Supplementary Materials for

Copper-Catalyzed Hydroformylation and Hydroxymethylation of Styrenes

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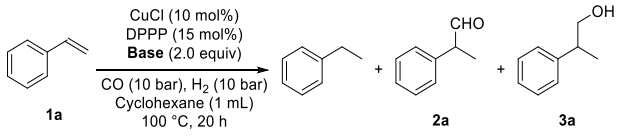
1. General Information

All commercially available reagents were bought from ABCR, Acros, Aldrich, Alfa, Fluka, Fluorochem, TCI, and Strem. The commercial reagents were used without further purification. Reactions were performed under argon atmosphere in oven-dried glassware. The primary sodium alkoxides used as base were dried under vacuum using a heat gun prior to reaction to minimize residual water. In addition, the batches were regularly replaced to maintain reproducibility. ICP measurements of the solid catalyst and directly from the reaction solution were performed to test for possible traces of rhodium. All testing conducted showed a negative result. Analytical data of literature known compounds were in accordance with previously reported data. NMR spectra were recorded on Bruker Avance 300 (300 MHz) or 400 (400 MHz) NMR spectrometers. Chemical shifts δ (ppm) are given relative to solvent: references for $CDCl_3$ were 7.26 ppm (^{1}H NMR) and 77.16 ppm (^{13}C NMR). ^{19}F NMR spectra are not calibrated by an internal reference. Signals were assigned as s (singlet), d (doublet), t (triplet), dd (doublet of doublet), h (hextet), m (multiplet), and bs (broad singlet). Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). All GC yields were calculated using hexadecane as internal standard. All measurements were carried out at room temperature unless otherwise noted. High resolution mass spectra (HRMS) were recorded on Agilent 6210 Time-of-Flight LC/MS (Agilent) with electrospray ionization (ESI). The data are given as mass units per charge (m/z) and intensities of signals are given in brackets. For GC analyses, HP 6890 chromatograph with a 29 m HP5 column was used. ICP measurements were conducted on a simultaneous measuring optical emission spetrometer: "Spectroblue SOP" from Spectro Analytical Instruments GmbH. The following measurement conditions were set: Plasma power: 1490 W; Pump rate: 20 rpm; Cooling gas: 14 L min^{-1}; Auxiliary gas: 1.4 L min^{-1}; Sputter gas: 0.45 L min^{-1}; Make-up gas O_2: 0.02 L min^{-1}.

2. Experimental Section

2.1. Optimization of Reaction Conditions of Alcohol

Table S1. **Screening of Base.**

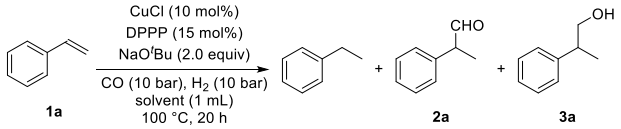


Entry	**Base**	Conv (%)	**2a:3a**	^{b}GC-Yield (%)
1	NaOMe	82	45:55	42
2	LiOMe	N.R.	–	–
3	NaOEt	57	–	–
4	KO^{t}Bu	59	<1:99	17
5	LiO^{t}Bu	65	–	–
6	NEt_3	N.R.	–	–

7	–	N.R.	–	–
8	NaO^{t}Bu	77	18:82	33

[a]Reaction conditions: **1a** (0.43 mmol), CuCl (10 mol%), DPPP (15 mol%), Base (2.0 equiv), Cyclohexane (1 mL), CO (10 bar), H_2 (10 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.**

Table S2. **Screening of Solvent.**



Entry	**Solvent**	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	Et_2O	47	47:53	19
2	*n*-Pentane	N.R.	–	–
3	Cycloheptane	83	25:75	36
4	*o*-Xylene	99	100:0	21
5	*p*-Xylene	70	60:40	45
6	PhF	79	46:54	28
7	PhCl	59	71:29	35
8	(*n*-Bu)$_2$O	80	54:46	39
9	DCE	N.R.	–	–
10	PhEt	90	10:90	42
11	$PhCF_3$	84	44:56	29
12	1,3-Difluorobenzene	93	33:67	30
13	MTBE	94	1:99	26
14	Acetone	N.R.	–	–
15	*n*-PrPh	85	17:83	23
16	Benzene	78	<1:99	12
17	PhOMe	85	32:68	28
18	Toluene	95	10:90	22
19	Cyclohexane	77	18:82	33
20	THF	65	88:12	2
21	1,4-Dioxane	71	–	3
22	Cyclohexane : Toluene (1:1, 1 mL)	92	<1:99	33
23	Cyclohexane : Toluene (9:1, 1 mL)	87	<1:99	37

[a]Reaction conditions: **1a** (0.43 mmol), CuCl (10 mol%), DPPP (15 mol%), NaO^{t}Bu (2 equiv), Solvent (1.0 mL), CO (10 bar), H_2 (10 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.**

In terms of chemoselectivity towards alcohol product **3a**, the cyclohexane/toluene mixture was superior.

Effects of Reaction Temperature

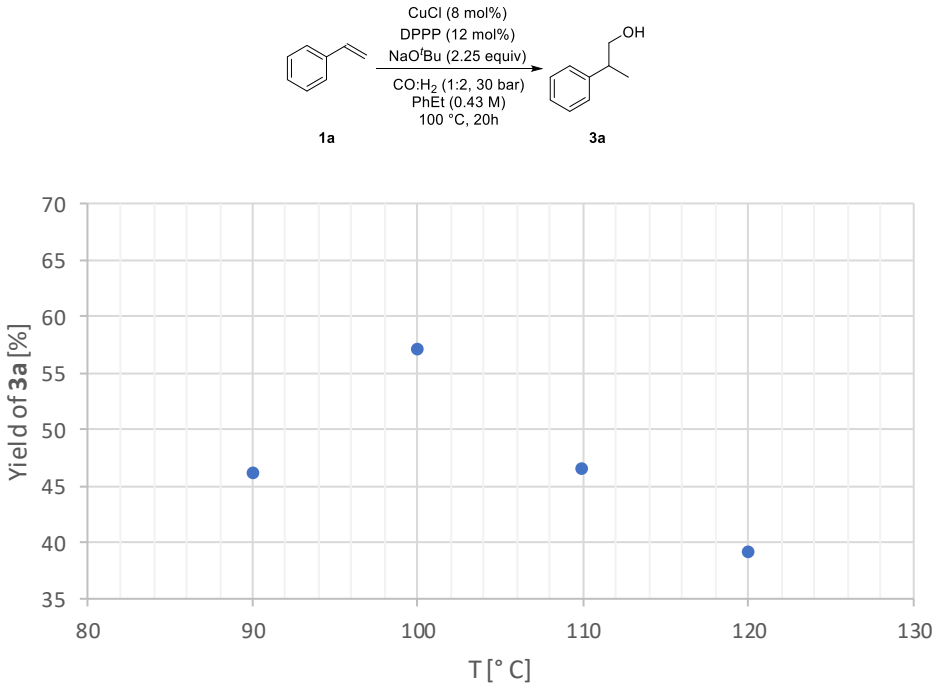
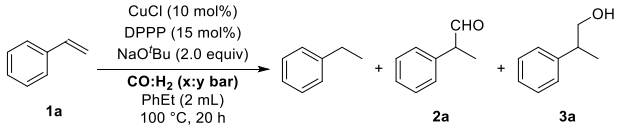


Figure S1. Effects of reaction temperature. aReaction conditionss: **1a** (0.43 mmol), CuCl (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv) in Cyclohexane : Toluene (9:1, 1 mL), 100 °C, 20 h.

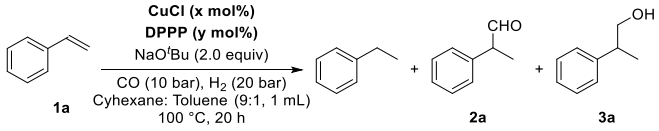
Table S3. **Screening of CO/H$_2$ Partial Pressures.**



Entry	CO (bar):H$_2$ (bar)	Conv. (%)	**2a:3a**	^{b}GC-Yield (%)
1	2:1, 15 bar	89	23:77	32
2	2:1, 30 bar	75	61:39	49
3	1:1, 20 bar	90	10:90	42
4	1:3, 40 bar	91	<1:99	36
5	1:2, 30 bar	88	<1:99	44

^{a}Reaction conditions: **1a** (0.43 mmol), CuCl (10 mol%), DPPP (15 mol%), NaO^{t}Bu (2.0 equiv), Cyclohexane: Toluene (9:1, 1.0 mL), 100 °C, 20 h. ^{b}GC-Yield of **2a** and **3a.**

Table S4. **Optimization of the Amount of CuCl.**

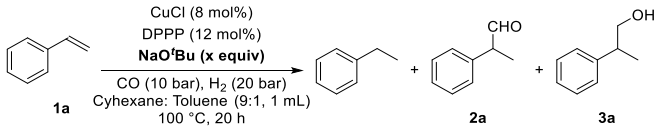


Entry	CuCl (mol%)	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	10	88	<1:99	44
2	8	90	<1:99	48
3	5	89	<1:99	46
4	3	86	47:53	38
5	1	53	53:47	26

[a]Reaction conditions: **1a** (0.43 mmol), CuCl: DPPP = 1:1.5, NaO^{t}Bu (2.0 equiv), CO (10 bar), H_2 (20 bar), Cyclohexane: Toluene (9:1, 1.0 mL),100 °C, 20 h. [b]GC-Yield of **2a** and **3a.**

T*able S5.* **Optimization of the Amount of NaO^{t}Bu.**

We screened the amounts of base under further optimized conditions.

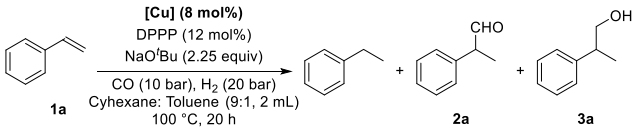


Entry	NaO^{t}Bu (equiv)	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	2.0	90	<1:99	48
2	2.25	89	11:89	56
3[c]	2.25	94	<1:99	53
4	2.5	81	66:34	58
5	2.75	88	43:57	56
6	3.0	91	58:42	60

[a]Reaction conditions: **1a** (0.43 mmol), CuCl (8 mol%), DPPP (12 mol%), NaO^{t}Bu, CO (10 bar), H_2 (20 bar), Cyclohexane: Toluene (9:1, 1.0 mL), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.** [c]Cyclohexane: Toluene (9:1, 2.0 mL).

T*able S6.* **Optimization of Cu Source.**

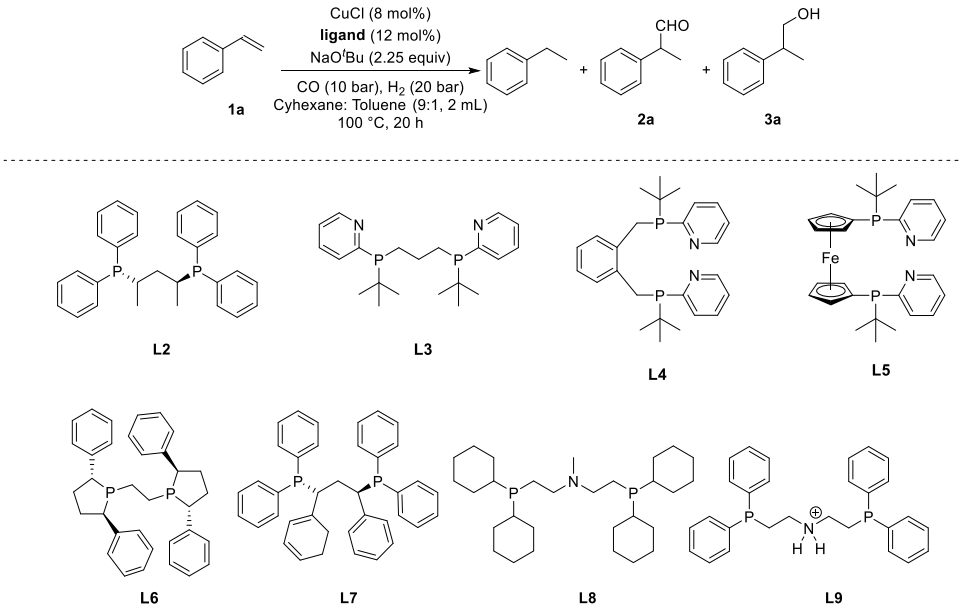
We screened different copper salts under further optimized conditions. We find that when we use CuCN as catalyst, the ratio fof 2a/3a will be higher.



Entry	Cu	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	CuCl	94	<1:99	53
2	CuO	15	100:0	3
3	CuBr	92	9:91	60
4[c]	$[CuOTf]_2$•Toluene	86	31:69	34
5	$CuBr_2$	69	52:48	27
6	$Cu(NO_3)_2$	97	11:89	44
7	CuCN	88	85:15	60
8	$CuSO_4$	67	73:27	41
9	$Cu(HCO_2)_2$	82	73:27	52

[a]Reaction conditions: **1a** (0.43 mmol), [Cu] (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Cyclohexane: Toluene (9:1, 2.0 mL), CO (10 bar), H_2 (20 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.** [c]$[CuOTf]_2$•Toluene (4 mol%)

Table S7. **Screening of the Ligand.**

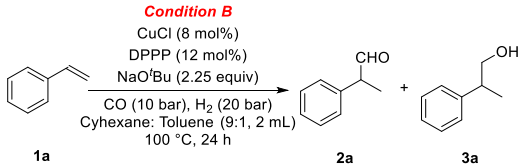


Entry	**Ligand**	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	Xantphos	100	25:75	16
2	BINAP	N.R.	–	–
3	DPPE	61	75:25	28
4	DPPB	85	58:42	51
5	DPPPE	61	81:19	26
6	DPPF	100	80:20	51
7	DCyPE	100	<1:99	3
8	$BuPAd_2$	47	<1:99	3
11	DPPP	94	<1:99	53

12[c]	DPPP	92	<1:99	58
13	**L2**	100	16:84	54
15	**L3**	71	91:9	18
16	**L4**	86	30:70	29
17	**L5**	49	81:19	19
18	**L6**	91	65:35	42
19	**L7**	70	–	n.d.
20	**L8**	57	–	5
21	**L9**	82	–	3

[a]Reaction conditions: **1a** (0.43 mmol), CuCl (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Cyclohexane (1.8 mL), Toluene (0.2 mL), CO (10 bar), H_2 (20 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.** [c]24 h.

Table S8. **Final Optimization of Cu-Promoted Hydroformylation Towards 3a.**



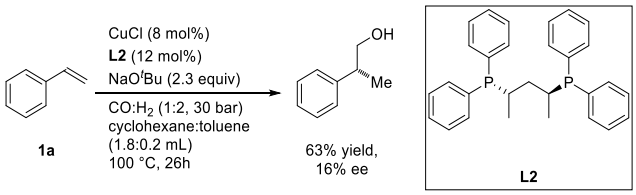
Entry	Varying from the condition B	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	Standard condition	92	<1:99	58
2	Cyclohexane: Toluene (9:1, 2.5 mL)	84	<1:99	61
3	Cyclohexane: Toluene (9:1, 3.0 mL)	84	<1:99	56
4	Toluene (2.5 mL)	86	<1:99	68
5	Xylene[c] (2.5 mL)	–[d]	<1:99	66
6	Additive of H_2O (0.5 equiv)	44	82:18	34
7	Additive of H_2O (1.25 equiv)	65	n.d.	14
8	Additive of H_2O (2.5 equiv)	–	–	0
9	Additive of PHMS (8 mol%)	88	<1:99	37
10	Additive of DEMS (8 mol%)	92	50:50	42
11[c]	Cu-H Source (8 mol%)	–	–	0
12	CuBr (8 mol%)	95	<1:99	54
13	CuBr·DMS (8 mol%),	91	<1:99	48
14	KCN (8 mol%) was added	71	72:28	61

[a]Reaction conditions: **1a** (0.43 mmol), CuCl (mol%), DPPP (mol%), NaO^{t}Bu (2.25 equiv), solvents, CO (10 bar), H_2 (20 bar), 100°C, 26 h. [b]GC-Yield of **2a** and **3a.**

[c]Commercially available "Hot" Stryker's reagent in Toluene (CAS: 13517-00-5)

***Table S9*. Stereoselectivity**

The use chiral ligand L2 gave no notable enantiomeric excess.

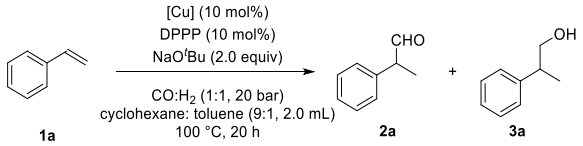


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.937	MM	0.1679	519.94867	51.61646	41.6467
2	9.960	MM	0.2096	728.52716	57.93745	58.3533

2.2. Optimization of Reaction Conditions of Aldehyde

***Table S10.* Optimization of Copper source.**

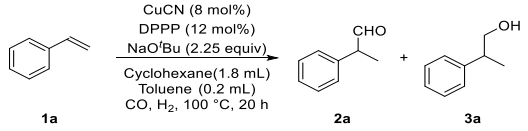
This table is in accordance with Table S9 and was used as a starting point for the optimization towards aldehyde product **2a**.



Entry	Cu	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
2	CuCl	94	<1:99	53
3	CuO	15	100:0	3
4	CuBr	92	9:91	60
5[c]	$[CuOTf]_2 \cdot$ Toluene	86	31:69	34
6	$CuBr_2$	69	52:48	27
7	$Cu(NO_3)_2$	97	11:89	44
8	CuCN	88	85:15	60
9	$CuSO_4$	67	73:27	41
10	$Cu(HCO_2)_2$	82	73:27	52

[a]Reaction conditions: **1a** (0.43 mmol), [Cu] (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Cyclohexane: Toluene (9:1, 2.0 mL), CO (10 bar), H_2 (20 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.** [c][Cu] (4 mol%).

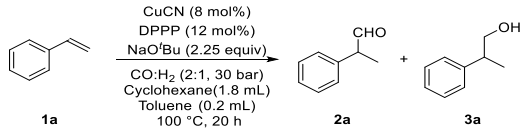
Table S11. **Optimization of the ratio of CO and H_2.**



Entry	CO (bar): H_2 (bar)	Conv. (%)	**2a:3a**	^{b}GC-Yield (%)
1	10:20	88	83/17	62
2	15:15	89	72/18	74
3	20:10	88	89/11	60
4	24:6	52	64/36	56

^{a}Reaction conditions: **1a** (0.43 mmol), CuCN (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Cyclohexane: Toluene (9:1, 2.0 mL), CO, H_2, 100 °C, 20 h. ^{b}GC-Yield of **2a** and **3a.**

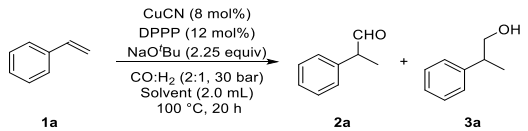
Table S12. **Optimization of addition.**



Entry	addition	Conv. (%)	**2a:3a**	^{b}GC-Yield (%)
1	w/o	88	89/11	60
2	KCN (1 equiv.)	84	84/16	57
3	PTSA 5 mol%	84	77/23	56
4	PTSA 10 mol%	91	91/9	56
5	PTSA 30 mol%	70	77/23	31

^{a}Reaction conditions: **1a** (0.43 mmol), CuCN (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Cyclohexane: Toluene (9:1, 2.0 mL), CO (20 bar), H_2 (10 bar), 100 °C, 20 h. ^{b}GC-Yield of **2a** and **3a.**

Table S13. **Optimization of solvent.**

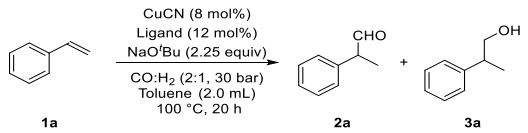


Entry	Solvent	Conv. (%)	**2a:3a**	^{b}GC-Yield (%)
1	Cyclohexane :Toluene (9:1, 2.0 mL)	88	89/11	60
2	PhCl	73	79/21	70
3	Toluene	91	92/8	68
4	*o*-Xylene	88	76/24	73

5	Cyclohexane	87	76/24	42
6	Benzene	84	74/26	68
7	PhEt	69	74/26	65
8	PhOMe	75	80/20	69

[a] Reaction condition: **1a** (0.43 mmol), CuCN (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Solvent (2.0 mL), CO (20 bar), H_2 (10 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.**

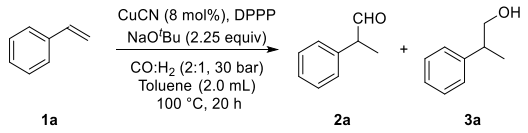
Table S14. **Optimization of ligand.**



Entry	Ligand	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	DPPP	91	92/8	68
2	DPPF	73	86/14	57
3	DPPE	59	88/12	33
4	DPPB	60	87/13	32
5	DPPPE	58	88/12	25
6	BiPhePhos	30	100:0	5

[a]Reaction condition: **1a** (0.43 mmol), CuCN (8 mol%), Ligand (12 mol%), NaO^{t}Bu (2.25 equiv), Toluene (2.0 mL), CO (20 bar), H_2 (10 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.**

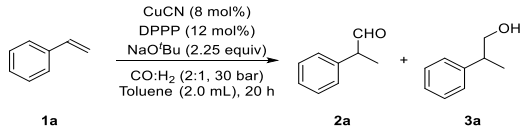
Table S15. **Optimization of the amount of ligand.**



Entry	DPPP	Conv. (%)	**2a:3a**	[b]GC-Yield (%)
1	10%	87	72/28	64
2	12%	91	92/8	68
3	15%	83	70/30	64
4	20%	88	75/25	57

[a]Reaction conditions: **1a** (0.43 mmol), CuCN (8 mol%), DPPP, NaO^{t}Bu (2.25 equiv), Toluene (2.0 mL), CO (20 bar), H_2 (10 bar), 100 °C, 20 h. [b]GC-Yield of **2a** and **3a.**

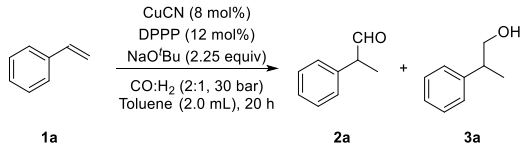
Table S16. **Optimization of temperature.**



Entry	Temperature (℃)	Conv. (%)	**2a:3a**	GC-Yield (%)[b]
1	100	91	92/8	68
2	90	92	73/27	66
3	110	88	94/6	57

[a]Reaction condition: **1a** (0.43 mmol), CuCN (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Toluene (2.0 mL), CO (20 bar), H_2 (10 bar), 20 h. [b]GC-Yield of **2a** and **3a.**

Table S17. **Optimization of addition.**

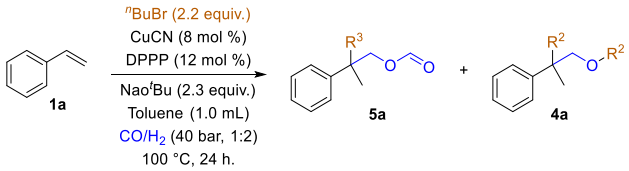


Entry	addition	Conv. (%)	**2a:3a**	GC-Yield (%)[b]
1	w/o	91	92/8	68
2	$(EtO)_3SiH$ (8 mol%)	96	33/67	36
3	PMHS (8 mol%)	85	83/17	42
4	DEMS (8 mol%)	96	45/55	40
5	KCN (8 mol%)	89	84/16	55
6	MeCN (8 mol%)	93	87/13	55
7	*o* -Tolunitrile (8 mol%)	91	89/11	57

[a]Reaction condition: **1a** (0.43 mmol), CuCN (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.25 equiv), Toluene (2.0 mL), CO (20 bar), H_2 (10 bar), 100 ℃, 20 h. [b]GC-Yield of **2a** and **3a.**

2.3. Optimization of 4a

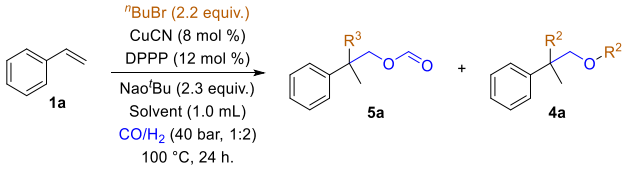
Table S18. **Optimization of addition.**



Entry	Addition	**3a**	**4a**
1	-	trace	14
2	NaI (20%)	5	12

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN (8.0 mol%), DPPP (12.0 mol%), Base (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 24 h.

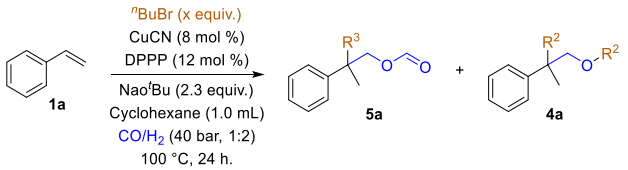
Table S19. **Optimization of solvent.**



Entry	Solvent	Yield of **4a** (%)
1	Anisole	5
2	*n*-Heptane	16
3	*n*-hexane	14
4	Cyclohexane	17
5	PhCl	10
6	PhF	7
7	MeCN	5
8	Toluene	14

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN (8 mol%), DPPP (12 mol%), NaO^{t}Bu (2.3 equiv.), solvent (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 24 h.

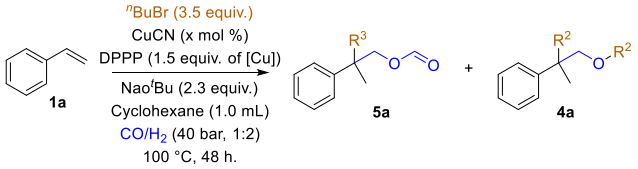
Table S20. **Optimization of the amount of alkyl halides.**



Entry	^{n}BuBr	Yield of **4a** (%)
1	2.2	17
2	2.5	20
3	3.0	20
4	3.5	23
5	4.0	24
6[b]	3.5	30

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN (5 mol%), DPPP (7.5 mol%), NaO^{t}Bu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 24 h. [b] 48 h

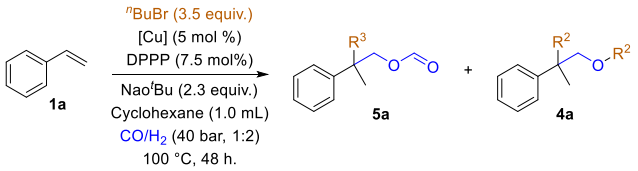
Table S21. **Optimization of the amount of catalysis.**



Entry	The amount of catalysis	Yield of **4a** (%)
1	5%	30
2 [c]	5%	32
3	1%	14
4	2%	18
5	8%	29
6	12%	22
7	15 %	22

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN, DPPP (1.5 equiv. of CuCN), NaO^{t}Bu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h. [b] 72 h.

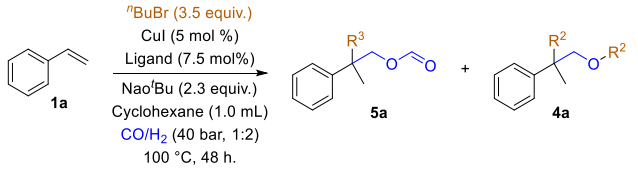
Table S22. **Optimization of catalysis.**



Entry	[Cu]	Yield of **4a** (%)
1	CuCl	19
2	$CuCl_2$	22
3	$Cu(OTf)_2$	19
4	CuBr	20
5	CuI	30
6	$Cu(acac)_2$	26
7	$Cu(OAc)_2$	24

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN (5 mol%), DPPP (1.5 equiv. of CuCN), NaO^{t}Bu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

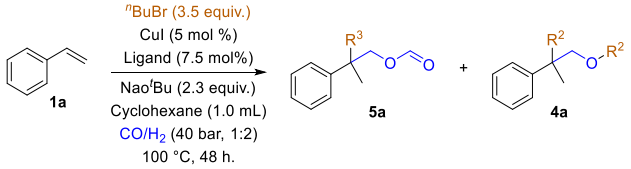
Table S23. **Optimization of ligand.**



Entry	Ligand	Yield of **4a** (%)
1	CyJohnphos	-
2	PPh_3	-
3	DPPB	11
4	Xantphos	29
5	DPEphos	19
6	DPPF	28
7	BINAP	-
8	$BuPAd_2$	2
	4-OMe-Dppp	33

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN (5 mol%), DPPP (1.5 equiv. of CuCN), NaO^{t}Bu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

Table S24. **Optimization of the amount of base.**

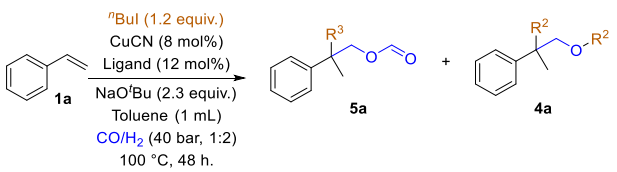


Entry	The amount of Base	Yield of **4a** (%)
1	2.0 equiv.	28
2	2.3 equiv.	33
3	2.5 equiv.	31
4	3.0 equiv.	34
5	3.5 equiv.	37
6	4.0 equiv.	45
7	4.5 equiv	35
8	5.0 equiv	36

[a] Reaction condition: styrene (0.2 mmol), 1-bromobutane (2.2 equiv.), CuCN (5 mol%), DPPP (1.5 equiv. of CuCN), NaO^{t}Bu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

2.4. Optimization of 5a

Table S25. **Optimization of ligand.**

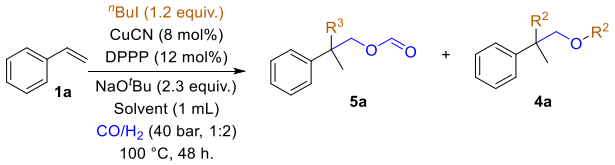


Entry	Ligand	**5a**	**4a**
1[b]	CyJohnphos	-	4
2[b]	PPh_3	-	-

3	DPPM	1	0
4	DPPB	-	3
5	BINAP	1	1
6	DPPE	7	2
7	DPPPe	1	1
8	DCyPE	5	2
9	DPPP	27	5
10	Xantphos	4	3
11	DPEphos	4	5
12	DPPF	6	5

[a] Reaction condition: Styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), CuCN (8.0 mol%), Ligand (12.0 mol%), NaO^{t}Bu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h. [b] Ligand (24 mol%).

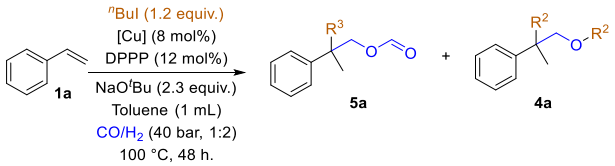
Table S26. **Optimization of Solvent.**



Entry	Solvent	**5a**	**4a**
1	Toluene	27	5
2	PhF	16	4
3	PhCl	7	5
4	$PhCF_3$	-	4
5	PhEt	22	5
6	*p*-Xylene	17	4
7	Anisole	9	3
8	*n*-Heptane	-	7
9	DMF	-	-
10	THF	-	-
11	DCM	-	-
12	CH_3CN	-	8

[a] Reaction condition: styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), CuCN (8.0 mol%), DPPP (12.0 mol%), NaO^{t}Bu (2.3 equiv.), Solvent (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

Table S27. **Optimization of [Cu] Source.**

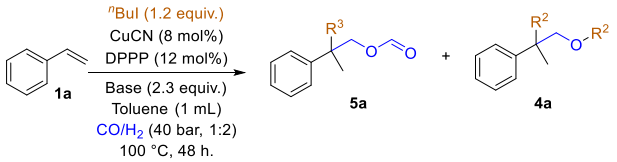


Entry	[Cu]	**5a**	**4a**
1	CuCN	27	5
2	CuCl	13	5

3	$CuCl_2$	10	4
4	$Cu(OAc)_2$	11	2
5	CuI	15	4
6	CuBr	14	3
7	IPrCuCl	-	-

[a] Reaction condition: styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), [Cu] (8.0 mol%), DPPP (12.0 mol%), NaO^tBu (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

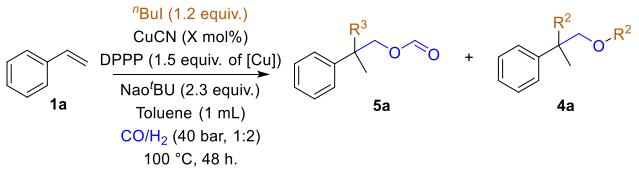
Table S32. **Optimization of Base.**



Entry	Base	**5a**	**4a**
1	NaO^tBu	27	5
2	KO^tBu	-	-
3	LiO^tBu	-	-
4	NaOMe	-	-
5	NaOEt	-	-
6	KOMe	-	-
7	NaOPh	-	-

[a] Reaction condition: styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), CuCN (8.0 mol%), DPPP (12.0 mol%), Base (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

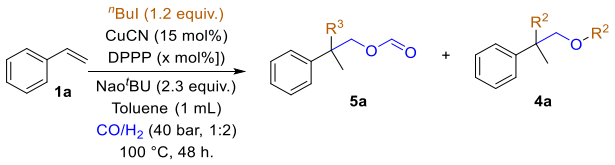
Table S28. **Optimization of the amount of catalysis.**



Entry	The amount of catalysis	**5a**	**4a**
	5%	26	7
1	8%	27	5
2	12%	28	4
3	15 %	27	3
4	17%	7	3

[a] Reaction condition: styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), CuCN (8.0 mol%), DPPP (1.5 equiv. of CuCN), Base (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

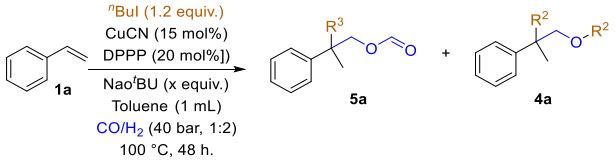
Table S29. **Optimization of the amount of ligand.**



Entry	The amount of Ligand	**5a**	**4a**
1	/	/	/
2	15%	30	5
3	20%	35	4
4	25%	25	4
5	30%	19	4

[a] Reaction condition: styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), CuCN (15 mol%), DPPP (1.5 equiv. of catalysis), Base (2.3 equiv.), Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

Table S30. **Optimization of the amount of Base**



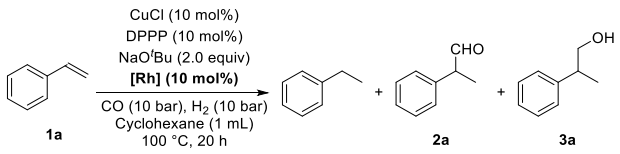
Entry	The amount of Base	**5a**	**4a**
1	1.2 equiv.	5	2
2	1.5 equiv.	4	2
3	2.0 equiv.	17	3
4	2.5 equiv.	26	4
5	3.0 equiv.	19	4

[a] Reaction condition: styrene (0.2 mmol), 1-iodobutane (1.2 equiv.), CuCN (15 mol%), DPPP (20 mol%), NaO^{t}Bu, Toluene (1.0 mL), CO/H_2 (40 bar, 1:2), 100 °C, 48 h.

2.5. Control Experiments

2.5.1 Control Experiments with Additional Rh Catalysts.

Table S31.

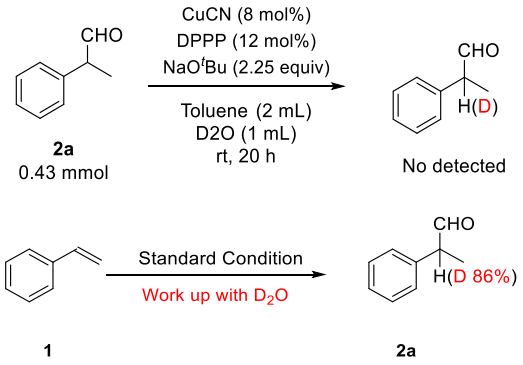


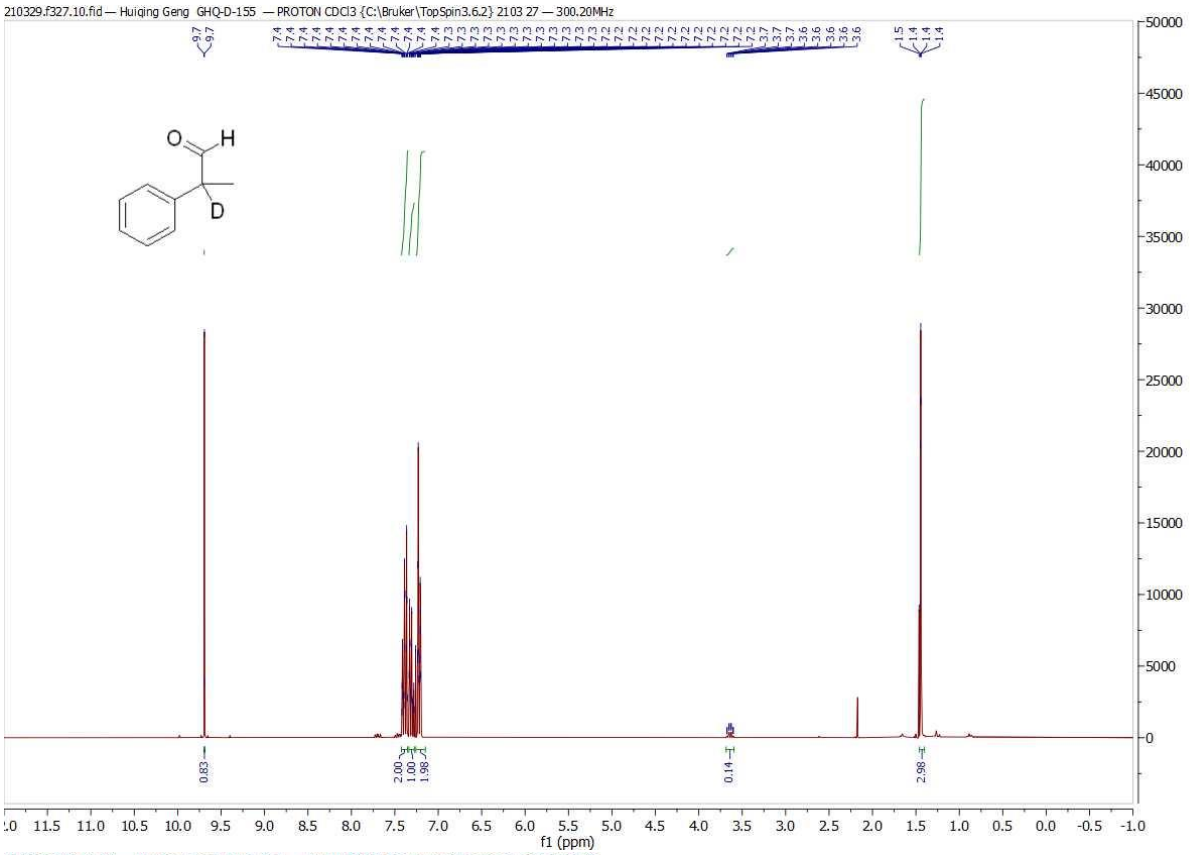
Entry	CuCl	**[Rh]**	**2a**	**3a**
1	–	$[RhCl(COD)]_2$	observed	Not detected
2	10 mol%	$[RhCl(COD)]_2$	observed	Not detected

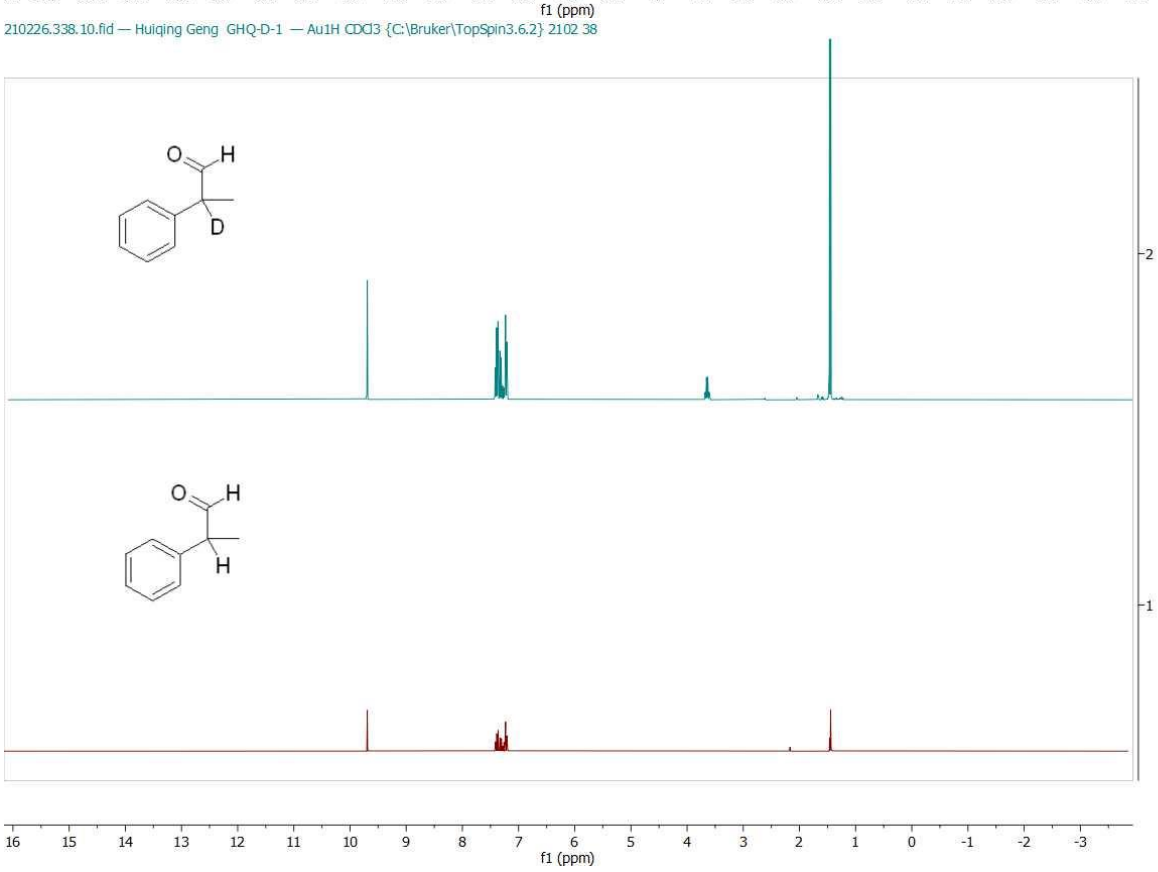
3	10 mol%	$[Rh(CO)_2acac]$	observed	Not detected
4	10 mol%	$RhCl_3$	observed	Not detected

[a] Reaction conditions: **1a** (0.43 mmol), DPPP (10 mol%), NaO^{t}Bu (2 equiv), Cyclohexane (1.0 mL), CO (10 bar), H_2 (10 bar), 100 °C, 20 h.

2.5.2 Deuterium-labeling experiments




210329.f327.10.fid — Huiqing Geng GHQ-D-155 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 27 — 300.20MHz
O
H
D
f1 (ppm)


210226.338.10.fid — Huiqing Geng GHQ-D-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2102 38
O
H
D
O
H
H
f1 (ppm)

2.5.3 ICP of CuCl Salt

An ICP measurement was made of the solid CuCl and analyzed for traces of other metals. Special attention was paid to Rh, as it could be active in ppm traces. The detection limits were Pd 0.06w%, Rh 0.17w%, Ru 0.07w%, Ir 0.06w%, Co 0.02w%, Fe 0.01w%, and Pt 0.05w%. The ICP-MS experiment of the diluted sample was performed on a Thermo Fisher Scientific X Series. With the exception of Fe, no trace metals were detected. The possible concentration of metals in a standard reaction mixture according to General Procedure A (taking into account the detection limit) would consequently give:

Metal	Concentration in ppb
Pd	< 9
Rh	< 25
Ru	< 10
Ir	< 9
Co	< 3
Fe	> 1
Pt	< 7

ICP-LABOR

A. Simmula, Tel. 0381 1281 315
ANALYSENERGEBNIS / KURZBERICHT

18059 Rostock
Albert-Einstein-Str. 29a

AUFTRAG

Datum:05.02.2021

MS-Auftragsnummer:	**10659**	
Probenbezeichnung:	**TM-HY-62**	
Auftraggeber:	**Meyer, Tim**	
Institution/Projekt:	LIKAT	Tel: 143

ANALYSE

Methode: ICP

durchgeführt von: as

Ergebnis :

Element	erwartet*	1. Messung	2. Messung
Pd	-%	nn%	nn%
Rh	-%	nn%	nn%
Ru	-%	nn%	nn%
Ir	-%	nn%	nn%
Pt	-%	nn%	nn%
Co	-%	nn%	nn%
Fe	-%	nn%	n%

* ev.zusätzliche Messung, s. Bemerkungen

Bemerkungen:

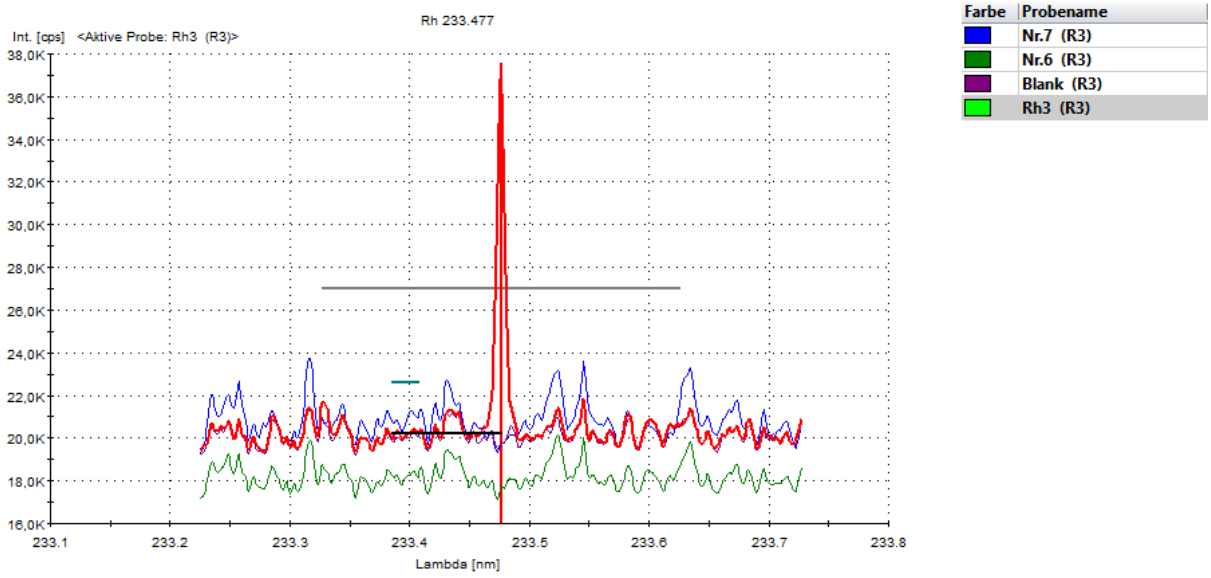
nn = nicht nachweisbar (German) = not detectable

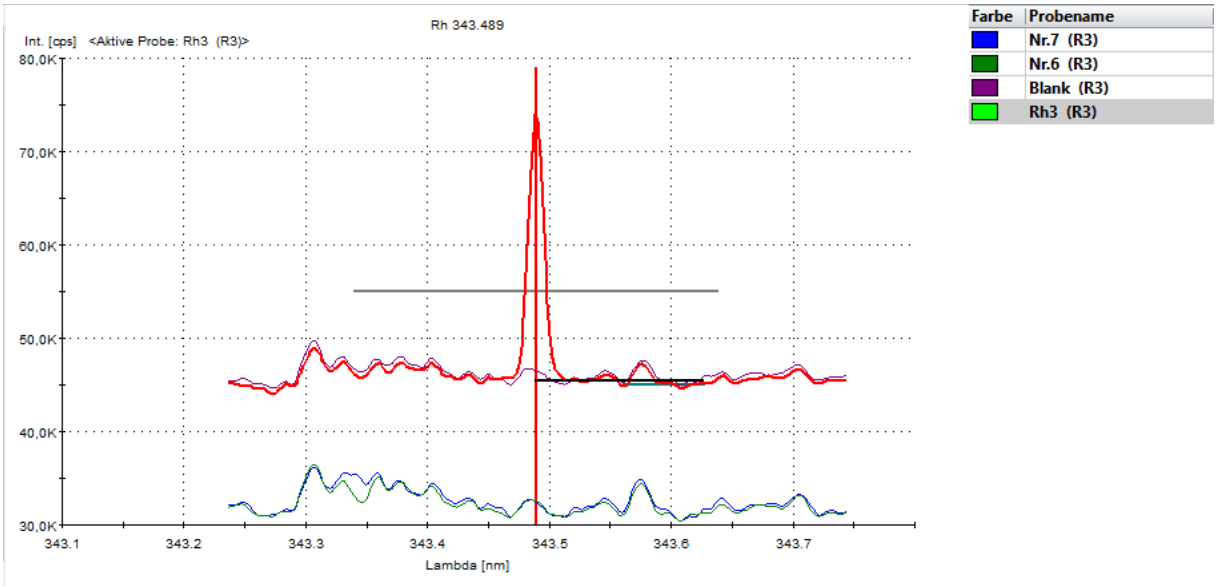
Unterschrift / Datum:

Anlagen (Erläuterungen / Abildungen ☐

2.5.4 ICP of the Reaction Mixtures

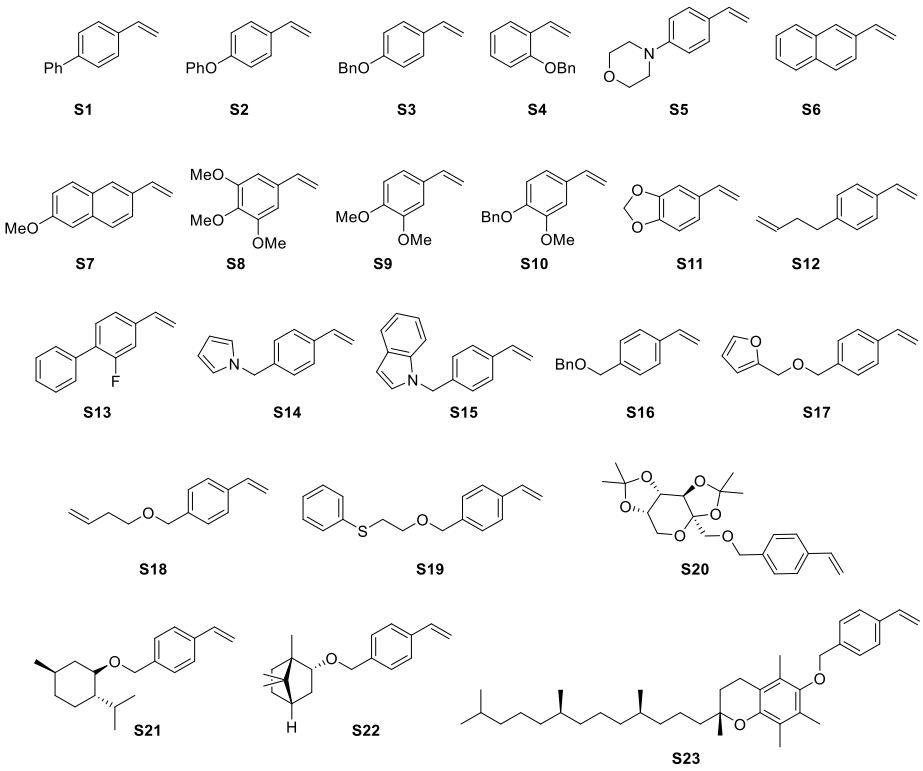
To exclude the possibility of Rh impurities from other sources (stir bar, base, ligand, etc.), ICP measurements were taken directly after a reaction run from the reaction solution. For this purpose, an aliquot of the reaction solution was diluted with xylene and examined. First, a reference solution was prepared. For this purpose, Wilkinson's catalyst [$RhCl(PPh_3)_3$] (0.323 mg) was first dissolved in xylene (10.000 g) and stirred for 1 h. This was further diluted with xylene to create a 0.3 ppm reference solution (red line). According to General Procedure A, a dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with CuCl (3.4 mg, 8 mol%), DPPP (21.3 mg, 12 mol%), and NaO^{t}Bu (93.0 mg, 2.25 equiv). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. Xylene (2.5 mL) was added *via* syringe. To this suspension, styrene **1a** (0.43 mmol) was added *via* Hamilton® syringe. The vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (10 bar) and subsequent with H_2 (20 bar) to reach the desired pressure of 30 bar. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 26 h at 100 °C. Then it was cooled to room temperature and the pressure was released carefully. An aliquot of 1.038g of the reaction solution was diluted to 5.067g of xylene (mixture of isomers). As can be seen in the figures below, no Rh could be detected (Sample "Nr.6", dark green line).



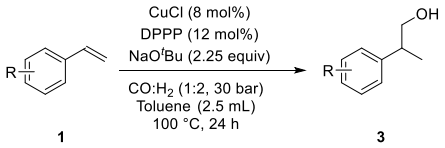


2.6. General Procedures for Preparing Vinylarenes

Vinylarenes **S1-S11**[1], **S12**[2], **S13**[3], **S14**[4], **S15**[4], **S16-S20**[5], **S21**[6], **S22**[6], **S23**[7] were prepared according to known literature procedures.



2.7. General Procedure A for Preparing Alcohols

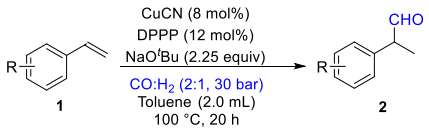


A dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with CuCl (3.4 mg, 8 mol%), DPPP (21.3 mg, 12 mol%), and NaO^{t}Bu (93.0 mg, 2.25 equiv). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. Toluene (2.5 mL) were added *via* syringe. To this suspension, substrate **1** (0.43 mmol) was added *via* Hamilton® syringe. The vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (10 bar) and subsequent with H_2 (20 bar) to reach the desired pressure of 30 bar. *Special attention needs to be paid while charging two gases: After the autoclave is charged with CO (10 bar), the tube needs to be purged with high pressure hydrogen. It is mandatory to have higher pressure inside the tube than in the autoclave! Therefore, the tab towards the main gas supply was left open. By that it was ensured, that no CO could come out while charging H_2, keeping the ratio 1:2.* The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 24 h at 100 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction was quenched with saturated NH_4Cl solution (1.0 mL), extracted with Et_2O (3x), washed with water (3.0 mL), dried over Na_2SO_4. Combined organic layers were concentrated with the aid of a rotary evaporator. The residue was purified by gradient column chromatography (pentane:EtOAc = 9:1 → 3:1) to yield product **3**.

In the case of GC-Analysis:

After the gases were released, *n*-hexadecane was added as an internal standard to the reaction mixture, followed by a small amount of Et_2O (0.8 mL) (*to facilitate an extraction and to ensure that all the internal standard is in solution*). Hereto saturated NH_4Cl solution was added (1.0 mL). The two-phase vials were stirred rigorously (1200 rpm) for 5 min to quench the reaction. From the top organic phase, an aliquot (1.0 mL) of the mixture was filtered with a short pipette flash column (silica) and Na_2SO_4. (~3 cm). Samples prepared in this way were subjected to GC analysis.

2.8. General Procedure B for Preparing Aldehydes.



A dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with CuCN (3.1 mg, 8 mol%), DPPP (21.3 mg, 12 mol%), and NaO^{t}Bu (93.0 mg, 2.25 equiv). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times.

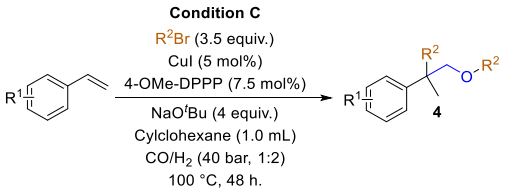
Toluene (2.0 mL) were added *via* syringe. To this suspension, substrate **1** (0.43 mmol) was added *via* Hamilton® syringe.

The vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (< 10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (20 bar) and subsequent with H_2 (10 bar) to reach the desired pressure of 30 bar. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 20 h at 100 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction was quenched with saturated NH_4Cl solution (1.0 mL), extracted with Et_2O (3x). The combined organic layers were washed with water (3.0 mL) and dried over Na_2SO_4, filtered, and concentrated in vacuum. The crude reaction mixture was purified by flash chromatograph on silica gel and evaporated to give the aldehyde product.

In the case of GC-Analysis:

After the gases were released, *n*-hexadecane was added as an internal standard to the reaction mixture, followed by a small amount of Et_2O (0.8 mL) (*to facilitate an extraction and to ensure that all the internal standard is in solution*). Hereto saturated NH_4Cl solution was added (1.0 mL). The two-phase vials were stirred rigorously (1200 rpm) for 5 min to quench the reaction. From the top organic phase, an aliquot (1.0 mL) of the mixture was filtered with a short pipette flash column (silica) and Na_2SO_4. (~3 cm). Samples prepared in this way were subjected to GC analysis.

2.9. General Procedure C for Preparing Esters 4

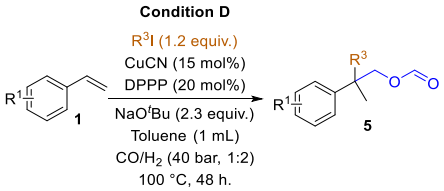


A dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with CuI (1.9 mg, 5 mol%), 4-OMe-DPPP (8.0 mg, 7.5 mol%), and NaO^{t}Bu (96.9 mg, 4.0 equiv). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. Cyclohexane (1.0 mL) were added *via* syringe. To this suspension, substrate **1** (0.2 mmol) was added *via* Hamilton® syringe.

The vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (13 bar) and subsequent with H_2 (27 bar) to reach the desired pressure of 40 bar. *Special attention needs to be paid while charging two gases: After the autoclave is charged with CO (13 bar), the tube needs to be purged with high pressure hydrogen. It is mandatory to have higher pressure inside the tube than in the autoclave! Therefore, the tab towards the main gas supply was left open. By that it was ensured, that no CO could come out while charging H_2, keeping the ratio 1:2.* The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 48 h at 100 °C. Then it was cooled to room

temperature and the pressure was released carefully. The reaction purified by gradient column chromatography (Pentane:DCM = 20:1) to yield product **4**.

2.10. General Procedure D for formic acid esters 5.



A dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with CuCN (2.7 mg, 15 mol%), DPPP (16.5 mg, 20 mol%), and NaO^{t}Bu (44.2 mg, 2.3 equiv). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. Toluene (1.0 mL) were added *via* syringe. To this suspension, substrate **1** (0.2 mmol) was added *via* Hamilton® syringe. The vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (13 bar) and subsequent with H_2 (27 bar) to reach the desired pressure of 40 bar. *Special attention needs to be paid while charging two gases: After the autoclave is charged with CO (13 bar), the tube needs to be purged with high pressure hydrogen. It is mandatory to have higher pressure inside the tube than in the autoclave! Therefore, the tab towards the main gas supply was left open. By that it was ensured, that no CO could come out while charging H_2, keeping the ratio 1:2.* The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (600 rpm) for 48 h at 100 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction was quenched with saturated NH_4Cl solution (1.0 mL), extracted with Et_2O (3x). The combined organic layers were washed with water (3.0 mL) and dried over Na_2SO_4, filtered, and concentrated in vacuum. The reaction purified by gradient column chromatography (Pentane:EtOAc = 100:1) to yield product **5**.

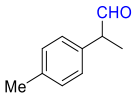
3. Characterization Data

3.1 Characterization Data of Aldehydes

2-Phenylpropanal (2a)

The title compound was prepared from styrene (44.8 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.7) to give the product as a colorless oil (39 mg, 68%).

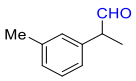
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.69 (d, *J* = 1.4 Hz, 1H), 7.43 – 7.35 (m, 2H), 7.35 – 7.27 (m, 1H), 7.26 – 7.17 (m, 2H), 3.64 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.45 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 137.8, 129.2, 128.4, 127.6, 53.1, 14.7. All data was consistent with that previously reported.[8]



2-(*p*-Tolyl)propanal (2b)

The title compound was prepared from 1-methyl-4-vinylbenzene (50.1 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.7) to give the product as a colorless oil (44 mg, 69%).

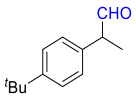
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.57 (d, *J* = 1.5 Hz, 1H), 7.10 (d, *J* = 7.8 Hz, 2H), 7.00 (d, *J* = 8.2 Hz, 2H), 3.50 (qd, *J* = 7.1, 1.3 Hz, 1H), 2.25 (s, 3H), 1.33 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.4, 137.4, 134.8, 129.9, 128.4, 52.8, 21.2, 14.8. All data was consistent with that previously reported.[9]



2-(*m*-Tolyl)propanal (2c)

The title compound was prepared from 1-methyl-3-vinylbenzene (50.1 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.7) to give the product as a colorless oil (40.1 mg, 63%).

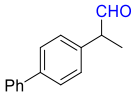
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.64 (d, *J* = 1.4 Hz, 1H), 7.24 – 7.21 (m, 1H), 7.08 (d, *J* = 7.3 Hz, 1H), 7.01 – 6.94 (m, 2H), 3.56 (qd, *J* = 7.0, 1.4 Hz, 1H), 2.32 (s, 3H), 1.39 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 138.8, 137.6, 129.1, 129.0, 128.3, 125.4, 53.0, 21.4, 14.6. All data was consistent with that previously reported.[9]



2-(4-(*tert*-Butyl)phenyl)propanal (2d)

The title compound was prepared from 1-(*tert*-butyl)-4-vinylbenzene (68.8 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.8) to give the product as a colorless oil (54.8 mg, 67%).

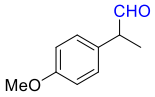
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.68 (d, *J* = 1.5 Hz, 1H), 7.41 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.2 Hz, 2H), 3.61 (qd, *J* = 7.0, 1.4 Hz, 1H), 1.44 (d, *J* = 7.1 Hz, 3H), 1.33 (s, 9H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.3, 150.5, 134.6, 128.0, 126.0, 52.5, 34.5, 31.3, 14.6. **HRMS** (EI): calcd for [M] $C_{13}H_{18}O$ 190.1352, found: 190.1350. All data was consistent with that previously reported.[10]



2-([1,1'-Biphenyl]-4-yl)propanal (2e)

The title compound was prepared from 4-vinyl-1,1'-biphenyl (77.4 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a white solid (24.4 mg, 27%).

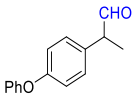
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.74 (d, *J* = 1.4 Hz, 1H), 7.65 – 7.58 (m, 4H), 7.49 – 7.42 (m, 2H), 7.39 – 7.28 (m, 3H), 3.70 (qd, *J* = 7.1, 1.2 Hz, 1H), 1.50 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.1, 140.7, 136.8, 129.0, 128.9, 127.9, 127.6, 127.2, 52.8, 14.8. All data was consistent with that previously reported.[10]



2-(4-Methoxyphenyl)propanal (2f)

The title compound was prepared from 1-methoxy-4-vinylbenzene (57.6 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.3) to give the product as a colorless oil (34.6 mg, 49%).

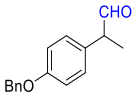
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.65 (d, *J* = 1.5 Hz, 1H), 7.13 (dd, *J* = 8.8, 0.4 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.81 (s, 3H), 3.59 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.42 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.2, 159.0, 129.6, 129.4, 114.5, 55.3, 52.2, 14.7. All data was consistent with that previously reported.[8]



2-(4-Phenoxyphenyl)propanal (2g)

The title compound was prepared from 1-phenoxy-4-vinylbenzene (84.3 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.3) to give the product as a colorless oil (54.4 mg, 56%).

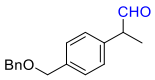
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.59 (d, *J* = 1.5 Hz, 1H), 7.29 – 7.21 (m, 2H), 7.09 – 6.99 (m, 3H), 6.97 – 6.88 (m, 4H), 3.53 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.35 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.1, 157.1, 156.9, 132.4, 129.9, 129.7, 123.6, 119.4, 119.2, 52.4, 14.8. **HRMS** (EI): calcd for [M] $C_{15}H_{14}O_2$ 226.0988, found: 219.0988.



2-(4-(Benzyloxy)phenyl)propanal (2h)

The title compound was prepared from 1-(benzyloxy)-4-vinylbenzene (90.3 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.3) to give the product as a colorless oil (67.1 mg, 65%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.52 (d, *J* = 1.5 Hz, 1H), 7.35 – 7.15 (m, 5H), 7.05 – 6.95 (m, 2H), 6.90 – 6.82 (m, 2H), 4.93 (s, 2H), 3.45 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.29 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 158.3, 136.9, 129.9, 129.5, 128.7, 128.1, 127.5, 115.5, 70.1, 52.2, 14.7. All data was consistent with that previously reported.[8]



2-(4-((Benzyloxy)methyl)phenyl)propanal (2i)

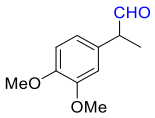
The title compound was prepared from 1-((benzyloxy)methyl)-4-vinylbenzene (96.3 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.2) to give the product as a white solid (65.6 mg, 60%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.69 (d, *J* = 1.4 Hz, 1H), 7.41 (dd, *J* = 2.5, 1.0 Hz, 1H), 7.40 – 7.34 (m, 5H), 7.34 – 7.28 (m, 1H), 7.24 – 7.19 (m, 2H), 4.59 (s, 2H), 4.57 (s, 2H), 3.65 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.46 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.1, 138.3, 137.8, 137.2, 128.6, 128.6, 128.5, 127.9, 127.8, 72.4, 71.8, 52.9, 14.8. All data was consistent with that previously reported.[11]

2-(2-(Benzyloxy)phenyl)propanal (2j)

The title compound was prepared from 1-(benzyloxy)-2-vinylbenzene (90.3 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (47.5 mg, 46%).

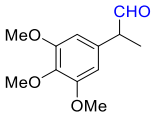
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.73 (d, *J* = 0.6 Hz, 1H), 7.42 – 7.25 (m, 6H), 7.18 – 7.12 (m, 1H), 7.05 – 6.97 (m, 2H), 5.12 (s, 2H), 3.98 (q, *J* = 7.1 Hz, 1H), 1.43 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 202.0, 156.4, 136.8, 129.3, 128.8, 128.7, 128.1, 127.4, 127.4, 121.4, 112.2, 70.3, 47.4, 13.6. All data was consistent with that previously reported.[13]



2-(3,4-Dimethoxyphenyl)propanal (2k)

The title compound was prepared from 1,2-dimethoxy-4-vinylbenzene (70.5 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.3) to give the product as a colorless oil (30.0 mg, 36%).

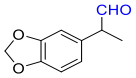
^{1}H NMR (300 MHz, Chloroformkk-*d*) δ 9.65 (d, *J* = 1.5 Hz, 1H), 6.88 (d, *J* = 8.3 Hz, 1H), 6.77 (ddd, *J* = 8.3, 2.1, 0.5 Hz, 1H), 6.68 (d, *J* = 2.1 Hz, 1H), 3.88 (s, 6H), 3.58 (qd, *J* = 7.1, 1.5 Hz, 1H), 1.42 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 149.6, 148.6, 130.1, 120.6, 111.8, 111.4, 56.1, 56.1, 52.7, 14.7. All data was consistent with that previously reported.[9]



2-(3,4,5-Trimethoxyphenyl)propanal (2l)

The title compound was prepared from 1,2,3-trimethoxy-5-vinylbenzene (83.4 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/EA = 10:1, Rf = 0.2) to give the product as a colorless oil (45,3 mg, 47%).

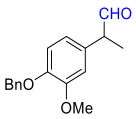
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.66 (d, *J* = 1.4 Hz, 1H), 6.39 (s, 2H), 3.85 (s, 6H), 3.83 (s, 3H), 3.56 (qd, *J* = 7.0, 1.2 Hz, 1H), 1.42 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.9, 153.9, 137.6, 133.4, 105.5, 61.1, 56.4, 53.4, 14.8. All data was consistent with that previously reported.[16]



2-(Benzo[*d*][1,3]dioxol-5-yl)propanal (2m)

The title compound was prepared from 5-vinylbenzo[*d*][1,3]dioxole (63.7 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (57.4 mg, 75%).

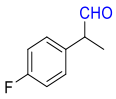
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.63 (d, *J* = 1.5 Hz, 1H), 6.81 (dd, *J* = 7.6, 0.7 Hz, 1H), 6.71 – 6.64 (m, 2H), 5.96 (s, 2H), 3.55 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.40 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.8, 148.3, 147.1, 131.3, 121.6, 108.8, 108.5, 101.2, 52.6, 14.7. All data was consistent with that previously reported.[14]



2-(4-(Benzyloxy)-3-methoxyphenyl)propanal (2n)

The title compound was prepared from 1-(benzyloxy)-2-methoxy-4-vinylbenzene (103.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (37.2 mg, 32%).

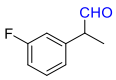
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.64 (d, *J* = 1.5 Hz, 1H), 7.46 – 7.42 (m, 2H), 7.40 – 7.30 (m, 3H), 6.92 – 6.86 (m, 1H), 6.73 – 6.66 (m, 2H), 5.15 (s, 2H), 3.89 (s, 3H), 3.56 (qd, *J* = 7.0, 1.5 Hz, 1H), 1.42 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.1, 150.2, 147.8, 137.2, 130.7, 128.7, 128.0, 127.4, 120.6, 114.5, 112.0, 71.2, 56.2, 52.7, 14.7. **HRMS** (EI): calcd for [M] $C_{17}H_{18}O_3$ 270.1251, found: 270.1251.



2-(4-Fluorophenyl)propanal (2o)

The title compound was prepared from 1-fluoro-4-vinylbenzene (52.5 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.5) to give the product as a colorless oil (53.0 mg, 81%).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.66 (d, *J* = 1.4 Hz, 1H), 7.21 – 7.14 (m, 2H), 7.11 – 7.03 (m, 2H), 3.63 (qd, *J* = 7.1, 1.3 Hz, 1H), 1.44 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.9, 162.4 (d, *J* = 246.0 Hz), 133.5 (d, *J* = 3.2 Hz), 130.0 (d, *J* = 8.1 Hz), 116.1 (d, *J* = 21.5 Hz), 52.3, 14.9. All data was consistent with that previously reported.[8]



2-(3-Fluorophenyl)propanal (2p)

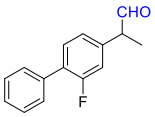
The title compound was prepared from 1-fluoro-3-vinylbenzene (52.5 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (44.5 mg, 68%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.58 (d, *J* = 1.4 Hz, 1H), 7.29 – 7.22 (m, 1H), 6.94 – 6.82 (m, 3H), 3.54 (qd, *J* = 7.4, 1.2 Hz, 1H), 1.35 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.5, 163.3 (d, *J* = 246.6 Hz), 140.3, 130.7 (d, *J* = 8.3 Hz), 124.1 (d, *J* = 3.0 Hz), 115.4 (d, *J* = 21.6 Hz), 114.7 (d, *J* = 21.0 Hz), 52.8, 14.6. All data was consistent with that previously reported.[12]

2-(2-Fluorophenyl)propanal (2q)

The title compound was prepared from 1-fluoro-2-vinylbenzene (52.5 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (43.8 mg, 67%).

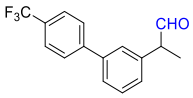
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.73 (dd, *J* = 1.7, 0.8 Hz, 1H), 7.33 – 7.23 (m, 1H), 7.22 – 7.05 (m, 3H), 3.91 (qd, *J* = 7.2, 0.6 Hz, 1H), 1.45 (dd, *J* = 7.2, 0.4 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.3, 161.0 (d, *J* = 246.0 Hz), 129.6 (d, *J* = 4.5 Hz), 129.4 (d, *J* = 8.5 Hz), 125.4 (d, *J* = 15.5 Hz), 124.8 (d, *J* = 3.5 Hz), 115.9 (d, *J* = 22.0 Hz), 46.6, 13.8. All data was consistent with that previously reported.[12]



2-(2-Fluoro-[1,1'-biphenyl]-4-yl)propanal (2r)

The title compound was prepared from 2-fluoro-4-vinyl-1,1'-biphenyl (85.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.5) to give the product as a colorless oil (24.5 mg, 25%).

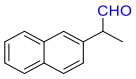
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.72 (d, *J* = 1.4 Hz, 1H), 7.57 – 7.52 (m, 2H), 7.49 – 7.37 (m, 4H), 7.10 – 7.01 (m, 2H), 3.68 (qd, *J* = 7.0, 1.0 Hz, 1H), 1.50 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.4, 160.3 (d, *J* = 248.0 Hz), 139.17 (d, *J* = 7.7 Hz), 135.42, 131.4 (d, *J* = 4.5 Hz), 129.1 (d, *J* = 2.9 Hz), 128.6, 128.0, 124.4 (d, *J* = 3.4 Hz), 116.1 (d, *J* = 23.5 Hz), 52.5, 14.6. All data was consistent with that previously reported.[15]



2-(4'-(Trifluoromethyl)-[1,1'-biphenyl]-3-yl)propanal (2s)

The title compound was prepared from 4'-(trifluoromethyl)-3-vinyl-1,1'-biphenyl (106.7 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (50.2 mg, 42%).

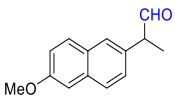
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.32 (d, *J* = 1.4 Hz, 1H), 7.30 – 7.19 (m, 4H), 7.14 – 7.02 (m, 2H), 7.01 – 6.94 (m, 1H), 6.85 – 6.81 (m, 1H), 3.30 (qd, *J* = 7.1, 1.3 Hz, 1H), 1.08 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.9, 144.4, 140.9, 138.7, 129.9, 129.6, 128.2, 127.7, 127.4, 126.7, 125.9 (q, *J* = 3.0 Hz), 124.4 (q, *J* = 271.8 Hz), 53.2, 14.9. **HRMS** (EI): calcd for [M] $C_{16}H_{13}F_3O$ 278.0913, found: 278.0912.



2-(Naphthalen-2-yl)propanal (2t)

The title compound was prepared from 2-vinylnaphthalene (66.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.5) to give the product as a colorless oil (58.6 mg, 74%).

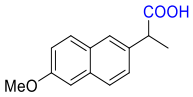
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.77 (d, *J* = 1.4 Hz, 1H), 7.90 – 7.81 (m, 3H), 7.70 – 7.67 (m, 1H), 7.54 – 7.46 (m, 2H), 7.33 (dd, *J* = 8.5, 1.9 Hz, 1H), 3.81 (qd, *J* = 7.0, 1.3 Hz, 1H), 1.55 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 135.3, 133.8, 132.8, 129.0, 127.9, 127.3, 126.6, 126.3, 126.3, 53.3, 14.8. All data was consistent with that previously reported.[8]



2-(6-Methoxynaphthalen-2-yl)propanal (2u)

The title compound was prepared from 2-methoxy-6-vinylnaphthalene (79.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.3) to give the product as a white solid (80.1 mg, 87%).

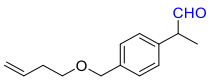
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.75 (d, *J* = 1.5 Hz, 1H), 7.78 – 7.70 (m, 2H), 7.61 (d, *J* = 1.3 Hz, 1H), 7.28 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.20 – 7.13 (m, 2H), 3.93 (s, 3H), 3.77 (qd, *J* = 7.0, 1.3 Hz, 1H), 1.53 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 157.9, 133.9, 132.7, 129.2, 129.1, 127.7, 127.0, 126.7, 119.3, 105.6, 55.3, 53.0, 14.7. All data was consistent with that previously reported.[15]



2-(6-Methoxynaphthalen-2-yl)propanoic acid (2u')

The title compound was prepared from **2u** (80.1 mg, 0.37 mmol), according to known literature procedures[17]. The crude residue was purified by flash chromatography (pentane/EA = 3:1, Rf = 0.3) to give the product as a white solid (76.6 mg, 90%).

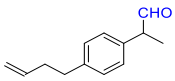
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.74 – 7.66 (m, 3H), 7.42 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.18 – 7.09 (m, 2H), 3.91 (s, 3H), 3.87 (q, *J* = 6.0 Hz, 1H), 1.59 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 180.8, 157.9, 135.0, 134.0, 129.4, 129.0, 127.4, 126.3, 126.3, 119.2, 105.7, 55.4, 45.4, 18.3. All data was consistent with that previously reported.[17]



2-(4-((But-3-en-1-yloxy)methyl)phenyl)propanal (2v)

The title compound was prepared from 1-((but-3-en-1-yloxy)methyl)-4-vinylbenzene (80.8 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.3) to give the product as a colorless oil (54.4 mg, 58%).

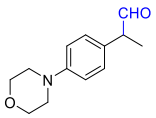
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.67 (d, *J* = 1.4 Hz, 1H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 5.85 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 5.15 – 5.02 (m, 2H), 4.51 (s, 2H), 3.63 (qd, *J* = 7.1, 1.3 Hz, 1H), 3.54 (t, *J* = 6.8 Hz, 2H), 2.39 (qt, *J* = 6.8, 1.4 Hz, 2H), 1.44 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.2, 138.0, 137.1, 135.3, 128.5, 116.6, 72.6, 69.9, 52.9, 34.4, 14.8.



2-(4-(But-3-en-1-yl)phenyl)propanal (2w)

The title compound was prepared from 1-(but-3-en-1-yl)-4-vinylbenzene (68.0 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.7) to give the product as a colorless oil (51.0 mg, 63%).

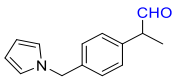
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.67 (d, *J* = 1.5 Hz, 1H), 7.23 – 7.11 (m, 4H), 5.86 (ddt, *J* = 16.8, 10.2, 6.5 Hz, 1H), 5.11 – 4.96 (m, 2H), 3.61 (qd, *J* = 7.0, 1.3 Hz, 1H), 2.71 (dd, *J* = 8.9, 6.7 Hz, 2H), 2.42 – 2.33 (m, 2H), 1.43 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.4, 141.4, 138.1, 135.2, 129.3, 128.4, 115.2, 52.8, 35.5, 35.1, 14.7. **HRMS** (EI): calcd for [M] $C_{13}H_{16}O$ 188.1196, found: 188.1193.



2-(4-Morpholinophenyl)propanal (2x)

The title compound was prepared from 4-(4-vinylphenyl)morpholine (81.3 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 20:1, Rf = 0.2) to give the product as a colorless oil (38.6 mg, 41%).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.64 (d, *J* = 1.5 Hz, 1H), 7.12 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.86 (t, *J* = 4.0 Hz, 4H), 3.56 (qd, *J* = 7.0, 1.4 Hz, 1H), 3.16 (t, *J* = 4.0 Hz, 4H), 1.41 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.4, 150.8, 129.3, 128.8, 116.2, 67.0, 52.3, 49.3, 14.7. **HRMS** (EI): calcd for [M] $C_{13}H_{17}NO_2$ 219.1254, found: 219.1260.

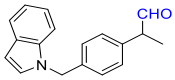


2-(4-((1*H*-Pyrrol-1-yl)methyl)phenyl)propanal (2y)

The title compound was prepared from 1-(4-vinylbenzyl)-1*H*-pyrrole (78.7 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 20:1, Rf = 0.5) to give the product as a colorless oil (42.2 mg, 46%).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.67 (d, *J* = 1.4 Hz, 1H), 7.19 – 7.12 (m, 4H), 6.70 (t, *J* = 2.1 Hz, 2H), 6.21 (t, *J* = 2.1 Hz, 2H), 5.08 (s, 2H), 3.63 (qd, *J* = 7.1, 1.3 Hz, 1H), 1.44 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.0, 137.8, 137.3, 128.8, 127.8, 121.2, 108.8, 53.0, 52.7, 14.7.

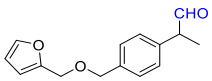
HRMS (EI): calcd for [M] $C_{14}H_{15}NO$ 213.1148, found: 213.1146.



2-(4-((1*H*-Indol-1-yl)methyl)phenyl)propanal (2z)

The title compound was prepared from 1-(4-vinylbenzyl)-1*H*-indole (100.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 20:1, Rf = 0.5) to give the product as a colorless oil (75.8 mg, 67%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.77 (d, *J* = 1.4 Hz, 1H), 7.87 – 7.76 (m, 1H), 7.45 – 7.40 (m, 1H), 7.36 – 7.23 (m, 7H), 6.72 (d, *J* = 3.2 Hz, 1H), 5.45 (s, 2H), 3.72 (qd, *J* = 7.1, 1.4 Hz, 1H), 1.55 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 200.9, 137.2, 137.1, 136.3, 128.8, 128.8, 128.3, 127.5, 121.9, 121.1, 119.7, 109.7, 101.9, 52.7, 49.8, 14.7. **HRMS** (ESI-TOF) m/z: Calcd. for $C_{18}H_{17}NO$ $[M+H]^+$: 264.1388, found: 264.1392.

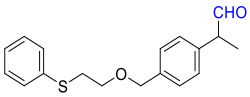


2-(4-((Furan-2-ylmethoxy)methyl)phenyl)propanal (2aa)

The title compound was prepared from 2-(((4-vinylbenzyl)oxy)methyl)furan (92. mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 20:1, Rf = 0.5) to give the product as a colorless oil (49.3 mg, 47%).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.67 (d, *J* = 1.4 Hz, 1H), 7.43 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.1 Hz, 2H), 6.37 – 6.32 (m, 2H), 4.54 (s, 2H), 4.50 (s, 2H), 3.63 (qd, *J* = 7.0, 1.3 Hz, 1H), 1.44 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.1, 151.7, 143.0, 137.4,

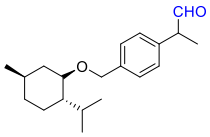
137.3, 128.7, 128.5, 110.4, 109.6, 71.5, 64.1, 52.8, 14.7. **HRMS** (EI): calcd for [M] $C_{15}H_{16}O_3$ 244.1094, found: 244.1092.



2-(4-((2-(Phenylthio)ethoxy)methyl)phenyl)propanal (2bb)

The title compound was prepared from phenyl(2-((4-vinylbenzyl)oxy)ethyl)sulfane (116.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 20:1, Rf = 0.5) to give the product as a colorless oil (67.1 mg, 52%).

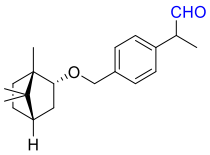
^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.70 (d, *J* = 1.4 Hz, 1H), 7.41 – 7.34 (m, 4H), 7.33 – 7.28 (m, 2H), 7.24 – 7.18 (m, 3H), 4.55 (s, 2H), 3.71 (t, *J* = 6.9 Hz, 2H), 3.66 (qd, *J* = 7.0, 6.5, 0.7 Hz, 1H), 3.18 (t, *J* = 6.9 Hz, 2H), 1.47 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.1, 137.5, 137.3, 136.1, 129.5, 129.1, 128.5, 128.5, 126.3, 72.7, 69.0, 52.8, 33.4, 14.8. **HRMS** (EI): calcd for [M] $C_{18}H_{20}O_2S$ 300.1179, found: 300.1175.



2-(4-((((1S,2S,5R)-2-Isopropyl-5-methylcyclohexyl)oxy)methyl)phenyl)propanal (2cc)

The title compound was prepared from 1-((((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)oxy)methyl)-4-vinylbenzene (117.1 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (54.6 mg, 42%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.66 (d, *J* = 1.4 Hz, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 4.52 (dd, *J* = 79.8, 11.4 Hz, 2H), 3.62 (qd, *J* = 7.0, 1.3 Hz, 1H), 3.18 (td, *J* = 10.6, 4.2 Hz, 1H), 2.34 – 2.16 (m, 2H), 1.64 (ddd, *J* = 9.5, 7.9, 5.6 Hz, 2H), 1.43 (d, *J* = 7.1 Hz, 3H), 1.38 – 1.22 (m, 2H), 1.07 – 0.87 (m, 9H), 0.72 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.1, 138.7, 136.9, 128.7, 128.4, 79.0, 70.1, 52.9, 48.4, 40.4, 34.7, 31.7, 25.7, 23.4, 22.5, 21.1, 16.2, 14.8. **HRMS** (EI): calcd for [M] $C_{20}H_{30}O_2$ 302.2240, found: 302.2239.

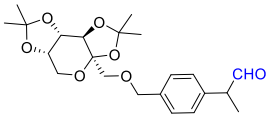


2-(4-((((1S,2R,4S)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)oxy)methyl)phenyl)propanal (2dd)

The title compound was prepared from (1S,2R,4S)-1,7,7-trimethyl-2-((4-vinylbenzyl)oxy)bicyclo[2.2.1]heptane (116.2 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 50:1, Rf = 0.4) to give the product as a colorless oil (58.1 mg, 45%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.68 (d, *J* = 1.4 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 4.59 – 4.41 (m, 2H), 3.70 (ddd, *J* = 9.3, 3.3, 1.7 Hz, 1H), 3.63 (qd, *J* = 7.2, 1.3 Hz, 1H), 2.19 – 2.04 (m, 2H), 1.71 (ddd, *J* = 18.4, 9.5, 4.2 Hz, 2H), 1.44 (d, *J* = 7.1 Hz, 3H), 1.25 (td, *J* = 10.5, 9.6,

3.3 Hz, 2H), 1.10 (dd, *J* = 13.0, 3.3 Hz, 1H), 0.93 – 0.83 (m, 9H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.3, 139.2, 136.6, 128.3, 128.0, 84.7, 71.3, 52.9, 49.5, 48.0, 45.2, 36.3, 28.4, 26.9, 19.9, 19.0, 14.8, 14.2. **HRMS** (EI): calcd for [M] $C_{20}H_{28}O_2$ 300.2084, found: 300.2076.



2-(4-((((3aR,5aS,8aS,8bR)-2,2,7,7-Tetramethyltetrahydro-3aH-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methoxy)methyl)phenyl)propanal (2ee)

The title compound was prepared from (3aR,5aS,8aS,8bR)-2,2,7,7-tetramethyl-3a-(((4-vinylbenzyl)oxy)methyl)tetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran (161.76 mg, 0.43 mmol), according to general procedure B. The crude residue was purified by flash chromatography (pentane/Et_2O = 20:1, Rf = 0.2) to give the product as a colorless oil (71.6 mg, 41%).

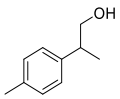
^{1}H NMR (300 MHz, Chloroform-*d*) δ 9.65 (d, *J* = 1.4 Hz, 1H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 4.69 – 4.54 (m, 3H), 4.41 (d, *J* = 2.6 Hz, 1H), 4.21 (dd, *J* = 7.9, 1.1 Hz, 1H), 3.90 (dd, *J* = 13.0, 1.9 Hz, 1H), 3.71 (dd, *J* = 13.0, 0.7 Hz, 1H), 3.65 – 3.55 (m, 3H), 1.53 (s, 3H), 1.41 (dd, *J* = 6.8, 4.3 Hz, 9H), 1.31 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 201.0, 137.6, 137.0, 128.3, 109.0, 108.6, 102.7, 73.3, 71.7, 71.1, 70.3, 70.2, 61.1, 52.8, 26.7, 25.9, 25.5, 24.1, 14.7. **HRMS** (ESI-TOF) m/z: Calcd. for $C_{22}H_{30}O_7$ $[M+Na]^+$: 429.1888, found: 429.1887.

3.2 Characterization Data of Alcohols

2-Phenylpropan-1-ol (3a)

The title compound was prepared from styrene (45 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a colorless oil (41.0 mg, 70%).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.30 (m, 2H), 7.28 – 7.20 (m, 3H), 3.69 – 3.67 (m, 2H), 2.94 (h, *J* = 6.9 Hz, 1H), 1.81 (bs, 1H), 1.28 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.8, 128.7, 127.6, 126.7, 68.7, 42.5, 17.7. All data was consistent with that previously reported.[18] **HR-MS (EI)**: m/z calc. for $[C_9H_{12}O]^+$ ($[M]^+$): 136.0883, measured: 136.0880.

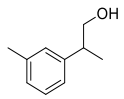


2-(*p*-Tolyl)propan-1-ol (3b)

The title compound was prepared from 1-methyl-4-vinylbenzene (56 µL, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a slightly yellow oil (41.4 mg, 64%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.20 – 7.10 (m, 4H), 3.68 (d, *J* = 7.0 Hz, 2H), 2.92 (h, *J* = 6.9 Hz, 1H), 2.34 (s, 3H), 1.38 (bs, 1H), 1.26 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, CDCl3) δ 140.7, 136.4,

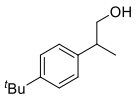
129.5, 127.5, 68.9, 42.2, 21.1, 17.8. All data was consistent with that previously reported.[18] **HR-MS (EI)**: m/z calc. for $[C_{10}H_{14}O]^+$ ($[M]^+$): 150.1039, measured: 150.1037.



2-(*m*-Tolyl)propan-1-ol (3c)

The title compound was prepared from 1-methyl-3-vinylbenzene (56 µL, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a slightly yellow oil (52.8 mg, 82%).

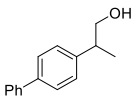
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.26 – 7.20 (m, 1H), 7.12 – 6.98 (m, 3H), 3.70 (d, *J* = 6.9 Hz, 2H), 2.92 (h, *J* = 7.0 Hz, 1H), 2.36 (s, 3H), 1.40 (bs, 1H), 1.27 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, CDCl3) δ 143.7, 138.4, 128.7, 128.4, 127.6, 124.6, 68.8, 42.5, 21.6, 17.8. All data was consistent with that previously reported.[19] **HR-MS (EI)**: m/z calc. for $[C_{10}H_{14}O]^+$ ($[M]^+$): 150.1039, measured: 150.1037.



2-(4-(*tert*-Butyl)phenyl)propan-1-ol (3d)

The title compound was prepared from 1-(*tert*-butyl)-4-vinylbenzene (68.9 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 80:20, Rf = 0.5) to give the product as a slightly yellow oil (67.5 mg, 82%).

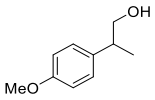
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.33 (m, 2H), 7.22 – 7.14 (m, 2H), 3.70 (d, *J* = 6.8 Hz, 2H), 2.94 (h, *J* = 6.9 Hz, 1H), 1.42 (bs, 1H), 1.33 (s, 9H), 1.28 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.6, 140.6, 127.3, 125.7, 68.9, 42.0, 34.5, 31.5, 31.5, 31.4, 17.7. All data was consistent with that previously reported. [20] **HR-MS (EI)**: m/z calc. for $[C_{13}H_{20}O]^+$ ($[M]^+$): 192.1509, measured: 192.1506.



2-([1,1'-Biphenyl]-4-yl)propan-1-ol (3e)

The title compound was prepared from 4-vinyl-1,1'-biphenyl (83.4 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.3) to give the product as a colorless viscous oil (34.1 mg, 41%).

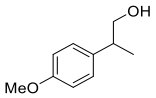
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.63 – 7.54 (m, 4H), 7.49 – 7.40 (m, 2H), 7.39 – 7.29 (m, 3H), 3.76 (d, *J* = 6.8 Hz, 2H), 3.02 (h, *J* = 6.9 Hz, 1H), 1.44 (s, 1H), 1.33 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 142.9, 141.0, 139.8, 128.9, 128.0, 127.5, 127.3, 127.2, 68.8, 42.2, 17.7. All data was consistent with that previously reported.[20] **HR-MS (EI)**: m/z calc. for $[C_{15}H_{16}O]^+$ ($[M]^+$): 212.1196, measured: 212.1192.



2-(4-Methoxyphenyl)propan-1-ol (3f)

The title compound was prepared from 1-methoxy-4-vinylbenzene (57 µL, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography pentane/EtOAc = 4:1, Rf = 0.3) to give the product as a colorless oil (36 mg, 50%).

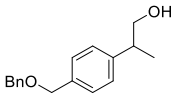
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.22 – 7.11 (m, 2H), 6.93 – 6.82 (m, 2H), 3.80 (s, 3H), 3.66 (dd, *J* = 6.9, 2.1 Hz, 2H), 2.91 (h, *J* = 7.0 Hz, 1H), 1.38 (bs, 1H), 1.25 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 158.5, 135.7, 128.5, 114.2, 68.9, 55.4, 41.7, 17.9. All data was consistent with that previously reported.[18] **HR-MS (EI)**: m/z calc. for $[C_{10}H_{14}O_2]^+$ ($[M]^+$): 166.0988, measured: 166.0987.



2-(4-Phenoxyphenyl)propan-1-ol (3g)

The title compound was prepared from 1-phenoxy-4-vinylbenzene (80 µL, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a colorless oil (58 mg, 59%).

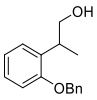
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.38 – 7.29 (m, 2H), 7.25 – 7.17 (m, 2H), 7.15 – 7.06 (m, 1H), 7.05 – 6.94 (m, 4H), 3.70 (d, *J* = 7.0 Hz, 2H), 2.95 (h, *J* = 6.9 Hz, 1H), 1.50 (bs, 1H), 1.28 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 157.4, 156.0, 138.6, 129.8, 128.8, 123.3, 119.1, 118.9, 68.8, 41.9, 17.8. All data was consistent with that previously reported.[19] **HR-MS (EI)**: m/z calc. for $[C_{15}H_{16}O_2]^+$ ($[M]^+$): 228.1145, measured: 228.1143.



2-(4-((Benzyloxy)methyl)phenyl)propan-1-ol (3h)

The title compound was prepared from 1-((benzyloxy)methyl)-4-vinylbenzene (96.5 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a slightly yellow oil (64.3 mg, 58%).

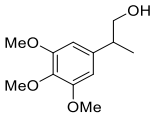
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.41 – 7.20 (m, 9H), 4.57 (s, 2H), 4.54 (s, 2H), 3.75 – 3.66 (m, 2H), 2.96 (h, *J* = 6.9 Hz, 1H), 1.33 (bs, 1H), 1.28 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.3, 138.4, 136.8, 128.5, 128.4, 127.9, 127.8, 127.7, 72.3, 72.0, 68.8, 42.3, 17.7. **HR-MS (EI)**: m/z calc. for $[C_{17}H_{20}O_2]^+$ ($[M]^+$): 256.1458, measured: 256.1458.



2-(2-(Benzyloxy)phenyl)propan-1-ol (3i)

The title compound was prepared from 1-(benzyloxy)-2-vinylbenzene (90.4 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a slightly yellow oil (38.1 mg, 40%).

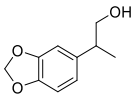
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 – 7.39 (m, 4H), 7.39 – 7.32 (m, 1H), 7.29 – 7.19 (m, 2H), 7.04 – 6.95 (m, 2H), 5.11 (s, 2H), 3.86 – 3.67 (m, 2H), 3.53 (h, *J* = 6.8 Hz, 1H), 1.64 (s, 1H), 1.30 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.6, 137.2, 132.3, 128.7, 128.0, 127.6, 127.5, 127.4, 121.2, 112.1, 70.3, 67.8, 35.5, 16.8. **HR-MS (EI)**: m/z calc. for $[C_{16}H_{18}O_2]^+$ ($[M]^+$): 242.1301, measured: 242.1300.



2-(3,4,5-Trimethoxyphenyl)propan-1-ol (3j)

The title compound was prepared from 1,2,3-trimethoxy-5-vinylbenzene (83.4 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 3:2, Rf = 0.3) to give the product as a colorless oil (40.0 mg, 41%).

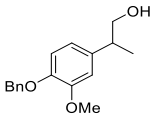
^{1}H NMR (300 MHz, Chloroform-*d*) δ 6.44 (s, 2H), 3.85 (s, 6H), 3.82 (s, 3H), 3.71 – 3.63 (m, 2H), 2.88 (h, *J* = 7.0 Hz, 1H), 1.51 (bs, 1H), 1.25 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.1, 153.4, 138.6, 104.3, 68.8, 61.0, 56.3, 39.4, 18.4. All data was consistent with that previously reported.[20] **HR-MS (EI)**: m/z calc. for $[C_{12}H_{18}O_4]^+$ ($[M]^+$): 226.1200, measured: 226.1199.



2-(Benzo[*d*][1,3]dioxol-5-yl)propan-1-ol (3k)

The title compound was prepared from 5-vinylbenzo[*d*][1,3]dioxole (56 µL, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc =5:1, Rf = 0.3) to give the product as a colorless oil (24 mg, 31%).

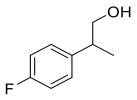
^{1}H NMR (300 MHz, Chloroform-*d*) δ 6.80 – 6.64 (m, 3H), 5.93 (s, 2H), 3.73 – 3.57 (m, 2H), 2.87 (h, *J* = 7.0 Hz, 1H), 1.47 (bs, 1H), 1.23 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 148.0, 146.4, 137.7, 120.7, 108.5, 107.7, 101.0, 68.8, 42.3, 17.9. All data was consistent with that previously reported.[21] **HR-MS (EI)**: m/z calc. for $[C_{10}H_{12}O_3]^+$ ($[M]^+$): 180.0781, measured: 180.0781.



2-(4-(Benzyloxy)-3-methoxyphenyl)propan-1-ol (3l)

The title compound was prepared from 1-(benzyloxy)-2-methoxy-4-vinylbenzene (103.3 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.2) to give the product as a yellow oil (36.0 mg, 31%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.49 – 7.41 (m, 2H), 7.40 – 7.27 (m, 3H), 6.85 (d, *J* = 8.2 Hz, 1H), 6.78 (d, *J* = 2.0 Hz, 1H), 6.72 (dd, *J* = 8.2, 2.1 Hz, 1H), 5.14 (s, 2H), 3.90 (s, 3H), 3.73 – 3.60 (m, 2H), 2.89 (h, *J* = 7.0 Hz, 1H), 1.34 (bs, 1H), 1.25 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.9, 147.1, 137.5, 136.9, 128.7, 127.9, 127.4, 119.4, 114.4, 111.5, 71.3, 68.9, 56.2, 42.2, 17.8. **HR-MS (EI)**: m/z calc. for $[C_{17}H_{20}O_3]^+$ ([M]$^+$): 272.1407, measured: 272.1406.



2-(4-Fluorophenyl)propan-1-ol (3m)

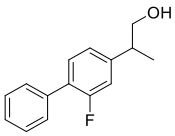
The title compound was prepared from 1-fluoro-4-vinylbenzene (51 µL, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.2) to give the product as a colorless oil (42 mg, 63%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.26 – 7.14 (m, 2H), 7.06 – 6.97 (m, 2H), 3.68 (d, *J* = 7.1 Hz, 2H), 2.94 (h, *J* = 6.9 Hz, 1H), 1.46 (bs, 1H), 1.26 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, Chloroform-*d*) δ 161.8 (d, *J* = 244.4 Hz), 139.5 (d, *J* = 3.0 Hz), 129.0 (d, *J* = 8.0 Hz), 115.5 (d, *J* = 21.2 Hz), 68.8, 41.8, 17.8; **^{19}F NMR** (282 MHz, $CDCl_3$) δ -116.59.[20] **HR-MS (EI)**: m/z calc. for $[C_9H_{11}OF]^+$ ([M]$^+$): 154.0788, measured: 154.0779.

2-(2-Fluorophenyl)propan-1-ol (3n)

The title compound was prepared from 1-fluoro-2-vinylbenzene (52.5 mg, 0.43 mmoL), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.2) to give the product as a colorless oil (40 mg, 60%).

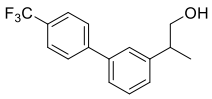
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.30 – 7.16 (m, 2H), 7.16 – 7.11 (m, 1H), 7.10 – 6.99 (m, 1H), 3.87 – 3.67 (m, 2H), 3.32 (h, *J* = 7.0 Hz, 1H), 1.35 (bs, 1H), 1.30 (dd, *J* = 7.1, 0.6 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.2 (d, *J* = 244.9 Hz), 130.5 (d, *J* = 14.4 Hz), 128.5 (d, *J* = 5.0 Hz), 128.1 (d, *J* = 8.3 Hz), 124.3 (d, *J* = 3.3 Hz), 115.6 (d, *J* = 22.9 Hz), 67.5, 35.8, 16.7. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -118.40. All data was consistent with that previously reported.[22] **HR-MS (EI)**: m/z calc. for $[C_9H_{11}OF]^+$ ([M]$^+$): 154.0788, measured: 154.0777.



2-(2-Fluoro-[1,1'-biphenyl]-4-yl)propan-1-ol (3o)

The title compound was prepared from 2-fluoro-4-vinyl-1,1'-biphenyl (85.2 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.3) to give the product as a slightly yellow solid (48.5 mg, 42%).

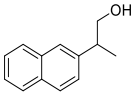
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.57 – 7.50 (m, 2H), 7.48 – 7.32 (m, 4H), 7.13 – 7.08 (m, 1H), 7.08 – 7.01 (m, 1H), 3.81 – 3.71 (m, 2H), 3.00 (h, *J* = 6.9 Hz, 1H), 1.37 (bs, 1H), 1.31 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, Chloroform-*d*) δ 130.9 (d, *J* = 4.1 Hz), 129.1 (d, *J* = 2.8 Hz), 128.6, 127.7, 123.7 (d, *J* = 3.3 Hz), 115.3, 115.0, 68.6, 42.2, 17.6. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -117.90. All data was consistent with that previously reported.[23] **HR-MS (EI)**: m/z calc. for $[C_{15}H_{15}OF]^+$ ($[M]^+$): 230.1101, measured: 230.1101.



2-(4'-(Trifluoromethyl)-[1,1'-biphenyl]-3-yl)propan-1-ol (3p)

The title compound was prepared from 4'-(trifluoromethyl)-3-vinyl-1,1'-biphenyl (106.8 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a colorless viscous oil (97.6 mg, 81%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.69 (s, 4H), 7.50 – 7.41 (m, 3H), 7.32 – 7.27 (m, 1H), 3.77 (d, *J* = 6.8 Hz, 2H), 3.05 (h, *J* = 6.9 Hz, 1H), 1.42 (s, 1H), 1.34 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 144.9, 144.8, 140.3, 130.2, 129.53 (q, *J* = 32.3 Hz), 129.4, 127.6, 127.4, 126.7, 125.8 (q, *J* = 3.6 Hz), 124.5 (q, *J* = 271.7 Hz), 68.8, 42.7, 17.8; **^{19}F NMR** (282 MHz, CDCl3) δ -62.39. **HR-MS (EI)**: m/z calc. for $[C_{16}H_{15}OF_3]^+$ ($[M]^+$): 280.1070, measured: 280.1072.

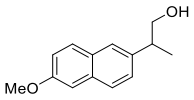


2-(2-Fluoro-[1,1'-biphenyl]-4-yl)propan-1-ol (3q)

The title compound was prepared from 2-vinylnaphthalene (66.3 mg, 0.43 mmol), CuCl (3.4 mg, 8 mol%), dppp (21.3 mg, 12 mol%), and NaO*t*Bu (93.0 mg, 2.25 equiv) in toluene (2.5 mL, 0.17 M). following General Procedure A. It was isolated as a slightly yellow solid (52.8 mg, 66%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.85 – 7.80 (m, 3H), 7.71 – 7.68 (m, 1H), 7.51 – 7.45 (m, 2H), 7.39 (dd, *J* = 8.4, 1.8 Hz, 1H), 3.87 – 3.76 (m, 2H), 3.14 (h, *J* = 6.8 Hz, 1H), 1.38 (d, *J* = 7.1 Hz, 4H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 141.2, 133.7, 132.6, 128.5, 127.8, 126.2, 126.2, 125.9, 125.7, 68.7, 42.7, 17.7.

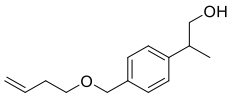
All data was consistent with that previously reported.[23] **HR-MS (EI)**: m/z calc. for $[C_{13}H_{14}O]^+$ ($[M]^+$): 186.1039, measured: 186.1040.



2-(6-Methoxynaphthalen-2-yl)propan-1-ol (3r)

The title compound was prepared from 2-methoxy-6-vinylnaphthalene (79.2 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.3) to give the product as a white solid (69.8 mg, 75%).

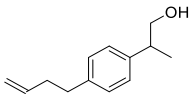
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.71 (dd, *J* = 8.6, 4.6 Hz, 2H), 7.64 – 7.60 (m, 1H), 7.35 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.19 – 7.11 (m, 2H), 3.92 (s, 3H), 3.77 (d, *J* = 6.8 Hz, 2H), 3.09 (h, *J* = 6.9 Hz, 1H), 1.44 (s, 1H), 1.36 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 157.6, 138.8, 133.7, 129.2, 129.2, 127.3, 126.4, 126.0, 119.0, 105.7, 68.8, 55.4, 42.5, 17.8. All data was consistent with that previously reported.[23] **HR-MS (EI)**: m/z calc. for $[C_{14}H_{16}O_2]^+$ ($[M]^+$): 216.1145, measured: 216.1142.



2-(4-((Benzyloxy)methyl)phenyl)propan-1-ol (3s)

The title compound was prepared from 1-((but-3-en-1-yloxy)methyl)-4-vinylbenzene (81.0 mg, 0.43 mmol), , according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.3) to give the product as a slightly yellow oil (46.1 mg, 49%).

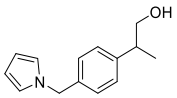
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.36 – 7.28 (m, 2H), 7.25 – 7.18 (m, 2H), 5.93 – 5.77 (m, 1H), 5.15 – 5.11 (m, 1H), 5.11 – 5.00 (m, 1H), 4.50 (s, 2H), 3.69 (d, *J* = 6.9 Hz, 2H), 3.54 (t, *J* = 6.8 Hz, 2H), 2.95 (h, *J* = 7.0 Hz, 1H), 2.38 (qt, *J* = 6.7, 1.4 Hz, 2H), 1.34 (bs, 1H), 1.27 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.2, 137.0, 135.4, 128.2, 127.7, 116.5, 72.8, 69.8, 68.8, 42.3, 34.4, 17.7. **HR-MS (EI)**: m/z calc. for $[C_{14}H_{19}O_2]^+$ ($[M]^+$): 219.1380, measured: 219.1381.



2-(4-(But-3-en-1-yl)phenyl)propan-1-ol (3t)

The title compound was prepared from 1-(but-3-en-1-yl)-4-vinylbenzene (68.0 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.4) to give the product as a yellow oil (49.0 mg, 60%).

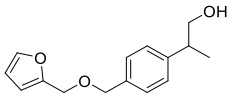
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.16 (s, 4H), 5.98 – 5.77 (m, 1H), 5.12 – 4.95 (m, 2H), 3.69 (d, *J* = 6.8 Hz, 2H), 2.93 (h, *J* = 6.9 Hz, 1H), 2.74 – 2.65 (m, 2H), 2.45 – 2.29 (m, 2H), 1.35 (s, 1H), 1.27 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 141.1, 140.4, 138.3, 128.8, 127.5, 115.0, 68.9, 42.2, 35.6, 35.1, 17.7. **HR-MS (EI)**: m/z calc. for $[C_{13}H_{18}O]^+$ ($[M]^+$): 190.1352, measured: 190.1347.



2-(4-((1*H*-Pyrrol-1-yl)methyl)phenyl)propan-1-ol (3u)

The title compound was prepared from 1-(4-vinylbenzyl)-1*H*-pyrrole (78.8 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a slightly yellow oil (43.7 mg, 47%).

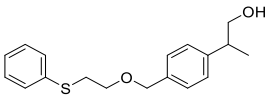
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.24 – 7.17 (m, 2H), 7.13 – 7.04 (m, 2H), 6.70 (t, *J* = 2.2 Hz, 2H), 6.25 – 6.15 (m, 2H), 5.06 (s, 2H), 3.69 (d, *J* = 6.4 Hz, 2H), 2.94 (h, *J* = 6.9 Hz, 1H), 1.37 (s, 1H), 1.26 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.3, 136.7, 127.9, 127.4, 121.2, 108.6, 68.7, 53.2, 42.2, 17.7. **HR-MS (EI)**: m/z calc. for $[C_{14}H_{17}ON]^+$ ($[M]^+$): 215.1305, measured: 215.1302.



2-(4-((Furan-2-ylmethoxy)methyl)phenyl)propan-1-ol (3v)

The title compound was prepared from 2-(((4-vinylbenzyl)oxy)methyl)furan (92.1 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 3:1, Rf = 0.3) to give the product as a colorless oil (49.0 mg, 46%).

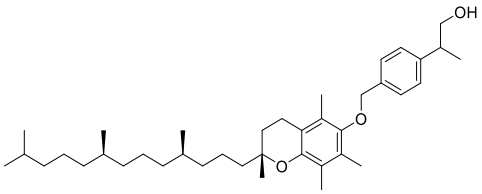
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.42 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.25 – 7.20 (m, 2H), 6.37 – 6.32 (m, 2H), 4.51 (d, *J* = 9.7 Hz, 4H), 3.69 (d, *J* = 6.8 Hz, 2H), 2.95 (h, *J* = 6.9 Hz, 1H), 1.40 (s, 1H), 1.27 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 151.9, 143.4, 142.9, 136.4, 128.5, 127.7, 110.4, 109.5, 71.8, 68.8, 64.0, 42.3, 17.7. **HR-MS (EI)**: m/z calc. for $[C_{15}H_{18}O_3]^+$ ($[M]^+$): 246.1251, measured: 246.1247.



2-(4-((2-(Phenylthio)ethoxy)methyl)phenyl)propan-1-ol (3w)

The title compound was prepared from phenyl(2-((4-vinylbenzyl)oxy)-ethyl)sulfane (116.3 mg, 0.43 mmoL), , according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 4:1, Rf = 0.3) to give the product as a yellow oil (89.4 mg, 81%).

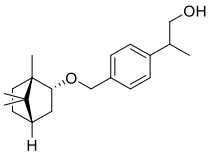
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.34 – 7.03 (m, 9H), 4.42 (s, 2H), 3.66 – 3.52 (m, 4H), 3.07 (t, *J* = 6.9 Hz, 2H), 2.87 (h, *J* = 6.9 Hz, 1H), 1.55 (s, 1H), 1.19 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.4, 136.5, 136.1, 129.5, 129.0, 128.3, 127.7, 126.3, 73.0, 68.9, 68.8, 42.3, 33.4, 17.7. **HR-MS (EI)**: m/z calc. for $[C_{18}H_{22}O_2S]^+$ ($[M]^+$): 302.1335, measured: 302.1337.



2-(4-((((*R*)-2,5,7,8-Tetramethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)oxy)methyl)phenyl)pro-pan-1-ol (3x)

The title compound was prepared from (R)-2,5,7,8-tetramethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)-6-((4-vinylbenzyl)oxy)-chromane (235.2 mg, 0.43 mmol), according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a colorless viscous oil (109 mg, 44%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 4.68 (s, 2H), 3.73 (d, *J* = 6.8 Hz, 2H), 2.99 (h, *J* = 7.0 Hz, 1H), 2.60 (t, *J* = 6.7 Hz, 2H), 2.23 (s, 3H), 2.18 (s, 3H), 2.11 (s, 3H), 1.90 – 1.70 (m, 2H), 1.59 – 1.04 (m, 28H), 0.94 – 0.78 (m, 12H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 148.2, 148.1, 143.4, 136.6, 128.3, 128.0, 127.8, 126.1, 123.1, 117.7, 75.0, 74.6, 68.9, 42.4, 40.2, 40.2, 39.5, 37.7, 37.6, 37.5, 37.4, 32.9, 32.9, 32.8, 32.8, 31.4, 31.4, 28.1, 24.9, 24.6, 24.0, 22.9, 22.8, 21.2, 20.8, 19.9, 19.8, 17.8, 13.0, 12.1, 12.0. **HR-MS (ESI)**: m/z calc. for $[C_{39}H_{62}NaO_3]^+$ ($[M+Na]^+$): 601.4591, measured: 601.4589.

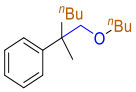


2-(4-((((1S,2R,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)methyl)phenyl)propan-1-ol (3y)

The title compound was prepared from (1S,2R,4S)-1,7,7-trimethyl-2-((4-vinylbenzyl)oxy)bicyclo[2.2.1]heptane (116.3 mg, 0.43 mmol, according to general procedure A. The crude residue was purified by flash chromatography (pentane/EtOAc = 5:1, Rf = 0.3) to give the product as a slightly yellow oil (55.1 mg, 42%).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.09 (m, 4H), 4.68 – 4.28 (m, 2H), 3.83 – 3.52 (m, 3H), 2.95 (h, *J* = 6.8 Hz, 1H), 2.24 – 1.95 (m, 2H), 1.84 – 1.56 (m, 2H), 1.36 (bs, 1H), 1.31 – 1.24 (m, 5H), 1.10 (dd, *J* = 13.0, 3.3 Hz, 1H), 0.91 (s, 3H), 0.86 (s, 3H), 0.84 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 142.6, 138.1, 127.7, 127.5, 84.6, 71.5, 68.9, 49.5, 48.0, 45.2, 42.3, 36.3, 28.4, 26.9, 19.9, 19.0, 17.8, 14.2. **HR-MS (EI)**: m/z calc. for $[C_{20}H_{30}O_2]^+$ ($[M]^+$): 302.2240, measured: 302.2240.

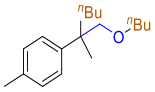
3.3 Characterization Data of ethers and formates



(1-Butoxy-2-methylhexan-2-yl)benzene (4a)

The title compound was prepared from styrene (23 µL, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a colorless oil (21 mg, 42%).

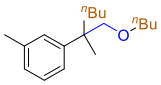
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 4H), 7.21 – 7.17 (m, 1H), 3.44 (s, 2H), 3.37 (td, *J* = 6.5, 1.3 Hz, 2H), 1.76 – 1.51 (m, 4H), 1.34 (s, 3H), 1.32 – 1.19 (m, 4H), 1.14 – 0.99 (m, 2H), 0.92 – 0.81 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.6, 127.9, 126.5, 125.6, 79.8, 71.3, 42.3, 38.8, 31.7, 26.2, 23.4, 22.9, 19.3, 14.0, 13.9.



1-(1-Butoxy-2-methylhexan-2-yl)-4-methylbenzene (4b)

The title compound was prepared from 1-methyl-4-vinylbenzene (23.6 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a colorless oil (21 mg, 41%).

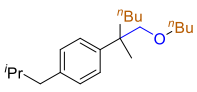
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 – 7.19 (m, 2H), 7.13 – 7.08 (m, 2H), 3.41 – 3.34 (m, 4H), 2.32 (s, 3H), 1.69 – 1.51 (m, 4H), 1.34 – 1.24 (m, 7H), 1.10 – 0.95 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.82 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.6, 135.0, 128.7, 126.4, 79.9, 71.3, 41.9, 38.8, 31.7, 26.3, 23.5, 22.9, 20.9, 19.4, 14.1, 13.9.



1-(1-Butoxy-2-methylhexan-2-yl)-3-methylbenzene (4c)

The title compound was prepared from 1-methyl-3-vinylbenzene (23.6 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a colorless oil (23 mg, 44%).

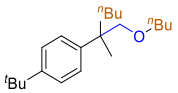
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 – 7.11 (m, 3H), 7.00 (dtd, *J* = 7.2, 1.6, 0.8 Hz, 1H), 3.42 (s, 2H), 3.37 (td, *J* = 6.5, 0.9 Hz, 2H), 2.36 – 2.33 (m, 3H), 1.82 – 1.61 (m, 2H), 1.55 – 1.49 (m, 2H), 1.36 – 1.23 (m, 7H), 1.14 – 0.97 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.83 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.5, 137.2, 127.7, 127.3, 126.3, 123.5, 79.7, 71.3, 42.1, 38.8, 31.7, 26.2, 23.4, 22.9, 21.7, 19.3, 14.0, 13.9.



1-(1-Butoxy-2-methylhexan-2-yl)-4-isobutylbenzene (4d)

The title compound was prepared from 1-isobutyl-4-vinylbenzene (32 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a colorless oil (21 mg, 34%).

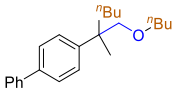
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.4 Hz, 2H), 7.06 (d, *J* = 8.5 Hz, 2H), 3.41 (d, *J* = 0.7 Hz, 2H), 3.36 (td, *J* = 6.5, 2.1 Hz, 2H), 2.44 (d, *J* = 7.2 Hz, 2H), 1.89 – 1.81 (m, 1H), 1.73 – 1.60 (m, 2H), 1.55 – 1.41 (m, 4H), 1.34 – 1.25 (m, 7H), 0.92 – 0.83 (m, 12H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 143.7, 138.7, 128.6, 126.1, 79.9, 71.3, 45.0, 41.9, 38.8, 31.6, 30.2, 26.2, 23.4, 22.8, 22.4, 19.3, 14.1, 13.9.



1-(1-Butoxy-2-methylhexan-2-yl)-4-(*tert*-butyl)benzene (4e)

The title compound was prepared from 1-(*tert*-butyl)-4-vinylbenzene (32 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a colorless oil (23 mg, 37%).

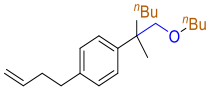
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 4H), 3.44 (d, *J* = 1.6 Hz, 2H), 3.39 (td, *J* = 6.5, 1.7 Hz, 2H), 1.76 – 1.63 (m, 2H), 1.59 – 1.46 (m, 4H), 1.33 (s, 9H), 1.33 (s, 3H), 1.31 – 1.23 (m, 4H), 0.93 – 0.85 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 148.1, 143.5, 126.0, 124.7, 79.7, 71.3, 41.8, 38.7, 34.2, 31.7, 31.4, 26.3, 23.5, 22.9, 19.3, 14.1, 13.9.



4-(1-Butoxy-2-methylhexan-2-yl)-1,1'-biphenyl (4f)

The title compound was prepared from 4-vinyl-1,1'-biphenyl (36 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a yellow oil (28 mg, 43%).

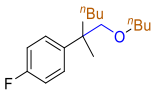
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 – 7.52 (m, 4H), 7.45 – 7.39 (m, 4H), 7.34 – 7.30 (m, 1H), 3.47 (d, *J* = 1.8 Hz, 2H), 3.39 (td, *J* = 6.5, 0.9 Hz, 2H), 1.62 – 1.45 (m, 6H), 1.36 – 1.27 (m, 7H), 0.91 – 0.82 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 145.8, 141.1, 138.3, 138.1, 128.7, 127.0, 127.0, 126.6, 79.7, 71.4, 42.2, 38.9, 31.7, 26.3, 23.5, 23.0, 19.4, 14.1, 13.9.



1-(But-3-en-1-yl)-4-(1-butoxy-2-methylhexan-2-yl)benzene (4g)

The title compound was prepared from 1-(but-3-en-1-yl)-4-vinylbenzene (32 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a yellow oil (20 mg, 33%).

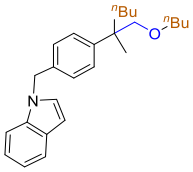
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (d, *J* = 9.4 Hz, 2H), 7.04 (d, *J* = 8.5 Hz, 2H), 5.80 (ddt, *J* = 16.8, 10.2, 6.5 Hz, 1H), 5.01 – 4.87 (m, 2H), 3.34 (s, 2H), 3.29 (td, *J* = 6.5, 1.4 Hz, 2H), 2.65 – 2.56 (m, 2H), 2.34 – 2.25 (m, 2H), 1.67 – 1.54 (m, 2H), 1.46 – 1.37 (m, 2H), 1.26 – 1.18 (m, 7H), 1.01 – 0.88 (m, 2H), 0.84 – 0.73 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 144.0, 138.9, 138.4, 127.9, 126.4, 114.7, 79.8, 71.3, 41.9, 38.8, 35.4, 34.8, 31.6, 26.2, 23.4, 22.9, 19.3, 14.0, 13.9.



1-(1-Butoxy-2-methylhexan-2-yl)-4-fluorobenzene (4h)

The title compound was prepared from 1-fluoro-4-vinylbenzene (24 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as yellow oil (30 mg, 57%).

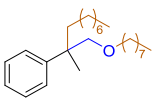
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 – 7.19 (m, 2H), 6.90 (t, *J* = 8.9 Hz, 2H), 3.36 – 3.25 (m, 4H), 1.63 – 1.42 (m, 4H), 1.26 – 1.13 (m, 7H), 1.05 – 0.86 (m, 2H), 0.78 (dt, *J* = 15.6, 7.4 Hz, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.0 (d, *J* = 249.1 Hz), 142.2 (d, *J* = 3.2 Hz), 128.0 (d, *J* = 7.6 Hz), 114.5 (d, *J* = 20.7 Hz),79.8, 71.3, 41.9, 39.0, 31.6, 26.1, 23.4, 22.9, 19.3, 14.0, 13.9.



1-(4-(1-Butoxy-2-methylhexan-2-yl)benzyl)-1*H*-indole (4i)

The title compound was prepared from 1-(4-vinylbenzyl)-1*H*-indole (46.6 mg, 0.2 mmol) and 1-bromobutane (76 µL, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a yellow oil (23 mg, 31%).

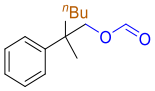
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 – 7.64 (m, 1H), 7.37 – 7.31 (m, 1H), 7.29 (d, *J* = 2.1 Hz, 1H), 7.27 (d, *J* = 2.0 Hz, 1H), 7.22 – 7.17 (m, 1H), 7.17 – 7.12 (m, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.57 (dd, *J* = 3.2, 0.8 Hz, 1H), 5.32 (s, 2H), 3.40 (d, *J* = 2.0 Hz, 2H), 3.36 (td, *J* = 6.5, 1.6 Hz, 2H), 1.76 – 1.60 (m, 2H), 1.57 – 1.47 (m, 4H), 1.33 (d, *J* = 11.3 Hz, 2H), 1.30 (s, 3H), 1.25 – 1.16 (m, 2H), 0.92 – 0.78 (m, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.1, 136.4, 134.6, 128.3, 127.5, 126.9, 126.5, 121.6, 120.9, 119.4, 109.7, 101.5, 79.6, 71.3, 49.8, 42.1, 38.8, 31.6, 26.2, 23.4, 22.9, 19.3, 14.1, 13.9.



(2-Methyl-1-(octyloxy)decan-2-yl)benzene (4j)

The title compound was prepared from styrene (23 µL, 0.2 mmol) and 1-bromooctane (134 mg, 3.5 equiv.), according to general procedure C. The crude residue was purified by flash chromatography (pentane/DCM = 20:1, Rf = 0.25) to give the product as a yellow oil (25 mg, 35%).

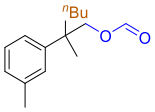
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 4H), 7.22 – 7.14 (m, 1H), 3.44 (s, 2H), 3.36 (td, *J* = 6.5, 1.2 Hz, 2H), 1.82 – 1.61 (m, 2H), 1.52 (ddd, *J* = 21.8, 15.7, 5.7 Hz, 4H), 1.33 (s, 3H), 1.28 (dd, *J* = 8.5, 2.1 Hz, 12H), 1.21 (s, 8H), 0.88 (q, *J* = 6.8 Hz, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 146.6, 127.9, 126.5, 125.5, 79.7, 71.6, 42.3, 39.0, 31.9, 31.8, 30.4, 29.5, 29.5, 29.4, 29.3, 26.2, 24.0, 22.9, 22.7, 22.6, 14.1.



2-Methyl-2-phenylhexyl formate (5a)

The title compound was prepared from styrene (23 µL, 0.2 mmol) and 1-iodobutane (27 µL, 1.2 equiv.), according to general procedure D. The crude residue was purified by flash chromatography (pentane/EtOAc = 100:1, Rf = 0.25) to give the product as a colorless oil (15 mg, 33%).

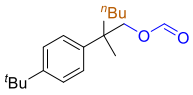
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (t, *J* = 0.9 Hz, 1H), 7.36 – 7.30 (m, 4H), 7.25 – 7.21 (m, 1H), 4.37 – 4.19 (m, 2H), 1.84 – 1.60 (m, 2H), 1.39 (s, 3H), 1.29 – 1.22 (m, 2H), 1.13 – 0.97 (m, 2H), 0.83 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.1, 144.4, 128.3, 126.3, 126.2, 71.8, 41.2, 38.7, 25.9, 23.3, 22.3, 13.9.



2-Methyl-2-(*m*-tolyl)hexyl formate (5b)

The title compound was prepared from 1-methyl-3-vinylbenzene (23.6 mg, 0.2 mmol) and 1-iodobutane (27 µL, 1.2 equiv.), according to general procedure D. The crude residue was purified by flash chromatography (pentane/EtOAc = 100:1, Rf = 0.25) to give the product as a colorless oil (14 mg, 30%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (t, *J* = 0.9 Hz, 1H), 7.23 – 7.20 (m, 1H), 7.12 (dd, *J* = 1.8, 1.1 Hz, 1H), 7.04 – 7.02 (m, 1H), 6.94 (d, *J* = 7.5 Hz, 1H), 4.33 – 4.17 (m, 2H), 2.36 (s, 3H), 1.37 (s, 3H), 1.26 (s, 2H), 1.10 – 0.98 (m, 4H), 0.83 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.2, 144.4, 137.7, 128.1, 127.1, 127.0, 123.4, 71.9, 41.1, 38.8, 26.0, 23.3, 22.4, 21.7, 14.0.

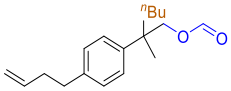


2-(4-(*tert*-Butyl)phenyl)-2-methylhexyl formate (5c)

The title compound was prepared from 1-(*tert*-butyl)-4-vinylbenzene (32 mg, 0.2 mmol) and 1-iodobutane (27 µL, 1.2 equiv.), according to general procedure D. The crude residue was purified by

flash chromatography (pentane/EtOAc = 100:1, Rf = 0.25) to give the product as a colorless oil (17 mg, 37%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 (t, *J* = 0.8 Hz, 1H), 7.33 (d, *J* = 8.7 Hz, 2H), 7.23 (d, *J* = 8.8 Hz, 2H), 4.24 (qd, *J* = 10.9, 0.8 Hz, 2H), 1.73 – 1.59 (m, 2H), 1.36 (s, 3H), 1.31 (s, 9H), 1.30 (d, *J* = 1.4 Hz, 4H), 0.83 (d, *J* = 7.4 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.2, 148.8, 141.3, 125.9, 125.1, 71.8, 40.7, 38.7, 31.3, 26.9, 26.0, 23.3, 22.5, 14.0.



2-(4-(But-3-en-1-yl)phenyl)-2-methylhexyl formate (5d)

The title compound was prepared from 1-(but-3-en-1-yl)-4-vinylbenzene (32 mg, 0.2 mmol) and 1-iodobutane (27 µL, 1.2 equiv.), according to general procedure D. The crude residue was purified by flash chromatography (pentane/EtOAc = 100:1, Rf = 0.25) to give the product as a yellow oil (16 mg, 29%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (t, *J* = 0.9 Hz, 1H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.6 Hz, 2H), 5.92 – 5.82 (m, 1H), 5.08 – 5.02 (m, 1H), 5.00 – 4.96 (m, 1H), 4.24 (qd, *J* = 10.9, 0.8 Hz, 2H), 2.72 – 2.66 (m, 2H), 2.40 – 2.34 (m, 2H), 1.56 (s, 2H), 1.36 (s, 3H), 1.24 (dd, *J* = 15.9, 8.1 Hz, 4H), 0.83 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 161.2, 139.7, 138.2, 128.3, 126.2, 114.9, 71.9, 40.9, 38.7, 35.3, 34.8, 26.0, 23.3, 22.4, 14.0.

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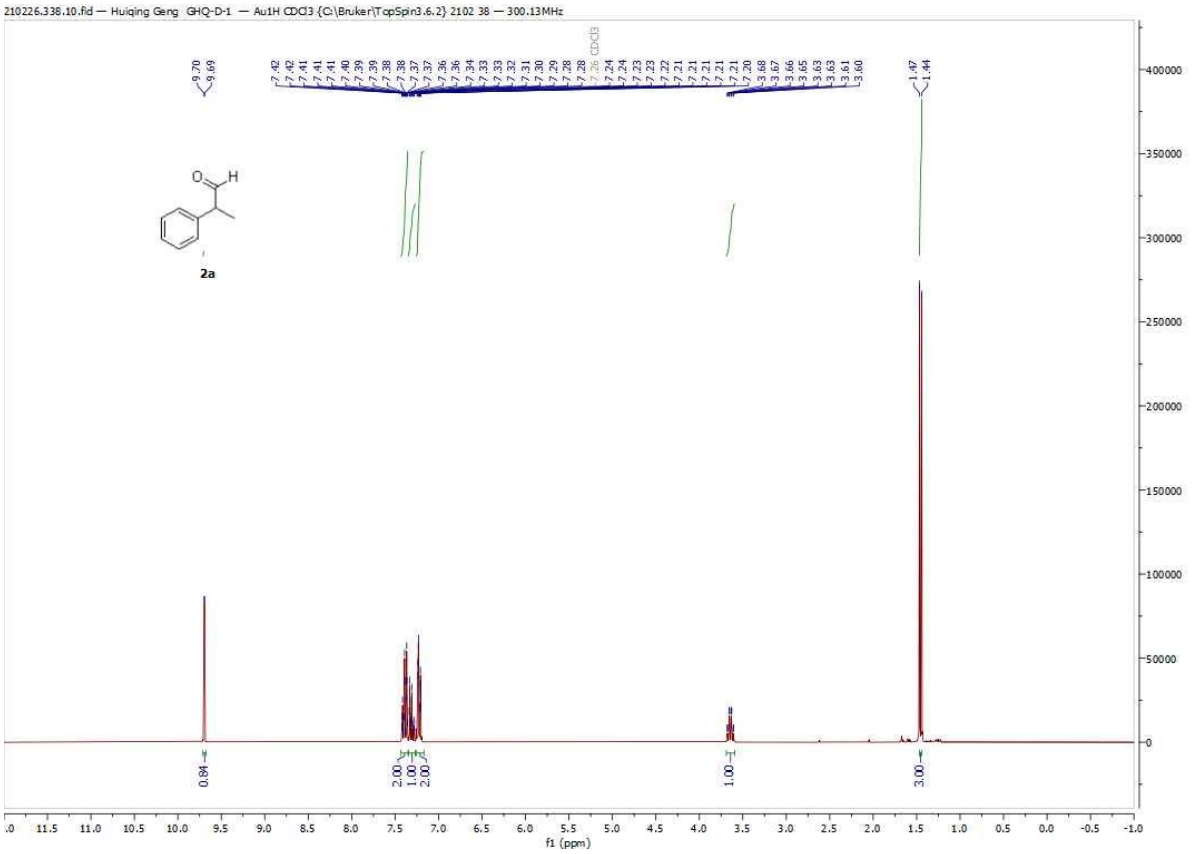
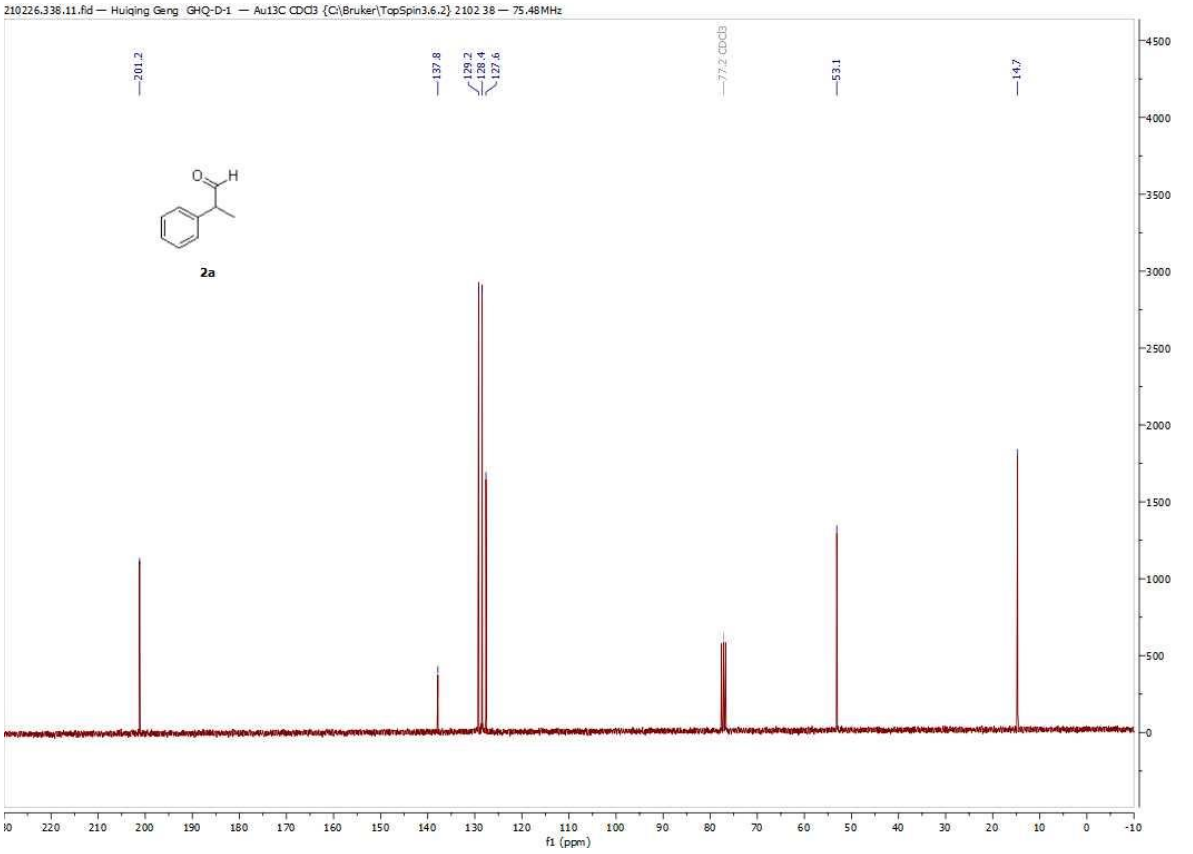
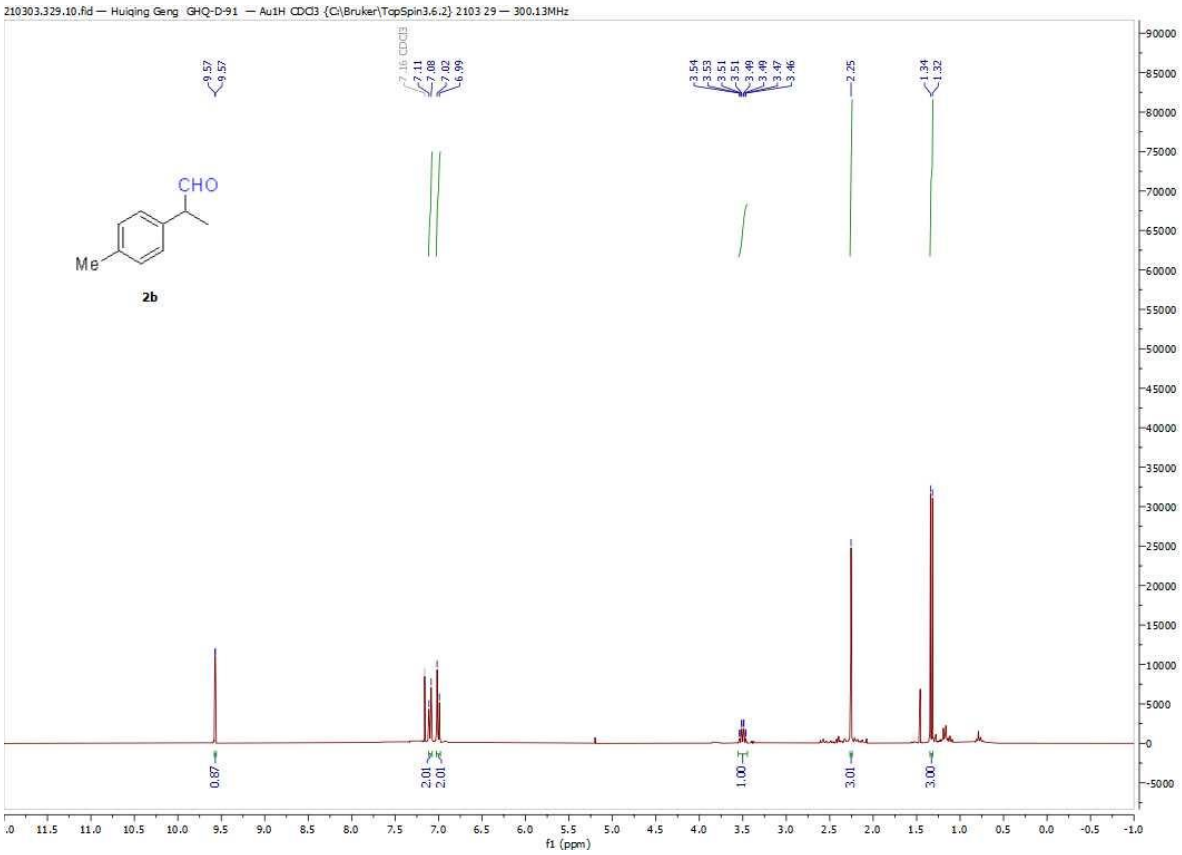
5. NMR Spectra



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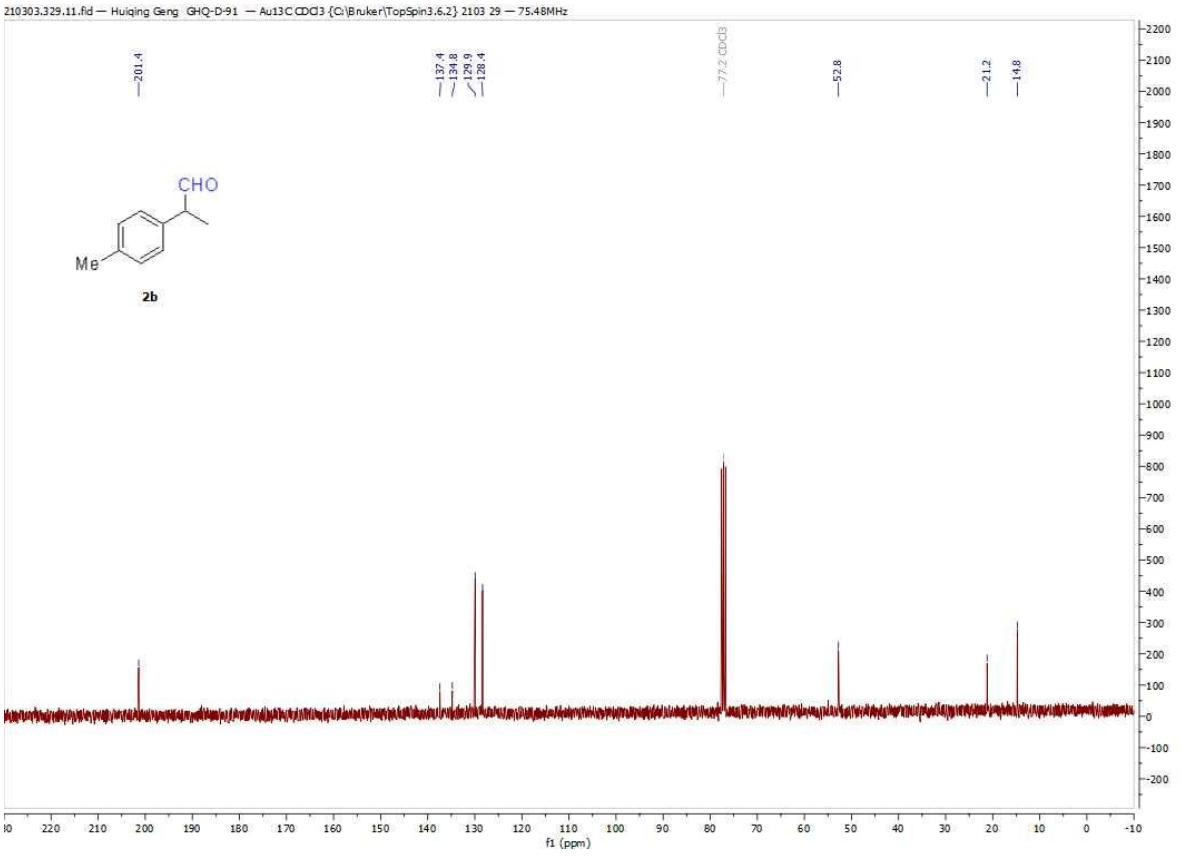
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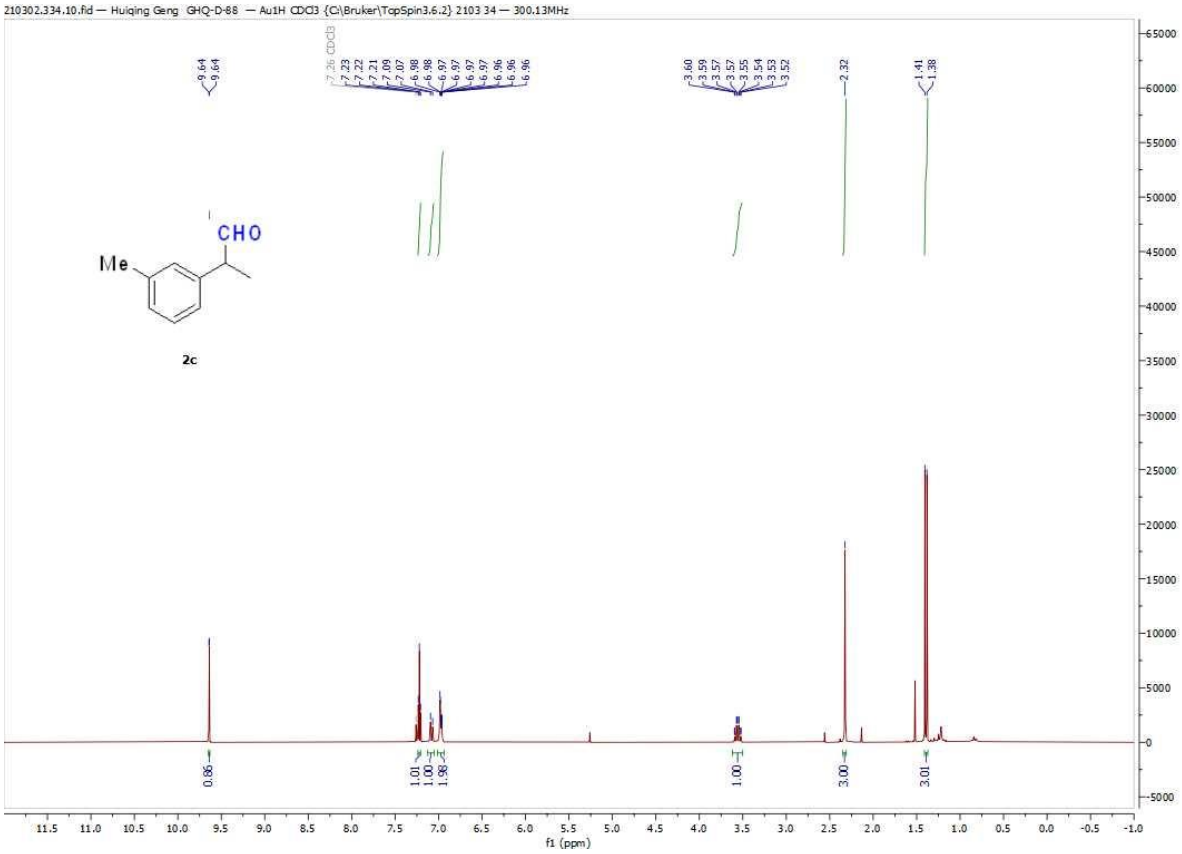
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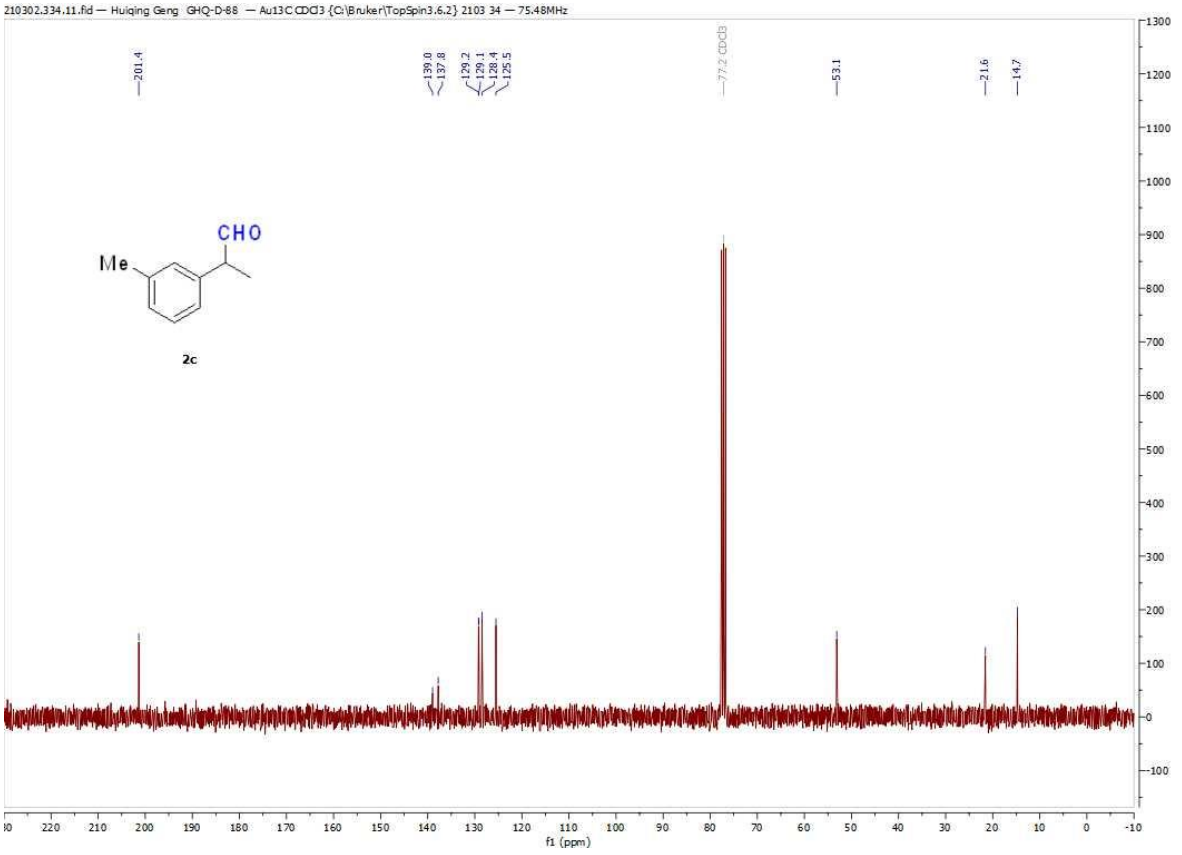
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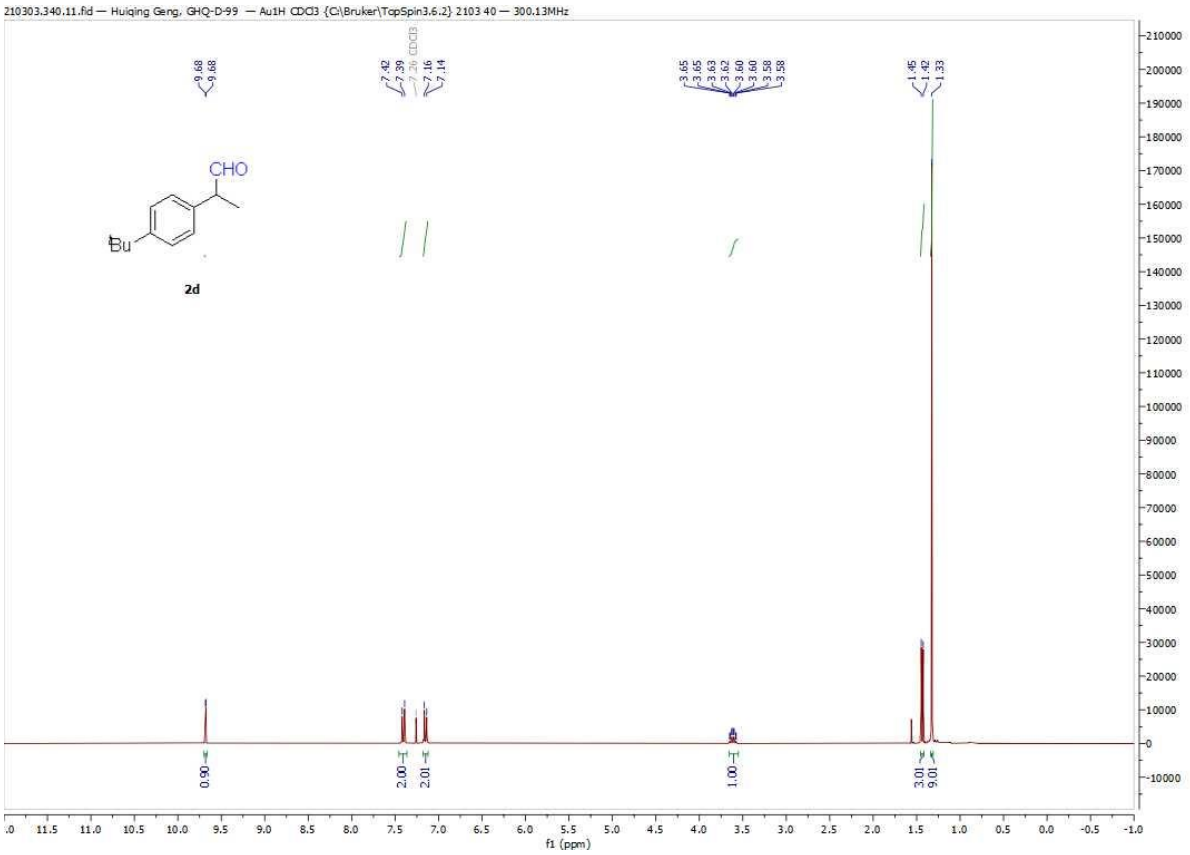
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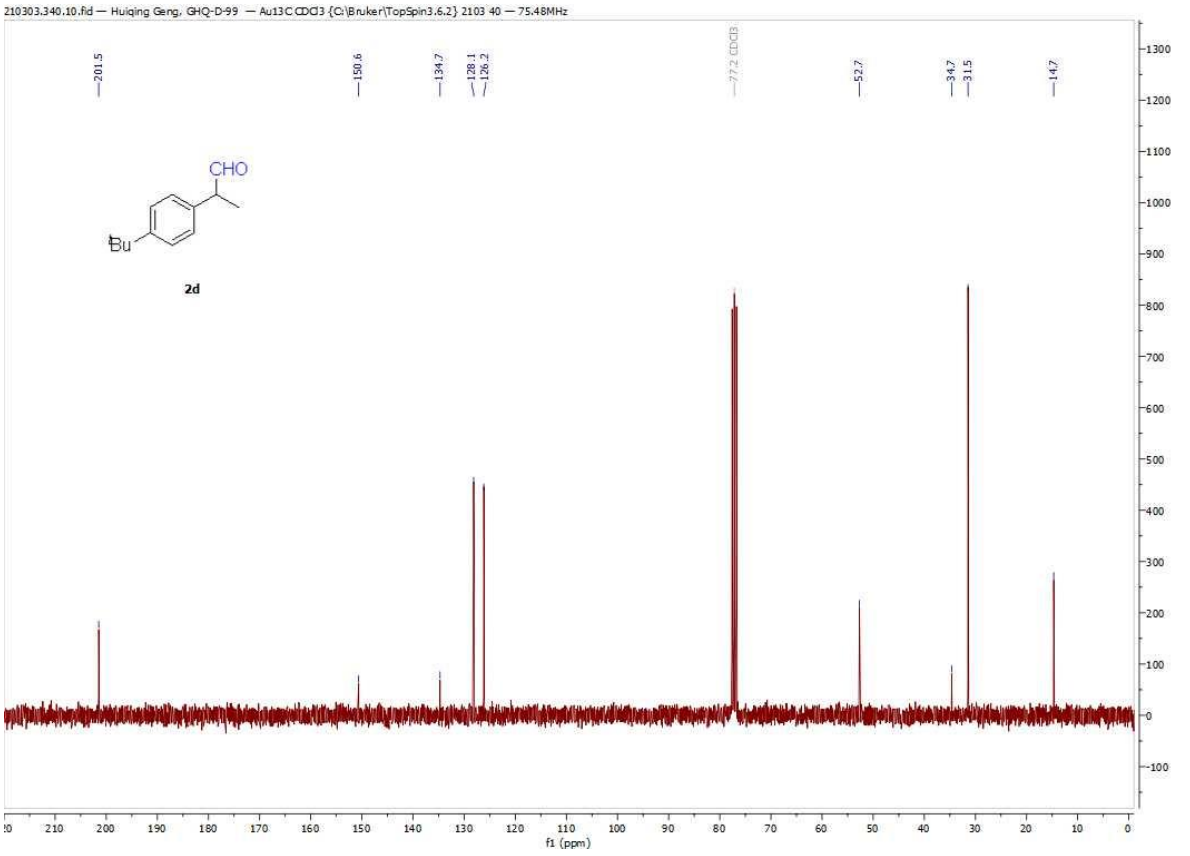
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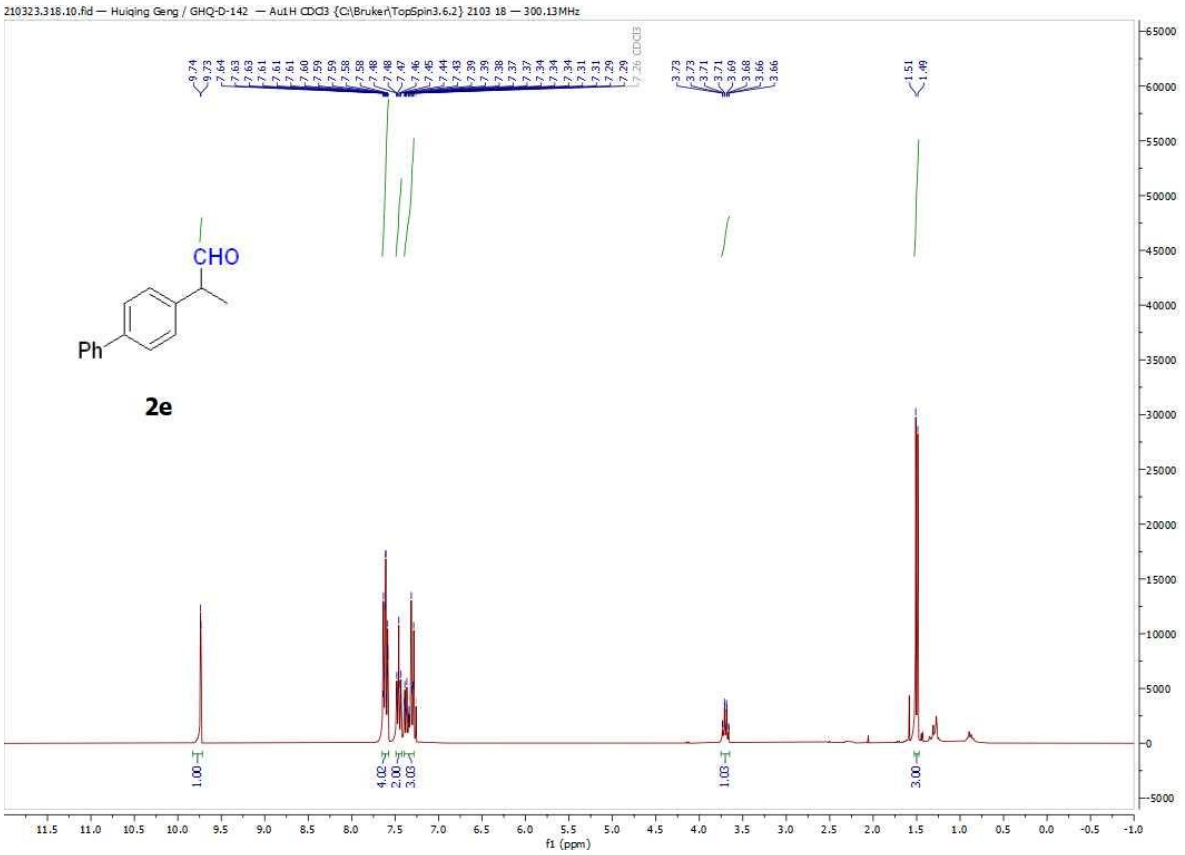
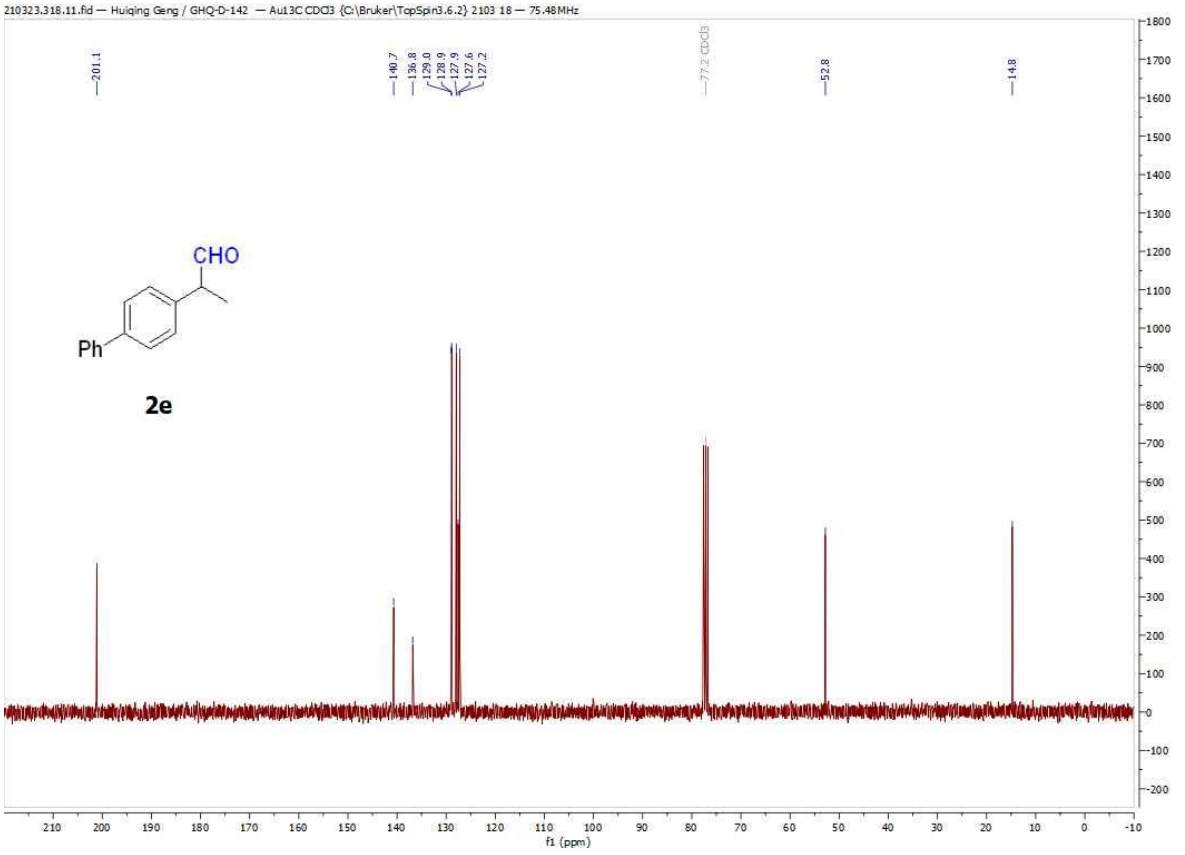
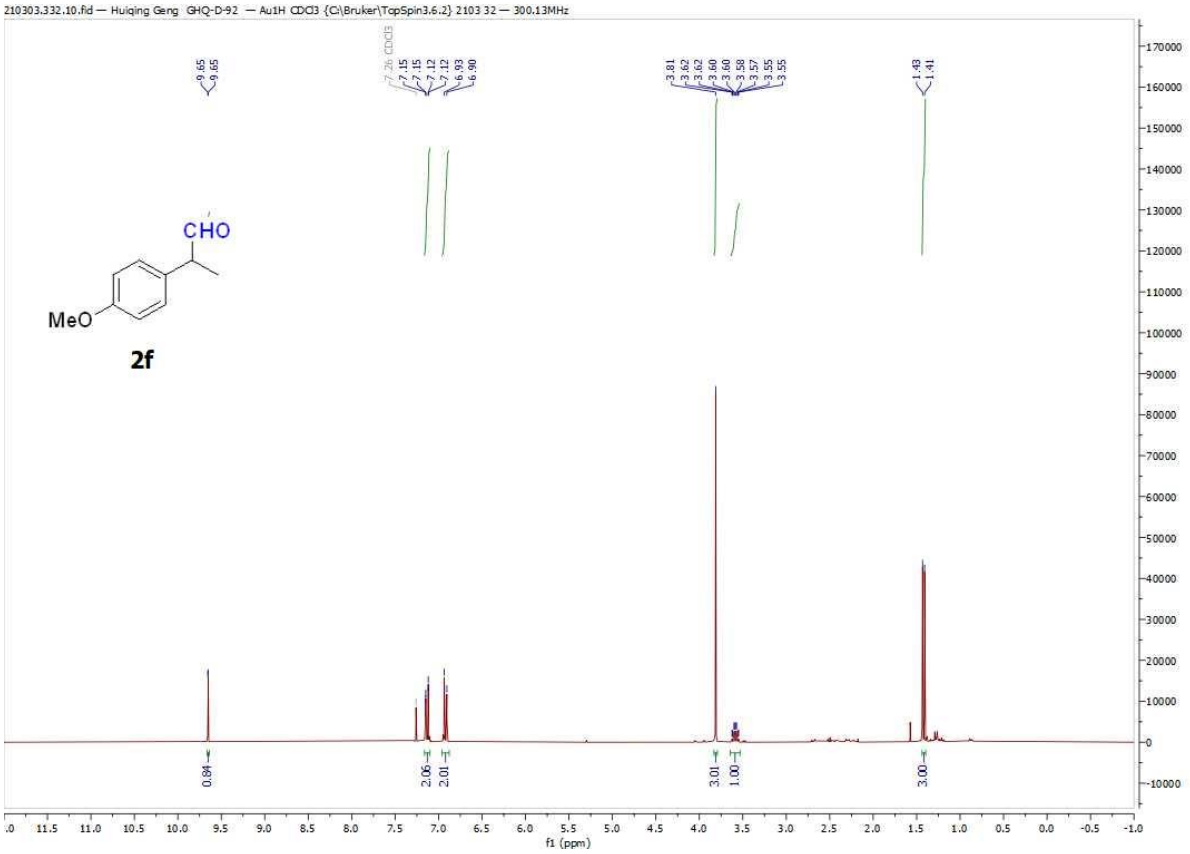
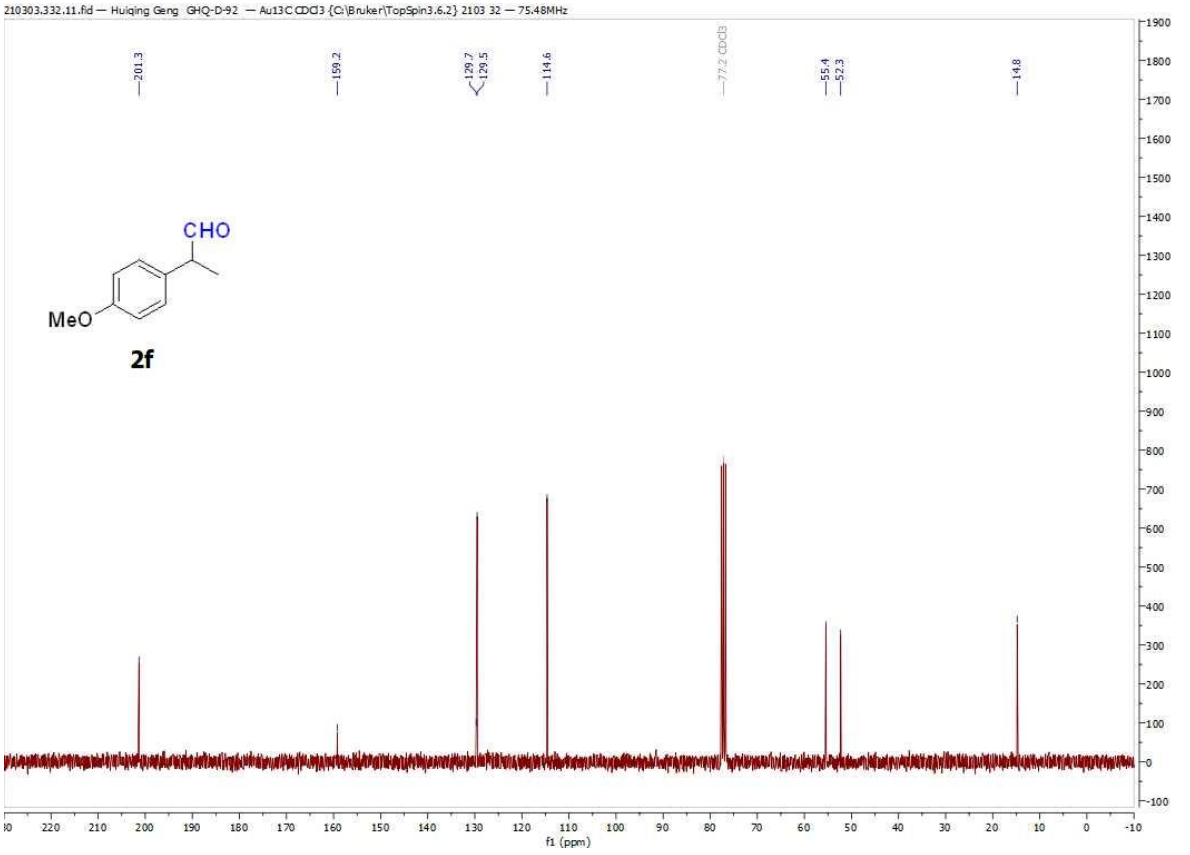
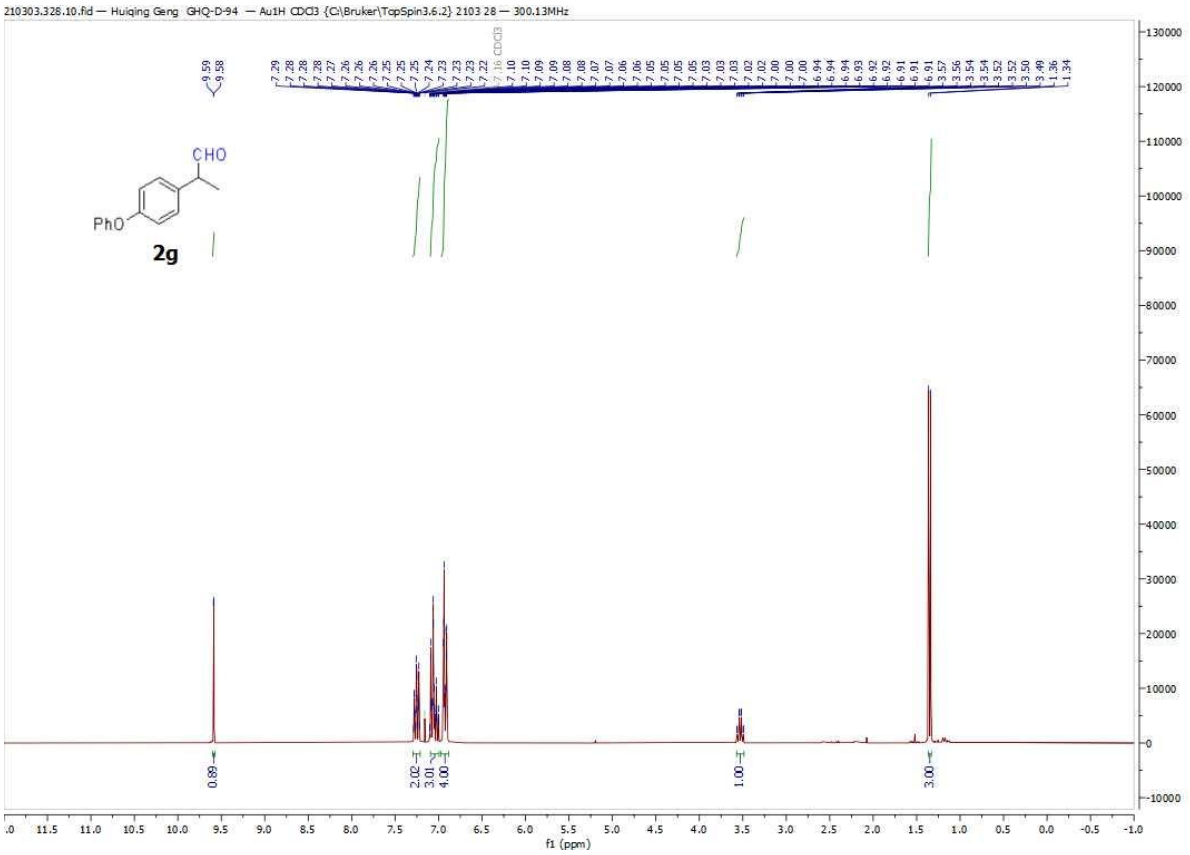
2d
f1 (ppm)

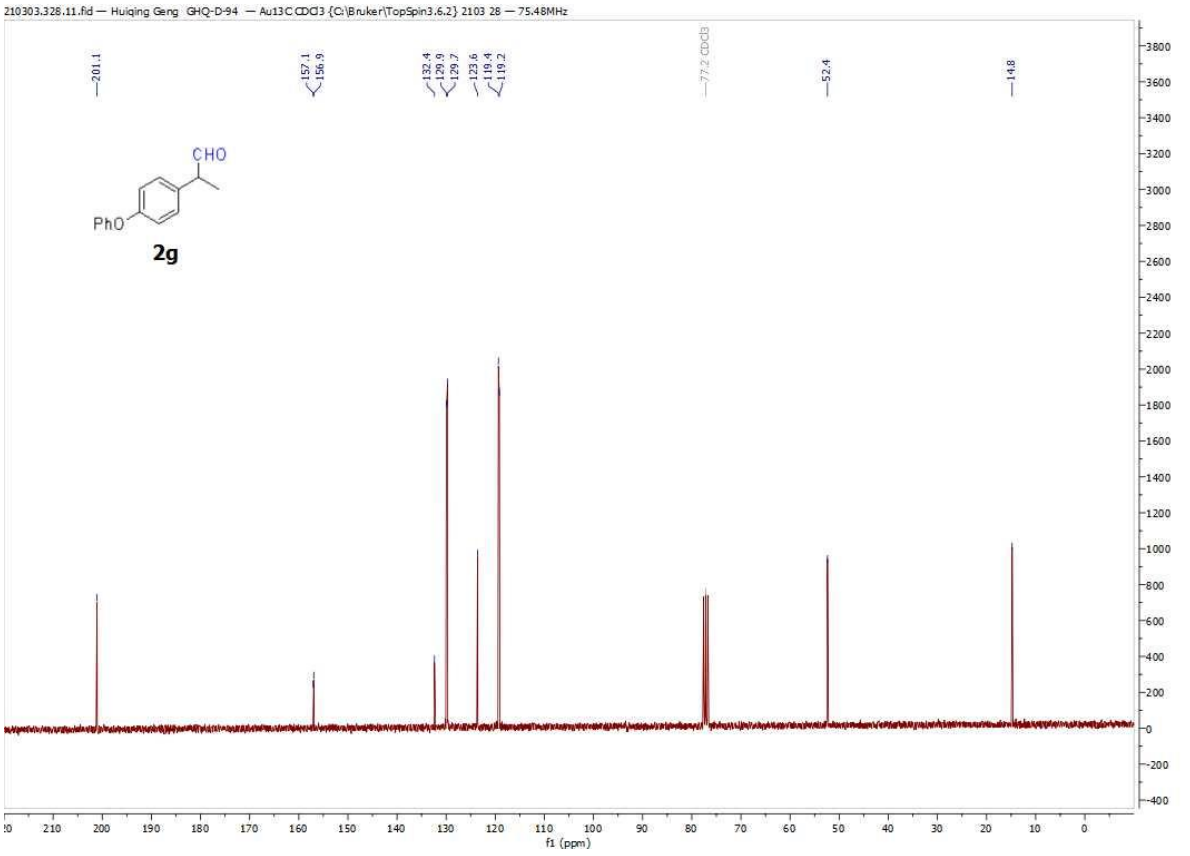

210323.318.10.fid — Huiqing Geng / GHQ-D-142 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 18 — 300.13MHz
CHO
Ph
2e
f1 (ppm)

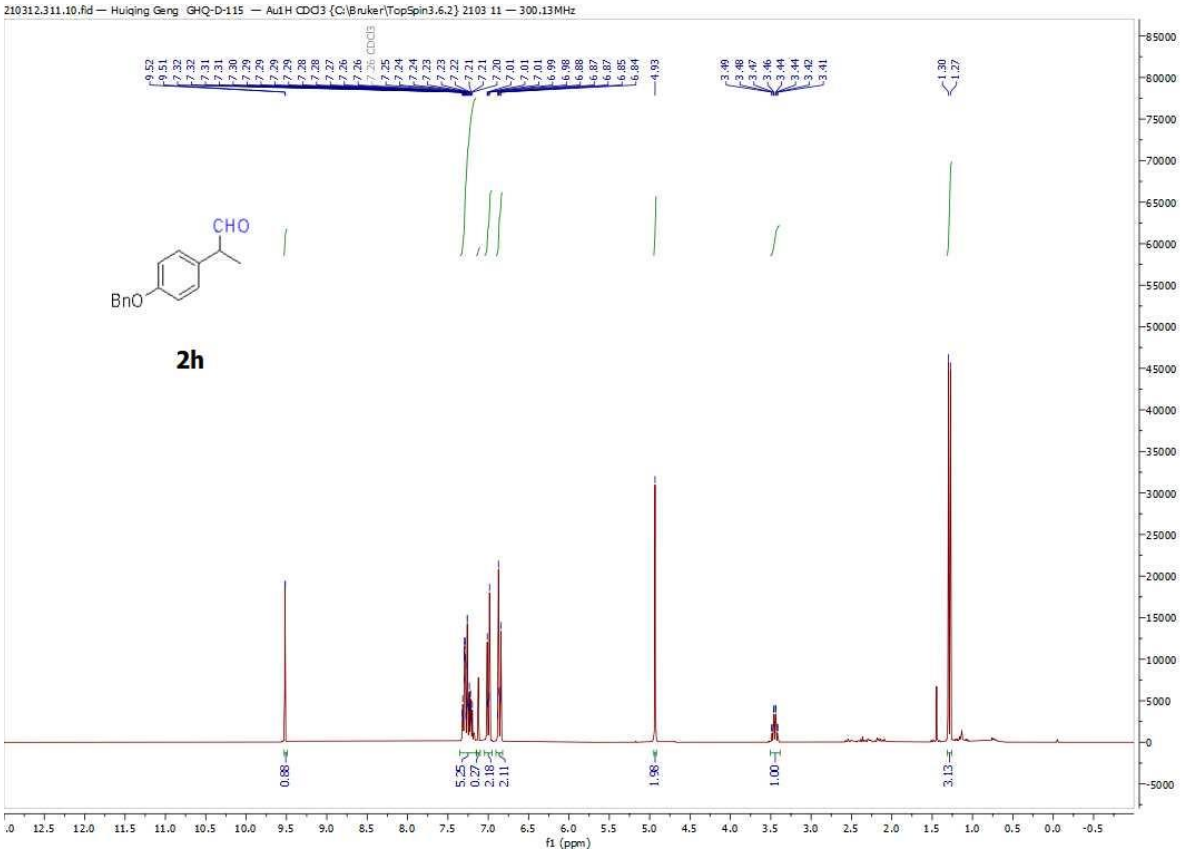

210323.318.11.fid — Huiqing Geng / GHQ-D-142 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 18 — 75.48MHz
201.1
140.7
136.8
129.0
128.9
127.9
127.6
127.2
77.2 CDCl3
52.8
14.8
CHO
Ph
2e
f1 (ppm)

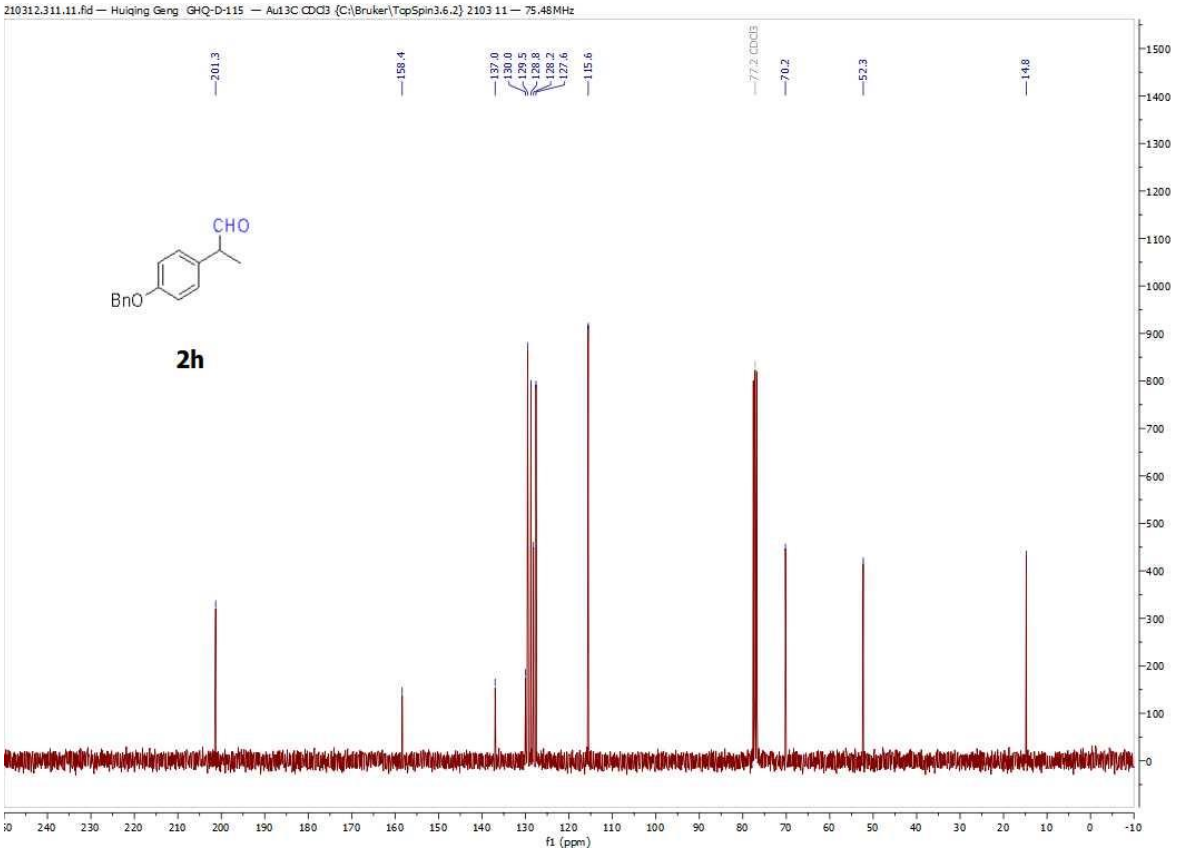

210303.332.10.fid — Huiqing Geng GHQ-D-92 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 32 — 300.13MHz
CHO
MeO
2f
f1 (ppm)

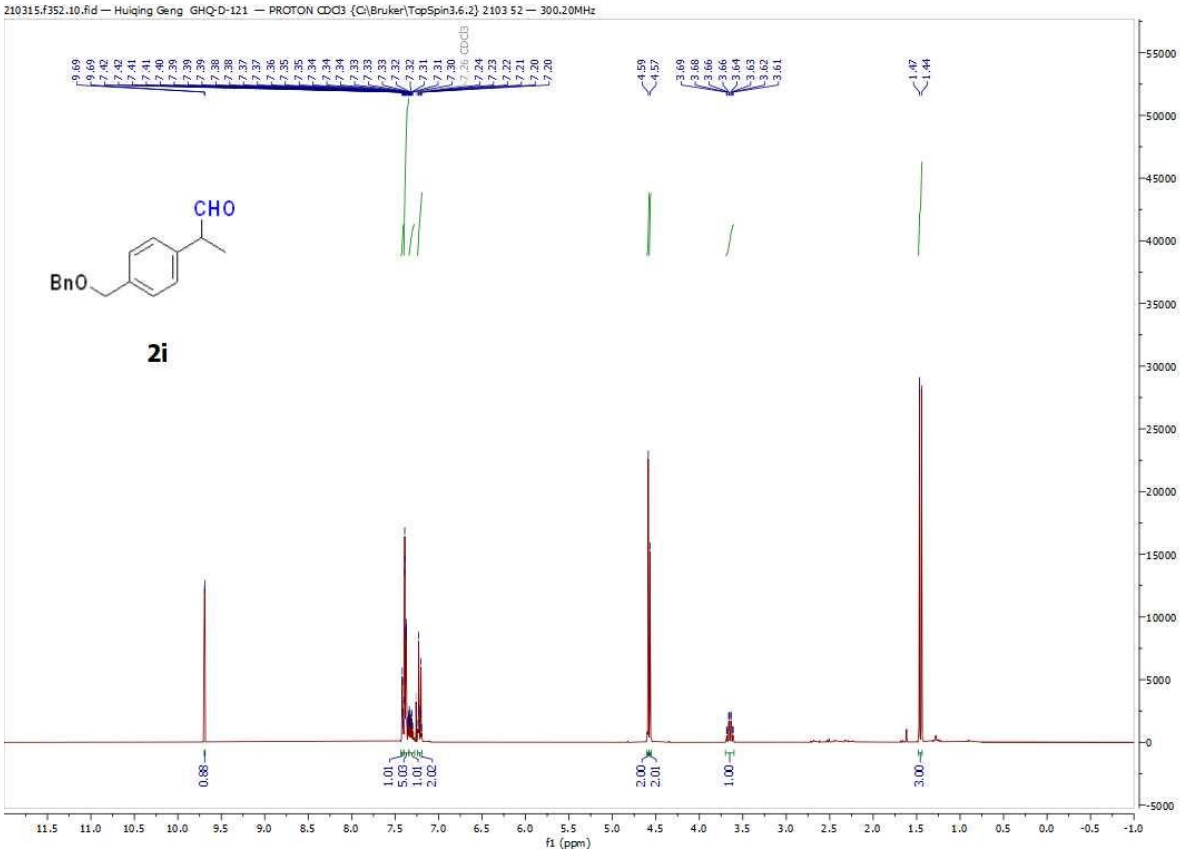

210303.332.11.fid — Huiqing Geng GHQ-D-92 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 32 — 75.48MHz
CHO
MeO
2f
f1 (ppm)

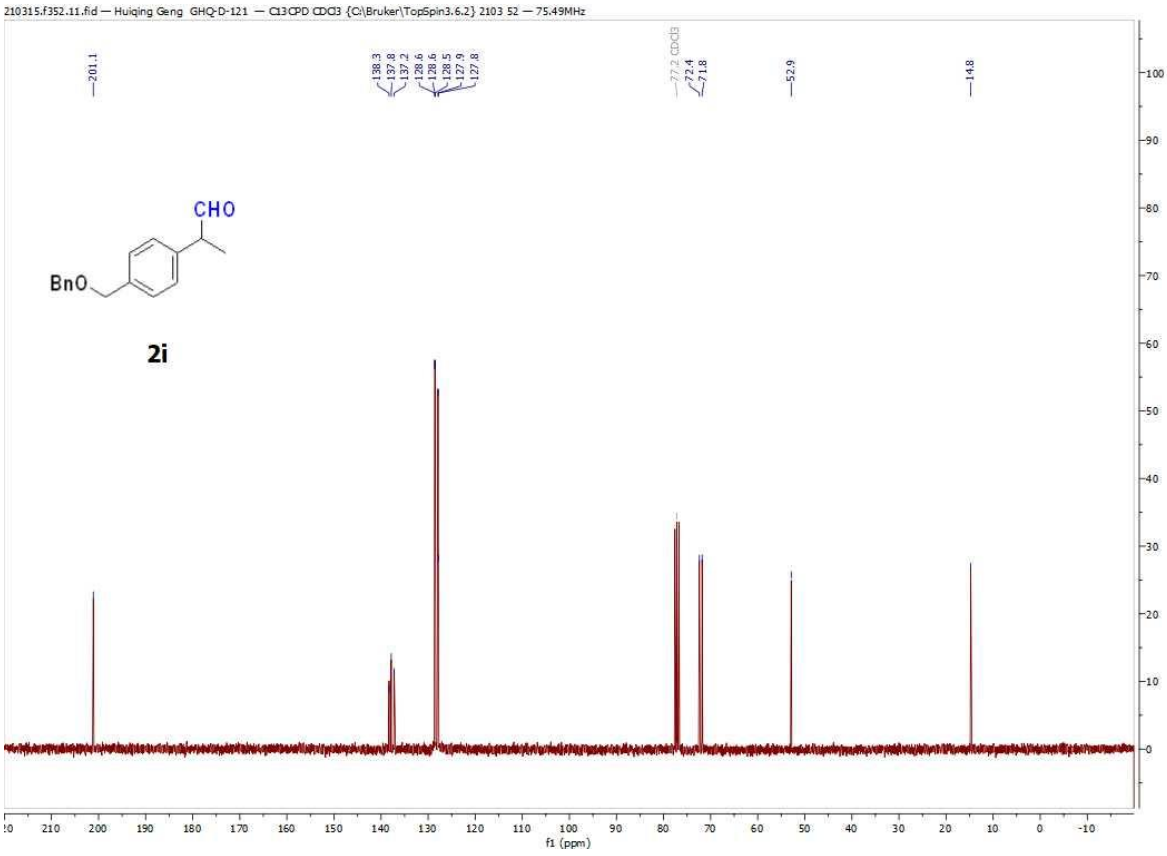

210303.328.10.fid — Huiqing Geng GHQ-D-94 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 28 — 300.13MHz
CHO
PhO
2g
f1 (ppm)

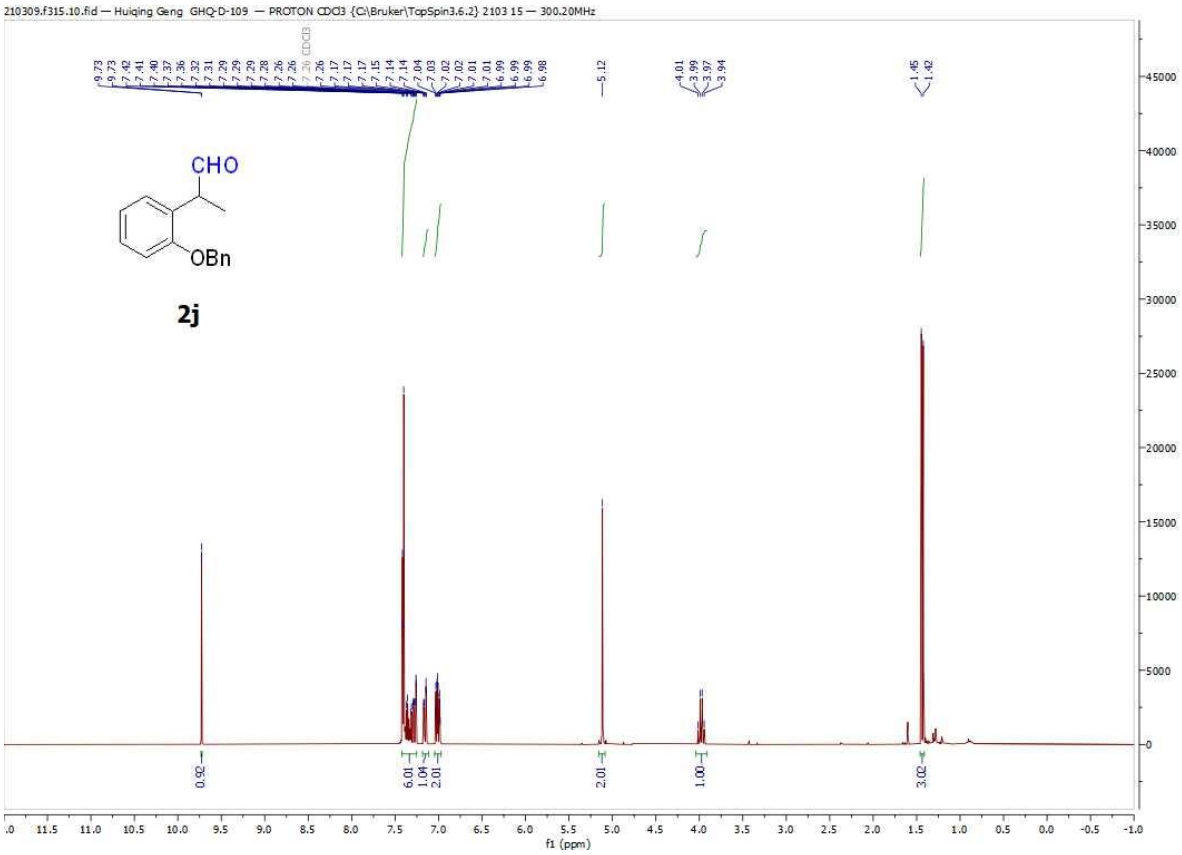

210303.328.11.fid — Huiqing Geng GHQ-D-94 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 28 — 75.48MHz
CHO
PhO
2g
f1 (ppm)

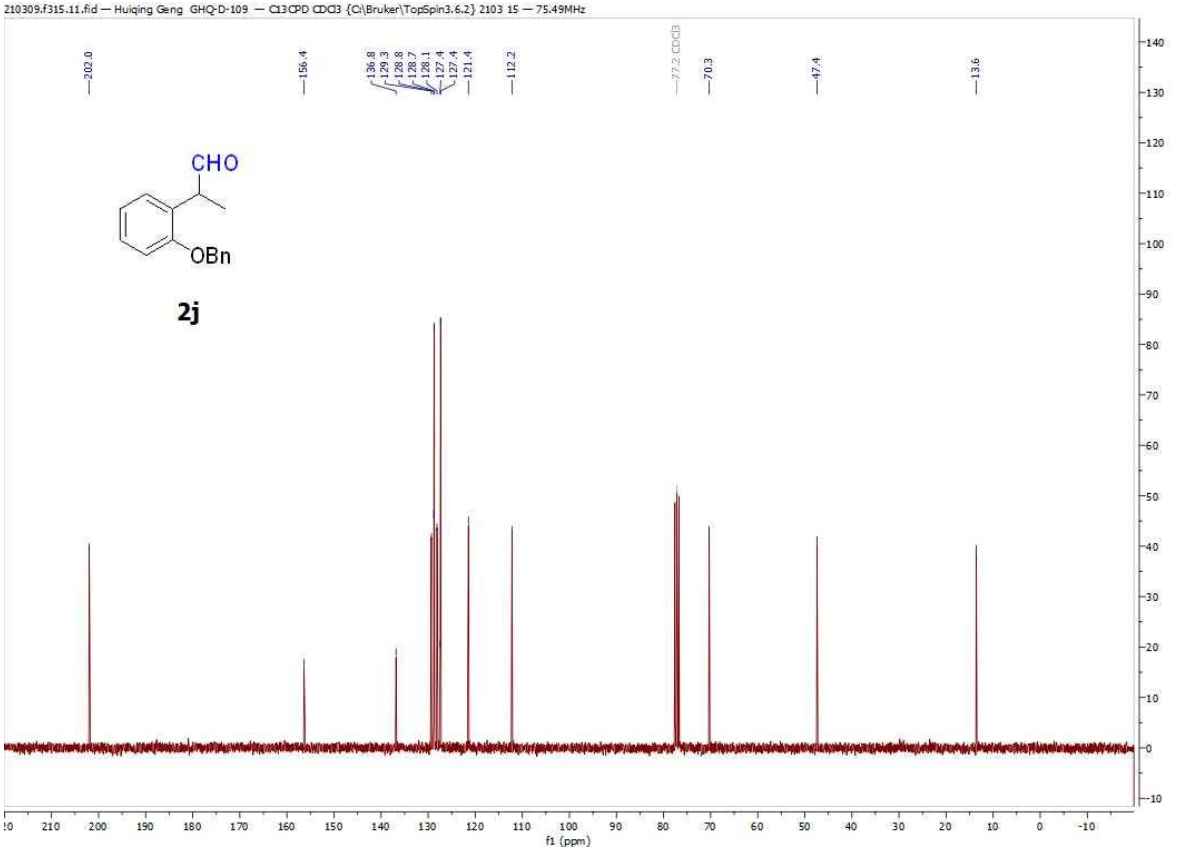

210312.311.10.fid — Huiqing Geng GHQ-D-115 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 11 — 300.13MHz
CHO
BnO
2h
f1 (ppm)

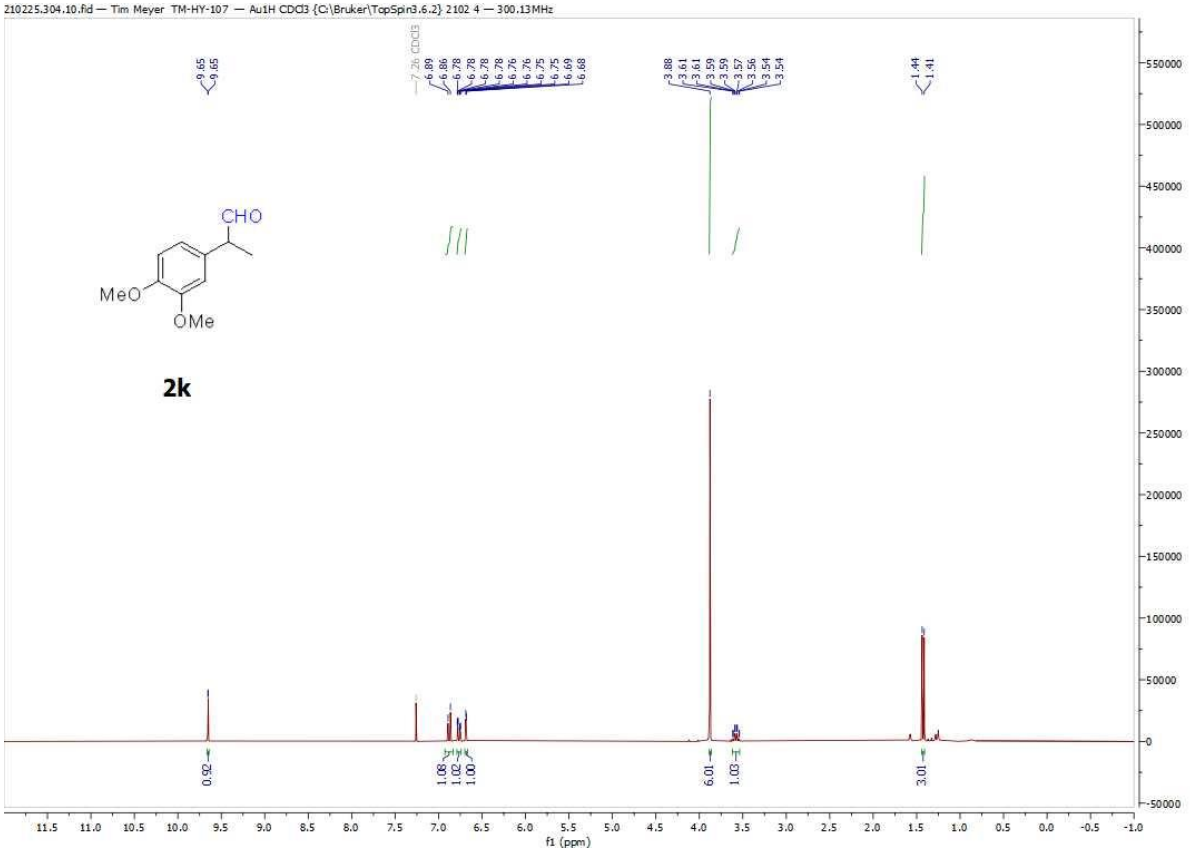

210312.311.11.fid — Huiqing Geng GHQ-D-115 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 11 — 75.48MHz
CHO
BnO
2h
f1 (ppm)

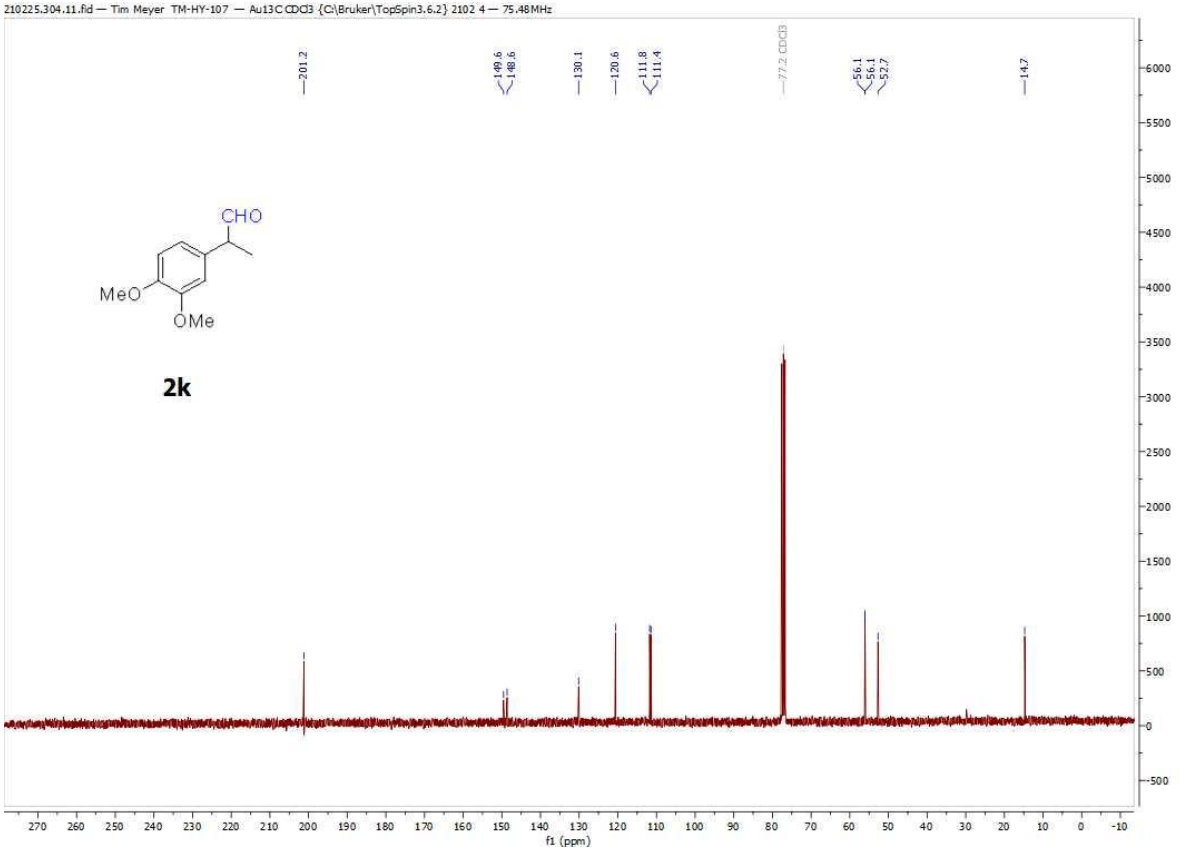

CHO
BnO
2i
f1 (ppm)

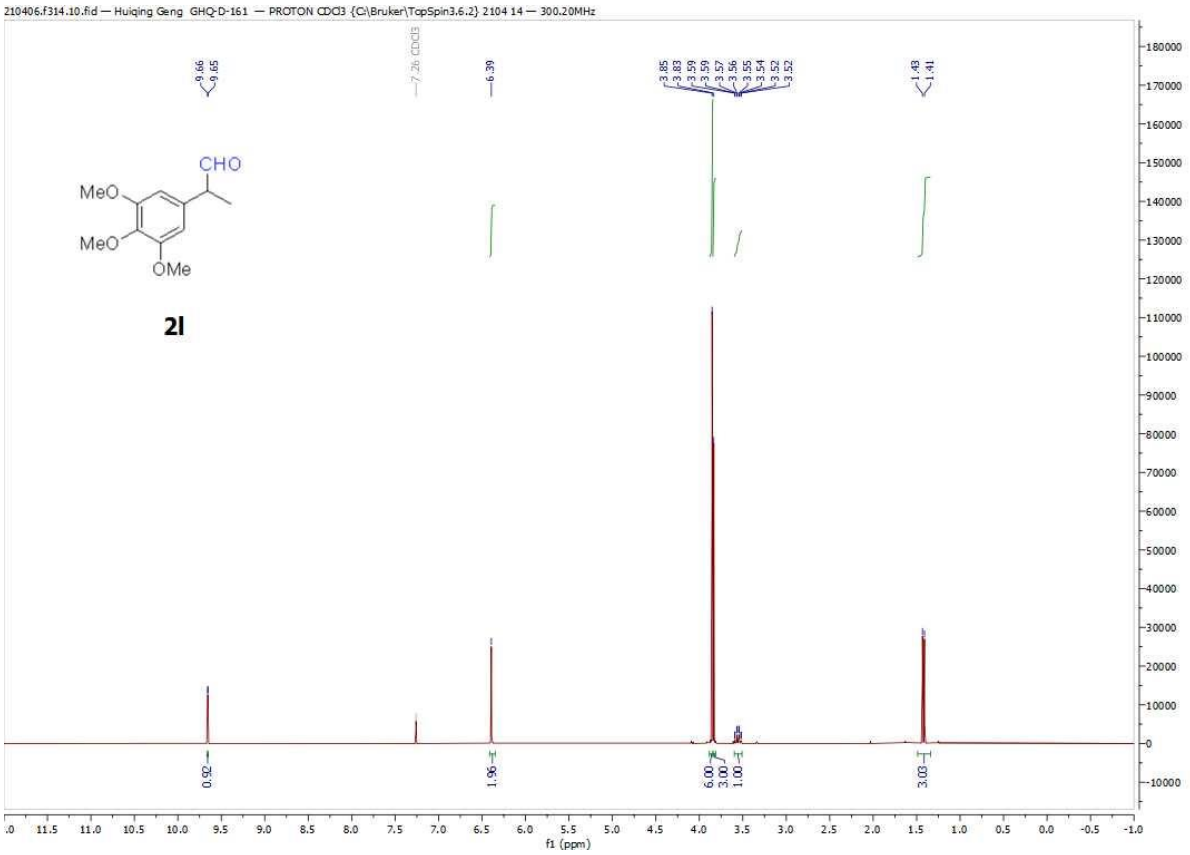

CHO
BnO
2i
f1 (ppm)


210309.f315.10.fid — Huiqing Geng GHQ-D-109 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 15 — 300.20MHz
CHO
OBn
2j
f1 (ppm)

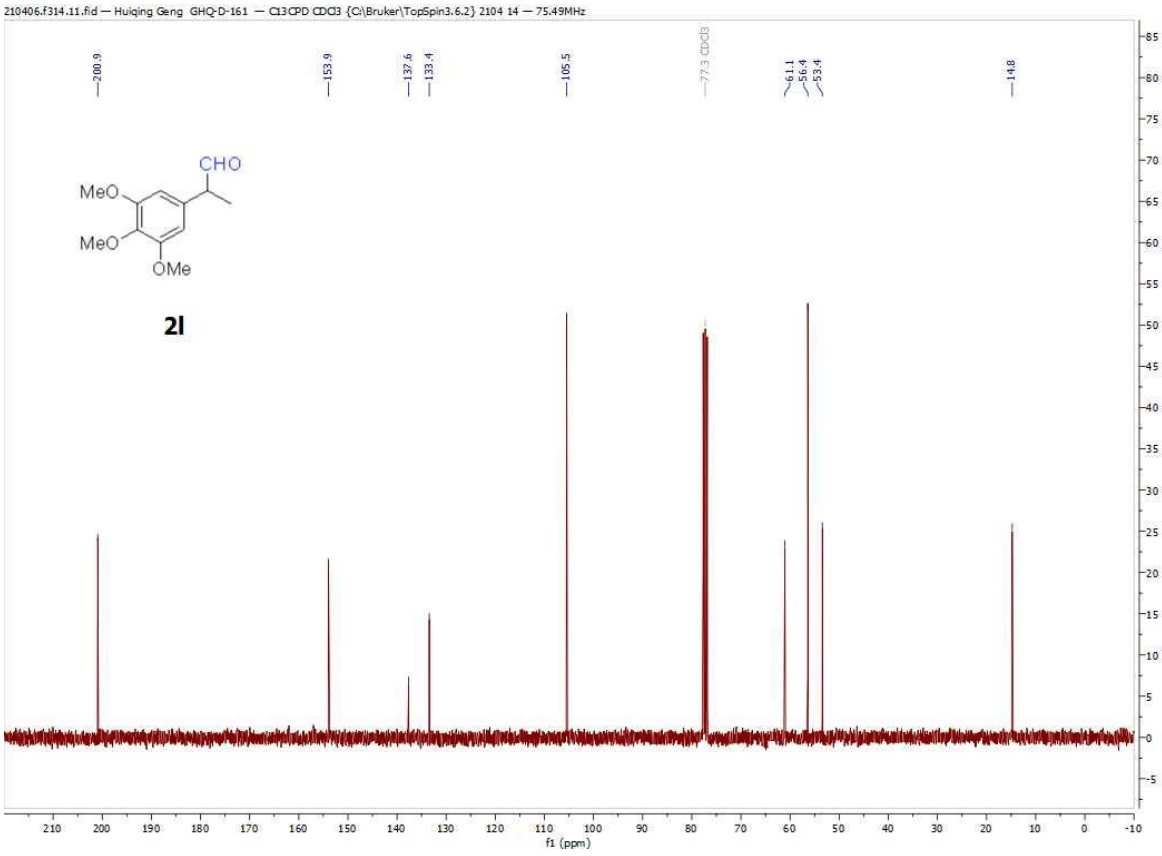

210309.f315.11.fid — Huiqing Geng GHQ-D-109 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 15 — 75.49MHz
CHO
OBn
2j
f1 (ppm)


210225.304.10.fid — Tim Meyer TM-HY-107 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2102 4 — 300.13MHz
CHO
MeO
OMe
2k
f1 (ppm)

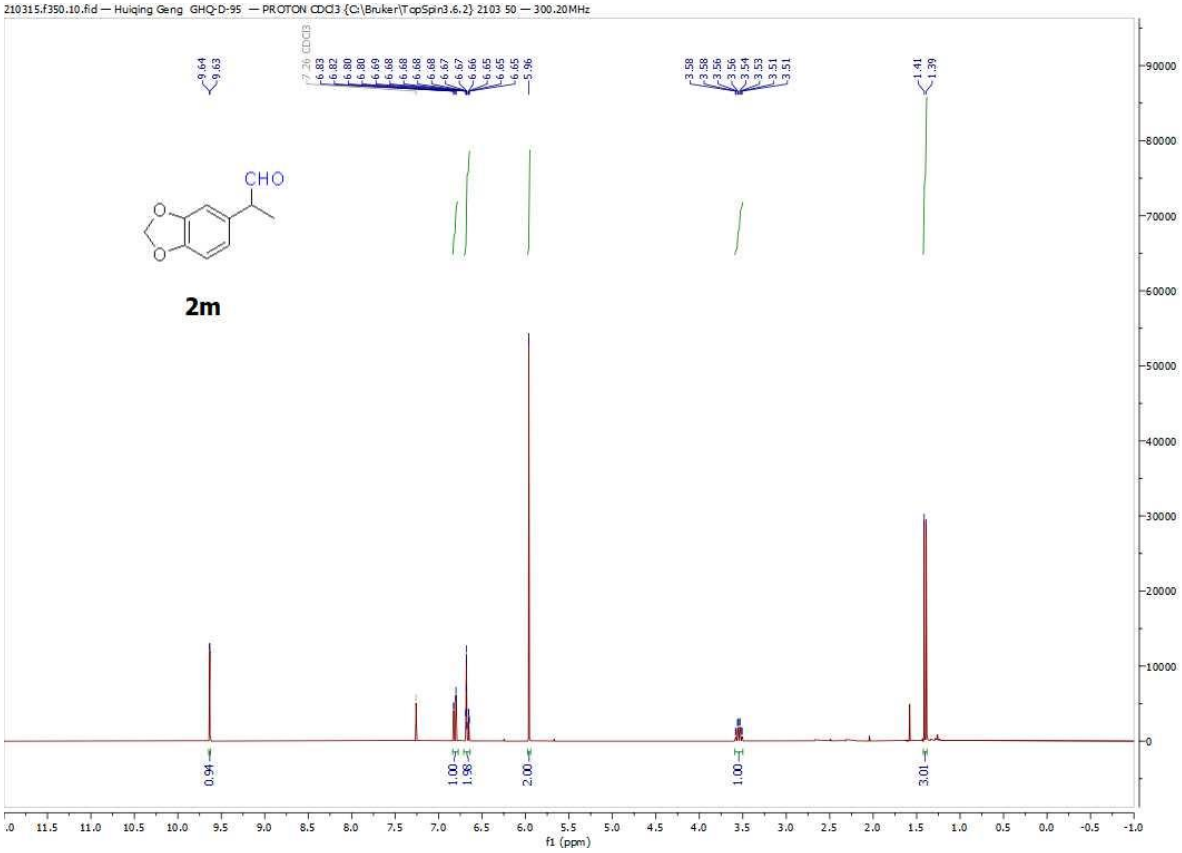

210225.304.11.fid — Tim Meyer TM-HY-107 — Au13CCDCl3 {C:\Bruker\TopSpin3.6.2} 2102 4 — 75.48MHz
CHO
MeO
OMe
2k
f1 (ppm)

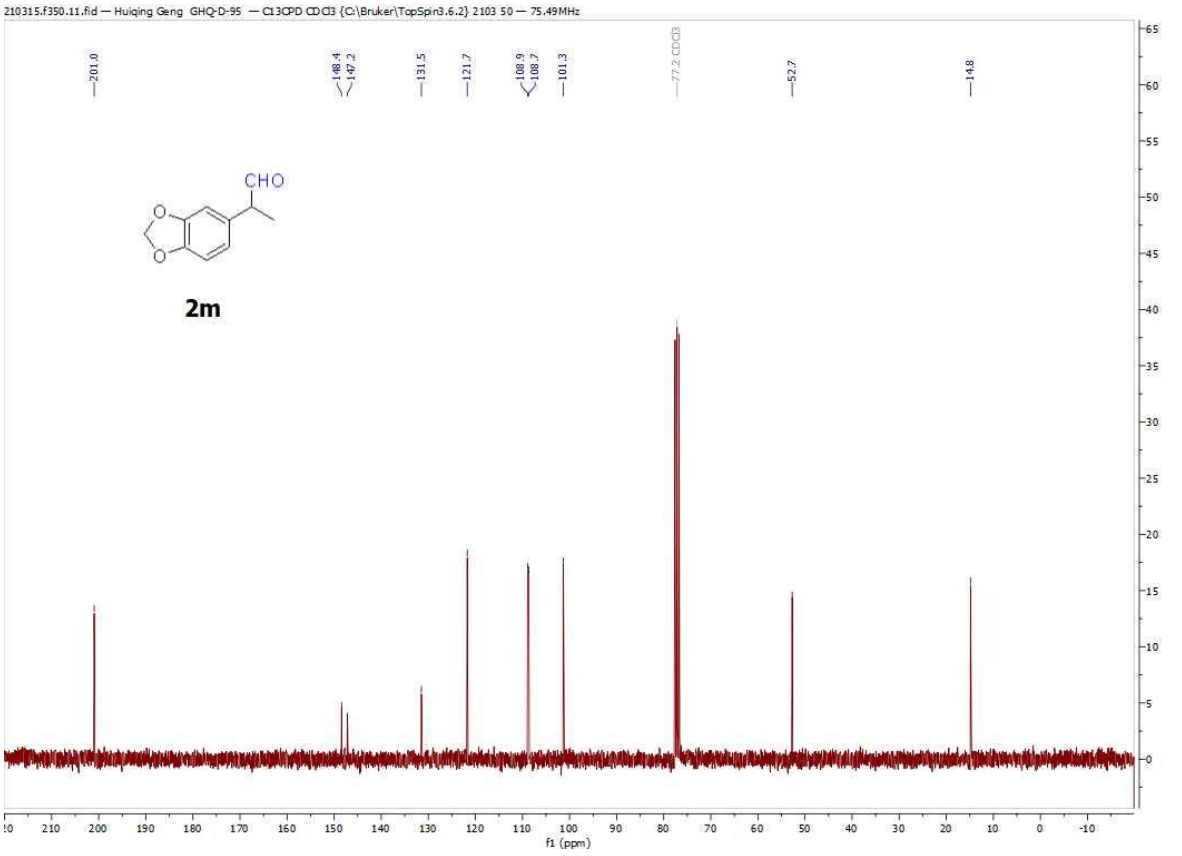

210406.f314.10.fid — Huiqing Geng GHQ-D-161 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2104 14 — 300.20MHz
9.66
9.65
7.26 CDCl3
6.39
3.85
3.83
3.59
3.59
3.57
3.56
3.55
3.54
3.52
3.52
1.43
1.41
CHO
MeO
MeO
OMe
0.92
1.96
6.00
3.00
1.00
3.03
f1 (ppm)

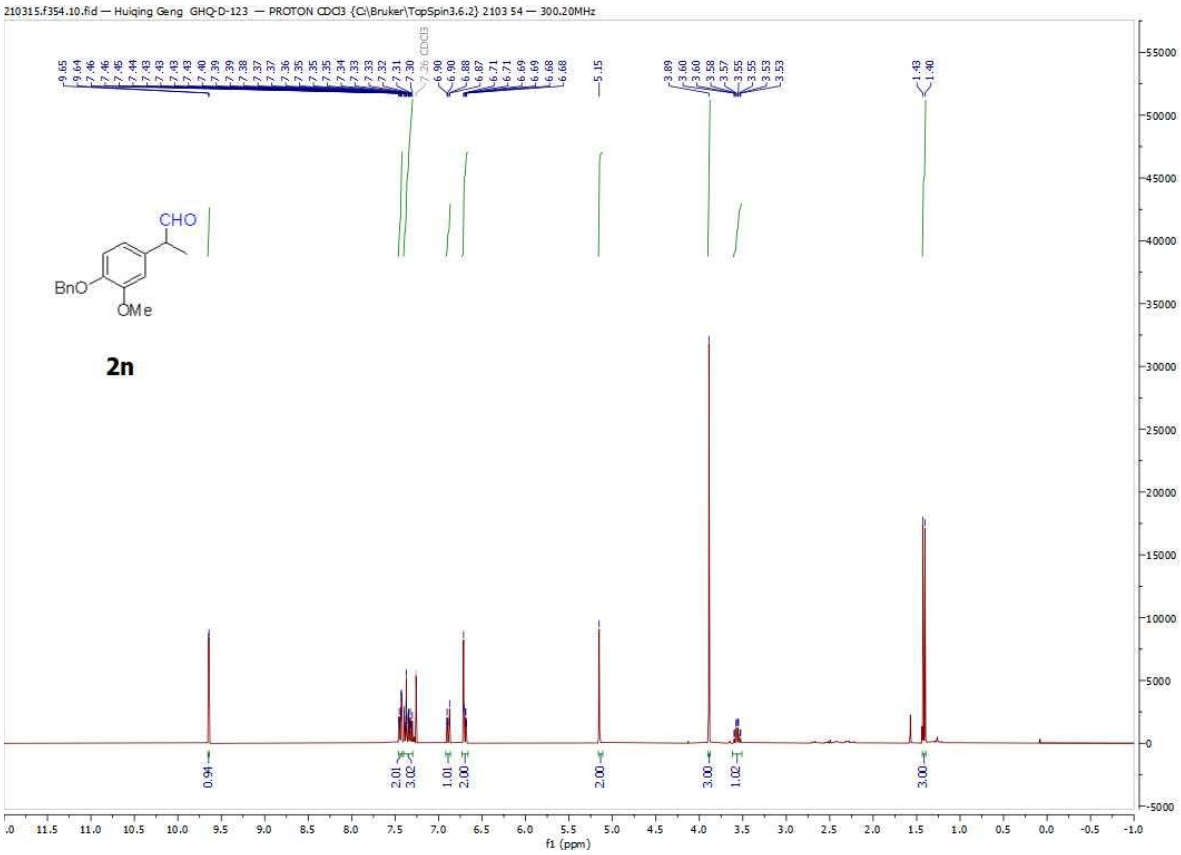
2l

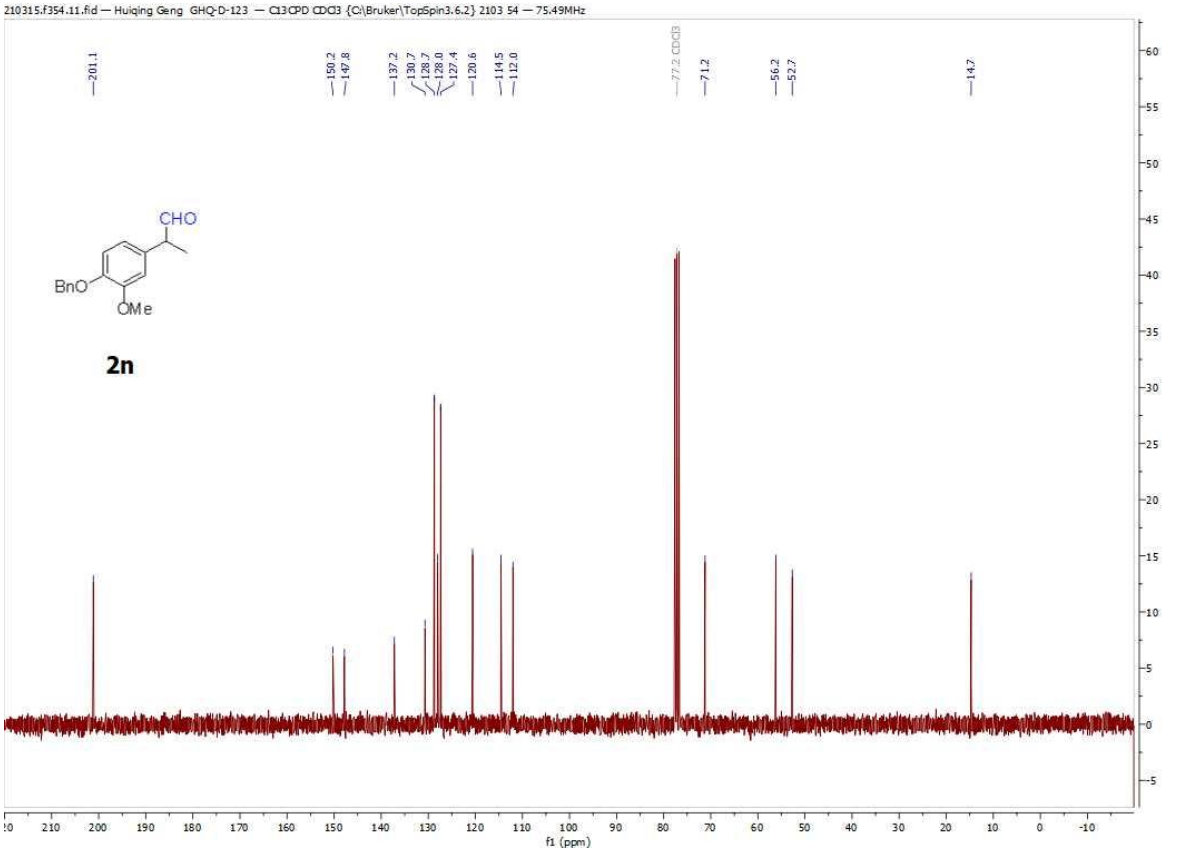

210406.f314.11.fid — Huiqing Geng GHQ-D-161 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2104 14 — 75.49MHz
200.9
153.9
137.6
133.4
105.5
77.3 CDCl3
61.1
56.4
53.4
14.8
CHO
MeO
MeO
OMe
f1 (ppm)

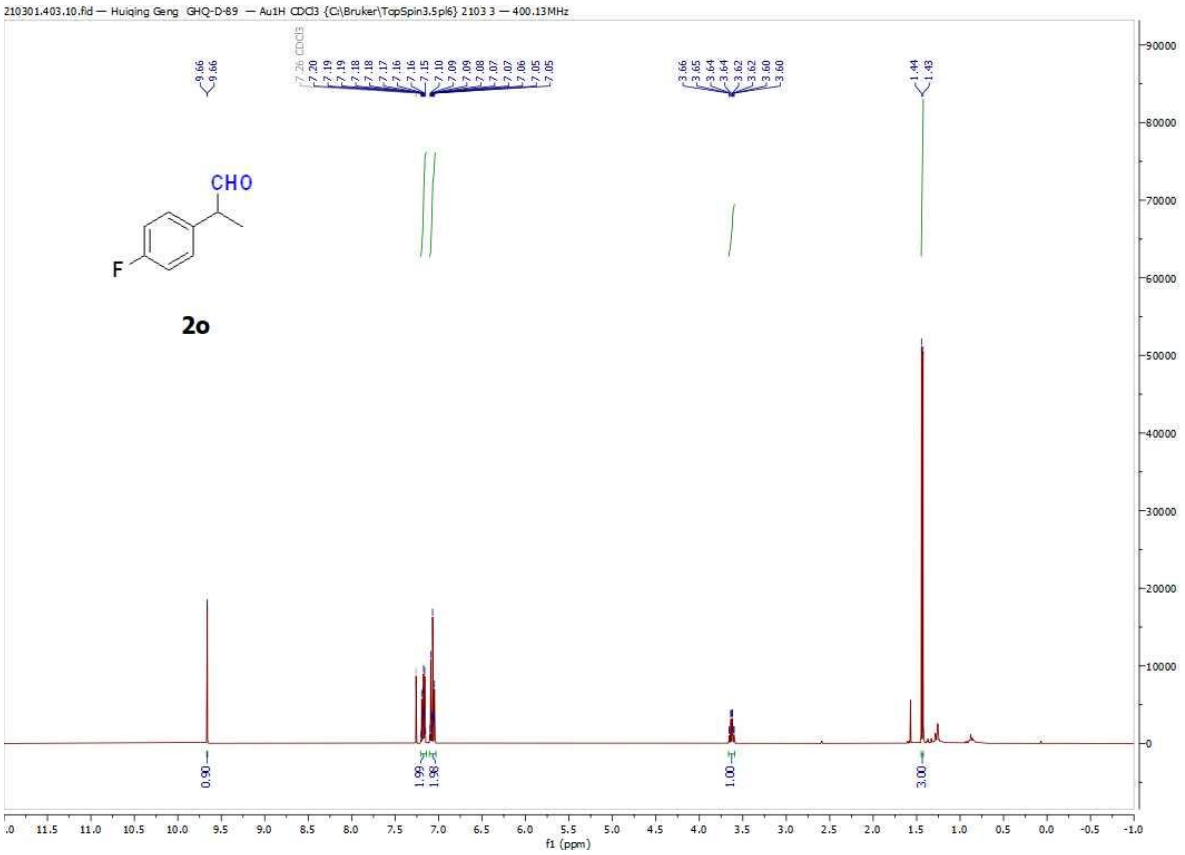
2l

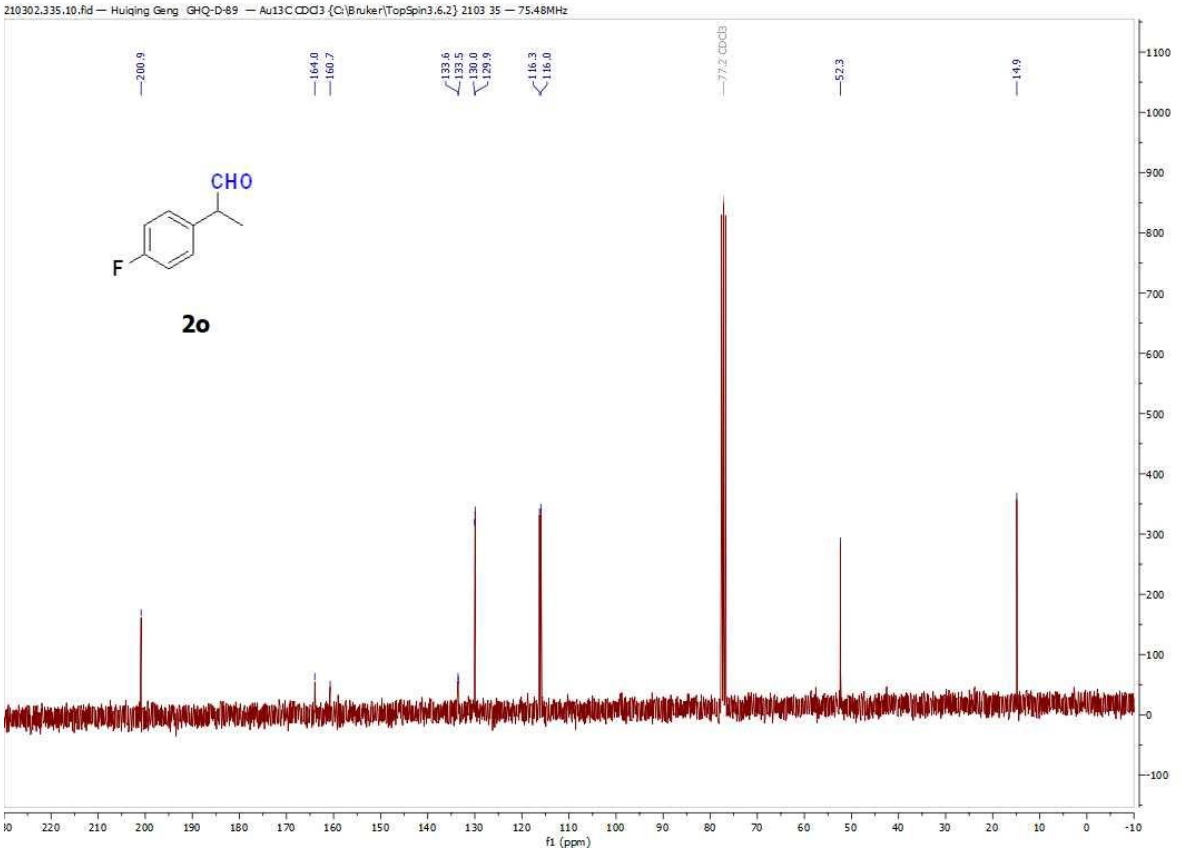

210315.f350.10.fid — Huiqing Geng GHQ-D-95 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 50 — 300.20MHz
CHO
2m
f1 (ppm)

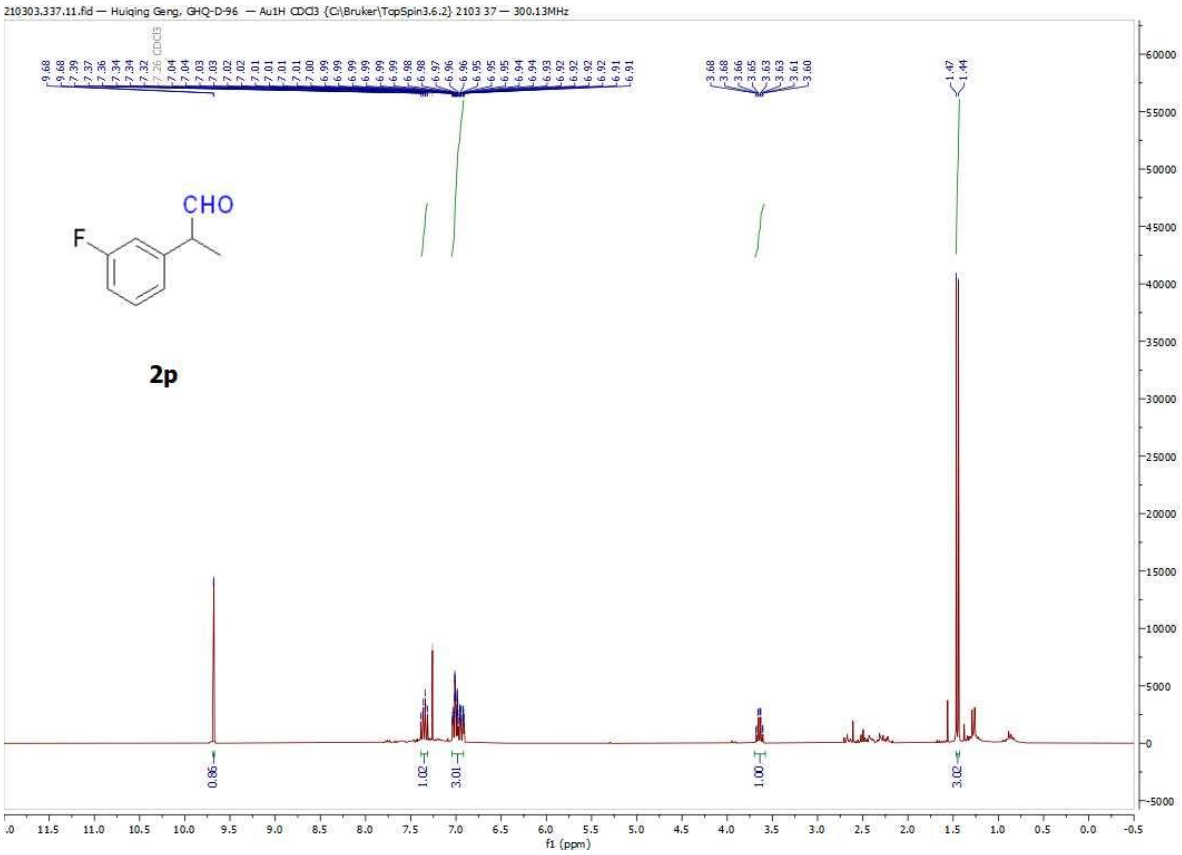

210315.f350.11.fid — Huiqing Geng GHQ-D-95 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 50 — 75.49MHz
CHO
2m
f1 (ppm)

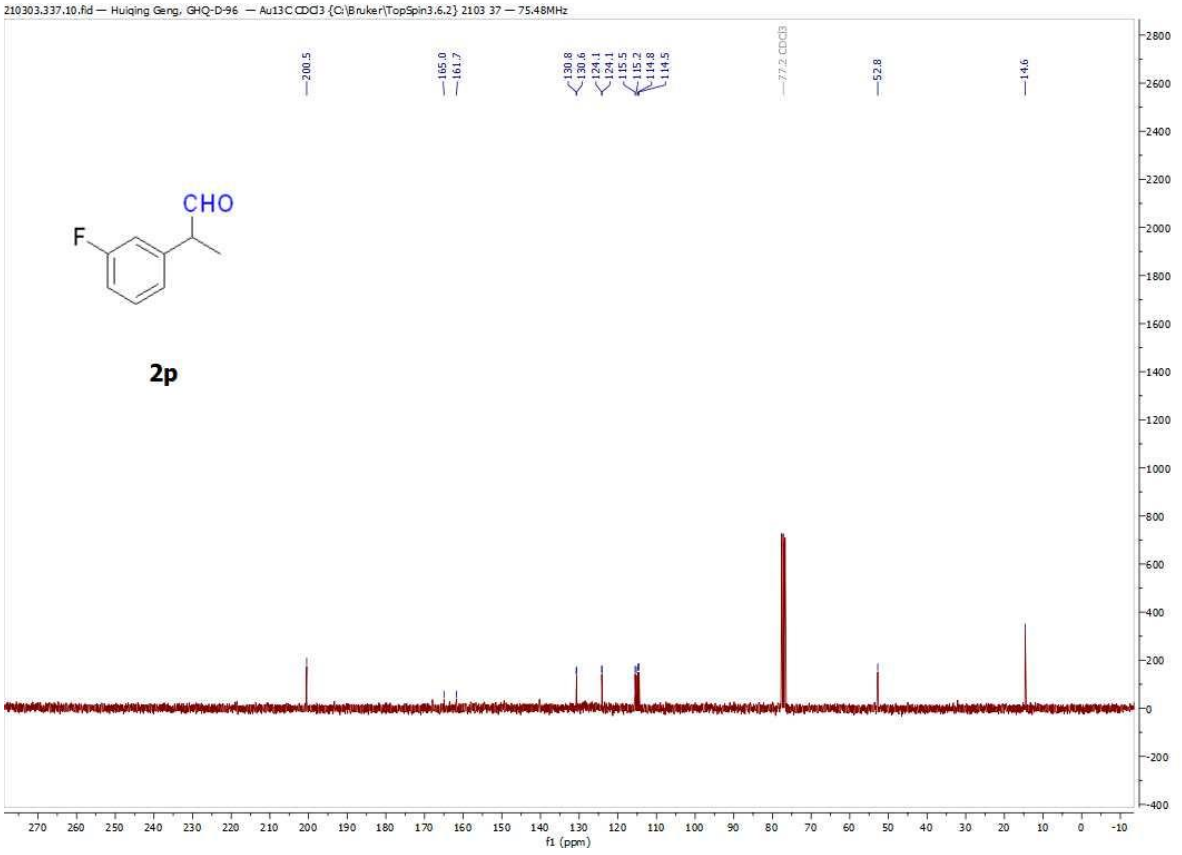

210315.f354.10.fid — Huiqing Geng GHQ-D-123 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 54 — 300.20MHz
CHO
BnO
OMe
2n
f1 (ppm)

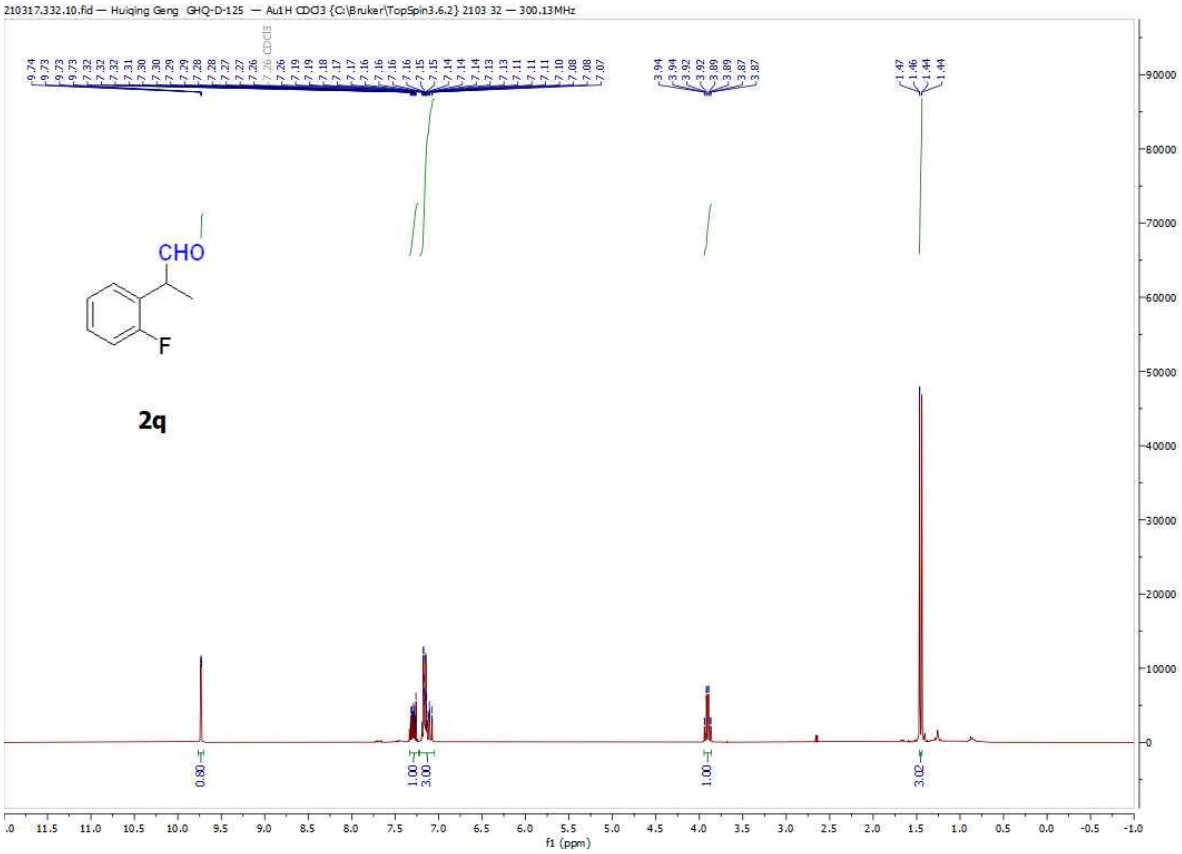

210315.f354.11.fid — Huiqing Geng GHQ-D-123 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 54 — 75.49MHz
CHO
BnO
OMe
2n
f1 (ppm)

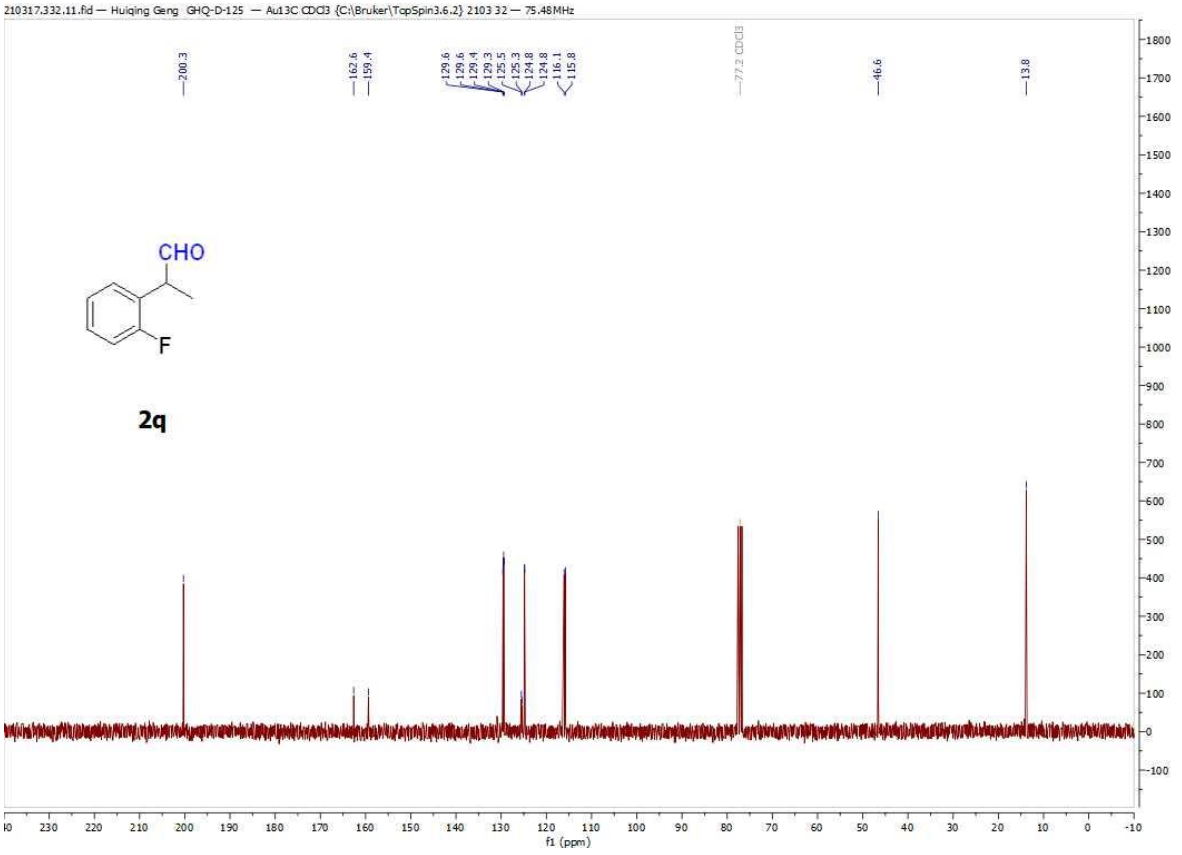

210301.403.10.fid — Huiqing Geng GHQ-D-89 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 3 — 400.13MHz
CHO
F
2o
f1 (ppm)

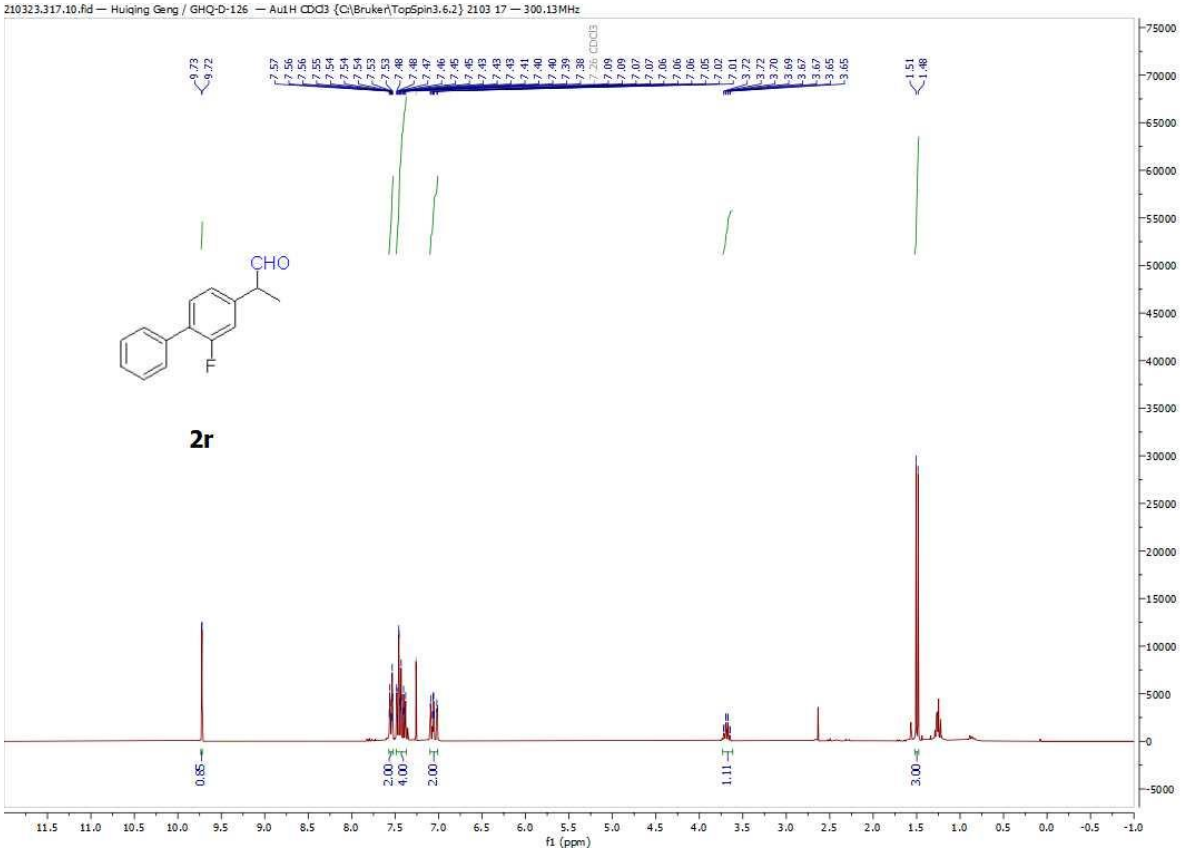

210302.335.10.fid — Huiqing Geng GHQ-D-89 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 35 — 75.48MHz
CHO
F
2o
f1 (ppm)

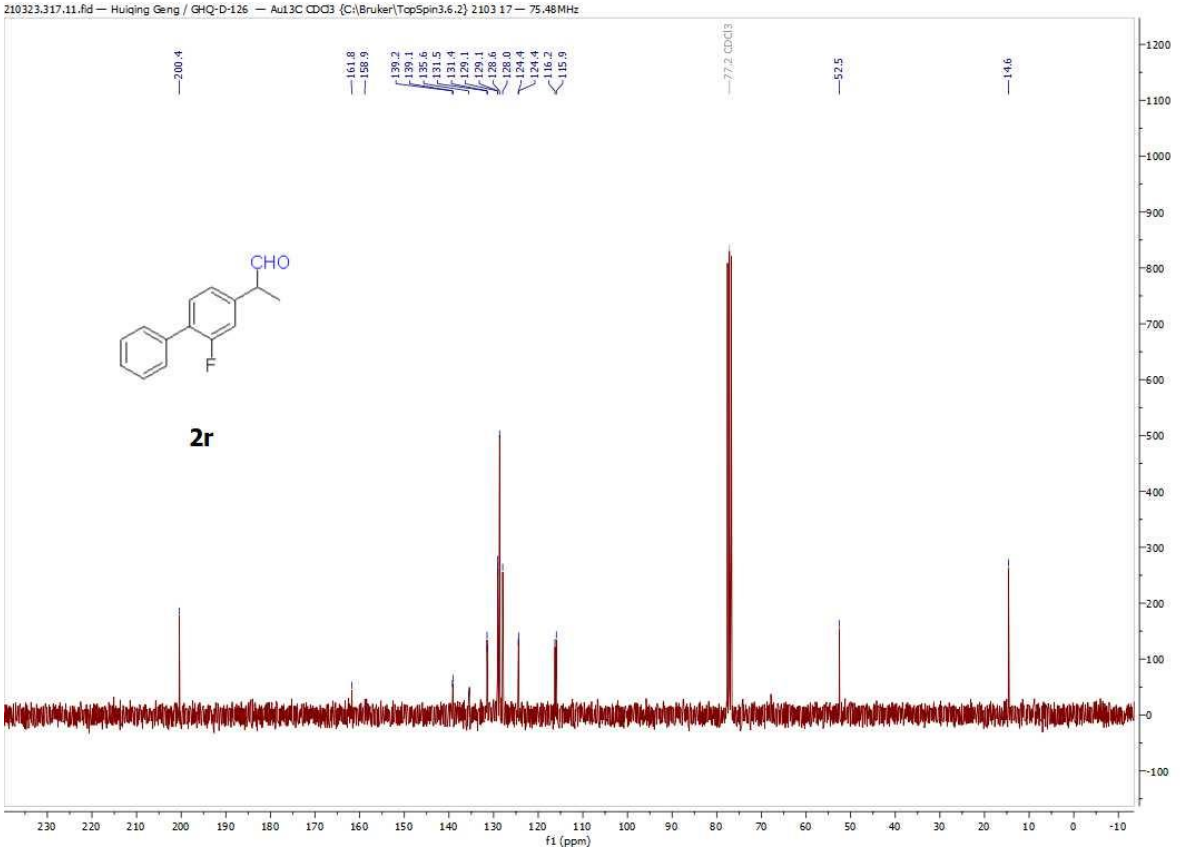

210303.337.11.fid — Huiqing Geng, GHQ-D-96 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 37 — 300.13MHz
CHO
F
2p
f1 (ppm)

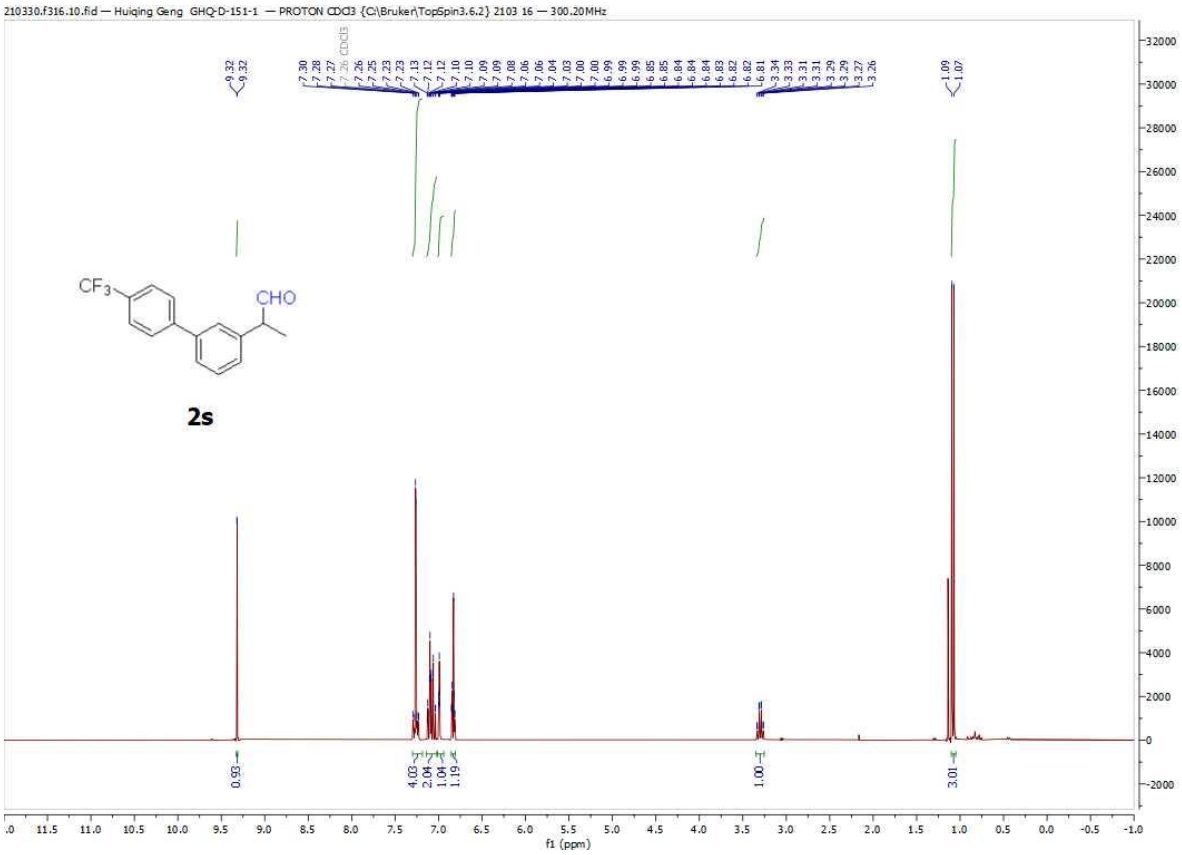

210303.337.10.fid — Huiqing Geng, GHQ-D-96 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 37 — 75.48MHz
CHO
F
2p
f1 (ppm)

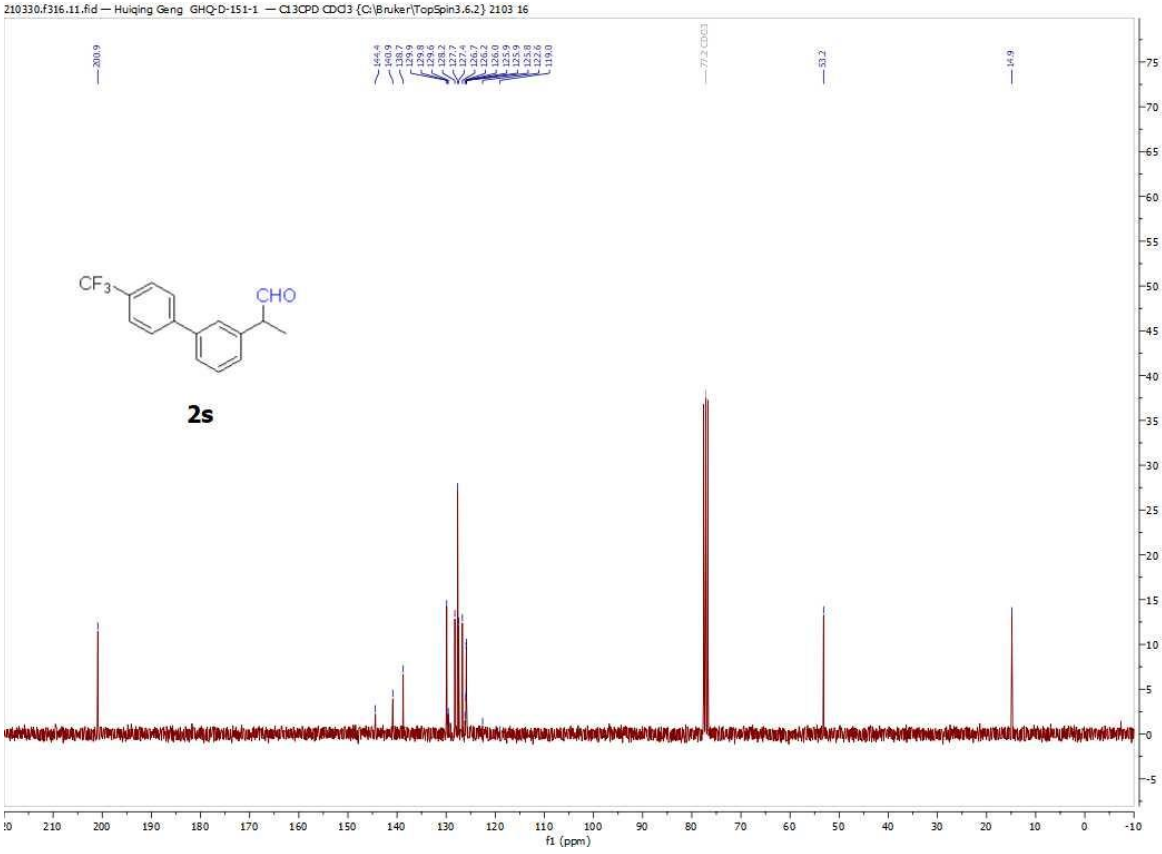

210317.332.10.fid — Huiqing Geng GHQ-D-125 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 32 — 300.13MHz
CHO
F
2q
f1 (ppm)

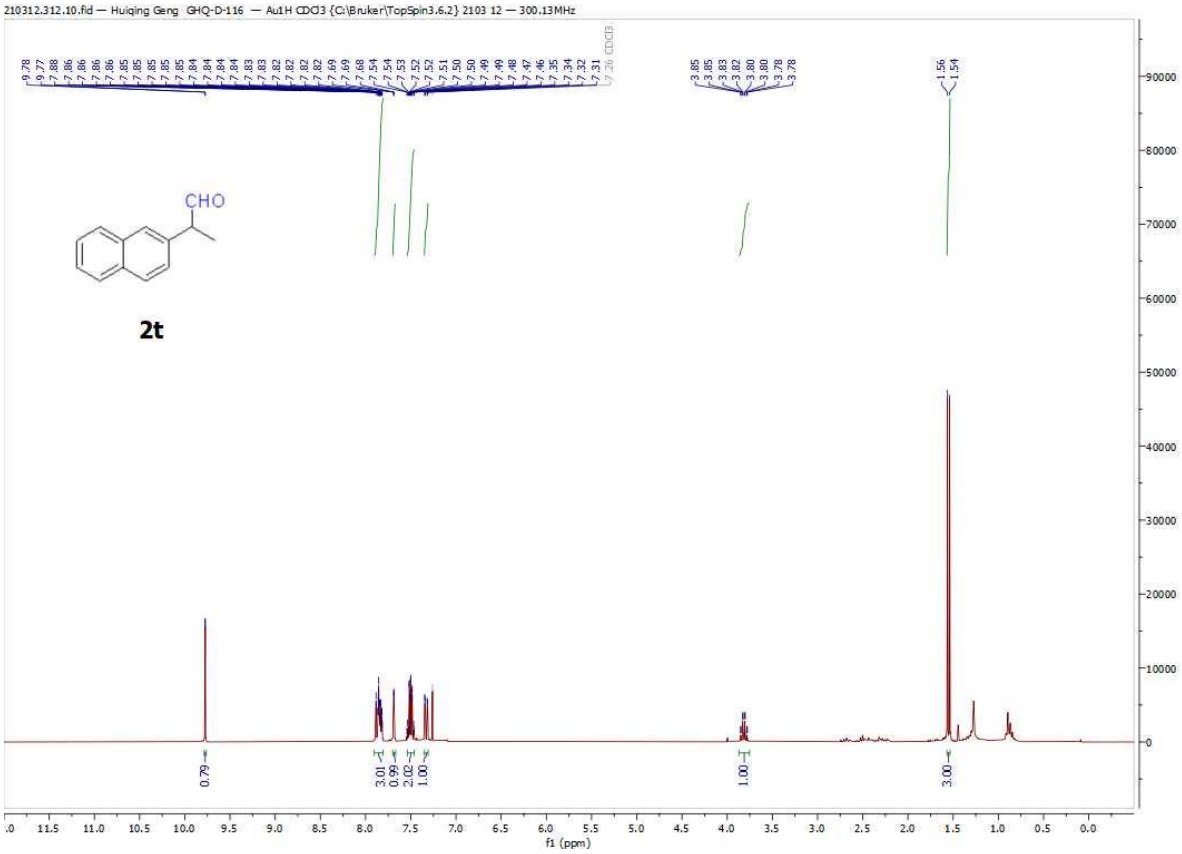

210317.332.11.fid — Huiqing Geng GHQ-D-125 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 32 — 75.48MHz
CHO
F
2q
f1 (ppm)

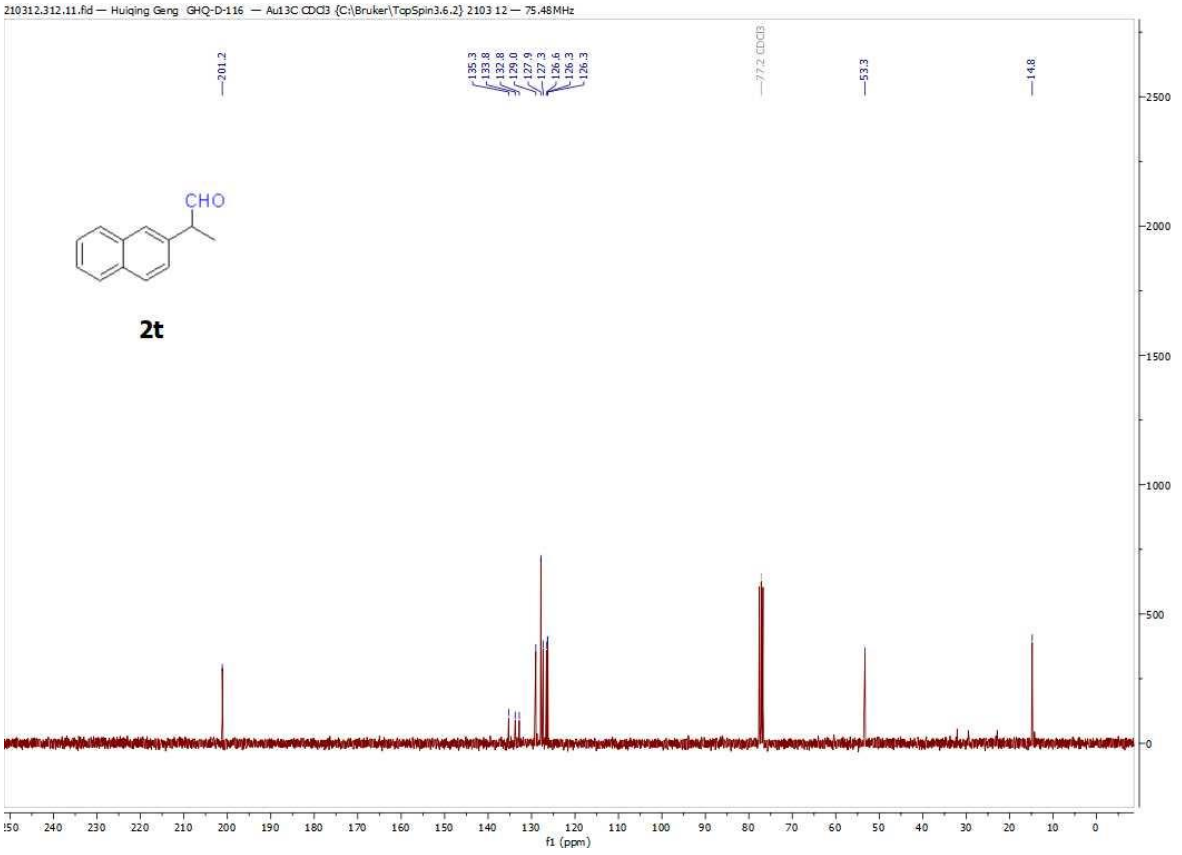

210323.317.10.fid — Huiqing Geng / GHQ-D-126 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 17 — 300.13MHz
CHO
F
2r
f1 (ppm)

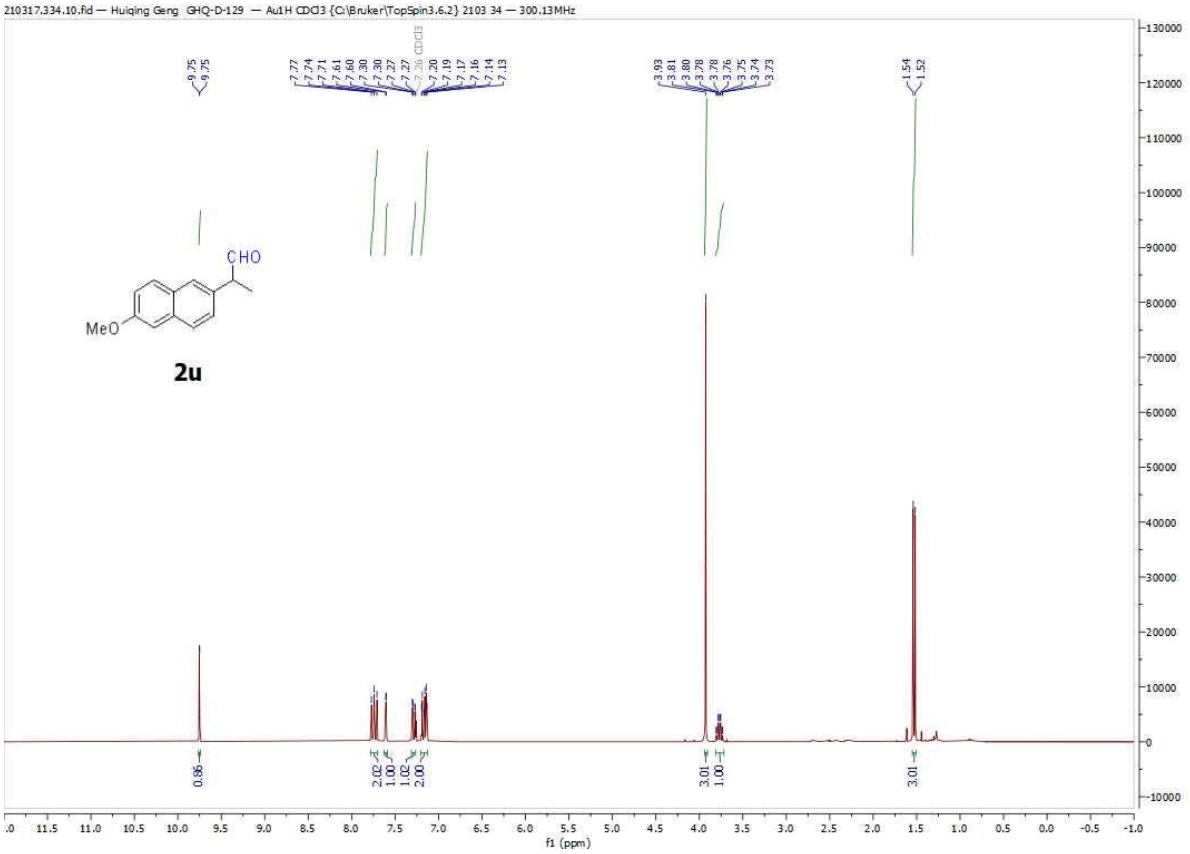

210323.317.11.fid — Huiqing Geng / GHQ-D-126 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 17 — 75.48MHz
CHO
F
2r
f1 (ppm)

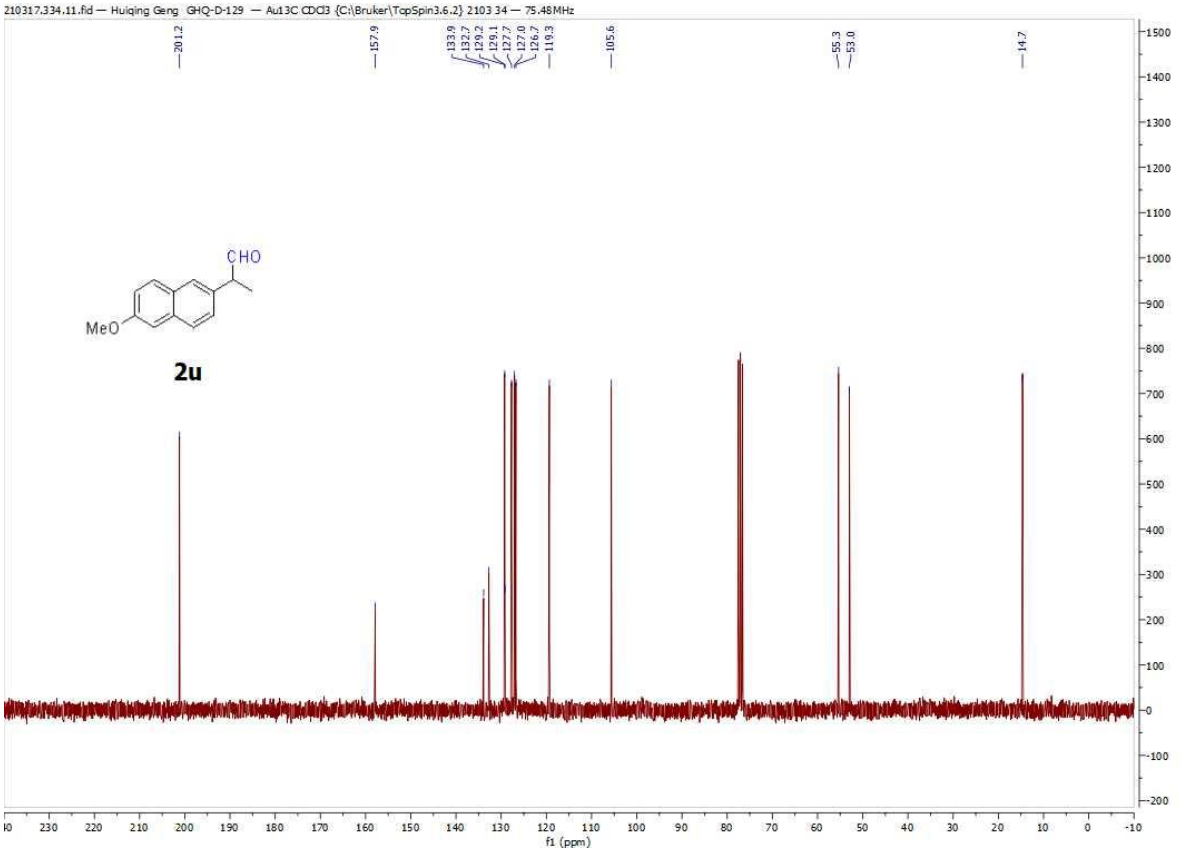

210330.f316.10.fid — Huiqing Geng GHQ-D-151-1 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 16 — 300.20MHz
CF3
CHO
2s
f1 (ppm)

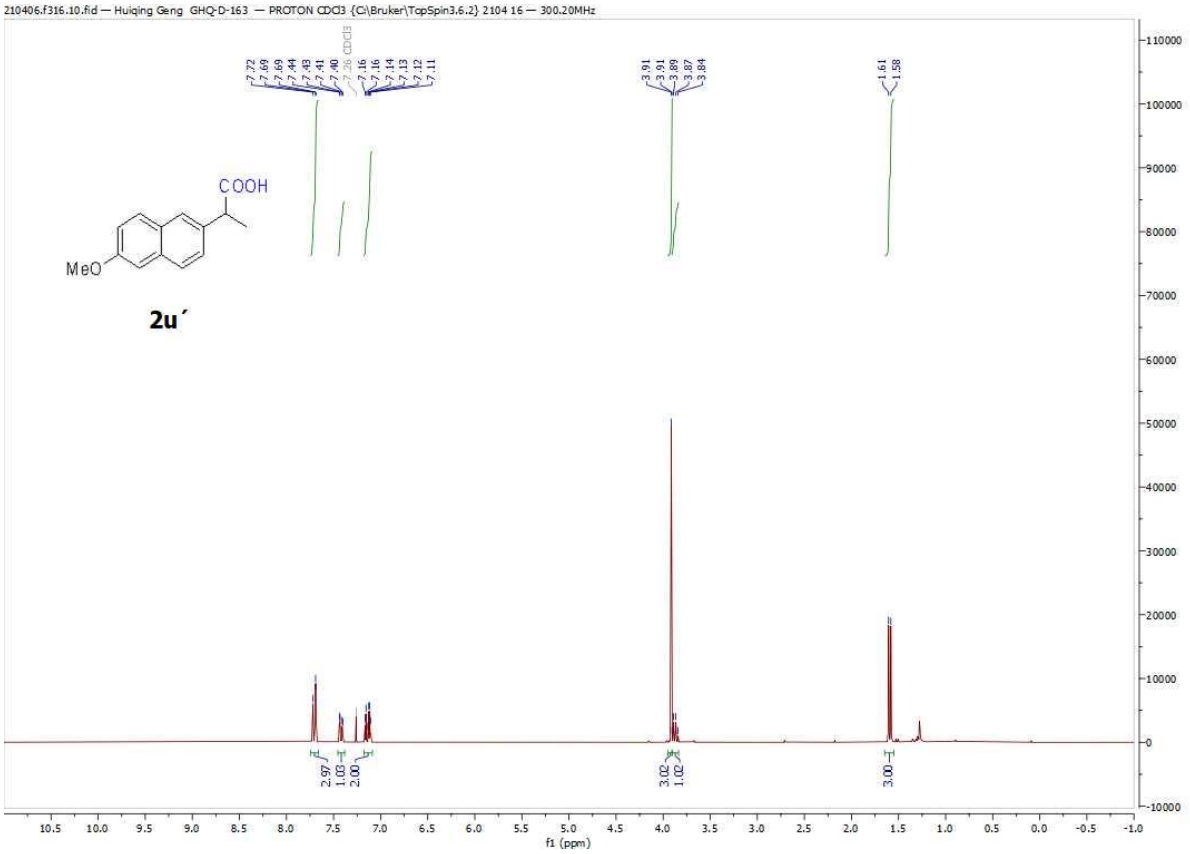

210330.f316.11.fid — Huiqing Geng GHQ-D-151-1 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 16
CF3
CHO
2s
f1 (ppm)

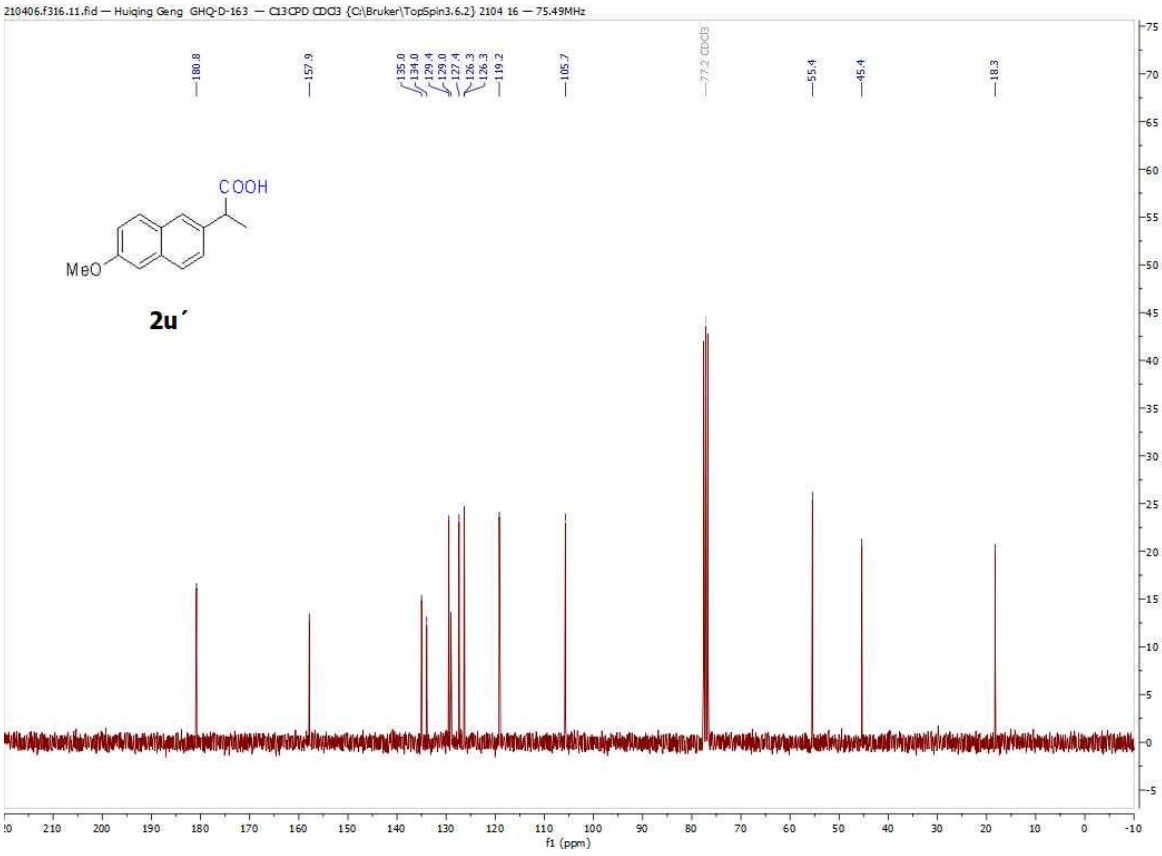

210312.312.10.fid — Huiqing Geng GHQ-D-116 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 12 — 300.13MHz
CHO
2t
f1 (ppm)

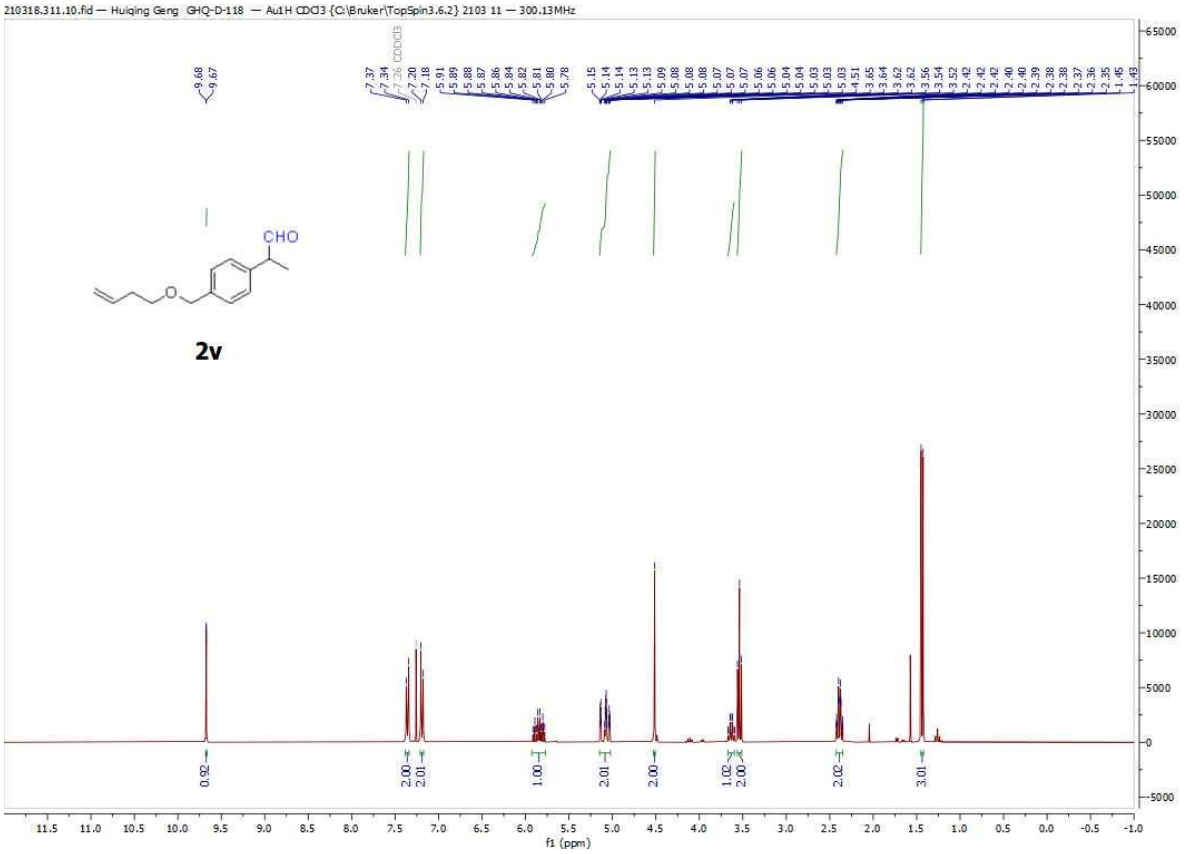

210312.312.11.fid — Huiqing Geng GHQ-D-116 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 12 — 75.48MHz
CHO
2t
f1 (ppm)

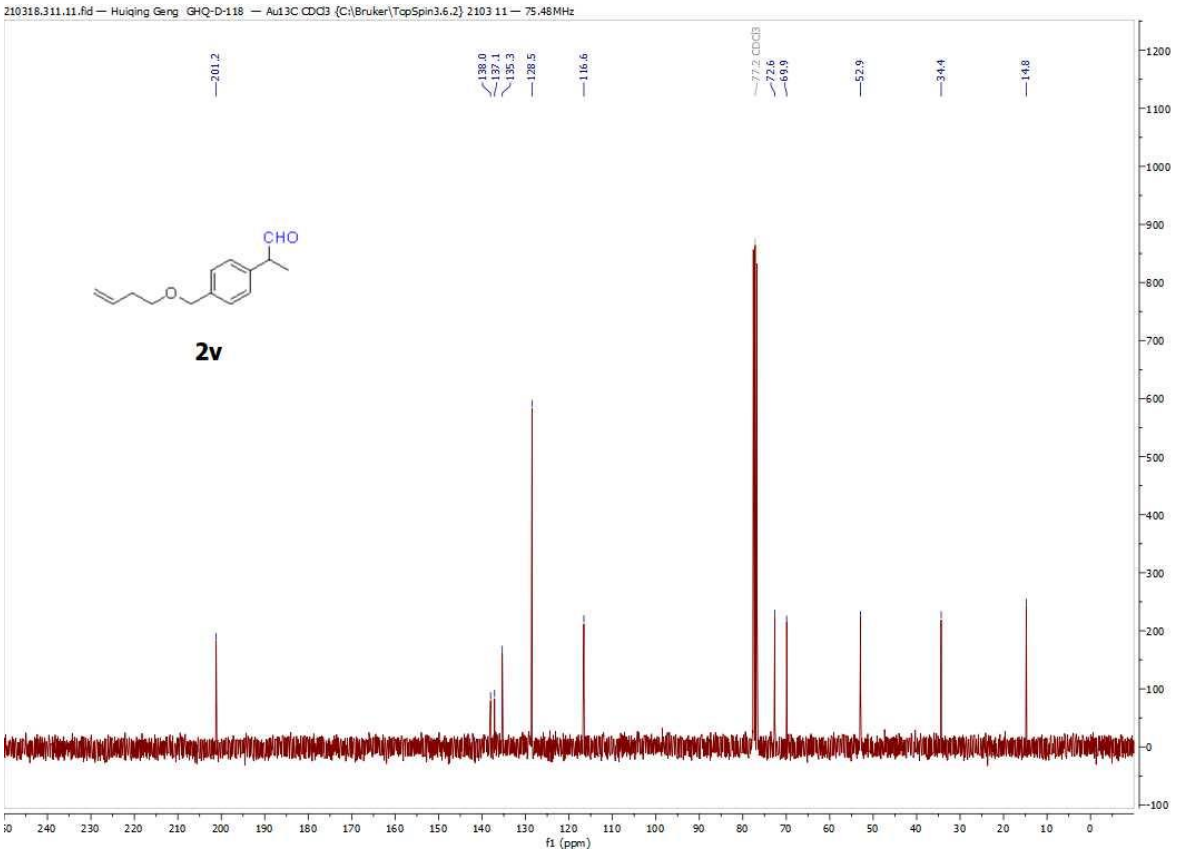

210317.334.10.fid — Huiqing Geng GHQ-D-129 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 34 — 300.13MHz
9.75
9.75
7.77
7.74
7.71
7.61
7.60
7.30
7.30
7.27
7.26 CDCl3
7.20
7.19
7.17
7.16
7.14
7.13
3.93
3.81
3.80
3.78
3.78
3.76
3.75
3.74
3.73
1.54
1.52
CHO
MeO
2u
0.86
2.02
1.00
1.02
2.00
3.01
1.00
3.01
f1 (ppm)

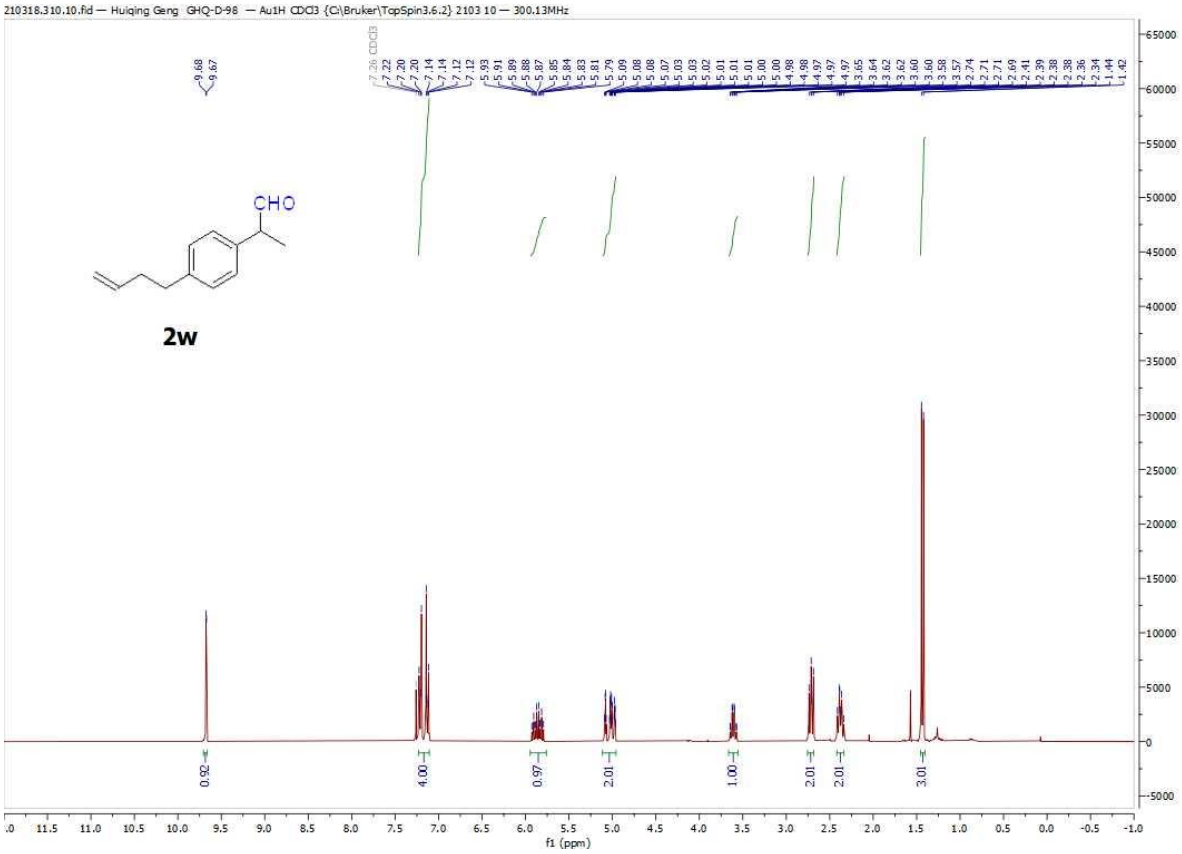

210317.334.11.fid — Huiqing Geng GHQ-D-129 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 34 — 75.48MHz
201.2
157.9
133.9
132.7
129.2
129.1
127.7
127.0
126.7
119.3
105.6
55.3
53.0
14.7
CHO
MeO
2u
f1 (ppm)

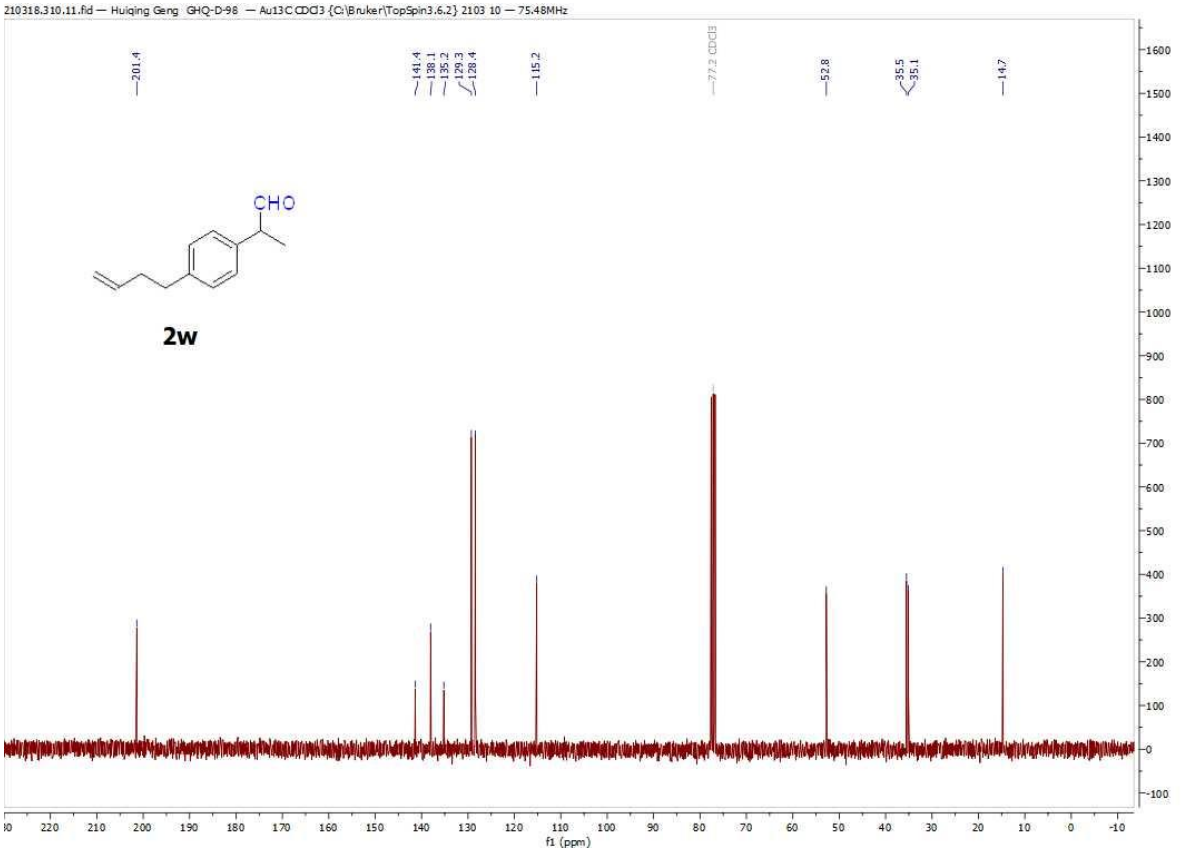

210406.f316.10.fid — Huiqing Geng GHQ-D-163 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2104 16 — 300.20MHz
COOH
MeO
2u´
f1 (ppm)

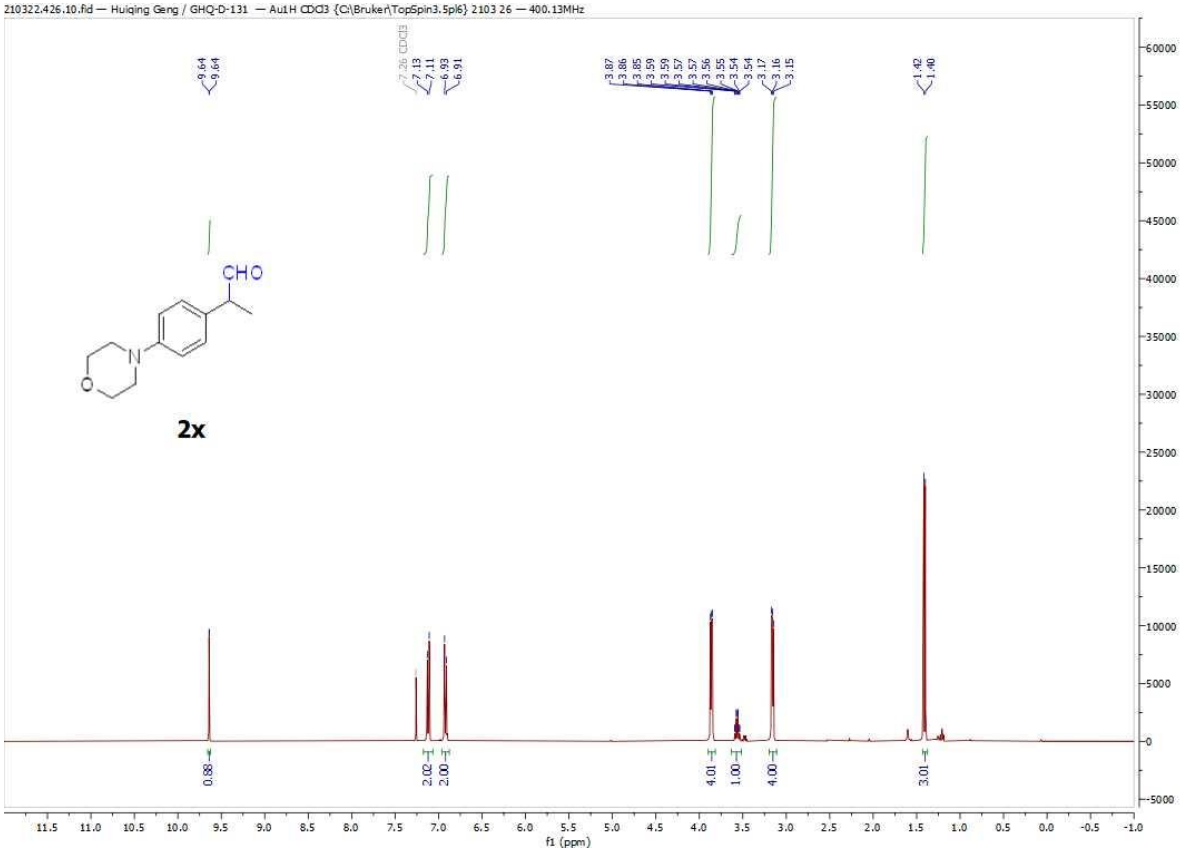

210406.f316.11.fid — Huiqing Geng GHQ-D-163 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2104 16 — 75.49MHz
COOH
MeO
2u´
f1 (ppm)

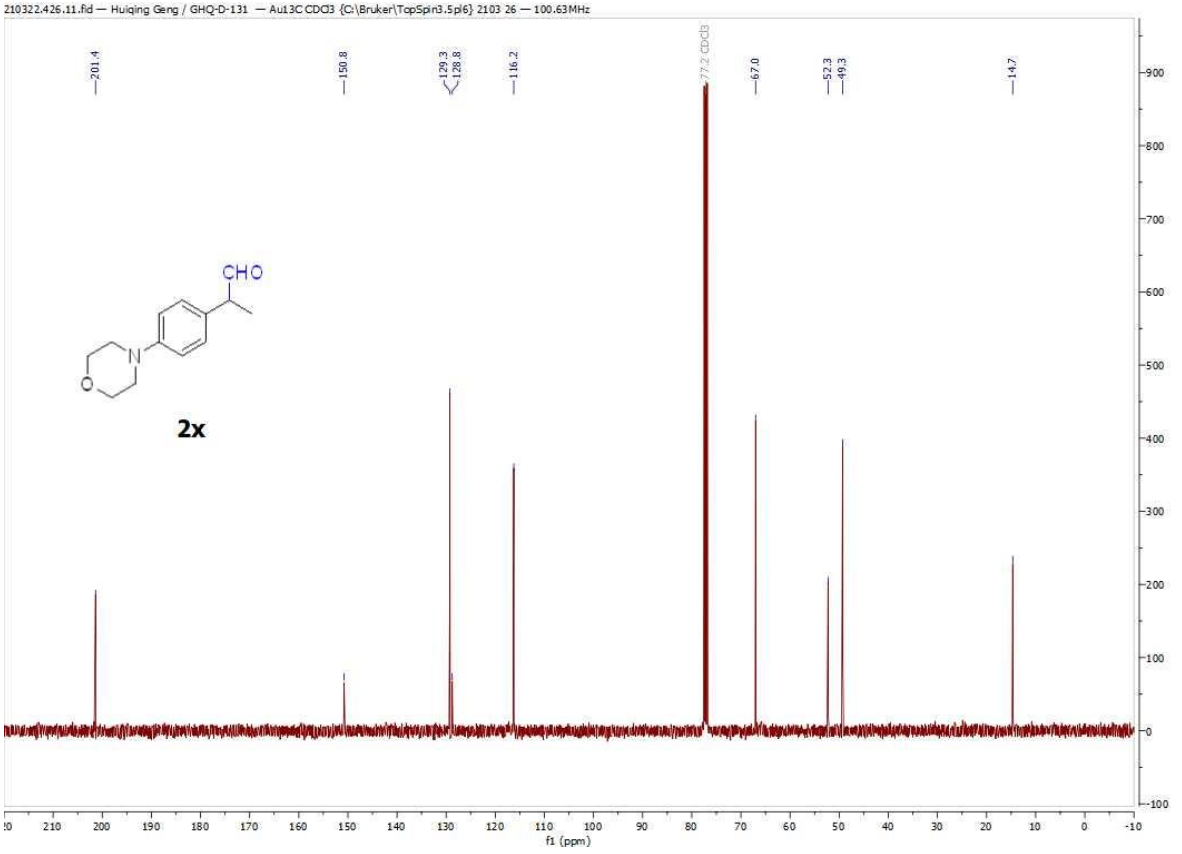

210318.311.10.fid — Huiqing Geng GHQ-D-118 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 11 — 300.13MHz
CHO
2v
f1 (ppm)

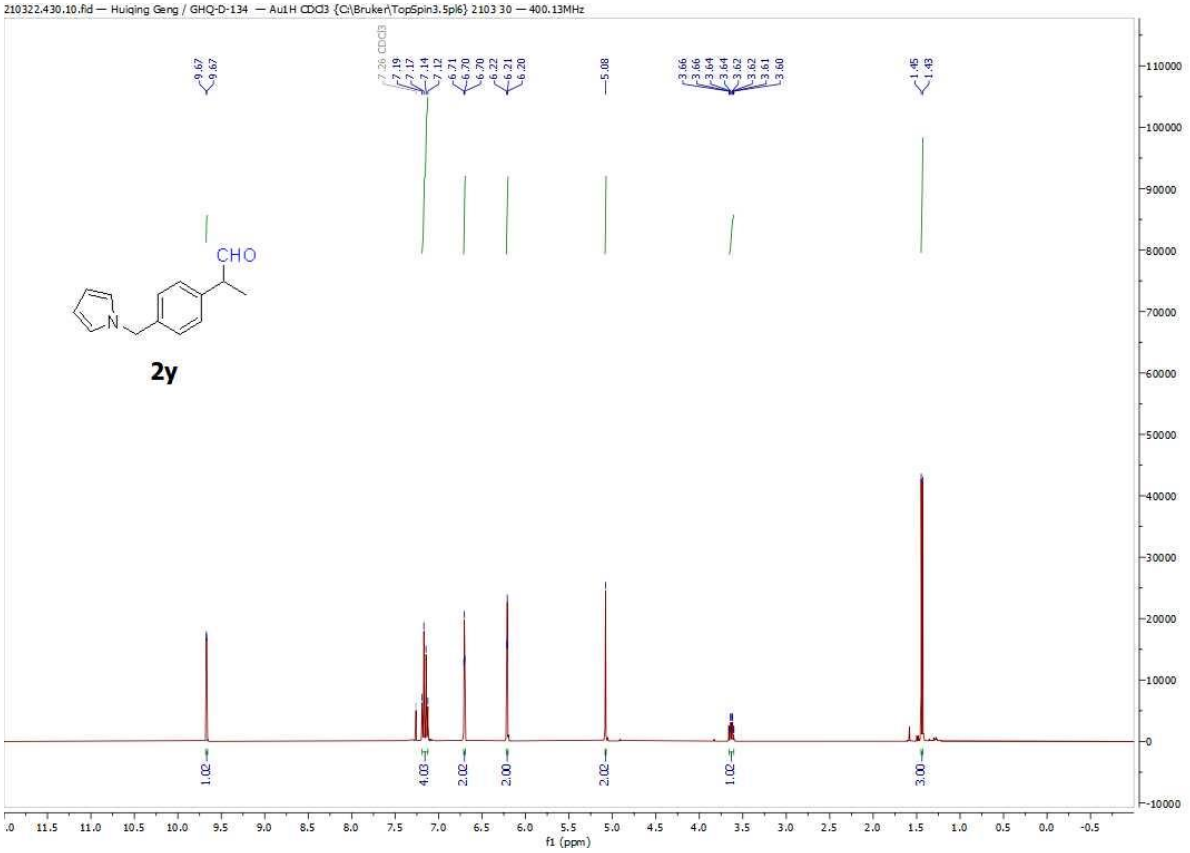

210318.311.11.fid — Huiqing Geng GHQ-D-118 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 11 — 75.48MHz
CHO
2v
f1 (ppm)

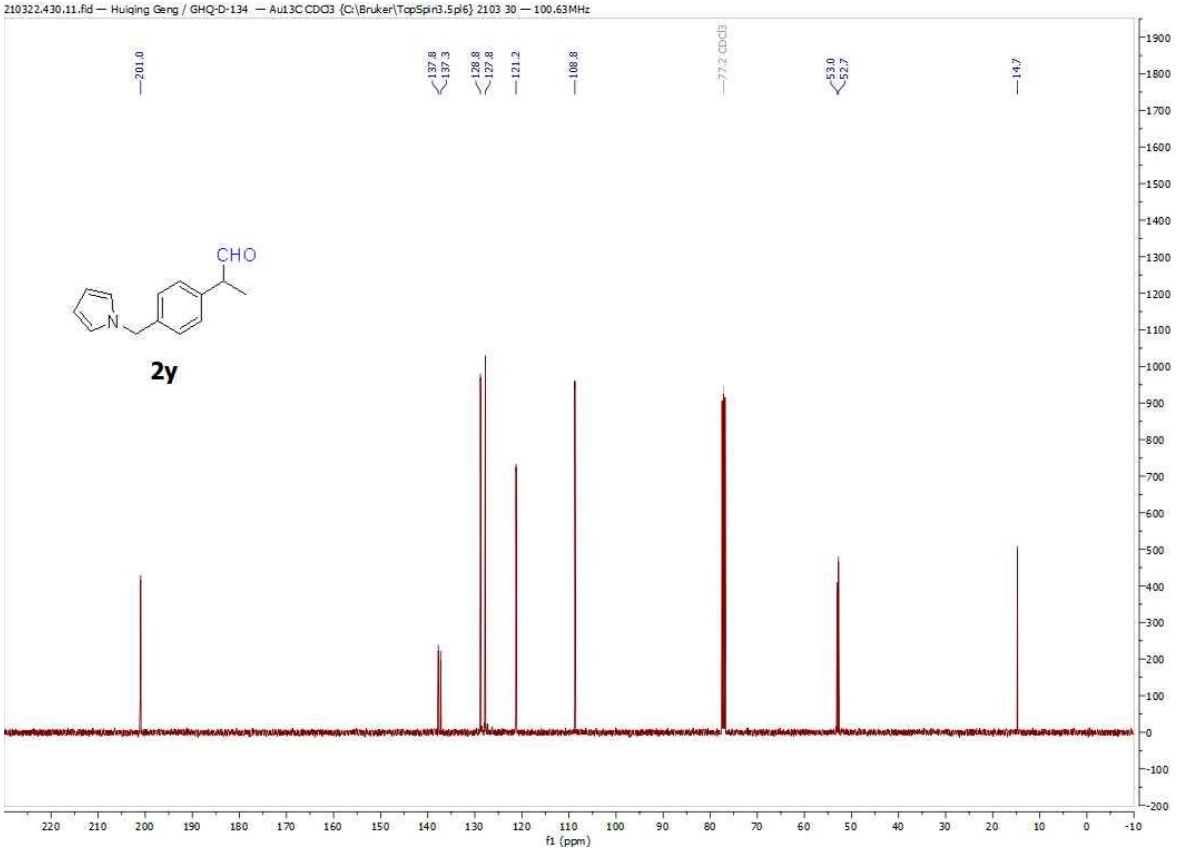

210318.310.10.fid — Huiqing Geng GHQ-D-98 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 10 — 300.13MHz
CHO
2w
f1 (ppm)

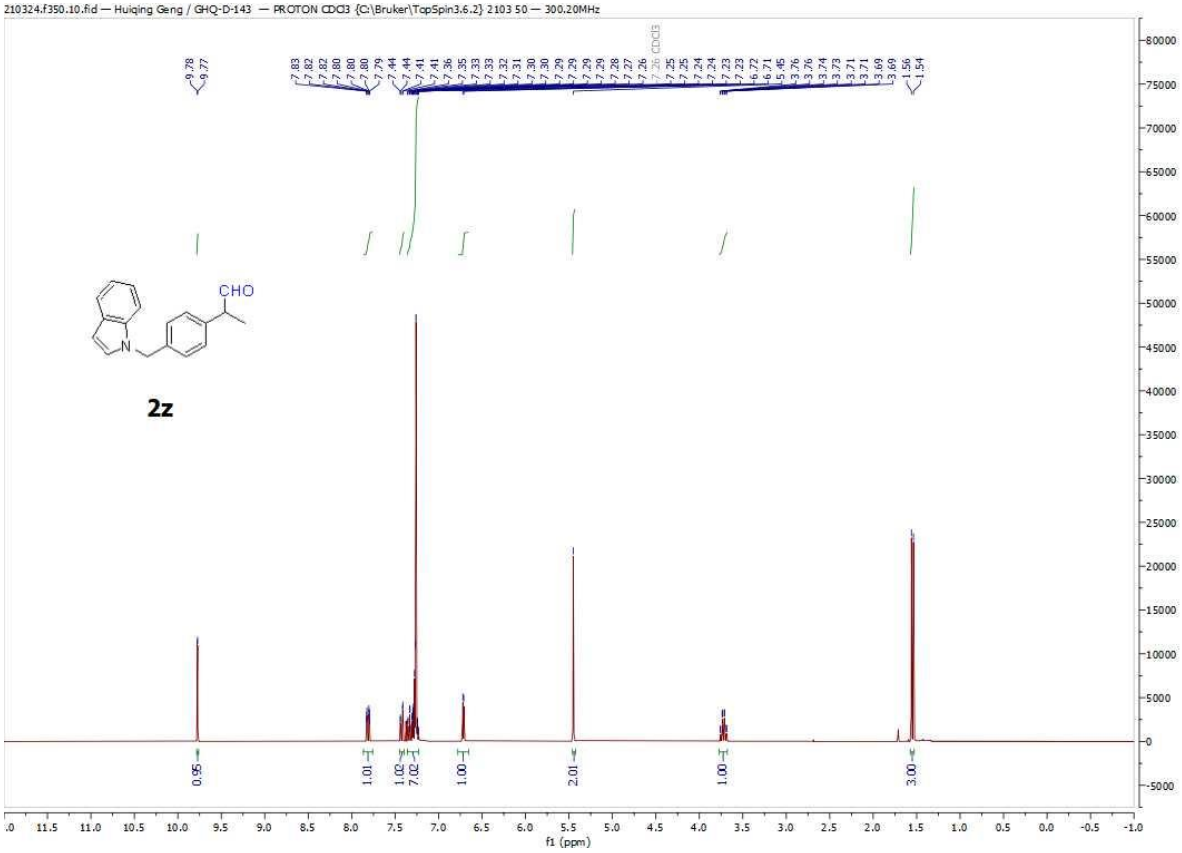

210318.310.11.fid — Huiqing Geng GHQ-D-98 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 10 — 75.48MHz
CHO
2w
f1 (ppm)

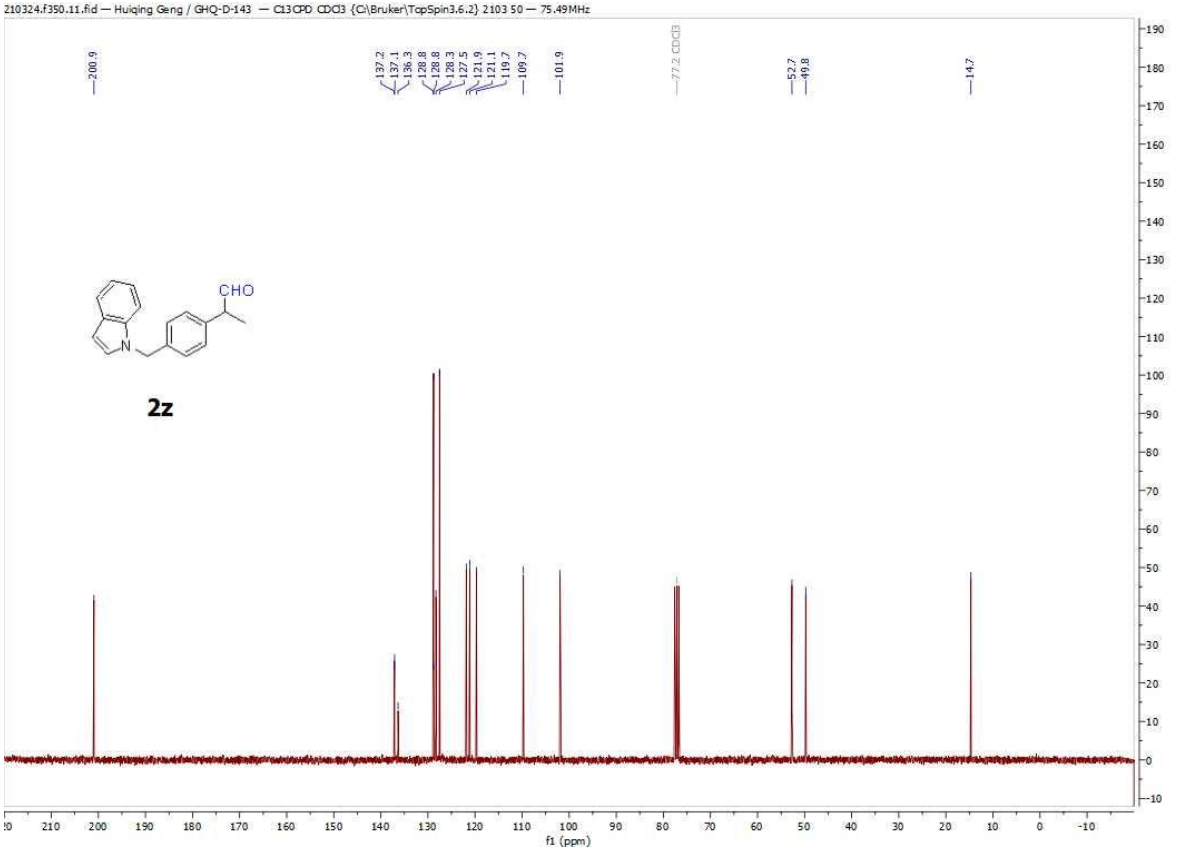

210322.426.10.fid — Huiqing Geng / GHQ-D-131 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 26 — 400.13MHz
CHO
2x
f1 (ppm)

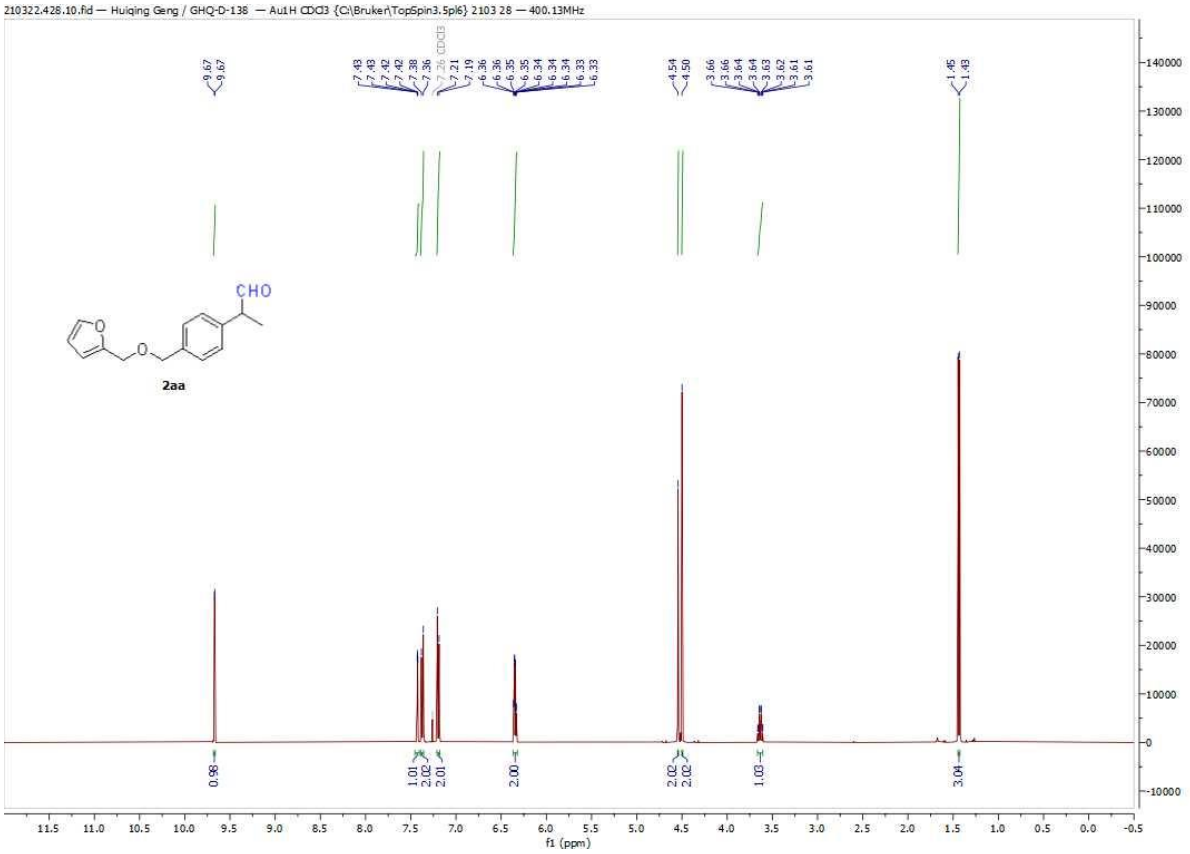

210322.426.11.fid — Huiqing Geng / GHQ-D-131 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 26 — 100.63MHz
CHO
2x
f1 (ppm)

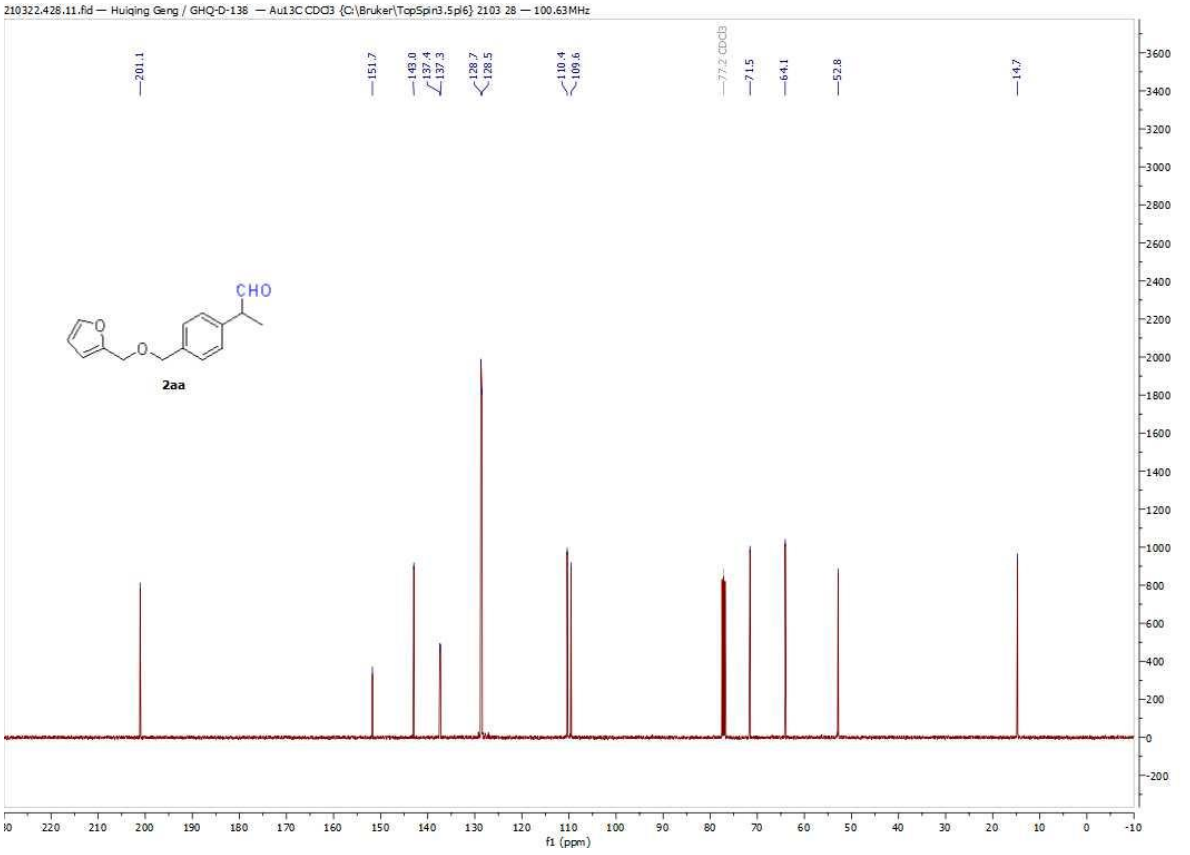

210322.430.10.fid — Huiqing Geng / GHQ-D-134 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 30 — 400.13MHz
9.67
9.67
7.26 CDCl3
7.19
7.17
7.14
7.12
6.71
6.70
6.70
6.22
6.21
6.20
5.08
3.66
3.66
3.64
3.64
3.62
3.62
3.61
3.60
1.45
1.43
CHO
N
2y
1.02
4.03
2.02
2.00
2.02
1.02
3.00
f1 (ppm)

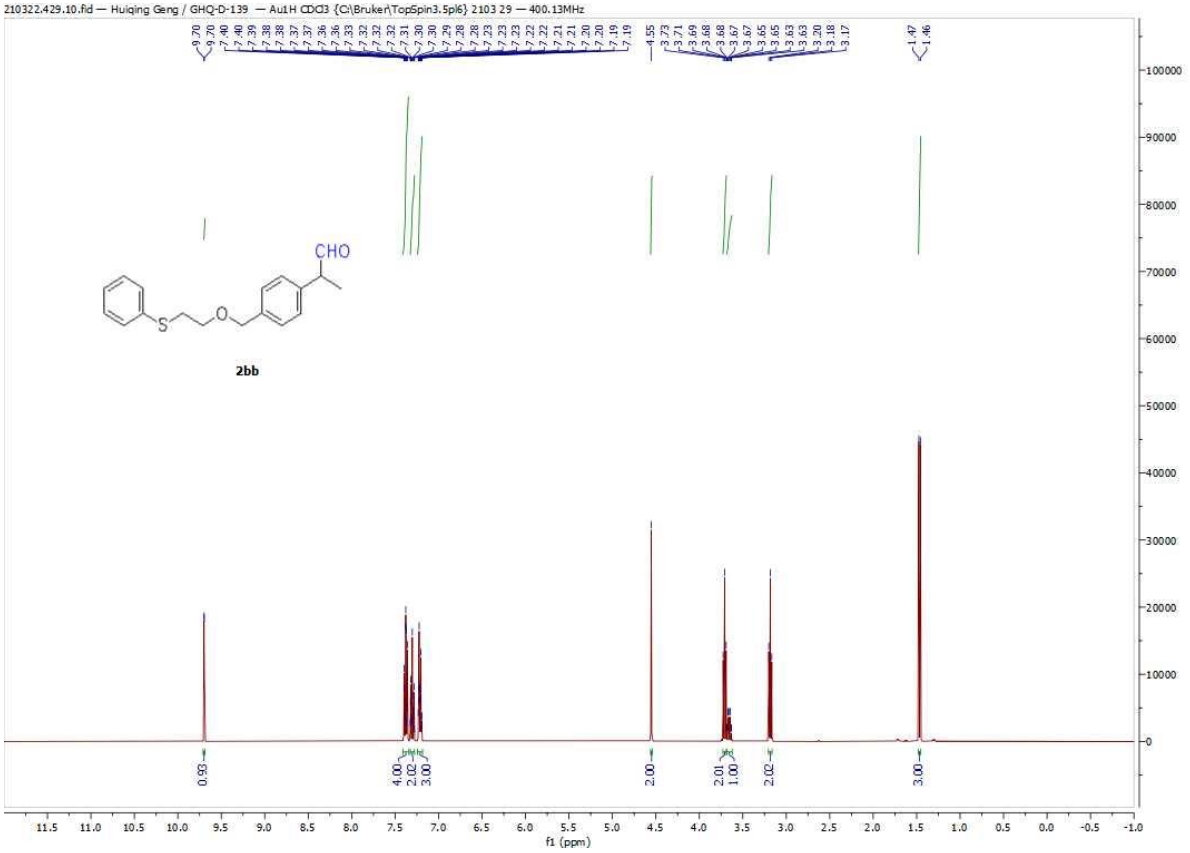

210322.430.11.fid — Huiqing Geng / GHQ-D-134 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 30 — 100.63MHz
201.0
137.8
137.3
128.8
127.8
121.2
108.8
77.2 CDCl3
53.0
52.7
14.7
CHO
N
2y
f1 (ppm)

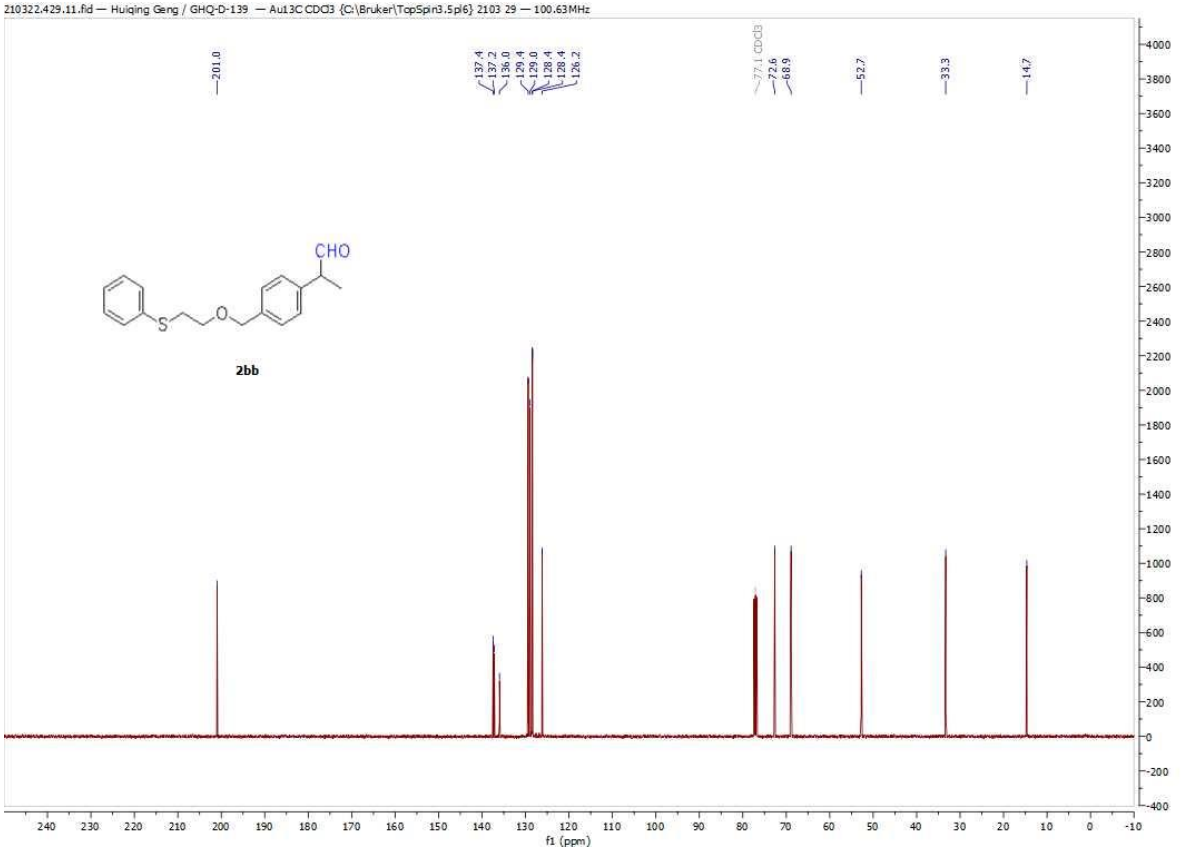

210324.f350.10.fid — Huiqing Geng / GHQ-D-143 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 50 — 300.20MHz
CHO
2z
f1 (ppm)

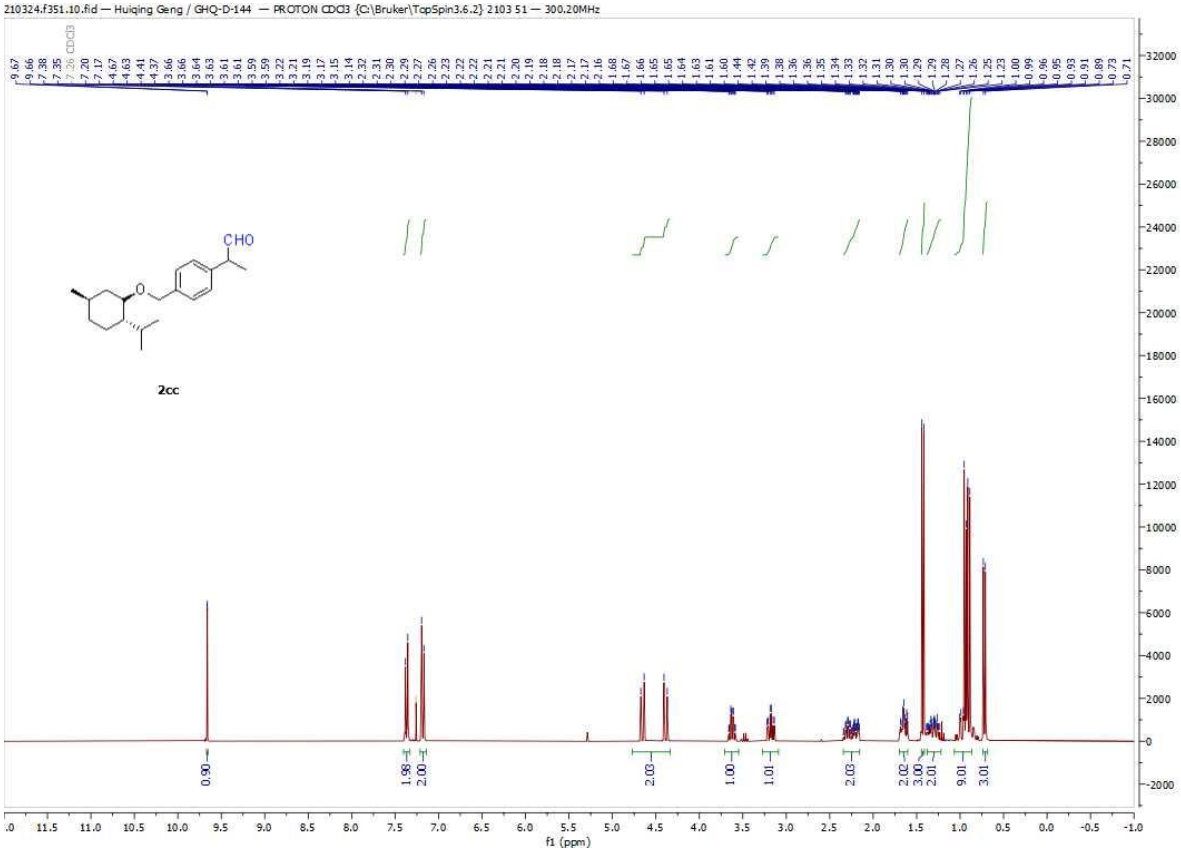

210324.f350.11.fid — Huiqing Geng / GHQ-D-143 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 50 — 75.49MHz
CHO
2z
f1 (ppm)

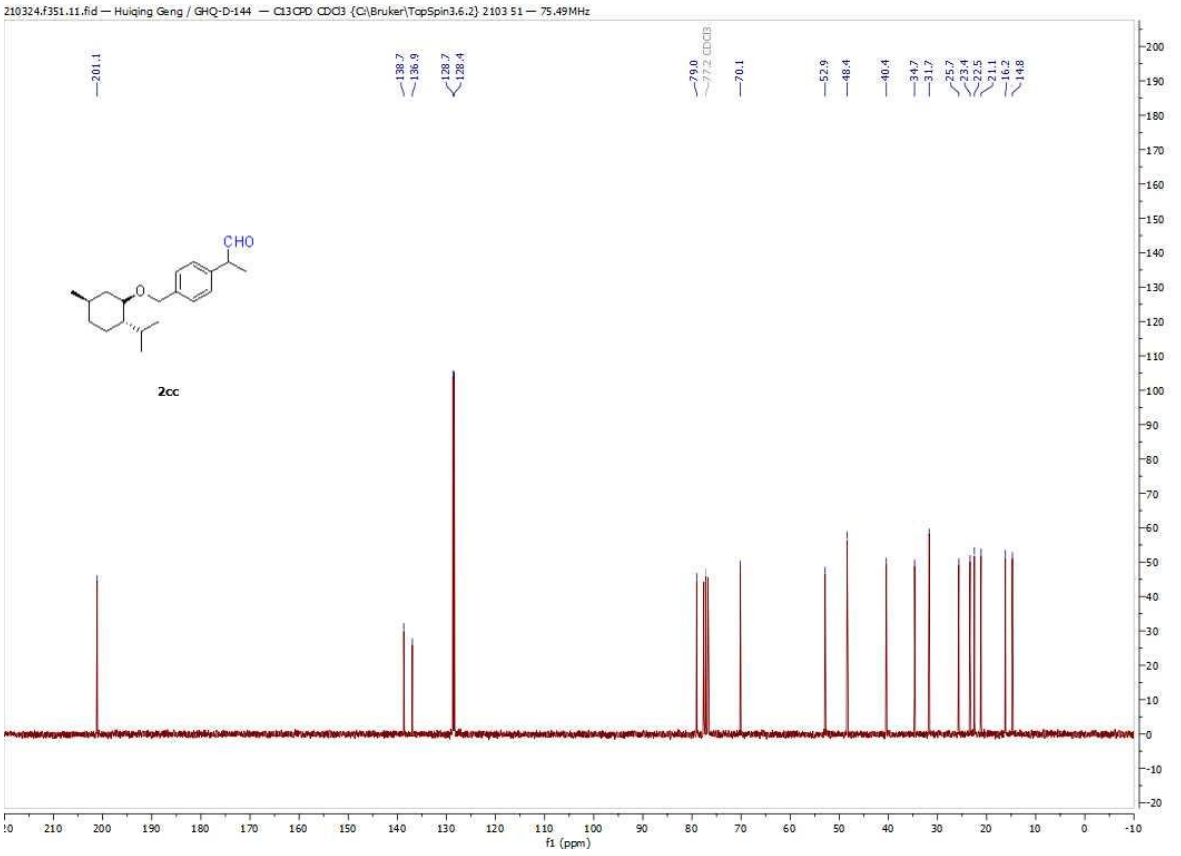

210322.428.10.fid — Huiqing Geng / GHQ-D-138 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 28 — 400.13MHz
CHO
2aa
f1 (ppm)

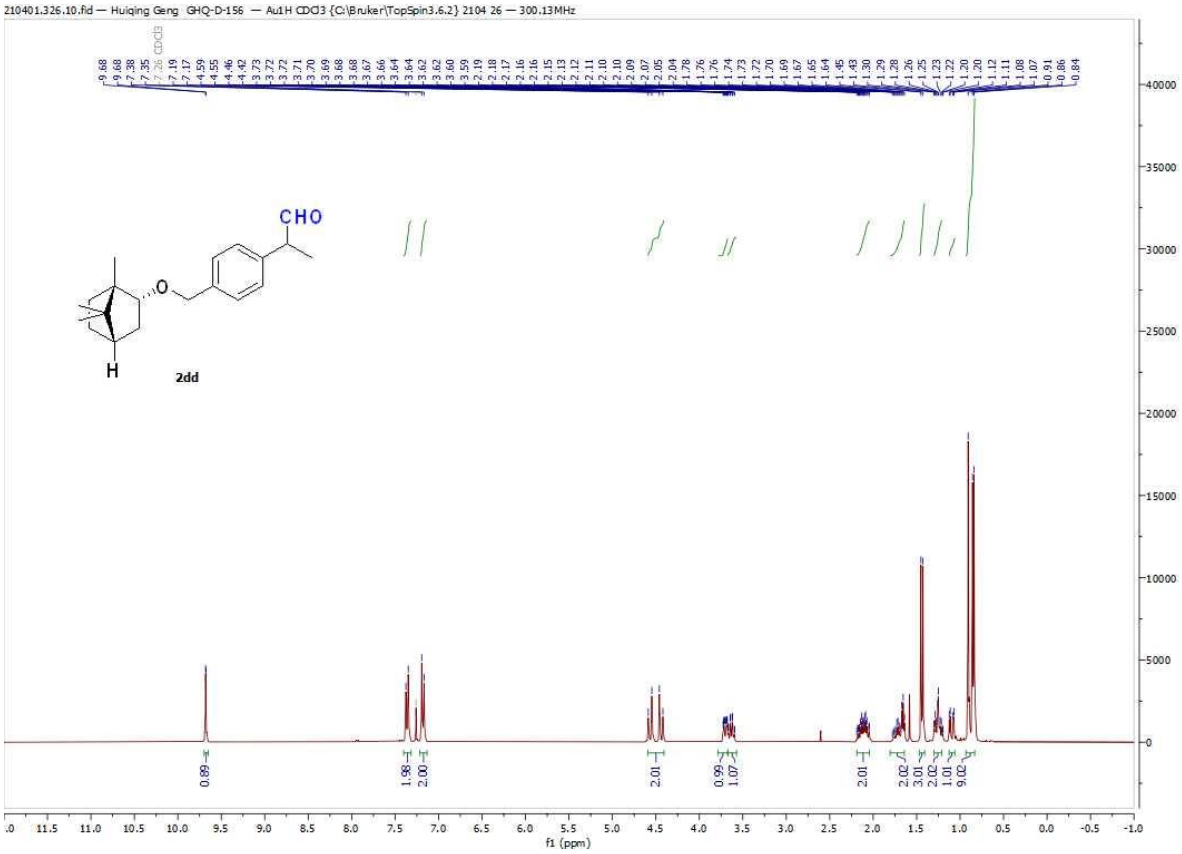

210322.428.11.fid — Huiqing Geng / GHQ-D-138 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 28 — 100.63MHz
CHO
2aa
f1 (ppm)

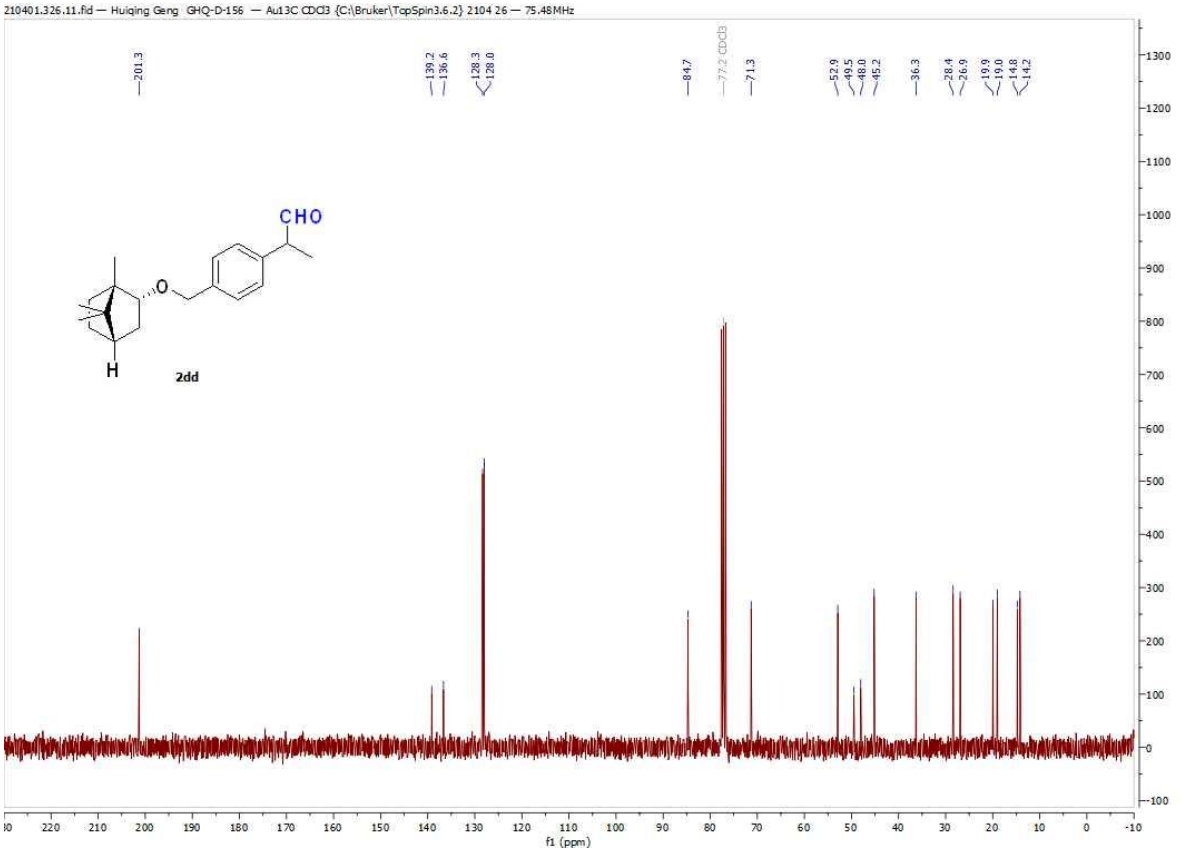

210322.429.10.fid — Huiqing Geng / GHQ-D-139 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 29 — 400.13MHz
CHO
S
O
2bb
f1 (ppm)

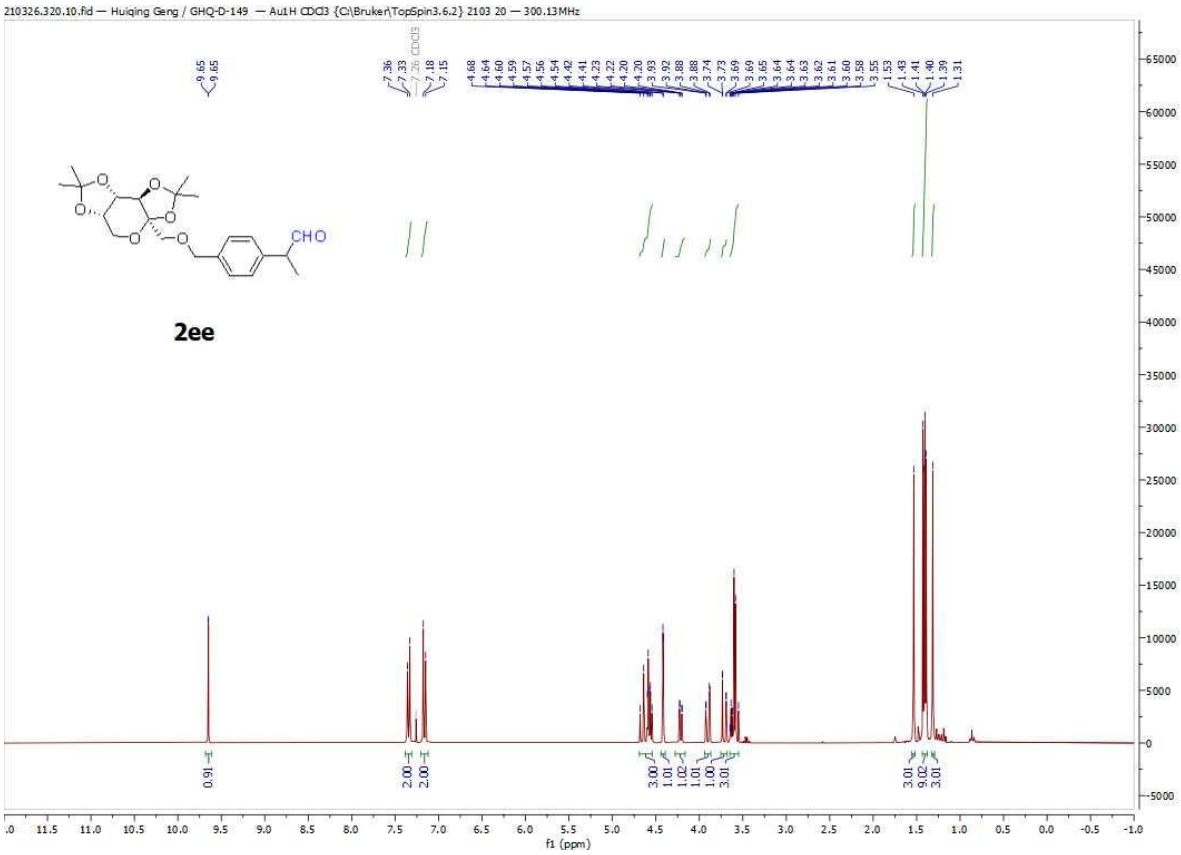

210322.429.11.fid — Huiqing Geng / GHQ-D-139 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2103 29 — 100.63MHz
201.0
137.4
137.2
136.0
129.4
129.0
128.4
128.4
126.2
77.1 CDCl3
72.6
68.9
52.7
33.3
14.7
CHO
S
O
2bb
f1 (ppm)

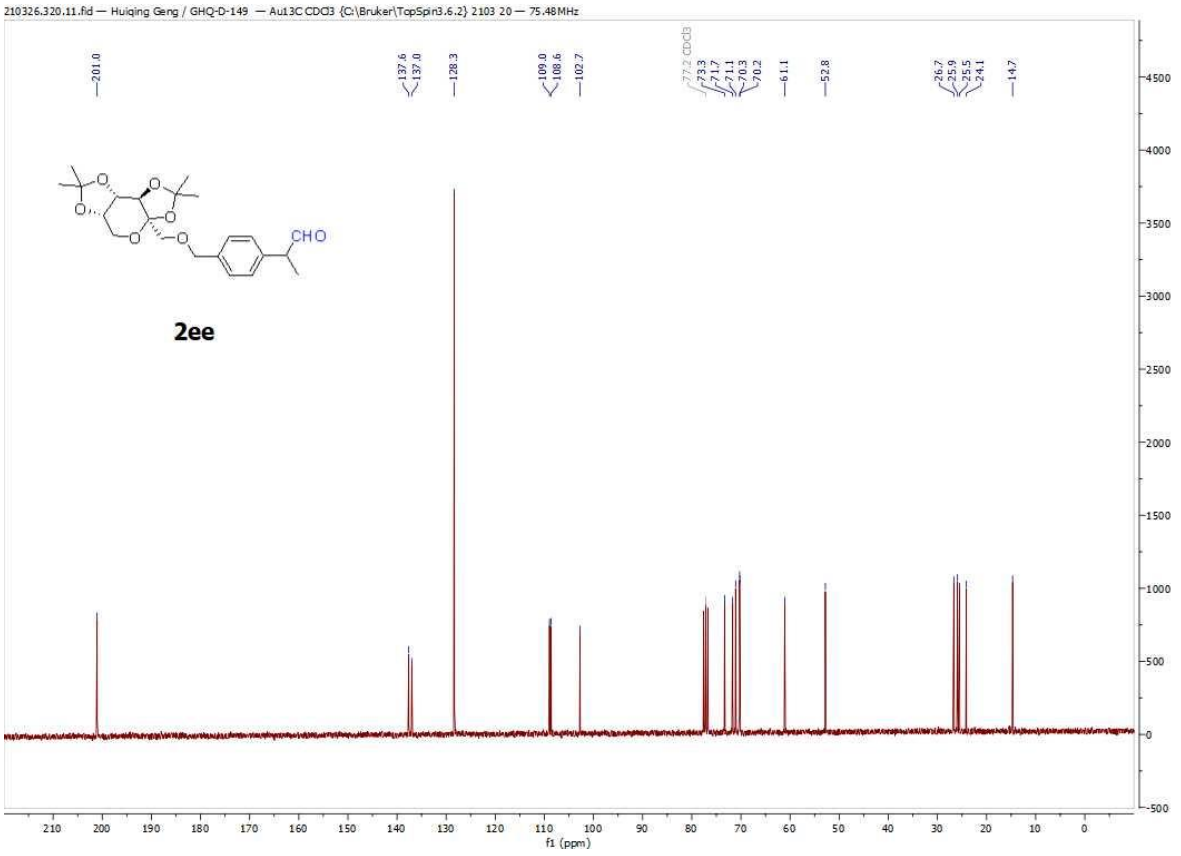

210324.f351.10.fid — Huiqing Geng / GHQ-D-144 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 51 — 300.20MHz
CHO
2cc
f1 (ppm)

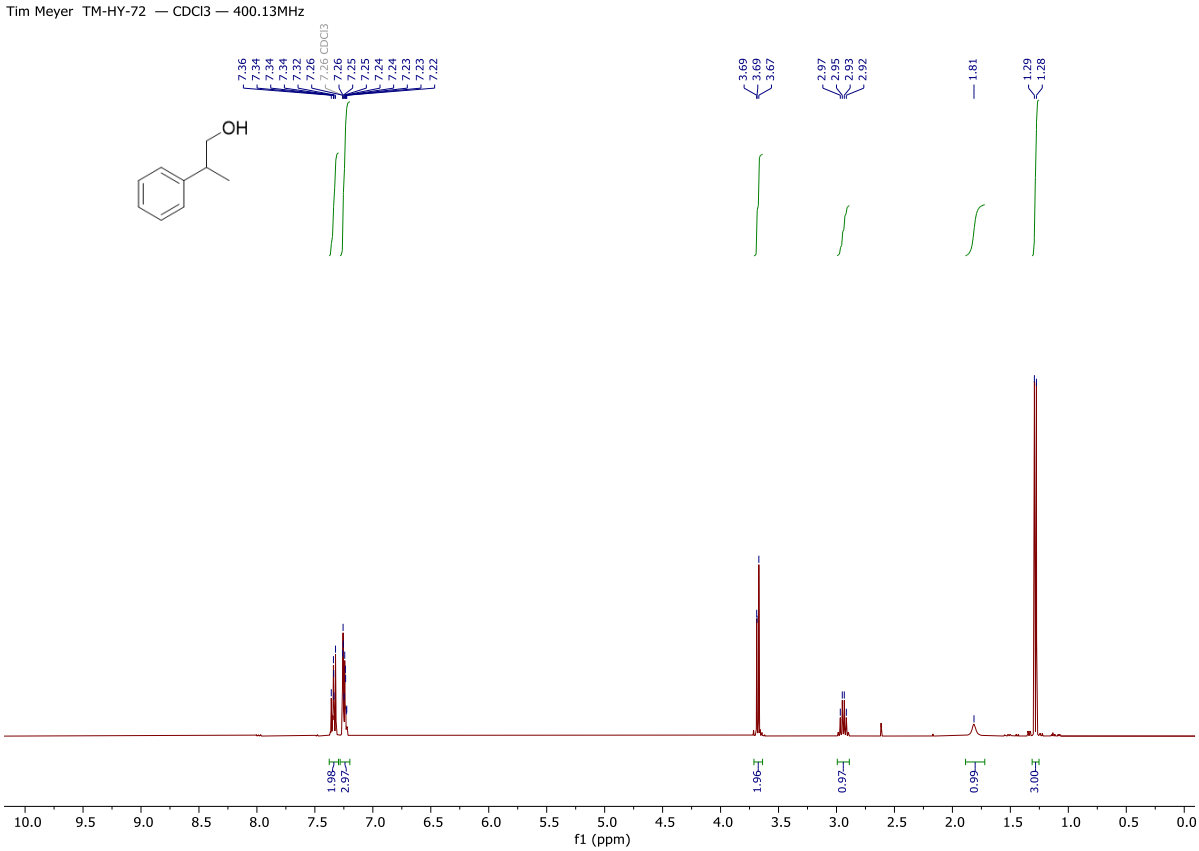

210324.f351.11.fid — Huiqing Geng / GHQ-D-144 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 51 — 75.49MHz
CHO
2cc
f1 (ppm)

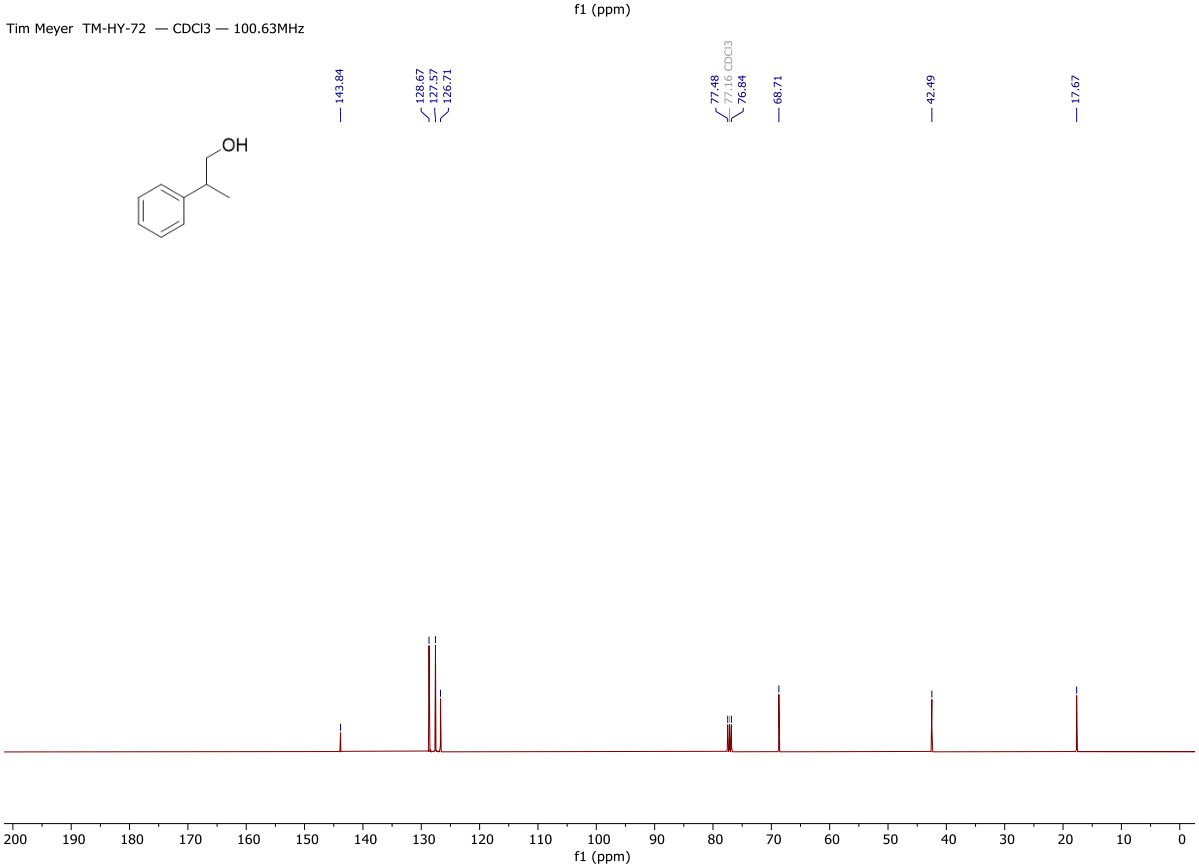

210401.326.10.fid — Huiqing Geng GHQ-D-156 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2104 26 — 300.13MHz
CHO
O
H
2dd
f1 (ppm)

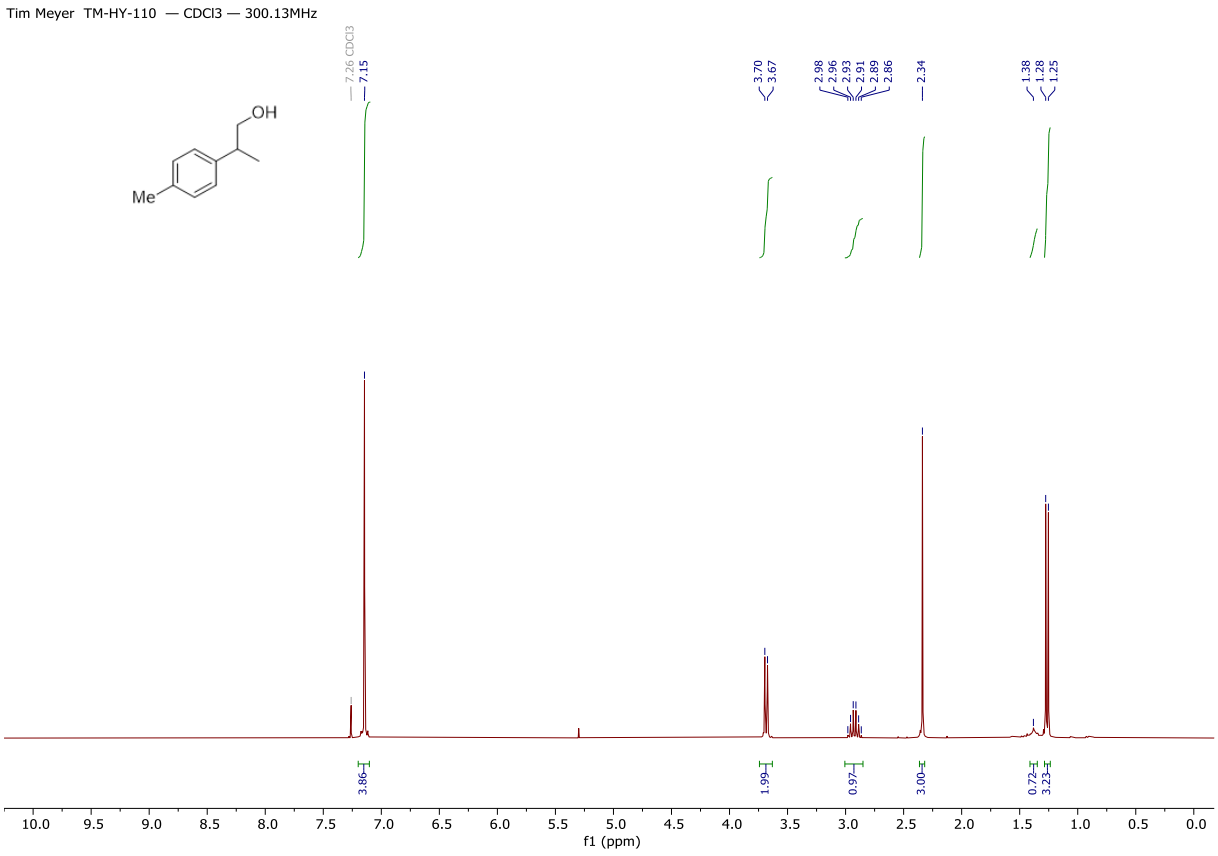

210401.326.11.fid — Huiqing Geng GHQ-D-156 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2104 26 — 75.48MHz
CHO
O
H
2dd
f1 (ppm)

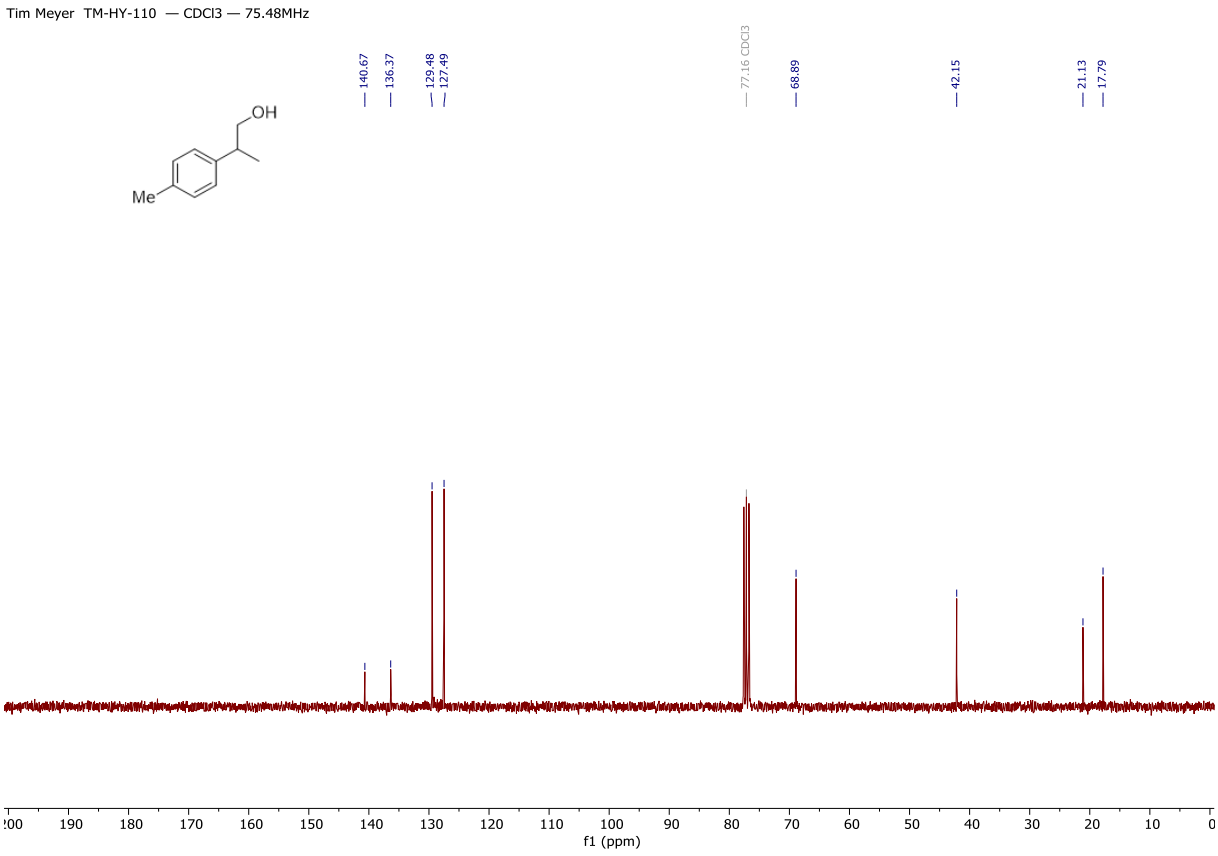

210326.320.10.fid — Huiqing Geng / GHQ-D-149 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 20 — 300.13MHz
CHO
2ee
f1 (ppm)


210326.320.11.fid — Huiqing Geng / GHQ-D-149 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2103 20 — 75.48MHz
CHO
2ee
f1 (ppm)

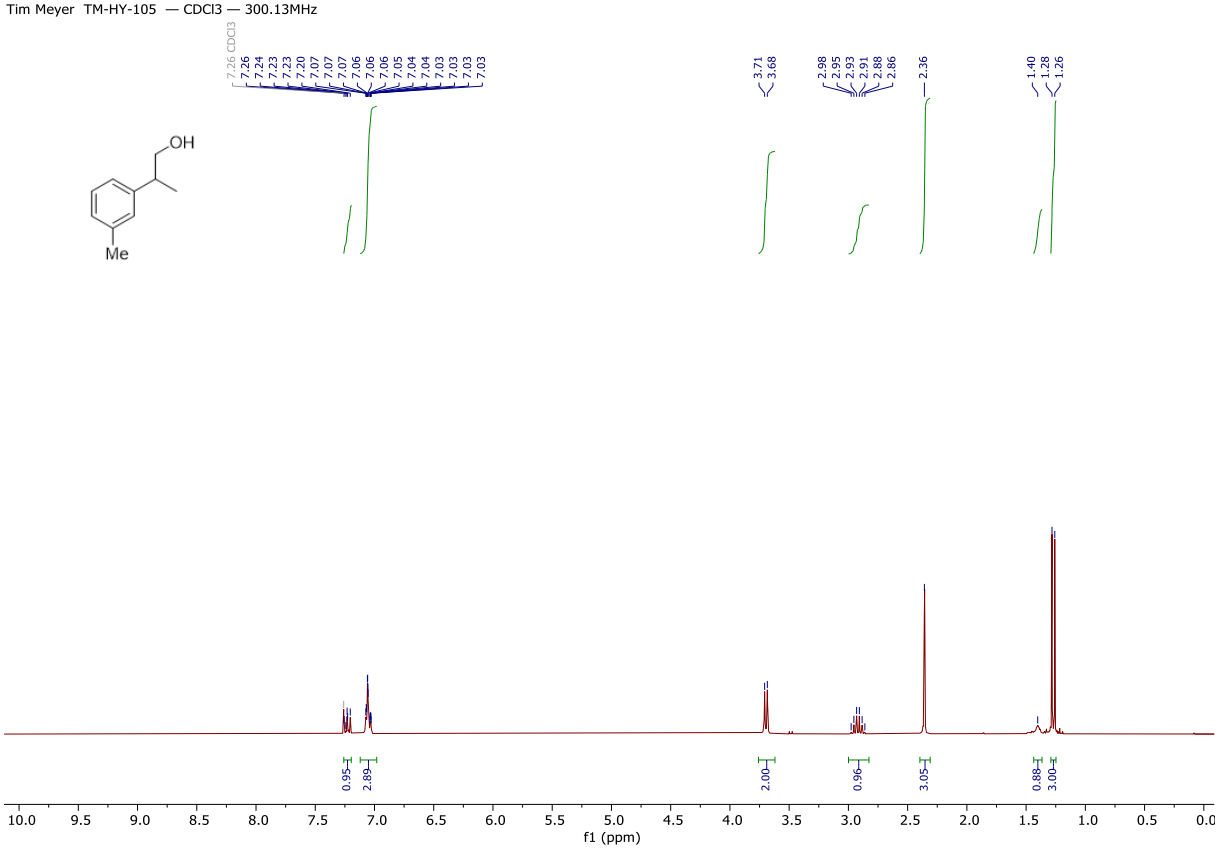

Tim Meyer TM-HY-72 — CDCl3 — 400.13MHz
7.36
7.34
7.34
7.34
7.32
7.26
7.26 CDCl3
7.26
7.25
7.25
7.24
7.24
7.23
7.23
7.22
3.69
3.69
3.67
2.97
2.95
2.93
2.92
1.81
1.29
1.28
OH
1.98
2.97
1.96
0.97
0.99
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Tim Meyer TM-HY-72 — CDCl3 — 100.63MHz
143.84
128.67
127.57
126.71
77.48
77.16 CDCl3
76.84
68.71
42.49
17.67
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

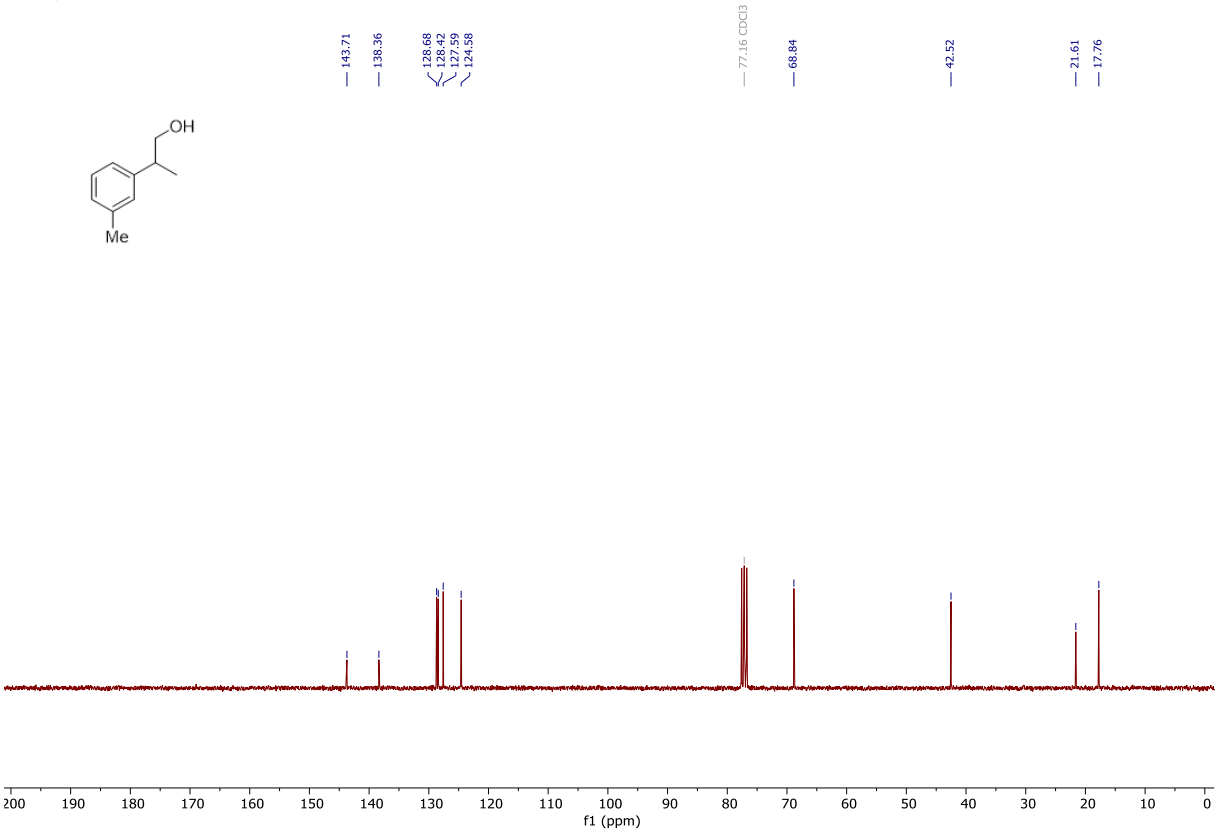

Tim Meyer TM-HY-110 — CDCl3 — 300.13MHz
OH
Me
7.26 CDCl3
7.15
3.70
3.67
2.98
2.96
2.93
2.91
2.89
2.86
2.34
1.38
1.28
1.25
3.86
1.99
0.97
3.00
0.72
3.23
f1 (ppm)


Tim Meyer TM-HY-110 — CDCl3 — 75.48MHz
OH
Me
140.67
136.37
129.48
127.49
77.16 CDCl3
68.89
42.15
21.13
17.79
f1 (ppm)

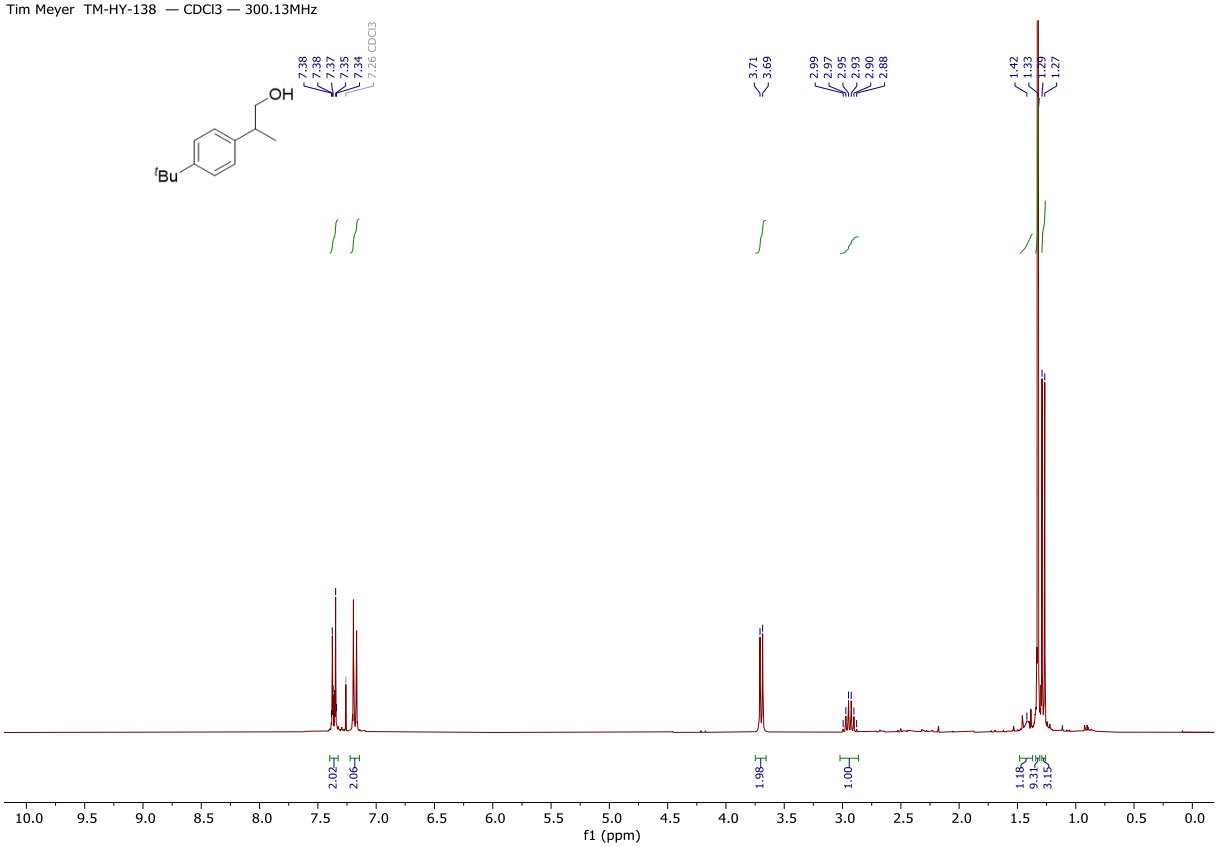
Tim Meyer TM-HY-105 — CDCl3 — 300.13MHz



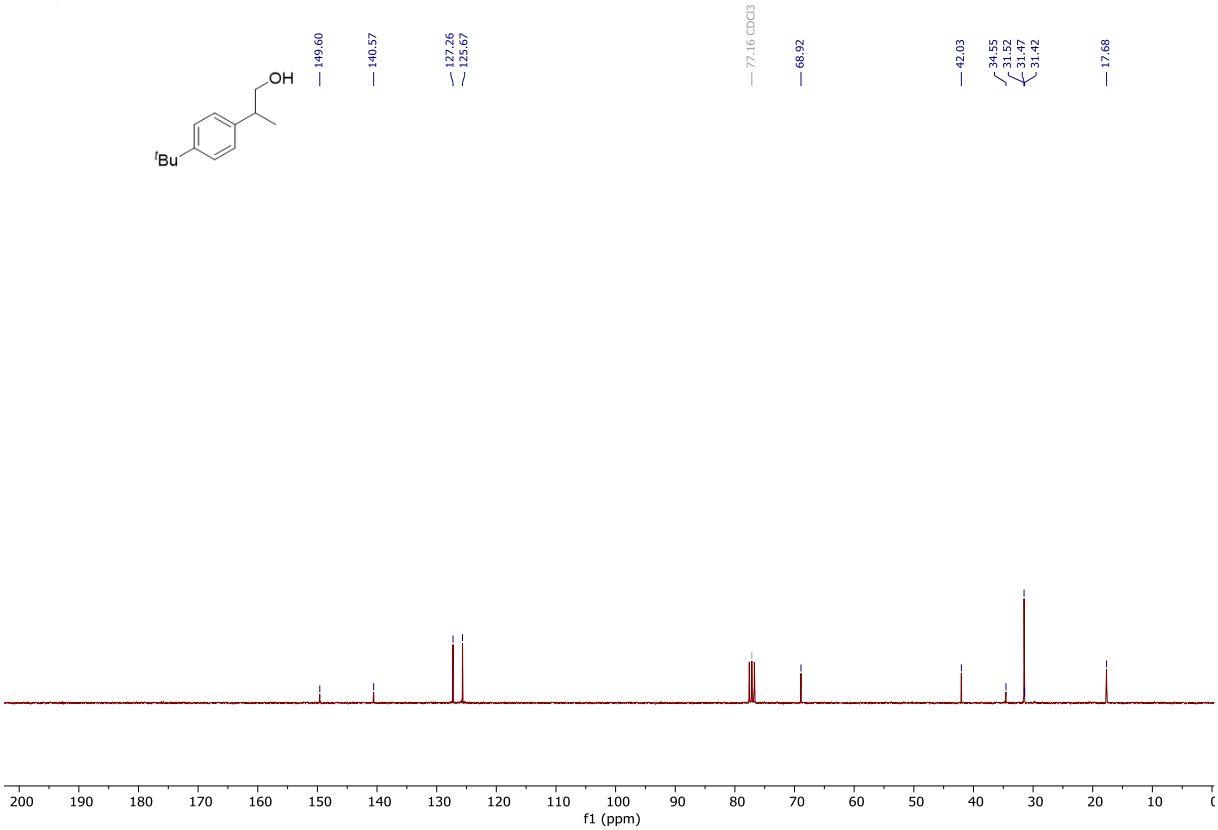
Tim Meyer TM-HY-105 — CDCl3 — 75.48MHz



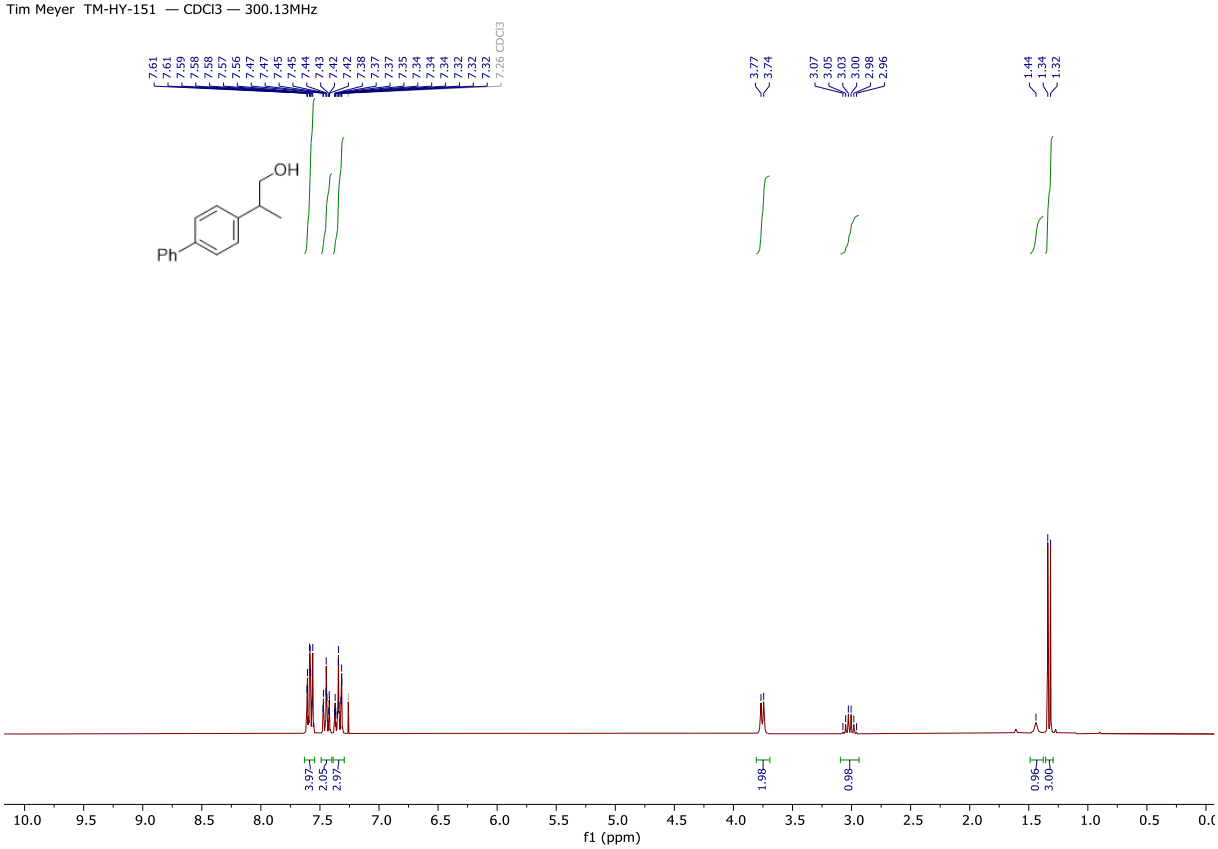
Tim Meyer TM-HY-138 — CDCl3 — 300.13MHz



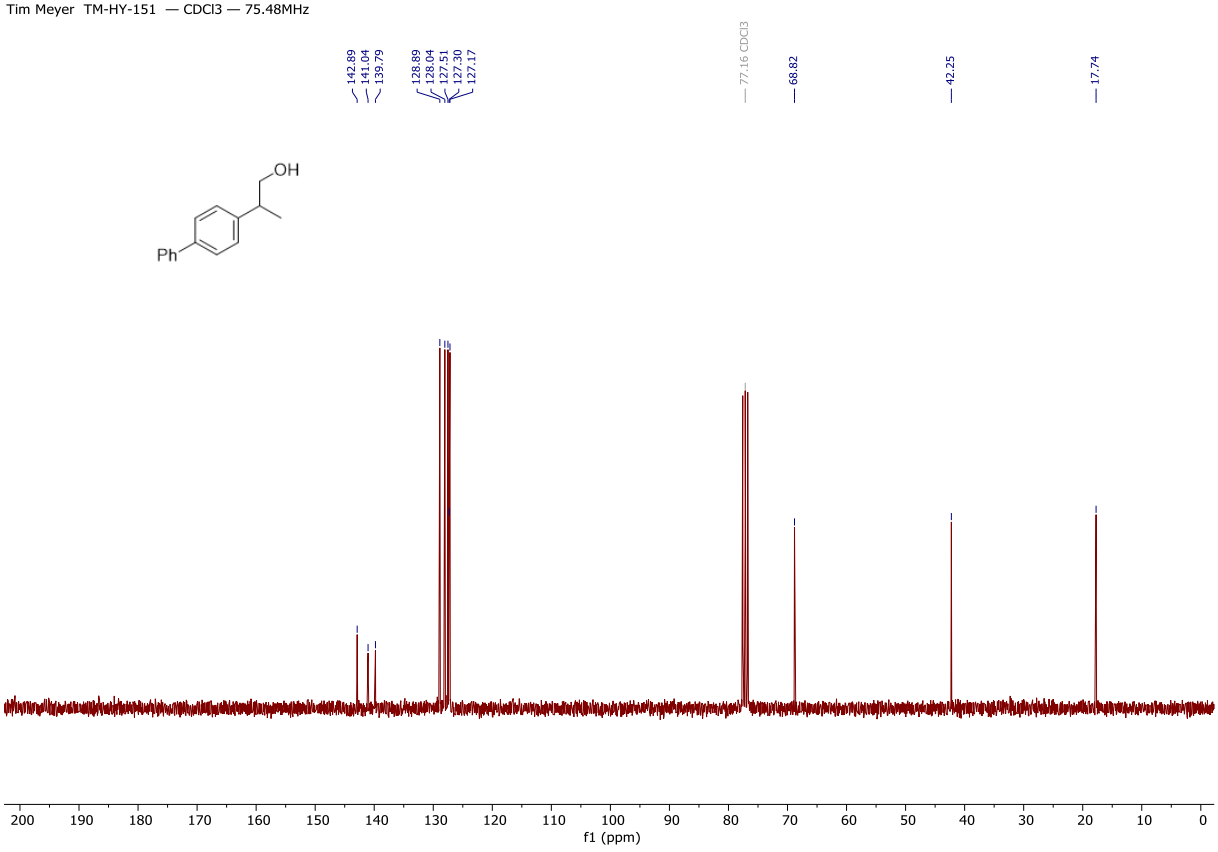
Tim Meyer TM-HY-138 — CDCl3 — 75.48MHz

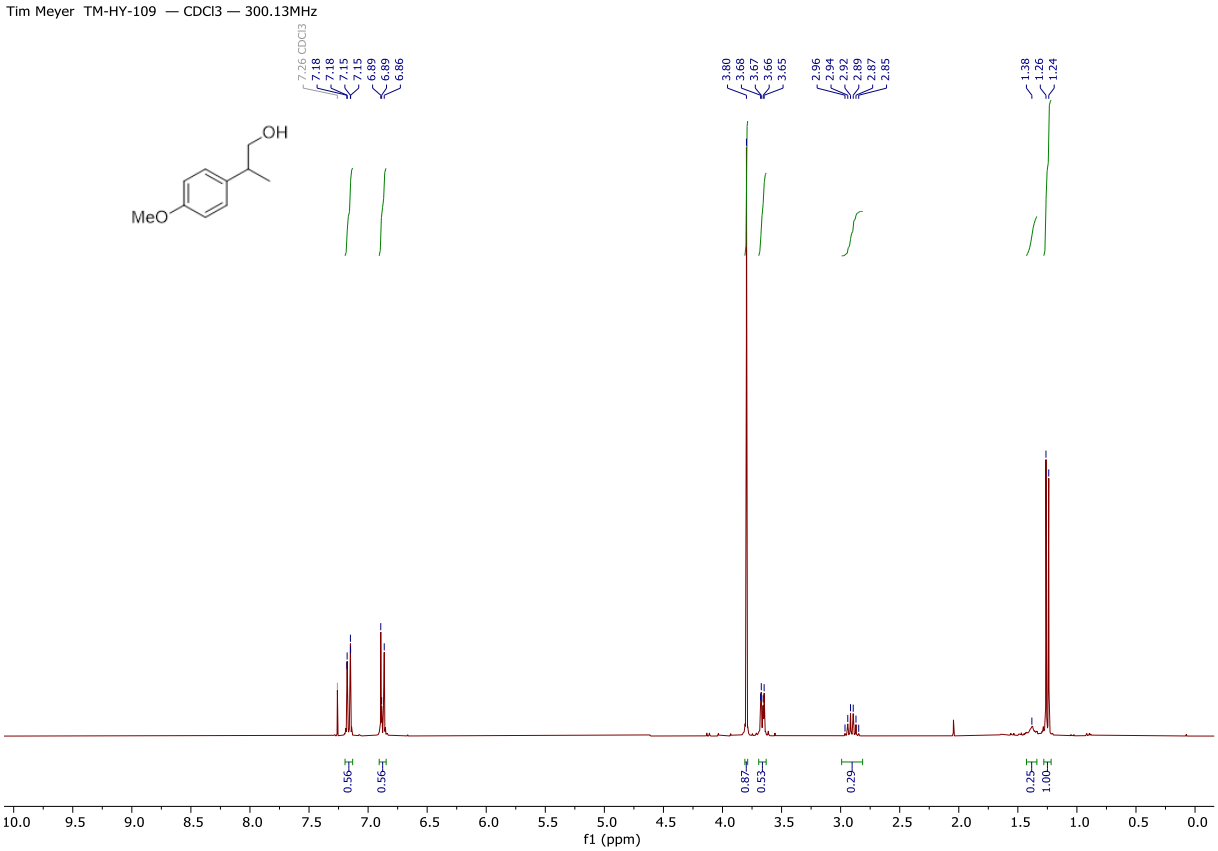


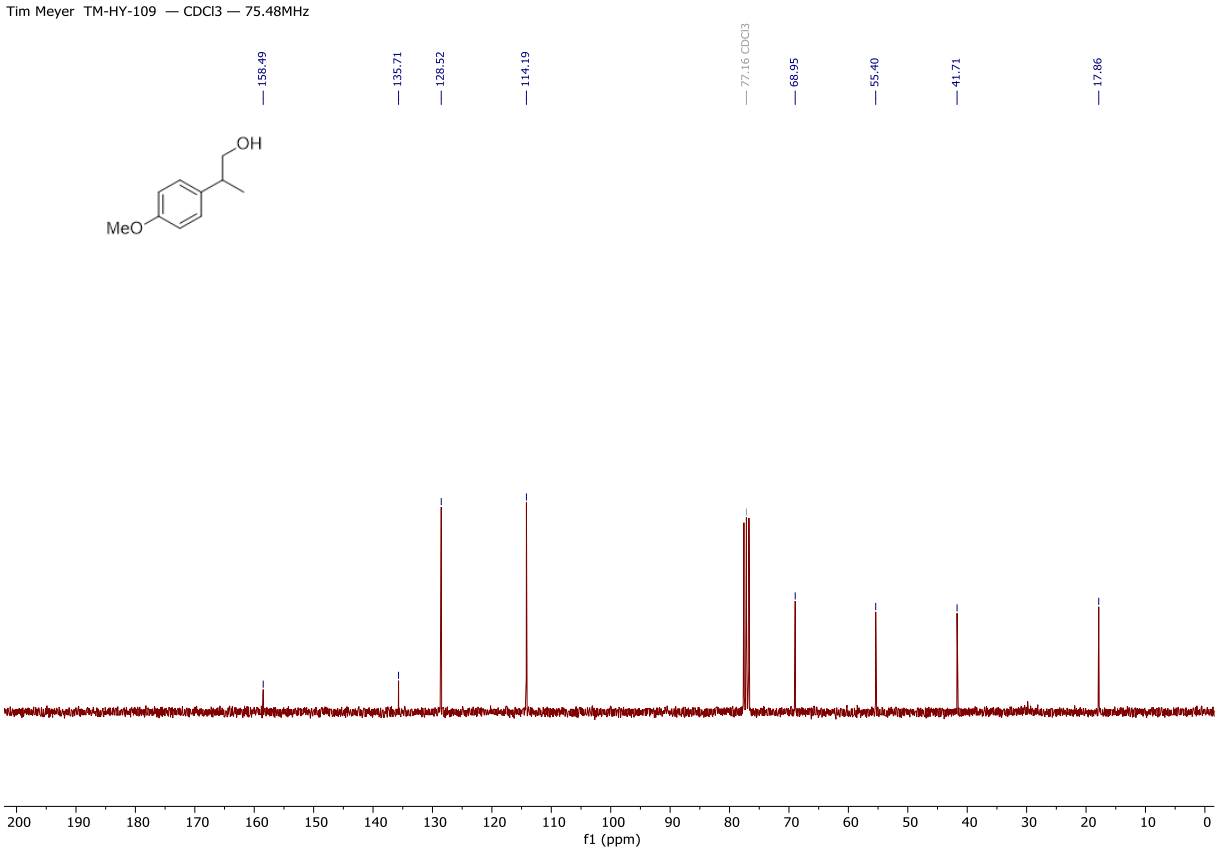
Tim Meyer TM-HY-151 — CDCl3 — 300.13MHz

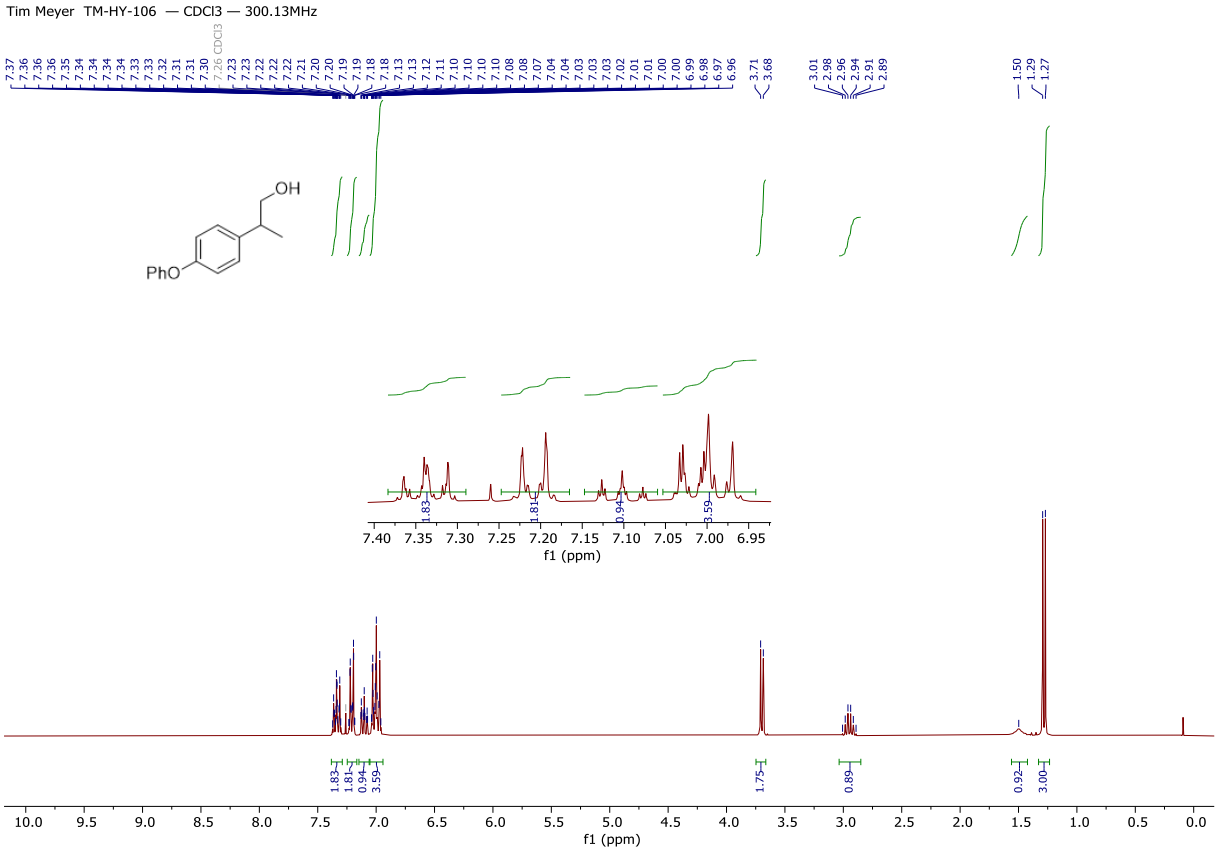


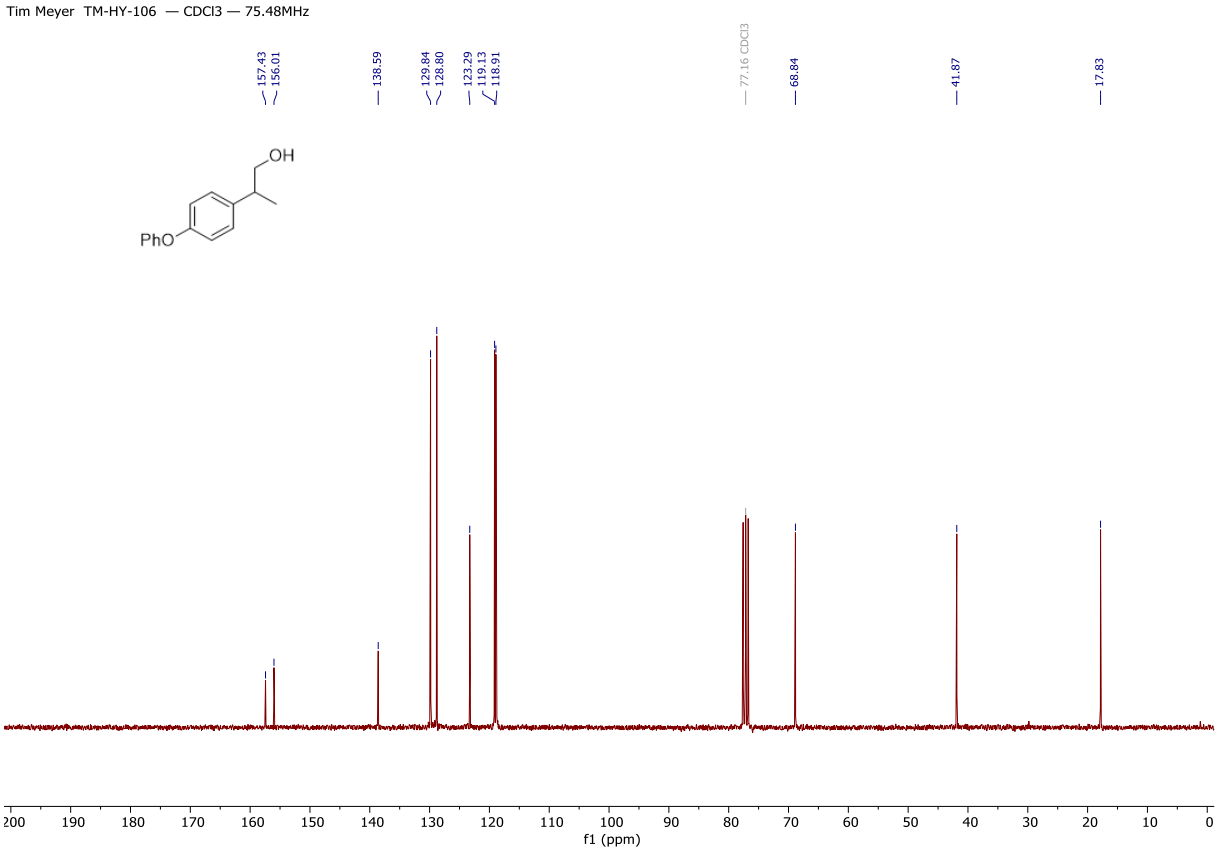
Tim Meyer TM-HY-151 — CDCl3 — 75.48MHz



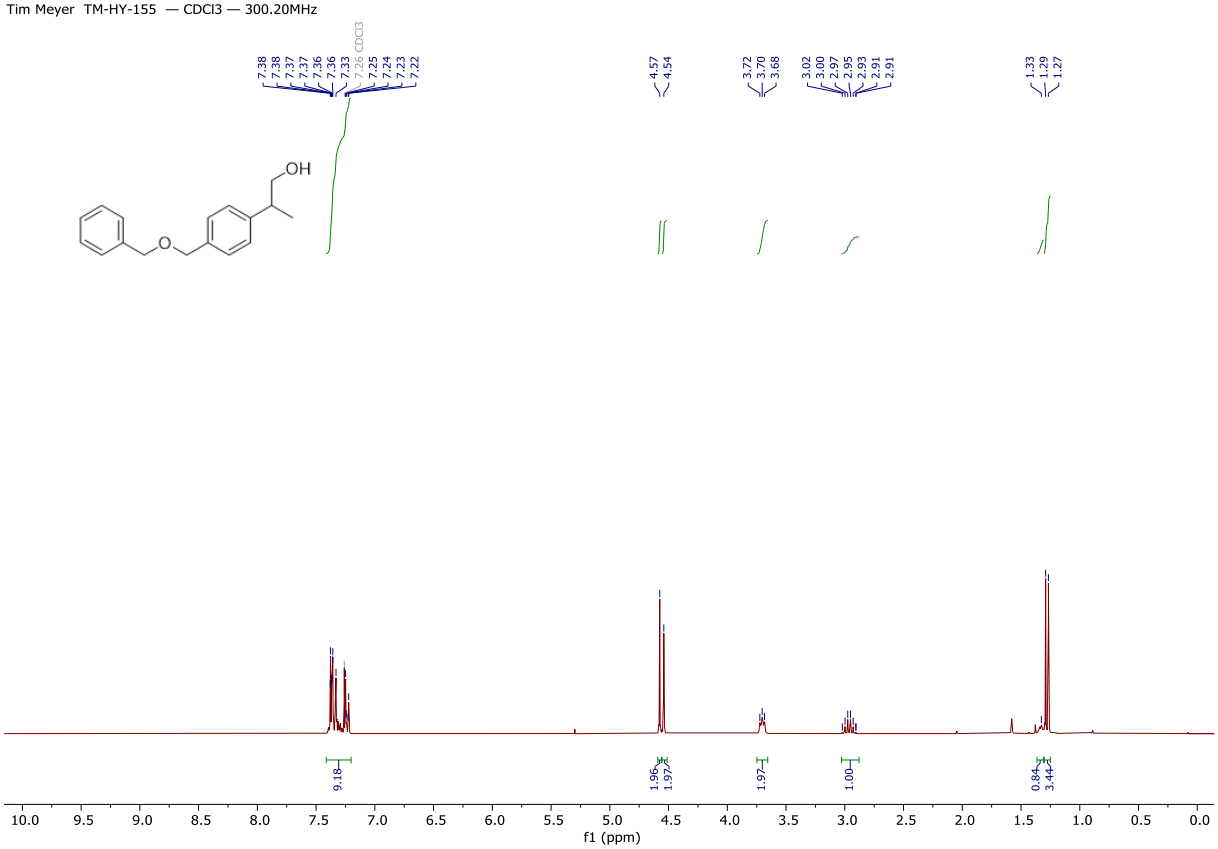

Tim Meyer TM-HY-109 — CDCl3 — 300.13MHz
7.26 CDCl3
7.18
7.18
7.15
7.15
6.89
6.89
6.86
3.80
3.68
3.67
3.66
3.65
2.96
2.94
2.92
2.89
2.87
2.85
1.38
1.26
1.24
OH
MeO
0.56
0.56
0.87
0.53
0.29
0.25
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Tim Meyer TM-HY-109 — CDCl3 — 75.48MHz
158.49
135.71
128.52
114.19
77.16 CDCl3
68.95
55.40
41.71
17.86
OH
MeO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

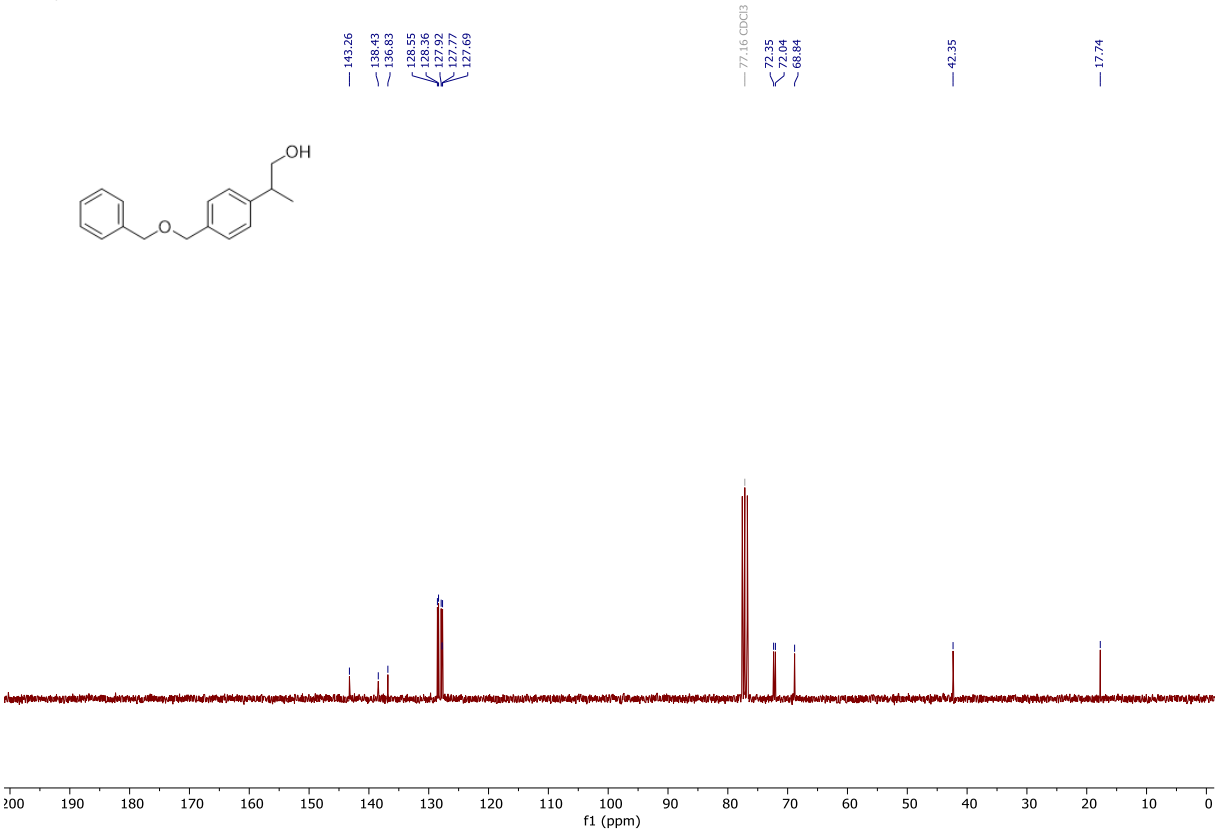

Tim Meyer TM-HY-106 — CDCl3 — 300.13MHz
OH
PhO
f1 (ppm)


Tim Meyer TM-HY-106 — CDCl3 — 75.48MHz
OH
PhO
f1 (ppm)

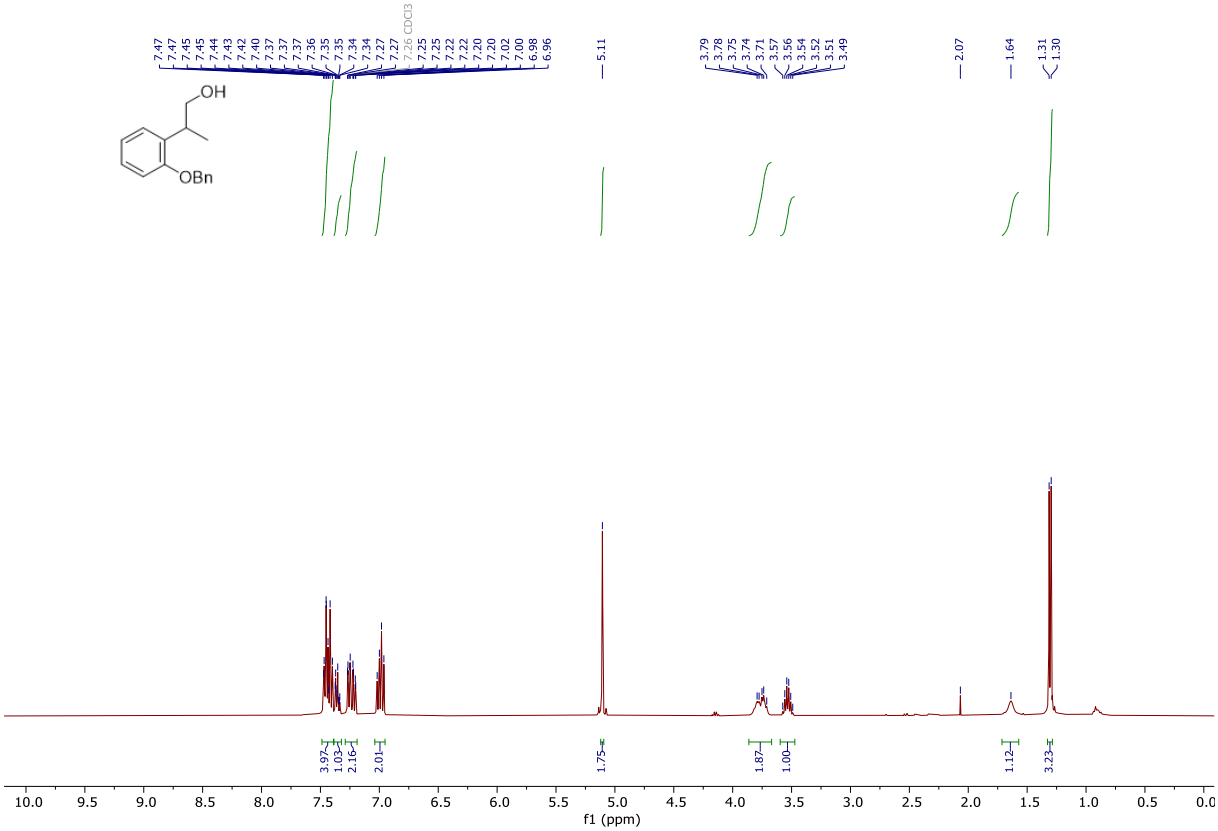
Tim Meyer TM-HY-155 — CDCl3 — 300.20MHz



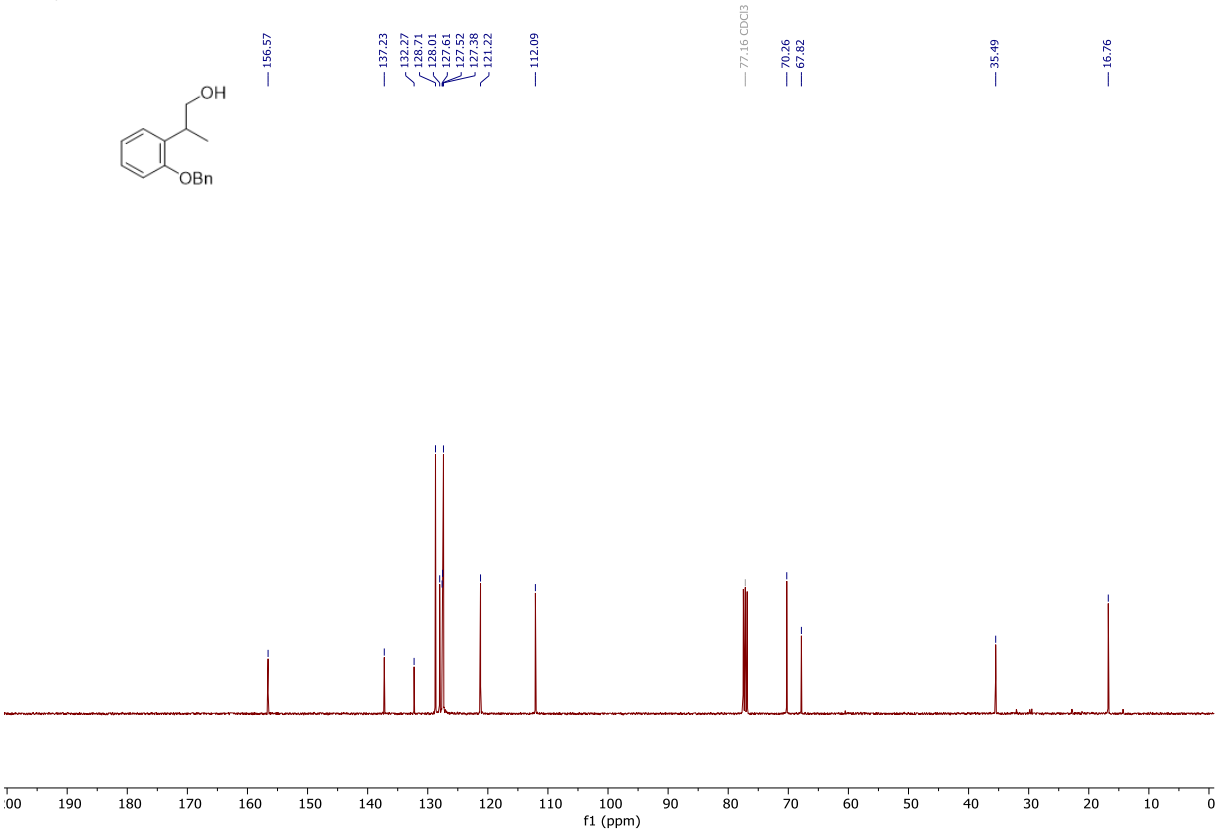
Tim Meyer TM-HY-156 — CDCl3 — 75.49MHz



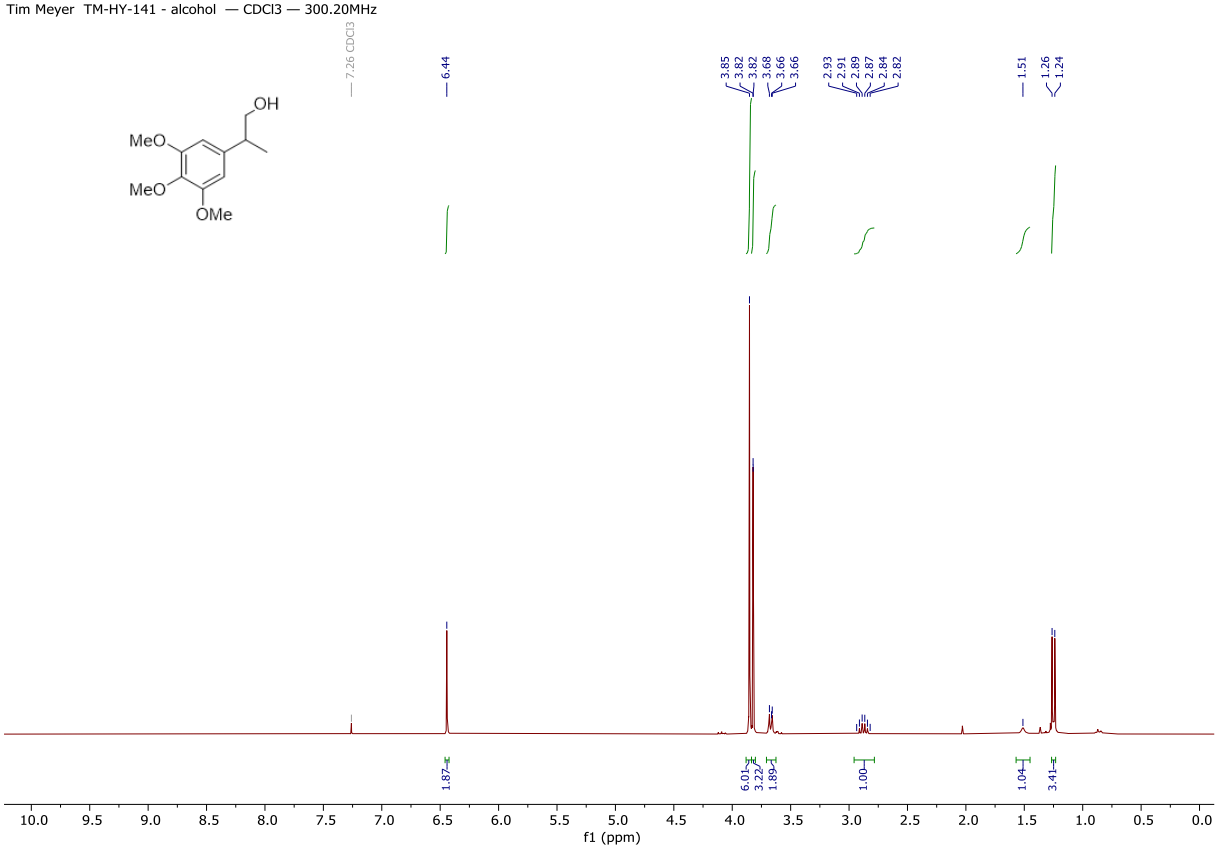
Tim Meyer TM-HY-144 — CDCl3 — 400.13MHz



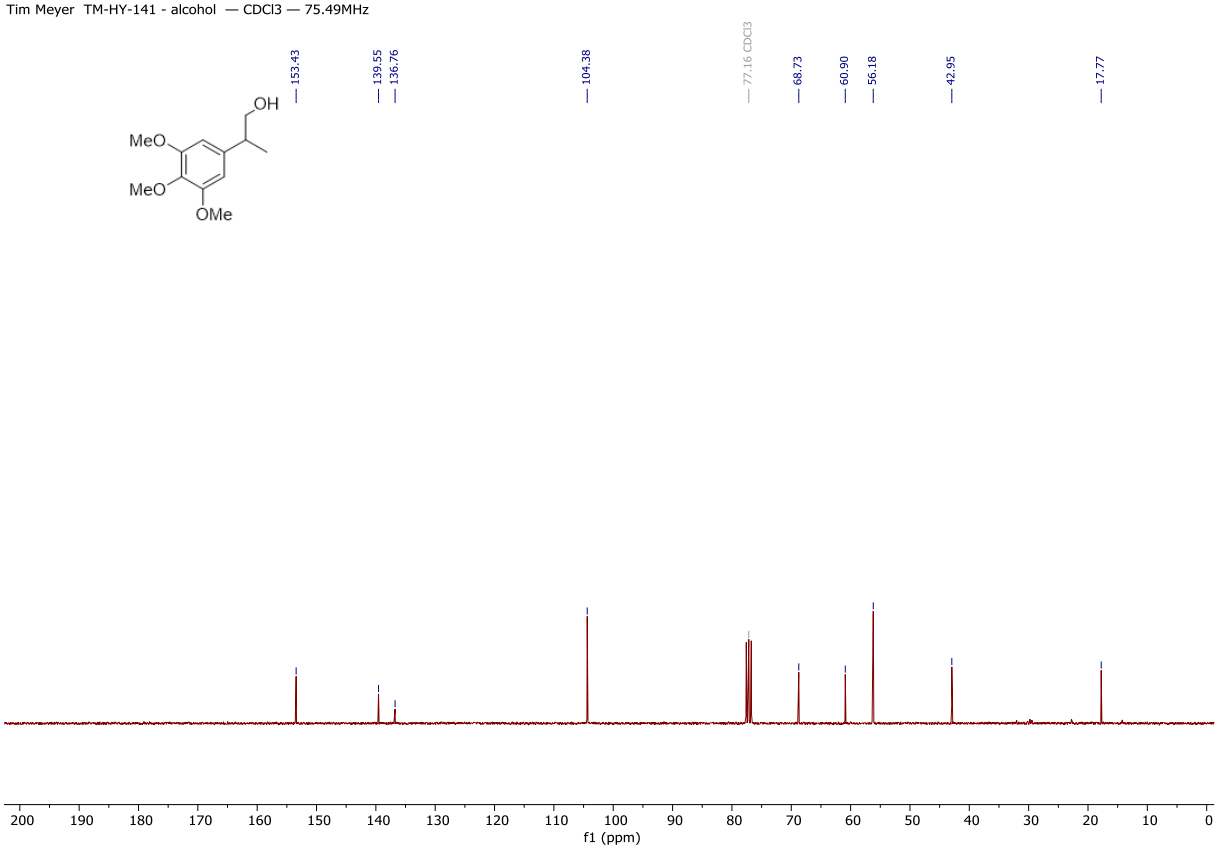
Tim Meyer TM-HY-144 — CDCl3 — 100.63MHz



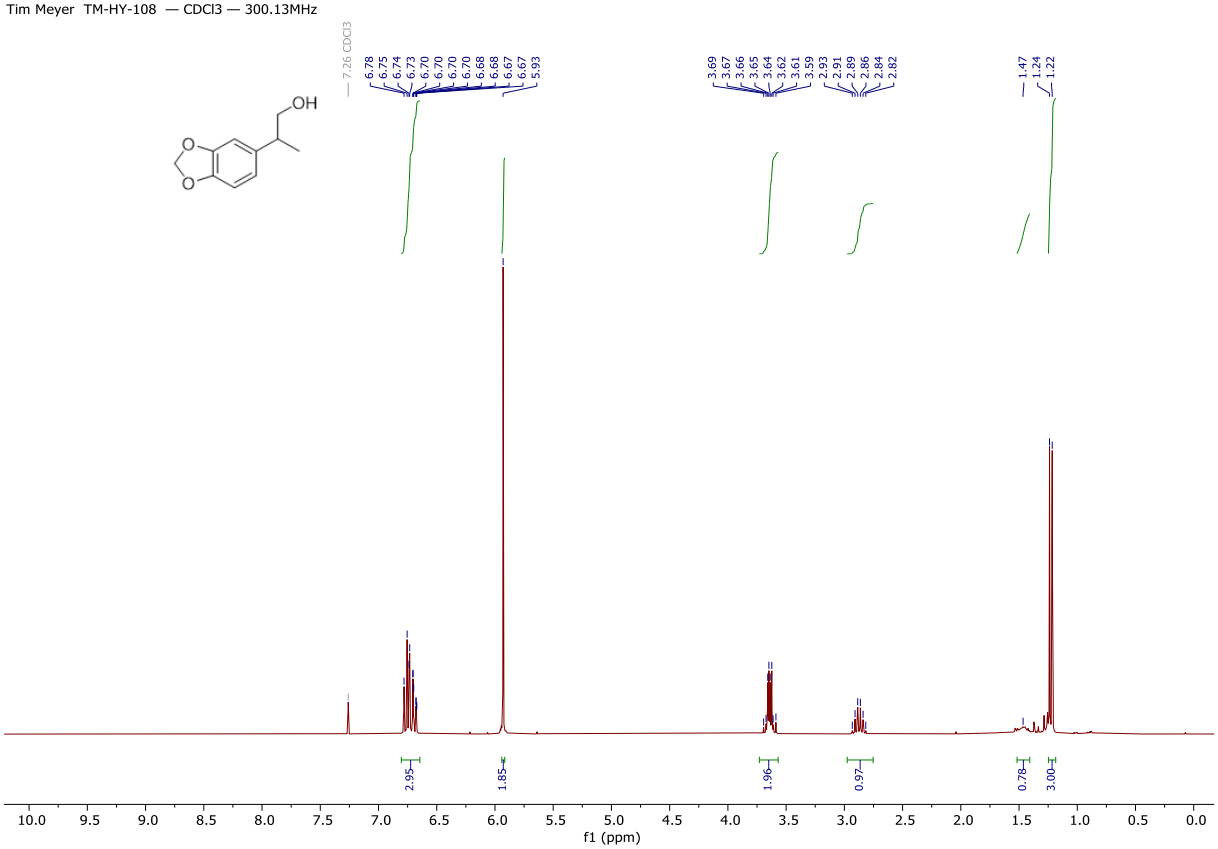
Tim Meyer TM-HY-141 - alcohol — CDCl3 — 300.20MHz



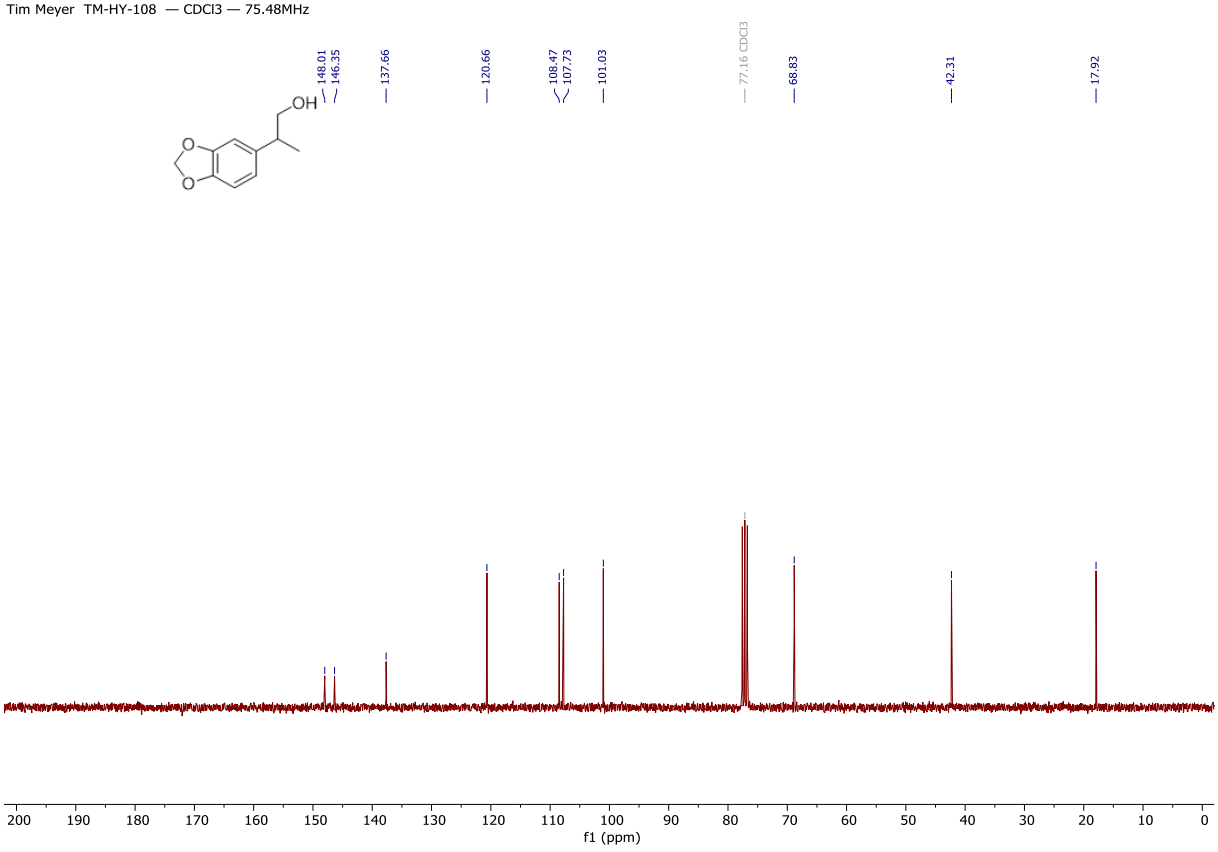
Tim Meyer TM-HY-141 - alcohol — CDCl3 — 75.49MHz



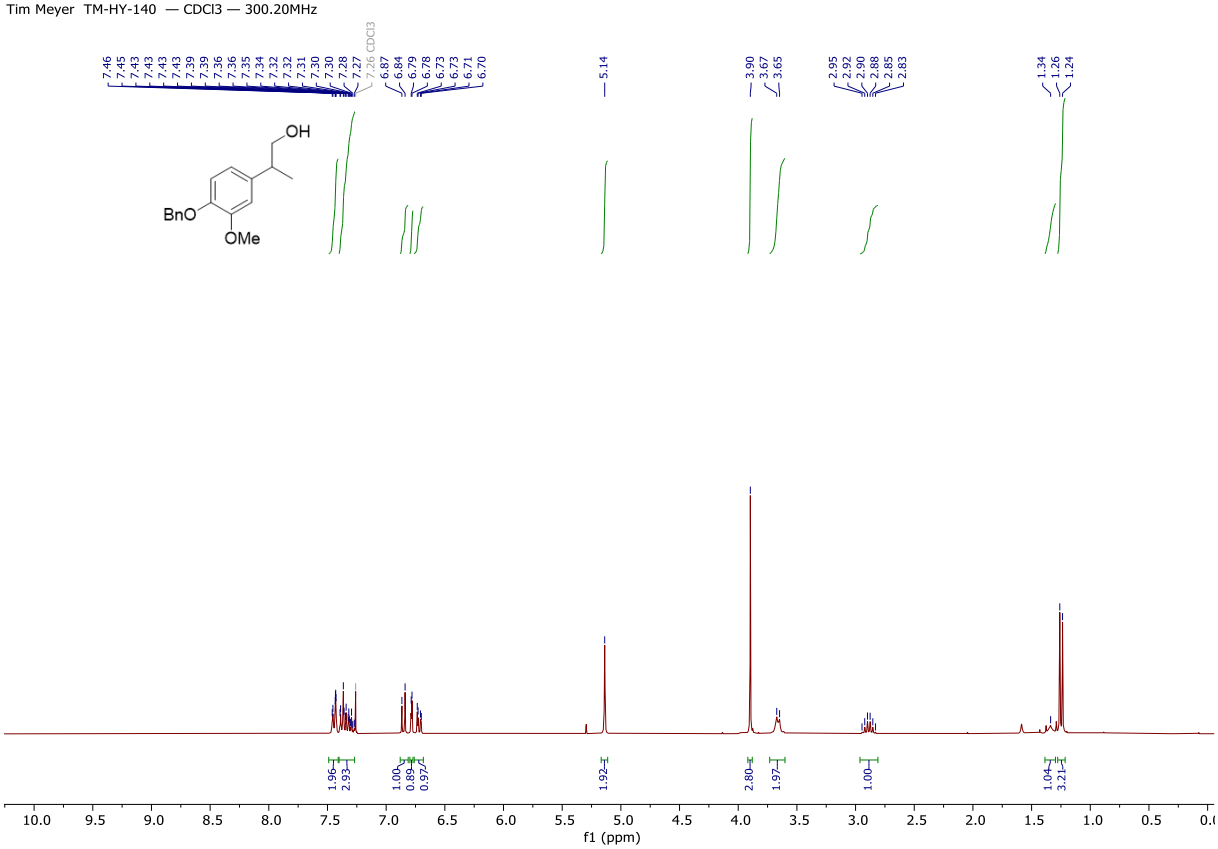
Tim Meyer TM-HY-108 — CDCl3 — 300.13MHz



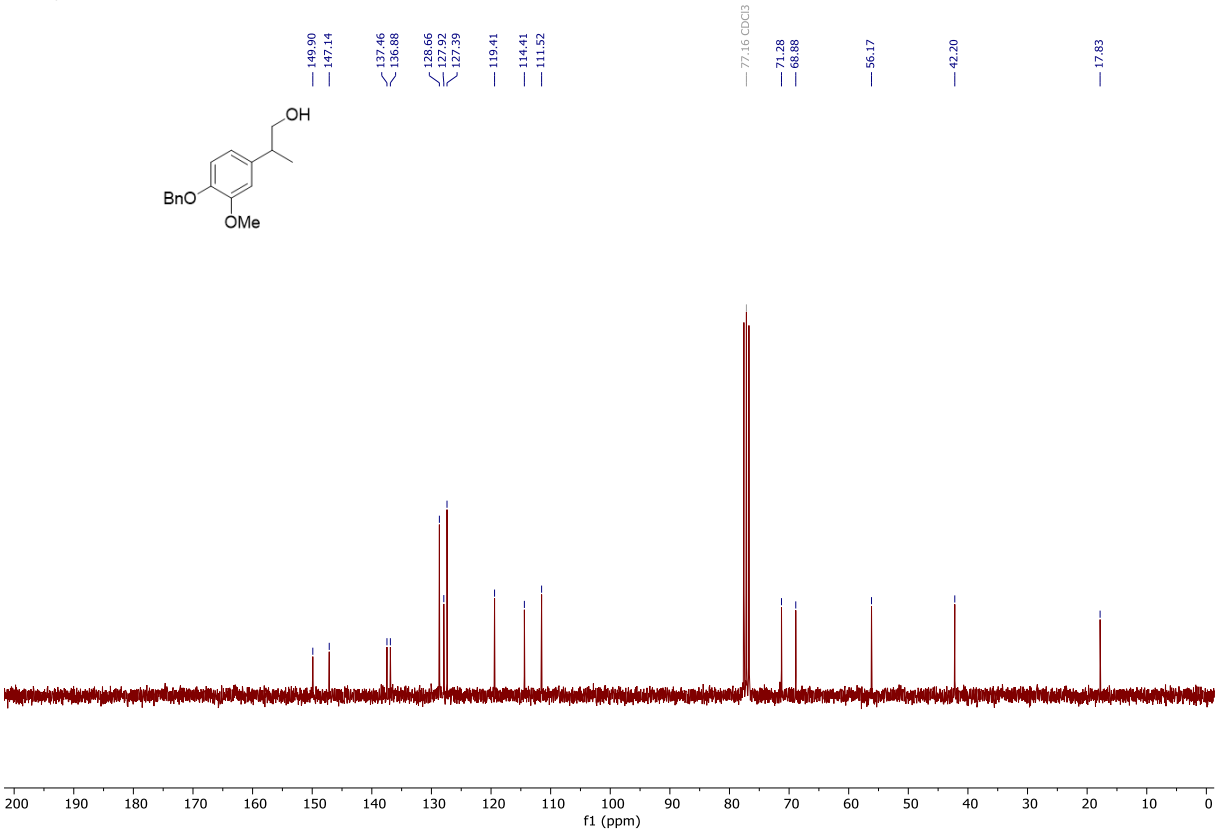
Tim Meyer TM-HY-108 — CDCl3 — 75.48MHz



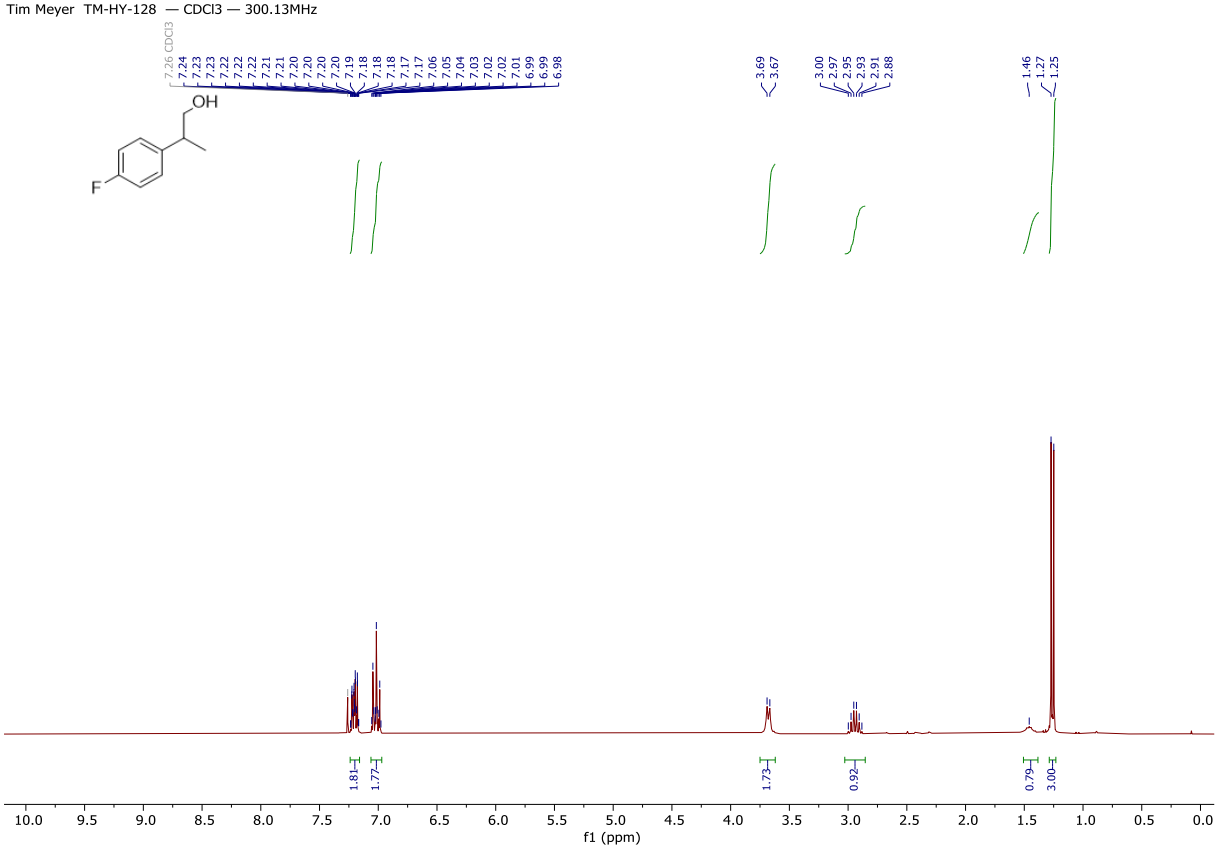
Tim Meyer TM-HY-140 — CDCl3 — 300.20MHz



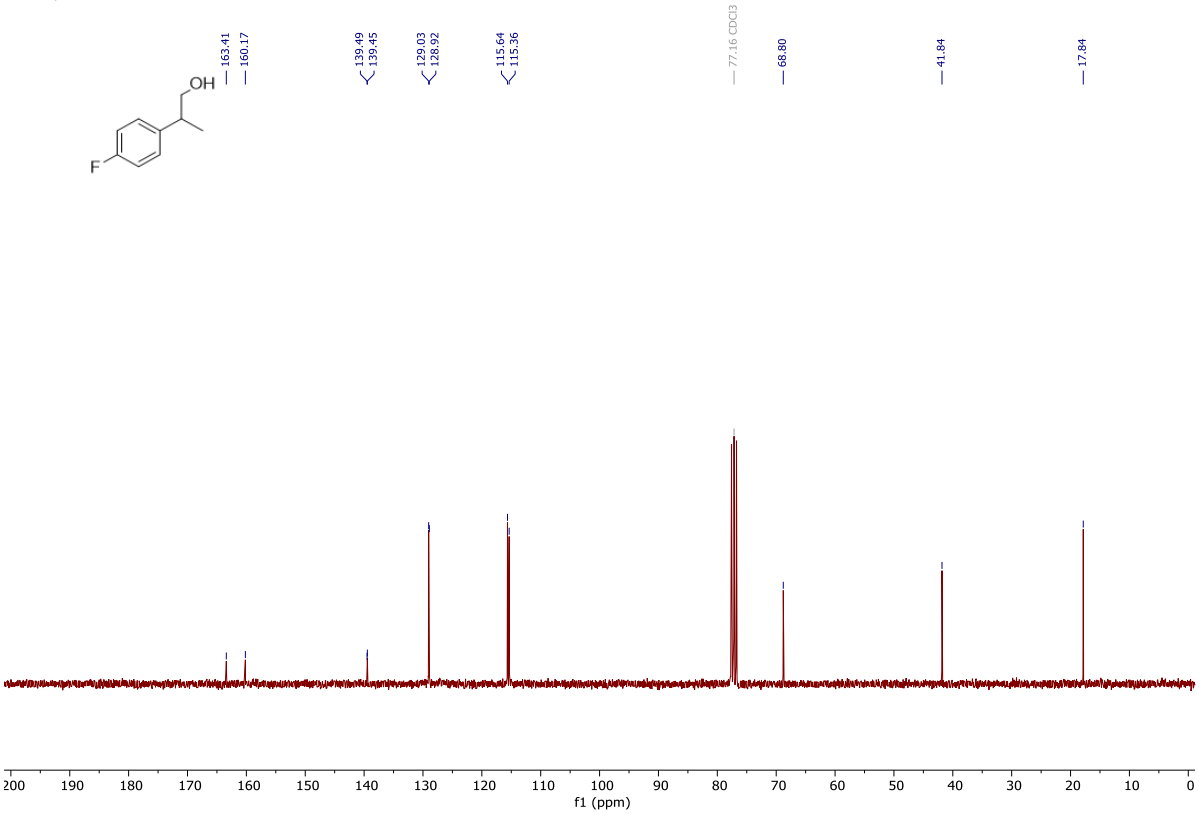
Tim Meyer TM-HY-140 — CDCl3 — 75.49MHz

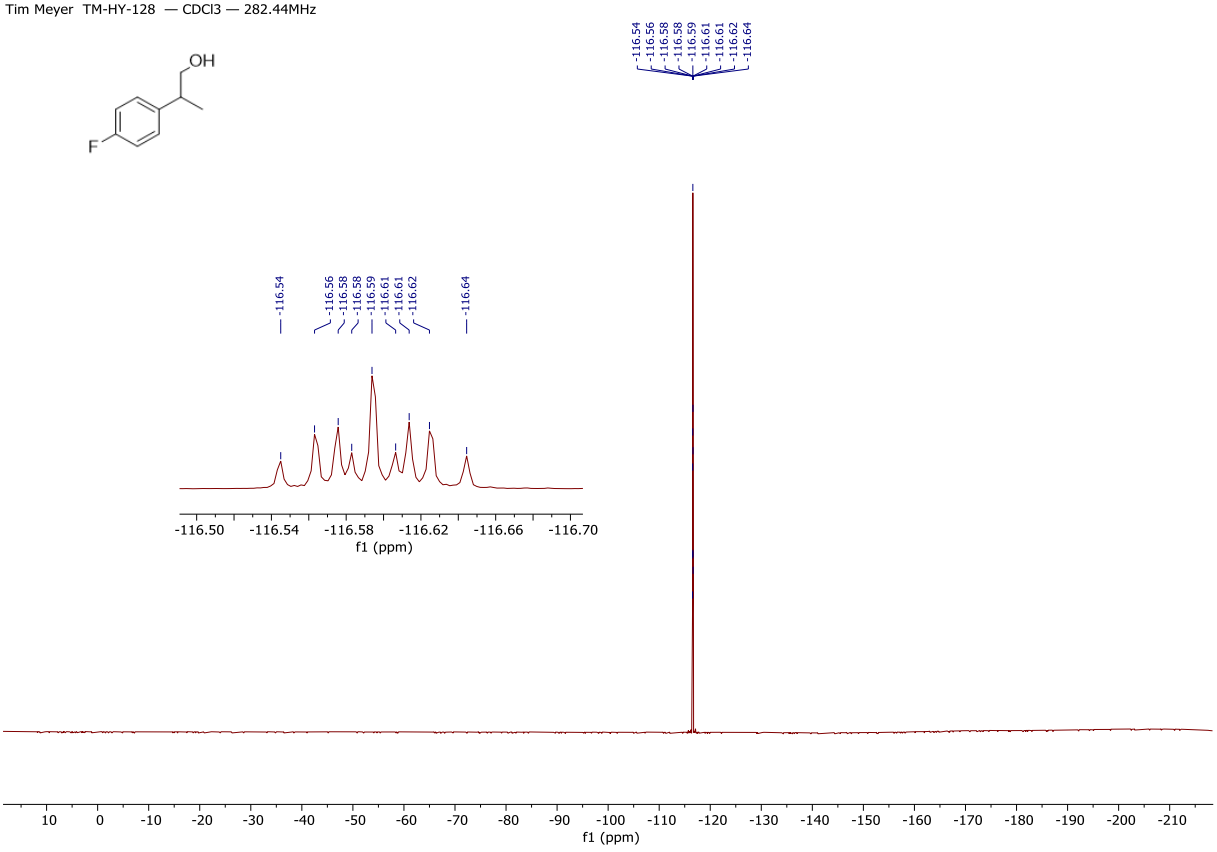


Tim Meyer TM-HY-128 — CDCl3 — 300.13MHz

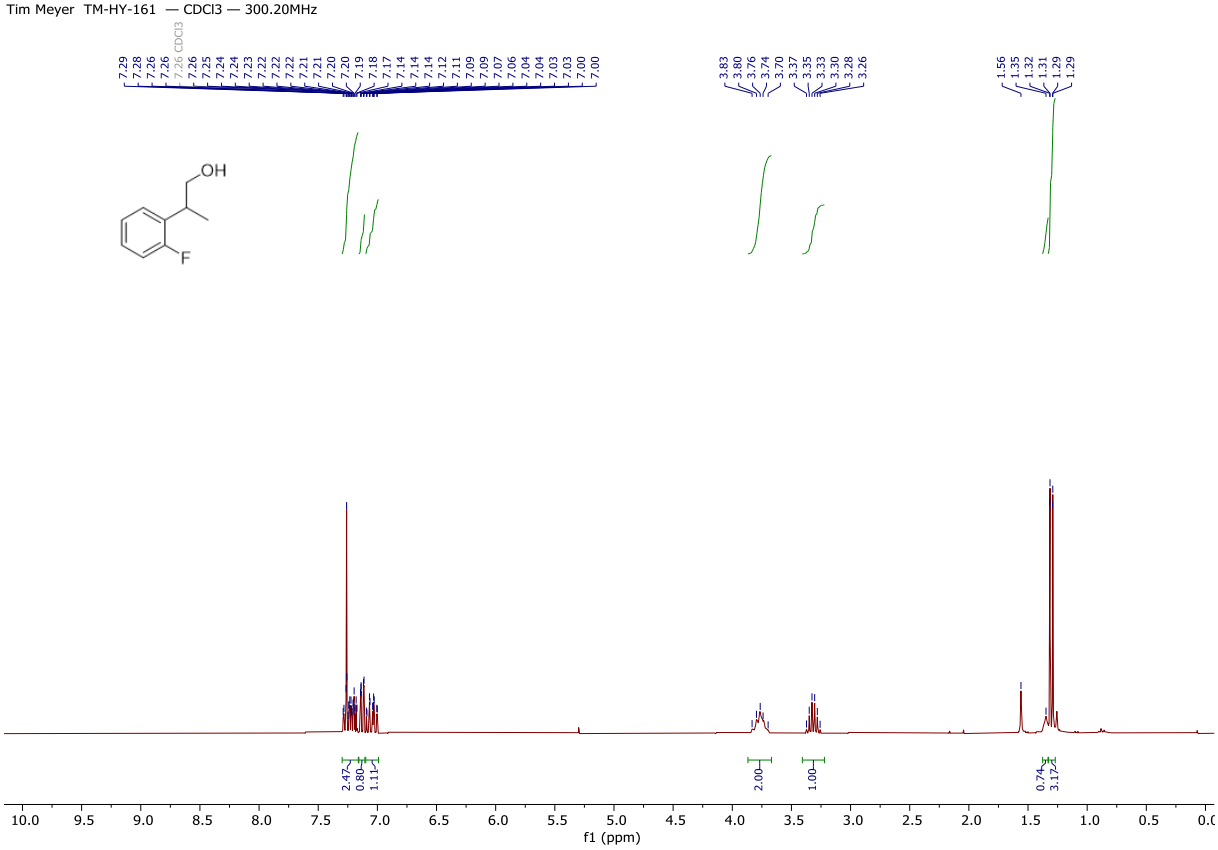


Tim Meyer TM-HY-128 — CDCl3 — 75.48MHz

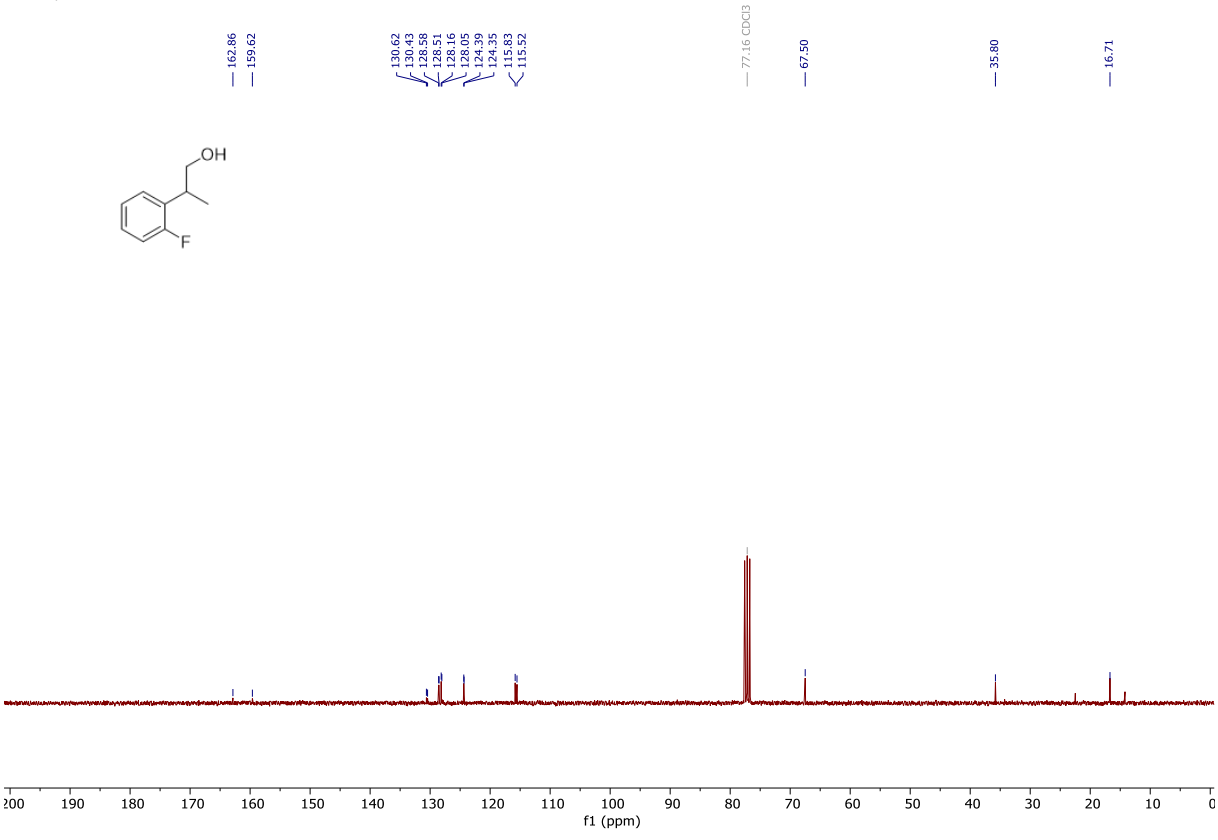



Tim Meyer TM-HY-128 — CDCl3 — 282.44MHz
OH
F
-116.54
-116.56
-116.58
-116.58
-116.59
-116.61
-116.61
-116.62
-116.64
-116.50
-116.54
-116.58
-116.62
-116.66
-116.70
f1 (ppm)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

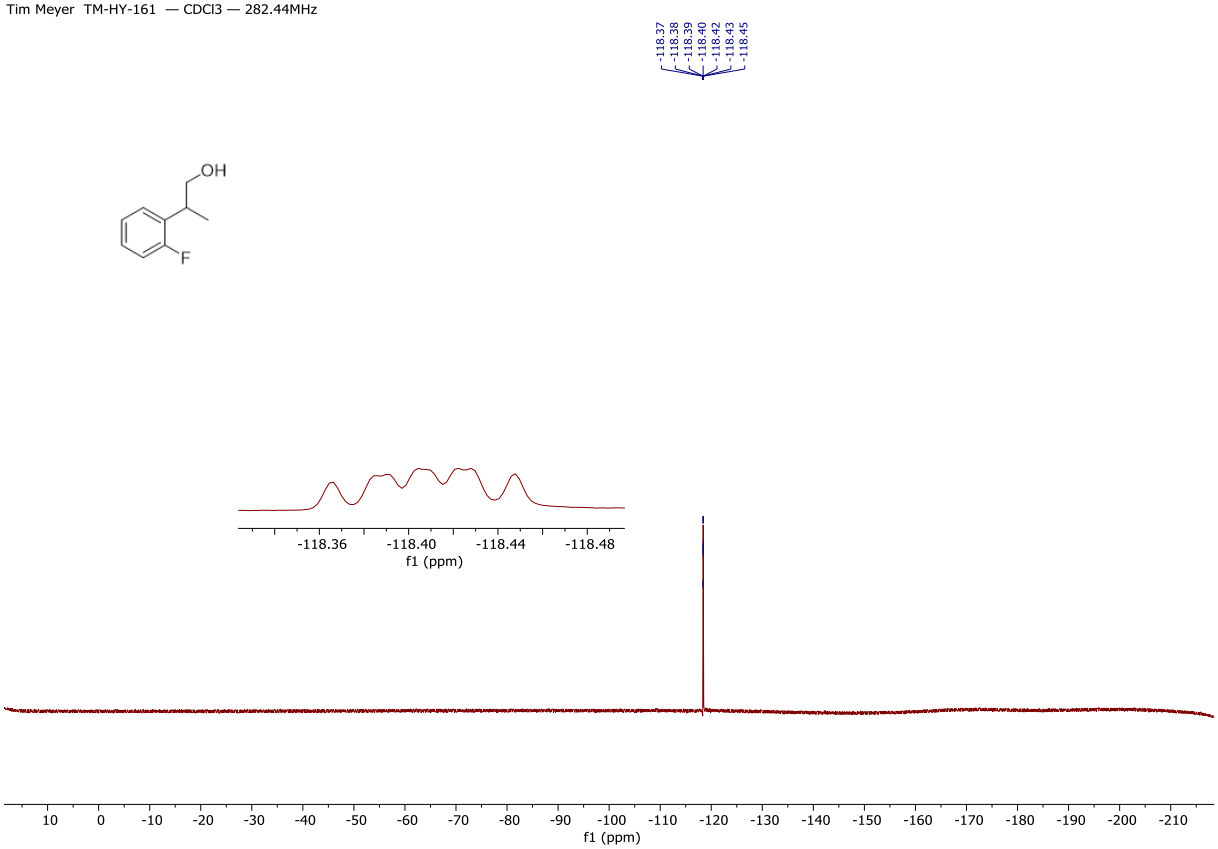
Tim Meyer TM-HY-161 — CDCl3 — 300.20MHz

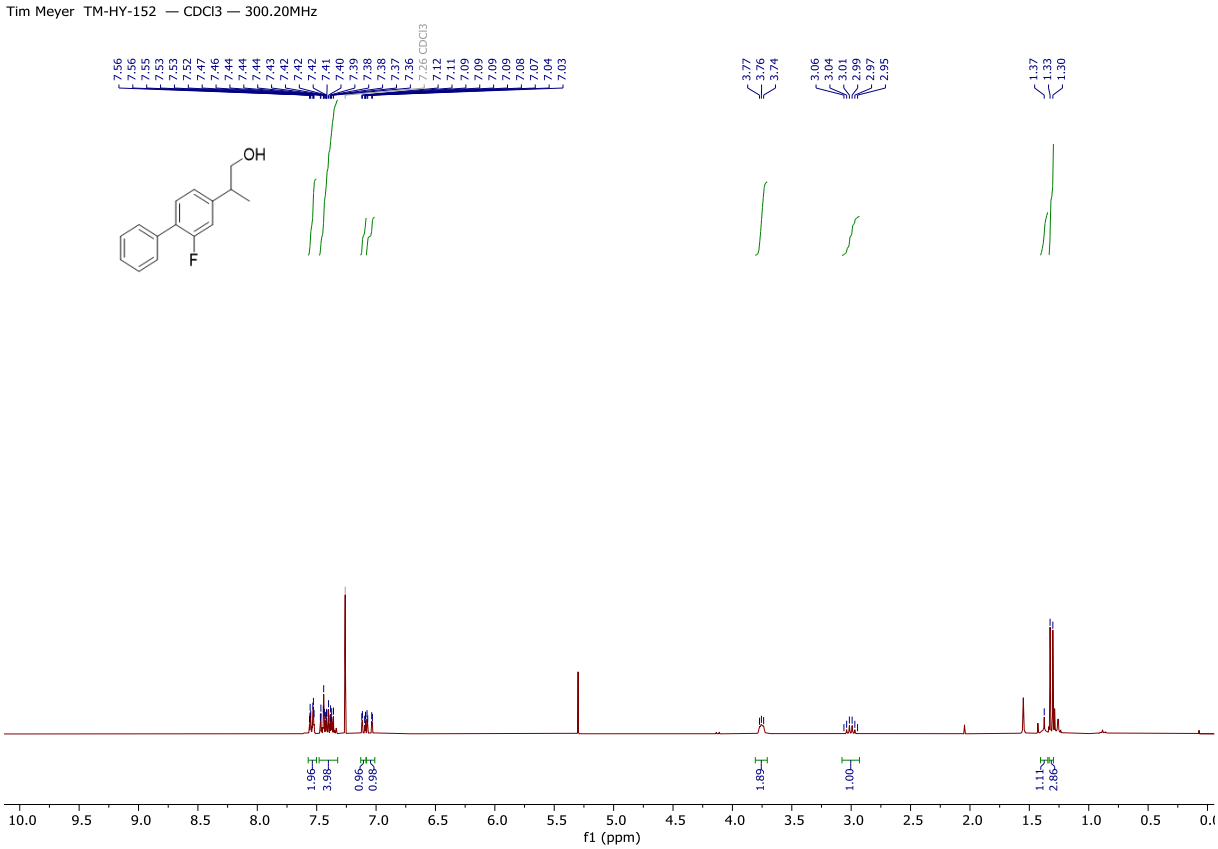


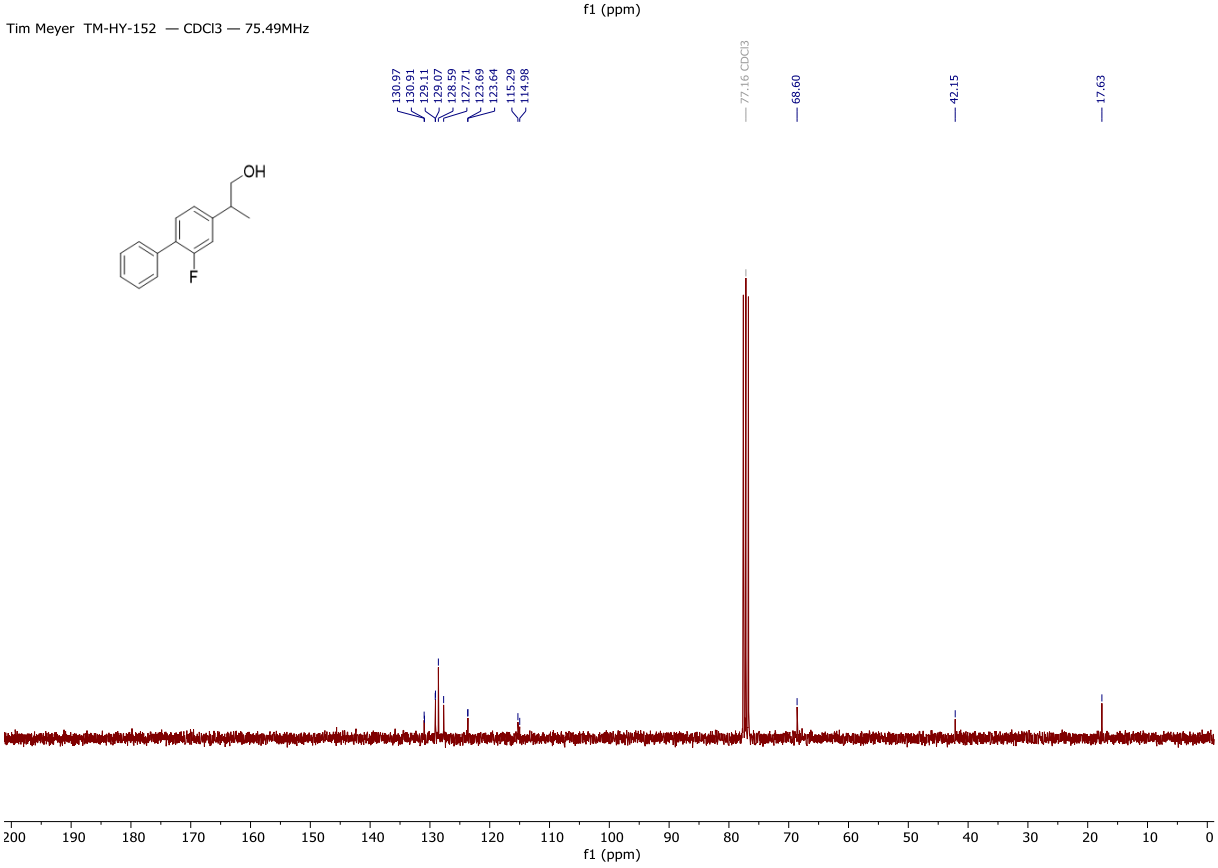
Tim Meyer TM-HY-161 — CDCl3 — 75.49MHz

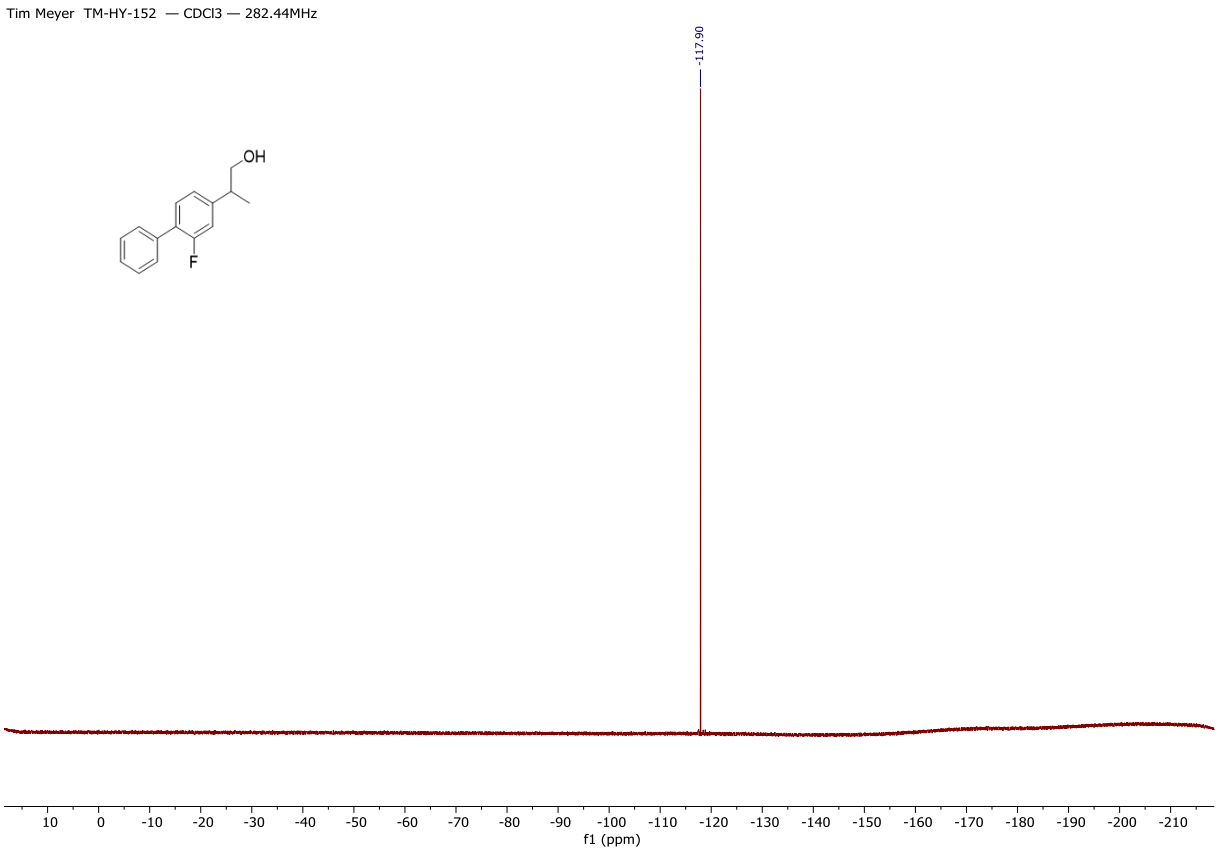


Tim Meyer TM-HY-161 — CDCl3 — 282.44MHz

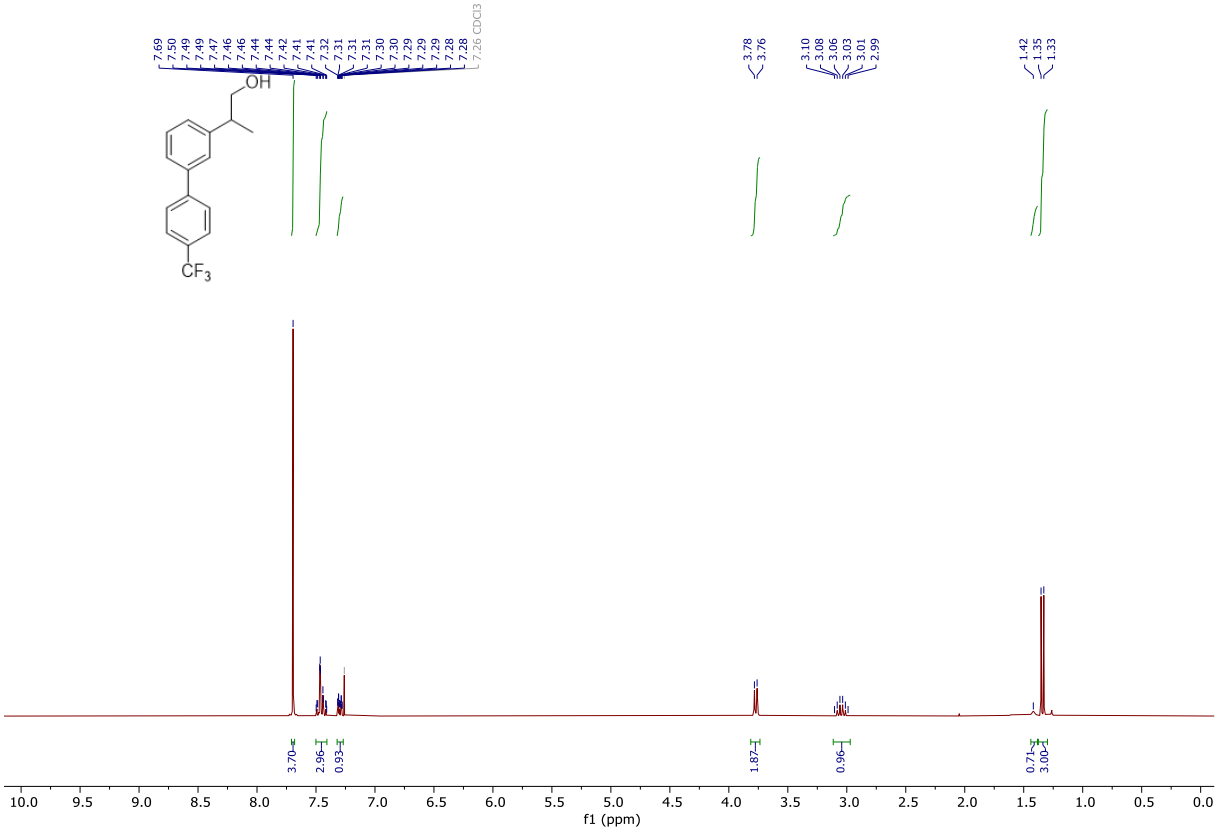



Tim Meyer TM-HY-152 — CDCl3 — 300.20MHz
7.56
7.56
7.55
7.53
7.53
7.52
7.47
7.46
7.44
7.44
7.44
7.43
7.42
7.42
7.42
7.41
7.40
7.39
7.38
7.38
7.37
7.36
7.26 CDCl3
7.12
7.11
7.09
7.09
7.09
7.09
7.08
7.07
7.04
7.03
3.77
3.76
3.74
3.06
3.04
3.01
2.99
2.97
2.95
1.37
1.33
1.30
OH
F
1.96
3.98
0.96
0.98
1.89
1.00
1.11
2.86
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

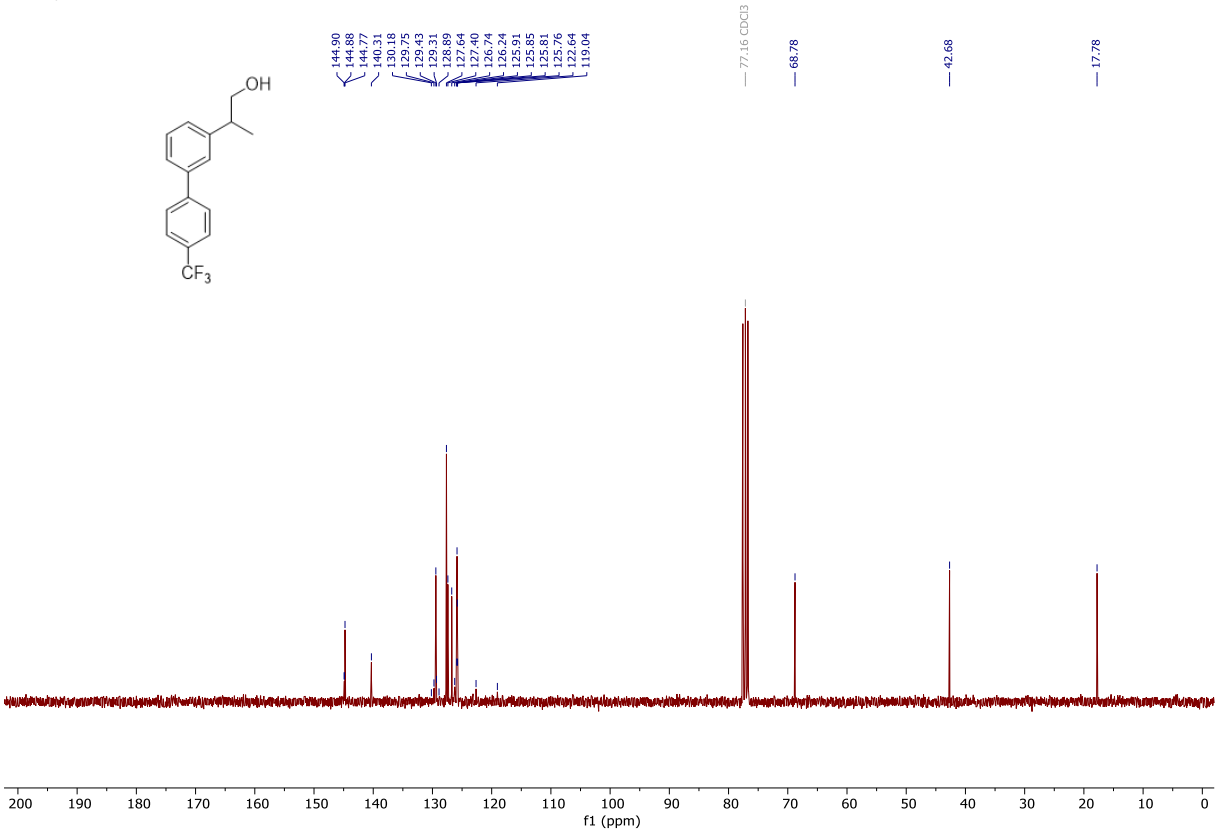

Tim Meyer TM-HY-152 — CDCl3 — 75.49MHz
130.97
130.91
129.11
129.07
128.59
127.71
123.69
123.64
115.29
114.98
77.16 CDCl3
68.60
42.15
17.63
OH
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

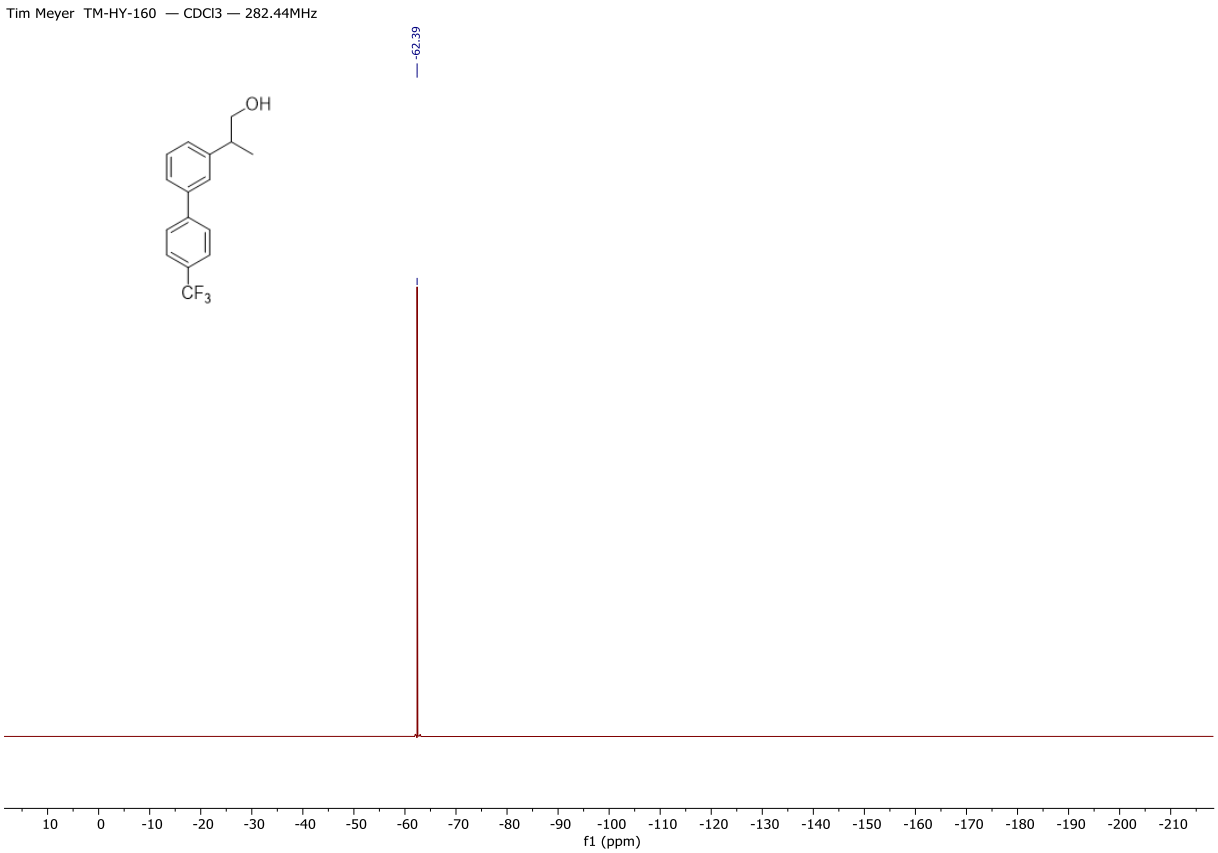

Tim Meyer TM-HY-152 — CDCl3 — 282.44MHz
-117.90
OH
F
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

Tim Meyer TM-HY-160 — CDCl3 — 300.20MHz

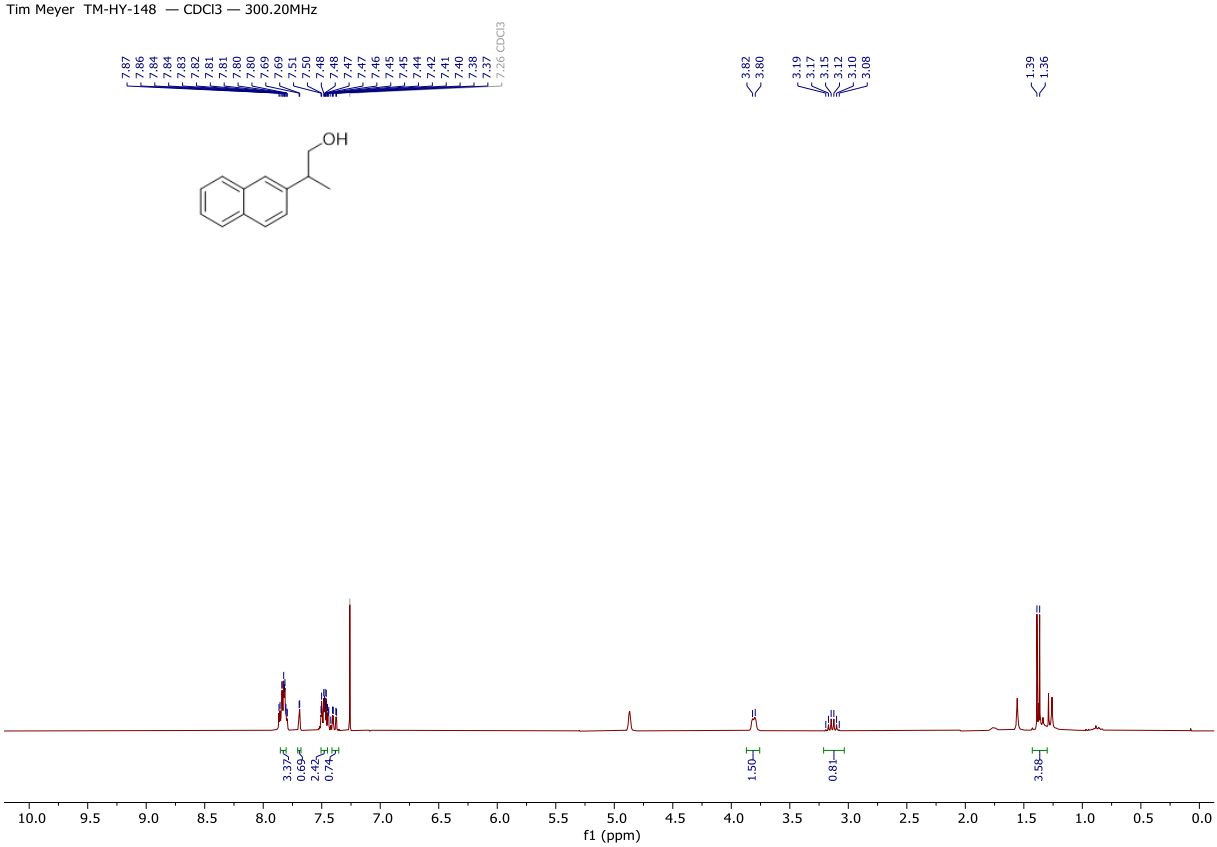


Tim Meyer TM-HY-160 — CDCl3 — 75.49MHz

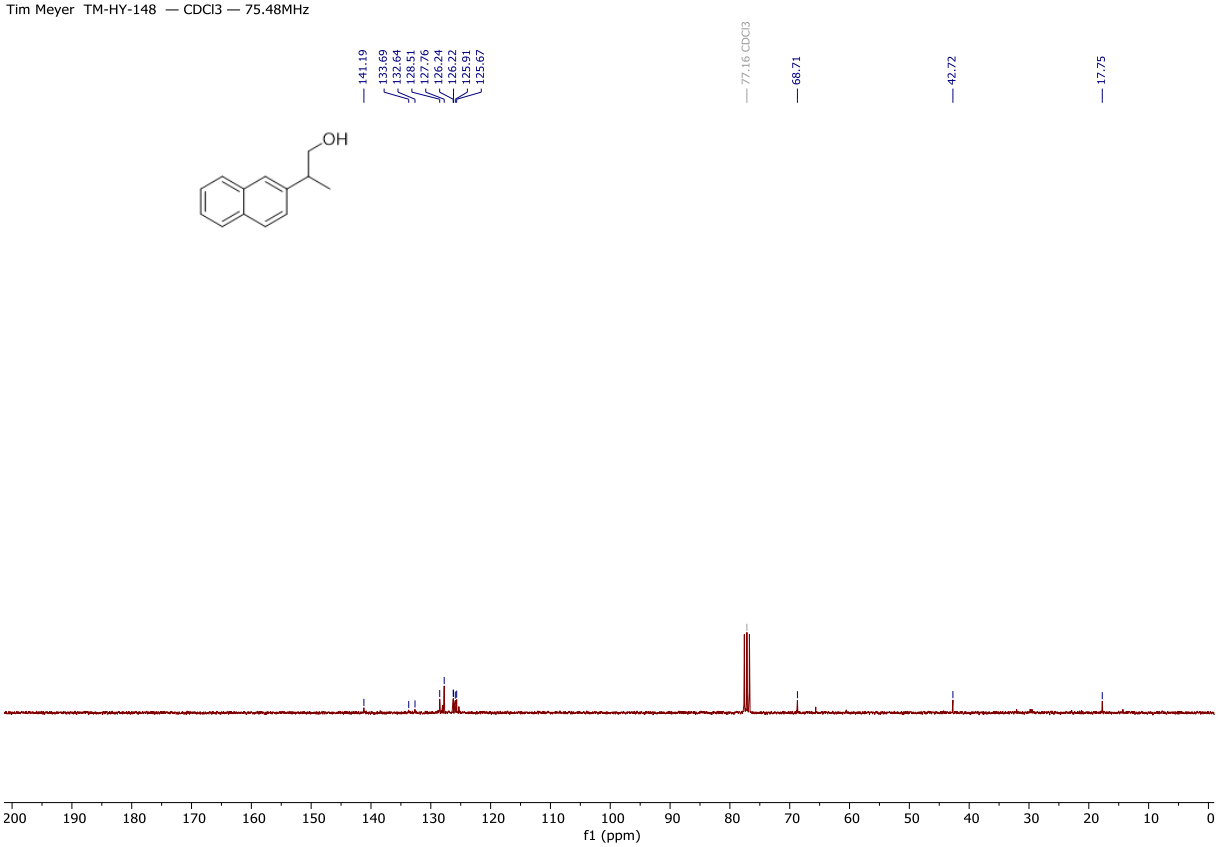


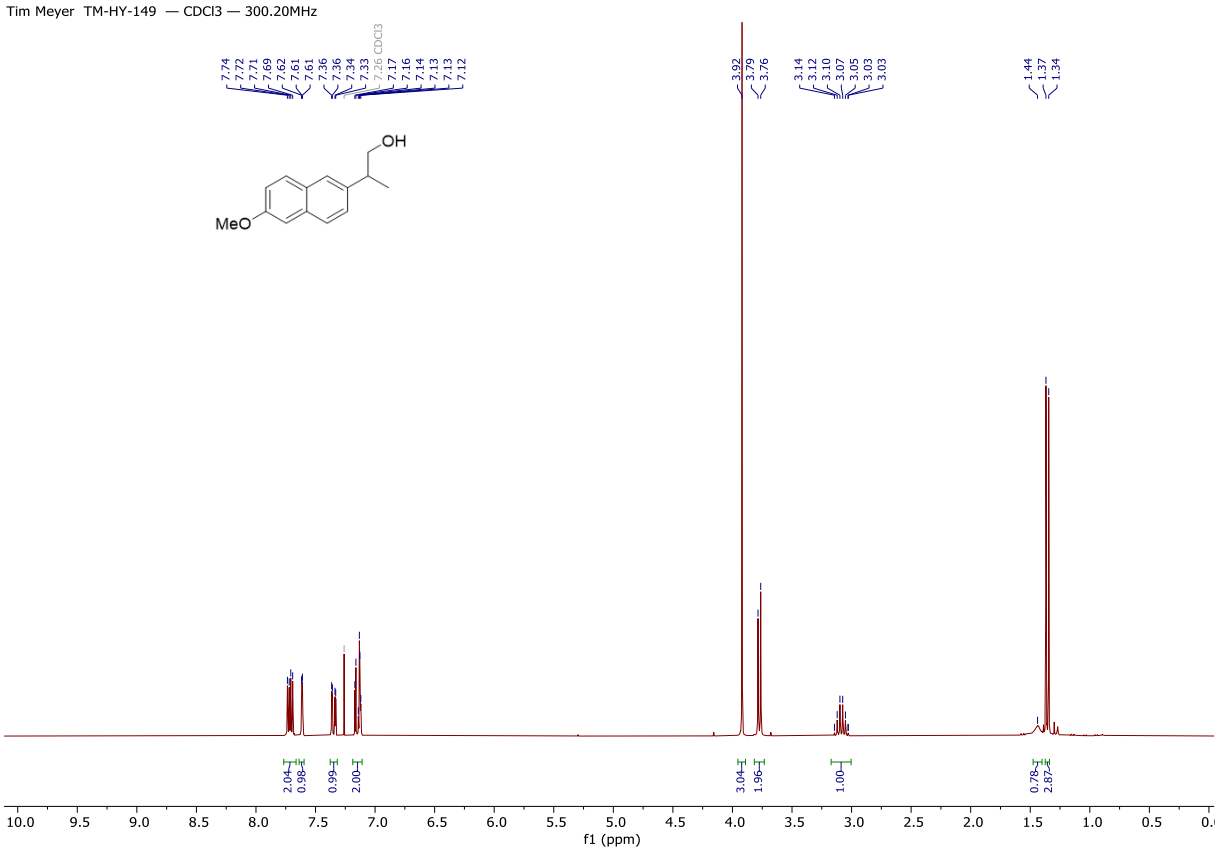

Tim Meyer TM-HY-160 — CDCl3 — 282.44MHz
-62.39
OH
CF3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

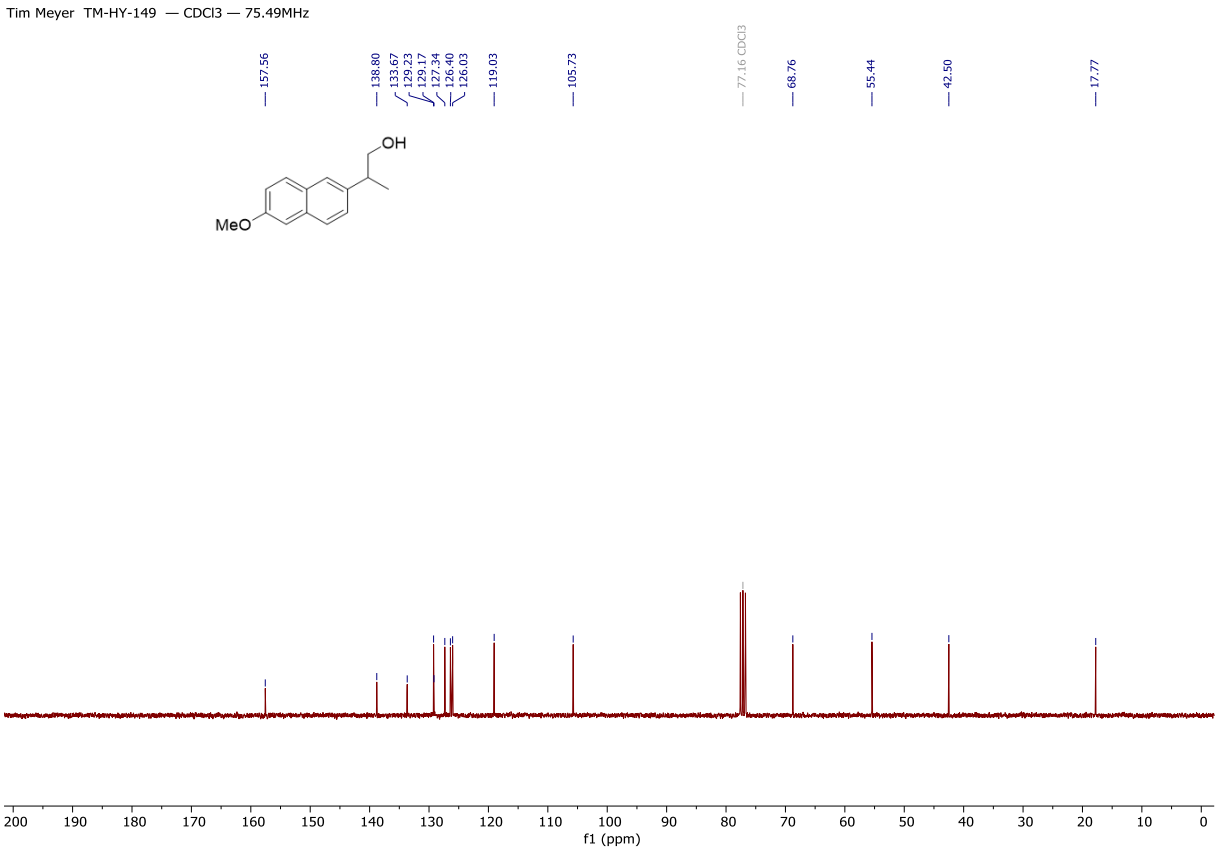
Tim Meyer TM-HY-148 — CDCl3 — 300.20MHz

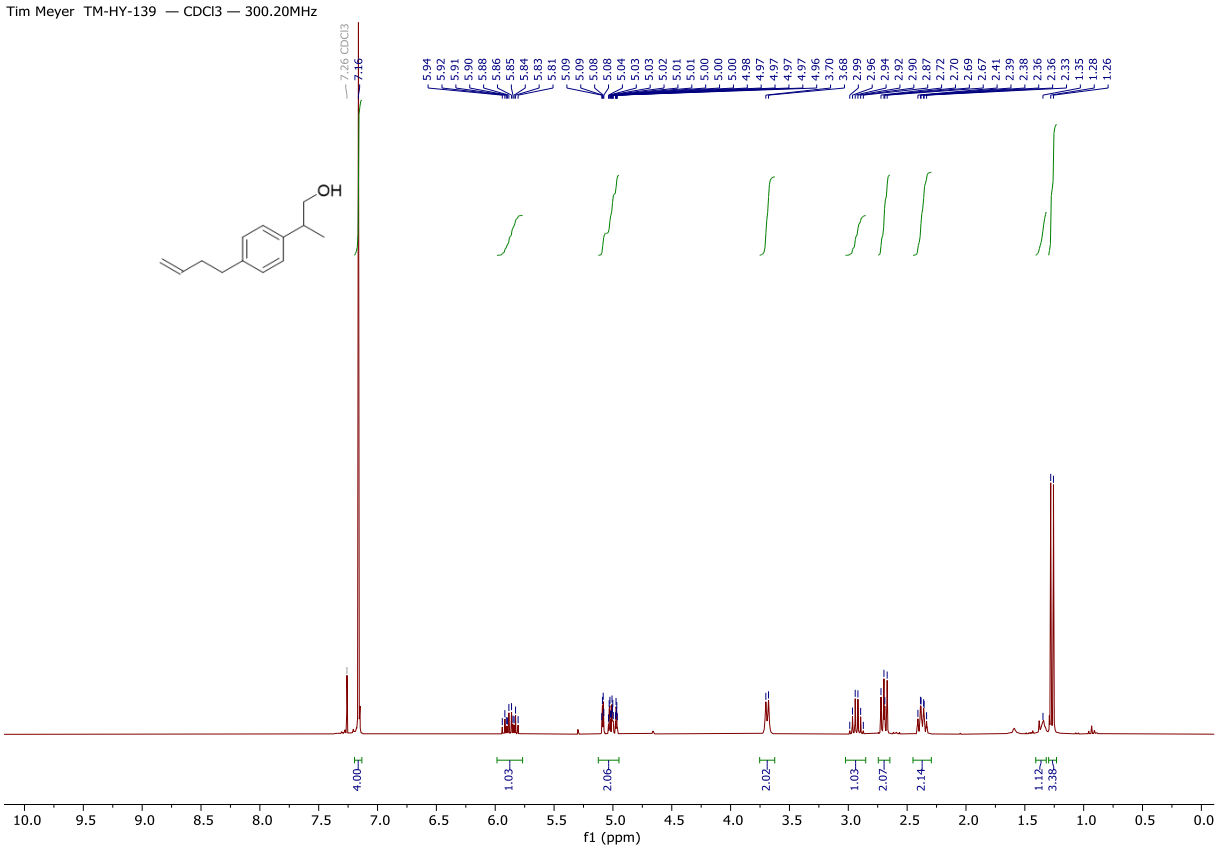


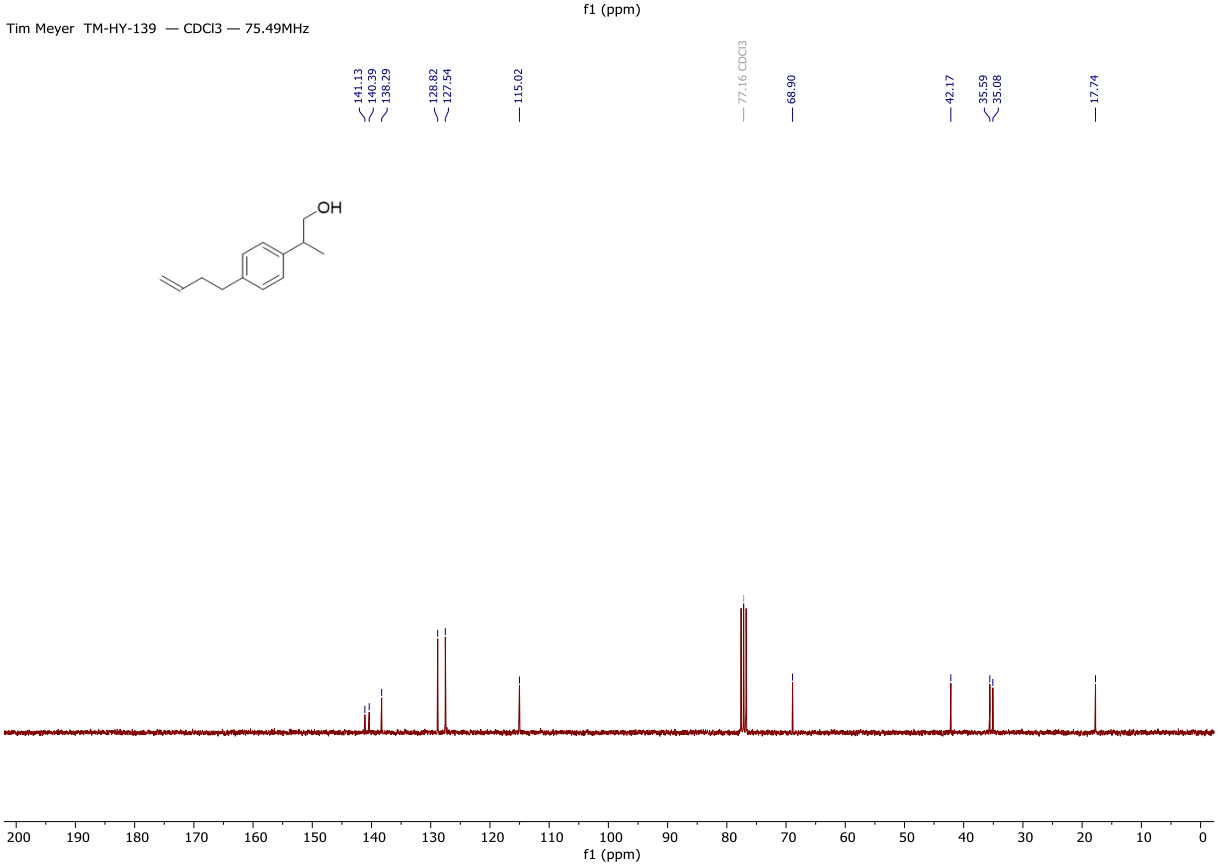
Tim Meyer TM-HY-148 — CDCl3 — 75.48MHz



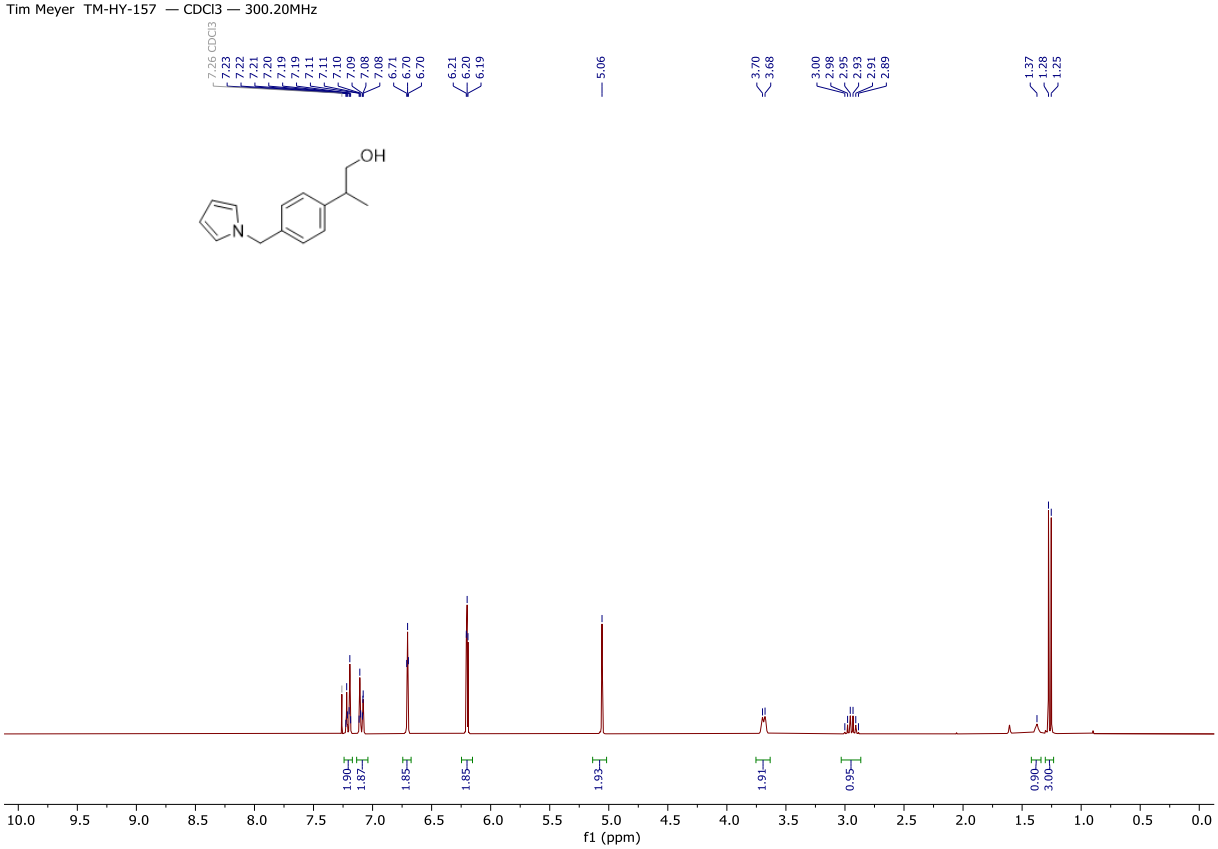

Tim Meyer TM-HY-149 — CDCl3 — 300.20MHz
7.74
7.72
7.71
7.69
7.62
7.61
7.61
7.36
7.36
7.34
7.33
7.26 CDCl3
7.17
7.16
7.14
7.13
7.13
7.12
3.92
3.79
3.76
3.14
3.12
3.10
3.07
3.05
3.03
3.03
1.44
1.37
1.34
OH
MeO
2.04
0.98
0.99
2.00
3.04
1.96
1.00
0.78
2.87
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


Tim Meyer TM-HY-149 — CDCl3 — 75.49MHz
157.56
138.80
133.67
129.23
129.17
127.34
126.40
126.03
119.03
105.73
77.16 CDCl3
68.76
55.44
42.50
17.77
OH
MeO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

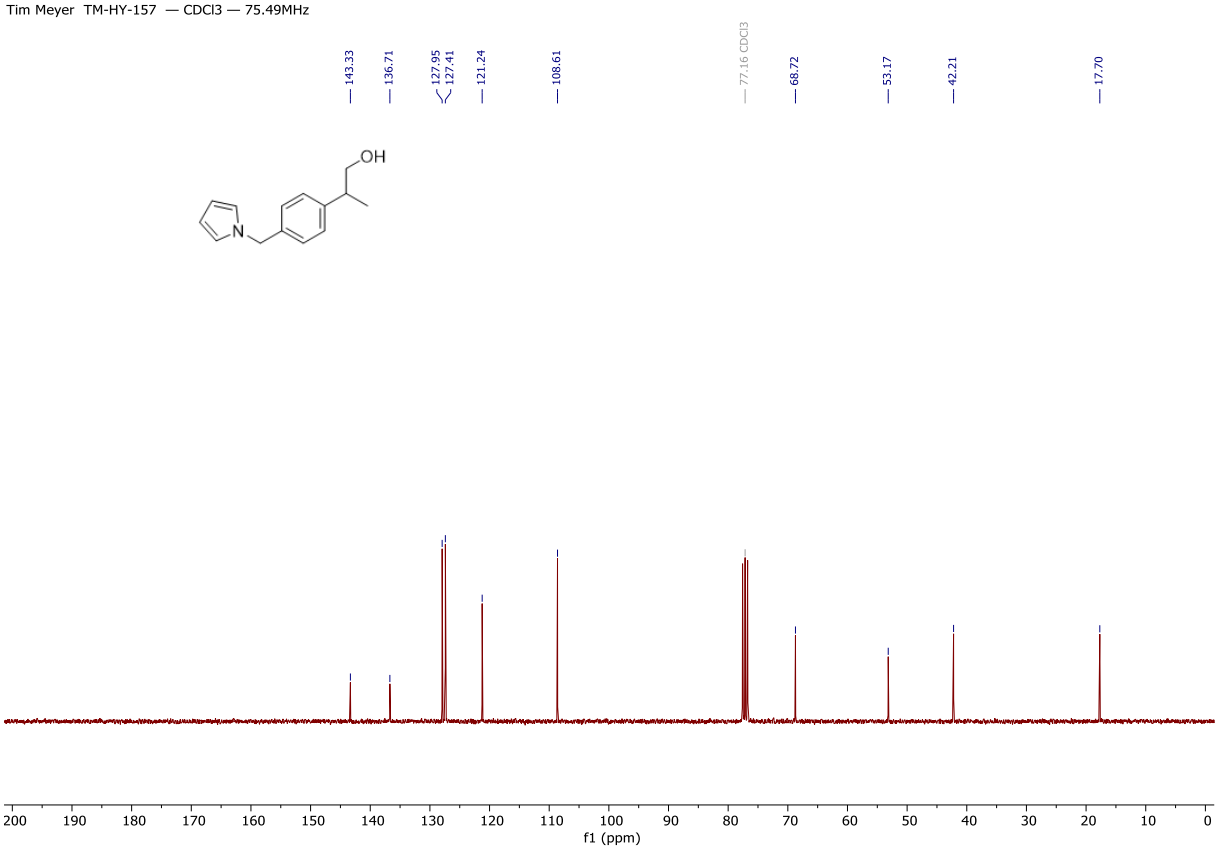

Tim Meyer TM-HY-139 — CDCl3 — 300.20MHz
7.26 CDCl3
7.16
5.94
5.92
5.91
5.90
5.88
5.86
5.85
5.84
5.83
5.81
5.09
5.08
5.04
5.03
5.02
5.01
5.00
5.00
4.98
4.97
4.97
4.97
4.96
3.70
3.68
2.99
2.96
2.94
2.92
2.90
2.87
2.72
2.70
2.69
2.67
2.41
2.39
2.38
2.36
2.36
2.33
1.35
1.28
1.26
OH
4.00
1.03
2.06
2.02
1.03
2.07
2.14
1.12
3.38
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Tim Meyer TM-HY-139 — CDCl3 — 75.49MHz
141.13
140.39
138.29
128.82
127.54
115.02
77.16 CDCl3
68.90
42.17
35.59
35.08
17.74
OH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

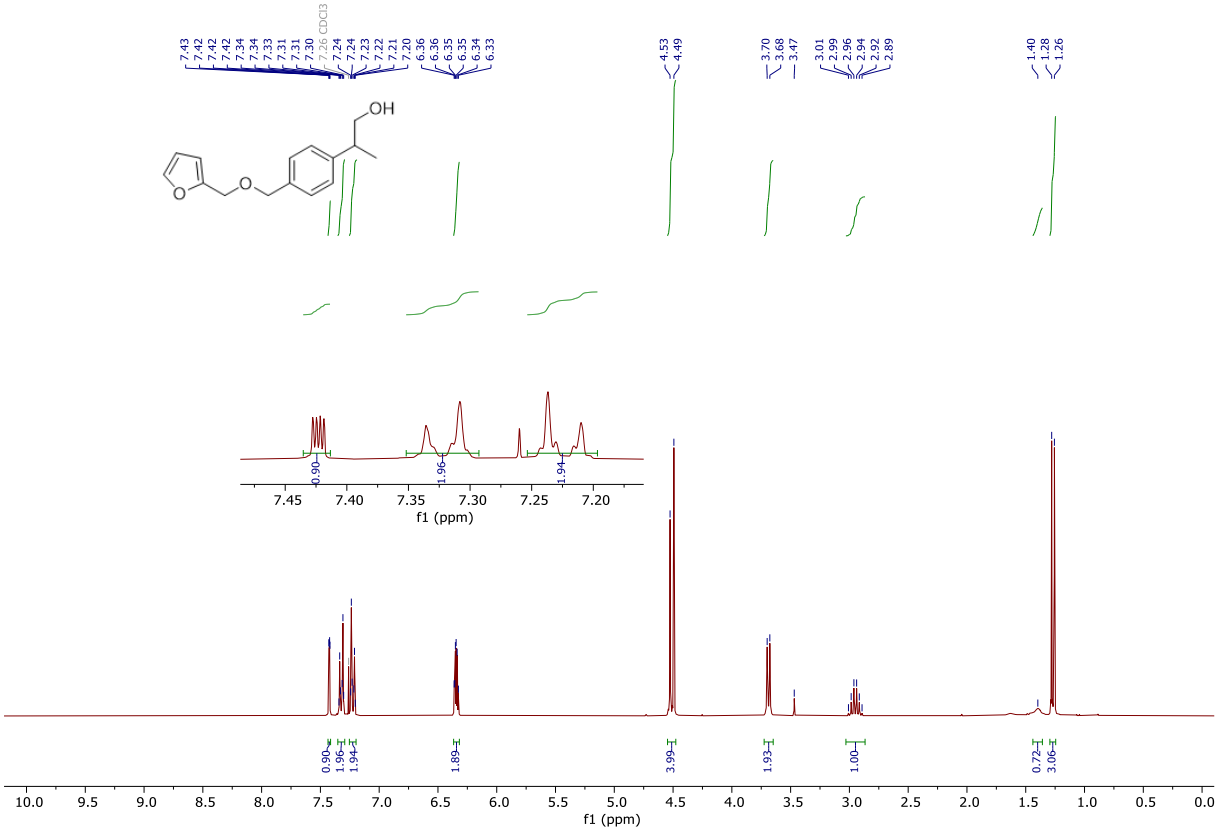
Tim Meyer TM-HY-157 — CDCl3 — 300.20MHz



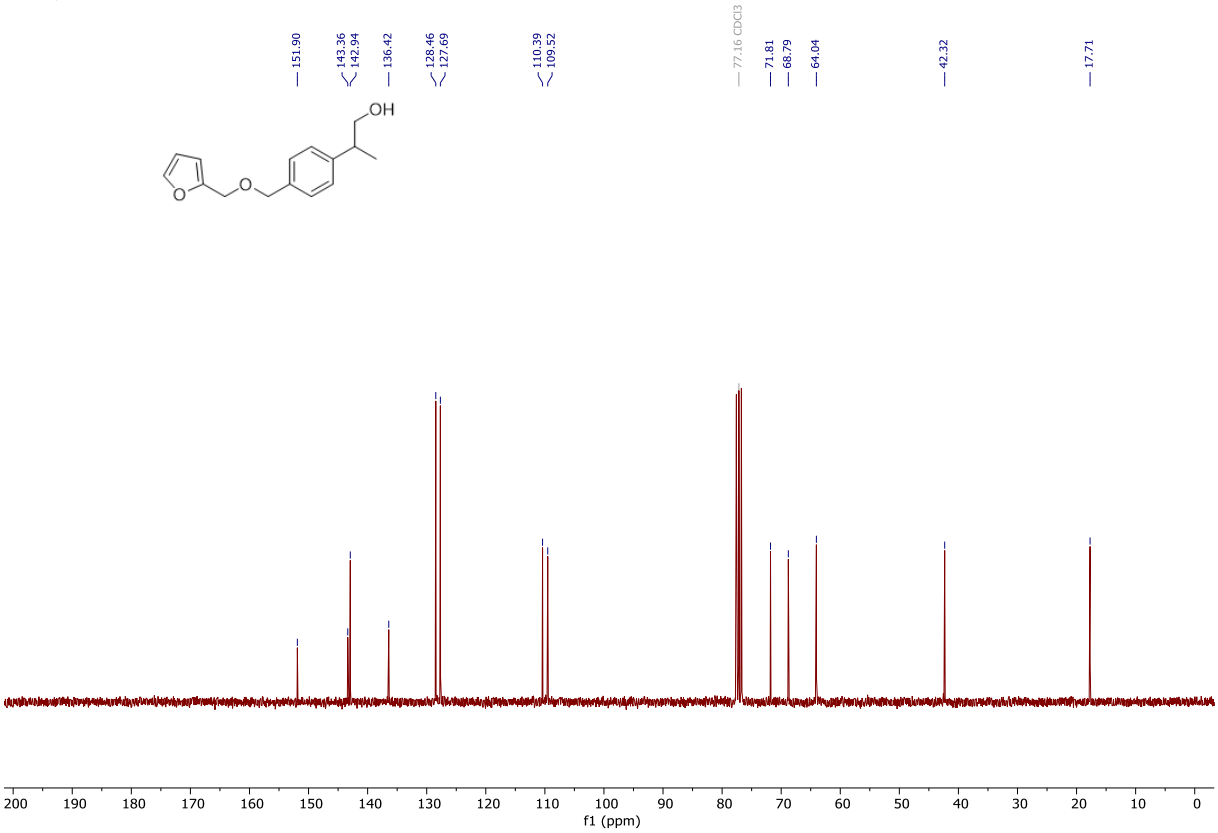
Tim Meyer TM-HY-157 — CDCl3 — 75.49MHz

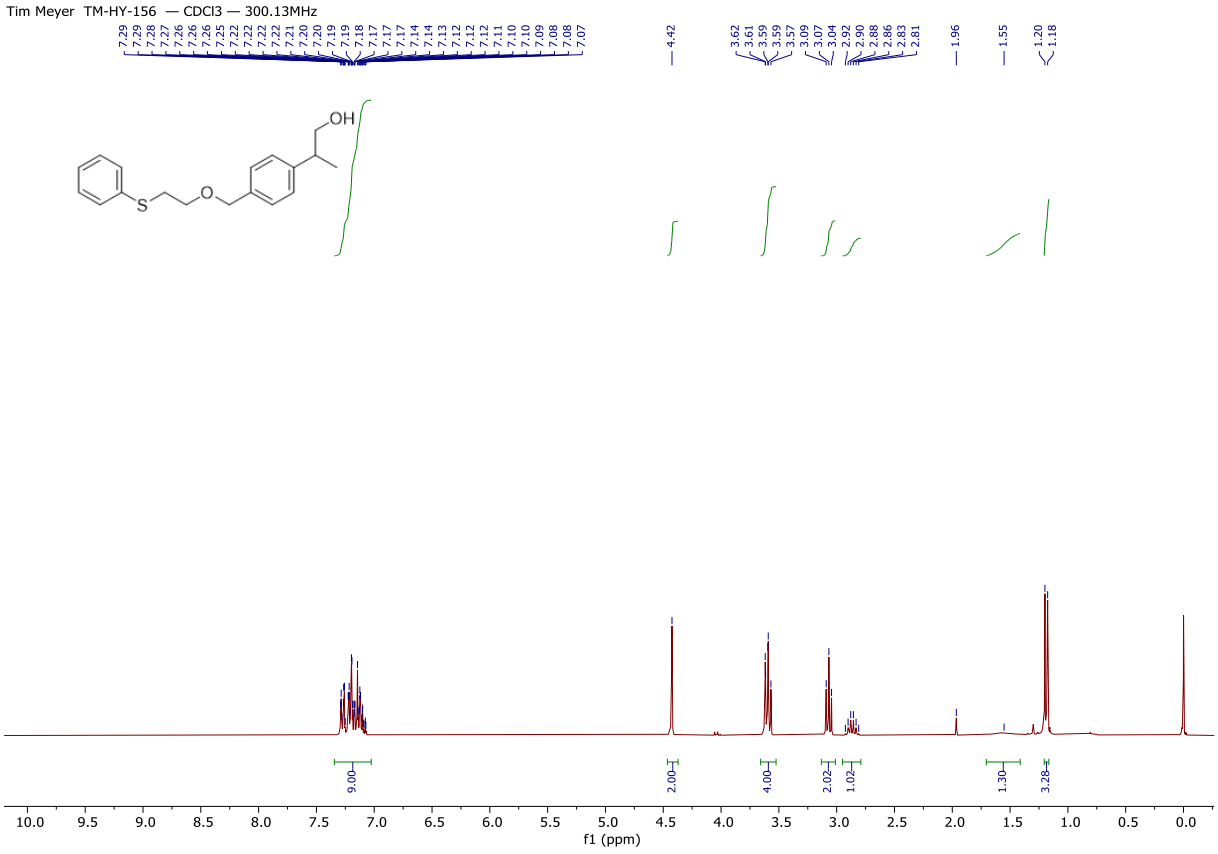


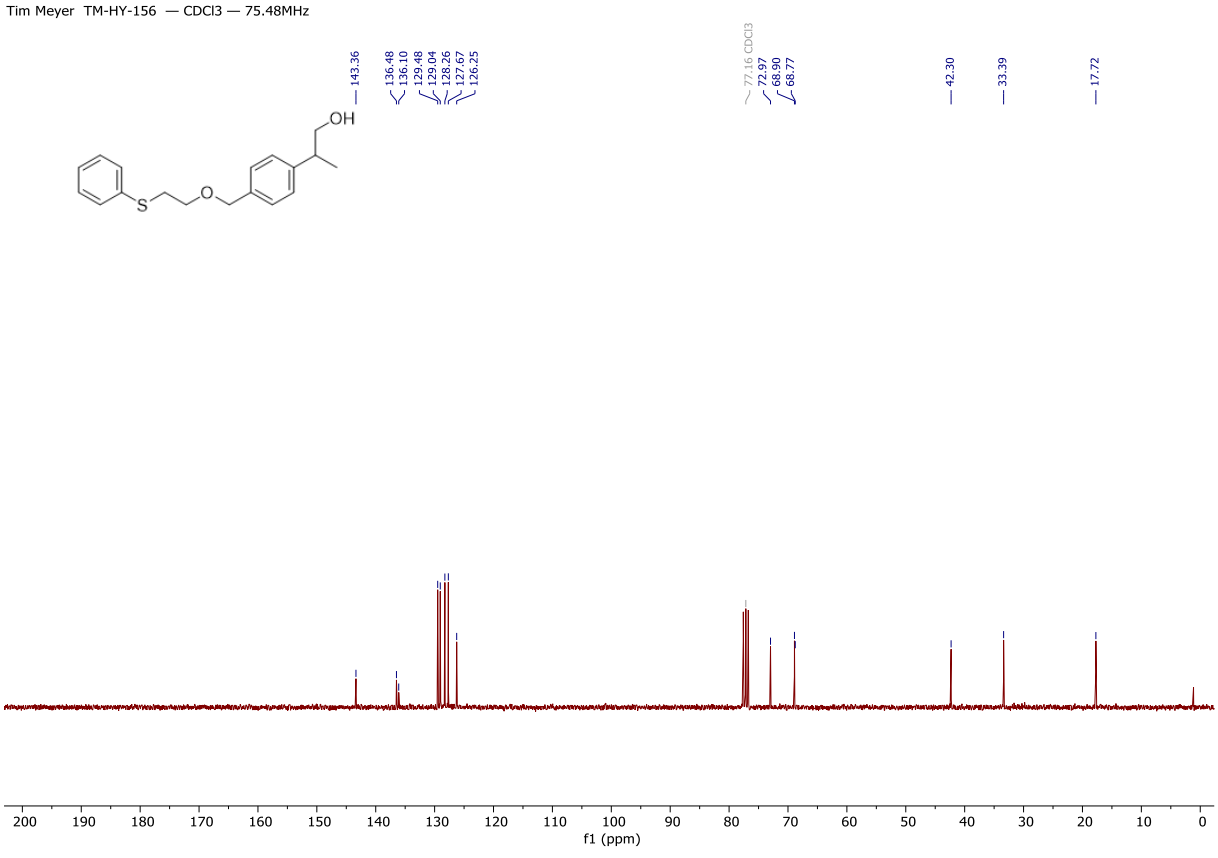
Tim Meyer TM-HY-158 — CDCl3 — 300.20MHz

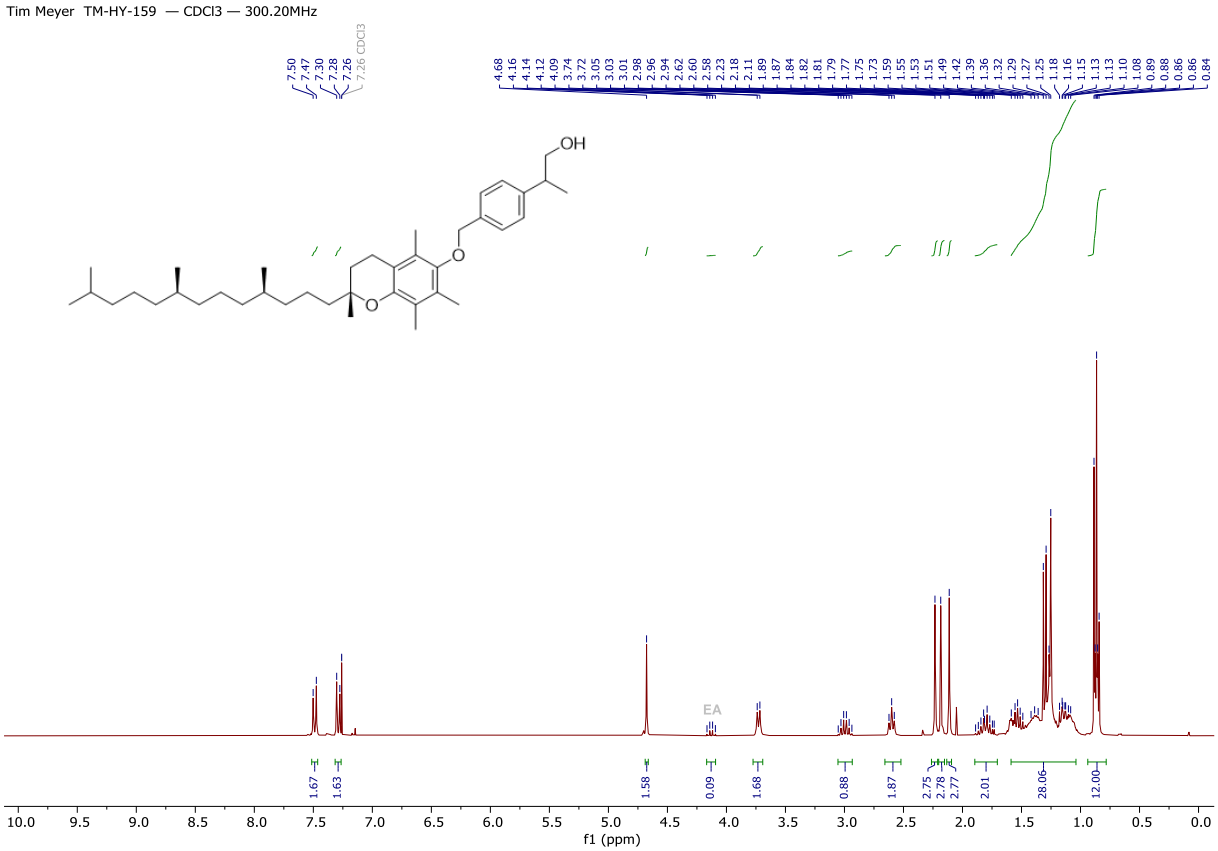


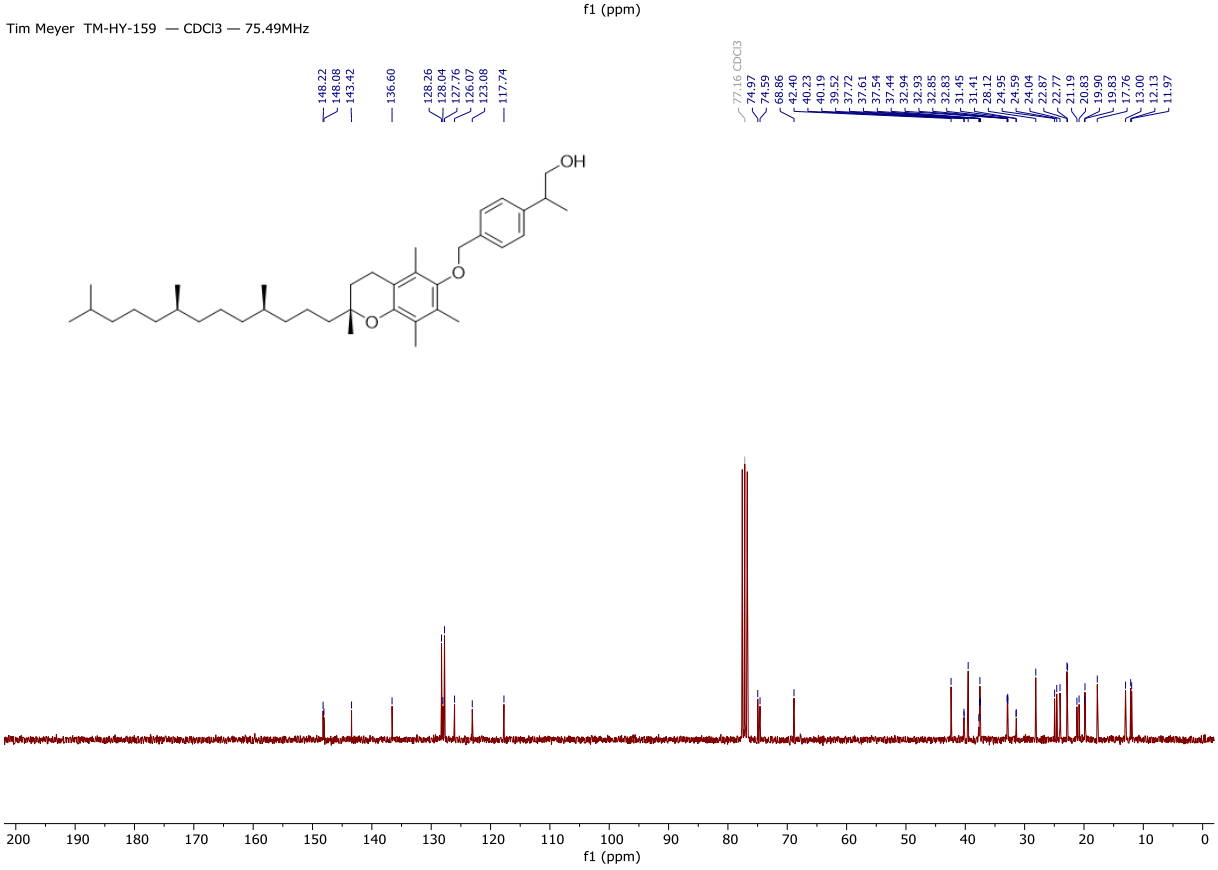
Tim Meyer TM-HY-158 — CDCl3 — 75.49MHz




Tim Meyer TM-HY-156 — CDCl3 — 300.13MHz
7.29 7.29 7.28 7.27 7.26 7.26 7.26 7.25 7.22 7.22 7.22 7.21 7.20 7.20 7.19 7.19 7.18 7.17 7.17 7.17 7.14 7.14 7.13 7.12 7.12 7.12 7.11 7.10 7.09 7.08 7.08 7.07
4.42
3.62 3.61 3.59 3.59 3.57 3.09 3.07 3.04 2.92 2.90 2.88 2.86 2.83 2.81
1.96
1.55
1.20 1.18
OH
S
O
9.00
2.00
4.00
2.02
1.02
1.30
3.28
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

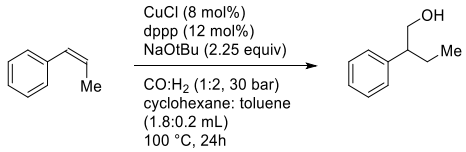

Tim Meyer TM-HY-156 — CDCl3 — 75.48MHz
143.36
136.48
136.10
129.48
129.04
128.26
127.67
126.25
77.16 CDCl3
72.97
68.90
68.77
42.30
33.39
17.72
OH
S
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Tim Meyer TM-HY-159 — CDCl3 — 300.20MHz
OH
O
O
EA
f1 (ppm)


Tim Meyer TM-HY-159 — CDCl3 — 75.49MHz
OH
O
O
f1 (ppm)

Internal Alkenes

2-Phenylbutan-1-ol (3z)



The title compound was prepared from (*Z*)-prop-1-en-1-ylbenzene (56 µL, 0.43 mmol), CuCl (3.5 mg, 8 mol%), DPPP (20.5 mg, 12 mol%), and NaO*t*Bu (93.5 mg, 2.25 equiv) in toluene (2.5 mL, 0.17 M). following General Procedure A.

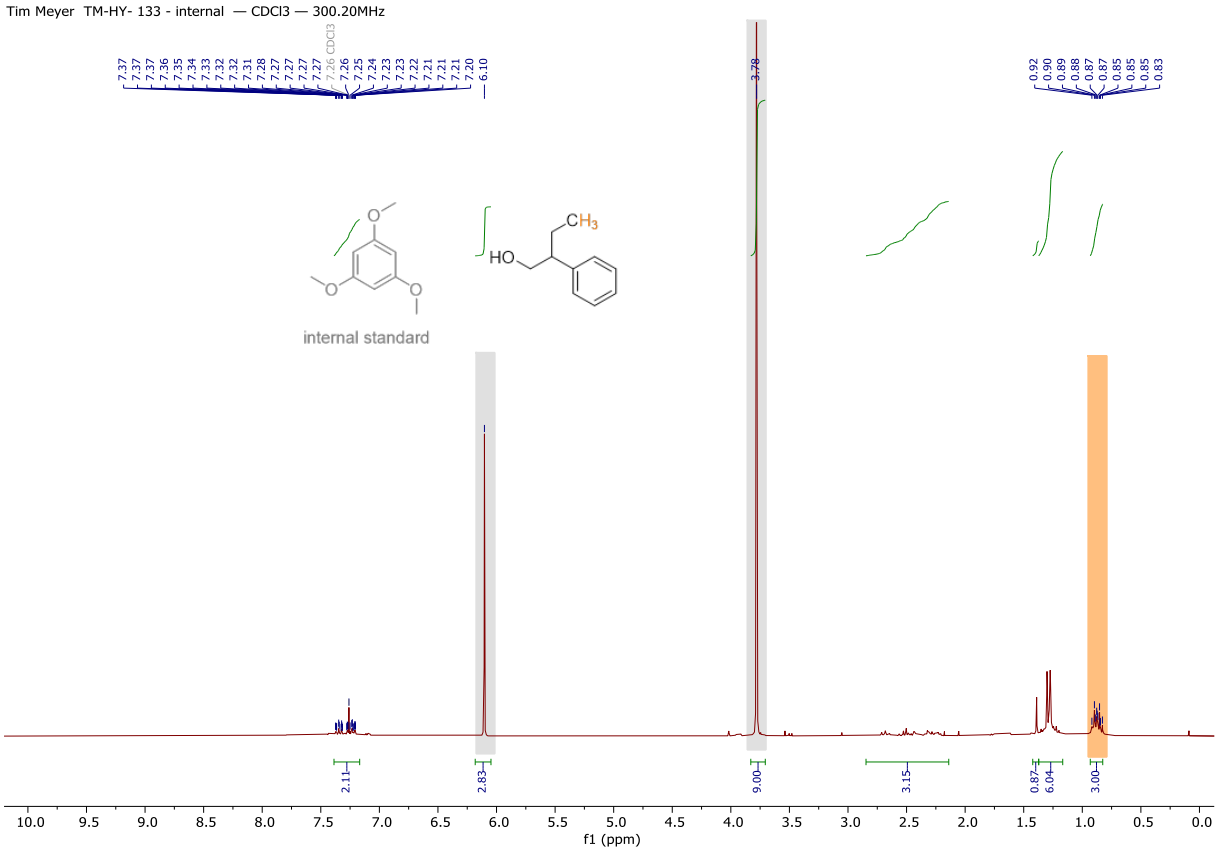
*** Yield was determined by ^{1}H NMR spectroscopy due to the low conversion (obviously a polymerization took place, see Figure #1).***

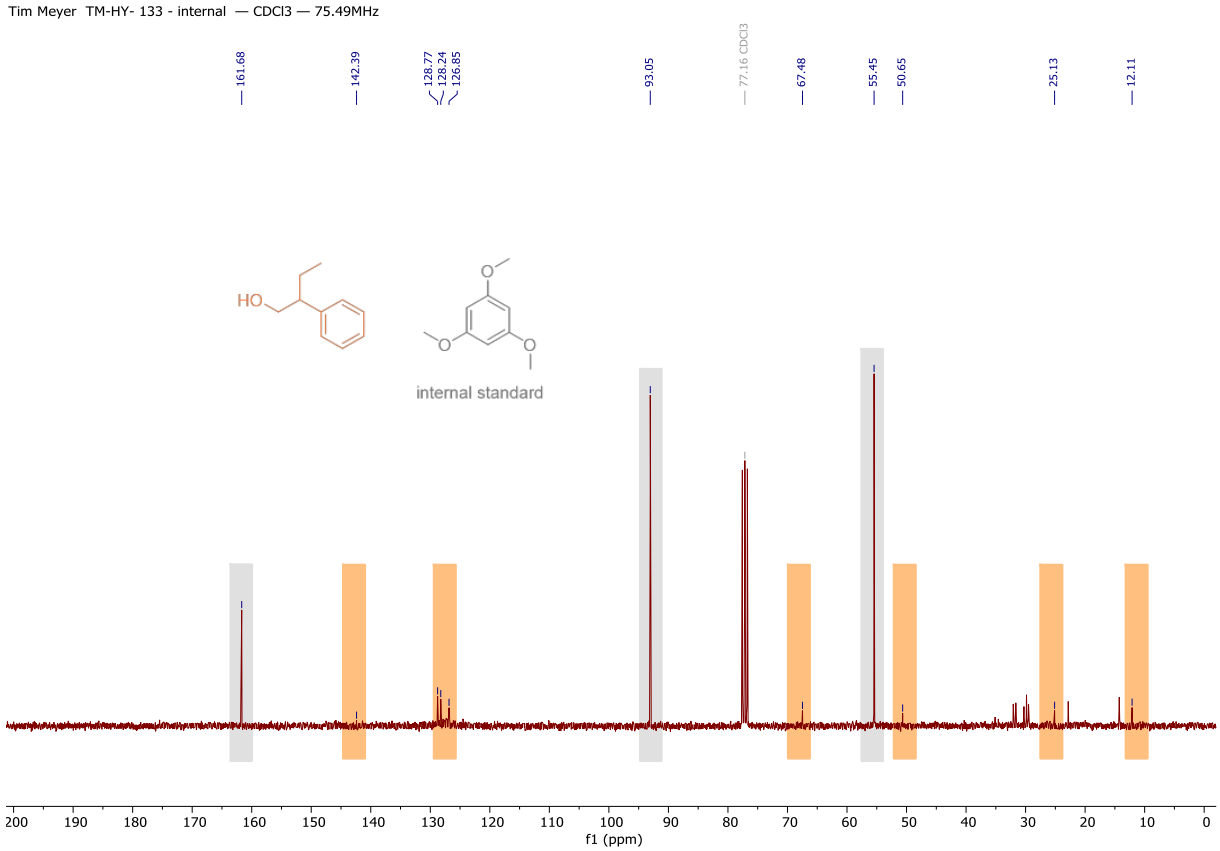
TMB (8.4 mg, 0.05 mmol) was added as an internal standard. From this the NMR-yield was calculated to be around 12%.

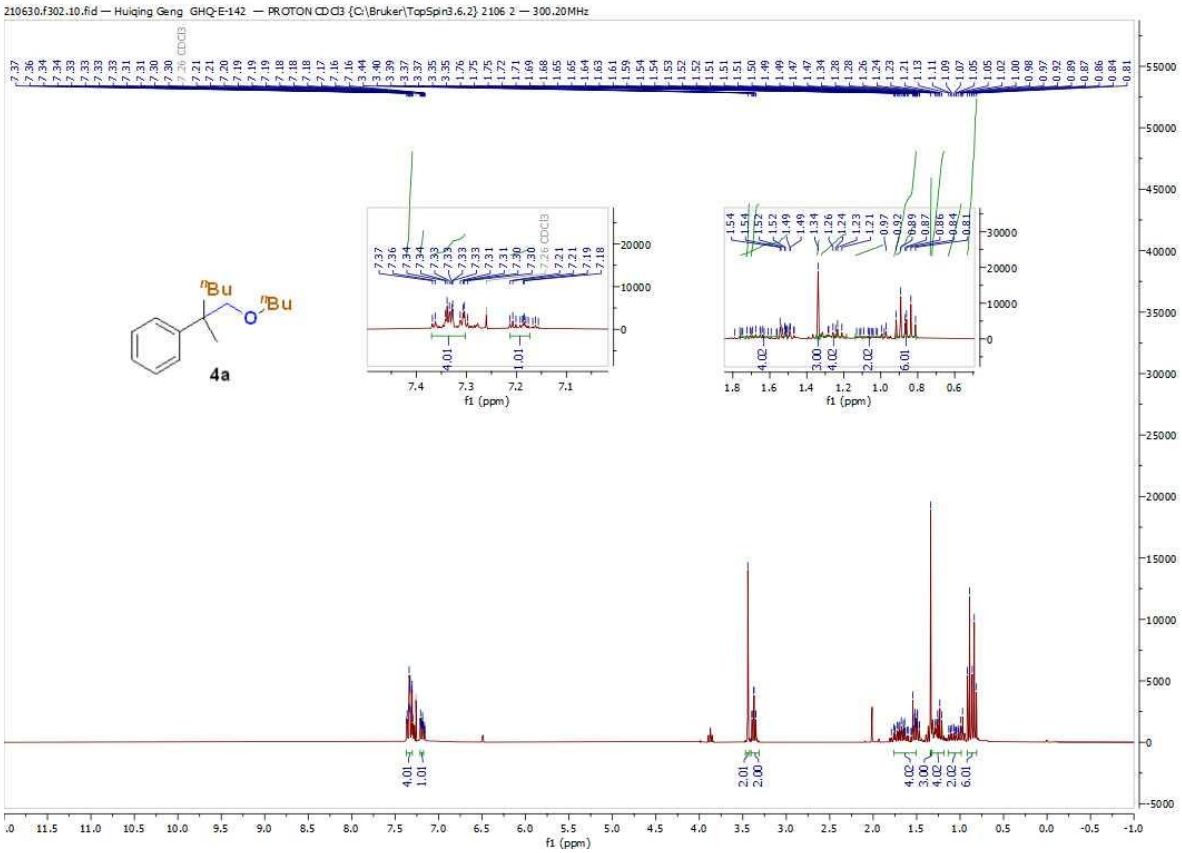
^{1}H NMR (300 MHz, Chloroform-*d*) δ 0.93 – 0.83 (m, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 142.4, 128.8, 128.2, 126.9, 67.5, 50.7, 25.1, 12.1.

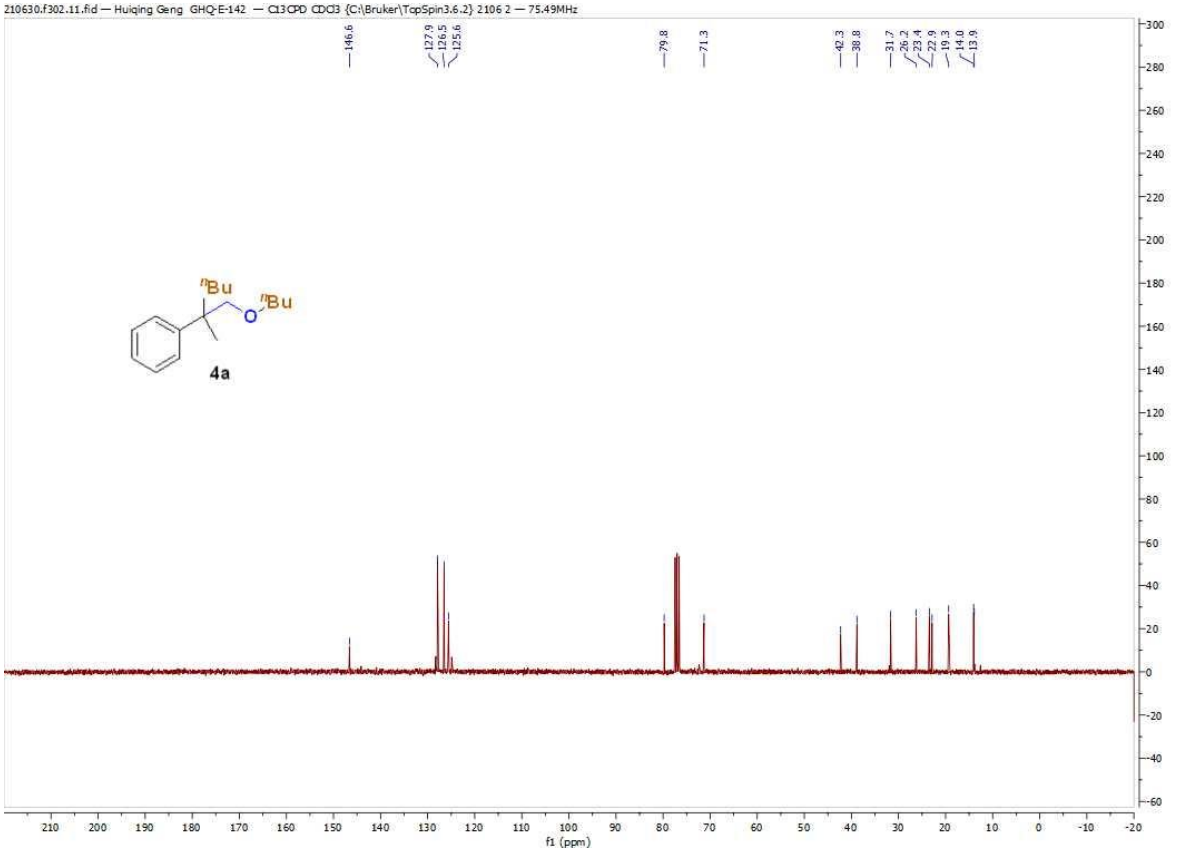
For ^{13}C NMR the data were in agreement with published values.*

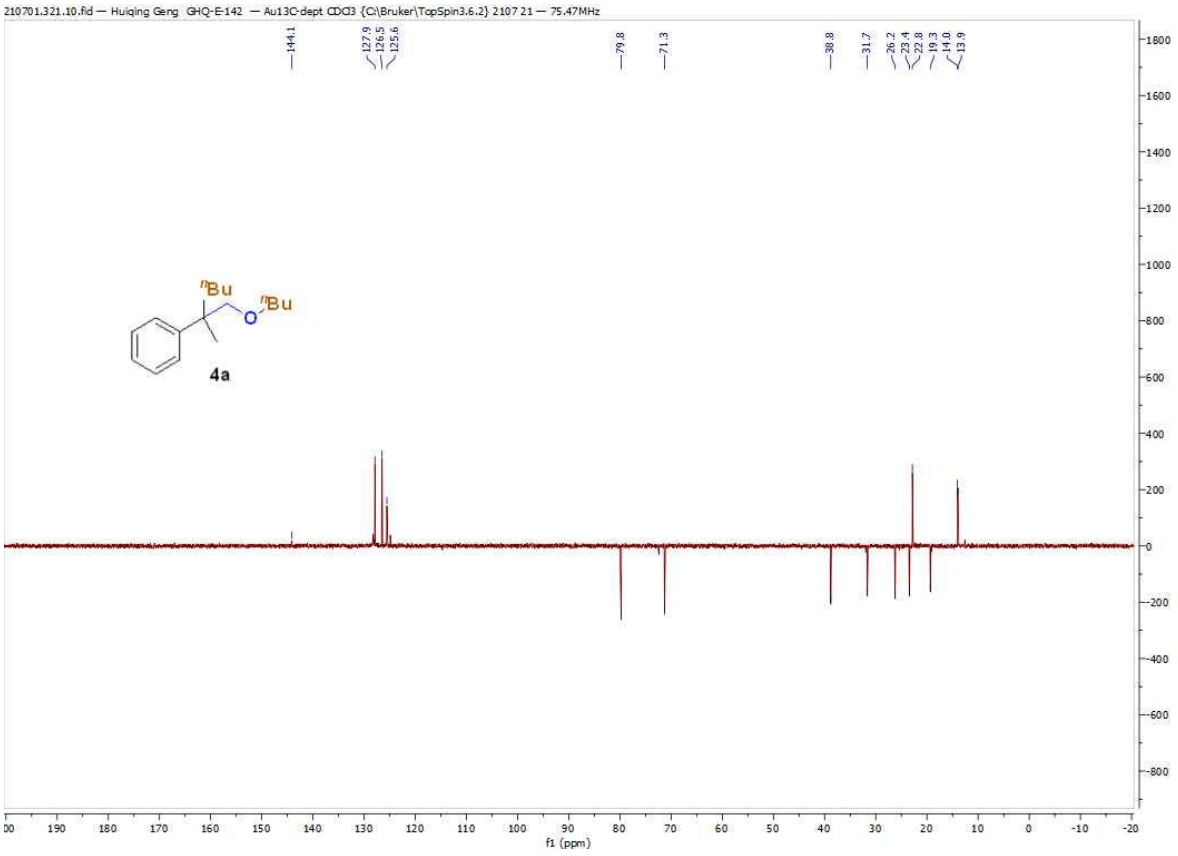
*M. Magre, E. Paffenholz, B. Maity, L. Cavallo, and M. Rueping, *J. Am. Chem. Soc.* **2020**, *142*, 14286.

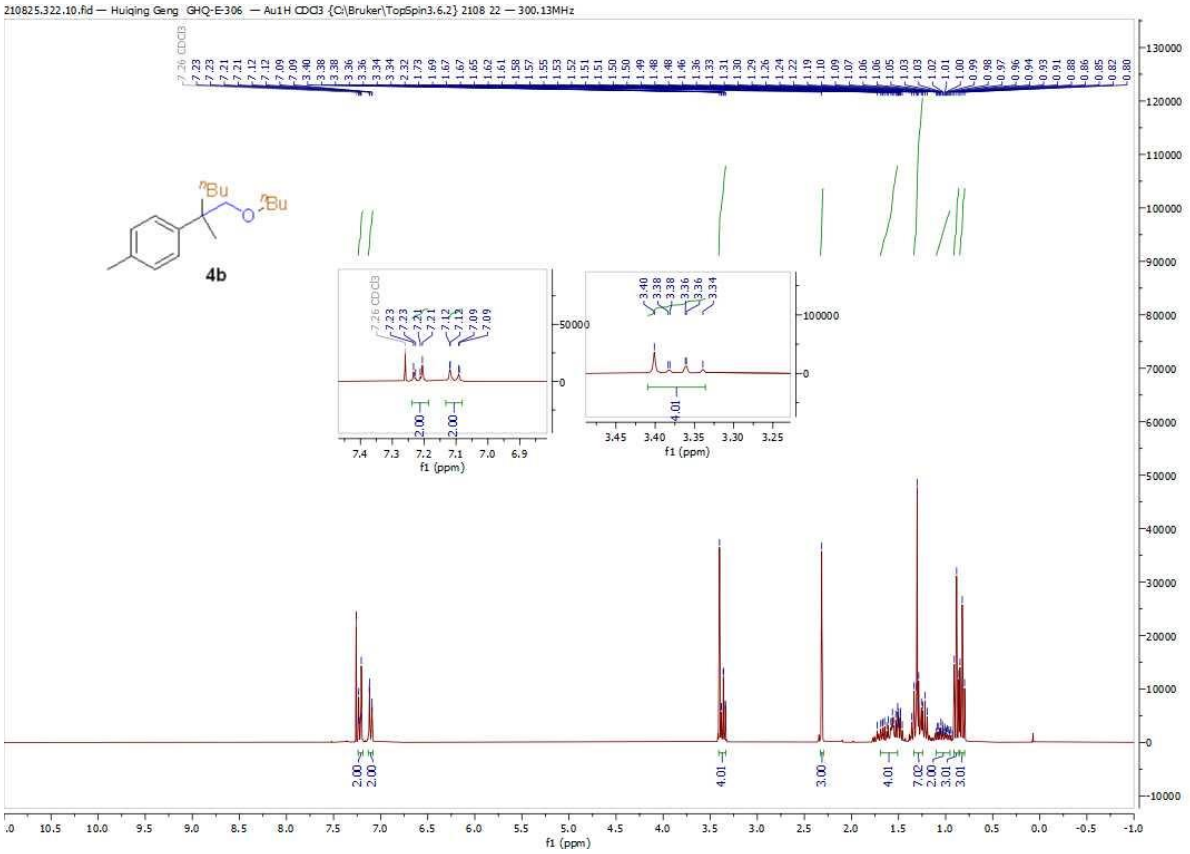

Tim Meyer TM-HY- 133 - internal — CDCl3 — 300.20MHz
internal standard
HO
CH3
f1 (ppm)

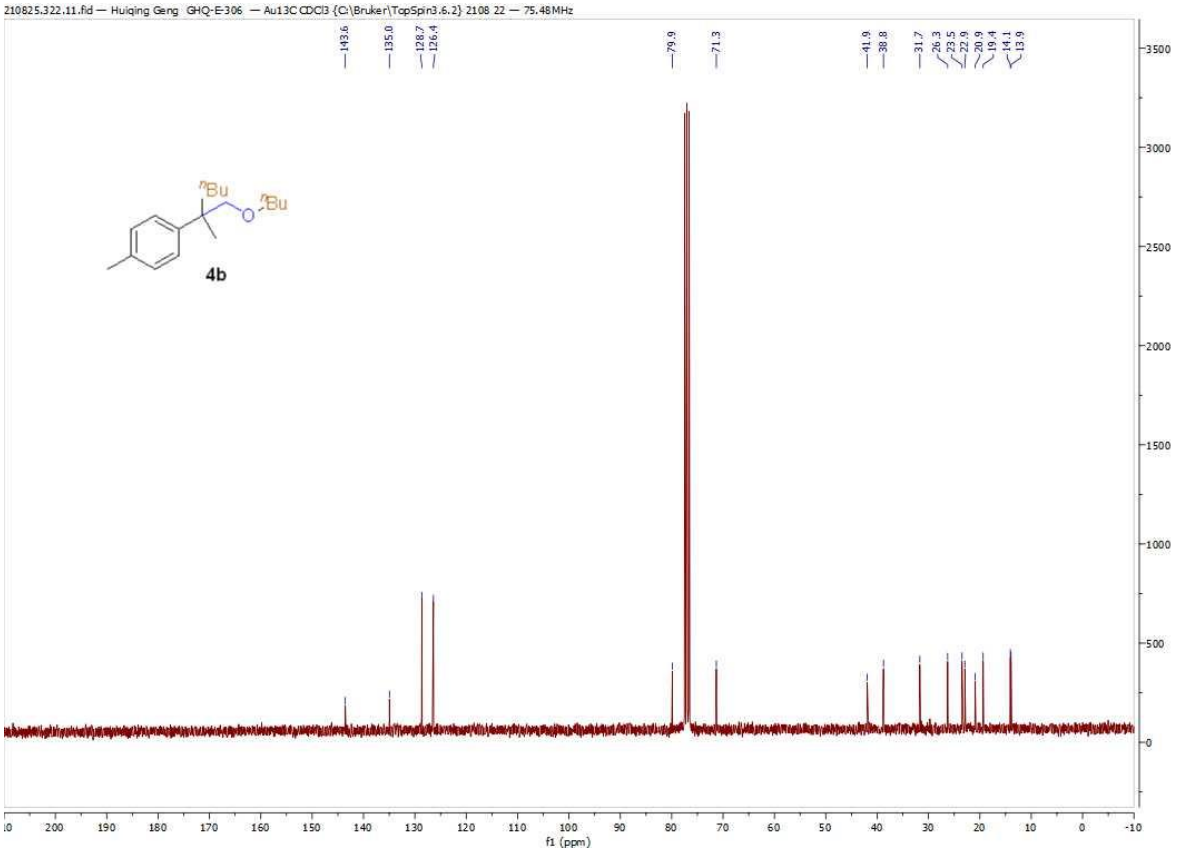

Tim Meyer TM-HY- 133 - internal — CDCl3 — 75.49MHz
161.68
142.39
128.77
128.24
126.85
93.05
77.16 CDCl3
67.48
55.45
50.65
25.13
12.11
HO
internal standard
f1 (ppm)

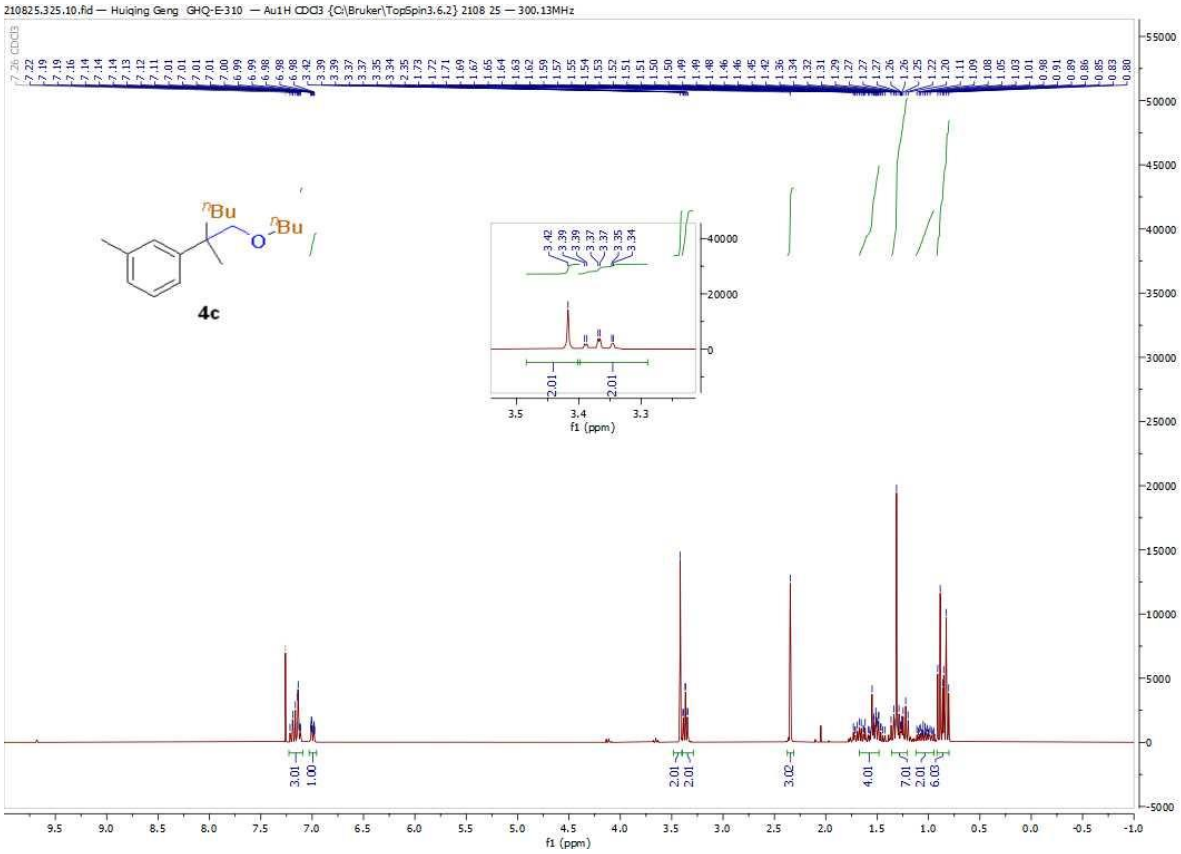

210630.f302.10.fid — Huiqing Geng GHQ-E-142 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 300.20MHz
nBu
nBu
4a
4.01
1.01
2.01
2.00
4.02
3.00
4.02
2.02
6.01
f1 (ppm)

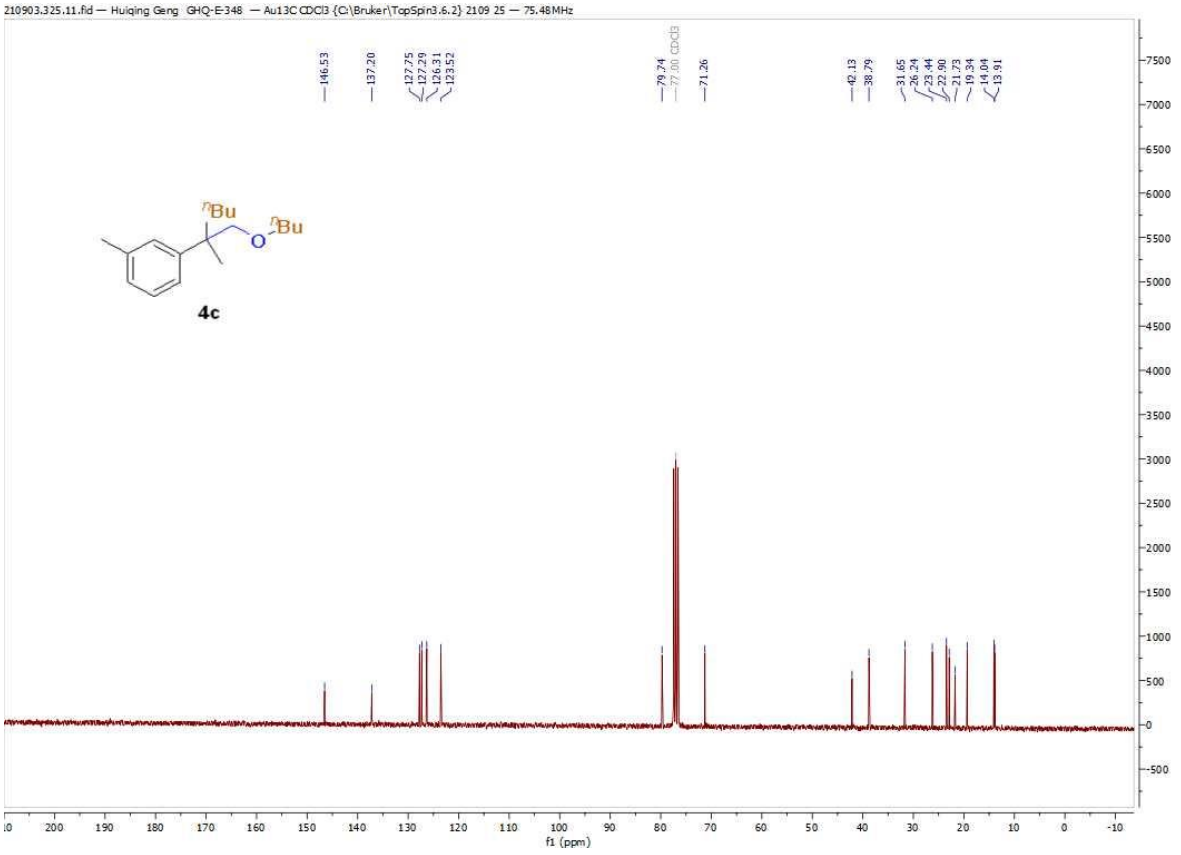

210630.f302.11.fid — Huiqing Geng GHQ-E-142 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 2 — 75.49MHz
146.6
127.9
126.5
125.6
79.8
71.3
42.3
38.8
31.7
26.2
23.4
22.9
19.3
14.0
13.9
nBu
nBu
O
4a
f1 (ppm)

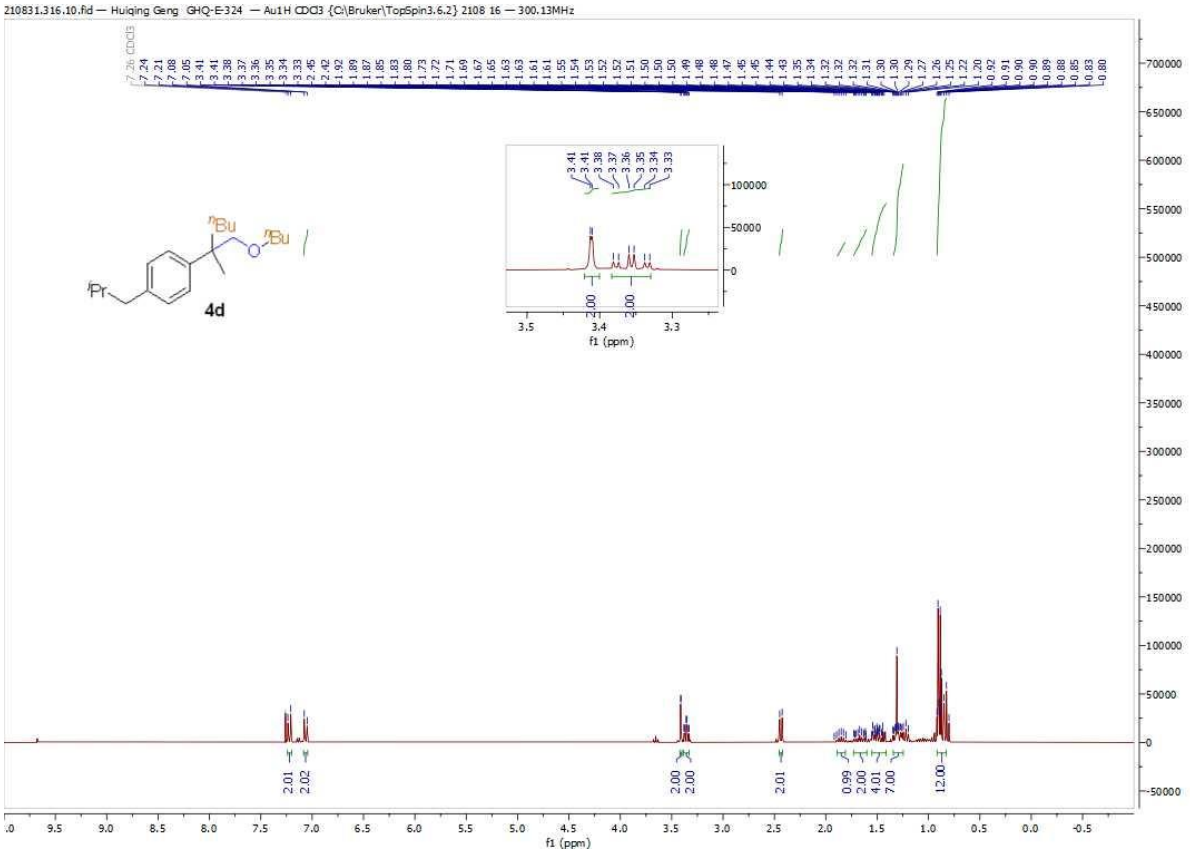

210701.321.10.fid — Huiqing Geng GHQ-E-142 — Au13C-dept CDCl3 {C:\Bruker\TopSpin3.6.2} 2107 21 — 75.47MHz
144.1
127.9
126.5
125.6
79.8
71.3
38.8
31.7
26.2
23.4
22.8
19.3
14.0
13.9
nBu
nBu
O
4a
f1 (ppm)

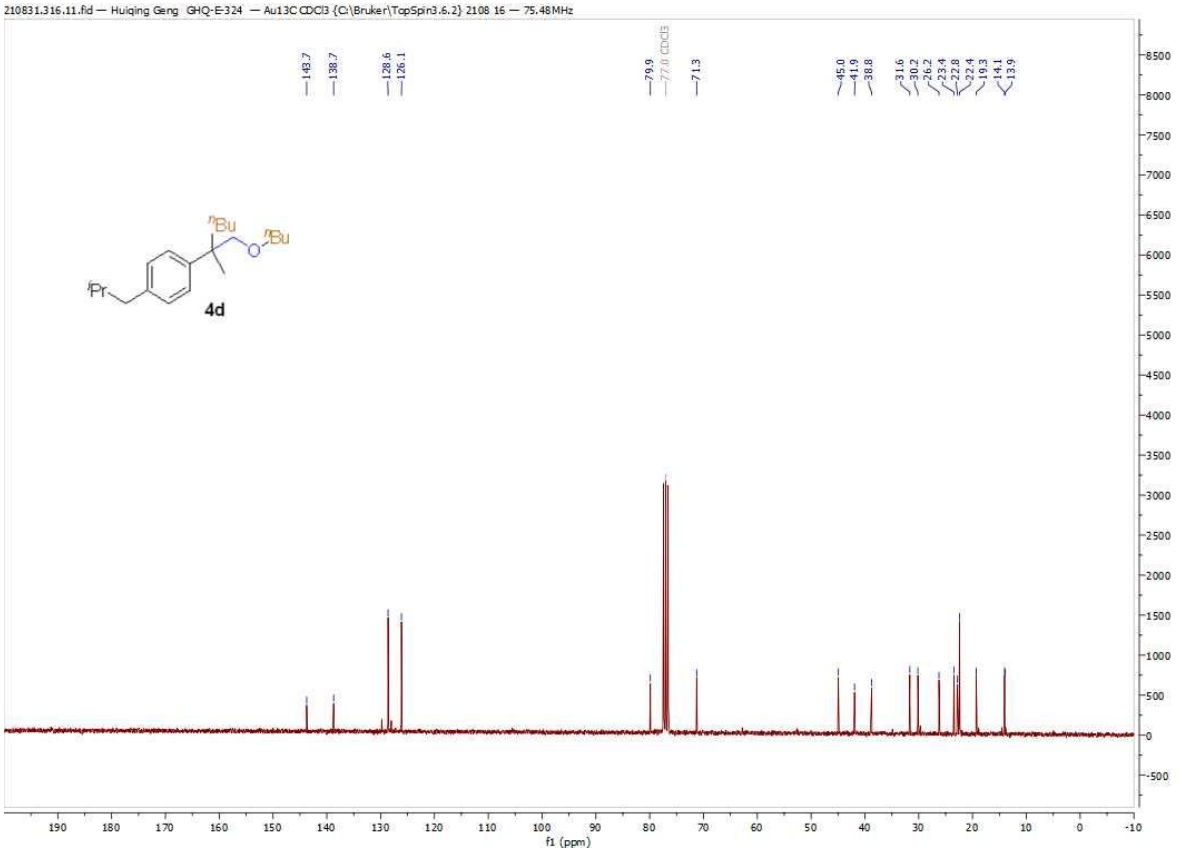

210825.322.10.fid — Huiqing Geng GHQ-E-306 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 22 — 300.13MHz
4b
f1 (ppm)

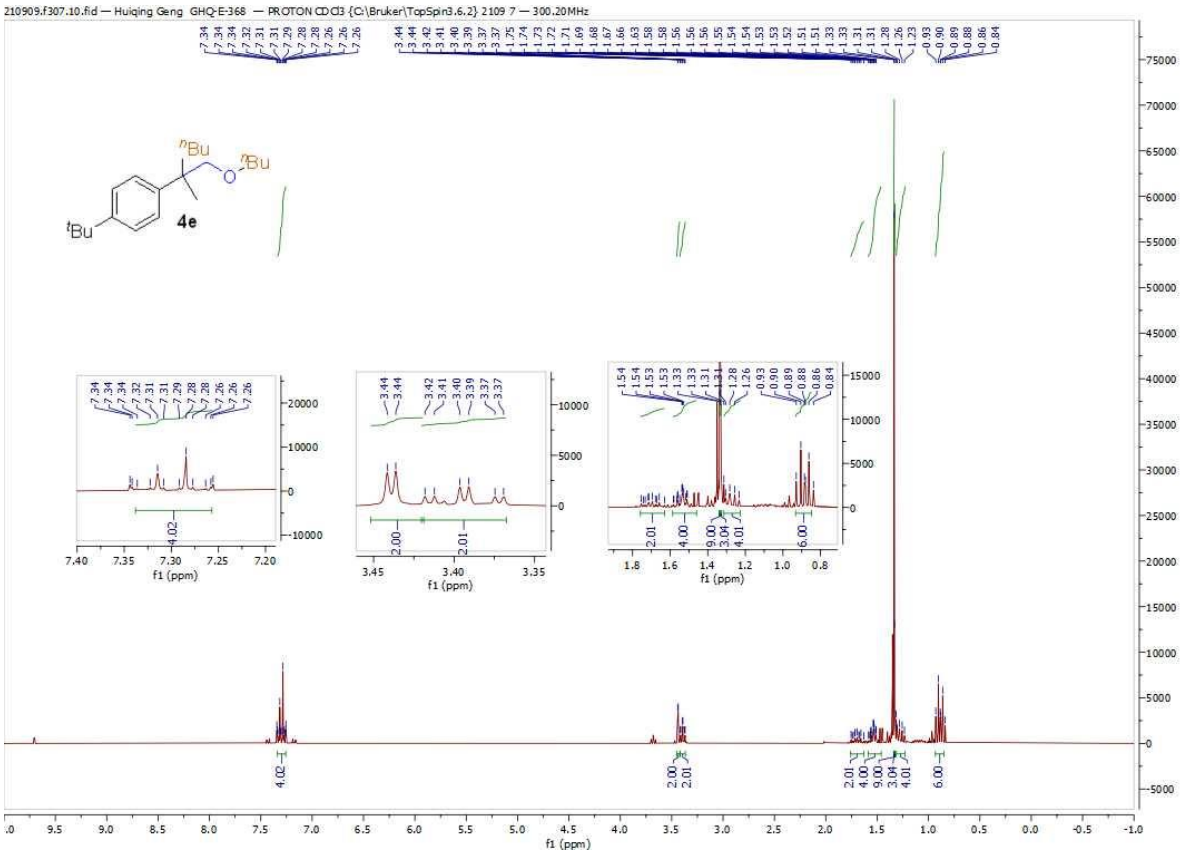

210825.322.11.fid — Huiqing Geng GHQ-E-306 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 22 — 75.48MHz
4b
f1 (ppm)

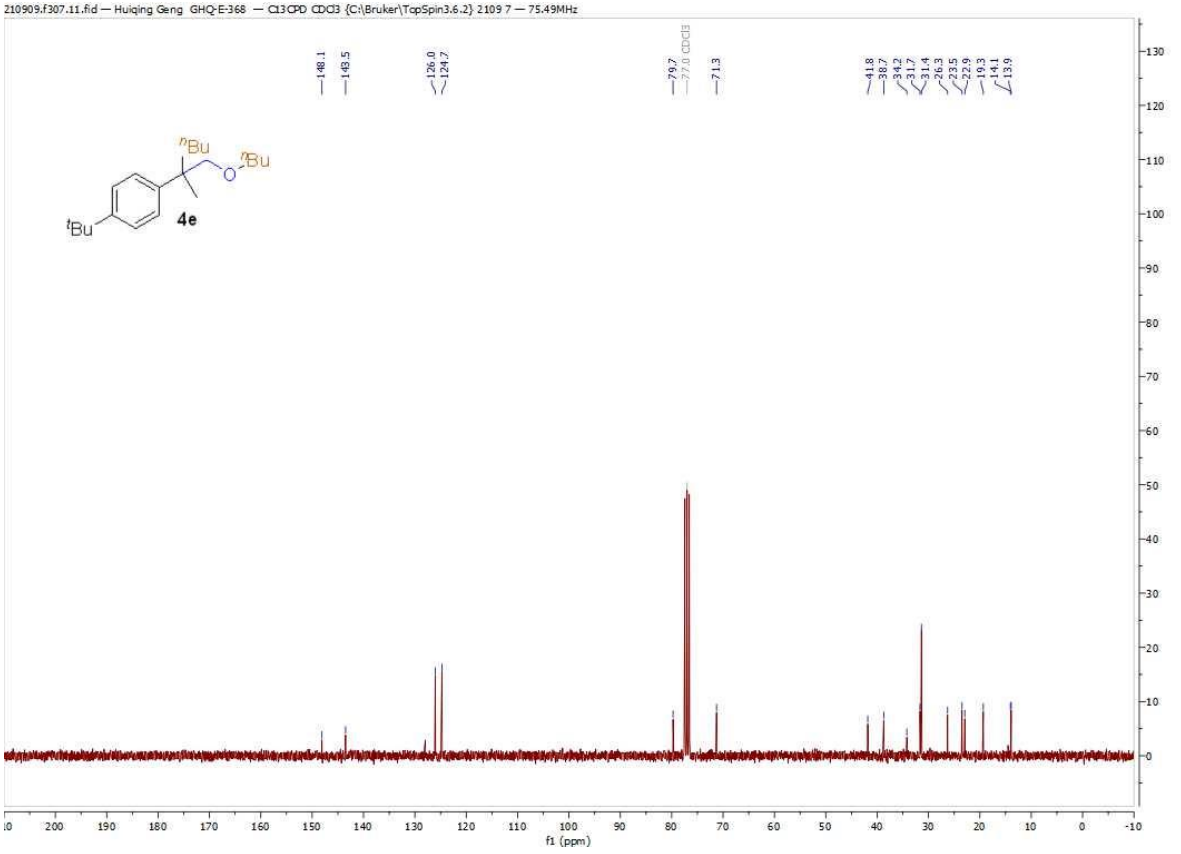

210825.325.10.fid — Huiqing Geng GHQ-E-310 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 25 — 300.13MHz
nBu
nBu
O
4c
f1 (ppm)

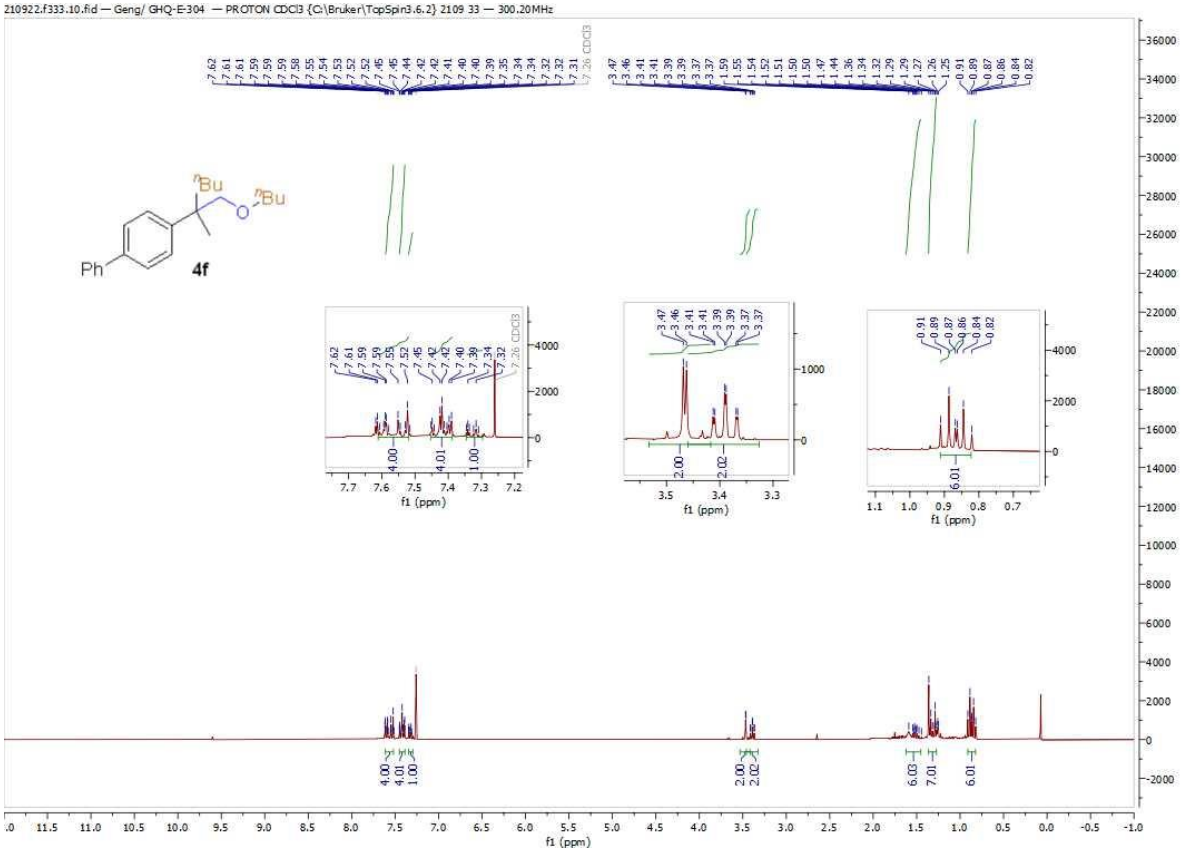

210903.325.11.fid — Huiqing Geng GHQ-E-348 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 25 — 75.48MHz
146.53
137.20
127.75
127.29
126.31
123.52
79.74
77.00 CDCl3
71.26
42.13
38.79
31.65
26.24
23.44
22.90
21.73
19.34
14.04
13.91
nBu
nBu
O
4c
f1 (ppm)

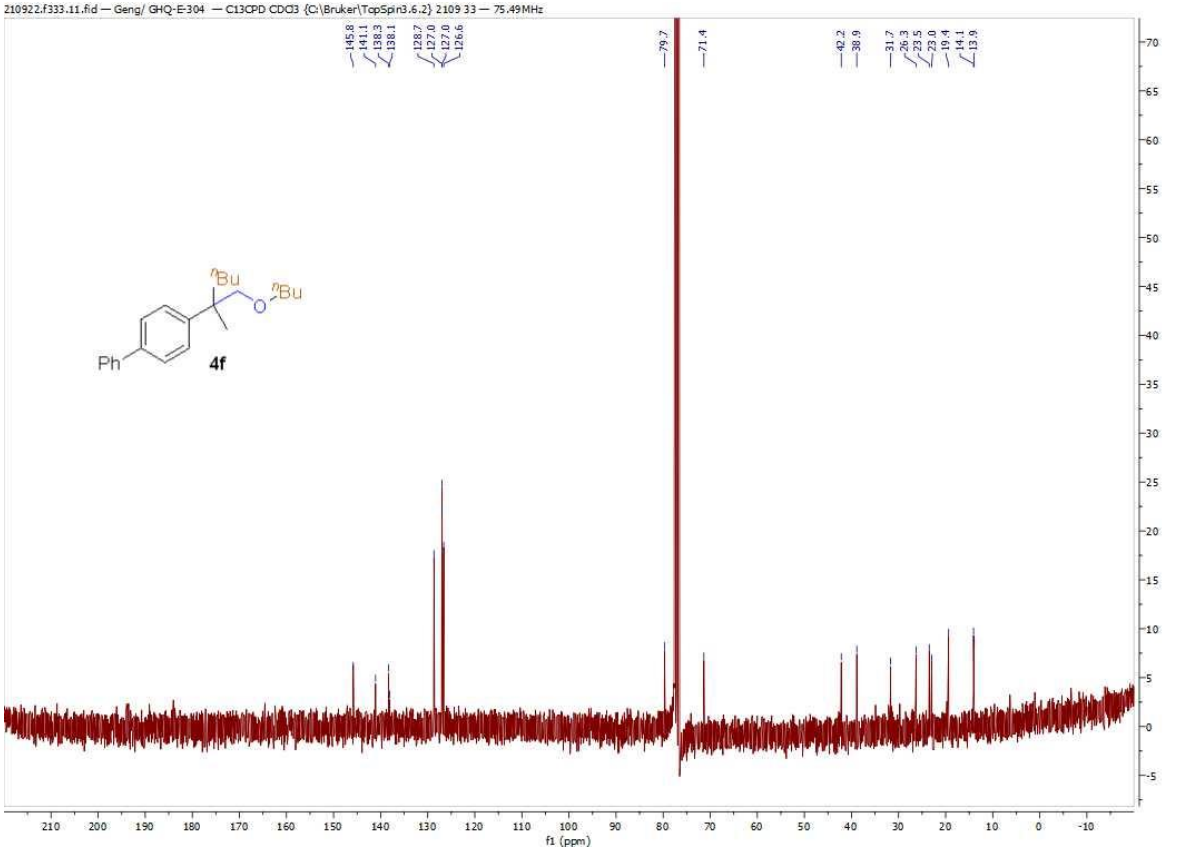

210831.316.10.fid — Huiqing Geng GHQ-E-324 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 16 — 300.13MHz
4d
f1 (ppm)

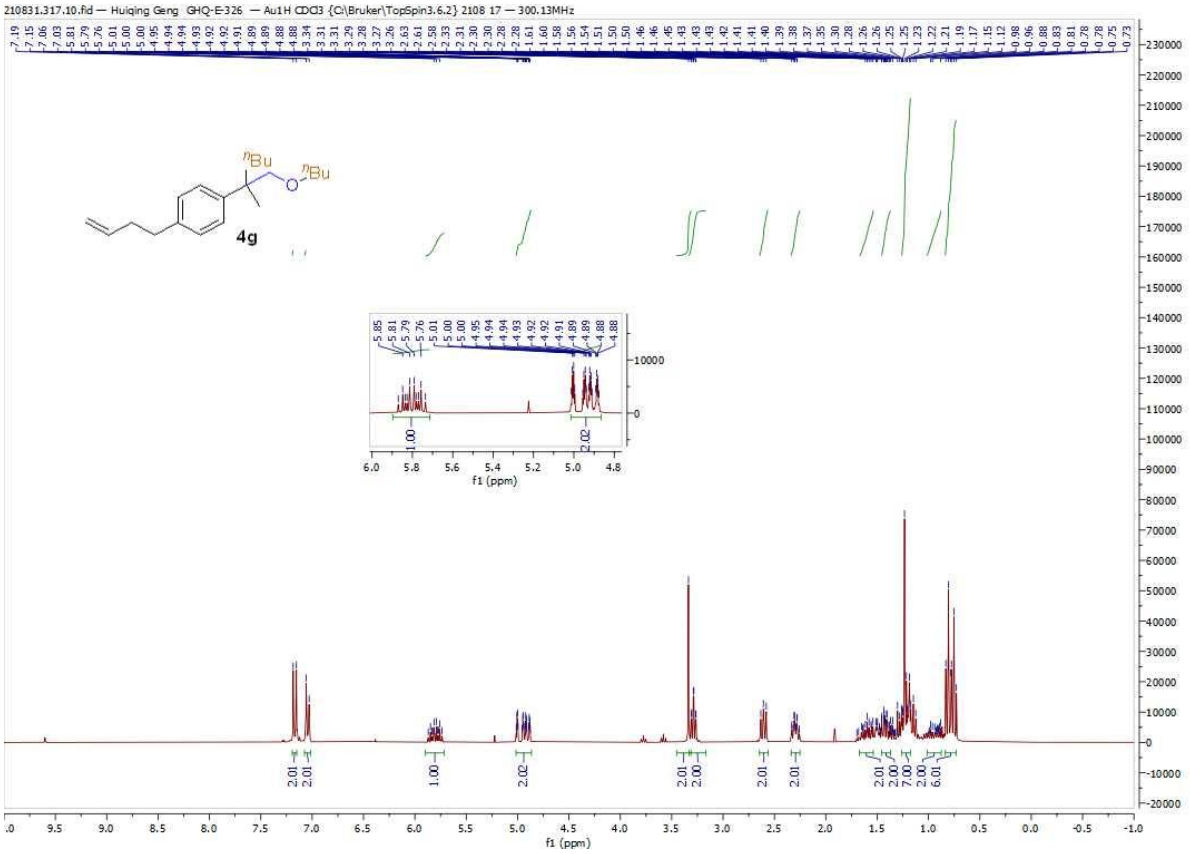

210831.316.11.fid — Huiqing Geng GHQ-E-324 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 16 — 75.48MHz
4d
f1 (ppm)

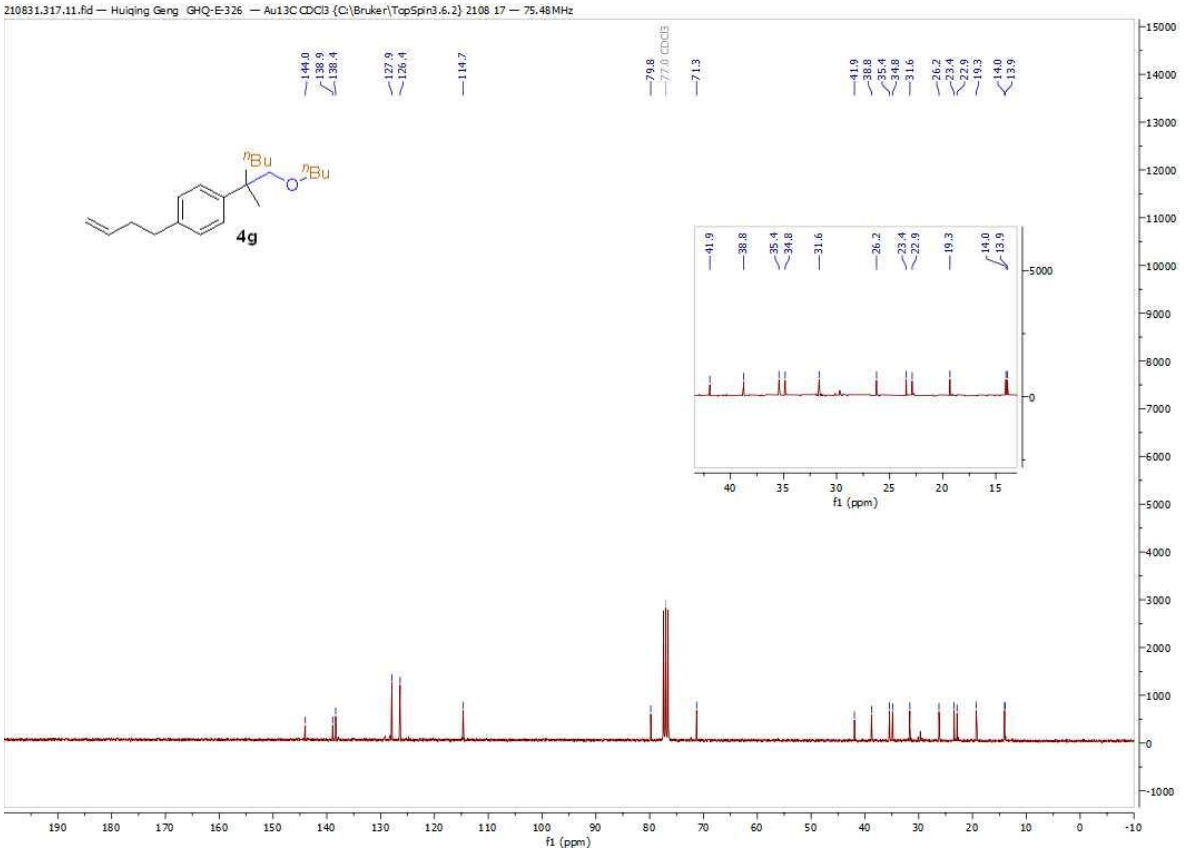

210909.f307.10.fid — Huiqing Geng GHQ-E-368 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 7 — 300.20MHz
nBu
nBu
tBu
4e
f1 (ppm)

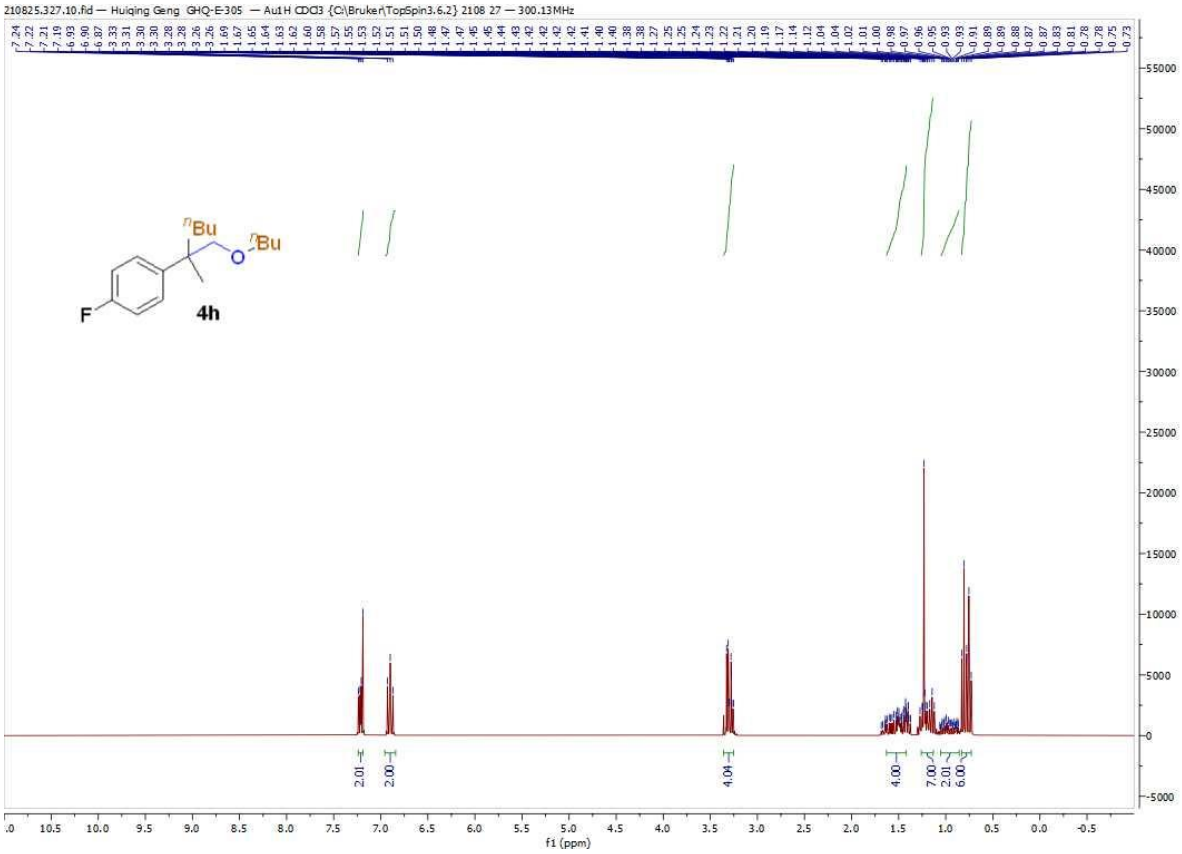

210909.f307.11.fid — Huiqing Geng GHQ-E-368 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 7 — 75.49MHz
nBu
nBu
tBu
4e
f1 (ppm)

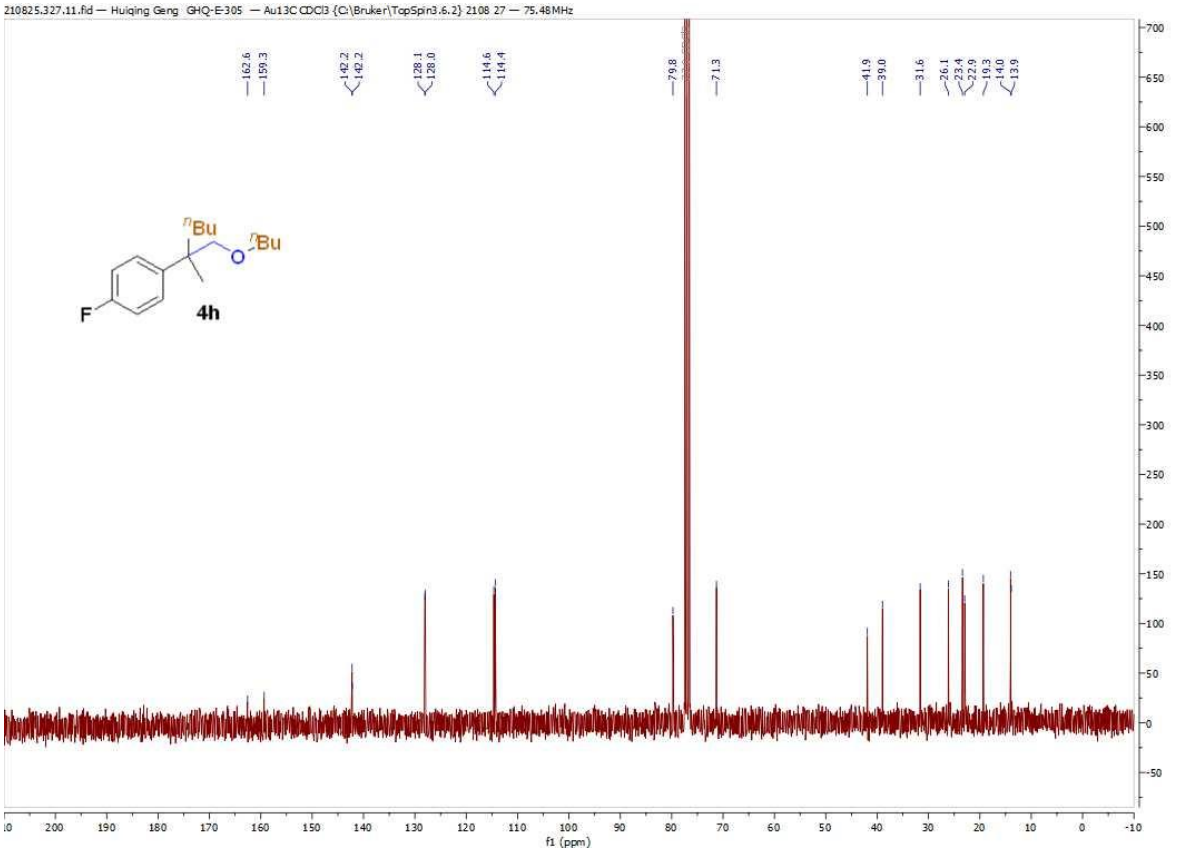

210922.f333.10.fid — Geng/ GHQ-E-304 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 33 — 300.20MHz
nBu
nBu
O
Ph
4f
f1 (ppm)

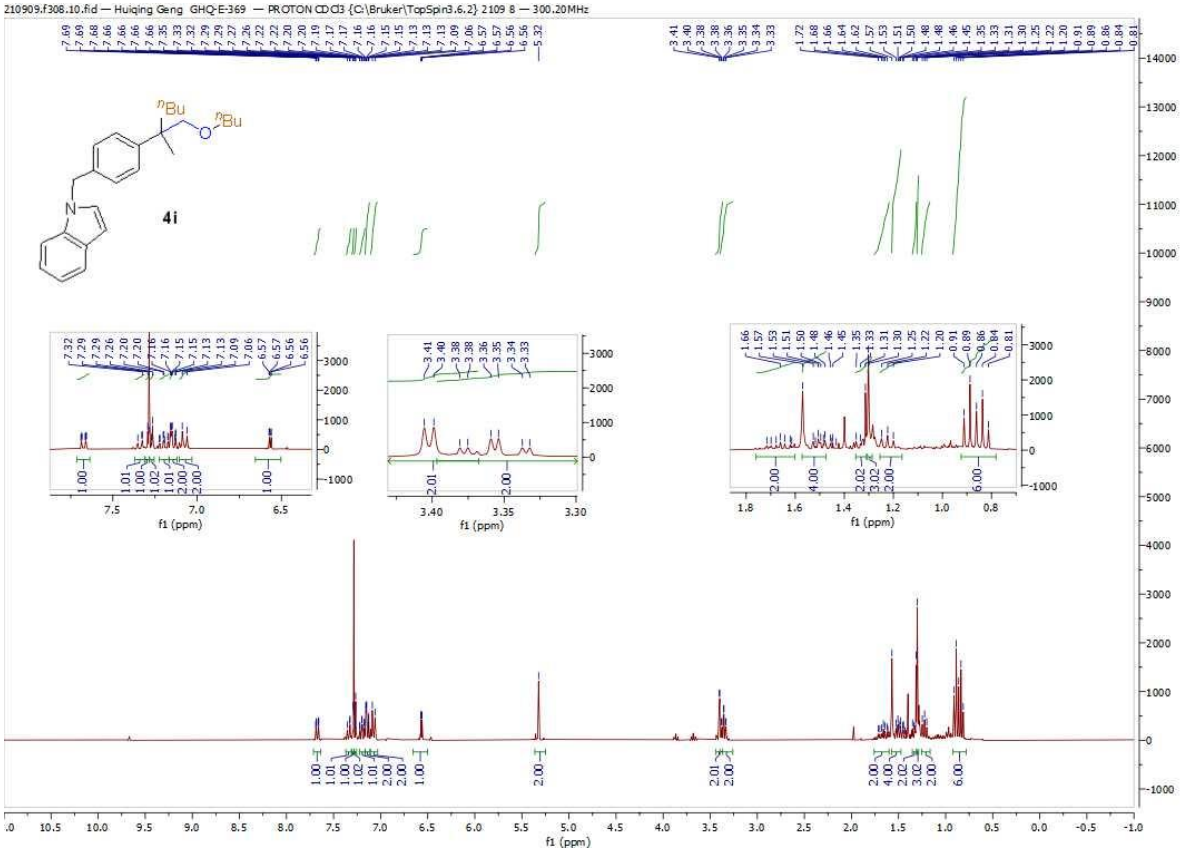

210922.f333.11.fid — Geng/ GHQ-E-304 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 33 — 75.49MHz
nBu
nBu
O
Ph
4f
f1 (ppm)

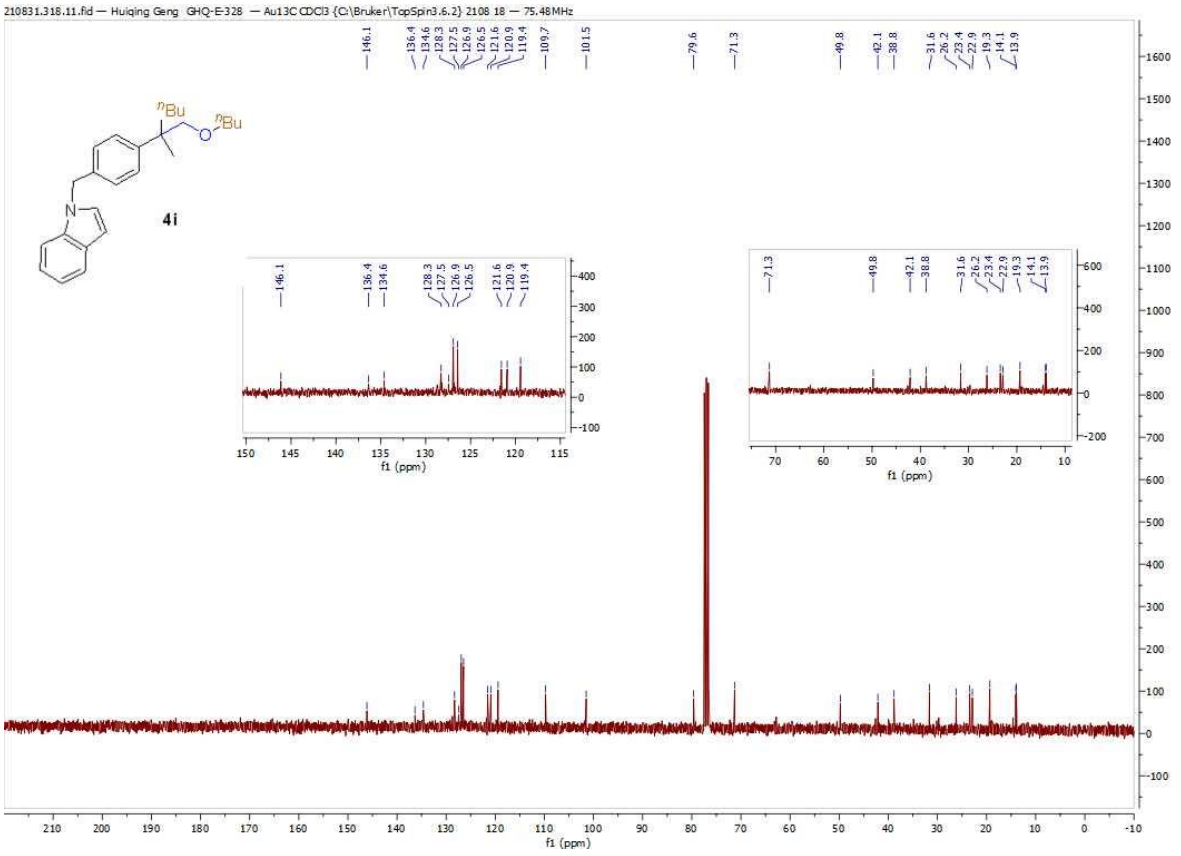

210831.317.10.fid — Huiqing Geng GHQ-E-326 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 17 — 300.13MHz
nBu
nBu
4g
f1 (ppm)

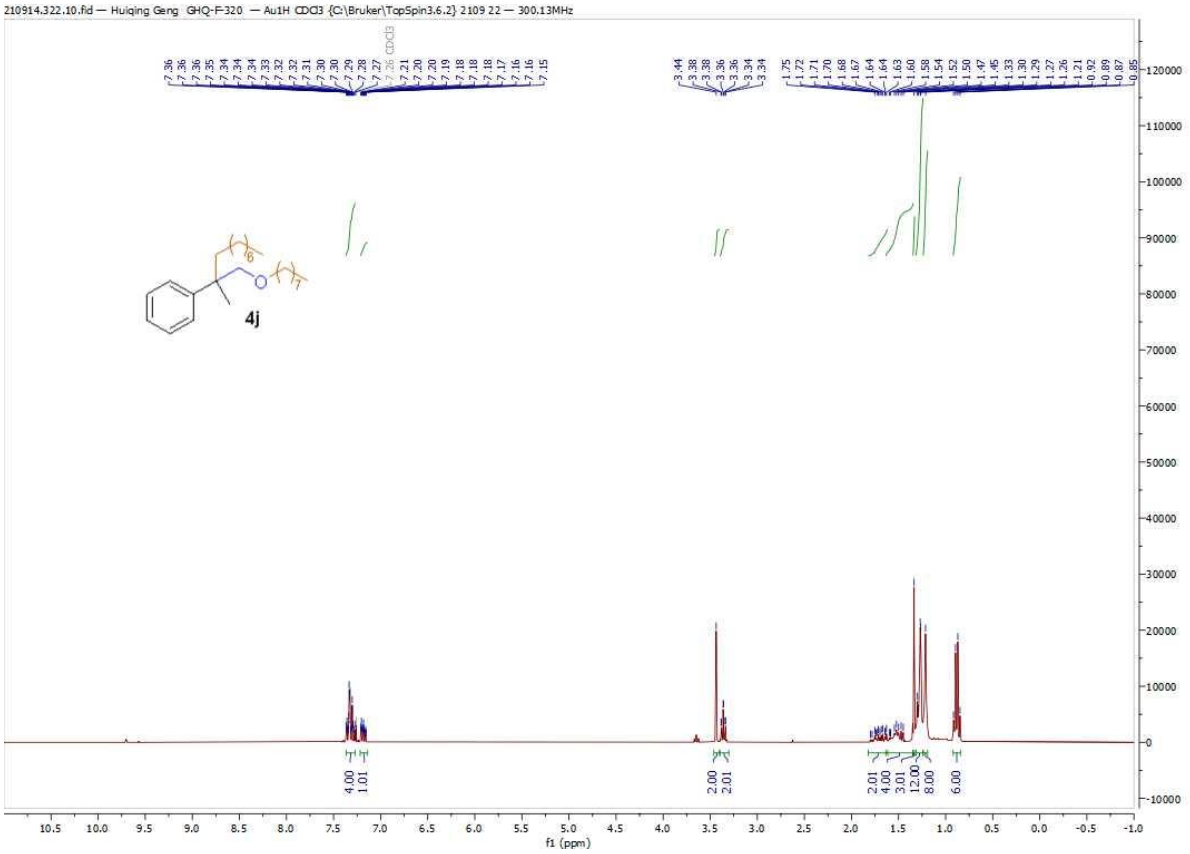

210831.317.11.fid — Huiqing Geng GHQ-E-326 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 17 — 75.48MHz
nBu
nBu
4g
f1 (ppm)

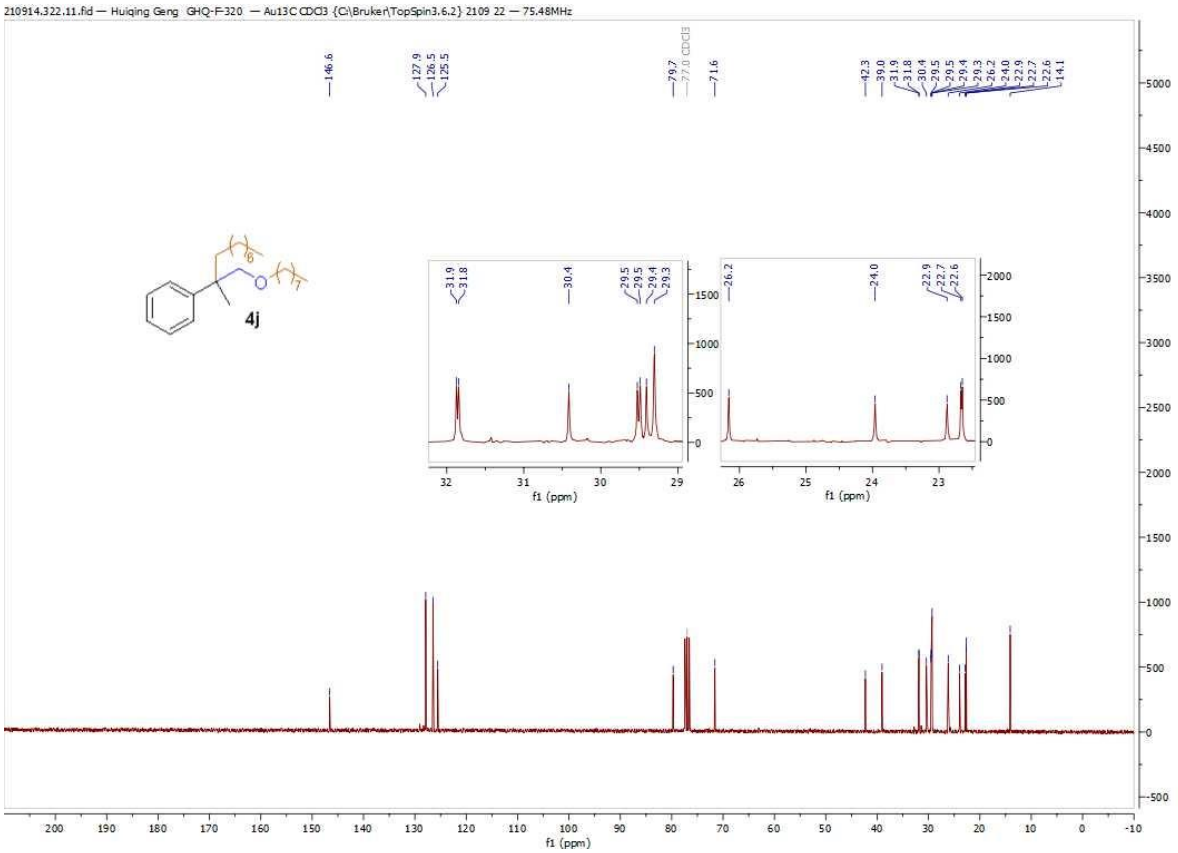

210825.327.10.fid — Huiqing Geng GHQ-E-305 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 27 — 300.13MHz
nBu
nBu
O
F
4h
2.01
2.00
4.04
4.00
7.00
2.01
6.00
f1 (ppm)

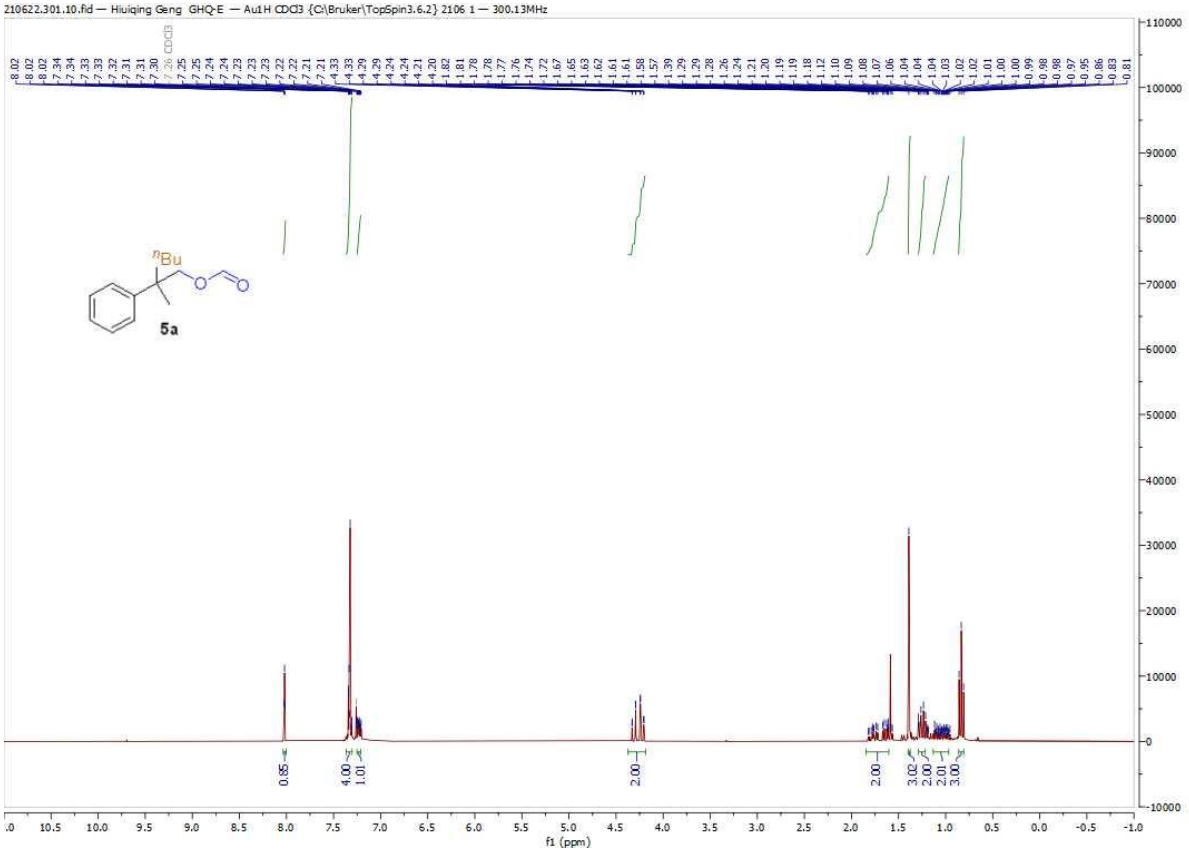

210825.327.11.fid — Huiqing Geng GHQ-E-305 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 27 — 75.48MHz
162.6
159.3
142.2
142.2
128.1
128.0
114.6
114.4
79.8
71.3
41.9
39.0
31.6
26.1
23.4
22.9
19.3
14.0
13.9
nBu
nBu
O
F
4h
f1 (ppm)

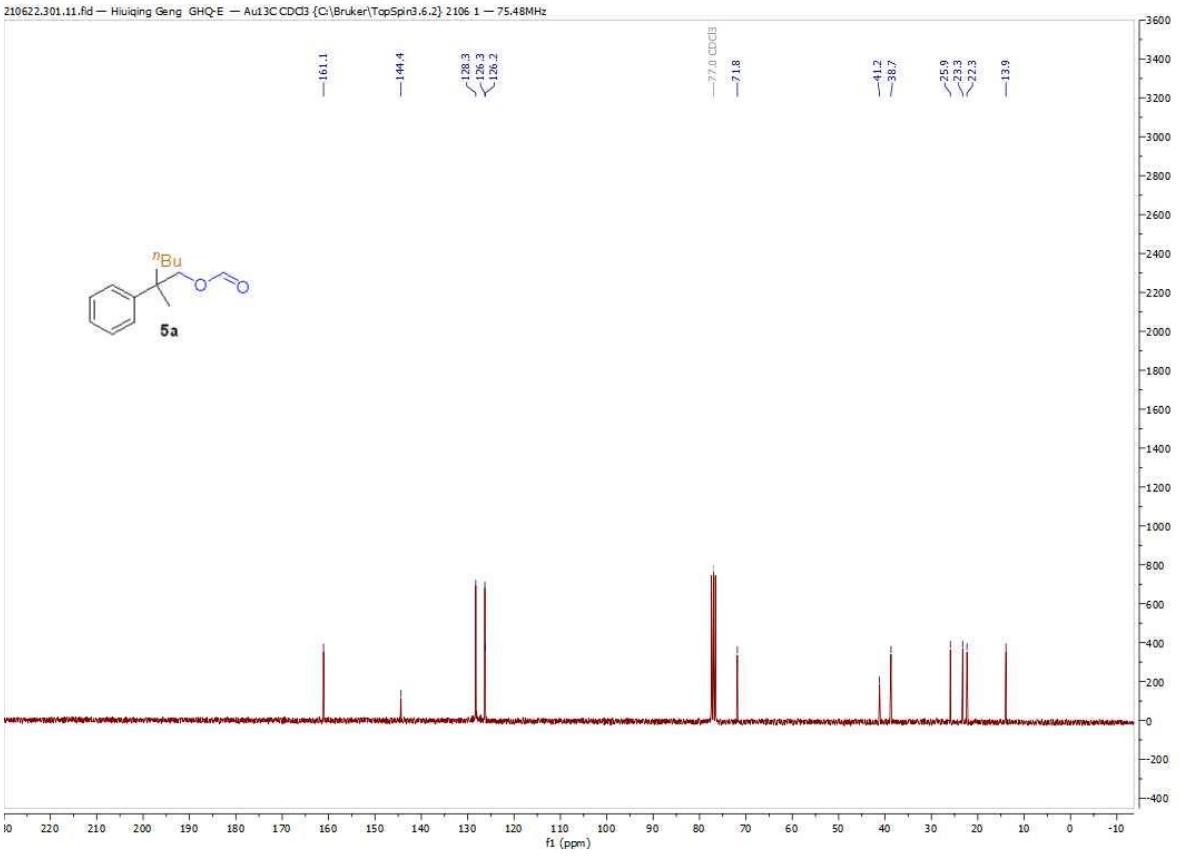

210909.f308.10.fid — Huiqing Geng GHQ-E-369 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 8 — 300.20MHz
4i
f1 (ppm)

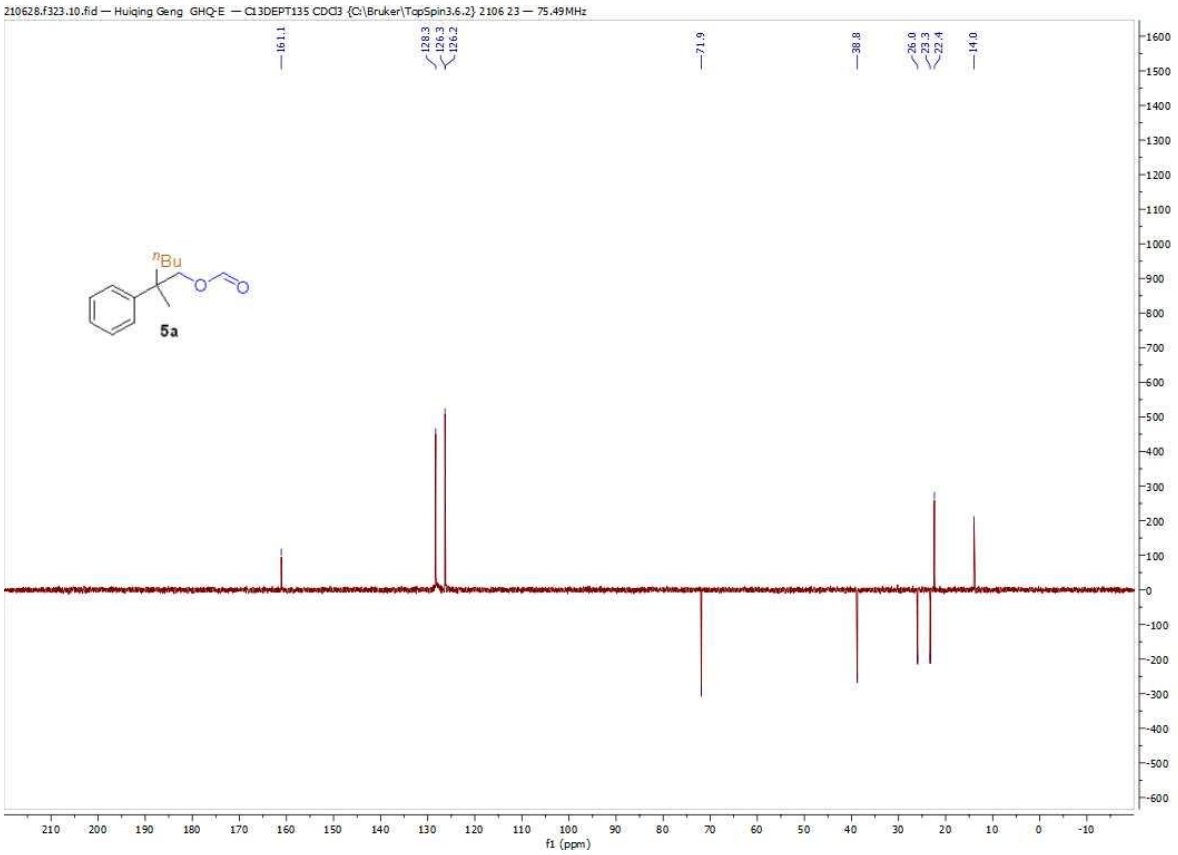

210831.318.11.fid — Huiqing Geng GHQ-E-328 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2108 18 — 75.48MHz
4i
f1 (ppm)

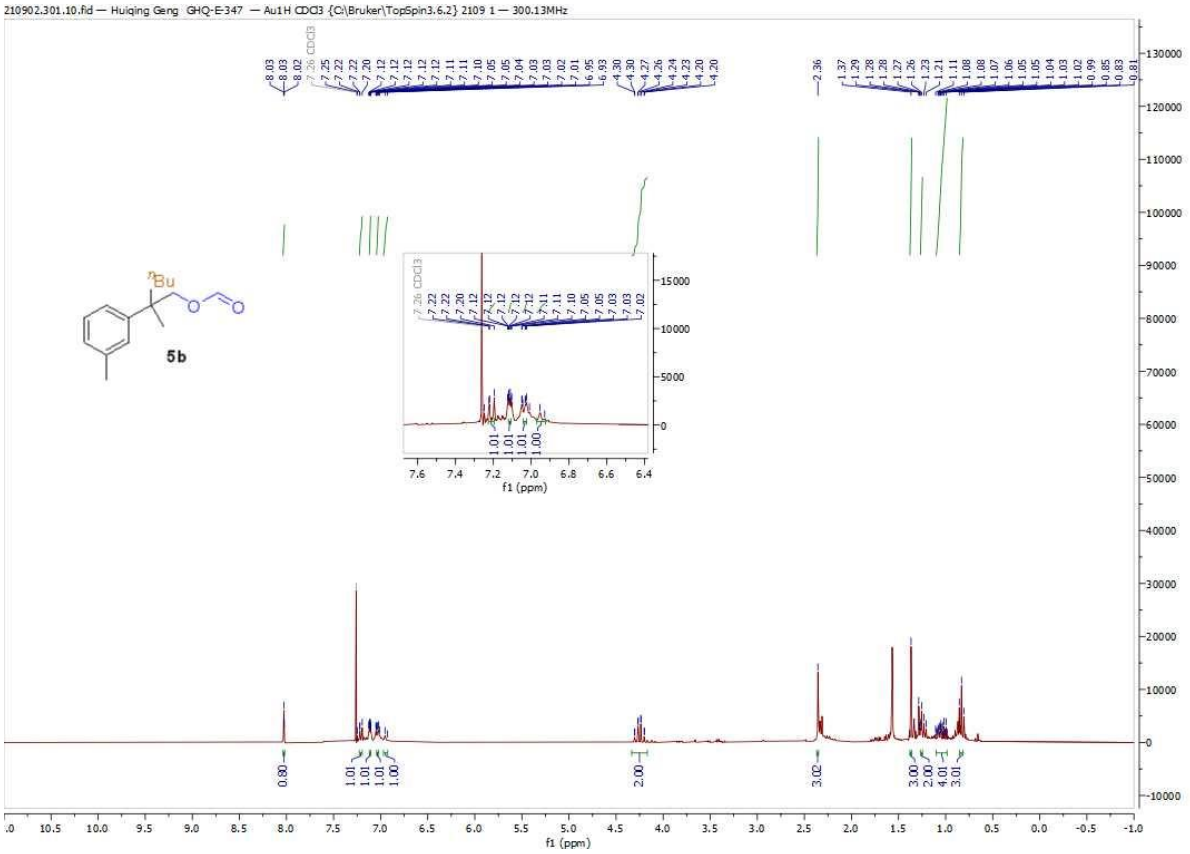

210914.322.10.fid — Huiqing Geng GHQ-F320 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 22 — 300.13MHz
4j
f1 (ppm)

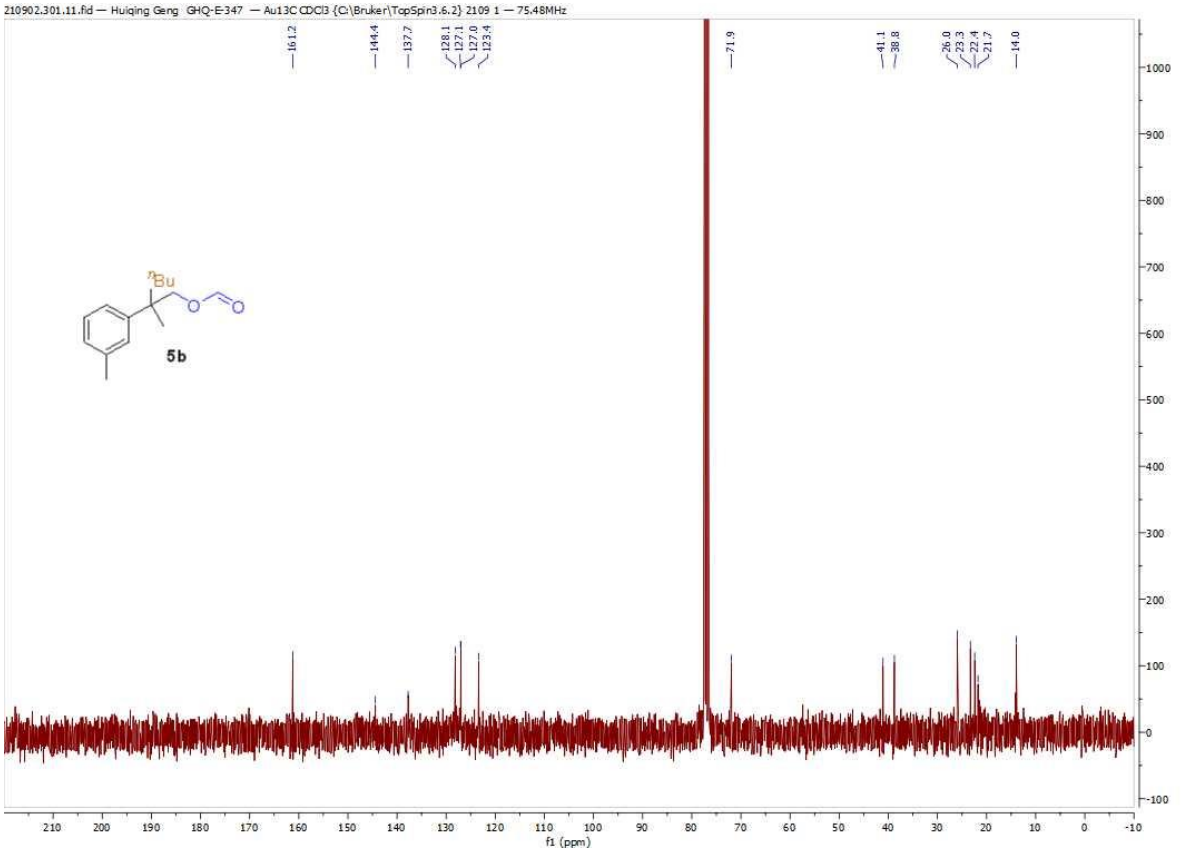

210914.322.11.fid — Huiqing Geng GHQ-F320 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 22 — 75.48MHz
4j
f1 (ppm)

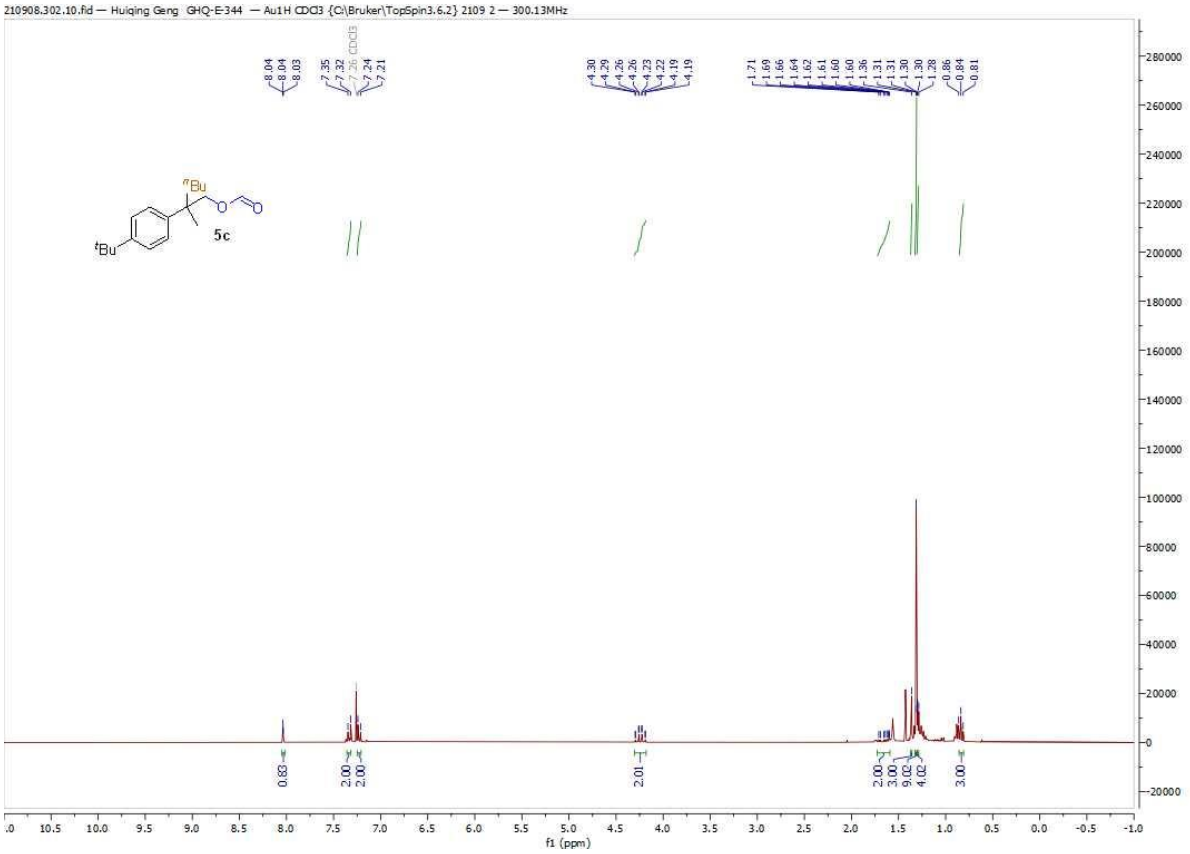

210622.301.10.fid — Hiuiqing Geng GHQ-E — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 1 — 300.13MHz
nBu
5a
f1 (ppm)

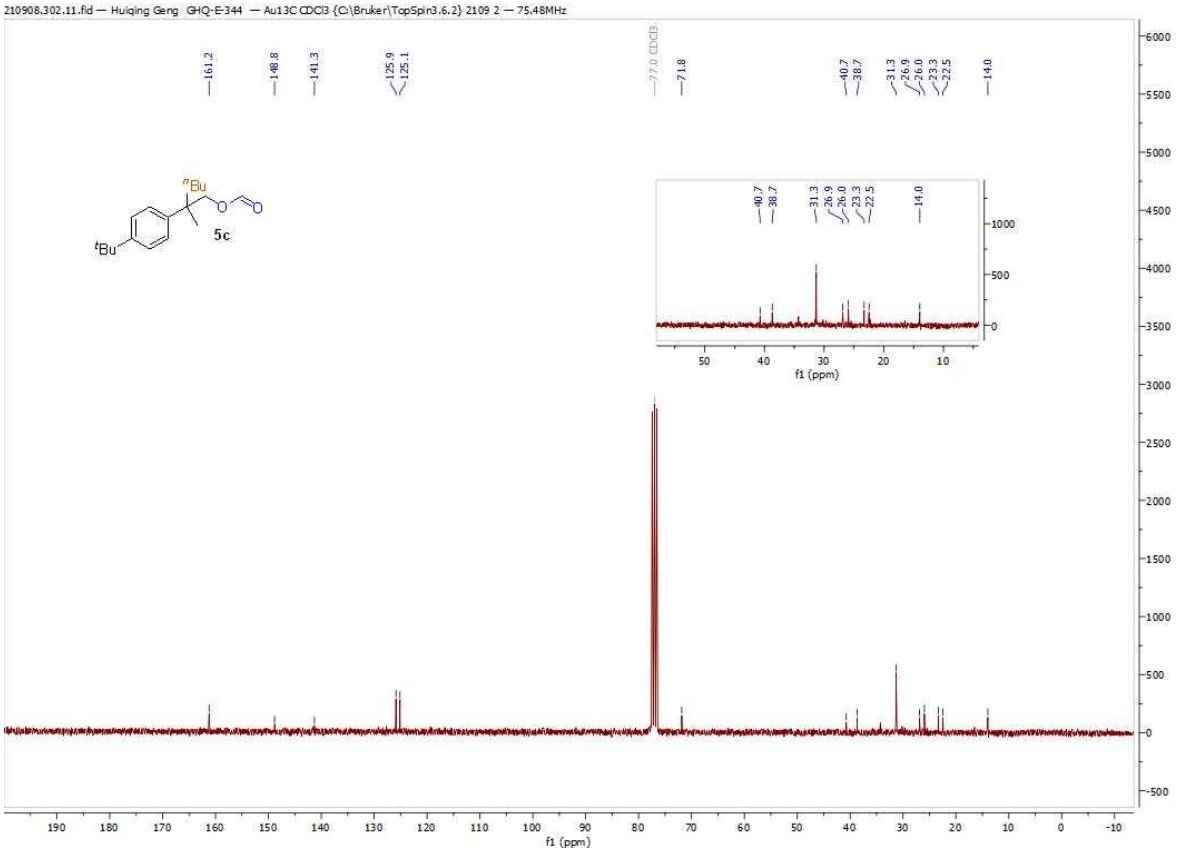

210622.301.11.fid — Hiuiqing Geng GHQ-E — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 1 — 75.48MHz
161.1
144.4
128.3
126.3
126.2
77.0 CDCl3
71.8
41.2
38.7
25.9
23.3
22.3
13.9
nBu
5a
f1 (ppm)

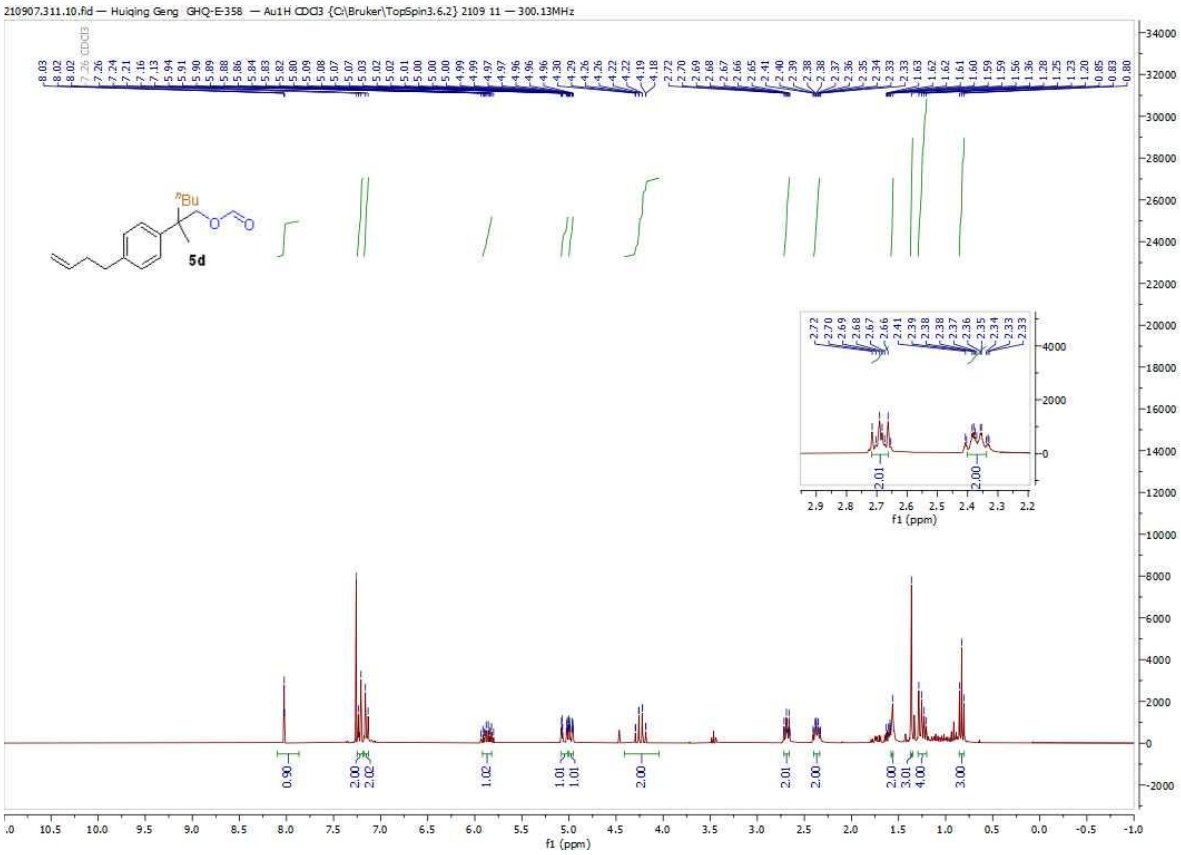

210628.f323.10.fid — Huiqing Geng GHQ-E — C13DEPT135 CDCl3 {C:\Bruker\TopSpin3.6.2} 2106 23 — 75.49MHz
161.1
128.3
126.3
126.2
71.9
38.8
26.0
23.3
22.4
14.0
nBu
5a
f1 (ppm)

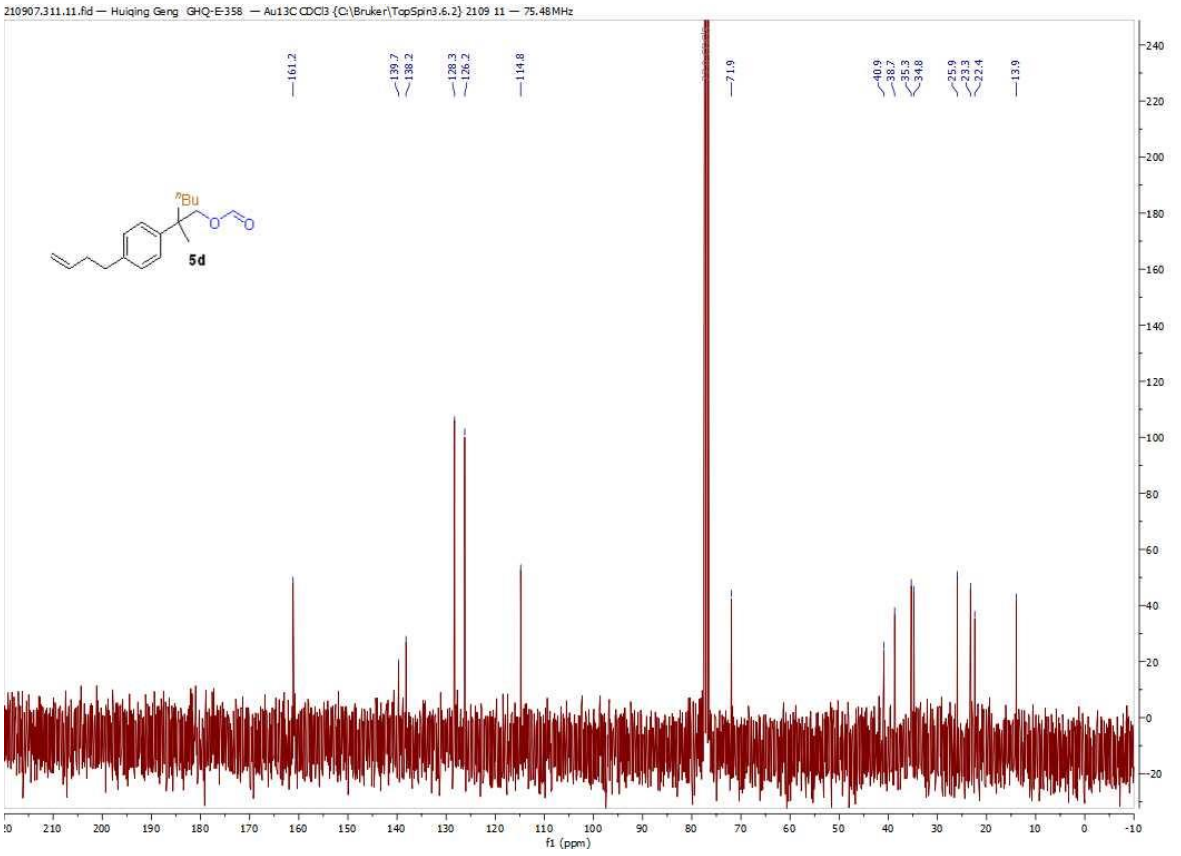

210902.301.10.fid — Huiqing Geng GHQ-E-347 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 1 — 300.13MHz
5b
f1 (ppm)


210902.301.11.fid — Huiqing Geng GHQ-E-347 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 1 — 75.48MHz
5b
f1 (ppm)


210908.302.10.fid — Huiqing Geng GHQ-E-344 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 2 — 300.13MHz
8.04
8.04
8.03
7.35
7.32
7.26 CDCl3
7.24
7.21
4.30
4.29
4.26
4.26
4.23
4.22
4.19
4.19
1.71
1.69
1.66
1.64
1.62
1.61
1.60
1.60
1.36
1.31
1.31
1.30
1.30
1.28
0.86
0.84
0.81
5c
0.83
2.00
2.00
2.01
2.00
3.00
9.02
4.02
3.00
f1 (ppm)


210908.302.11.fid — Huiqing Geng GHQ-E-344 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 2 — 75.48MHz
161.2
148.8
141.3
125.9
125.1
77.0 CDCl3
71.8
40.7
38.7
31.3
26.9
26.0
23.3
22.5
14.0
5c
f1 (ppm)


210907.311.10.fid — Huiqing Geng GHQ-E-358 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 11 — 300.13MHz
nBu
O
O
5d
f1 (ppm)


210907.311.11.fid — Huiqing Geng GHQ-E-358 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2109 11 — 75.48MHz
161.2
139.7
138.2
128.3
126.2
114.8
71.9
40.9
38.7
35.3
34.8
25.9
23.3
22.4
13.9
nBu
O
O
5d
f1 (ppm)