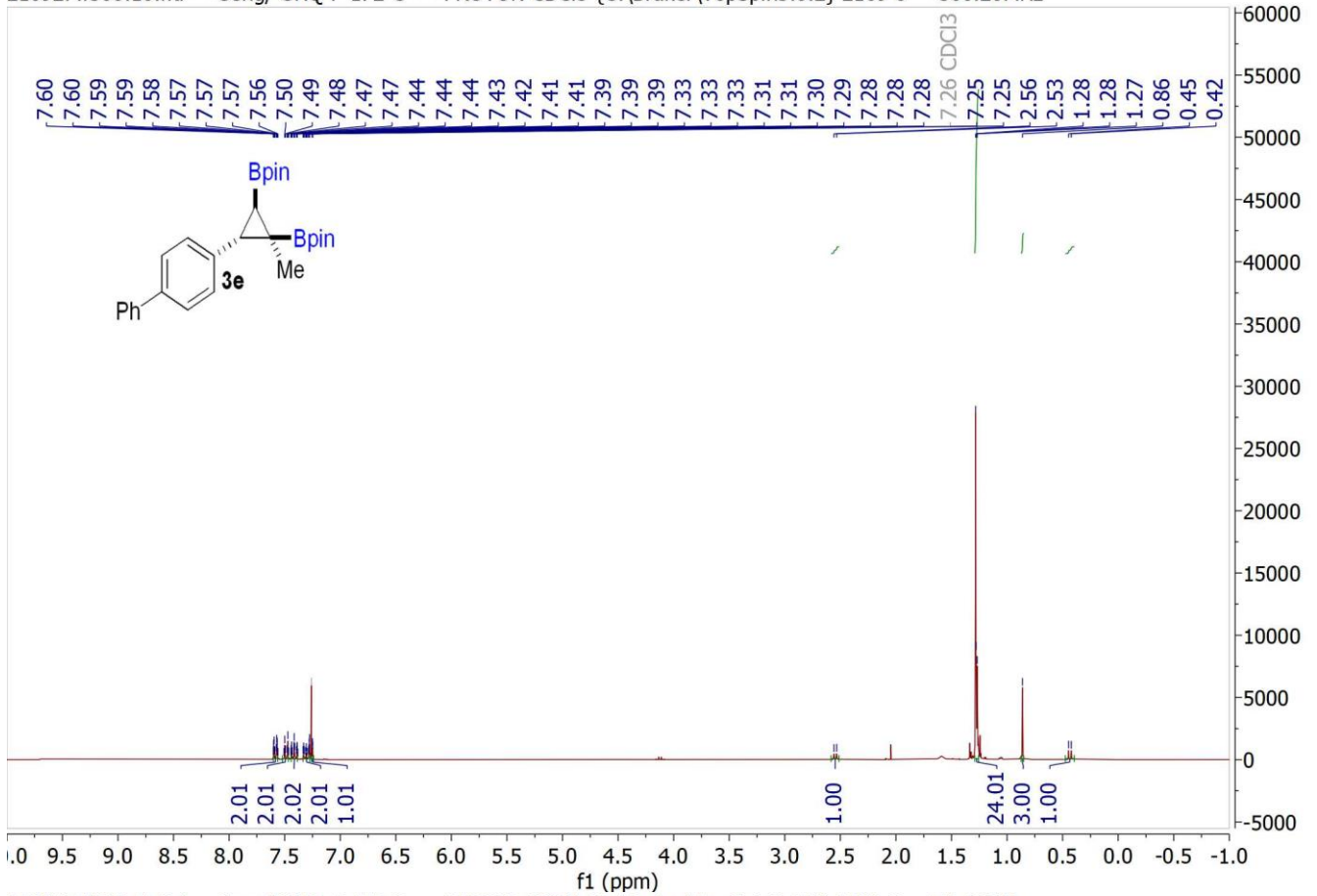
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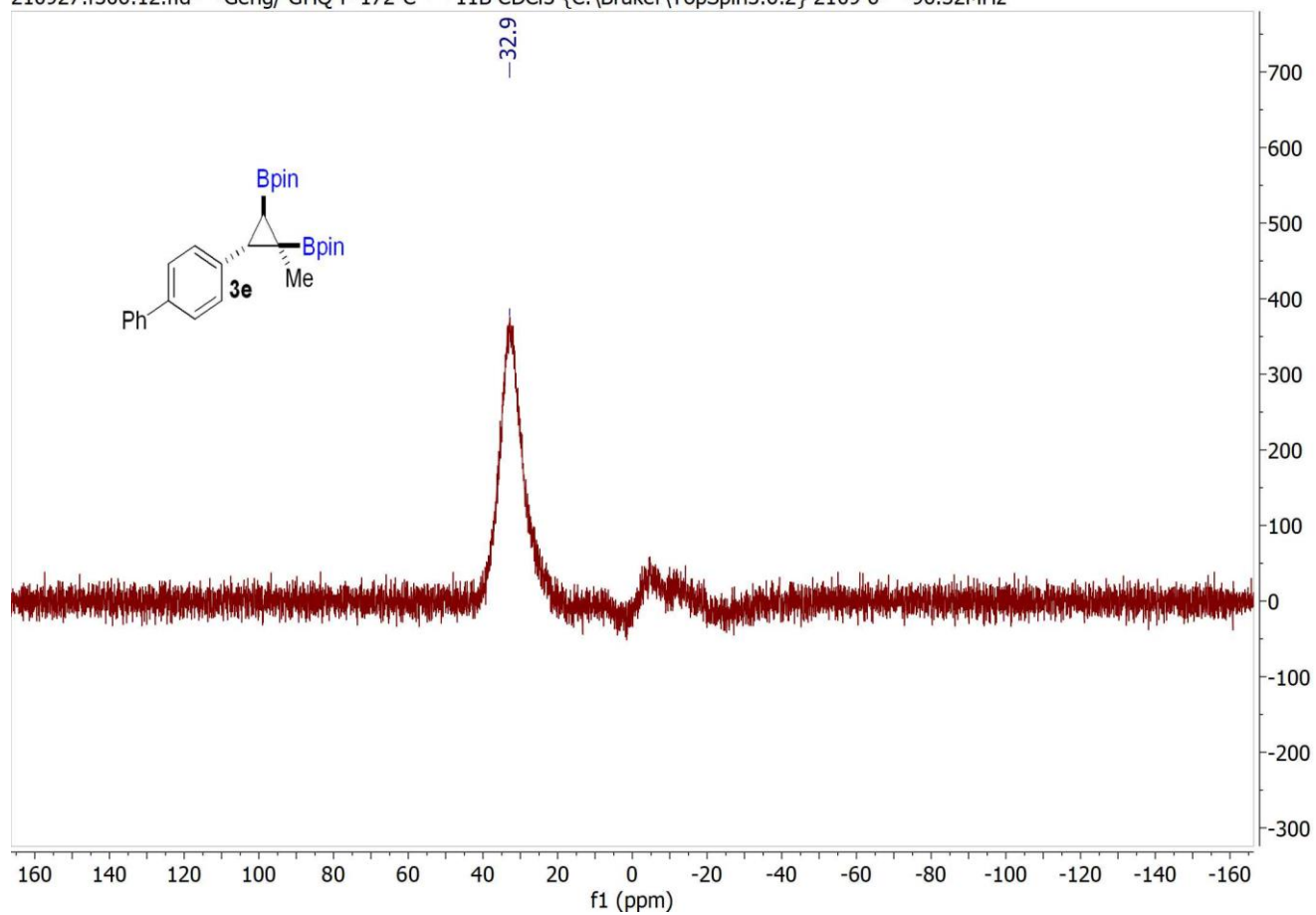


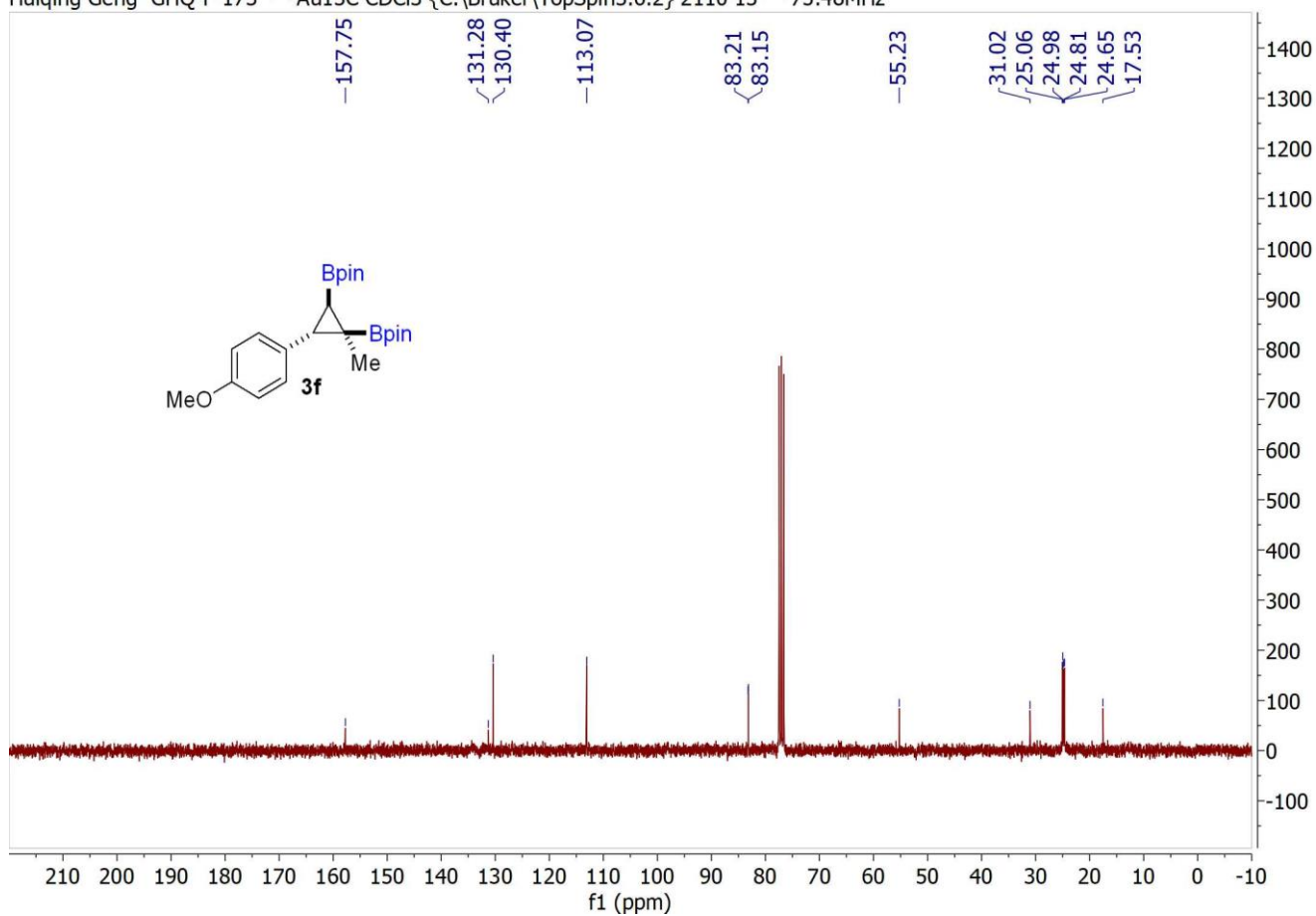
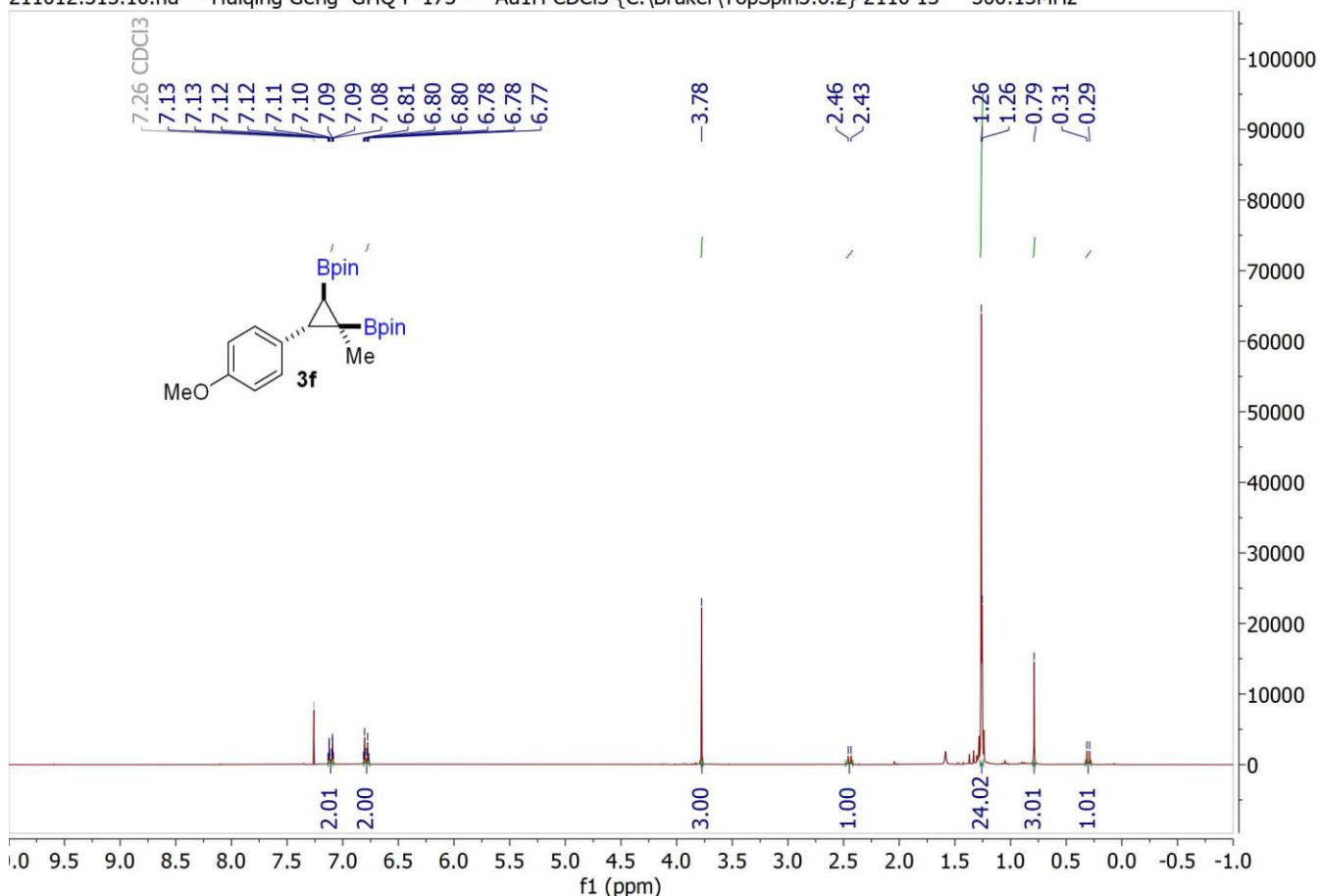
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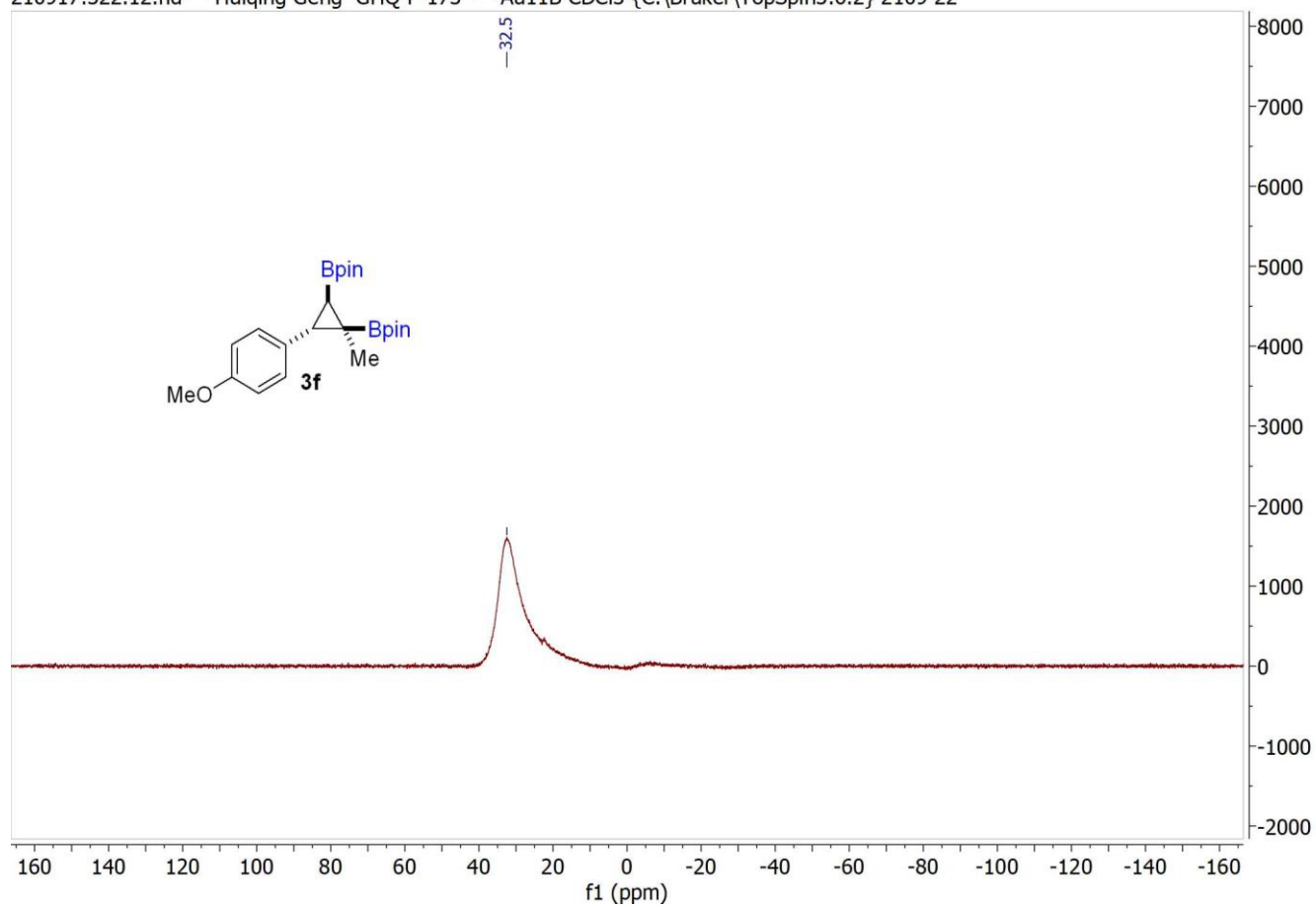


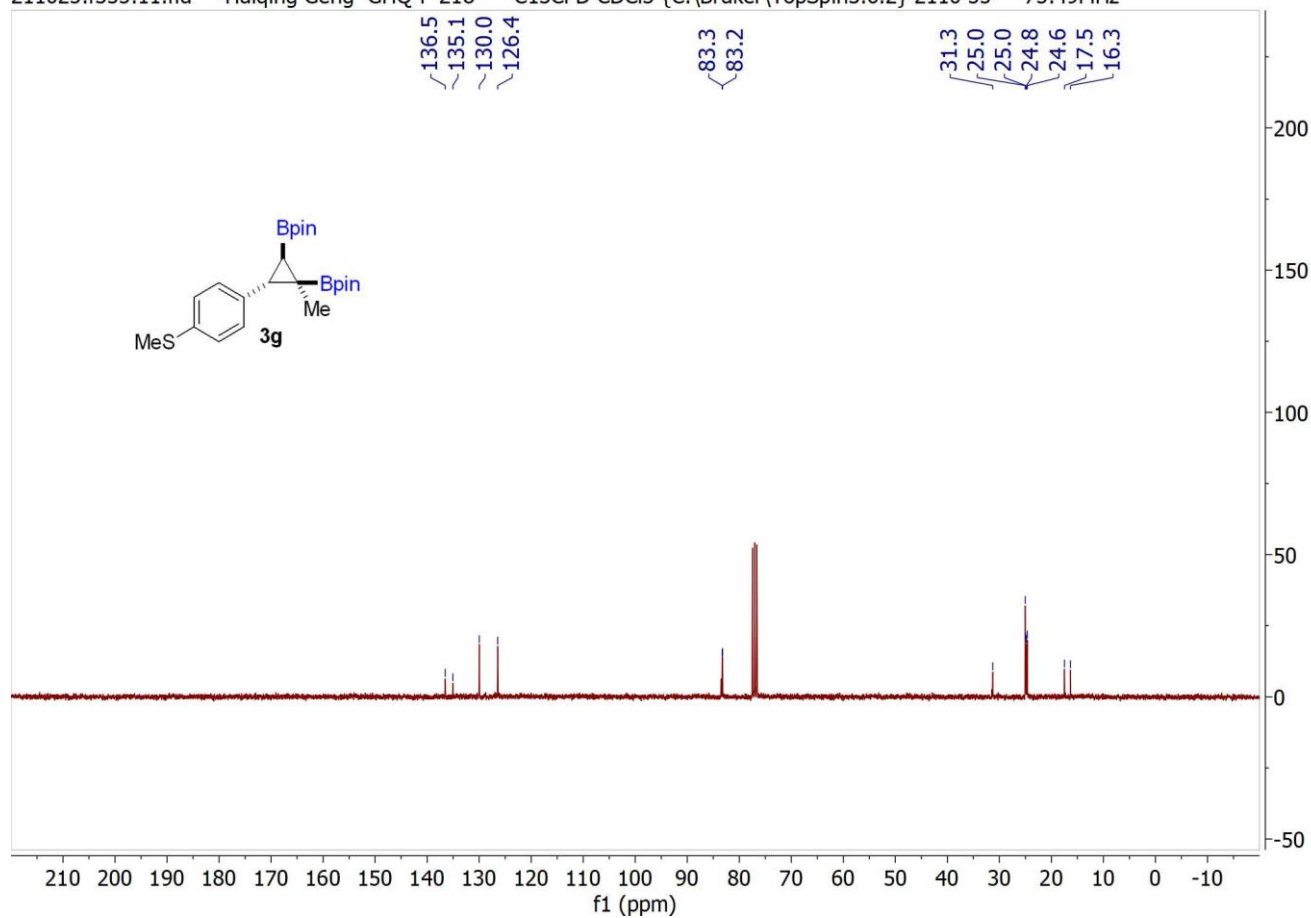
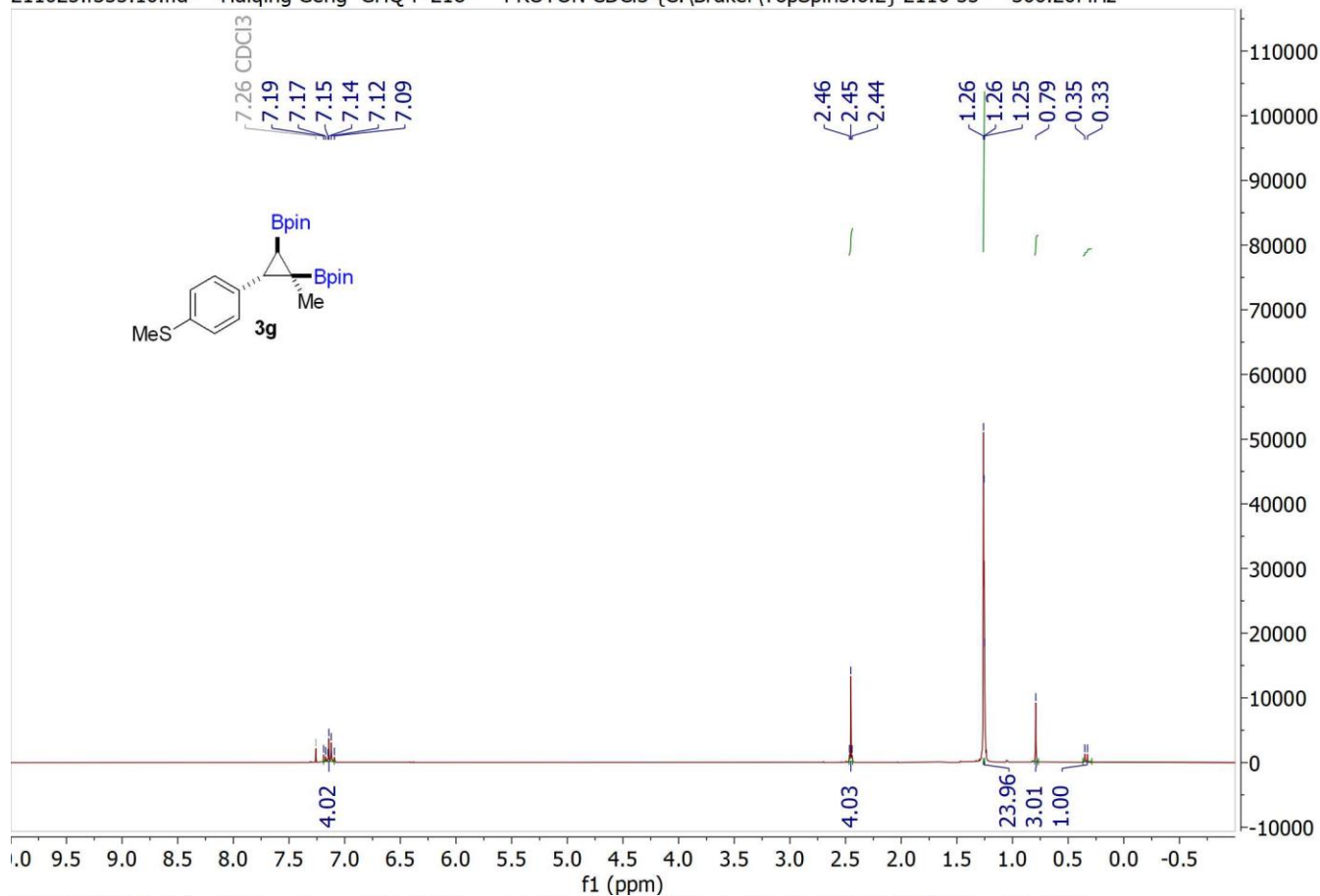


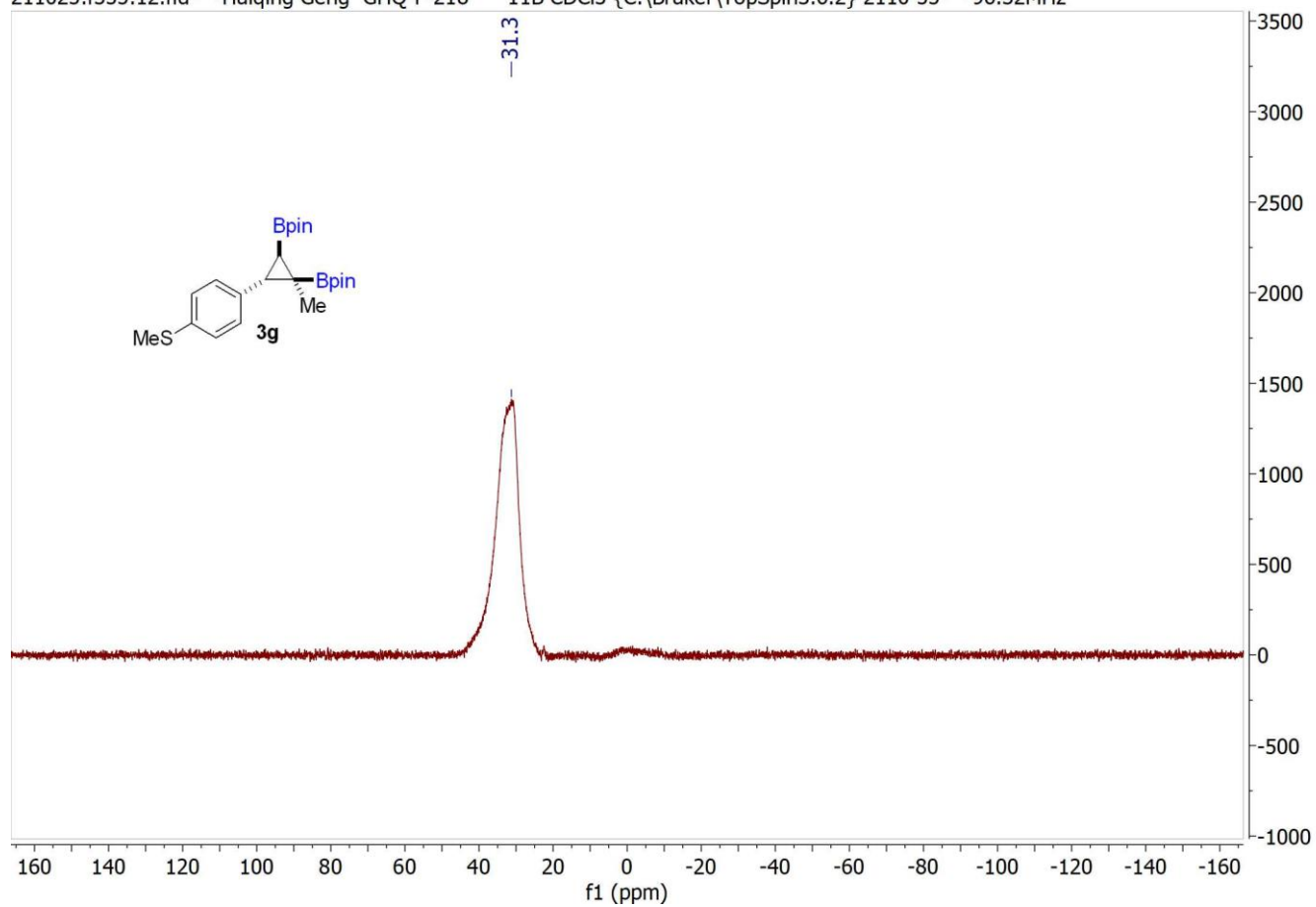


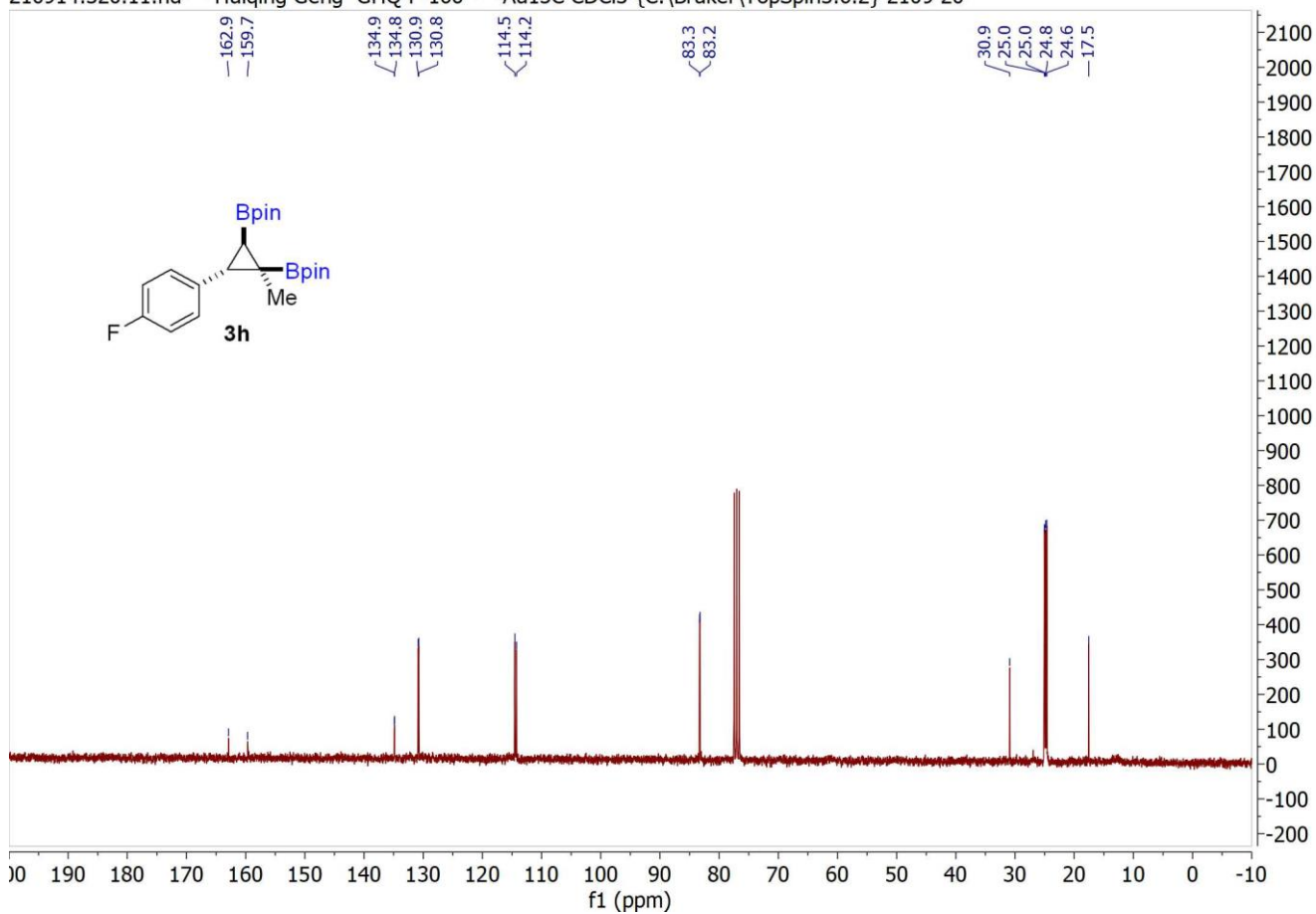
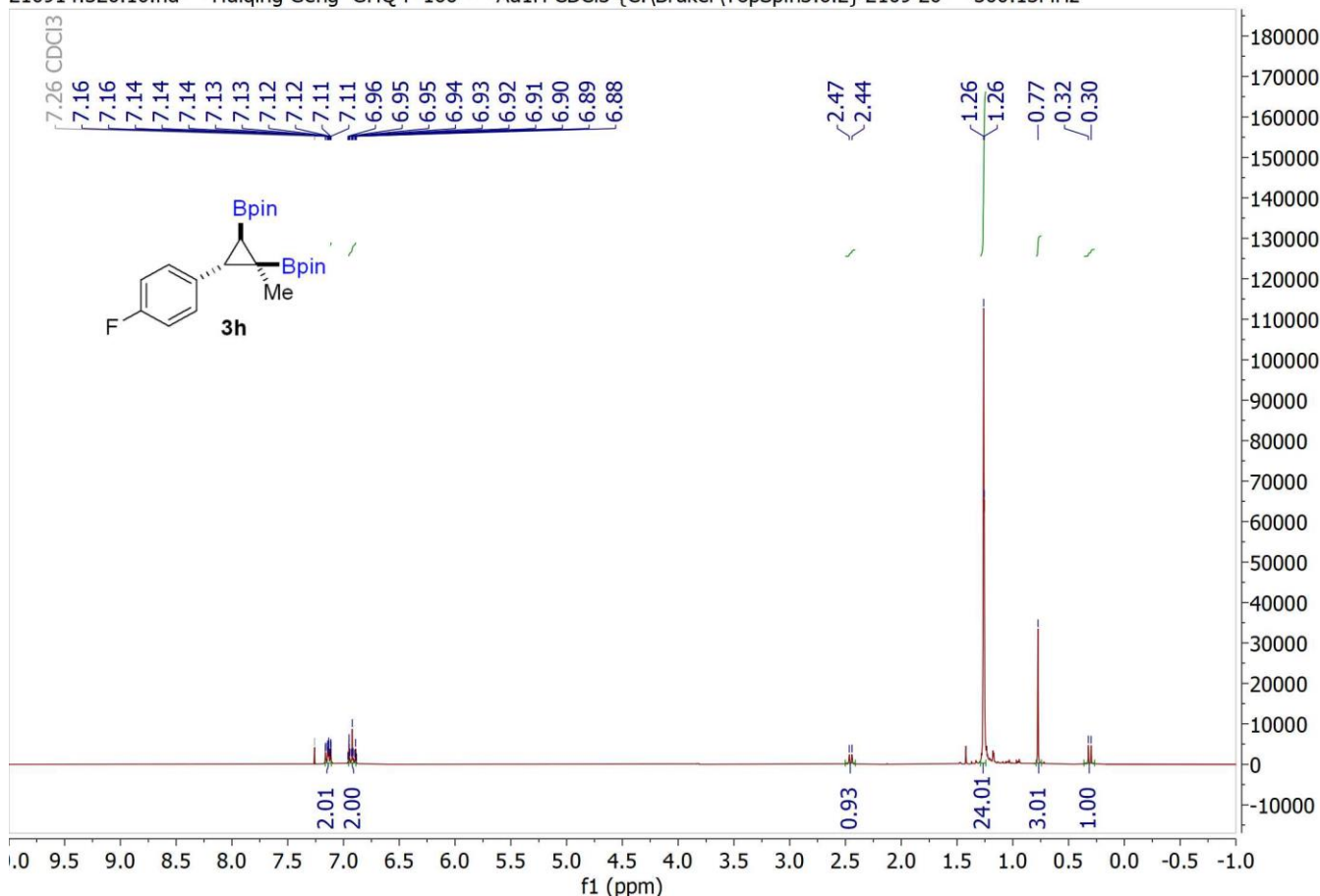


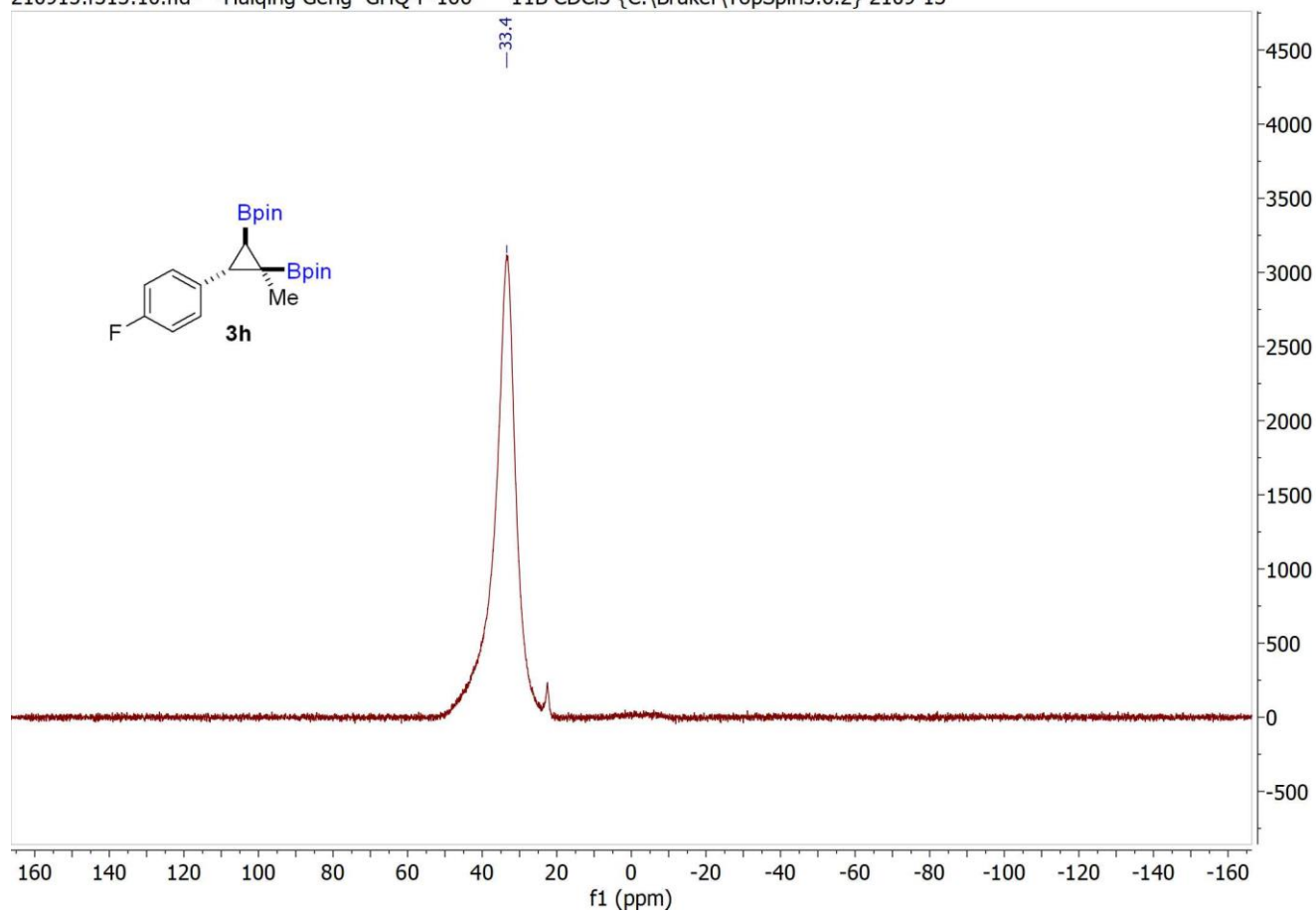


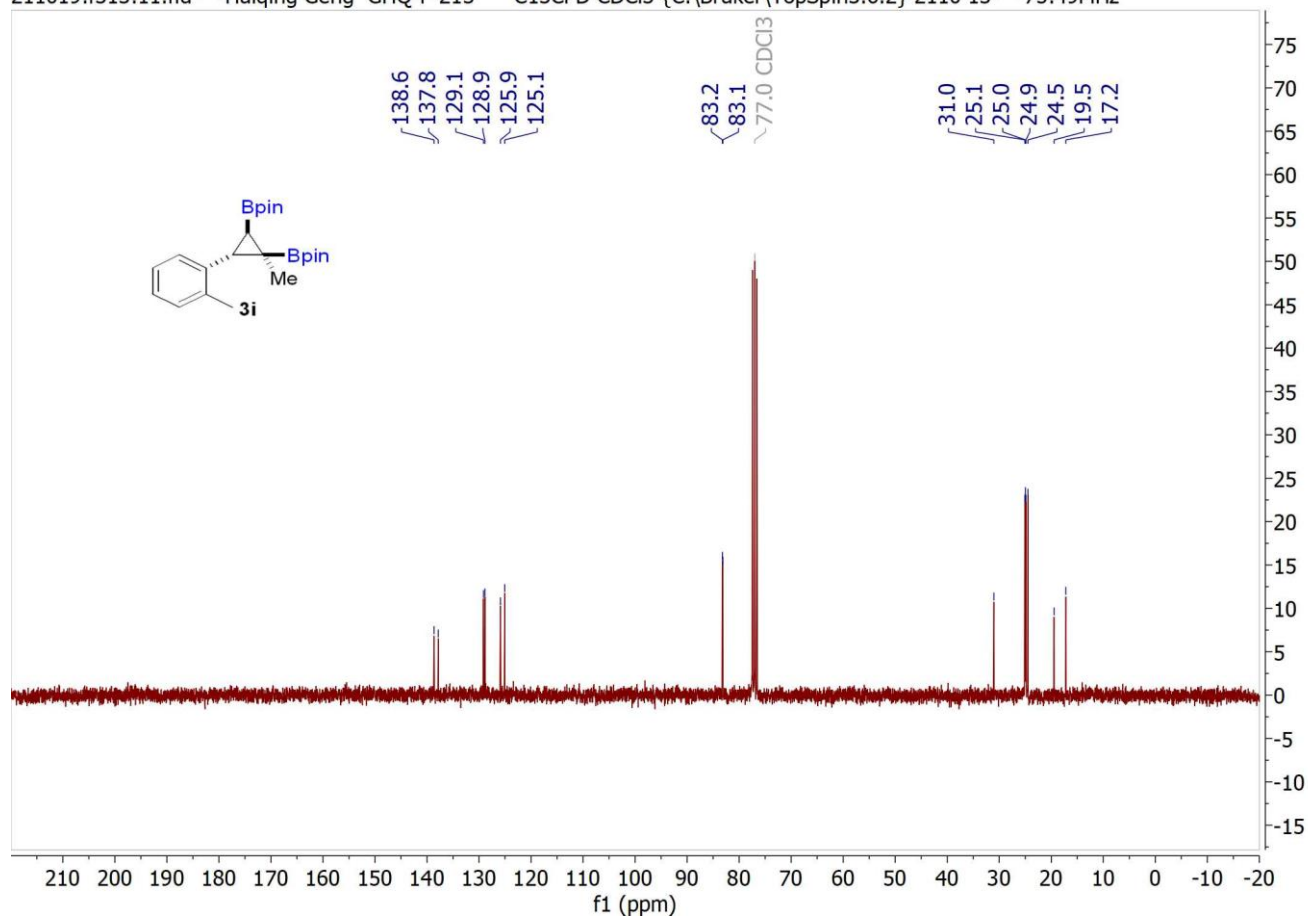
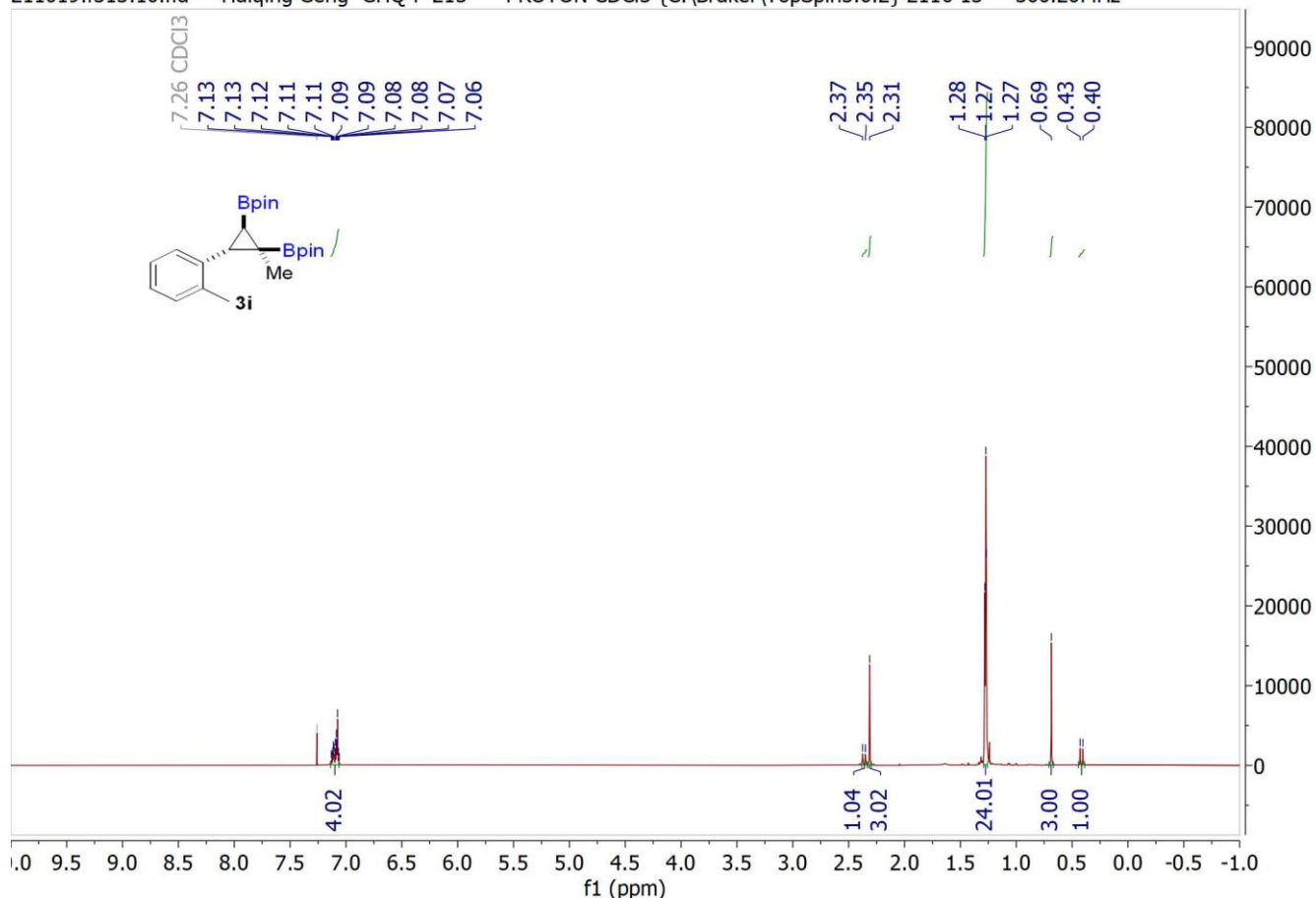


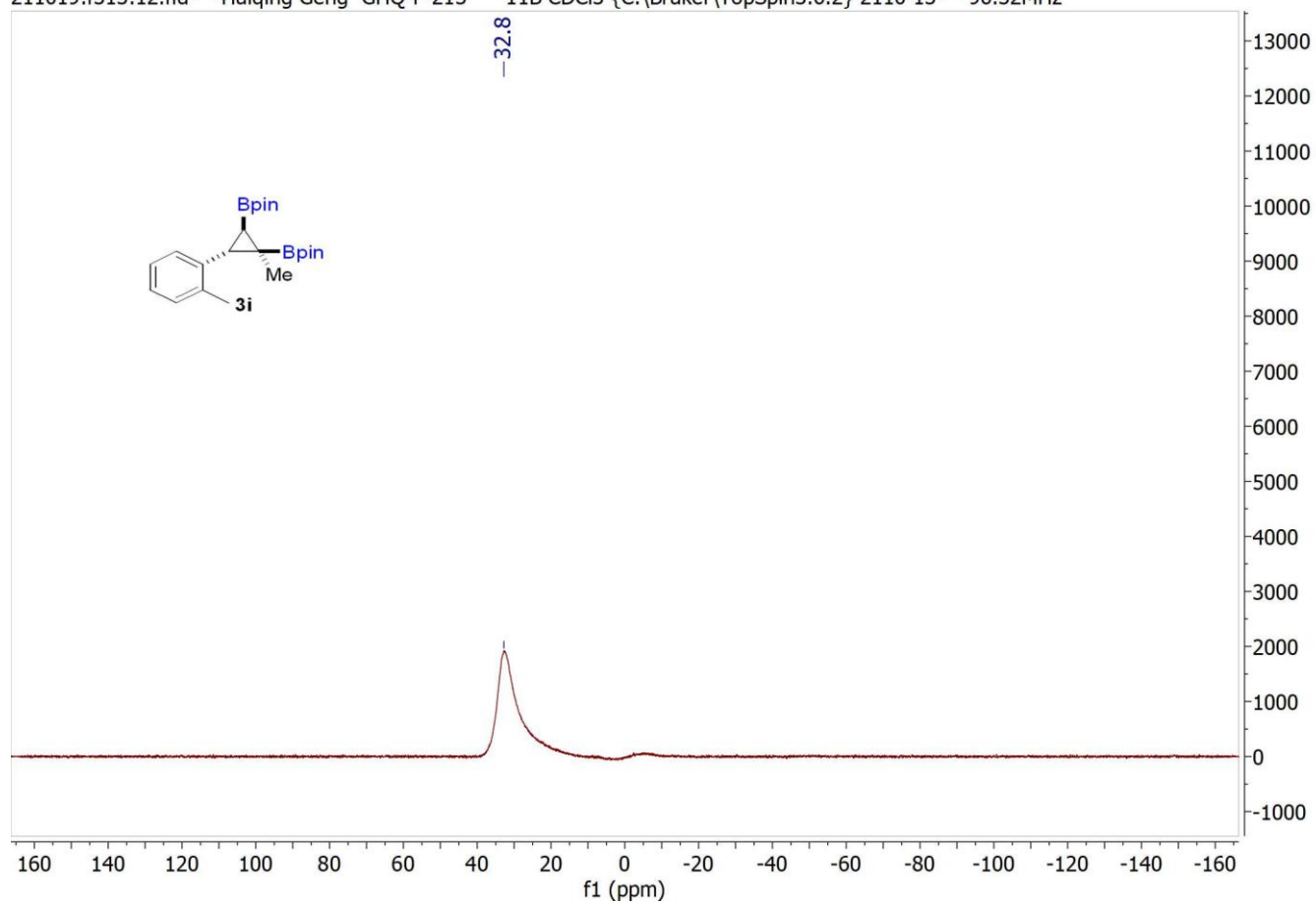


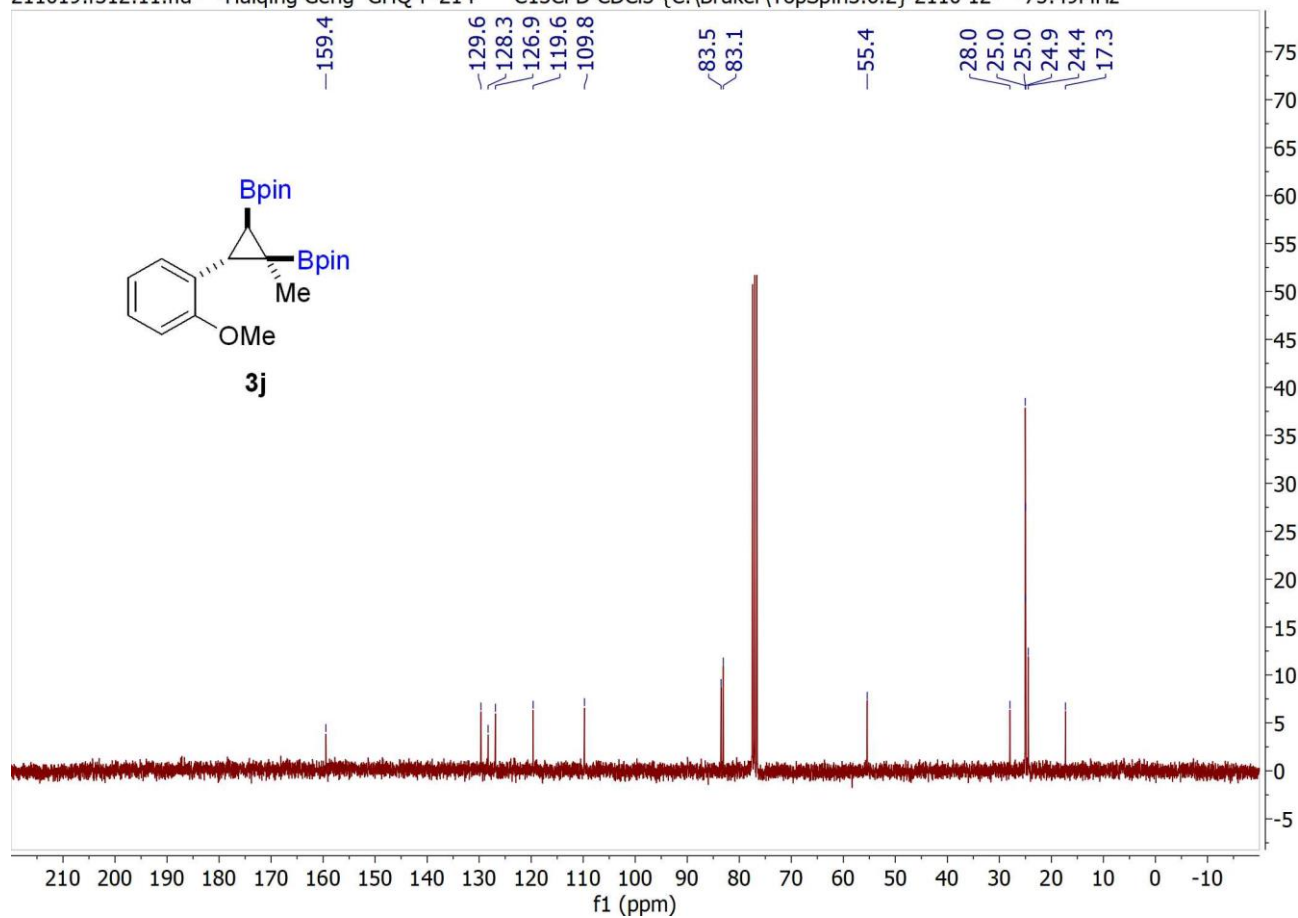
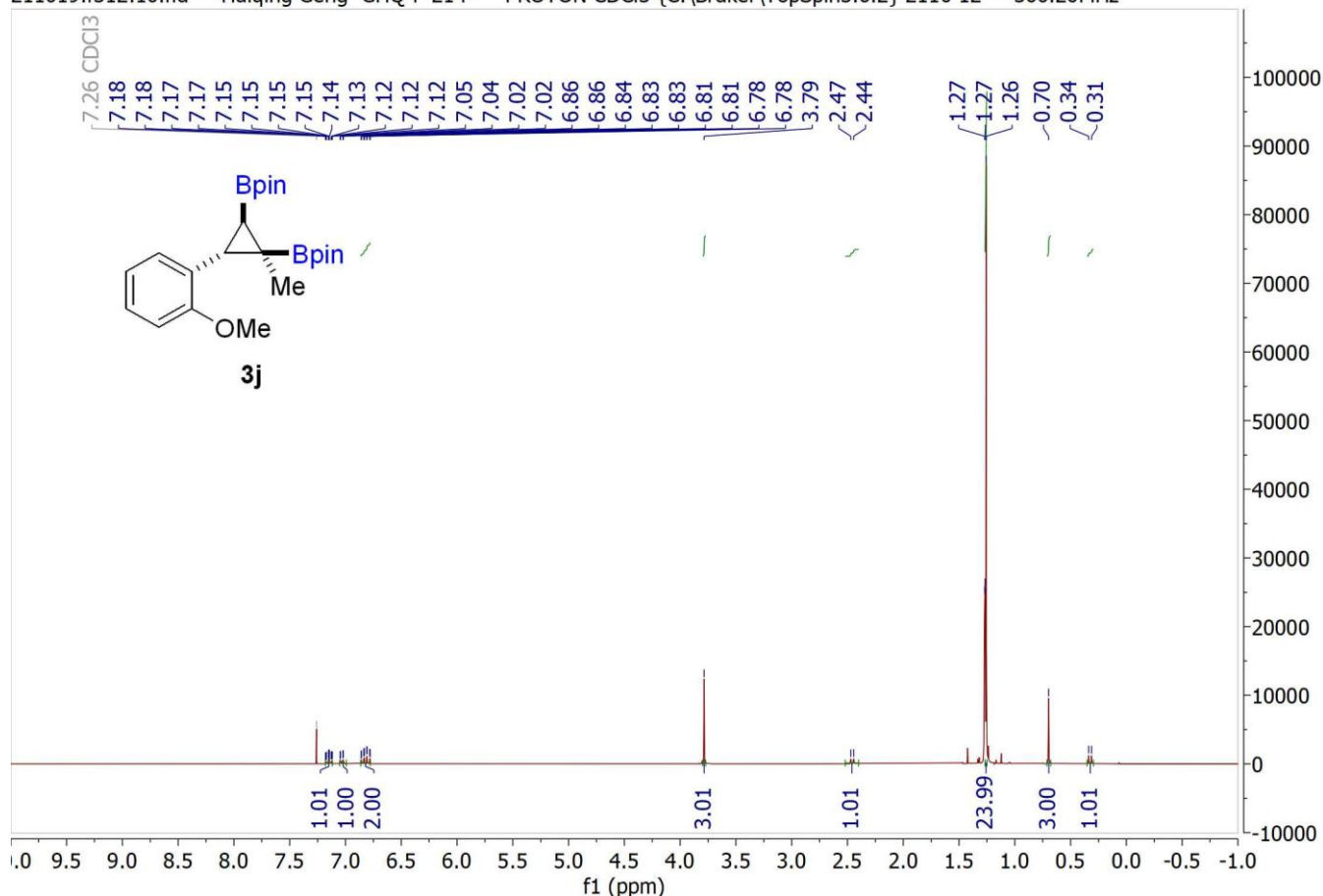


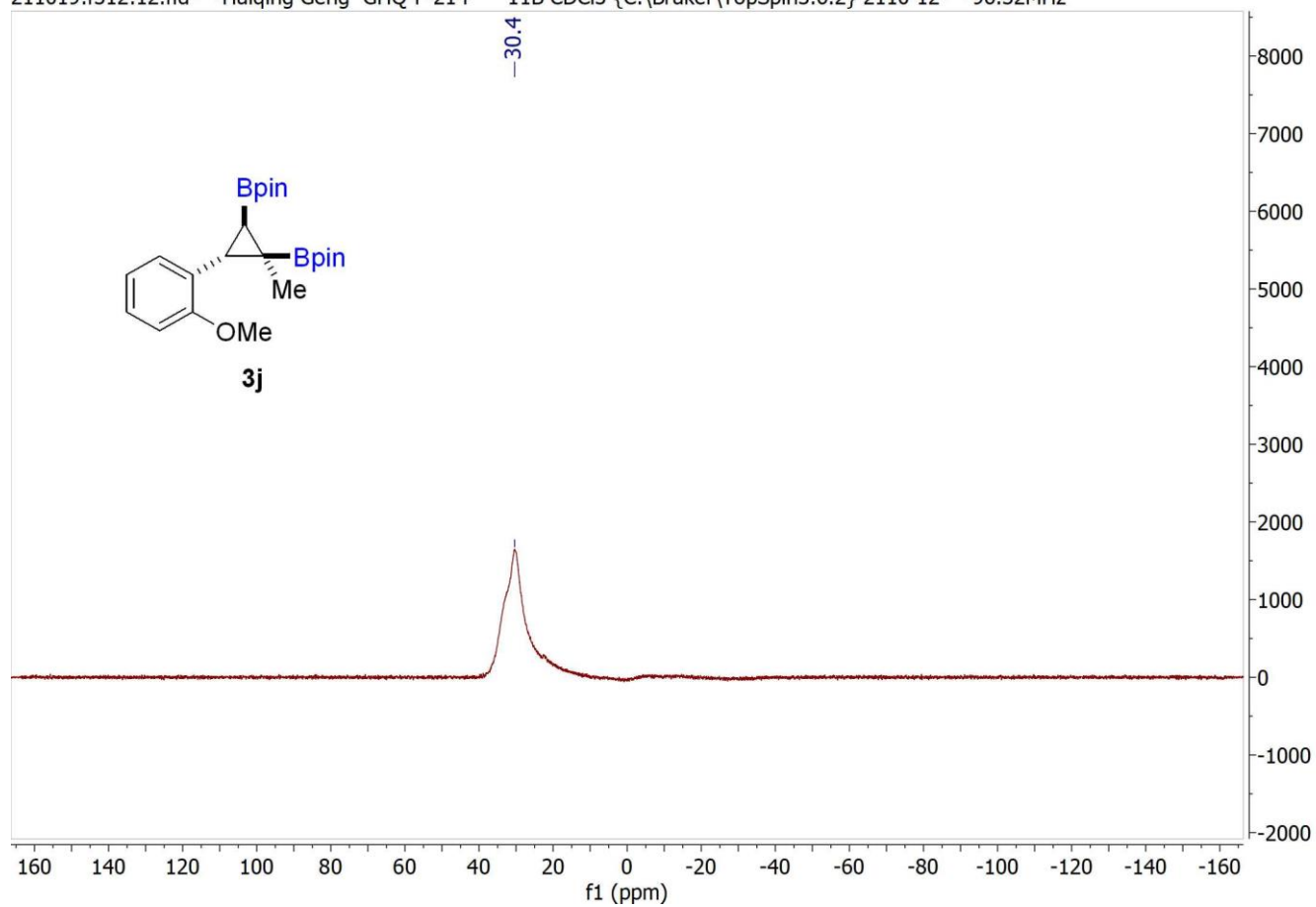


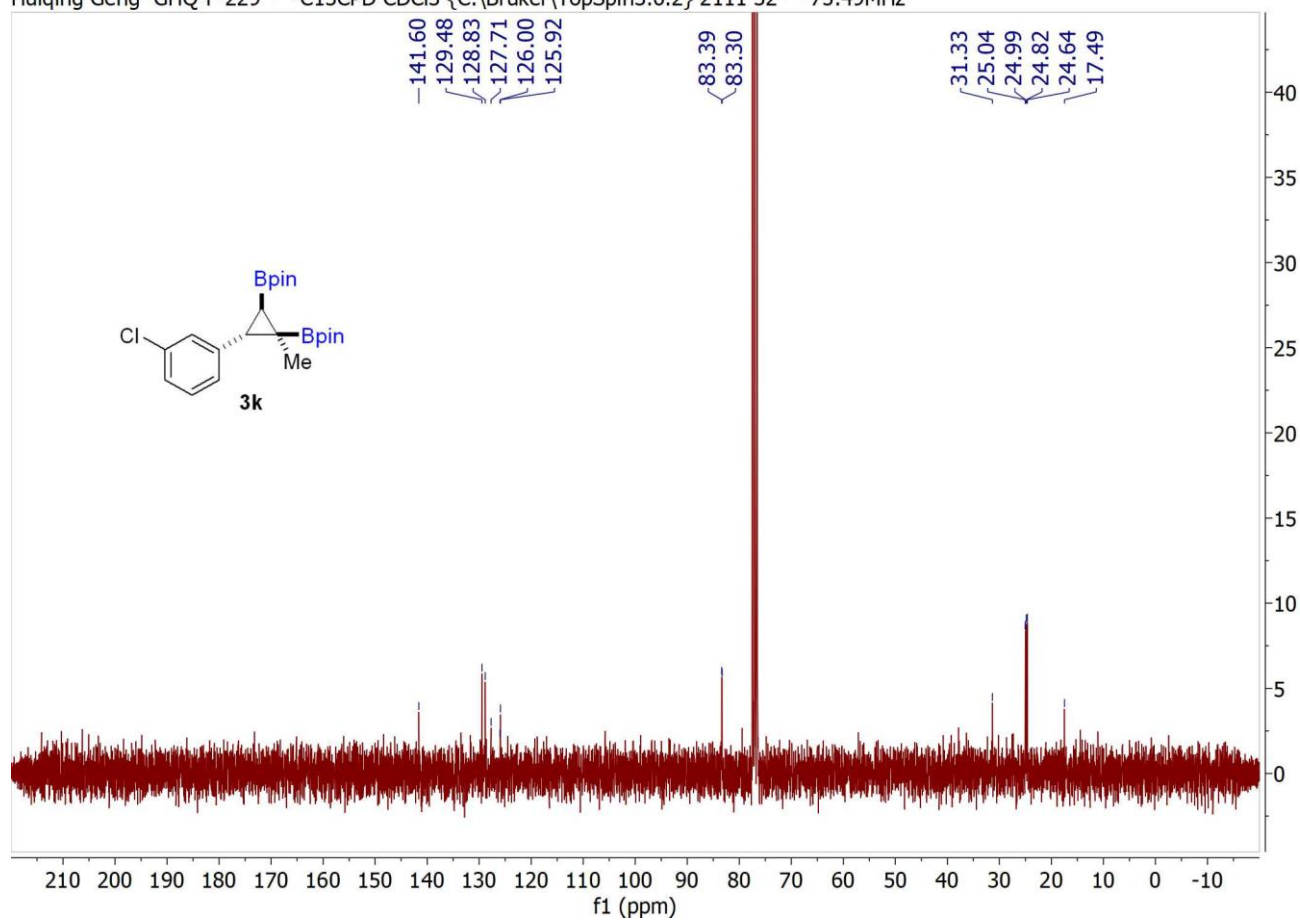
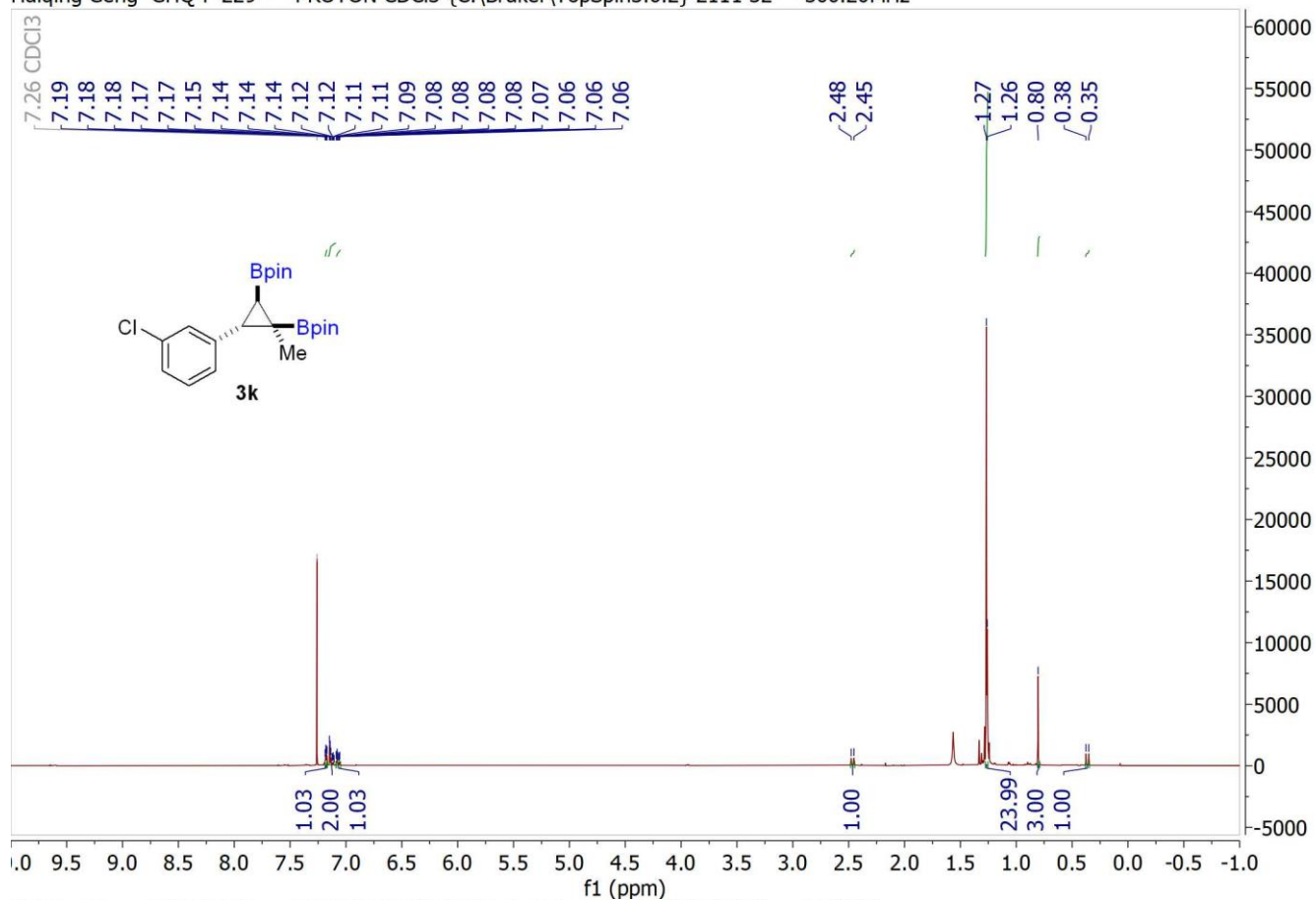


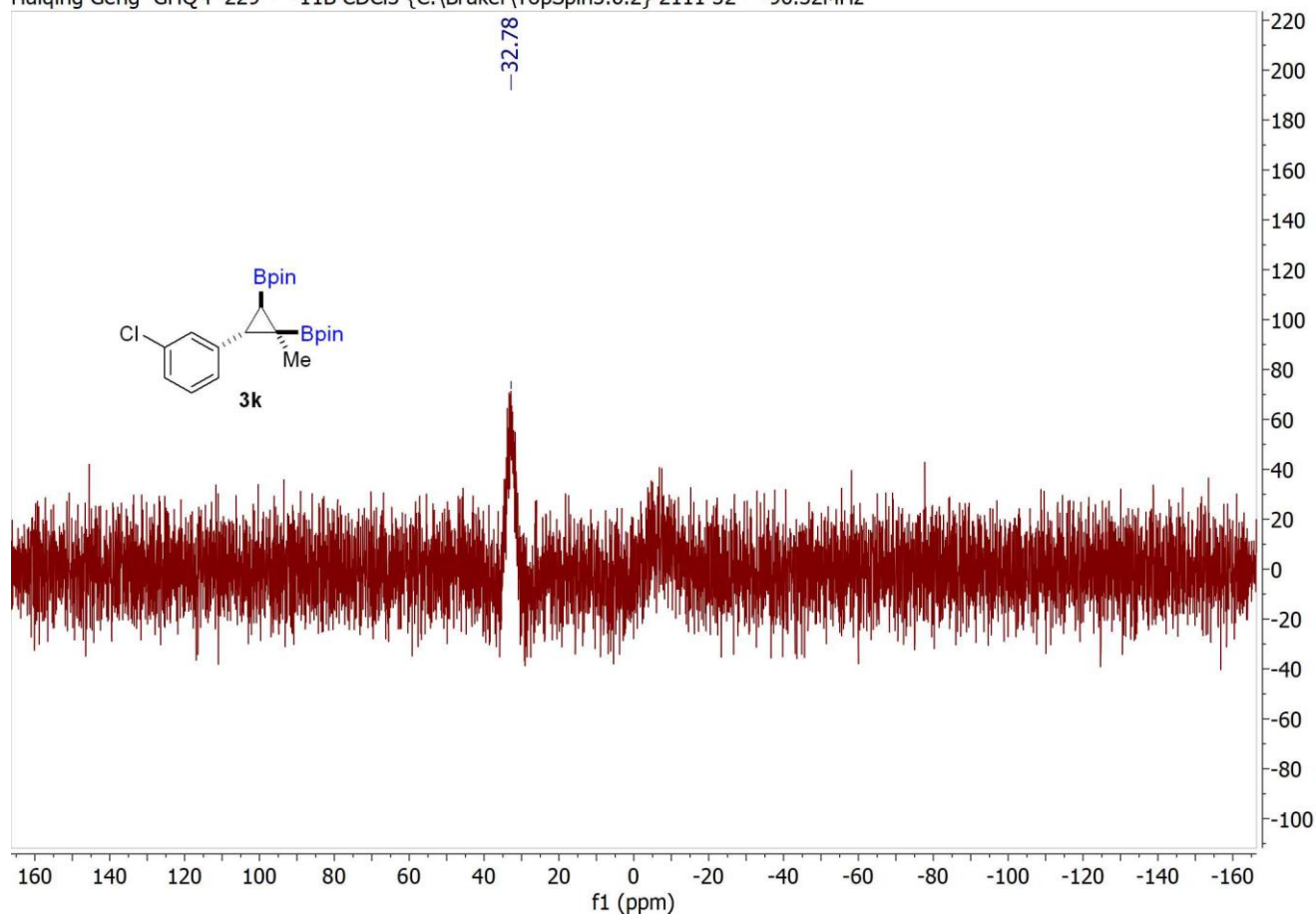


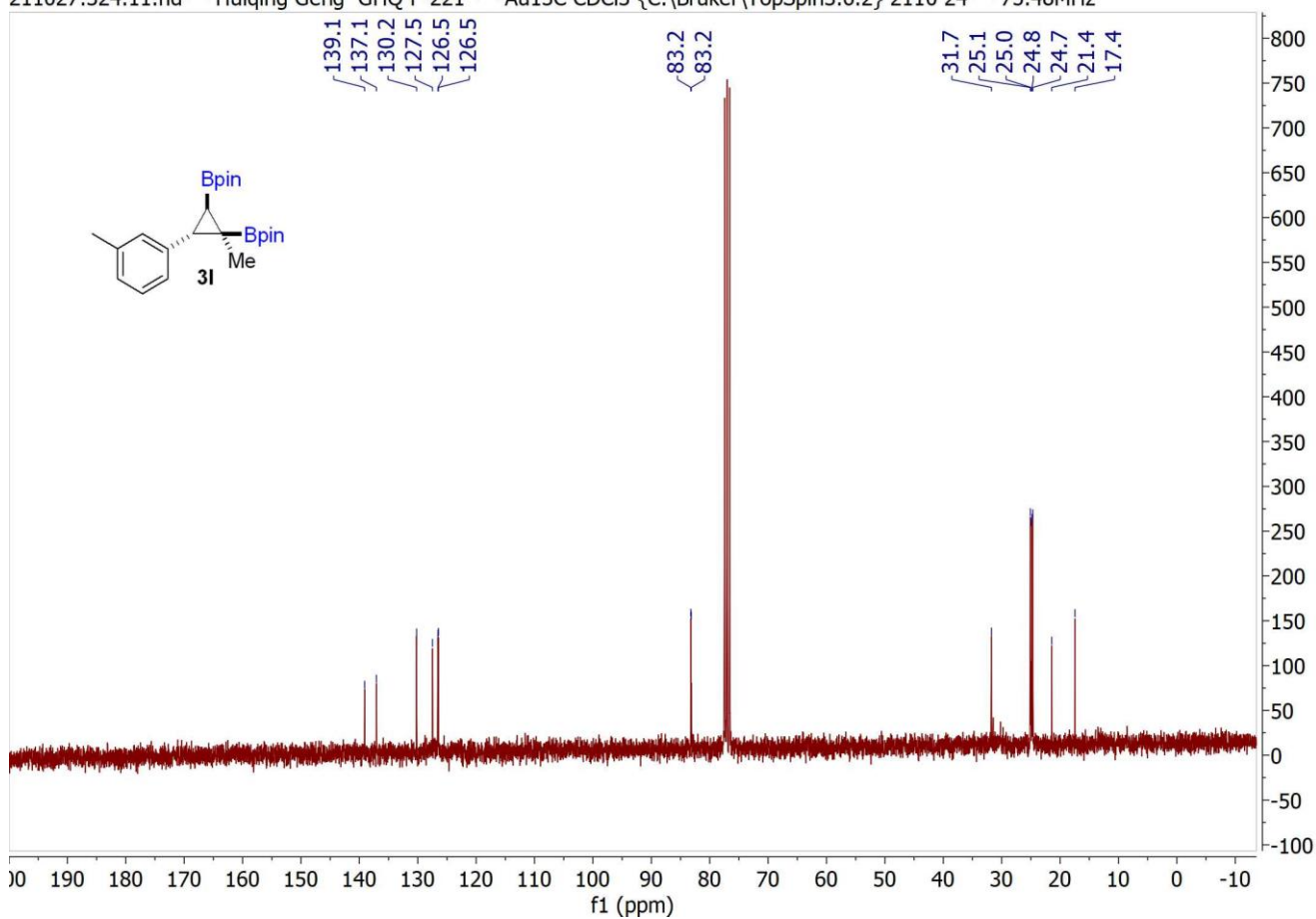
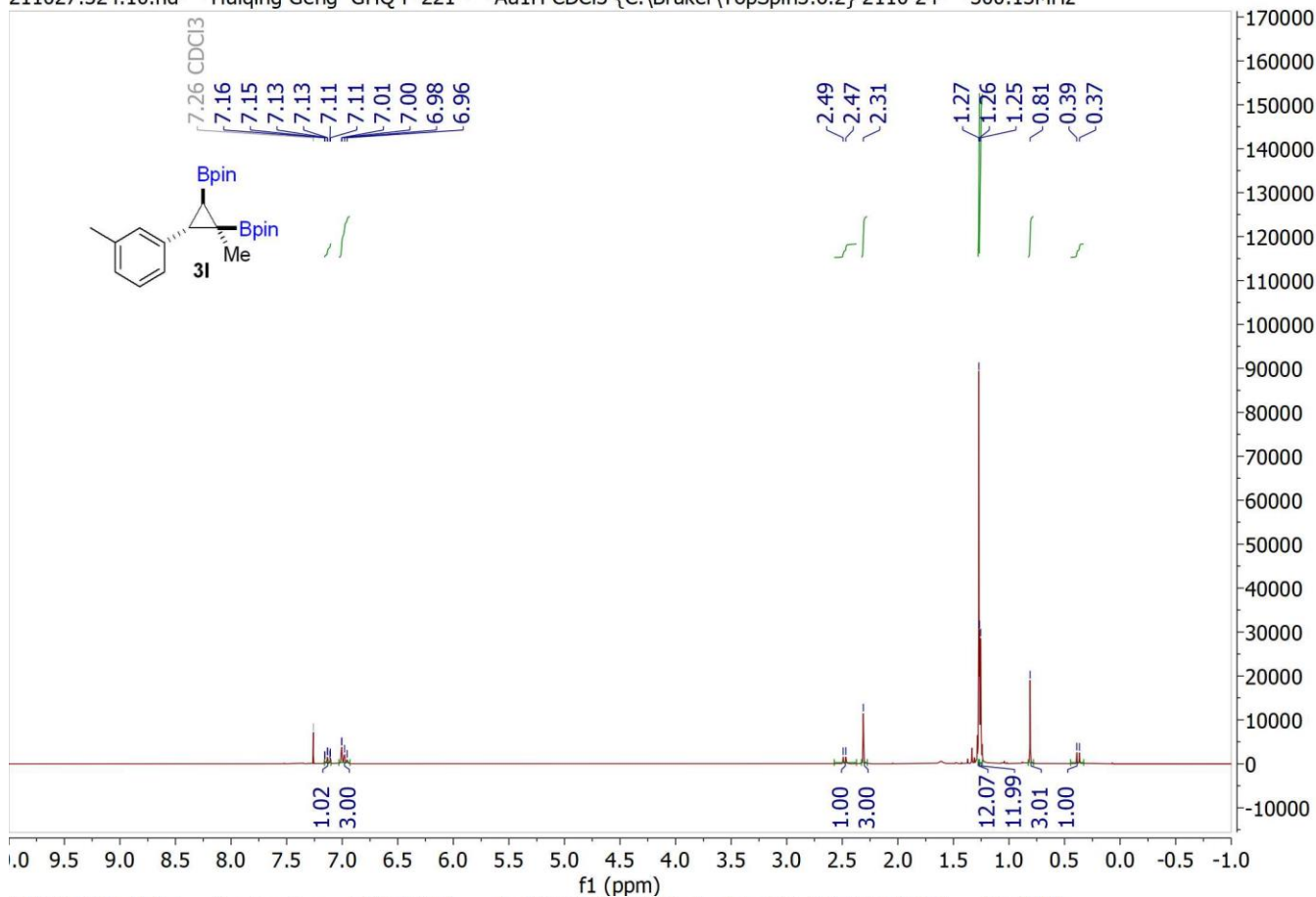


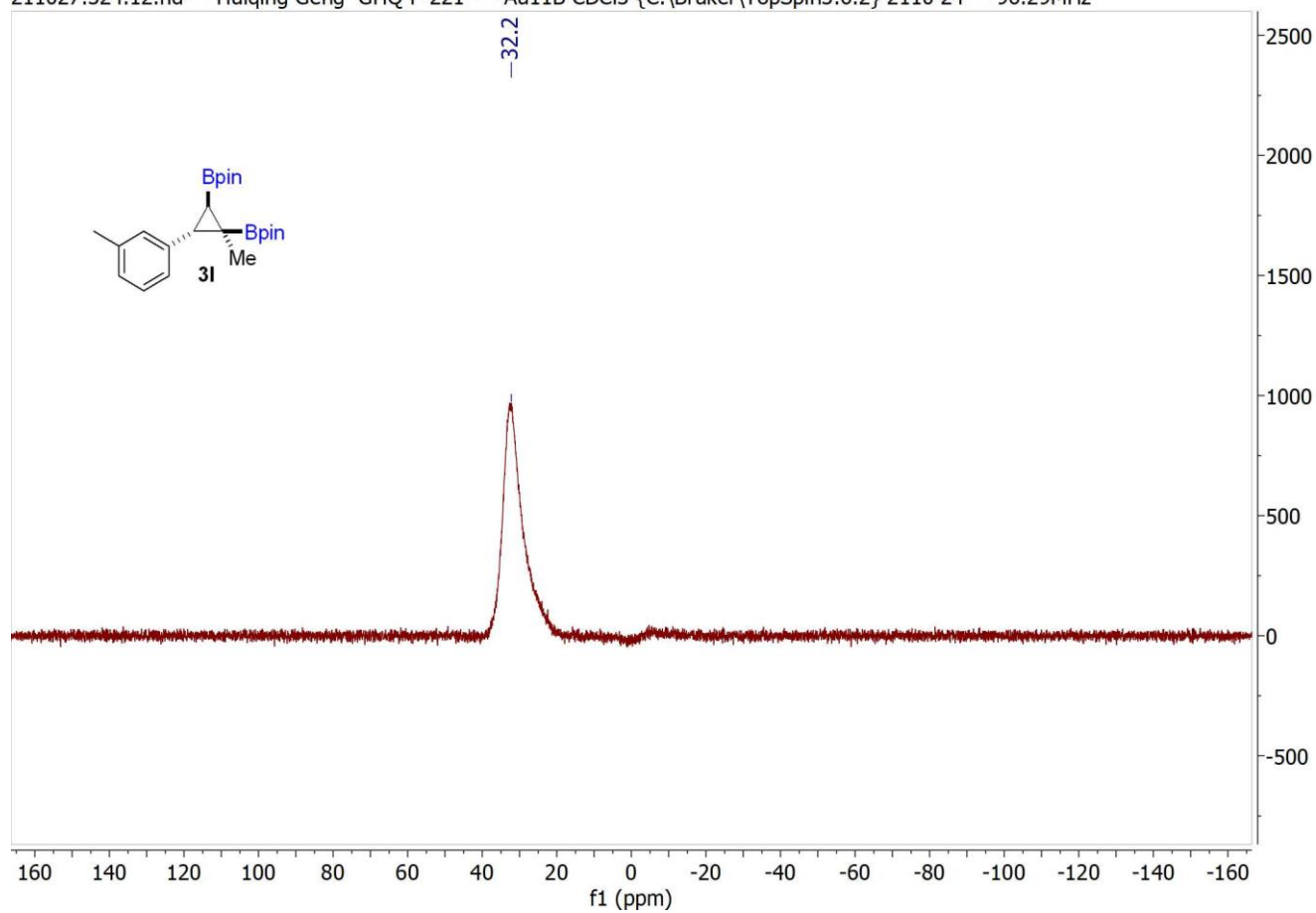


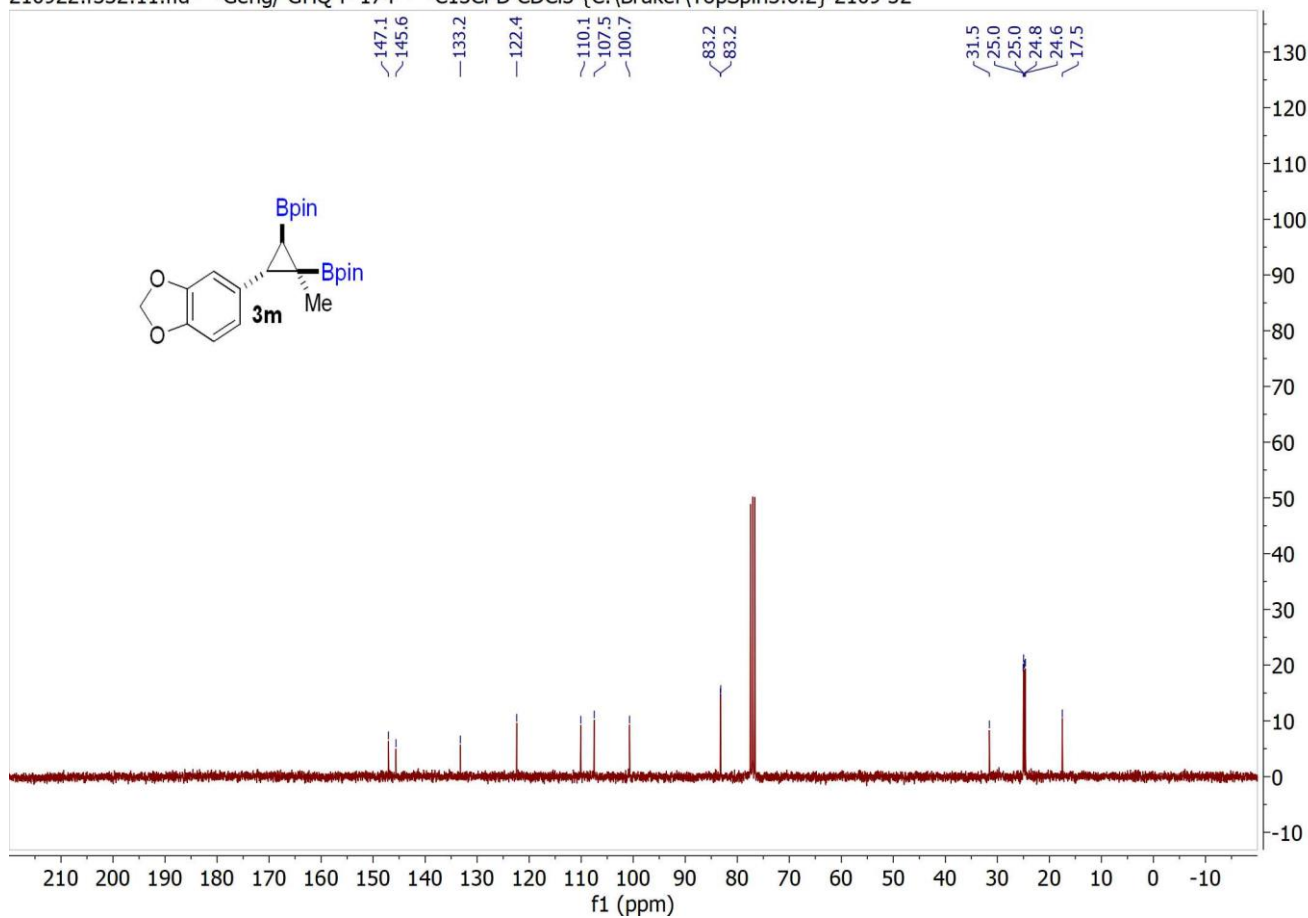
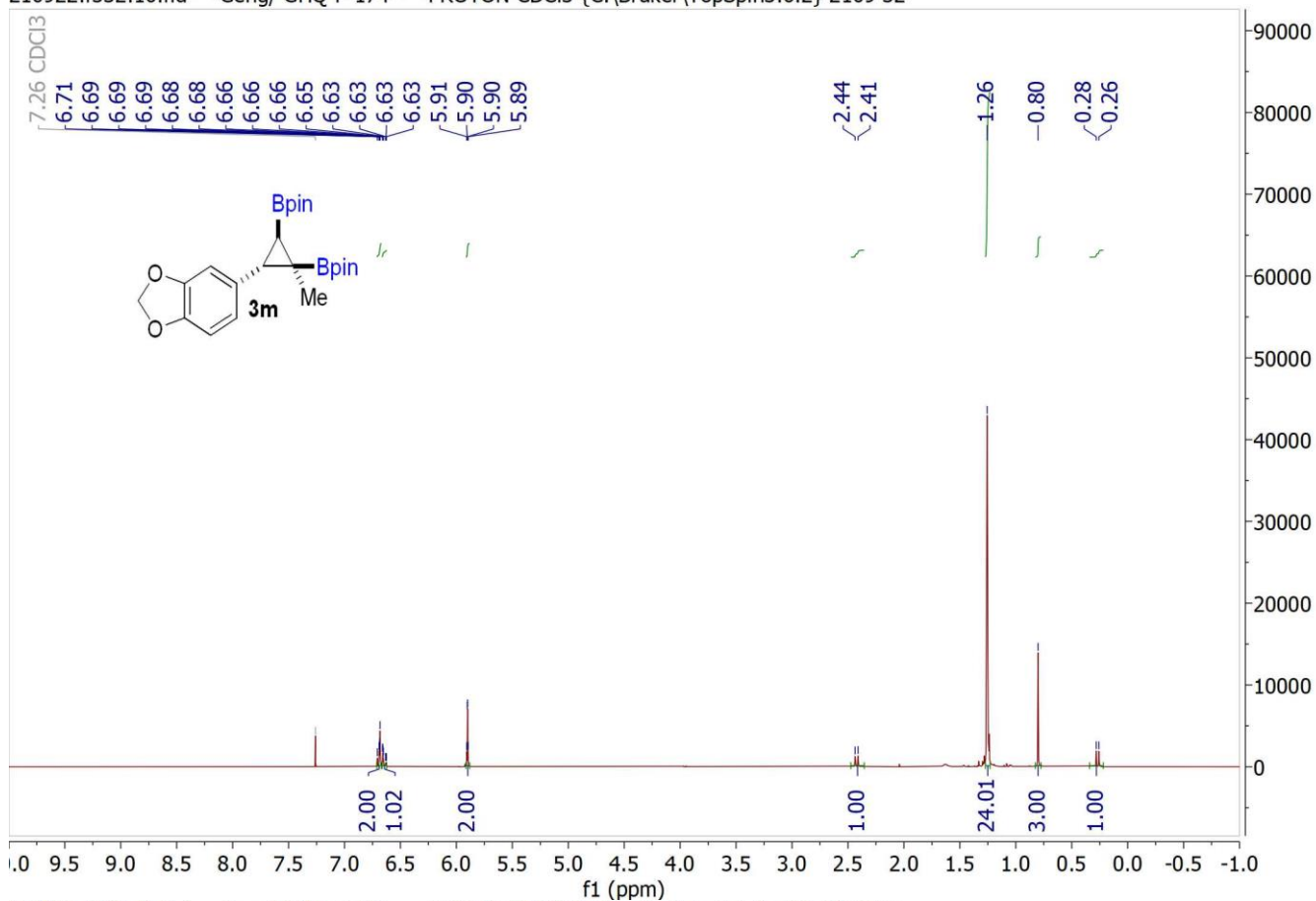


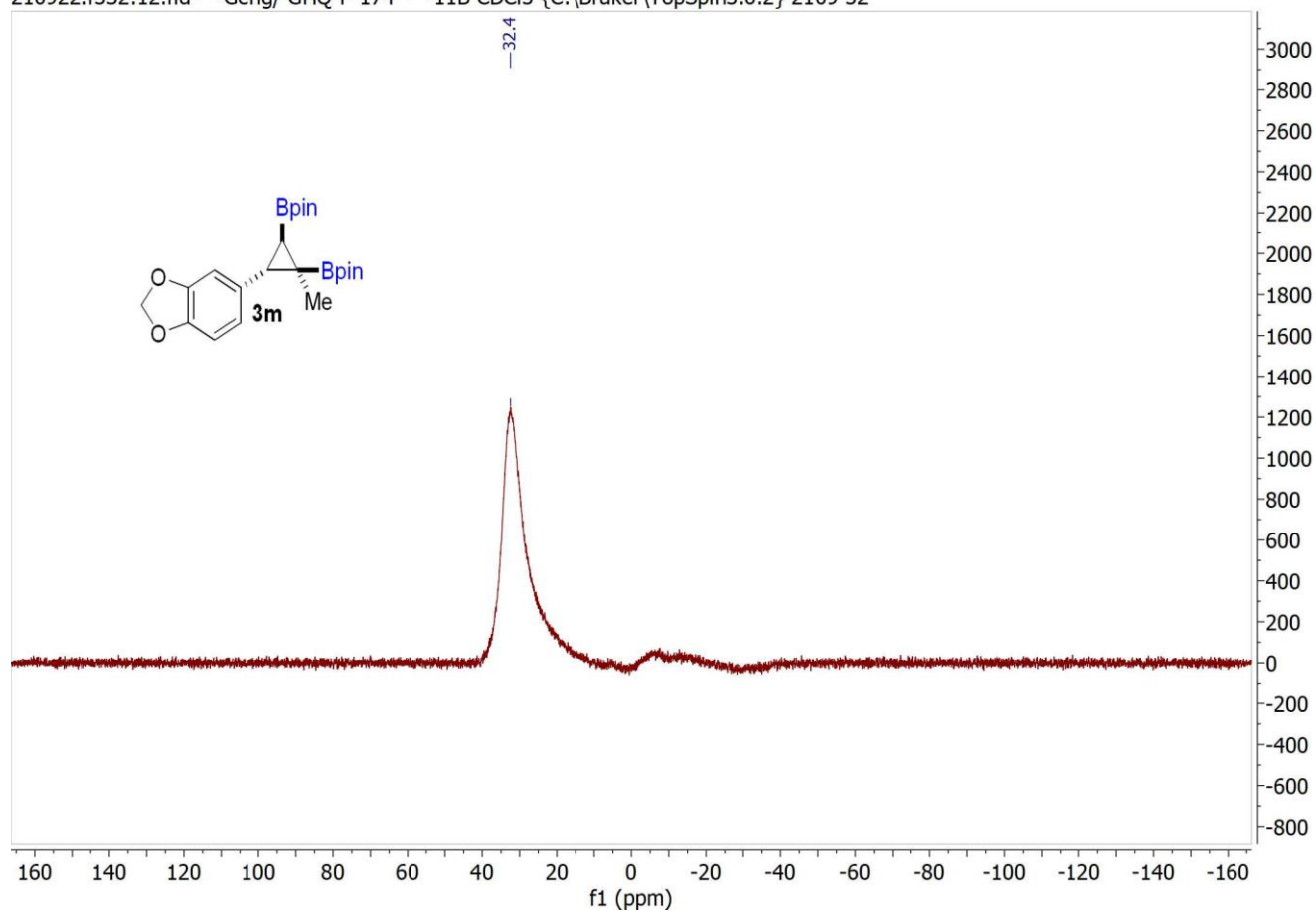


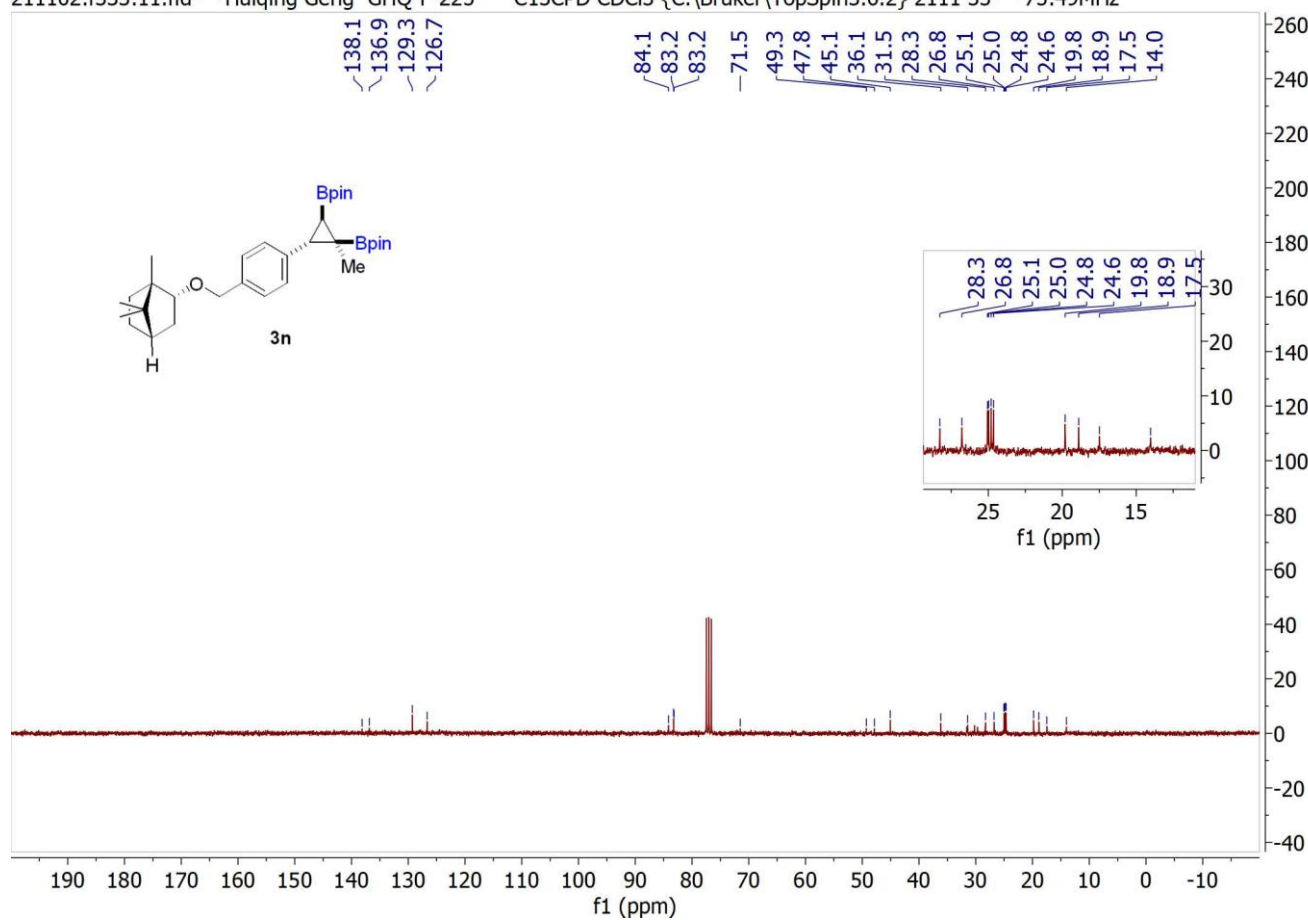
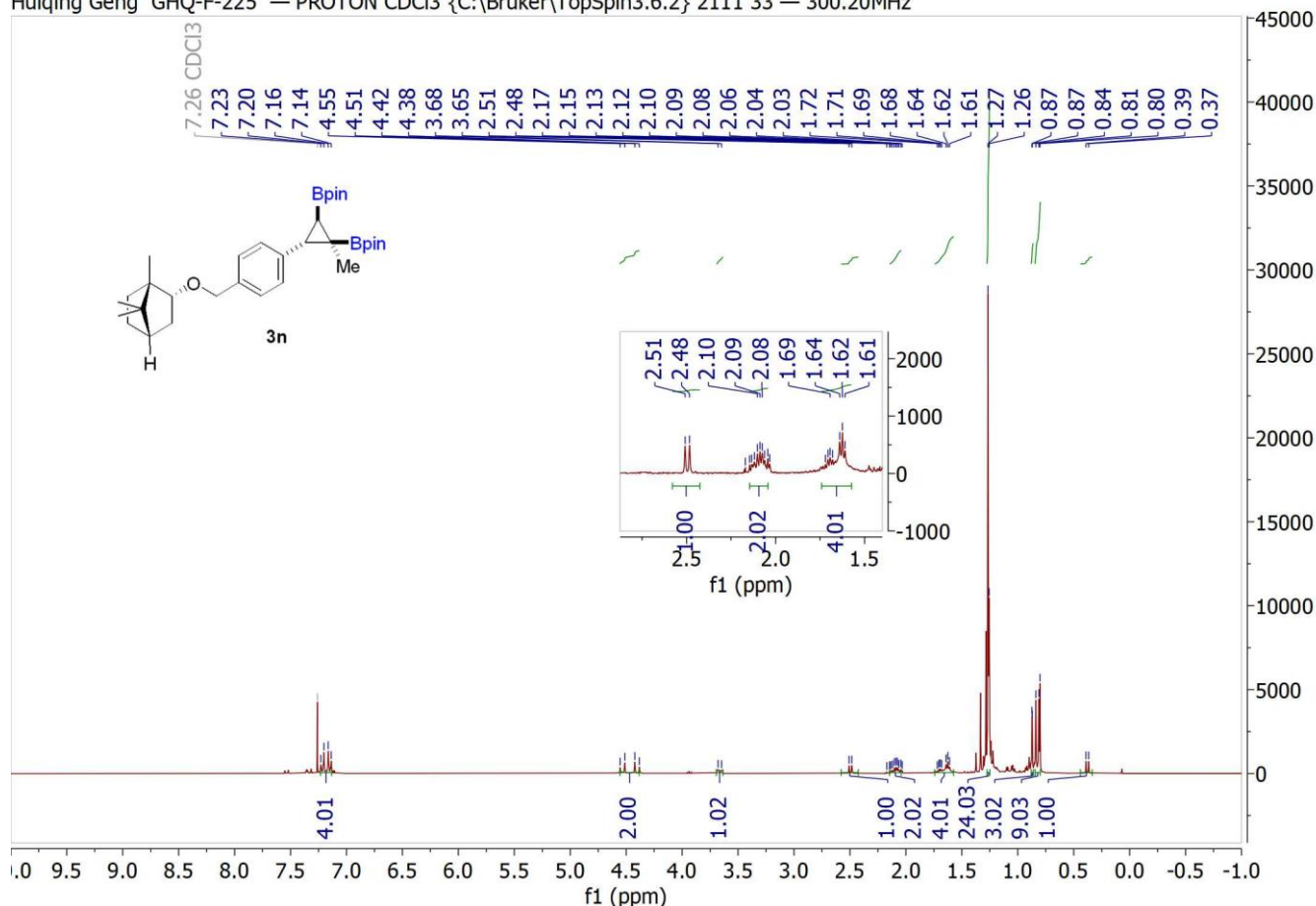


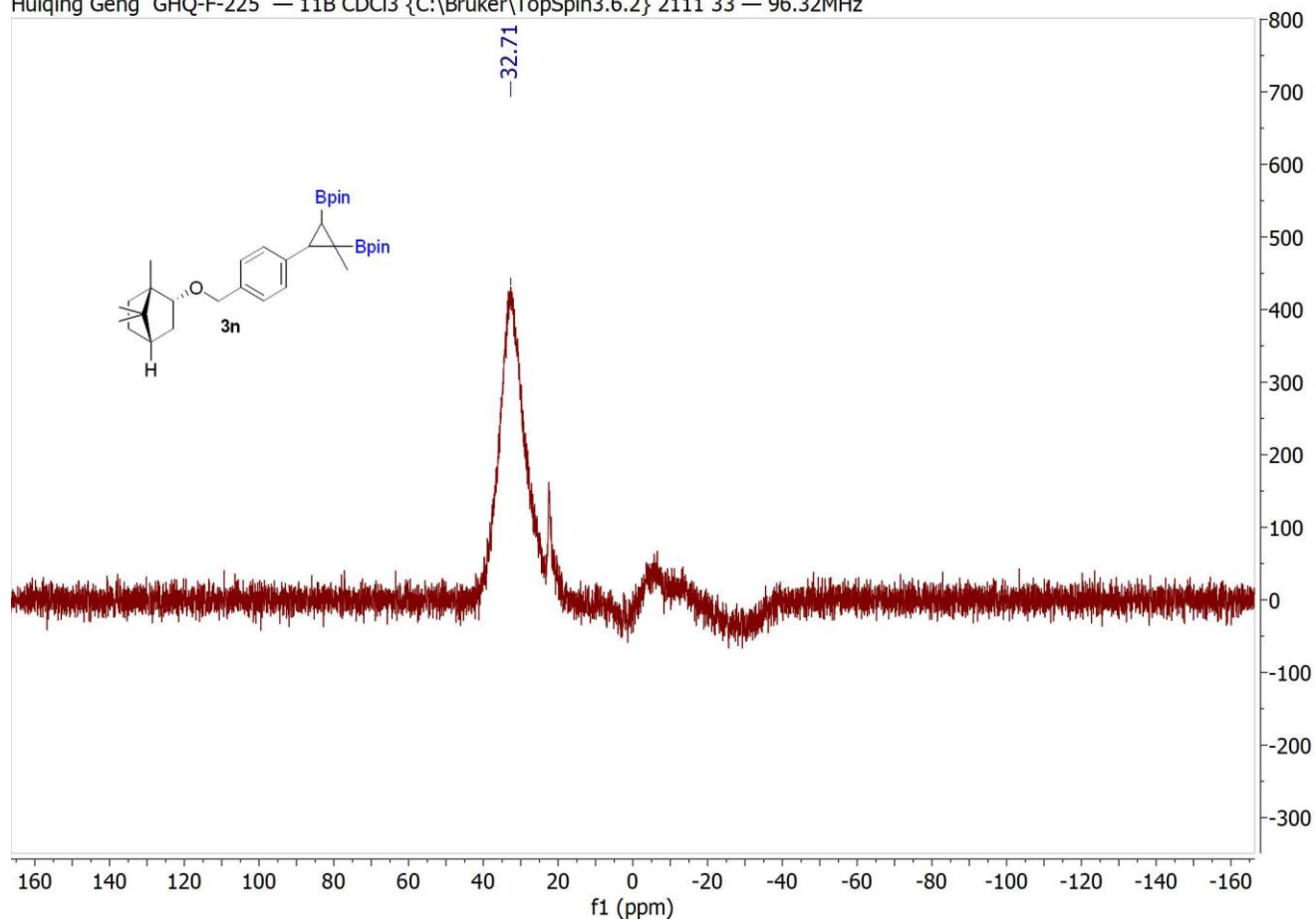


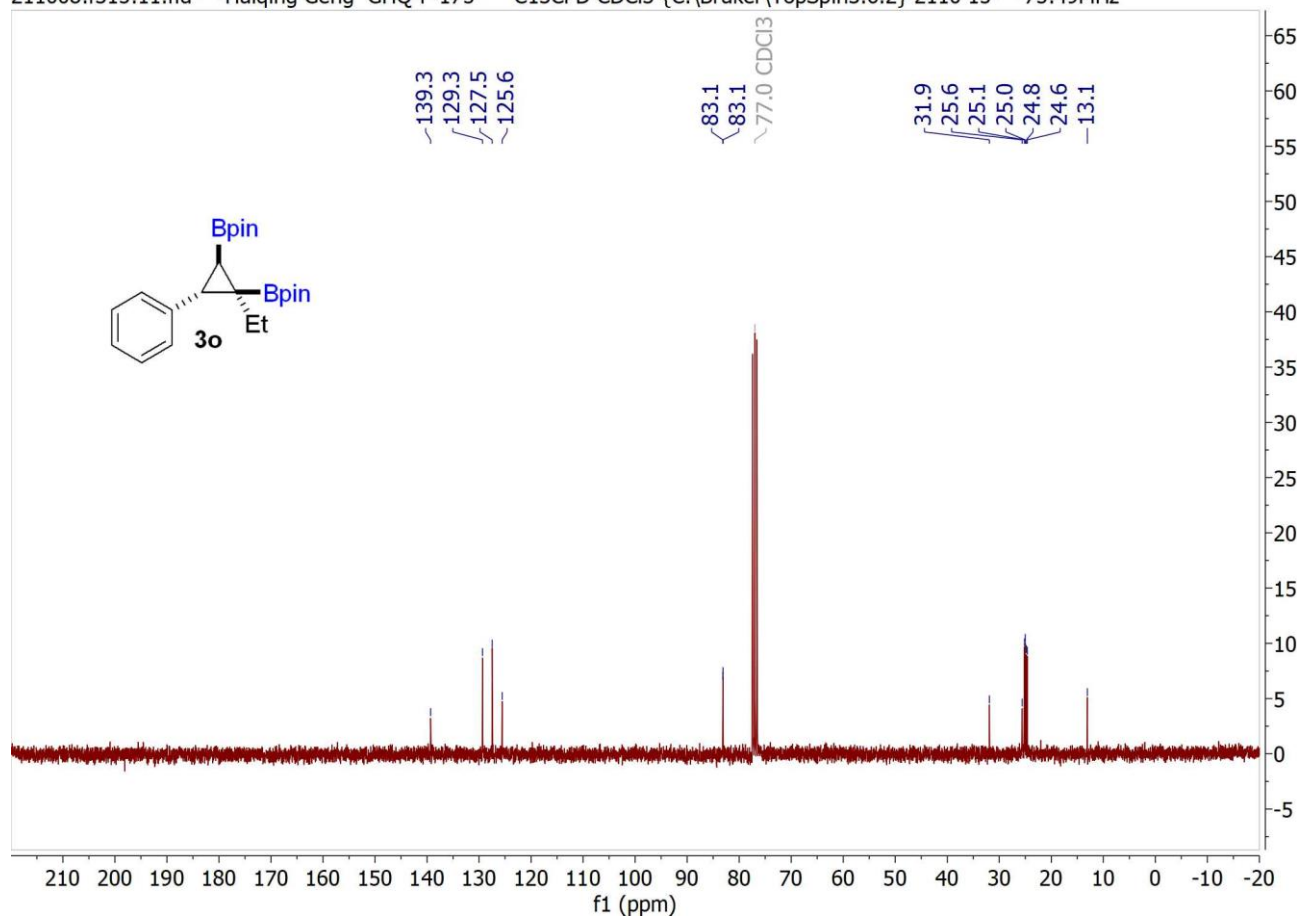
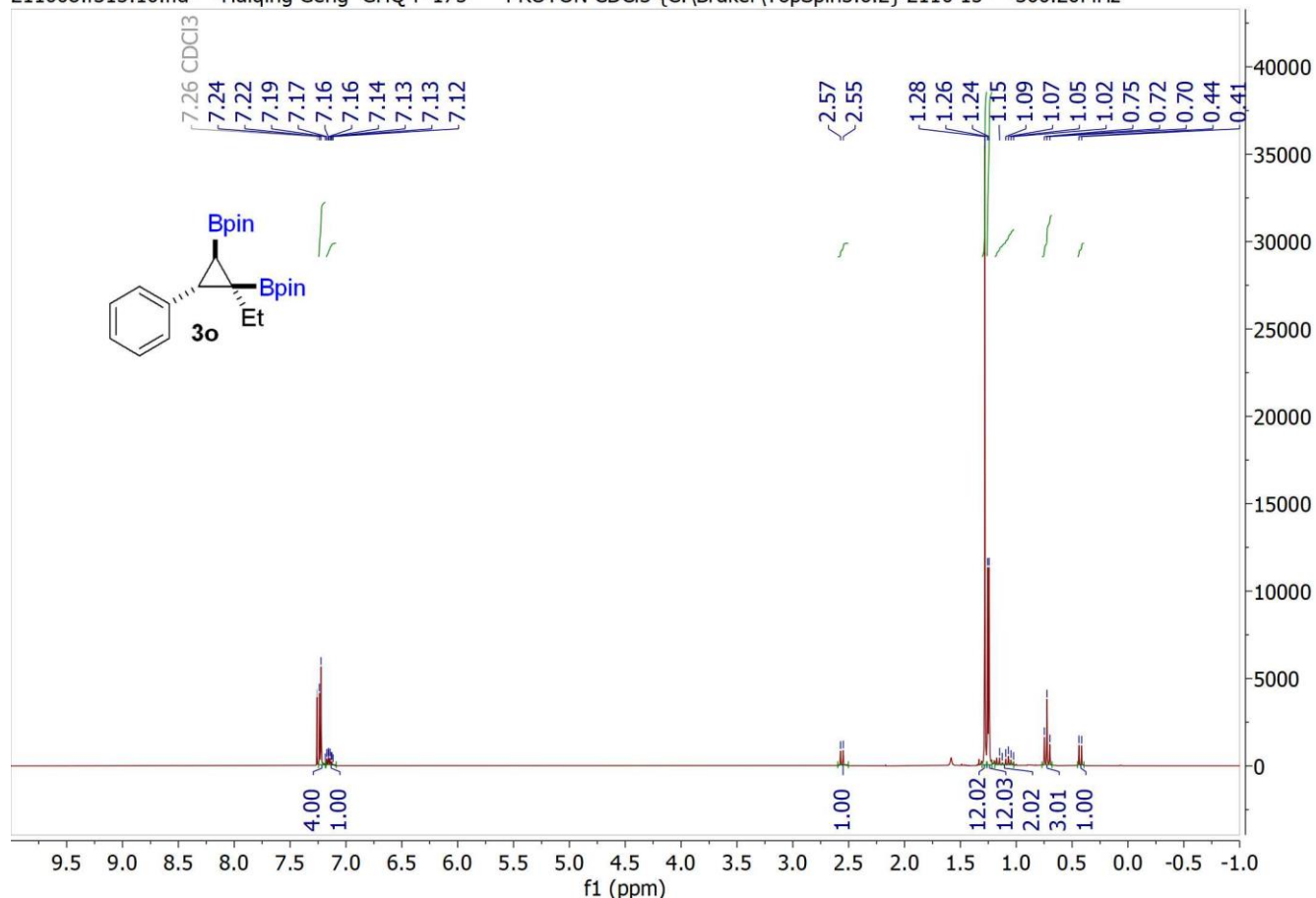


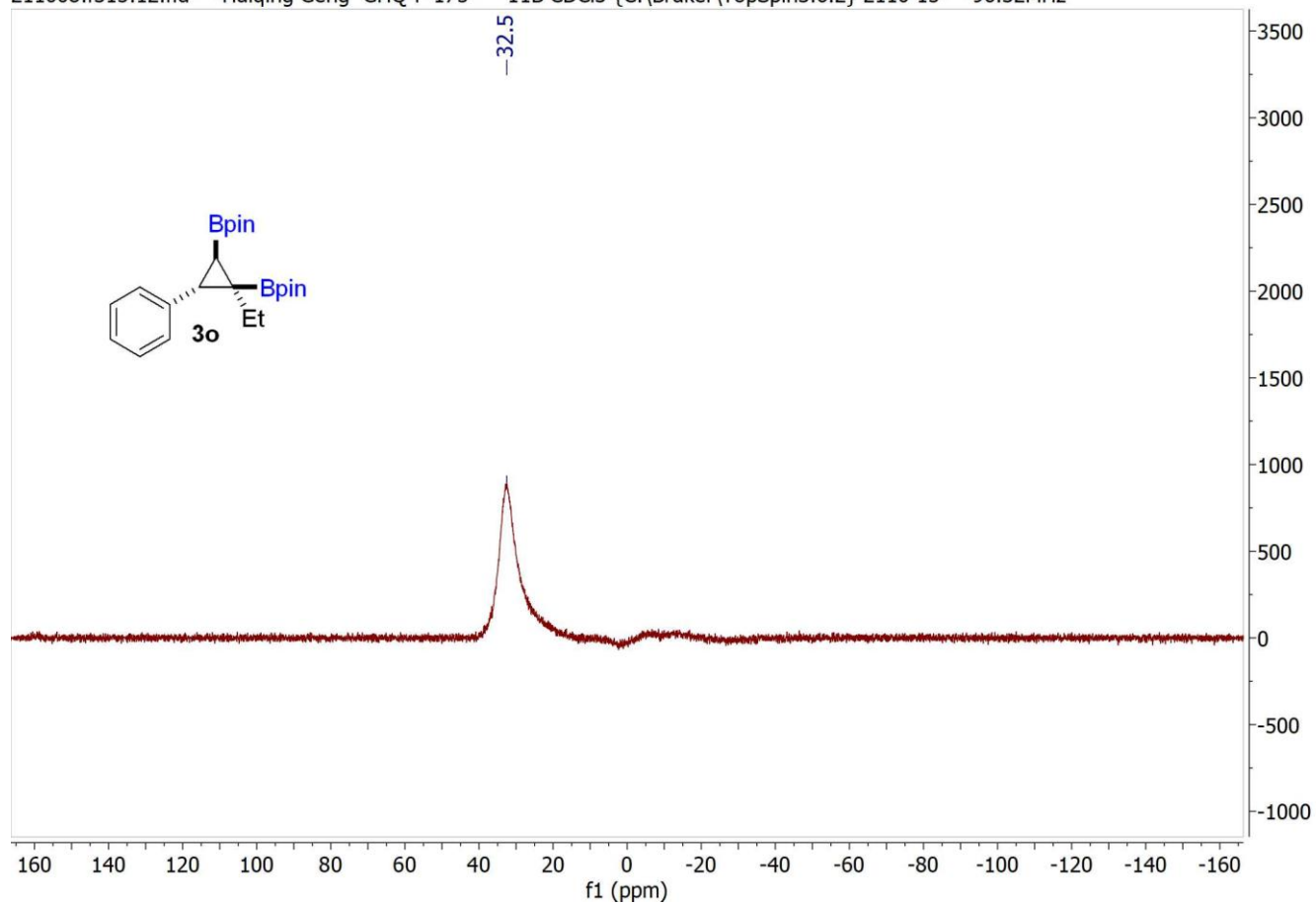


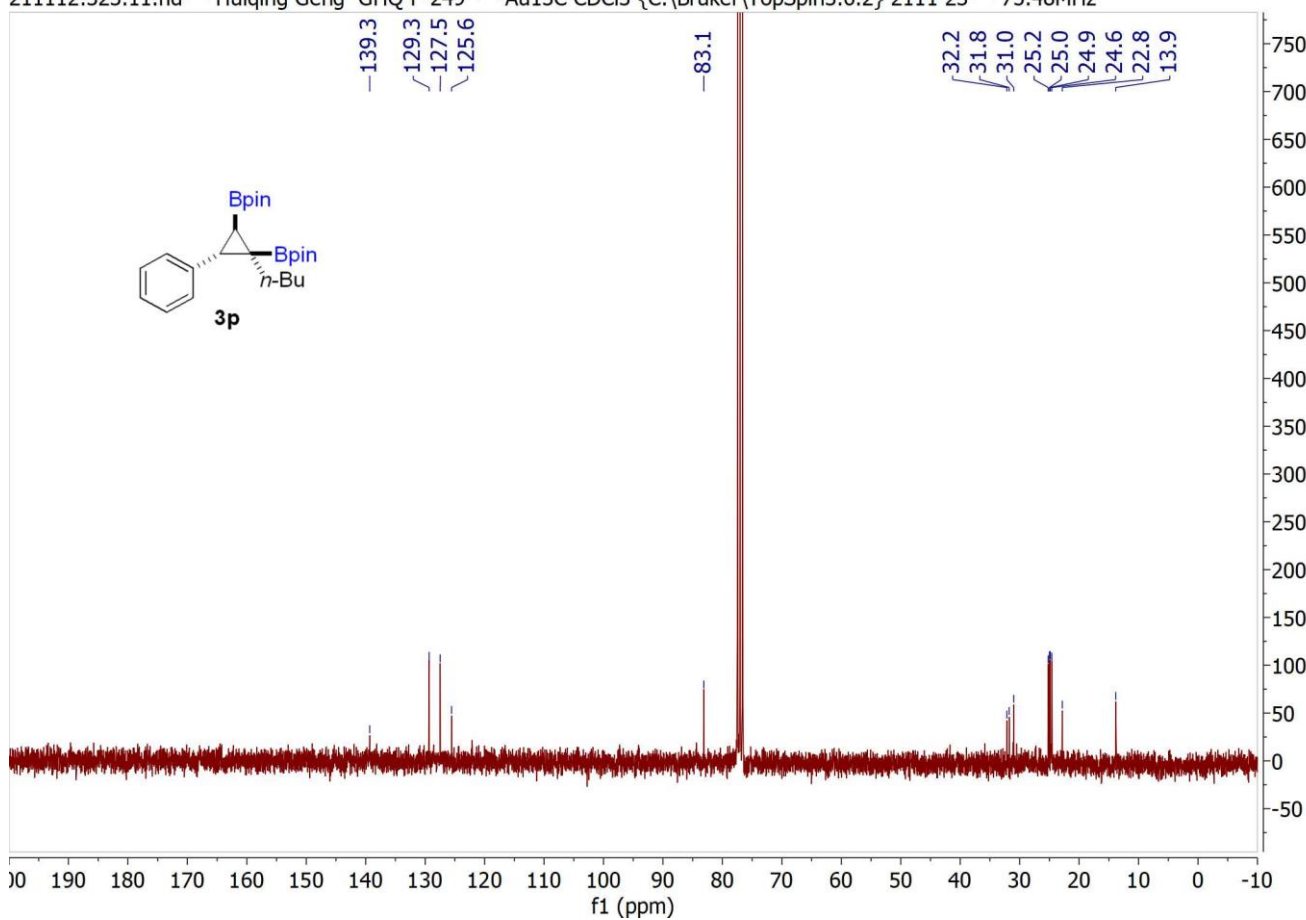
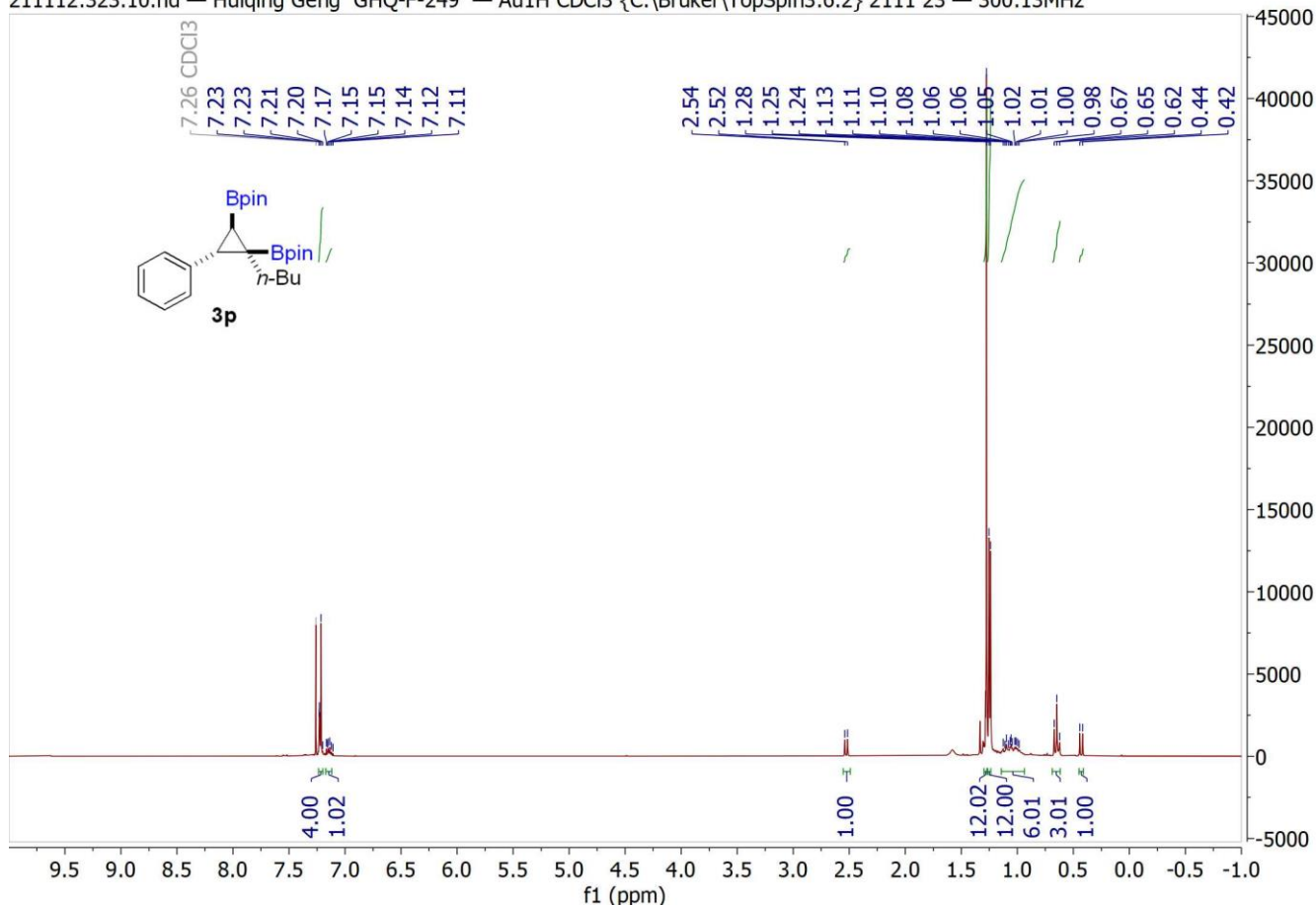


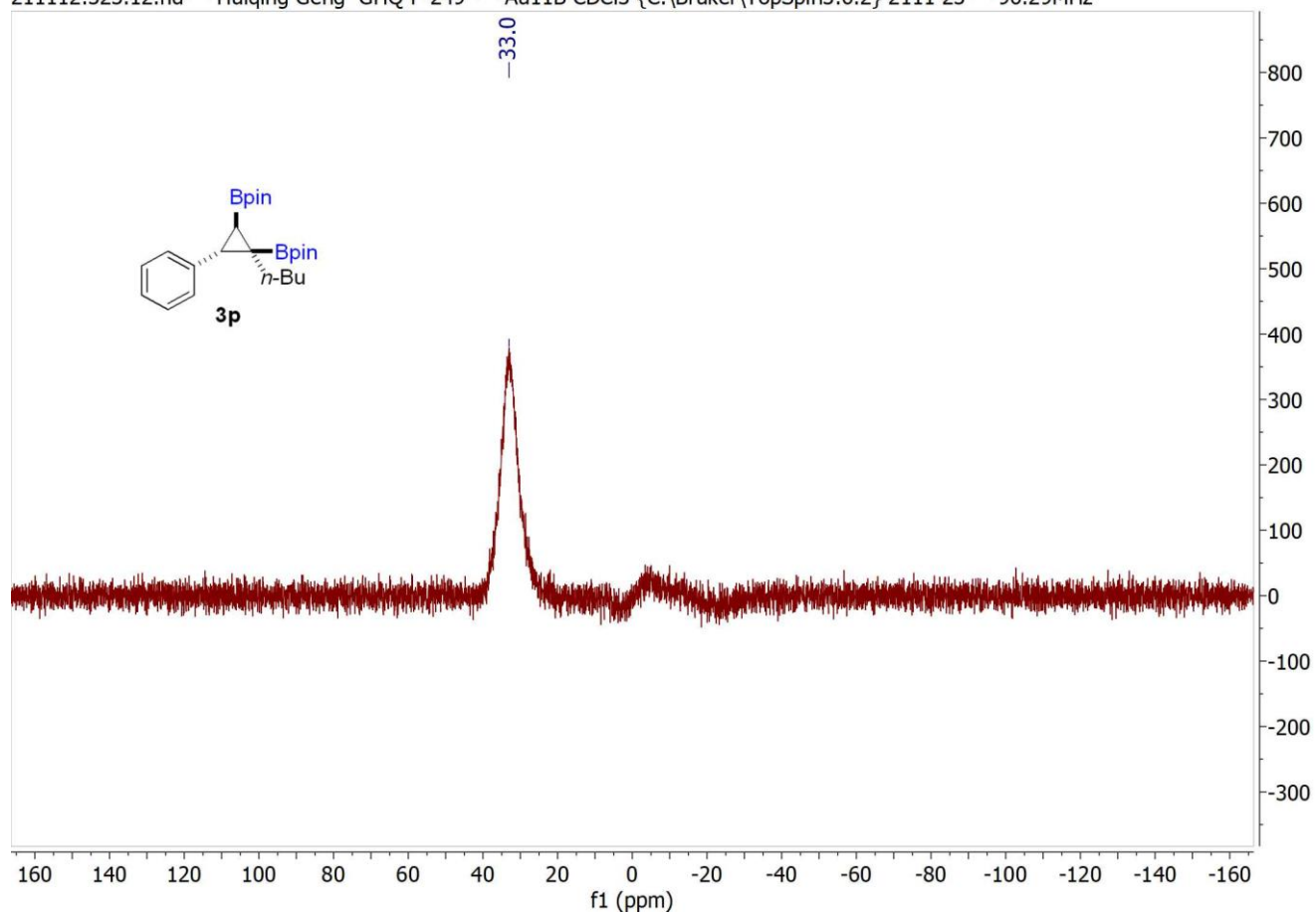


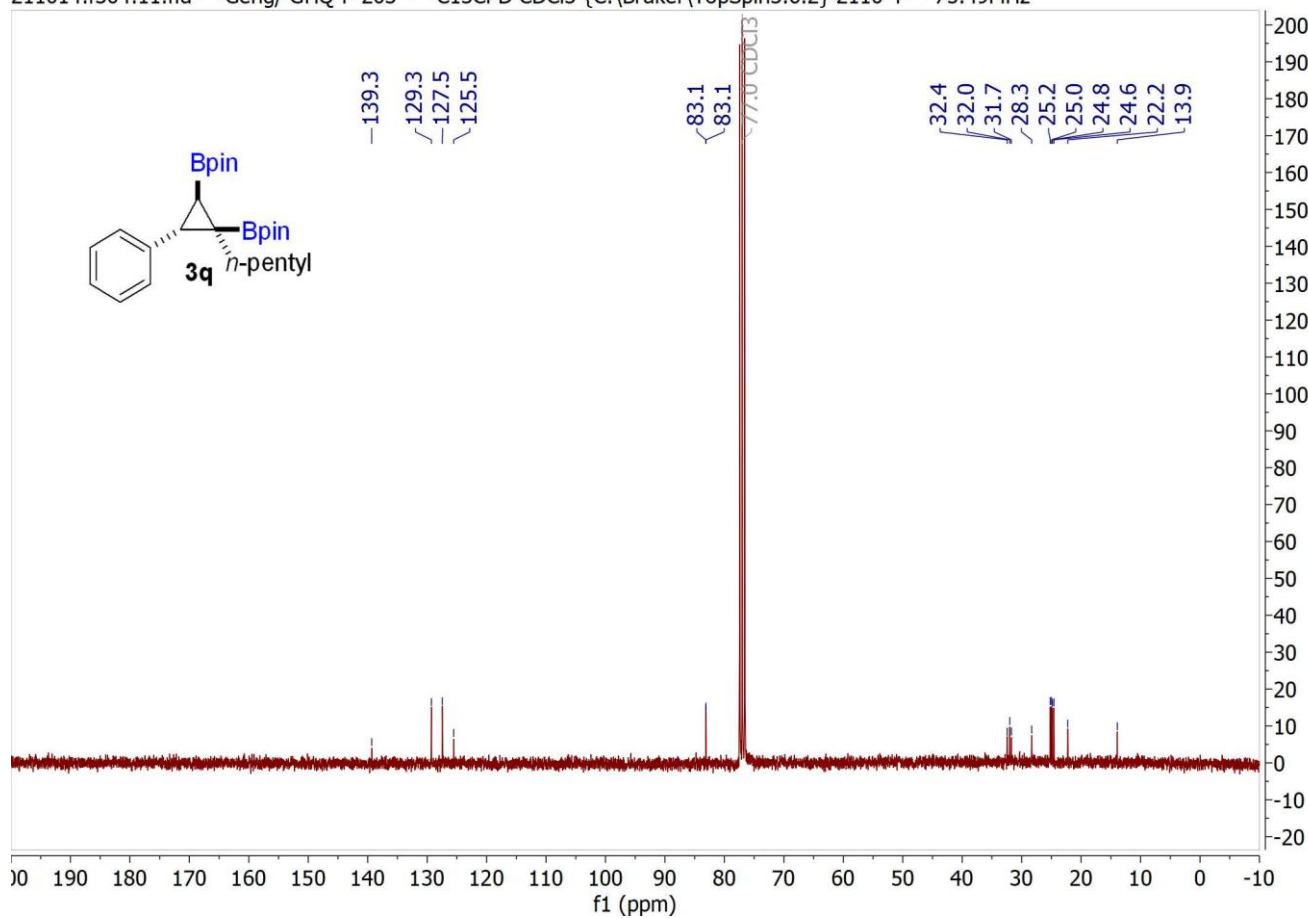
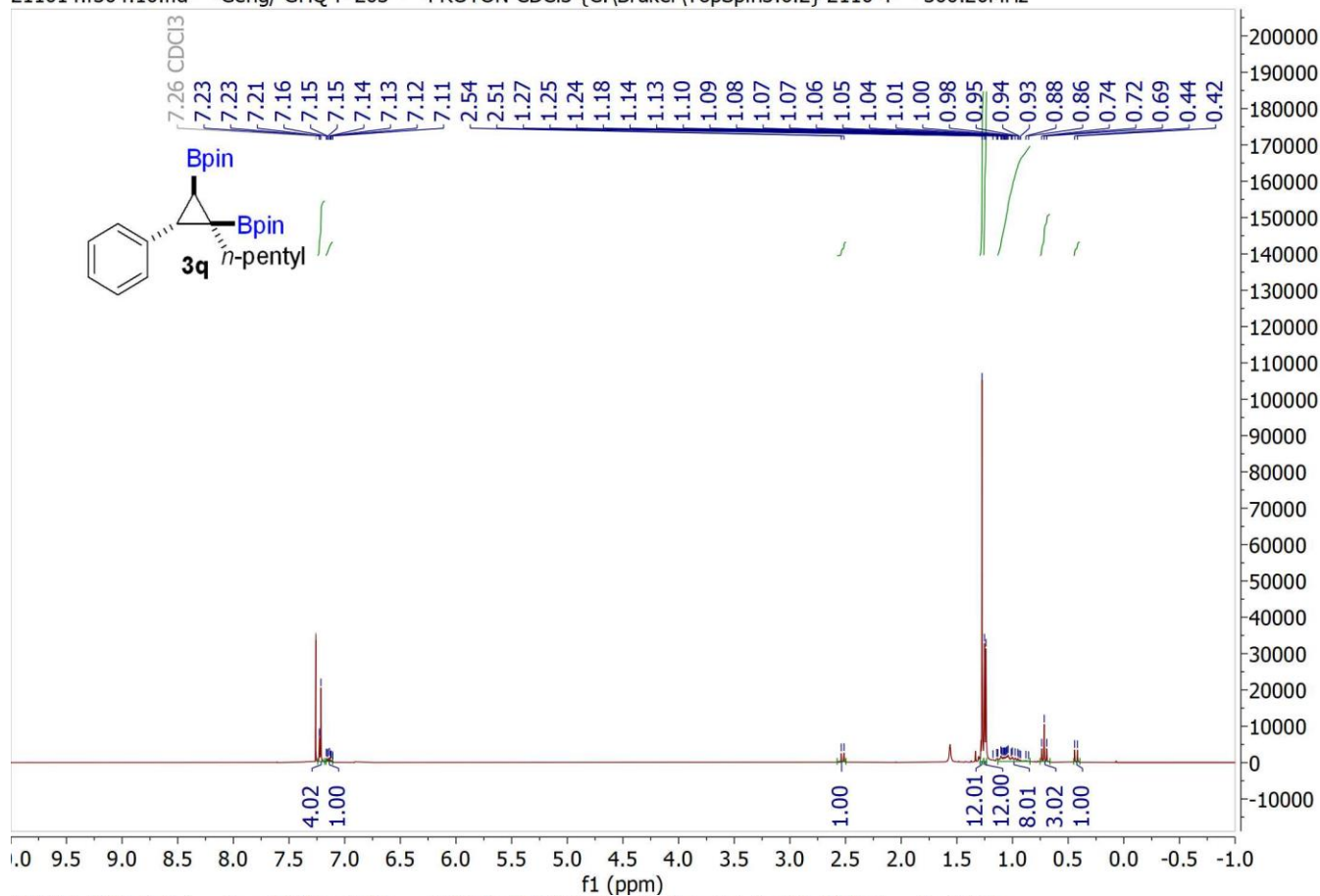


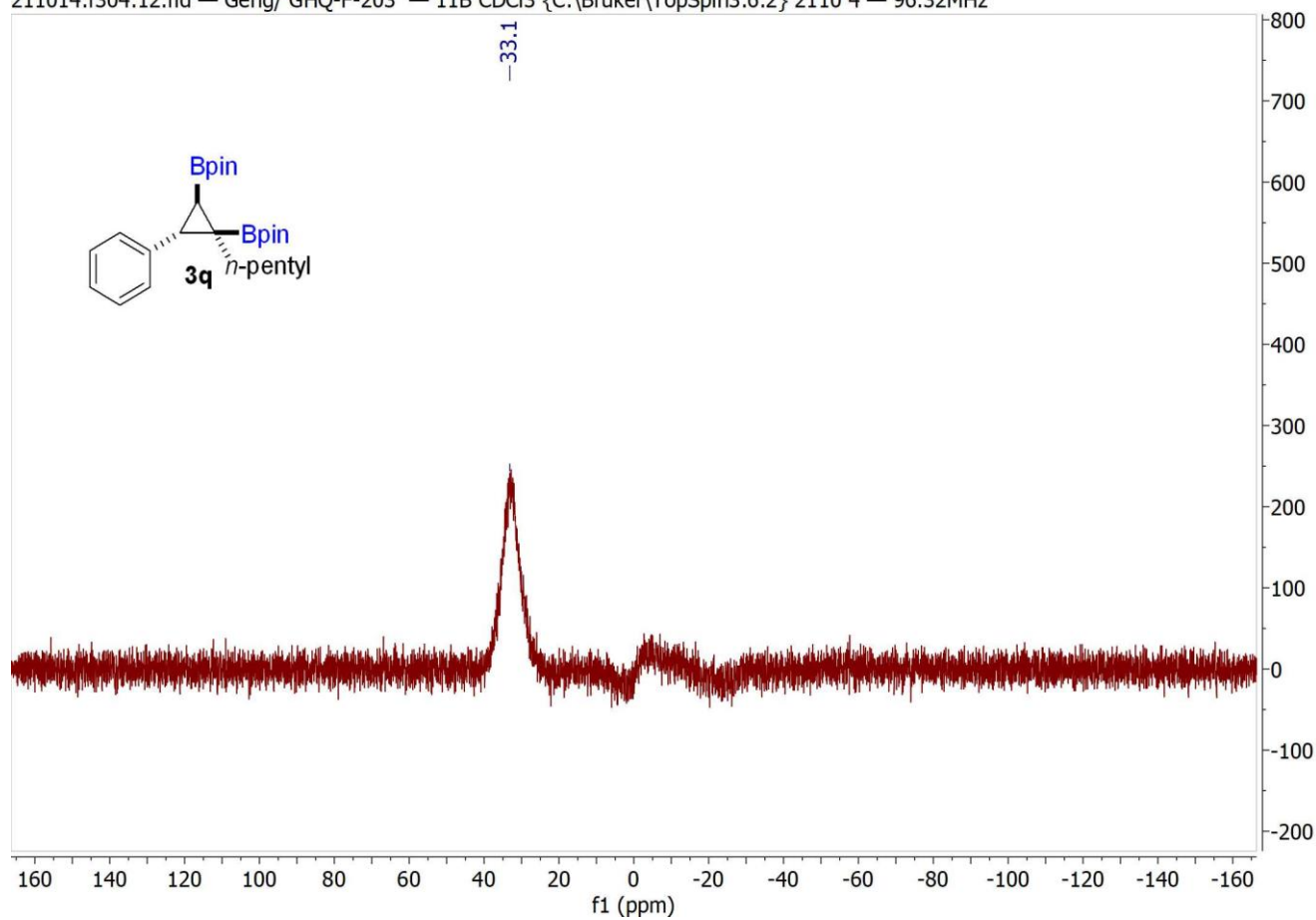




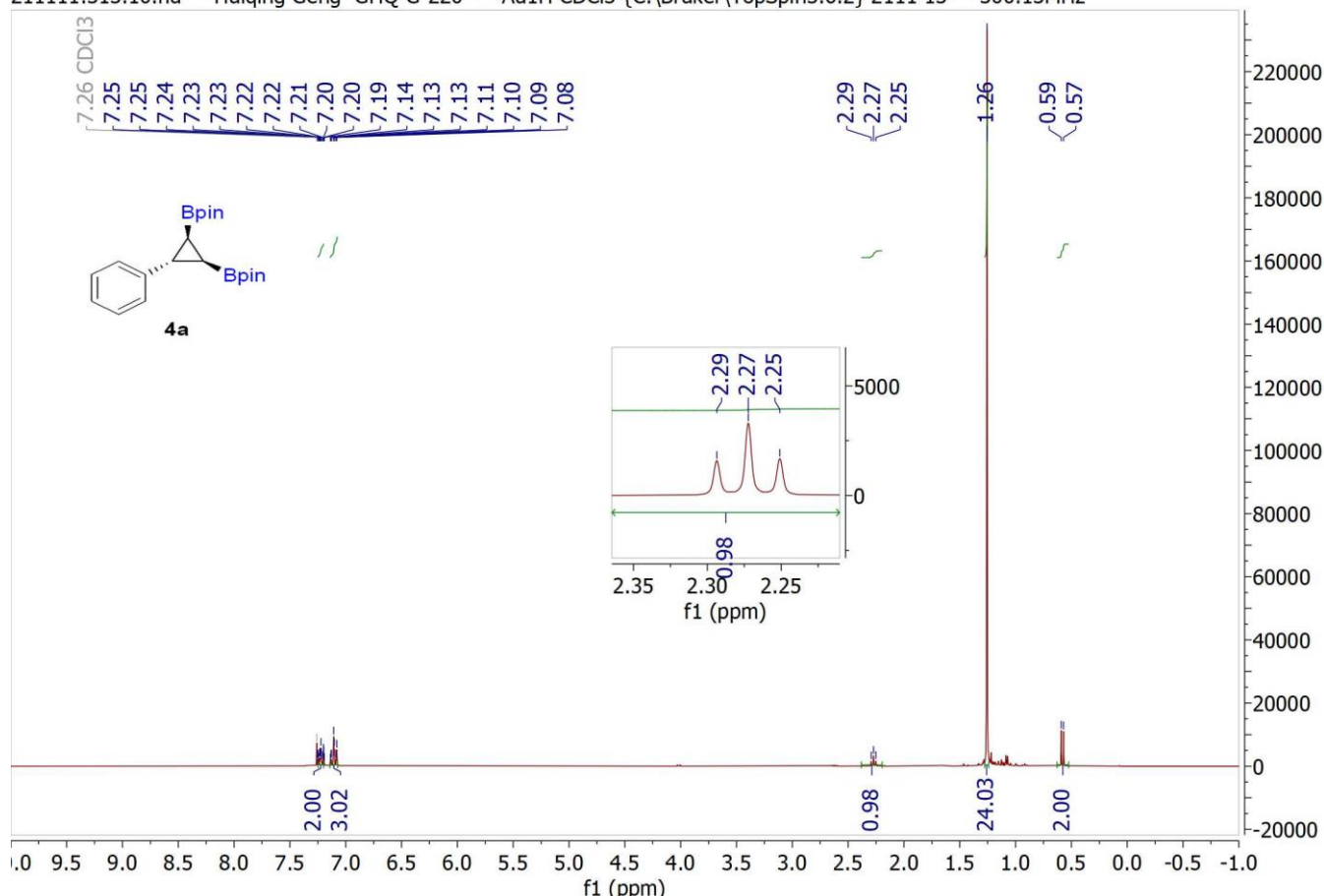




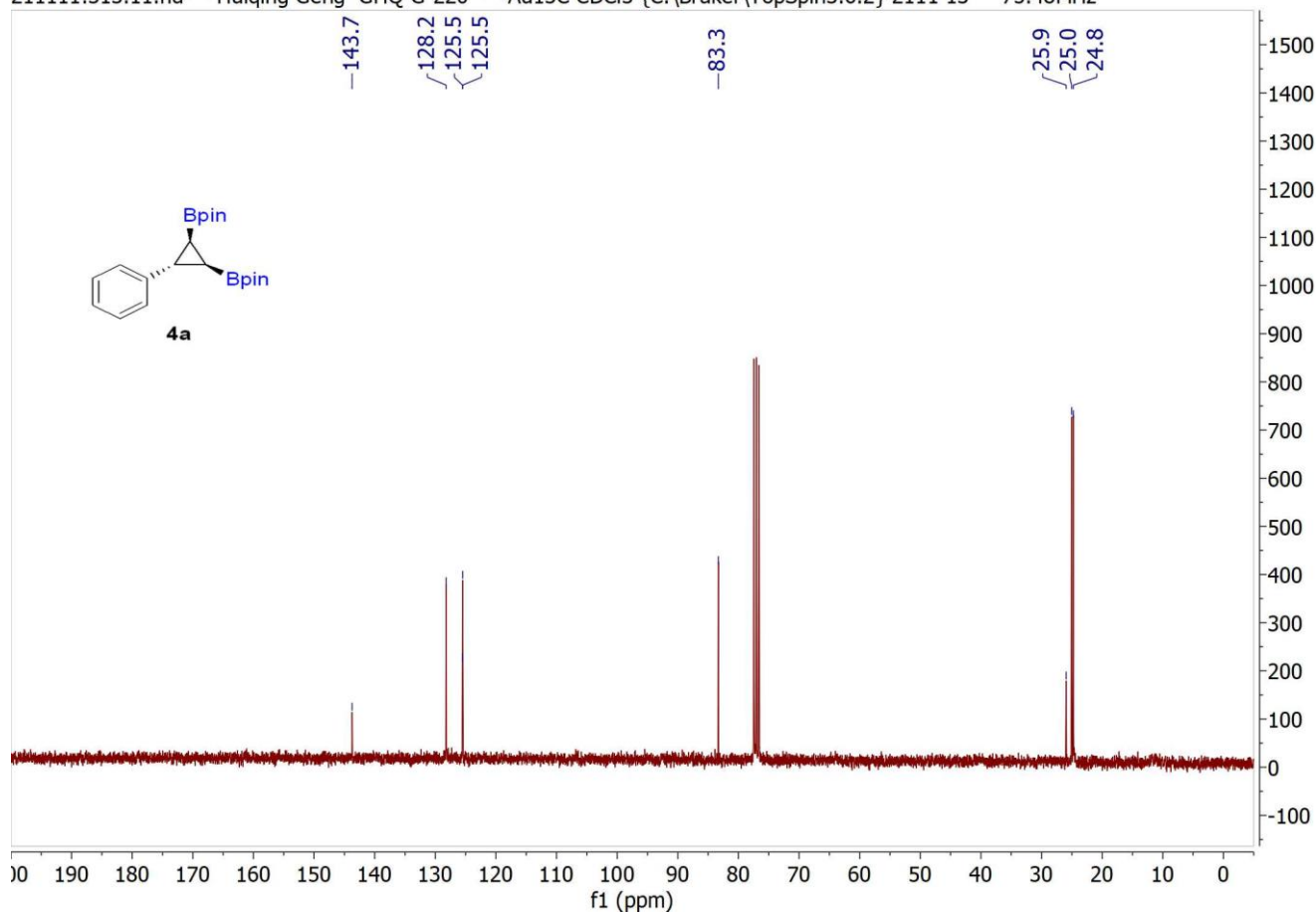


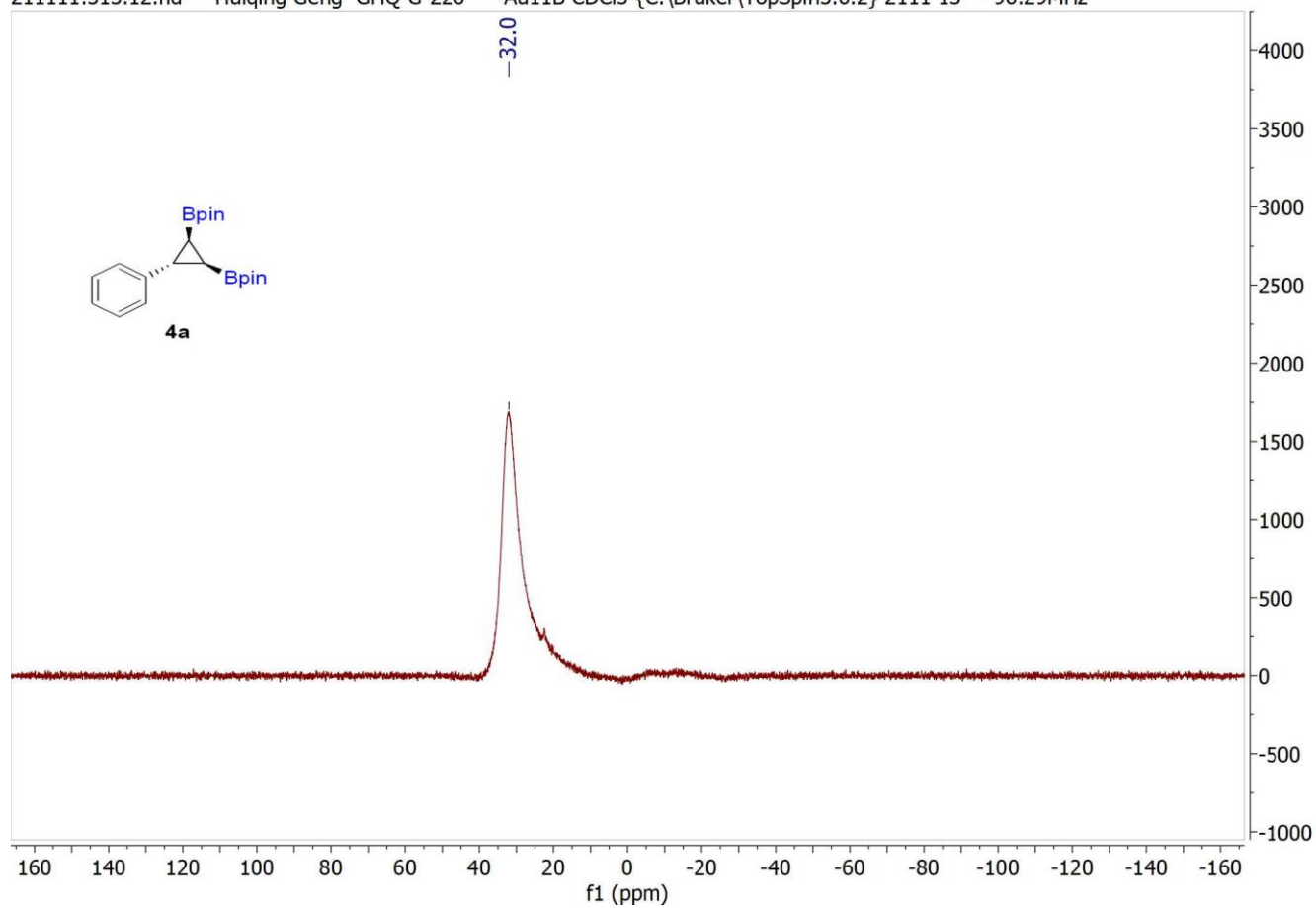


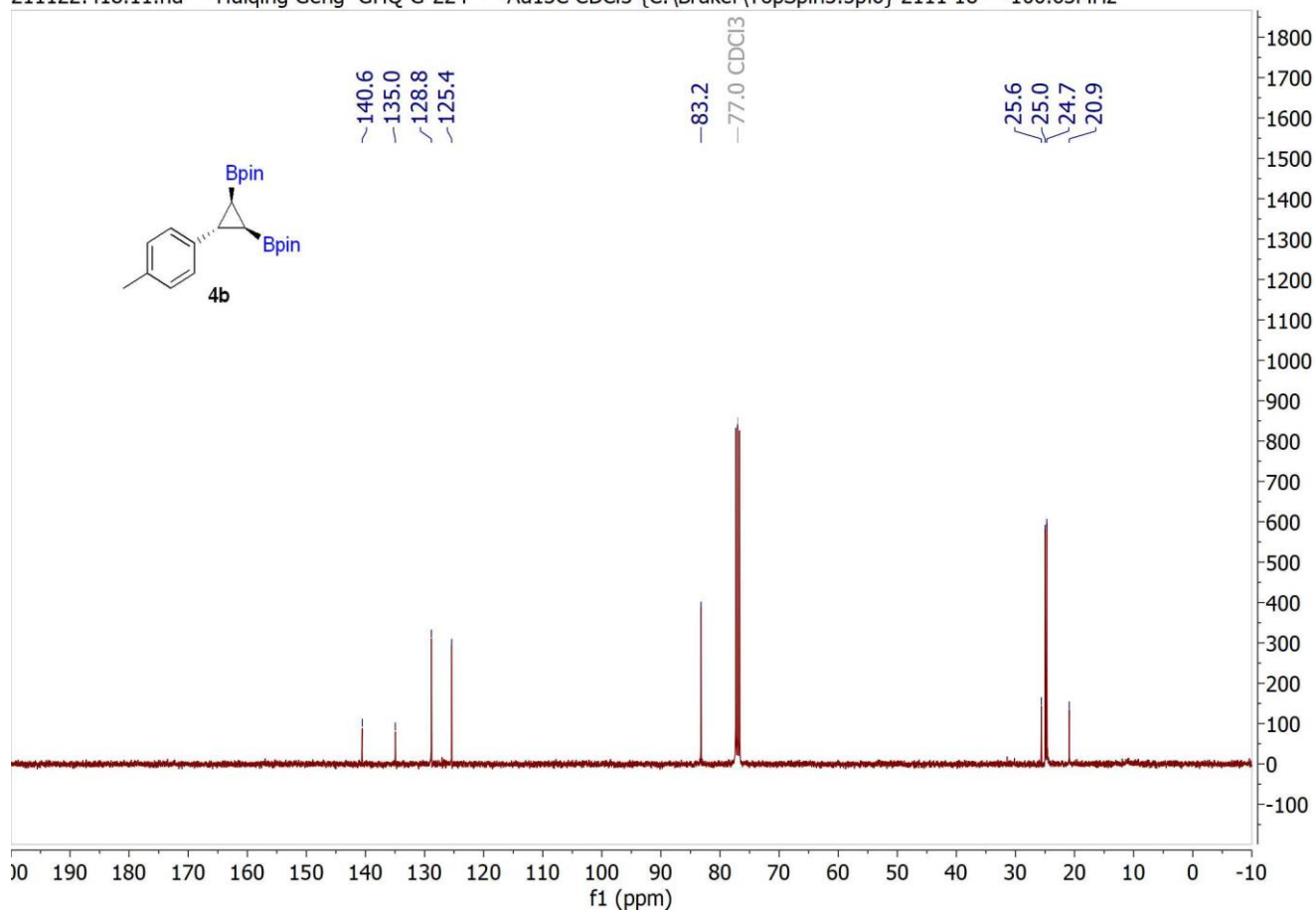
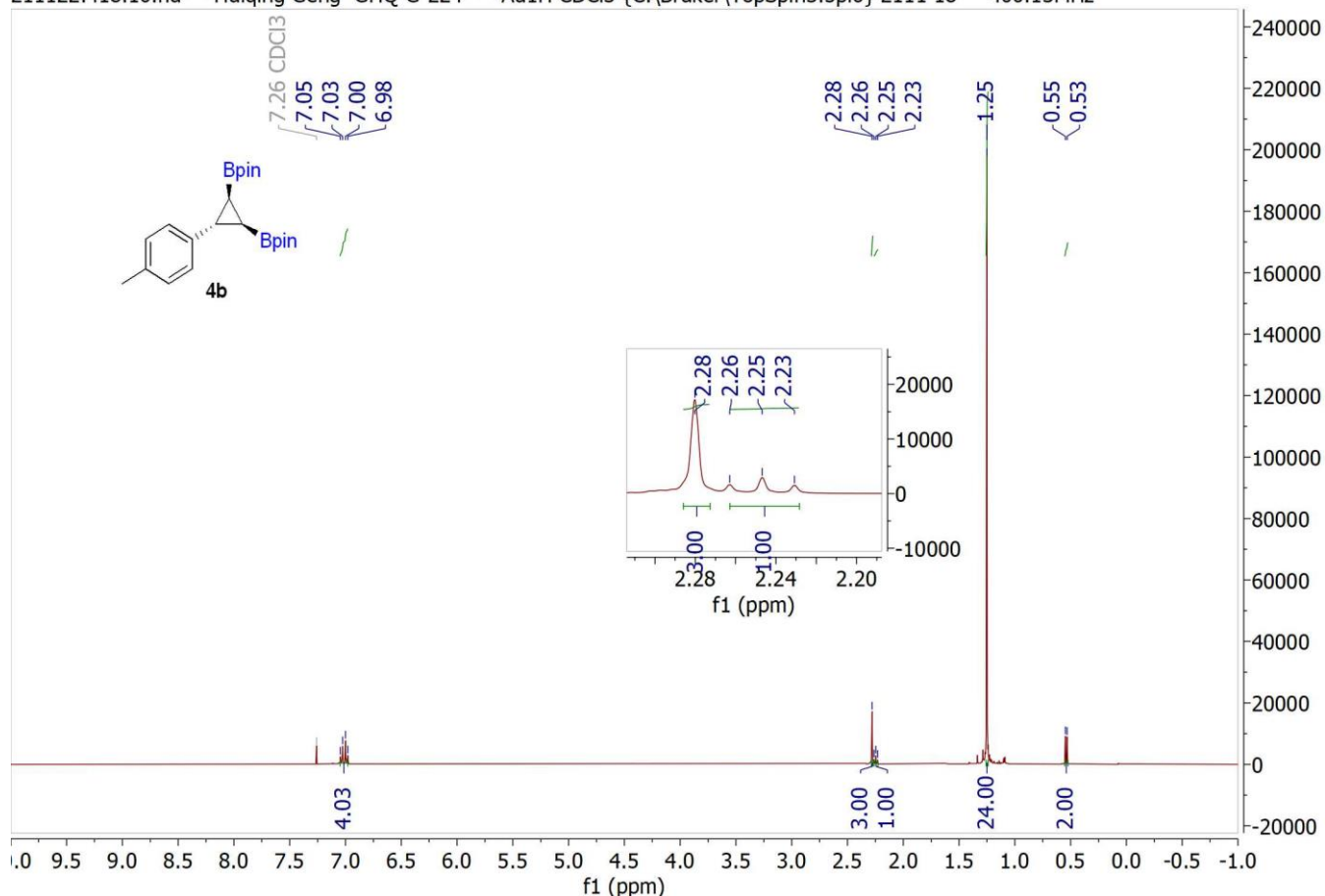
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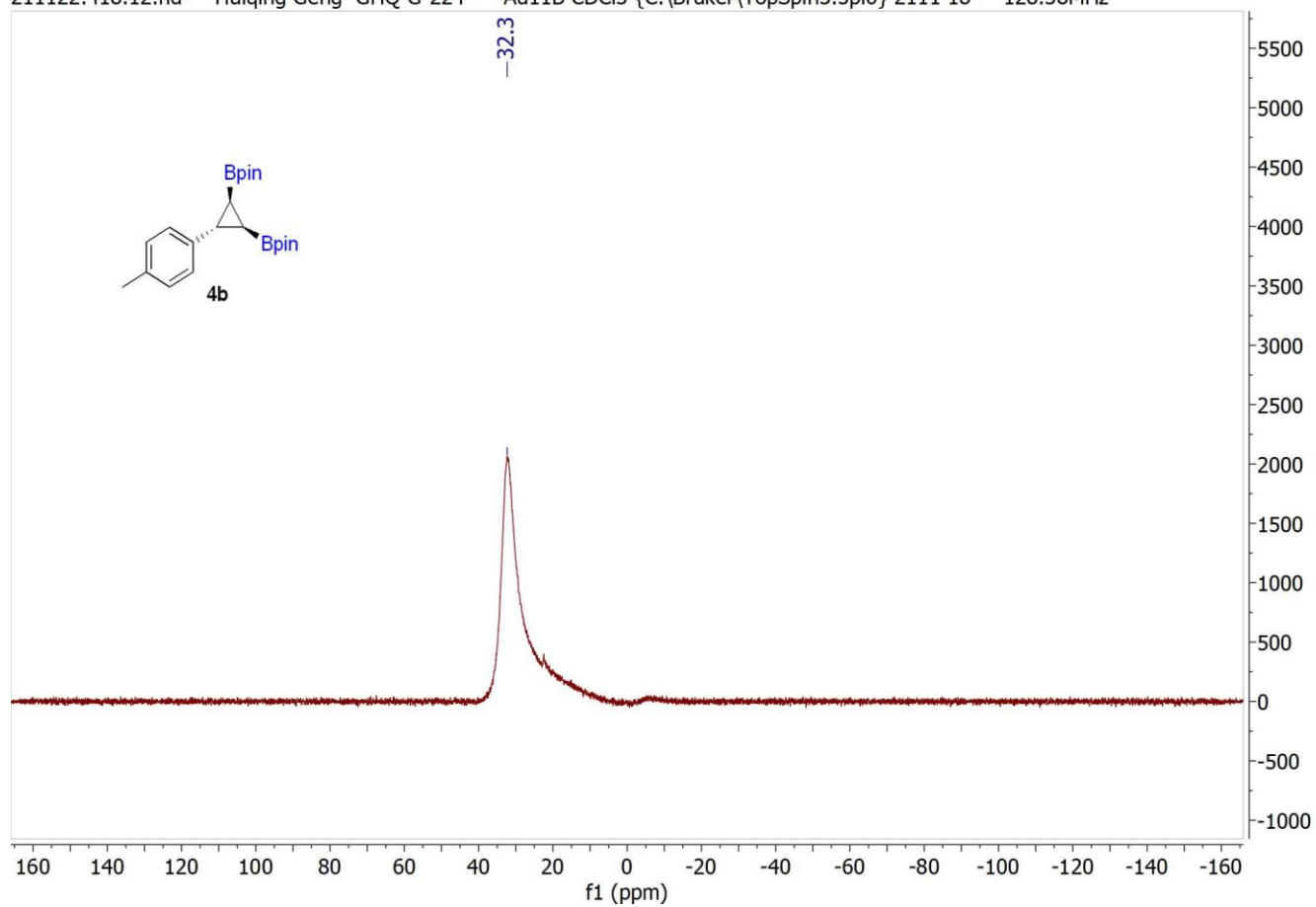


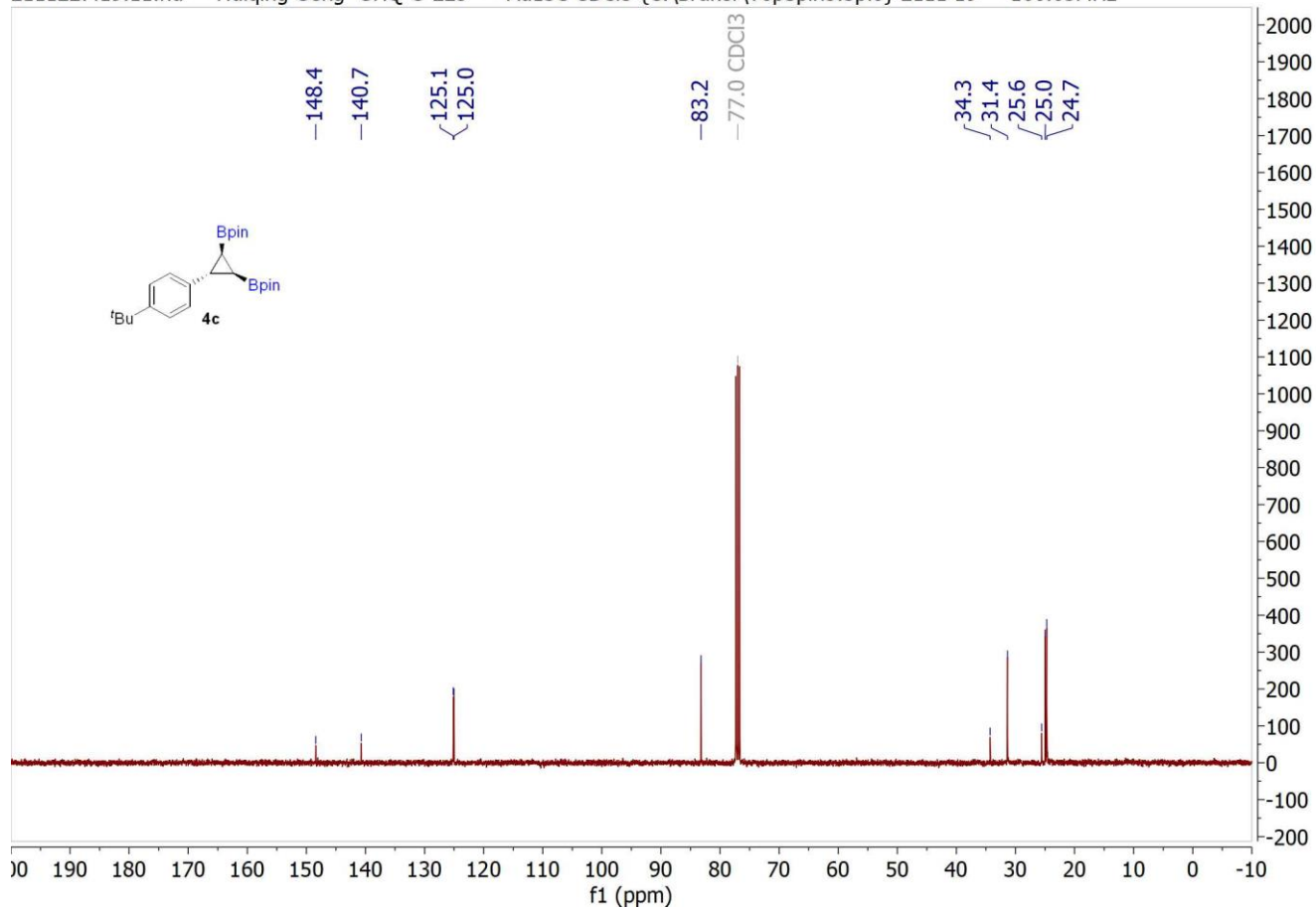
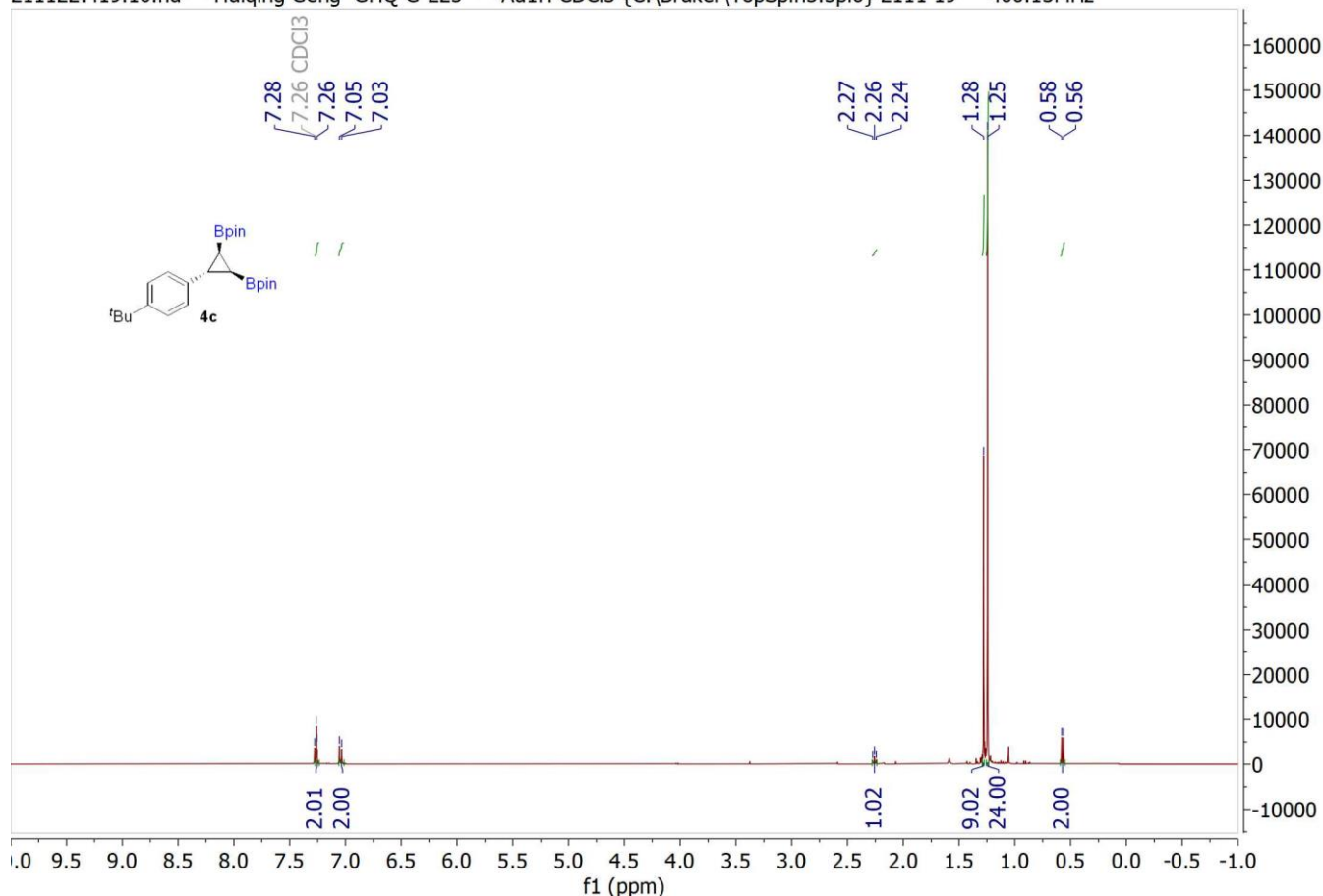
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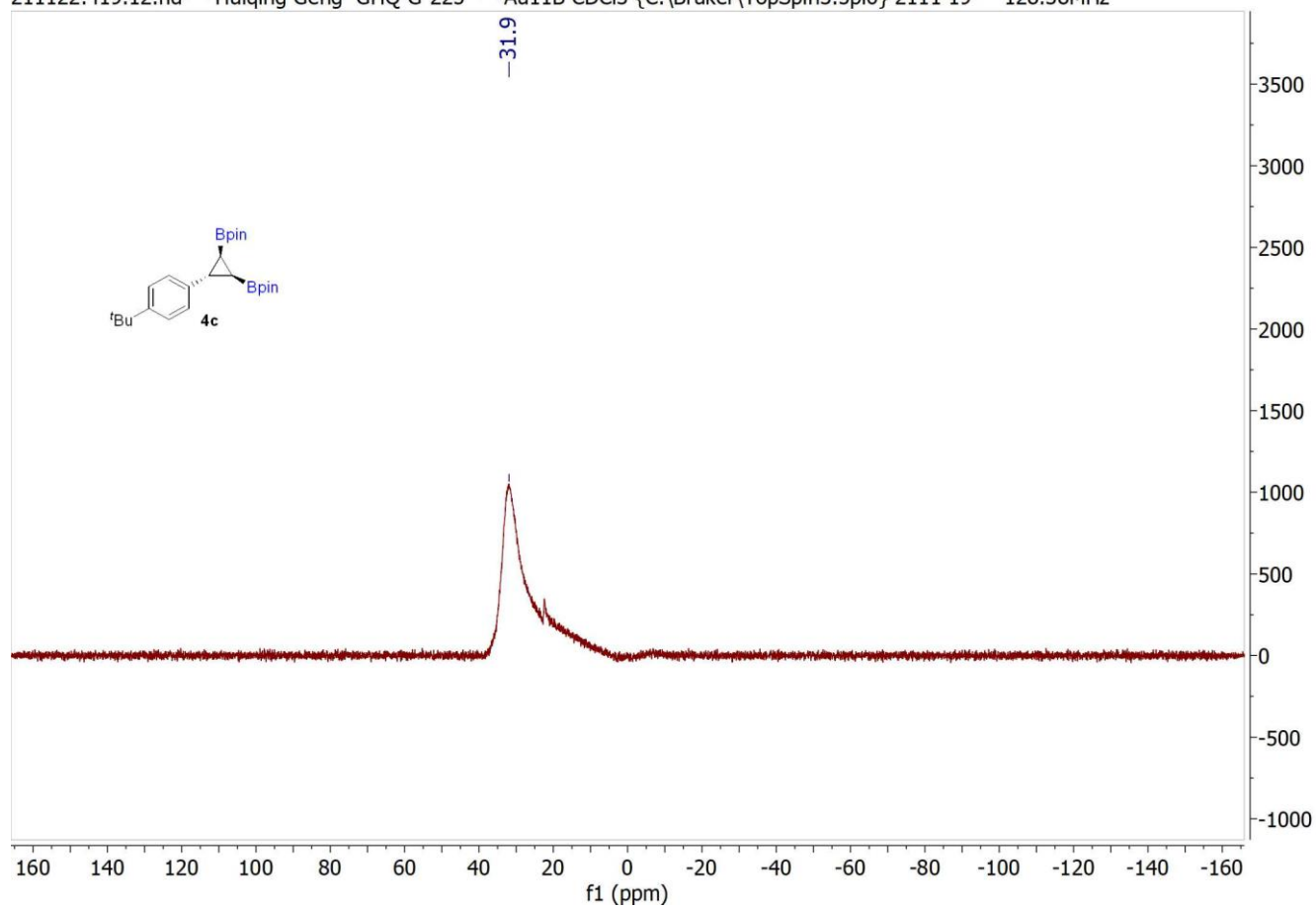


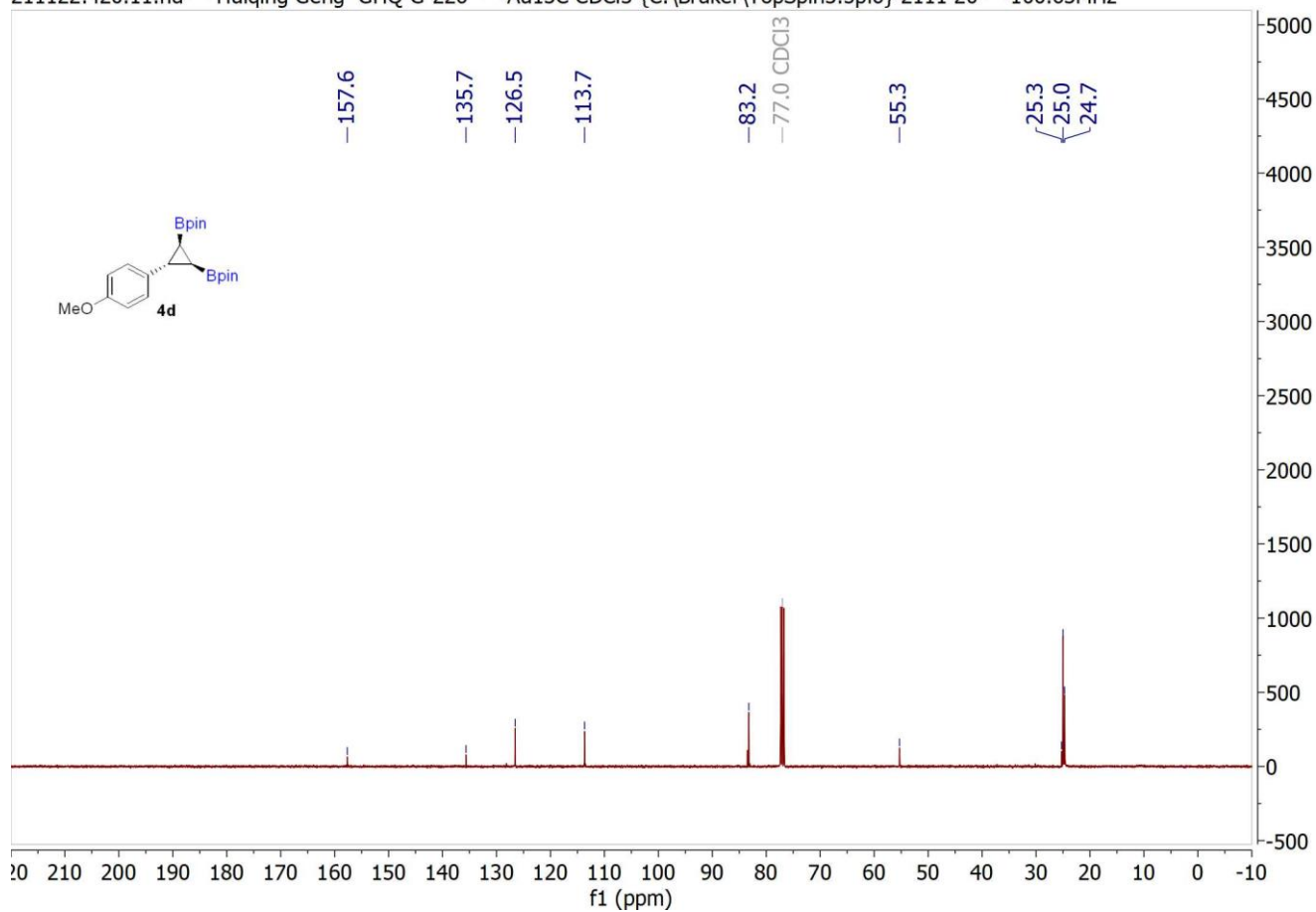
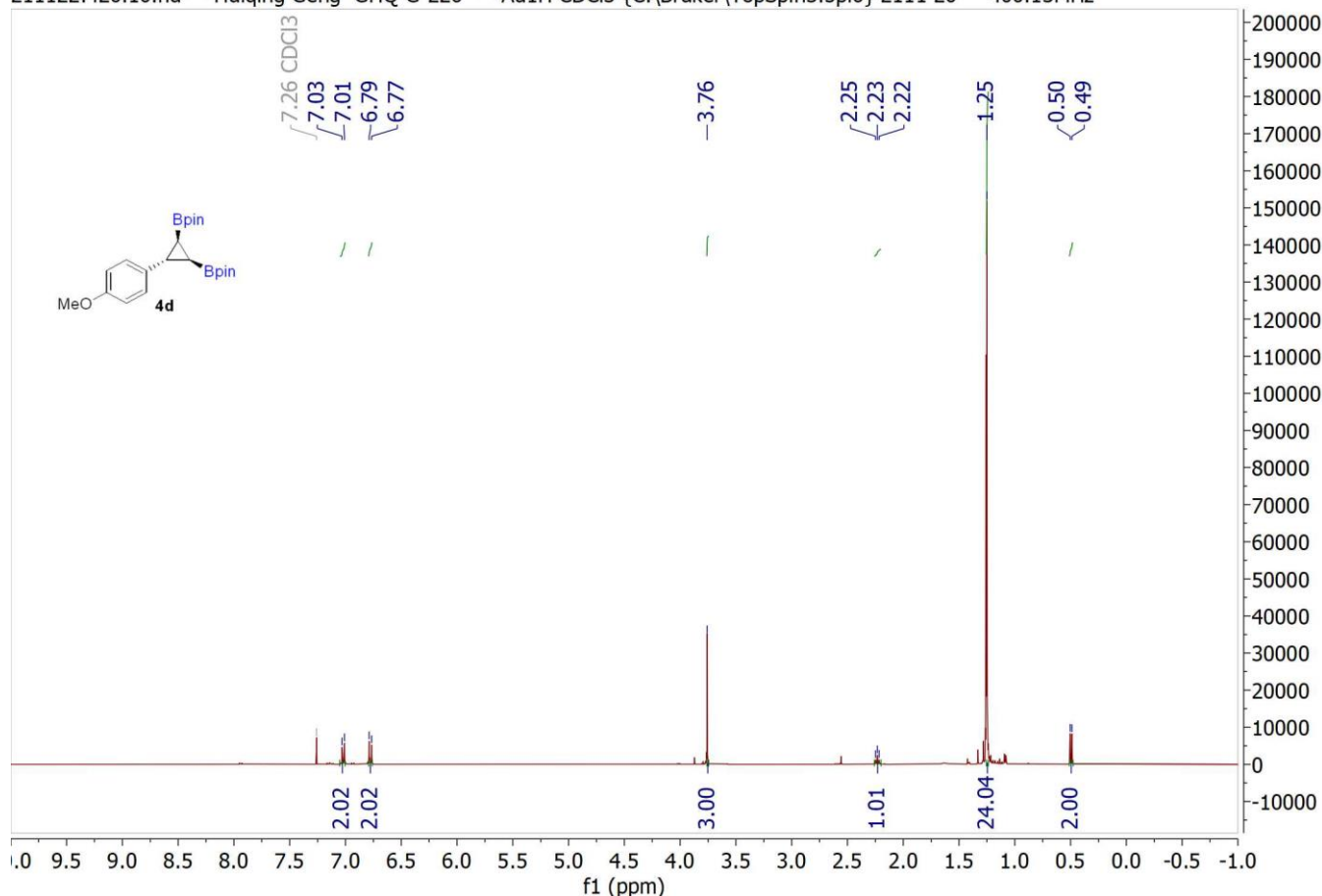


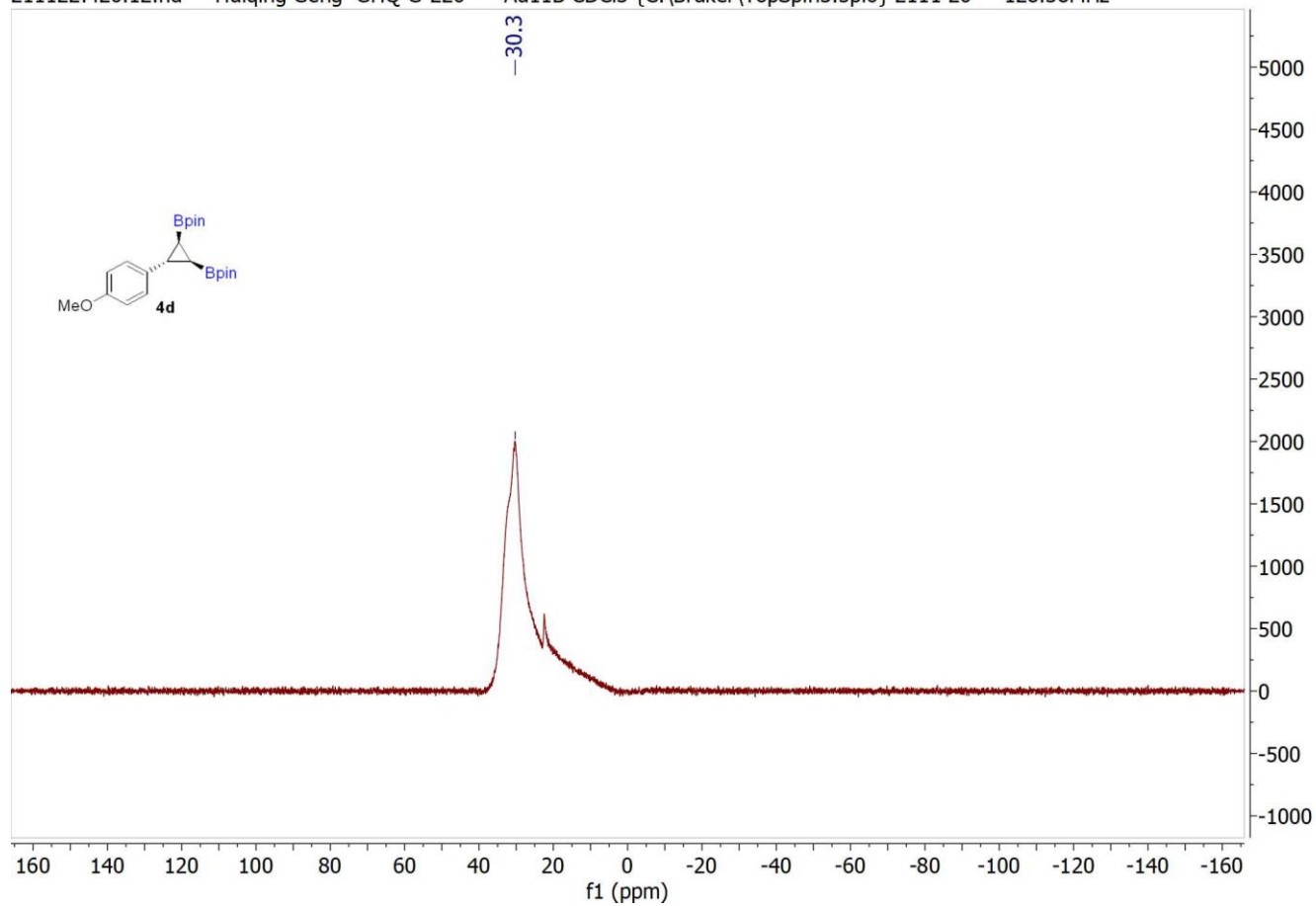


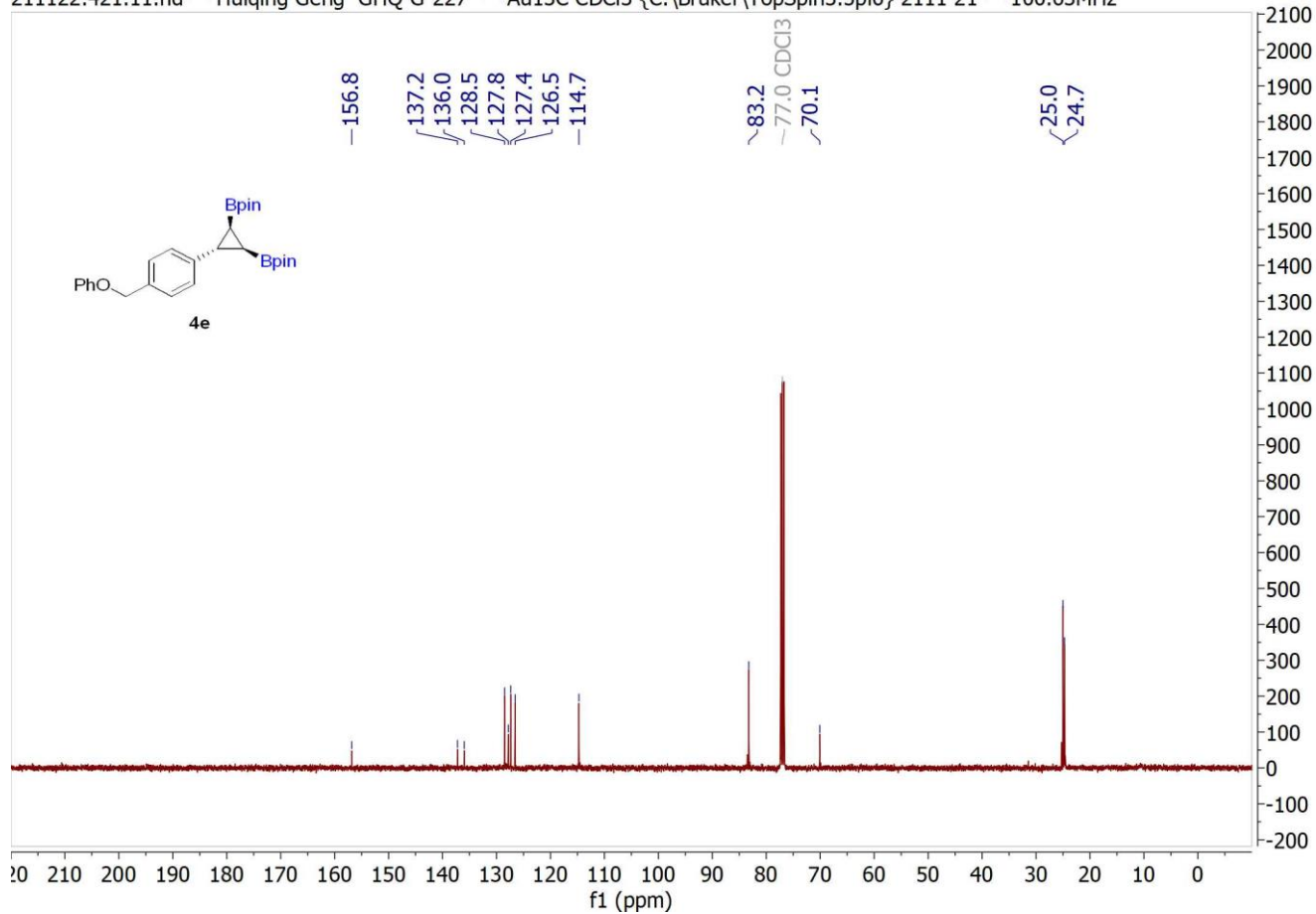
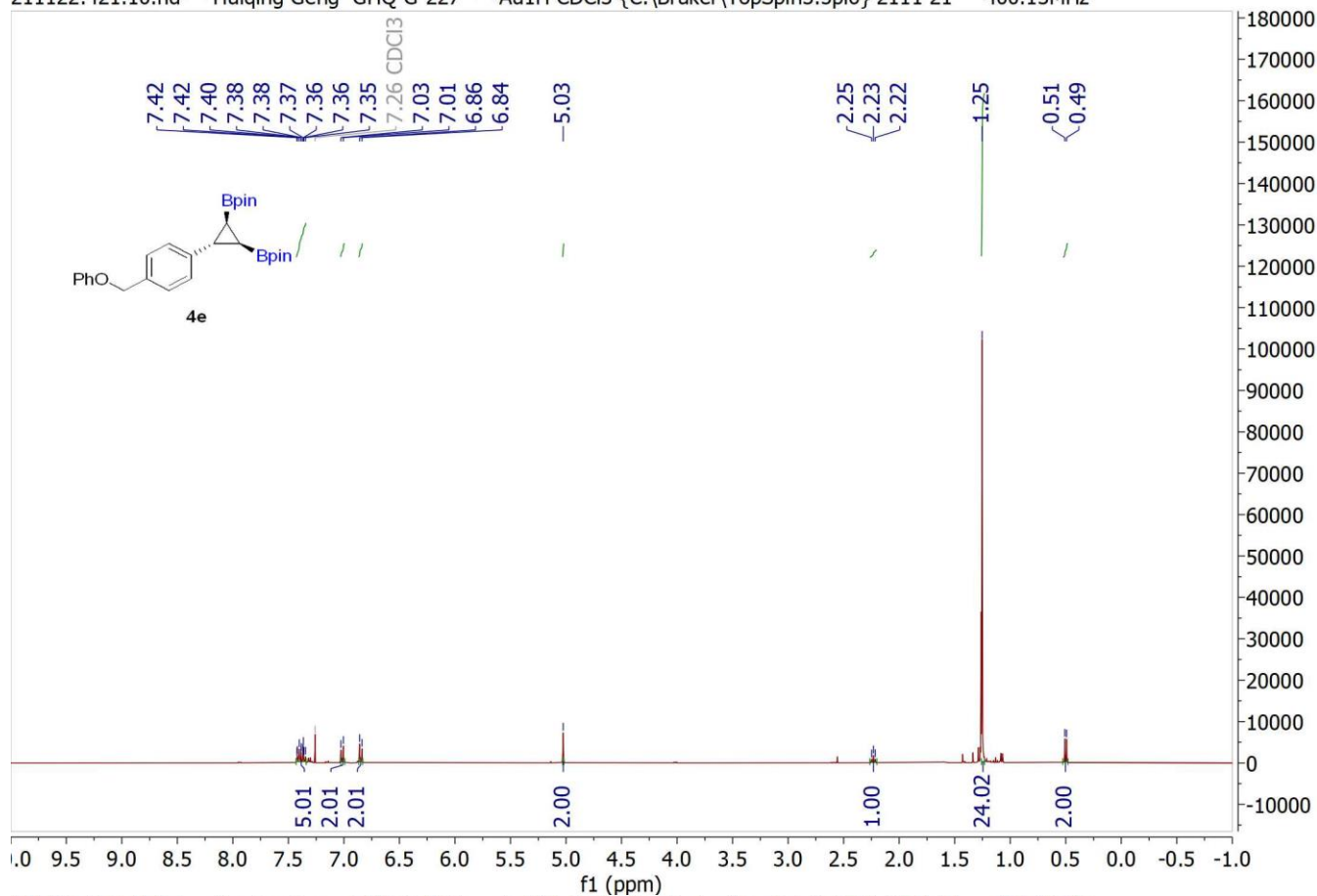


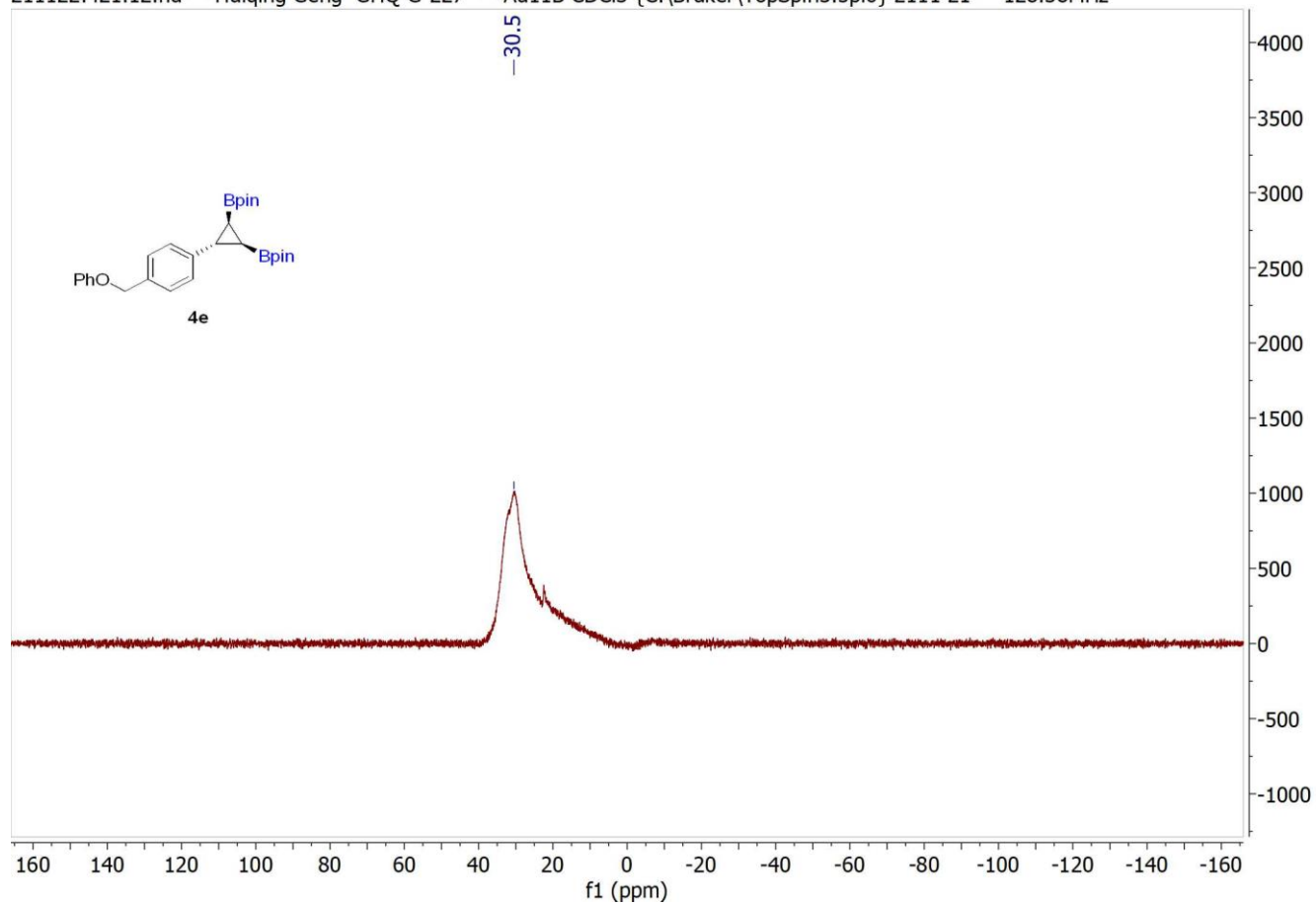


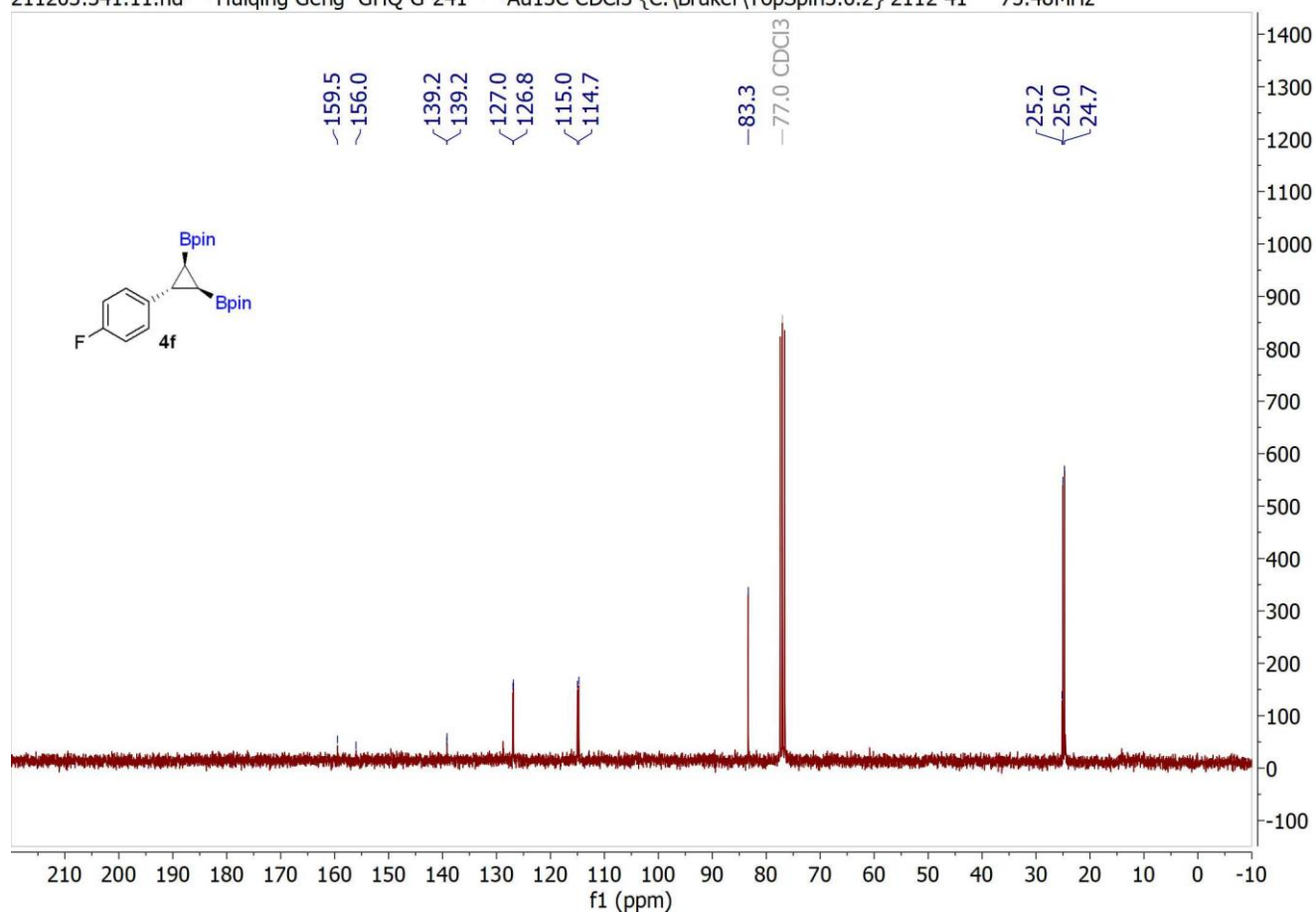
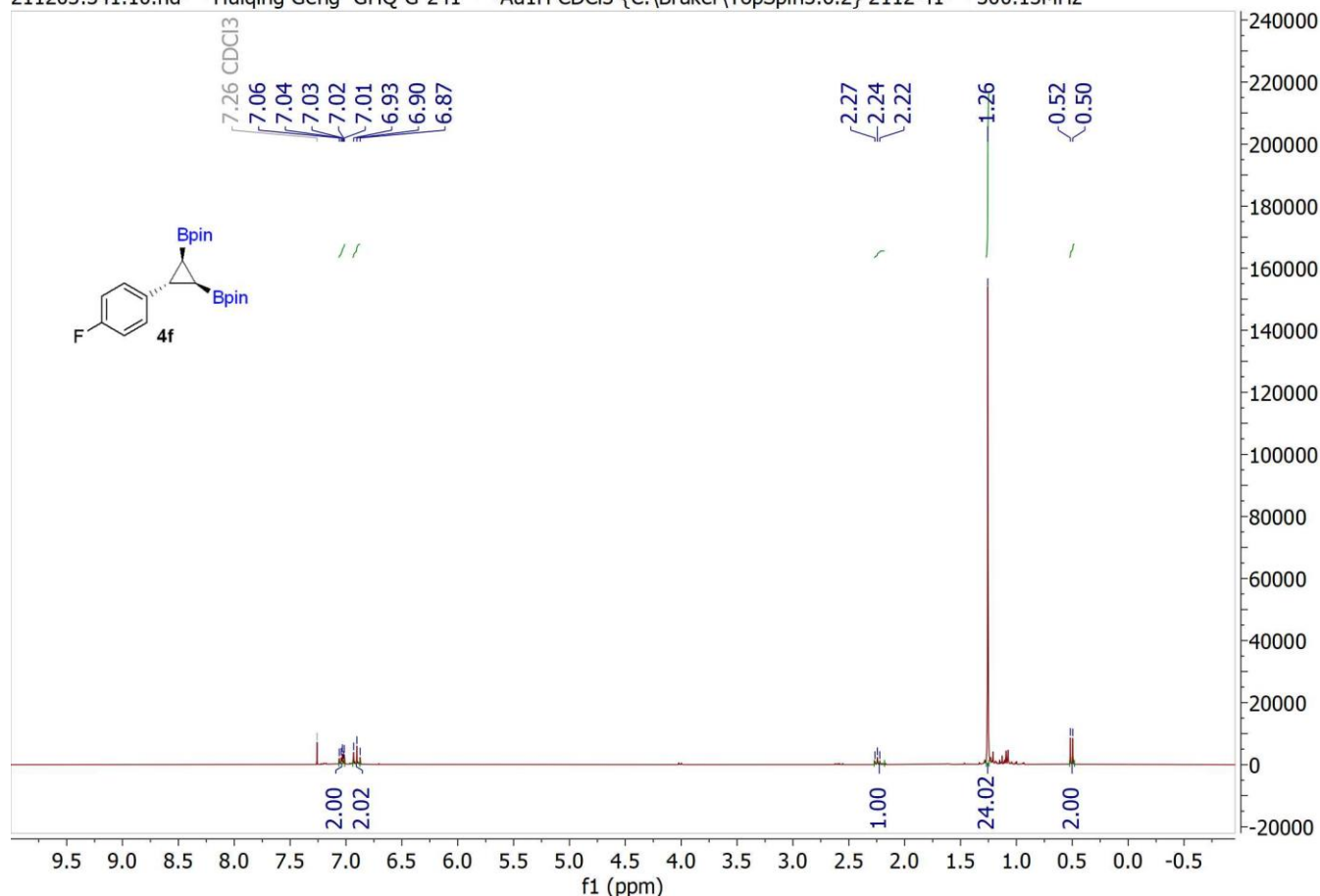


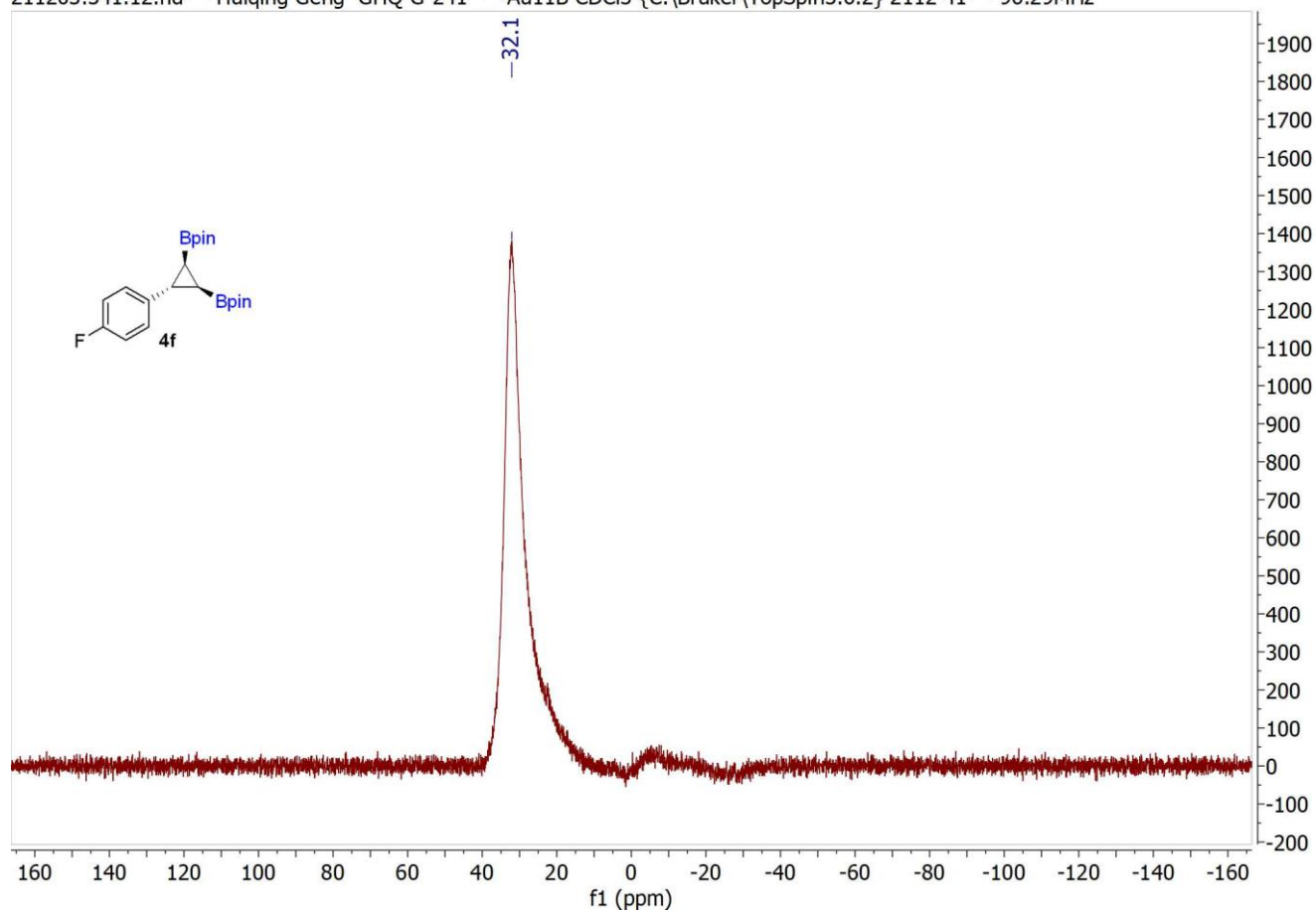


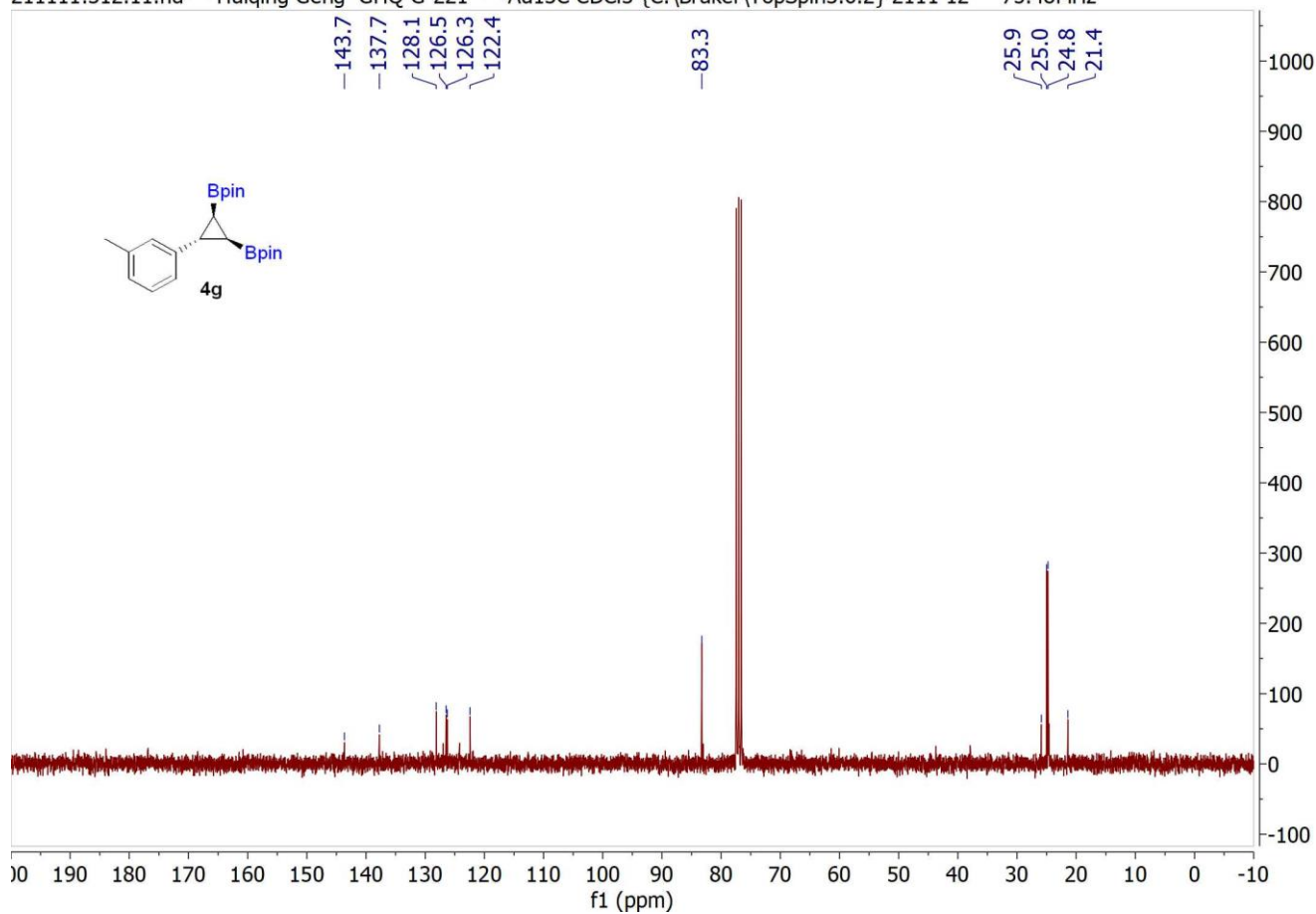
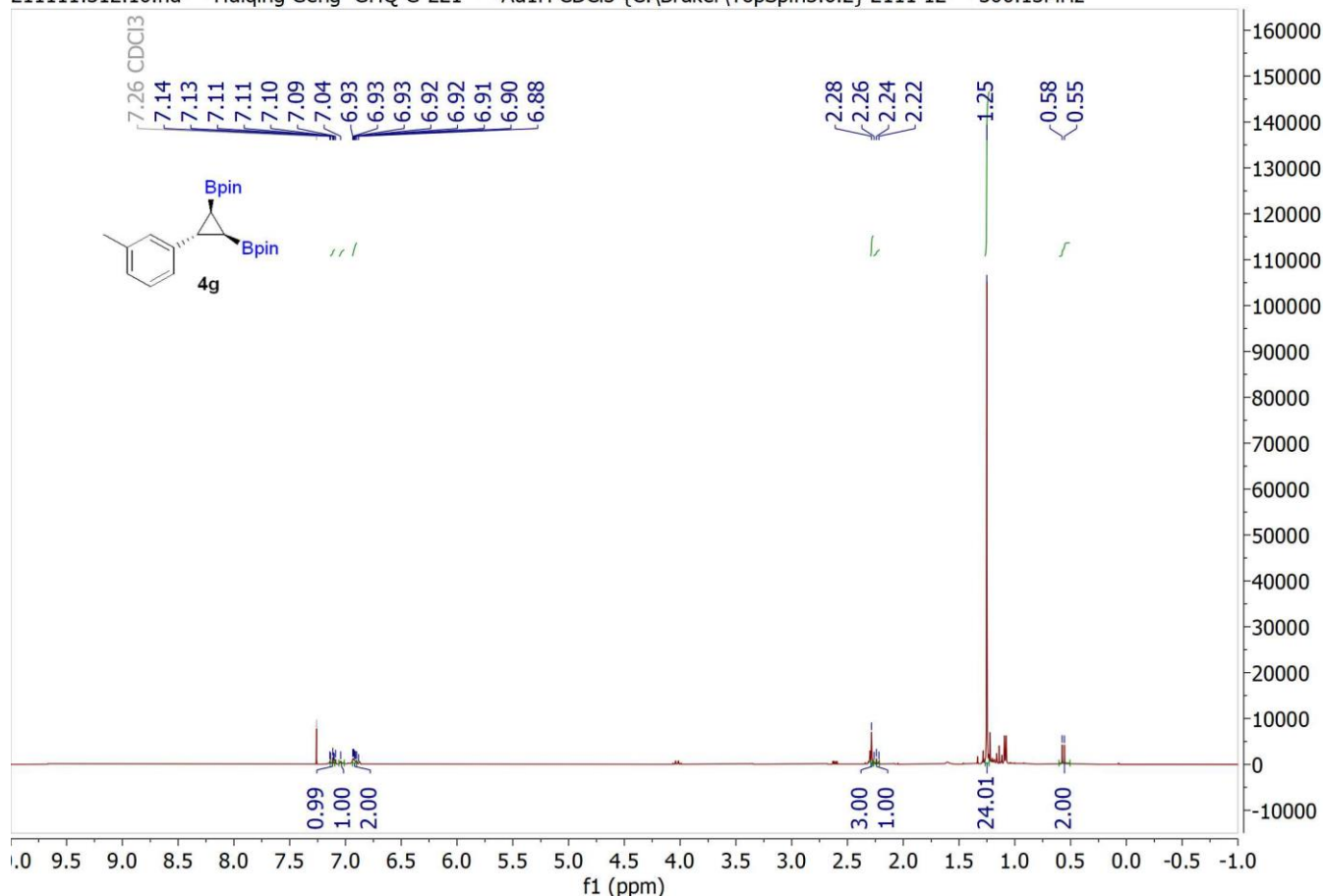


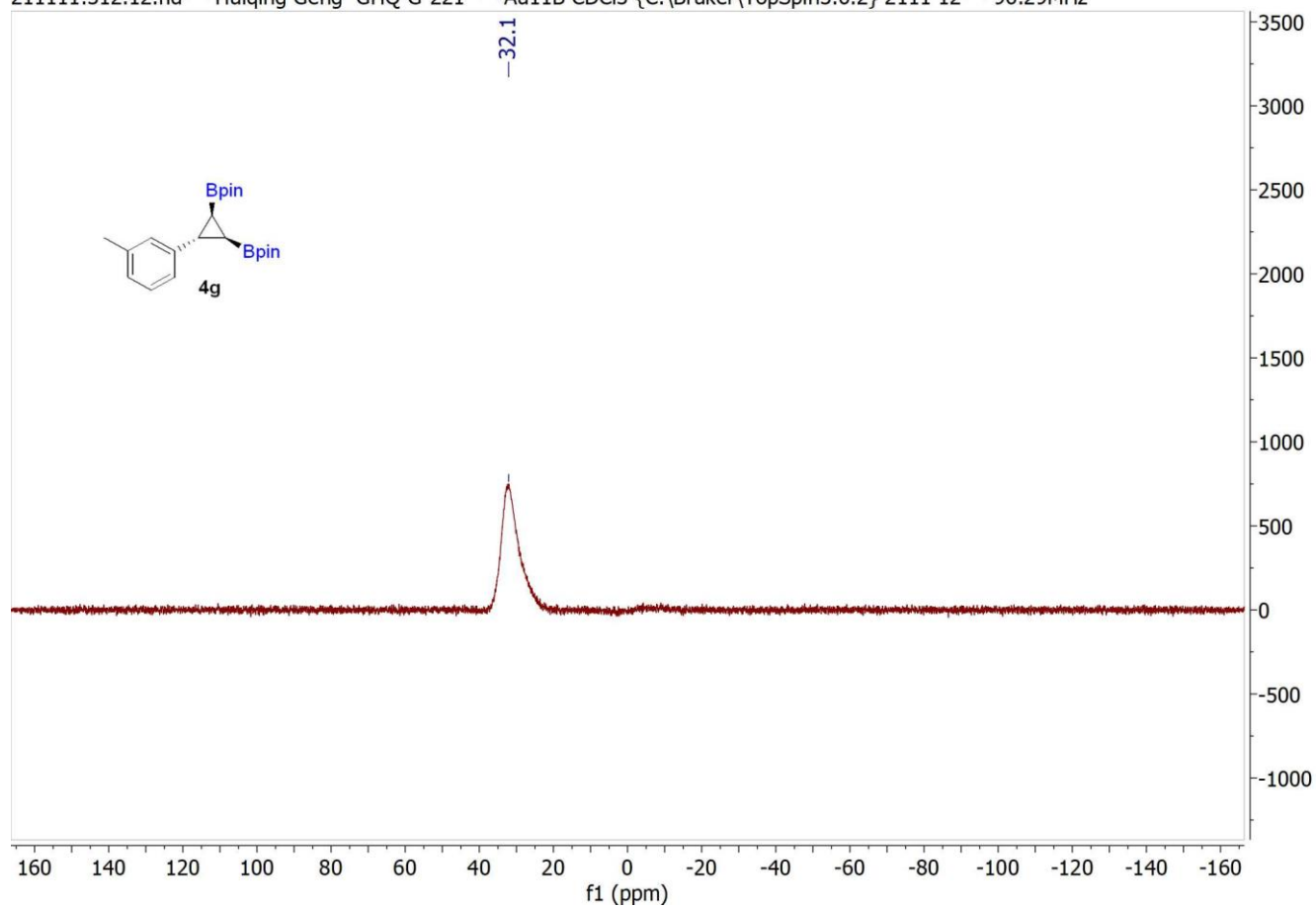


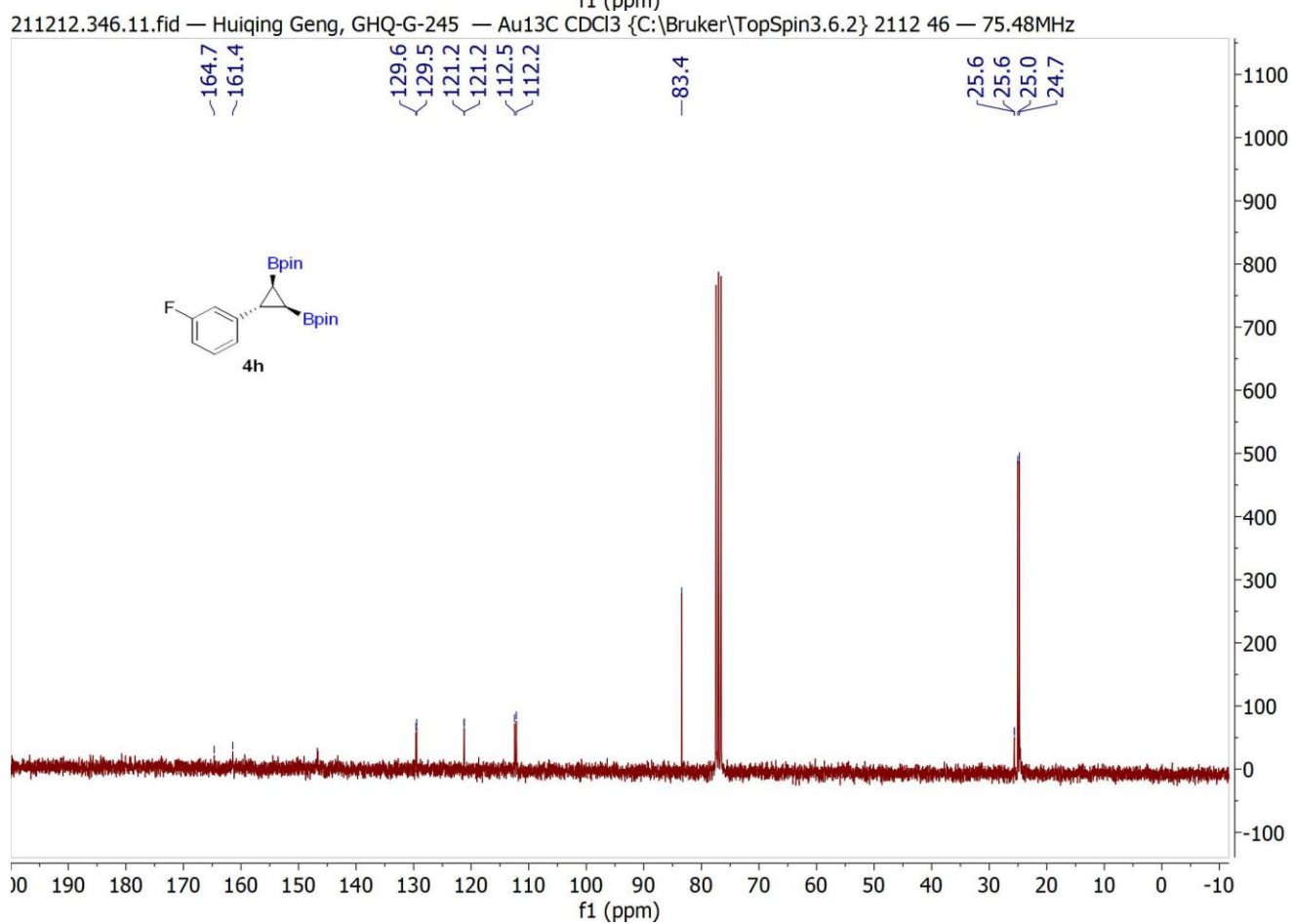
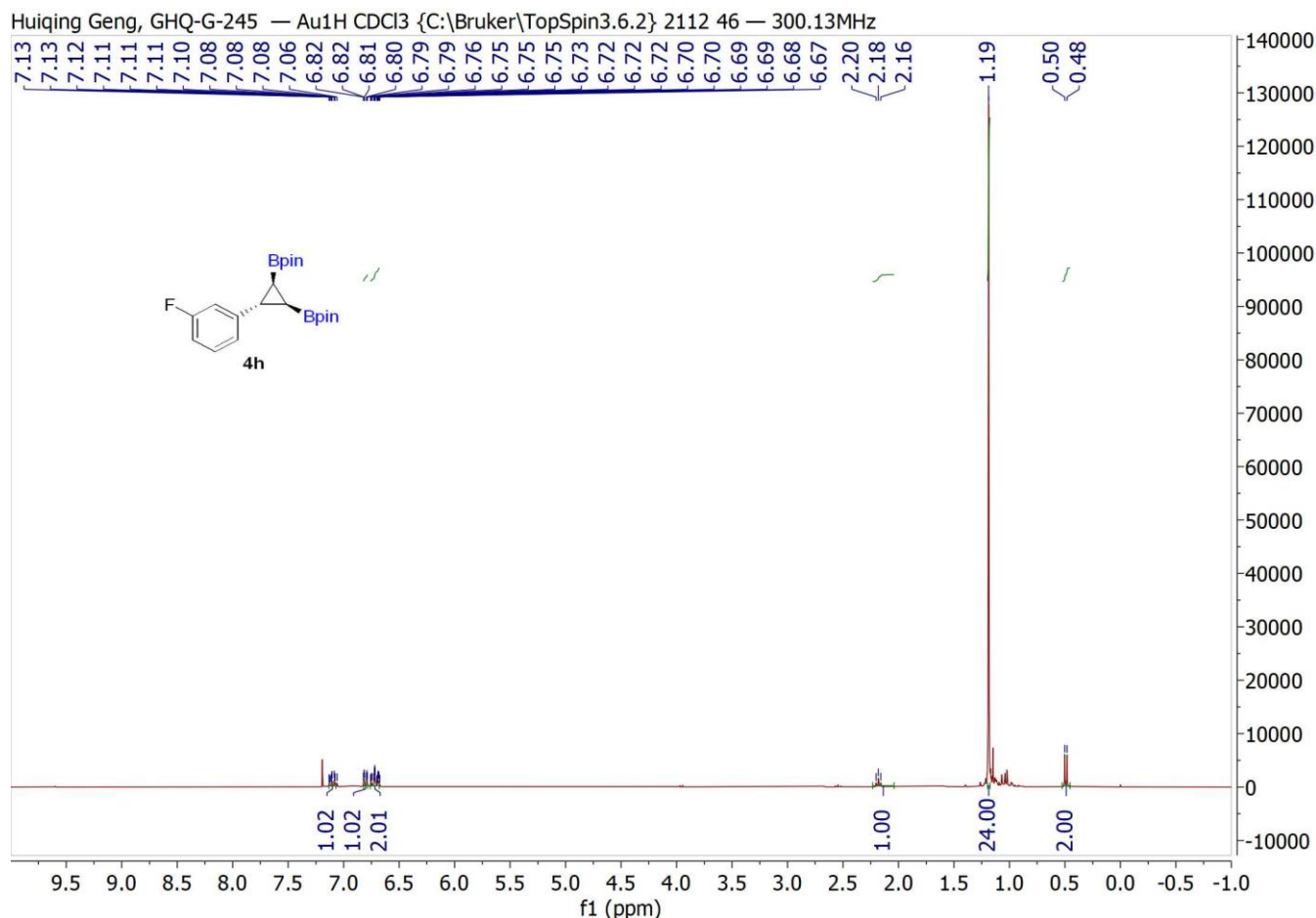


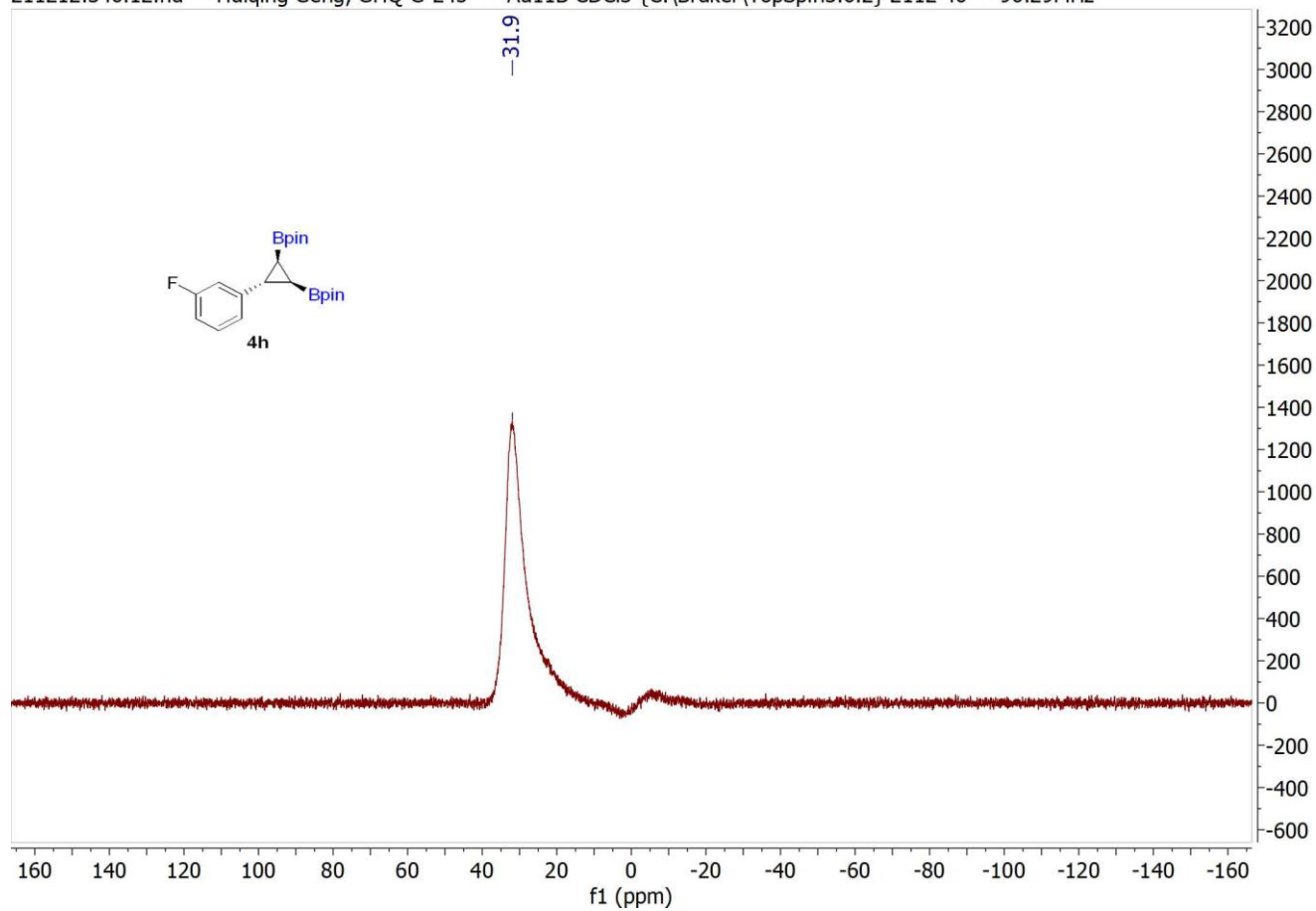




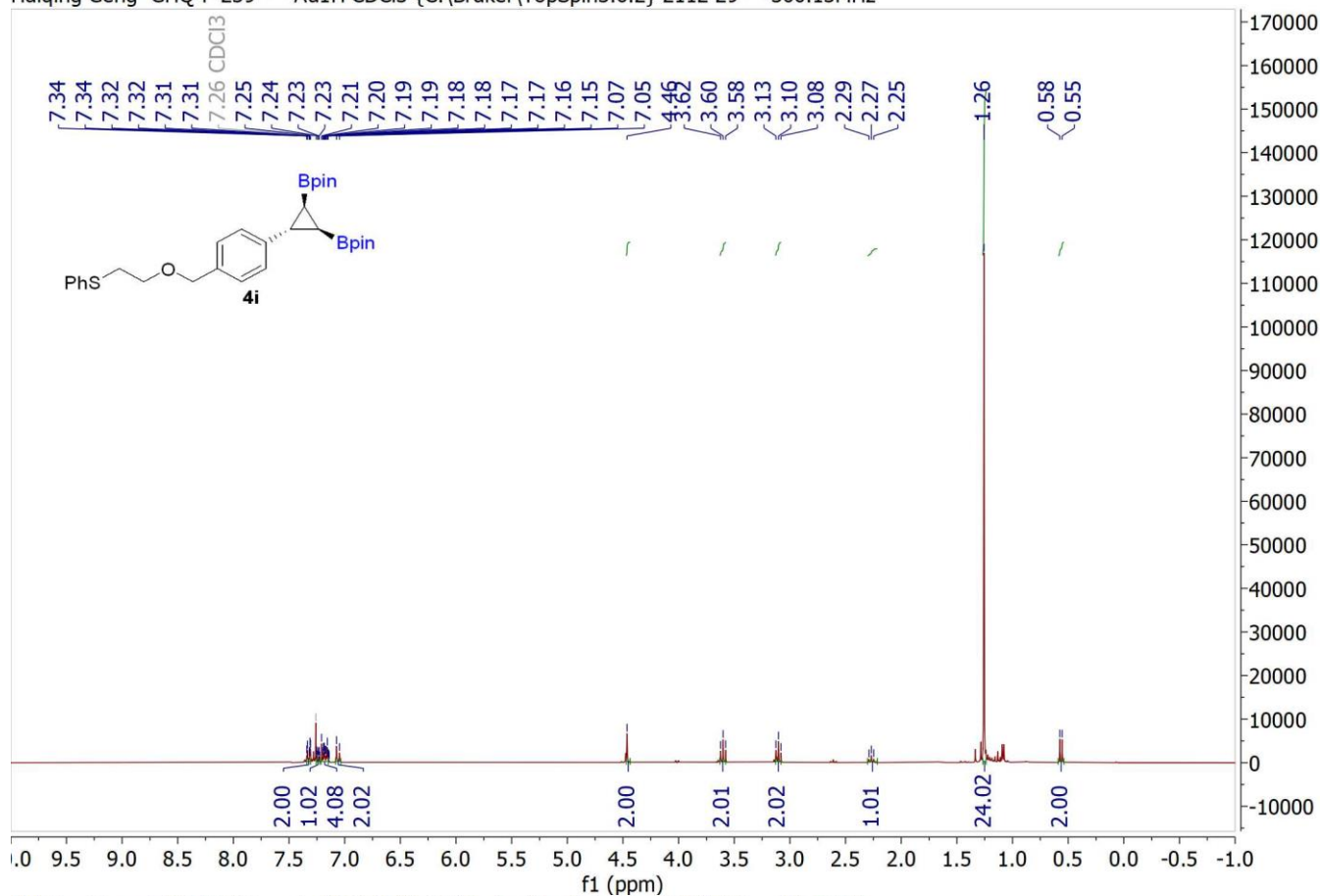




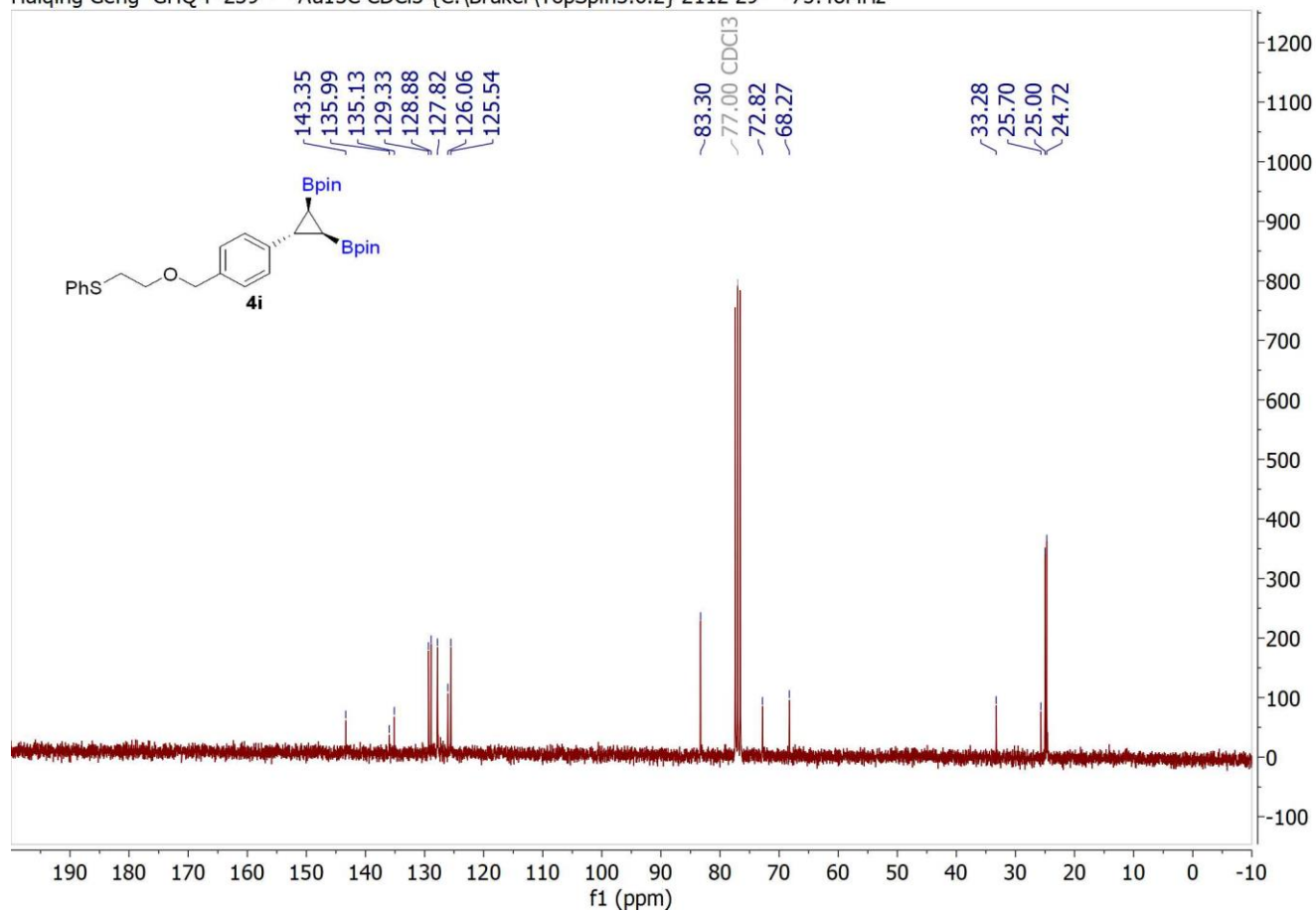


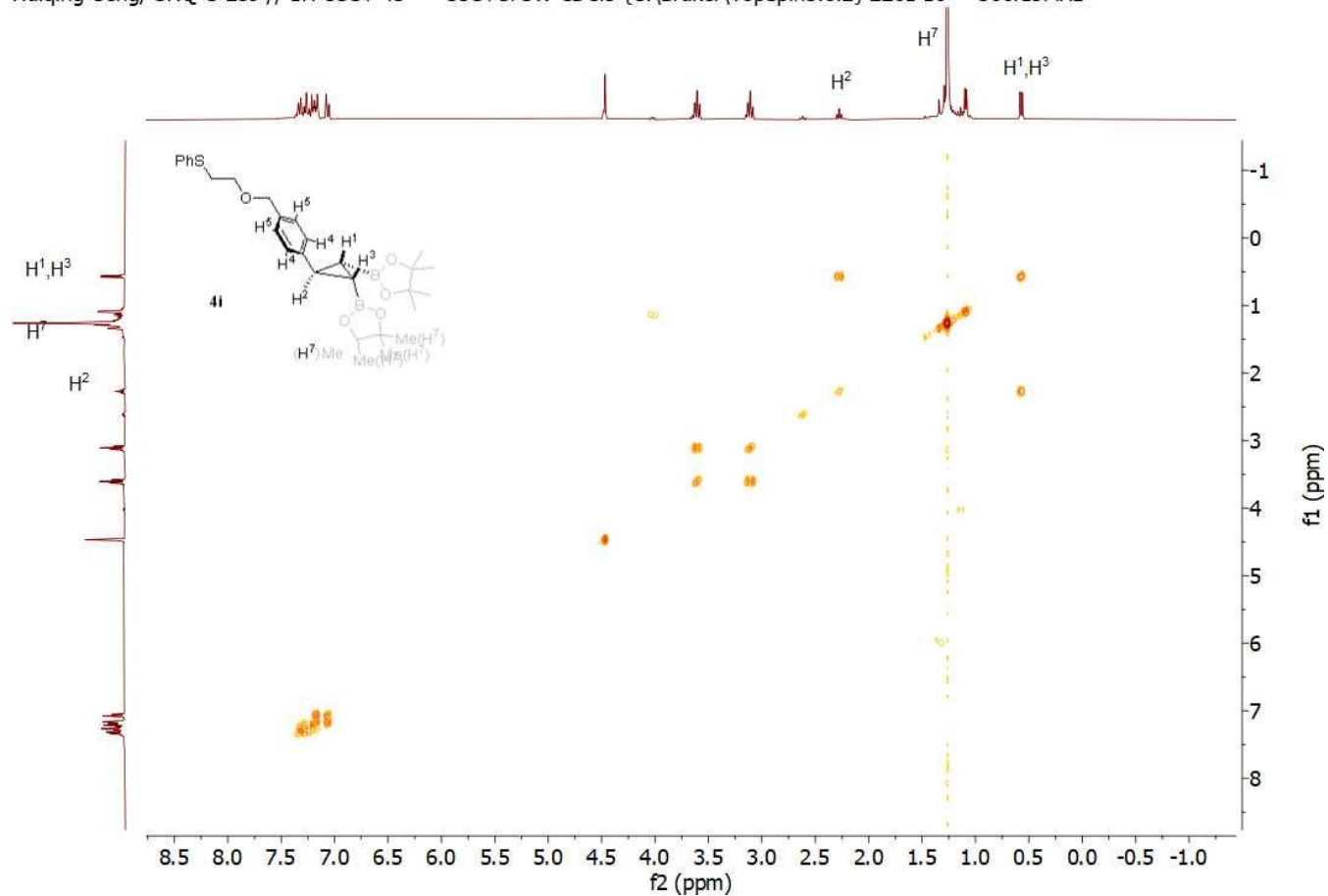
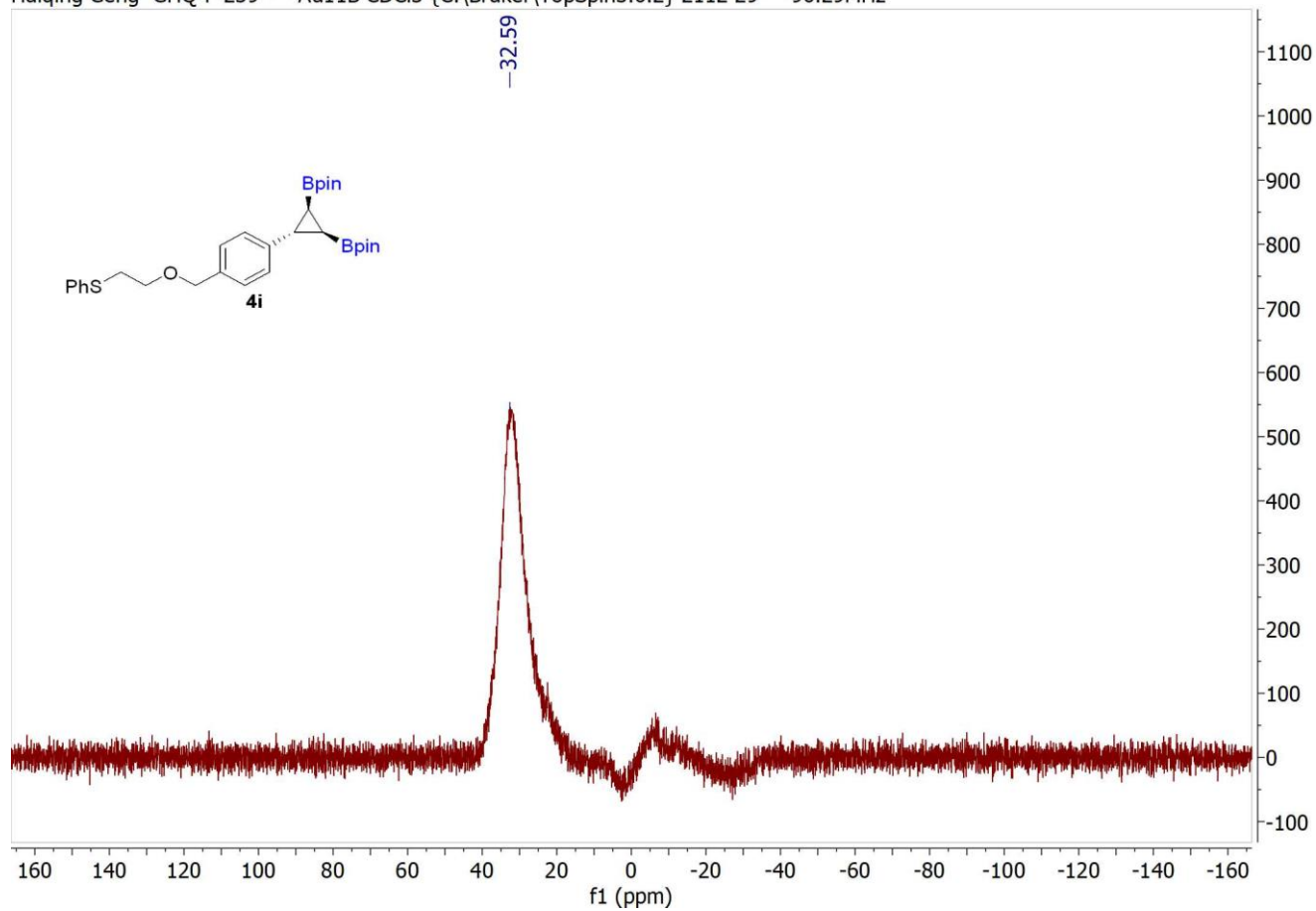


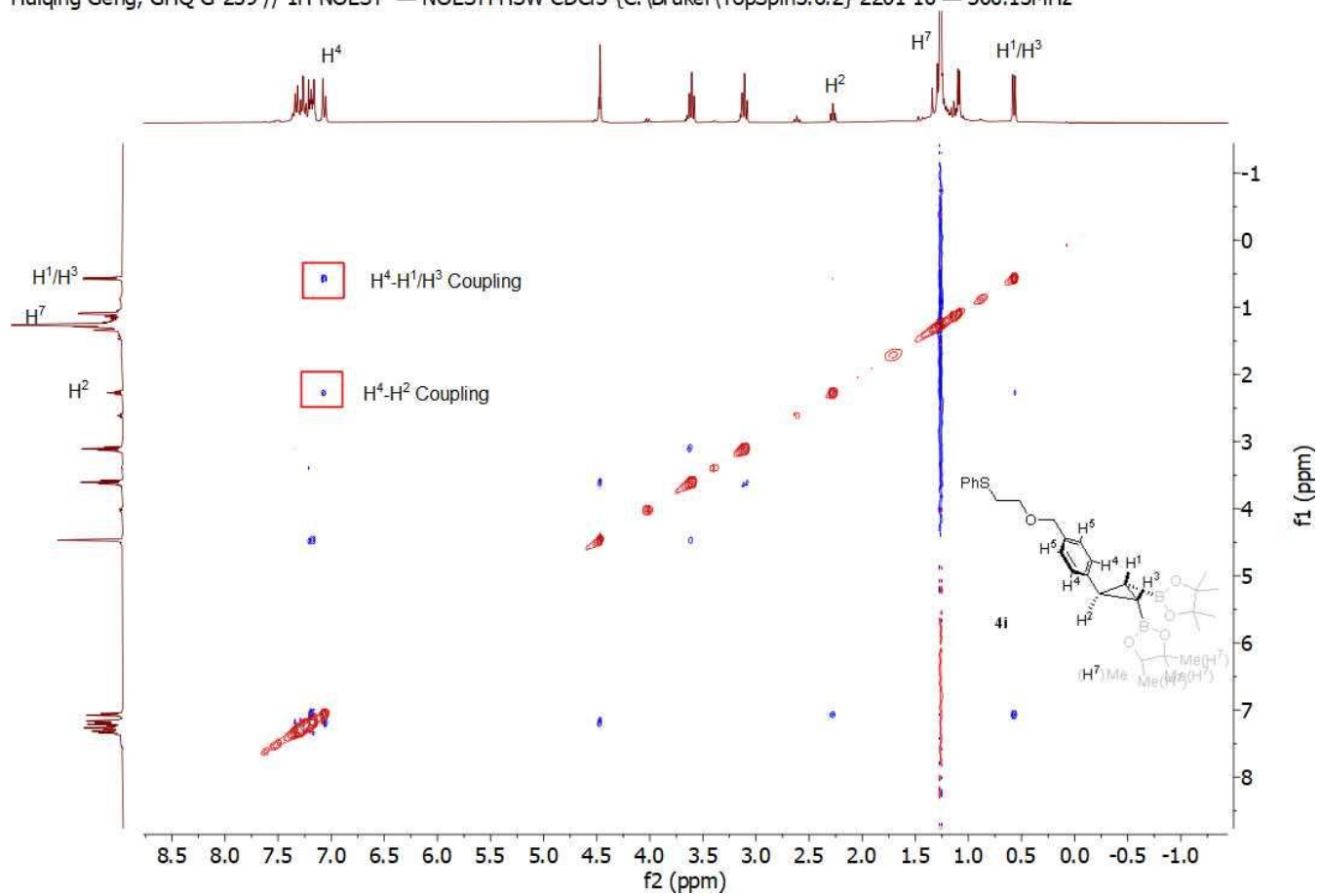
Huiqing Geng GHQ-F-239 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 29 — 300.13MHz

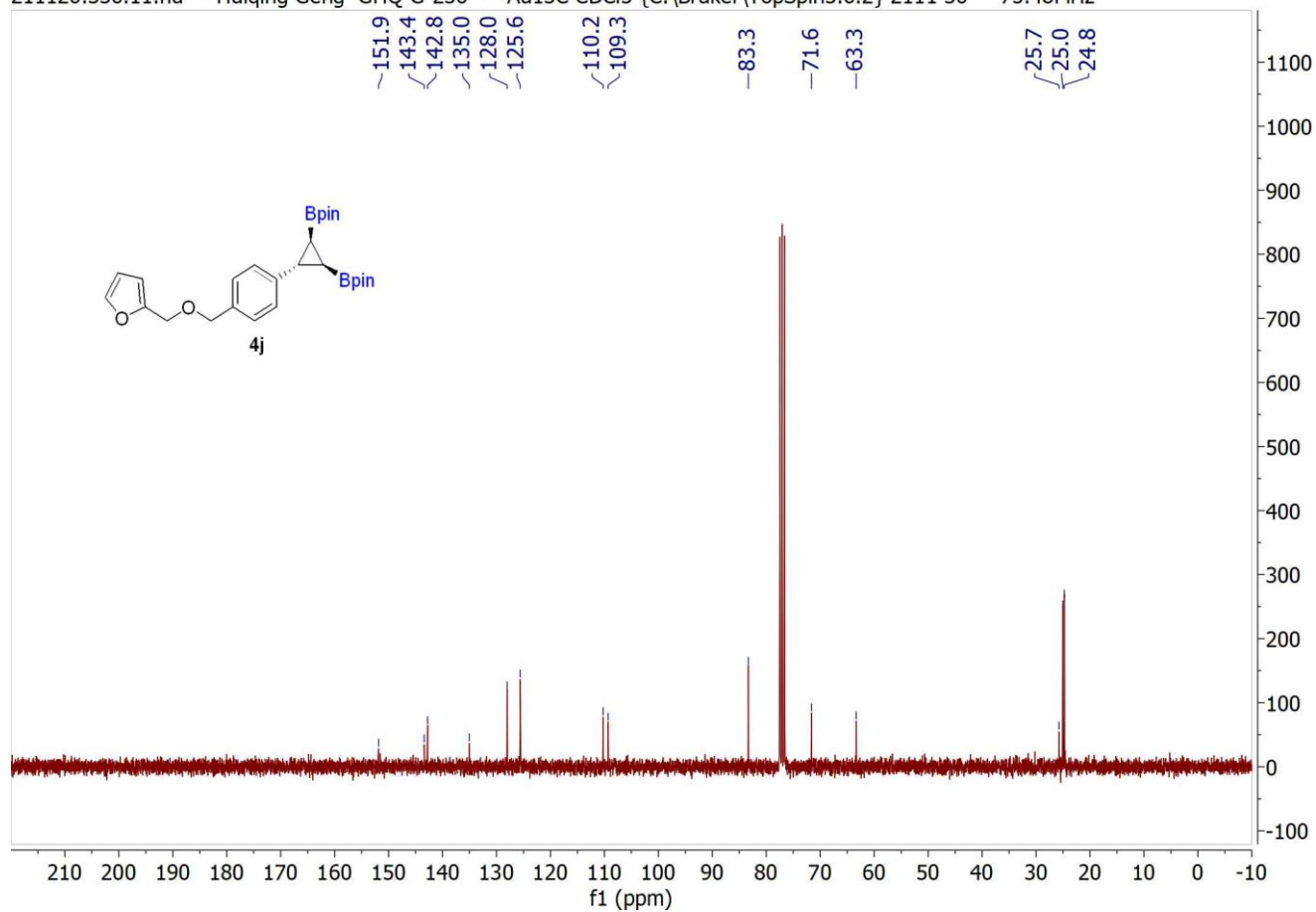
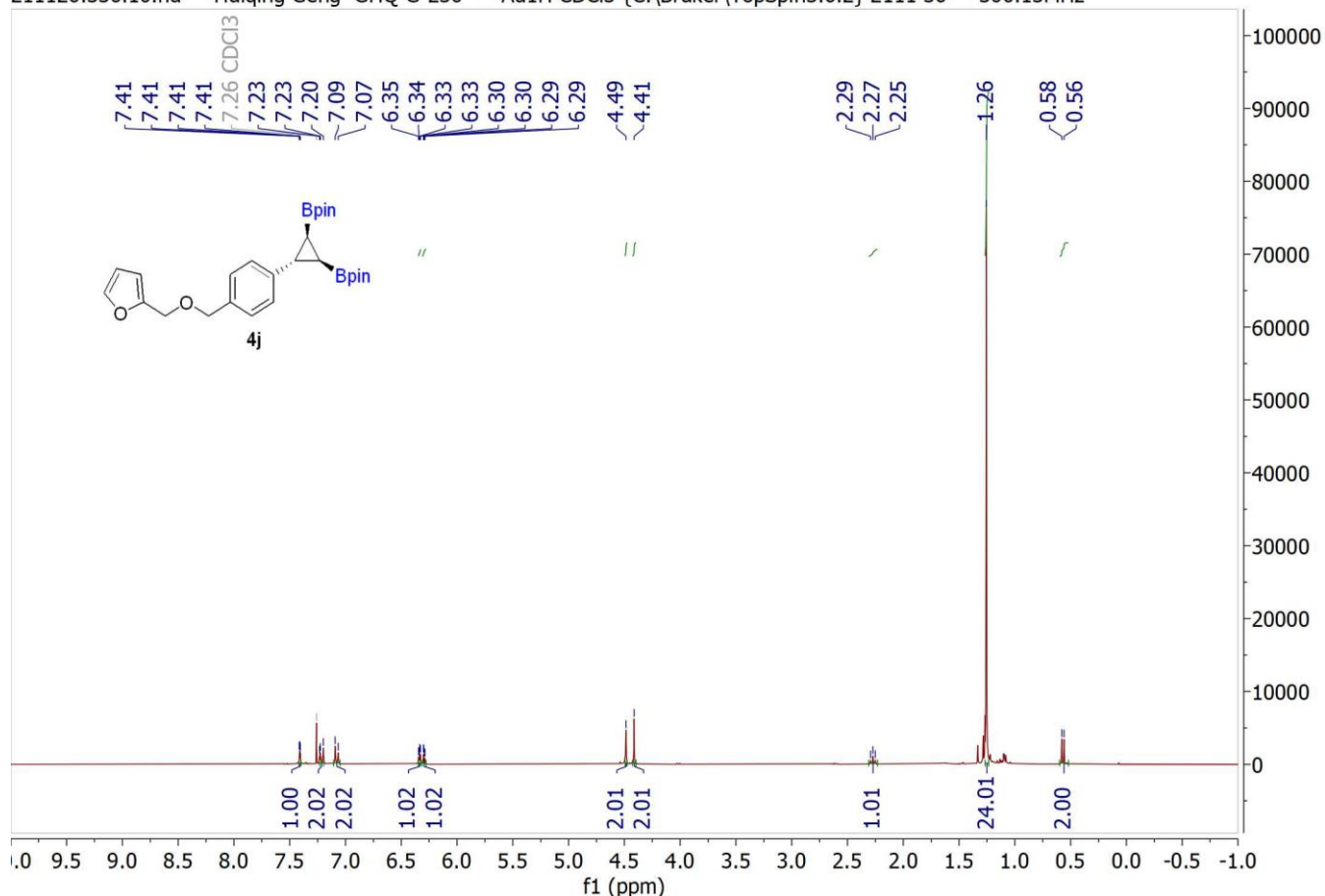


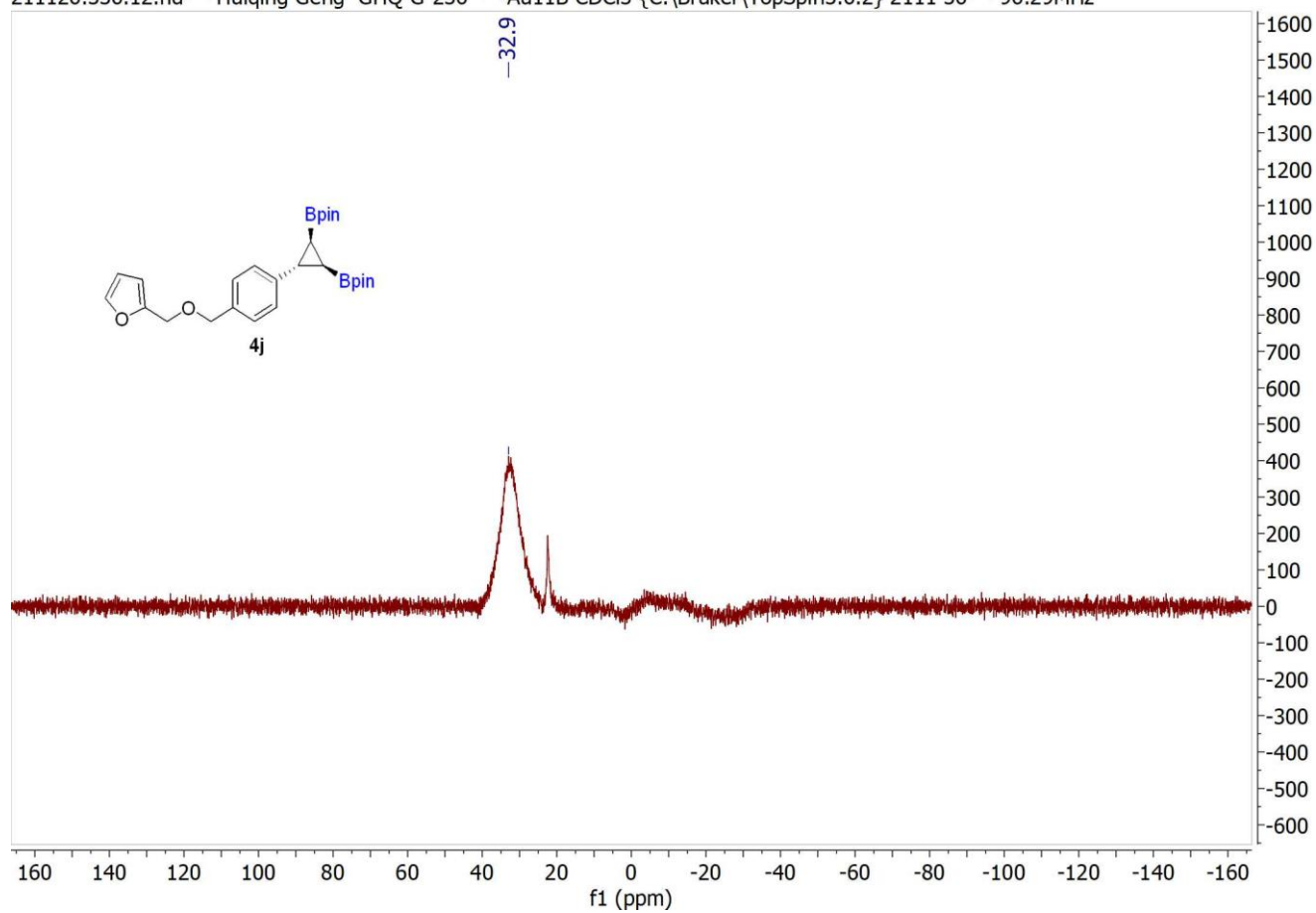
Huiqing Geng GHQ-F-239 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 29 — 75.48MHz

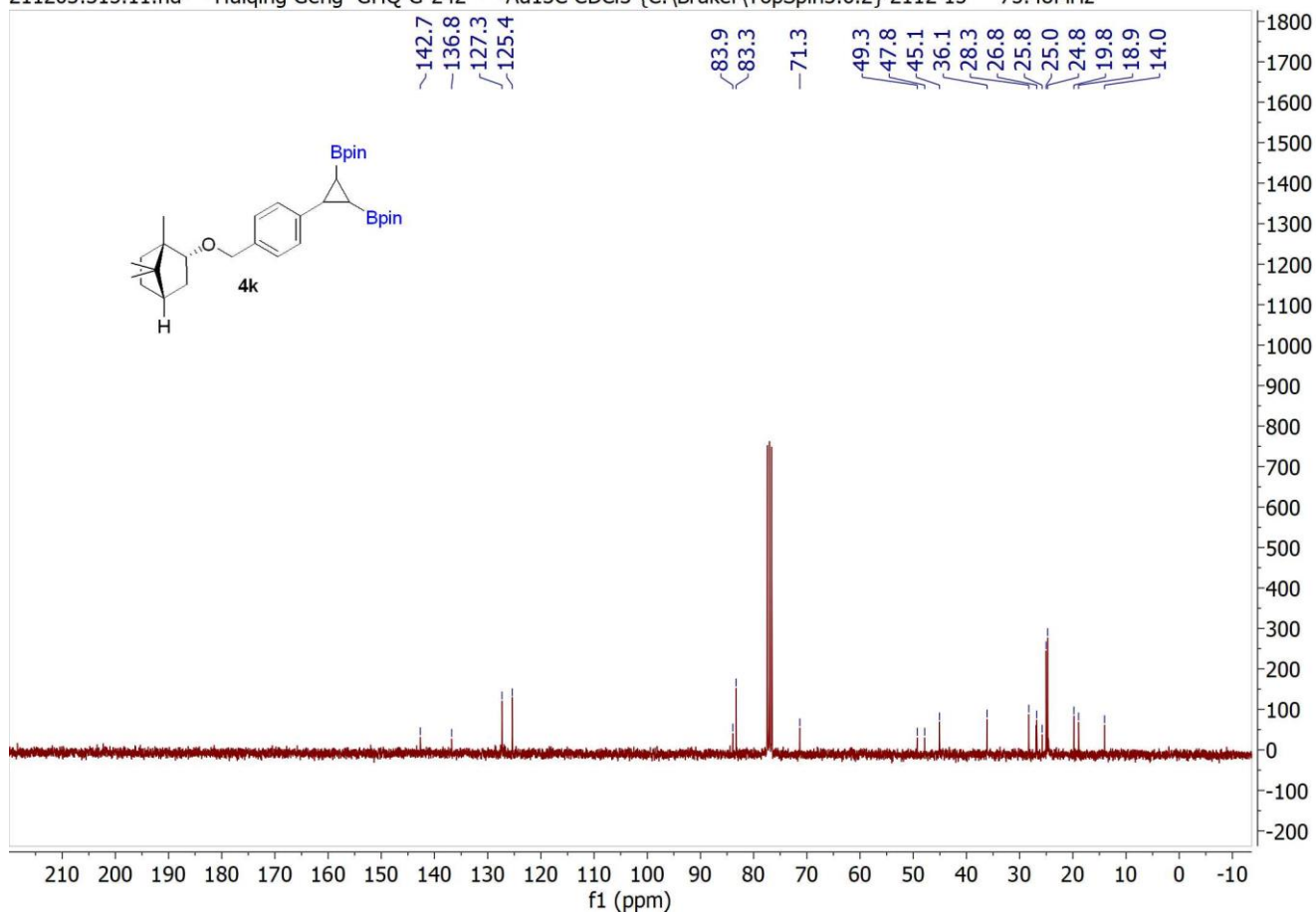
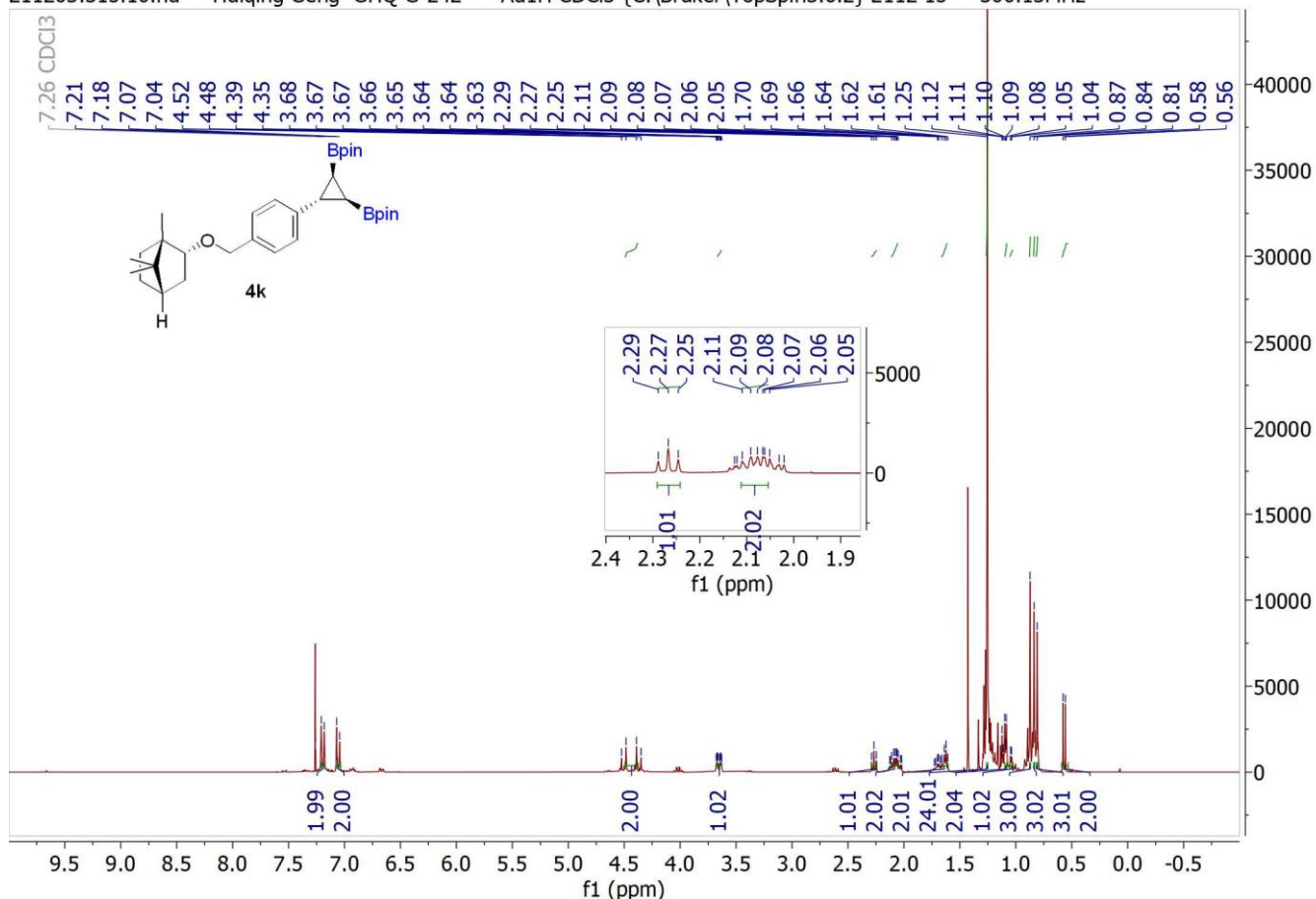


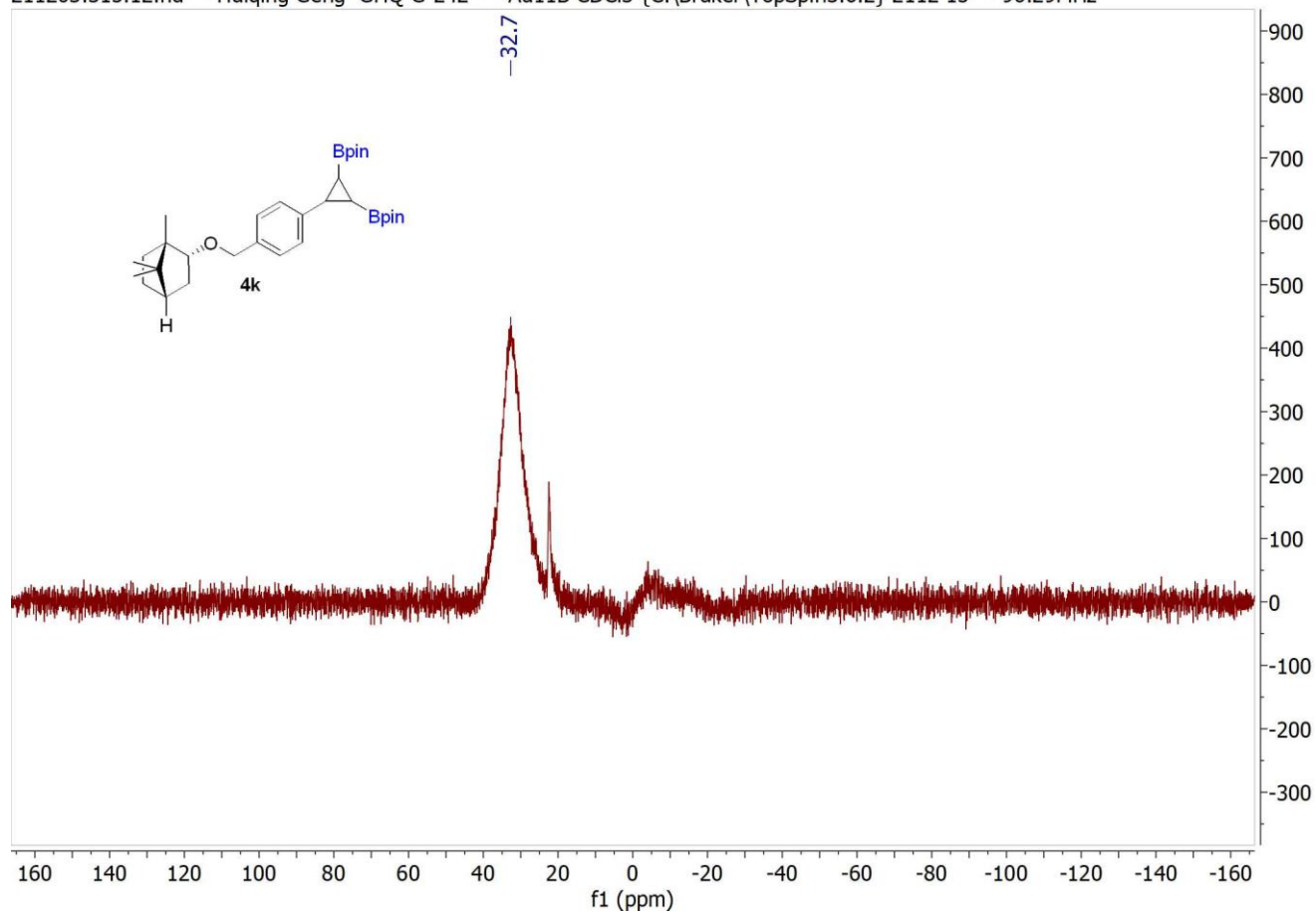


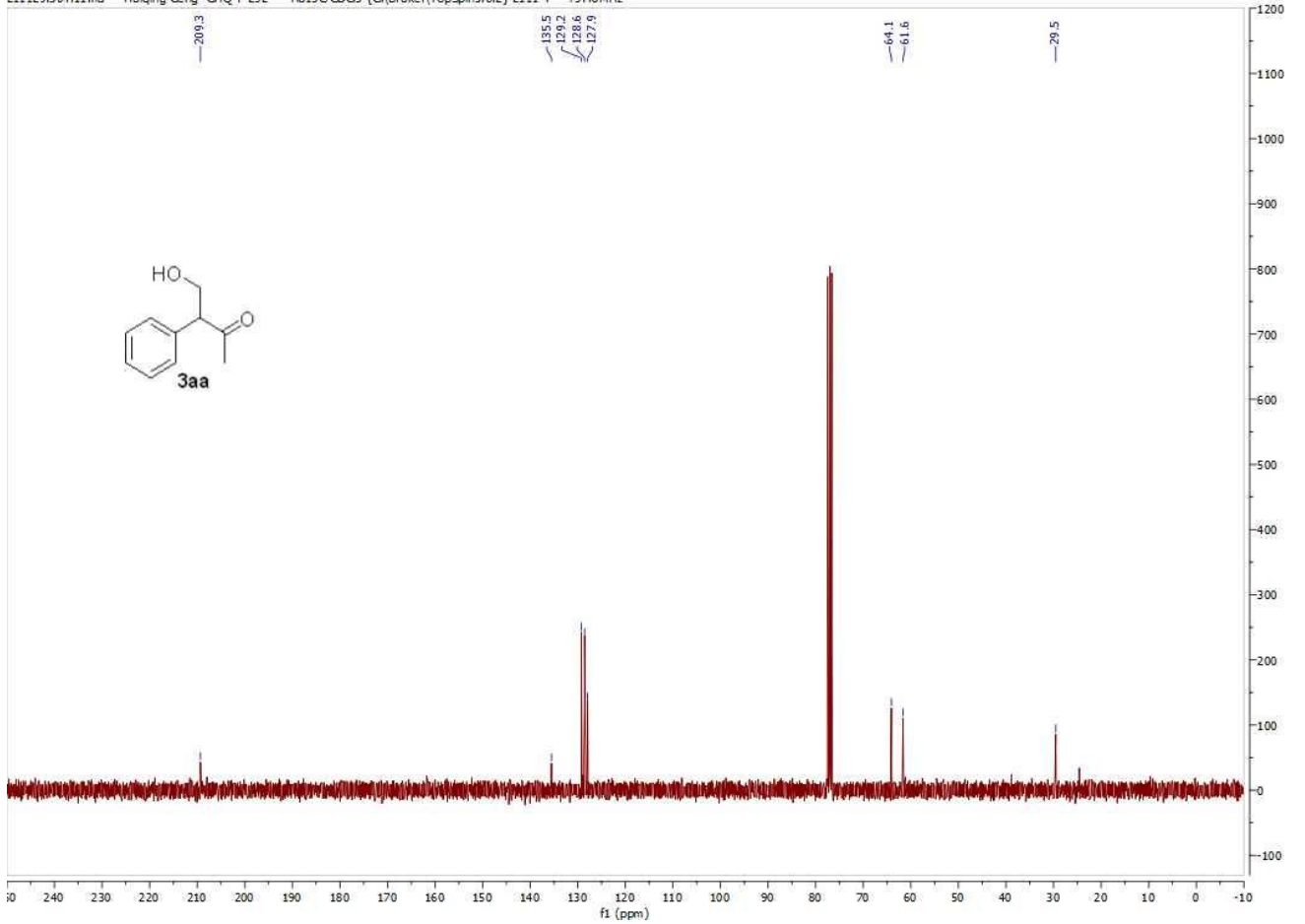
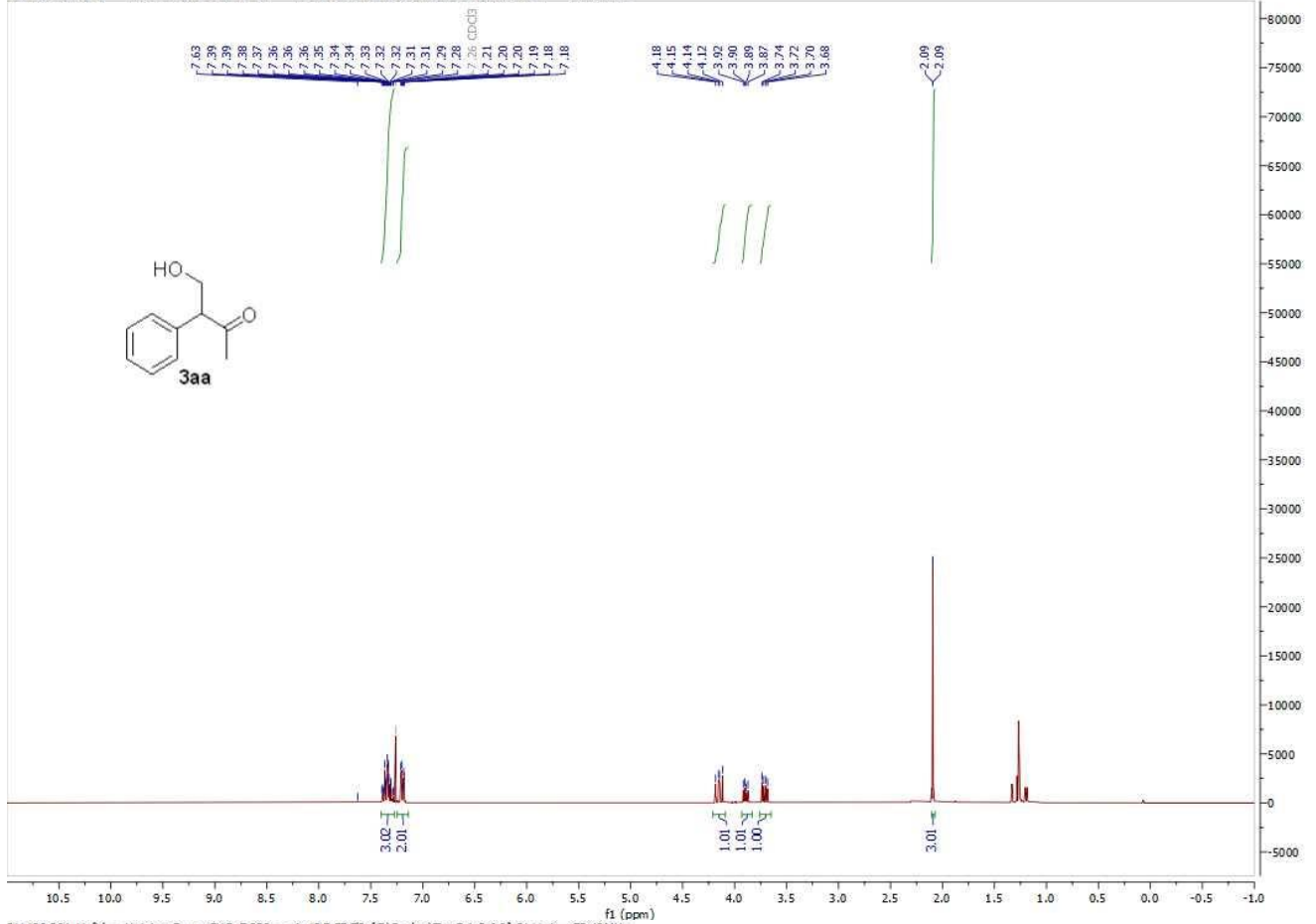




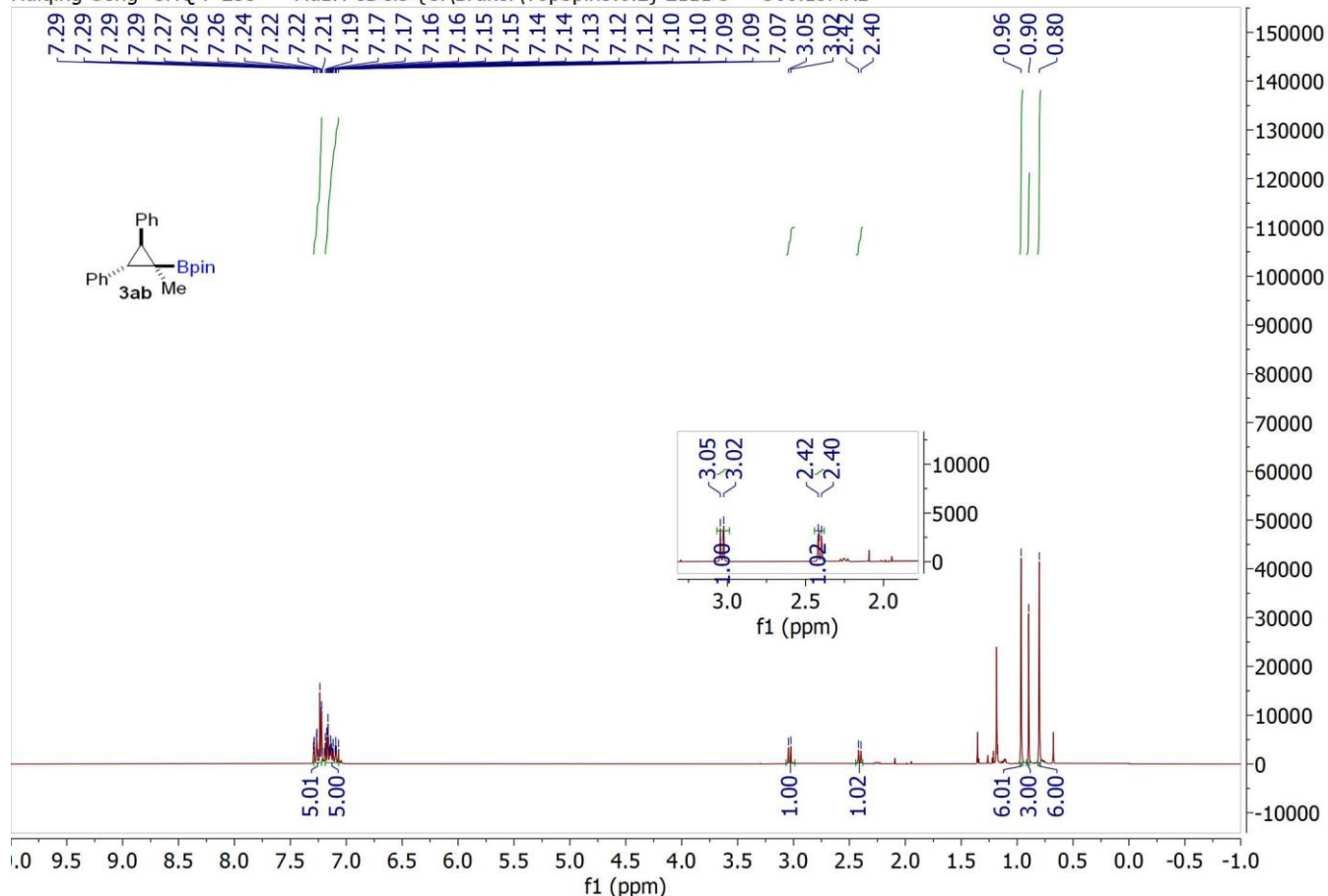




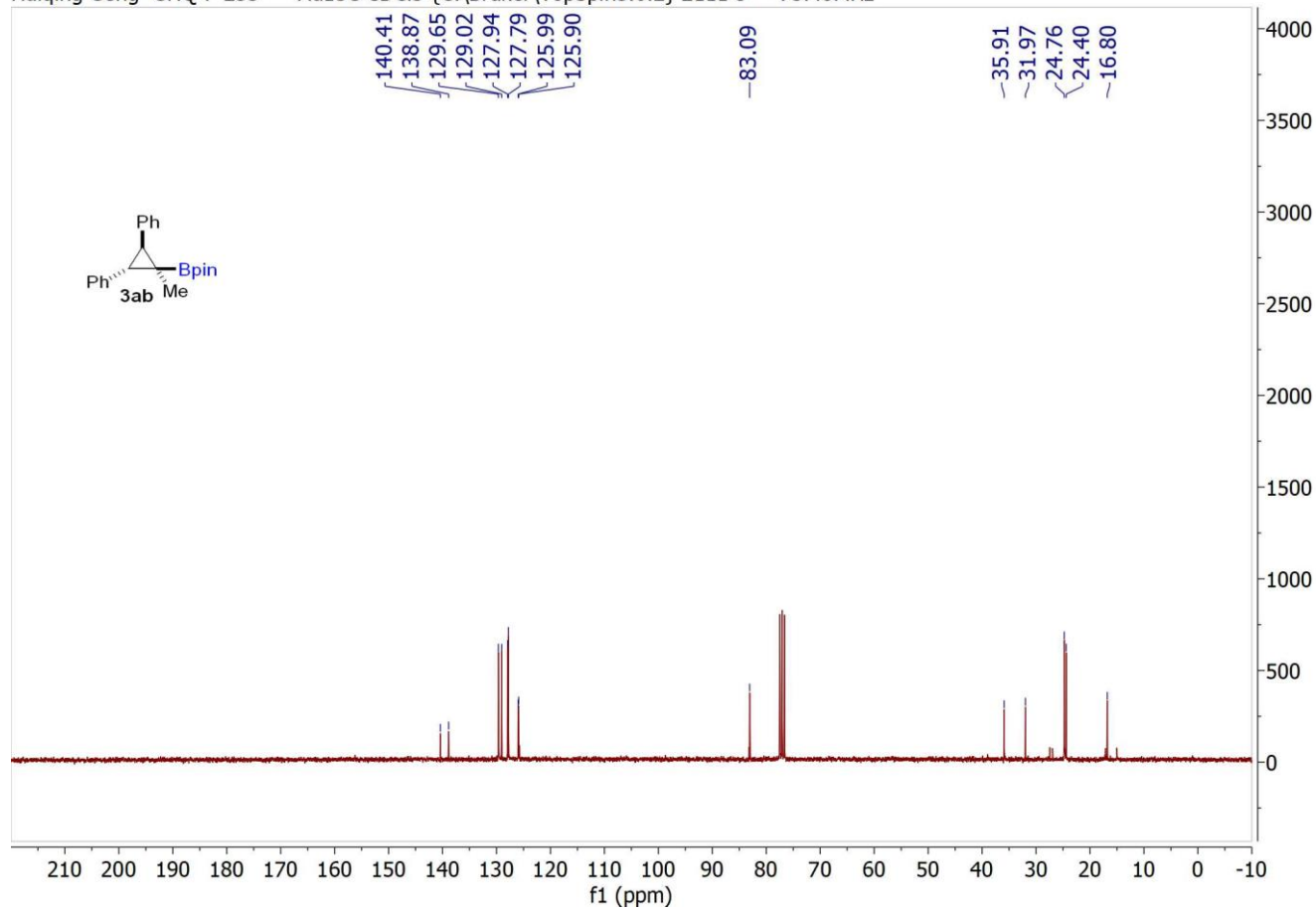


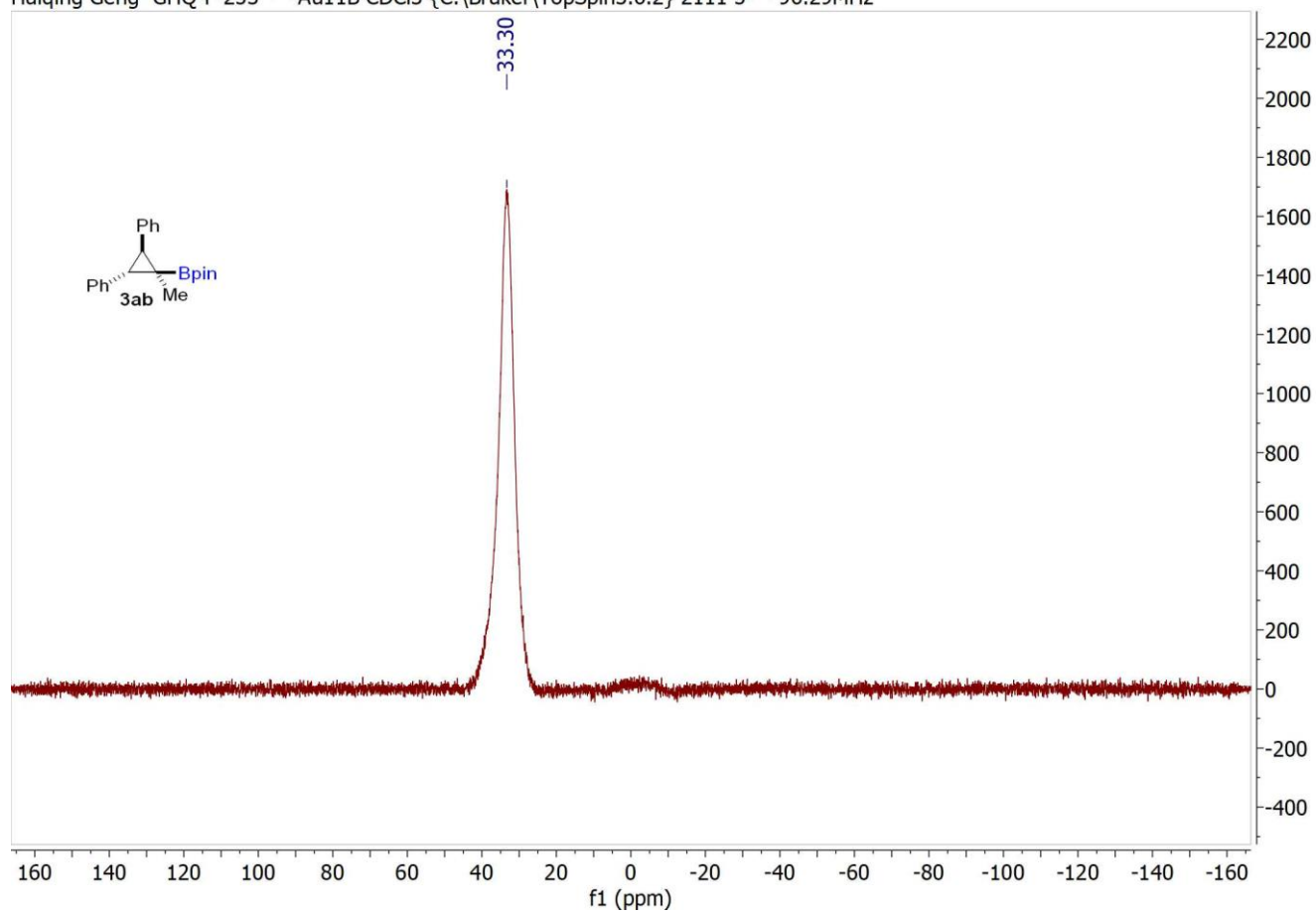


Huiqing Geng GHQ-F-253 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2111 5 — 300.13MHz

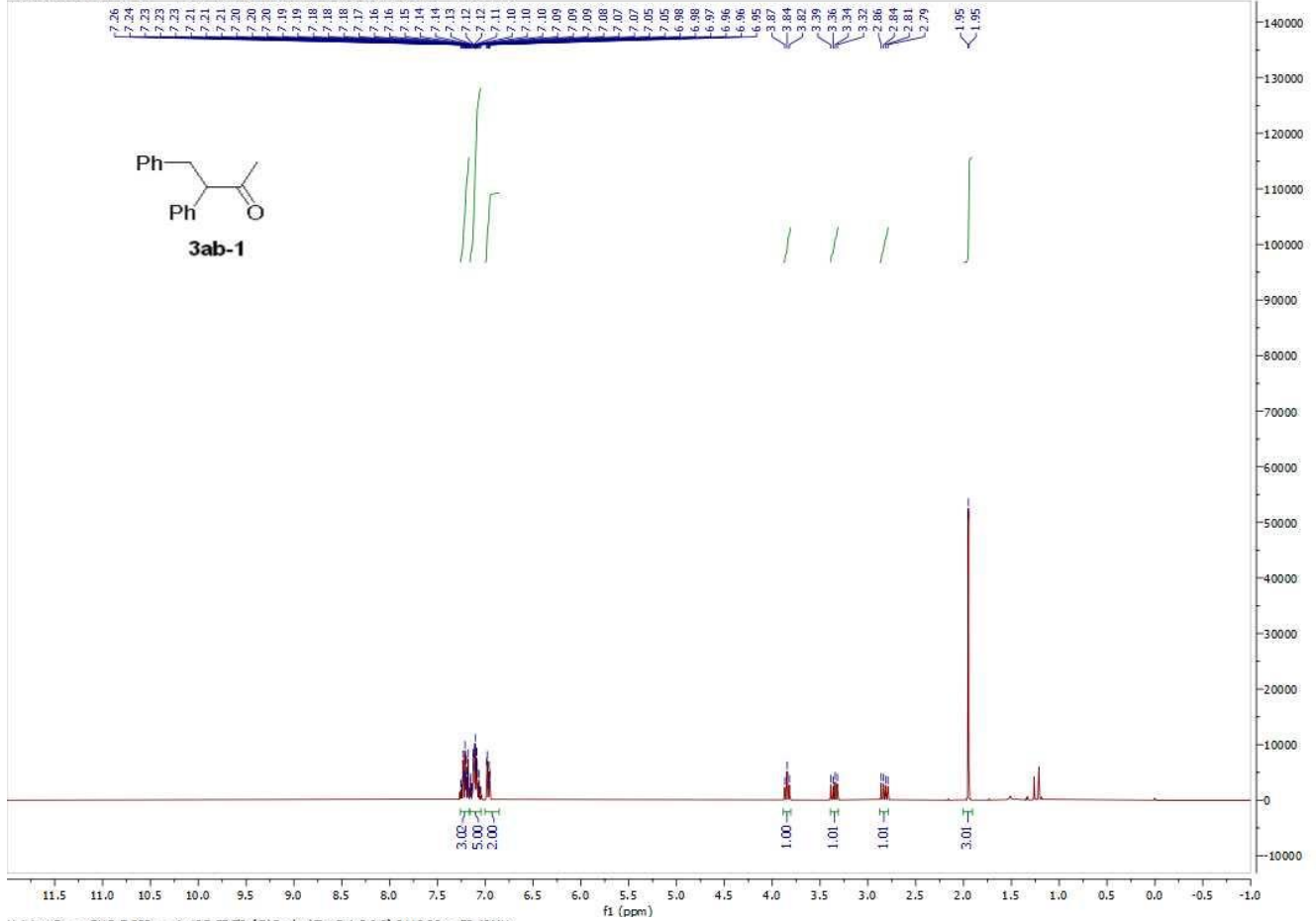


Huiqing Geng GHQ-F-253 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2111 5 — 75.48MHz

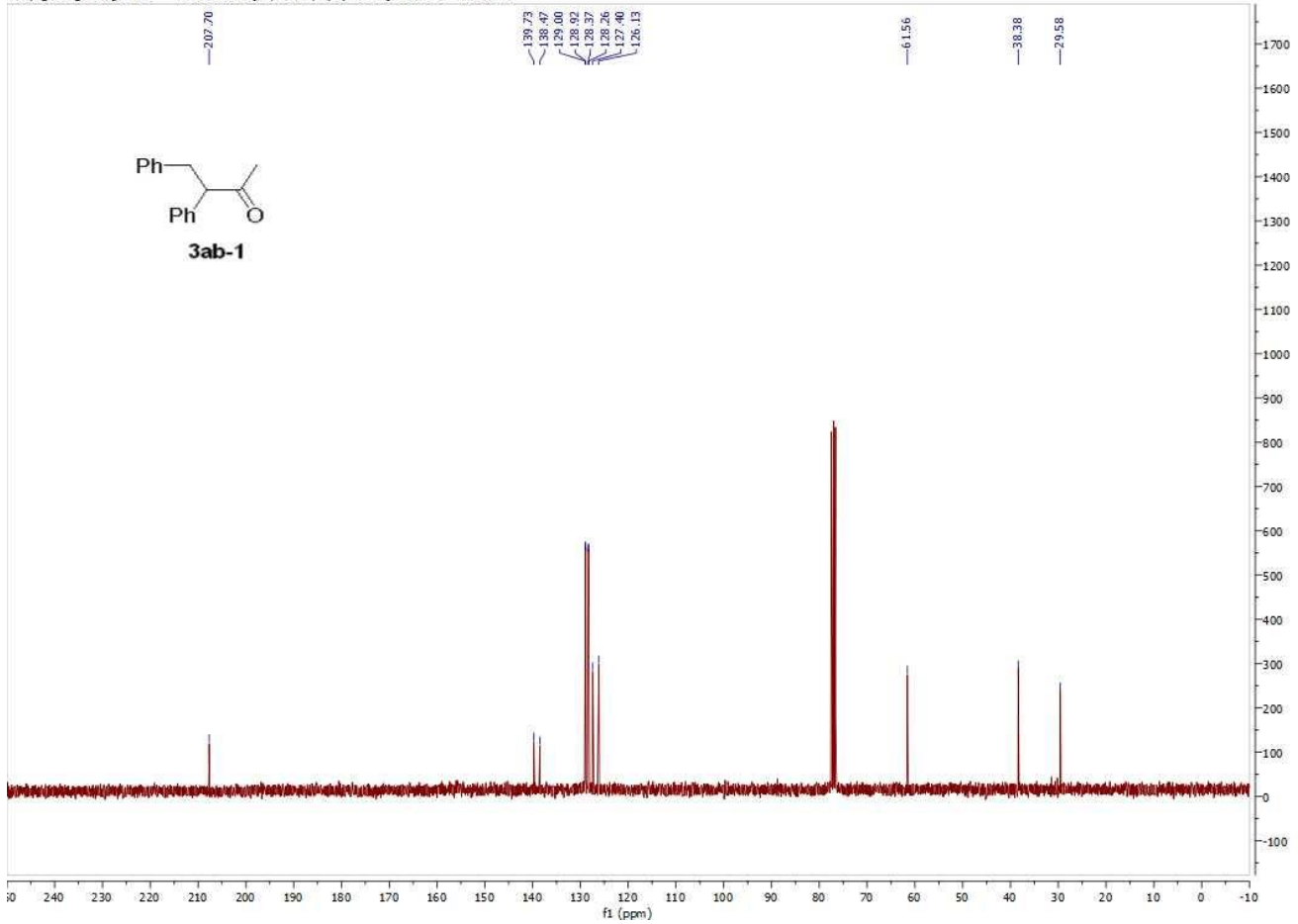




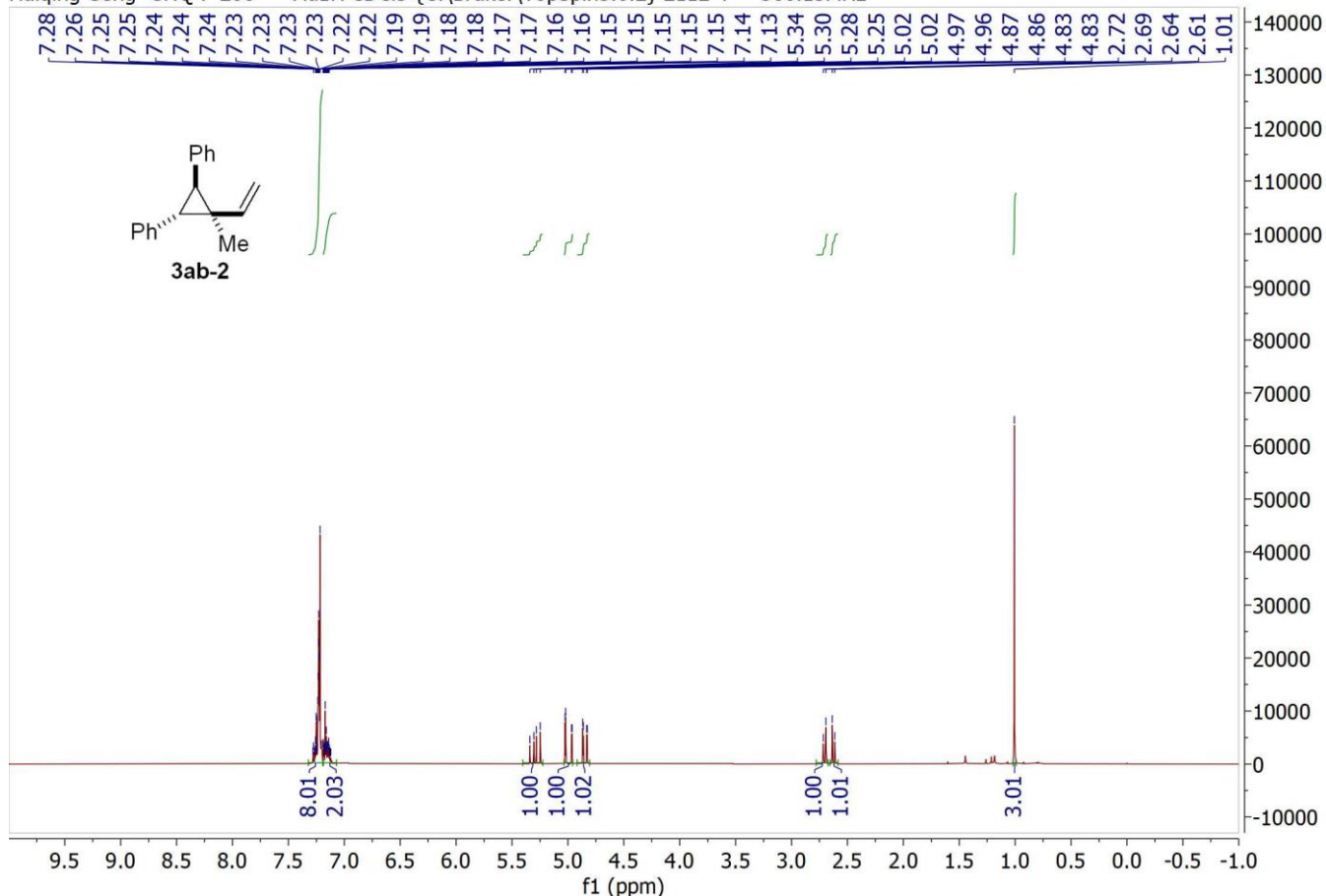
Huiqing Geng GHQ-F-255 — Au1H CDCl3 (C)Bruker(TopSpin3.6.2) 2112 28 — 300.13MHz



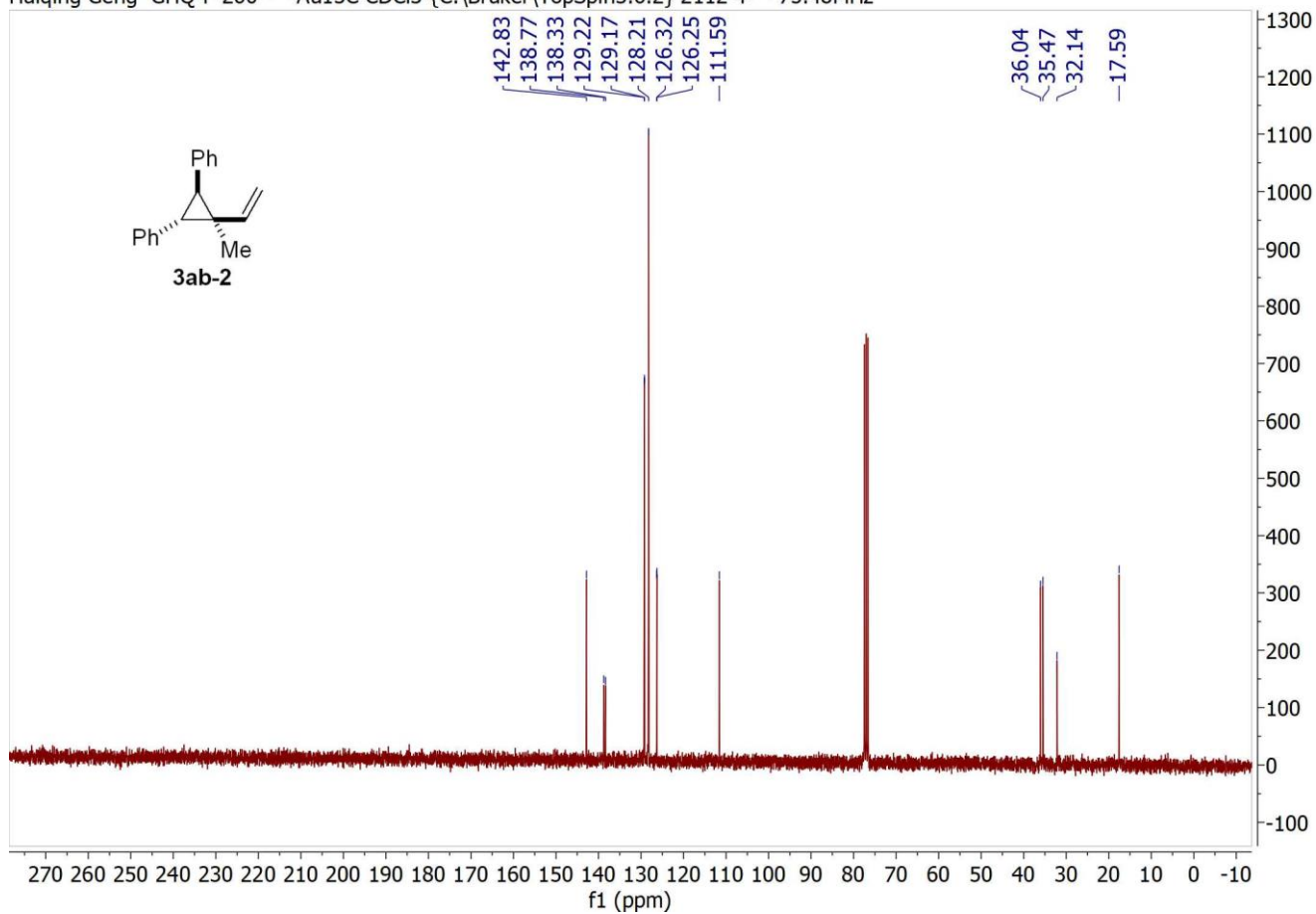
Huiqing Geng GHQ-F-255 — Au13C CDCl3 (C)Bruker(TopSpin3.6.2) 2112 28 — 75.48MHz



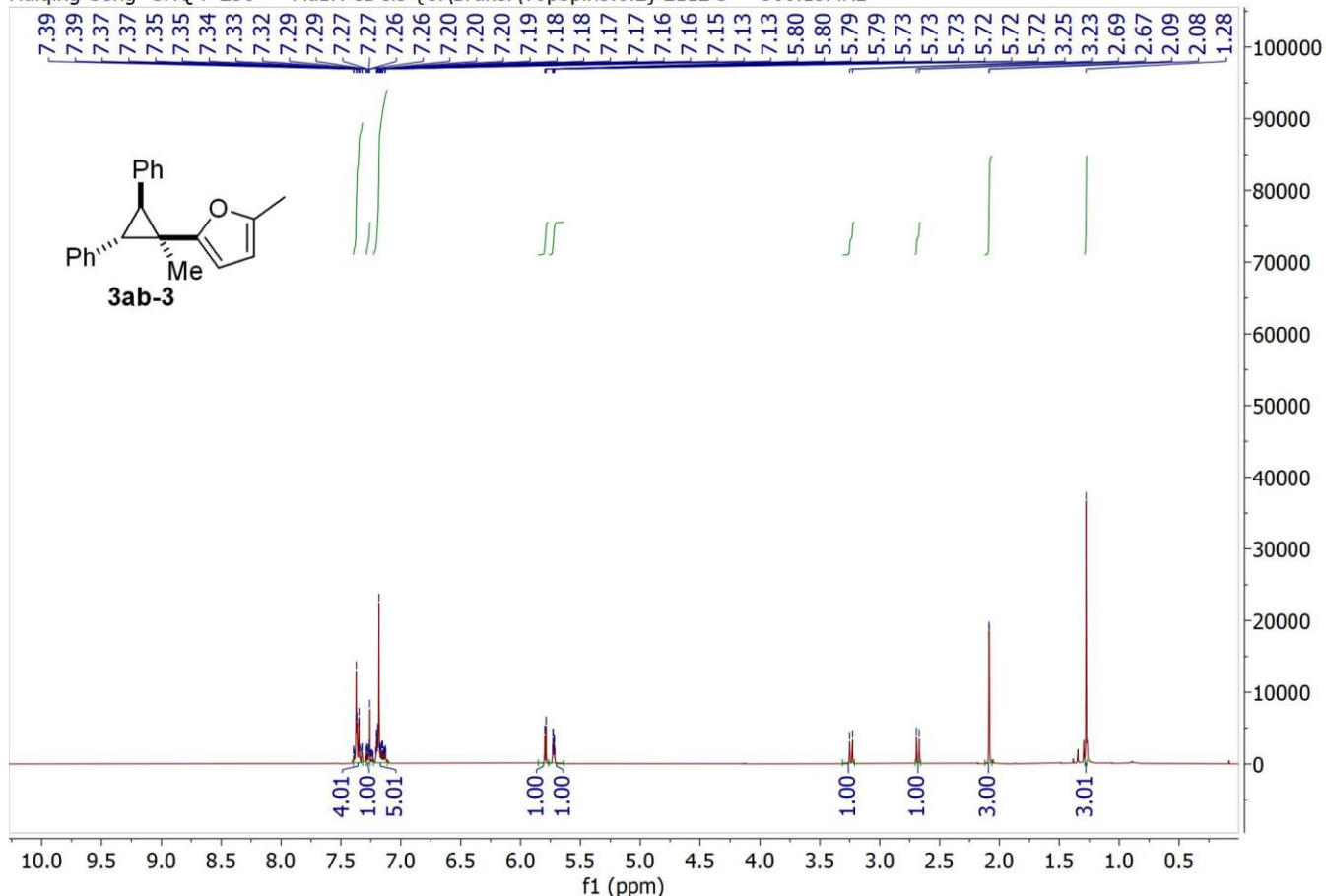
Huiqing Geng GHQ-F-260 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 4 — 300.13MHz



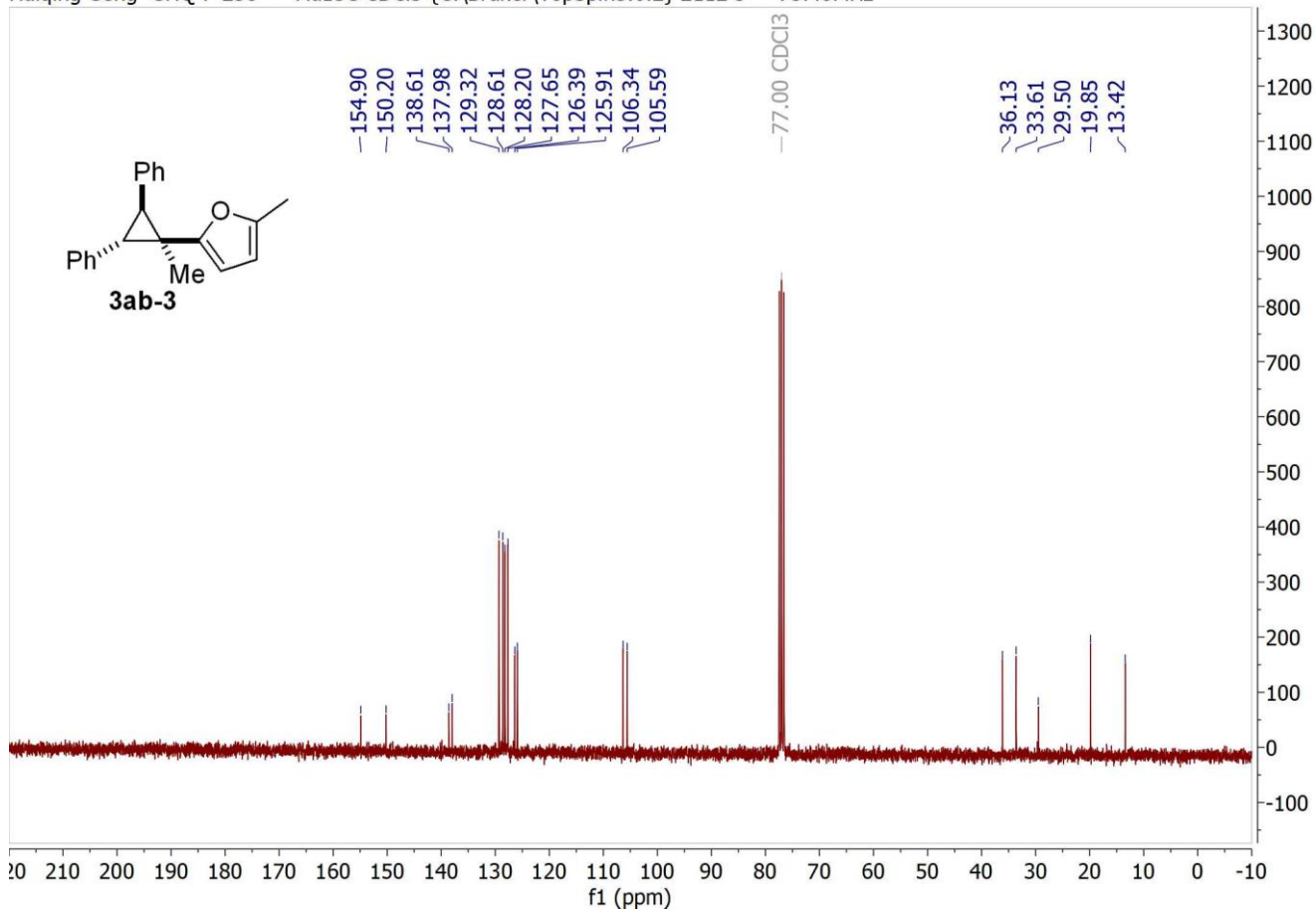
Huiqing Geng GHQ-F-260 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 4 — 75.48MHz



Huiqing Geng GHQ-F-258 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 3 — 300.13MHz

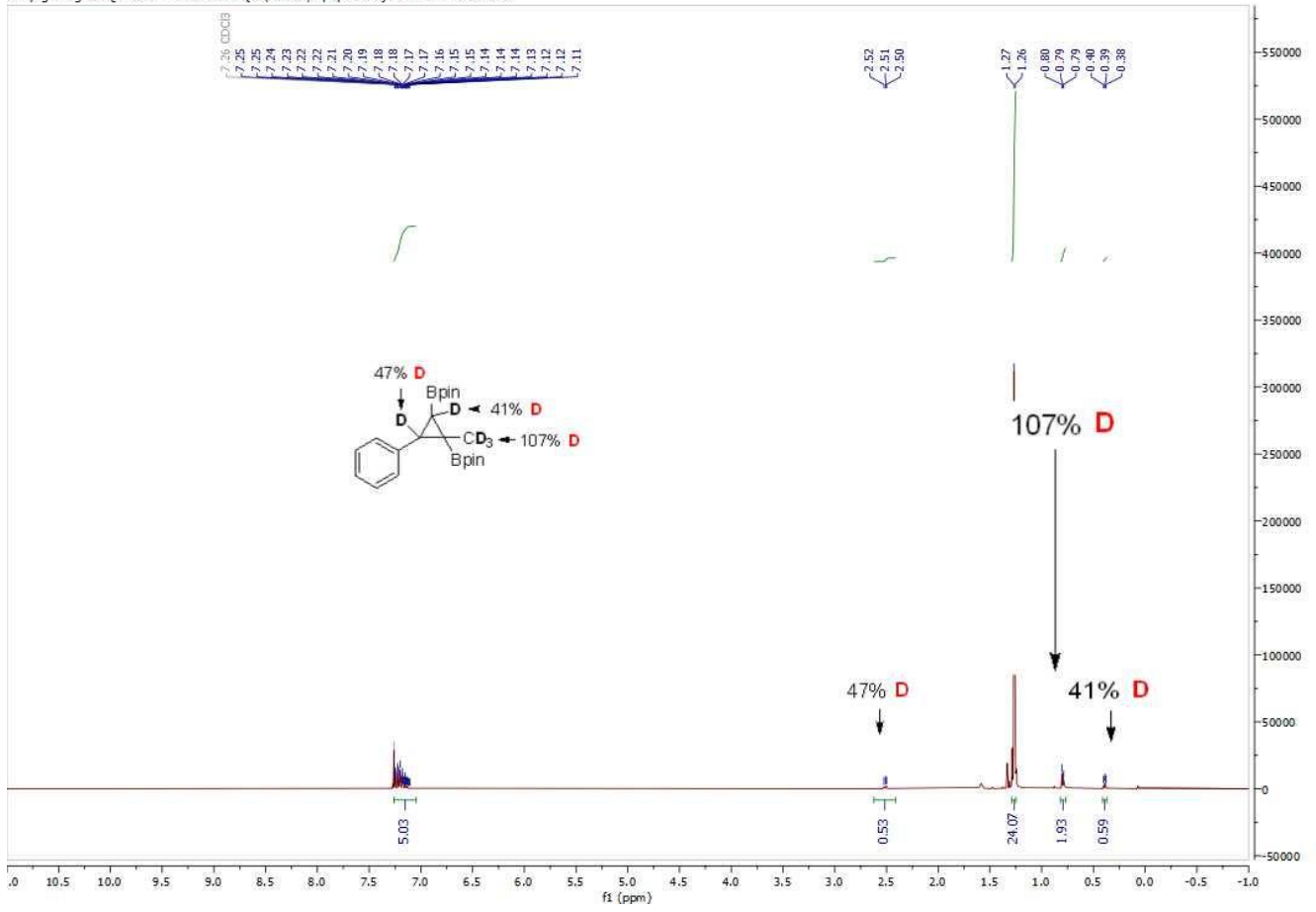


Huiqing Geng GHQ-F-258 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2112 3 — 75.48MHz



CH₄

Huiqing Geng GHQ-F-221 — Au1H CDCl₃ (C:\Bruker\TopSpin3.6.2) 2110 25 — 300.13MHz



8. X-Ray data

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) AX2331

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: AX2331

Bond precision: C-C = 0.0037 A Wavelength=0.71073
Cell: a=23.931 (5) b=9.526 (2) c=9.259 (2)
alpha=90 beta=90 gamma=90
Temperature: 150 K

	Calculated	Reported
Volume	2110.7 (8)	2110.6 (8)
Space group	P n a 21	P n a 21
Hall group	P 2c -2n	P 2c -2n
Moiety formula	C21 H32 B2 O4	?
Sum formula	C21 H32 B2 O4	C21 H32 B2 O4
Mr	370.09	370.08
Dx, g cm ⁻³	1.165	1.165
Z	4	4
Mu (mm ⁻¹)	0.077	0.077
F000	800.0	800.0
F000'	800.36	
h, k, lmax	31, 12, 12	31, 12, 12
Nref	5101 [2707]	5099
Tmin, Tmax	0.963, 0.986	0.960, 0.990
Tmin'	0.963	

Correction method= # Reported T Limits: Tmin=0.960 Tmax=0.990
AbsCorr = MULTI-SCAN

Data completeness= 1.88/1.00 Theta (max)= 27.989

R(reflections)= 0.0454 (4752) wR2 (reflections)=
0.1232 (5099)
S = 1.016 Npar= 252

