Supporting Information

Copper-catalyzed synthesis of *β*-boryl cyclopropanes via 1,2-borocyclopropanation of aryl olefins with CO as the C1 source

Hui-Qing Geng, [a] and Xiao-Feng Wu* [a,b]

[a] Leibniz-Institut für Katalyse e.V., Albert-Einstein-Straße 29a, 18059 Rostock (Germany).
[b] Dalian National Laboratory for Clean Energy, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, 116023, Dalian, Liaoning, China, E-mail: xwu2020@dicp.ac.cn

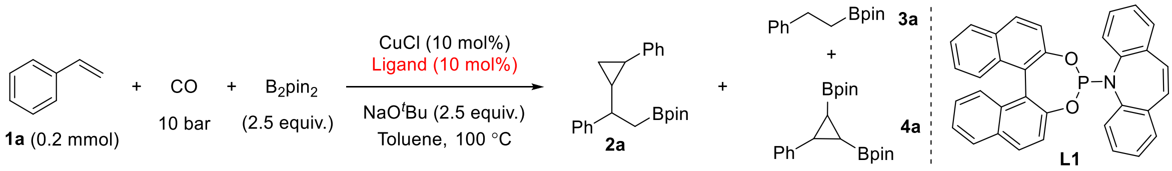
Table of Contents

1. General information

Unless otherwise stated, all experiments were performed under carbon monoxide or nitrogen atmosphere. Aryl olefins were prepared according to previous references. All chemical reagents were purchased Sigma-Aldrich, TCI, ABCR, and Acros of the highest purity grade and used without purification. All solvents were dried by standard techniques. Silica gel column chromatography was carried out using silica gel (200–300 mesh) and *n*-pentane and ethyl acetate as eluent. Analytical thin layer chromatography (TLC) was performed on silica gel (silica gel 60 DC-Platten ALUGRAM® Xtra SIL G / UV_{254}). TLC plates were visualized with UV light, and/or submersion in $KMnO_4$ solution). All NMR spectra were recorded with Bruker Avance III HD 300 NMR (^{1}H, 300 MHz; $^{13}C\{^{1}H\}$, 75 MHz; ^{11}B, 96 MHz), Bruker ARX 400 NMR spectrometers (^{1}H, 400 MHz; $^{13}C\{^{1}H\}$, 101 MHz). Chemical shifts are reported as δ values relative to internal chloroform (δ 7.26 for ^{1}H NMR) and TMS (δ 0.00 for ^{1}H NMR) chloroform (δ 77.16 for $^{13}C\{^{1}H\}$ NMR) in parts per million (ppm). The following abbreviations are used for the NMR spectra' multiplicities: s: singlet, d: doublet, dd: doublet of doublet, t: triplet, q: quartet, m: multiplet, and br: broad signal for proton spectra. All ^{13}C NMR spectra were broad-band ^{1}H decoupled. However, it is hard to observe the signals for the carbon attached to boron in the $^{13}C\{^{1}H\}$ NMR spectra. Gas chromatography (GC) analyses were performed on an Agilent HP-7890A instrument with an 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 were recorded on an Agilent 6210 system. All the reactions which used CO were performed in an autoclave. The laboratory should be well-equipped with a CO detector and alarm system.

2. Optimization of reaction conditions.

***Table S1*. Screening of ligand**[a]

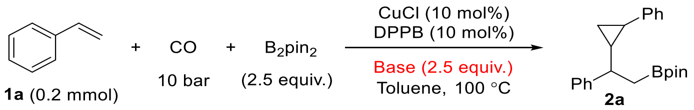


Entry	Ligand	**2a** (%)	**dr**	**3a** (%)	**4a** (%)
1	DPPP	-	-	Trace	16
2	DPPE	-	-	Trace	10
3	DPPF	29	7:1	Trace	-
4	Xantphos	-	-	Major[c]	-
5	BINAP	-	-	Major[c]	-
6	DPEphos	19	8:1	Trace	11
7[b]	PPh_3	-	-	Major[c]	-
8	IPr	-	-	-	-
9	IMes	-	-	-	-
10	DPPM	6	-	Major[c]	-
11	DPPB	42	8:1	Trace	-
12	DPPPe	13	7:1	Trace	6
13	DPPBz	-	-	Trace	-
14	BPY	-	-	-	-

15	**L1**	-	-	Trace	-

[a] Reaction conditions: **1a** (0.2 mmol), B_2Pin_2 (2.5 equiv.), CuCl (10 mol%), ligand (10 mol%), NaO^tBu (2.5 equiv.), Toluene (1 mL), CO (10 Bar), 100 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC. [b] PPh_3 (20 mol%). [c] **3a** is the major product.

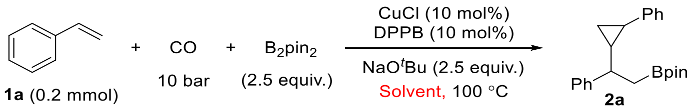
***Table S2*. Screening of base**[a]



Entry	Base	**2a (%)**	**dr**
1	LiO^tBu	Trace	-
2	LiOMe	Trace	-
3	NaOEt	Trace	-
4	KO^tBu	7	4:1
5	KOMe	Trace	-
6	NaO^tBu	42	8:1

[a] Reaction Conditions: **1a** (0.2 mmol), CuCl (10 mol%), DPPB (10 mol%), Base (2.5 equiv.), B_2Pin_2 (2.5 equiv.), Toluene (1 mL), CO (10 bar), 100 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

***Table S3*. Screening of solvent**[a]

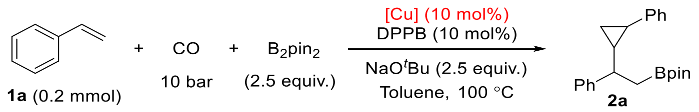


Entry	Solvent	**2a (%)**	**dr**
1	DMAc	4	8:1
2	1,4-dioxane	2	-
3	DCE	18	8:1
4	MeCN	Trace	-
5	DMF	6	-
6	Et_2O	10	-
7	DMSO	4	-
8	Cyalohexane	Trace	-
9	PhF	36	11:1

10	PhCl	34	5:1
11	PhCF$_3$	35	7:1
12	PhEt	39	9:1
13	Toluene	42	8:1

[a] Reaction Conditions: **1a** (0.2 mmol), CuCl (10 mol%), DPPB (10 mol%), NaO^{t}Bu (2.5 equiv.), B$_2$Pin$_2$ (2.5 equiv.), solvent (1 mL), CO (10 bar), 100 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

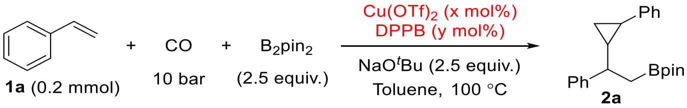
***Table S4*. Screening of copper source**^{a}



Entry	[Cu]	**2a** (%)	**dr**
1	CuCl$_2$	39	8:1
2	CuI	26	8:1
3	CuBr	25	13:1
4	Cu(OAc)$_2$	14	9:1
5	Cu(OTf)$_2$	48	12:1
6	CuCN	4	-
7	Cu(acac)$_2$	47	8:1
8	CuCl$_2$ + AgBF$_4$	37	11:1
9	CuCl$_2$ + AgNO$_3$	n.d.	-
10	Cu(PPh$_3$)$_2$NO$_3$	10	5:1
11	Cu(NO$_3$)$_2$•3H$_2$O	29	6:1
12	Cu(TFA)$_2$•xH$_2$O	31	6:1
13	CuSO$_4$	43	5:1

[a] Reaction Conditions: **1a** (0.2 mmol), [Cu] (10 mol%), DPPB (10 mol%), NaO^{t}Bu (2.5 equiv.), B$_2$Pin$_2$ (2.5 equiv.), Toluene (1 mL), CO (10 bar), 100 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

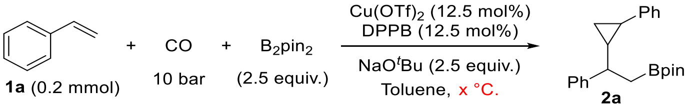
***Table S5*. Screening of amount of Cu(OTf)$_2$ and DPPB**[a]



Entry	Cu(OTf)$_2$	DPPB	**2a** (%)	**dr**
1	2.5 mol%	2.5 mol%	15	13:1
2	5 mol%	5 mol%	37	12:1
3	7.5 mol%	7.5 mol%	47	13:1
4	10 mol%	10 mol%	48	13:1
5	12.5 mol%	12.5 mol%	53	13:1
6	15 mol%	15 mol%	38	13:1
7	10 mol%	12.5 mol%	28	12:1
8	10 mol%	15 mol%	35	13:1
9	10 mol%	20 mol%	20	12:1

[a] Reaction Conditions: **1a** (0.2 mmol), Cu(OTf)$_2$ (x mol%), DPPB (y mol%), NaO^{t}Bu (2.5 equiv.), B$_2$Pin$_2$ (2.5 equiv.), Toluene (1 mL), CO (10 bar), 100 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC. The value of dr determined by GC.

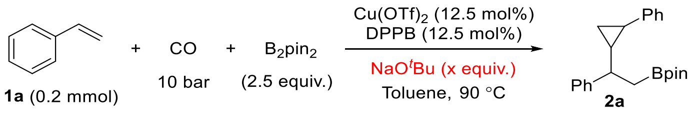
***Table S6*. Screening of temperature**[a]



Entry	Temperature	**2a** (%)	**dr**
1	80 °C	43	11:1
2	90 °C	57	10:1
3	100 °C	53	10:1
4	110 °C	33	10:1
5	120 °C	54	10:1
6	130 °C	52	10:1
7	140 °C	38	10:1

[a] Reaction Conditions: **1a** (0.2 mmol), Cu(OTf)$_2$ (12.5 mol%), DPPB (12.5 mol%), NaO^{t}Bu (2.5 equiv.), B$_2$Pin$_2$ (2.5 equiv.), Toluene (1 mL), CO (10 bar), x °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

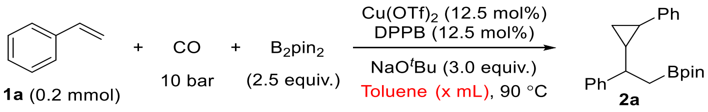
***Table S7*. Screening of the amount of NaO^{t}Bu**[a]



Entry	The amount of NaO^{t}Bu	**2a** (%)	**dr**
1	w/o	n.d.	-
2	12.5 mol%	n.d.	-
3	1 equiv.	8	18:1
4	1.25 equiv.	31	13:1
5	1.75 equiv.	20	11:1
6	2 equiv.	29	11:1
7	2.25 equiv.	45	11:1
8	2.5 equiv.	57	12:1
9	2.75 equiv.	61	12:1
10	3.0 equiv.	63	12:1
11	3.5 equiv.	56	11:1
12	3.75 equiv.	48	10:1

[a] Reaction Conditions: **1a** (0.2 mmol), Cu(OTf)$_2$ (12.5 mol%), DPPB (12.5 mol%), NaO^{t}Bu (x equiv.), B$_2$Pin$_2$ (2.5 equiv.), Toluene (1 mL), CO (10 bar), 90 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

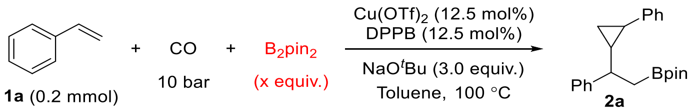
***Table S8*. Screening of the amount of toluene**[a]



Entry	The amount of toluene	**2a** (%)	**dr**
1	0.5 mL	37	11:1
2	1.0 mL	63	12:1
3	2 mL	46	12:1
4	2.5 mL	24	12:1

[a] Reaction Conditions: **1a** (0.2 mmol), Cu(OTf)$_2$ (12.5 mol%), DPPB (12.5 mol%), NaO^{t}Bu (3.0 equiv.), B$_2$Pin$_2$ (2.5 equiv.), Toluene (x mL), CO (10 bar), 90 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

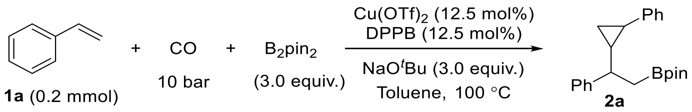
***Table S9*. Screening of the amount of B_2pin_2[a]**



Entry	The amount of B_2pin_2	**2a** (%)	**dr**
1	1 equiv.	trace	-
2	1.5 equiv.	24	12:1
3	1.75 equiv.	62	12:1
4	2 equiv.	64	13:1
5	2.5 equiv.	63	12:1
6	3.0 equiv.	74	12:1
7	3.5 equiv.	53	13:1

[a] Reaction Conditions: **1a** (0.2 mmol), $Cu(OTf)_2$ (12.5 mol%), DPPB (12.5 mol%), NaO^{t}Bu (3.0 equiv.), B_2Pin_2 (x equiv.), Toluene (1 mL), CO (10 bar), 90 °C, 16 h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC.

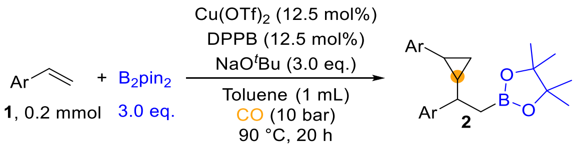
***Table S10*. Screening of the reaction time[a]**



Entry	Reaction time	**2a** (%)	**dr**
1	16 h	74	12:1
2	20 h	77 (69[c])	14:1
3[b]	20 h	62	14:1

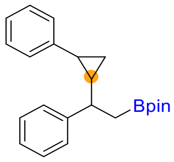
[a] Reaction Conditions: **1a** (0.2 mmol), $Cu(OTf)_2$ (12.5 mol%), DPPB (12.5 mol%), NaO^{t}Bu (3.0 equiv.), B_2Pin_2 (3.0 equiv.), Toluene (1 mL), CO (10 bar), 90 °C, x h. Yields are determined by GC with hexadecane as an internal standard. The yield of **2a** is calculated based on 0.1 mmol styrene. Because two equivalents of **1a** are required to produce one equivalent of **2a**. The value of dr determined by GC. [b] 4Å MS (30 mg). [c] Isolated yield.

3. General procedures for synthesis of 2.



A dried vial (4 mL) was charged with $Cu(OTf)_2$ (12.5 mol%), DPPB (12.5 mol%), B_2pin_2 (3.0 equiv.), NaO^tBu (3.0 equiv.), and a stirring bar. The vial was sealed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, aryl olefins (0.2 mmol) and toluene (1.0 mL) were added under argon by using a syringe. The vial (or several vials) was placed in an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. The autoclave was charged with CO (10 bar) after flushing two times with N_2 and two times with CO. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction was performed for 20 h at 90 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After that the residue was directly purified by column chromatography to afford the corresponding products.

4. Characterization Data.



2a, 69%

4,4,5,5-Tetramethyl-2-(2-phenyl-2-(2-phenylcyclopropyl)ethyl)-1,3,2-dioxaborolane (2a)

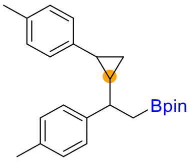
The title compound was prepared from styrene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 100:1, Rf = 0.2) to give the product as a colorless oil (24 mg, 69%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.29 – 7.26 (m, 2H), 7.25 – 7.05 (m, 8H), 2.39 (td, *J* = 9.3, 6.3 Hz, 1H), 1.92 – 1.81 (m, 1H), 1.40 – 1.32 (m, 3H), 1.06 (s, 6H), 1.02 (s, 6H), 0.87 – 0.77 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 146.4, 143.6, 128.2, 128.1, 127.4, 126.0, 126.0, 125.2, 83.0, 46.1, 31.8, 24.7, 24.5, 23.8, 15.8.

^{11}B NMR (96 MHz, CDCl$_3$) δ 33.0.

HRMS (**EI**): calcd for [M]$^+$ $C_{23}H_{29}BO_2$ 348.2255, found: 348.2259.



2b, 55%

4,4,5,5-Tetramethyl-2-(2-(*p*-tolyl)-2-(2-(*p*-tolyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2b)

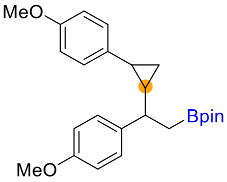
The title compound was prepared from 1-methyl-4-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 100:1, Rf = 0.2) to give the product as a colorless oil (21 mg, 55%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.18 – 7.14 (m, 2H), 7.11 – 7.03 (m, 4H), 6.98 (d, *J* = 8.2 Hz, 2H), 2.37 – 2.28 (m, 7H), 1.85 (dd, *J* = 9.0, 4.5 Hz, 1H), 1.31 (dt, *J* = 13.2, 3.4 Hz, 3H), 1.07 (d, *J* = 8.5 Hz, 12H), 0.85 – 0.73 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 143.4, 140.6, 135.3, 134.7, 128.8, 128.8, 127.2, 125.9, 82.9, 45.6, 31.6, 24.7, 24.6, 23.4, 21.0, 20.9, 15.5.

^{11}B NMR (96 MHz, CDCl$_3$) δ 33.2.

HRMS (**EI**): calcd for [M]$^+$ $C_{25}H_{33}BO_2$ 376.2568, found: 376.2574.



2c, 54%

2-(2-(4-Methoxyphenyl)-2-(2-(4-methoxyphenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2c)

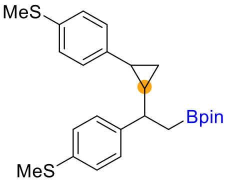
The title compound was prepared from 1-methoxy-4-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.2) to give the product as a colorless oil (22 mg, 54%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.22 – 7.14 (m, 2H), 7.11 – 6.94 (m, 2H), 6.85 – 6.76 (m, 4H), 3.79 (s, 3H), 3.77 (s, 3H), 2.34 (td, *J* = 9.3, 6.1 Hz, 1H), 1.81 (dt, *J* = 9.1, 5.0 Hz, 1H), 1.30 – 1.25 (m, 3H), 1.08 (s, 6H), 1.05 (s, 6H), 0.82 – 0.67 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 157.8, 157.5, 138.7, 135.6, 128.3, 127.1, 113.6, 113.5, 83.0, 55.3, 55.2, 45.2, 31.4, 24.8, 24.6, 22.9, 15.1.

^{11}B NMR (96 MHz, CDCl$_3$) δ 33.1.

HRMS (**EI**): calcd for [M]$^+$ $C_{25}H_{33}BO_4$ 408.2466, found: 408.2474.



2d, 41%

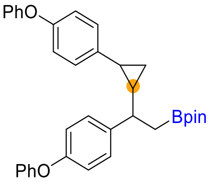
4,4,5,5-Tetramethyl-2-(2-(4-(methylthio)phenyl)-2-(2-(4-(methylthio)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2d)

The title compound was prepared from methyl(4-vinylphenyl)sulfane (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 30:1, Rf = 0.3) to give the product as a colorless oil (18 mg, 41%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 – 7.15 (m, 6H), 7.01 (d, *J* = 8.2 Hz, 2H), 2.46 (s, 3H), 2.45 (s, 3H), 2.40 – 2.32 (m, 1H), 1.87 – 1.77 (m, 1H), 1.35 – 1.26 (m, 3H), 1.08 (s, 6H), 1.05 (s, 6H), 0.84 – 0.74 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 140.9, 128.0, 127.4, 127.1, 126.6, 83.1, 45.5, 31.6, 24.8, 24.6, 23.3, 16.6, 16.4, 15.6.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.1.



2e, 52%

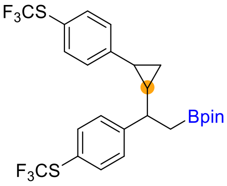
4,4,5,5-Tetramethyl-2-(2-(4-phenoxyphenyl)-2-(2-(4-phenoxyphenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2e)

The title compound was prepared from 1-phenoxy-4-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.2) to give the product as a colorless oil (28 mg, 52%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 4H), 7.28 (s, 2H), 7.14 – 7.05 (m, 4H), 7.04 – 6.85 (m, 8H), 2.42 (td, *J* = 9.3, 6.1 Hz, 1H), 1.95 – 1.85 (m, 1H), 1.43 – 1.30 (m, 3H), 1.12 (d, *J* = 9.7 Hz, 12H), 0.87 – 0.79 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 157.8, 129.6, 128.6, 127.3, 122.8, 122.8, 119.1, 119.0, 118.4, 118.3, 83.1, 45.4, 31.6, 24.8, 24.5, 23.2, 15.6.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.54.



2f, 69%

4,4,5,5-Tetramethyl-2-(2-(4-((trifluoromethyl)thio)phenyl)-2-(2-(4-((trifluoromethyl)thio)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2f)

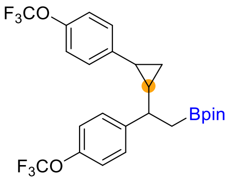
The title compound was prepared from (trifluoromethyl)(4-vinylphenyl)sulfane (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 30:1, Rf = 0.2) to give the product as a colorless oil (38 mg, 69%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 – 7.50 (m, 4H), 7.35 – 7.31 (m, 2H), 7.12 – 7.09 (m, 2H), 2.43 (td, *J* = 9.4, 6.1 Hz, 1H), 1.91 (dt, *J* = 8.8, 5.0 Hz, 1H), 1.43 – 1.37 (m, 3H), 1.05 (s, 6H), 1.00 (s, 6H), 0.89 – 0.83 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 149.4, 146.9, 136.4, 131.1 (q, *J* = 309.5 Hz), 129.60 (q, *J* = 307.7 Hz), 128.6, 126.9, 83.2, 45.9, 29.7, 24.6, 24.4, 23.8, 16.5.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.9.

^{19}F NMR (282 MHz, $CDCl_3$) δ -43.13, -43.22.



2g, 51%

4,4,5,5-Tetramethyl-2-(2-(4-(trifluoromethoxy)phenyl)-2-(2-(4-(trifluoromethoxy)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2g)

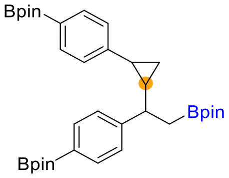
The title compound was prepared from 1-(trifluoromethoxy)-4-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 30:1, Rf = 0.2) to give the product as a colorless oil (26 mg, 51%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.36 – 7.27 (m, 2H), 7.13 (ddd, *J* = 8.6, 3.4, 1.7 Hz, 2H), 7.09 (s, 4H), 2.40 (td, *J* = 9.4, 6.1 Hz, 1H), 1.88 (dt, *J* = 8.6, 5.2 Hz, 1H), 1.37 (t, *J* = 3.0 Hz, 3H), 1.06 (s, 6H), 1.02 (s, 6H), 0.87 – 0.84 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 145.0, 142.1, 128.6, 127.2, 120.9, 120.8, 120.5 (q, *J* = 256.6 Hz), 83.2, 45.5, 31.5, 24.7, 24.5, 23.3, 15.9.

^{11}B NMR (96 MHz, CDCl$_3$) δ 33.6.

^{19}F NMR (282 MHz, CDCl$_3$) δ -58.0, -58.0.



2h, 39%

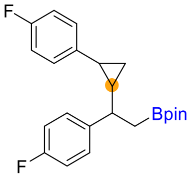
4,4,5,5-Tetramethyl-2-(4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(2-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)cyclopropyl)ethyl)phenyl)-1,3,2-dioxaborolane (2h)

The title compound was prepared from 4,4,5,5-tetramethyl-2-(4-vinylphenyl)-1,3,2-dioxaborolane (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 50:1, Rf = 0.2) to give the product as a colorless oil (23 mg, 39%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.73 (dd, *J* = 16.9, 8.1 Hz, 4H), 7.30 – 7.24 (m, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 2.44 (td, *J* = 9.1, 6.5 Hz, 1H), 2.00 – 1.83 (m, 1H), 1.35 (dd, *J* = 2.9, 1.8 Hz, 27H), 1.09 (d, *J* = 11.4 Hz, 12H), 0.91 – 0.82 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 149.8, 147.2, 134.8, 126.9, 125.3, 83.6, 83.1, 46.2, 32.0, 24.9, 24.9, 24.6, 24.2, 16.2.

^{11}B NMR (96 MHz, CDCl$_3$) δ 30.0.



2i, 57%

2-(2-(4-Fluorophenyl)-2-(2-(4-fluorophenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2i)

The title compound was prepared from 1-fluoro-4-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 100:1, Rf = 0.2) to give the product as a colorless oil (22 mg, 39%).

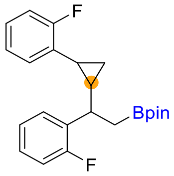
^{1}H NMR (300 MHz, CDCl$_3$) δ 7.25 – 7.17 (m, 2H), 7.07 – 6.89 (m, 6H), 2.38 (td, *J* = 9.3, 6.1 Hz, 1H), 1.92 – 1.74 (m, 1H), 1.28 (d, *J* = 4.8 Hz, 3H), 1.08 (s, 6H), 1.05 (s, 6H), 0.83 – 0.74 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 161.3 (d, *J* = 243.1 Hz), 161.0 (d, *J* = 243.1 Hz), 142.0 (d, *J* = 3.1 Hz), 138.9 (d, *J* = 3.0 Hz), 128.7 (d, *J* = 7.8 Hz), 127.5 (d, *J* = 7.8 Hz), 114.9 (d, *J* = 21.1 Hz), 114.8 (d, *J* = 21.1 Hz), 83.1, 45.2, 31.5, 24.7, 24.5, 23.1, 15.4.

^{11}B NMR (96 MHz, CDCl$_3$) δ 32.8.

^{19}F NMR (282 MHz, CDCl$_3$) δ -117.5, -118.2.

HRMS (EI): calcd for [M]$^+$ $C_{23}H_{27}BF_2O_2$ 384.2067, found: 384.2070.



2j, 67%

2-(2-(2-Fluorophenyl)-2-(2-(2-fluorophenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2j)

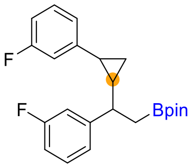
The title compound was prepared from 1-fluoro-2-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 100:1, Rf = 0.2) to give the product as a colorless oil (26 mg, 67%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 1H), 7.19 – 7.08 (m, 3H), 6.99 (ddt, *J* = 7.9, 6.7, 2.1 Hz, 3H), 6.95 – 6.89 (m, 1H), 2.76 (td, *J* = 9.5, 6.3 Hz, 1H), 2.11 – 2.08 (m, 1H), 1.48 – 1.34 (m, 3H), 1.06 (s, 6H), 1.02 (s, 6H), 0.93 – 0.81 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 161.7 (d, *J* = 245.3 Hz), 160.6 (d, *J* = 244.6 Hz), 133.1 (d, *J* = 15.1 Hz), 130.2 (d, *J* = 14.5 Hz), 128.8 (d, *J* = 5.3 Hz), 127.4 (d, *J* = 8.1 Hz), 126.6 (d, *J* = 7.5 Hz), 126.5 (d, *J* = 4.5 Hz), 123.8 (d, *J* = 3.8 Hz), 123.8 (d, *J* = 3.7 Hz), 115.2 (d, *J* = 23.1 Hz), 114.9 (d, *J* = 22.2 Hz), 83.0, 39.3, 29.5, 24.7, 24.4, 17.1 (d, *J* = 5.0 Hz).14.2.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.0.

^{19}F NMR (282 MHz, $CDCl_3$) δ -117.74 (dt, *J* = 11.3, 6.5 Hz), -119.11 (ddd, *J* = 10.3, 7.4, 5.2 Hz).



2k, 61%

2-(2-(3-Fluorophenyl)-2-(2-(3-fluorophenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2k)

The title compound was prepared from 1-fluoro-3-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 100:1, Rf = 0.2) to give the product as a colorless oil (23 mg, 61%).

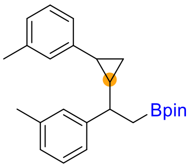
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 – 7.11 (m, 2H), 7.06 – 6.96 (m, 2H), 6.92 – 6.73 (m, 4H), 2.39 (td, *J* = 9.2, 6.1 Hz, 1H), 1.96 – 1.82 (m, 1H), 1.38 – 1.28 (m, 3H), 1.10 (s, 6H), 1.06 (s, 6H), 0.89 – 0.81 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0 (d, *J* = 244.6 Hz), 162.8 (d, *J* = 245.3 Hz), 149.0 (d, *J* = 6.6 Hz), 146.2 (d, *J* = 7.4 Hz), 129.6 (d, *J* = 7.8 Hz), 129.6 (d, *J* = 8.3 Hz), 123.0 (d, *J* = 2.8 Hz), 121.7 (d, *J* = 2.7 Hz), 114.3 (d, *J* = 21.0 Hz), 112.9 (d, *J* = 21.1 Hz), 112.7 (d, *J* = 21.9 Hz), 112.2 (d, *J* = 21.1 Hz), 83.2, 45.8, 31.6, 24.8, 24.6, 23.7, 16.0.

^{11}B NMR (96 MHz, $CDCl_3$) δ 32.9.

^{19}F NMR (282 MHz, $CDCl_3$) δ -113.80 – -113.86 (m), -113.99 – -114.13 (m).

HRMS (**EI**): calcd for $[M]^+$ $C_{23}H_{27}BF_2O_2$ 384.2067, found: 384.2076.



2l, 64%

4,4,5,5-Tetramethyl-2-(2-(*m*-tolyl)-2-(2-(*m*-tolyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2l)

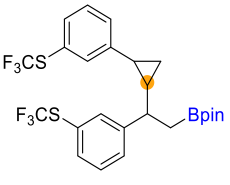
The title compound was prepared from 1-fluoro-3-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 100:1, Rf = 0.2) to give the product as a colorless oil (24 mg, 64%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 – 7.05 (m, 4H), 7.02 – 6.86 (m, 4H), 2.37 – 2.26 (m, 1H), 2.32 (d, *J* = 7.9 Hz, 6H), 1.84 (dt, *J* = 8.6, 5.0 Hz, 1H), 1.37 – 1.26 (m, 3H), 1.08 (s, 6H), 1.05 (s, 6H), 0.87 – 0.78 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 146.4, 143.6, 137.7, 137.5, 128.2, 128.1, 128.0, 126.8, 126.7, 126.0, 124.4, 123.0, 82.9, 46.2, 31.7, 24.7, 24.5, 23.8, 21.5, 21.4, 15.8.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.3.

HRMS (**EI**): calcd for $[M]^+$ $C_{25}H_{33}BO_2$ 376.2568, found: 376.2573.



2m, 73%

4,4,5,5-Tetramethyl-2-(2-(3-((trifluoromethyl)thio)phenyl)-2-(2-(3-((trifluoromethyl)thio)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2m)

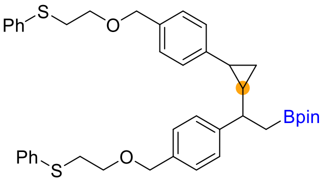
The title compound was prepared from (trifluoromethyl)(3-vinylphenyl)sulfane (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 30:1, Rf = 0.2) to give the product as a colorless oil (40 mg, 73%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 – 7.29 (m, 7H), 7.19 – 7.09 (m, 1H), 2.45 (td, *J* = 9.0, 6.3 Hz, 1H), 1.95 – 1.87 (m, 1H), 1.48 – 1.38 (m, 3H), 1.05 (d, *J* = 12.7 Hz, 12H), 0.89 – 0.85 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 147.8, 145.0, 135.5, 134.0, 133.9, 133.3, 129.9, 129.3, 128.4, 128.5 (q, *J* = 309.4 Hz), 128.3 (q, *J* = 309.5 Hz), 124.4, 83.2, 45.6, 31.5, 24.7, 24.5, 23.5, 15.9.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.2.

^{19}F NMR (282 MHz, $CDCl_3$) δ -42.59, -42.61.



2n,43%

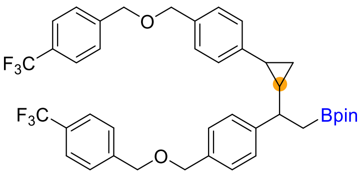
4,4,5,5-Tetramethyl-2-(2-(4-((2-(phenylthio)ethoxy)methyl)phenyl)-2-(2-(4-((2-(phenylthio)ethoxy)methyl)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2n)

The title compound was prepared from phenyl(2-((4-vinylbenzyl)oxy)ethyl)sulfane (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 7:1, Rf = 0.2) to give the product as a colorless oil (29 mg, 43%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 7.24 (m, 4H), 7.22 – 7.15 (m, 8H), 7.14 – 7.09 (m, 4H), 6.97 (d, *J* = 8.1 Hz, 2H), 4.41 (d, *J* = 8.8 Hz, 4H), 3.56 (td, *J* = 6.9, 3.1 Hz, 4H), 3.07 (s, 4H), 2.32 (td, *J* = 9.2, 6.3 Hz, 1H), 1.79 (dt, *J* = 8.7, 4.9 Hz, 1H), 1.32 – 1.21 (m, 3H), 0.97 (d, *J* = 10.0 Hz, 12H), 0.79 – 0.70 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 146.0, 143.2, 135.6, 134.9, 129.4, 128.9, 127.9, 127.7, 127.5, 126.1, 126.1, 83.5, 83.0, 72.9, 45.8, 33.3, 31.8, 24.8, 24.6, 23.6, 15.9.

^{11}B NMR (96 MHz, $CDCl_3$) δ 30.1.



2o, 54%

4,4,5,5-Tetramethyl-2-(2-(4-(((4-(trifluoromethyl)benzyl)oxy)methyl)phenyl)-2-(2-(4-(((4-(trifluoromethyl)benzyl)oxy)methyl)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2o)

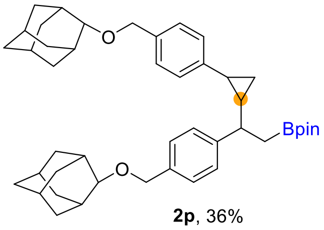
The title compound was prepared from 1-(trifluoromethyl)-4-(((4-vinylbenzyl)oxy)methyl)benzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.3) to give the product as a colorless oil (39 mg, 54%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.1 Hz, 4H), 7.50 (d, *J* = 7.6 Hz, 4H), 7.35 – 7.24 (m, 6H), 7.12 (d, *J* = 8.1 Hz, 2H), 4.64 – 4.54 (m, 8H), 2.46 (td, *J* = 9.1, 6.2 Hz, 1H), 1.93 (dt, *J* = 9.0, 4.9 Hz, 1H), 1.46 – 1.34 (m, 3H), 1.09 (d, *J* = 9.6 Hz, 12H), 0.93 – 0.80 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 146.2, 143.4, 142.6, 135.5, 134.8, 129.7 (q, *J* = 30.2 Hz), 128.0, 127.9, 127.6, 127.6, 127.6, 126.1, 125.28 (q, *J* = 3.7 Hz), 124.2 (q, *J* = 272.0 Hz), 83.1, 72.4, 72.4, 71.0, 45.8, 31.9, 24.8, 24.6, 23.6, 15.9.

^{11}B NMR (96 MHz, $CDCl_3$) δ 31.5.

^{19}F NMR (282 MHz, $CDCl_3$) δ -62.46.



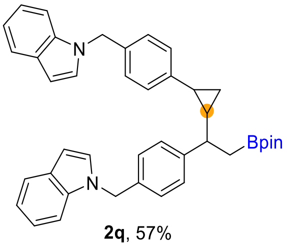
2p, 36%

2-(2-(4-((((1*r*,3*r*,5*r*,7*r*)-Adamantan-2-yl)oxy)methyl)phenyl)-2-(2-(4-((((1*r*,3*r*,5*r*,7*r*)-adamantan-2-yl)oxy)methyl)phenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2p)

The title compound was prepared from (1*r*,3*r*,5*r*,7*r*)-2-((4-vinylbenzyl)oxy)adamantane (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.3) to give the product as a colorless oil (24 mg, 36%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.24 – 7.13 (m, 6H), 6.97 (d, J = 8.1 Hz, 2H), 4.43 (d, J = 9.2 Hz, 4H), 3.45 (t, J = 2.9 Hz, 2H), 2.31 (td, J = 9.2, 6.3 Hz, 1H), 2.20 – 1.84 (m, 9H), 1.81 – 1.51 (m, 19H), 1.30 – 1.22 (m, 3H), 0.99 (d, J = 9.6 Hz, 12H), 0.79 – 0.69 (m, 2H).
^{13}C NMR (75 MHz, CDCl$_3$) δ 145.4, 142.6, 137.1, 136.5, 127.4, 127.3, 125.9, 83.0, 80.9, 80.8, 69.1, 45.8, 37.6, 36.6, 31.9, 31.7, 31.7, 27.5, 24.8, 24.6, 23.6, 15.8.

^{11}B NMR (96 MHz, CDCl$_3$) δ 33.6.



2q, 57%

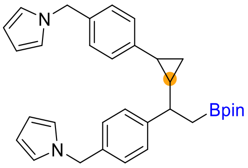
1-(4-(2-(1-(4-((1*H*-Indol-1-yl)methyl)phenyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)cyclopropyl)benzyl)-1*H*-indole (2q)

The title compound was prepared from 1-(4-vinylbenzyl)-1*H*-indole (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.2) to give the product as a colorless oil (35 mg, 57%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.61 – 7.46 (m, 2H), 7.20 – 6.95 (m, 12H), 6.89 (s, 4H), 6.44 (d, J = 3.1 Hz, 2H), 5.17 (d, J = 6.4 Hz, 4H), 2.25 (td, J = 9.3, 6.2 Hz, 1H), 1.73 (dt, J = 9.2, 4.9 Hz, 1H), 1.22 – 1.16 (m, 3H), 0.91 (d, J = 11.3 Hz, 12H), 0.74 – 0.62 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 145.8, 142.9, 136.2, 135.1, 134.4, 128.6, 128.1, 127.7, 126.7, 126.2, 121.5, 121.5, 120.9, 120.8, 119.4, 119.4, 109.7, 109.6, 101.5, 101.4, 82.9, 49.9, 49.8, 45.7, 31.8, 24.7, 24.5, 23.4, 15.8.

^{11}B NMR (96 MHz, CDCl$_3$) δ 31.6.



2r, 62%

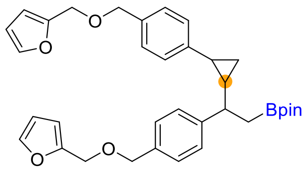
1-(4-(2-(1-(4-((1*H*-Pyrrol-1-yl)methyl)phenyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)cyclopropyl)benzyl)-1*H*-pyrrole (2r)

The title compound was prepared from 1-(4-vinylbenzyl)-1*H*-pyrrole (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.2) to give the product as a colorless oil (31 mg, 62%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.21 (d, J = 8.1 Hz, 2H), 7.06 (d, J = 8.3 Hz, 2H), 7.04 – 6.95 (m, 4H), 6.67 (t, J = 2.1 Hz, 4H), 6.17 (t, J = 2.1 Hz, 4H), 5.03 (s, 2H), 5.01 (s, 2H), 2.36 (td, J = 9.2, 6.1 Hz, 1H), 1.85 (dt, J = 9.0, 4.9 Hz, 1H), 1.36 – 1.29 (m, 3H), 1.04 (d, J = 13.3 Hz, 12H), 0.85 – 0.75 (m, 2H).

^{13}C NMR (75 MHz, CDCl$_3$) δ 135.7, 135.0, 127.7, 127.0, 126.2, 121.0, 108.3, 108.2, 83.0, 53.1, 53.0, 45.7, 31.8, 25.0, 24.7, 23.4, 15.8.

^{11}B NMR (96 MHz, CDCl$_3$) δ 30.2.



2s, 37%

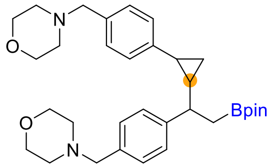
2-(2-(4-((Furan-2-ylmethoxy)methyl)phenyl)-2-(2-(4-((furan-2-ylmethoxy)methyl)phenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2s)

The title compound was prepared from 2-(((4-vinylbenzyl)oxy)methyl)furan (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 20:1, Rf = 0.3) to give the product as a colorless oil (21 mg, 37%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (td, *J* = 1.7, 0.9 Hz, 2H), 7.29 – 7.23 (m, 6H), 7.14 – 7.01 (m, 2H), 6.39 – 6.31 (m, 4H), 4.55 (s, 2H), 4.52 (s, 2H), 4.48 (d, *J* = 1.8 Hz, 4H), 2.43 (td, *J* = 9.2, 6.2 Hz, 1H), 1.90 (dt, *J* = 8.9, 5.0 Hz, 1H), 1.40 – 1.30 (m, 3H), 1.08 (d, *J* = 9.9 Hz, 12H), 0.87 – 0.77 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 151.8, 146.0, 143.2, 142.7, 135.4, 134.8, 128.0, 127.9, 127.5, 126.0, 110.2, 109.3, 83.0, 71.8, 71.7, 63.5, 45.7, 31.8, 24.7, 24.6, 23.6, 15.8.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.3.



2t, 61%

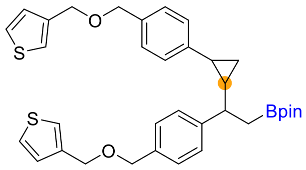
4-(4-(2-(1-(4-(Morpholinomethyl)phenyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)cyclopropyl)benzyl)morpholine (2t)

The title compound was prepared from 4-(4-vinylbenzyl)morpholine (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (EA/MeOH = 20:1, Rf = 0.2) to give the product as a colorless oil (33 mg, 61%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.17 (m, 6H), 7.02 (d, *J* = 8.0 Hz, 2H), 3.72 – 3.68 (m, 8H), 3.50 (d, *J* = 8.0 Hz, 4H), 2.46 (s, 8H), 2.37 (dd, *J* = 9.3, 3.4 Hz, 1H), 1.85 (dt, *J* = 8.9, 4.9 Hz, 1H), 1.40 – 1.28 (m, 3H), 1.03 (d, *J* = 12.5 Hz, 12H), 0.87 – 0.80 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.5, 142.8, 129.3, 129.3, 127.4, 125.9, 83.0, 66.8, 63.1, 53.4, 45.8, 31.9, 24.7, 24.6, 23.6, 15.9.

^{11}B NMR (128 MHz, $CDCl_3$) δ 33.3.



2u, 51%

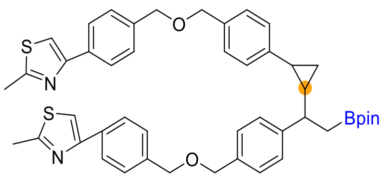
4,4,5,5-Tetramethyl-2-(2-(4-((thiophen-3-ylmethoxy)methyl)phenyl)-2-(2-(4-((thiophen-3-ylmethoxy)methyl)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2u)

The title compound was prepared from 3-(((4-vinylbenzyl)oxy)methyl)thiophene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 30:1, Rf = 0.3) to give the product as a colorless oil (31 mg, 51%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 – 7.13 (m, 10H), 7.03 – 6.97 (m, 4H), 4.46 – 4.41 (m, 8H), 2.33 (td, *J* = 9.2, 6.3 Hz, 1H), 1.80 (dt, *J* = 8.9, 5.0 Hz, 1H), 1.26 – 1.15 (m, 3H), 0.98 (d, *J* = 9.6 Hz, 12H), 0.79 – 0.70 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 145.9, 143.1, 139.5, 135.7, 135.0, 127.9, 127.8, 127.4, 127.4, 126.0, 125.9, 125.9, 122.7, 83.0, 71.9, 71.8, 67.0, 45.7, 31.8, 24.7, 24.6, 23.6, 15.8.

^{11}B NMR (96 MHz, $CDCl_3$) δ 33.3.



2v, 47%

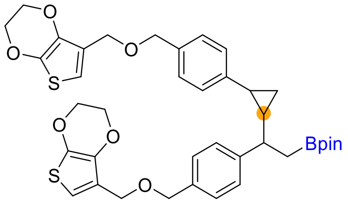
2-methyl-4-(4-(((4-(2-(1-(4-(((4-(2-methylthiazol-4-yl)benzyl)oxy)methyl)phenyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)cyclopropyl)benzyl)oxy)methyl)phenyl)thiazole (2v)

The title compound was prepared from 2-methyl-4-(4-(((4-vinylbenzyl)oxy)methyl)phenyl)thiazole (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 4:1, Rf = 0.2) to give the product as a colorless oil (37 mg, 47%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 8.2, 1.7 Hz, 4H), 7.42 (d, *J* = 7.5 Hz, 4H), 7.29 (ddd, *J* = 14.2, 5.2, 2.8 Hz, 8H), 7.10 (d, *J* = 8.1 Hz, 2H), 4.66 – 4.49 (m, 8H), 2.80 (s, 6H), 2.44 (td, *J* = 9.2, 6.2 Hz, 1H), 1.91 (dq, *J* = 8.9, 4.4 Hz, 1H), 1.45 – 1.32 (m, 3H), 1.08 (d, *J* = 9.6 Hz, 12H), 0.90 – 0.79(m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 154.9, 146.0, 143.2, 138.2, 135.8, 135.1, 133.8, 128.2, 128.0, 127.9, 127.5, 126.4, 126.1, 112.2, 83.0, 75.0, 72.0, 71.9, 71.6, 45.8, 31.9, 24.9, 24.8, 24.6, 23.6, 19.3, 15.9.

^{11}B NMR (96 MHz, $CDCl_3$) δ 22.6.



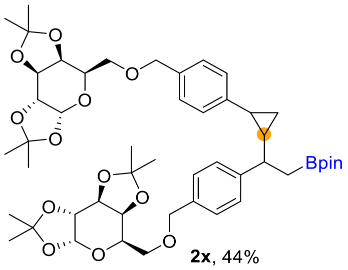
2w, 53%

2-(2-(4-(((2,3-Dihydrothieno[2,3-*b*][1,4]dioxin-7-yl)methoxy)methyl)phenyl)-2-(2-(4-(((2,3-dihydrothieno[2,3-*b*][1,4]dioxin-7-yl)methoxy)methyl)phenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2w)

The title compound was prepared from 7-(((4-vinylbenzyl)oxy)methyl)-2,3-dihydrothieno[2,3-*b*][1,4]dioxine (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 5:1, Rf = 0.2) to give the product as a colorless oil (38 mg, 53%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.06 (m, 8H), 6.98 (d, *J* = 8.1 Hz, 2H), 6.23 (dd, *J* = 4.6, 1.3 Hz, 2H), 4.46 – 4.42 (m, 8H), 4.12 (qq, *J* = 2.8, 1.3 Hz, 8H), 2.37 – 2.28 (m, 1H), 1.79 (dt, *J* = 8.9, 5.0 Hz, 1H), 1.32 – 1.26 (m, 3H), 0.98 (d, *J* = 9.9 Hz, 12H), 0.77 – 0.70 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 145.9, 143.1, 141.3, 139.7, 135.7, 135.0, 128.0, 127.9, 127.4, 126.0, 113.7, 113.2, 98.9, 83.0, 75.0, 71.5, 71.5, 64.7, 64.6, 62.2, 62.2, 45.8, 31.8, 24.9, 24.8, 24.6, 15.8.
^{11}B NMR (96 MHz, $CDCl_3$) δ 22.6.

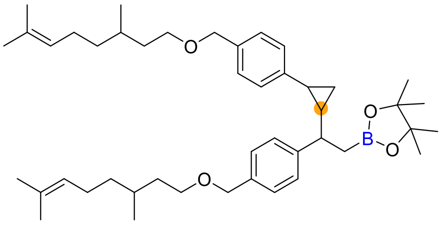


2x, 44%

4,4,5,5-Tetramethyl-2-(2-(4-((((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-b:4',5'-*d*]pyran-5-yl)methoxy)methyl)phenyl)-2-(2-(4-((((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-b:4',5'-*d*]pyran-5-yl)methoxy)methyl)phenyl)cyclopropyl)ethyl)-1,3,2-dioxaborolane (2x)

The title compound was prepared from (3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyl-5-(((4-vinylbenzyl)oxy)methyl)tetrahydro-5*H*-bis([1,3]dioxolo)[4,5-b:4',5'-*d*]pyran (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 1:1, Rf = 0.2) to give the product as a colorless oil (39 mg, 44%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.23 – 7.07 (m, 6H), 6.96 (d, *J* = 8.2 Hz, 2H), 4.59 – 4.41 (m, 6H), 4.39 – 4.34 (m, 2H), 4.19 – 4.13 (m, 2H), 3.89 – 3.83 (m, 1H), 3.82 (d, *J* = 2.0 Hz, 1H), 3.66 (ddd, *J* = 13.0, 1.9, 0.7 Hz, 2H), 3.57 – 3.41 (m, 4H), 2.31 (td, *J* = 9.2, 6.3 Hz, 1H), 1.78 (dt, *J* = 9.0, 5.0 Hz, 1H), 1.37 (d, *J* = 4.5 Hz, 3H), 1.18 (d, *J* = 6.4 Hz, 24H), 0.97 (d, *J* = 11.2 Hz, 12H), 0.78 – 0.63 (m, 2H).
^{13}C NMR (75 MHz, CDCl$_3$) δ 145.6, 142.9, 135.7, 135.0, 127.6, 127.4, 127.3, 125.8, 108.9, 108.5, 102.7, 102.7, 82.9, 74.9, 73.6, 73.5, 71.4, 71.4, 71.3, 71.0, 70.2, 70.2, 70.1, 60.9, 45.7, 31.7, 26.5, 25.8, 25.8, 25.4, 24.8, 24.7, 24.6, 24.5, 24.5, 24.0, 23.6, 15.8.



2y, 43%

2-(2-(4-(((3,7-Dimethyloct-6-en-1-yl)oxy)methyl)phenyl)-2-(2-(4-(((3,7-dimethyloct-6-en-1-yl)oxy)methyl)phenyl)cyclopropyl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2y)

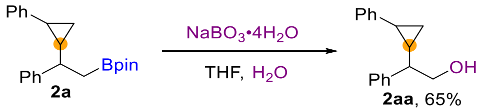
The title compound was prepared from 1-(((3,7-dimethyloct-6-en-1-yl)oxy)methyl)-4-vinylbenzene (0.2 mmol), according to general procedure. The crude residue was purified by flash chromatography (*n*-pentane/EA = 30:1, Rf = 0.4) to give the product as a colorless oil (29 mg, 43%).

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.25 (s, 4H), 7.22 – 7.19 (m, 2H), 7.05 (d, *J* = 8.1 Hz, 2H), 5.09 (dddd, *J* = 7.1, 5.8, 2.9, 1.4 Hz, 2H), 4.46 (d, *J* = 8.4 Hz, 4H), 3.47 (ddq, *J* = 6.9, 4.7, 2.4 Hz, 4H), 2.39 (td, *J* = 9.2, 6.2 Hz, 1H), 2.01 – 1.84 (m, 5H), 1.68 (s, 6H), 1.60 (s, 6H), 1.49 – 1.20 (m, 11H), 1.14 (t, *J* = 2.3 Hz, 2H), 1.06 (d, *J* = 9.8 Hz, 12H), 0.88 (dd, *J* = 6.6, 1.2 Hz, 6H), 0.84 – 0.74 (m, 2H).
^{13}C NMR (75 MHz, CDCl$_3$) δ 145.7, 142.9, 136.3, 135.6, 131.1, 127.7, 127.6, 127.4, 125.9, 124.8, 83.0, 72.8, 68.5, 45.7, 37.2, 36.7, 31.8, 29.6, 29.6, 25.7, 25.5, 24.7, 24.6, 23.6, 19.5, 17.6, 15.8.

^{11}B NMR (96 MHz, CDCl$_3$) δ 34.8.

5. Derivatization of 2a.

5.1 Oxidation of **2a.**



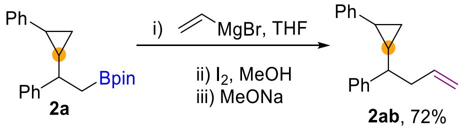
The title compound **2aa** was synthesized according to the following literature[1]: $NaBO_3 \bullet 4H_2O$ (5 equiv.) was added to a solution of boration product **2a** (0.1 mmol) in THF/H_2O (2.5 mL/2.5 mL). The resulting mixture was stirred vigorously for 2 h at room temperature. The reaction mixture was quenched with water and then extracted with ethyl acetate (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 **2aa** as a colorless oil.

2-Phenyl-2-(2-phenylcyclopropyl)ethan-1-ol (2aa)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.37 (m, 2H), 7.34 – 7.28 (m, 5H), 7.22 – 7.17 (m, 1H), 7.16 – 7.09 (m, 2H), 4.03 – 3.89 (m, 2H), 2.36 (ddd, *J* = 9.7, 7.5, 6.2 Hz, 1H), 1.95 (dt, *J* = 9.1, 4.9 Hz, 1H), 1.41 – 1.31 (m, 2H), 0.97 (dt, *J* = 8.6, 5.2 Hz, 1H), 0.86 (dt, *J* = 8.7, 5.4 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.8, 141.6, 128.7, 128.4, 128.0, 126.9, 125.7, 125.6, 67.3, 53.2, 25.8, 23.8, 14.1.

5.2 Vinylation of **2a.**

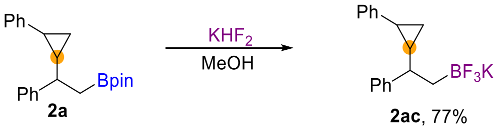


The title compound **2ab** was synthesized according to the following literature[2]: a solution of **2a** (0.1 mmol) in THF (2 mL) was added to an oven-dried round bottom flask containing a stirring bar. Vinylmagnesium bromide (1 M, 0.4 mL, 4.0 equiv.) was added dropwise to the mixture. The mixture was stirred at room temperature for 0.5 h. Then the flask was cooled to -78 ℃. I_2 (2.0 equiv.) in methanol (3.0 mL) was added dropwise. The reaction mixture was allowed to stir 0.5 h at the same temperature, followed by the dropwise addition of a solution of NaOMe (8.0 equiv.) in methanol (3 mL). Then the reaction mixture was warmed to room temperature and stirred for another 1.5 h, diluted with EtOAc (10 mL), and washed sequentially with 10% aqueous solution of $Na_2S_2O_3$ (5 mL). Then, the mixture was extracted with EtOAc (10 mL), dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel to afford the corresponding product **2ab** as a colorless oil.

(2-(1-Phenylbut-3-en-1-yl)cyclopropyl)benzene (2ab)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.01 (m, 10H), 5.66 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 4.97 – 4.81 (m, 2H), 2.56 – 2.42 (m, 2H), 2.17 – 2.03 (m, 1H), 1.75 (dt, *J* = 9.1, 4.9 Hz, 1H), 1.26 (d, *J* = 3.5 Hz, 1H), 0.85 – 0.71 (m, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 144.5, 143.3, 136.8, 128.3, 128.3, 127.6, 126.2, 125.7, 125.4, 116.0, 51.0, 41.0, 29.5, 24.3, 14.9.

5.3 BF_3K preparation.



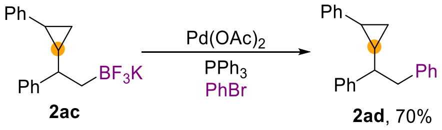
The title compound **2ac** was prepared according to the following literature[3]: a solution of **2a** (0.1 mmol) and KHF_2 (7 equiv.) in MeOH (2 mL) was added to an oven-dried round bottom flask containing a stirring bar. The reaction mixture was stirred vigorously for 3 h at room temperature. The resulting slurry was stirred concentrated, then placed under high vacuum. The dried solids were triturated with hot acetone and filtered to remove inorganic salts. The resulting filtrate was concentrated to a minimal volume and wash with *n*-pentane (5 x 2 mL) was added to afford **2ac** as a white solid. (There is still some **2a** that has not reacted completely, and acetone cannot wash it off completely. Therefore, there is still about 23% of the **2a**)

Trifluoro(2-phenyl-2-(2-phenylcyclopropyl)ethyl)-λ⁴-borane, potassium salt (2ac)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.22 (m, 6H), 7.12 – 7.08 (m, 4H), 2.38 (td, *J* = 9.3, 6.1 Hz, 1H), 2.20 – 2.09 (m, 2H), 1.92 – 1.78 (m, 2H), 0.82 – 0.80 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 146.4, 143.6, 128.4, 128.2, 128.2, 127.5, 126.0, 125.7, 46.2, 31.8, 24.8, 24.6, 23.9, 15.8.

5.4 Suzuki–Miyaura Reaction of **2ad.**



The title compound **2ad** was prepared according to a modified methods[3]: A 4 mL screw-cap vial with a magnetic stir bar was charged was charged with $Pd(OAc)_2$ (1 mol %), RuPhos (1 mol %), K_2CO_3 (3 equiv.), and **2ac** (0.12 mmol). The vial was closed with a Teflon septum and cap and connected to the atmosphere via a needle. Toluene (0.5 mL) was added, followed by H_2O (50 μL), and bromobenzene (1 equiv.). The vial was quickly exchanged for a Teflon-lined screw cap and the reaction was stirred at 80 ℃ for 24 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 Na2SO4 and then concentrated in vacuo. The crude product was purified by column chromatography on silica gel (*n*-pentane/EA = 100/1) to afford the corresponding product **2ad** as a colorless oil.

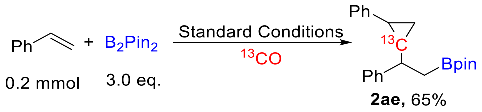
(1-(2-Phenylcyclopropyl)ethane-1,2-diyl)dibenzene (2ad)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.02 (m, 12H), 6.91 – 6.88 (m, 1H), 6.86 – 6.77 (m, 2H), 3.07 – 2.90 (m, 2H), 2.28 (dt, *J* = 9.4, 7.3 Hz, 1H), 1.50 (dt, *J* = 9.0, 4.9 Hz, 1H), 1.29 – 1.24 (m, 1H), 0.72 (ddt, *J* = 29.6, 8.6, 5.2 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 144.7, 143.2, 140.3, 129.3, 128.3, 128.1, 128.1, 127.7, 126.3, 125.8, 125.7, 125.3, 53.1, 43.5, 29.5, 24.6, 15.0.

6. Mechanism studies.

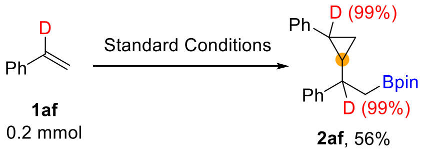
6.1 ^{13}C labeling experiment.



A dried vial (4 mL) was charged with Cu(OTf)$_2$ (12.5 mol%), DPPB (12.5 mol%), B$_2$pin$_2$ (3.0 equiv.), NaO^{t}Bu (3.0 equiv.), and a stirring bar. The vial was sealed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, styrene (0.2 mmol) and toluene (1.0 mL) were added under argon by using a syringe. The vial (or several vials) was placed in an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. The autoclave was charged with ^{13}CO (1 bar) and CO (9 bar) after flushing two times with N$_2$ and two times with CO. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction was performed for 20 h at 90 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After that the residue was directly purified by column chromatography to afford the corresponding products **2ae**.

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.20 – 6.99 (m, 10H), 2.31 (ddt, *J* = 12.6, 6.2, 3.2 Hz, 1H), 1.81 (dt, *J* = 8.8, 5.0 Hz, 1H), 1.34 – 1.25 (m, 3H), 0.98 (d, *J* = 10.1 Hz, 12H), 0.79 – 0.71 (m, 2H).
^{13}C NMR (75 MHz, CDCl$_3$) δ 146.4, 143.6, 128.2, 128.1, 127.4, 126.0, 126.0, 125.3, 83.0, 46.1 (t, *J* = 21.13 Hz), 31.8, 24.7, 24.5, 23.8 (t, *J* = 6.0 Hz), 15.8 (t, *J* = 6.8 Hz), 15.7.
^{11}B NMR (96 MHz, CDCl$_3$) δ 33.6.
(By the result of this reaction, we confirm that the carbon at the *β*-position of the benzene ring comes from this carbon monoxide.)

6.2 Deuterated Styrene as starting material.



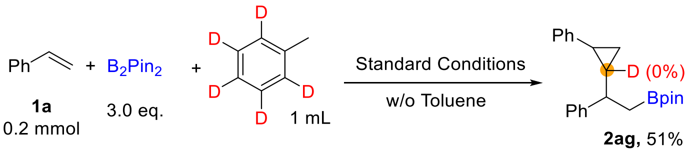
A dried vial (4 mL) was charged with Cu(OTf)$_2$ (12.5 mol%), DPPB (12.5 mol%), B$_2$pin$_2$ (3.0 equiv.), NaO^{t}Bu (3.0 equiv.), and a stirring bar. The vial was sealed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, **1af** (0.2 mmol) and toluene (1.0 mL) were added under argon by using a syringe. The vial (or several vials) was placed in an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. The autoclave was charged with CO (10 bar) after flushing two times with N$_2$ and two times with CO. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction was performed for 20 h at 90 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After that the residue was directly purified by column chromatography to afford the corresponding products **2af**.

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.28 – 7.23 (m, 5H), 7.21 – 7.03 (m, 5H), 1.36 (dd, *J* = 6.8, 2.3 Hz, 3H), 1.06 (s, 6H), 1.02 (s, 6H), 0.81 (dd, *J* = 9.0, 7.1 Hz, 2H).

^{11}B NMR (96 MHz, CDCl$_3$) δ 32.9.

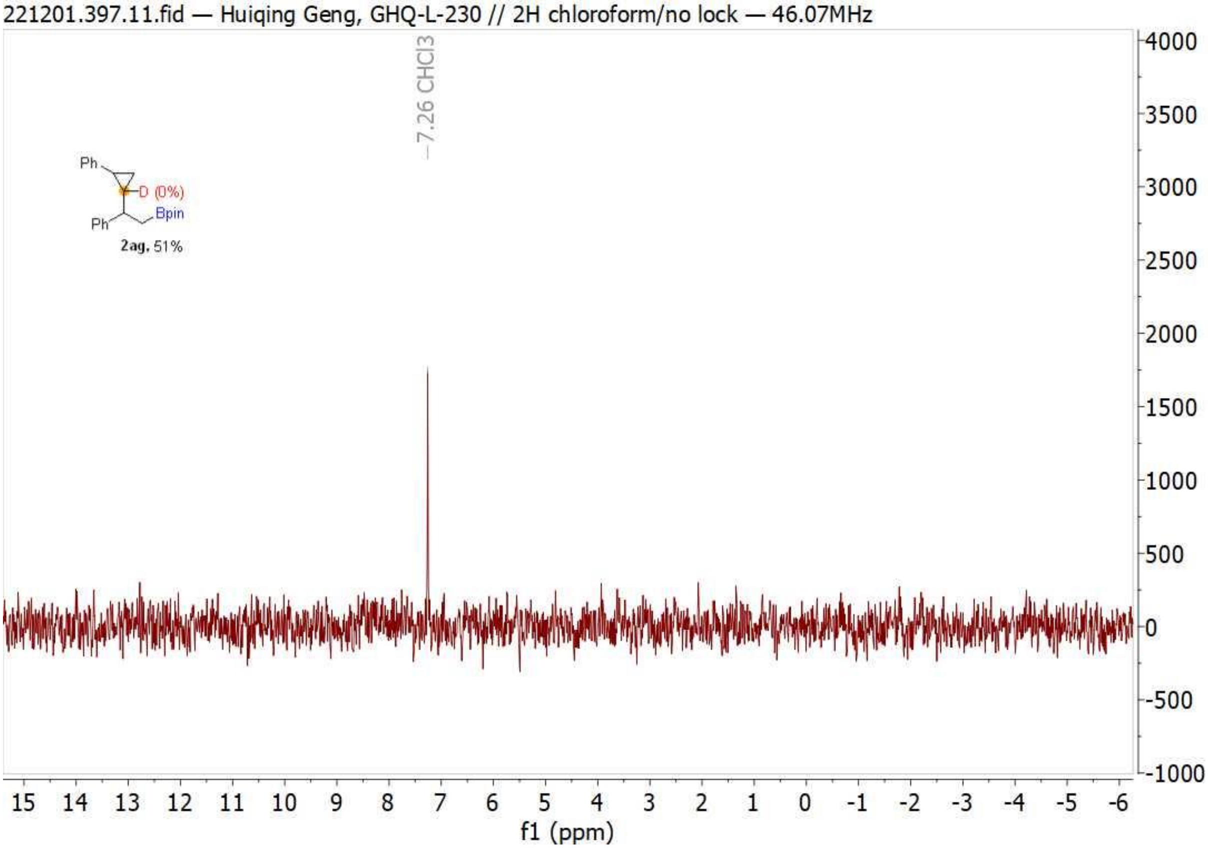
(Compared with the 1H NMR of standard product **2a**, the hydrogen in the *α*-position of styrene does not undergo intramolecular transfer.)

6.3 Deuterated solvent.

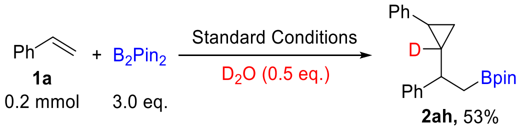


A dried vial (4 mL) was charged with Cu(OTf)$_2$ (12.5 mol%), DPPB (12.5 mol%), B$_2$pin$_2$ (3.0 equiv.), NaO^{t}Bu (3.0 equiv.), and a stirring bar. The vial was sealed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, **1a** (0.2 mmol) and toluene-D$_5$ (1.0 mL) were added under argon by using a syringe. The vial (or several vials) was placed in an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. The autoclave was charged with CO (10 bar) after flushing two times with N$_2$ and two times with CO. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction was performed for 20 h at 90 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After that the residue was directly purified by column chromatography to afford the corresponding products **2ag**.

^{1}H NMR (300 MHz, CDCl$_3$) δ 7.22 – 7.16 (m, 5H), 7.16 – 6.99 (m, 5H), 2.32 (td, *J* = 9.3, 6.2 Hz, 1H), 1.97 – 1.72 (m, 1H), 1.33 – 1.20 (m, 3H), 0.98 (d, *J* = 10.1 Hz, 12H), 0.81 – 0.71 (m, 2H).
^{13}C NMR (75 MHz, CDCl$_3$) δ 146.4, 143.6, 128.2, 128.1, 127.4, 126.0, 126.0, 125.2, 83.0, 46.1, 31.8, 24.7, 24.5, 23.8, 15.8.
^{11}B NMR (96 MHz, CDCl$_3$) δ 33.2.
(No peaks of deuterium atoms were found in ^{2}H NMR spectra of **2ag**, proving that the hydrogen at the β-position of the benzene ring does not come from toluene.)

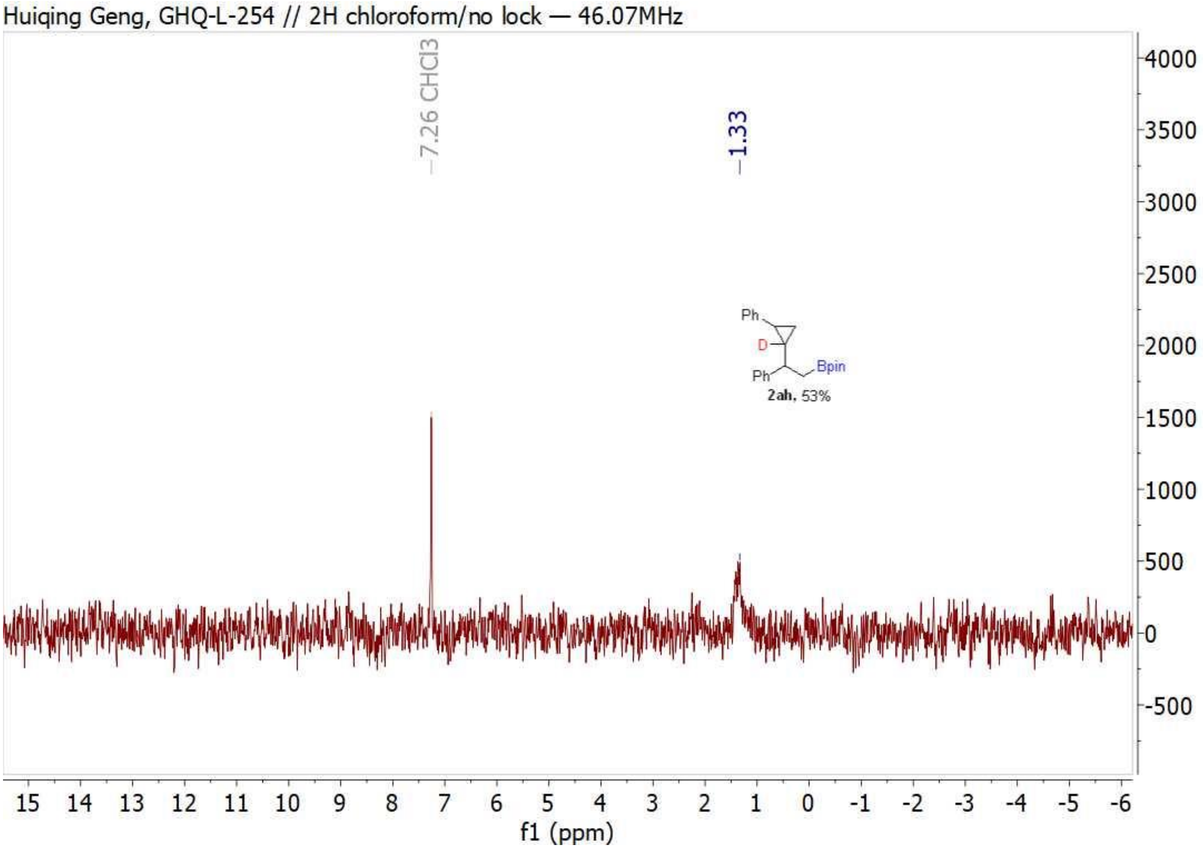


6.4 Additional deuterium water.



A dried vial (4 mL) was charged with $Cu(OTf)_2$ (12.5 mol%), DPPB (12.5 mol%), B_2pin_2 (3.0 equiv.), NaO^tBu (3.0 equiv.), and a stirring bar. The vial was sealed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. The vial was evacuated under vacuum and recharged with argon for three times. Then, **1a** (0.2 mmol) and toluene (1.0 mL) and D_2O (1.8 μL, 0.5 equiv.) were added under argon by using a syringe. The vial (or several vials) was placed in an alloy plate and transferred into a 300 mL autoclave of the 4560 series from Parr Instruments. The autoclave was charged with CO (10 bar) after flushing two times with N_2 and two times with CO. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction was performed for 20 h at 90 °C. After the reaction was complete, the autoclave was cooled down with ice water to room temperature and the pressure was released carefully. After that the residue was directly purified by column chromatography to afford the corresponding products **2ah**.

(The hydrogen at *β*-position overlaps with other alkyl region hydrogens in the ^1H NMR spectra, so the specific deuteration content cannot be seen qualitatively. In this control reaction, we added about Volumetric ratio (500:1) of solvent to D_2O and obtain 53% of the product **2ah**. In the ^2H NMR spectra, we can see that the deuterium peak. This result allowed us to qualitatively determine that the hydrogen at the *β*-position of the benzene ring comes from the trace amount of water present in the solvent.)



7. References.

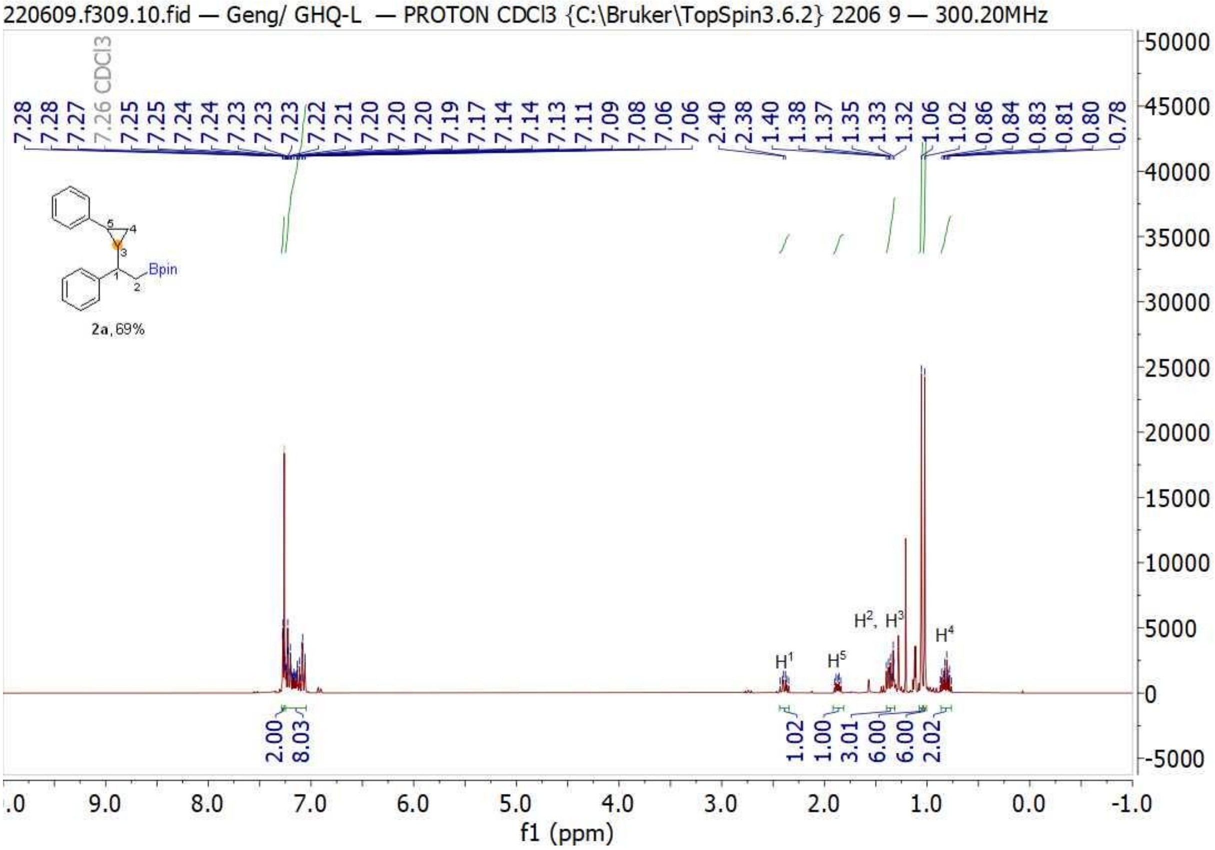
(1) Lee, J.-E.; Yun, J. Catalytic Asymmetric Boration of Acyclic *α, β*-Unsaturated Esters and Nitriles. *Angew. Chem. Int. Ed.* **2008**, *47*, 145-147.

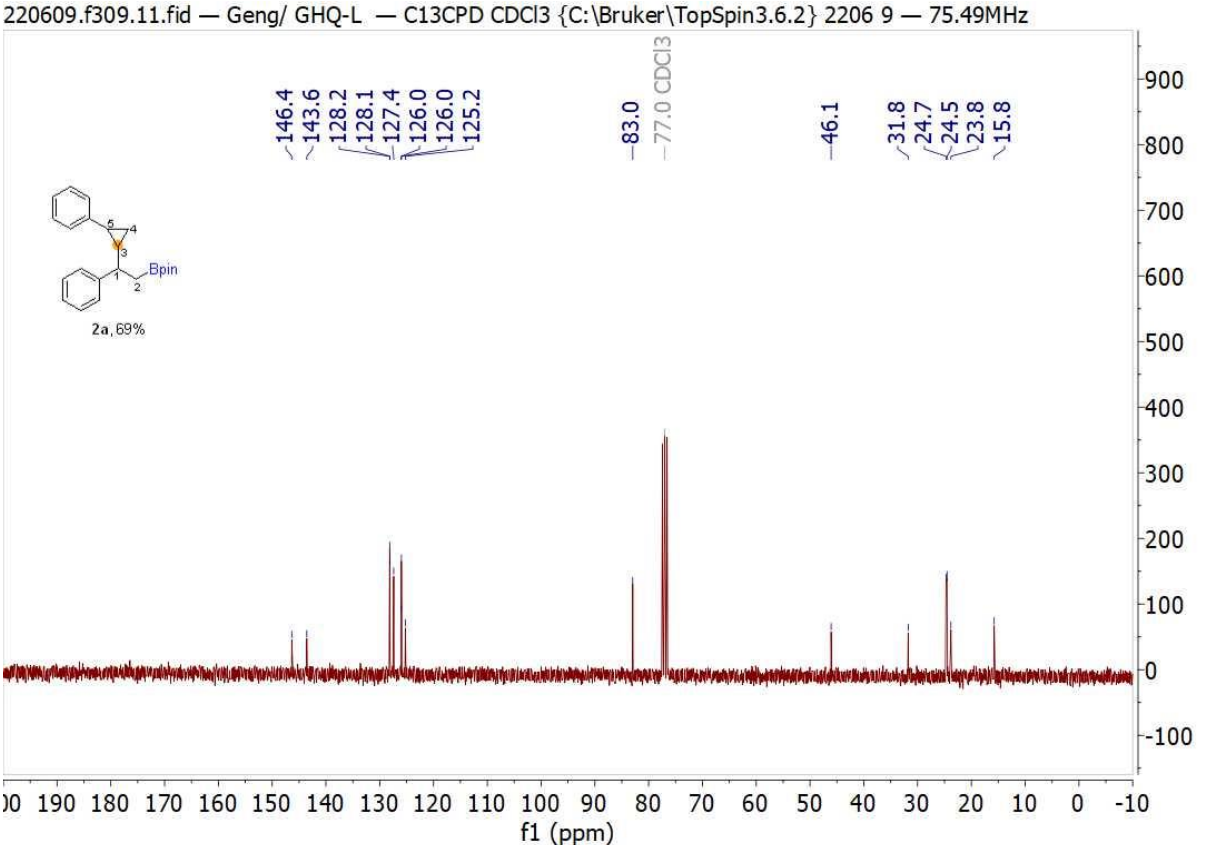
(2) Sun S.-Z.; Martin R. Nickel-Catalyzed Umpolung Arylation of Ambiphilic *α*-Bromoalkyl Boronic Esters. *Angew. Chem. Int. Ed.* **2018**, *57*, 3684-3687

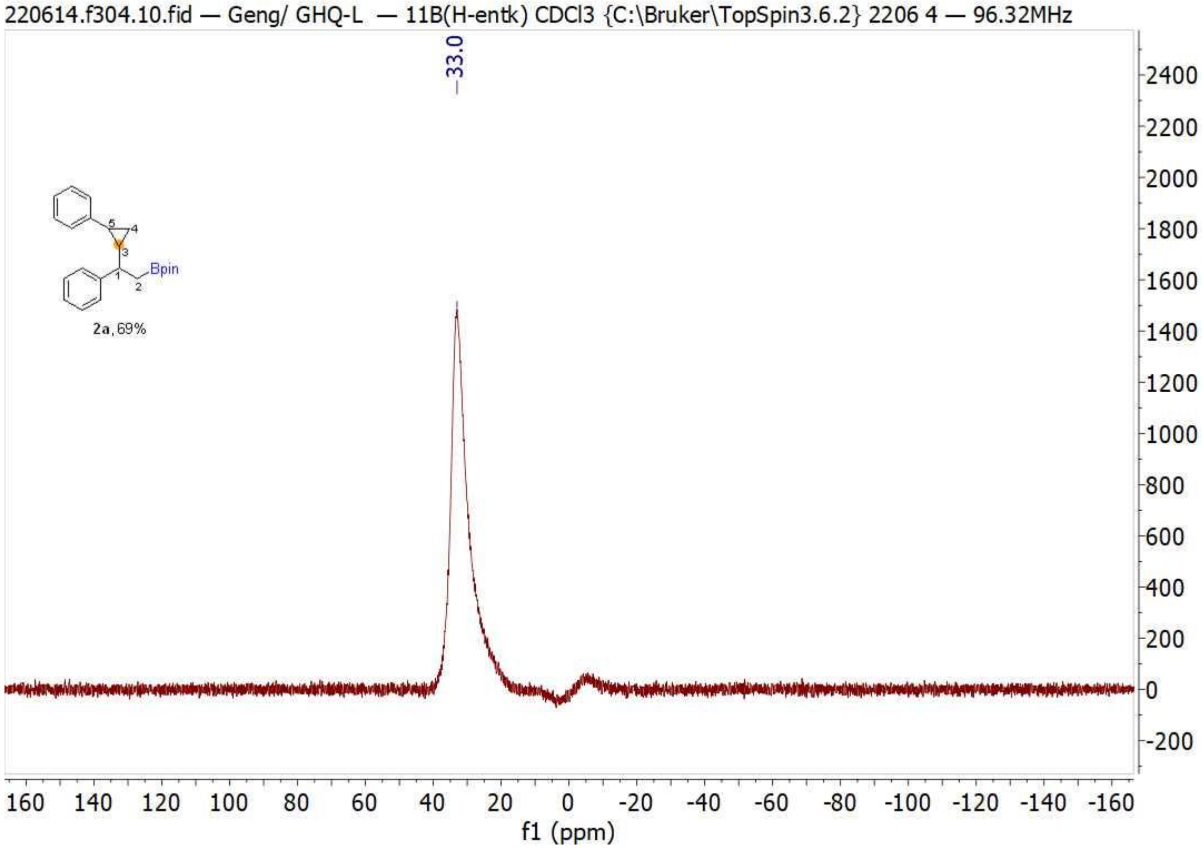
(3) Fang, G.-H.; Yan, Z.-J.; Deng, M.-Z. Palladium-Catalyzed Cross-Coupling of Stereospecific Potassium Cyclopropyl Trifluoroborates with Aryl Bromides. *Org. Lett*. **2004**, *6*, 357–360.

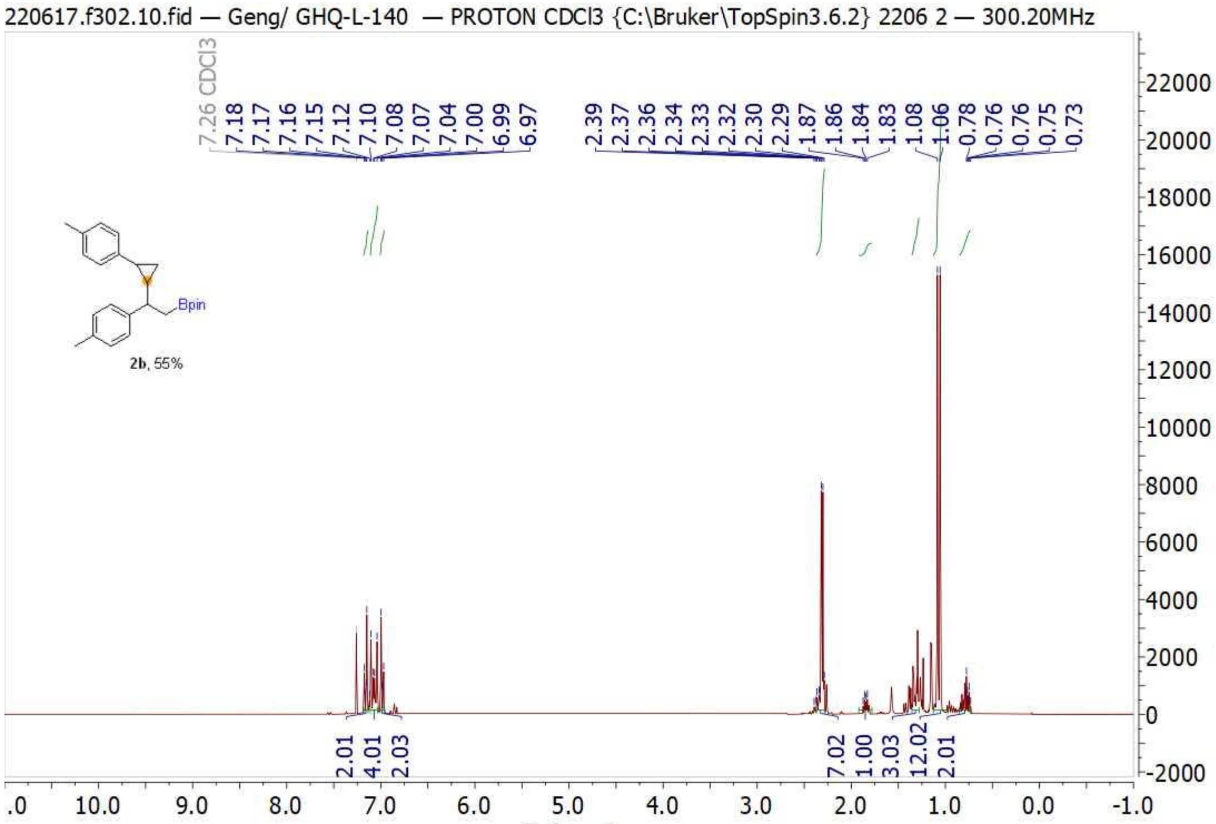
(4) Dreher, S. D.; Lim, S.-E.; Sandrock, D. L.; Molander, G. A. Suzuki–Miyaura Cross-Coupling Reactions of Primary Alkyltrifluoroborates with Aryl Chlorides. *J. Org. Chem*. **2009**, *74*, 3626–3631.

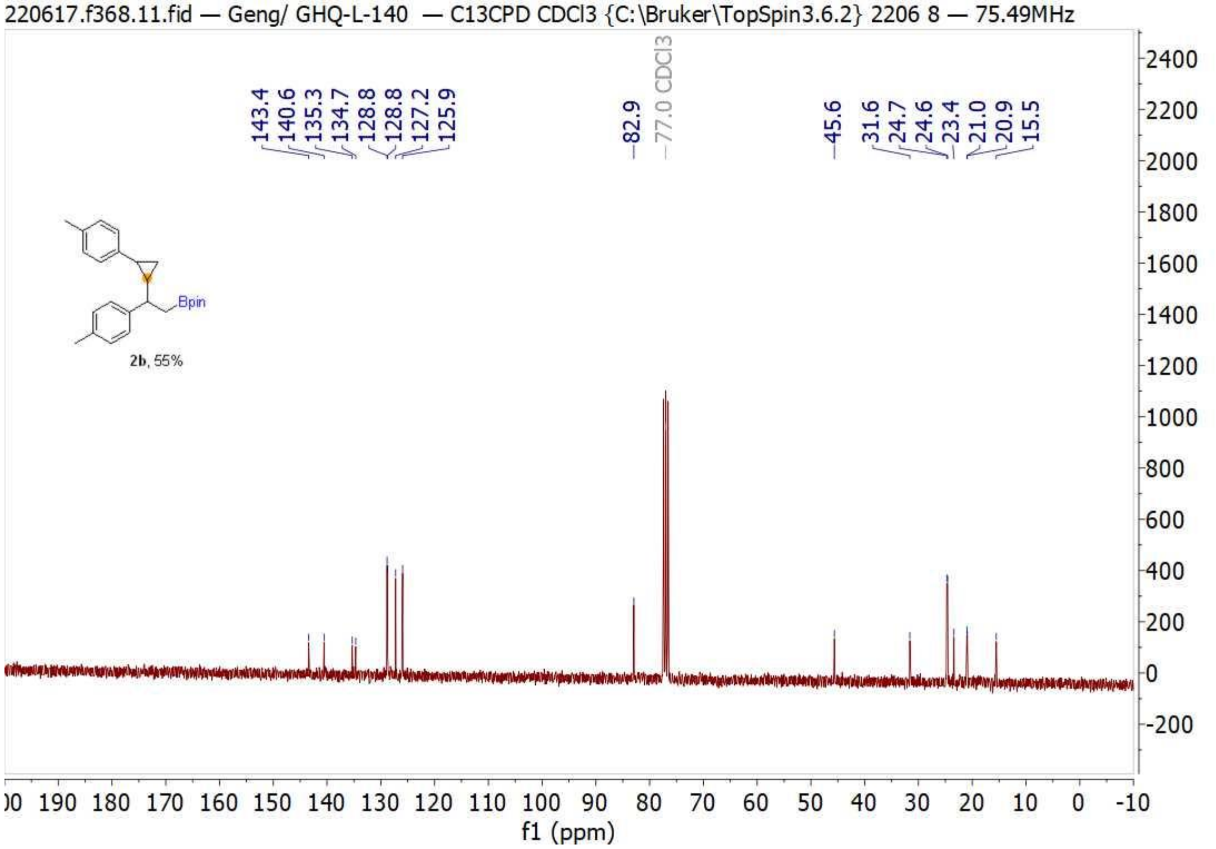
8. Spectra Copies.

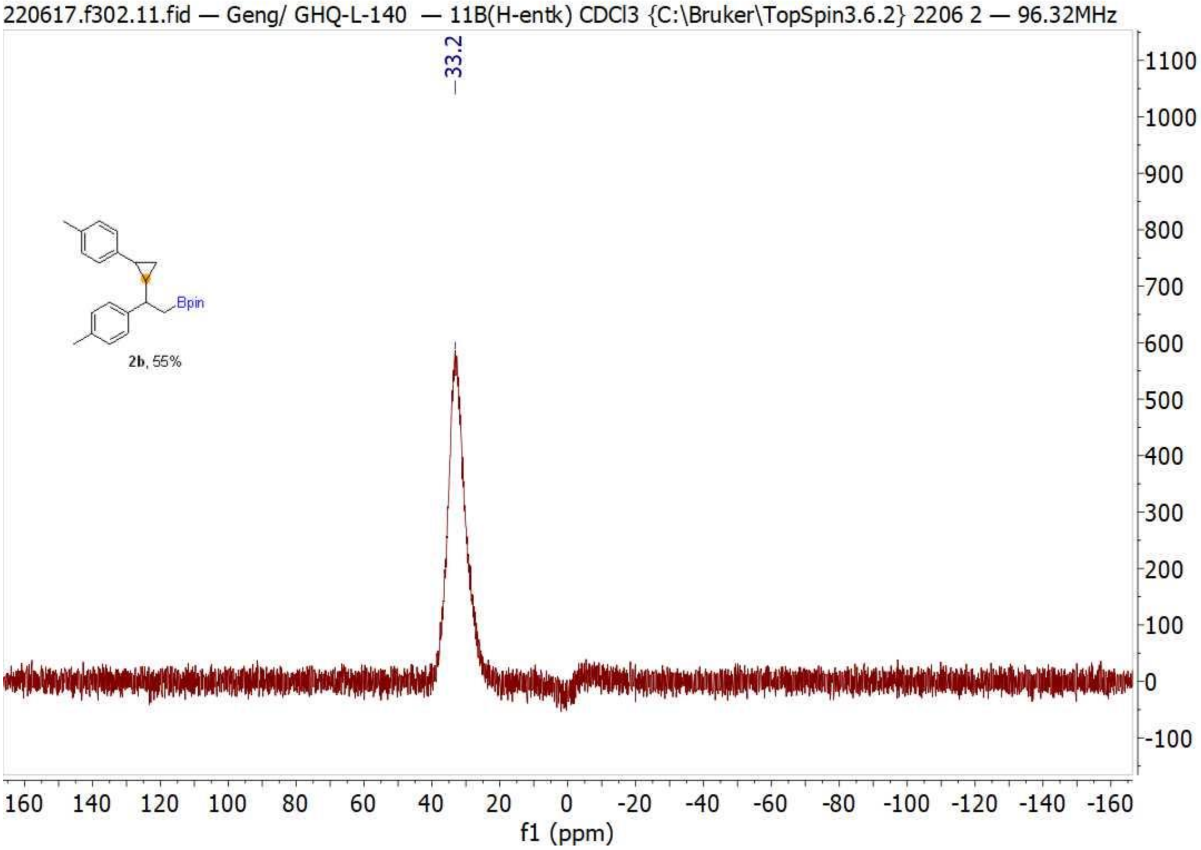




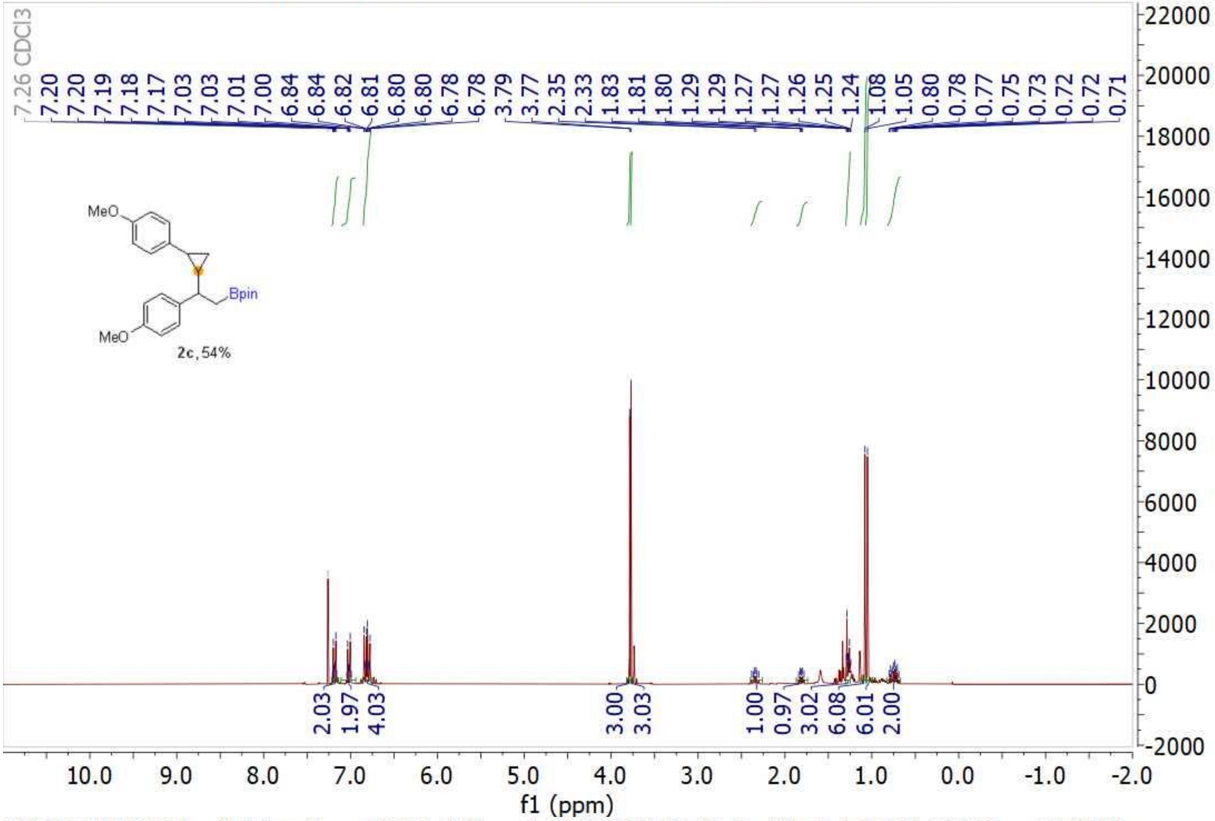

220614.f304.10.fid — Geng/ GHQ-L — 11B(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 4 — 96.32MHz
33.0
5
4
3
1
2
Bpin
2a, 69%
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
f1 (ppm)


220617.f302.10.fid — Geng/ GHQ-L-140 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 2 — 300.20MHz
7.26 CDCl3
7.18
7.17
7.16
7.15
7.12
7.10
7.08
7.07
7.04
7.00
6.99
6.97
2.39
2.37
2.36
2.34
2.33
2.32
2.30
2.29
1.87
1.86
1.84
1.83
1.08
1.06
0.78
0.76
0.76
0.75
0.73
Bpin
2b, 55%
2.01
4.01
2.03
7.02
1.00
3.03
12.02
2.01
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

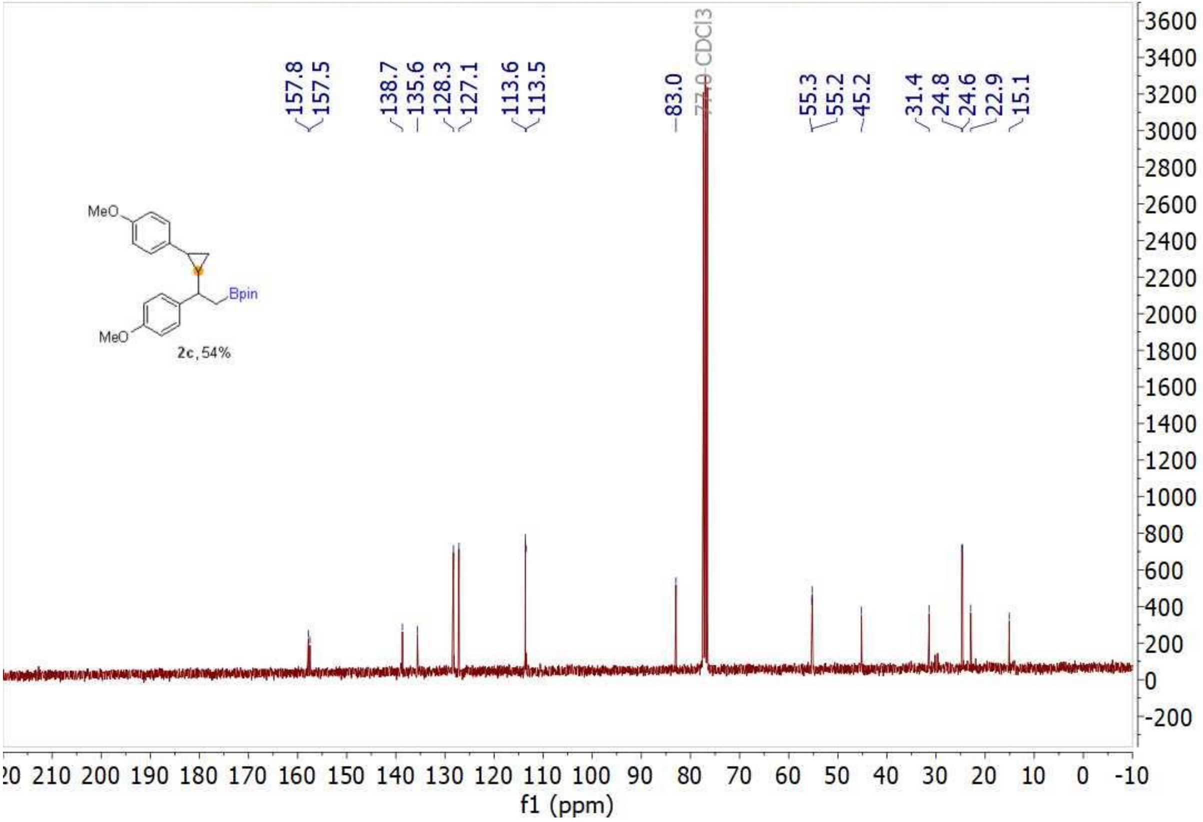

220617.f368.11.fid — Geng/ GHQ-L-140 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 8 — 75.49MHz
143.4
140.6
135.3
134.7
128.8
128.8
127.2
125.9
82.9
77.0 CDCl3
45.6
31.6
24.7
24.6
23.4
21.0
20.9
15.5
Bpin
2b, 55%
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

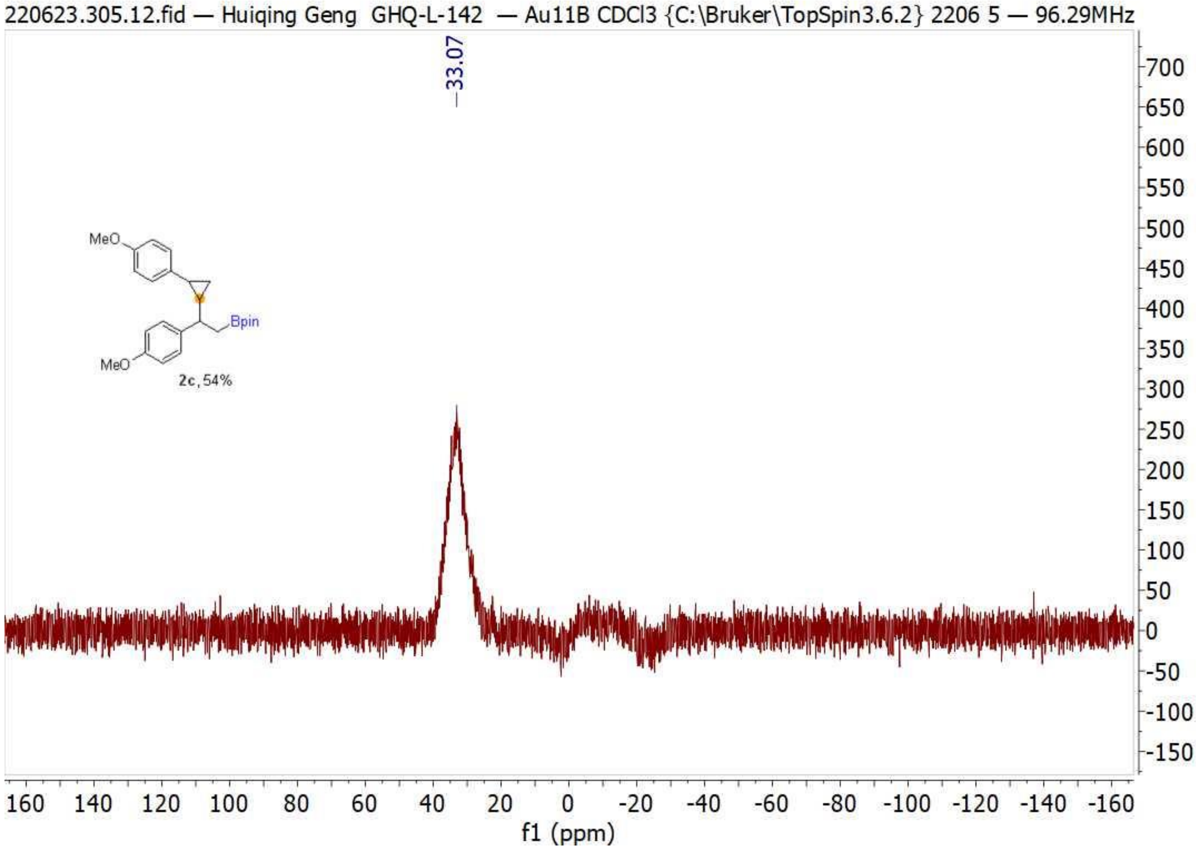

220617.f302.11.fid — Geng/ GHQ-L-140 — 11B(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 2 — 96.32MHz
33.2
Bpin
2b, 55%
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
f1 (ppm)

220622.f318.10.fid — Huiqing Geng GHQ-L-142 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 18 — 300.20MHz

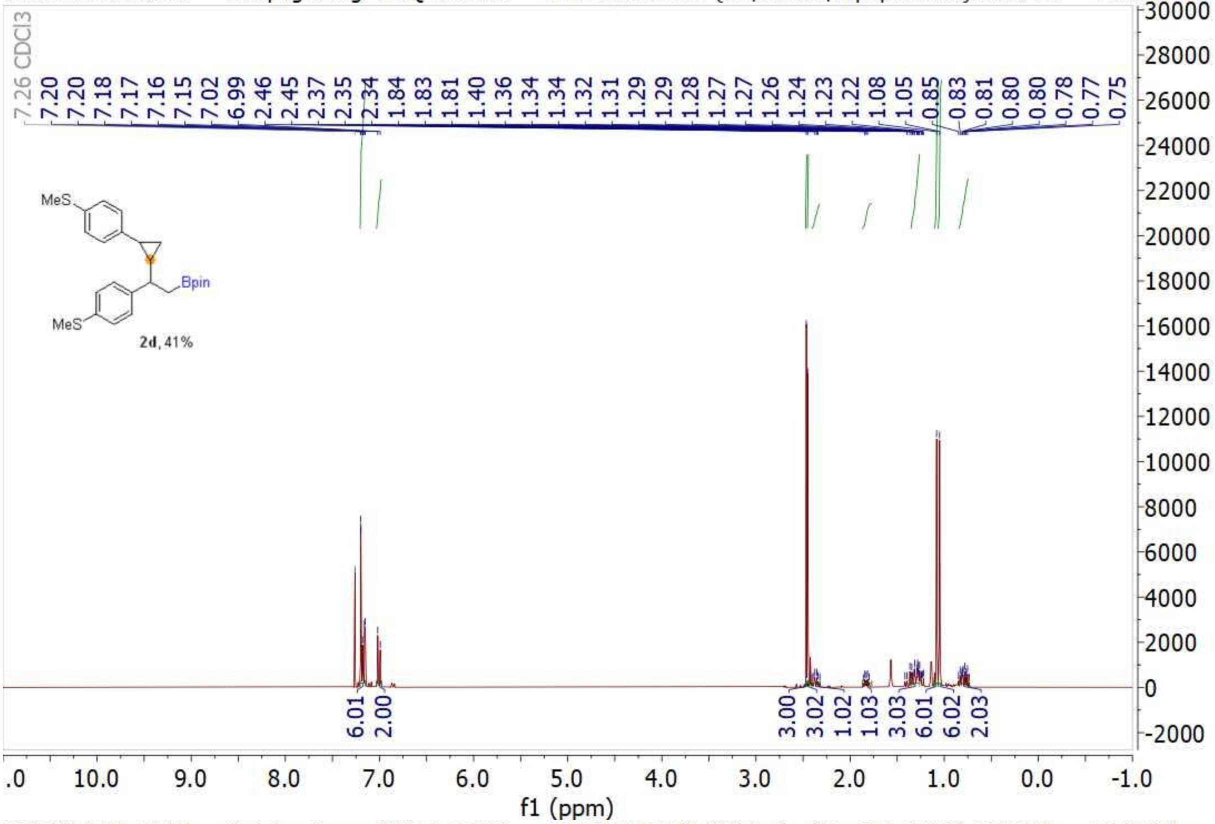


220623.305.11.fid — Huiqing Geng GHQ-L-142 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 5 — 75.48MHz

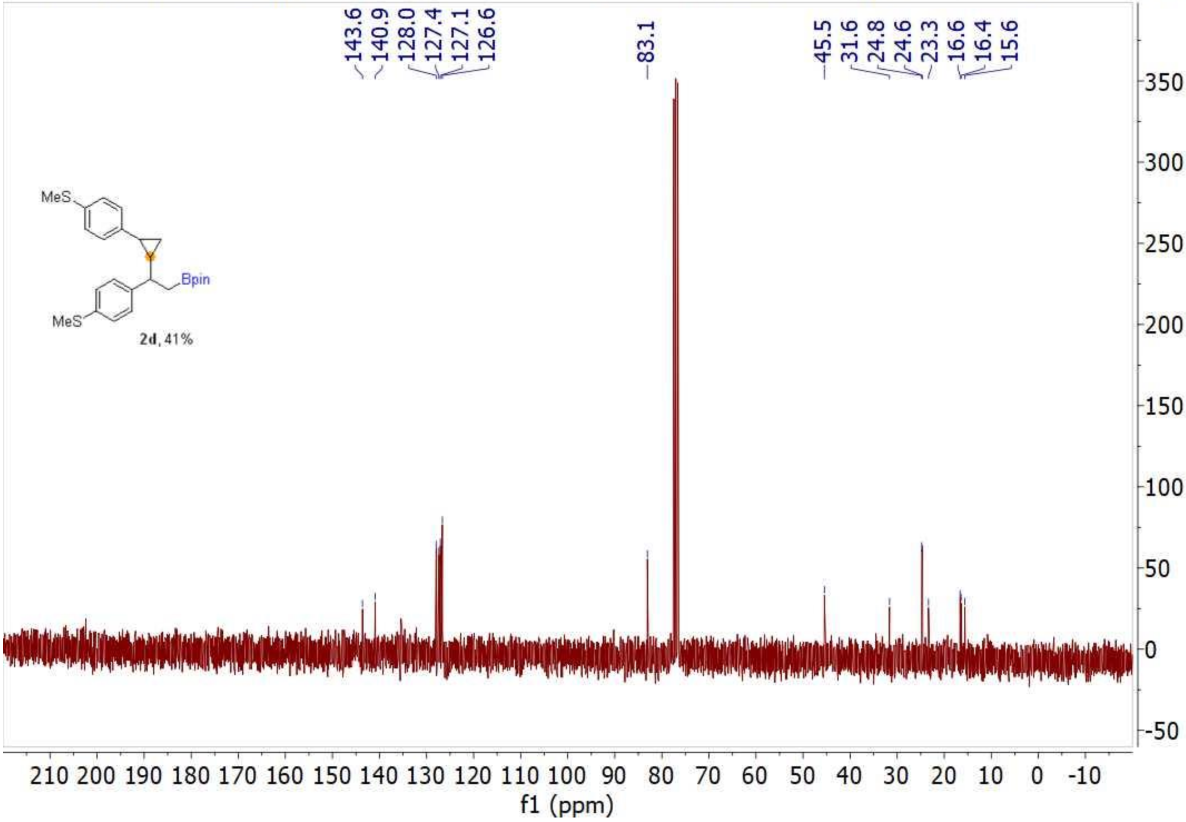


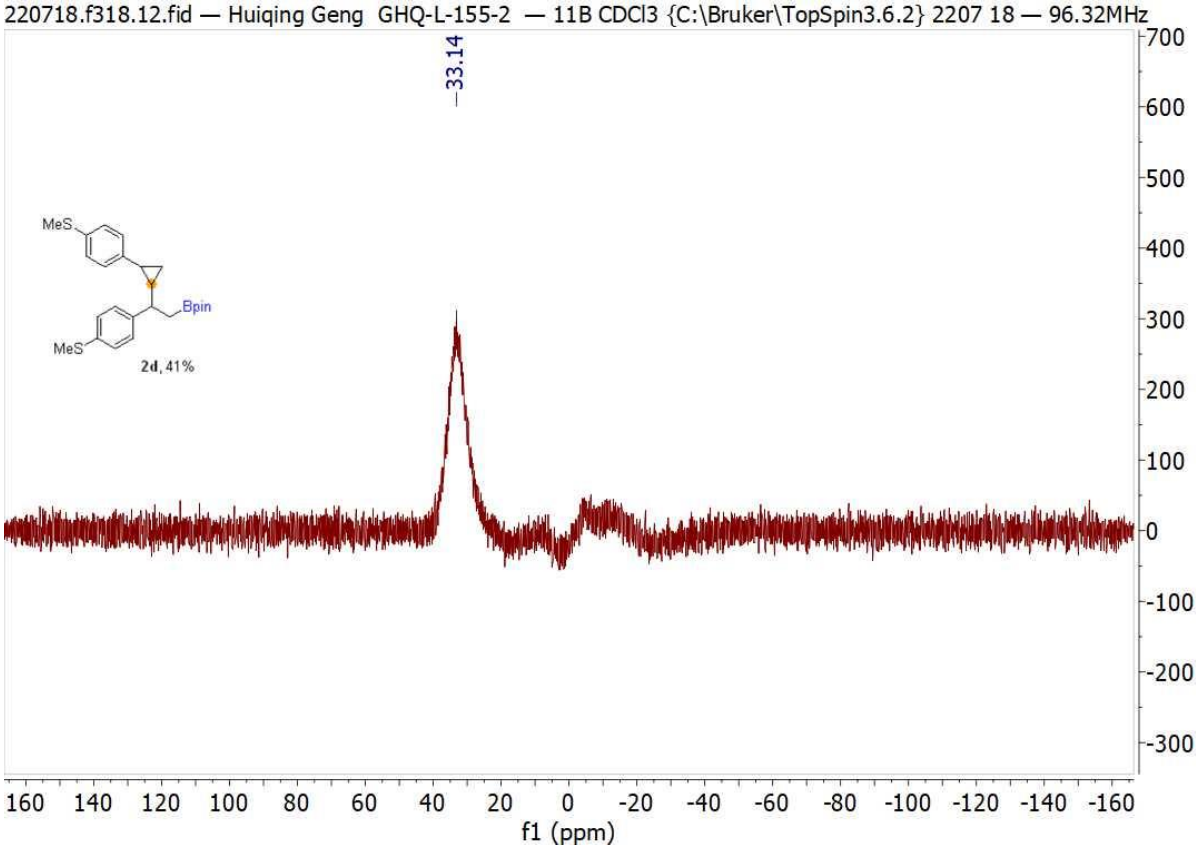

220623.305.12.fid — Huiqing Geng GHQ-L-142 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 5 — 96.29MHz
33.07
MeO
Bpin
MeO
2c, 54%
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
-100
-150
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
f1 (ppm)

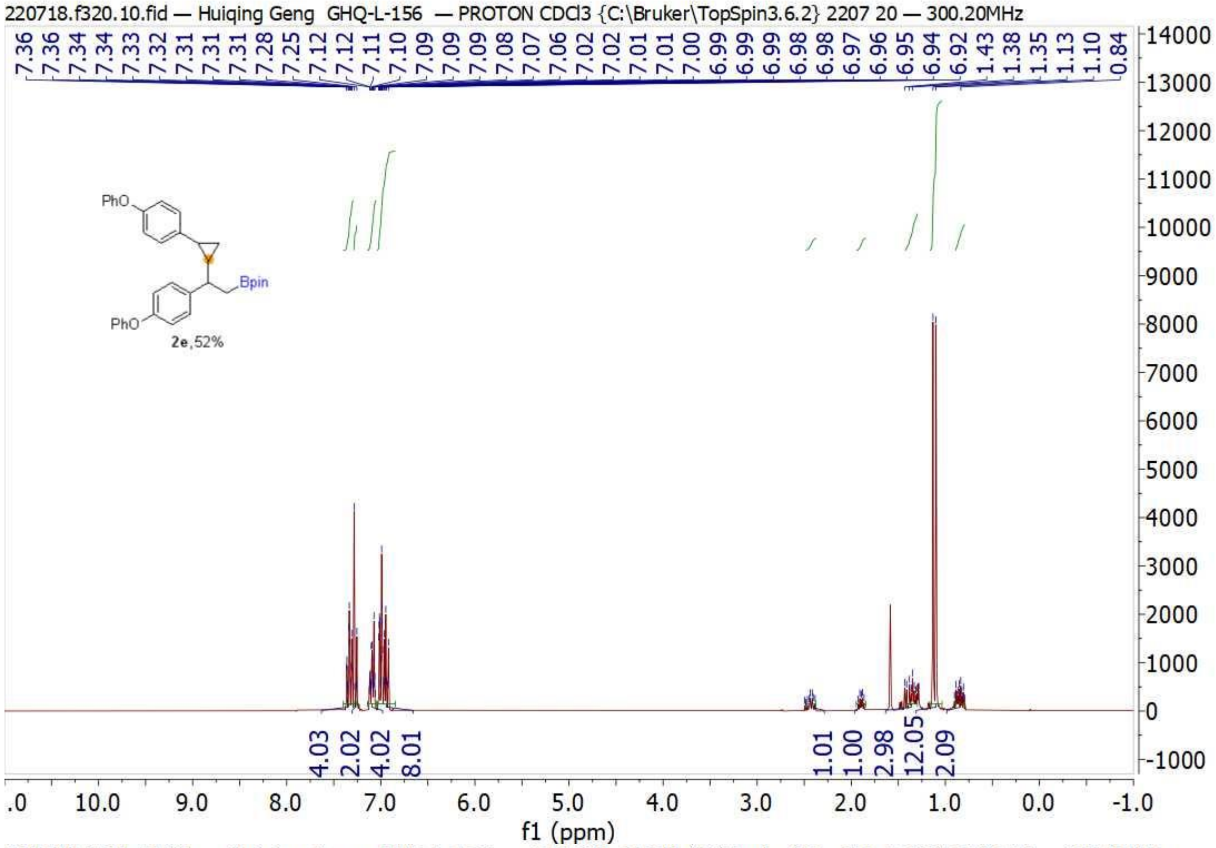
220718.f318.10.fid — Huiqing Geng GHQ-L-155-2 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 18 — 300.20MHz

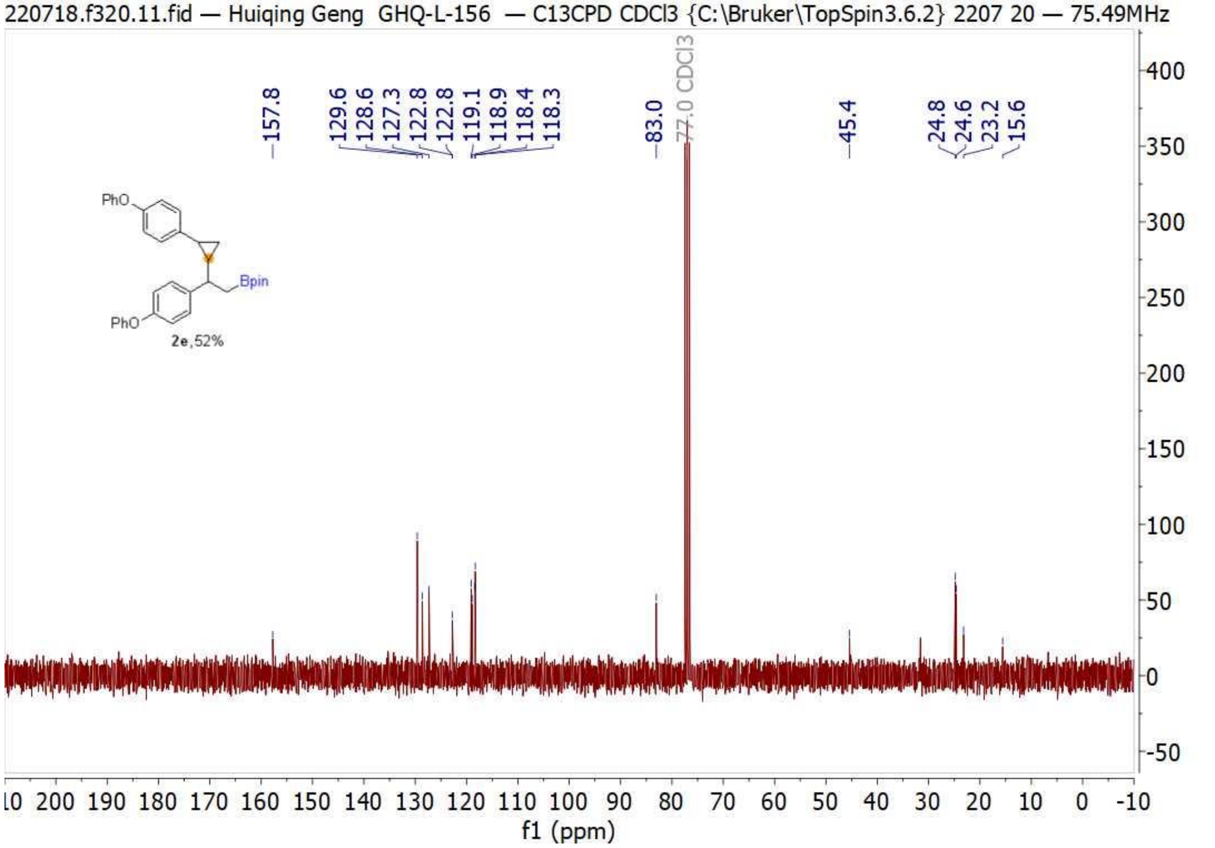


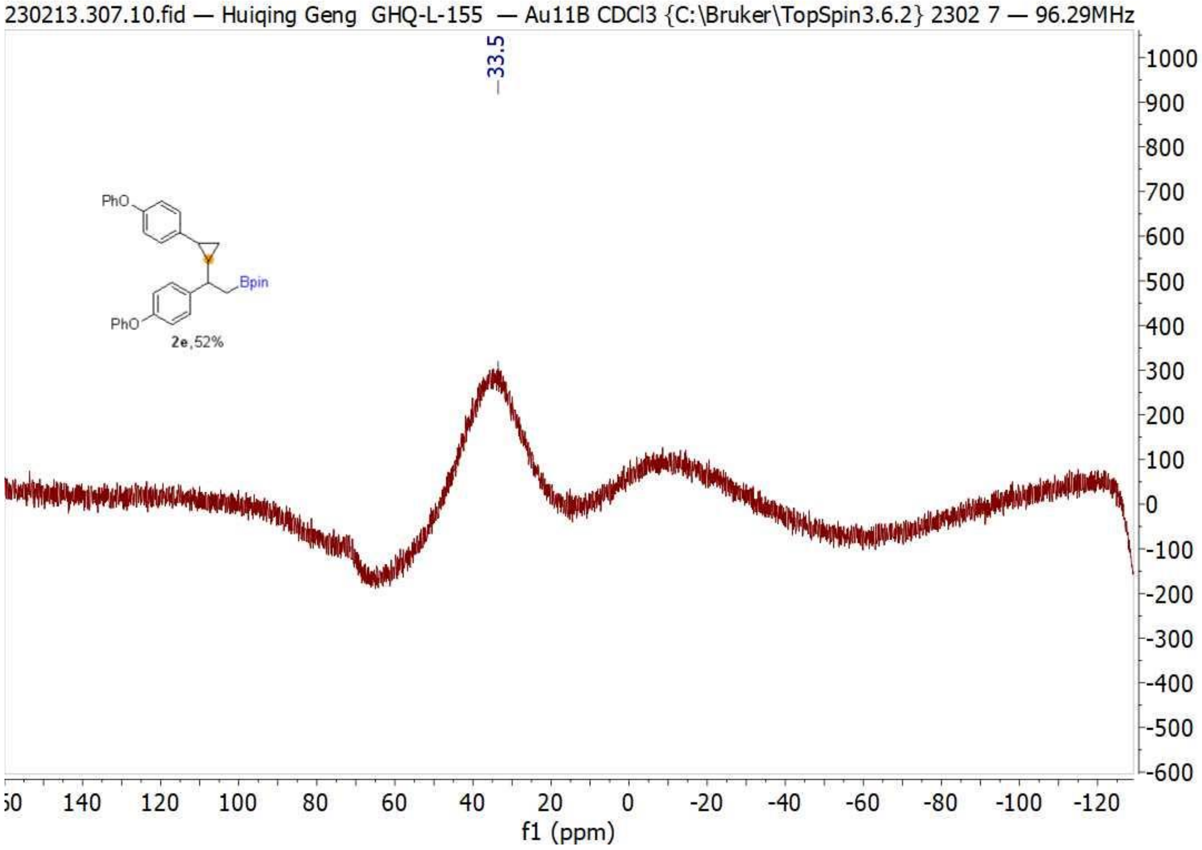
220718.f318.11.fid — Huiqing Geng GHQ-L-155-2 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 18 — 75.49MHz

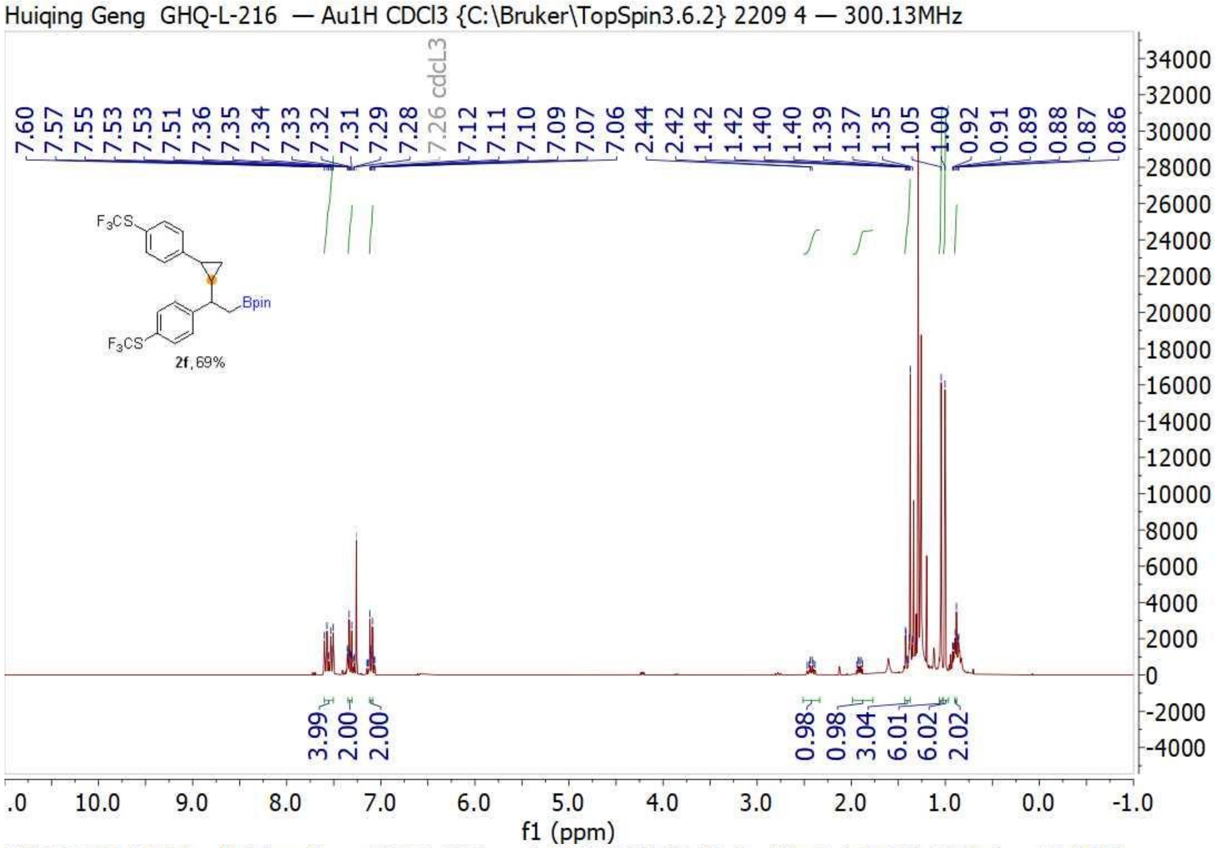


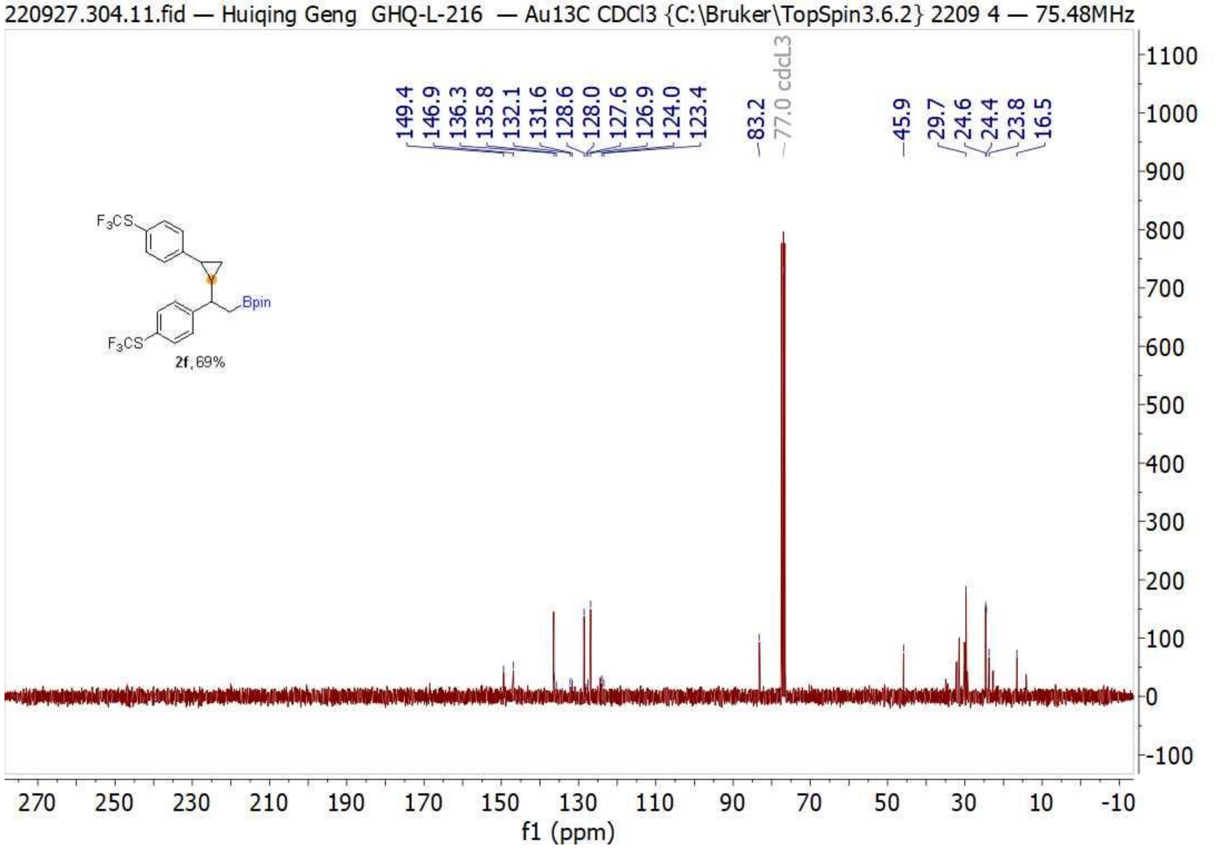

220718.f318.12.fid — Huiqing Geng GHQ-L-155-2 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 18 — 96.32MHz
—33.14
MeS
Bpin
MeS
2d, 41%
700
600
500
400
300
200
100
0
-100
-200
-300
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
f1 (ppm)

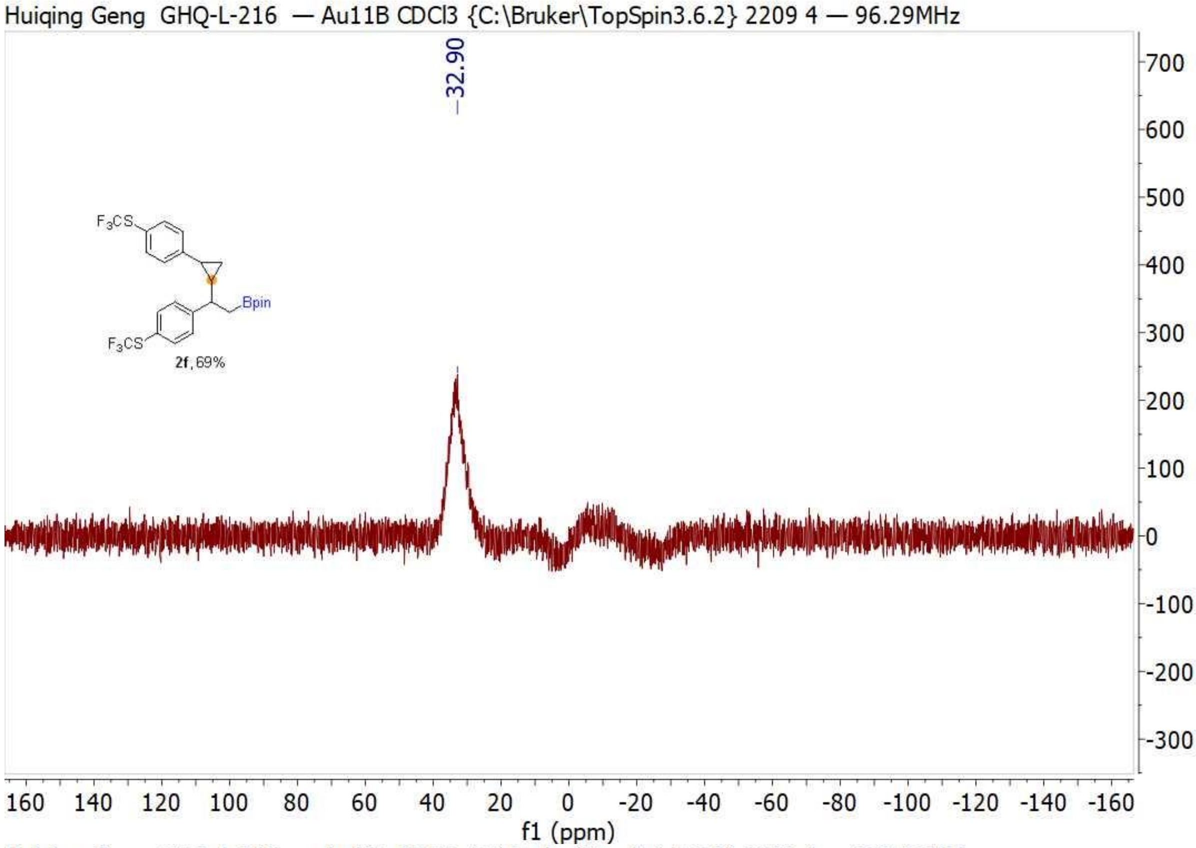

220718.f320.10.fid — Huiqing Geng GHQ-L-156 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 20 — 300.20MHz
7.36 7.36 7.34 7.34 7.33 7.32 7.31 7.31 7.31 7.28 7.25 7.12 7.12 7.11 7.10 7.09 7.09 7.09 7.08 7.07 7.06 7.02 7.02 7.01 7.01 7.00 6.99 6.99 6.99 6.98 6.98 6.97 6.96 6.95 6.94 6.92 1.43 1.38 1.35 1.13 1.10 0.84
PhO
Bpin
PhO
2e,52%
4.03 2.02 4.02 8.01
1.01 1.00 2.98 12.05 2.09
14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000
.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

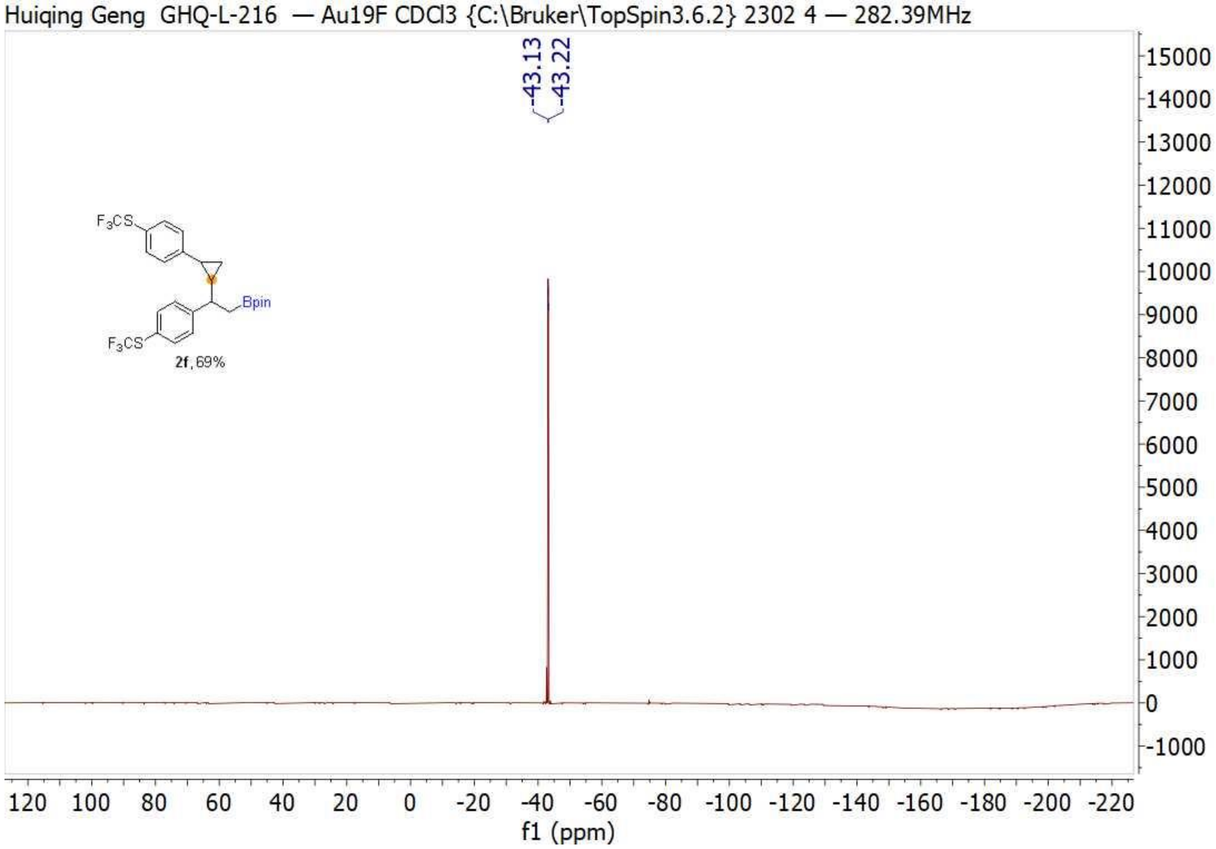

220718.f320.11.fid — Huiqing Geng GHQ-L-156 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 20 — 75.49MHz
157.8
129.6 128.6 127.3 122.8 122.8 119.1 118.9 118.4 118.3
83.0
77.0 CDCl3
45.4
24.8 24.6 23.2 15.6
PhO
Bpin
PhO
2e,52%
400 350 300 250 200 150 100 50 0 -50
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

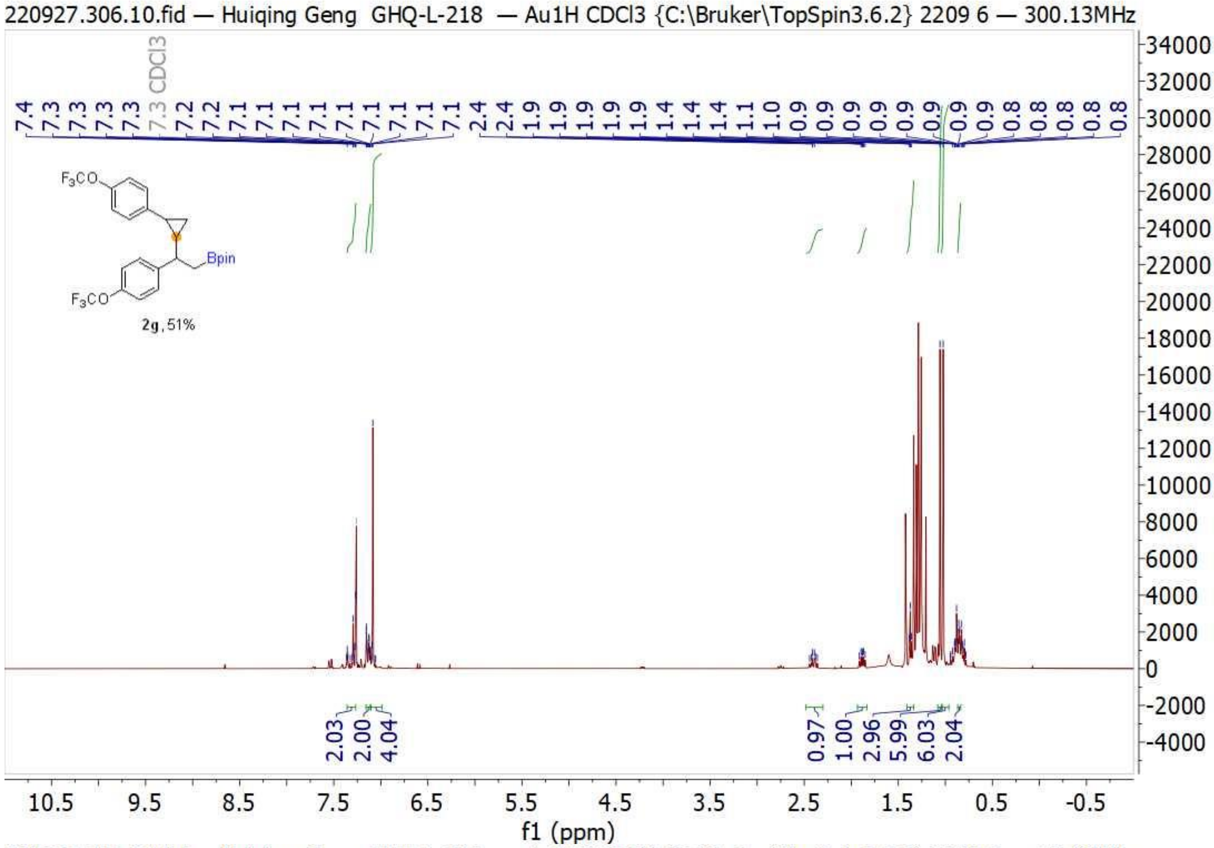

230213.307.10.fid — Huiqing Geng GHQ-L-155 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2302 7 — 96.29MHz
—33.5
PhO
Bpin
PhO
2e, 52%
1000
900
800
700
600
500
400
300
200
100
0
-100
-200
-300
-400
-500
-600
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
f1 (ppm)

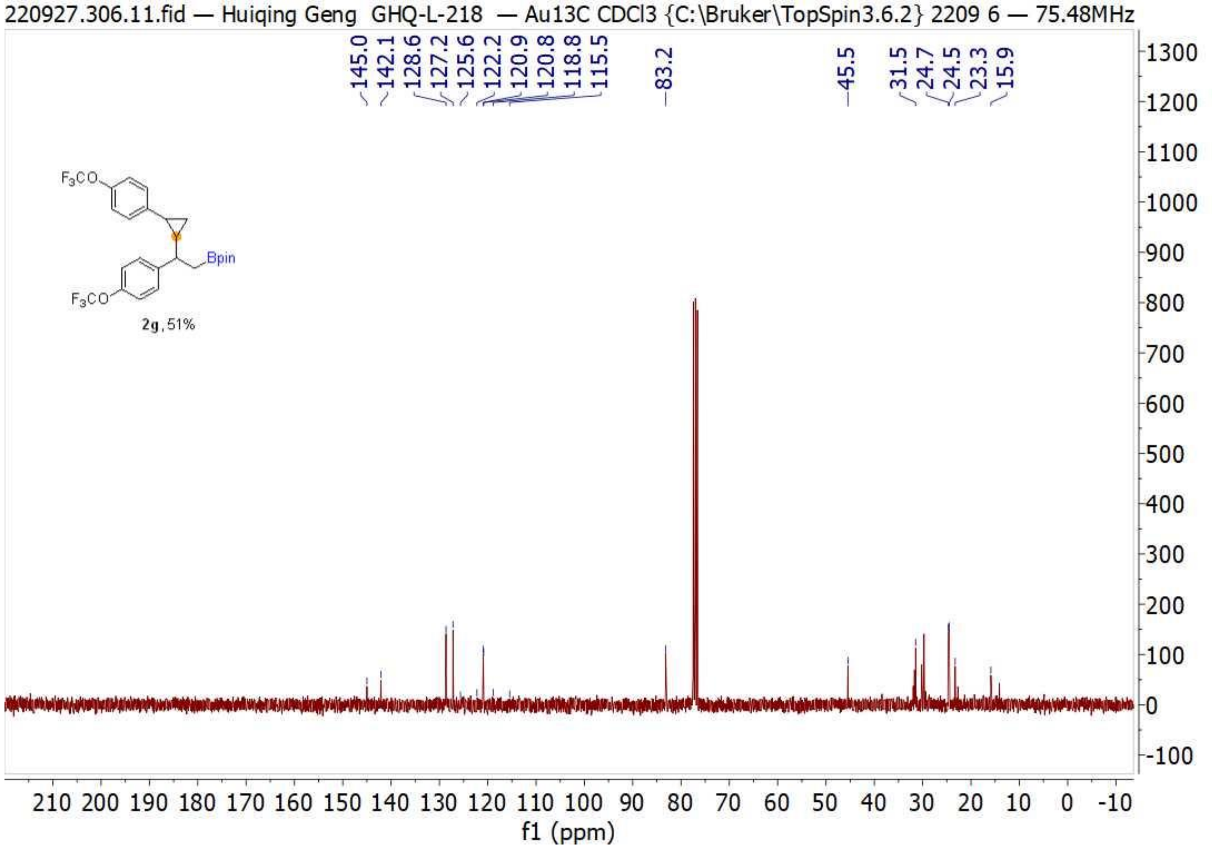

Huiqing Geng GHQ-L-216 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 4 — 300.13MHz
7.60
7.57
7.55
7.53
7.53
7.51
7.36
7.35
7.34
7.33
7.32
7.31
7.29
7.28
7.26 cdcL3
7.12
7.11
7.10
7.09
7.07
7.06
2.44
2.42
1.42
1.42
1.40
1.40
1.39
1.37
1.35
1.05
1.00
0.92
0.91
0.89
0.88
0.87
0.86
F3CS
Bpin
F3CS
2f, 69%
3.99
2.00
2.00
0.98
0.98
3.04
6.01
6.02
2.02
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


220927.304.11.fid — Huiqing Geng GHQ-L-216 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 4 — 75.48MHz
149.4
146.9
136.3
135.8
132.1
131.6
128.6
128.0
127.6
126.9
124.0
123.4
83.2
77.0 cdcL3
45.9
29.7
24.6
24.4
23.8
16.5
F3CS
Bpin
F3CS
2f, 69%
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
270
250
230
210
190
170
150
130
110
90
70
50
30
10
-10
f1 (ppm)

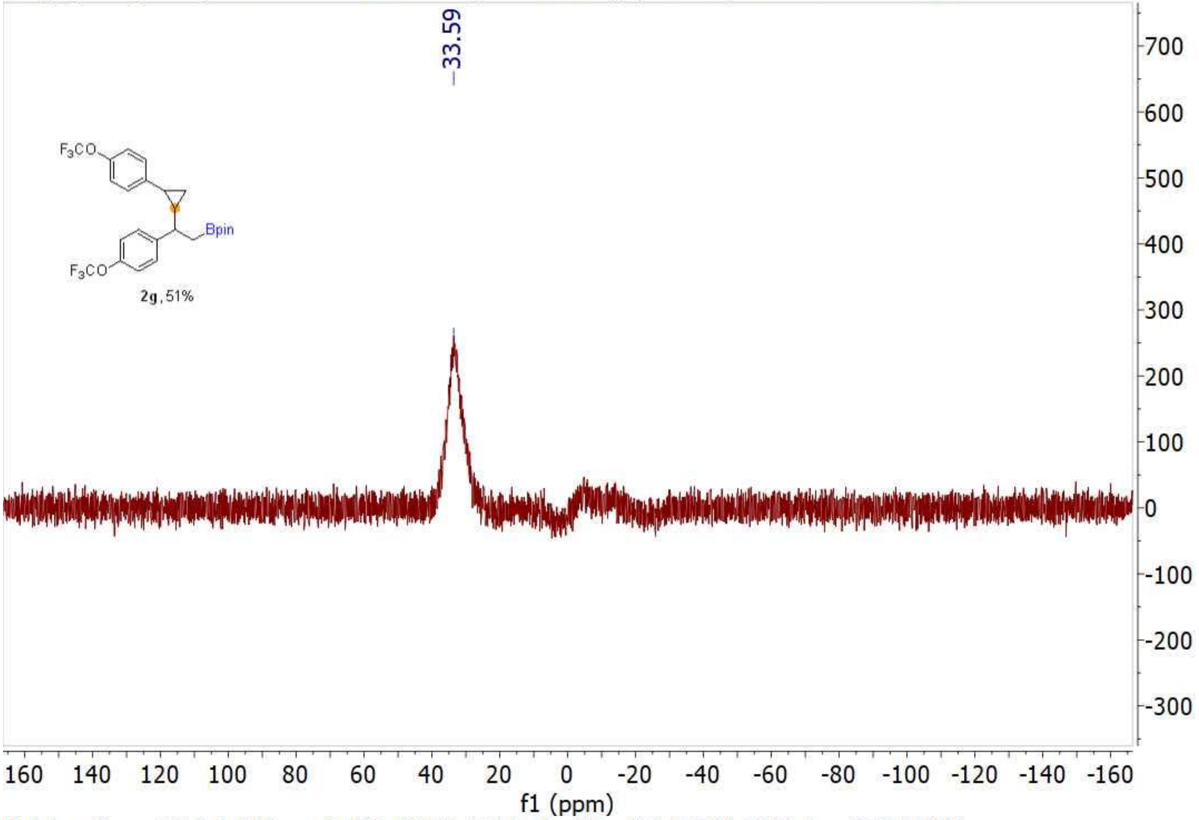

Huiqing Geng GHQ-L-216 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 4 — 96.29MHz
32.90
F3CS
Bpin
F3CS
2f, 69%
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)


Huiqing Geng GHQ-L-216 — Au19F CDCl3 {C:\Bruker\TopSpin3.6.2} 2302 4 — 282.39MHz
-43.13
-43.22
F3CS
Bpin
F3CS
2f, 69%
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220
f1 (ppm)

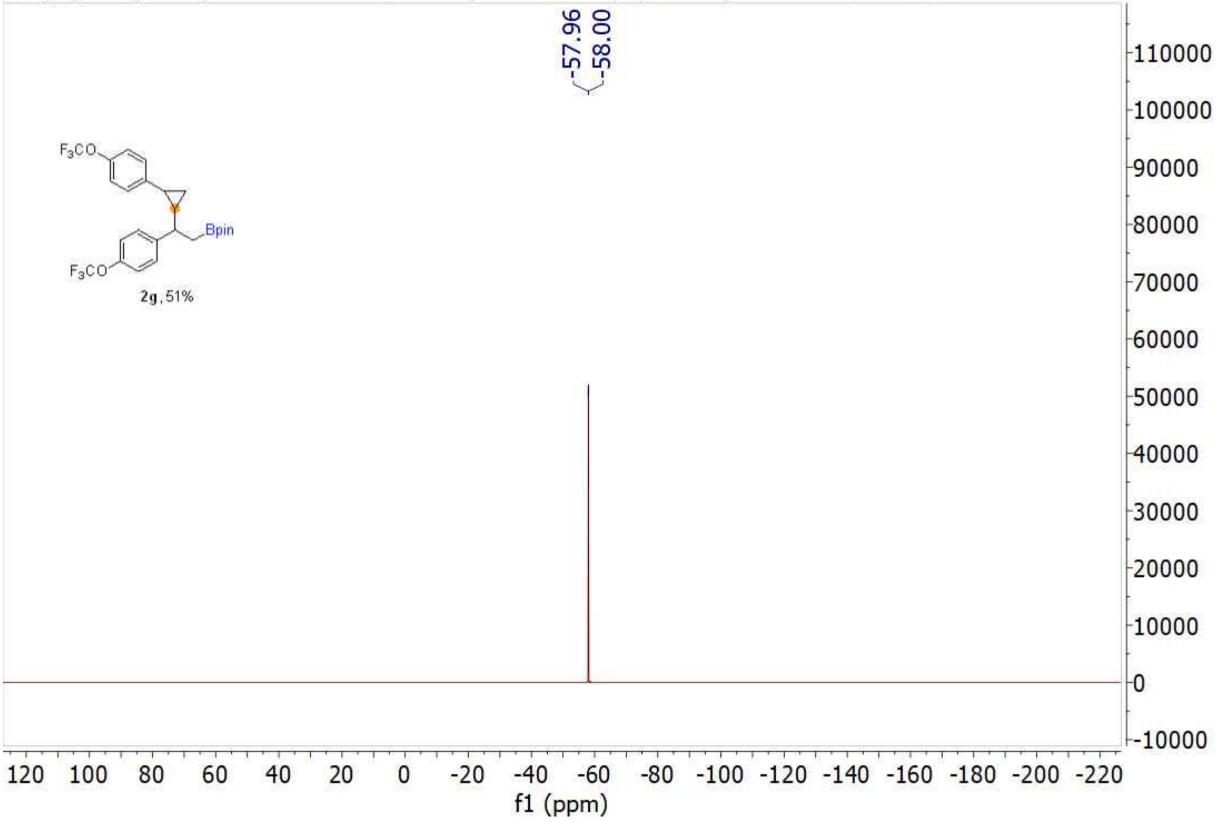

220927.306.10.fid — Huiqing Geng GHQ-L-218 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 6 — 300.13MHz
7.4
7.3
7.3
7.3
7.3
7.3 CDCl3
7.2
7.2
7.1
7.1
7.1
7.1
7.1
7.1
7.1
7.1
7.1
2.4
2.4
1.9
1.9
1.9
1.9
1.9
1.4
1.4
1.4
1.1
1.0
0.9
0.9
0.9
0.9
0.9
0.9
0.9
0.8
0.8
0.8
0.8
0.8
F3CO
Bpin
F3CO
2g, 51%
2.03
2.00
4.04
0.97
1.00
2.96
5.99
6.03
2.04
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-4000
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

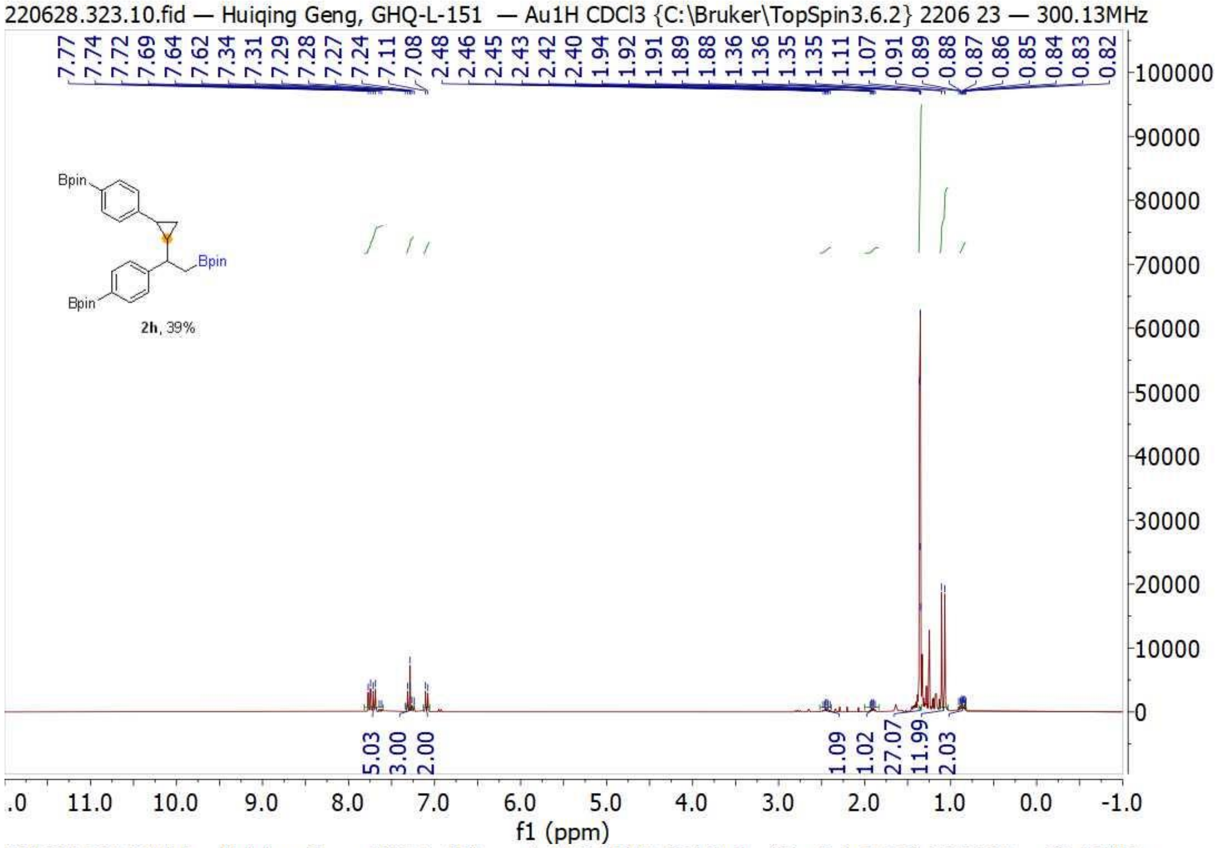

220927.306.11.fid — Huiqing Geng GHQ-L-218 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 6 — 75.48MHz
145.0
142.1
128.6
127.2
125.6
122.2
120.9
120.8
118.8
115.5
83.2
45.5
31.5
24.7
24.5
23.3
15.9
F3CO
Bpin
F3CO
2g, 51%
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

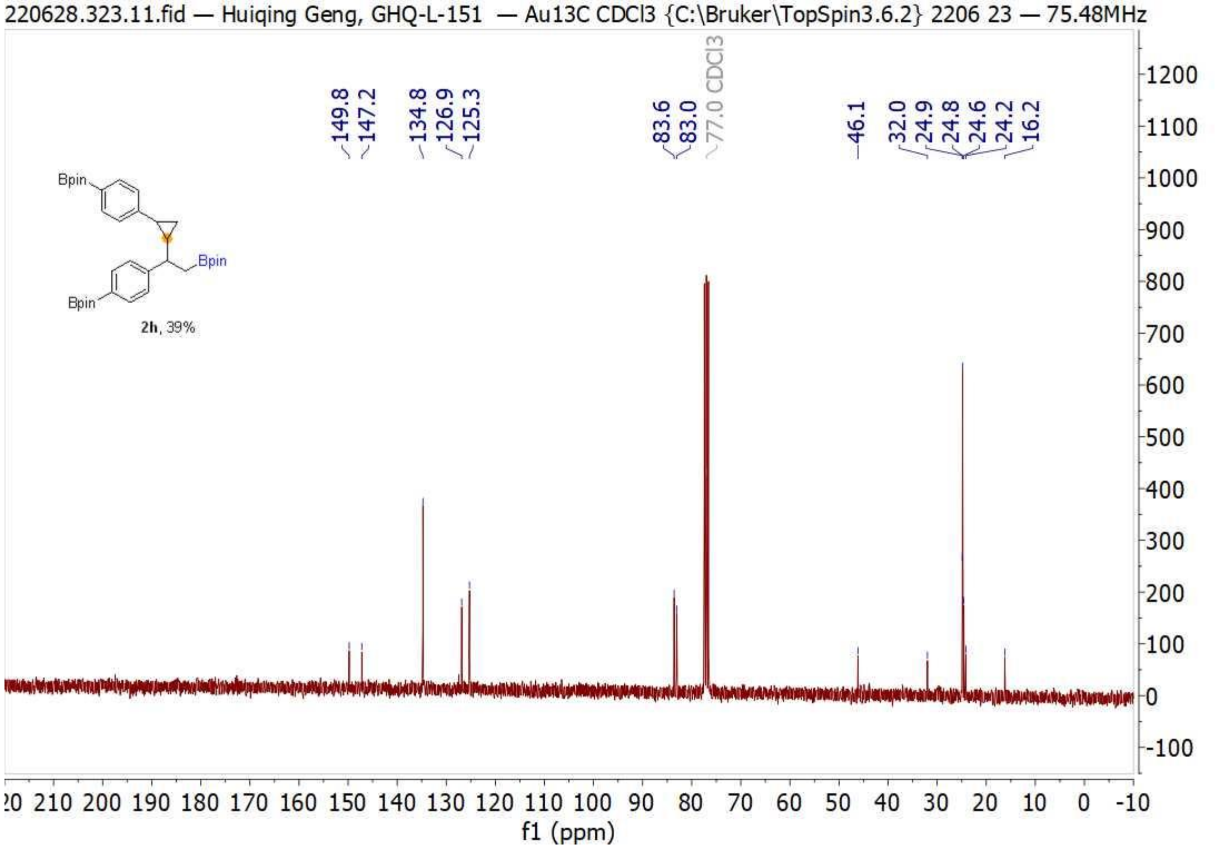
Huiqing Geng GHQ-L-218 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 6 — 96.29MHz

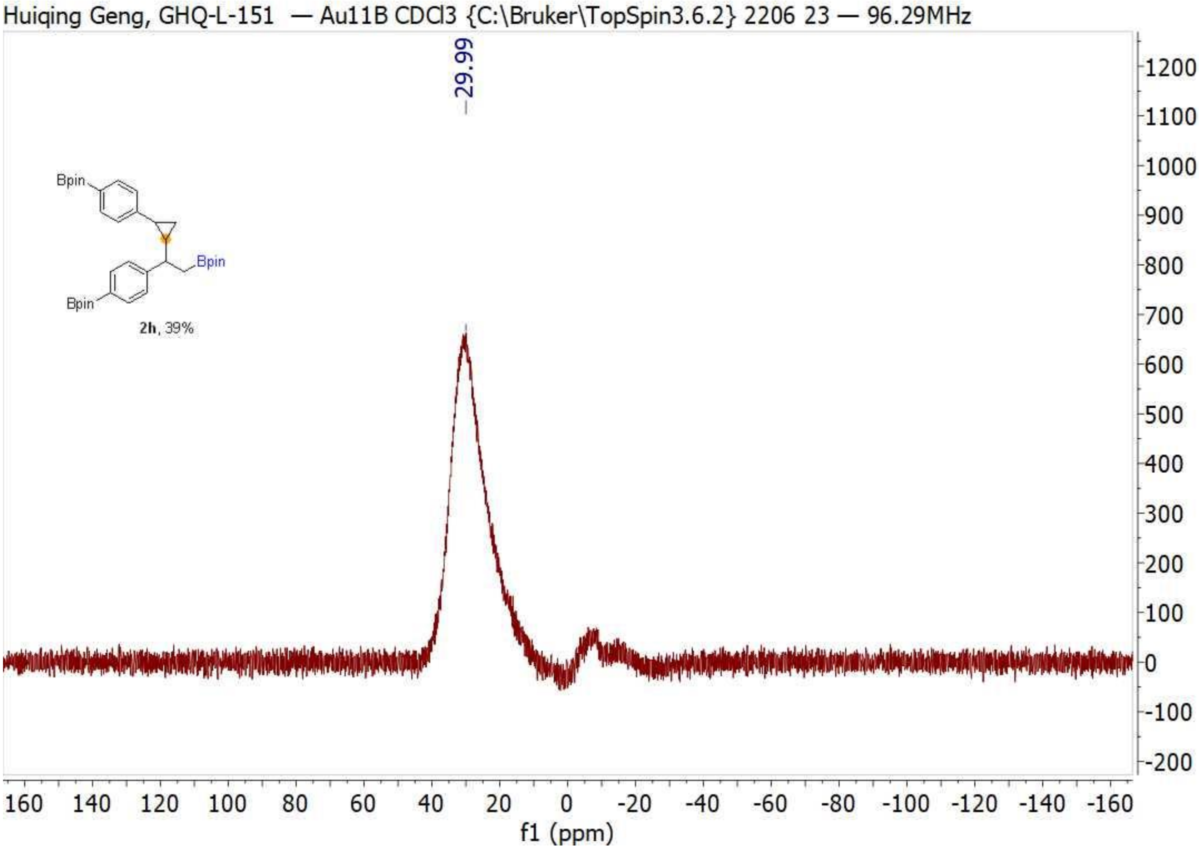


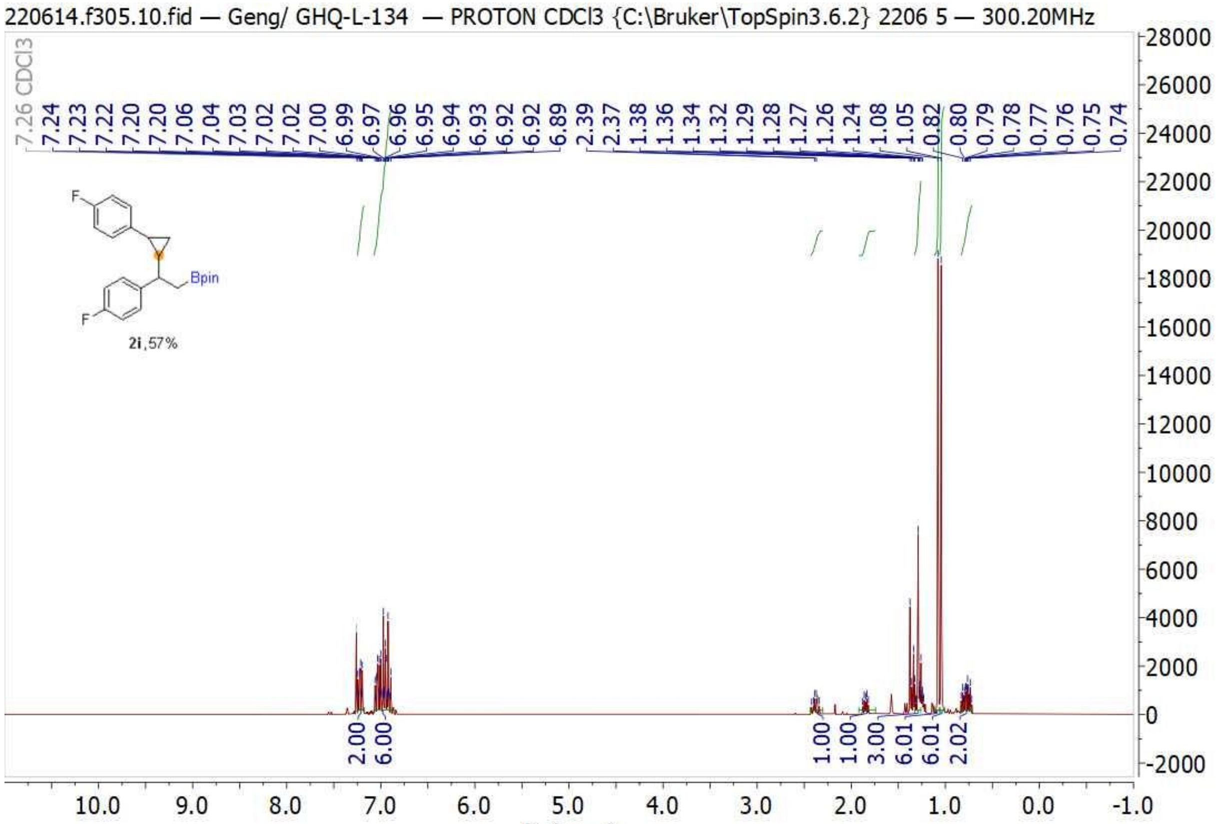
Huiqing Geng GHQ-L-218 — Au19F CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 6 — 282.39MHz

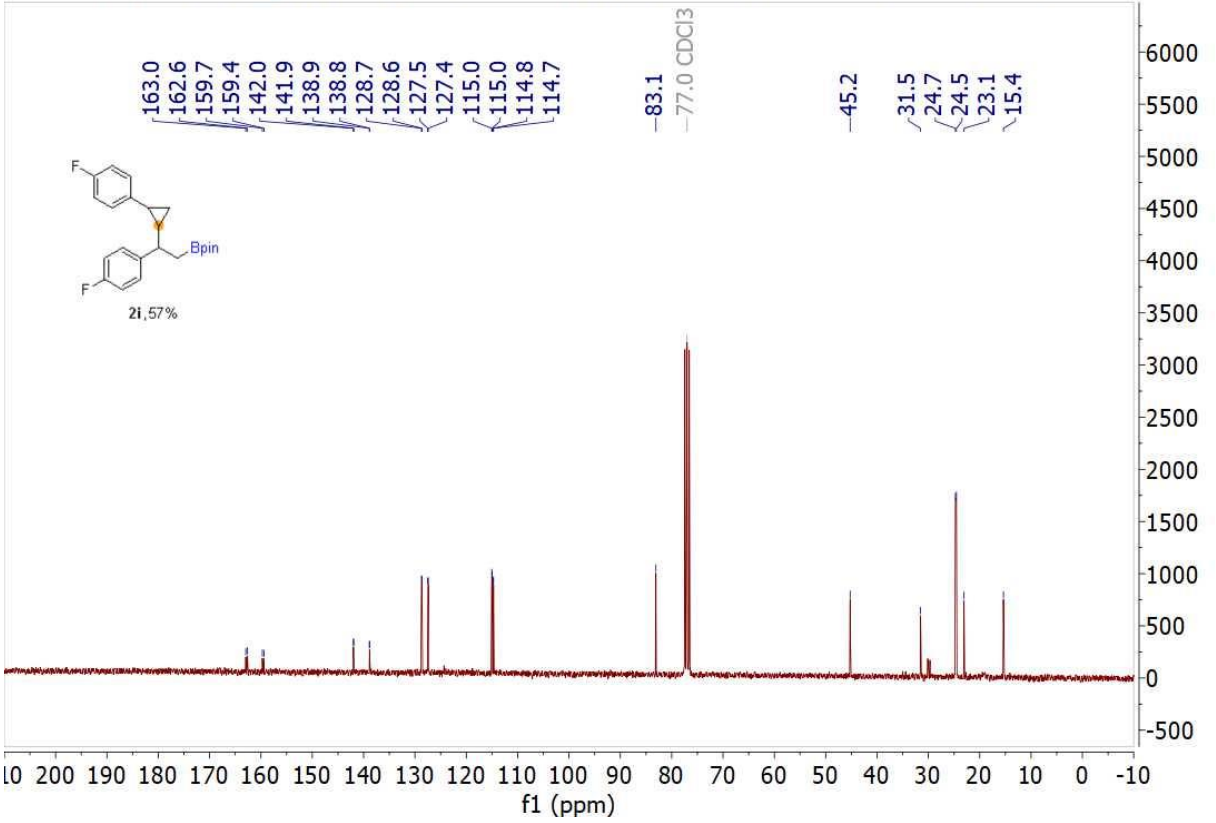



220628.323.10.fid — Huiqing Geng, GHQ-L-151 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 23 — 300.13MHz
7.77
7.74
7.72
7.69
7.64
7.62
7.34
7.31
7.29
7.28
7.27
7.24
7.11
7.08
2.48
2.46
2.45
2.43
2.42
2.40
1.94
1.92
1.91
1.89
1.88
1.36
1.36
1.35
1.35
1.11
1.07
0.91
0.89
0.88
0.87
0.86
0.85
0.84
0.83
0.82
Bpin
Bpin
Bpin
2h, 39%
5.03
3.00
2.00
1.09
1.02
27.07
11.99
2.03
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

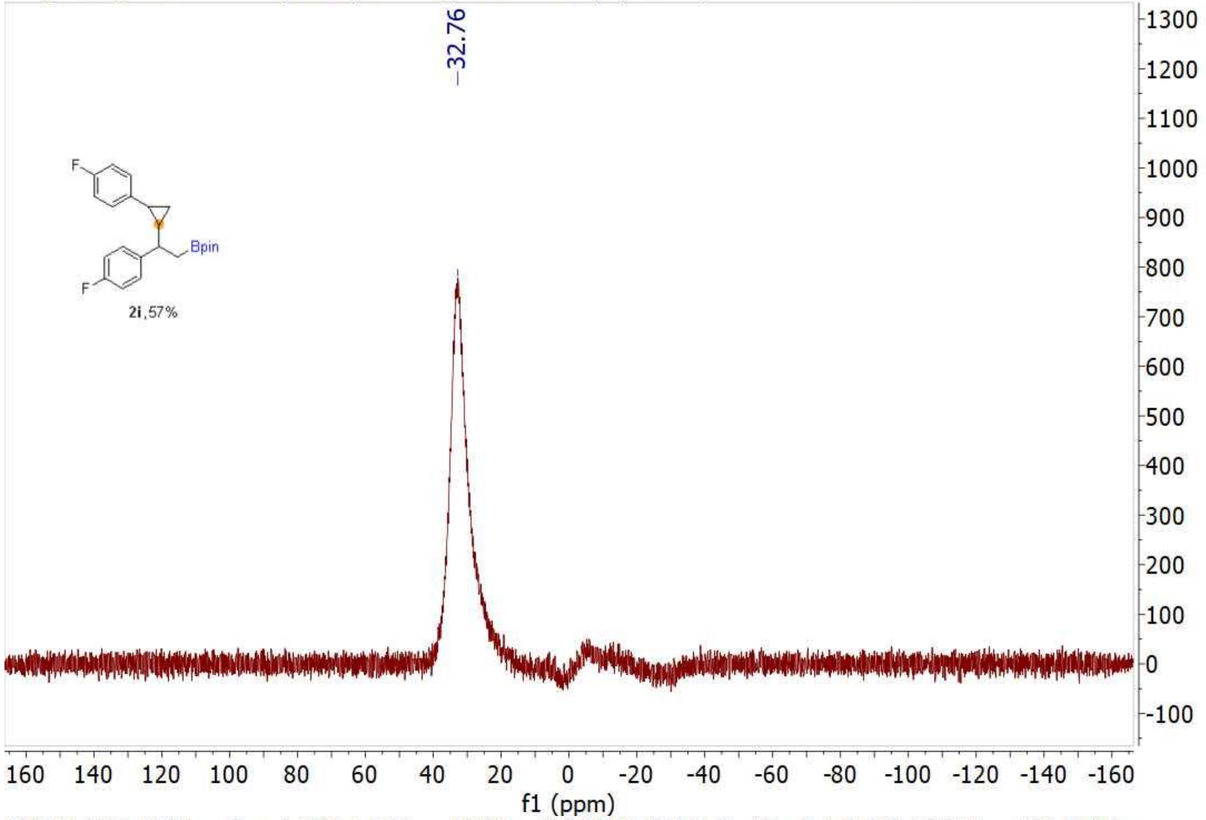

220628.323.11.fid — Huiqing Geng, GHQ-L-151 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 23 — 75.48MHz
149.8
147.2
134.8
126.9
125.3
83.6
83.0
77.0 CDCl3
46.1
32.0
24.9
24.8
24.6
24.2
16.2
Bpin
Bpin
Bpin
2h, 39%
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Huiqing Geng, GHQ-L-151 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 23 — 96.29MHz
29.99
Bpin
Bpin
Bpin
2h, 39%
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
-200
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

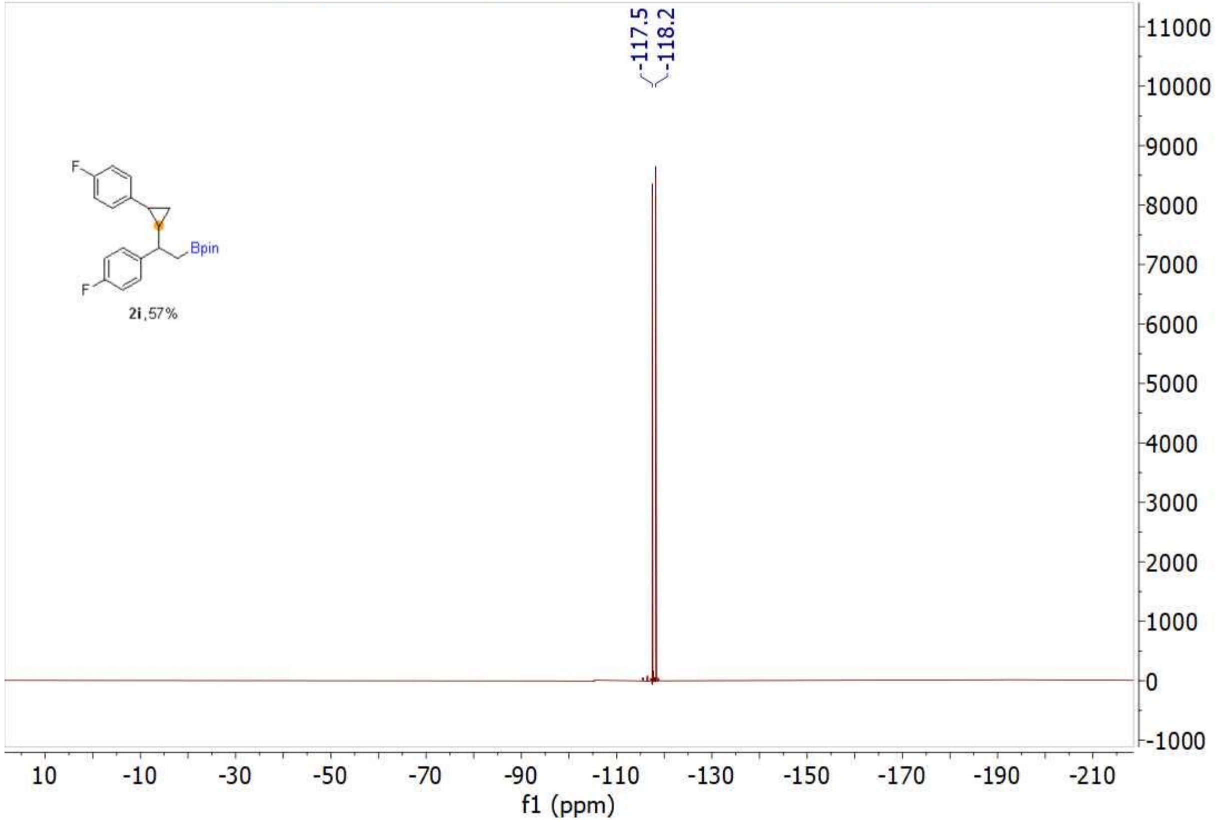

220614.f305.10.fid — Geng/ GHQ-L-134 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 5 — 300.20MHz
7.26 CDCl3
7.24
7.23
7.22
7.20
7.20
7.06
7.04
7.03
7.02
7.02
7.00
6.99
6.97
6.96
6.95
6.94
6.93
6.92
6.92
6.89
2.39
2.37
1.38
1.36
1.34
1.32
1.29
1.28
1.27
1.26
1.24
1.08
1.05
0.82
0.80
0.79
0.78
0.77
0.76
0.75
0.74
F
F
Bpin
2i,57%
2.00
6.00
1.00
1.00
3.00
6.01
6.01
2.02
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

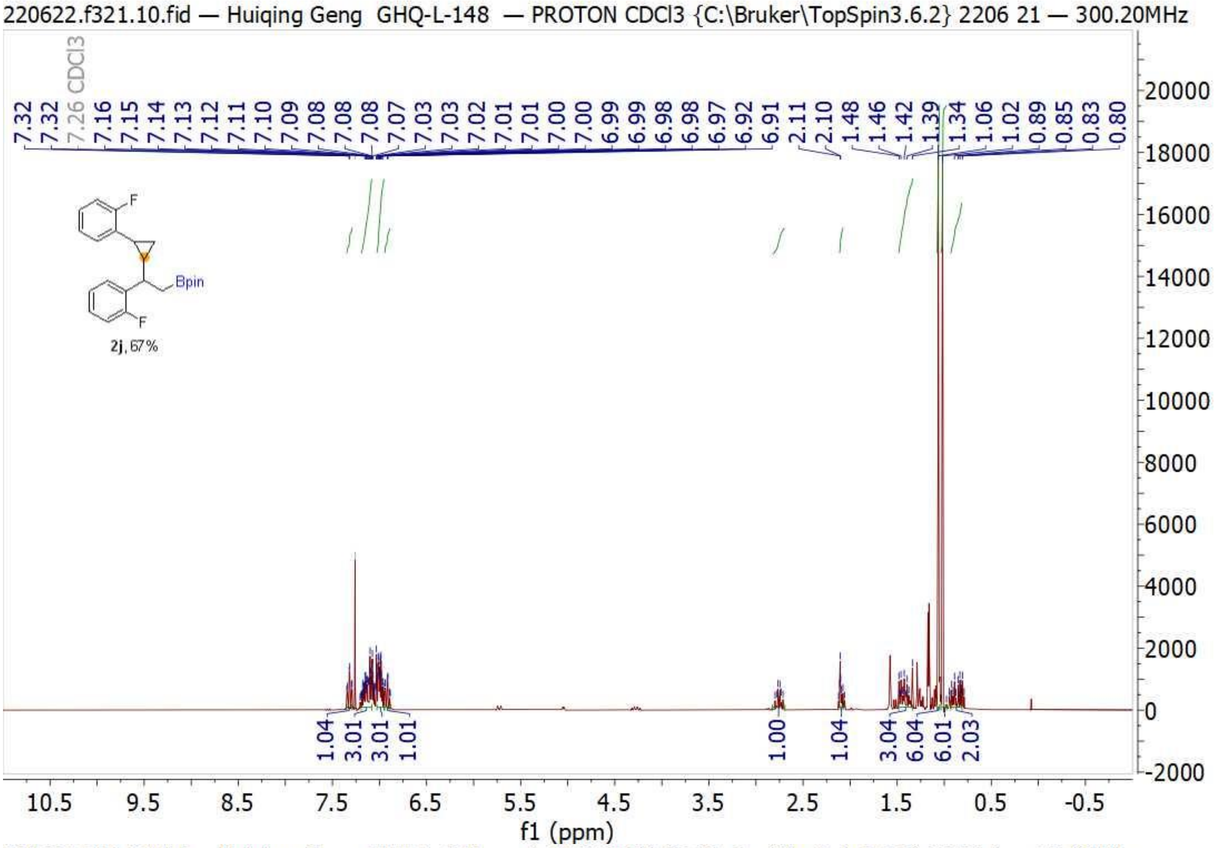

220615.303.11.fid — Geng/ GHQ-L-134 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 3 — 75.48MHz
163.0
162.6
159.7
159.4
142.0
141.9
138.9
138.8
128.7
128.6
127.5
127.4
115.0
115.0
114.8
114.7
83.1
77.0 CDCl3
45.2
31.5
24.7
24.5
23.1
15.4
F
F
Bpin
2i,57%
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

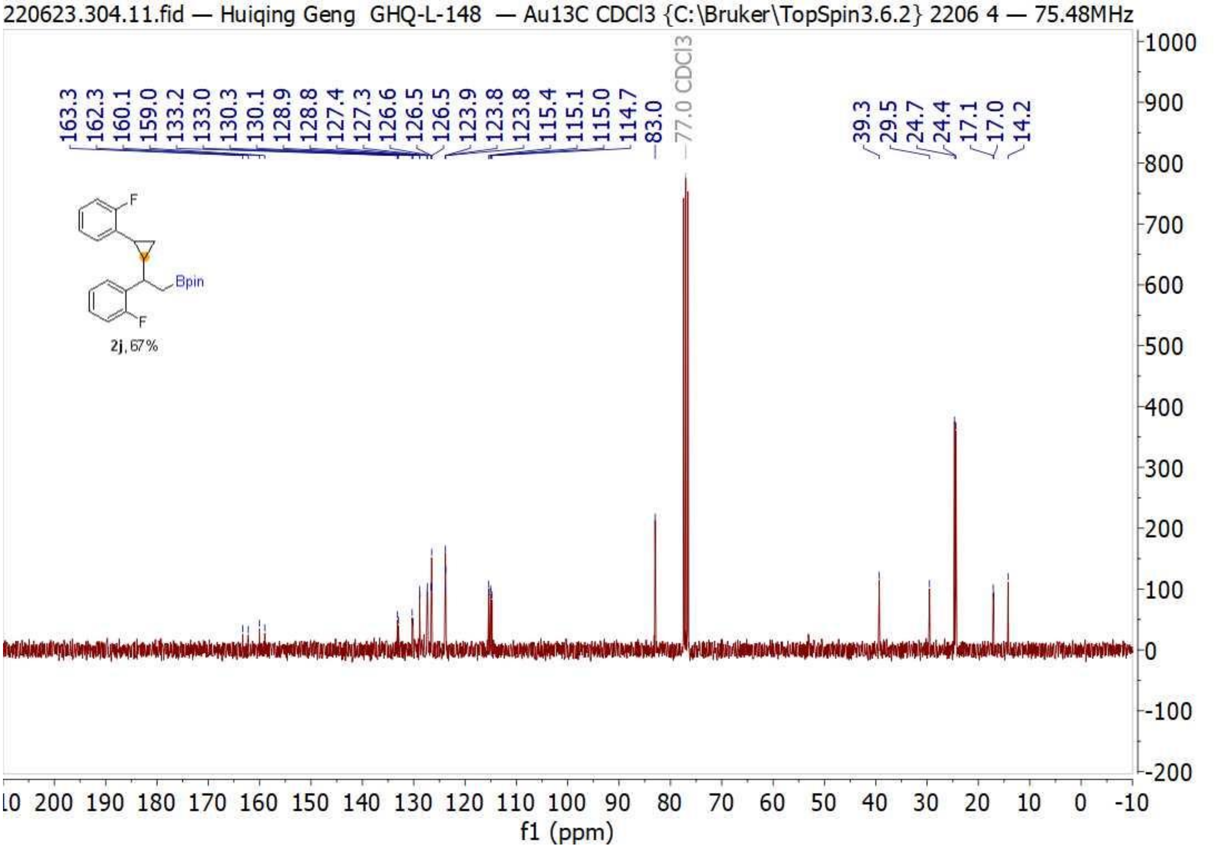
Geng/ GHQ-L-134 — 11B(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 5 — 96.32MHz



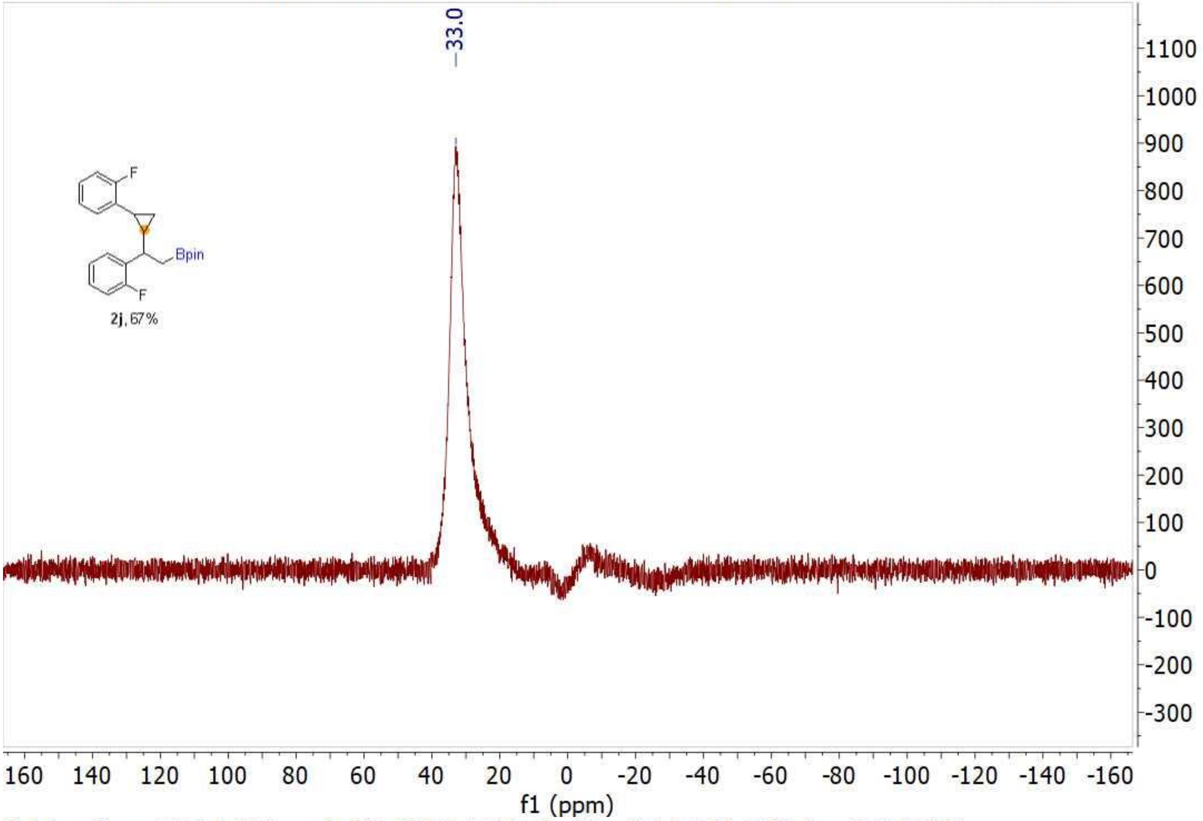
220614.f305.12.fid — Geng/ GHQ-L-134 — 19F(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 5 — 282.44MHz



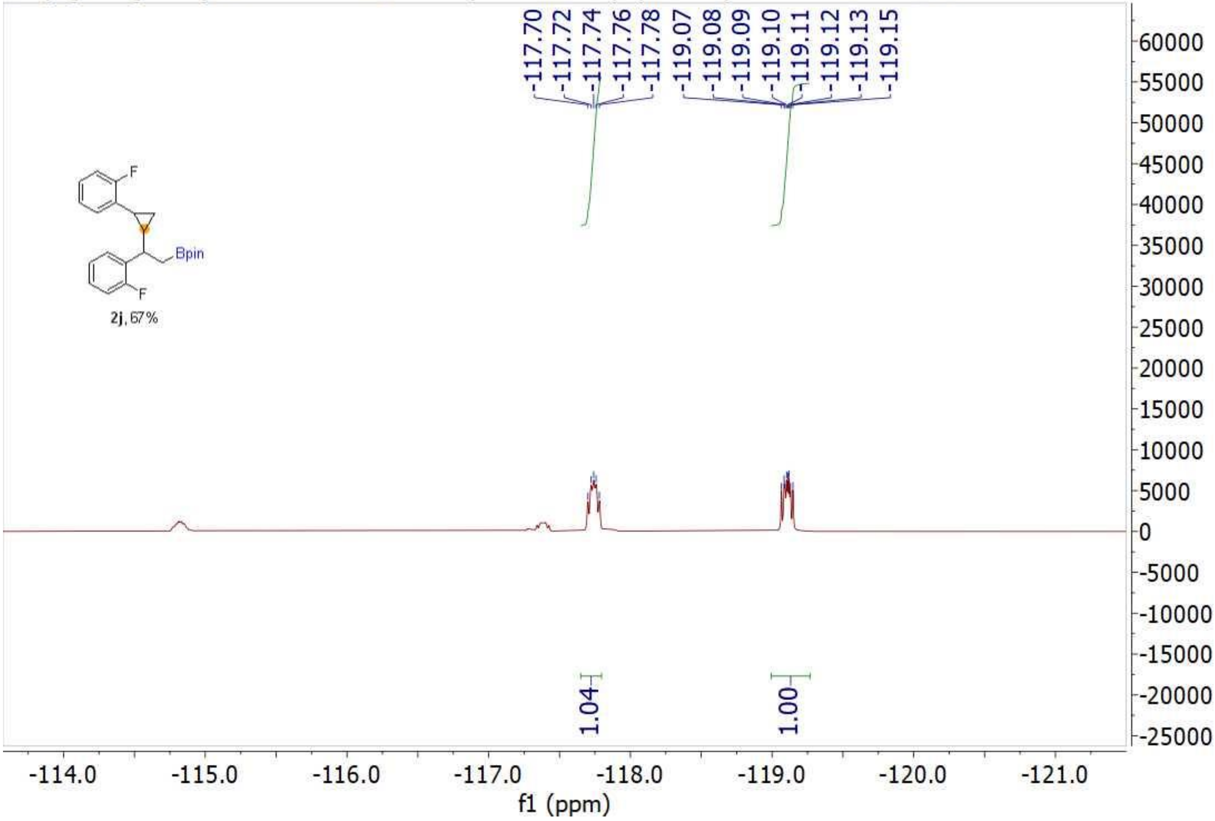

220622.f321.10.fid — Huiqing Geng GHQ-L-148 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 21 — 300.20MHz
7.32
7.32
7.26 CDCl3
7.16
7.15
7.14
7.13
7.12
7.11
7.10
7.09
7.08
7.08
7.08
7.07
7.03
7.03
7.02
7.01
7.01
7.00
7.00
6.99
6.99
6.98
6.98
6.97
6.92
6.91
2.11
2.10
1.48
1.46
1.42
1.39
1.34
1.06
1.02
0.89
0.85
0.83
0.80
F
Bpin
F
2j, 67%
1.04
3.01
3.01
1.01
1.00
1.04
3.04
6.04
6.01
2.03
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


220623.304.11.fid — Huiqing Geng GHQ-L-148 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 4 — 75.48MHz
163.3
162.3
160.1
159.0
133.2
133.0
130.3
130.1
128.9
128.8
127.4
127.3
126.6
126.5
126.5
123.9
123.8
123.8
115.4
115.1
115.0
114.7
83.0
77.0 CDCl3
39.3
29.5
24.7
24.4
17.1
17.0
14.2
F
Bpin
F
2j, 67%
1000
900
800
700
600
500
400
300
200
100
0
-100
-200
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

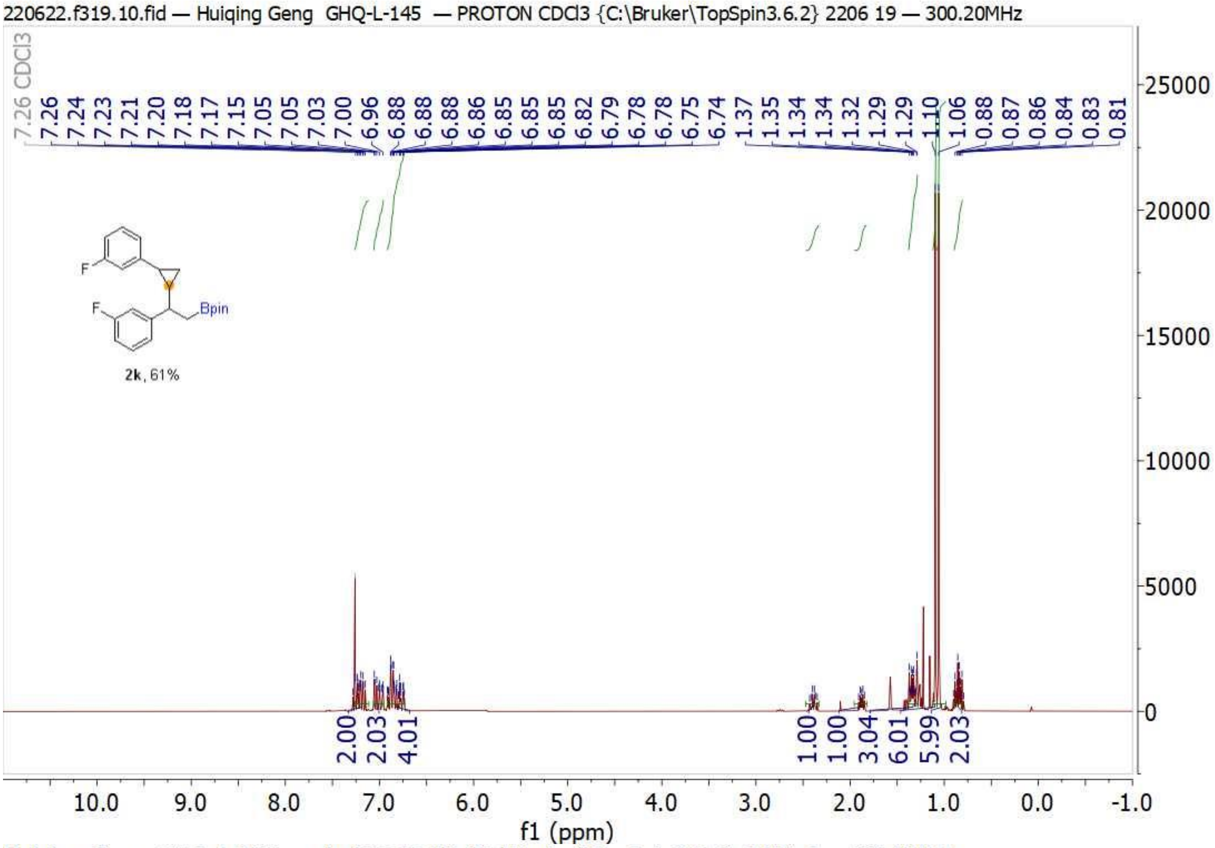
220622.f321.11.fid — Huiqing Geng GHQ-L-148 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 21 — 96.32MHz



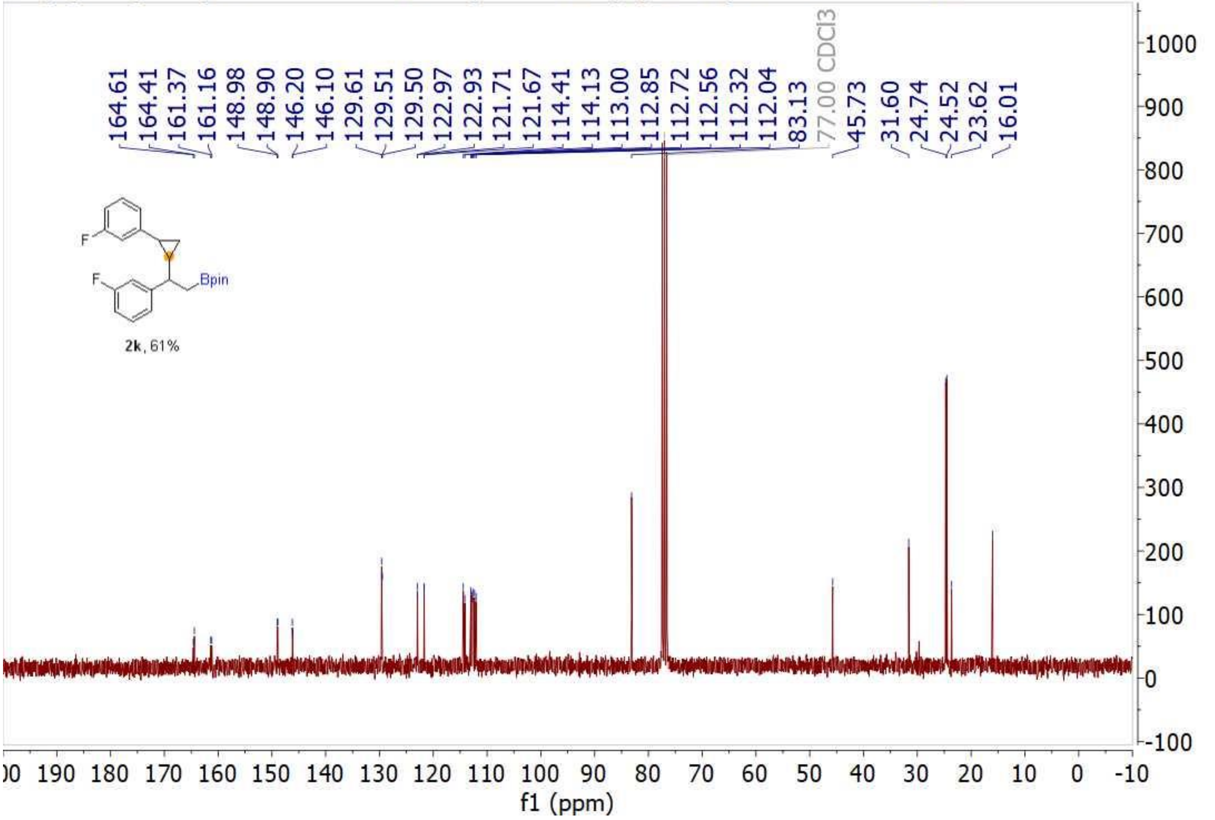
Huiqing Geng GHQ-L-148 — Au19F CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 4 — 282.39MHz



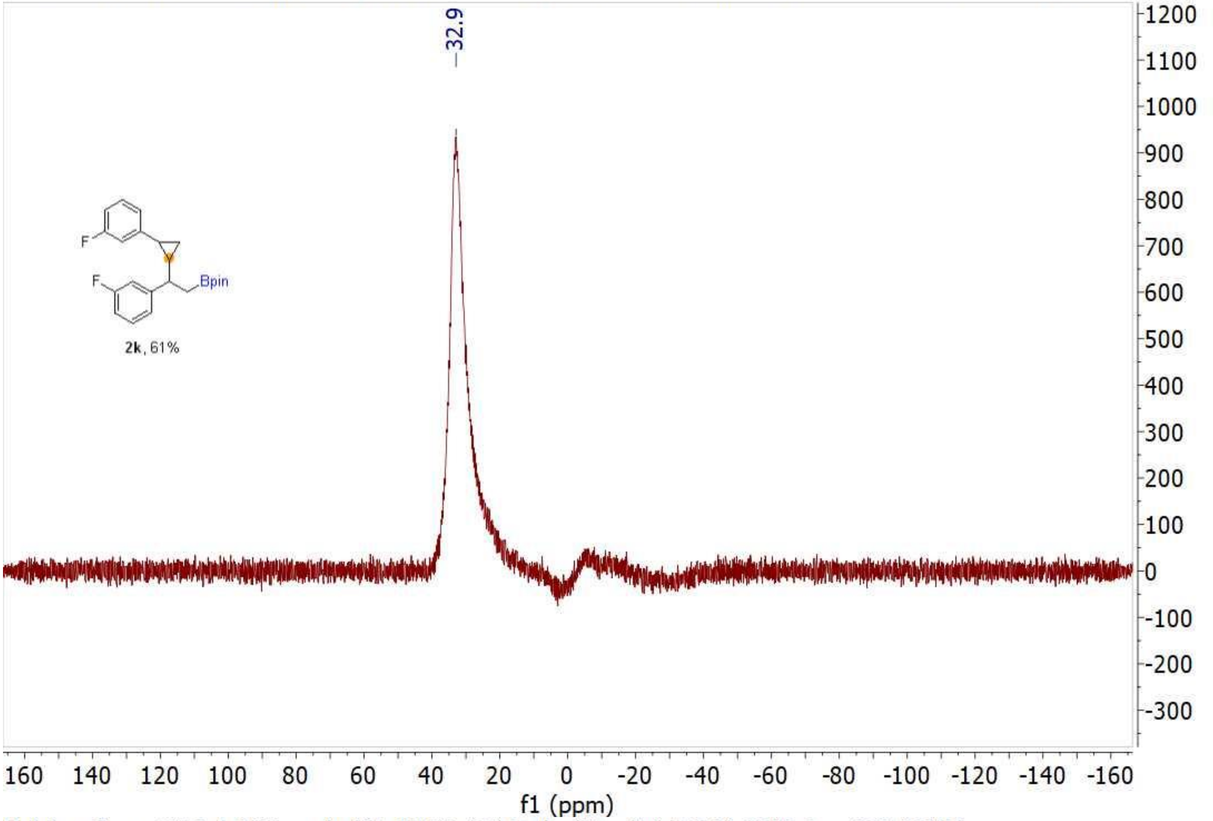
220622.f319.10.fid — Huiqing Geng GHQ-L-145 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 19 — 300.20MHz



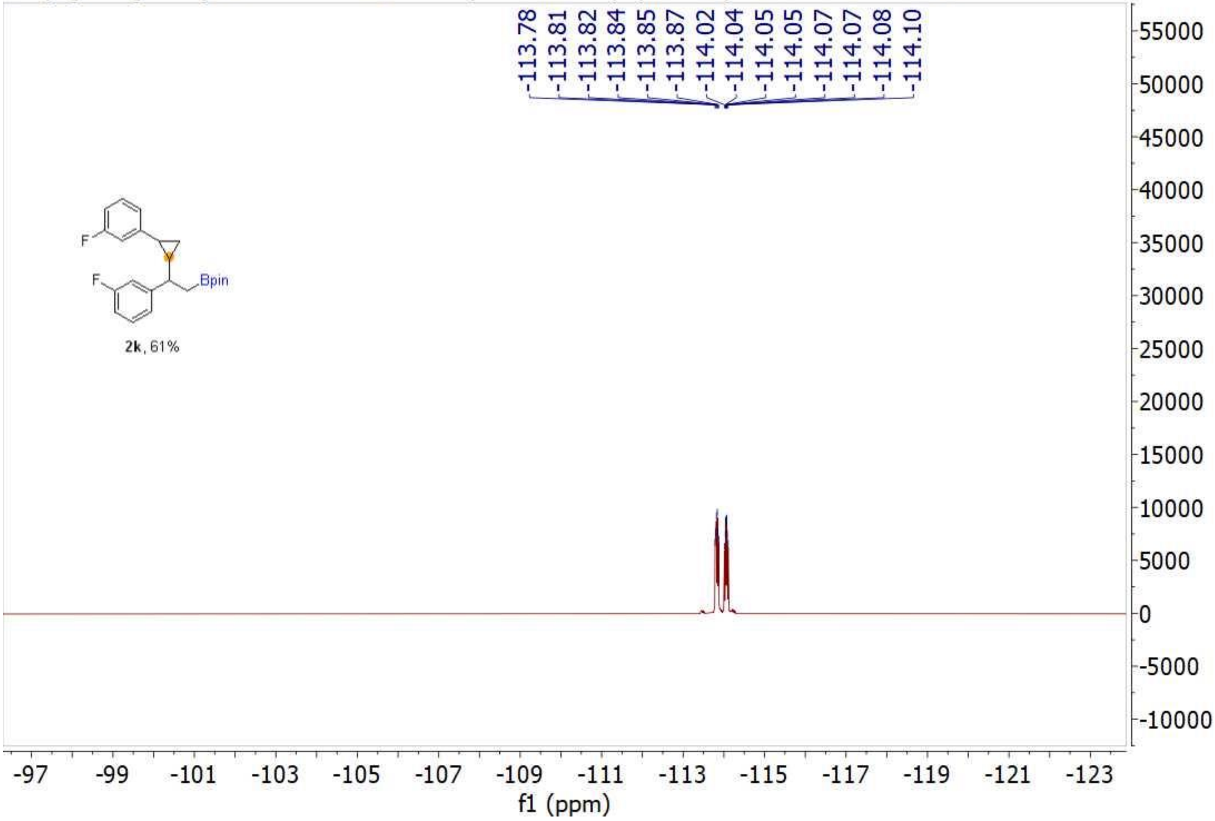
Huiqing Geng GHQ-L-145 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 6 — 75.48MHz

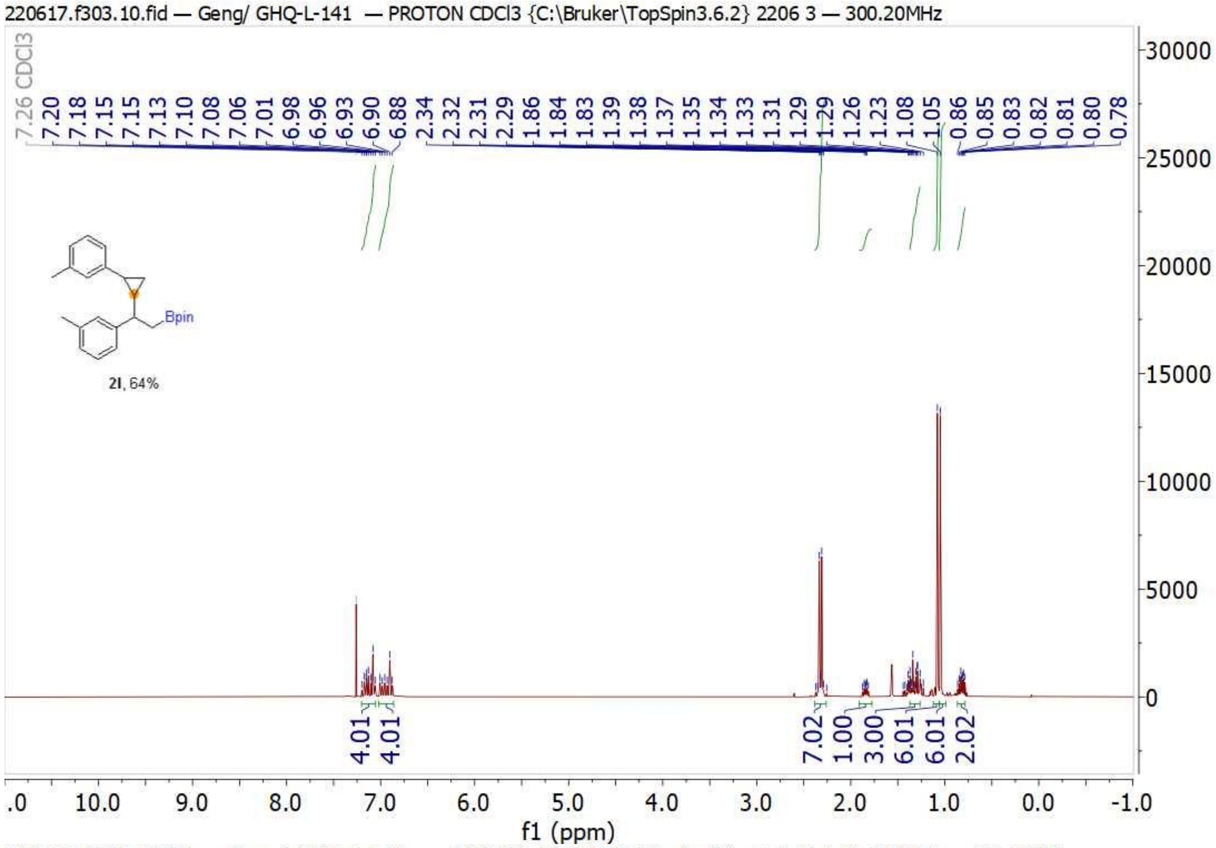


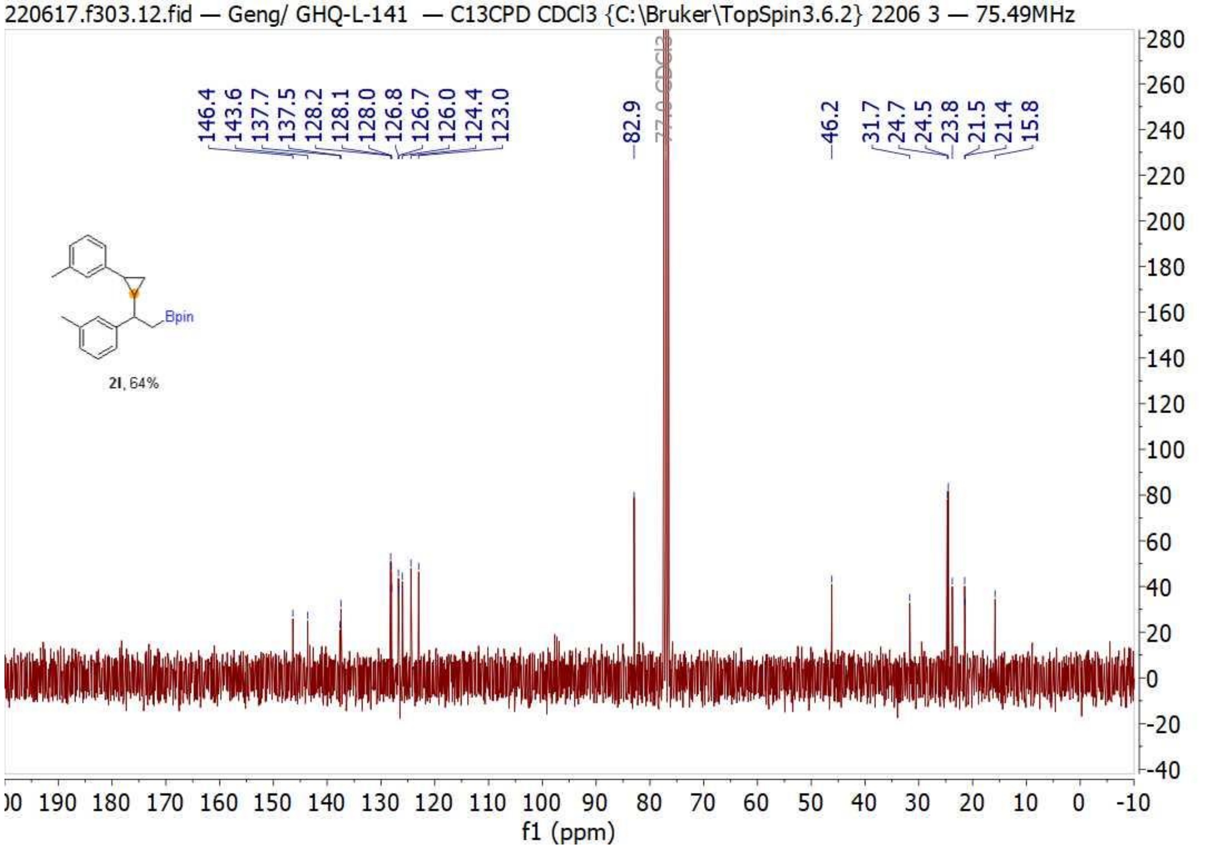
220622.f319.11.fid — Huiqing Geng GHQ-L-145 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 19 — 96.32MHz

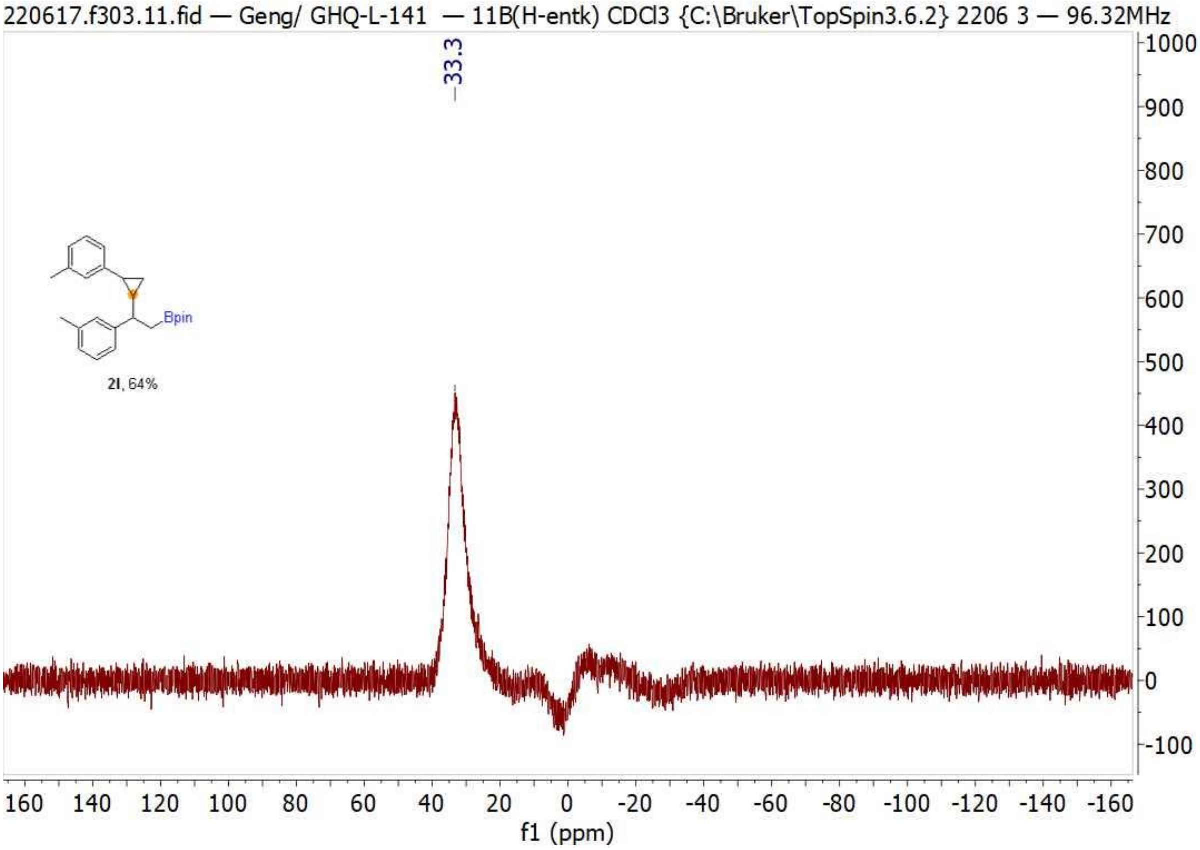


Huiqing Geng GHQ-L-145 — Au19F CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 6 — 282.39MHz

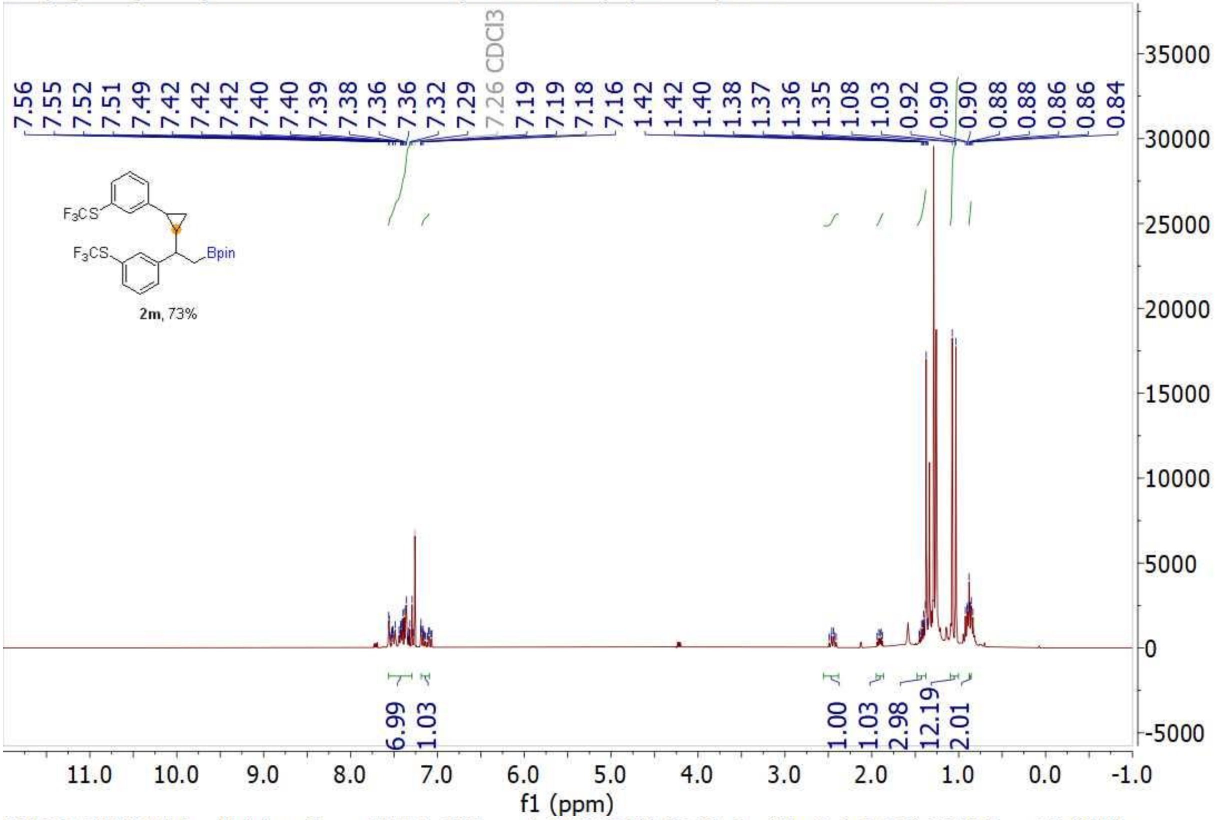



220617.f303.10.fid — Geng/ GHQ-L-141 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 3 — 300.20MHz
7.26 CDCl3
7.20
7.18
7.15
7.15
7.13
7.10
7.08
7.06
7.01
6.98
6.96
6.93
6.90
6.88
2.34
2.32
2.31
2.29
1.86
1.84
1.83
1.39
1.38
1.37
1.35
1.34
1.33
1.31
1.29
1.29
1.26
1.23
1.08
1.05
0.86
0.85
0.83
0.82
0.81
0.80
0.78
Bpin
2l, 64%
4.01
4.01
7.02
1.00
3.00
6.01
6.01
2.02
30000
25000
20000
15000
10000
5000
0
.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

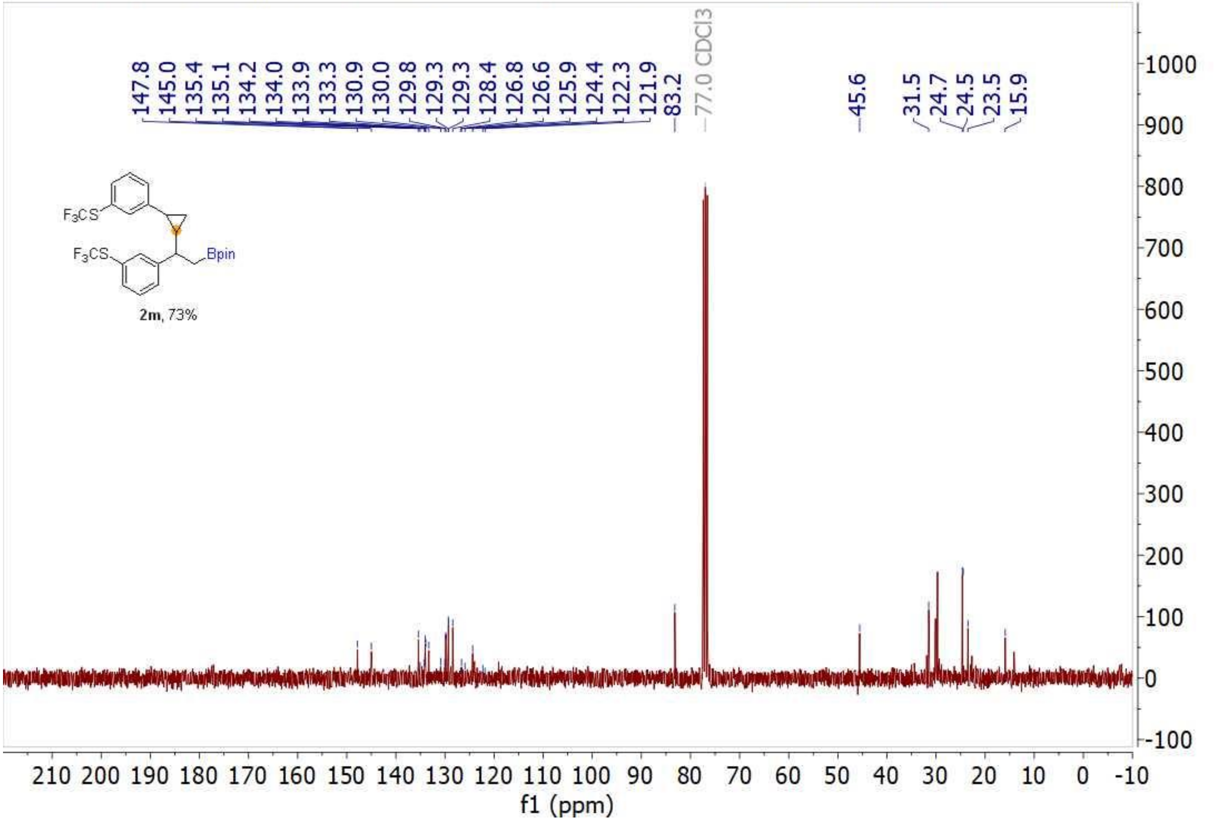

220617.f303.12.fid — Geng/ GHQ-L-141 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 3 — 75.49MHz
146.4
143.6
137.7
137.5
128.2
128.1
128.0
126.8
126.7
126.0
124.4
123.0
82.9
77.0 CDCl3
46.2
31.7
24.7
24.5
23.8
21.5
21.4
15.8
Bpin
2l, 64%
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

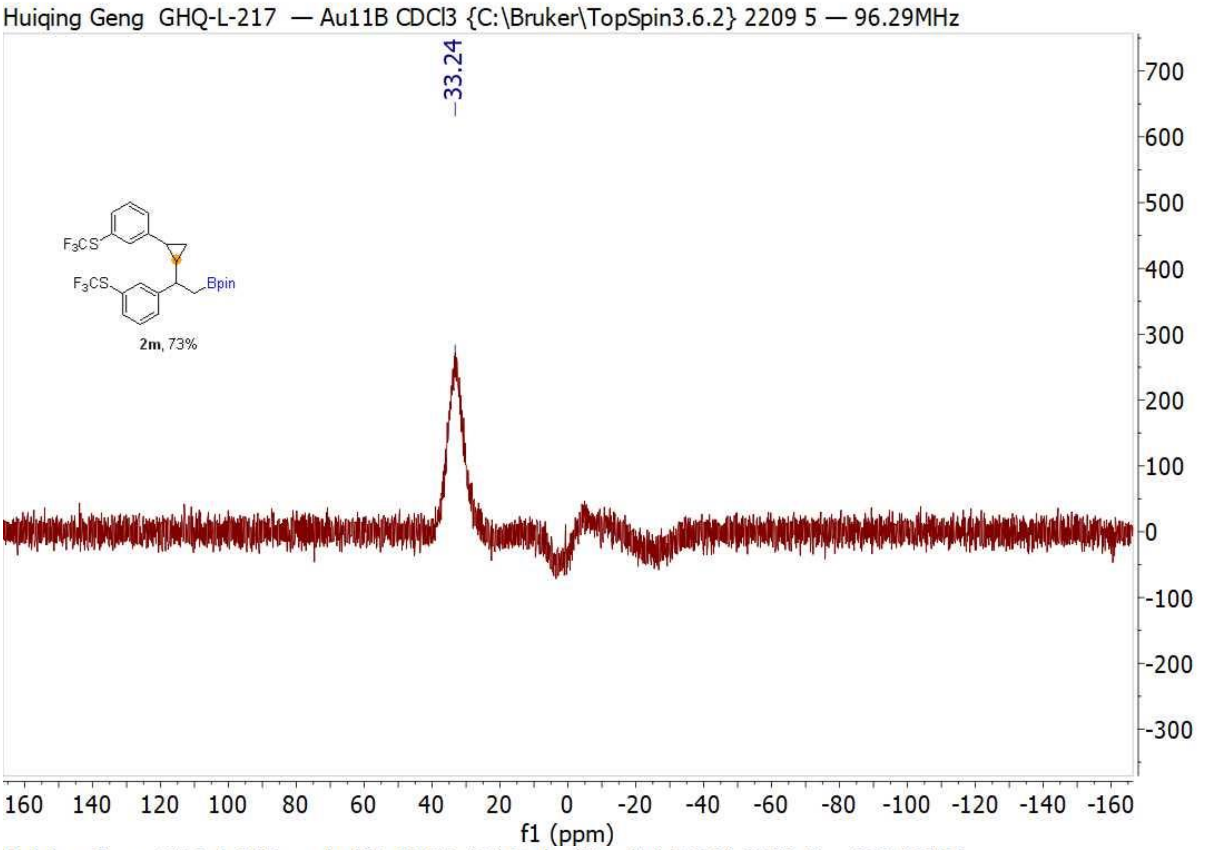

220617.f303.11.fid — Geng/ GHQ-L-141 — 11B(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 3 — 96.32MHz
33.3
Bpin
2l, 64%
1000
900
800
700
600
500
400
300
200
100
0
-100
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
f1 (ppm)

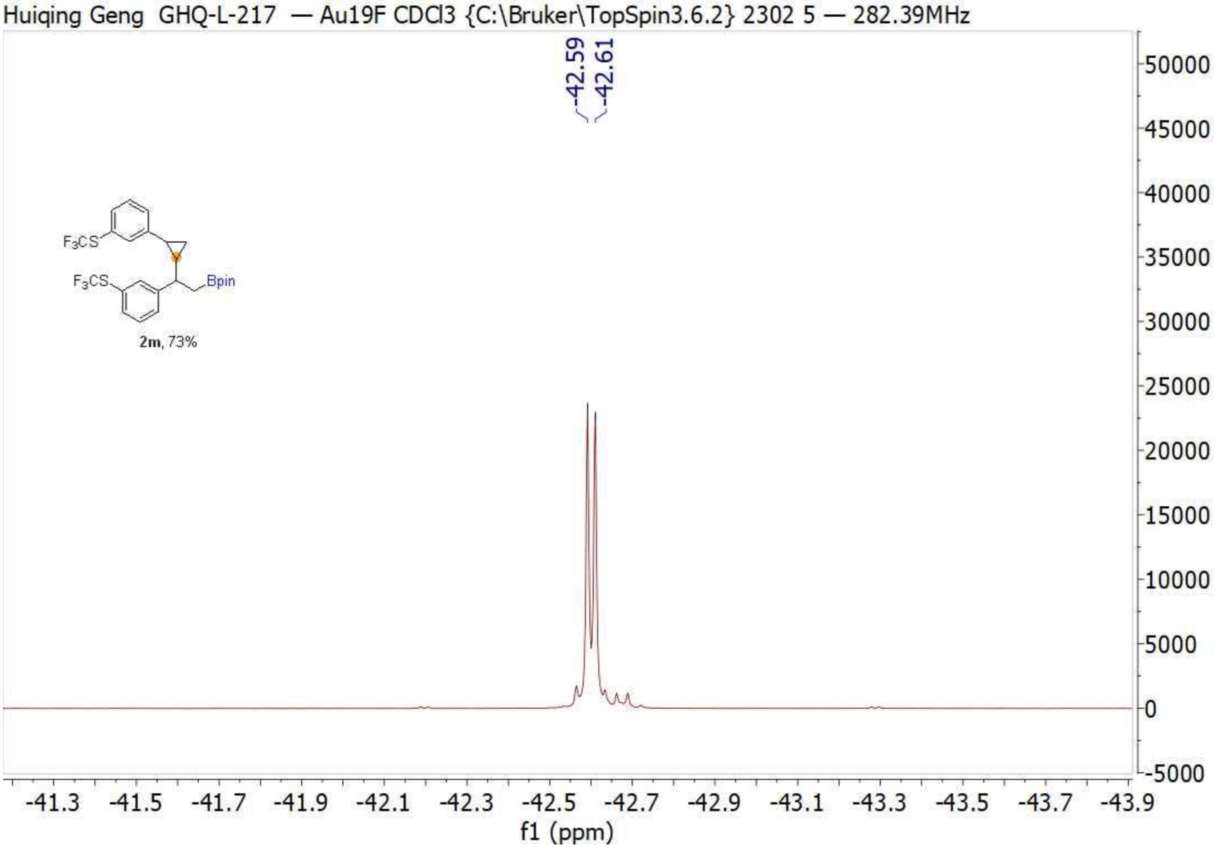
Huiqing Geng GHQ-L-217 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 5 — 300.13MHz

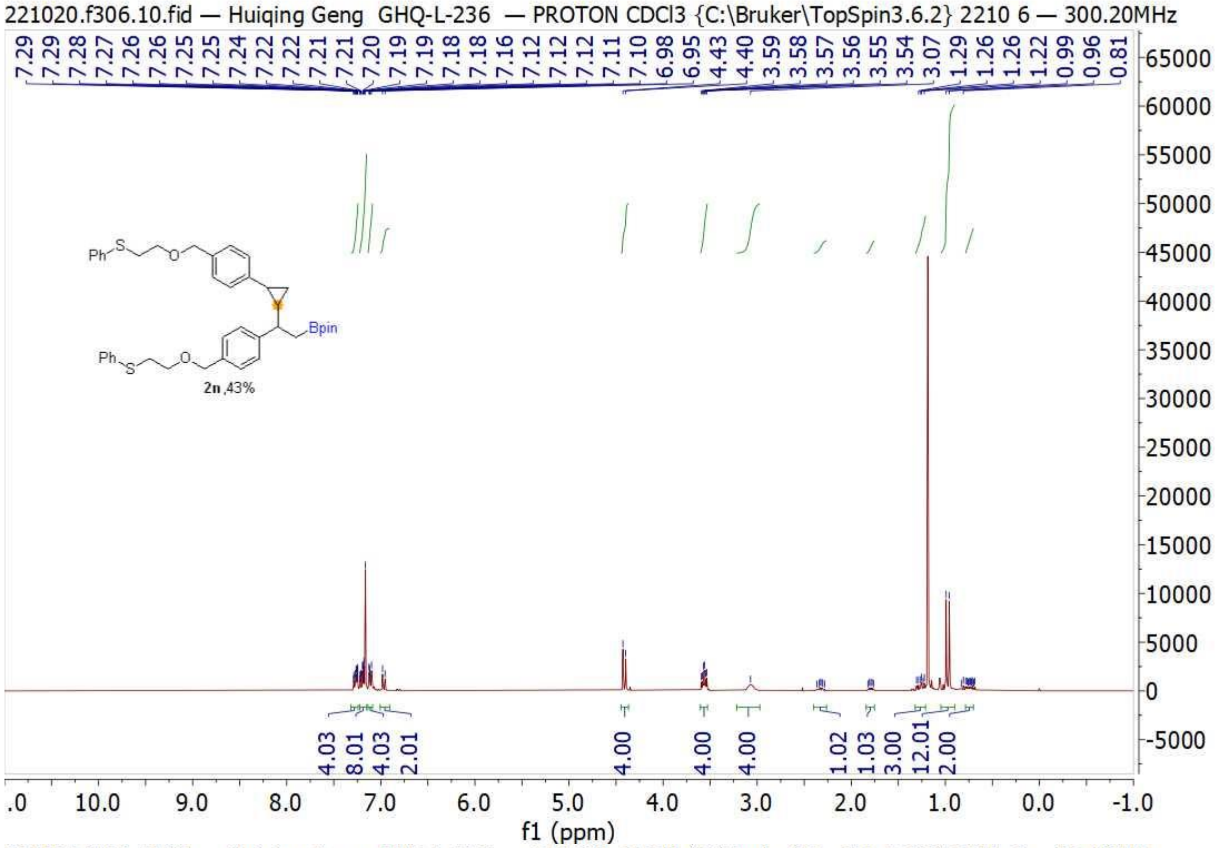


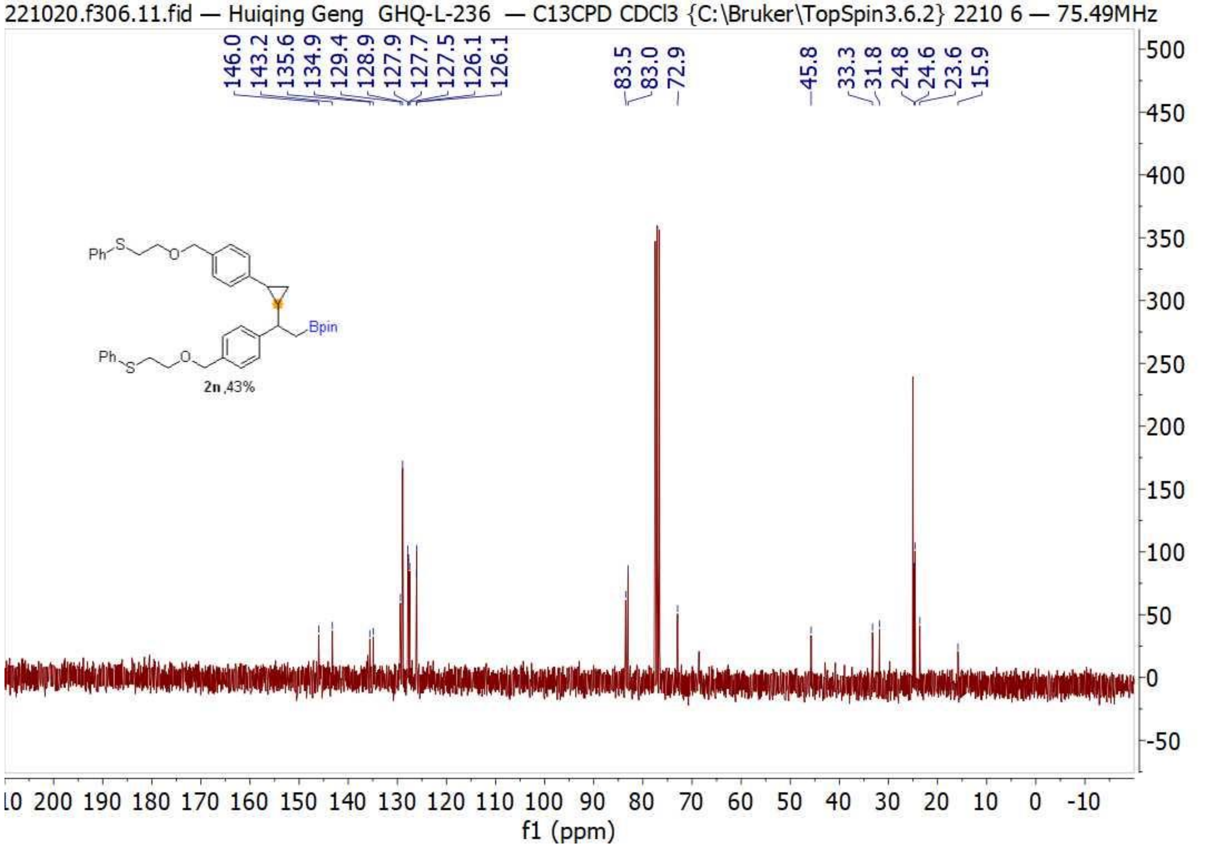
220927.305.11.fid — Huiqing Geng GHQ-L-217 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 5 — 75.48MHz

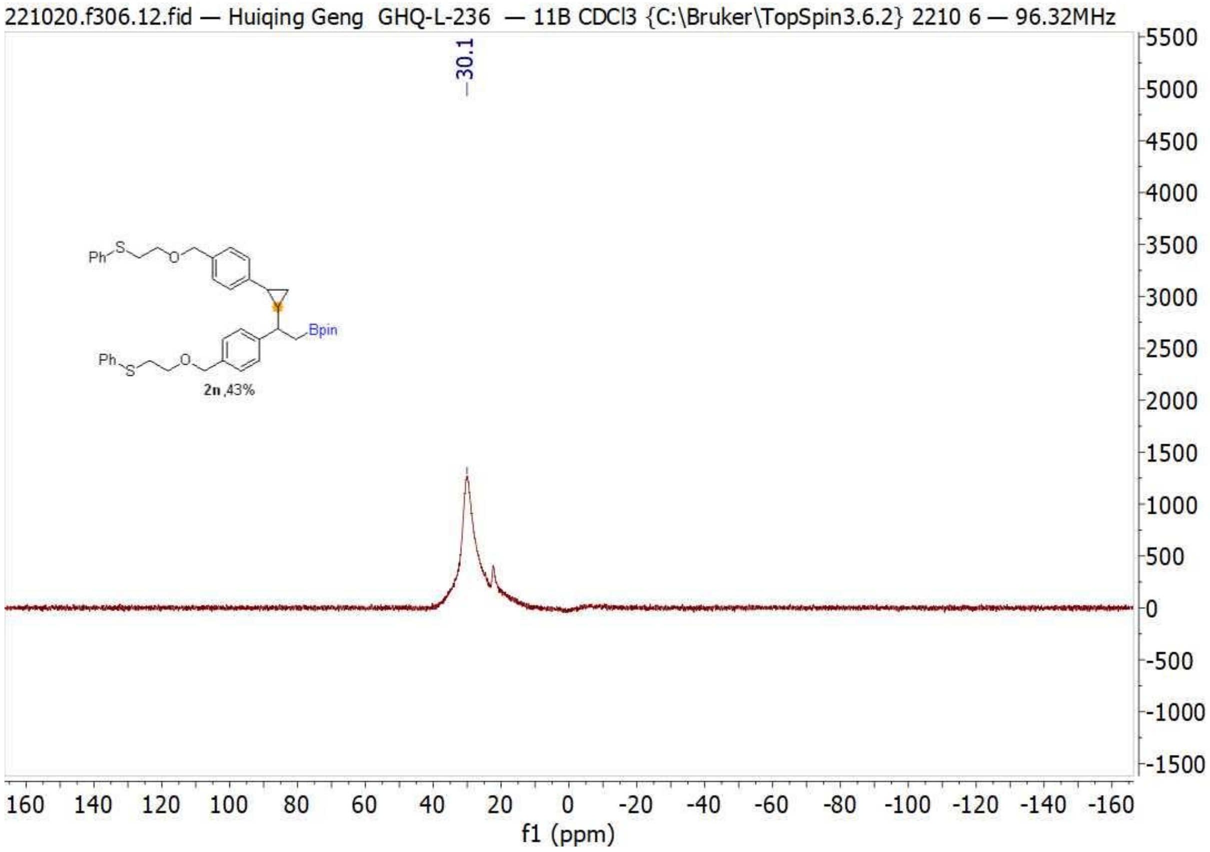


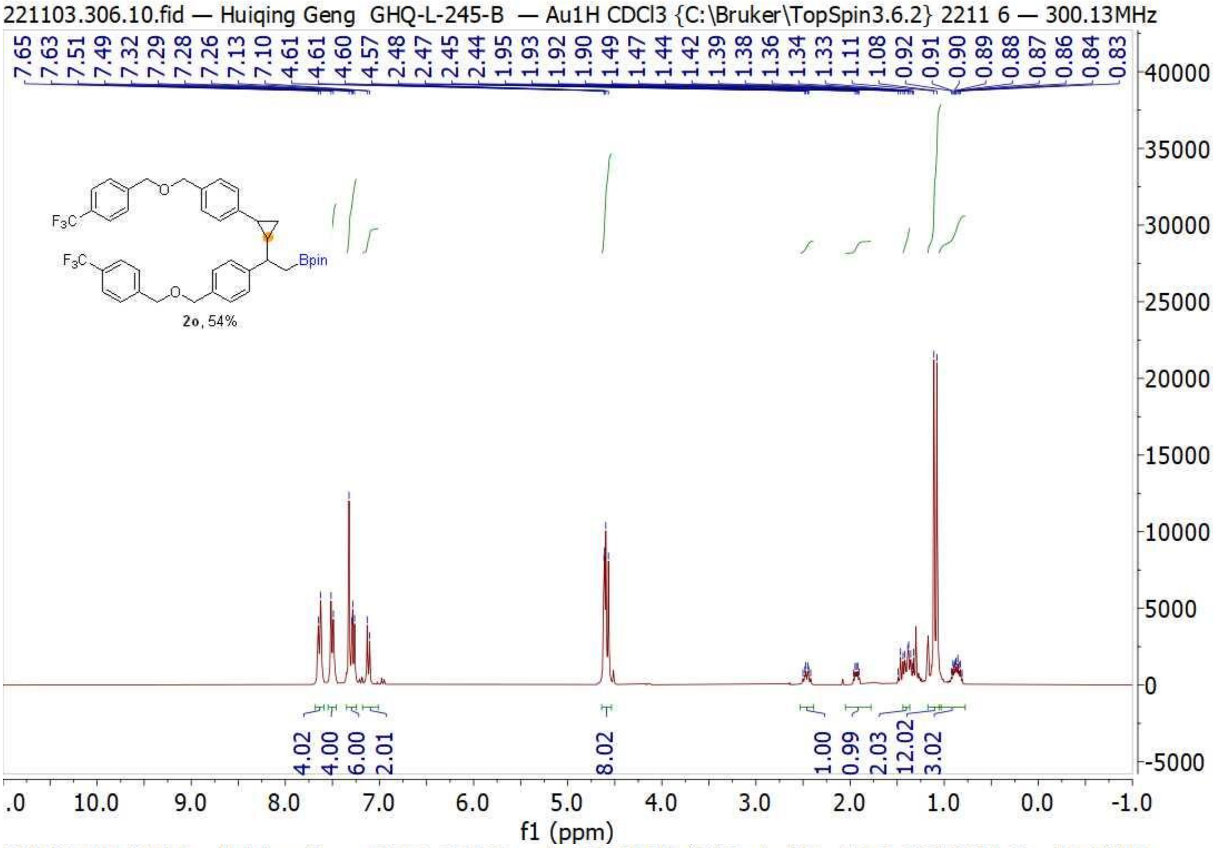

Huiqing Geng GHQ-L-217 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 5 — 96.29MHz
33.24
F3CS
F3CS
Bpin
2m, 73%
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

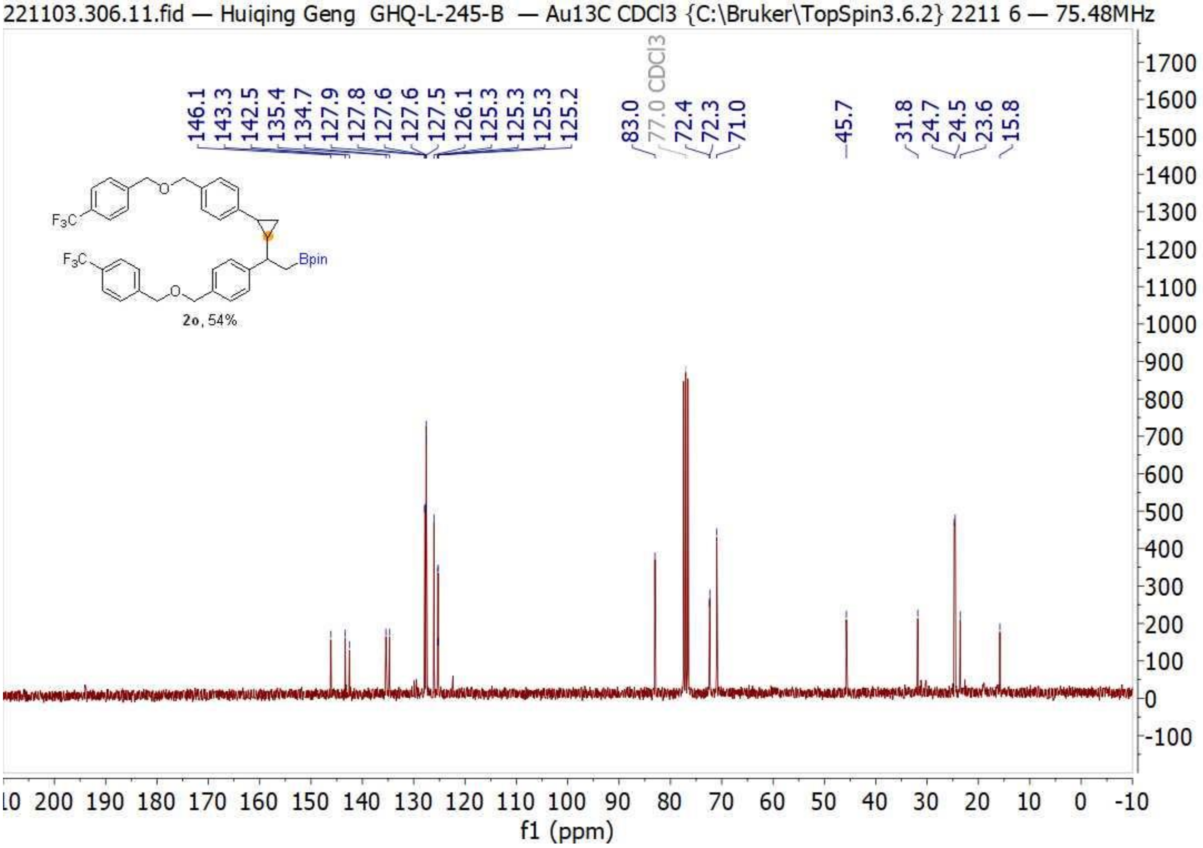

Huiqing Geng GHQ-L-217 — Au19F CDCl3 {C:\Bruker\TopSpin3.6.2} 2302 5 — 282.39MHz
-42.59
-42.61
F3CS
F3CS
Bpin
2m, 73%
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-41.3 -41.5 -41.7 -41.9 -42.1 -42.3 -42.5 -42.7 -42.9 -43.1 -43.3 -43.5 -43.7 -43.9
f1 (ppm)

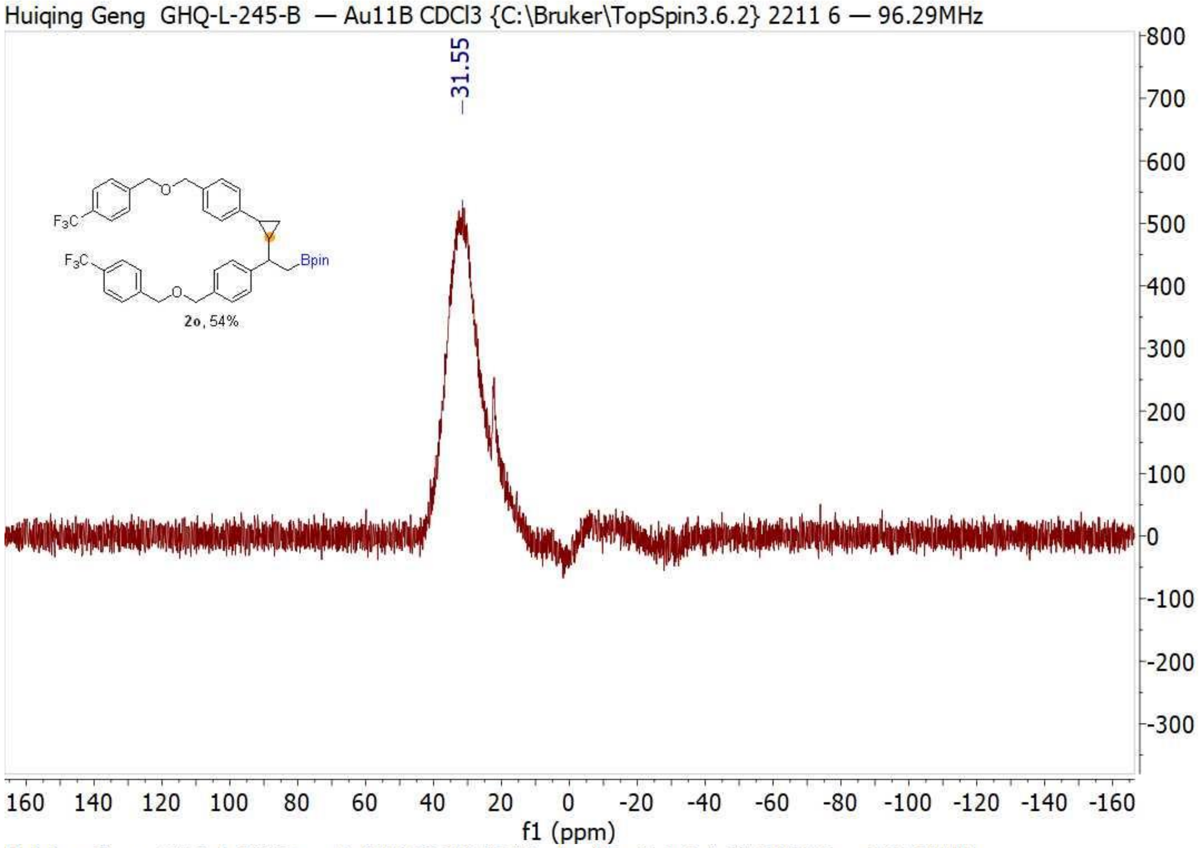

221020.f306.10.fid — Huiqing Geng GHQ-L-236 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 6 — 300.20MHz
7.29 7.29 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.18 7.16 7.12 7.12 7.12 7.11 7.10 6.98 6.95 4.43 4.40 3.59 3.58 3.57 3.56 3.55 3.54 3.07 1.29 1.26 1.26 1.22 0.99 0.96 0.81
Ph S O
Bpin
Ph S O
2n,43%
4.03 8.01 4.03 2.01 4.00 4.00 4.00 1.02 1.03 3.00 12.01 2.00
65000 60000 55000 50000 45000 40000 35000 30000 25000 20000 15000 10000 5000 0 -5000
.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

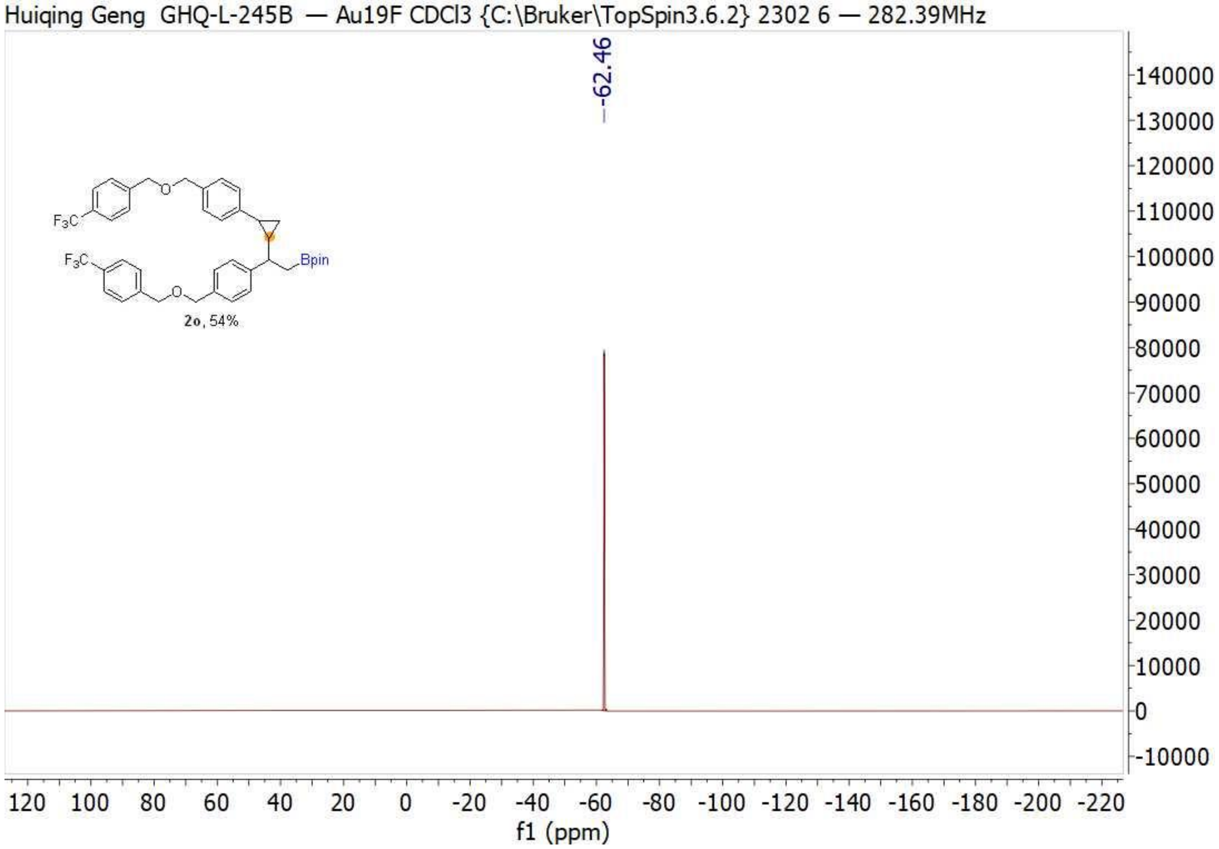

221020.f306.11.fid — Huiqing Geng GHQ-L-236 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 6 — 75.49MHz
146.0 143.2 135.6 134.9 129.4 128.9 127.9 127.7 127.5 126.1 126.1
83.5 83.0 72.9
45.8 33.3 31.8 24.8 24.6 23.6 15.9
Ph S O
Bpin
Ph S O
2n,43%
500 450 400 350 300 250 200 150 100 50 0 -50
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

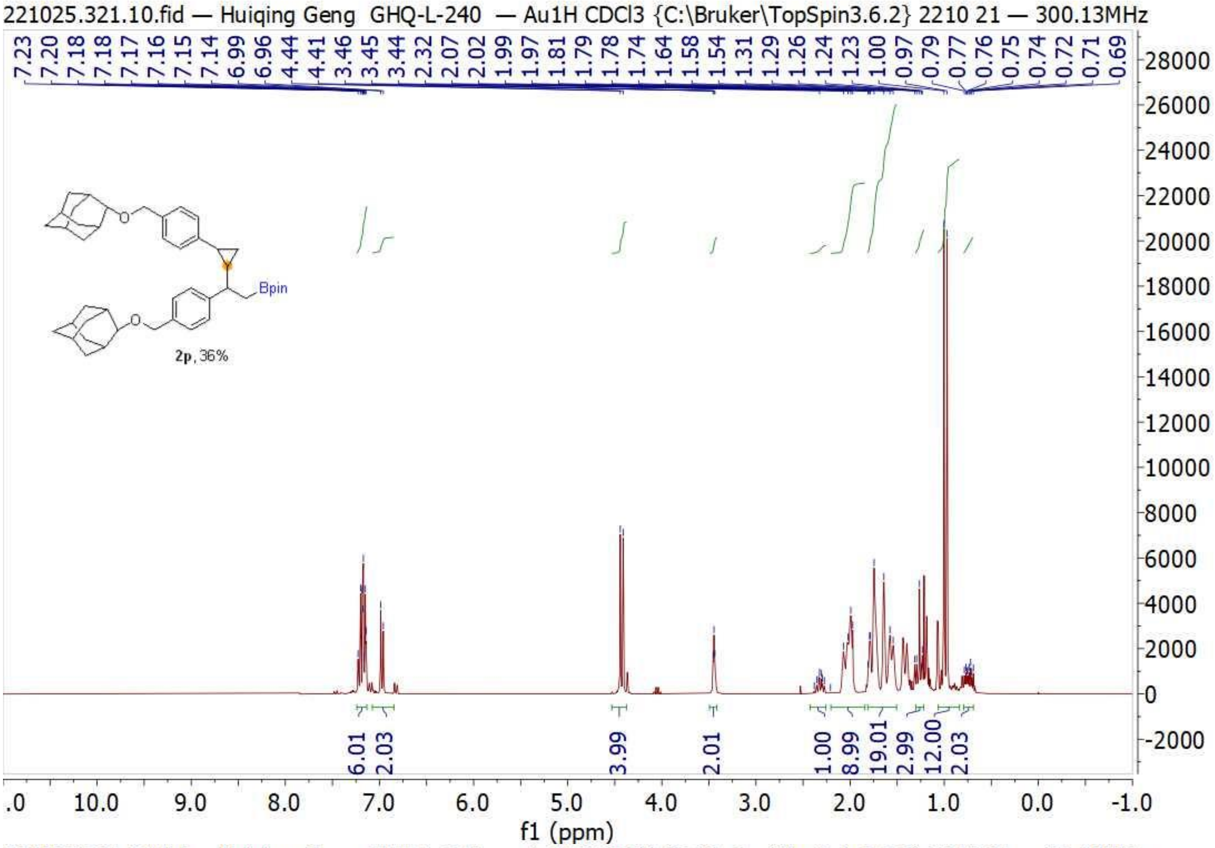

221020.f306.12.fid — Huiqing Geng GHQ-L-236 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 6 — 96.32MHz
30.1
Ph
S
O
Bpin
2n,43%
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
-1500
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

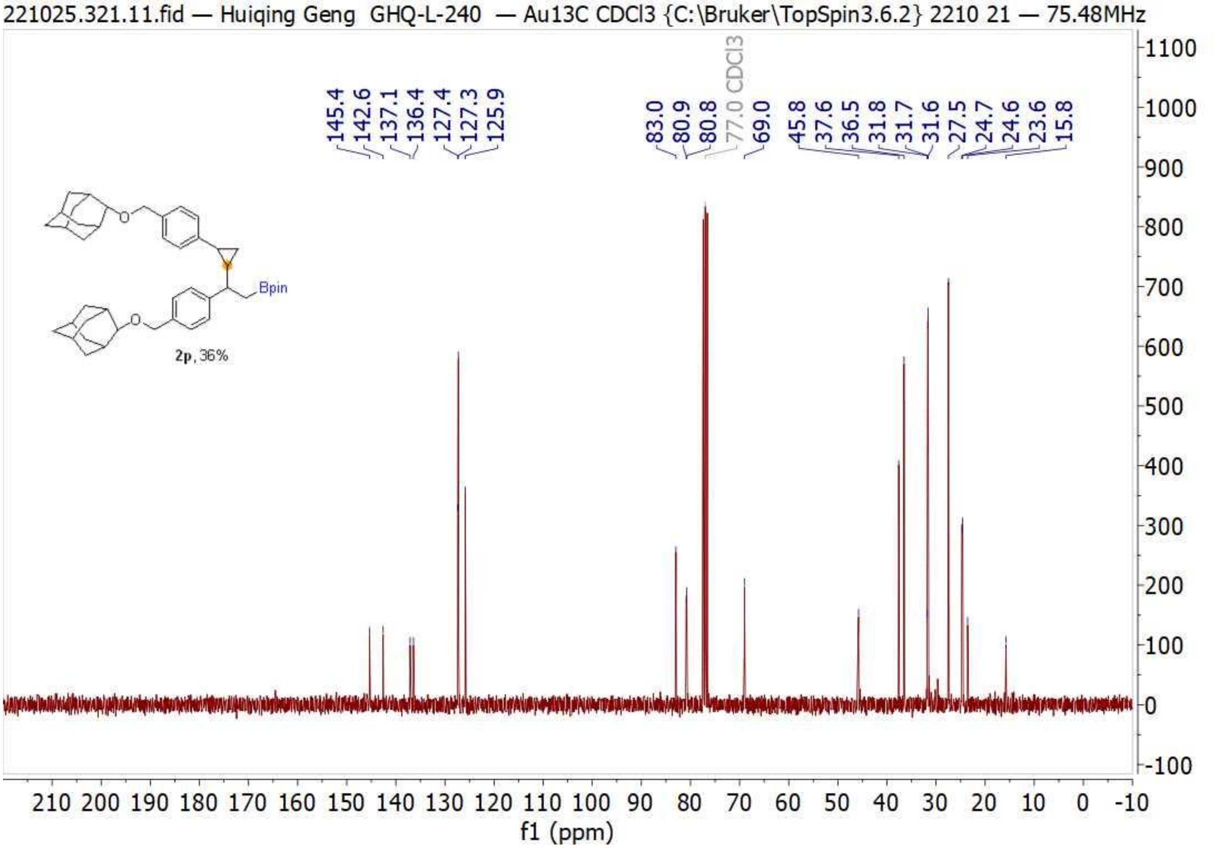

221103.306.10.fid — Huiqing Geng GHQ-L-245-B — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 6 — 300.13MHz
7.65 7.63 7.51 7.49 7.32 7.29 7.28 7.26 7.13 7.10 4.61 4.61 4.60 4.57 2.48 2.47 2.45 2.44 1.95 1.93 1.92 1.90 1.49 1.47 1.44 1.42 1.39 1.38 1.36 1.34 1.33 1.11 1.08 0.92 0.91 0.90 0.89 0.88 0.87 0.86 0.84 0.83
F_3C
Bpin
2o, 54%
4.02 4.00 6.00 2.01 8.02 1.00 0.99 2.03 12.02 3.02
40000 35000 30000 25000 20000 15000 10000 5000 0 -5000
.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

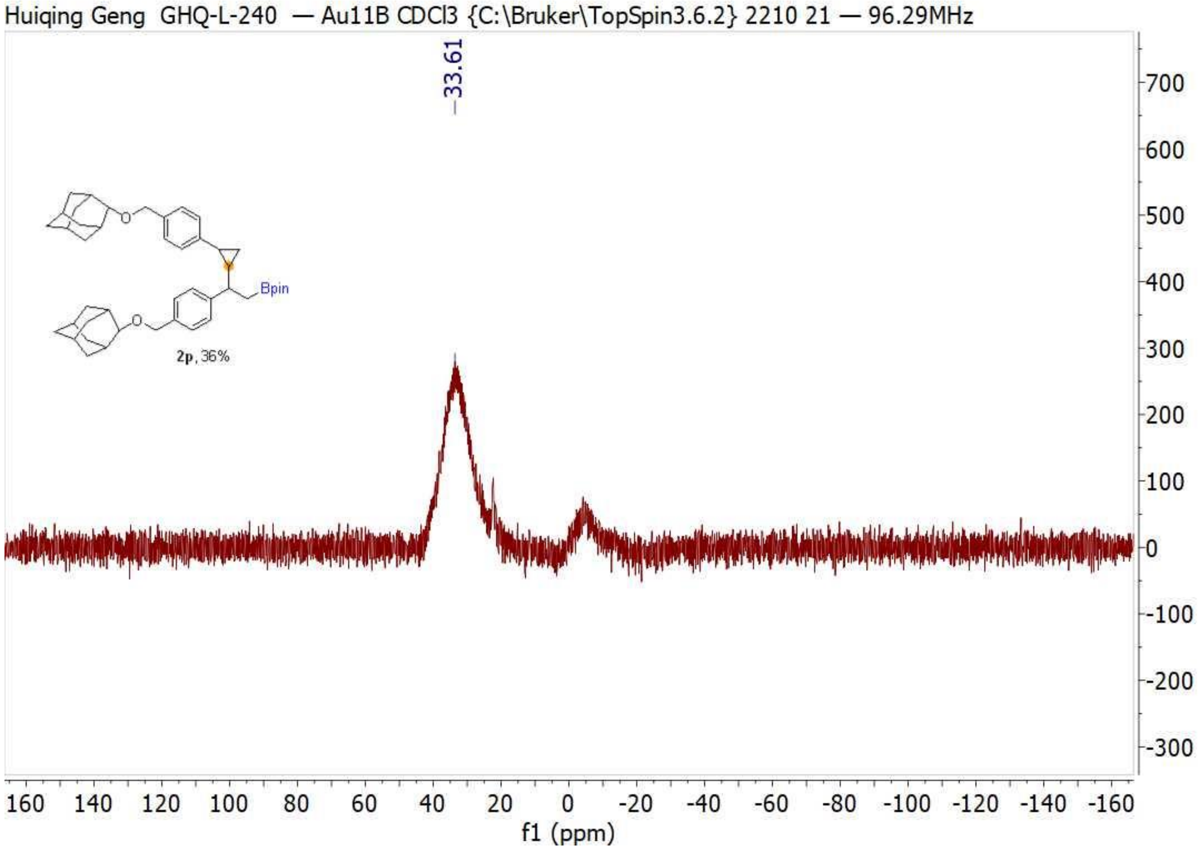

221103.306.11.fid — Huiqing Geng GHQ-L-245-B — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 6 — 75.48MHz
146.1 143.3 142.5 135.4 134.7 127.9 127.8 127.6 127.5 126.1 125.3 125.3 125.3 125.2
83.0 77.0 CDCl3 72.4 72.3 71.0
45.7
31.8 24.7 24.5 23.6 15.8
F_3C
Bpin
2o, 54%
1700 1600 1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 -100
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

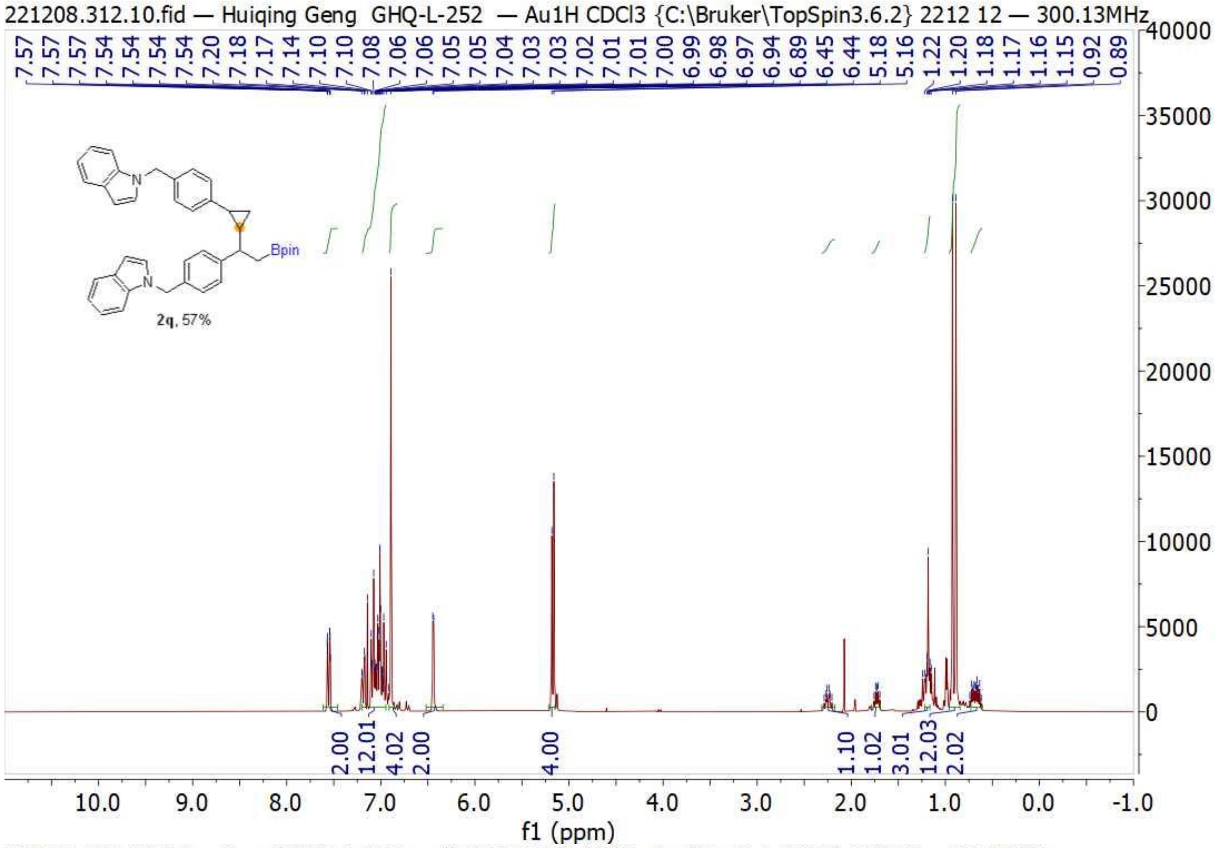

Huiqing Geng GHQ-L-245-B — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 6 — 96.29MHz
31.55
F3C
O
Bpin
F3C
O
2o, 54%
800
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

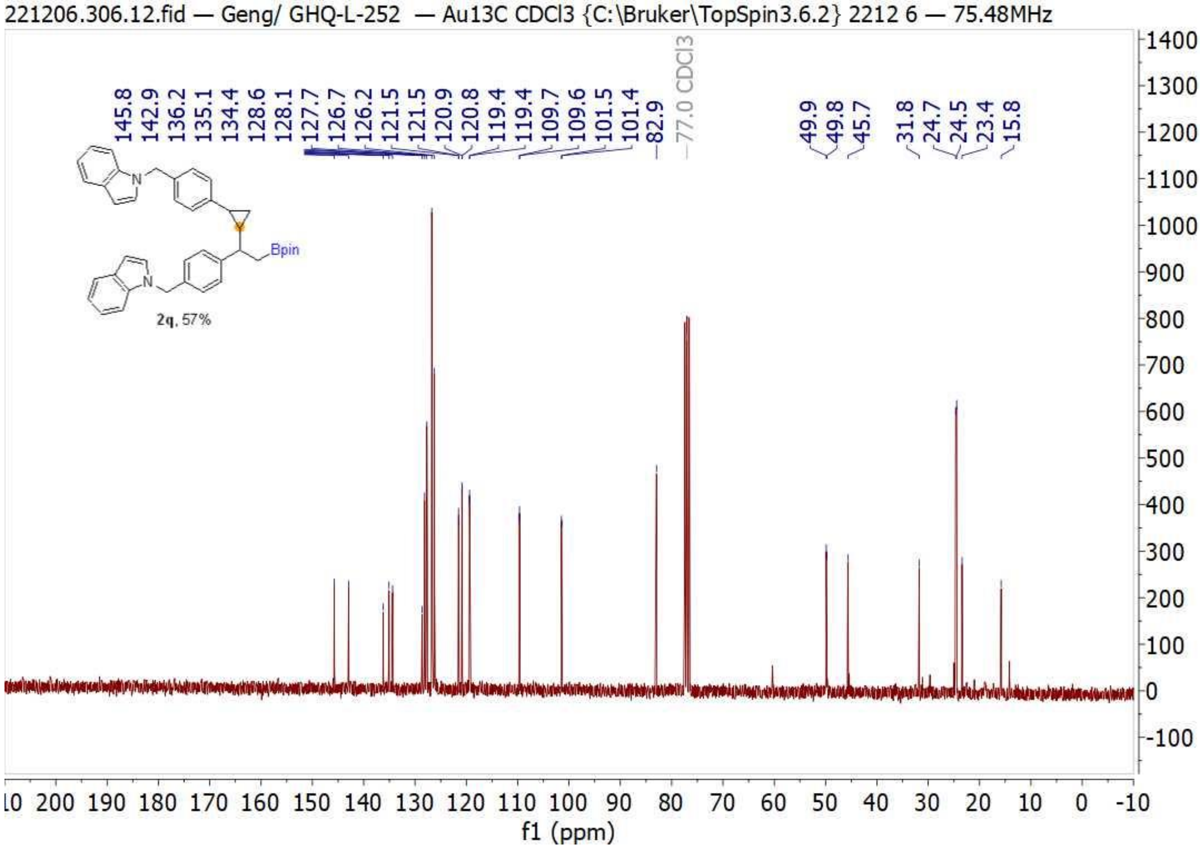

Huiqing Geng GHQ-L-245B — Au19F CDCl3 {C:\Bruker\TopSpin3.6.2} 2302 6 — 282.39MHz
-62.46
F3C
O
Bpin
F3C
O
2o, 54%
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220
f1 (ppm)

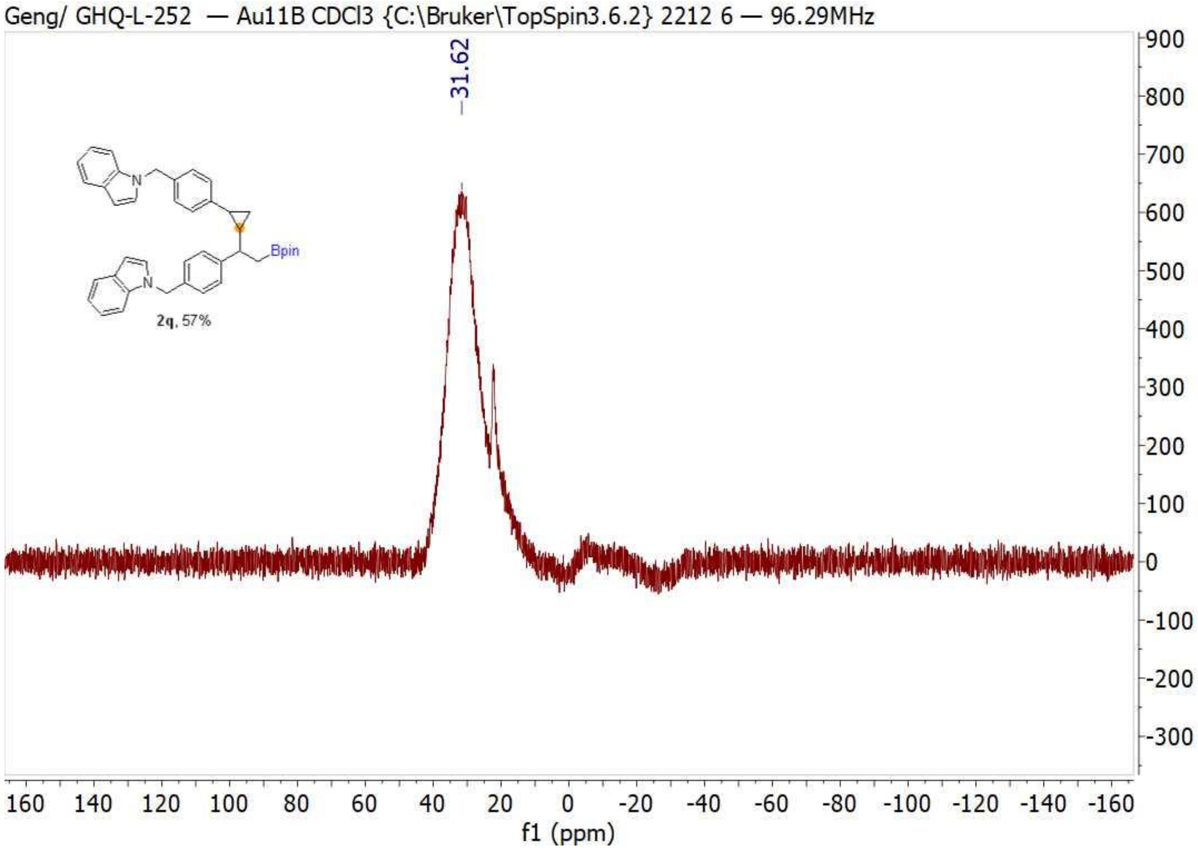

221025.321.10.fid — Huiqing Geng GHQ-L-240 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 21 — 300.13MHz
7.23 7.20 7.18 7.18 7.17 7.16 7.15 7.14 6.99 6.96 4.44 4.41 3.46 3.45 3.44 2.32 2.07 2.02 1.99 1.97 1.81 1.79 1.78 1.74 1.64 1.58 1.54 1.31 1.29 1.26 1.24 1.23 1.00 0.97 0.79 0.77 0.76 0.75 0.74 0.72 0.71 0.69
Bpin
2p, 36%
6.01 2.03 3.99 2.01 1.00 8.99 19.01 2.99 12.00 2.03
28000 26000 24000 22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000
.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

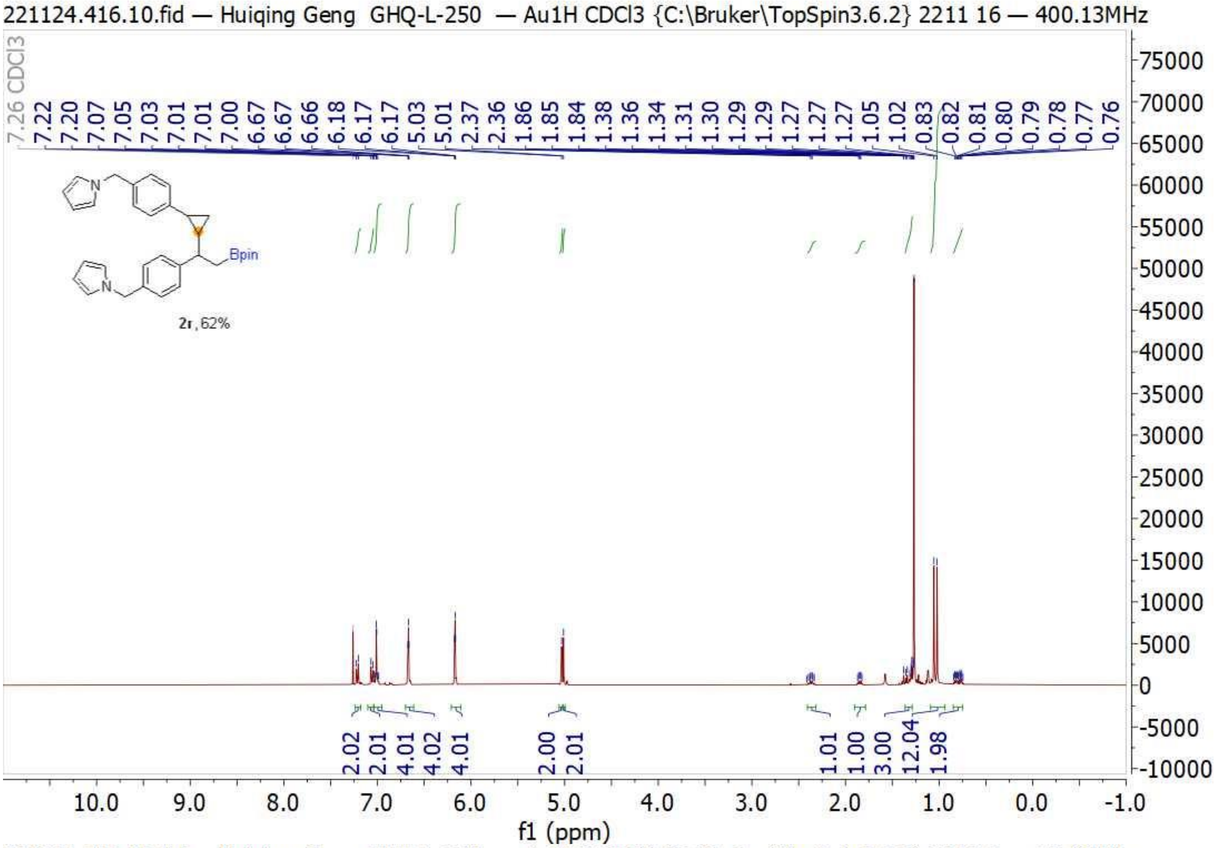

221025.321.11.fid — Huiqing Geng GHQ-L-240 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 21 — 75.48MHz
145.4 142.6 137.1 136.4 127.4 127.3 125.9
83.0 80.9 80.8 77.0 CDCl3 69.0
45.8 37.6 36.5 31.8 31.7 31.6 27.5 24.7 24.6 23.6 15.8
Bpin
2p, 36%
1100 1000 900 800 700 600 500 400 300 200 100 0 -100
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

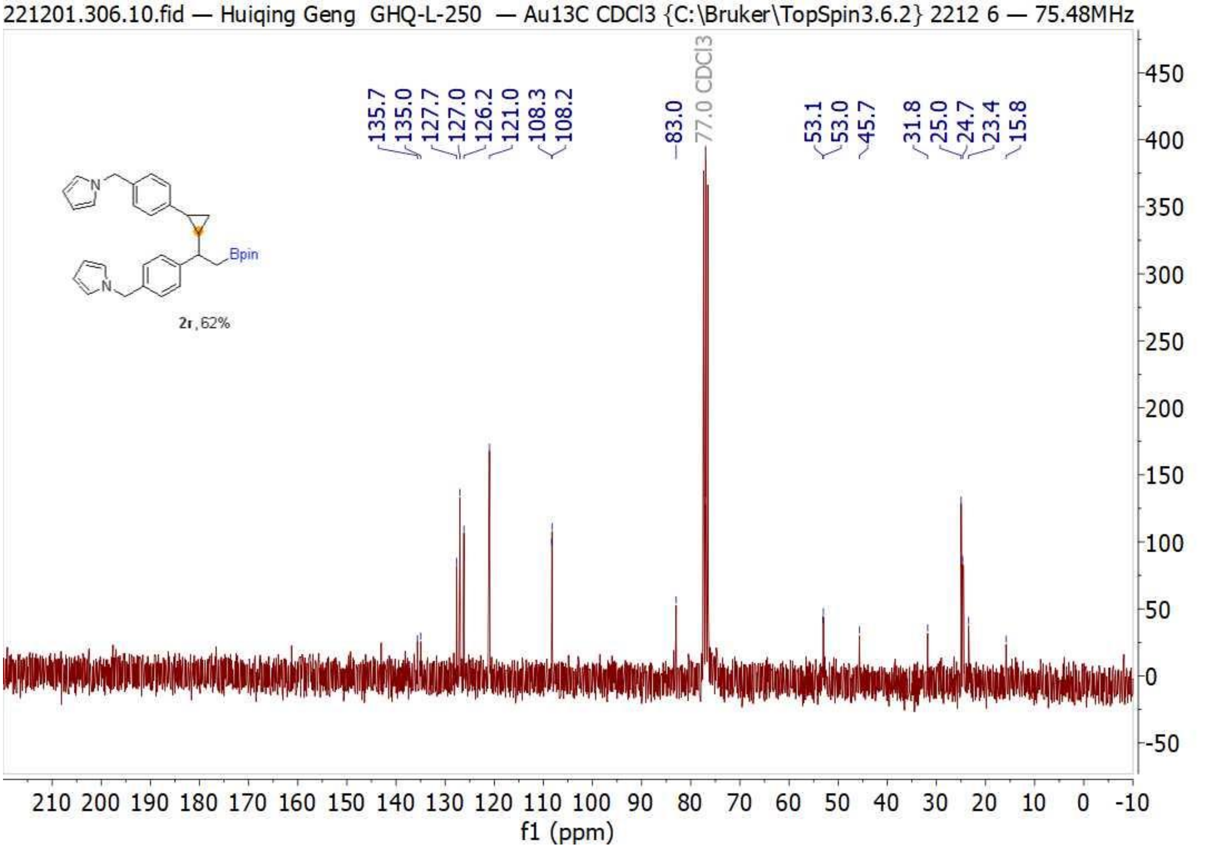

Huiqing Geng GHQ-L-240 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 21 — 96.29MHz
33.61
Bpin
O
O
2p, 36%
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

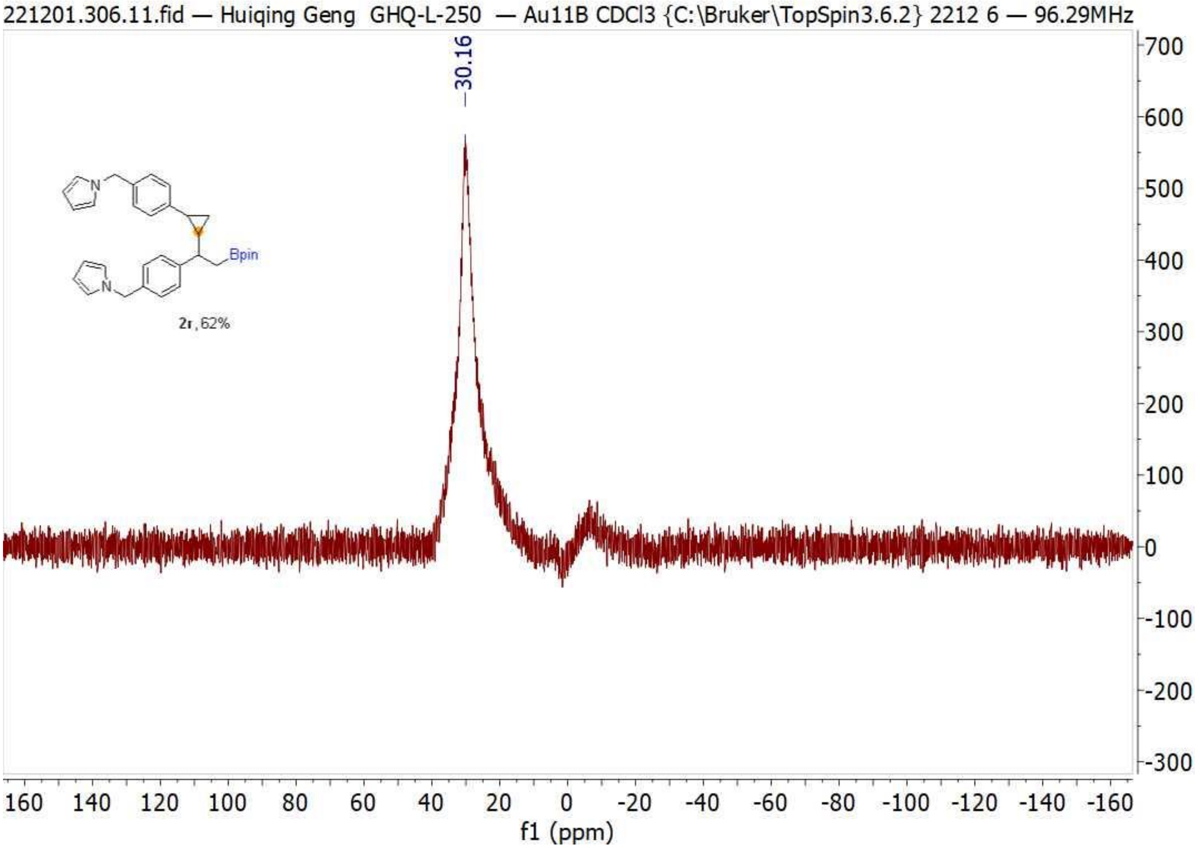

221208.312.10.fid — Huiqing Geng GHQ-L-252 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 12 — 300.13MHz
7.57
7.57
7.57
7.54
7.54
7.54
7.54
7.20
7.18
7.17
7.14
7.10
7.10
7.08
7.06
7.06
7.05
7.05
7.04
7.03
7.03
7.02
7.01
7.01
7.00
6.99
6.98
6.97
6.94
6.89
6.45
6.44
5.18
5.16
1.22
1.20
1.18
1.17
1.16
1.15
0.92
0.89
N
N
Bpin
2q, 57%
2.00
12.01
4.02
2.00
4.00
1.10
1.02
3.01
12.03
2.02
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

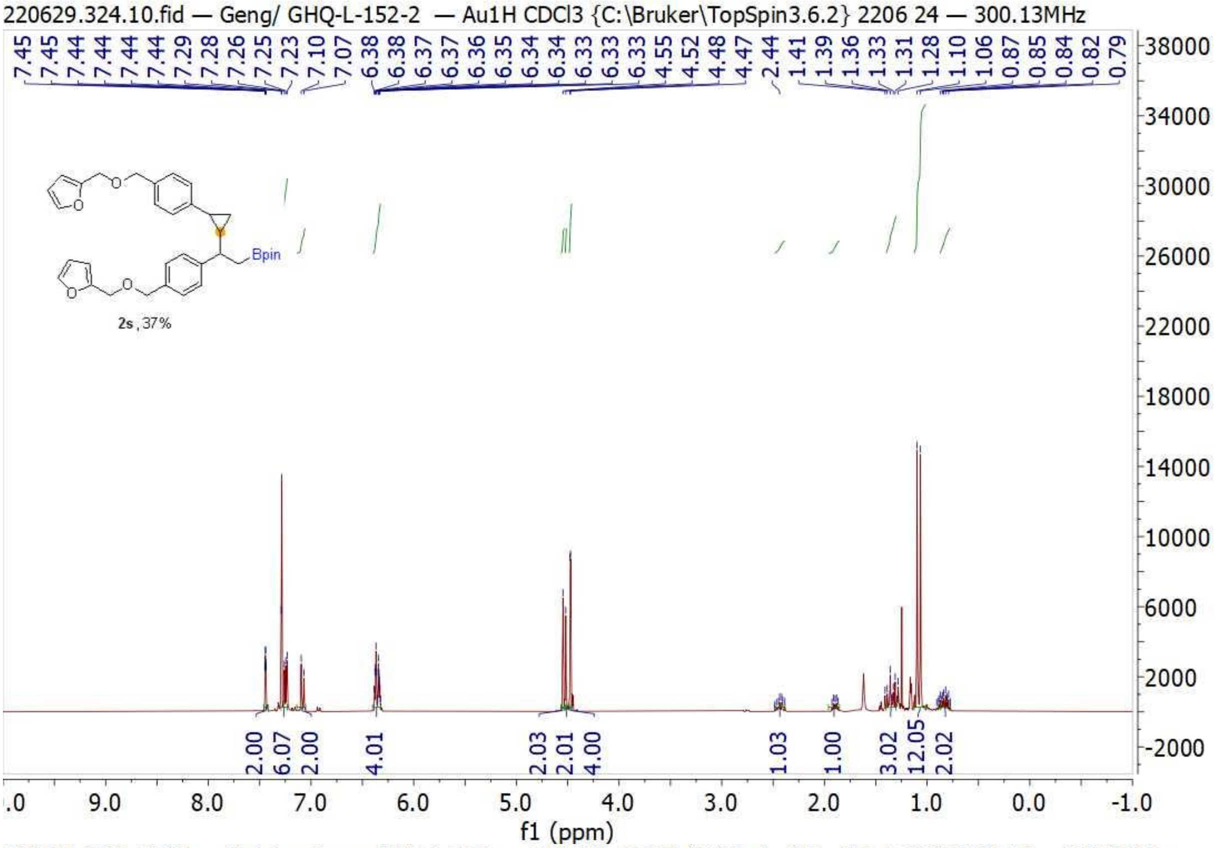

221206.306.12.fid — Geng/ GHQ-L-252 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 6 — 75.48MHz
145.8
142.9
136.2
135.1
134.4
128.6
128.1
127.7
126.7
126.2
121.5
121.5
120.9
120.8
119.4
119.4
109.7
109.6
101.5
101.4
82.9
77.0 CDCl3
49.9
49.8
45.7
31.8
24.7
24.5
23.4
15.8
N
N
Bpin
2q, 57%
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

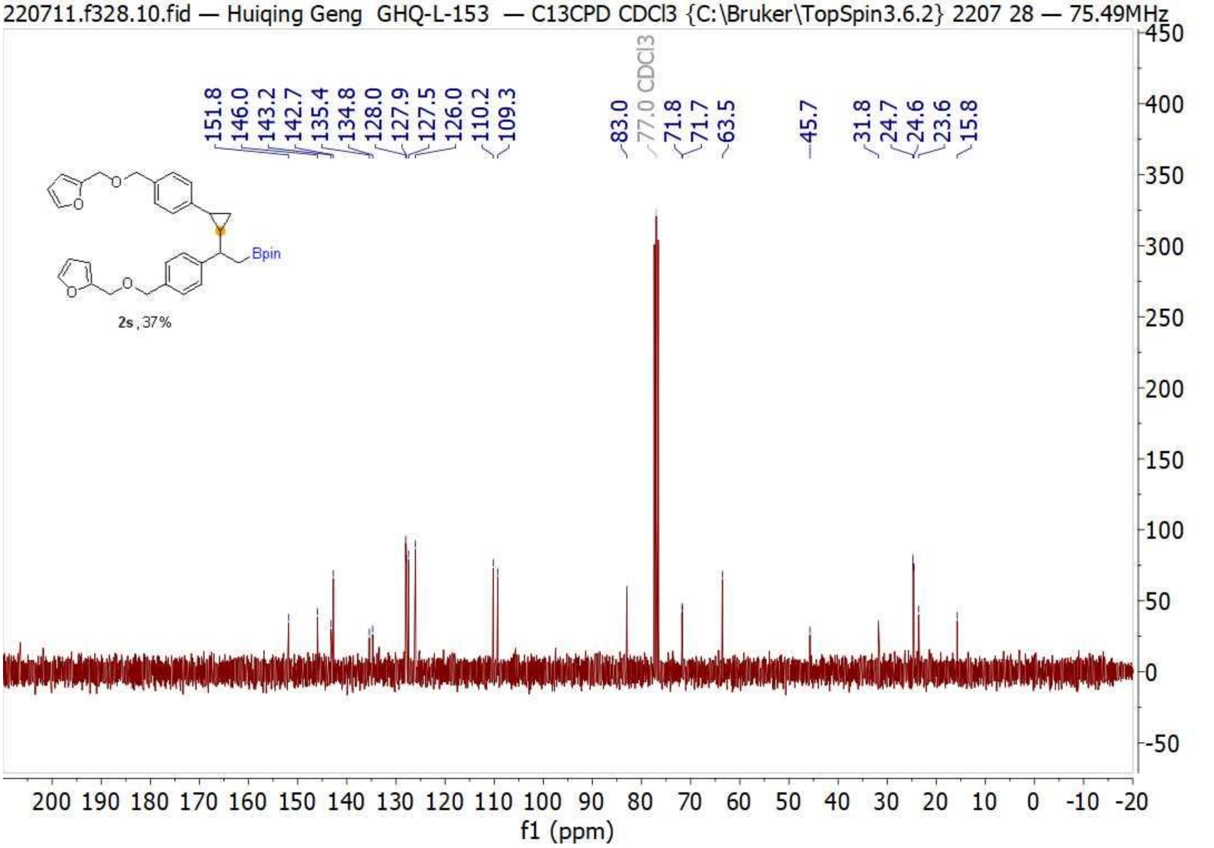

Geng/ GHQ-L-252 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 6 — 96.29MHz
—31.62
N
N
Bpin
2q, 57%
900
800
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

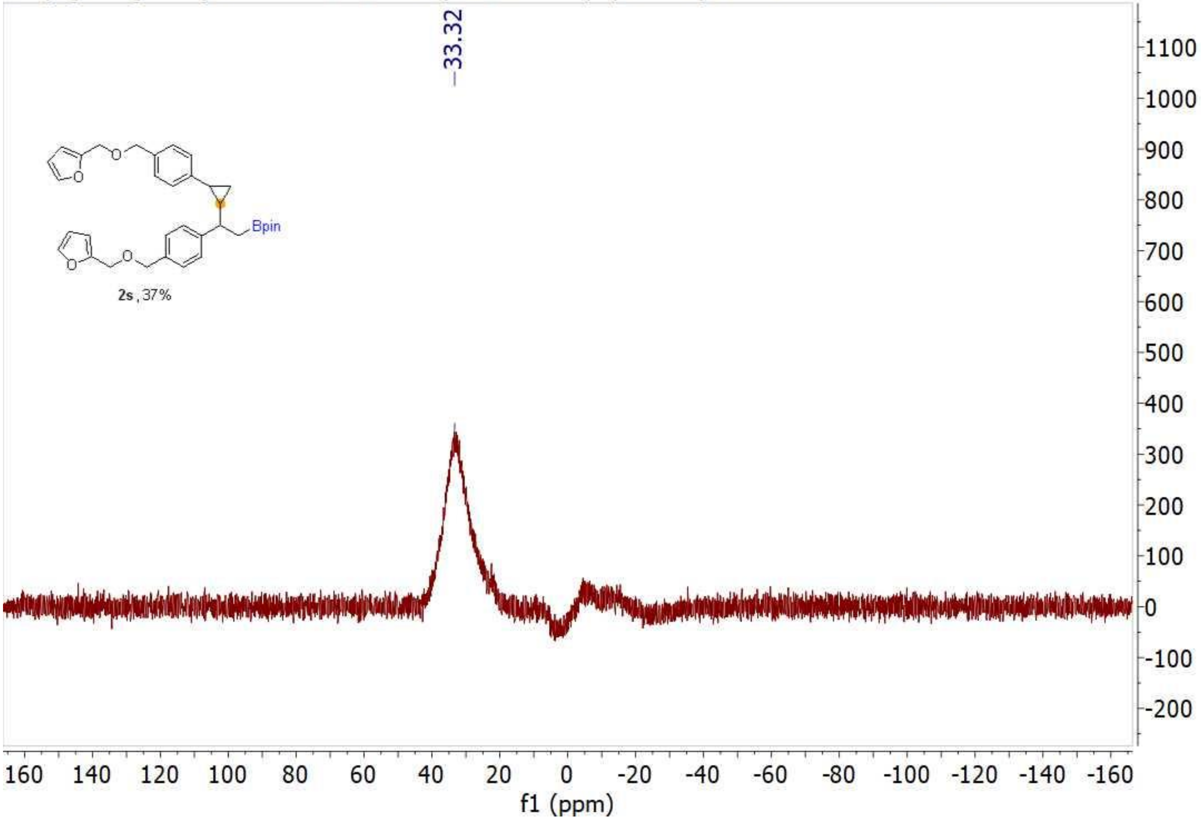

221124.416.10.fid — Huiqing Geng GHQ-L-250 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 16 — 400.13MHz
7.26 CDCl3
7.22
7.20
7.07
7.05
7.03
7.01
7.01
7.00
6.67
6.67
6.66
6.18
6.17
6.17
5.03
5.01
2.37
2.36
1.86
1.85
1.84
1.38
1.36
1.34
1.31
1.30
1.29
1.29
1.27
1.27
1.27
1.05
1.02
0.83
0.82
0.81
0.80
0.79
0.78
0.77
0.76
Bpin
2r, 62%
2.02
2.01
4.01
4.02
4.01
2.00
2.01
1.01
1.00
3.00
12.04
1.98
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-10000
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


221201.306.10.fid — Huiqing Geng GHQ-L-250 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 6 — 75.48MHz
135.7
135.0
127.7
127.0
126.2
121.0
108.3
108.2
83.0
77.0 CDCl3
53.1
53.0
45.7
31.8
25.0
24.7
23.4
15.8
Bpin
2r, 62%
450
400
350
300
250
200
150
100
50
0
-50
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


221201.306.11.fid — Huiqing Geng GHQ-L-250 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 6 — 96.29MHz
30.16
N
N
Bpin
2r, 62%
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)

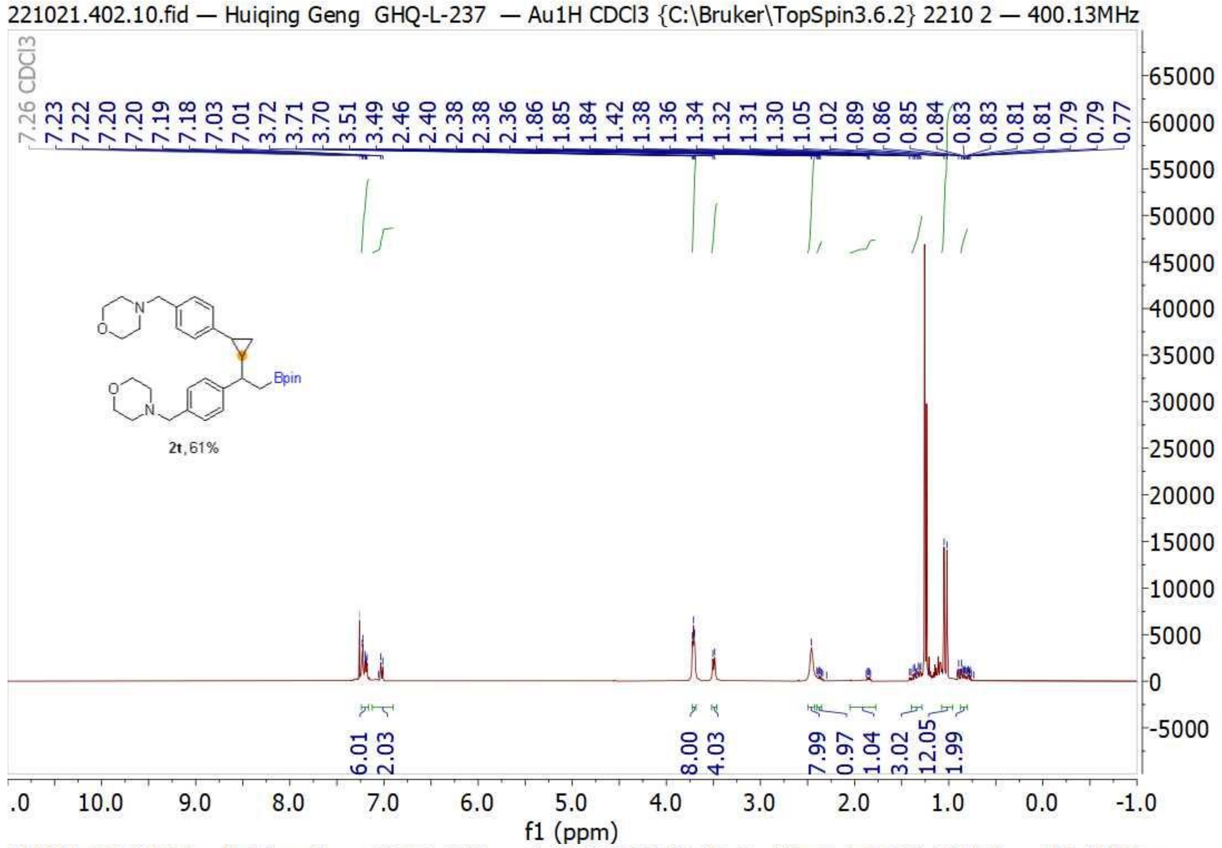

220629.324.10.fid — Geng/ GHQ-L-152-2 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2206 24 — 300.13MHz
7.45 7.45 7.44 7.44 7.44 7.44 7.29 7.28 7.26 7.25 7.23 7.10 7.07 6.38 6.38 6.37 6.37 6.36 6.35 6.34 6.34 6.33 6.33 6.33 4.55 4.52 4.48 4.47 2.44 1.41 1.39 1.36 1.33 1.31 1.28 1.10 1.06 0.87 0.85 0.84 0.82 0.79
Bpin
2s, 37%
2.00 6.07 2.00 4.01 2.03 2.01 4.00 1.03 1.00 3.02 12.05 2.02
38000 34000 30000 26000 22000 18000 14000 10000 6000 2000 -2000
.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

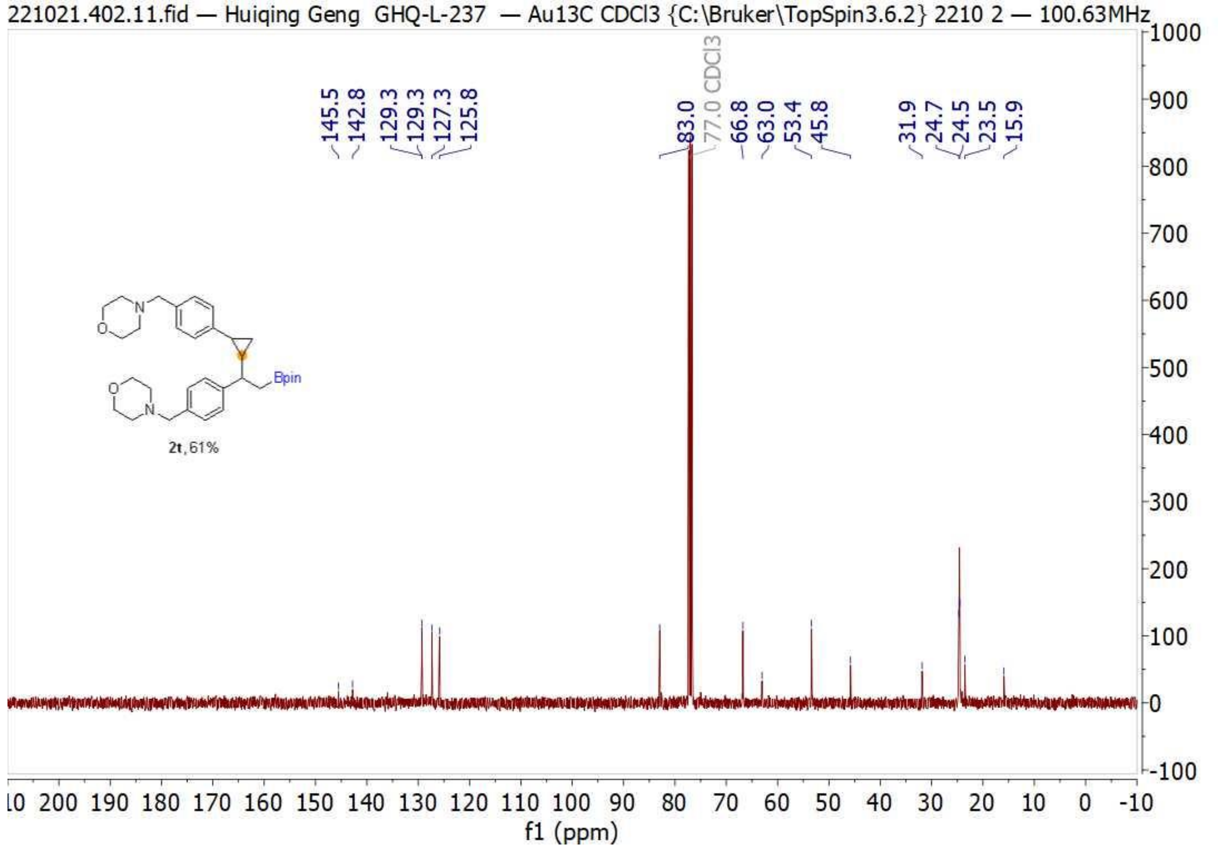

220711.f328.10.fid — Huiqing Geng GHQ-L-153 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 28 — 75.49MHz
151.8 146.0 143.2 142.7 135.4 134.8 128.0 127.9 127.5 126.0 110.2 109.3 83.0 77.0 CDCl3 71.8 71.7 63.5 45.7 31.8 24.7 24.6 23.6 15.8
Bpin
2s, 37%
450 400 350 300 250 200 150 100 50 0 -50
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

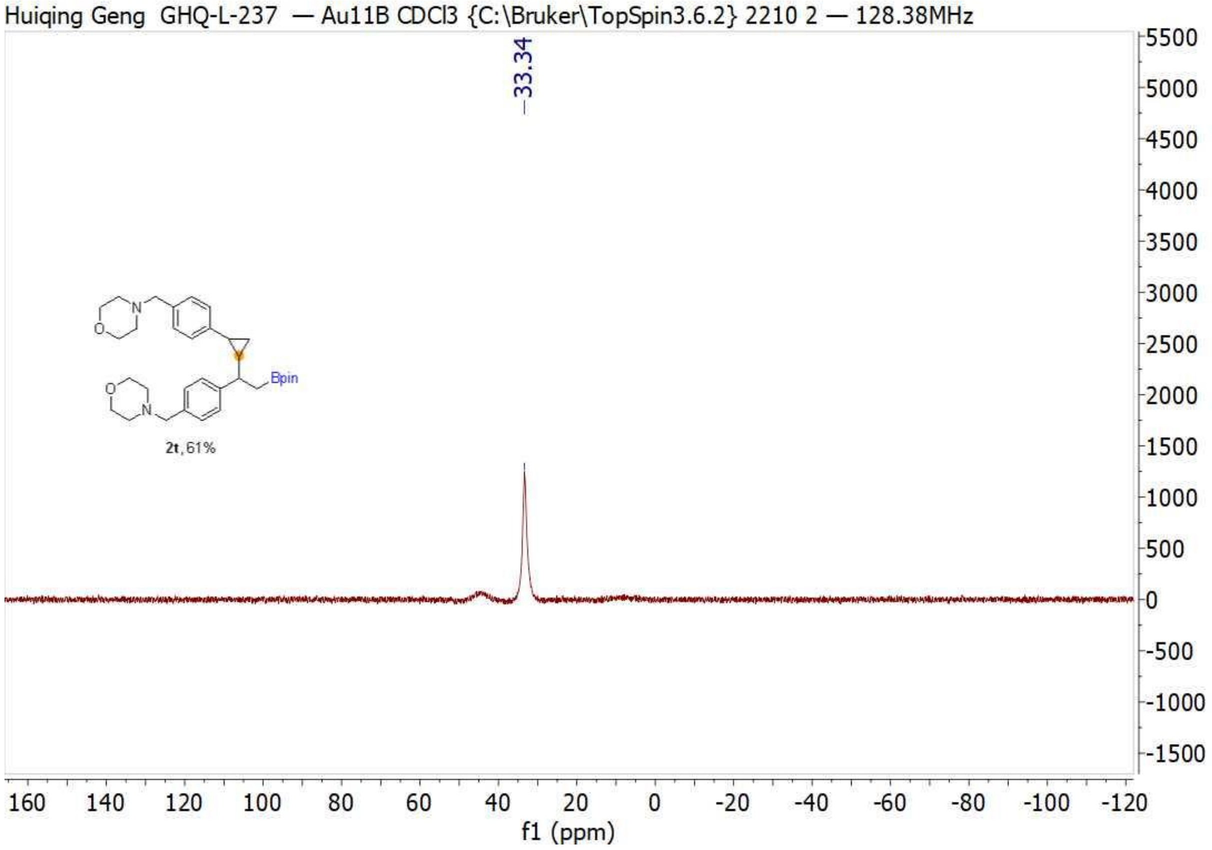


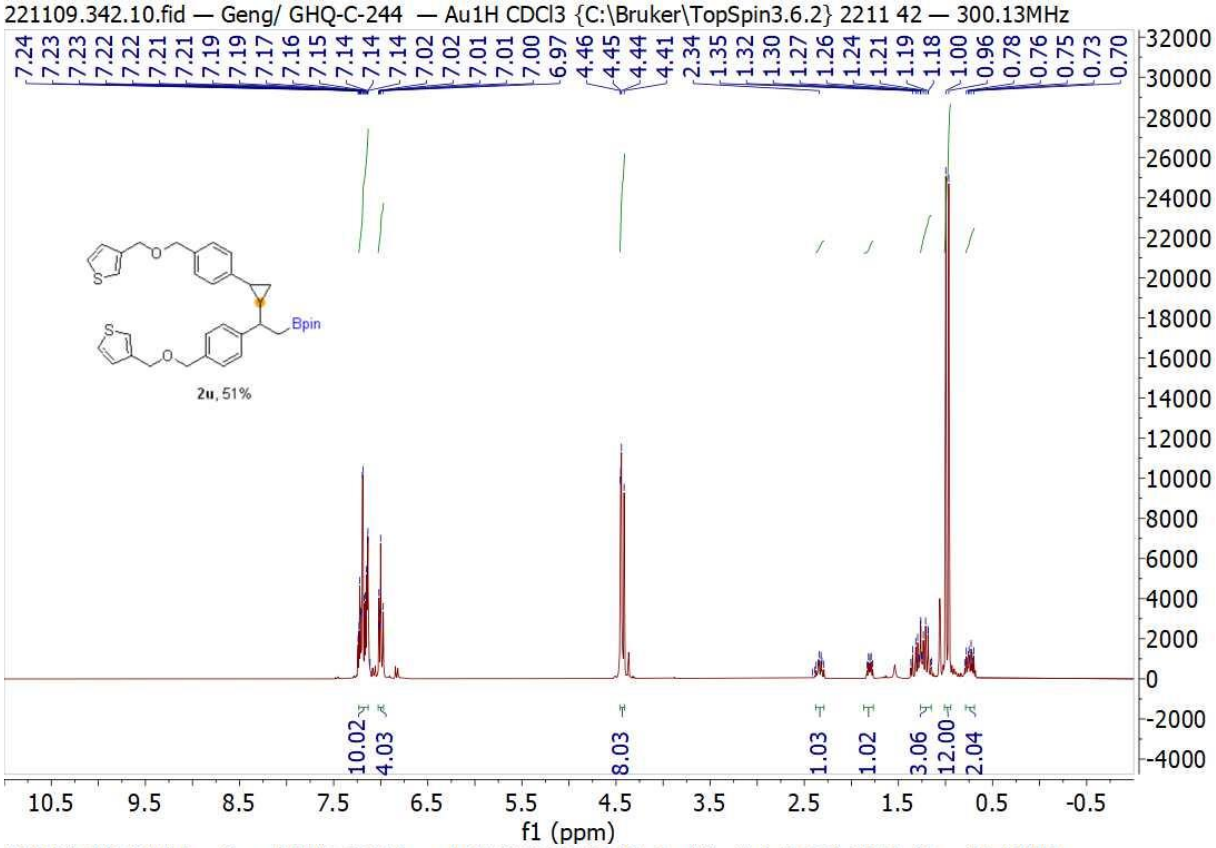
Huiqing Geng GHQ-L-153 — 11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2207 28 — 96.32MHz

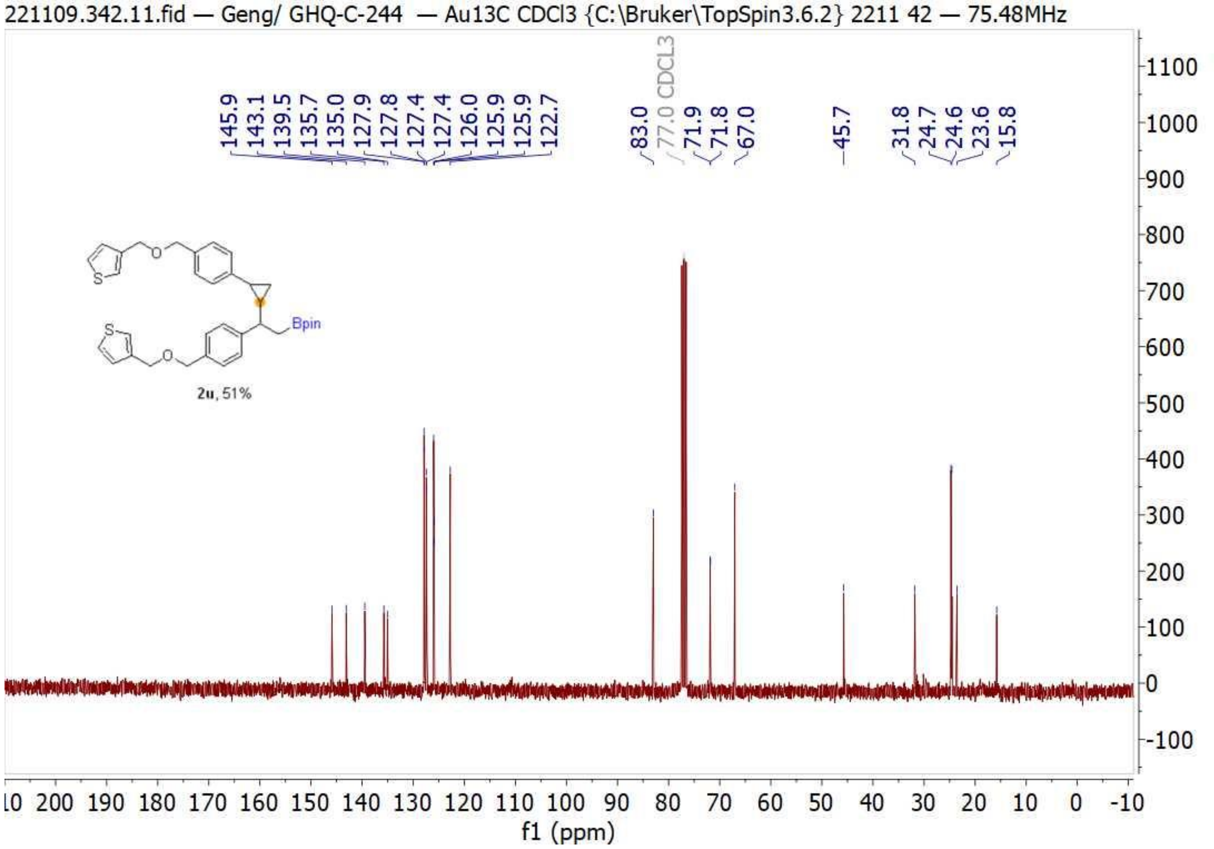
2s, 37%

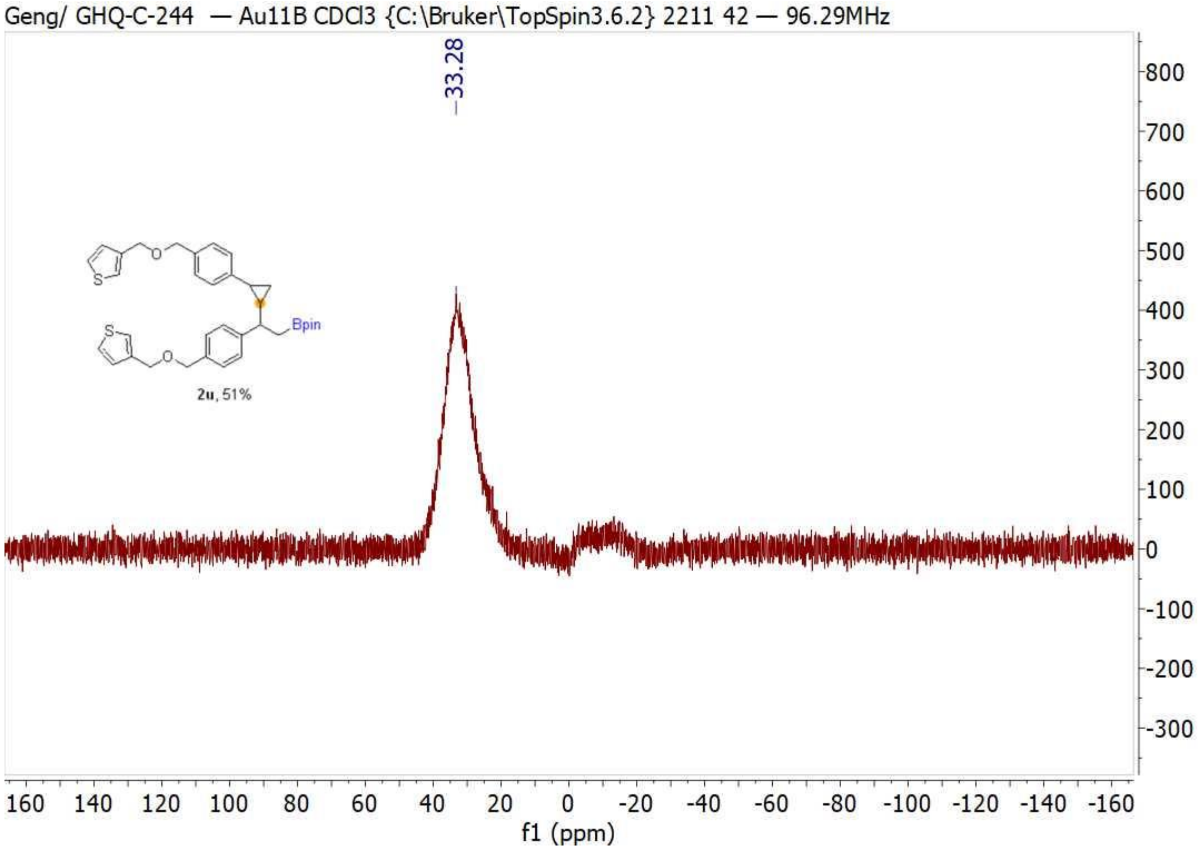

221021.402.10.fid — Huiqing Geng GHQ-L-237 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 2 — 400.13MHz
7.26 CDCl3
7.23
7.22
7.20
7.20
7.19
7.18
7.03
7.01
3.72
3.71
3.70
3.51
3.49
2.46
2.40
2.38
2.38
2.36
1.86
1.85
1.84
1.42
1.38
1.36
1.34
1.32
1.31
1.30
1.05
1.02
0.89
0.86
0.85
0.84
0.83
0.83
0.81
0.81
0.79
0.79
0.77
Bpin
N
O
N
O
2t, 61%
6.01
2.03
8.00
4.03
7.99
0.97
1.04
3.02
12.05
1.99
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)

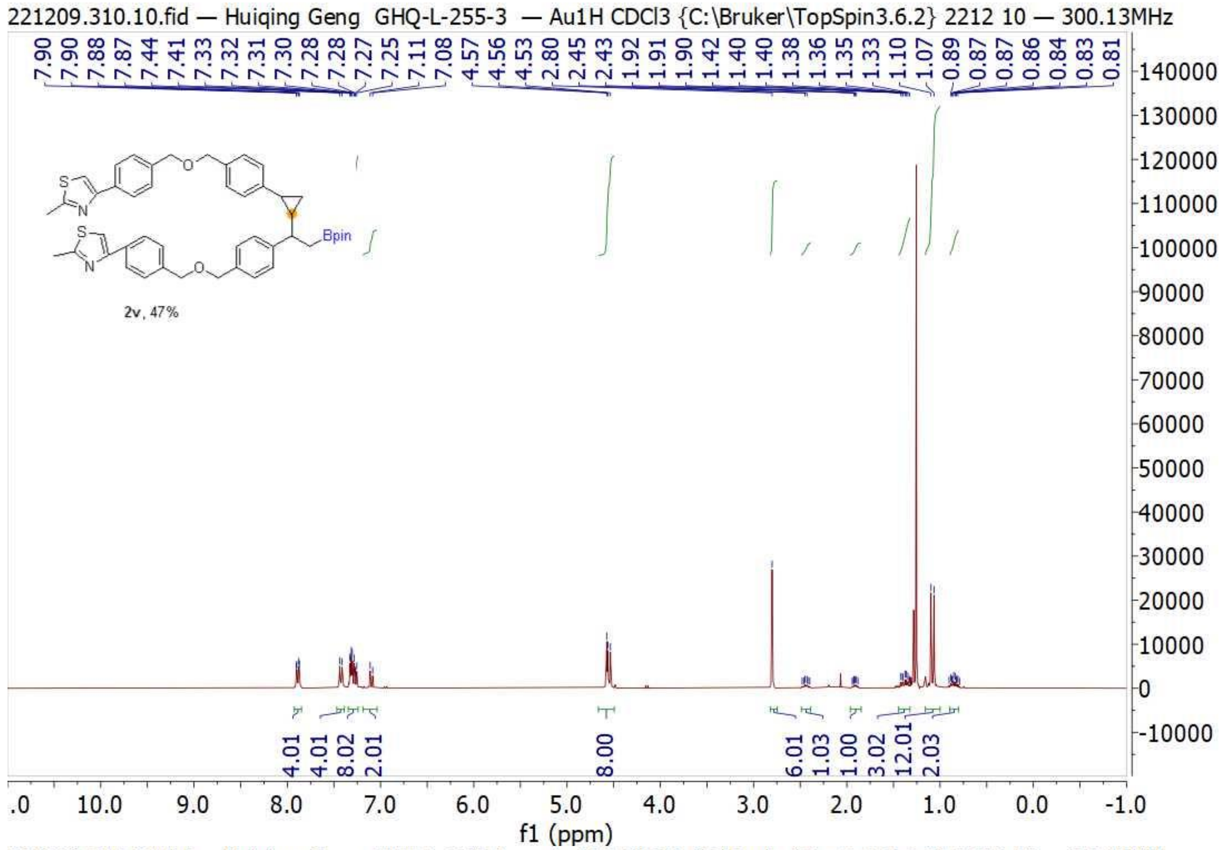

221021.402.11.fid — Huiqing Geng GHQ-L-237 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 2 — 100.63MHz
145.5
142.8
129.3
129.3
127.3
125.8
83.0
77.0 CDCl3
66.8
63.0
53.4
45.8
31.9
24.7
24.5
23.5
15.9
Bpin
N
O
N
O
2t, 61%
1000
900
800
700
600
500
400
300
200
100
0
-100
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

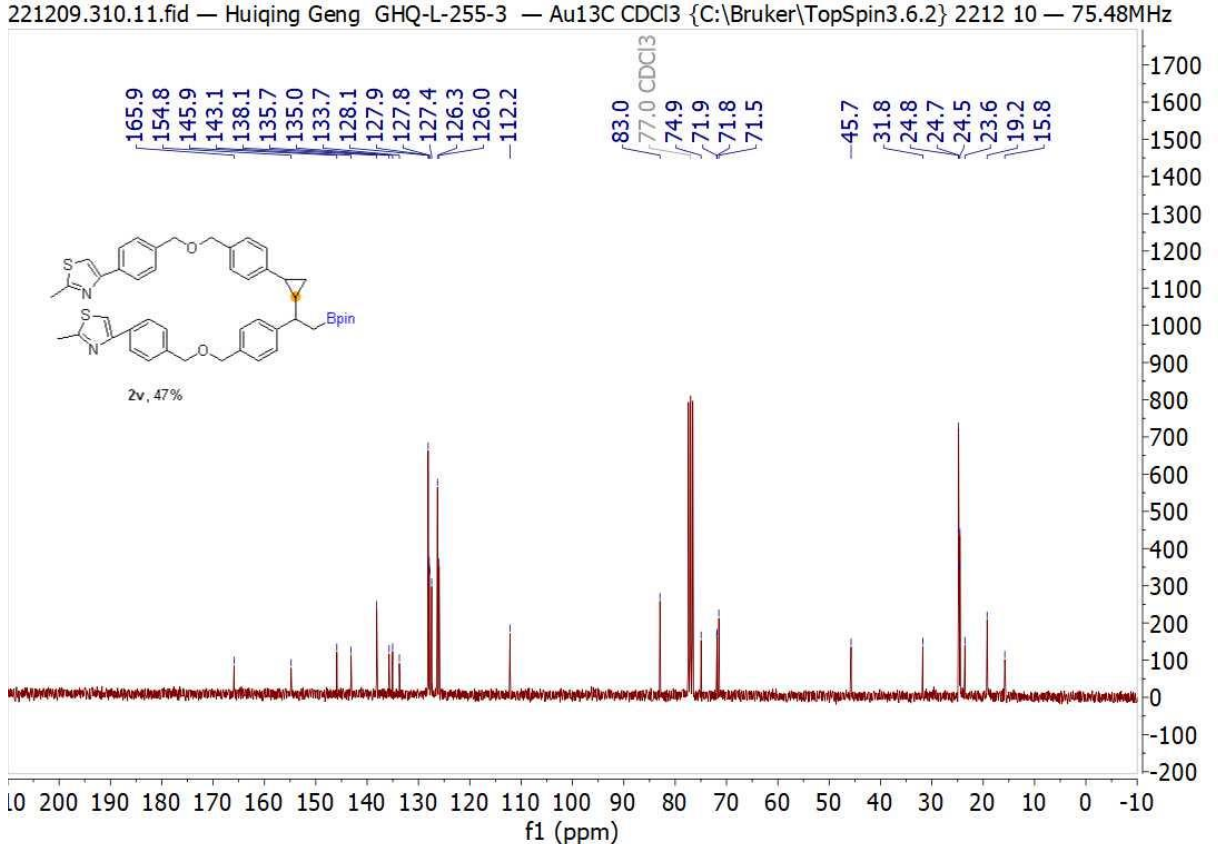

Huiqing Geng GHQ-L-237 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 2 — 128.38MHz
—33.34
Bpin
O
N
O
N
2t, 61%
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
-1500
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
f1 (ppm)

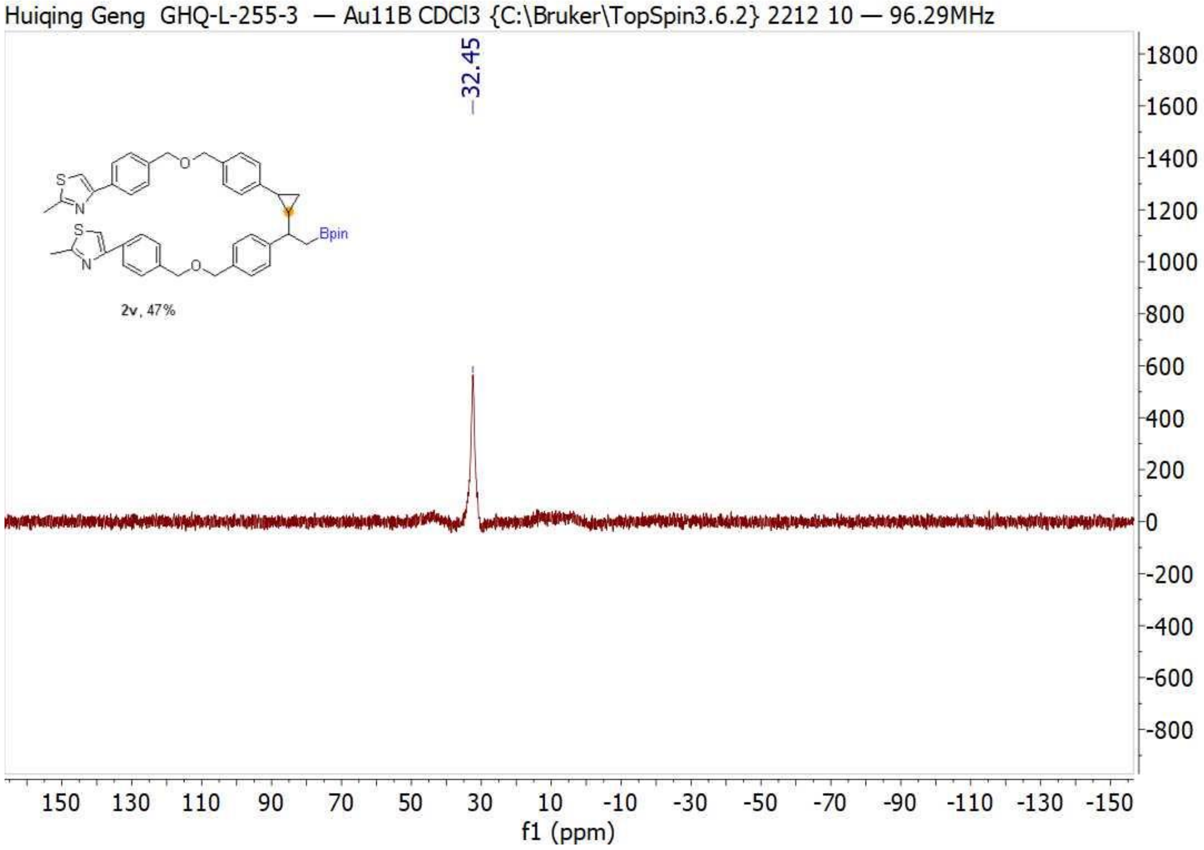

221109.342.10.fid — Geng/ GHQ-C-244 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 42 — 300.13MHz
7.24 7.23 7.23 7.22 7.22 7.21 7.21 7.19 7.19 7.17 7.16 7.15 7.14 7.14 7.14 7.02 7.02 7.01 7.01 7.00 6.97 4.46 4.45 4.44 4.41 2.34 1.35 1.32 1.30 1.27 1.26 1.24 1.21 1.19 1.18 1.00 0.96 0.78 0.76 0.75 0.73 0.70
S
O
S
O
Bpin
2u, 51%
10.02
4.03
8.03
1.03
1.02
3.06
12.00
2.04
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)
32000 30000 28000 26000 24000 22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000 -4000


221109.342.11.fid — Geng/ GHQ-C-244 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 42 — 75.48MHz
145.9 143.1 139.5 135.7 135.0 127.9 127.8 127.4 127.4 126.0 125.9 125.9 122.7
83.0 77.0 CDCL3 71.9 71.8 67.0
45.7
31.8 24.7 24.6 23.6 15.8
S
O
S
O
Bpin
2u, 51%
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
1100 1000 900 800 700 600 500 400 300 200 100 0 -100

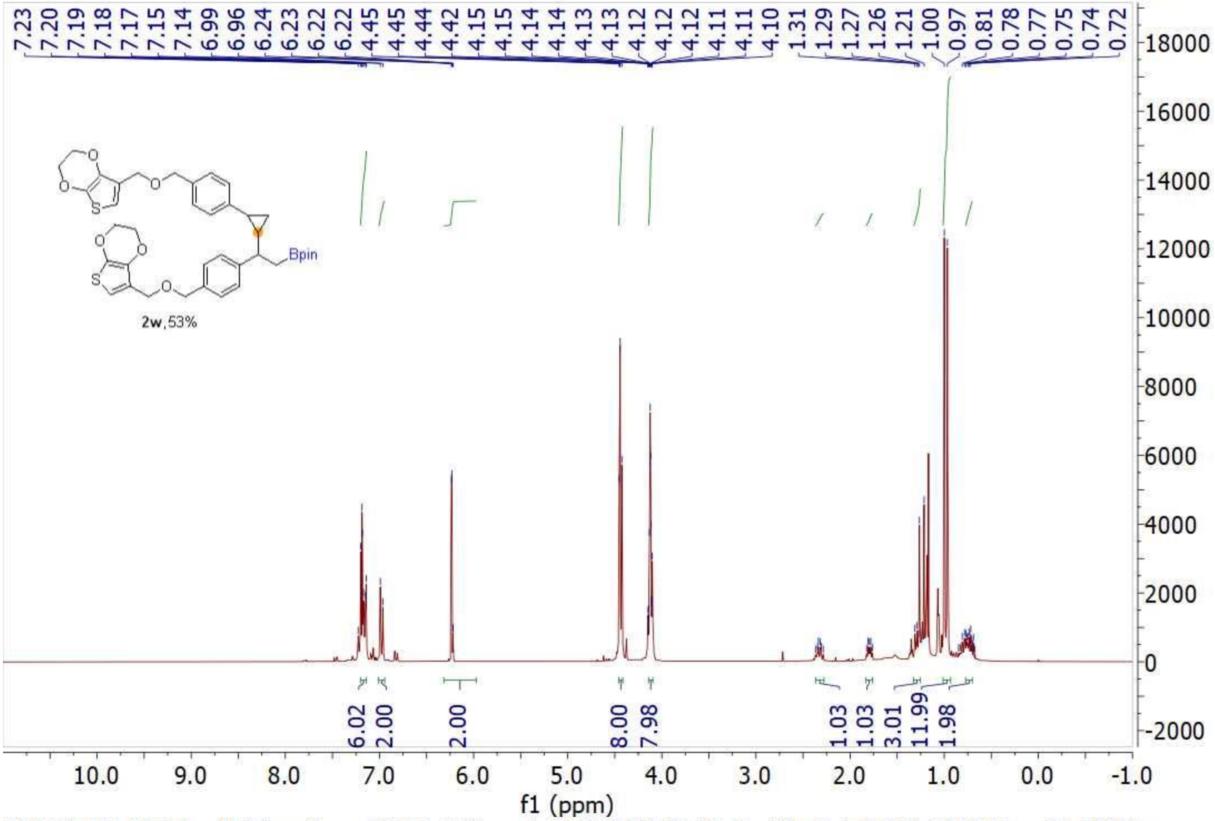

Geng/ GHQ-C-244 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 42 — 96.29MHz
33.28
S
O
S
O
Bpin
2u, 51%
800
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)


221209.310.10.fid — Huiqing Geng GHQ-L-255-3 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 10 — 300.13MHz
7.90 7.90 7.88 7.87 7.44 7.41 7.33 7.32 7.31 7.30 7.28 7.28 7.27 7.25 7.11 7.08 4.57 4.56 4.53 2.80 2.45 2.43 1.92 1.91 1.90 1.42 1.40 1.40 1.38 1.36 1.35 1.33 1.10 1.07 0.89 0.87 0.87 0.86 0.84 0.83 0.81
4.01 4.01 8.02 2.01 8.00 6.01 1.03 1.00 3.02 12.01 2.03
S
N
S
N
O
O
Bpin
2v, 47%
140000 130000 120000 110000 100000 90000 80000 70000 60000 50000 40000 30000 20000 10000 0 -10000
.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

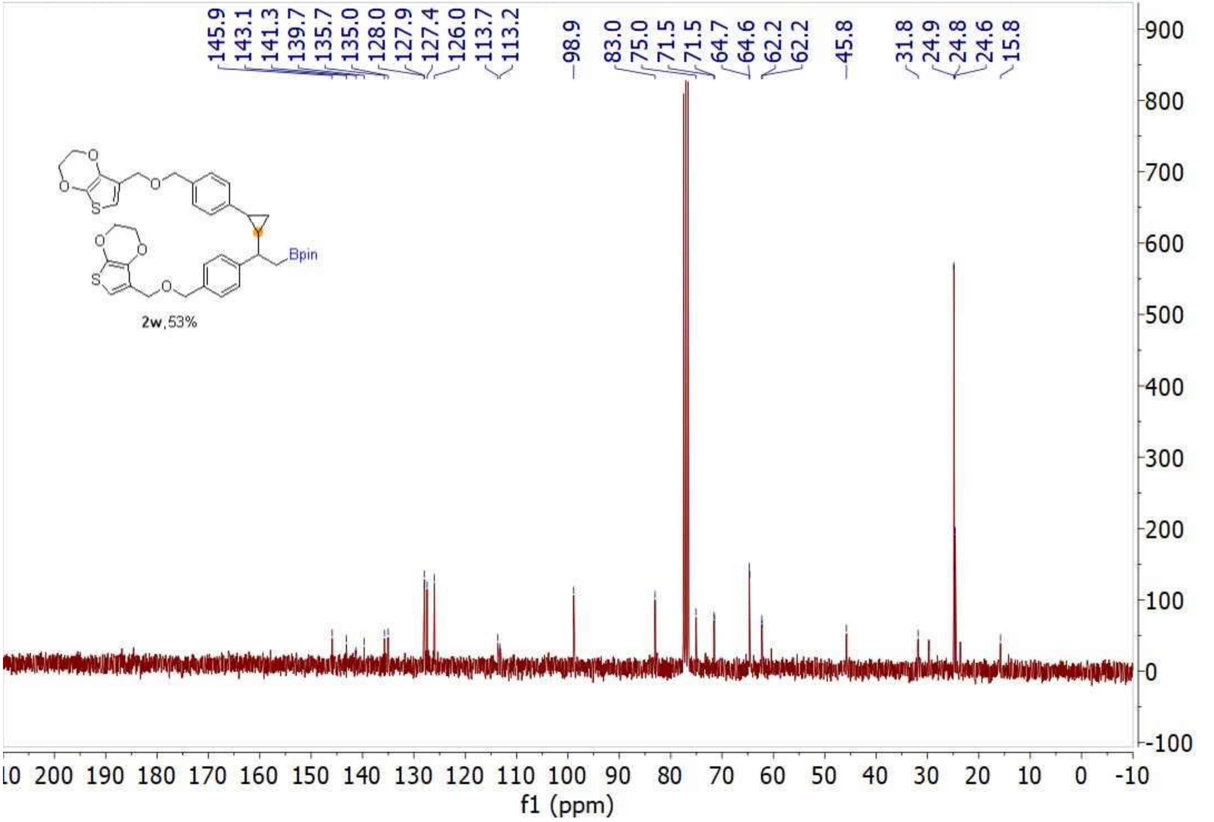

221209.310.11.fid — Huiqing Geng GHQ-L-255-3 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 10 — 75.48MHz
165.9 154.8 145.9 143.1 138.1 135.7 135.0 133.7 128.1 127.9 127.8 127.4 126.3 126.0 112.2
83.0 77.0 CDCl3 74.9 71.9 71.8 71.5
45.7 31.8 24.8 24.7 24.5 23.6 19.2 15.8
S
N
S
N
O
O
Bpin
2v, 47%
1700 1600 1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 -100 -200
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

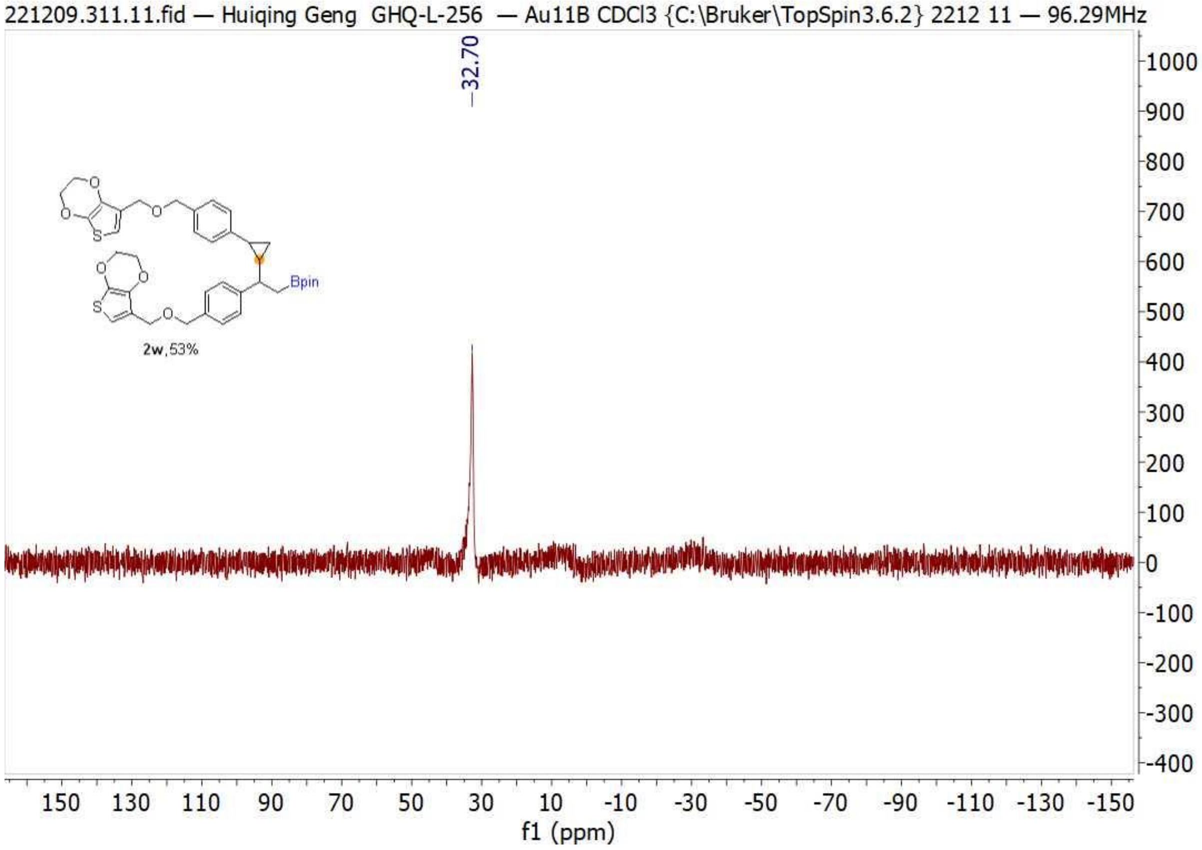

Huiqing Geng GHQ-L-255-3 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 10 — 96.29MHz
32.45
S
N
O
S
N
O
Bpin
2v, 47%
1800
1600
1400
1200
1000
800
600
400
200
0
-200
-400
-600
-800
150
130
110
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
f1 (ppm)

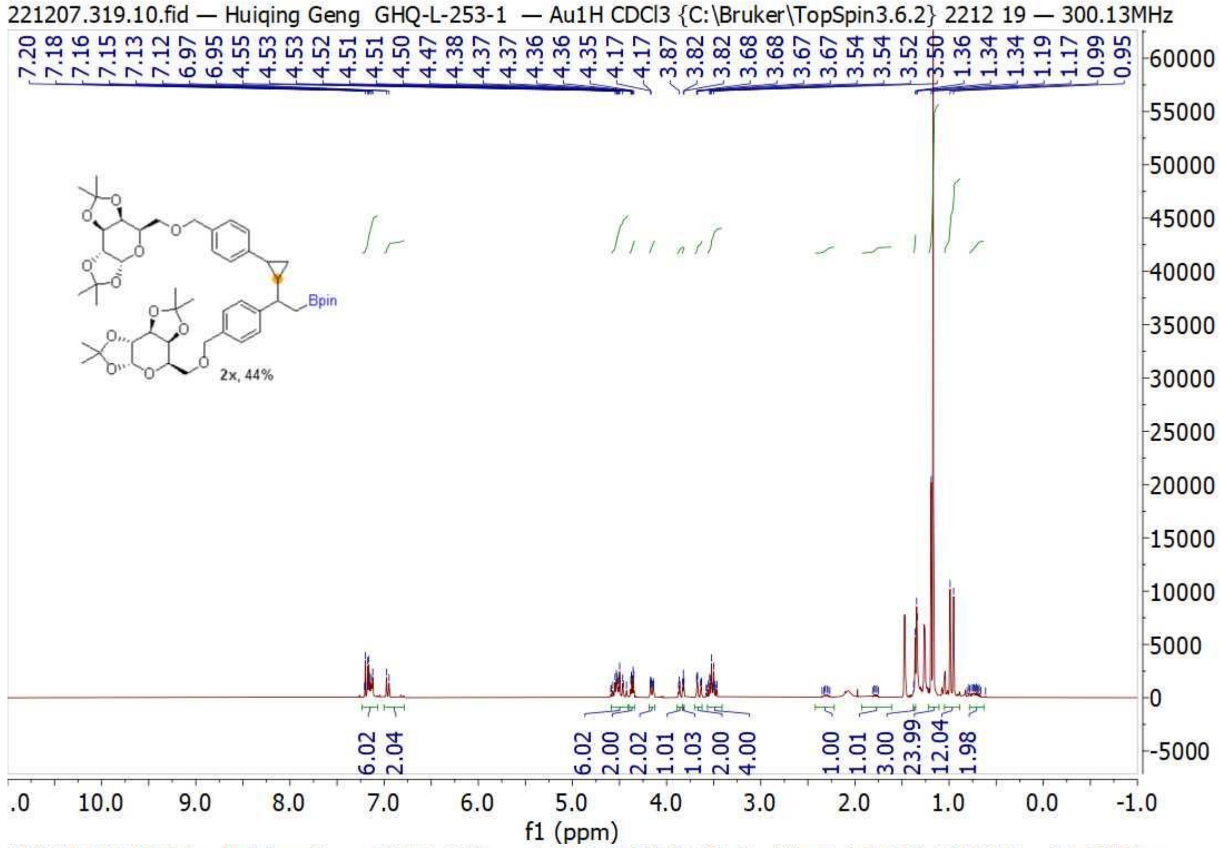
221209.312.10.fid — Huiqing Geng GHQ-L-256-3 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 12 — 300.13MHz

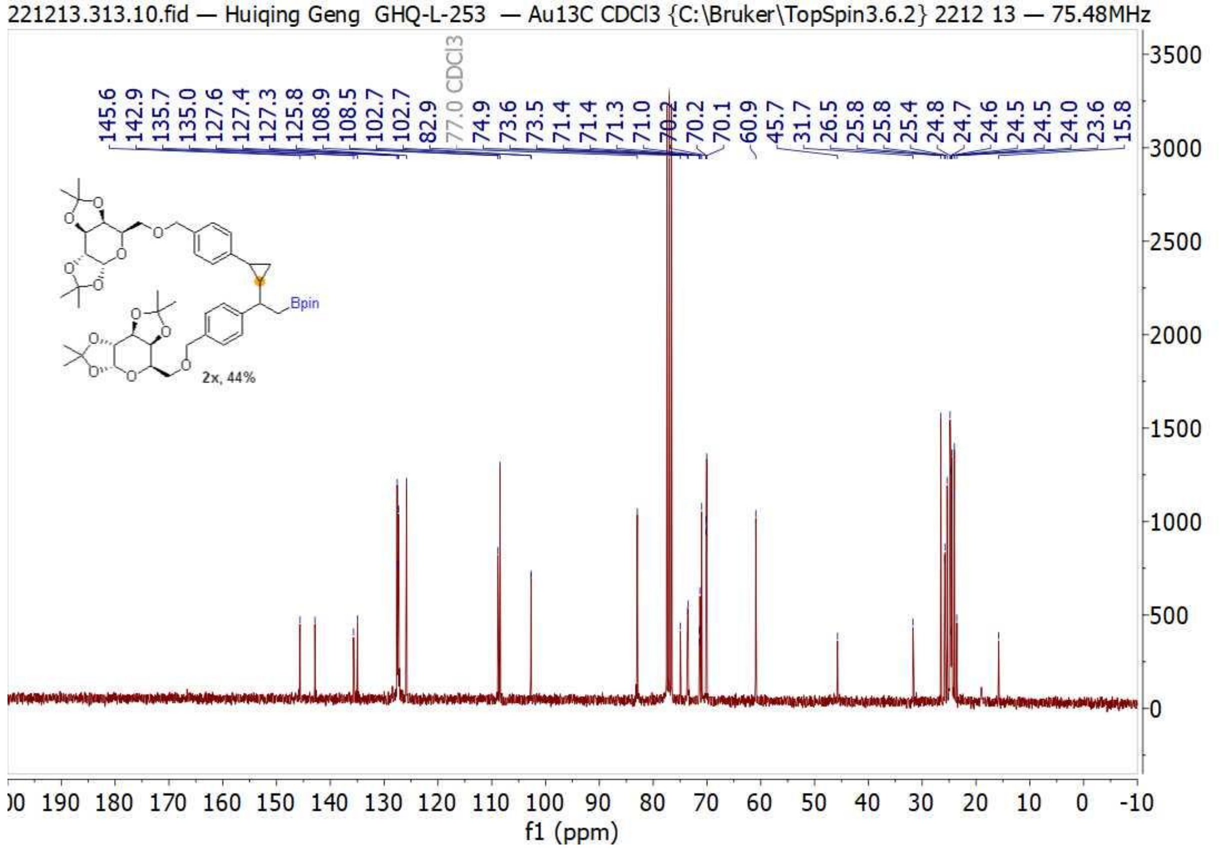


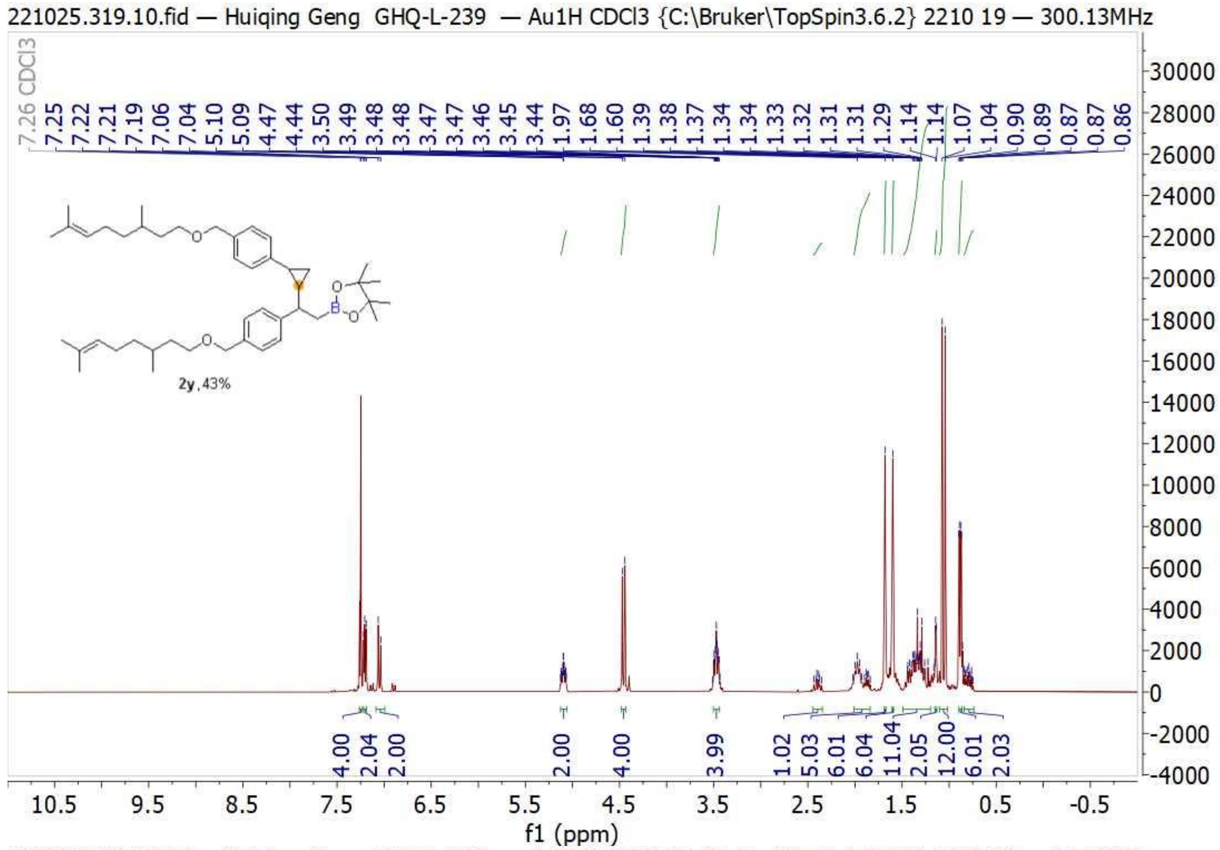
221209.311.10.fid — Huiqing Geng GHQ-L-256 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 11 — 75.48MHz

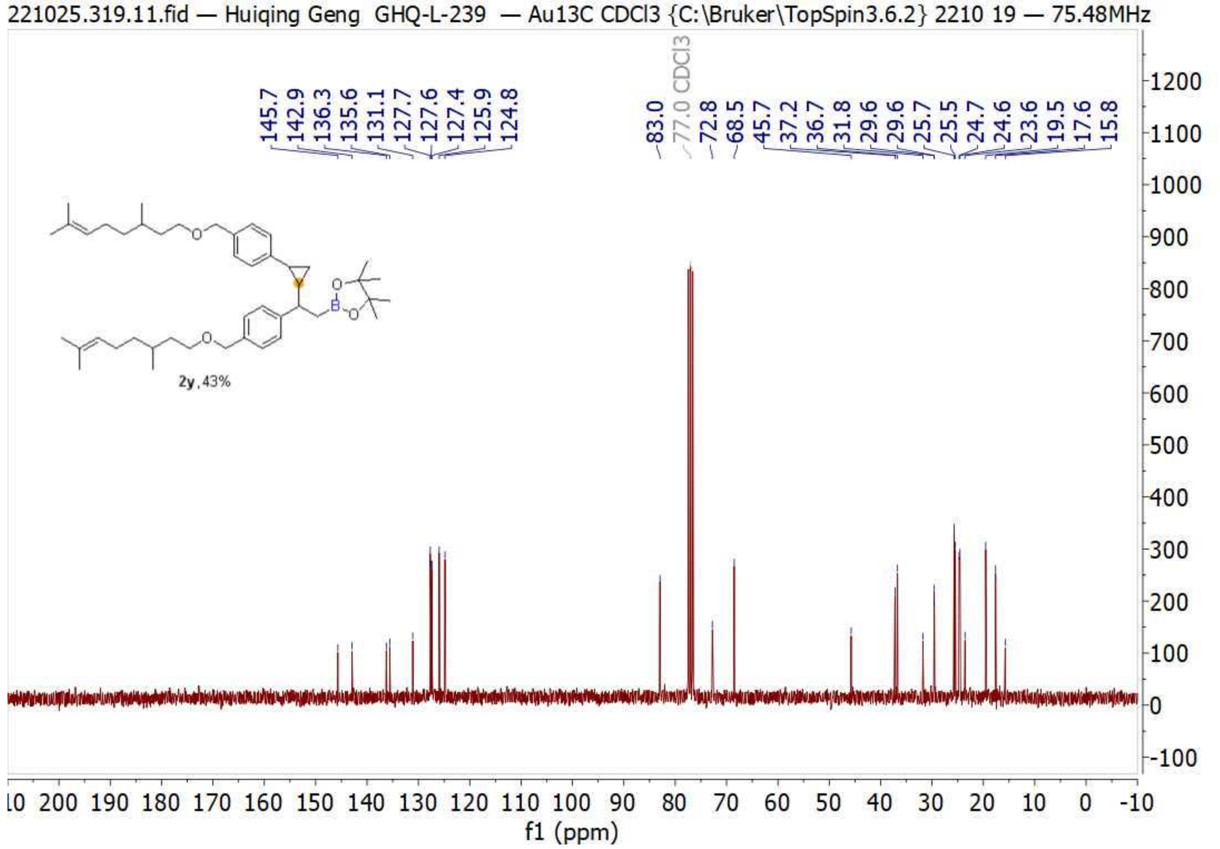


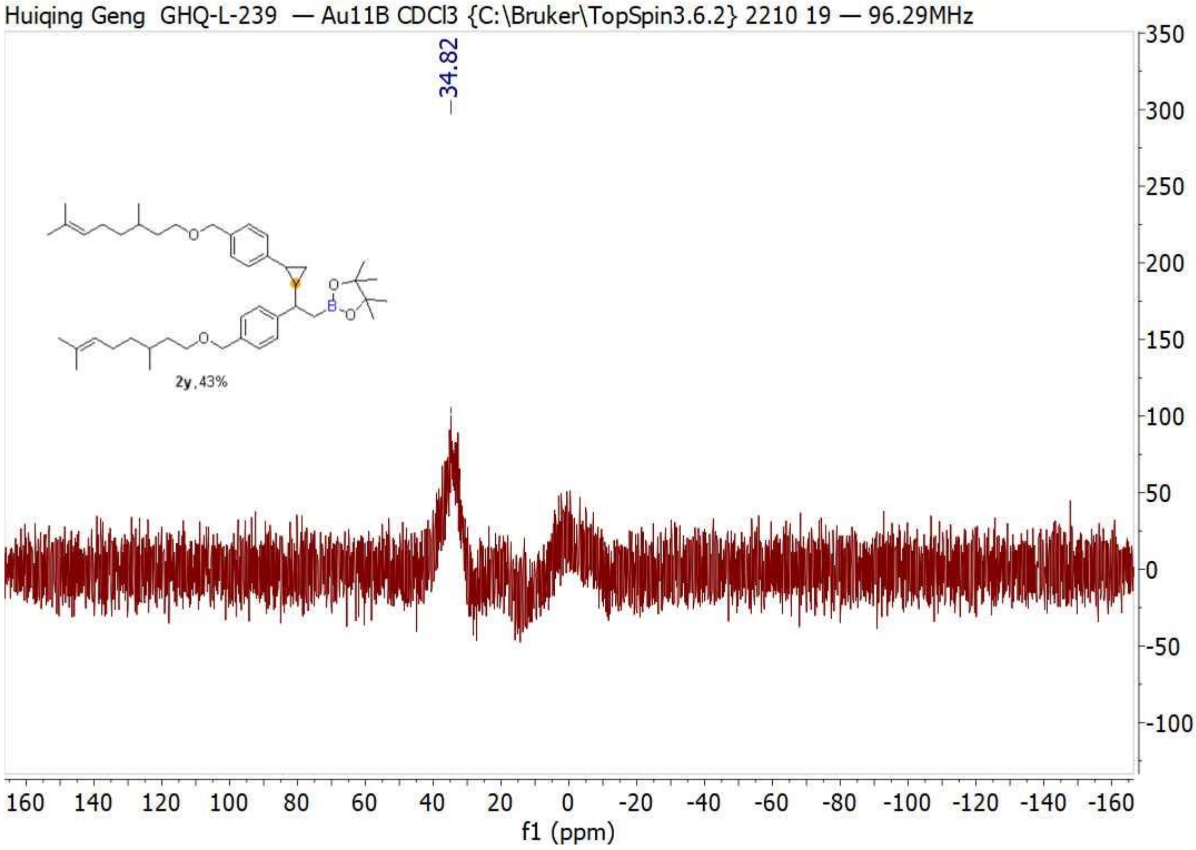

221209.311.11.fid — Huiqing Geng GHQ-L-256 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 11 — 96.29MHz
32.70
Bpin
2w, 53%
1000
900
800
700
600
500
400
300
200
100
0
-100
-200
-300
-400
150 130 110 90 70 50 30 10 -10 -30 -50 -70 -90 -110 -130 -150
f1 (ppm)

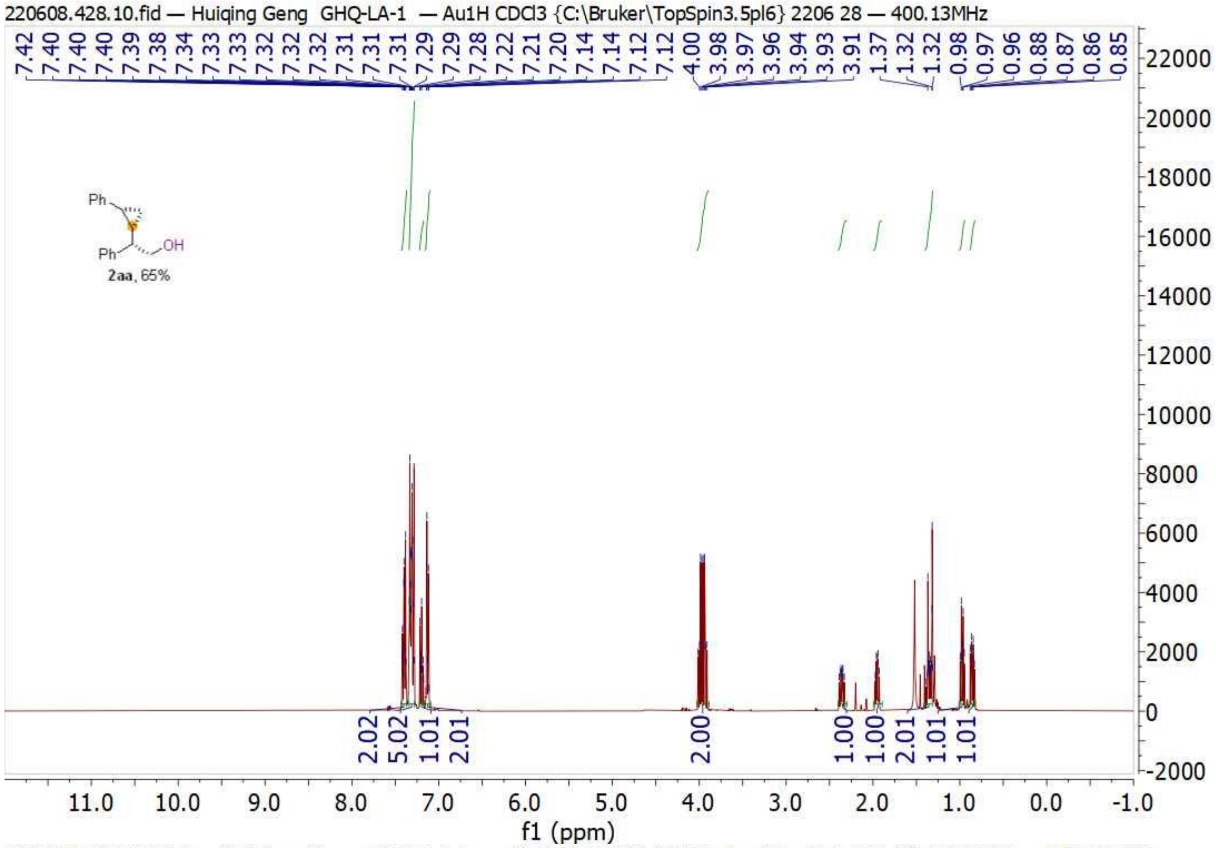

221207.319.10.fid — Huiqing Geng GHQ-L-253-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 19 — 300.13MHz
7.20 7.18 7.16 7.15 7.13 7.12 6.97 6.95 4.55 4.53 4.53 4.52 4.51 4.51 4.50 4.47 4.38 4.37 4.37 4.36 4.36 4.35 4.17 4.17 3.87 3.82 3.82 3.68 3.68 3.67 3.67 3.54 3.54 3.52 3.50 1.36 1.34 1.34 1.19 1.17 0.99 0.95
6.02 2.04 6.02 2.00 2.02 1.01 1.03 2.00 4.00 1.00 1.01 3.00 23.99 12.04 1.98
Bpin
2x, 44%
f1 (ppm)
.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
60000 55000 50000 45000 40000 35000 30000 25000 20000 15000 10000 5000 0 -5000

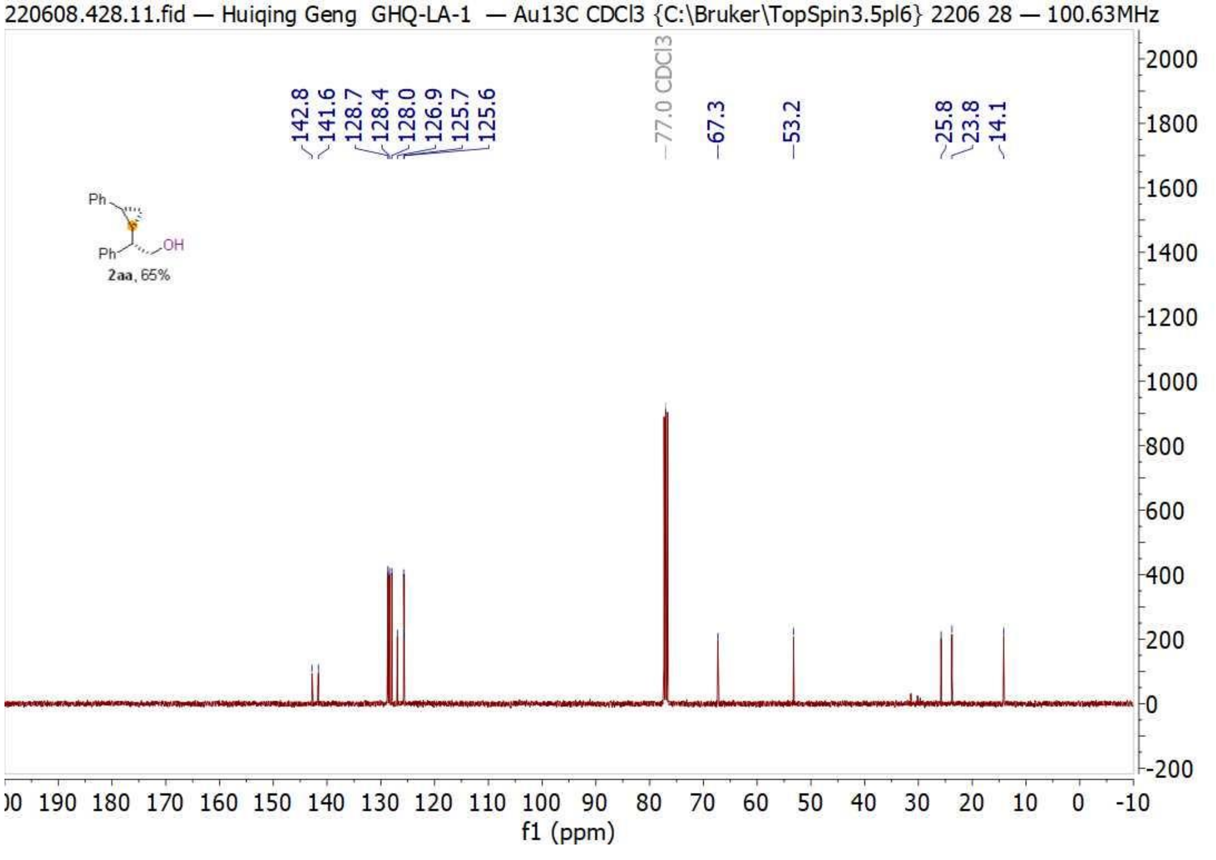

221213.313.10.fid — Huiqing Geng GHQ-L-253 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2212 13 — 75.48MHz
145.6 142.9 135.7 135.0 127.6 127.4 127.3 125.8 108.9 108.5 102.7 102.7 82.9 77.0 CDCl3 74.9 73.6 73.5 71.4 71.4 71.3 71.0 70.2 70.2 70.1 60.9 45.7 31.7 26.5 25.8 25.8 25.4 24.8 24.7 24.6 24.5 24.5 24.0 23.6 15.8
Bpin
2x, 44%
f1 (ppm)
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
3500 3000 2500 2000 1500 1000 500 0


221025.319.10.fid — Huiqing Geng GHQ-L-239 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 19 — 300.13MHz
7.26 CDCl3
7.25 7.22 7.21 7.19 7.06 7.04 5.10 5.09 4.47 4.44 3.50 3.49 3.48 3.48 3.47 3.47 3.46 3.45 3.44 1.97 1.68 1.60 1.39 1.38 1.37 1.34 1.34 1.33 1.32 1.31 1.31 1.29 1.14 1.14 1.07 1.04 0.90 0.89 0.87 0.87 0.86
O
B
2y, 43%
4.00 2.04 2.00 2.00 4.00 3.99 1.02 5.03 6.01 6.04 11.04 2.05 12.00 6.01 2.03
30000 28000 26000 24000 22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000 -4000
10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

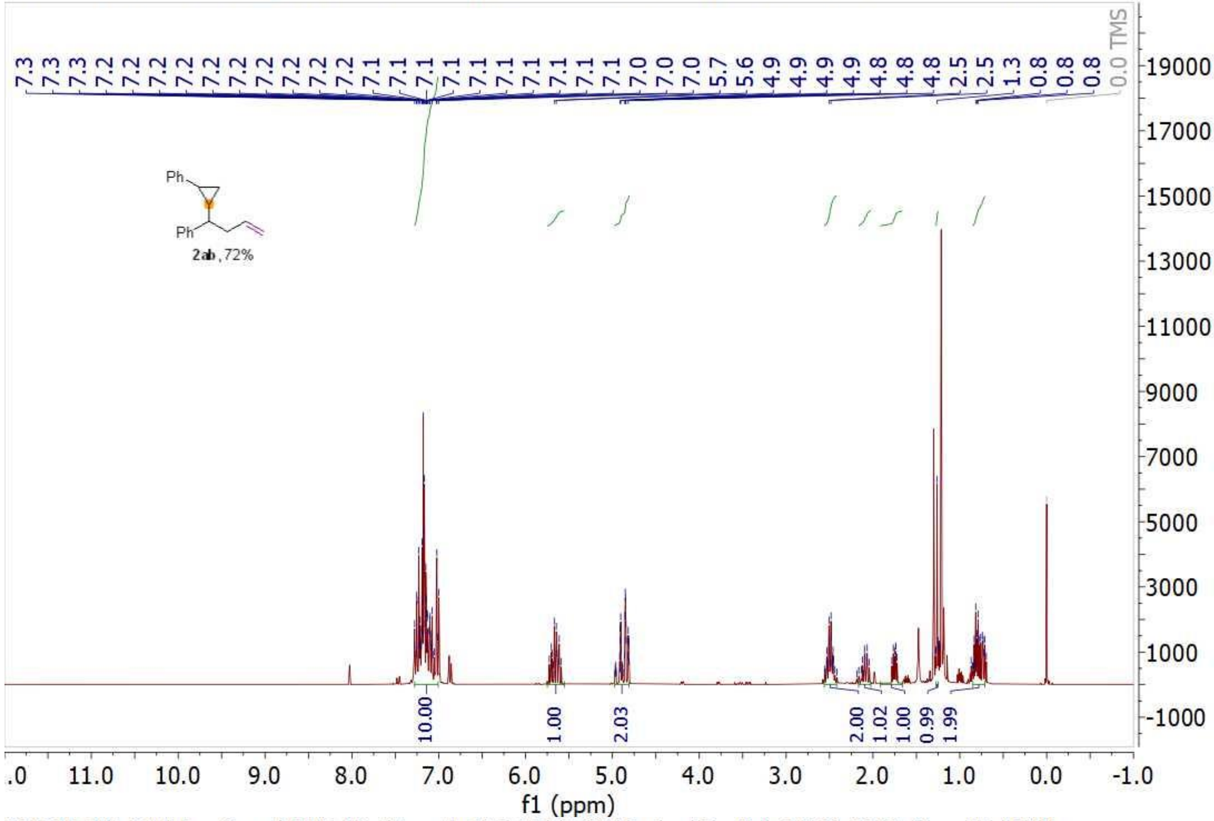

221025.319.11.fid — Huiqing Geng GHQ-L-239 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 19 — 75.48MHz
145.7 142.9 136.3 135.6 131.1 127.7 127.6 127.4 125.9 124.8
83.0 77.0 CDCl3 72.8 68.5 45.7 37.2 36.7 31.8 29.6 29.6 25.7 25.5 24.7 24.6 23.6 19.5 17.6 15.8
O
B
2y, 43%
1200 1100 1000 900 800 700 600 500 400 300 200 100 0 -100
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Huiqing Geng GHQ-L-239 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 19 — 96.29MHz
34.82
2y, 43%
350
300
250
200
150
100
50
0
-50
-100
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
f1 (ppm)

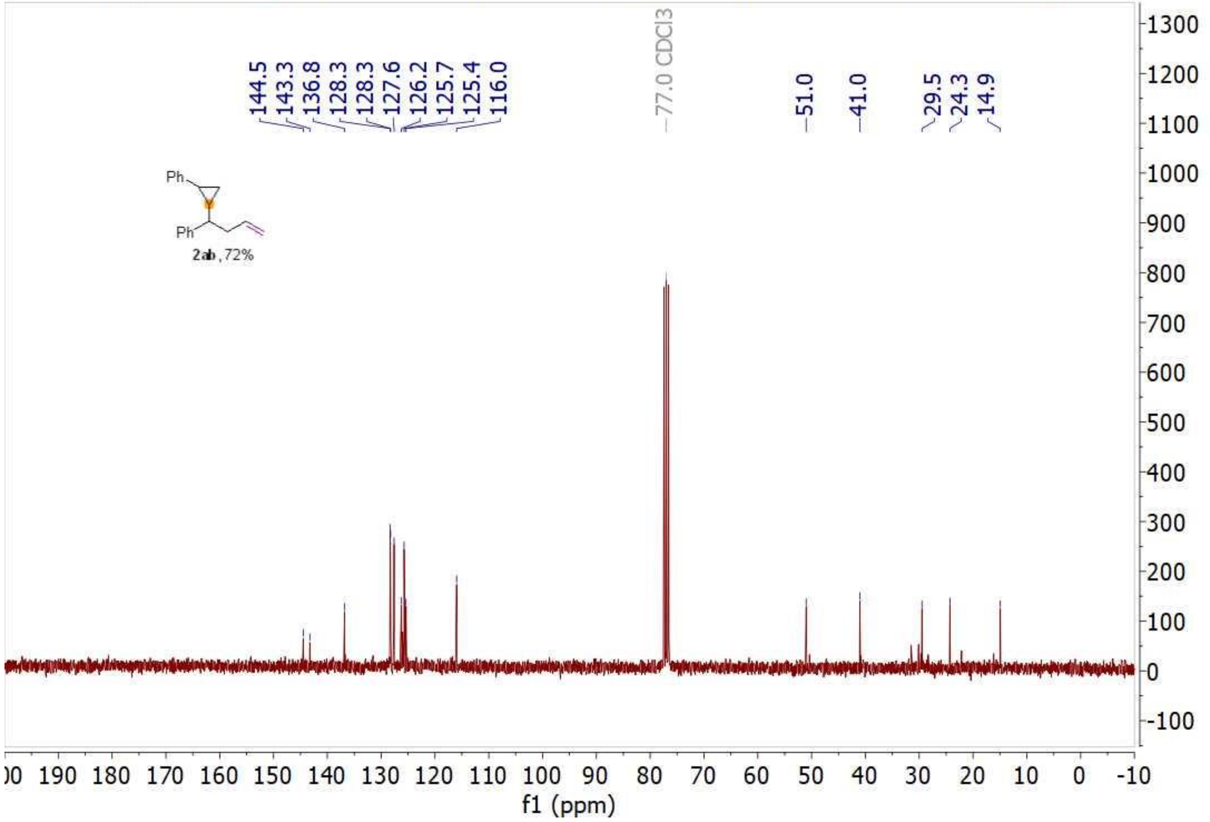

220608.428.10.fid — Huiqing Geng GHQ-LA-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2206 28 — 400.13MHz
7.42 7.40 7.40 7.40 7.39 7.38 7.34 7.33 7.33 7.32 7.32 7.32 7.31 7.31 7.31 7.29 7.29 7.28 7.22 7.21 7.20 7.14 7.14 7.12 7.12 4.00 3.98 3.97 3.96 3.94 3.93 3.91 1.37 1.32 1.32 0.98 0.97 0.96 0.88 0.87 0.86 0.85
Ph
Ph
OH
2aa, 65%
2.02 5.02 1.01 2.01 2.00 1.00 1.00 2.01 1.01 1.01
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)
22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000

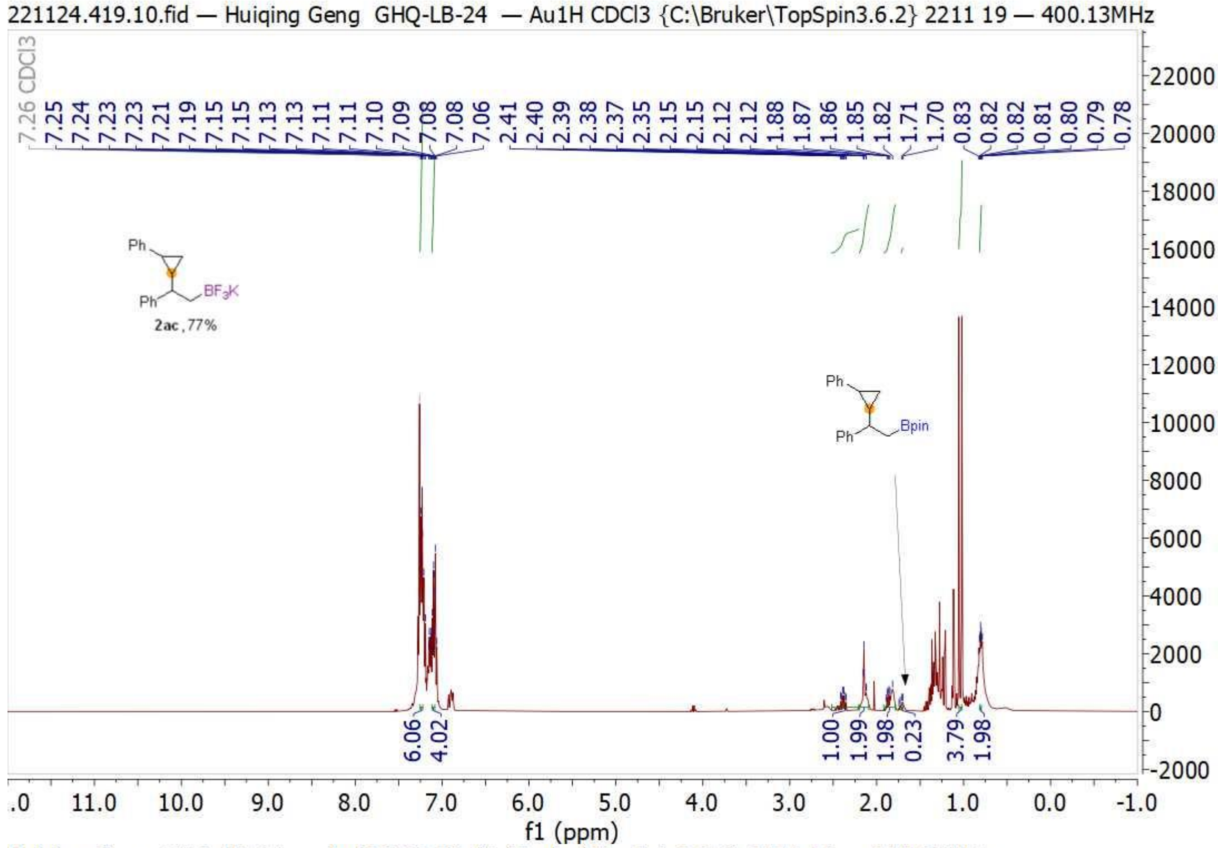

220608.428.11.fid — Huiqing Geng GHQ-LA-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2206 28 — 100.63MHz
142.8 141.6 128.7 128.4 128.0 126.9 125.7 125.6
77.0 CDCl3
67.3
53.2
25.8 23.8 14.1
Ph
Ph
OH
2aa, 65%
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200

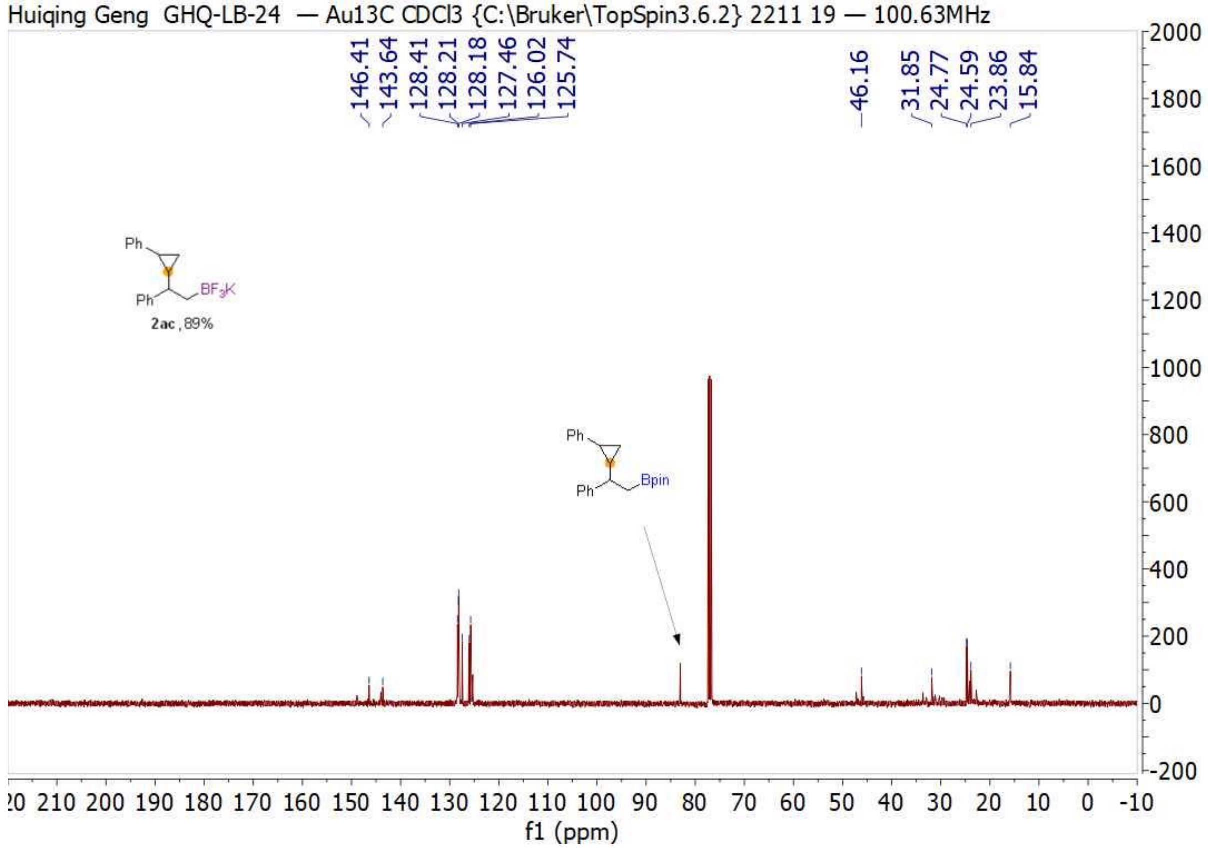
221110.341.10.fid — Geng/ GHQ-LB-17 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 41 — 300.13MHz

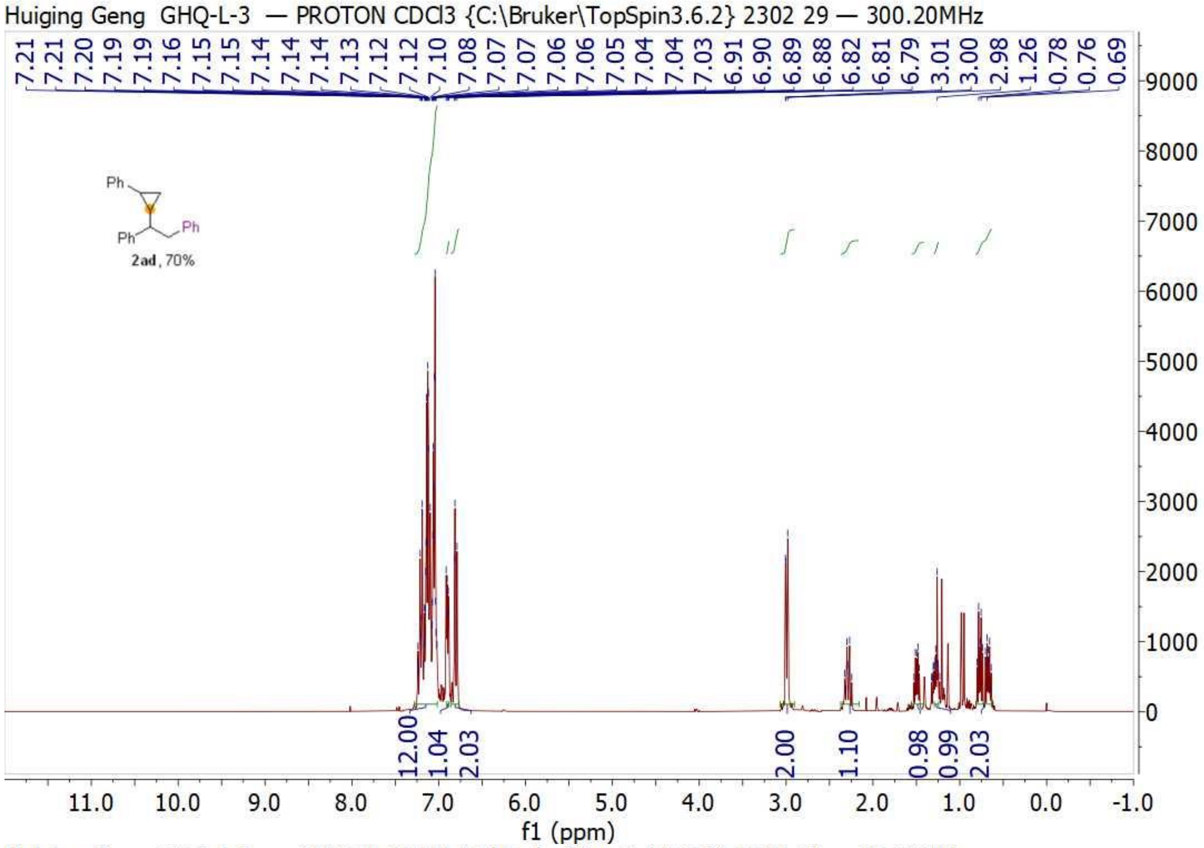


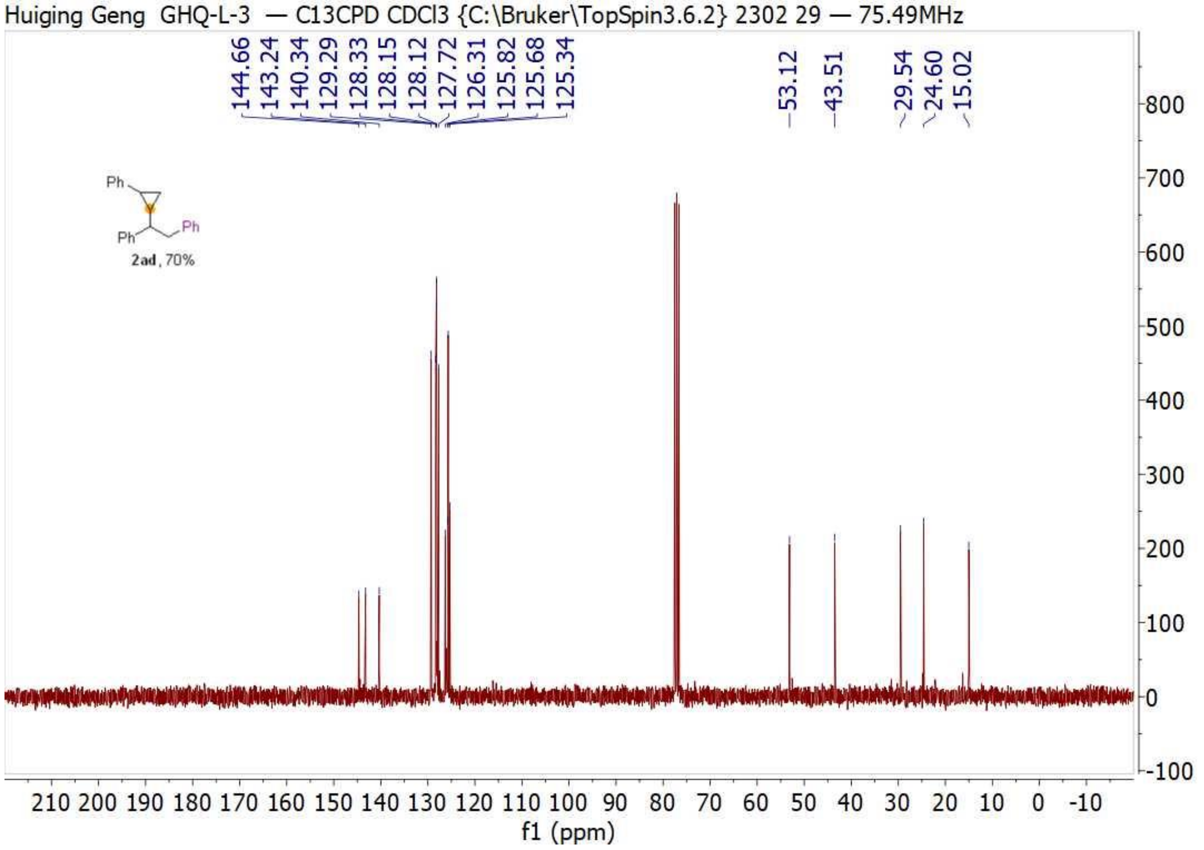
221110.341.11.fid — Geng/ GHQ-LB-17 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 41 — 75.48MHz

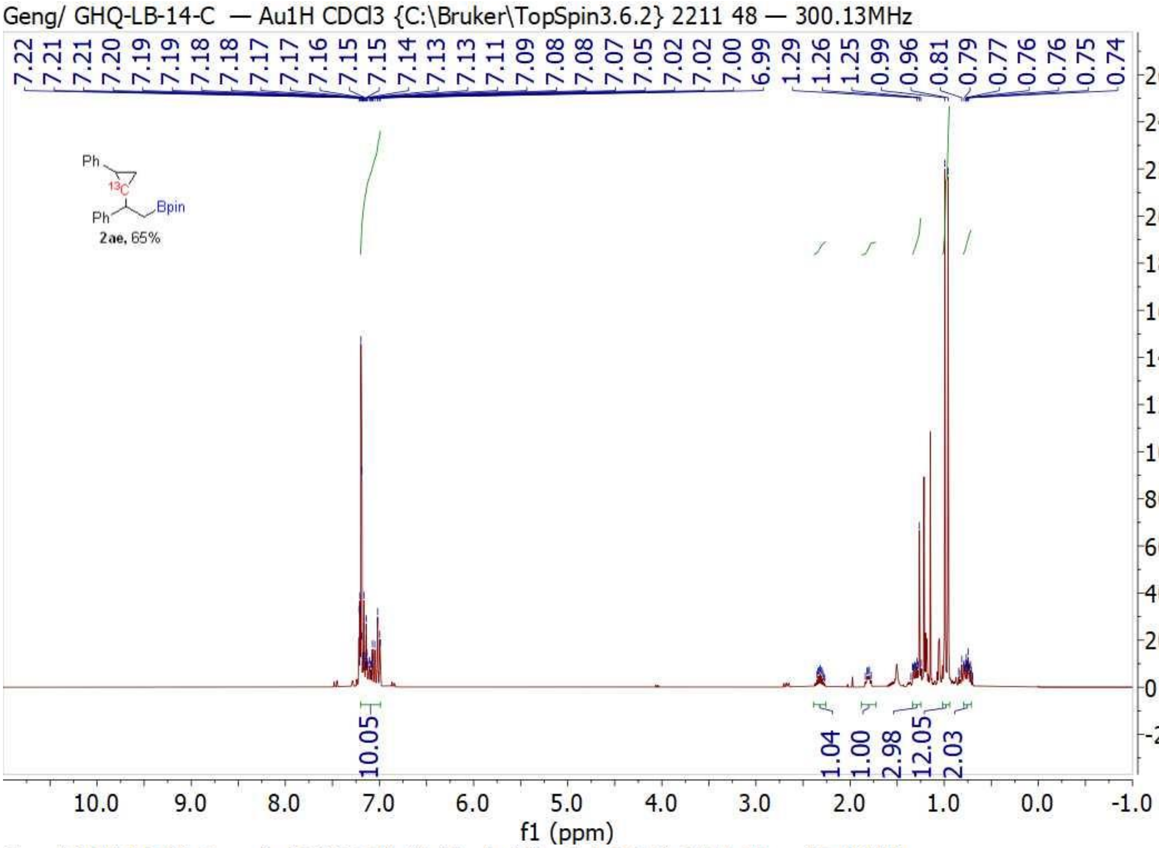


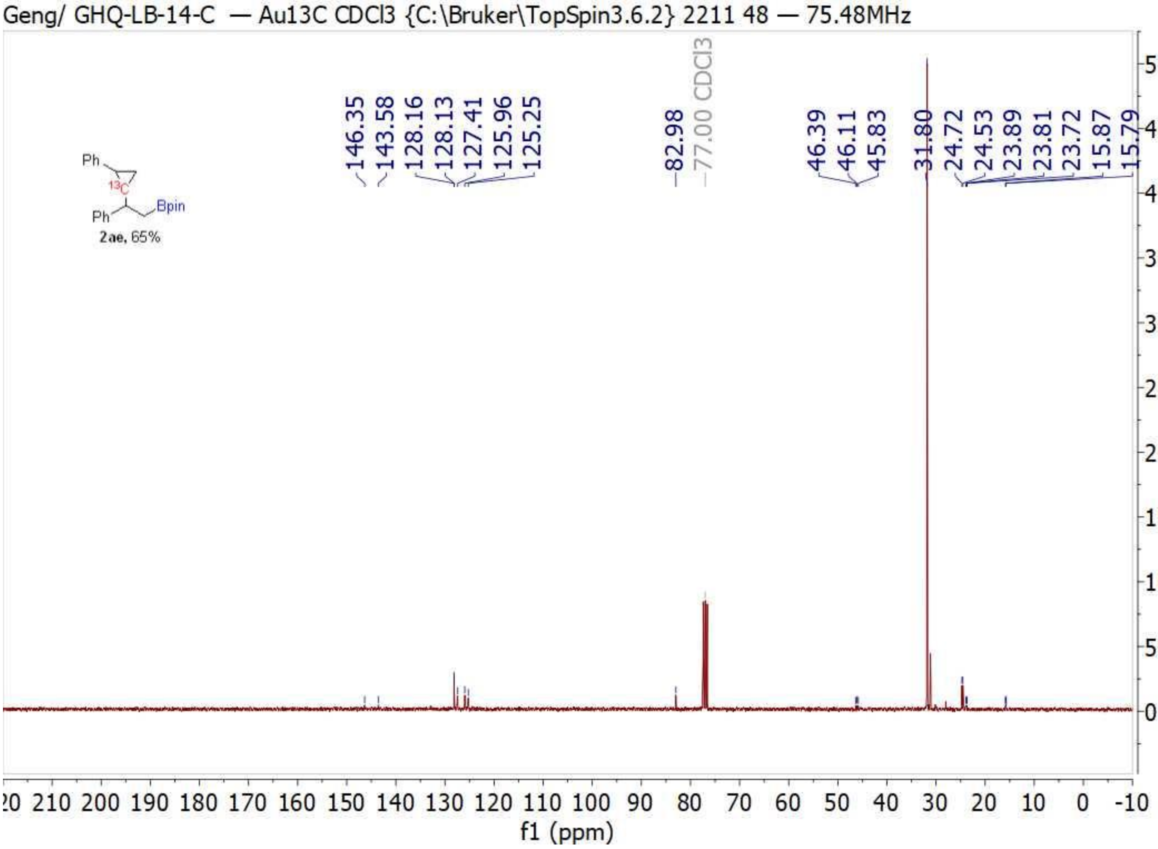

221124.419.10.fid — Huiqing Geng GHQ-LB-24 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 19 — 400.13MHz
7.26 CDCl3
7.25 7.24 7.23 7.23 7.21 7.19 7.15 7.15 7.13 7.13 7.11 7.11 7.10 7.09 7.08 7.08 7.06 2.41 2.40 2.39 2.38 2.37 2.35 2.15 2.15 2.12 2.12 1.88 1.87 1.86 1.85 1.82 1.71 1.70 0.83 0.82 0.82 0.81 0.80 0.79 0.78
Ph
Ph
BF3K
2ac, 77%
Ph
Ph
Bpin
6.06 4.02 1.00 1.99 1.98 0.23 3.79 1.98
22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 -2000
.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0
f1 (ppm)

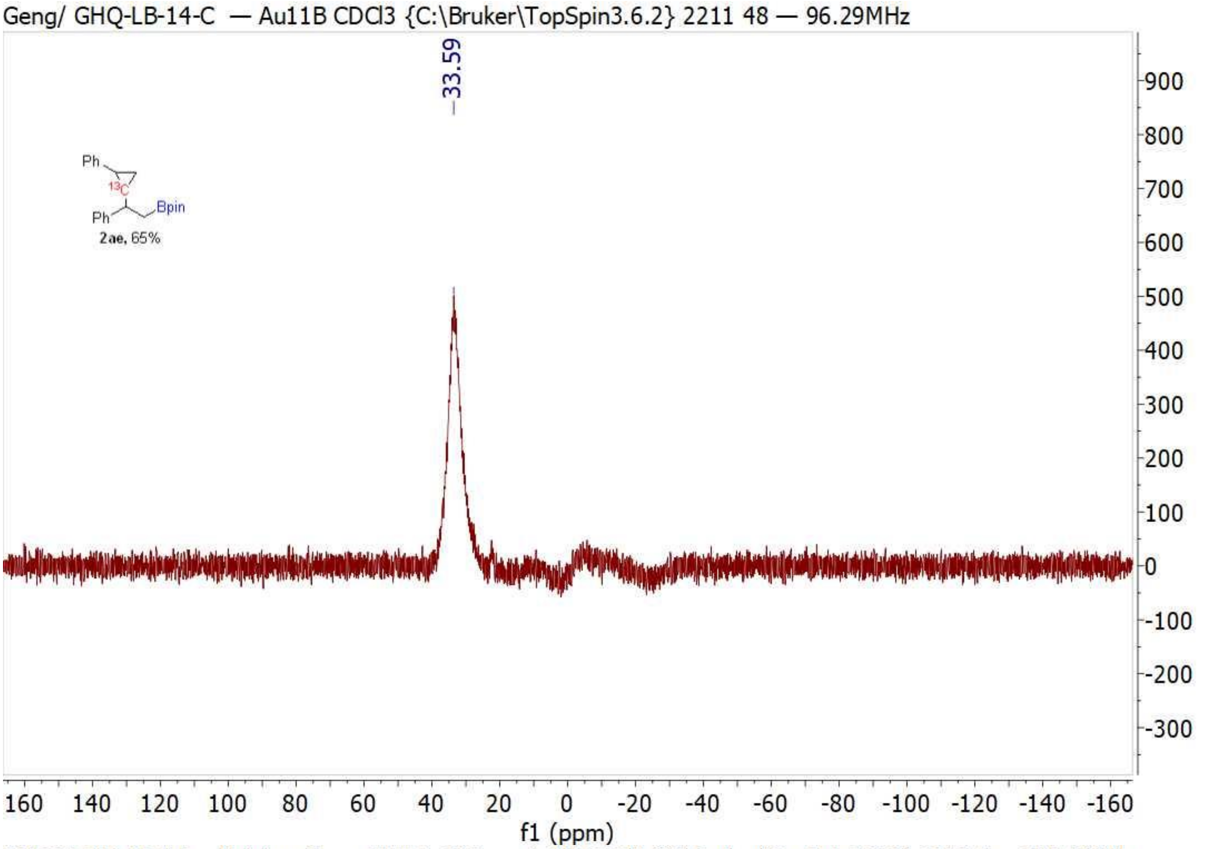

Huiqing Geng GHQ-LB-24 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 19 — 100.63MHz
146.41 143.64 128.41 128.21 128.18 127.46 126.02 125.74
46.16
31.85 24.77 24.59 23.86 15.84
Ph
Ph
BF3K
2ac, 89%
Ph
Ph
Bpin
2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

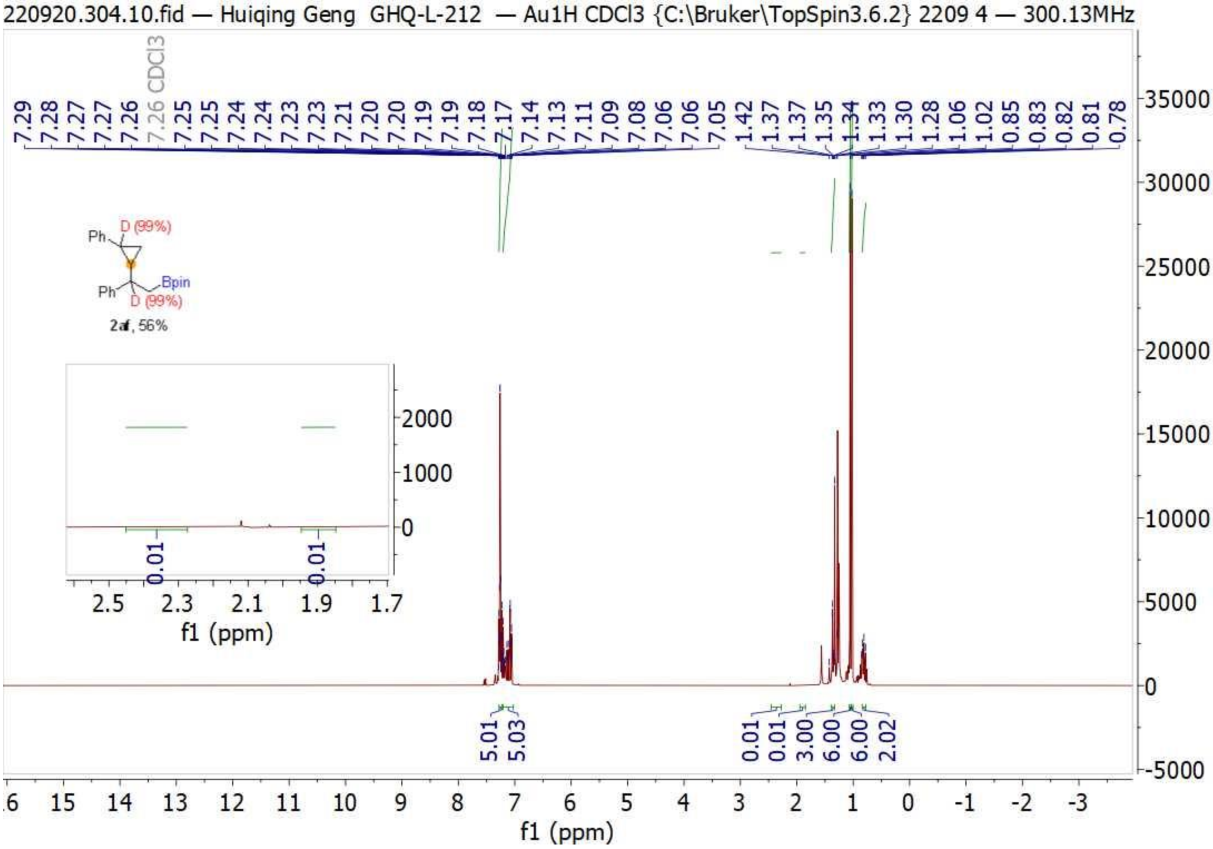

Huiging Geng GHQ-L-3 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2302 29 — 300.20MHz
7.21
7.21
7.20
7.19
7.19
7.16
7.15
7.15
7.14
7.14
7.14
7.13
7.12
7.12
7.10
7.08
7.07
7.07
7.06
7.06
7.05
7.04
7.04
7.03
6.91
6.90
6.89
6.88
6.82
6.81
6.79
3.01
3.00
2.98
1.26
0.78
0.76
0.69
Ph
Ph
Ph
2ad, 70%
12.00
1.04
2.03
2.00
1.10
0.98
0.99
2.03
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


Huiging Geng GHQ-L-3 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2302 29 — 75.49MHz
144.66
143.24
140.34
129.29
128.33
128.15
128.12
127.72
126.31
125.82
125.68
125.34
53.12
43.51
29.54
24.60
15.02
Ph
Ph
Ph
2ad, 70%
800
700
600
500
400
300
200
100
0
-100
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

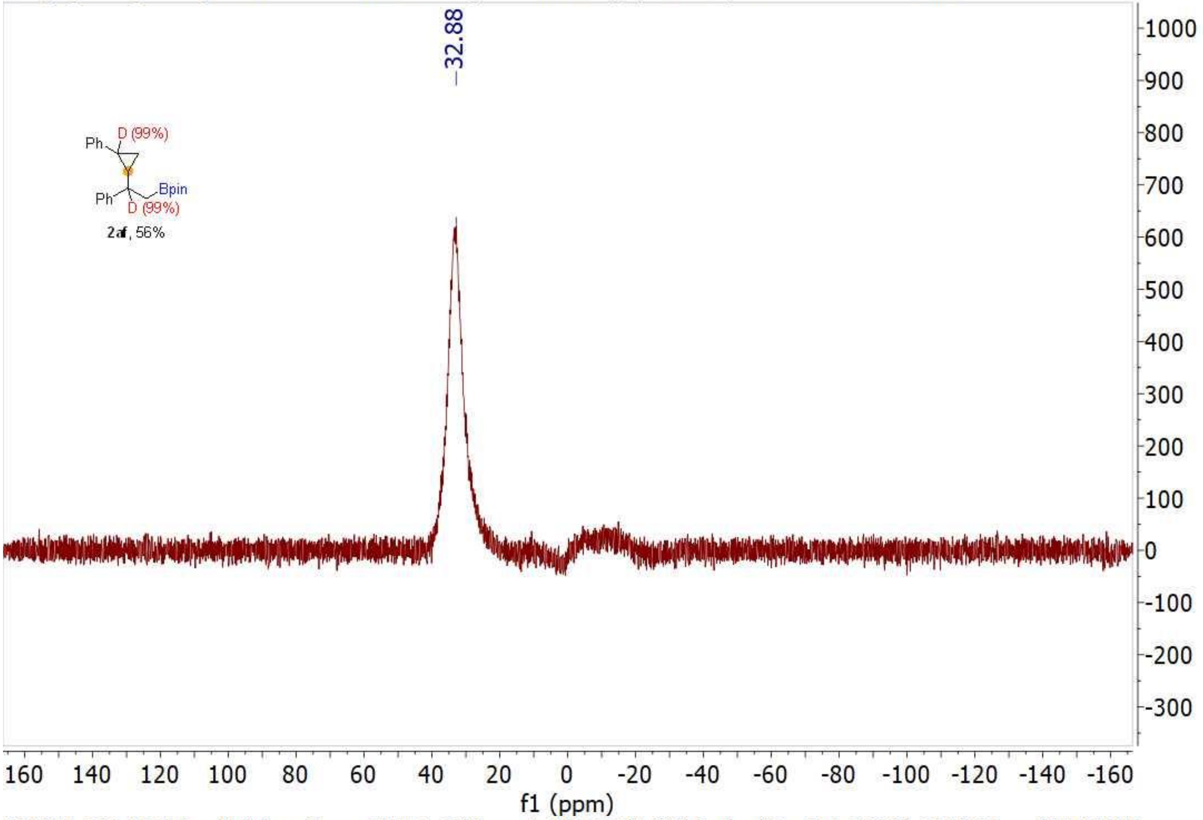

Geng/ GHQ-LB-14-C — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 48 — 300.13MHz
7.22
7.21
7.21
7.20
7.19
7.19
7.18
7.18
7.17
7.17
7.16
7.15
7.15
7.14
7.13
7.13
7.11
7.09
7.08
7.08
7.07
7.05
7.02
7.02
7.00
6.99
1.29
1.26
1.25
0.99
0.96
0.81
0.79
0.77
0.76
0.76
0.75
0.74
Ph
13C
Ph
Bpin
2ae, 65%
10.05
1.04
1.00
2.98
12.05
2.03
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
f1 (ppm)


Geng/ GHQ-LB-14-C — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 48 — 75.48MHz
146.35
143.58
128.16
128.13
127.41
125.96
125.25
82.98
77.00 CDCl3
46.39
46.11
45.83
31.80
24.72
24.53
23.89
23.81
23.72
15.87
15.79
Ph
13C
Ph
Bpin
2ae, 65%
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

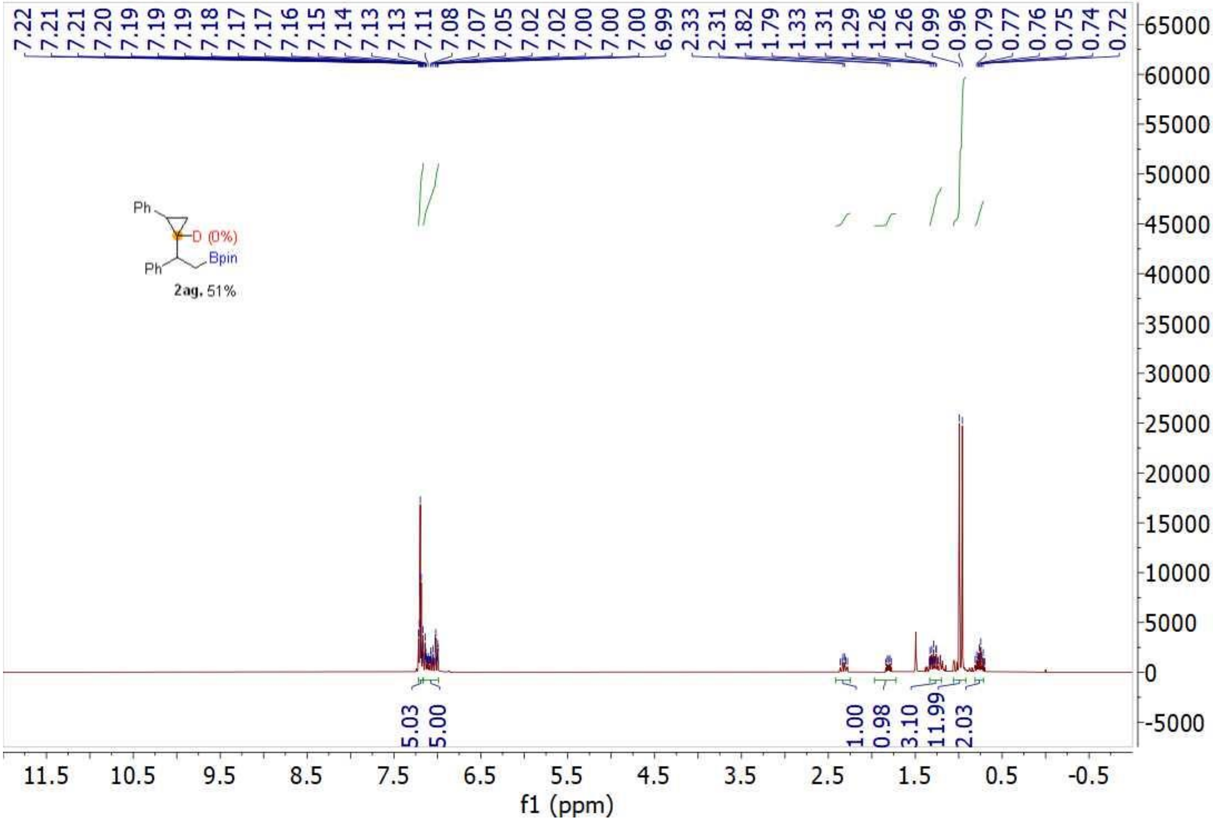

Geng/ GHQ-LB-14-C — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2211 48 — 96.29MHz
33.59
Ph
13C
Ph
Bpin
2ae, 65%
900
800
700
600
500
400
300
200
100
0
-100
-200
-300
160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160
f1 (ppm)


220920.304.10.fid — Huiqing Geng GHQ-L-212 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 4 — 300.13MHz
7.29 7.28 7.27 7.27 7.26 7.26 CDCl3 7.25 7.25 7.24 7.24 7.23 7.23 7.21 7.20 7.20 7.19 7.19 7.18 7.17 7.14 7.13 7.11 7.09 7.08 7.06 7.06 7.05 1.42 1.37 1.37 1.35 1.34 1.33 1.30 1.28 1.06 1.02 0.85 0.83 0.82 0.81 0.78
Ph
D (99%)
Ph
Bpin
D (99%)
2af, 56%
2000
1000
0
0.01
0.01
2.5 2.3 2.1 1.9 1.7
f1 (ppm)
35000
30000
25000
20000
15000
10000
5000
0
-5000
5.01 5.03
0.01 0.01 3.00 6.00 6.00 2.02
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

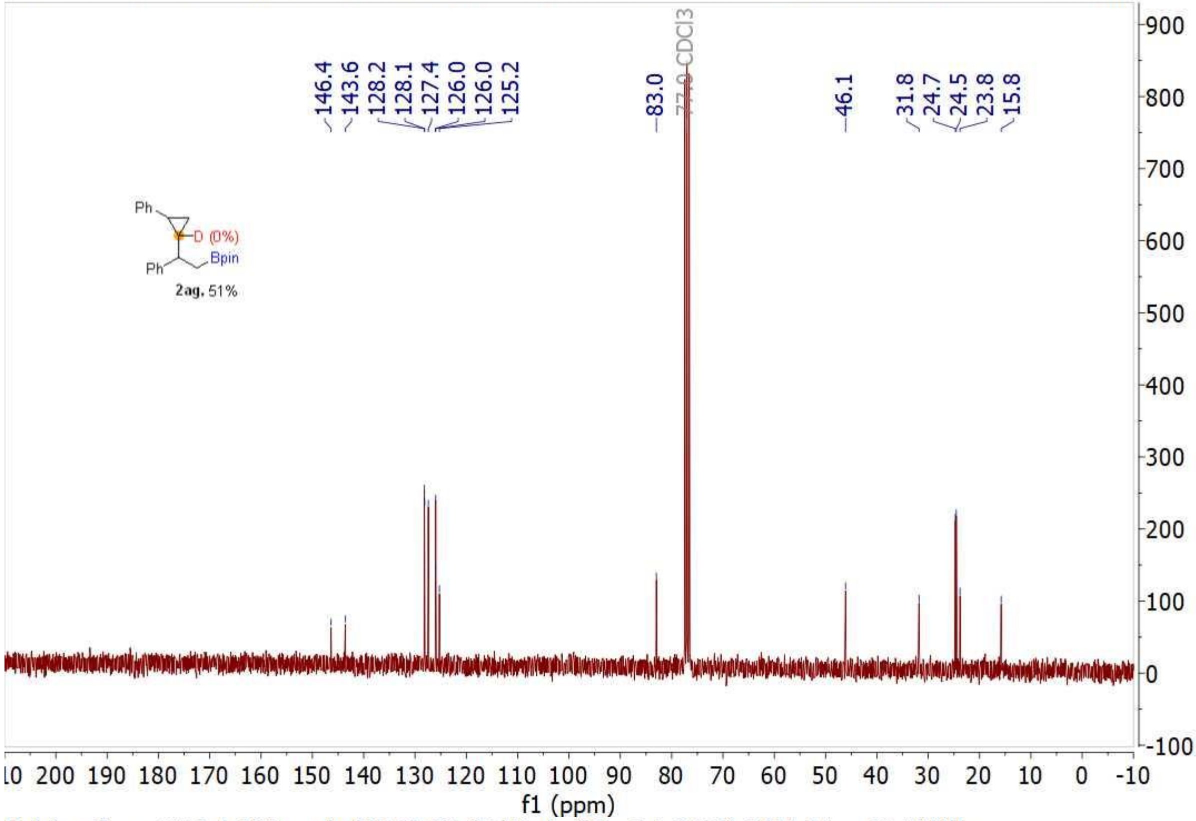
Huiqing Geng GHQ-L-212 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2209 4 — 96.29MHz



221011.324.10.fid — Huiqing Geng GHQ-L-230 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 24 — 300.13MHz



221011.324.11.fid — Huiqing Geng GHQ-L-230 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 24 — 75.48MHz



Huiqing Geng GHQ-L-230 — Au11B CDCl3 {C:\Bruker\TopSpin3.6.2} 2210 24 — 96.29MHz