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SUPPLEMENTARY INFORMATION

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Catalytic enantioselective desymmetrizing functionalization of alkyl radicals via Cu(I)/CPA cooperative catalysis

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Supplementary Information

Catalytic enantioselective desymmetrising functionalization of alkyl radicals via Cu(I)/CPA cooperative catalysis

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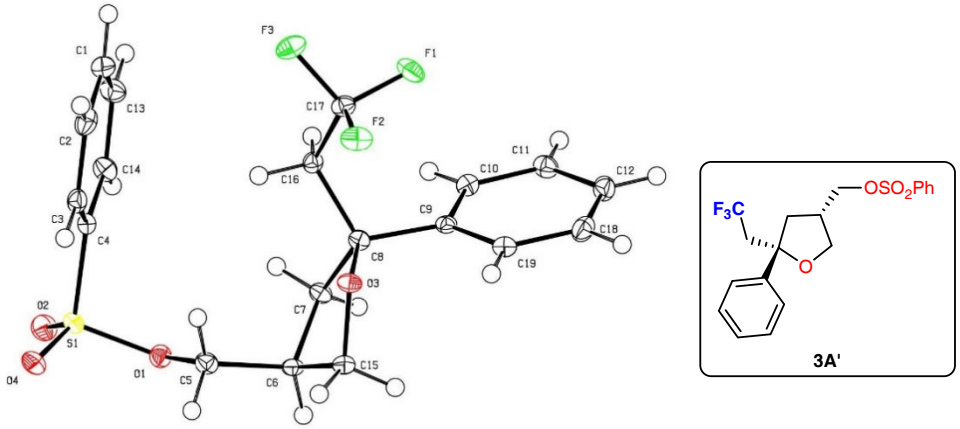

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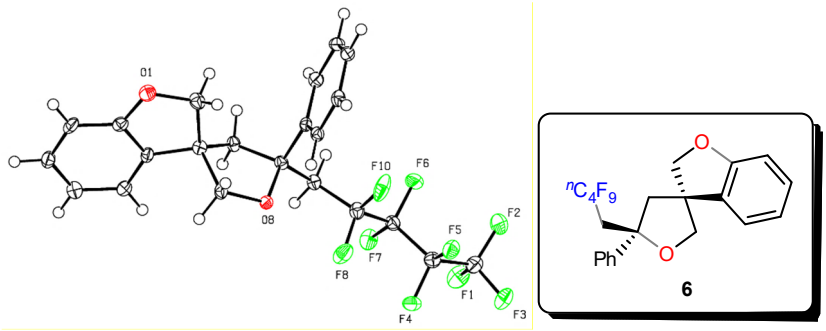

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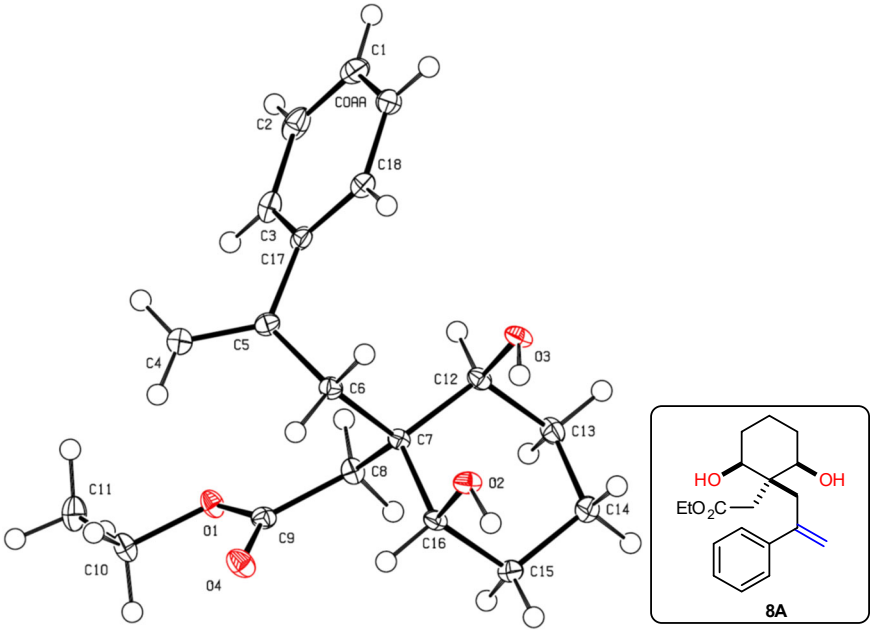

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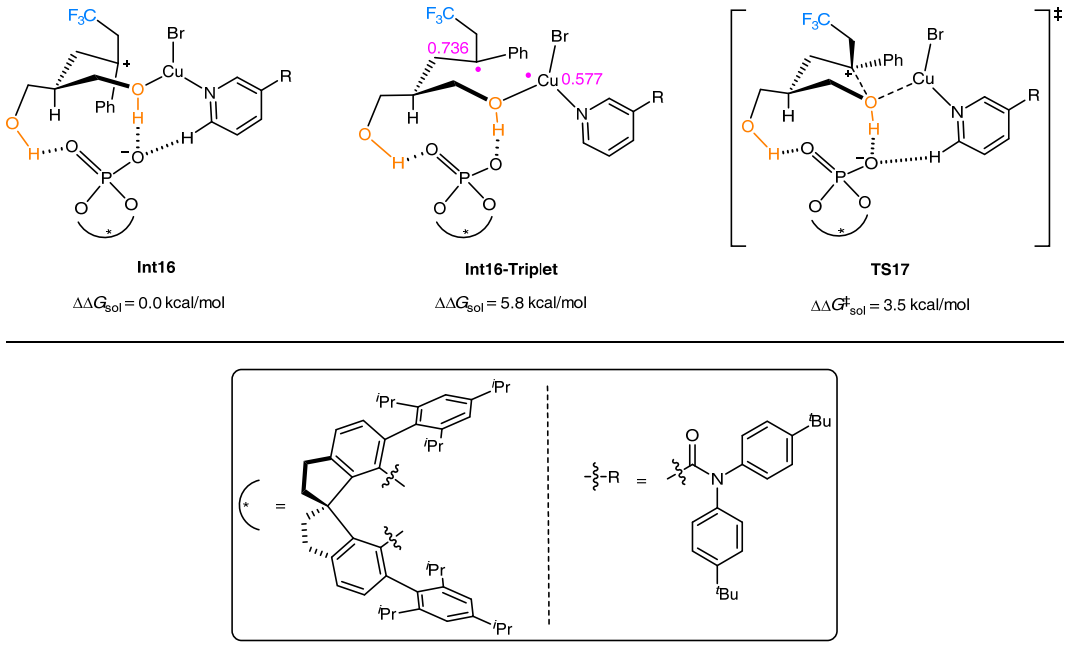

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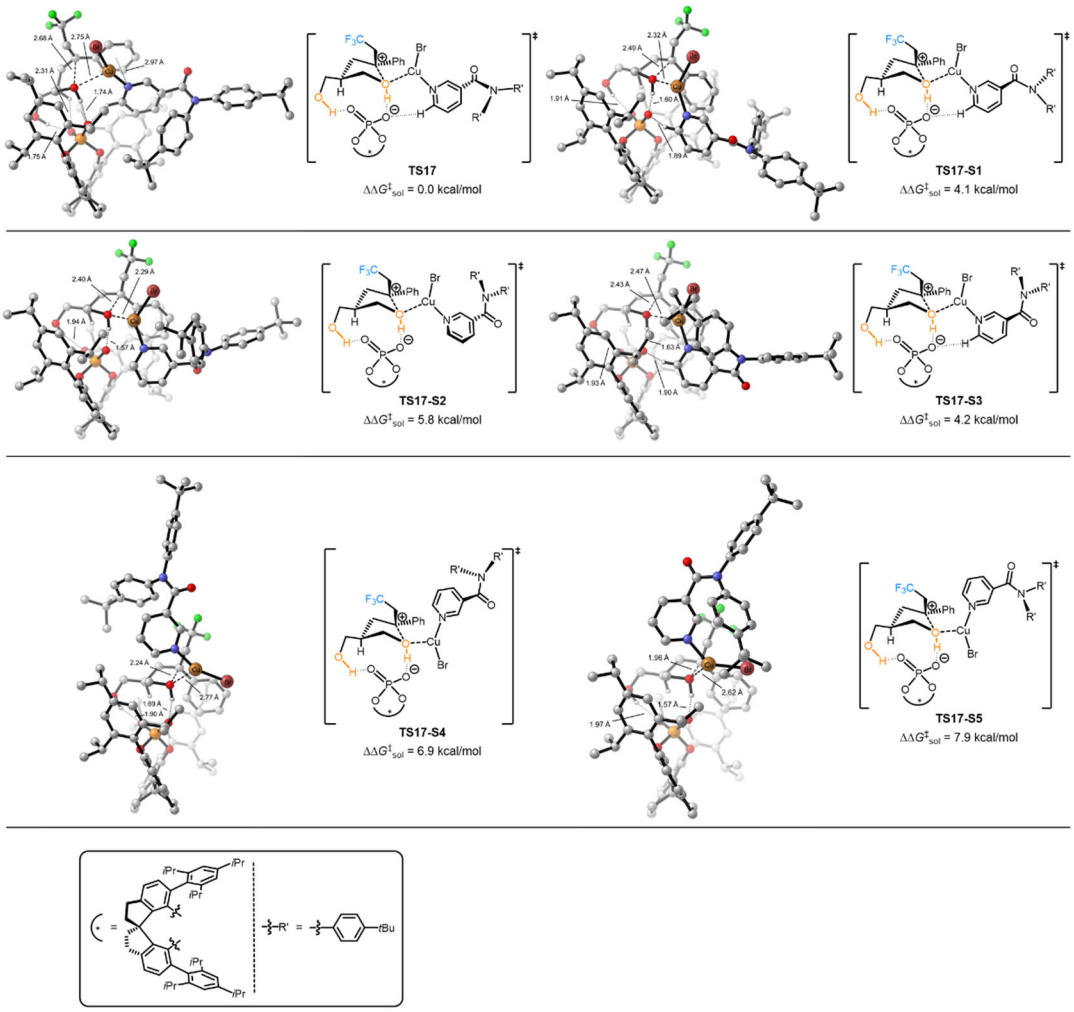

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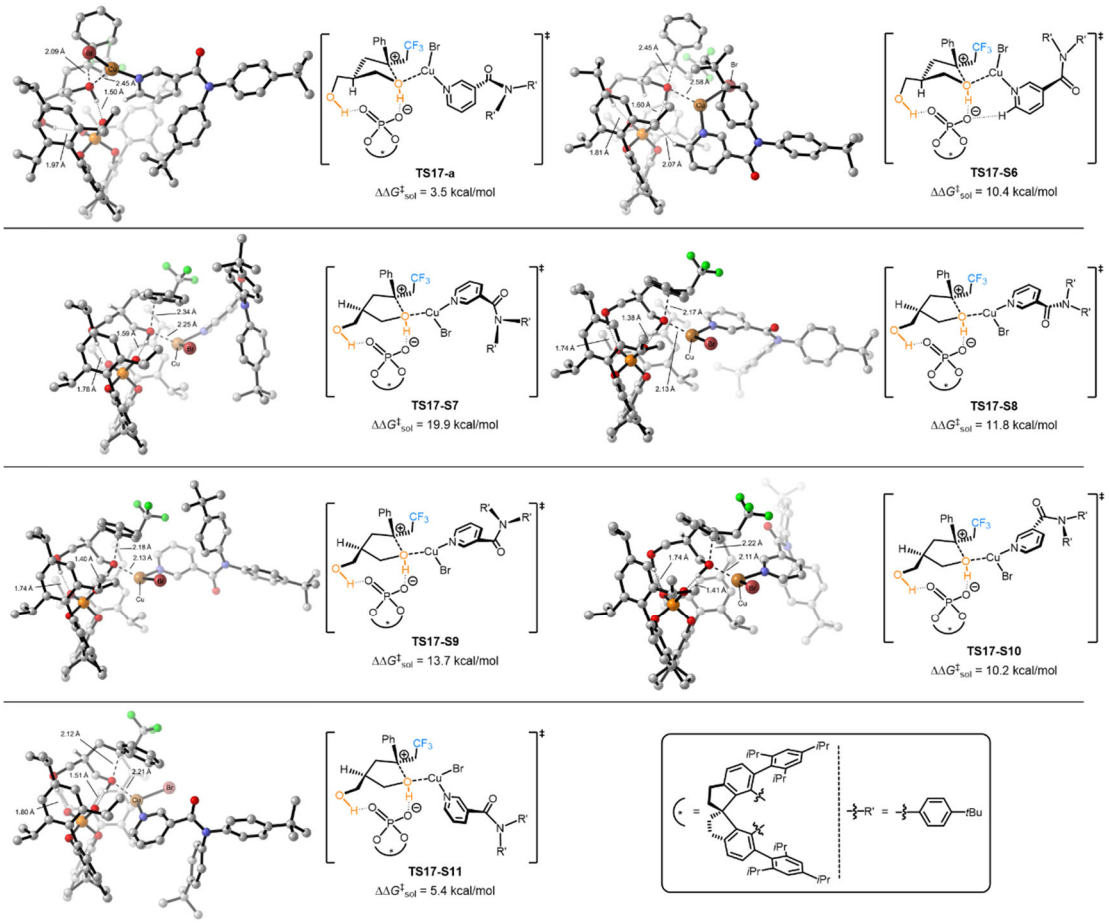

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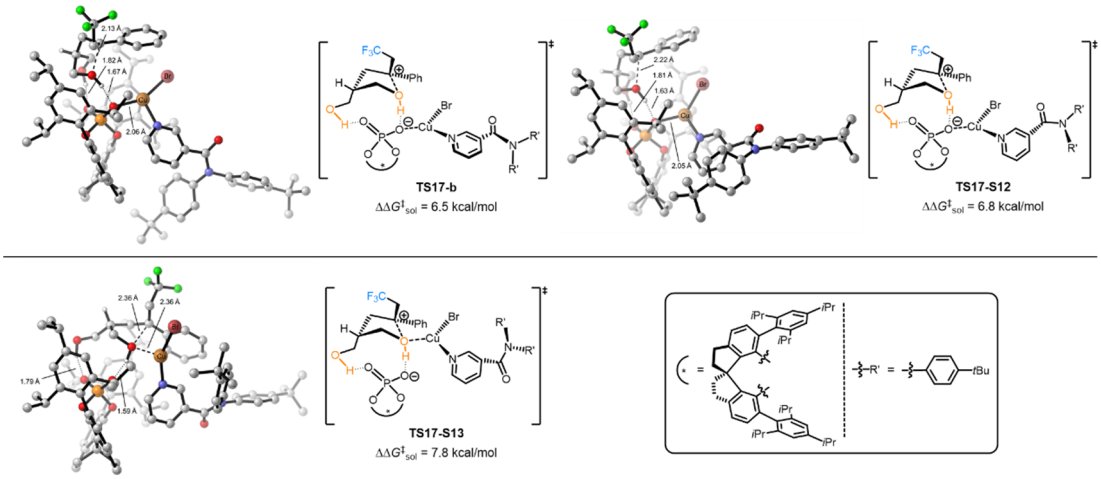

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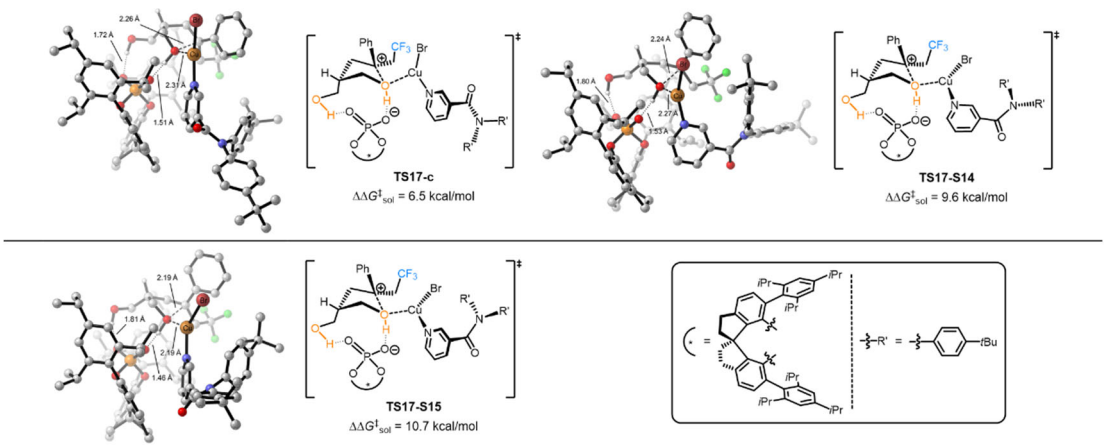

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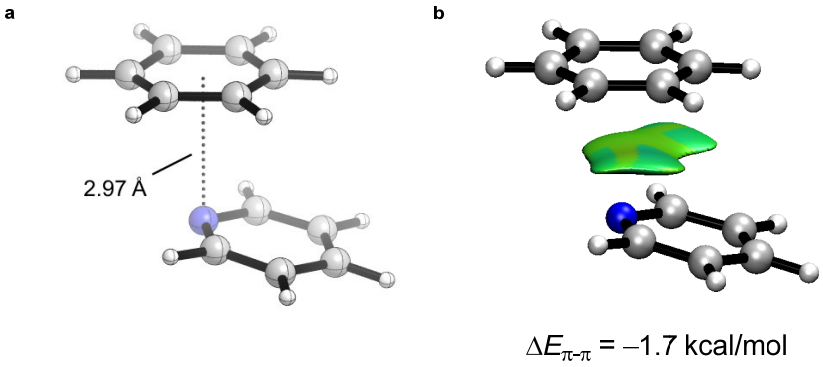

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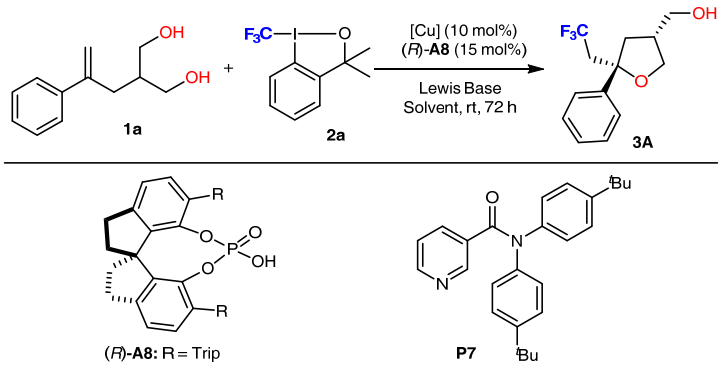
2. Supplementary Tables

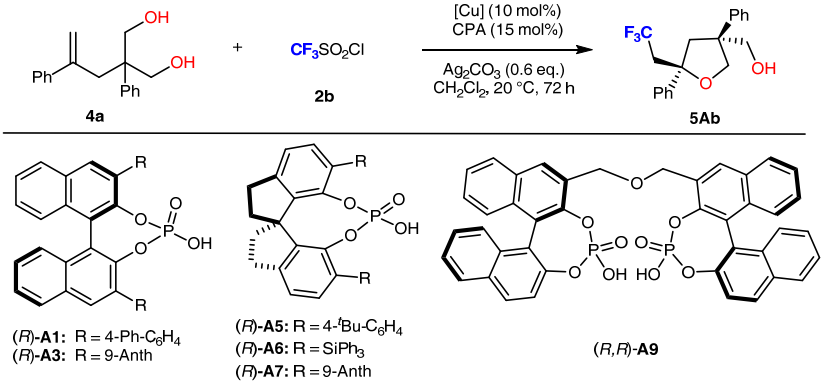
Supplementary Table 1. Optimization of reaction conditions for other parameters[a]



Entry	[Cu]	Solvent	LB	Yield (%)	Dr	Ee (%)
1	$CuBH_4(PPh_3)_2$	AcO^{i}Pr	-	25	4:1	39/5
2[b]	$CuBH_4(PPh_3)_2$	AcO^{i}Pr	-	85	3:1	38/5
3[b]	$Cu(CH_3CN)_4BF_4$	AcO^{i}Pr	-	45	2:1	38/-7
4[b]	Cu_2O	AcO^{i}Pr	-	90	4:1	43/15
5[b]	CuI	AcO^{i}Pr	-	81	2:1	22/10
6[b]	CuCl	AcO^{i}Pr	-	93	2:1	36/7
7[b]	CuTc	AcO^{i}Pr	-	62	2:1	39/7
8	Cu_2O	AcO^{i}Pr	**P7** (40 mol%)	86	>20:1	91/-
9	Cu_2O	PhMe	**P7** (40 mol%)	55	>20:1	69/-
10	Cu_2O	DCM	**P7** (40 mol%)	77	>20:1	70/-
11	Cu_2O	THF	**P7** (40 mol%)	75	1:4	30/5
12	Cu_2O	MeCN	**P7** (40 mol%)	70	1:4	47/16

[a]Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), Cu^I (10 mol%), (*R*)-**A8** (15 mol%) and solvent (0.5 mL) at rt for 3 d; Yield and dr values were based on ^{1}H NMR analysis of the crude product using CH_2Br_2 as an internal standard; Ee value was based on HPLC analysis. [b]50 °C.

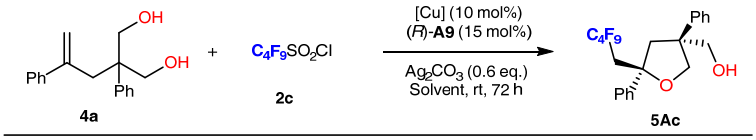
Supplementary Table 2. Optimization of reaction conditions to access two congested quaternary stereocenters with CF_3 radical[a]

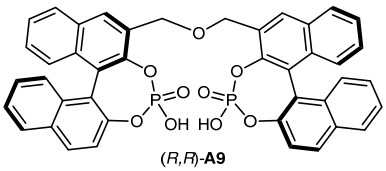


Entry	[Cu]	CPA	Solvent	Yield (%)	Dr	Ee (%)
1	CuBr	(*R*)-**A1**	CH_2Cl_2	89	4:1	20
2	CuBr	(*R*)-**A3**	CH_2Cl_2	78	3:1	32
3	CuBr	(*R*)-**A5**	CH_2Cl_2	71	3:1	22
4	CuBr	(*R*)-**A6**	CH_2Cl_2	83	3:1	30
5	CuBr	(*R*)-**A7**	CH_2Cl_2	80	3:1	23
6	CuI	(*R,R*)-**A9**	CH_2Cl_2	84	10:1	74
7	CuCl	(*R,R*)-**A9**	CH_2Cl_2	86	10:1	83
8	CuBr	(*R,R*)-**A9**	CH_2Cl_2	83	9:1	80
9	CuOAc	(*R,R*)-**A9**	CH_2Cl_2	68	8:1	58
10	Cu_2O	(*R,R*)-**A9**	CH_2Cl_2	74	6:1	53
11	CuBr·SMe_2	(*R,R*)-**A9**	CH_2Cl_2	84	11:1	78

[a]Reaction conditions: **4a** (0.025 mmol), **2b** (0.03 mmol), Cu^I (10 mol%), Ag_2CO_3 (0.015 mmol), CPA (15 mol%) and solvent (0.3 mL) at 20 °C for 3 d under argon; Yield was based on ^{1}H NMR analysis of the crude product using CH_2Br_2 as an internal standard; Dr and ee values were determined by ^{1}H NMR and HPLC analysis, respectively.

Supplementary Table 3. Optimization of reaction conditions to access two congested quaternary stereocenters with C_4F_9 radical[a]



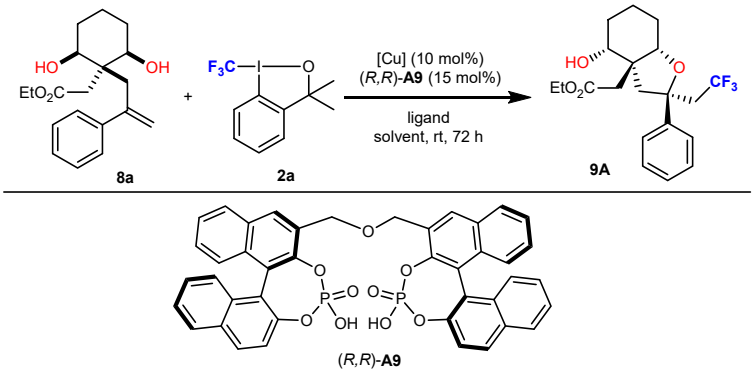


(*R*,*R*)-**A9**

Entry	[Cu]	Solvent	Yield (%)	Dr	Ee (%)
1	CuI	^{i}PrCO$_2$Et	96	15:1	79
2	CuCl	^{i}PrCO$_2$Et	95	11:1	71
3	CuBr	^{i}PrCO$_2$Et	97	13:1	82
4	CuOAc	^{i}PrCO$_2$Et	93	6:1	64
5	CuBr	EtOAc	95	12:1	85
6	CuBr	CH_3CN	57	3:1	8
7	CuBr	CH_2Cl_2	96	>20:1	93
8	CuBr	$CHCl_3$	96	18:1	85
9	CuBr	CCl_4	94	16:1	76
10	CuBr	THF	62	4:1	31

[a]Reaction conditions: **4a** (0.025 mmol), **2c** (0.03 mmol), Cu^{I} (10 mol%), (*R,R*)-**A9** (15 mol%), Ag_2CO_3 (0.015 mmol), and solvent (0.3 mL) at rt for 3 d under argon; Yield was based on ^{1}H NMR analysis of the crude product using CH_2Br_2 as an internal standard; Dr and ee values were determined by ^{1}H NMR and HPLC analysis, respectively.

Supplementary Table 4. Optimization of reaction conditions of *meso* olefinic 1,3-diol[a]



entry	[Cu]	solvent	ligand	yield (%)	dr	ee (%)
1	$CuBH_4(PPh_3)_2$	AcO*i*Pr	-	82	1:3	7/72
2	$CuBH_4(PPh_3)_2$	$PhCF_3$	-	45	1:2	2/55
3	$CuBH_4(PPh_3)_2$	DCE	-	40	2:1	12/20
4	$CuBH_4(PPh_3)_2$	$CHCl_3$	-	81	1:2	2/67
5	$CuBH_4(PPh_3)_2$	CCl_4	-	91	1:5	13/90
6	$CuBH_4(PPh_3)_2$	MTBE	-	70	1:3	0/80
7	CuCl	CCl_4	-	56	5:1	11/5
8	CuI	CCl_4	-	50	5:1	0/10
9	Cu_2O	CCl_4	-	58	2:1	10/6
10	CuTc	CCl_4	-	70	3:1	27/26
11	CuTc	CCl_4	PPh_3 (20 mol%)	82	1:6	4/91

[a]Reaction conditions: **8a** (0.05 mmol), **2a** (0.075 mmol), Cu^I (10 mol%), (*R,R*)-**A9** (15 mol%) and solvent (0.5 mL) at r.t for 3 d; Yield based on ^1H NMR analysis of the crude product using CH_2Br_2 as an internal standard; dr and ee values were determined by ^1H NMR and HPLC analysis, respectively.

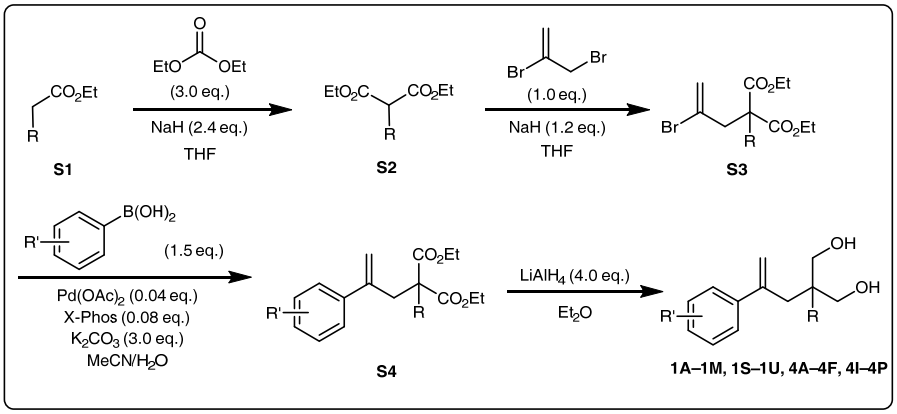
3. Supplementary Methods

3.1 General Information

All reactions were carried out under argon atmosphere using Schlenk techniques. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. Chiral phosphoric acid (**CPA**) was purchased from Daicel Chiral Technologies (China). Extra dry solvents were purchased from Acros® and J&K®. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 GF254 plates. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040–0.063 mm). ^{1}H, ^{13}C, ^{19}F and 2D-NMR spectra were recorded on Bruker Avance-400 or 500 spectrometers. Mass spectrometric data were obtained using Bruker Apex IV RTMS. Circular dichroism (CD) spectra were recorded on an Applied PhotoPhysics Chirascan CD spectropolarimeter, using a 1 mm quartz cuvette. Enantiomeric excess (ee) was determined using Agilent high-performance liquid chromatography (HPLC) with a Hatachi detector, column conditions are reported in the experimental section below. X-ray diffraction was measured on a 'Bruker APEX-II CCD' diffractometer with Cu–Kα radiation.

3.2 General Procedure for the Synthesis of Substrates

Synthesis of substrates **1A**–**1M, 1S**–**1U, 4A**–**4F, 4I**–**4P** (According to literature procedures with minor revision[1]):



Synthesis of substrate **S2**: To a suspension of NaH (60% dispensed in mineral oil, 2.4 eq.) in dry THF at 0 °C was slowly added the corresponding ester **S1** (1.0 eq.). After stirring for 30 min, ethyl carbonate (3.0 eq.) was added dropwise. The mixture was warmed to rt and then heated to reflux under inert atmosphere for 24 h. After cooled to rt, saturated NH_4Cl (aq.) was carefully added to the above reaction solution to quench the reaction and the mixture was extracted with EtOAc (3x). The combined organic layers were dried over Na_2SO_4, filtered and then concentrated. The crude product **S2** was used directly in the next step without further purification.

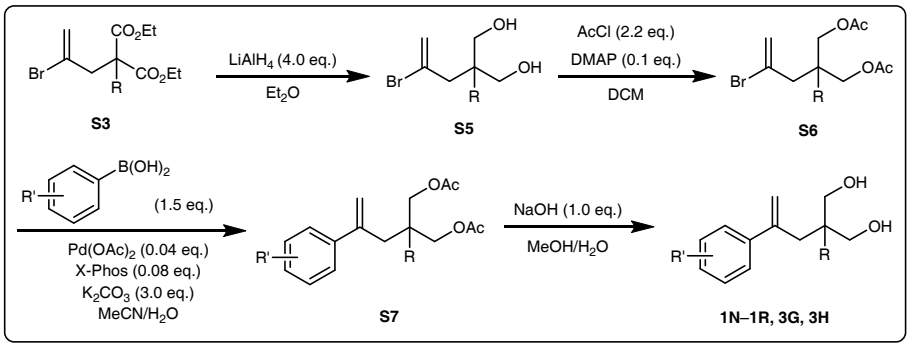
Synthesis of substrate **S3**: To a suspension of NaH (60% dispensed in mineral oil, 1.2 eq.) in dry THF (20 mL) at 0 °C was slowly added **S2** (1.0 eq.). After stirring for 30 min, 2,3-dibromopropene (1.0 eq.) was added dropwise. The reaction mixture was warmed to rt and stirred overnight. Then the reaction was quenched with saturated NH_4Cl (aq.) and extracted with CH_2Cl_2 (3x). The combined organic layers were dried (Na_2SO_4) and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (eluent: petroleum ether/ EtOAc ~ 20:1) to give **S3**.

Synthesis of substrates **S4**: To a solution of **S3** (1.0 eq.), arylboronic acids (1.5 eq.), K_2CO_3 (3.0 eq.) and 2-dicyclohexylphosphino-2′,4′,6′-triisopropylbiphenyl (X-Phos, 0.08 eq.) in CH_3CN/H_2O (v/v = 3/1) was added $Pd(OAc)_2$ (0.04 eq.). The flask was evacuated and then backfilled with argon three times. Then the reaction mixture was stirred at 80 °C for 12 h under argon atmosphere. After cooling to rt, the mixture was extracted with CH_2Cl_2 (3x). The combined organic layer was washed with brine, dried (Na_2SO_4) and concentrated *in vacuo.* The residue was purified by flash chromatography to give **S4**.

Synthesis of substrates **1A**–**1M, 1S-1U, 4A-4F, 4I-4P**: To a suspension of $LiAlH_4$ (4.0 eq.) in Et_2O at 0 °C was slowly added a solution of **S4** (1.0 eq.) in Et_2O. Then the reaction mixture was warmed to rt and stirred for 2 h. Next, it was quenched by slow, portionwise addition of wet Na_2SO_4 (4.0 mL water in 32.0 g Na_2SO_4) at 0 °C. Upon

completion, the mixture was warmed to rt, stirred for additional 30 min, filtered, and concentrated. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc ~ 2/1) to give **1A**–**1M, 1S-1U, 4A-4F, 4I-4P**.

Synthesis of substrates **1N**–**1R, 3G, 3H** (According to literature procedures with minor revision[1]):



Synthesis of substrate **S5**: To a suspension of $LiAlH_4$ (4.0 eq.) in Et_2O at 0 °C was slowly added a solution of **S3** (1.0 eq.) in Et_2O. Then the reaction mixture was warmed to rt and stirred for 2 h. Next, it was quenched by slow, portionwise addition of wet Na_2SO_4 (4.0 mL water in 32.0 g Na_2SO_4) at 0 °C. Upon completion, the mixture was warmed to rt, stirred for additional 30 min, filtered, and concentrated. The crude product **S5** was used directly in the next step.

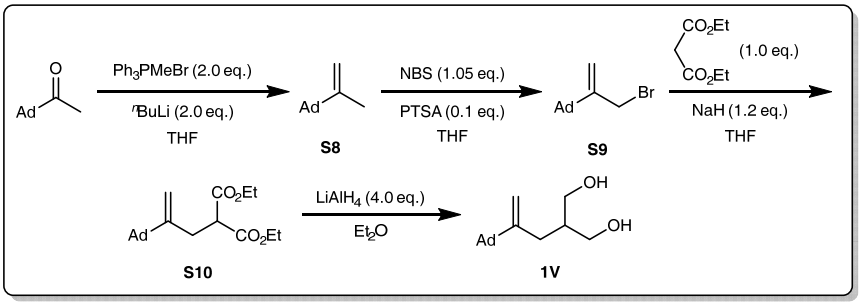
Synthesis of substrate **S6**: To a solution of **S5** (1.0 eq.) and 4-(dimethylamino)pyridine (DMAP, 0.1 eq.) in CH_2Cl_2 at 0 °C was slowly added acetyl chloride (2.2 eq.). The reaction mixture was stirred at rt for additional 4 h. Upon completion, water was added and the aqueous layer was extracted with CH_2Cl_2 (3x). The combined organic layer was washed with brine, dried (Na_2SO_4) and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 5/1) to give **S6** as an oil.

Synthesis of substrates **S7**: To a solution of **S6** (1.0 eq.), arylboronic acids (1.5 eq.), K_2CO_3 (3.0 eq.) and 2-dicyclohexylphosphino-2′,4′,6′-triisopropylbiphenyl (X-Phos, 0.08 eq.) in CH_3CN/H_2O (v/v = 3/1) was added $Pd(OAc)_2$ (0.04 eq.). The flask was evacuated and then backfilled with argon three times. Then the reaction mixture was stirred at 80 °C for 12 h under argon atmosphere. After cooled to rt, the mixture was extracted with CH_2Cl_2 (3x). The combined organic layer was washed with brine, dried (Na_2SO_4) and concentrated *in vacuo.* The residue was purified by flash chromatography to give **S7**.

Synthesis of substrates **1N-1R, 3G, 3H**: To a solution of **S7** (1.0 eq.) in $MeOH/H_2O$ (v/v = 1/1) was added NaOH (2.2 eq.). The reaction mixture was stirred at rt. Upon completion, water was added and the aqueous layer was extracted with ethyl acetate (3x). The combined organic layer was washed with brine, dried (Na_2SO_4) and concentrated *in vacuo.* The residue was purified by silica gel column chromatography

to give **1N-1R, 3G, 3H**.

Synthesis of substrate **1V**



Synthesis of substrate **S8**: To a stirred solution of Ph_3PMeBr (10.0 g, 28.0 mmol) in THF (40 mL) at 0 °C was added *n*-BuLi (10.5 mL in *n*-Hexane, 2.4 M, 25.2 mmol) dropwise over 30 min. The reaction mixture was stirred for 2 hours at rt. 1-(Adamantan-1-yl)ethanone (2.5 g, 14 mmol) in THF (5 mL) was added dropwise to the cooled reaction mixture at 0 °C. The reaction was then stirred at rt for 72 h, quenched with saturated NH_4Cl, and extracted with EtOAc. The combined organic extracts were washed with brine, dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography to yield **S8** as a colorless oil (2.34 g, 13.3 mmol, 95%).

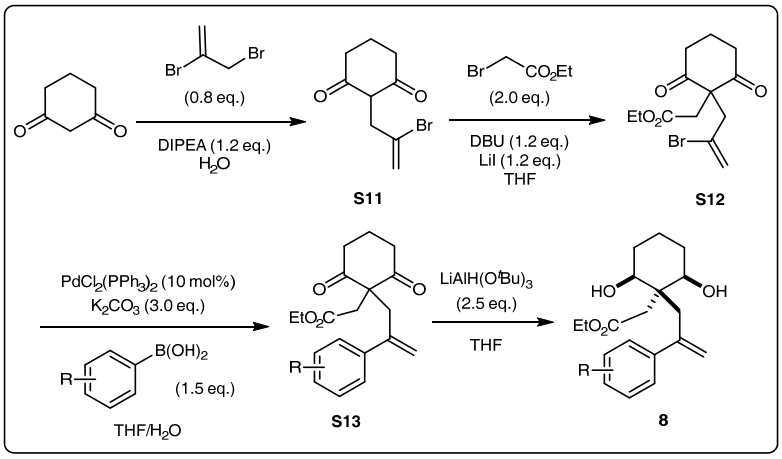
Synthesis of substrate **S9**: To a solution of **S8** (2.34 g, 13.3 mmol) in dry THF (40 mL) in an oven-dried flask were added *N*-bromosuccinimide (2.5 g, 14 mmol) and *p*-toluenesulfonic acid (PTSA) (0.23 g, 1.3 mmol). The resulting mixture was refluxed at 100 °C for 4 h. The mixture was then cooled to rt, diluted with petroleum ether (15 mL/mmol), and washed by H_2O (15 mL × 3). The organic phase was dried over Na_2SO_4 and concentrated under reduced pressure to give a yellow oil. Purification by column chromatography over silica gel using petroleum ether as eluent afforded **S9** as a yellow oil which was used directly in the next step without further purification.

Synthesis of substrate **S10**: To a suspension of NaH (60% dispensed in mineral oil, 0.38 g, 16 mmol) in dry THF (60 mL) at 0 °C was slowly added diethyl malonate (2.0 mL, 13 mmol). After stirring for 30 min, **S9** was added dropwise. The reaction mixture was warmed to rt and stirred overnight. Then the reaction was quenched with saturated NH_4Cl (aq.) and extracted with CH_2Cl_2 (3x). The combined organic layers were dried (Na_2SO_4) and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc $\approx$ 20/1) to give **S10** (3.6 g, 11 mmol, 81% yield in two steps).

Synthesis of substrate **1V**: To a suspension of $LiAlH_4$ (4.0 eq.) in Et_2O at 0 °C was slowly added a solution of **S10** (3.6 g, 11 mmol) in Et_2O. Then the reaction mixture was warmed to rt and stirred for 2 h. Next, it was quenched by slow, portionwise addition of wet Na_2SO_4 (4.0 mL water in 32.0 g Na_2SO_4) at 0 °C. Upon completion, the mixture was warmed to rt, stirred for additional 30 min, filtered, and concentrated.

The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc ≈ 2/1) to give **1V** as a white solid (2.32 g, 9.3 mmol, 86%).

Synthesis of substrates **8A** and **8B**



Synthesis of substrate **S11** (According to literature procedures with minor revision[2]): To a cooled (0 °C) solution of 1,3-cyclohexanedione (11.2 g, 100 mmol) in water (20 mL) were added dropwise *N,N*-diisopropylethylamine (DIPEA) (19.8 mL, 120 mmol) and 2,3-dibromopropene (8.3 mL, 80 mmol). The mixture was warm to rt and stirred for 48 h. The reaction mixture was diluted with EtOAc (300 mL) and washed with 1N HCl (2 x 100 mL), water (2 x 100 mL), and then brine (2 x 100 mL). The mixture was dried (Na_2SO_4) and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 1/1) to give **S11** (11.6 g, 50.0 mmol) as a yellow solid in 63% yield.

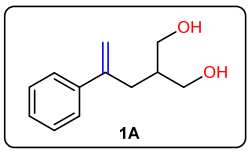
Synthesis of substrate **S12** (According to literature procedures with minor revision[3]): To a solution of **S11** (11.6 g, 50 mmol) in THF (150 mL) at rt were added 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) (9.0 mL, 60 mmol) and anhydrous LiI (8.04 g, 60 mmol) and the resulting mixture was stirred for 30 min at rt followed by addition of ethyl bromoacetate (11.1 mL, 100 mmol). The reaction mixture was then warmed to 65 °C and stirred for 24 h at that temperature. Upon completion, the reaction mixture was cooled to rt, and brine (150 mL) was added followed by extraction with ethyl acetate (3 x 200 mL). The combined organic extracts were dried(Na_2SO_4) and concentrated *in vacuo.* The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 3/1) to give **S12** (10.3 g, 32.5 mmol) as a yellow solid in 65% yield.

Synthesis of substrates **S13**

To a solution of **S12** (1.0 eq.), arylboronic acid (1.5 eq.), and K_2CO_3 (3.0 eq.) in THF/H_2O (v/v = 3/1) was added $PdCl_2(PPh_3)_2$ (0.1 eq.). The flask was evacuated and then backfilled with argon three times. Then the reaction mixture was stirred at rt for 10 h under argon atmosphere. The mixture was extracted with CH_2Cl_2 (3x). The combined organic layer was washed with brine, dried (Na_2SO_4), and concentrated *in*

vacuo. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 4/1) to give **S13**.

Synthesis of substrates **8** (According to literature procedures with minor revision[3]): To a solution of **S13** (1.0 eq.) in THF (0.1 M) at 0 °C was slowly added $LiAlH(O^tBu)_3$ (1.1 M in THF, 2.5 eq.). The resulting solution was stirred at 0 °C for 0.5 h and then at 25 °C for 2 h. Upon completion, the reaction was quenched by saturated NH_4Cl (aq.). Then EtOAc was added and the reaction mixture was acidified to pH 5 by slow addition of 1N HCl to facilitate the removal of the aluminum salt. After stirring for 10 min, the mixture was extract with EtOAc (3x). The combined organic extracts were washed with saturated $NaHCO_3$ (aq.), dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 3/1) to give **8**.

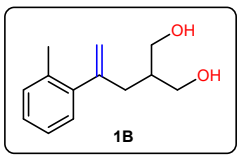


2-(2-phenylallyl)propane-1,3-diol (1A)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.37 (m, 2H), 7.34–7.30 (m, 2H), 7.28–7.24 (m, 1H), 5.32 (d, *J* = 1.4 Hz, 1H), 5.09 (d, *J* = 1.1 Hz, 1H), 3.73–3.70 (m, 2H), 3.62–3.57 (m, 2H), 2.79 (brs, 2H), 2.50 (d, *J* = 7.4 Hz, 2H), 1.85–1.80 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 140.4, 128.4, 127.6, 126.2, 114.3, 65.3, 40.0, 33.8.

HRMS (ESI) calcd for $C_{12}H_{16}NaO_2$ $[M+Na]^+$ 215.1043, found: 215.1040.

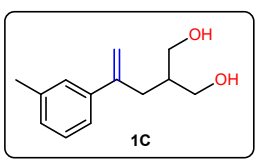


2-(2-(*o*-tolyl)allyl)propane-1,3-diol (1B)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17–7.11 (m, 3H), 7.08 (d, *J* = 7.0 Hz, 1H), 5.23 (s, 1H), 4.97 (s, 1H), 3.77 (dd, *J* = 10.7, 3.4 Hz, 2H), 3.63 (dd, *J* = 10.3, 7.5 Hz, 2H), 2.60 (brs, 2H), 2.34 (d, *J* = 7.1 Hz, 2H), 2.31 (s, 3H) 1.79–1.74 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.4, 142.0, 134.8, 130.3, 128.3, 127.0, 125.5, 116.2, 65.7, 39.8, 35.9, 19.9.

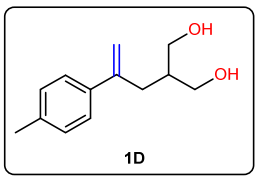
HRMS (ESI) calcd for $C_{13}H_{18}NaO_2$ $[M+Na]^+$ 229.1199, found: 229.1197.



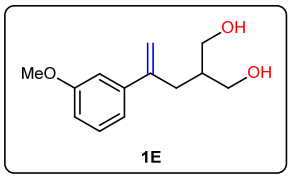
2-(2-(*m*-tolyl)allyl)propane-1,3-diol (1C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.16 (m, 3H), 7.08 (d, *J* = 7.0 Hz, 1H), 5.29 (s,

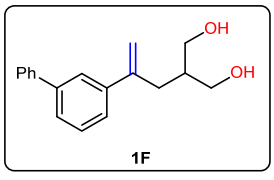
1H), 5.07 (s, 1H), 3.74–3.71 (m, 2H), 3.65–3.58 (m, 2H), 2.79 (brs, 2H), 2.48 (d, J = 6.7 Hz, 2H), 2.35 (s, 3H), 1.85–1.82 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 146.3, 140.5, 137.9, 128.3, 128.2, 126.9, 123.3, 114.1, 65.3, 40.0, 33.9, 21.4.
HRMS (ESI) calcd for $C_{13}H_{18}NaO_2$ $[M+Na]^+$ 229.1199, found: 229.1198.



2-(2-(*p*-tolyl)allyl)propane-1,3-diol (1D)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, J = 8.2 Hz, 2H), 7.11 (d, J = 7.9 Hz, 2H), 5.27 (d, J = 1.6 Hz, 1H), 5.02 (d, J = 1.2 Hz, 1H), 3.65 (dd, J = 10.8, 3.9 Hz, 2H), 3.53 (dd, J = 10.8, 7.0 Hz, 2H), 3.37 (brs, 2H), 2.43 (d, J = 7.3 Hz, 2H), 2.31 (s, 3H), 1.83–1.74 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 145.8, 137.5, 137.2, 129.0, 125.9, 113.4, 64.7, 40.0, 33.8, 20.9.
HRMS (ESI) calcd for $C_{13}H_{18}NaO_2$ $[M+Na]^+$ 229.1199, found: 229.1197.

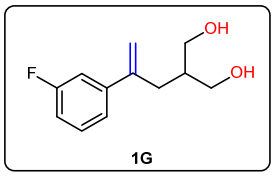


2-(2-(3-methoxyphenyl)allyl)propane-1,3-diol (1E)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (t, J = 7.9 Hz, 1H), 6.98 (d, J = 7.7 Hz, 1H), 6.93 (s, 1H), 6.82 (d, J = 8.2 Hz, 1H), 5.32 (s, 1H), 5.09 (s, 1H), 3.81 (s, 3H), 3.74–3.72 (m, 2H), 3.63–3.59 (m, 2H), 2.78 (brs, 2H), 2.48 (d, J = 7.3 Hz, 2H), 1.88–1.80 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 146.1, 142.1, 129.4, 118.7, 114.5, 112.6, 112.3, 65.3, 55.2, 40.1, 33.9.
HRMS (ESI) calcd for $C_{13}H_{18}NaO_3$ $[M+Na]^+$ 245.1148, found: 245.1145.



2-(2-([1,1'-biphenyl]-3-yl)allyl)propane-1,3-diol (1F)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60–7.58 (m, 3H), 7.51–7.33 (m, 6H), 5.38 (s, 1H), 5.14 (s, 1H), 3.76 (dd, J = 10.7, 3.8 Hz, 2H), 3.64 (dd, J = 10.7, 7.0 Hz, 2H), 2.56 (d, J = 7.4 Hz, 2H), 2.50 (brs, 2H), 1.91–1.85 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 146.2, 141.4, 141.1, 141.0, 128.8, 128.7, 127.4, 127.2, 126.5, 125.13, 125.09, 114.7, 65.5, 40.1, 33.9.

HRMS (ESI) calcd for $C_{18}H_{20}NaO_2$ $[M+Na]^+$ 291.1356, found: 291.1352.



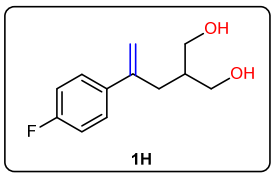
2-(2-(3-fluorophenyl)allyl)propane-1,3-diol (1G)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (dd, *J* = 14.4, 7.5 Hz, 1H), 7.15 (d, *J* = 7.7 Hz, 1H), 7.08 (d, *J* = 10.4 Hz, 1H), 6.94 (t, *J* = 8.1 Hz, 1H), 5.33 (s, 1H), 5.11 (s, 1H), 3.66 (dd, *J* = 10.7, 3.6 Hz, 2H), 3.57–3.53 (m, 4H), 2.44 (d, *J* = 7.3 Hz, 2H), 1.78–1.75 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8 (d, *J* = 243.8 Hz), 144.9 (d, *J* = 2.0 Hz), 142.8 (d, *J* = 7.3 Hz), 129.8 (d, *J* = 8.4 Hz), 121.8 (d, *J* = 2.7 Hz), 115.2, 114.3 (d, *J* = 21.0 Hz), 113.0 (d, *J* = 21.6 Hz), 64.4, 39.9, 33.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -113.12 – -113.19 (m, 1F).

HRMS (ESI) calcd for $C_{12}H_{15}FNaO_2$ $[M+Na]^+$ 233.0948, found: 233.0946.



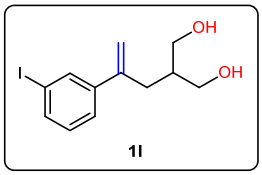
2-(2-(4-fluorophenyl)allyl)propane-1,3-diol (1H)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.34 (m, 2H), 7.01 (t, *J* = 8.1 Hz, 2H), 5.27 (s, 1H), 5.08 (s, 1H), 3.75–3.72 (m, 2H), 3.64–3.59 (m, 2H), 2.75 (brs, 2H), 2.48 (d, *J* = 7.3 Hz, 2H), 1.86–1.78 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3 (d, *J* = 245.1 Hz), 145.1, 136.5, 127.8 (d, *J* = 8.0 Hz), 115.2 (d, *J* = 21.2 Hz), 114.3, 65.3, 40.0, 33.9.

^{19}F NMR (376 MHz, $CDCl_3$) δ -114.76 – -114.84 (m, 1F).

HRMS (ESI) calcd for $C_{12}H_{15}FNaO_2$ $[M+Na]^+$ 233.0948, found: 233.0945.

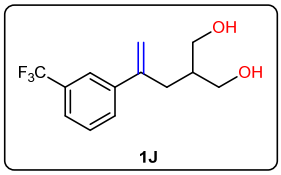


2-(2-(3-iodophenyl)allyl)propane-1,3-diol (1I)

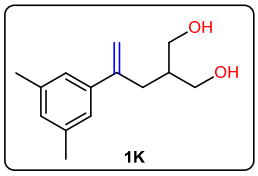
Diisobutyl aluminium hydride (DIBAH) (8.0 eq.) was used instead of $LiAlH_4$ in the reduction step.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.06 (t, *J* = 7.7 Hz, 1H), 5.31 (s, 1H), 5.13 (s, 1H), 3.77–3.73 (m, 2H), 3.66–3.60 (m, 2H), 2.62–2.41 (m, 4H), 1.82 (s, 1H).

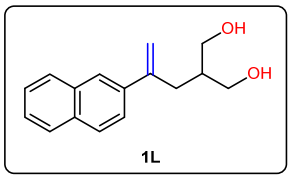
^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 142.9, 136.5, 135.2, 130.1, 125.4, 115.50, 94.6, 65.4, 39.9, 33.7.
HRMS (ESI) calcd for $C_{12}H_{16}IO_2$ $[M+H]^+$ 319.0189, found: 319.0187.



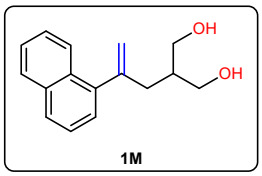
2-(2-(3-(trifluoromethyl)phenyl)allyl)propane-1,3-diol (1J)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (s, 1H), 7.56 (d, *J* = 7.7 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 1H), 5.37 (s, 1H), 5.17 (s, 1H), 3.69 (d, *J* = 6.6 Hz, 2H), 3.59 (d, *J* = 6.3 Hz, 2H), 3.40 (s, 2H), 2.50 (d, *J* = 6.9 Hz, 2H), 1.81–1.74 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 141.4, 130.7 (q, *J* = 31.9 Hz), 129.4, 128.9, 124.2 (q, *J* = 3.6 Hz), 124.1 (d, *J* = 270.7 Hz), 122.8 (q, *J* = 3.8 Hz), 115.8, 64.6, 39.9, 33.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.6 (s, 3F).
HRMS (ESI) calcd for $C_{13}H_{15}F_3NaO_2$ $[M+Na]^+$ 283.0916, found: 283.0912.



2-(2-(3,5-dimethylphenyl)allyl)propane-1,3-diol (1K)
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (s, 2H), 6.91 (s, 1H), 5.27 (s, 1H), 5.04 (s, 1H), 3.71 (dd, *J* = 10.7, 3.3 Hz, 2H), 3.59 (dd, *J* = 10.5, 7.3 Hz, 2H), 2.96 (brs, 2H), 2.45 (d, *J* = 7.4 Hz, 2H), 2.30 (s, 6H), 1.86–1.80 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4, 140.6, 137.8, 129.2, 124.0, 113.9, 65.2, 39.9, 33.9, 21.3.
HRMS (ESI) calcd for $C_{14}H_{20}NaO_2$ $[M+Na]^+$ 243.1356, found: 243.1354.



2-(2-(naphthalen-2-yl)allyl)propane-1,3-diol (1L)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81–7.78 (m, 4H), 7.55 (d, *J* = 8.5 Hz, 1H), 7.47–7.43 (m, 2H), 5.46 (s, 1H), 5.19 (s, 1H), 3.76 (d, *J* = 11.0 Hz, 2H), 3.67–3.63 (m, 2H), 2.62 (d, *J* = 7.3 Hz, 2H), 2.40 (brs, 2H), 1.88 (s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 137.8, 133.3, 132.8, 128.1, 128.0, 127.5, 126.2, 125.9, 124.9, 124.6, 114.9, 65.6, 40.2, 33.9.
HRMS (ESI) calcd for $C_{16}H_{18}NaO_2$ $[M+Na]^+$ 265.1199, found: 265.1195.

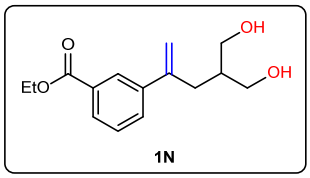


2-(2-(naphthalen-1-yl)allyl)propane-1,3-diol (1M)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 6.1, 3.6 Hz, 1H), 7.85 (dd, *J* = 6.3, 3.2 Hz, 1H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.49–7.46 (m, 2H), 7.45–7.40 (m, 1H), 7.29 (d, *J* = 7.0 Hz, 1H), 5.46 (s, 1H), 5.20 (d, *J* = 1.6 Hz, 1H), 3.80 (d, *J* = 10.5 Hz, 2H), 3.69–3.65 (m, 2H), 2.56 (d, *J* = 7.2 Hz, 2H), 2.13 (brs, 2H), 1.81–1.75 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.2, 140.3, 133.8, 131.1, 128.4, 127.5, 125.9, 125.7, 125.5, 125.2, 125.1, 117.7, 65.9, 40.2, 36.8.

HRMS (ESI) calcd for $C_{16}H_{18}NaO_2$ $[M+Na]^+$ 265.1199, found: 265.1196.

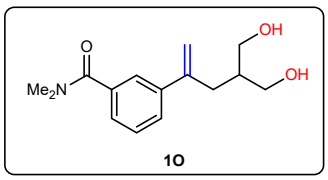


ethyl 3-(5-hydroxy-4-(hydroxymethyl)pent-1-en-2-yl)benzoate (1N)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.92 (d, *J* = 7.5 Hz, 1H), 7.58 (d, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 5.37 (s, 1H), 5.15 (s, 1H), 4.35 (q, *J* = 6.8 Hz, 2H), 3.91 (brs, 2H), 3.69–3.66 (m, 2H), 3.60–3.56 (m, 2H), 2.52 (d, *J* = 7.0 Hz, 2H), 1.78 (s, 1H), 1.38 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 145.1, 140.7, 136.0, 130.4, 128.3, 128.2, 127.0, 115.0, 63.8, 61.0, 40.0, 33.4, 14.0.

HRMS (ESI) calcd for $C_{15}H_{20}NaO_4$ $[M+Na]^+$ 287.1254, found: 287.1250.

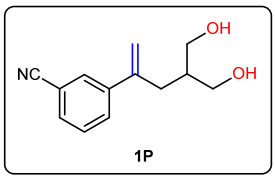


3-(5-hydroxy-4-(hydroxymethyl)pent-1-en-2-yl)-N,N-dimethylbenzamide (1O)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46–7.43 (m, 2H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 5.33 (s, 1H), 5.12 (s, 1H), 3.63 (d, *J* = 10.7 Hz, 2H), 3.55–3.51 (m, 2H), 3.45 (brs, 2H), 3.10 (s, 3H), 2.99 (s, 3H), 2.48 (d, *J* = 7.3 Hz, 2H), 1.75 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 145.7, 141.1, 136.0, 128.4, 127.6, 125.8, 124.9, 115.0, 64.6, 40.3, 39.6, 35.4, 33.8.

HRMS (ESI) calcd for $C_{15}H_{21}NNaO_3$ $[M+Na]^+$ 286.1414, found: 286.1410.

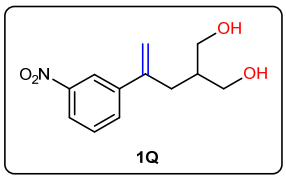


3-(5-hydroxy-4-(hydroxymethyl)pent-1-en-2-yl)benzonitrile (1P)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.64 (d, *J* = 7.9 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 5.39 (s, 1H), 5.23 (s, 1H), 3.77 (d, *J* = 10.5 Hz, 2H), 3.68–3.64 (m, 2H), 2.55 (d, *J* = 7.4 Hz, 2H), 2.48 (brs, 2H), 1.81 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.3, 141.9, 131.0, 130.6, 129.8, 129.3, 118.8, 116.5, 112.6, 65.2, 39.9, 33.5.

HRMS (ESI) calcd for $C_{13}H_{15}NNaO_2$ $[M+Na]^+$ 240.0995, found: 240.0993.

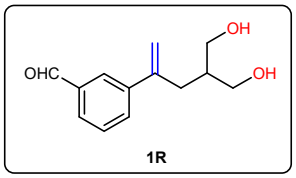


2-(2-(3-nitrophenyl)allyl)propane-1,3-diol (1Q)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (s, 1H), 8.13 (d, *J* = 8.1 Hz, 1H), 7.74 (d, *J* = 7.7 Hz, 1H), 7.52 (t, *J* = 7.9 Hz, 1H), 5.47 (s, 1H), 5.27 (s, 1H), 3.79–3.76 (m, 2H), 3.68–3.64 (m, 2H), 2.59–2.57 (m, 4H), 1.82 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 148.4, 144.2, 142.3, 132.2, 129.4, 122.3, 121.0, 116.8, 65.0, 40.0, 33.4.

HRMS (ESI) calcd for $C_{12}H_{15}NNaO_4$ $[M+Na]^+$ 260.0893, found: 260.0891.

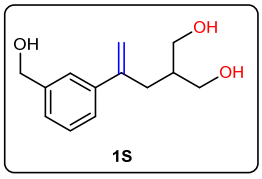


3-(5-hydroxy-4-(hydroxymethyl)pent-1-en-2-yl)benzaldehyde (1R)

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.89 (s, 1H), 7.76 (d, *J* = 7.5 Hz, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 5.40 (s, 1H), 5.19 (s, 1H), 3.71 (d, *J* = 10.5 Hz, 2H), 3.63–3.59 (m, 2H), 3.33 (brs, 2H), 2.54 (d, *J* = 7.2 Hz, 2H), 1.80–1.79 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 144.9, 141.5, 136.3, 132.2, 129.14, 129.11, 126.9, 115.7, 64.6, 40.0, 33.5.

HRMS (ESI) calcd for $C_{13}H_{16}NaO_3$ $[M+Na]^+$ 243.0992, found: 243.0989.

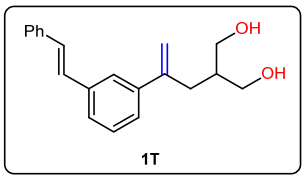


2-(2-(3-(hydroxymethyl)phenyl)allyl)propane-1,3-diol (1S)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.33–7.29 (m, 2H), 7.26–7.24 (m, 1H), 5.33 (s, 1H), 5.11 (s, 1H), 4.67 (s, 2H), 3.72 (dd, *J* = 10.7, 3.8 Hz, 2H), 3.61 (dd, *J* = 10.6, 7.1 Hz, 2H), 2.52–2.50 (m, 4H), 1.85–1.81 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.2, 141.0, 140.9, 128.7, 126.3, 125.5, 124.9, 114.6, 65.5, 65.2, 40.2, 34.0.

HRMS (ESI) calcd for $C_{13}H_{18}NaO_3$ $[M+Na]^+$ 245.1148, found: 245.1145.

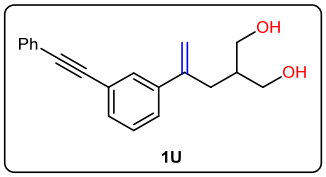


(*E*)-2-(2-(3-styrylphenyl)allyl)propane-1,3-diol (1T)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49–7.47 (m, 3H), 7.39 (d, *J* = 7.0 Hz, 1H), 7.32 (t, *J* = 7.3 Hz, 2H), 7.26–7.24 (m, 3H), 7.08 (s, 2H), 5.32 (s, 1H), 5.08 (s, 1H), 3.67 (dd, *J* = 10.6, 3.4 Hz, 2H), 3.57–3.53 (m, 2H), 3.24 (brs, 2H), 2.47 (d, *J* = 7.1 Hz, 2H), 1.79 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.0, 141.0, 137.3, 137.0, 128.9, 128.64, 128.57, 128.4, 127.6, 126.4, 125.4, 124.5, 114.5, 64.7, 40.0, 33.8.

HRMS (ESI) calcd for $C_{20}H_{22}NaO_2$ $[M+Na]^+$ 317.1512, found: 317.1508.

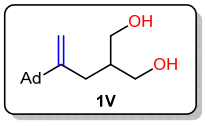


2-(2-(3-(phenylethynyl)phenyl)allyl)propane-1,3-diol (1U)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59–7.56 (m, 3H), 7.49–7.46 (m, 1H), 7.42–7.31 (m, 5H), 5.38 (d, *J* = 1.2 Hz, 1H), 5.18 (s, 1H), 3.82 (d, *J* = 10.3 Hz, 2H), 3.72–3.68 (m, 2H), 2.57 (d, *J* = 7.4 Hz, 2H), 2.20 (brs, 2H), 1.92–1.83 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.6, 140.8, 131.6, 130.7, 129.4, 128.5, 128.3, 126.2, 123.4, 123.1, 115.1, 89.5, 89.2, 65.7, 40.0, 33.8.

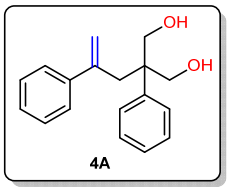
HRMS (ESI) calcd for $C_{20}H_{21}O_2$ $[M+H]^+$ 293.1536, found: 293.1533.



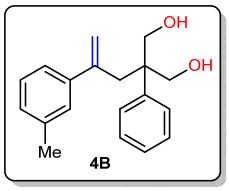
2-(2-(adamantan-1-yl)allyl)propane-1,3-diol (1V)

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.92 (s, 1H), 4.78 (d, *J* = 1.2 Hz, 1H), 3.84 (d, *J* = 10.1 Hz, 2H), 3.68–3.63 (m, 2H), 2.58 (brs, 2H), 2.10–2.03 (m, 1H), 2.00–1.96 (m, 5H), 1.74–1.63 (m, 12H).

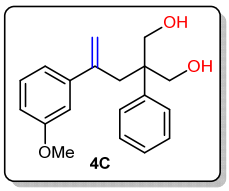
^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5, 107.9, 66.4, 41.04, 41.02, 38.0, 36.9, 28.61, 28.59.
HRMS (ESI) calcd for $C_{16}H_{27}O_2$ $[M+H]^+$ 251.2006, found: 251.2003.



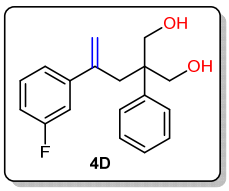
2-phenyl-2-(2-phenylallyl)propane-1,3-diol (4A)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.6 Hz, 2H), 7.18– 7.16 (m, 3H), 7.10–7.07 (m, 3H), 7.00 (d, *J* = 8.6 Hz, 2H), 5.06 (d, *J* = 1.6 Hz, 1H), 4.73 (s, 1H), 3.82 (d, *J* = 11.2 Hz, 2H), 3.73 (d, *J* = 11.2 Hz, 2H), 2.79 (brs, 2H), 2.71 (s, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 142.3, 140.0, 131.1, 129.0, 128.1, 127.1, 126.2, 120.2, 117.7, 67.1, 47.6, 40.5.
HRMS (ESI) m/z calcd. for $C_{18}H_{20}NaO_2$ $[M+Na]^+$ 291.1356, found 291.1351.



2-phenyl-2-(2-(*m*-tolyl)allyl)propane-1,3-diol (4B)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28–7.26 (m, 4H), 7.19–7.15 (m, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 7.04–6.97 (m, 3H), 5.10 (d, *J* = 1.8 Hz, 1H), 4.76–5.75 (m, 1H), 3.97 (dd, *J* = 11.3, 5.3 Hz, 2H), 3.88 (dd, *J* = 11.3, 6.0 Hz, 2H), 2.85 (s, 2H), 2.28 (s, 3H), 2.04 (brs, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 142.6, 141.1, 137.8, 128.4, 128.14, 128.08, 127.10, 127.06, 126.5, 123.3, 117.5, 67.8, 48.2, 40.6, 21.4.
HRMS (ESI) m/z calcd. for $C_{19}H_{22}NaO_2$ $[M+Na]^+$ 305.1512, found 305.1505.



2-(2-(3-methoxyphenyl)allyl)-2-phenylpropane-1,3-diol (4C)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29–7.28 (m, 4H), 7.21–7.14 (m, 2H), 6.84–6.82 (m, 1H), 6.77–6.72 (m, 2H), 5.12 (d, *J* = 1.7 Hz, 1H), 4.77 (s, 1H), 4.00 (dd, *J* = 11.3, 5.3 Hz, 2H), 3.90 (dd, *J* = 11.3, 6.1 Hz, 2H), 3.77 (s, 3H), 2.86 (s, 2H), 1.99 (br s, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 144.8, 144.2, 141.0, 129.2, 128.4, 127.1, 126.5, 118.8, 117.8, 112.6, 112.3, 67.7, 55.2, 48.2, 40.5.
HRMS (ESI) m/z calcd. for $C_{19}H_{22}NaO_3$ $[M+Na]^+$ 321.1461, found 321.1457.



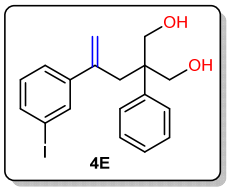
2-(2-(3-fluorophenyl)allyl)-2-phenylpropane-1,3-diol (4D)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22–7.15 (m, 6H), 7.00 (d, *J* = 7.8 Hz, 1H), 6.92 (d, *J* = 11.1 Hz, 1H), 6.83 (t, *J* = 8.2 Hz, 1H), 5.11 (s, 1H), 4.79 (s, 1H), 3.91 (d, *J* = 11.1 Hz, 2H), 3.84 (d, *J* = 11.2 Hz, 2H), 2.81 (s, 2H), 2.35 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5 (d, *J* = 243.8 Hz), 144.9 (d, *J* = 7.5 Hz), 143.9, 140.6, 129.5 (d, *J* = 8.4 Hz), 128.4, 127.1, 126.6, 122.0 (d, *J* = 2.8 Hz), 118.5, 113.9 (d, *J* = 21.1 Hz), 113.3 (d, *J* = 21.7 Hz), 67.7, 48.0, 40.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ -112.92 – -112.98 (m, 1F).

HRMS (ESI) m/z calcd. for $C_{18}H_{19}FNaO_2$ $[M+Na]^+$ 309.1261, found 309.1257.



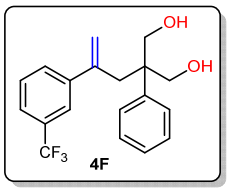
2-(2-(3-iodophenyl)allyl)-2-phenylpropane-1,3-diol (4E)

Diisobutyl aluminium hydride (DIBAH) (8.0 eq.) was used instead of $LiAlH_4$ in the reduction step.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.9 Hz, 1H), 7.36 (s, 1H), 7.21–7.16 (m, 2H), 7.12–7.08 (m, 3H), 7.05 (d, *J* = 7.9 Hz, 1H), 6.85 (t, *J* = 7.6 Hz, 1H), 5.06 (s, 1H), 4.82 (s, 1H), 3.92 (d, *J* = 10.8 Hz, 2H), 3.82 (d, *J* = 11.2 Hz, 2H), 2.74 (s, 2H), 2.66 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 143.7, 140.3, 135.7, 135.3, 129.5, 128.2, 126.9, 126.6, 125.5, 118.4, 94.1, 67.2, 47.5, 40.2.

HRMS (ESI) m/z calcd. for $C_{18}H_{19}INaO_2$ $[M+Na]^+$ 417.0322, found 417.0316.



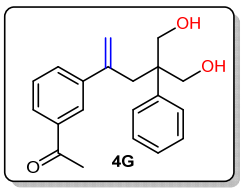
2-phenyl-2-(2-(3-(trifluoromethyl)phenyl)allyl)propane-1,3-diol (4F)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.34 (m, 1H), 7.27–7.21 (m, 3H), 7.17–7.12 (m, 4H), 7.09–7.05 (m, 1H), 5.15 (d, *J* = 1.6 Hz, 1H), 4.96 (s, 1H), 4.02 (d, *J* = 10.8 Hz, 2H), 3.91 (d, *J* = 10.8 Hz, 2H), 2.86 (s, 2H), 2.32 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 143.2, 140.1, 130.1 (q, *J* = 32.1 Hz), 129.6, 128.4, 128.3, 127.0, 126.6, 124.0 (q, *J* = 270.8 Hz), 123.6 (q, *J* = 3.6 Hz), 123.1 (q, *J* = 3.9 Hz), 118.9, 67.5, 47.5, 40.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.6 (s, 3F).

HRMS (ESI) m/z calcd. for $C_{19}H_{19}F_3NaO_2$ $[M+Na]^+$ 359.1229, found 359.1225.

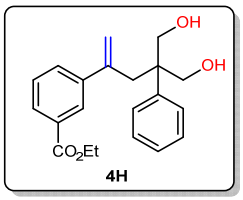


1-(3-(5-hydroxy-4-(hydroxymethyl)-4-phenylpent-1-en-2-yl)phenyl)ethan-1-one (4G)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.7 Hz, 1H), 7.58 (s, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.21 (t, *J* = 7.7 Hz, 1H), 7.11 (d, *J* = 4.3 Hz, 4H), 7.05–7.00 (m, 1H), 5.13 (d, *J* = 1.3 Hz, 1H), 4.91 (s, 1H), 3.95 (d, *J* = 11.1 Hz, 2H), 3.86 (d, *J* = 11.1 Hz, 2H), 2.91 (brs, 2H), 2.86 (s, 2H), 2.51 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 144.3, 142.9, 140.6, 136.4, 131.0, 128.11, 128.06, 126.9, 126.7, 126.2, 126.1, 118.3, 67.2, 47.4, 40.2, 26.6.

HRMS (ESI) m/z calcd. for $C_{20}H_{22}NaO_3$ $[M+Na]^+$ 333.1461, found 333.1456.

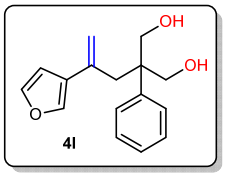


methyl 3-(5-hydroxy-4-(hydroxymethyl)-4-phenylpent-1-en-2-yl)benzoate (4H)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.7 Hz, 1H), 7.74 (s, 1H), 7.27 (d, *J* = 8.0 Hz, 1H), 7.19 (t, *J* = 7.7 Hz, 1H), 7.14–7.13 (m, 4H), 7.08–7.02 (m, 1H), 5.14 (s, 1H), 4.89 (s, 1H), 4.36 (q, *J* = 7.1 Hz, 2H), 3.95 (d, *J* = 11.1 Hz, 2H), 3.86 (d, *J* = 11.1 Hz, 2H), 3.08 (brs, 2H), 2.85 (s, 2H), 1.40 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 144.2, 142.7, 140.6, 130.7, 129.8, 128.0, 127.80, 127.78, 127.3, 126.9, 126.1, 118.1, 67.0, 60.9, 47.4, 40.2, 14.2.

HRMS (ESI) m/z calcd. for $C_{21}H_{24}NaO_4$ $[M+Na]^+$ 363.1567, found 363.1561.

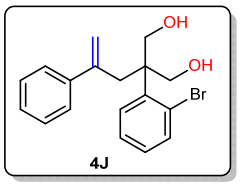


2-(2-(furan-3-yl)allyl)-2-phenylpropane-1,3-diol (4I)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29–7.21 (m, 5H), 7.19–7.14 (m, 2H), 6.33 (dd, *J* = 2.0, 0.9 Hz, 1H), 5.15 (d, *J* = 1.4 Hz, 1H), 4.50 (s, 1H), 3.96 (d, *J* = 11.2 Hz, 2H), 3.87 (d, *J* = 11.2 Hz, 2H), 2.98 (brs, 2H), 2.57 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8, 141.3, 139.1, 134.7, 128.3, 127.8, 127.1, 126.4, 114.7, 108.1, 66.8, 47.5, 39.6.

HRMS (ESI) m/z calcd. for $C_{16}H_{18}NaO_3$ $[M+Na]^+$ 281.1148, found 281.1143.



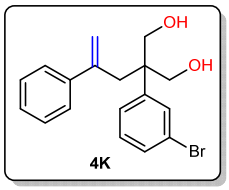
2-(2-bromophenyl)-2-(2-phenylallyl)propane-1,3-diol (4J)

Diisobutyl aluminium hydride (DIBAH) (8.0 eq.) was used instead of $LiAlH_4$ in the reduction step.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.13–7.08 (m, 6H), 6.97 (dd, *J* = 7.2, 1.6 Hz, 1H), 5.05 (d, *J* = 1.8 Hz, 1H), 4.93 (s, 1H), 4.46 (d, *J* = 11.6 Hz, 2H), 3.92 (d, *J* = 11.2 Hz, 2H), 3.14 (s, 2H), 2.34 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 142.3, 138.4, 135.7, 131.9, 128.3, 128.0, 127.2, 127.1, 126.3, 122.4, 117.4, 68.0, 50.0, 36.5.

HRMS (ESI) m/z calcd. for $C_{18}H_{19}BrNaO_2$ $[M+Na]^+$ 369.0461, found 369.0456.



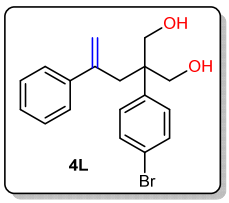
2-(3-bromophenyl)-2-(2-phenylallyl)propane-1,3-diol (4K)

Diisobutyl aluminium hydride (DIBAH) (8.0 eq.) was used instead of $LiAlH_4$ in the reduction step.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23–7.14 (m, 5H), 7.10–7.07 (m, 3H), 7.02 (t, *J* = 8.0 Hz, 1H), 5.08 (s, 1H), 4.78 (s, 1H), 3.83 (d, *J* = 11.2 Hz, 2H), 3.75 (d, *J* = 11.2 Hz, 2H), 2.83 (brs, 2H), 2.73 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 143.5, 142.2, 130.5, 129.6, 129.3, 128.0, 127.2, 126.2, 125.8, 122.4, 117.7, 67.0, 47.8, 40.4.

HRMS (ESI) m/z calcd. for $C_{18}H_{19}BrNaO_2$ $[M+Na]^+$ 369.0461, found 369.0456.



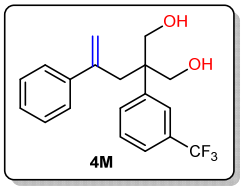
2-(4-bromophenyl)-2-(2-phenylallyl)propane-1,3-diol (4L)

Diisobutyl aluminium hydride (DIBAH) (8.0 eq.) was used instead of $LiAlH_4$ in the reduction step.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.2 Hz, 2H), 7.21–7.19 (m, 3H), 7.14–7.12 (m, 2H), 7.07 (d, *J* = 8.2 Hz, 2H), 5.10 (s, 1H), 4.77 (s, 1H), 3.90 (d, *J* = 11.2 Hz, 2H), 3.82 (d, *J* = 11.2 Hz, 2H), 2.78 (s, 2H), 2.29 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.7, 142.4, 140.1, 131.3, 129.0, 128.2, 127.2, 126.3, 120.4, 117.9, 67.6, 47.9, 40.6.

HRMS (ESI) m/z calcd. for $C_{18}H_{19}BrNaO_2$ $[M+Na]^+$ 369.0461, found 369.0456.



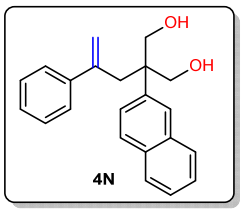
2-(2-phenylallyl)-2-(3-(trifluoromethyl)phenyl)propane-1,3-diol (4M)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.29 (m, 4H), 7.16–7.14 (m, 3H), 7.11–7.08 (m, 2H), 5.10 (s, 1H), 4.81 (s, 1H), 4.03–4.00 (m, 2H), 3.95–3.89 (m, 2H), 2.84 (d, J = 6.7 Hz, 2H), 2.30 (brs, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 144.7, 142.13, 142.09, 130.7, 130.4 (q, J = 31.6 Hz), 128.6, 128.2, 127.3, 126.2, 124.1 (q, J = 270.8 Hz), 124.1 (q, J = 3.8 Hz), 123.2 (q, J = 3.9 Hz), 117.9, 67.6, 47.9, 40.8.

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.5 (s, 3F).

HRMS (ESI) m/z calcd. for $C_{19}H_{19}F_3NaO_2$ $[M+Na]^+$ 359.1229, found 359.1225.

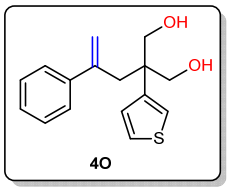


2-(naphthalen-2-yl)-2-(2-phenylallyl)propane-1,3-diol (4N)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.74 (m, 4H), 7.65 (s, 1H), 7.46–7.44 (m, 3H), 7.21–7.12 (m, 4H), 5.07 (s,1H), 4.77 (s, 1H), 4.10–4.07 (m, 2H), 3.99–3.95 (m, 2H), 2.95 (d, J = 3.6 Hz, 2H), 2.05 (brs, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 142.6, 138.3, 133.3, 132.0, 128.2, 128.1, 128.0, 127.31, 127.25, 126.5, 126.3, 125.9, 125.8, 124.9, 117.8, 67.9, 48.5, 40.3.

HRMS (ESI) m/z calcd. for $C_{22}H_{22}NaO_2$ $[M+Na]^+$ 341.1512, found 341.1507.

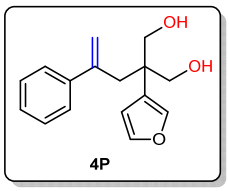


2-(2-phenylallyl)-2-(thiophen-3-yl)propane-1,3-diol (4O)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23–7.18 (m, 6H), 6.98–6.93 (m, 2H), 5.15 (d, J = 1.6 Hz, 1H), 4.85 (s, 1H), 3.86–3.80 (m, 4H), 2.87 (s, 2H), 2.07 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 142.6, 142.4, 128.2, 127.3, 126.2, 125.6, 121.5, 117.7, 67.6, 47.3, 39.7.

HRMS (ESI) m/z calcd. for $C_{16}H_{18}NaO_2S$ $[M+Na]^+$ 297.0920, found 297.0916.

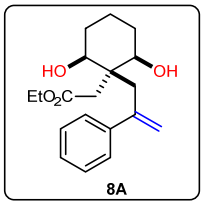


2-(furan-3-yl)-2-(2-phenylallyl)propane-1,3-diol (4P)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26–7.19 (m, 6H), 7.10 (s, 1H), 6.21 (s, 1H), 5.18 (d, *J* = 1.6 Hz, 1H), 4.93 (s, 1H), 3.64 (s, 4H), 2.77 (s, 2H), 2.52 (brs, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.8, 142.7, 139.6, 128.1, 127.2, 126.2, 125.6, 117.7, 108.8, 66.7, 44.1, 38.9.

HRMS (ESI) m/z calcd. for $C_{16}H_{18}NaO_3$ $[M+Na]^+$ 281.1148, found 281.1144.



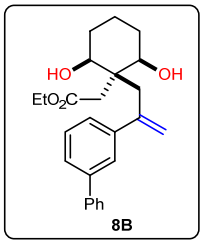
ethyl 2-(-2,6-dihydroxy-1-(2-phenylallyl)cyclohexyl)acetate (8A)

^{1}H NMR (400 MHz, DMSO) δ 7.41 (d, *J* = 7.3 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 2H), 7.24–7.19 (m, 1H), 5.30–5.24 (m, 2H), 4.68 (brs, 2H), 3.82 (q, *J* = 7.1 Hz, 2H), 3.71–3.67 (m, 2H), 2.90 (s, 2H), 2.21 (s, 2H), 1.71–1.66 (m, 1H), 1.59–1.53 (m, 2H), 1.50–1.42 (m, 2H), 1.21–1.13 (m, 1H), 1.05 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, DMSO, 80 °C) δ 171.7, 147.3, 144.0, 128.2, 127.2, 126.9, 118.2, 71.5, 59.6, 45.0, 36.5, 33.6, 29.3, 16.8, 14.4.

HRMS (ESI) m/z calcd. for $C_{19}H_{27}O_4$ $[M+H]^+$ 319.1904, found 319.1904.

The relative configuration of **8A** was determined by X-ray experiment (see Supplementary Figure 3).



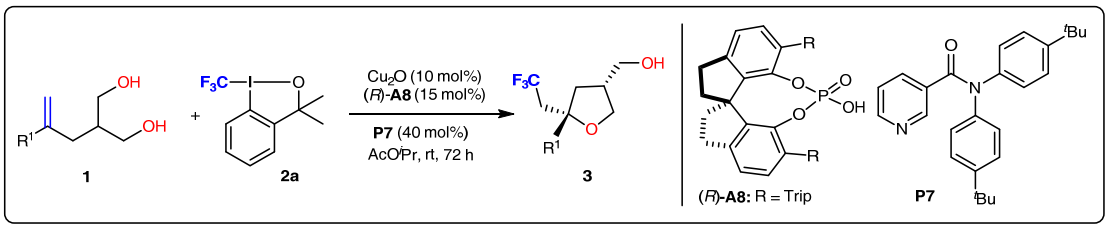
ethyl 2-(1-(2-([1,1'-biphenyl]-3-yl)allyl)-2,6-dihydroxycyclohexyl)acetate (8B)

^{1}H NMR (400 MHz, DMSO) δ 7.72–7.68 (m, 3H), 7.53 (dt, *J* = 7.1, 1.6 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.43–7.33 (m, 3H), 5.37 (d, *J* = 2.2 Hz, 1H), 5.33 (d, *J* = 1.9 Hz, 1H), 4.80 (brs, 2H), 3.81–3.75 (m, 4H), 2.99 (s, 2H), 2.27 (s, 2H), 1.72 (s, 1H), 1.62–1.57 (m, 2H), 1.54–1.46 (m, 2H), 1.23–1.14 (m, 1H), 1.02 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, DMSO, 80 °C) δ 171.7, 147.1, 144.6, 141.1, 140.3, 129.2, 128.8, 127.7, 127.2, 126.1, 125.7, 125.6, 118.6, 71.6, 59.6, 45.0, 36.6, 33.8, 29.3, 16.6, 14.3.

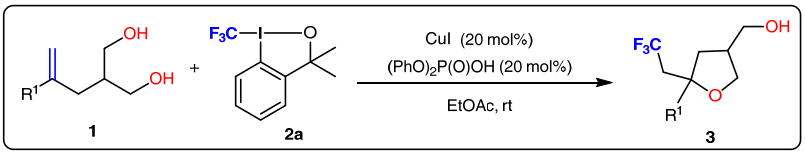
HRMS (ESI) m/z calcd. for $C_{25}H_{31}O_4$ $[M+H]^+$ 395.2217, found 395.2218.

3.3 General Procedure A: Cu/CPA-catalyzed enantioselective desymmetrising radical oxytrifluoromethylation of olefinic 1,3-diols

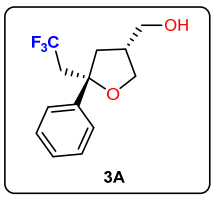


Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1** (0.2 mmol, 1.0 eq.), Cu_2O (2.9 mg, 0.02 mmol, 10 mol%), chiral phosphoric acid (*R*)-**A8** (21.6 mg, 0.03 mmol, 15 mol%), **P7** (30.9 mg, 0.08 mmol, 40 mol%), Togni's reagent **2a** (99.0 mg, 0.3 mmol, 1.5 eq.), and AcO^iPr (2.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. Upon completion (monitored by TLC), the solvent was removed *in vacuo*, and the residue was purified by a silica gel chromatography to afford the desired product **3**.

Note: Since the reaction is sensitive to water and air, the Schlenk tube and the reagents must be dried prior to use.



The racemate was prepared by following the same procedure as described above using substrate **1** (0.1 mmol, 1.0 eq.), Togni's reagent **2a** (49.5 mg, 0.15 mmol, 1.5 eq.) CuI (3.8 mg, 0.02 mmol, 20 mol%) and diphenyl phosphate (5.0 mg, 0.02 mmol, 20 mol%) at 25 °C in EtOAc (1.0 mL). Upon completion (monitored by TLC), the solvent was removed *in vacuo*, and the residue was purified by silica gel column chromatography to afford the desired product **3** as an inseparable mixture of diastereomers.



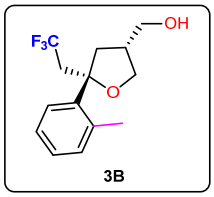
((2*S*,4*R*)-2-phenyl-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3A)

Product **3A** was obtained as a colorless oil in 79% (41.0 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.

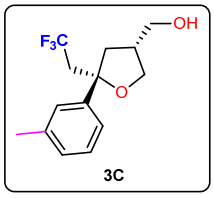
$[\alpha]_D^{27}$ = –42 (*c* 1.2, $CHCl_3$).

HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.4 mL/min, λ = 210 nm), t_R (major) = 32.12 min, t_R (minor) = 28.03 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.33 (m, 4H), 7.26 (t, *J* = 7.0 Hz, 1H), 3.98 (t, *J* = 8.4 Hz, 1H), 3.78 (t, *J* = 7.9 Hz, 1H), 3.67–3.59 (m, 2H), 2.73 (d, *J* = 10.8 Hz, 1H), 2.67 (d, *J* = 10.8 Hz, 1H), 2.57 (dd, *J* = 12.3, 7.4 Hz, 1H), 2.38–2.29 (m, 1H), 1.95 (dd, *J* = 12.0, 10.0 Hz, 1H), 1.89 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 128.3, 127.3, 125.26 (q, *J* = 276.6 Hz), 125.21, 83.4 (q, *J* = 2.1 Hz), 69.7, 64.4, 45.4 (q, *J* = 26.4 Hz), 40.9, 40.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{13}H_{15}F_3NaO_2$ $[M+Na]^+$ 283.0916, found: 283.0914.

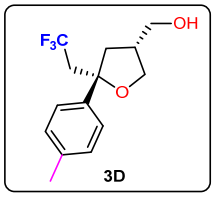


((2*S*,4*R*)-2-(*o*-tolyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3B)
Product **3B** was obtained as a colorless oil in 66% (36.2 mg, 0.13 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.
$[\alpha]_D^{27}$ = –40 (*c* 0.9, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 35.09 min, t_R (minor) = 39.37 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.54 (m, 1H), 7.20–7.17 (m, 3H), 3.98 (t, *J* = 8.1 Hz, 1H), 3.84 (t, *J* = 8.8 Hz, 1H), 3.66 (d, *J* = 6.6 Hz, 2H), 2.88–2.73 (m, 2H), 2.59 (dd, *J* = 12.5, 7.8 Hz, 1H), 2.42 (s, 3H), 2.39–2.32 (m, 1H), 2.04 (dd, *J* = 12.5, 7.9 Hz, 1H), 1.68 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 141.4, 133.7, 132.3, 127.5, 125.9, 125.8, 125.4 (q, *J* = 276.7 Hz), 83.6 (q, *J* = 2.0 Hz), 69.4, 64.4, 43.5 (q, *J* = 26.2 Hz), 41.4, 40.8, 21.3.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.8 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{14}H_{17}F_3NaO_2$ $[M+Na]^+$ 297.1073, found: 297.1070.

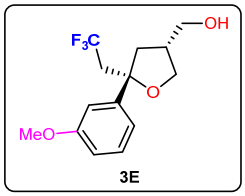


((2*S*,4*R*)-2-(*m*-tolyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3C)
Product **3C** was obtained as a colorless oil in 73% (40.0 mg, 0.15 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.
$[\alpha]_D^{27}$ = –16 (*c* 0.5, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 96/4, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 32.89 min, t_R (minor) = 31.52 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.16 (m, 3H), 7.07 (d, *J* = 7.2 Hz, 1H), 3.98 (t, *J* = 8.4 Hz, 1H), 3.77 (t, *J* = 7.9 Hz, 1H), 3.67–3.59 (m, 2H), 2.71 (d, *J* = 10.8 Hz, 1H),

2.66 (d, J = 10.8 Hz, 1H), 2.56 (dd, J = 12.3, 7.4 Hz, 1H), 2.38–2.30 (m, 4H), 1.97–1.91 (m, 1H), 1.86 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.6, 137.9, 128.1, 128.0, 125.8, 125.3 (q, J = 276.5 Hz), 122.3, 83.4 (q, J = 2.1 Hz), 69.7, 64.4, 45.4 (q, J = 26.3 Hz), 40.9, 40.8, 21.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, J = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{14}H_{17}F_3NaO_2$ $[M+Na]^+$ 297.1073, found: 297.1072.



((2*S*,4*R*)-2-(*p*-tolyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3D)
Product **3D** was obtained as a colorless oil in 80% (43.8 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.
$[\alpha]_D^{27}$ = –25 (*c* 0.8, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 30.76 min, t_R (minor) = 38.80 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, J = 8.0 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 3.96 (t, J = 8.4 Hz, 1H), 3.78 (t, J = 8.4 Hz, 1H), 3.69–3.61 (m, 2H), 2.71 (d, J = 10.8 Hz, 1H), 2.66 (d, J = 10.8 Hz, 1H), 2.57 (dd, J = 12.3, 7.5 Hz, 1H), 2.39–2.32 (m, 4H), 1.93 (dd, J = 12.3, 9.6 Hz, 1H), 1.62 (brs, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 140.4, 137.0, 129.0, 125.3 (q, J = 276.6 Hz), 125.2, 83.4 (q, J = 2.6 Hz), 69.6, 64.6, 45.5 (q, J = 26.3 Hz), 40.9, 40.8 (q, J = 1.2 Hz), 21.0.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, J = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{14}H_{17}F_3NaO_2$ $[M+Na]^+$ 297.1073, found: 297.1071.



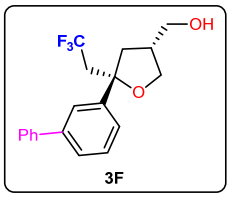
((2*S*,4*R*)-2-(3-methoxyphenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3E)
Product **3E** was obtained as a colorless oil in 83% (48.1 mg, 0.17 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.
$[\alpha]_D^{27}$ = –23 (*c* 0.5, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 210 nm), t_R (major) = 40.77 min, t_R (minor) = 44.54 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (t, J = 8.0 Hz, 1H), 6.98 (t, J = 2.1 Hz, 1H), 6.95 (d, J = 7.7 Hz, 1H), 6.81 (dd, J = 8.2, 2.5 Hz, 1H), 4.00 (t, J = 8.4 Hz, 1H), 3.82 (s, 3H), 3.81–3.77 (m, 1H), 3.69–3.61 (m, 2H), 2.72 (dd, J = 10.8, 1.8 Hz, 1H), 2.66 (dd,

J = 10.8, 1.5 Hz, 1H), 2.56 (dd, *J* = 12.3, 7.4 Hz, 1H), 2.42–2.30 (m, 1H), 1.95 (dd, *J* = 12.3, 9.6 Hz, 1H), 1.64 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 145.4, 129.4, 125.3 (q, *J* = 276.6 Hz), 117.6, 112.3, 111.4, 83.4 (q, *J* = 2.2 Hz), 69.8, 64.4, 55.2, 45.3 (q, *J* = 26.4 Hz), 40.91 (q, *J* = 1.2 Hz), 40.86.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, *J* = 10.8 Hz, 3F).

HRMS (ESI) calcd for $C_{14}H_{17}F_3NaO_3$ $[M+Na]^+$ 313.1022, found: 313.1018.



3F

((2*S*,4*R*)-2-([1,1'-biphenyl]-3-yl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3F)

Product **3F** was obtained as a colorless oil in 80% (53.8 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.

$[\alpha]_D^{27}$ = −52 (*c* 1.5, $CHCl_3$).

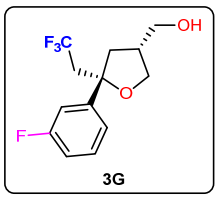
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 24.14 min, t_R (minor) = 32.04 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64–7.58 (m, 3H), 7.51–7.34 (m, 6H), 4.03 (t, *J* = 8.4 Hz, 1H), 3.82 (dd, *J* = 8.7, 7.2 Hz, 1H), 3.70–3.62 (m, 2H), 2.78 (dd, *J* = 10.7, 2.0 Hz, 1H), 2.72 (dd, *J* = 10.8, 1.7 Hz, 1H), 2.63 (dd, *J* = 12.3, 7.4 Hz, 1H), 2.44–2.33 (m, 1H), 2.00 (dd, *J* = 12.4, 9.6 Hz, 1H), 1.65 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 141.3, 141.0, 128.7, 127.4, 127.2, 126.2, 125.3 (q, *J* = 276.5 Hz), 124.2, 124.1, 83.5 (q, *J* = 2.3 Hz), 69.8, 64.4, 45.5 (q, *J* = 26.5 Hz), 41.0, 40.9.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.3 (t, *J* = 10.8 Hz, 3F).

HRMS (ESI) calcd for $C_{19}H_{19}F_3NaO_2$ $[M+Na]^+$ 359.1229, found: 359.1226.



3G

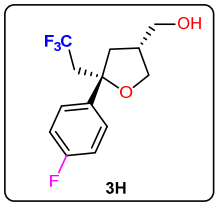
((2*S*,4*R*)-2-(3-fluorophenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3G)

Product **3G** was obtained as a colorless oil in 72% (40.0 mg, 0.14 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.

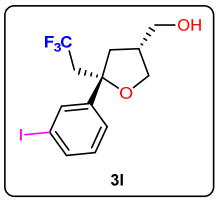
$[\alpha]_D^{27}$ = −26 (*c* 1.2, $CHCl_3$).

HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 210 nm), t_R (major) = 22.04 min, t_R (minor) = 20.59 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34–7.29 (m, 1H), 7.16–7.14 (m, 2H), 6.96 (t, *J* = 7.9 Hz, 1H), 3.99 (t, *J* = 8.4 Hz, 1H), 3.80 (t, *J* = 8.0 Hz, 1H), 3.69–3.62 (m, 2H), 2.72 (d, *J* = 10.6 Hz, 1H), 2.67 (d, *J* = 10.7 Hz, 1H), 2.54 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.39–2.31 (m, 1H), 1.97 (t, *J* = 10.8 Hz, 1H), 1.68 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9 (d, *J* = 244.3 Hz), 146.4 (d, *J* = 6.2 Hz), 129.9 (d, *J* = 8.0 Hz), 125.1 (q, *J* = 276.3 Hz), 120.9 (d, *J* = 2.9 Hz), 114.2 (d, *J* = 21.0 Hz), 112.6 (d, *J* = 22.7 Hz), 83.1 (q, *J* = 1.8 Hz), 69.8, 64.2, 45.3 (q, *J* = 26.7 Hz), 41.2, 40.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, *J* = 10.8 Hz, 3F), -112.7–-112.8 (m, 1F).
HRMS (ESI) calcd for $C_{13}H_{14}F_4NaO_2$ $[M+Na]^+$ 301.0822, found: 301.0822.



((2*S*,4*R*)-2-(4-fluorophenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3H)
Product **3H** was obtained as a colorless oil in 76% (42.3 mg, 0.15 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.
$[\alpha]_D^{27}$ = –28 (*c* 1.0, $CHCl_3$).
HPLC analysis: Chiralcel AS3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 22.39 min, t_R (minor) = 24.52 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.34 (m, 2H), 7.06–7.00 (m, 2H), 3.96 (t, *J* = 8.4 Hz, 1H), 3.79 (dd, *J* = 8.7, 7.1 Hz, 1H), 3.70–3.62 (m, 2H), 2.71 (d, *J* = 10.7 Hz, 1H), 2.66 (d, *J* = 10.8 Hz, 1H), 2.56 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.40–2.31 (m, 1H), 1.94 (dd, *J* = 12.4, 9.6 Hz, 1H), 1.65 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0 (d, *J* = 244.5 Hz), 139.0 (d, *J* = 3.1 Hz), 127.1 (d, *J* = 8.0 Hz), 125.2 (q, *J* = 276.4 Hz), 115.1 (d, *J* = 21.3 Hz), 83.1 (q, *J* = 2.2 Hz), 69.6, 64.4, 45.6 (q, *J* = 26.3 Hz), 41.1, 40.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, *J* = 10.8 Hz, 3F), -115.60–-115.64 (m, 1F).
HRMS (ESI) calcd for $C_{13}H_{14}F_4NaO_2$ $[M+Na]^+$ 301.0822, found: 301.0822.



((2*S*,4*R*)-2-(3-iodophenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3I)
Product **3I** was obtained as a colorless oil in 72% (55.6 mg, 0.14 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.

$[\alpha]_D^{27}$ = –20 (*c* 1.2, $CHCl_3$).

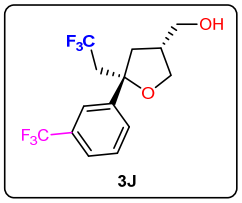
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 230 nm), t_R (major) = 12.33 min, t_R (minor) = 14.97 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.8 Hz, 1H), 3.99 (t, *J* = 8.4 Hz, 1H), 3.79 (t, *J* = 8.0 Hz, 1H), 3.69–3.62 (m, 2H), 2.70 (d, *J* = 10.7 Hz, 1H), 2.65 (d, *J* = 10.7 Hz, 1H), 2.52 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.40–2.29 (m, 1H), 1.96 (dd, *J* = 12.1, 10.0 Hz, 1H), 1.65 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 136.4, 134.4, 130.1, 125.1 (q, *J* = 276.6 Hz), 124.6, 94.4, 82.9 (q, *J* = 2.2 Hz), 69.8, 64.2, 45.3 (q, *J* = 26.6 Hz), 41.0 (q, *J* = 1.1 Hz), 40.8.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.7 Hz, 3F).

HRMS (ESI) calcd for $C_{13}H_{14}F_3INaO_2$ $[M+Na]^+$ 408.9883, found: 408.9883.



3J

((2*S*,4*R*)-2-(2,2,2-trifluoroethyl)-2-(3-(trifluoromethyl)phenyl)tetrahydrofuran-4-yl)methanol (3J)

Product **3J** was obtained as a colorless oil in 65% (42.6 mg, 0.13 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.

$[\alpha]_D^{27}$ = –22 (*c* 1.3, $CHCl_3$).

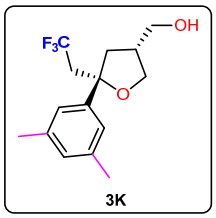
HPLC analysis: Chiralcel AS3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.3 mL/min, λ = 214 nm), t_R (major) = 29.85 min, t_R (minor) = 31.69 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (s, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.55 (d, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 1H), 4.00 (t, *J* = 8.4 Hz, 1H), 3.83 (dd, *J* = 8.7, 7.4 Hz, 1H), 3.71–3.64 (m, 2H), 2.75 (d, *J* = 10.6 Hz, 1H), 2.69 (d, *J* = 10.7 Hz, 1H), 2.57 (dd, *J* = 12.5, 7.4 Hz, 1H), 2.40–2.28 (m, 1H), 2.02 (dd, *J* = 12.5, 9.7 Hz, 1H), 1.64 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.7, 130.8 (q, *J* = 32.0 Hz), 128.9, 128.8, 125.1 (q, *J* = 276.5 Hz), 124.3 (q, *J* = 3.7 Hz), 124.1 (q, *J* = 270.6 Hz), 122.2 (q, *J* = 3.7 Hz), 83.2 (q, *J* = 2.4 Hz), 69.9, 64.1, 45.4 (q, *J* = 26.7 Hz), 41.3 (q, *J* = 1.6 Hz), 40.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, *J* = 10.7 Hz, 3F), -62.5 (s, 3F).

HRMS (ESI) calcd for $C_{14}H_{15}F_6O_2$ $[M+H]^+$ 329.0971, found: 329.0967.



3K

((2*S*,4*R*)-2-(3,5-dimethylphenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)met

hanol (3K)

Product **3K** was obtained as a colorless oil in 80% (46.1 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.

$[\alpha]_D^{27}$ = –22 (*c* 0.8, $CHCl_3$).

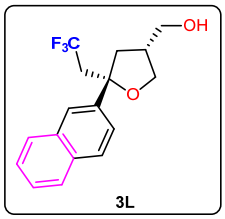
HPLC analysis: Chiralcel IC (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 26.57 min, t_R (minor) = 33.80 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (s, 2H), 6.90 (s, 1H), 4.00 (t, *J* = 8.3 Hz, 1H), 3.77 (t, *J* = 7.9 Hz, 1H), 3.68–3.61 (m, 2H), 2.70 (d, *J* = 10.8 Hz, 1H), 2.65 (d, *J* = 10.8 Hz, 1H), 2.54 (dd, *J* = 12.3, 7.4 Hz, 1H), 2.40–2.32 (m, 7H), 1.94 (t, *J* = 11.2 Hz, 1H), 1.71 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 137.7, 128.9, 125.3 (q, *J* = 276.4 Hz), 122.9, 83.4 (q, *J* = 2.2 Hz), 69.7, 64.5, 45.3 (q, *J* = 26.2 Hz), 40.9, 40.7, 21.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.8 Hz, 3F).

HRMS (ESI) calcd for $C_{15}H_{19}F_3NaO_2$ $[M+Na]^+$ 311.1229, found: 311.1225.



3L

((2*S*,4*R*)-2-(naphthalen-2-yl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3L)

Product **3L** was obtained as a colorless oil in 74% (45.9 mg, 0.15 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.

$[\alpha]_D^{27}$ = –11 (*c* 0.4, $CHCl_3$).

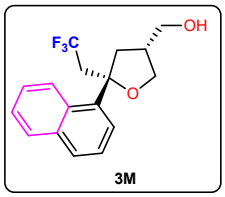
HPLC analysis: Chiralcel AD3 (*n*-Hexane/*i*-PrOH = 94/6, flow rate 0.5 mL/min, λ = 270 nm), t_R (major) = 32.51 min, t_R (minor) = 27.45 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.85–7.82 (m, 3H), 7.52–7.45 (m, 3H), 4.06 (t, *J* = 8.4 Hz, 1H), 3.85 (dd, *J* = 8.7, 7.2 Hz, 1H), 3.72–3.64 (m, 2H), 2.83 (d, *J* = 10.8 Hz, 1H), 2.77 (d, *J* = 10.8 Hz, 1H), 2.70 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.44–2.32 (m, 1H), 2.03 (dd, *J* = 12.4, 9.7 Hz, 1H), 1.58 (brs, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 140.7, 133.0, 132.5, 128.20, 128.16, 127.5, 126.3, 126.1, 125.2 (q, *J* = 276.4 Hz), 124.2, 123.4, 83.6 (q, *J* = 1.8 Hz), 69.8, 64.5, 45.3 (q, *J* = 26.5 Hz), 40.9.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.6 Hz, 3F).

HRMS (ESI) calcd for $C_{17}H_{17}F_3NaO_2$ $[M+Na]^+$ 333.1073, found: 333.1069.



((2*S*,4*R*)-2-(naphthalen-1-yl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3M)

Product **3M** was obtained as a colorless oil in 65% (40.3 mg, 0.13 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure, except that the reaction was stirred at 50 °C.

$[\alpha]_D^{27}$ = –20 (*c* 0.4, $CHCl_3$).

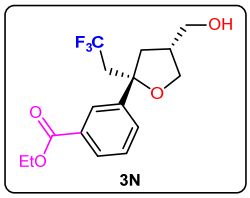
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 270 nm), t_R (major) = 12.41 min, t_R (minor) = 16.42 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.9 Hz, 1H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 2H), 7.54–7.43 (m, 3H), 4.07 (t, *J* = 8.0 Hz, 1H), 3.91 (dd, *J* = 8.7, 6.1 Hz, 1H), 3.68 (d, *J* = 6.5 Hz, 2H), 3.11–3.00 (m, 2H), 2.91 (dd, *J* = 12.1, 7.3 Hz, 1H), 2.40–2.26 (m, 2H), 1.82 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.9, 134.8, 129.7, 129.4, 128.8, 125.9, 125.5 (q, *J* = 276.8 Hz) 125.10, 125.07, 124.5, 123.9, 83.7 (q, *J* = 1.8 Hz), 69.1, 64.4, 44.3 (q, *J* = 26.2 Hz), 41.6, 40.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.6 (t, *J* = 10.9 Hz, 3F).

HRMS (ESI) calcd for $C_{17}H_{17}F_3NaO_2$ $[M+Na]^+$ 333.1073, found: 333.1072.



Ethyl 3-((2*S*,4*R*)-4-(hydroxymethyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-2-yl) benzoate (3N)

Product **3N** was obtained as a colorless oil in 70% (46.5 mg, 0.14 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 3/1) following the general procedure.

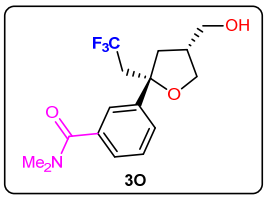
$[\alpha]_D^{27}$ = –25 (*c* 1.0, $CHCl_3$).

HPLC analysis: Chiralcel IB (*n*-Hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 230 nm), t_R (major) = 17.18 min, t_R (minor) = 19.10 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (t, *J* = 1.8 Hz, 1H), 7.96 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.65–7.62 (m, 1H), 7.43 (t, *J* = 7.8 Hz, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 4.00 (t, *J* = 8.4 Hz, 1H), 3.82 (dd, *J* = 8.7, 7.3 Hz, 1H), 3.70–3.63 (m, 2H), 2.76 (dd, *J* = 10.7, 1.8 Hz, 1H), 2.70 (dd, *J* = 10.7, 1.6 Hz, 1H), 2.60 (dd, *J* = 12.5, 7.4 Hz, 1H), 2.37–2.28 (m, 1H), 2.00 (dd, *J* = 12.4, 9.7 Hz, 1H), 1.78 (brs, 1H), 1.41 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 166.5, 144.0, 130.6, 129.8, 128.6, 128.4, 126.4, 125.1 (q, *J* = 276.5 Hz), 83.3 (q, *J* = 1.8 Hz), 69.8, 64.2, 61.1, 45.3 (q, *J* = 26.6 Hz), 41.2, 40.7, 14.3.

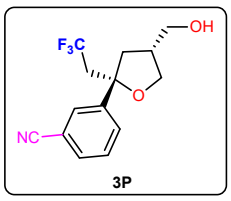
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{16}H_{19}F_3NaO_4$ $[M+Na]^+$ 355.1128, found: 355.1123.



3O

3-((2*S*,4*R*)-4-(hydroxymethyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-2-yl)-N,N-dimethylbenzamide (3O)

Product **3O** was obtained as a yellow oil in 75% (49.6 mg, 0.15 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 1/1) following the general procedure, except that the reaction was stirred at 50 °C.
$[\alpha]_D^{27}$ = –26 (*c* 1.0, $CHCl_3$).
HPLC analysis: Chiralcel INB (*n*-Hexane/*i*-PrOH = 75/25, flow rate 0.3 mL/min, λ = 230 nm), t_R (major) = 27.51 min, t_R (minor) = 40.32 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.38 (m, 3H), 7.32 (d, *J* = 7.3 Hz, 1H), 3.96 (t, *J* = 8.4 Hz, 1H), 3.78 (dd, *J* = 8.7, 7.2 Hz, 1H), 3.63–3.56 (m, 2H), 3.11 (s, 3H), 2.93 (s, 3H), 2.74 (dd, *J* = 10.7, 3.0 Hz, 1H), 2.68 (dd, *J* = 10.7, 2.7 Hz, 1H), 2.55 (dd, *J* = 12.4, 7.3 Hz, 1H), 2.33–2.24 (m, 1H), 1.93 (dd, *J* = 12.4, 9.6 Hz, 1H), 1.26 (brs, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 143.7, 136.3, 128.6, 126.5, 126.0, 125.2 (q, *J* = 276.5 Hz), 123.9, 83.2 (q, *J* = 1.5 Hz), 69.9, 64.0, 45.5 (q, *J* = 26.5 Hz), 41.5, 40.7, 39.4, 35.3.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{16}H_{21}F_3NO_3$ $[M+H]^+$ 332.1468, found: 332.1463.

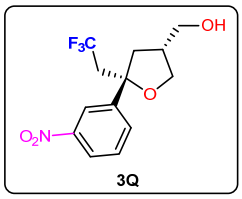


3P

3-((2*S*,4*R*)-4-(hydroxymethyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-2-yl)benzonitrile (3P)

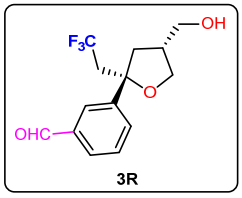
Product **3P** was obtained as a colorless oil in 84% (47.9 mg, 0.17 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 3/1) following the general procedure, except that the reaction was stirred at 50 °C.
$[\alpha]_D^{27}$ = –35 (*c* 1.3, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 210 nm), t_R (major) = 33.39 min, t_R (minor) = 35.79 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.65 (d, *J* = 7.9 Hz, 1H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 1H), 3.99 (t, *J* = 8.4 Hz, 1H), 3.84 (t, *J* = 8.0 Hz, 1H), 3.67 (d, *J* = 6.3 Hz, 2H), 2.74 (d, *J* = 10.6 Hz, 1H), 2.69 (d, *J* = 10.6 Hz, 1H), 2.53 (dd, *J* = 12.5, 7.4 Hz, 1H), 2.38–2.27 (m, 1H), 2.03 (dd, *J* = 12.6, 9.6 Hz, 1H), 1.78 (brs, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 131.1, 129.9, 129.22, 129.18, 124.9 (q, *J* = 276.6 Hz), 118.7, 112.5, 82.9 (q, *J* = 2.3 Hz), 69.9, 63.8, 45.3 (q, *J* = 26.9 Hz), 41.4, 40.7.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) calcd for $C_{13}H_{14}F_3NNaO_2$ $[M+Na]^+$ 308.0869, found: 308.0864.



3Q

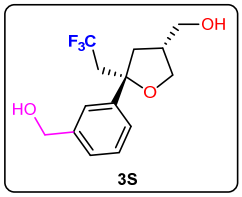
((2*S*,4*R*)-2-(3-nitrophenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3Q)
Product **3Q** was obtained as a yellow oil in 82% (50.0 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 1/1) following the general procedure.
$[\alpha]_D^{27}$ = –26 (*c* 1.2, $CHCl_3$).
HPLC analysis: Chiralcel OJ-H (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 50.29 min, t_R (minor) = 58.03 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (s, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.55 (t, *J* = 7.9 Hz, 1H), 4.03 (t, *J* = 8.4 Hz, 1H), 3.86 (t, *J* = 8.2 Hz, 1H), 3.68 (d, *J* = 6.3 Hz, 2H), 2.79 (d, *J* = 10.6 Hz, 1H), 2.73 (d, *J* = 10.6 Hz, 1H), 2.59 (dd, *J* = 12.6, 7.4 Hz, 1H), 2.38–2.31 (m, 1H), 2.07 (t, *J* = 10.0 Hz, 1H), 1.82 (brs, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 148.4, 146.0, 131.6, 129.4, 124.9 (q, *J* = 276.5 Hz), 122.5, 120.6, 83.0 (q, *J* = 1.8 Hz), 70.0, 63.8, 45.3 (q, *J* = 26.8 Hz), 41.6, 40.7.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.6 Hz, 3F).
HRMS (ESI) calcd for $C_{13}H_{15}F_3NO_4$ $[M+H]^+$ 306.0948, found: 306.0944.



3R

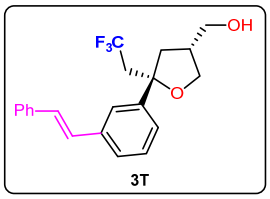
3-((2*S*,4*R*)-4-(hydroxymethyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-2-yl)benzaldehyde (3R)
Product **3R** was obtained as a colorless oil in 71% (40.9 mg, 0.14 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 2/1) following the general procedure, except that the reaction was stirred at 50 °C.
$[\alpha]_D^{27}$ = –45 (*c* 3.1, $CHCl_3$).
HPLC analysis: Chiralcel AD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 29.33 min, t_R (minor) = 33.00 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.03 (s, 1H), 7.93 (s, 1H), 7.80 (d, *J* = 7.4 Hz, 1H), 7.71 (d, *J* = 7.7 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 4.01 (t, *J* = 8.4 Hz, 1H), 3.84 (t, *J* = 8.0 Hz, 1H), 3.71–3.64 (m, 2H), 2.77 (d, *J* = 10.6 Hz, 1H), 2.72 (d, *J* = 10.7 Hz, 1H),

2.61 (dd, J = 12.5, 7.4 Hz, 1H), 2.40–2.29 (m, 1H), 2.02 (t, J = 9.6 Hz, 1H), 1.94 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 192.3, 144.9, 136.4, 131.5, 129.1, 129.0, 126.4, 125.1 (q, J = 276.3 Hz), 83.1 (q, J = 2.6 Hz), 69.9, 64.1, 45.4 (q, J = 26.6 Hz), 41.4, 40.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, J = 10.6 Hz, 3F).
HRMS (ESI) calcd for $C_{14}H_{16}F_3O_3$ $[M+H]^+$ 289.1046, found: 289.1042.



3S

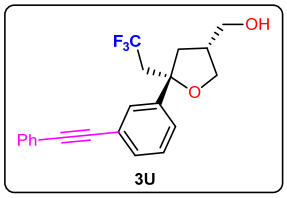
(3-((2*S*,4*R*)-4-(hydroxymethyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-2-yl)phenyl)methanol (3S)
Product **3S** was obtained as a colorless oil in 79% (45.8 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 1/1) following the general procedure.
$[\alpha]_D^{27}$ = –13 (*c* 0.6, $CHCl_3$).
HPLC analysis: Chiralcel AS3 (*n*-Hexane/*i*-PrOH = 87/13, flow rate 0.5 mL/min, λ = 210 nm), t_R (major) = 29.11 min, t_R (minor) = 26.27 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1H), 7.37–7.31 (m, 2H), 7.28–7.26 (m, 1H), 4.70 (s, 2H), 3.98 (t, J = 8.4 Hz, 1H), 3.78 (dd, J = 8.7, 7.3 Hz, 1H), 3.68–3.59 (m, 2H), 2.73 (dd, J = 10.8, 1.6 Hz, 1H), 2.68 (dd, J = 10.7, 1.3 Hz, 1H), 2.57 (dd, J = 12.3, 7.4 Hz, 1H), 2.39–2.28 (m, 1H), 1.95 (dd, J = 12.4, 9.7 Hz, 1H), 1.87 (brs, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 140.9, 128.5, 125.9, 125.2 (q, J = 276.5 Hz), 124.6, 123.7, 83.4 (q, J = 2.3 Hz), 69.8, 65.2, 64.4, 45.4 (q, J = 26.5 Hz), 41.1, 40.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, J = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{14}H_{18}F_3O_3$ $[M+H]^+$ 291.1203, found: 291.1200.



3T

((2*S*,4*R*)-2-(3-((*E*)-styryl)phenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3T)
Product **3T** was obtained as a colorless oil in 70% (50.7 mg, 0.14 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.
$[\alpha]_D^{27}$ = –10 (*c* 0.8, $CHCl_3$).
HPLC analysis: Chiralcel ID (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.4 mL/min, λ = 210 nm), t_R (major) = 29.11 min, t_R (minor) = 26.27 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56–7.51 (m, 3H), 7.44 (d, J = 7.6 Hz, 1H), 7.39–7.33 (m, 3H), 7.28–7.25 (m, 2H), 7.13 (s, 2H), 4.04 (t, J = 8.5 Hz, 1H), 3.82 (t, J = 8.0 Hz,

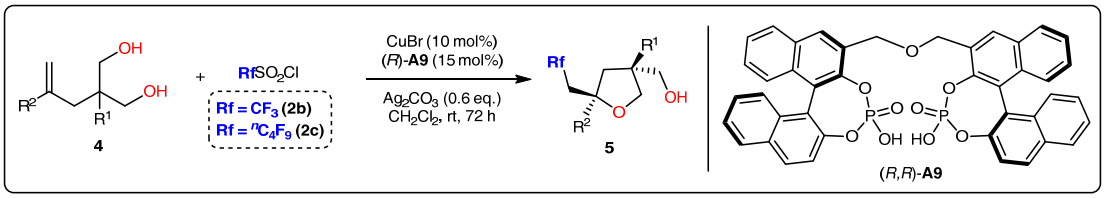
1H), 3.71–3.63 (m, 2H), 2.76 (d, *J* = 10.8 Hz, 1H), 2.71 (d, *J* = 10.7 Hz, 1H), 2.61 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.44–2.30 (m, 1H), 1.99 (t, *J* = 10.4 Hz, 1H), 1.72 (brs, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 144.1, 137.4, 137.1, 129.0, 128.7, 128.5, 127.7, 126.5, 125.4, 125.3 (q, *J* = 276.5 Hz), 124.6, 123.4, 83.4 (q, *J* = 2.4 Hz), 69.8, 64.4, 45.4 (q, *J* = 26.4 Hz), 40.94, 40.87.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) calcd for $C_{21}H_{21}F_3NaO_2$ $[M+Na]^+$ 385.1386, found: 385.1379.



3U

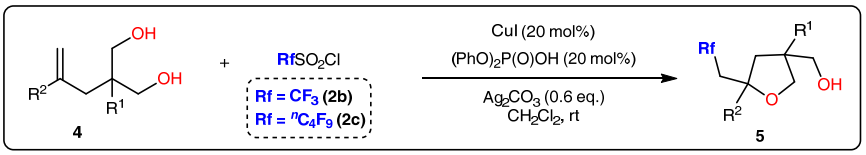
((2*S*,4*R*)-2-(3-(phenylethynyl)phenyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (3U)
Product **3U** was obtained as a colorless oil in 78% (56.2 mg, 0.16 mmol) yield in a 0.20 mmol scale synthesis by column chromatography (PE/EA = 4/1) following the general procedure.
$[\alpha]_D^{27}$ = −8 (*c* 0.6, $CHCl_3$).
HPLC analysis: Chiralcel OJ-H (*n*-Hexane/*i*-PrOH = 80/20, flow rate 0.4 mL/min, λ = 280 nm), t_R (major) = 37.18 min, t_R (minor) = 30.21 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.53 (m, 3H), 7.45 (dt, *J* = 7.1, 1.6 Hz, 1H), 7.39–7.32 (m, 5H), 4.02 (t, *J* = 8.4 Hz, 1H), 3.81 (dd, *J* = 8.7, 7.3 Hz, 1H), 3.70–3.63 (m, 2H), 2.74 (dd, *J* = 10.7, 1.5 Hz, 1H), 2.69 (dd, *J* = 10.7, 1.2 Hz, 1H), 2.59 (dd, *J* = 12.4, 7.4 Hz, 1H), 2.42–2.33 (m, 1H), 1.98 (dd, *J* = 12.4, 9.7 Hz, 1H), 1.62 (brs, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 143.9, 131.6, 130.6, 128.5, 128.41, 128.35, 125.3, 125.2 (q, *J* = 276.6 Hz), 123.3, 123.0, 89.5, 89.2, 83.2 (q, *J* = 2.1 Hz), 69.8, 64.3, 45.3 (q, *J* = 26.3 Hz), 41.0, 40.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) calcd for $C_{21}H_{20}F_3O_2$ $[M+H]^+$ 361.1410, found: 361.1406.

3.4 General Procedure B: Cu/CPA-catalyzed enantioselective desymmetrising radical oxy-perfluoroalkylation of olefinic 1,3-diols

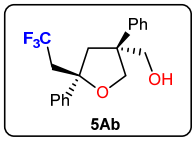


Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **4** (0.1 mmol, 1.0 eq.), CuBr (1.4 mg, 0.01 mmol, 10 mol%), chiral phosphoric acid (*R,R*)-**A9** (11.1 mg, 0.015 mmol, 15 mol%), **2b** (20.2 mg, 0.12 mmol, 1.2 eq.) or **2c** (38.2 mg, 0.12 mmol, 1.2 eq.), Ag_2CO_3 (16.5 mg, 0.06 mmol, 0.6 eq.) and CH_2Cl_2 (1.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. Upon completion (monitored by TLC), the solvent was removed *in vacuo*, and the residue was purified by a silica gel chromatography to afford the desired product **5**.

Note: Since the reaction is sensitive to water and air, the Schlenk tube and the reagents must be dried prior to use.



The racemate was prepared by following the same procedure as described above using substrate **1** (0.1 mmol, 1.0 eq.), **2b** or **2c** (0.12 mmol, 1.2 eq.), CuI (3.8 mg, 0.02 mmol, 20 mol%), diphenyl phosphate (5.0 mg, 0.02 mmol, 20 mol%), and Ag_2CO_3 (16.5 mg, 0.06 mmol, 0.6 eq.) at 25 °C in CH_2Cl_2 (1.0 mL). Upon completion (monitored by TLC), the solvent was removed *in vacuo*, and the residue was purified by silica gel column chromatography to afford the desired product **5** as a mixture of diastereomers (very similar Rf values).



((2*R*,4*S*)-2,4-diphenyl-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (5Ab)

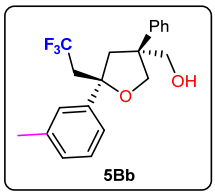
Product **5Ab** was obtained as a white solid (m.p. 101 °C) in 96% (32.3 mg, 0.10 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = –12 (*c* 0.9, $CHCl_3$).

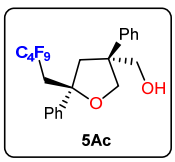
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min, λ = 214 nm), t_R (major) = 37.56 min, t_R (minor) = 50.38 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53–7.45 (m, 2H), 7.40–7.36 (m, 4H), 7.32–7.26 (m, 2H), 7.23–7.18 (m, 2H), 4.41 (d, *J* = 9.1 Hz, 1H), 4.26 (d, *J* = 9.0 Hz, 1H), 3.44 (d, *J* = 10.7 Hz, 1H), 3.35 (d, *J* = 10.8 Hz, 1H), 2.83 (d, *J* = 13.1 Hz, 1H), 2.68–2.57 (m, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 143.0, 128.8, 128.3, 127.2, 127.1, 127.0, 125.0, 124.99 (q, *J* = 276.8 Hz), 83.1 (q, *J* = 1.9 Hz), 73.2, 68.7, 54.0, 47.5, 45.6 (q, *J* = 25.8 Hz).
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, *J* = 10.5 Hz, 3F).
HRMS (ESI) calcd for $C_{19}H_{20}O_2F_3$ $[M+H]^+$ 337.1410, found: 337.1398.

Another product **5Ab** was obtained in 65% yield (determined by ^{1}H NMR using CH_2Br_2 as an internal standard) in a 0.025 mmol scale synthesis with moderate dr (12:1) and poor ee (32%) following general procedure A using the Togni's reagent **2a** as the •CF_3 radical precursor.



((2*R*,4*S*)-4-phenyl-2-(m-tolyl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran-4-yl)methanol (5Bb)
Product **5Bb** was obtained as a white solid (m.p. 104 °C) in 91% (31.8 mg, 0.09 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.
$[\alpha]_D^{27} = -10$ (*c* 0.7, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min, λ = 214 nm), t_R (major) = 28.03 min, t_R (minor) = 25.91 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (t, *J* = 6.9 Hz, 2H), 7.31–7.25 (m, 4H), 7.20 (d, *J* = 7.8 Hz, 2H), 7.08–7.04 (m, 1H), 4.42 (d, *J* = 9.0 Hz, 1H), 4.26 (d, *J* = 8.8 Hz, 1H), 3.45 (d, *J* = 10.7 Hz, 1H), 3.36 (d, *J* = 10.0 Hz, 1H), 2.80 (d, *J* = 13.1 Hz, 1H), 2.68–2.56 (m, 3H), 2.38 (s, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 143.0, 137.9, 128.8, 128.2, 127.9, 127.1, 127.0, 125.6, 125.0 (q, *J* = 277.0 Hz), 122.1, 83.1 (q, *J* = 1.9 Hz), 73.2, 68.7, 54.0, 47.5, 45.6 (q, *J* = 25.8 Hz), 21.6.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.3 Hz, 3F).
HRMS (ESI) calcd for $C_{20}H_{22}O_2F_3$ $[M+H]^+$ 351.1566, found: 351.1557.



((2*R*,4*S*)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2,4-diphenyltetrahydrofuran-4-yl)methanol (5Ac)

Product **5Ac** was obtained as a white solid (m.p. 130 °C) in 80% (38.9 mg, 0.08 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = –6 (*c* 0.3, $CHCl_3$).

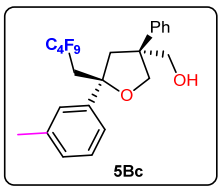
HPLC analysis: Chiralcel IC (*n*-Hexane/*i*-PrOH = 97/3, flow rate 0.4 mL/min, λ = 214 nm), t_R (major) = 19.83 min, t_R (minor) = 18.19 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.8 Hz, 2H), 7.37 (t, *J* = 7.7 Hz, 4H), 7.33–7.26 (m, 2H), 7.19 (d, *J* = 7.3 Hz, 2H), 4.41 (d, *J* = 9.0 Hz, 1H), 4.27 (d, *J* = 9.0 Hz, 1H), 3.42 (d, *J* = 10.8 Hz, 1H), 3.32 (d, *J* = 10.8 Hz, 1H), 2.88 (d, *J* = 13.1 Hz, 1H), 2.66–2.54 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 142.9, 128.8, 128.3, 127.2, 127.1, 127.0, 125.0, 121.1–106.3 (m), 83.7, 73.1, 68.6, 54.1, 47.9, 41.7 (t, *J* = 19.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -113.0 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{22}H_{20}O_2F_9$ $[M+H]^+$ 487.1314, found: 487.1306.



5Bc

((2*R*,4*S*)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyl-2-(m-tolyl)tetrahydrofuran-4-yl)methanol (5Bc)

Product **5Bc** was obtained as a white solid (m.p. 143 °C) in 75% (37.5 mg, 0.08 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = –18 (*c* 1.2, $CHCl_3$).

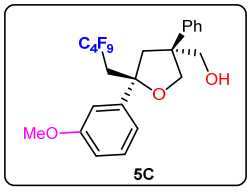
HPLC analysis: Chiralcel IB (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.4 mL/min, λ = 214 nm), t_R (major) = 25.92 min, t_R (minor) = 22.33 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.6 Hz, 2H), 7.33 (s, 1H), 7.31–7.23 (m, 3H), 7.21–7.18 (m, 2H), 7.09 (d, *J* = 7.0 Hz, 1H), 4.41 (d, *J* = 9.0 Hz, 1H), 4.27 (d, *J* = 9.0 Hz, 1H), 3.44 (d, *J* = 10.8 Hz, 1H), 3.34 (d, *J* = 10.8 Hz, 1H), 2.85 (d, *J* = 13.1 Hz, 1H), 2.67–2.52 (m, 3H), 2.38 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 145.3, 143.0, 137.9, 128.8, 128.2, 127.9, 127.1, 127.0, 125.6, 122.0, 120.7–106.3 (m), 83.8, 73.1, 68.6, 54.0, 47.9, 41.6 (t, *J* = 19.6 Hz), 21.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -113.0 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{23}H_{22}O_2F_9$ $[M+H]^+$ 501.1471, found: 501.1462.



5C

((2*R*,4*S*)-2-(3-methoxyphenyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyltetrahydrofuran-4-yl)methanol (5C)

Product **5C** was obtained as a white solid (m.p. 146 °C) in 82% (42.3 mg, 0.08 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −12 (*c* 0.8, $CHCl_3$).

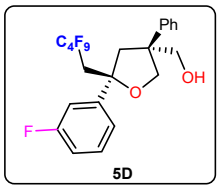
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 210 nm), t_R (major) = 15.96 min, t_R (minor) = 14.87 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (t, *J* = 7.6 Hz, 2H), 7.33–7.26 (m, 2H), 7.20 (d, *J* = 8.1 Hz, 2H), 7.12 (s, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.82 (d, *J* = 8.1 Hz, 1H), 4.41 (d, *J* = 9.0 Hz, 1H), 4.27 (d, *J* = 9.0 Hz, 1H), 3.83 (s, 3H), 3.45 (d, *J* = 10.8 Hz, 1H), 3.36 (d, *J* = 10.8 Hz, 1H), 2.87 (d, *J* = 13.1 Hz, 1H), 2.62–2.55 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 159.6, 147.0, 142.9, 129.4, 128.8, 127.1, 127.0, 121.0–106.3 (m), 117.4, 112.3, 111.1, 83.7, 73.2, 68.6, 55.2, 54.0, 47.9, 41.6 (t, *J* = 19.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.9 – -113.1 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{23}H_{22}O_3F_9$ $[M+H]^+$ 517.1420, found: 517.1408.



5D

((2*R*,4*S*)-2-(3-fluorophenyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyltetrahydrofuran-4-yl)methanol (5D)

Product **5D** was obtained as a white solid (m.p. 134 °C) in 81% (40.8 mg, 0.08 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −10 (*c* 0.5, $CHCl_3$).

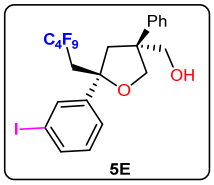
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 12.86 min, t_R (minor) = 10.72 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (t, *J* = 7.6 Hz, 2H), 7.36–7.23 (m, 4H), 7.19 (d, *J* = 7.2 Hz, 2H), 6.97 (td, *J* = 8.3, 1.9 Hz, 1H), 4.42 (d, *J* = 9.1 Hz, 1H), 4.27 (d, *J* = 9.1 Hz, 1H), 3.44 (d, *J* = 10.8 Hz, 1H), 3.34 (d, *J* = 10.8 Hz, 1H), 2.85 (d, *J* = 13.2 Hz, 1H), 2.62–2.54 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 162.8 (d, *J* = 245.7 Hz), 148.0 (d, *J* = 6.7 Hz), 142.7, 129.9 (d, *J* = 8.1 Hz), 128.8, 127.2, 126.9, 120.7 (d, *J* = 2.9 Hz), 114.2 (d, *J* = 21.2 Hz), 112.4 (d, *J* = 23.0 Hz), 121.1–106.3 (m), 83.3 (d, *J* = 2.1 Hz), 73.2, 68.5, 54.0, 48.1, 41.5 (t, *J* = 19.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -112.9 (m, 2F), -112.4 – -112.5 (m, 1F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{22}H_{19}O_2F_{10}$ $[M+H]^+$ 505.1220, found: 505.1214.



((2*R*,4*S*)-2-(3-iodophenyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyltetrahydrofuran-4-yl)methanol (5E)

Product **5E** was obtained as a white solid (m.p. 166 °C) in 72% (44.1 mg, 0.07 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −7 (*c* 0.3, $CHCl_3$).

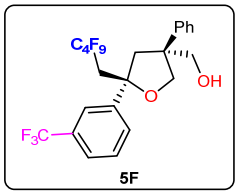
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.4 mL/min, λ = 240 nm), t_R (major) = 15.88 min, t_R (minor) = 13.89 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 7.9 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 2H), 7.11 (t, *J* = 7.8 Hz, 1H), 4.41 (d, *J* = 9.1 Hz, 1H), 4.26 (d, *J* = 9.0 Hz, 1H), 3.43 (d, *J* = 10.7 Hz, 1H), 3.34 (d, *J* = 10.8 Hz, 1H), 2.81 (d, *J* = 13.2 Hz, 1H), 2.68–2.46 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 147.6, 142.6, 136.4, 134.1, 130.0, 128.8, 127.2, 126.9, 124.4, 120.9–106.0 (m), 94.4, 83.1, 73.1, 68.5, 54.0, 48.0, 41.5 (t, *J* = 19.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.08 – -81.13 (m, 3F), -110.7 – -112.7 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{22}H_{19}O_2F_9I$ $[M+H]^+$ 613.0281, found: 613.0265.



((2*R*,4*S*)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyl-2-(3-(trifluoromethyl)phenyl)tetrahydrofuran-4-yl)methanol (5F)

Product **5F** was obtained as a white solid (m.p. 140 °C) in 55% (30.5 mg, 0.06 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −12 (*c* 0.8, $CHCl_3$).

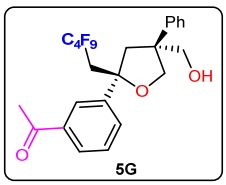
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 34.40 min, t_R (minor) = 27.45 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.70 (d, *J* = 7.7 Hz, 1H), 7.56 (d, *J* = 7.7 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.24–7.17 (m, 2H), 4.44 (d, *J* = 9.1 Hz, 1H), 4.30 (d, *J* = 9.1 Hz, 1H), 3.43 (d, *J* = 10.8 Hz, 1H), 3.33 (d, *J* = 10.8 Hz, 1H), 2.85 (d, *J* = 13.2 Hz, 1H), 2.70–2.51 (m, 3H).

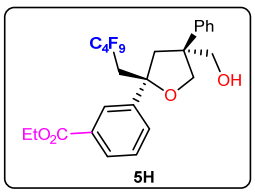
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.3, 142.4, 130.8 (q, *J* = 32.3 Hz), 128.9, 128.8, 128.5, 127.3, 127.0, 124.2 (q, *J* = 3.8 Hz), 124.1 (q, *J* = 270.8 Hz), 122.0 (q, *J* = 3.4 Hz), 120.9–106.0 (m), 83.4, 73.2, 68.6, 54.1, 48.2, 41.6 (t, *J* = 19.7 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.5 (s, 3F), -81.1 – -81.2 (m, 3F), -110.7 – -112.6 (m,

2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).
HRMS (ESI) calcd for $C_{23}H_{22}F_{12}NO_2$ $[M+NH_4]^+$ 572.1453, found: 572.1446.



1-(3-((2*R*,4*S*)-4-(hydroxymethyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyltetrahydrofuran-2-yl)phenyl)ethan-1-one (5G)
Product **5G** was obtained as a white solid (m.p. 98 °C) in 85% (44.9 mg, 0.09 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 2/1) following the general procedure.
$[\alpha]_D^{27}$ = –8 (*c* 0.8, $CHCl_3$).
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 240 nm), t_R (major) = 23.02 min, t_R (minor) = 14.90 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.87 (d, *J* = 7.7 Hz, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.23–7.17 (m, 2H), 4.45 (d, *J* = 9.1 Hz, 1H), 4.29 (d, *J* = 9.1 Hz, 1H), 3.42 (d, *J* = 10.8 Hz, 1H), 3.32 (d, *J* = 10.8 Hz, 1H), 2.88 (d, *J* = 13.2 Hz, 1H), 2.69–2.54 (m, 6H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 198.1, 146.0, 142.6, 137.1, 129.9, 128.8, 128.6, 127.5, 127.2, 127.0, 124.7, 120.9–106.0 (m), 83.5, 73.1, 68.5, 54.1, 48.3, 41.5 (t, *J* = 19.6 Hz), 26.7.
^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.7 – -112.6 (m, 2F), -124.62 – -124.64 (m, 2F), -125.86 – -125.92 (m, 2F).
HRMS (ESI) calcd for $C_{24}H_{22}O_3F_9$ $[M+H]^+$ 529.1420, found: 529.1408.



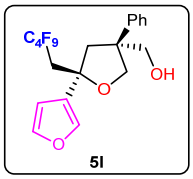
ethyl 3-((2*R*,4*S*)-4-(hydroxymethyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyltetrahydrofuran-2-yl)benzoate (5H)
Product **5H** was obtained as a white solid (m.p. 133 °C) in 59% (32.9 mg, 0.06 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 2/1) following the general procedure.
$[\alpha]_D^{27}$ = –12 (*c* 0.5, $CHCl_3$).
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 240 nm), t_R (major) = 32.88 min, t_R (minor) = 17.22 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.98 (d, *J* = 7.6 Hz, 1H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.3 Hz, 1H), 7.20 (d, *J* = 7.8 Hz, 2H), 4.45 (d, *J* = 9.1 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 4.29 (d, *J*

= 9.1 Hz, 1H), 3.42 (d, *J* = 10.8 Hz, 1H), 3.33 (d, *J* = 10.8 Hz, 1H), 2.88 (d, *J* = 13.2 Hz, 1H), 2.69–2.56 (m, 3H), 1.42 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 166.5, 145.7, 142.6, 130.6, 129.5, 128.8, 128.5, 128.4, 127.2, 127.0, 126.2, 120.9–106.0 (m), 83.5, 73.1, 68.6, 61.1, 54.1, 48.3, 41.5 (t, *J* = 19.6 Hz), 14.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -112.6 (m, 2F), -124.61 – -124.63 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{25}H_{24}O_4F_9$ $[M+H]^+$ 559.1525, found: 59.1516.



5I

((2*R*,4*S*)-2-(furan-3-yl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-4-phenyltetrahydrofuran-4-yl)methanol (5I)

Product **5I** was obtained as a white solid (m.p. 115 °C) in 46% (21.9 mg, 0.05 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = –13 (*c* 0.5, $CHCl_3$).

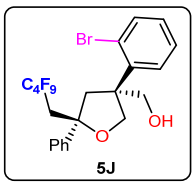
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 210 nm), t_R (major) = 23.29 min, t_R (minor) = 13.97 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (s, 1H), 7.41 (t, *J* = 1.6 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 2H), 6.42 (s, 1H), 4.35 (d, *J* = 9.0 Hz, 1H), 4.15 (d, *J* = 9.0 Hz, 1H), 3.60 (d, *J* = 10.8 Hz, 1H), 3.54 (d, *J* = 10.8 Hz, 1H), 2.82 (d, *J* = 13.2 Hz, 1H), 2.67–2.44 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 143.5, 142.9, 138.6, 130.5, 128.8, 127.2, 126.9, 120.7–106.4 (m), 108.7, 79.8, 73.0, 68.6, 54.5, 46.4, 41.6 (t, *J* = 19.7 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.07 – -81.12 (m, 3F), -110.6 – -113.4 (m, 2F), -124.7 – -124.8 (m, 2F), -125.8 – -126.0 (m, 2F).

HRMS (ESI) calcd for $C_{20}H_{18}O_3F_9$ $[M+H]^+$ 477.1107, found: 477.1104.



5J

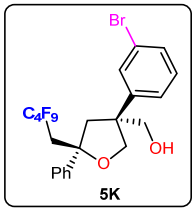
((2*R*,4*S*)-4-(2-bromophenyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyltetrahydrofuran-4-yl)methanol (5J)

Product **5J** was obtained as a white solid (m.p. 115 °C) in 88% (49.7 mg, 0.09 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

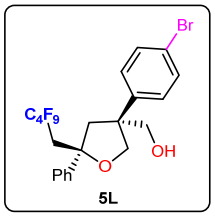
$[\alpha]_D^{27}$ = –10 (*c* 0.7, $CHCl_3$).

HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ =

230 nm), t_R (major) = 13.27 min, t_R (minor) = 11.27 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.1 Hz, 1H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.30 (dt, *J* = 15.5, 7.2 Hz, 2H), 7.20–7.11 (m, 2H), 4.69 (d, *J* = 9.2 Hz, 1H), 4.34 (d, *J* = 9.2 Hz, 1H), 3.64–3.52 (m, 2H), 3.17 (d, *J* = 13.3 Hz, 1H), 2.73–2.57 (m, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 141.0, 135.1, 131.0, 128.8, 128.3, 127.3, 127.2, 124.9, 122.2, 121.0–106.3 (m), 83.4, 72.9, 64.5, 55.1, 48.3, 41.2 (t, *J* = 19.6 Hz).
^{19}F NMR (376 MHz, $CDCl_3$) δ -81.3 – -81.4 (m, 3F), -111.1 – -113.1 (m, 2F), -124.8 (s, 2F), -126.0 – -126.1 (m, 2F).
HRMS (ESI) calcd for $C_{22}H_{19}O_2BrF_9$ $[M+H]^+$ 565.0419, found: 565.0407.

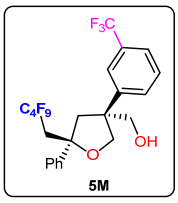


((2*R*,4*S*)-4-(3-bromophenyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyltetrahydrofuran-4-yl)methanol (5K)
Product **5K** was obtained as a white solid (m.p. 115 °C) in 73% (41.2 mg, 0.07 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.
$[\alpha]_D^{27}$ = –20 (*c* 1.2, $CHCl_3$).
HPLC analysis: Chiralcel IC (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 18.97 min, t_R (minor) = 16.41 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 7.6 Hz, 2H), 7.45–7.32 (m, 4H), 7.31–7.22 (m, 2H), 7.13 (d, *J* = 7.8 Hz, 1H), 4.37 (d, *J* = 9.1 Hz, 1H), 4.22 (d, *J* = 9.1 Hz, 1H), 3.41 (d, *J* = 10.8 Hz, 1H), 3.32 (d, *J* = 10.8 Hz, 1H), 2.88 (d, *J* = 13.2 Hz, 1H), 2.65–2.55 (m, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.5, 145.0, 130.2, 128.4, 127.3, 125.6, 125.0, 122.9, 120.8–106.6 (m), 83.7, 72.9, 68.2, 53.9, 47.7, 41.7 (t, *J* = 19.6 Hz).
^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -113.0 (m, 2F), -124.56 – -124.64 (m, 2F), -125.8 – -125.9 (m, 2F).
HRMS (ESI) calcd for $C_{22}H_{19}O_2BrF_9$ $[M+H]^+$ 565.0419, found: 565.0406.



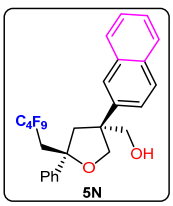
((2*R*,4*S*)-4-(4-bromophenyl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyltetrahydrofuran-4-yl)methanol (5L)
Product **5L** was obtained as a white solid (m.p. 118 °C) in 86% (48.6 mg, 0.09 mmol)

yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.
$[\alpha]_D^{27}$ = –18 (*c* 1.3, $CHCl_3$).
HPLC analysis: Chiralcel IC (*n*-Hexane/*i*-PrOH = 97/3, flow rate 0.4 mL/min, λ = 214 nm), t_R (major) = 21.23 min, t_R (minor) = 16.21 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50–7.48 (m, 4H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.28 (t, *J* = 7.3 Hz, 1H), 7.07 (d, *J* = 8.4 Hz, 2H), 4.35 (d, *J* = 9.0 Hz, 1H), 4.20 (d, *J* = 9.0 Hz, 1H), 3.38 (d, *J* = 10.5 Hz, 1H), 3.29 (d, *J* = 10.6 Hz, 1H), 2.87 (d, *J* = 13.1 Hz, 1H), 2.69–2.50 (m, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.0, 142.2, 131.8, 131.7, 128.7, 128.4, 127.3, 124.9, 121.0, 120.8–106.3 (m), 83.8, 73.0, 68.1, 53.7, 47.7, 41.7 (t, *J* = 19.5 Hz).
^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.7 – -113.0 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).
HRMS (ESI) calcd for $C_{22}H_{19}O_2BrF_9$ $[M+H]^+$ 565.0419, found: 565.0407.



5M

((2*R*,4*S*)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyl-4-(3-(trifluoromethyl)phenyl)tetrahydrofuran-4-yl)methanol (5M)
Product **5M** was obtained as a white solid (m.p. 120 °C) in 80% (44.3 mg, 0.08 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.
$[\alpha]_D^{27}$ = –10 (*c* 0.6, $CHCl_3$).
HPLC analysis: Chiralcel AD3 (*n*-Hexane/*i*-PrOH = 97/3, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 29.91 min, t_R (minor) = 17.47 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.8 Hz, 1H), 7.54–7.47 (m, 3H), 7.45 (s, 1H), 7.42–7.36 (m, 3H), 7.32–7.27 (m, 1H), 4.41 (d, *J* = 9.1 Hz, 1H), 4.26 (d, *J* = 9.1 Hz, 1H), 3.46 (d, *J* = 10.8 Hz, 1H), 3.37 (d, *J* = 10.8 Hz, 1H), 2.94 (d, *J* = 13.1 Hz, 1H), 2.68–2.51 (m, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.0, 144.3, 131.0 (q, *J* = 32.2 Hz), 130.5, 129.1, 128.4, 127.4, 125.0, 124.0 (q, *J* = 270.9 Hz), 123.9 (q, *J* = 3.8 Hz), 123.7 (q, *J* = 3.8 Hz), 120.9–106.3 (m), 83.8, 73.1, 68.0, 54.0, 47.7, 41.8 (t, *J* = 19.6 Hz).
^{19}F NMR (376 MHz, $CDCl_3$) δ -62.6 (s, 3F), -81.1 – -81.2 (m, 3F), -110.8 – -113.0 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).
HRMS (ESI) calcd for $C_{23}H_{19}O_2F_{12}$ $[M+H]^+$ 555.1188, found: 555.1185.



5N

((2*R*,4*S*)-4-(naphthalen-2-yl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyltetrahydrofuran-4-yl)methanol (5N)

Product **5N** was obtained as a white solid (m.p. 110 °C) in 64% (34.3 mg, 0.06 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −12 (*c* 0.5, $CHCl_3$).

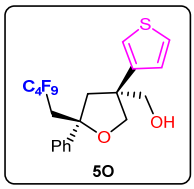
HPLC analysis: Chiralcel AD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 22.45 min, t_R (minor) = 12.71 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88–7.80 (m, 3H), 7.64 (d, *J* = 1.4 Hz, 1H), 7.58–7.48 (m, 4H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.33–7.27 (m, 2H), 4.51 (d, *J* = 9.0 Hz, 1H), 4.39 (d, *J* = 9.0 Hz, 1H), 3.50 (d, *J* = 10.9 Hz, 1H), 3.41 (d, *J* = 10.9 Hz, 1H), 2.97 (d, *J* = 13.1 Hz, 1H), 2.73 (d, *J* = 13.1 Hz, 1H), 2.68–2.56 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 140.2, 133.1, 132.3, 128.7, 128.4, 127.8, 127.5, 127.3, 126.5, 126.1, 125.7, 125.0, 119.7–107.8 (m), 83.8, 73.2, 68.4, 54.2, 48.0, 41.7 (t, *J* = 19.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -112.9 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{26}H_{25}O_2NF_9$ $[M+NH_4]^+$ 554.1736, found: 554.1730.



5O

((2*R*,4*S*)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyl-4-(thiophen-3-yl)tetrahydrofuran-4-yl)methanol (5O)

Product **5O** was obtained as a white solid (m.p. 117 °C) in 93% (45.7 mg, 0.09 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −8 (*c* 0.6, $CHCl_3$).

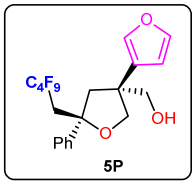
HPLC analysis: Chiralcel OX3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 240 nm), t_R (major) = 30.95 min, t_R (minor) = 32.71 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.6 Hz, 2H), 7.40–7.33 (m, 3H), 7.28 (t, *J* = 7.3 Hz, 1H), 7.15–7.08 (m, 1H), 7.05–6.99 (m, 1H), 4.30–4.22 (m, 2H), 3.44 (d, *J* = 10.8 Hz, 1H), 3.40 (d, *J* = 10.8 Hz, 1H), 2.79 (d, *J* = 13.2 Hz, 1H), 2.66–2.52 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 143.8, 128.3, 127.3, 126.8, 126.3, 125.0, 120.9, 120.8–106.3 (m), 83.8, 74.0, 68.0, 52.1, 48.7, 41.6 (t, *J* = 19.5 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.8 – -113.0 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

HRMS (ESI) calcd for $C_{20}H_{18}O_2F_9S$ $[M+H]^+$ 493.0878, found: 493.0868.



5P

((2*R*,4*S*)-4-(furan-3-yl)-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-2-phenyltetrahydrofuran-4-yl)methanol (5P)

Product **5P** was obtained as a white solid (m.p. 115 °C) in 53% (25.2 mg, 0.05 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.

$[\alpha]_D^{27}$ = −15 (*c* 0.5, $CHCl_3$).

HPLC analysis: Chiralcel ODH (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min, λ = 214 nm), t_R (major) = 26.91 min, t_R (minor) = 37.68 min.

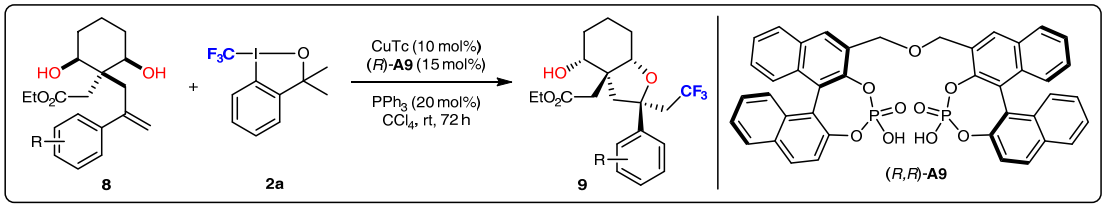
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49–7.44 (m, 3H), 7.42–7.35 (m, 3H), 7.30–7.26 (m, 1H), 6.36 (dd, *J* = 1.8, 1.0 Hz, 1H), 4.17–4.10 (m, 2H), 3.43 (d, *J* = 10.9 Hz, 1H), 3.39 (d, *J* = 10.7 Hz, 1H), 2.71 (d, *J* = 13.3 Hz, 1H), 2.66–2.56 (m, 2H), 2.51 (d, *J* = 13.2 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 144.0, 139.0, 128.4, 127.6, 127.3, 125.0, 120.7–106.4 (m), 109.1, 83.9, 74.1, 67.5, 48.5, 48.4, 41.6 (t, *J* = 19.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 – -81.2 (m, 3F), -110.7 – -113.0 (m, 2F), -124.6 – -124.7 (m, 2F), -125.8 – -125.9 (m, 2F).

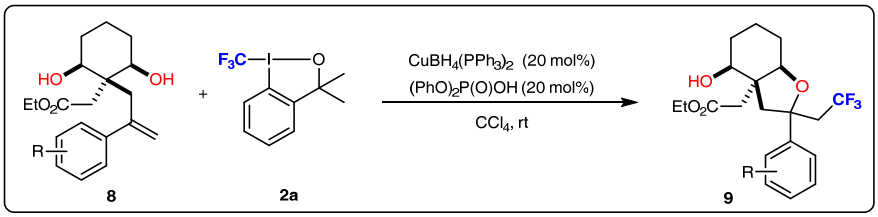
HRMS (ESI) calcd for $C_{20}H_{18}O_3F_9$ $[M+H]^+$ 477.1107, found: 477.1102.

3.5 General Procedure C: Cu/CPA-catalyzed enantioselective desymmetrising radical oxytrifluoromethylation of *meso* olefinic 1,3-diols

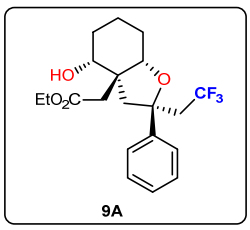


Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **8** (0.1 mmol, 1.0 eq.), CuTc (1.9 mg, 0.01 mmol, 10 mol%), chiral phosphoric acid (*R,R*)-**A9** (11.1 mg, 0.015 mmol, 15 mol%), **2a** (49.5 mg, 0.15 mmol, 1.5 eq.), PPh_3 (5.2 mg, 0.02 mmol, 20 mol%) and CCl_4 (1.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. Upon completion (monitored by TLC), the solvent was removed *in vacuo*, and the residue was purified by a silica gel chromatography to afford the desired product **9**.

Note: Since the reaction is sensitive to water and air, the Schlenk tube and the reagents must be dried prior to use.



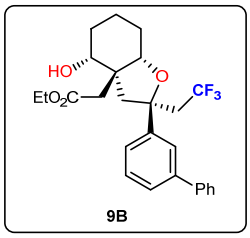
The racemate was prepared by following the same procedure as described above using substrate **8** (0.1 mmol, 1.0 eq.), **2a** (0.15 mmol, 1.5 eq.), $CuBH_4(PPh_3)_2$ (12.0 mg, 0.02 mmol, 20 mol%), and diphenyl phosphate (5.0 mg, 0.02 mmol, 20 mol%) at 25 °C in CCl_4 (1.0 mL). Upon completion (monitored by TLC), the solvent was removed *in vacuo*, and the residue was purified by silica gel column chromatography to afford the desired product **9** as a mixture of diastereomers (very similar Rf values).



ethyl 2-(4-hydroxy-2-phenyl-2-(2,2,2-trifluoroethyl)hexahydrobenzofuran-3a(4H)-yl)acetate (9A)

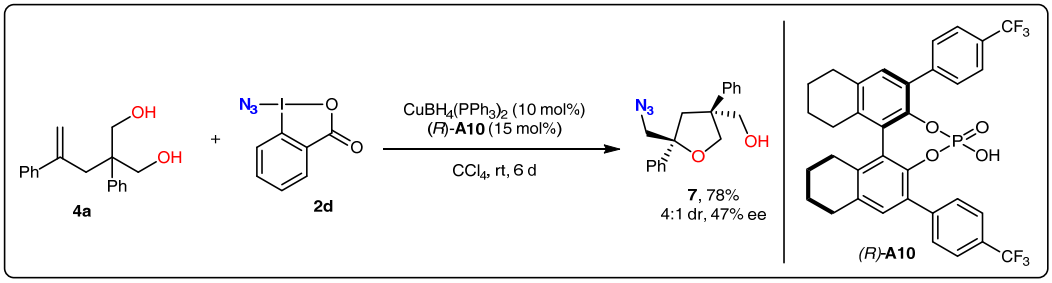
Product **9A** was obtained as a white solid in 80% (30.9 mg, 0.08 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general

procedure.
$[\alpha]_D^{27}$ = +20 (*c* 0.7, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 18.24 min, t_R (minor) = 21.51 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.8 Hz, 2H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 1H), 4.28–4.15 (m, 2H), 3.99–3.96 (m, 1H), 3.73–3.69 (m, 1H), 3.19 (brs, 1H), 2.85–2.79 (m, 2H), 2.75 (d, *J* = 13.8 Hz, 1H), 2.69–2.65 (m, 1H), 2.60 (d, *J* = 15.0 Hz, 1H), 2.46 (d, *J* = 14.0 Hz, 1H), 1.68–1.58 (m, 3H), 1.48–1.36 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.25–1.18 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 146.2, 128.0, 127.0, 125.1, 125.0 (q, *J* = 276.8 Hz), 82.6, 81.6 (q, *J* = 2.1 Hz), 73.9, 61.3, 49.7, 46.6 (q, *J* = 25.4 Hz), 44.1, 43.4, 28.9, 27.0, 16.9, 14.1.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.4 (t, *J* = 10.5 Hz, 3F).
HRMS (ESI) calcd for $C_{20}H_{26}F_3O_4$ $[M+H]^+$ 387.1778, found: 387.1778.



ethyl 2-(2-([1,1'-biphenyl]-3-yl)-4-hydroxy-2-(2,2,2-trifluoroethyl)hexahydrobenzofuran-3a(4H)-yl)acetate (9B)
Product **9B** was obtained as a white solid in 71% (32.8 mg, 0.07 mmol) yield in a 0.10 mmol scale synthesis by column chromatography (PE/EA = 6/1) following the general procedure.
$[\alpha]_D^{27}$ = +7 (*c* 0.2, $CHCl_3$).
HPLC analysis: Chiralcel IG (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.4 mL/min, λ = 254 nm), t_R (major) = 18.09 min, t_R (minor) = 19.79 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.60–7.57 (m, 2H), 7.50–7.38 (m, 5H), 7.37–7.32 (m, 1H), 4.27–4.17 (m, 2H), 4.00 (dd, *J* = 7.6, 4.7 Hz, 1H), 3.73 (dd, *J* = 8.9, 3.9 Hz, 1H), 3.21 (brs, 1H), 2.85 (d, *J* = 15.0 Hz, 1H), 2.80 (d, *J* = 14.0 Hz, 1H), 2.74–2.66 (m, 1H), 2.61 (d, *J* = 15.0 Hz, 1H), 2.50 (d, *J* = 13.9 Hz, 1H), 1.72–1.59 (m, 3H), 1.52–1.35 (m, 2H), 1.32 (t, *J* = 7.2 Hz, 3H), 1.26–1.18 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 173.2, 146.9, 141.1, 140.9, 128.7, 128.4, 127.3, 127.2, 125.8, 125.0 (q, *J* = 276.8 Hz), 124.1, 124.0, 82.8, 81.7 (q, *J* = 1.9 Hz), 74.1, 61.3, 49.9, 46.7 (q, *J* = 25.4 Hz), 43.9, 43.5, 28.9, 27.2, 17.1, 14.1.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.3 (t, *J* = 10.5 Hz, 3F).
HRMS (ESI) calcd for $C_{26}H_{30}F_3O_4$ $[M+H]^+$ 463.2091, found: 463.2091.

3.6 Procedure for the Synthesis of 7



Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **4a** (0.1 mmol, 1.0 eq.), $CuBH_4(PPh_3)_2$ (6.0 mg, 0.01 mmol, 10 mol%), chiral phosphoric acid (*R*)-**A10** (9.7 mg, 0.015 mmol, 15 mol%), **2d** (43.4 mg, 0.15 mmol, 1.5 eq.), and CCl_4 (1.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. After 6 d, the solvent was removed *in vacuo*, and the residue was purified by a silica gel chromatography to afford the desired product **7** (24.1 mg) as two separable diastereomers and the data of the major diastereomer (a colorless oil) was shown.

$[\alpha]_D^{27} = -6$ (*c* 0.7, $CHCl_3$).

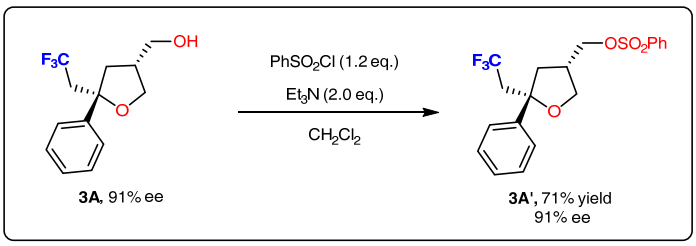
HPLC analysis: Chiralcel OD-H (*n*-Hexane/*i*-PrOH = 94/6, flow rate 0.8 mL/min, λ = 214 nm), t_R (major) = 30.96 min, t_R (minor) = 27.46 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.44 (m, 2H), 7.41–7.32 (m, 4H), 7.31–7.24 (m, 2H), 7.21–7.14 (m, 2H), 4.45 (d, *J* = 8.7 Hz, 1H), 4.24 (d, *J* = 8.7 Hz, 1H), 3.44–3.25 (m, 4H), 2.70 (d, *J* = 12.8 Hz, 1H), 2.61 (d, *J* = 12.8 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.3, 142.8, 128.7, 128.4, 127.4, 127.0, 126.98, 125.0, 86.8, 73.6, 68.3, 60.3, 54.4, 43.1.

HRMS (ESI) calcd for $C_{18}H_{20}NO_2$ $[M+H-N_2]^+$ 282.1489, found: 282.1486.

3.7 Transformation and Determination of Absolute Configurations



To a solution of **3A** (26 mg, 0.1 mmol, 1.0 eq.) and Et_3N (28 μL, 0.2 mmol, 2.0 eq.) in DCM (2 mL) was added $PhSO_2Cl$ (15 μL, 0.12 mmol, 1.2 eq.). The mixture was stirred for 2 d at rt. Saturated $NaHCO_3$ (aq.) was added to quench the reaction. The aqueous layer was extracted with EtOAc (3x) and the combined organic layer was dried over Na_2SO_4. The solvent was removed *in vacuo*, and the residue was purified by silica gel chromatography to afford the desired product **3A'** (28.4 mg) as a white solid (m.p. 135 °C).

$[\alpha]_D^{27} = -13$ (*c* 0.5, $CHCl_3$).

HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 31.39 min, t_R (minor) = 18.00 min.

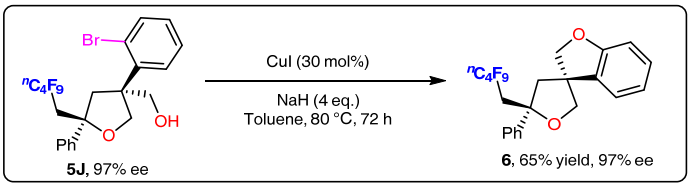
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91–7.89 (m, 2H), 7.70–7.66 (m, 1H), 7.59–7.55 (m, 2H), 7.37–7.32 (m, 4H), 7.29–7.25 (m, 1H), 4.07–3.99 (m, 2H), 3.89 (dd, *J* = 9.0, 8.0 Hz, 1H), 3.66 (dd, *J* = 9.0, 6.7 Hz, 1H), 2.69–2.55 (m, 3H), 2.53–2.41 (m, 1H), 1.87 (dd, *J* = 12.3, 9.3 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8, 135.7, 134.0, 129.4, 128.5, 127.8, 127.6, 125.2, 125.1 (q, *J* = 276.5 Hz), 83.5 (q, *J* = 1.9 Hz), 71.3, 68.8, 45.4 (q, *J* = 26.7 Hz), 40.6, 38.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.5 (t, *J* = 10.8 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{19}H_{20}F_3O_4S$ $[M+H]^+$ 401.1029, found 401.1024.

The absolute configuration of **3A** was assigned as 2*S*,4*R* by comparing with that of its sulfonylated derivative **3A'**. The absolute configuration of **3A'** was determined as 2*S*,4*S* through an X-ray experiment (see Supplementary Figure 1). The absolute configurations of **3B**–**3U** were assigned by analogy.

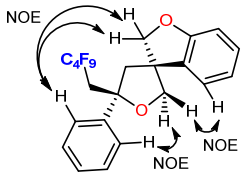


6 was synthesized according to the procedure previously reported.[4]

To a solution of **5J** (44 mg, 0.08 mmol) in toluene (2 mL) were added NaH (60% dispensed in mineral oil, ca. 13 mg, 0.32 mmol, 4.0 eq.) and CuI (ca. 5.0 mg, 0.026 mmol, 30 mol%). The solution was heated to 80 °C for 72 h. The solvent was removed under reduced pressure. The residue was purified by chromatography to give **6** (24.5 mg) as a white solid (m.p. 82 °C).

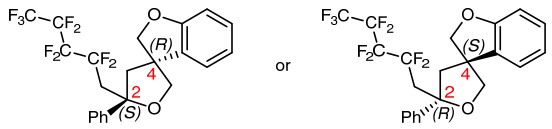
$[\alpha]_D^{27}$ = –23 (*c* 0.8, $CHCl_3$).
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 43.15 min, t_R (minor) = 51.19 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.1 Hz, 2H), 7.39 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.25 (d, *J* = 6.8 Hz, 1H), 7.16 (t, *J* = 7.7 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 4.19 (d, *J* = 9.1 Hz, 1H), 4.10 (d, *J* = 9.1 Hz, 1H), 3.97 (d, *J* = 9.5 Hz, 1H), 3.78 (d, *J* = 9.5 Hz, 1H), 2.90–2.60 (m, 4H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 143.6, 129.6, 129.1, 128.5, 127.7, 125.3, 122.7, 121.1, 109.8, 120.0–106.1 (m), 84.7, 82.8, 79.1, 53.3, 52.5, 41.7 (t, *J* = 19.8 Hz).
^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 (t, *J* = 9.9 Hz, 3F), -110.1 – -113.0 (m, 2F), -124.4 – -124.5 (m, 2F), -125.7 – -125.8 (m, 2F).
HRMS (ESI) calcd for $C_{22}H_{18}O_2F_9$ $[M+H]^+$ 485.1158, found: 485.1148.

The relative configuration of **6** was determined by 2D-NMR (Supplementary Figure 10).



Supplementary Figure 10. The NOE of compound 6

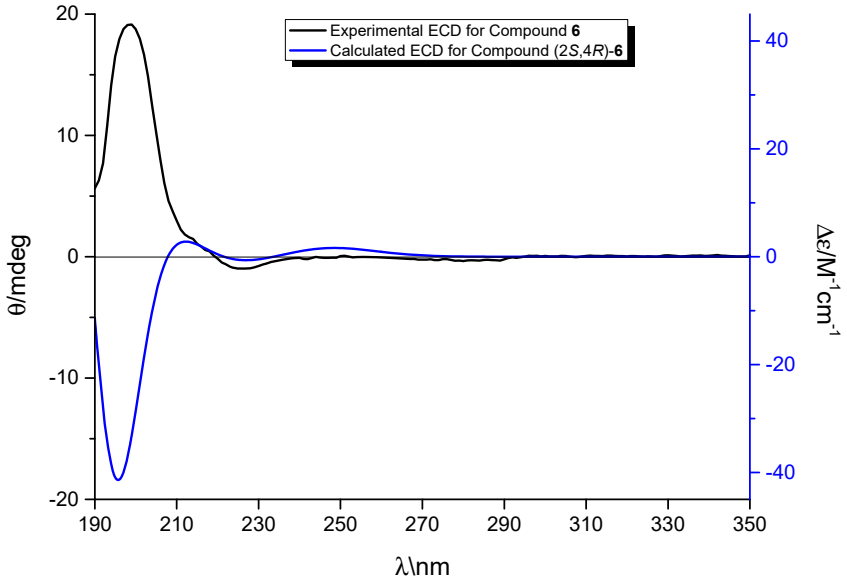
The absolute configuration of **6** was determined by ECD



(2*S*,4*R*)-**6** or (2*R*,4*S*)-**6**

Conformations of (2*S*,4*R*)-**6** was searched using Conflex 7 (Rev. D). Initial low-energy conformers with larger than 1% distribution in Gibb's free energy were chosen for geometry optimization and frequency calculation at the M062X/6-311++G(d,p) level in acetonitrile solvent by a self-consistent reaction field (SCRF) using the SMD implicit solvent model with Gaussian 16.[5] Then, conformers with distinct conformations were further subjected to TD-DFT calculation at the same level as aforementioned for dipole and rotational strengths of the first 40 excited states in the UV range. Next, ECD spectra of (2*S*,4*R*)-**6** (Supplementary Figure 11) was calculated from excitation energies and rotational strengths as averages weighed on Boltzmann conformer relative populations as a sum of Gaussian functions centered at the wavelength of each transition with appropriate widths of the band at half-height using SpecDis (version 1.71), respectively.
Samples of compounds **6** for ECD were dissolved in MeCN, and spectra were acquired in a 1.0-mm pathlength cuvette, respectively. The UV and ECD spectra were recorded using a Chirascan® Spectrophotometer with the following instrumental parameters: 185–350 nm with a 1 nm step and a 2 nm bandwidth with data averaging

over 1.0 sec per point. Three spectral acquisitions were taken for each sample and were averaged and smoothed thereafter.



Supplementary Figure 11. **Comparison of the calculated ECD of compound (2*S*,4*R*)-6 with the experimental one of compound 6.** Width of the band at half-height σ: 0.3 eV; Shift: 2 nm.

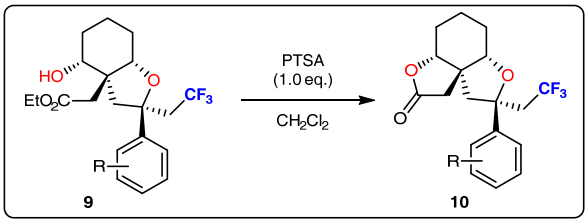
The calculated spectrum for (2*S*,4*R*)-**6** was opposite to the experimental one, and thus, the absolute configuration of compound **6** was assigned to 2*R* and 4*S*, accordingly. The absolute configurations of products **5** and **7** were assigned by analogue to that of **6**.

The relative and absolute configuration of **6** were further confirmed by X-ray crystallographic analysis (Supplementary Figure 2).

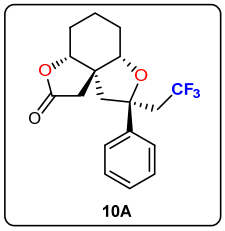
Supplementary Table 5. Energies of calculated conformers of (2*S*,4*R*)-6. Energies (*E*) (in Hartree) of the structures calculated at the M062X/6-311++G(d,p)-SMD(Acetonitrile) level of theory.

Structure	*E*
Conformer 1	−1897.5029516
Conformer 2	−1897.5006310
Conformer 3	−1897.5014146
Conformer 4	−1897.5007358
Conformer 5	−1897.5005500
Conformer 6	−1897.5012383
Conformer 7	−1897.501049
Conformer 8	−1897.5003922

Conformer 9	−1897.501967
Conformer 10	−1897.4991605
Conformer 11	−1897.4999599

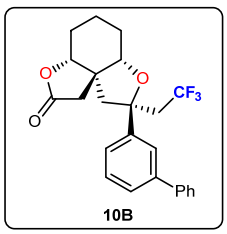


10 was synthesized according to the procedure previously reported.[6]
To a solution of **9** (1.0 eq.) in CH_2Cl_2 (0.05 M), PTSA (1.0 eq.) was added. The solution stirred at rt for 24 h. The solvent was removed under reduced pressure. The residue was purified by chromatography to give **10** as separable diastereomers.



8-phenyl-8-(2,2,2-trifluoroethyl)hexahydro-5H-benzo[2,1-b:6,1-b']difuran-2(1H)-one (10A)
Product **10A** was obtained as a white solid in 63% (17.2 mg, 0.051 mmol) yield by column chromatography (PE/EA = 6/1) following the general procedure.

HPLC analysis: Chiralcel AZ3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 49.45 min, t_R (minor) = 36.97 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.32 (m, 4H), 7.30–7.27 (m, 1H), 4.49 (t, J = 5.4 Hz, 1H), 4.23 (dd, J = 12.0, 5.8 Hz, 1H), 2.81 (d, J = 15.9 Hz, 1H), 2.78–2.65 (m, 3H), 2.62 (d, J = 13.6 Hz, 1H), 2.22 (d, J = 13.6 Hz, 1H), 2.06–1.98 (m, 1H), 1.79–1.73 (m, 1H), 1.64–1.58 (m, 2H), 1.46–1.38 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 174.7, 145.8, 128.2, 127.3, 124.9, 124.8 (q, J = 276.8 Hz), 83.4 (q, J = 2.1 Hz), 81.6, 81.5, 52.3, 46.3 (q, J = 26.1 Hz), 43.3, 28.2, 20.8, 17.7.
^{19}F NMR (376 MHz, $CDCl_3$) δ -60.2 (t, J = 10.3 Hz, 3F).
HRMS (ESI) calcd for $C_{18}H_{20}F_3O_3$ $[M+H]^+$ 341.1359, found: 341.1365.



8-([1,1'-biphenyl]-3-yl)-8-(2,2,2-trifluoroethyl)hexahydro-5H-benzo[2,1-b:6,1-b'] difuran-2(1H)-one (10B)

Product **10B** was obtained as a white solid in 75% (22.1 mg, 0.053 mmol) yield by column chromatography (PE/EA = 6/1) following the general procedure.

HPLC analysis: Chiralcel ID (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 29.38 min, t_R (minor) = 21.65 min.

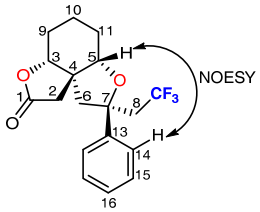
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (s, 1H), 7.59 (d, *J* = 7.6 Hz, 2H), 7.52 (d, *J* = 7.6 Hz, 1H), 7.47–7.41 (m, 3H), 7.36 (t, *J* = 8.6 Hz, 2H), 4.52–4.50 (m, 1H), 4.24 (dd, *J* = 11.6, 5.7 Hz, 1H), 2.83 (d, *J* = 16.0 Hz, 1H), 2.80–2.71 (m, 3H), 2.68 (d, *J* = 13.6 Hz, 1H), 2.27 (d, *J* = 13.6 Hz, 1H), 2.07–2.01 (m, 1H), 1.81–1.76 (m, 1H), 1.67–1.63 (m, 2H), 1.49–1.39 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 174.7, 146.4, 141.2, 140.9, 128.8, 128.7, 127.5, 127.2, 126.2, 123.9, 123.7, 83.5 (q, *J* = 1.8 Hz), 81.7, 81.6, 52.4, 46.3 (q, *J* = 25.8 Hz), 44.8, 43.3, 28.2, 20.9, 17.8.

^{19}F NMR (376 MHz, $CDCl_3$) δ -60.1 (t, *J* = 10.2 Hz, 3F).

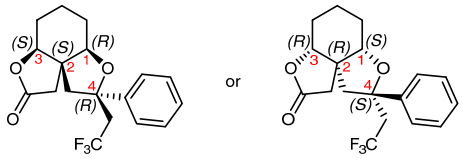
HRMS (ESI) calcd for $C_{24}H_{24}F_3O_3$ $[M+H]^+$ 417.1672, found: 417.1670.

The relative configuration of **10A** was determined by 2D-NMR (Supplementary Figure 12).



Supplementary Figure 12. The NOE of compound 10A

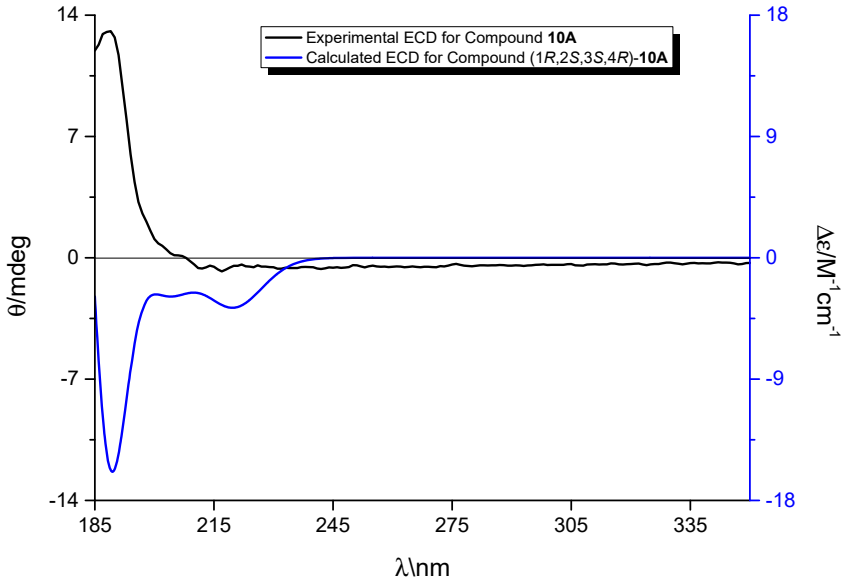
The absolute configuration of **10A** was determined by ECD



(1*R*,2*S*,3*S*,4*R*)-**10A** (1*S*,2*R*,3*R*,4*S*)-**10A**

Conformations of (1*R*,2*S*,3*S*,4*R*)-**10A** was searched using Conflex 7 (Rev. D). Initial low-energy conformers with larger than 1% distribution in Gibb's free energy were chosen for geometry optimization and frequency calculation at the M062X/6-311++G(d,p) level in acetonitrile solvent by a self-consistent reaction field (SCRF) using the SMD implicit solvent model with Gaussian 16.[5] Then, conformers with distinct conformations were further subjected to TD-DFT calculation at the same level as aforementioned for dipole and rotational strengths of the first 40 excited states in the UV range. Next, ECD spectra of (1*R*,2*S*,3*S*,4*R*)-**10A** (Supplementary Figure 13) were calculated from excitation energies and rotational strengths as averages weighed on Boltzmann conformer relative populations as a sum of Gaussian functions centered at the wavelength of each transition with appropriate widths of the

band at half-height using SpecDis (version 1.71), respectively.
Samples of compounds **10A** for ECD were dissolved in MeCN, and spectra were acquired in a 1.0-mm pathlength cuvette, respectively. The UV and ECD spectra were recorded using a Chirascan® Spectrophotometer with the following instrumental parameters: 185–350 nm with a 1 nm step and a 2 nm bandwidth with data averaging over 1.0 sec per point. Three spectral acquisitions were taken for each sample and were averaged and smoothed thereafter.



Supplementary Figure 13. Comparison of the calculated ECD of compound (1*R*,2*S*,3*S*,4*R*)-10A with the experimental one of compound 10A. Width of the band at half-height σ: 0.25 eV; Shift: 0 nm.

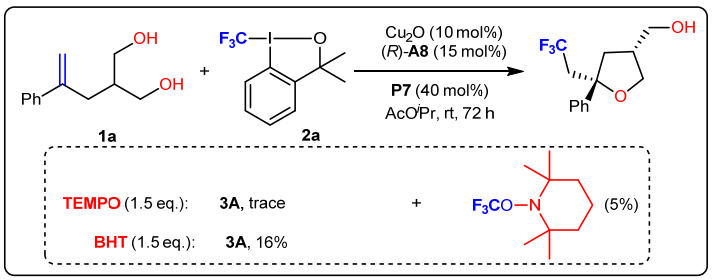
The calculated spectrum for (1*R*,2*S*,3*S*,4*R*)-**10A** was opposite to the experimental one, and thus, the absolute configuration of compound **10A** was assigned to 1*S*,2*R*,3*R*,4*S* accordingly. The absolute configurations of products **9** and **10B** were assigned by analogue to that of **10A**.

Supplementary Table 6. Energies of calculated conformers of (1*R*,2*S*,3*S*,4*R*)-10A. Energies (*E*) (in Hartree) of the structures calculated at the M062X/6-311++G(d,p)-SMD(Acetonitrile) level of theory.

Structure	*E*
Conformer 1	−1222.4993174
Conformer 2	−1222.5008069
Conformer 3	−1222.5011999
Conformer 4	−1222.5029266

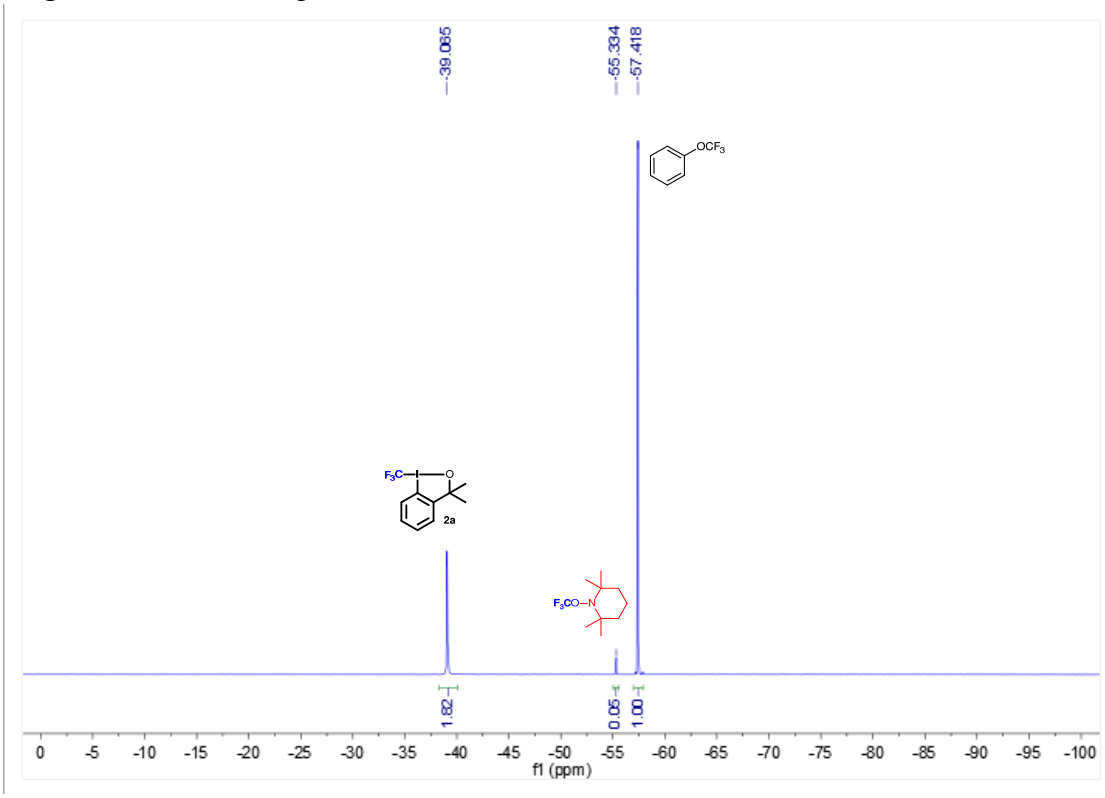
3.8 Mechanistic Study

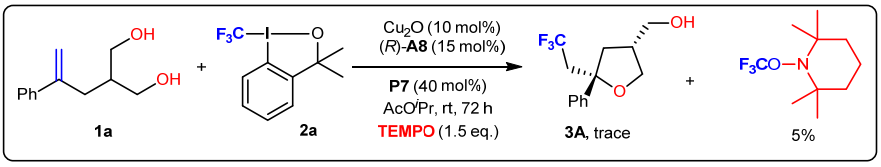
a) Radical trapping experiments

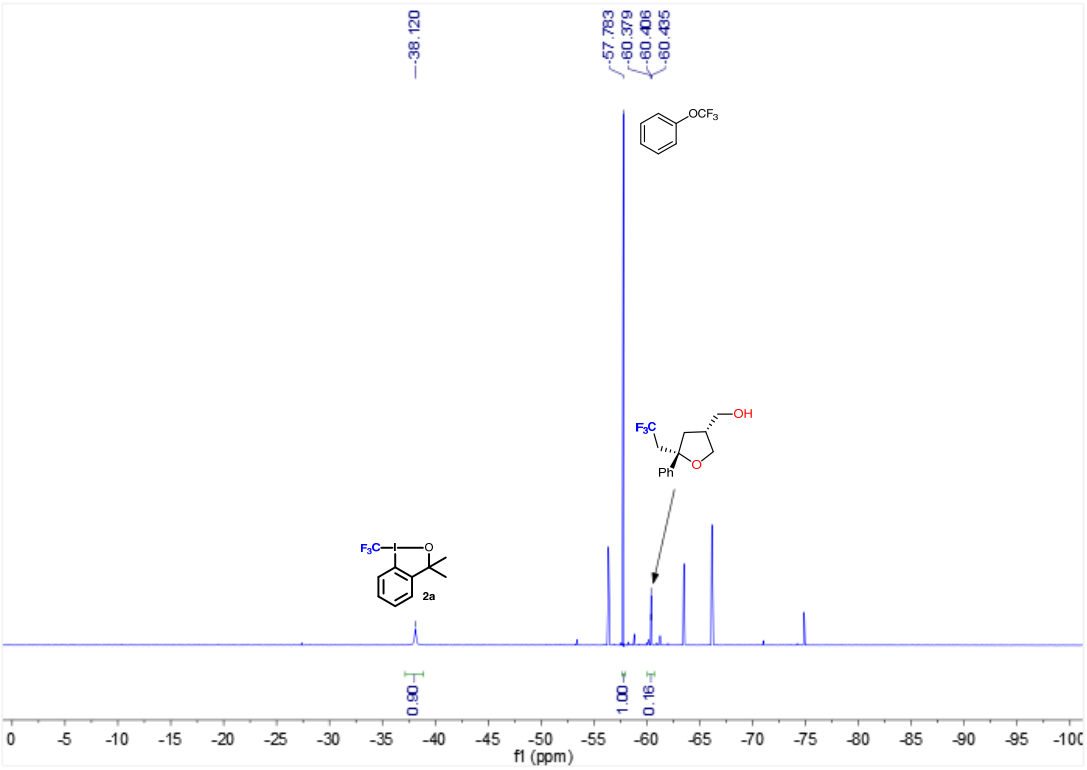


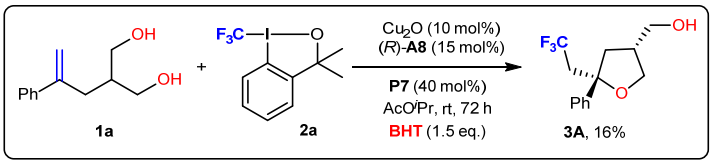
Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1a** (19.2 mg, 0.1 mmol, 1.0 eq.), Cu_2O (1.4 mg, 0.01 mmol, 10 mol%), chiral phosphoric acid (*R*)-**A8** (10.8 mg, 0.015 mmol, 15 mol%), **P7** (15.4 mg, 0.04 mmol, 40 mol%), Togni's reagent **2a** (49.5 mg, 0.15 mmol, 1.5 eq.), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO, 23.4 mg, 0.15 mmol, 1.5 eq.) or butylated hydroxytoluene (BHT, 33.0 mg, 0.15 mmol, 1.5 eq.), and AcO^{i}Pr (1.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C for 72 h. $PhOCF_3$ (internal standard, 0.10 mmol, 1.0 eq.) was added to the reaction mixture. Yield was calculated based on ^{19}F NMR analysis of the crude product.

Note: Since the reaction is sensitive to water and air, the Schlenk tube and the reagents must be dried prior to use.

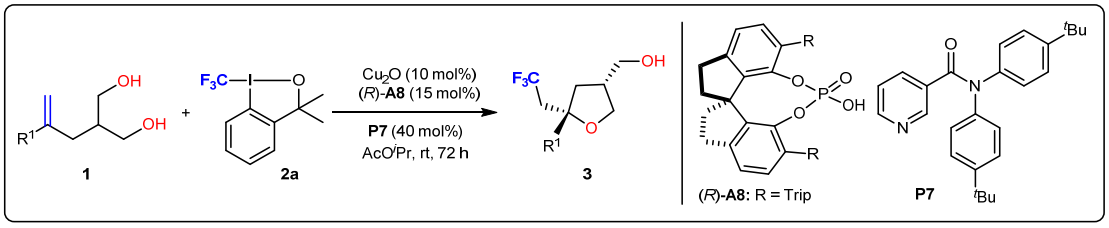




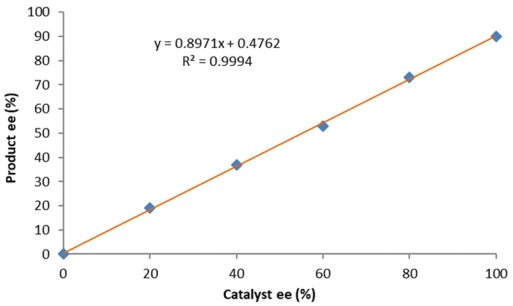




b) Linear effect experiments



These reactions were conducted according to the general procedure C. The catalysts with different *ee* values were prepared by mixing (*R*)-**A8** (99% *ee*) and (*S*)-**A8** (99% *ee*) in appropriate ratios. The product was separated by preparative TLC. The ee values of products was then determined by HPLC, which indicated a linear relationship between ee values of products and corresponding catalysts.


y = 0.8971x + 0.4762
R² = 0.9994
Product ee (%)
Catalyst ee (%)
100
90
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100

3.9 Computational Study

3.9.1 Computational Details

All density functional theory (DFT) calculations were performed using Gaussian 16 program [5] with the default ultrafine integral grid parameters. Geometry optimizations were conducted with B3LYP functional,[7,8] with LANL2DZ basis set[9–12] for the copper as well as bromine atom and 6-31G* basis set for other atoms. Frequency analysis was also performed at the same level of theory as geometry optimization to confirm whether optimized stationary points were either local minimum or transition state, as well as to evaluate zero-point vibrational energies and thermal corrections for enthalpies and free energies at 298.15 K. Single-point energies and solvent effects were evaluated with B3LYP functional and SDD basis set[9,13–17] for the copper as well as bromine atom and 6-311+G(d,p) basis set for other atoms. The solvation energies were calculated with a self-consistent reaction field (SCRF) using the SMD implicit solvent model.[18] Also, geometry optimization, frequency analysis and single point energy of open-shell local minimums were calculated with unrestricted DFT methods, while same computations for closed-shell transition states and local minimum were performed with restricted DFT methods.

Both geometry optimizations and single-point energy calculations include the empirical dispersion corrections.[19] Empirical dispersion corrections employed the D3 version of Grimme's dispersion corrections with Becke-Johnson damping.[20]

To correct the Gibbs free energies under 1 atm to the standard state in solution (1 mol/L), a correction of $RT\ln(c_s/c_g)$ is added to energies of all species. c_s stands for the standard molar concentration in solution (1 mol/L), c_g stands for the standard molar concentration in gas phase (about 0.04087 mol/L at 298.15 K), and R is the gas constant. For calculated intermediates at the standard state of 1 mol/L at 298.15 K, the correction value equaling to 1.89 kcal/mol was used.

Independent Gradient Model (IGM)[21] analysis was performed with Multiwfn[22] software package, using high quality grid option to generate files for further ploting, and the visualization of IGM analysis results are presented with VMD[23] visualization software. The 3D diagrams of optimized structures shown in the main text and Supplementary Figures 5–8 and 9a were generated with CYLview software.[24] The 3D diagram of Supplementary Figure 9b was generated with VMD.

3.9.2 Alternative Pathway Involving Radical Substitution

The triplet state of **Int16** (**Int16-Triplet**) is 5.8 kcal/mol less favorable than the singlet state **Int16**, which is higher in free energy as compared to the closed-shell C–O bond formation transition state **TS17** (Supplementary Figure 4). This indicates that the radical substitution pathway (path a, Fig 3) involving diradical species is unlikely. In addition, the RHF wavefunction stability was confirmed for closed-shell transition state **TS17**, which excludes the existence of the corresponding open-shell

singlet diradical state. Therefore, C–O bond formation proceed via the close-shell transition state **TS17**, instead of the radical substitution pathway (path a, Fig 3).

3.9.3 Located Conformers of C–O Bonding Transition State

See Supplementary Figures 5–8.

3.9.4 Calculations of π–π Interaction

Calculations of π–π interaction between the interacting fragments were performed at B3LYP-D3(BJ)/6-311+G(d,p) level of theory in gas-phase. The stabilization energy of intramolecular π-π interactions ($\Delta E_{\pi\text{-}\pi}$) was calculated using Gaussian 16[5] by defining pyridine as fragment-1 and benzene as fragment-2.

$$\Delta E_{\pi\text{-}\pi} = E_{\text{complex}} - E_{\text{separate}} = E_{\text{complex}} - (E_{\text{fragmemt-1}} + E_{\text{fragment-2}})$$

E_{complex} refers to the gas-phase single point energy of the interacting benzene and pyridine fragments. The geometry of the interacting fragments was taken from the optimized geometry of **TS17**, with appended C–H bonds (1.086Å for C(sp^2)–H bond both on pyridine and benzene fragments). E_{separate} ($E_{\text{fragmemt-1}}$ and $E_{\text{fragment-2}}$)) refers to the gas phase energies of each fragment. Supplementary Figure 9 includes the geometry of the interacting fragments from **TS17**. The interacting energy between these two fragments is 1.7 kcal/mol.

3.9.5 Table of Energies

Supplementary Table 7. Energies in Fig. 4, Fig. 5, and Supplementary Figures 4 to 8. Zero-point correction (*ZPE*), thermal correction to enthalpy (*TCH*), thermal correction to Gibbs free energy (*TCG*), energies (*E*), enthalpies (*H*), and Gibbs free energies (*G*) (in Hartree) of the structures calculated at B3LYP-D3(BJ)/6-311+G(d,p)-SDD-SMD(Propyl Ethanoate)//B3LYP-D3(BJ)/6-31G (d)-LANL2DZ level of theory.

Structure	*ZPE*	*TCH*	*TCG*	*E*	*H*	*G*	Imaginary Frequency
Int12	0.274936	0.293432	0.227017	−955.327939	−955.034507	−955.100922	
Int13	1.970986	2.084677	1.813142	−5068.262546	−5066.177869	−5066.449404	
Int14	1.742439	1.845289	1.600379	−4829.528505	−4827.683216	−4827.928126	
TS15	1.741194	1.843766	1.597279	−4829.523391	−4827.679625	−4827.926112	29.8*i*
Int16	1.741819	1.845268	1.595028	−4829.522656	−4827.677388	−4827.927628	

Int16-Triplet	1.741011	1.843963	1.597370	−4829.515692	−4827.671729	−4827.918322	
TS17	1.742511	1.844997	1.600049	−4829.522021	−4827.677024	−4827.921972	39.9*i*
Int18	0.507369	0.539871	0.439369	−1404.912642	−1404.372771	−1404.473273	
3A	0.265969	0.283115	0.220877	−954.749710	−954.466595	−954.528833	
(*R*)-A8	0.963839	1.016959	0.878394	−2469.828876	−2468.811917	−2468.950482	
P7	0.504703	0.533157	0.445327	−1194.054491	−1193.521334	−1193.609164	
TS17-a	1.741763	1.844334	1.599122	−4829.515486	−4827.671152	−4827.916364	85.9*i*
TS17-b	1.742801	1.844947	1.601932	−4829.513596	−4827.668649	−4827.911664	50.1*i*
TS17-c	1.740464	1.843146	1.596231	−4829.507800	−4827.664654	−4827.911569	53.5*i*
TS17-S1	1.741978	1.844762	1.598634	−4829.514107	−4827.669345	−4827.915473	26.4*i*
TS17-S2	1.741979	1.844717	1.599092	−4829.511830	−4827.667113	−4827.912738	21.6*i*
TS17-S3	1.741870	1.844814	1.597425	−4829.512677	−4827.667863	−4827.915252	43.9*i*
TS17-S4	1.741854	1.844930	1.595773	−4829.506763	−4827.661833	−4827.910990	44.6*i*
TS17-S5	1.741309	1.844287	1.594813	−4829.504223	−4827.659936	−4827.909410	84.2*i*
TS17-S6	1.741769	1.844524	1.597440	−4829.502876	−4827.658352	−4827.905436	57.2*i*
TS17-S7	1.741230	1.843552	1.599951	−4829.490265	−4827.646713	−4827.890314	19.6*i*
TS17-S8	1.739442	1.842009	1.591987	−4829.495218	−4827.653209	−4827.903231	56.0*i*
TS17-S9	1.739940	1.842294	1.596903	−4829.496964	−4827.654670	−4827.900061	48.6*i*
TS17-S10	1.740513	1.842815	1.596511	−4829.502149	−4827.659334	−4827.905638	34.7*i*
TS17-S11	1.742046	1.844352	1.599411	−4829.512765	−4827.668413	−4827.913354	31.6*i*
TS17-S12	1.741108	1.843948	1.595912	−4829.506984	−4827.663036	−4827.911072	20.8*i*
TS17-S13	1.742256	1.844372	1.600358	−4829.509971	−4827.665599	−4827.909613	61.1*i*
TS17-S14	1.742162	1.844283	1.601309	−4829.507999	−4827.663716	−4827.906690	48.8*i*
TS17-S15	1.741069	1.843378	1.597488	−4829.502477	−4827.659099	−4827.904989	56.6*i*

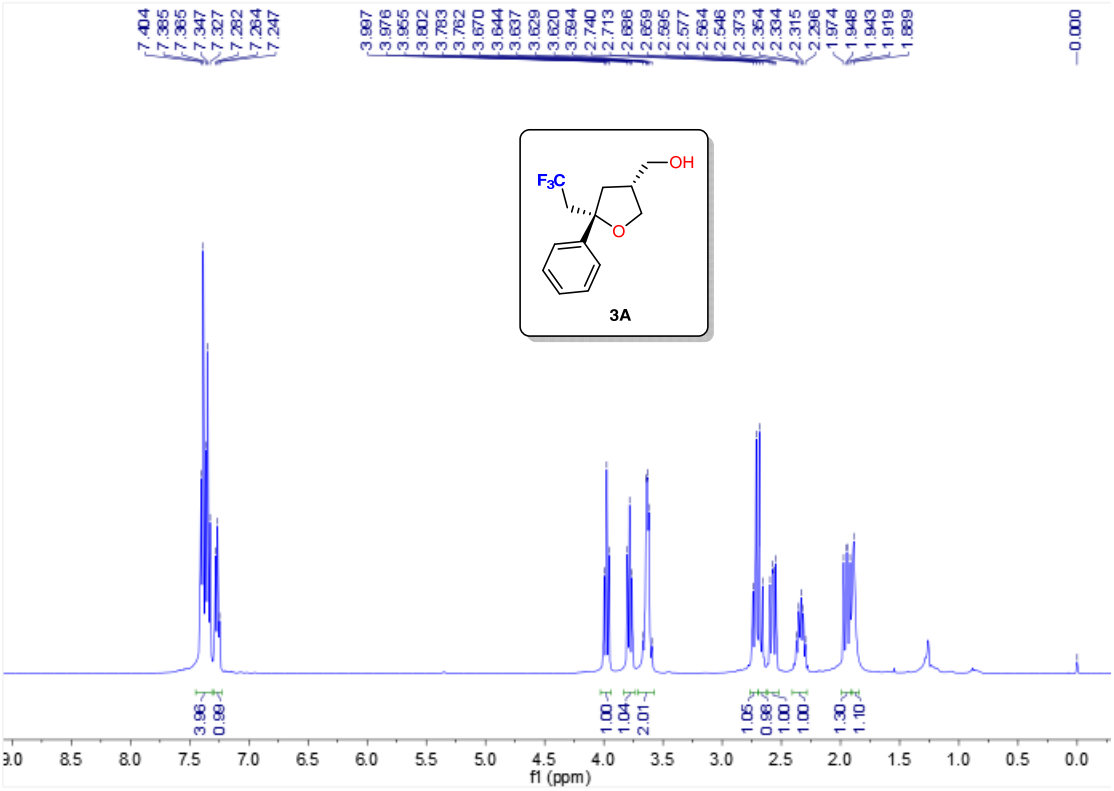
Supplementary Table 8. Energies in Fig. 5. Gas-phase energies (*E*) (in Hartree) of the structures calculated at the B3LYP-D3(BJ)/6-311+G(d,p)-SDD level of theory.

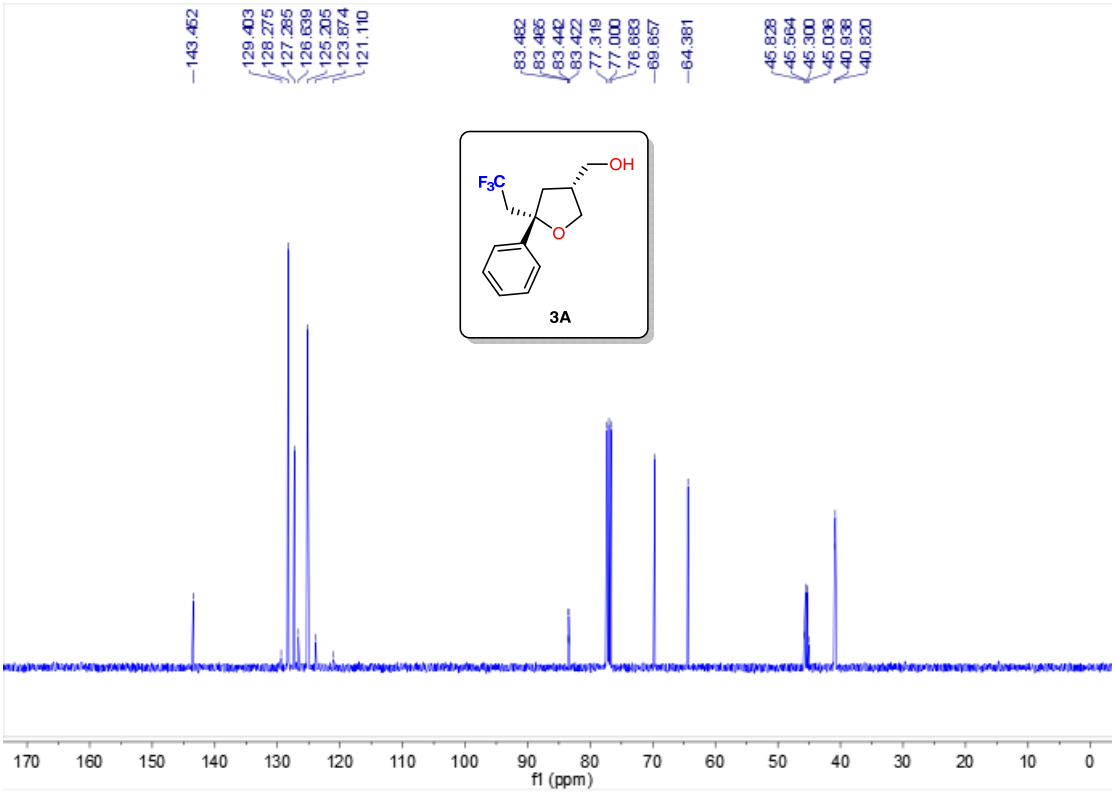
Structure	*E*
TS17	−4829.451737
TS17-a	−4829.443054
TS17-b	−4829.443669
TS17-c	−4829.427385

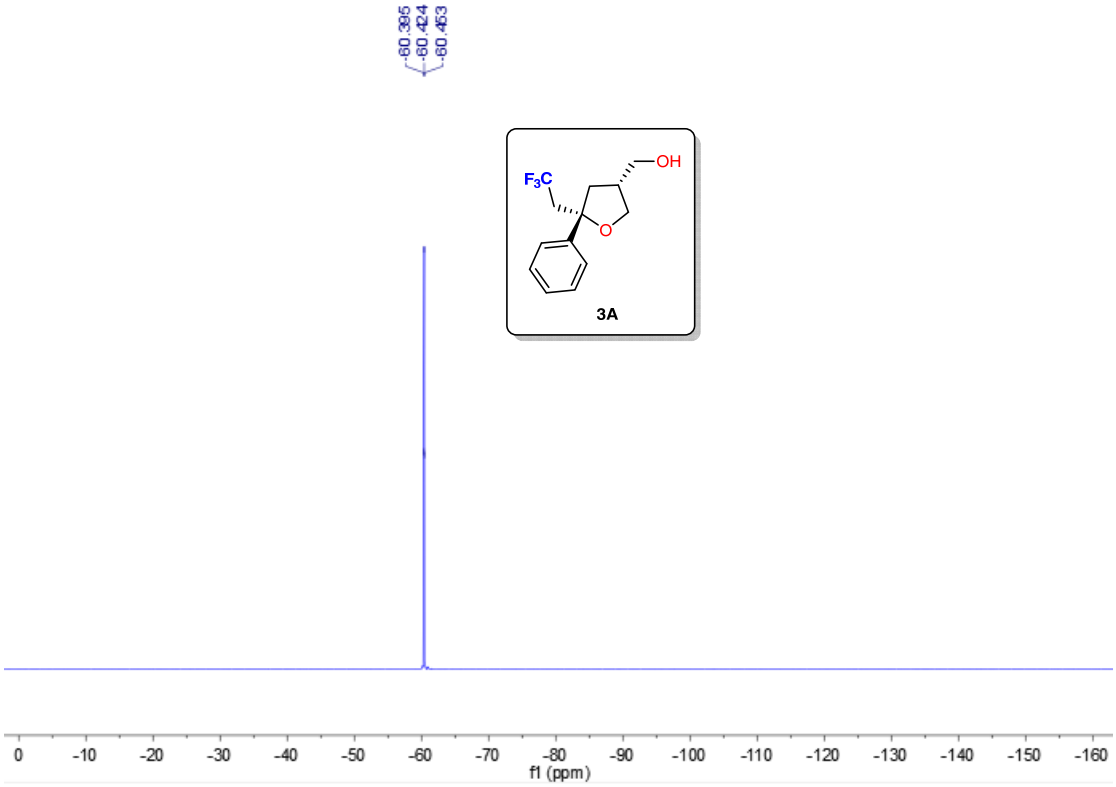
Supplementary Table 9. Energies in Supplementary Figure 9. Gas-phase energies (*E*) (in Hartree) of the structures calculated at the B3LYP-D3(BJ)/6-311+G(d,p) level of theory.

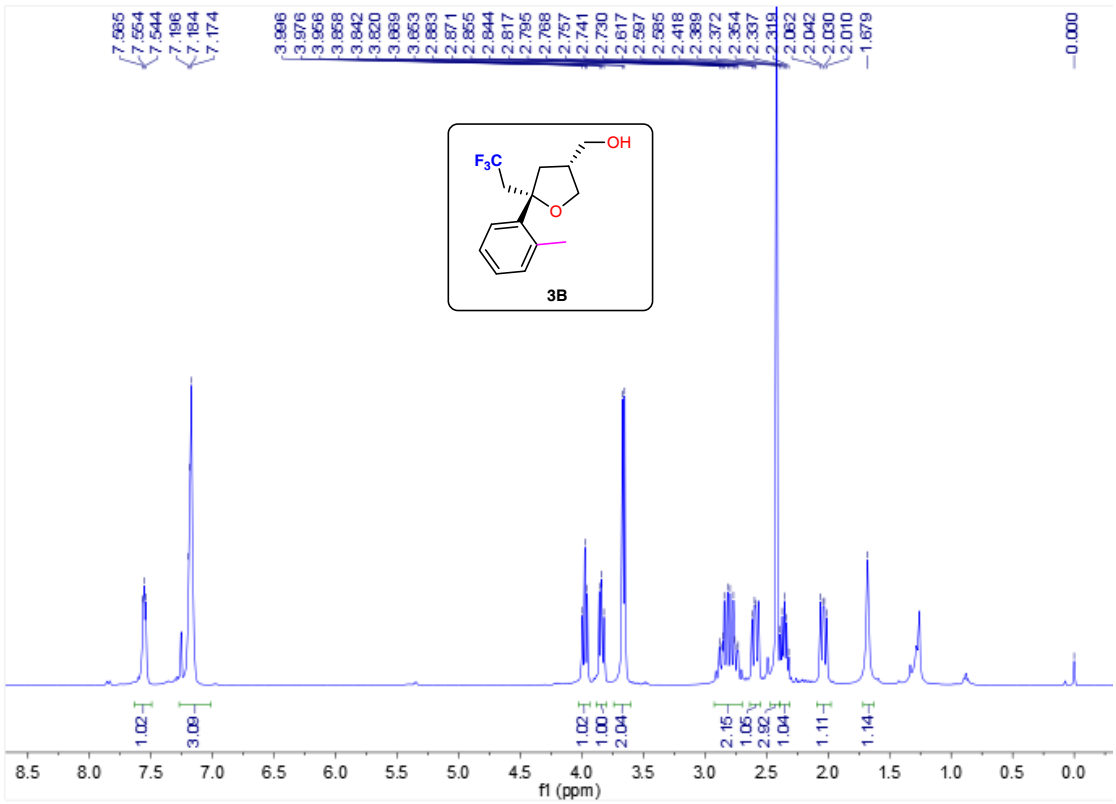
Structure	*E*
π–π Interacting Fragment	−480.697730
Fragment-1	−248.367468
Fragment-2	−232.327526

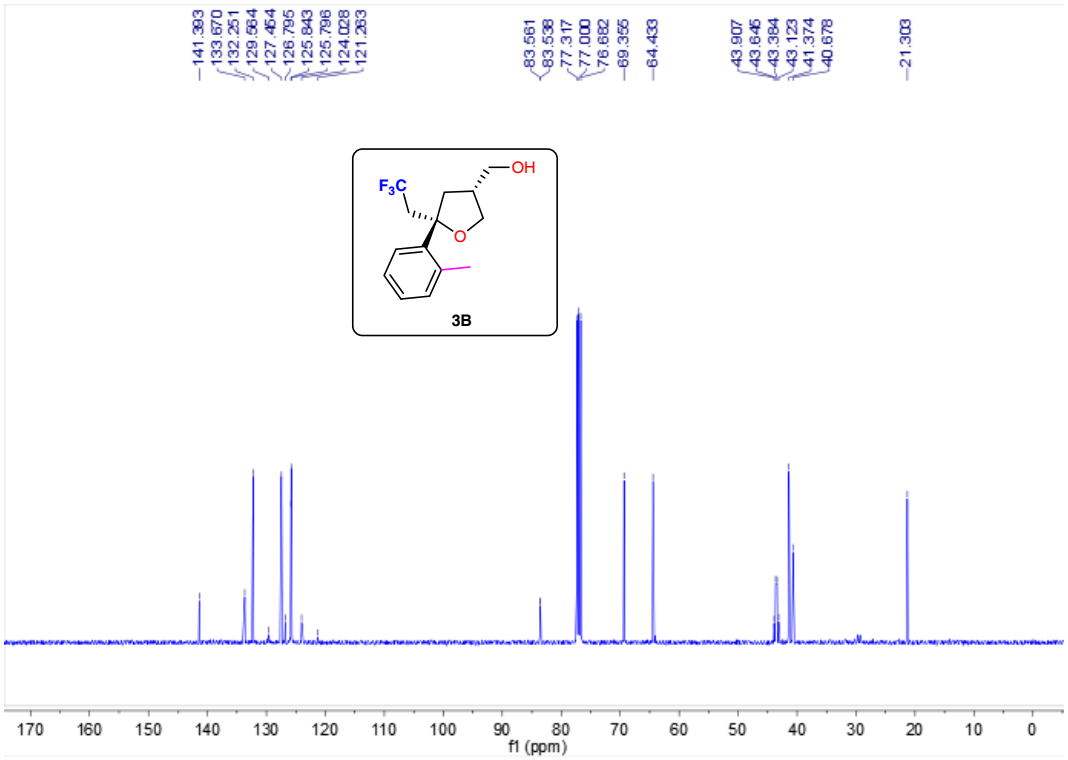
3.10 NMR Spectra

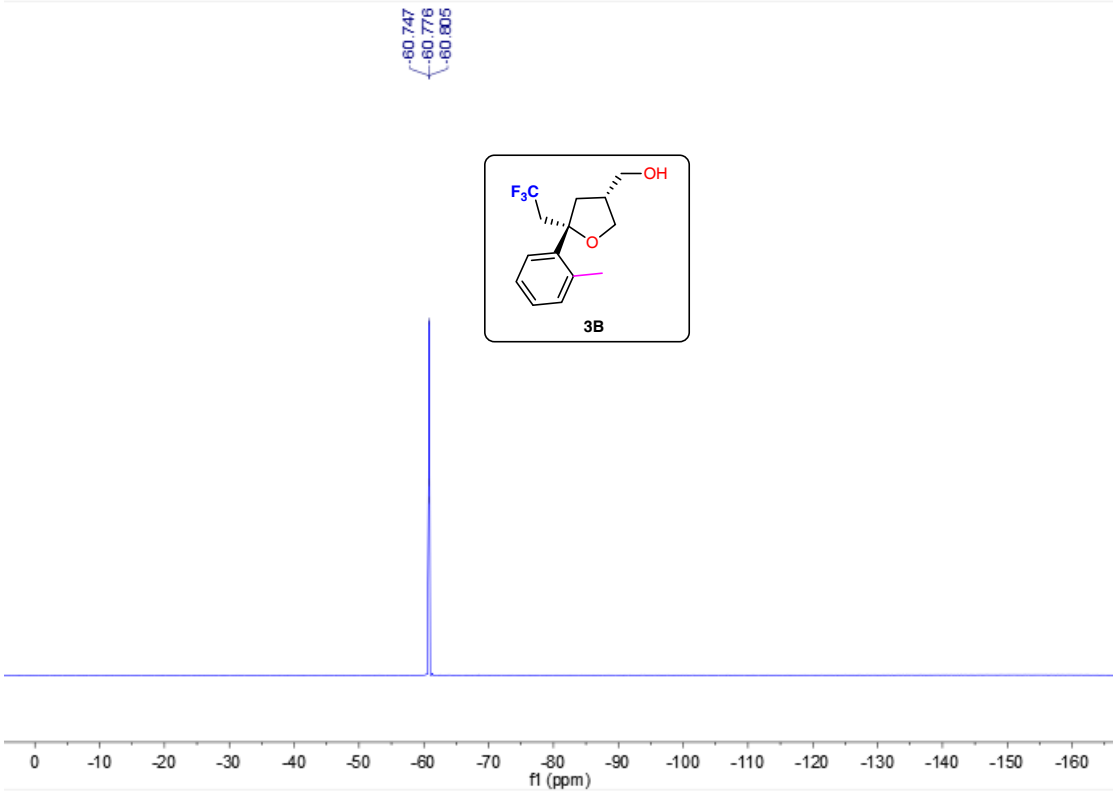


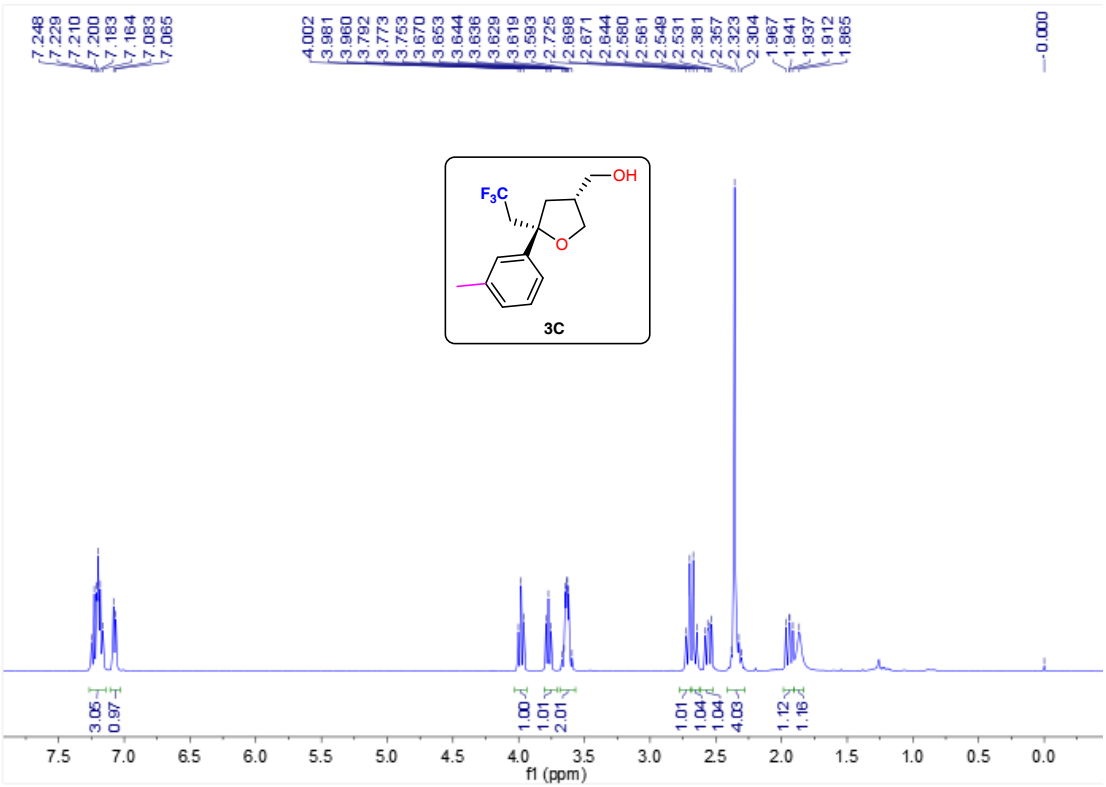


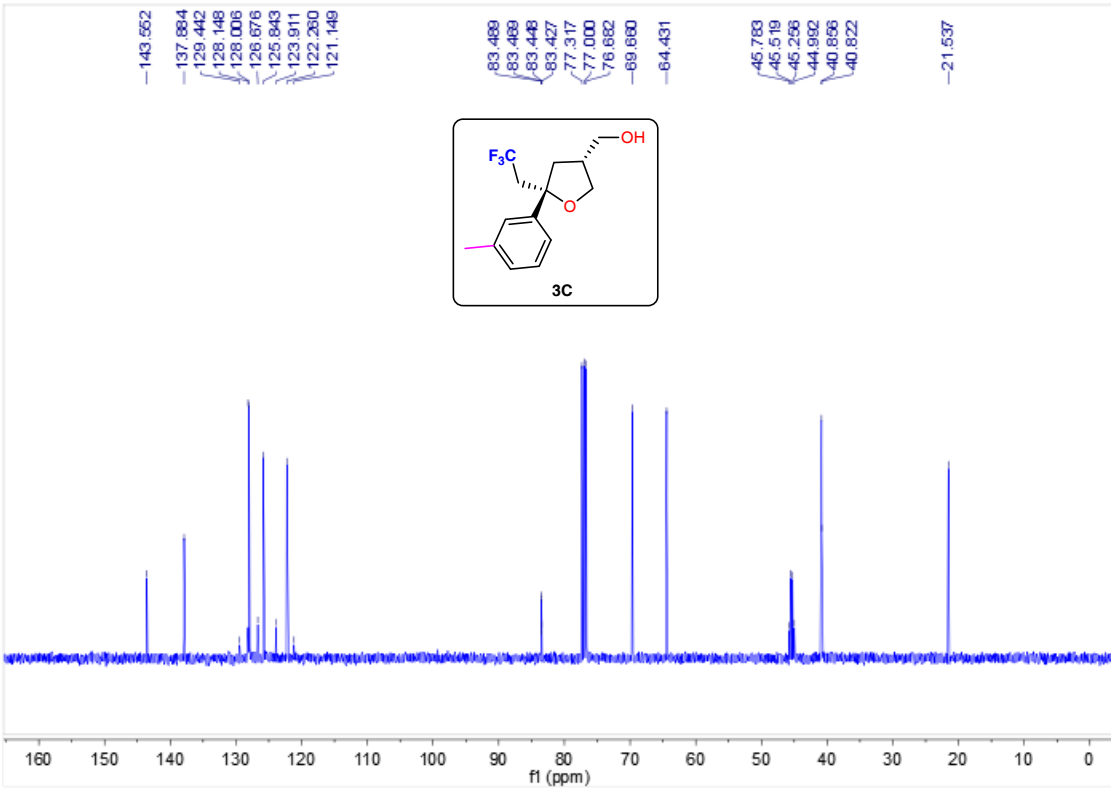

-60.395
-60.424
-60.453
F3C
OH
O
3A
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

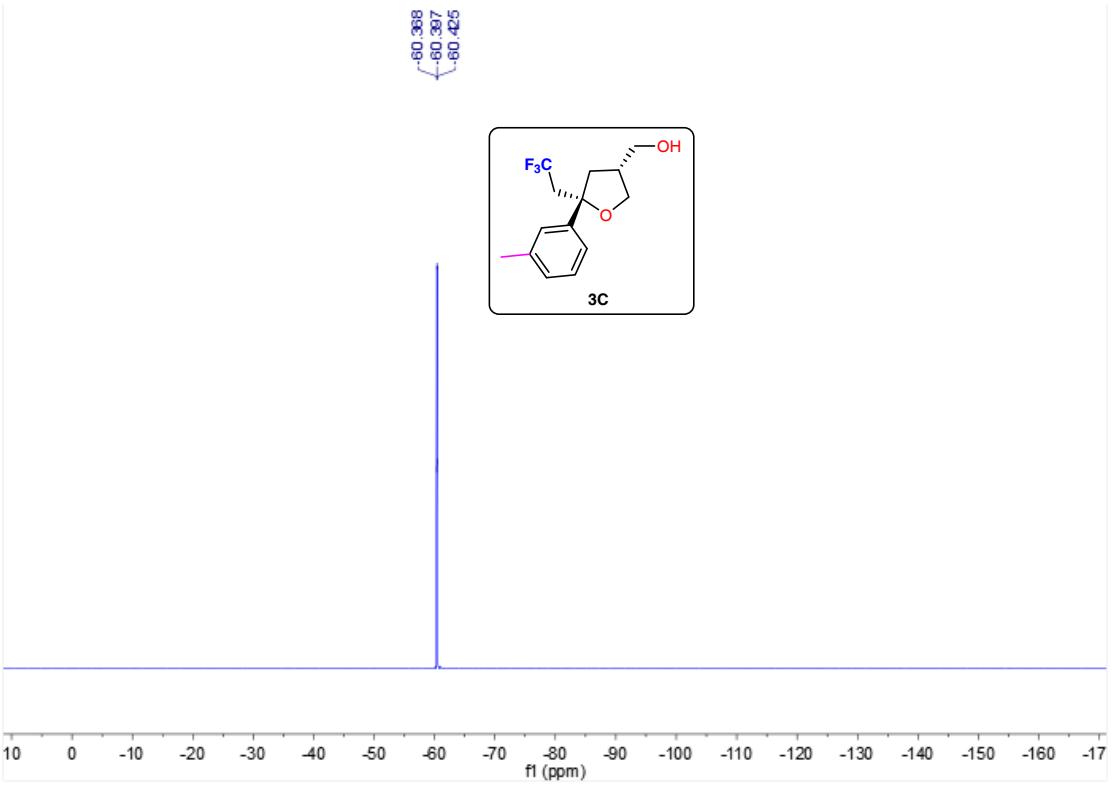

7.565
7.554
7.544
7.196
7.184
7.174
3.996
3.976
3.956
3.858
3.842
3.820
3.669
3.653
3.883
2.871
2.855
2.844
2.817
2.795
2.788
2.757
2.741
2.730
2.617
2.597
2.585
2.418
2.389
2.372
2.354
2.337
2.319
2.062
2.042
2.030
2.010
1.679
0.000
F3C
OH
O
3B
1.02
3.09
1.02
1.00
2.04
2.15
1.05
2.92
1.04
1.11
1.14
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)

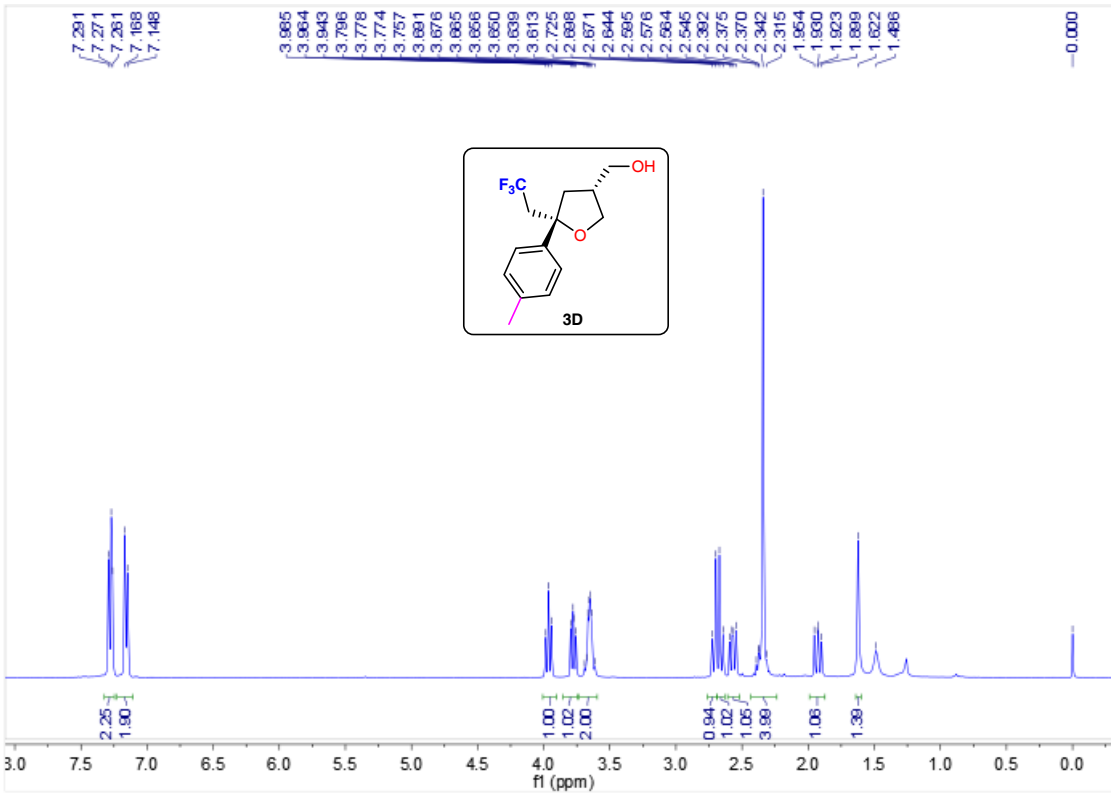

141.393
133.670
132.251
129.664
127.454
126.795
125.843
125.796
124.028
121.263
83.561
83.538
77.317
77.000
76.682
69.355
64.433
43.907
43.645
43.384
43.123
41.374
40.678
21.303
F3C
OH
O
3B
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

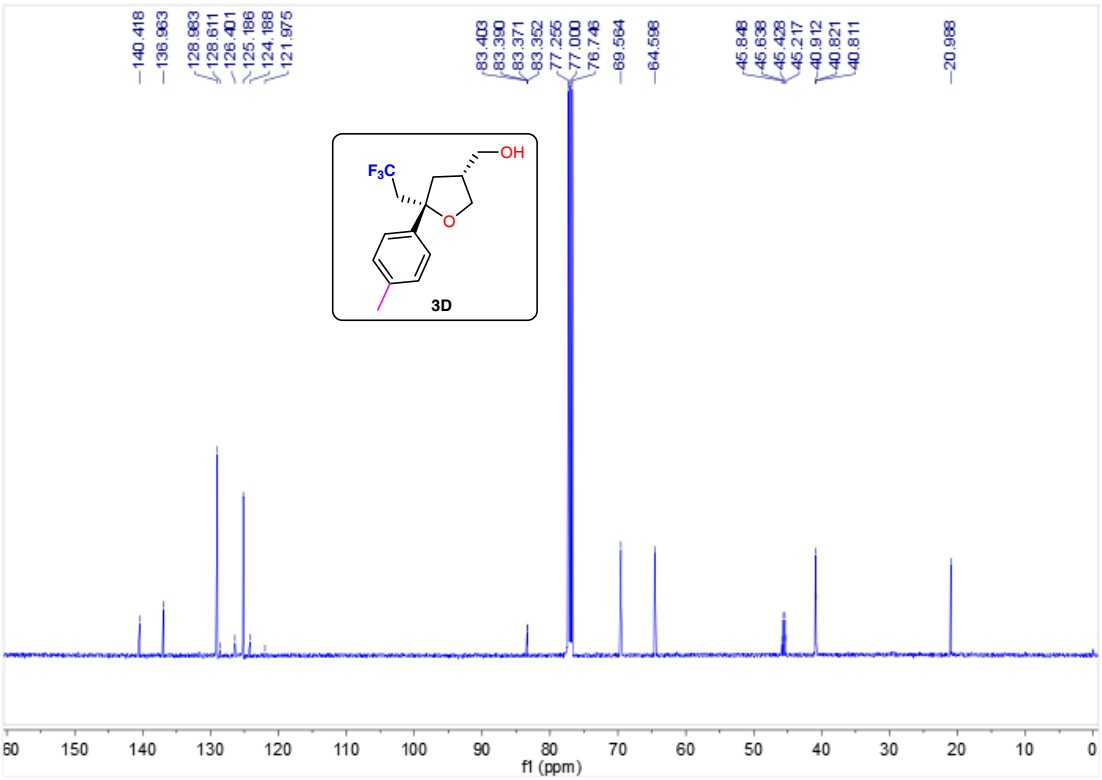

60.747
60.776
60.805
F3C
OH
O
3B
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

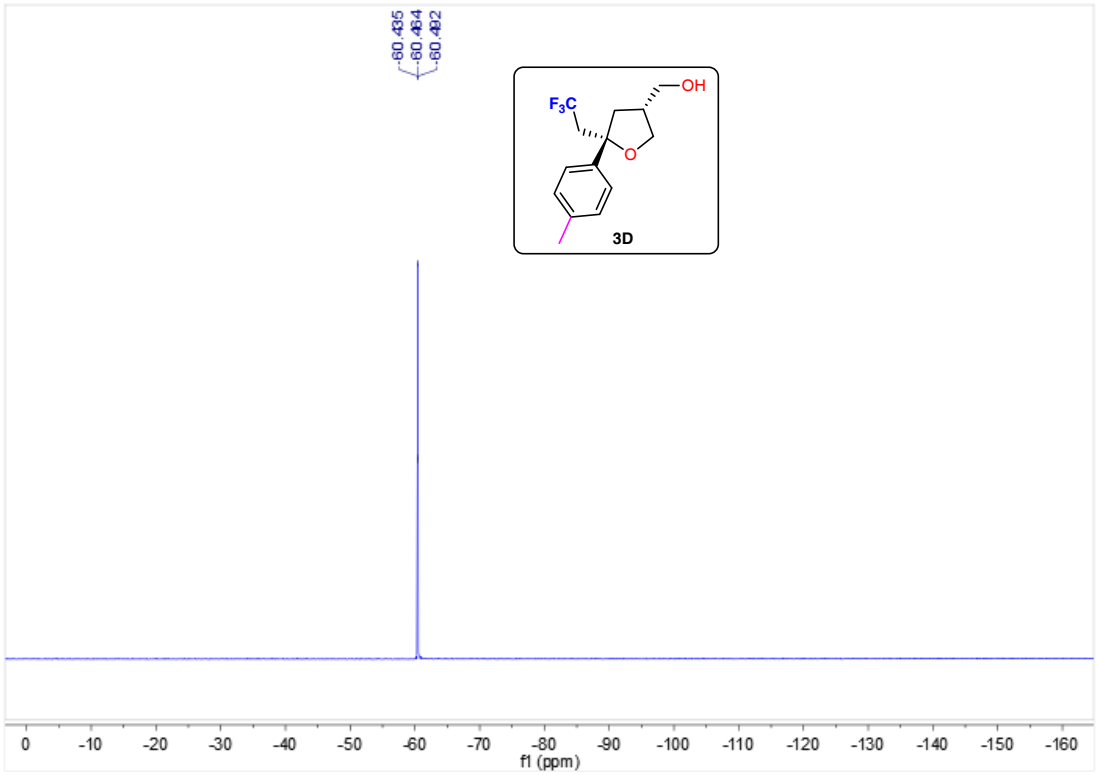

7.248
7.229
7.210
7.200
7.183
7.164
7.083
7.065
4.002
3.981
3.960
3.792
3.773
3.753
3.670
3.653
3.644
3.636
3.629
3.619
3.593
2.725
2.698
2.671
2.644
2.580
2.561
2.549
2.531
2.381
2.357
2.323
2.304
1.967
1.941
1.937
1.912
1.865
0.000
F3C
OH
O
3C
3.05
0.97
1.00
1.01
2.01
1.01
1.04
1.04
4.03
1.12
1.16
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)

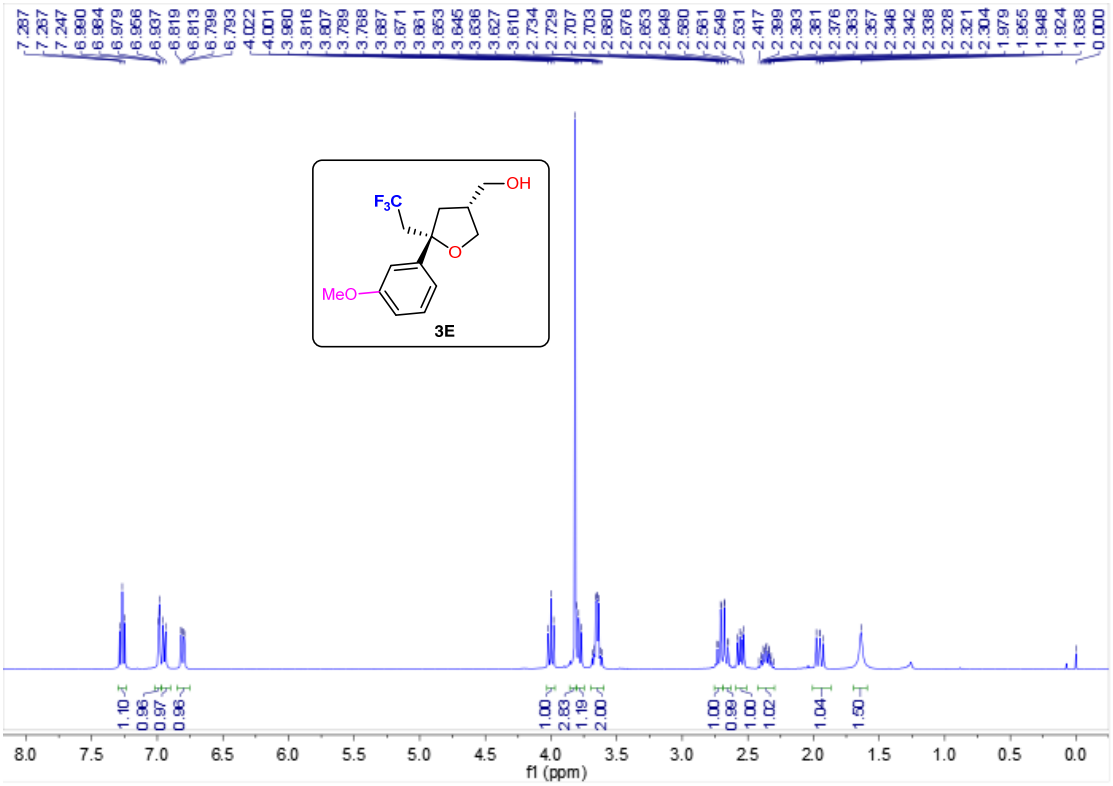

143.552
137.884
129.442
128.148
128.006
126.676
125.843
123.911
122.260
121.149
83.489
83.469
83.448
83.427
77.317
77.000
76.682
69.660
64.431
45.783
45.519
45.256
44.992
40.856
40.822
21.537
F3C
OH
O
3C
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

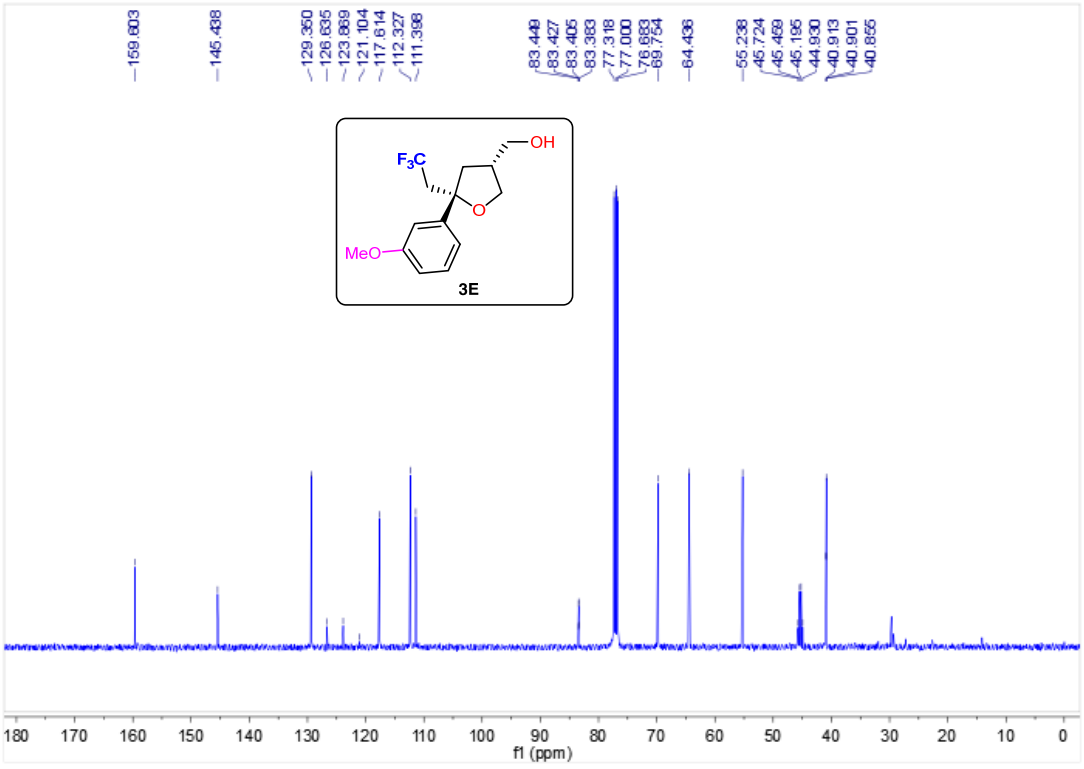

-60.368
-60.397
-60.425
F3C
OH
O
3C
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -17
f1 (ppm)

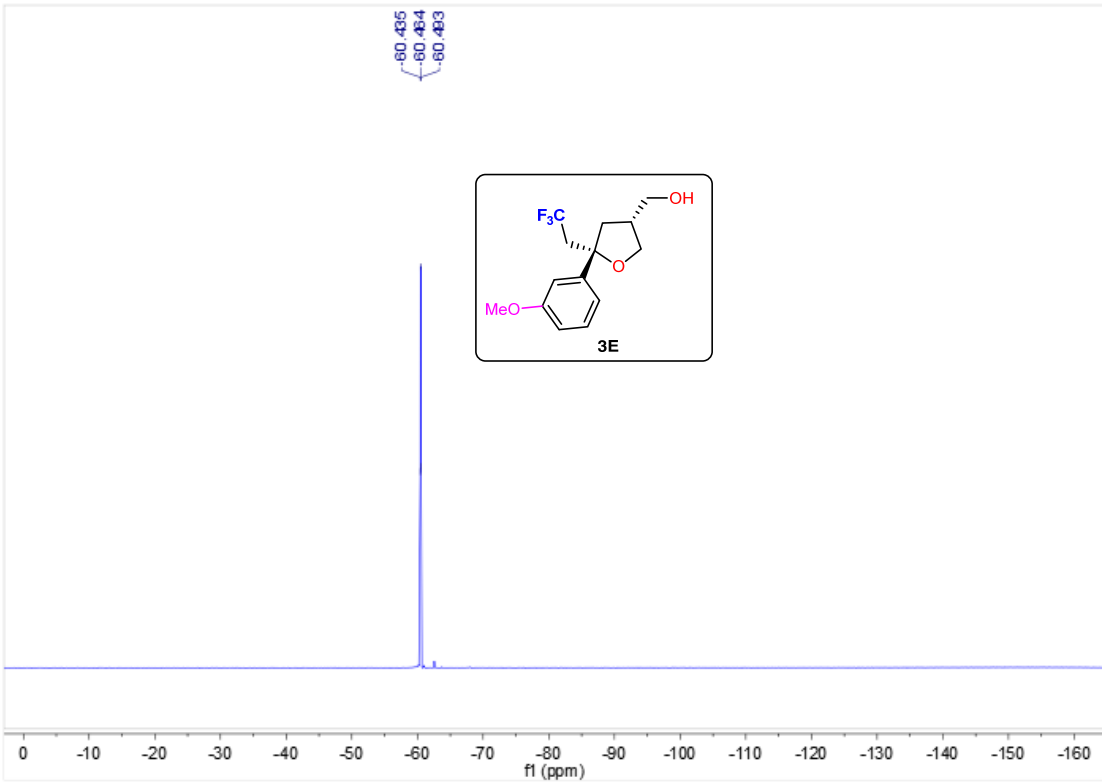

7.291
7.271
7.261
7.168
7.148
3.965
3.964
3.943
3.796
3.778
3.774
3.757
3.691
3.676
3.665
3.656
3.650
3.639
3.613
2.725
2.698
2.671
2.644
2.595
2.576
2.564
2.545
2.392
2.375
2.370
2.342
2.315
1.964
1.930
1.923
1.899
1.622
1.486
0.000
F3C
OH
O
3D
2.25
1.90
1.00
1.02
2.00
0.94
1.02
1.05
3.99
1.08
1.39
3.0 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)

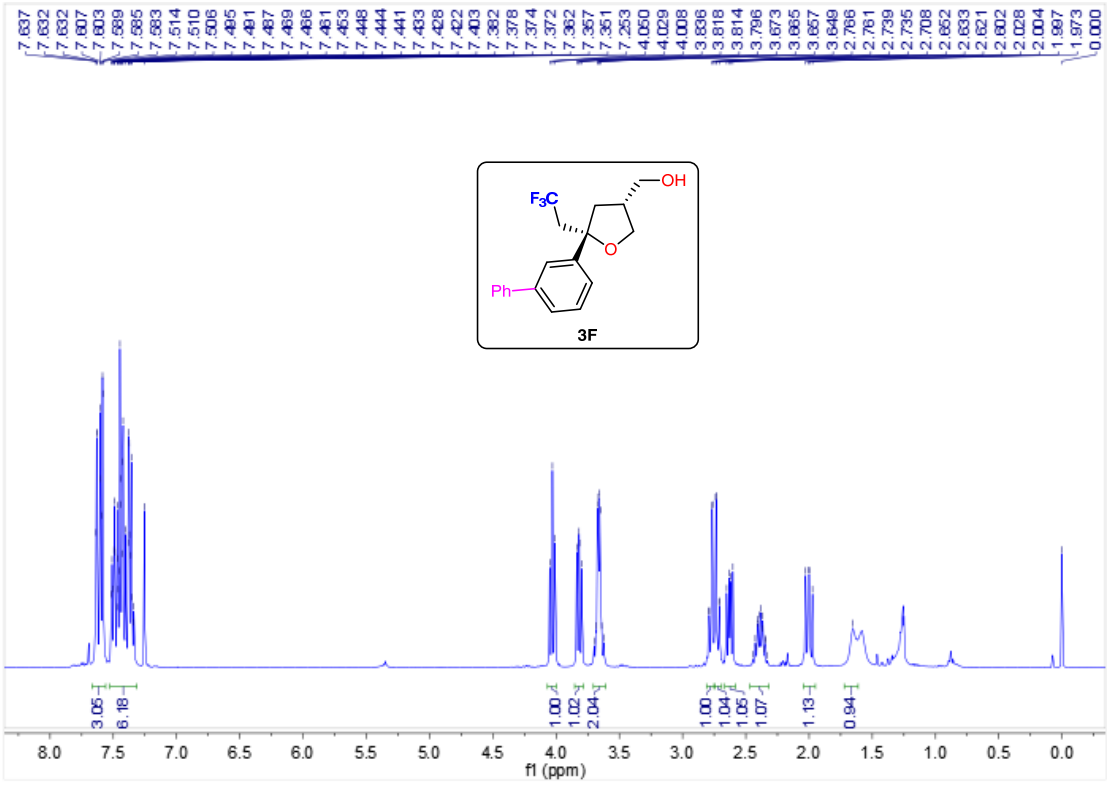

140.418
136.963
128.983
128.611
126.401
125.186
124.188
121.975
83.403
83.390
83.371
83.352
77.255
77.000
76.746
69.564
64.598
45.848
45.638
45.428
45.217
40.912
40.821
40.811
20.988
OH
F3C
O
3D
f1 (ppm)

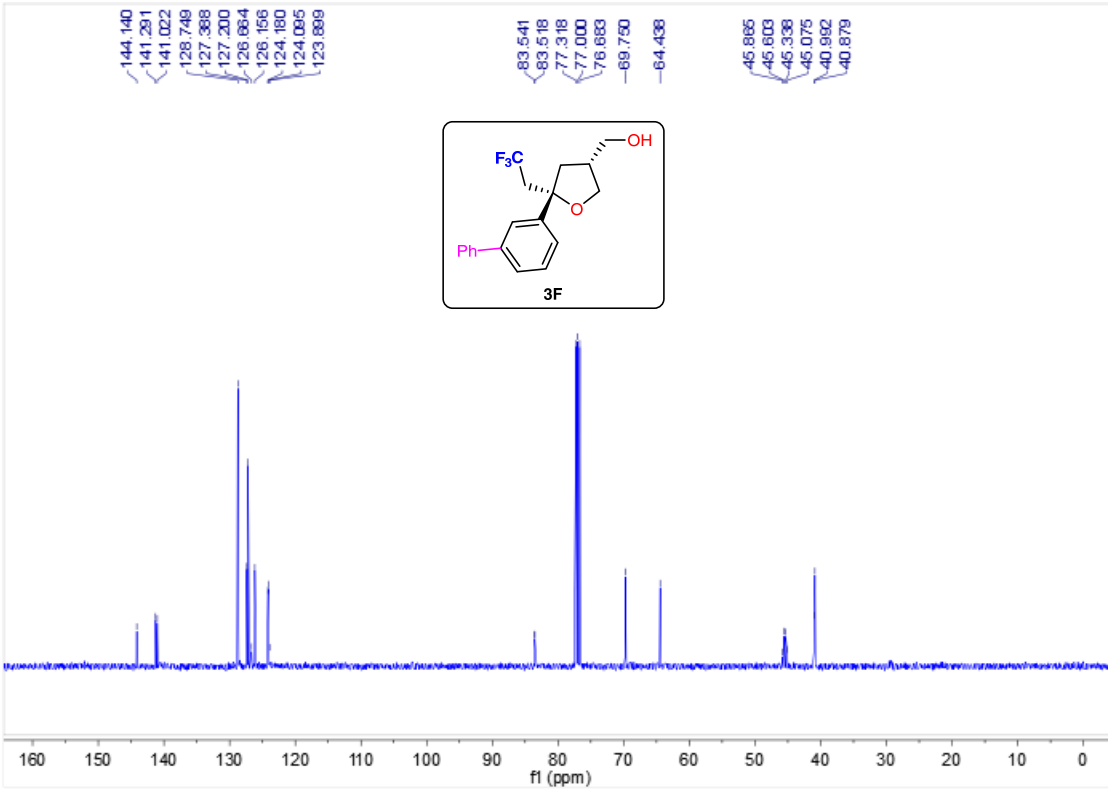

60.435
60.464
60.492
OH
F3C
O
3D
f1 (ppm)

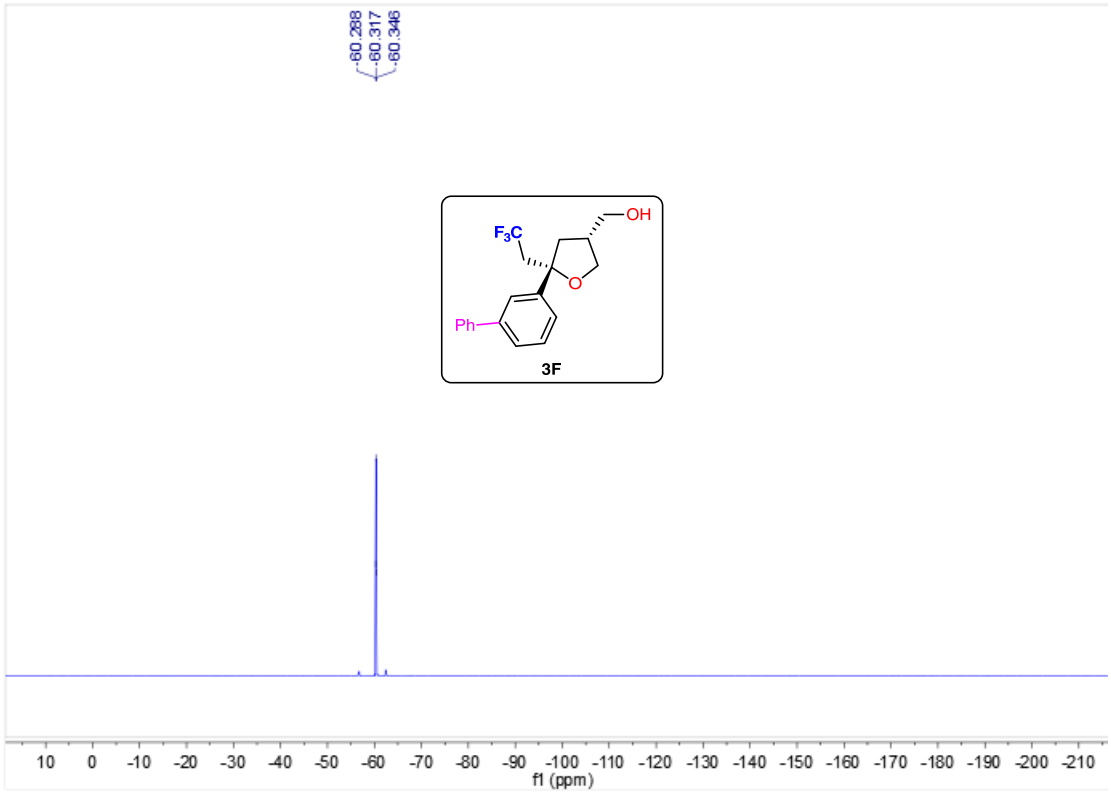

F3C
OH
O
MeO
3E
f1 (ppm)

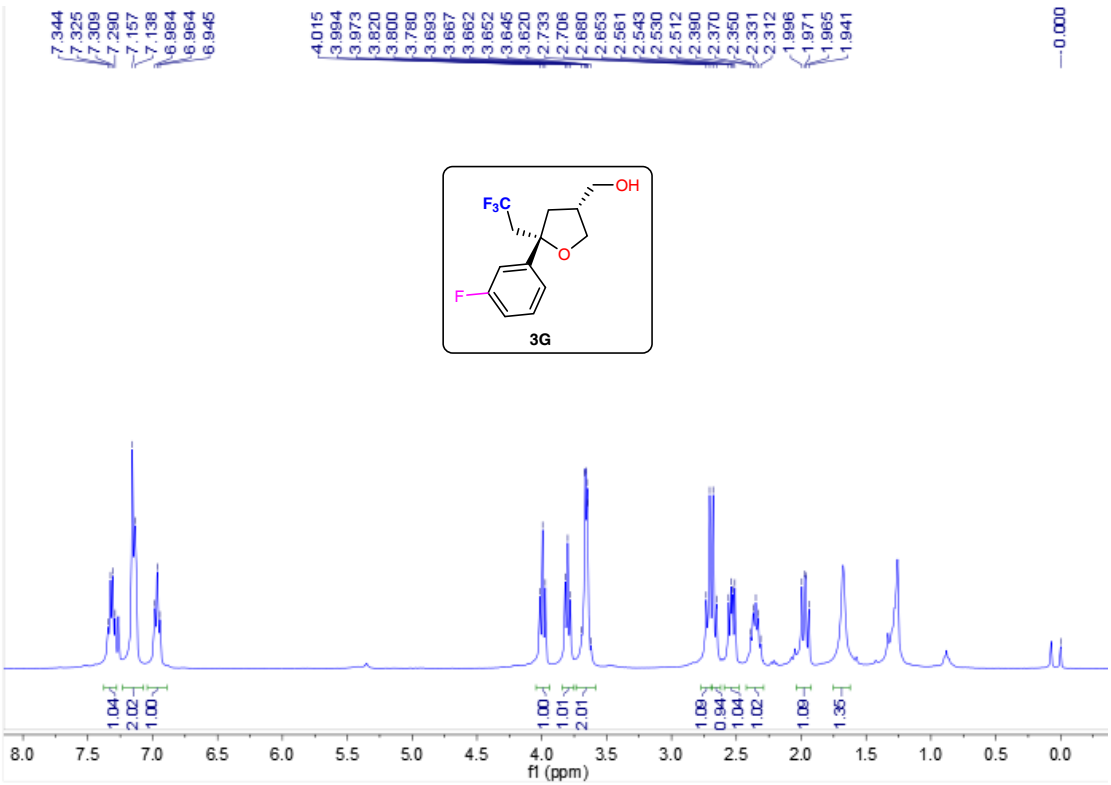

F3C
OH
O
MeO
3E
f1 (ppm)

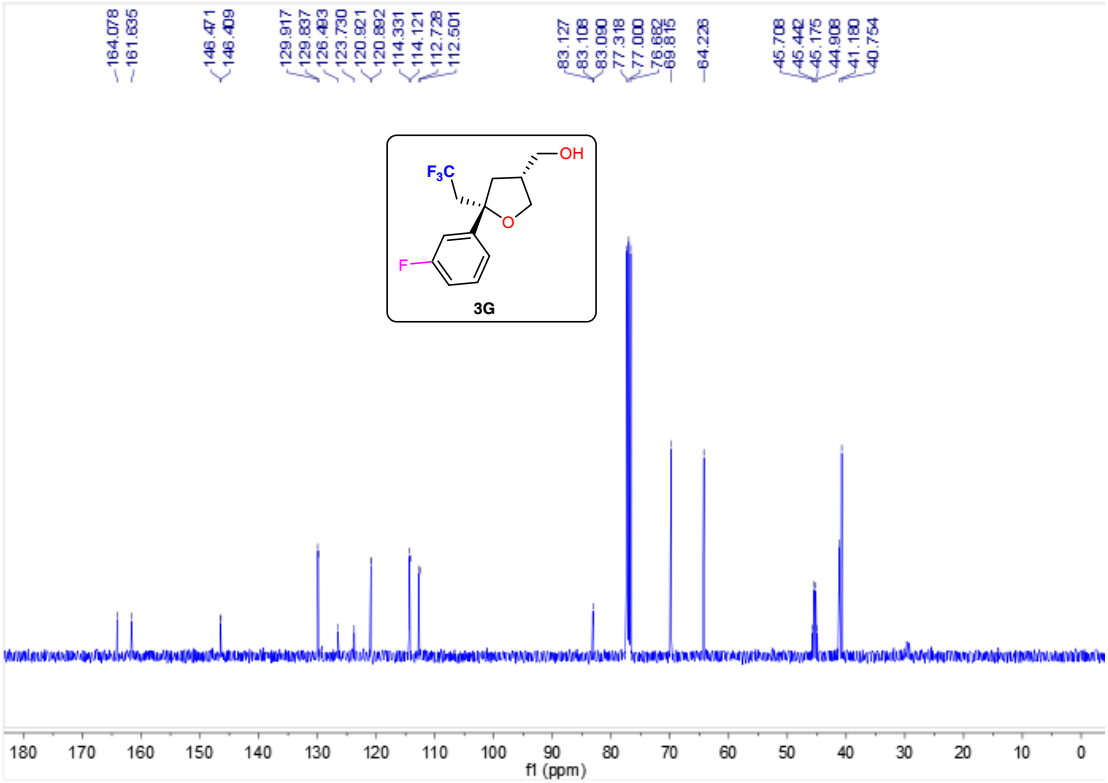

-60.435
-60.464
-60.493
F3C
OH
O
MeO
3E
f1 (ppm)

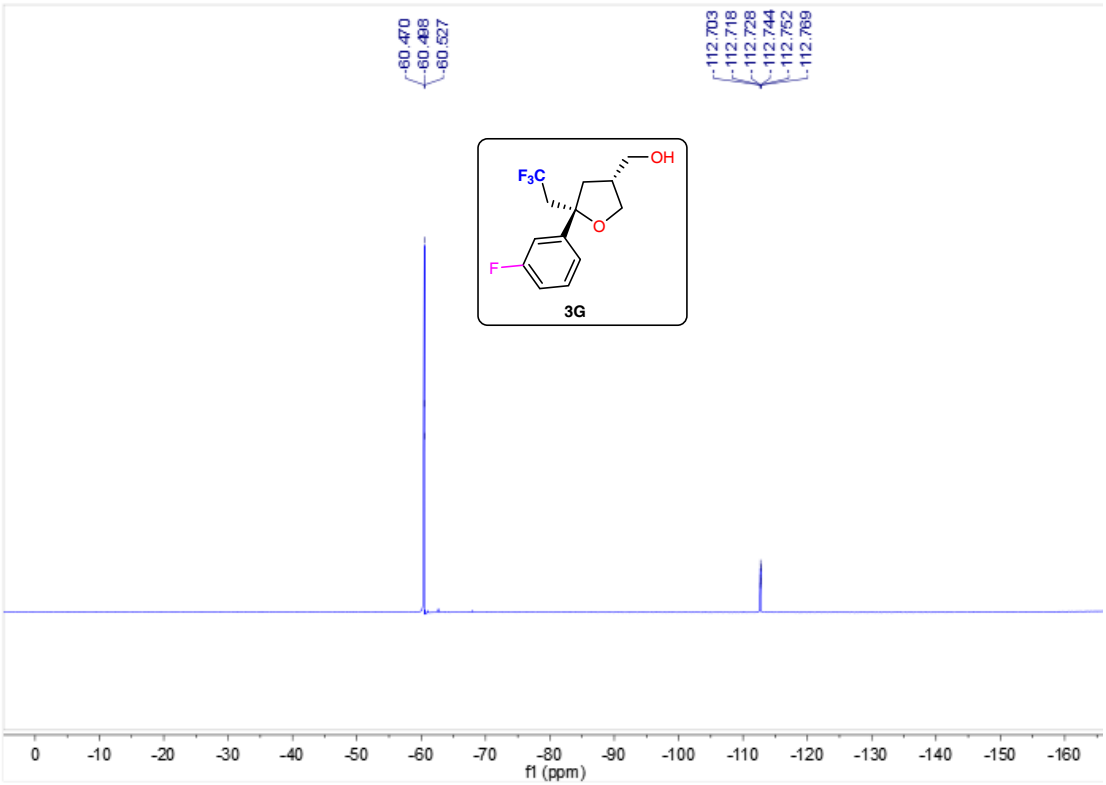

F3C
OH
O
Ph
3F
3.05
6.18
1.00
1.02
2.04
1.00
1.04
1.05
1.07
1.13
0.94
f1 (ppm)

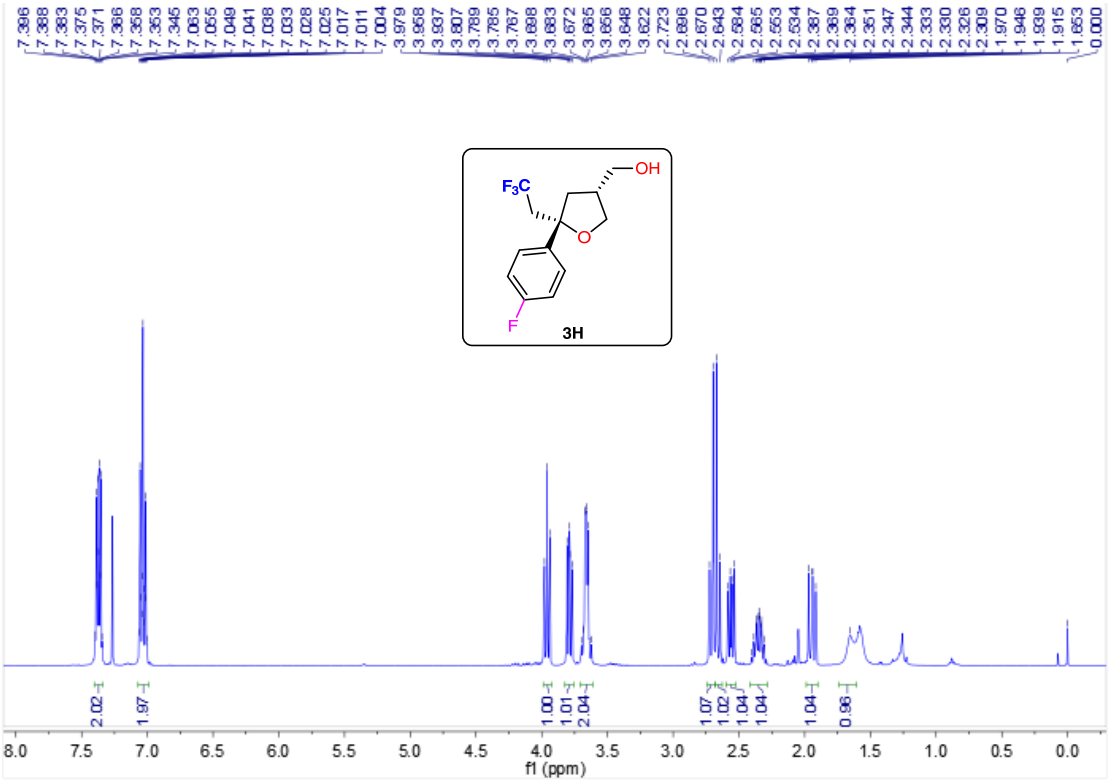

144.140
141.291
141.022
128.749
127.388
127.200
126.884
126.156
124.180
124.095
123.899
83.541
83.518
77.318
77.000
76.683
69.750
64.438
45.886
45.603
45.338
45.075
40.992
40.879
OH
F3C
O
Ph
3F
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

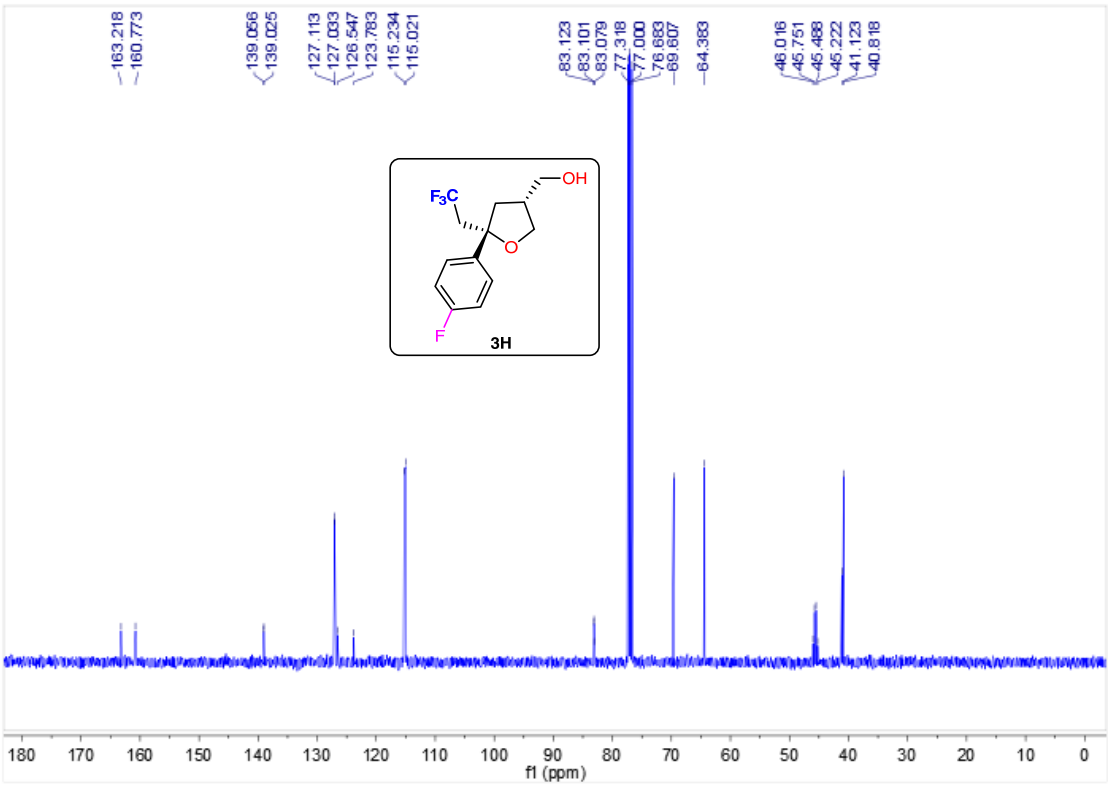

60.288
60.317
60.346
OH
F3C
O
Ph
3F
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

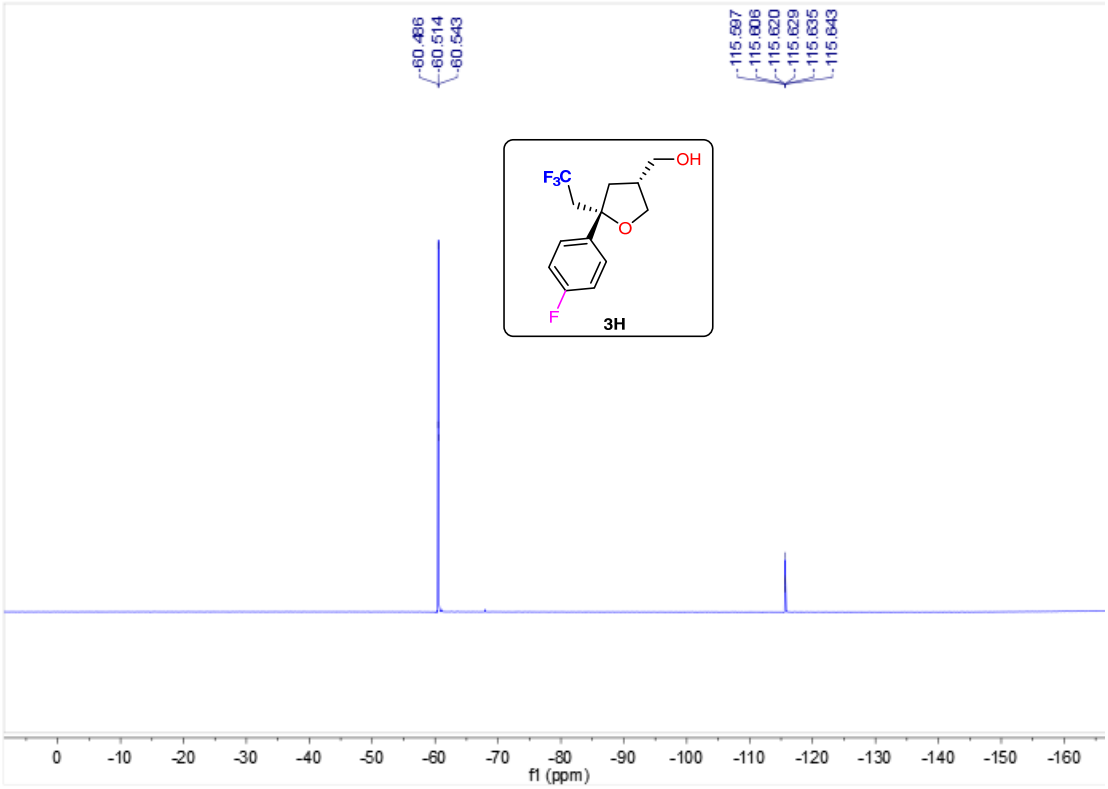

7.344
7.325
7.309
7.290
7.157
7.138
6.984
6.964
6.945
4.015
3.994
3.973
3.820
3.800
3.780
3.693
3.667
3.662
3.652
3.645
3.620
2.733
2.706
2.680
2.653
2.561
2.543
2.530
2.512
2.390
2.370
2.350
2.331
2.312
1.996
1.971
1.965
1.941
0.000
F3C
OH
O
F
3G
1.04
2.02
1.00
1.00
1.01
2.01
1.09
0.94
1.04
1.02
1.09
1.35
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)

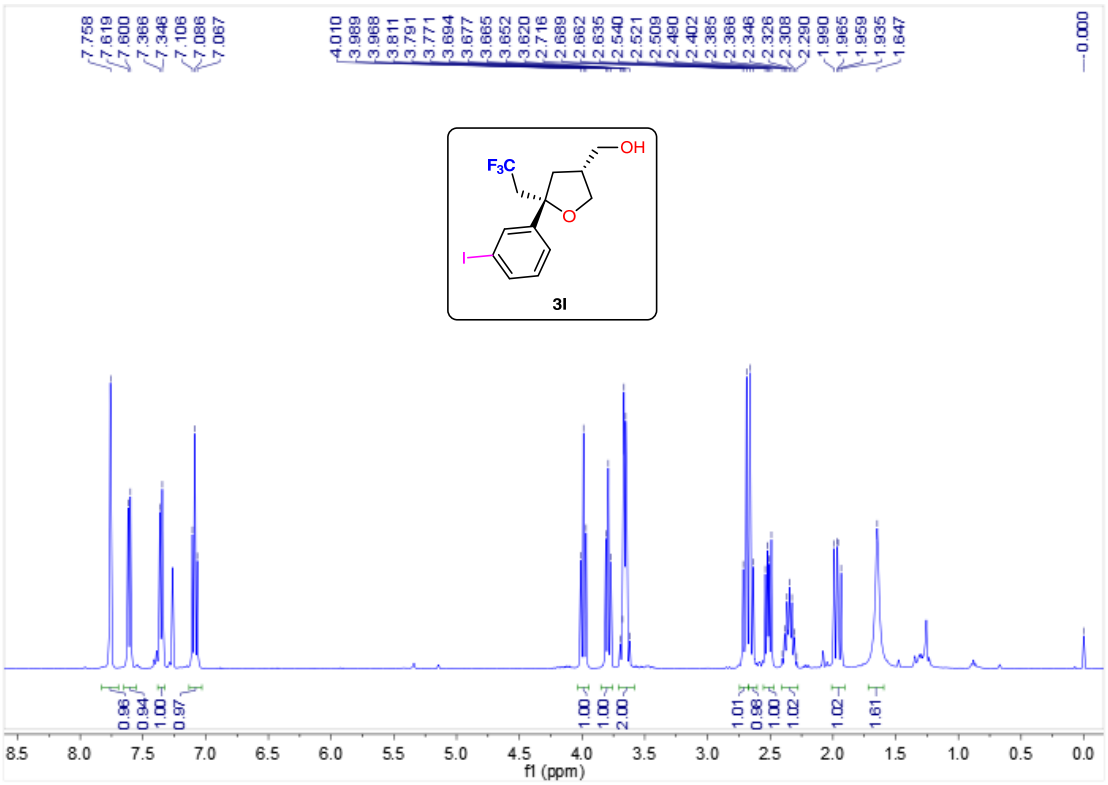

164.078
161.635
146.471
146.409
129.917
129.837
126.493
123.730
120.921
120.892
114.331
114.121
112.728
112.501
83.127
83.108
83.090
77.318
77.000
76.682
69.815
64.226
45.708
45.442
45.175
44.908
41.180
40.754
F3C
OH
O
F
3G
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

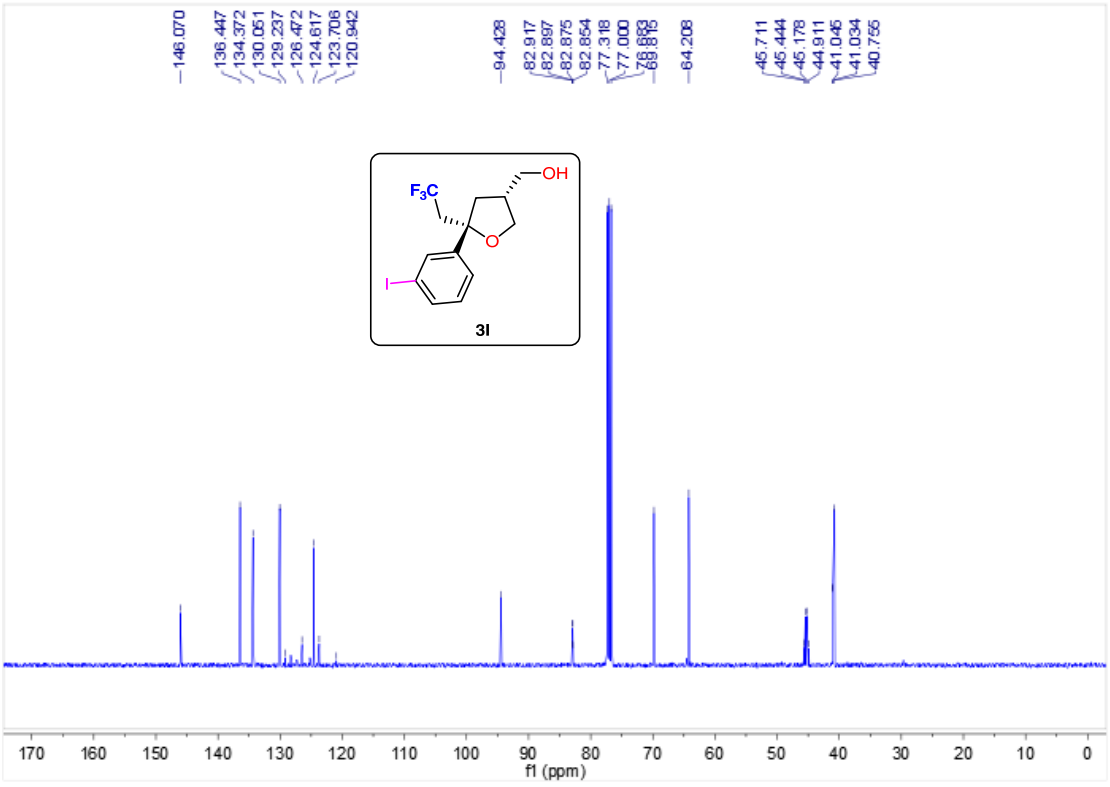

-60.470
-60.498
-60.527
-112.703
-112.718
-112.728
-112.744
-112.752
-112.769
F3C
OH
O
F
3G
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

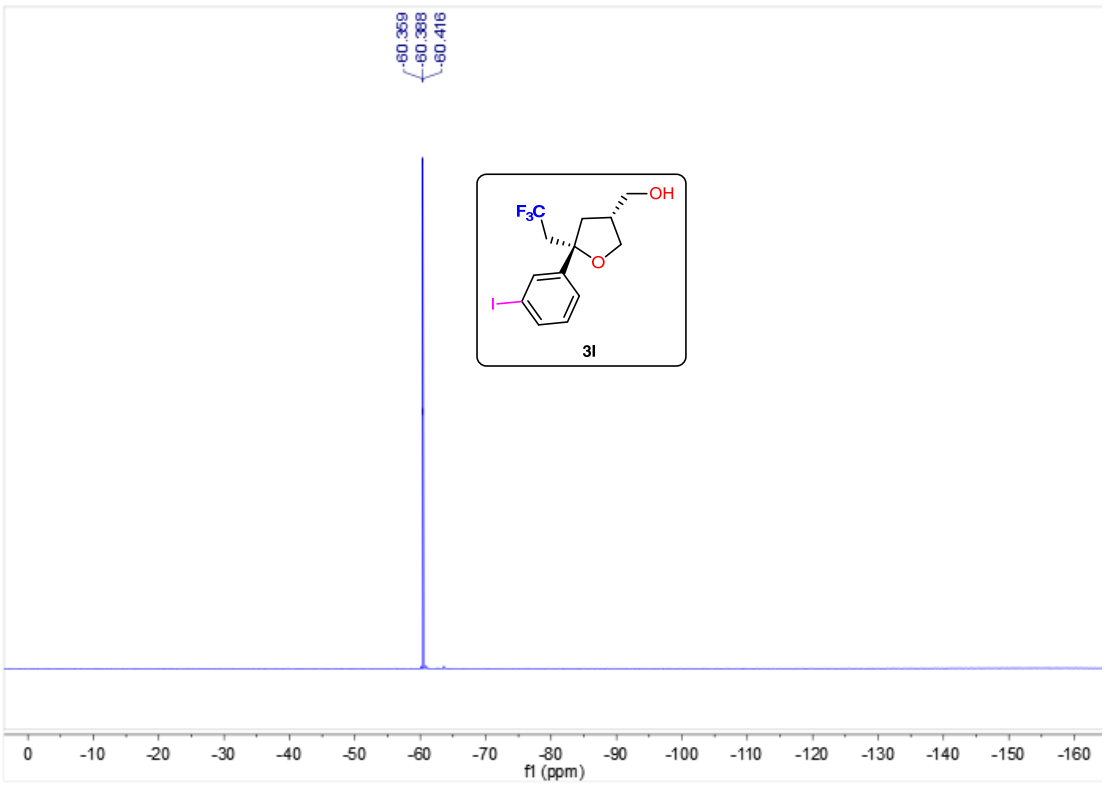

F3C
OH
O
F
3H
2.02
1.97
1.00
1.01
2.04
1.07
1.02
1.04
1.04
1.04
0.96
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

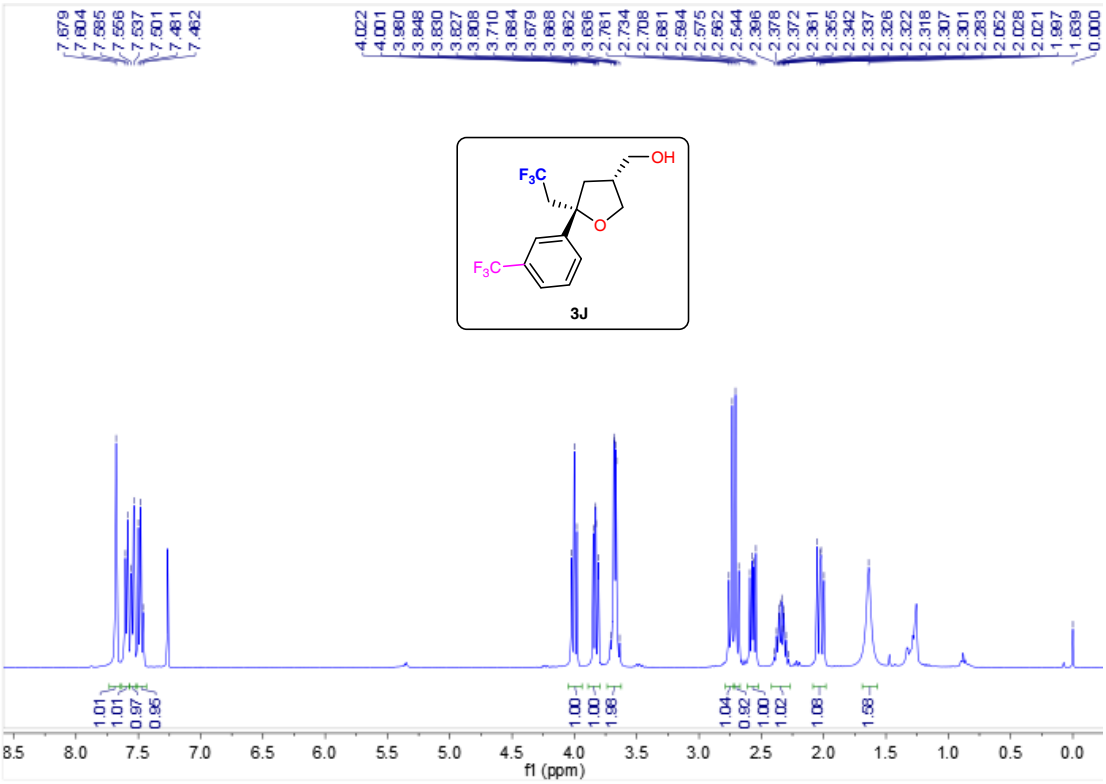

163.218
160.773
139.056
139.025
127.113
127.033
126.547
123.783
115.234
115.021
83.123
83.101
83.079
77.318
77.000
76.683
69.607
64.383
46.016
45.751
45.488
45.222
41.123
40.818
OH
F3C
O
F
3H
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

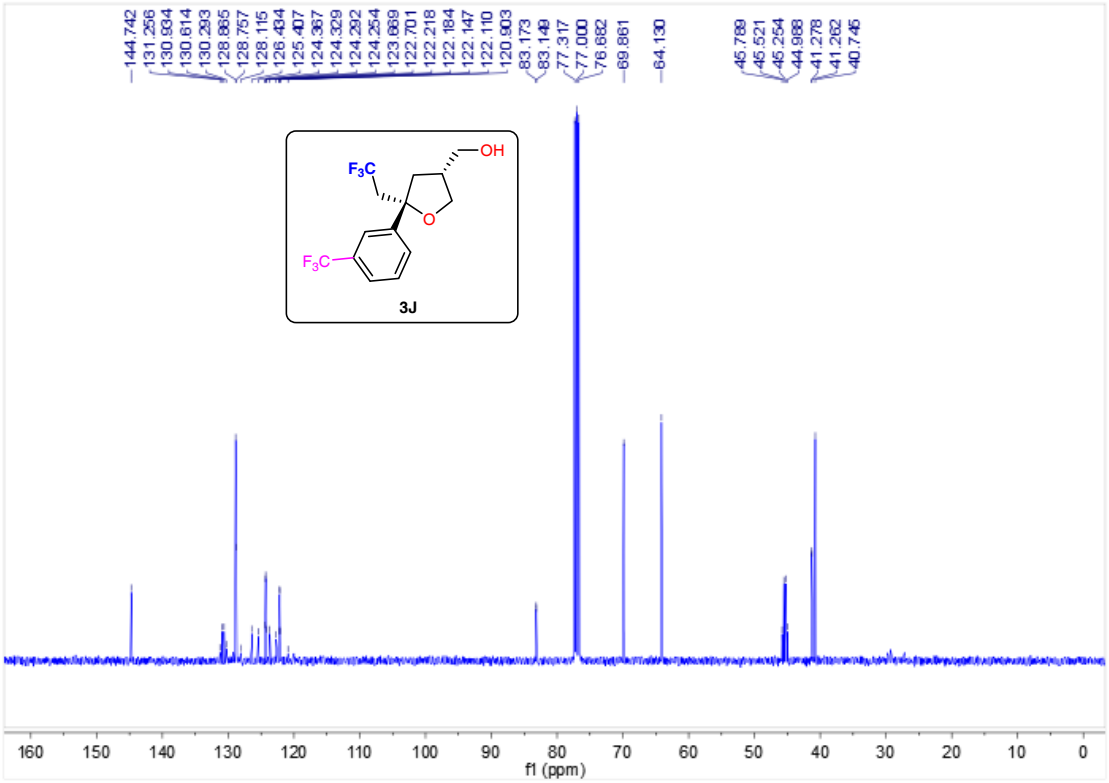

60.486
60.514
60.543
115.597
115.606
115.620
115.629
115.635
115.643
OH
F3C
O
F
3H
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


7.758
7.619
7.600
7.366
7.348
7.106
7.086
7.067
4.010
3.989
3.968
3.811
3.791
3.771
3.694
3.677
3.665
3.652
3.620
2.716
2.689
2.662
2.635
2.540
2.521
2.509
2.490
2.402
2.385
2.366
2.346
2.328
2.308
2.290
1.990
1.965
1.959
1.935
1.647
0.000
F3C
OH
O
I
3l
0.96
0.94
1.00
0.97
1.00
1.00
2.00
1.01
0.98
1.00
1.02
1.02
1.61
f1 (ppm)


146.070
136.447
134.372
130.051
129.237
126.472
124.617
123.706
120.942
94.428
82.917
82.897
82.875
82.854
77.318
77.000
76.683
69.816
64.208
45.711
45.444
45.178
44.911
41.045
41.034
40.755
F3C
OH
O
I
3l
f1 (ppm)


60.359
60.388
60.416
OH
F3C
O
I
3I
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)


7.679
7.604
7.585
7.556
7.537
7.501
7.481
7.462
4.022
4.001
3.980
3.848
3.830
3.827
3.808
3.710
3.684
3.679
3.668
3.662
3.636
2.761
2.734
2.708
2.681
2.594
2.575
2.562
2.544
2.396
2.378
2.372
2.361
2.355
2.342
2.337
2.326
2.322
2.318
2.307
2.301
2.283
2.052
2.028
2.021
1.997
1.639
0.000
OH
F3C
O
F3C
3J
1.01
1.01
0.97
0.95
1.00
1.00
1.98
1.04
0.92
1.00
1.02
1.08
1.58
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)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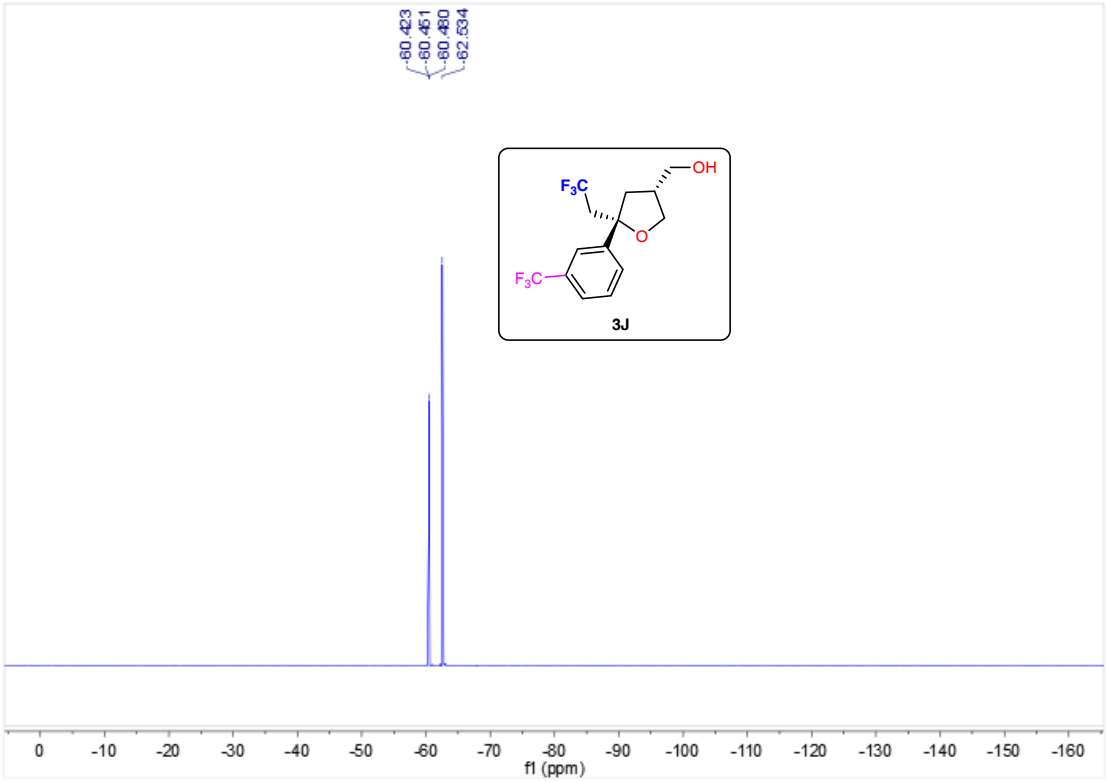
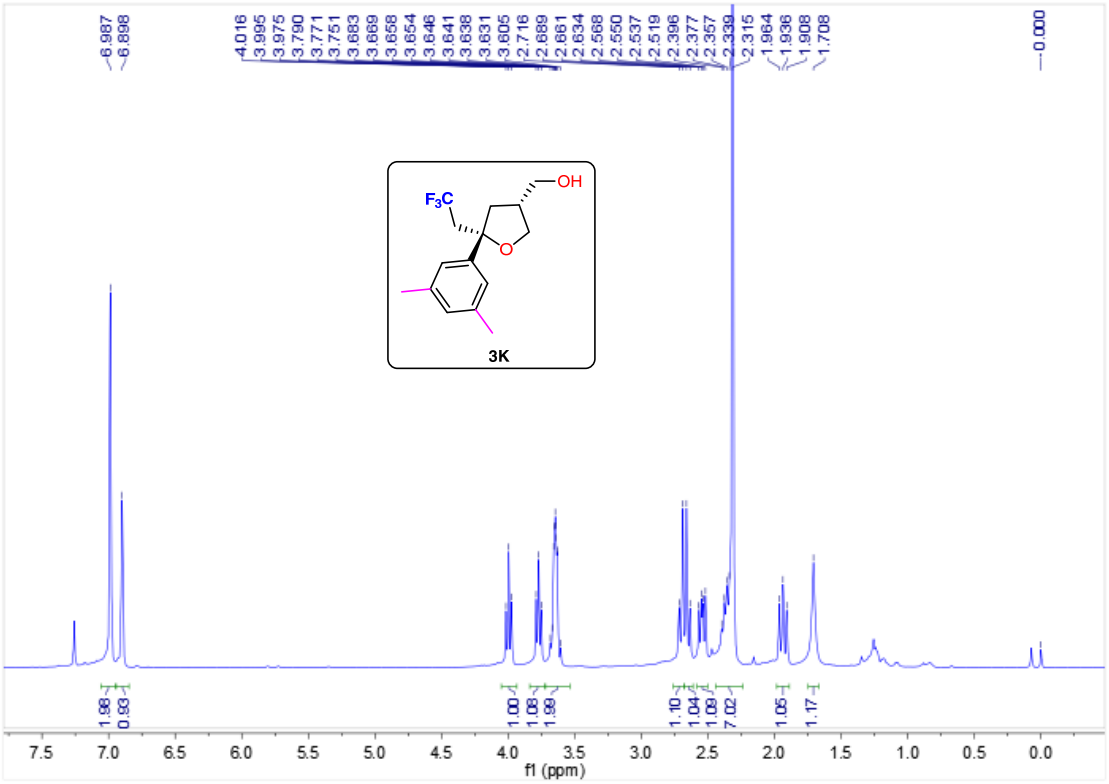
131.256
130.934
130.614
130.293
128.885
128.757
128.115
126.434
125.407
124.367
124.329
124.292
124.254
123.669
122.701
122.218
122.184
122.147
122.110
120.903
83.173
83.149
77.317
77.000
76.682
69.861
64.130
45.789
45.521
45.254
44.988
41.278
41.262
40.745
OH
F3C
O
F3C
3J
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


60.423
60.451
60.480
62.534
OH
F3C
O
F3C
3J
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)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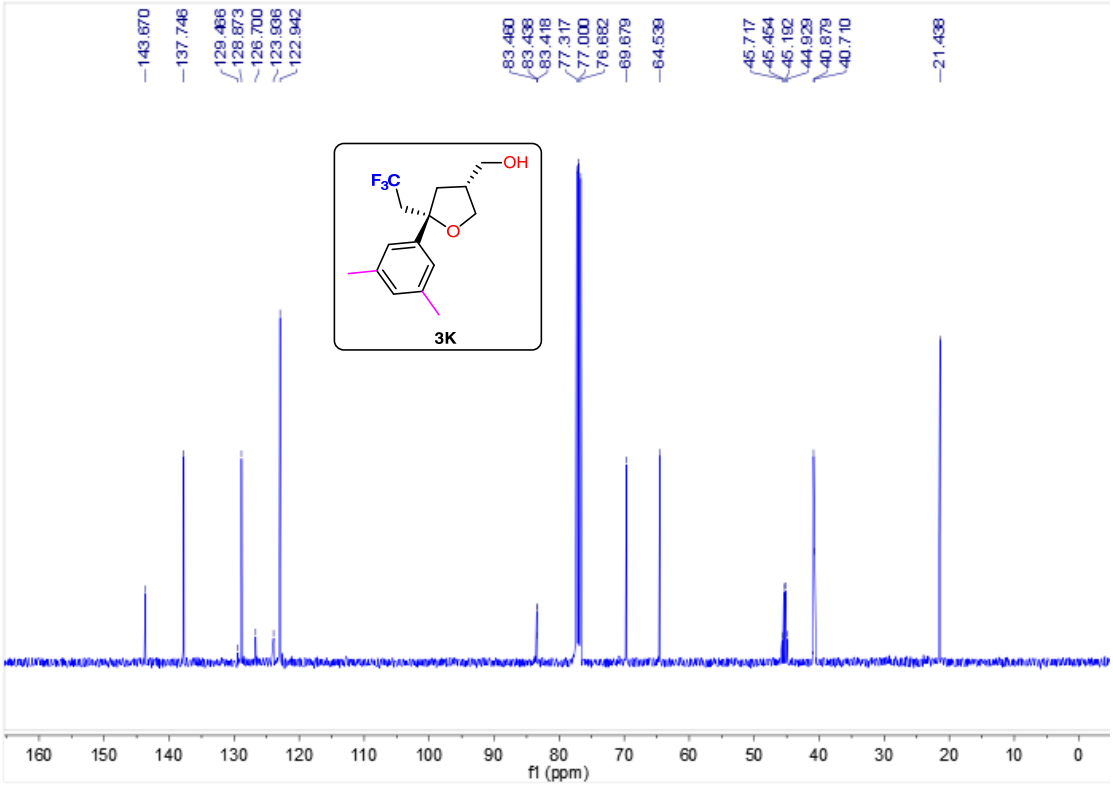
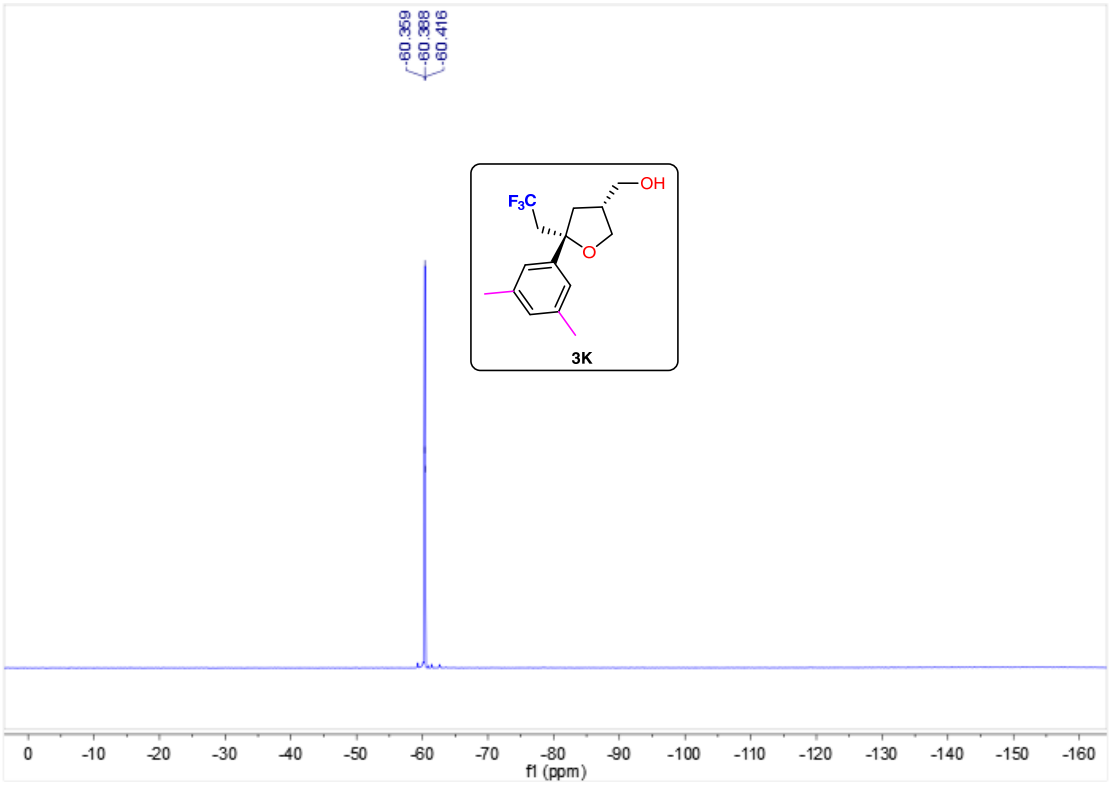
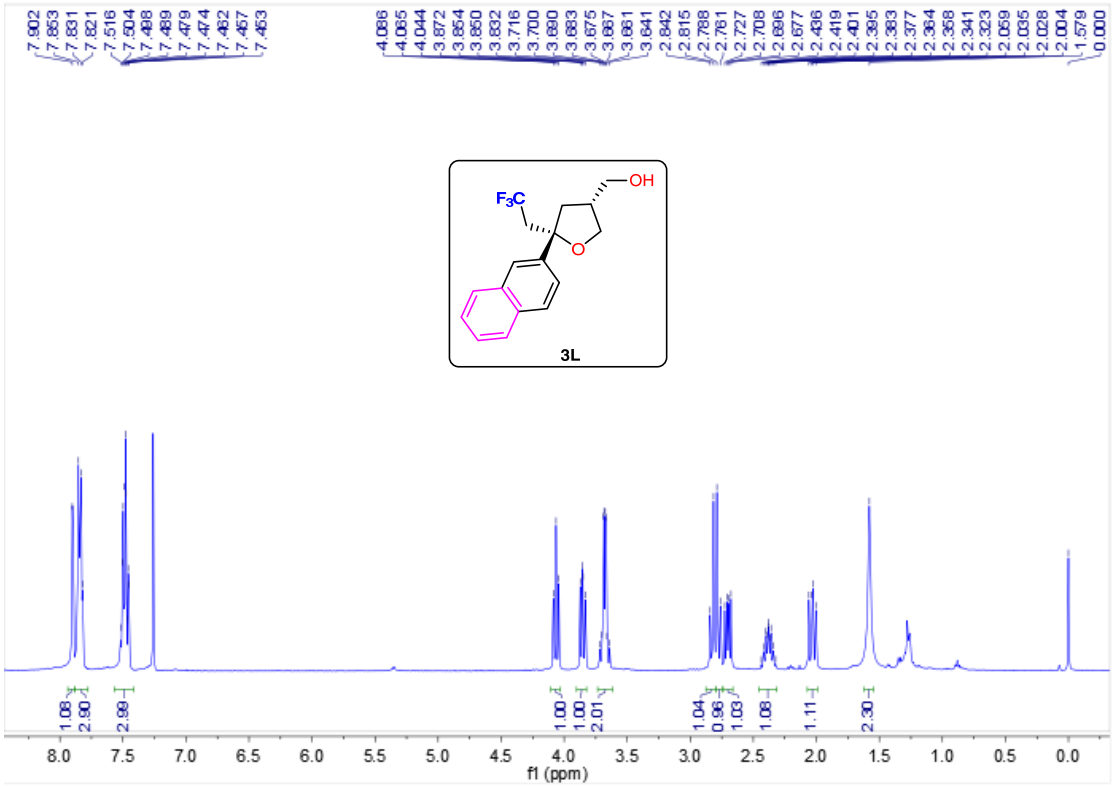
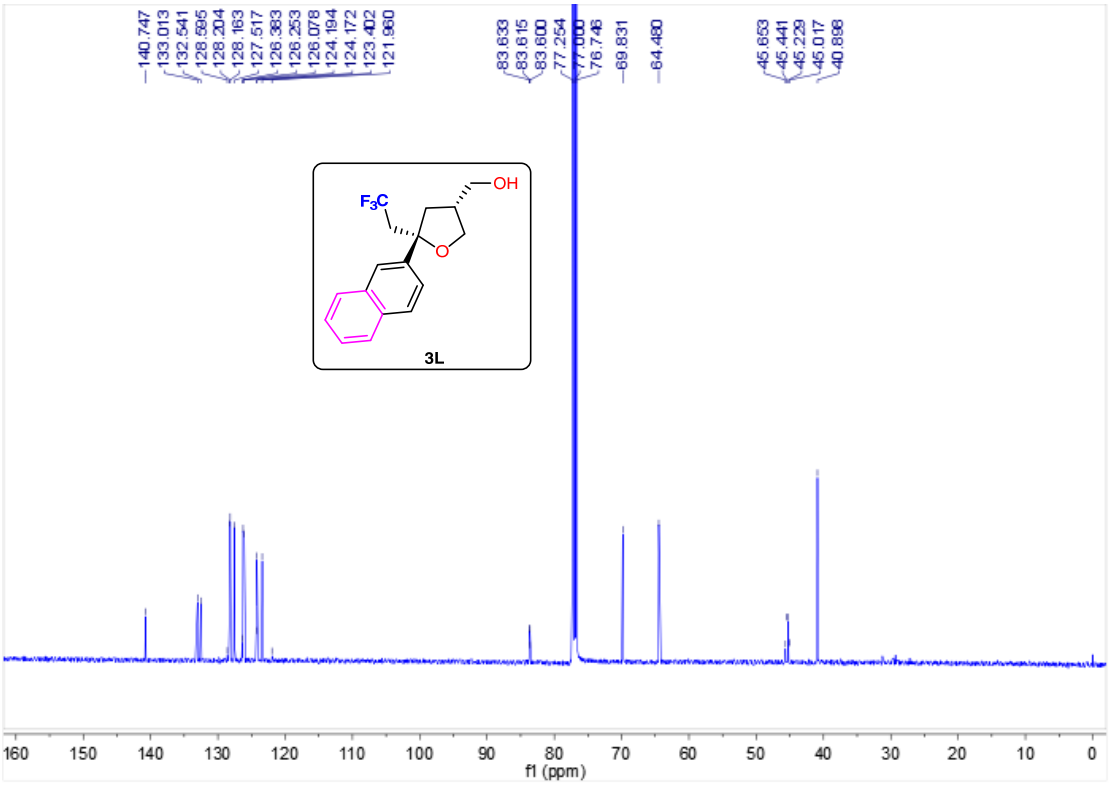
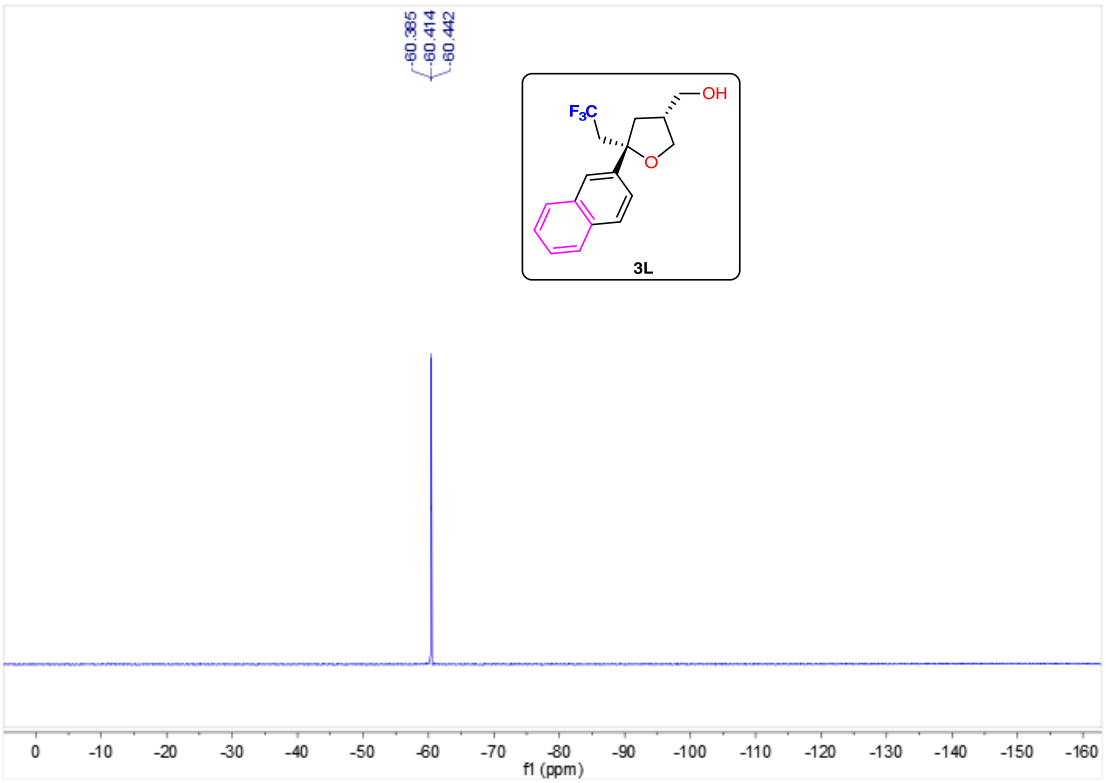
6.898
4.016
3.995
3.975
3.790
3.771
3.751
3.683
3.669
3.658
3.654
3.646
3.641
3.638
3.631
3.605
2.716
2.689
2.661
2.634
2.568
2.550
2.537
2.519
2.396
2.377
2.357
2.339
2.315
1.964
1.936
1.908
1.708
0.000
OH
F3C
O
3K
1.98
0.93
1.00
1.08
1.99
1.10
1.04
1.08
7.02
1.05
1.17
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

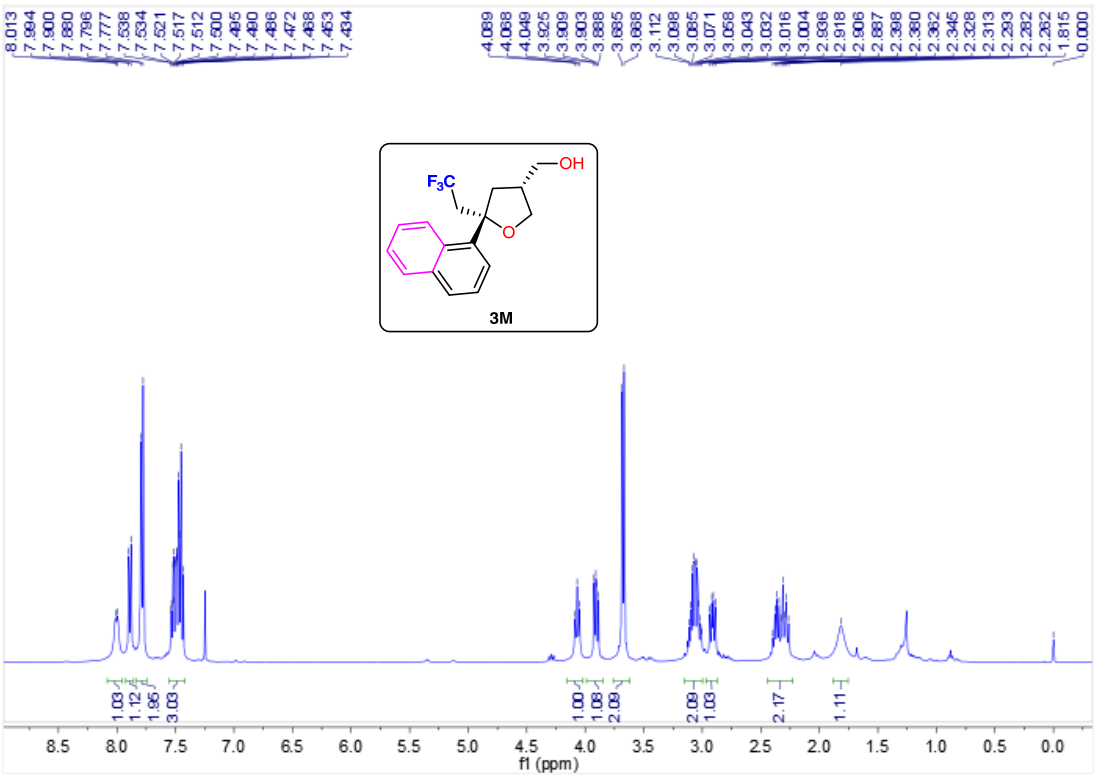

143.670
137.746
129.486
128.873
126.700
123.936
122.942
83.460
83.438
83.418
77.317
77.000
76.682
69.679
64.539
45.717
45.454
45.192
44.929
40.879
40.710
21.438
OH
F3C
O
3K
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

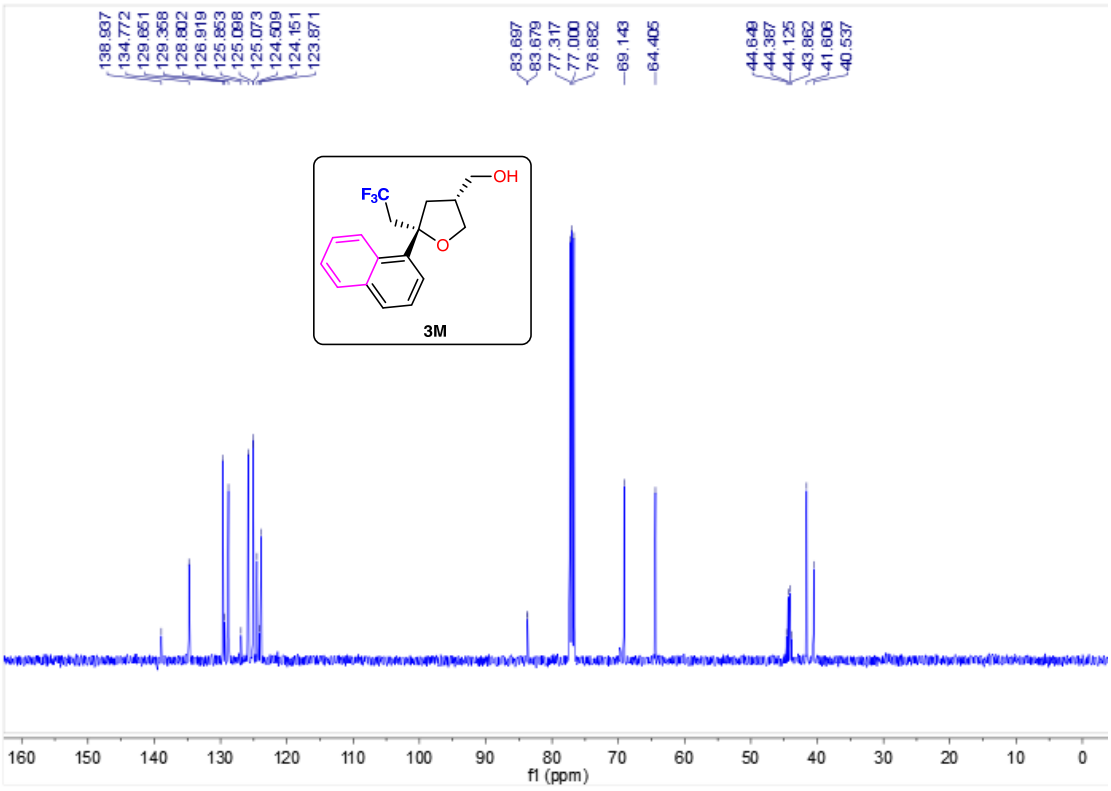

-60.359
-60.388
-60.416
F3C
OH
O
3K
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

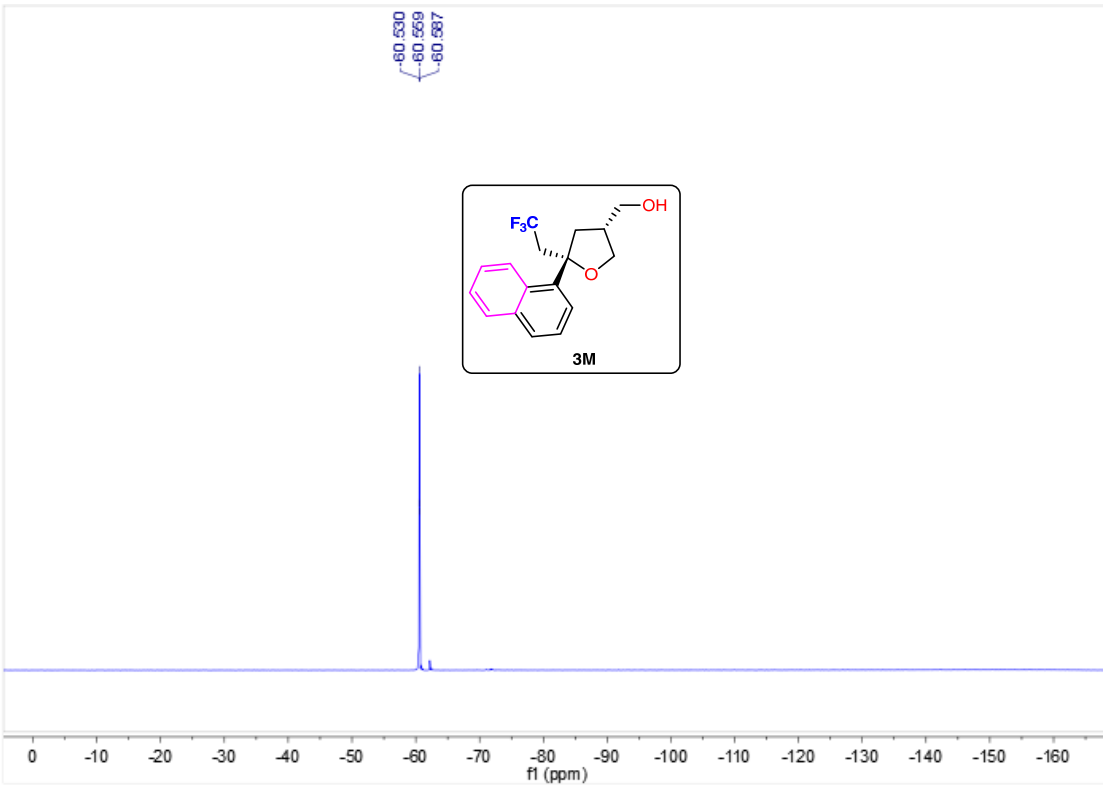

7.902
7.853
7.831
7.821
7.516
7.504
7.498
7.489
7.479
7.474
7.462
7.457
7.453
4.066
4.055
4.044
3.872
3.854
3.850
3.832
3.716
3.700
3.690
3.683
3.675
3.667
3.661
3.641
2.842
2.815
2.788
2.761
2.727
2.708
2.696
2.677
2.436
2.419
2.401
2.395
2.383
2.377
2.364
2.358
2.341
2.323
2.059
2.035
2.028
2.004
1.579
0.000
F3C
OH
O
3L
1.08
2.90
2.99
1.00
1.00
2.01
1.04
0.96
1.03
1.08
1.11
2.30
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)

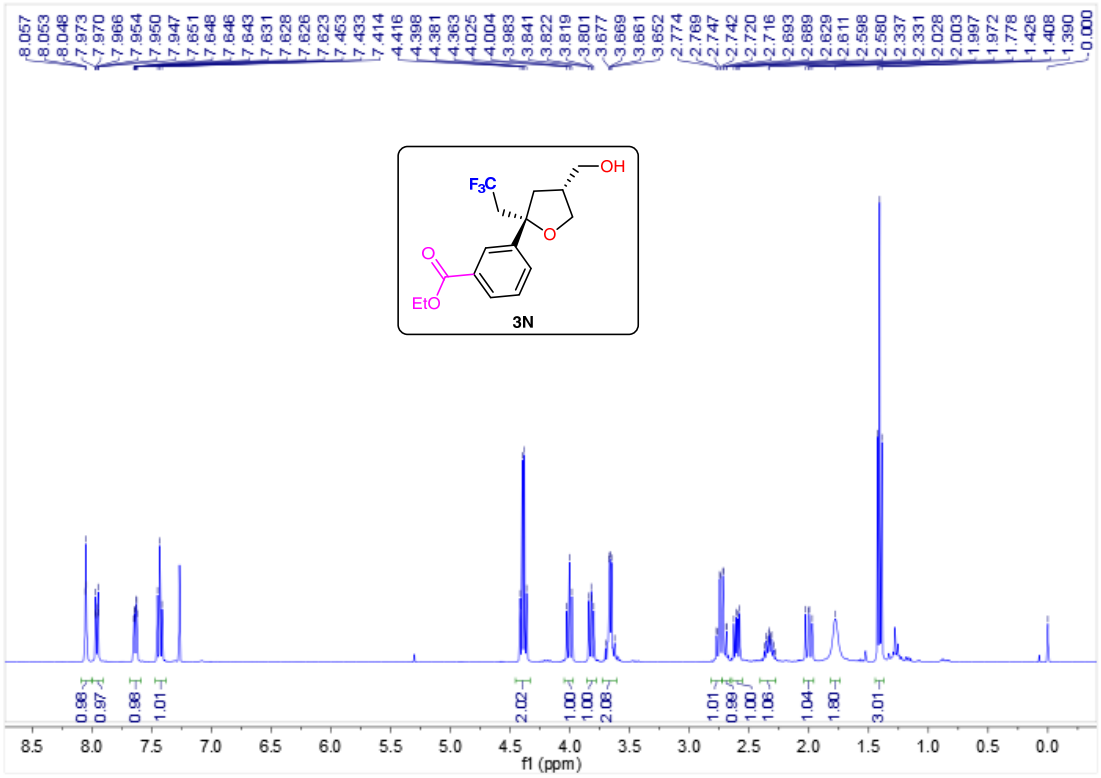

140.747
133.013
132.541
128.595
128.204
128.163
127.517
126.383
126.253
126.078
124.194
124.172
123.402
121.960
83.633
83.615
83.600
77.254
77.000
76.746
69.831
64.480
45.653
45.441
45.229
45.017
40.898
F3C
OH
O
3L
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

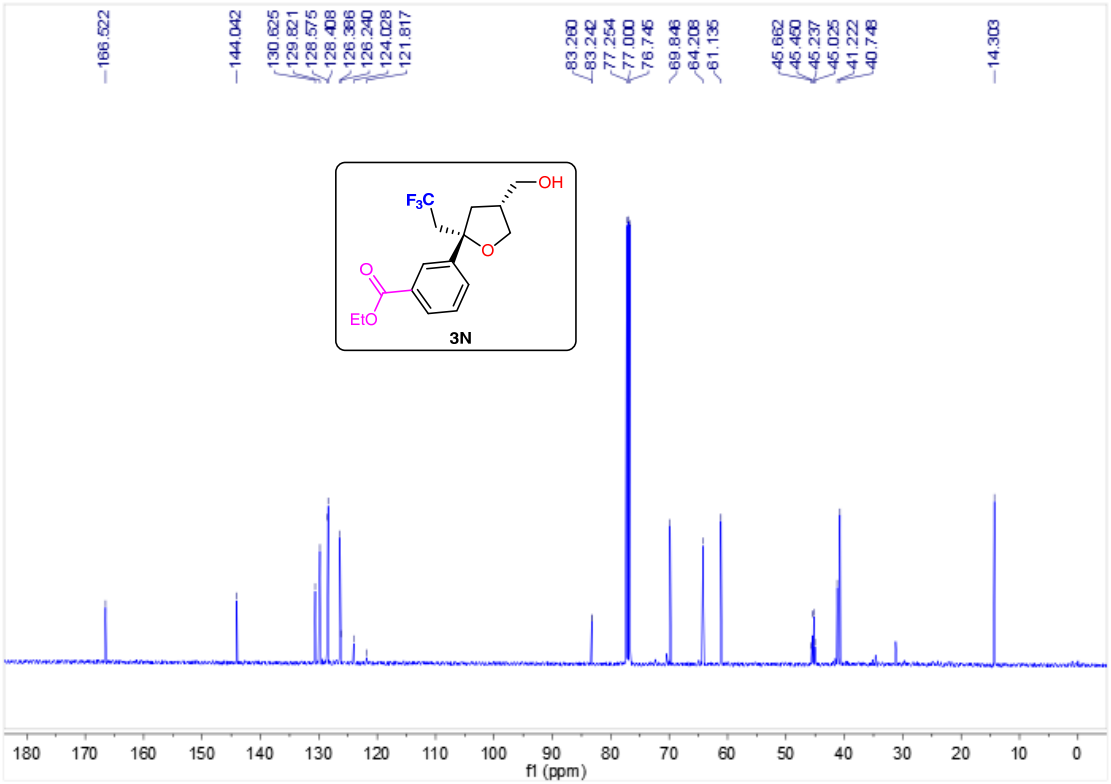

60.385
60.414
60.442
F3C
OH
O
3L
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

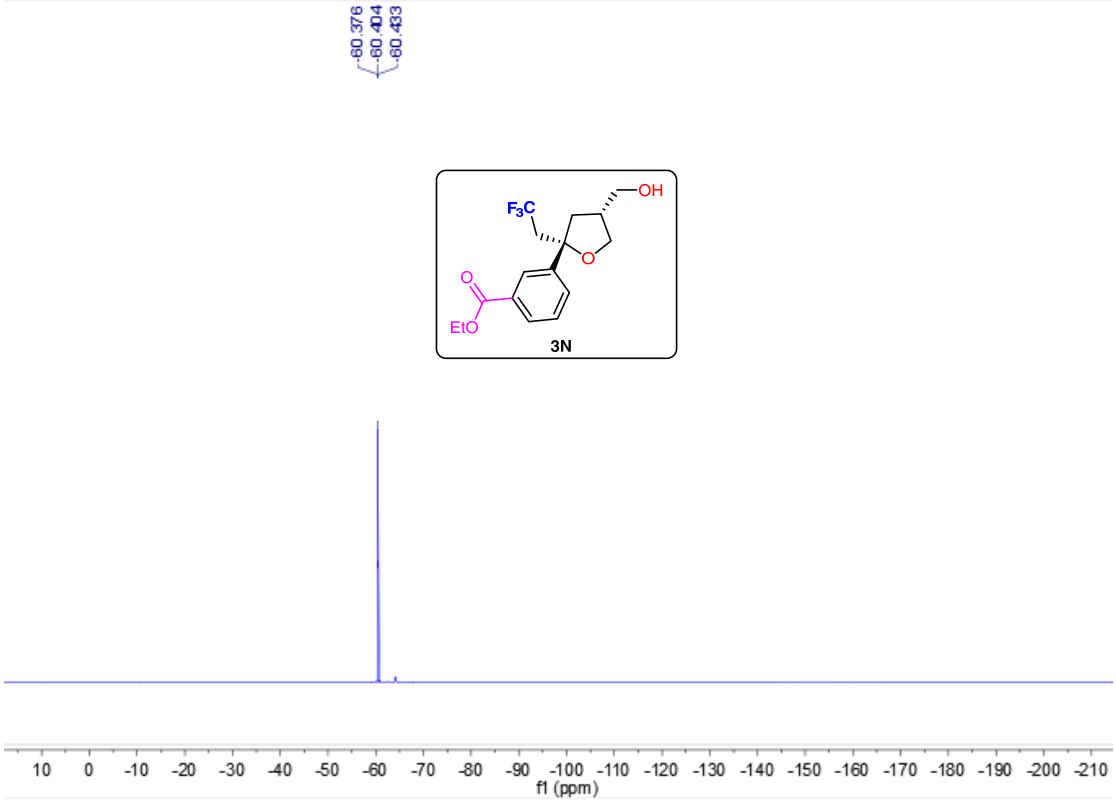

8.013
7.994
7.900
7.880
7.796
7.777
7.538
7.534
7.521
7.517
7.512
7.500
7.495
7.490
7.486
7.472
7.468
7.463
7.434
4.069
4.058
4.049
3.925
3.909
3.903
3.888
3.685
3.668
3.112
3.098
3.085
3.071
3.058
3.043
3.032
3.016
3.004
2.936
2.918
2.906
2.887
2.398
2.380
2.362
2.345
2.328
2.313
2.293
2.282
2.262
1.815
0.000
F3C
OH
O
3M
1.03
1.12
1.95
3.03
1.00
1.08
2.09
2.09
1.03
2.17
1.11
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)

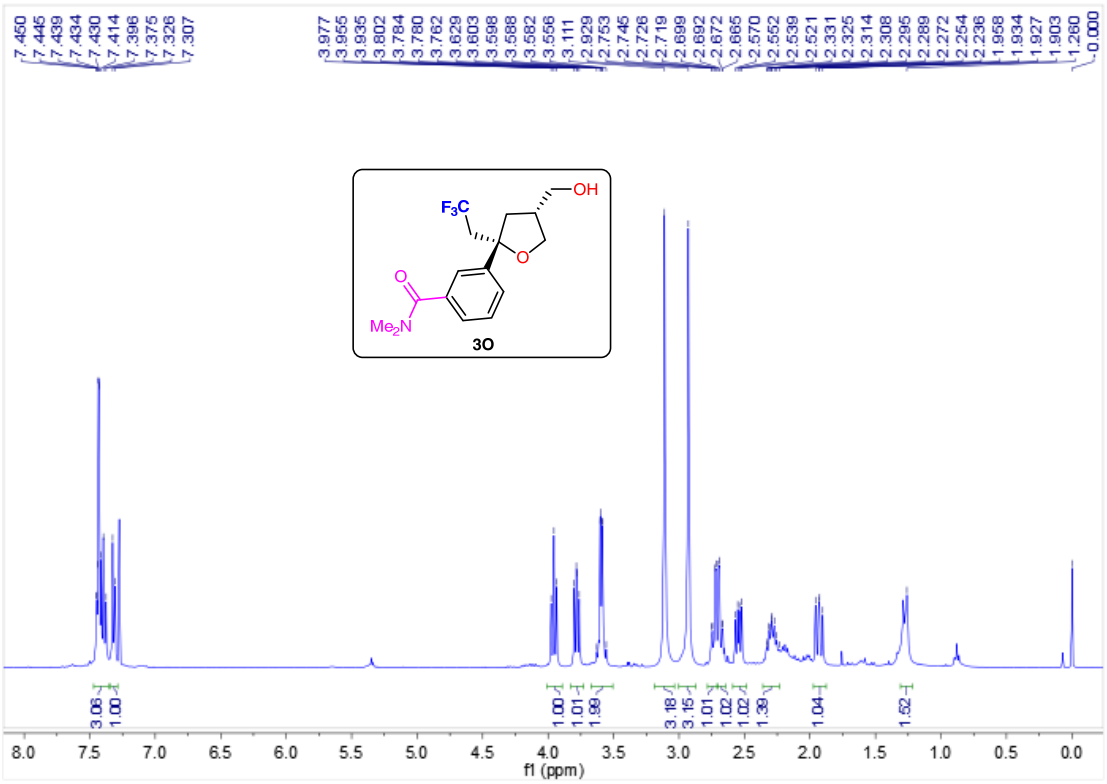

138.937
134.772
129.651
129.368
128.802
126.919
125.853
125.088
125.073
124.509
124.151
123.871
83.697
83.679
77.317
77.000
76.682
69.143
64.405
44.649
44.387
44.125
43.862
41.606
40.537
F3C
OH
O
3M
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

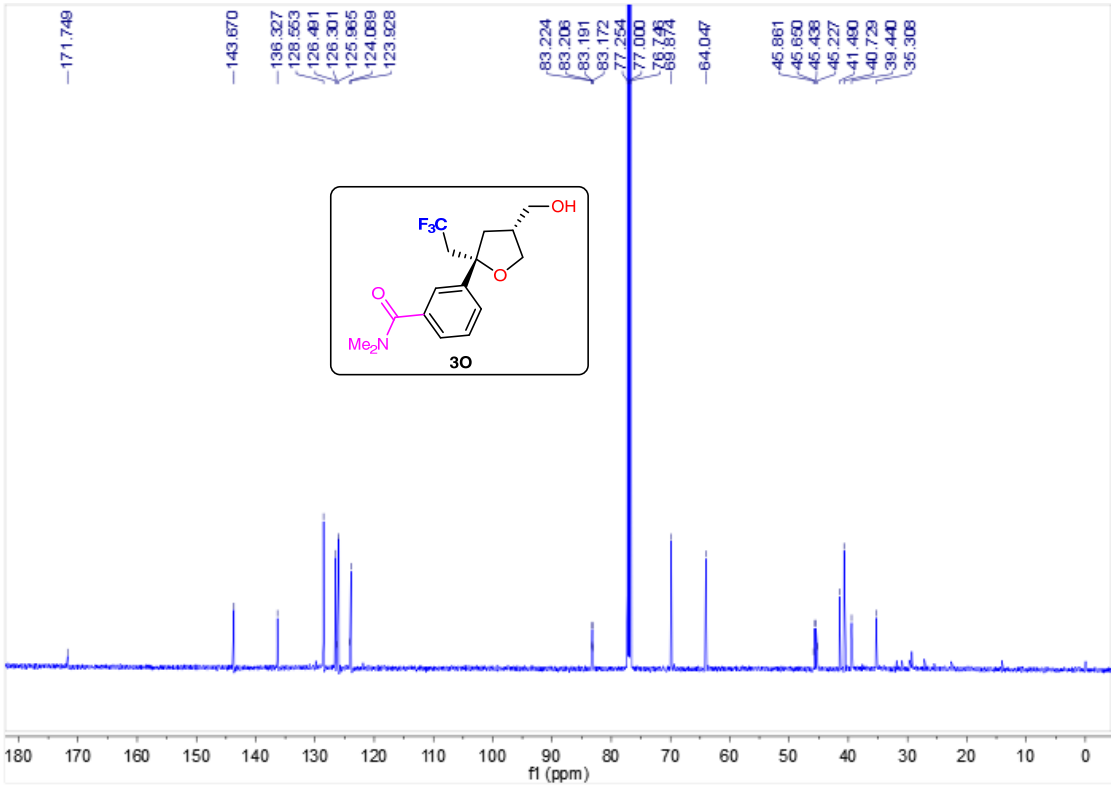

-60.530
-60.559
-60.587
F3C
OH
O
3M
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

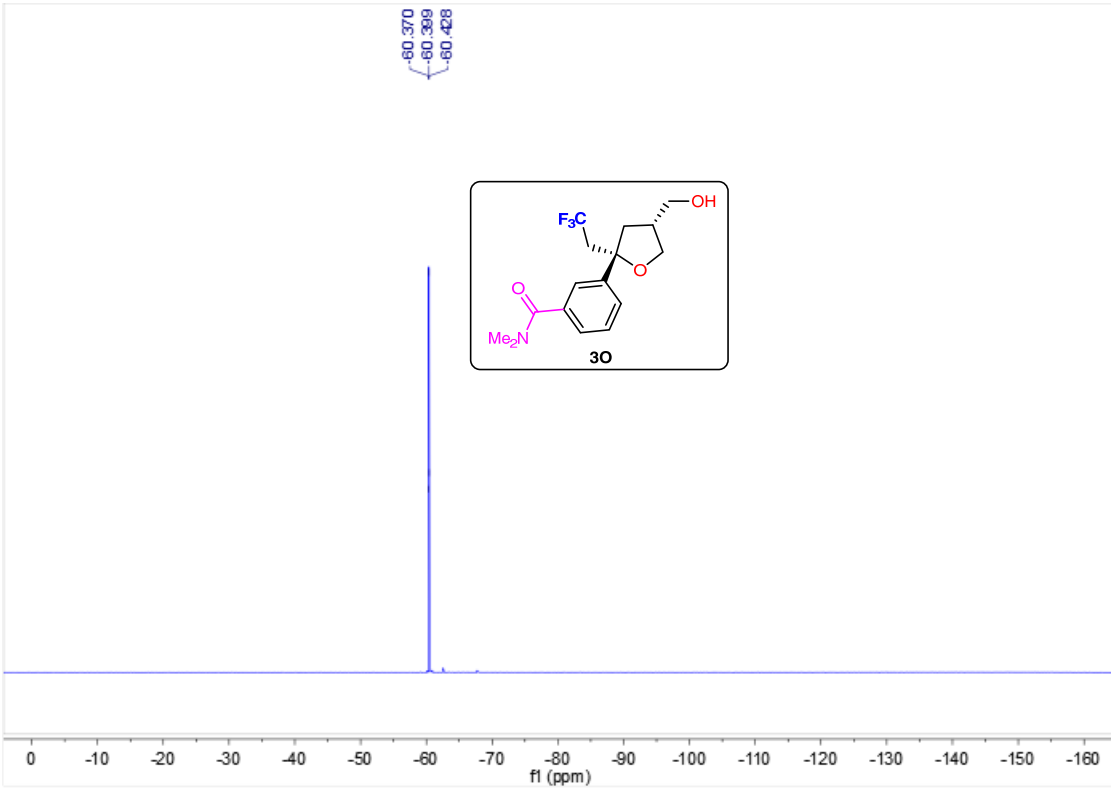

F3C
OH
O
O
EtO
3N
0.98
0.97
0.98
1.01
2.02
1.00
1.00
2.08
1.01
0.99
1.00
1.06
1.04
1.80
3.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

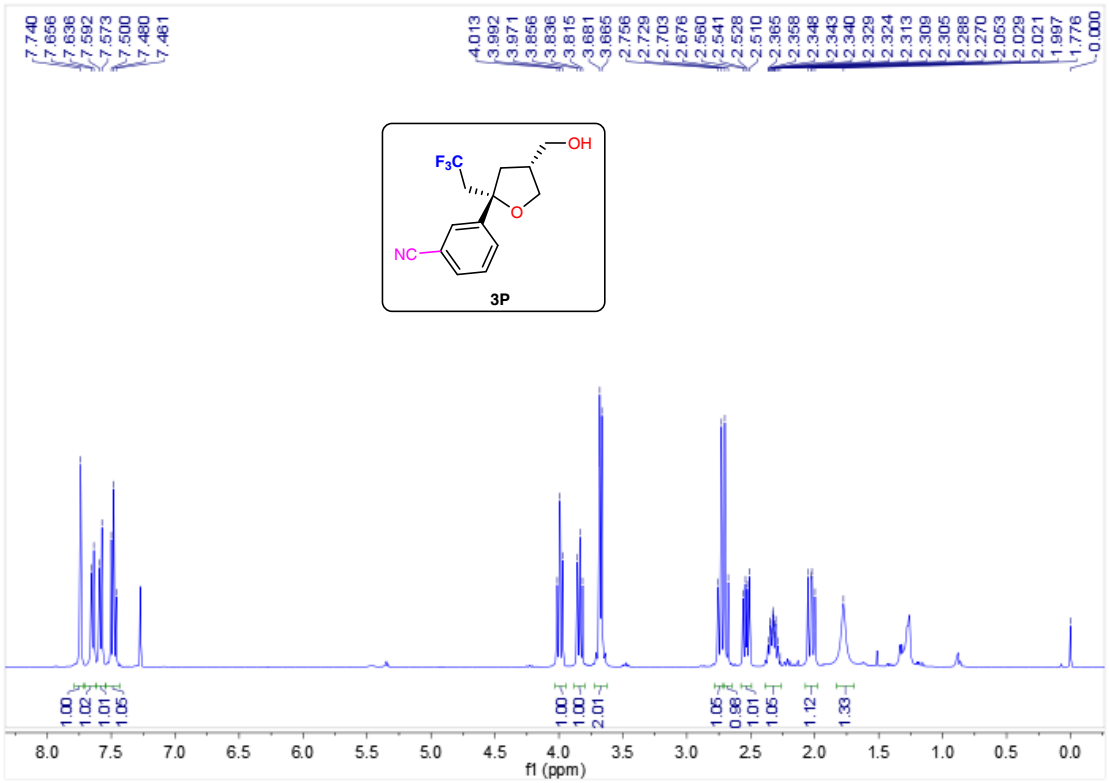

166.522
144.042
130.625
129.821
128.575
128.408
126.386
126.240
124.028
121.817
83.280
83.242
77.254
77.000
76.745
69.846
64.208
61.135
45.662
45.490
45.237
45.025
41.222
40.748
14.303
OH
F3C
O
O
EtO
3N
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

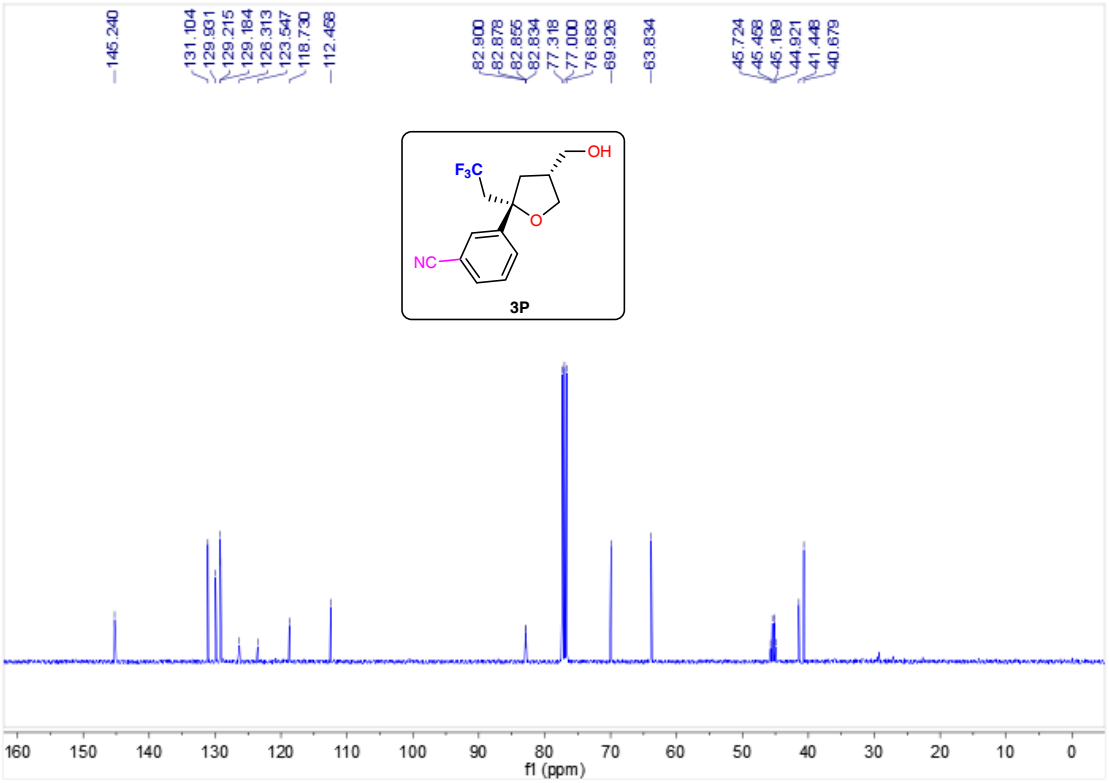

-60.376
-60.404
-60.433
OH
F3C
O
O
EtO
3N
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

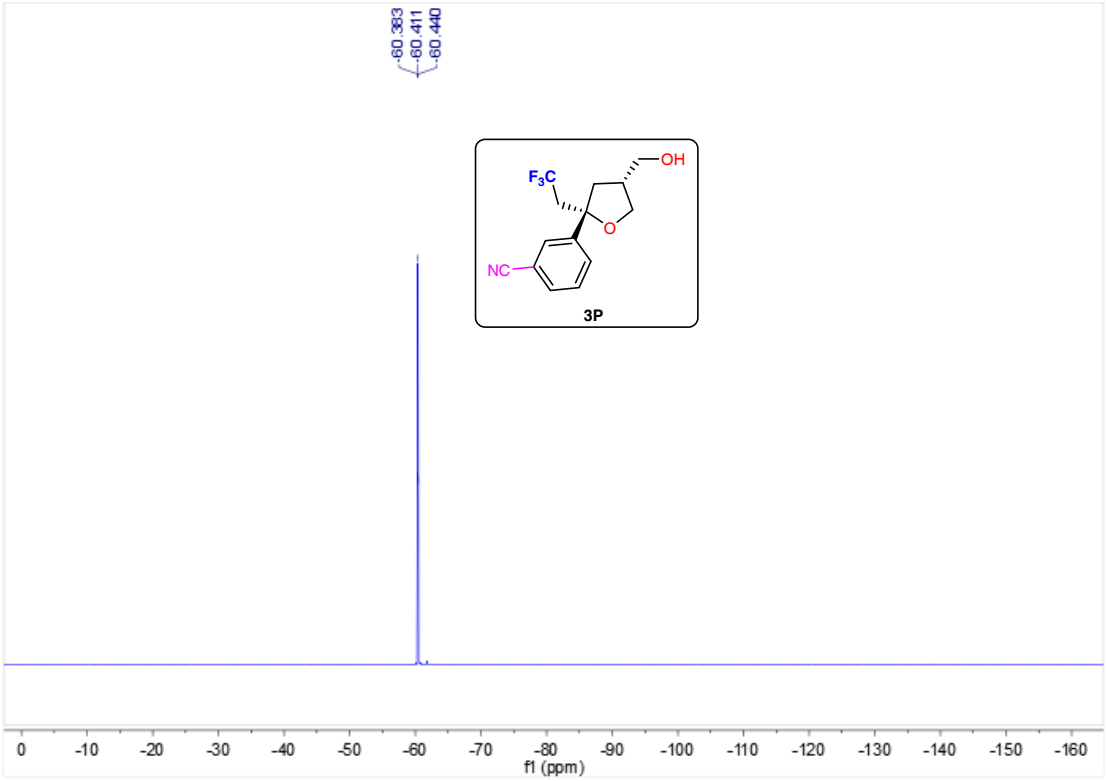

7.450
7.445
7.439
7.434
7.430
7.414
7.396
7.375
7.326
7.307
3.977
3.955
3.935
3.802
3.784
3.780
3.762
3.629
3.603
3.598
3.588
3.582
3.556
3.111
2.929
2.753
2.745
2.726
2.719
2.699
2.692
2.672
2.665
2.570
2.552
2.539
2.521
2.331
2.325
2.314
2.308
2.295
2.289
2.272
2.254
2.236
1.958
1.934
1.927
1.903
1.260
0.000
F3C
OH
O
Me2N
3O
3.06
1.00
1.00
1.01
1.99
3.18
3.15
1.01
1.02
1.02
1.39
1.04
1.52
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)

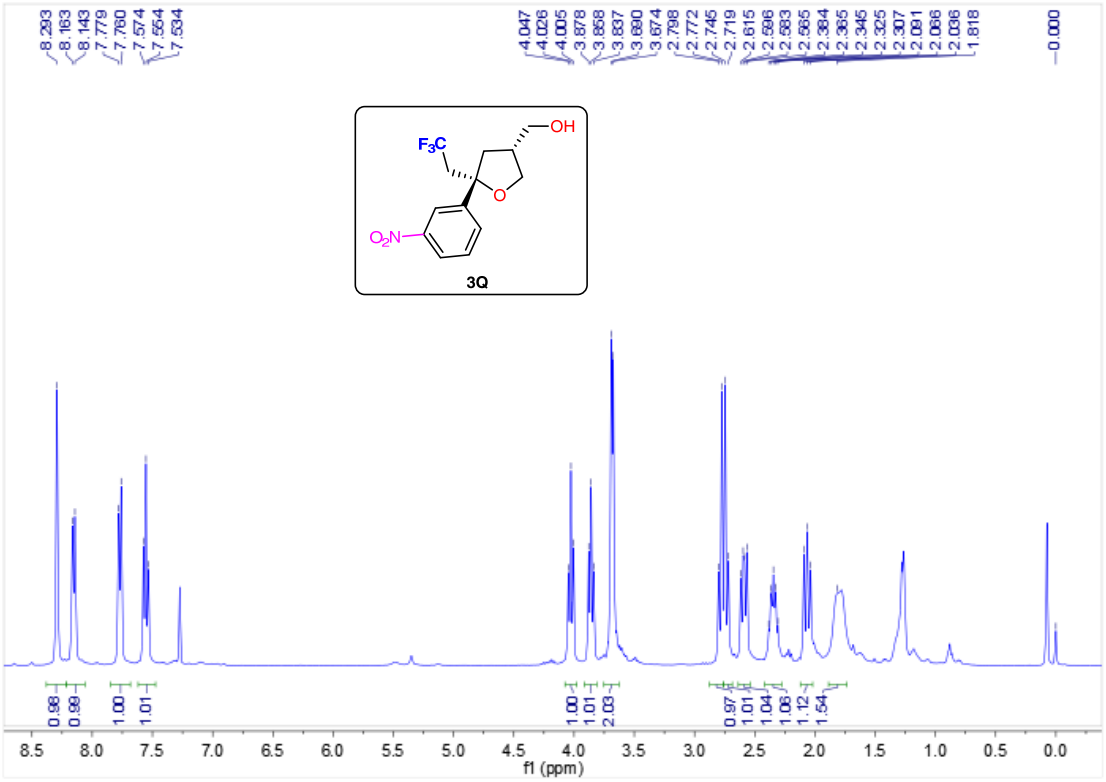

171.749
143.670
136.327
128.553
126.491
126.301
125.965
124.089
123.928
83.224
83.206
83.191
83.172
77.254
77.000
64.047
45.861
45.650
45.438
45.227
41.490
40.729
39.440
35.308
F3C
OH
O
Me2N
3O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

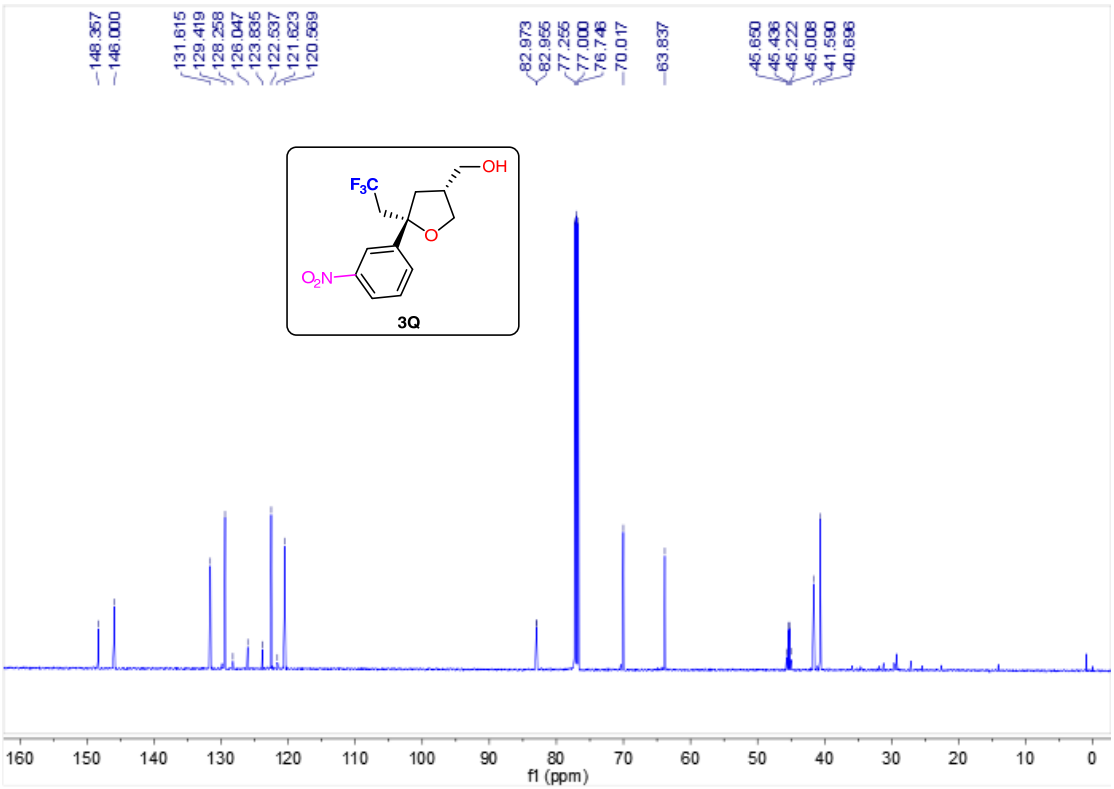

-60.370
-60.399
-60.428
F3C
OH
O
O
Me2N
3O
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

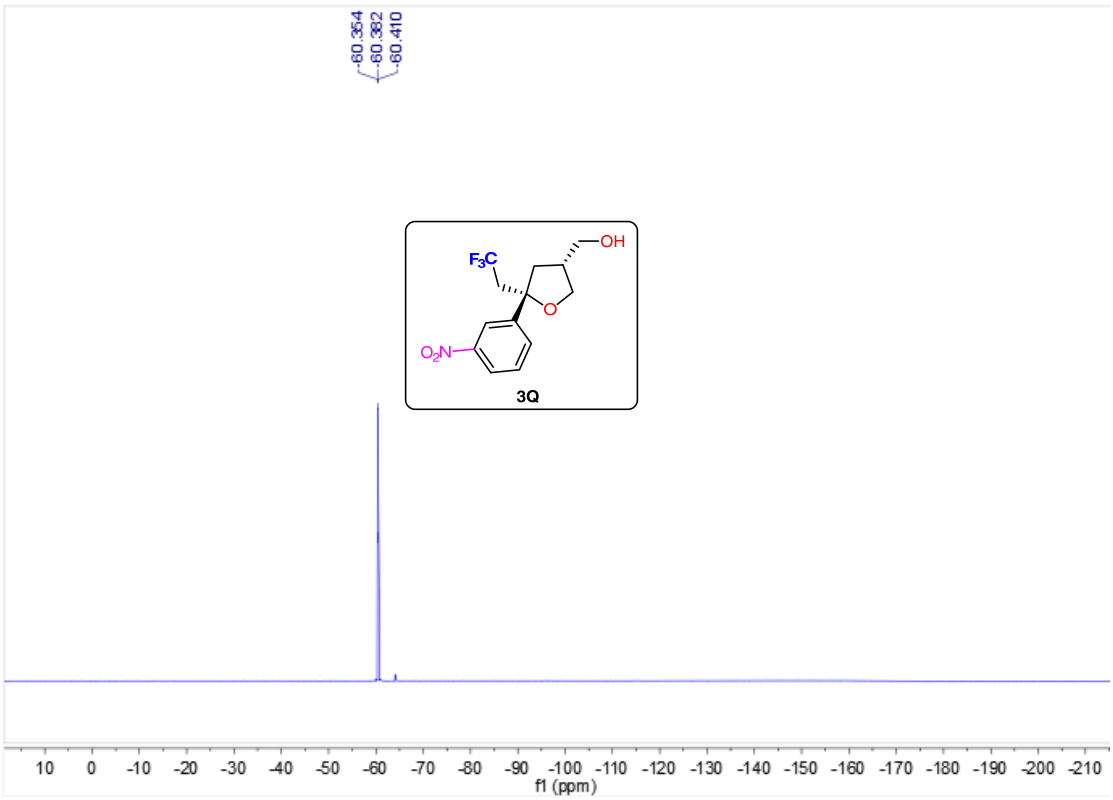

7.740
7.656
7.636
7.592
7.573
7.500
7.480
7.461
4.013
3.992
3.971
3.856
3.836
3.815
3.681
3.665
2.756
2.729
2.703
2.676
2.560
2.541
2.528
2.510
2.365
2.358
2.348
2.343
2.340
2.329
2.324
2.313
2.309
2.305
2.288
2.270
2.053
2.029
2.021
1.997
1.776
0.000
F3C
OH
O
NC
3P
1.00
1.02
1.01
1.05
1.00
1.00
2.01
1.05
0.98
1.01
1.05
1.12
1.33
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)

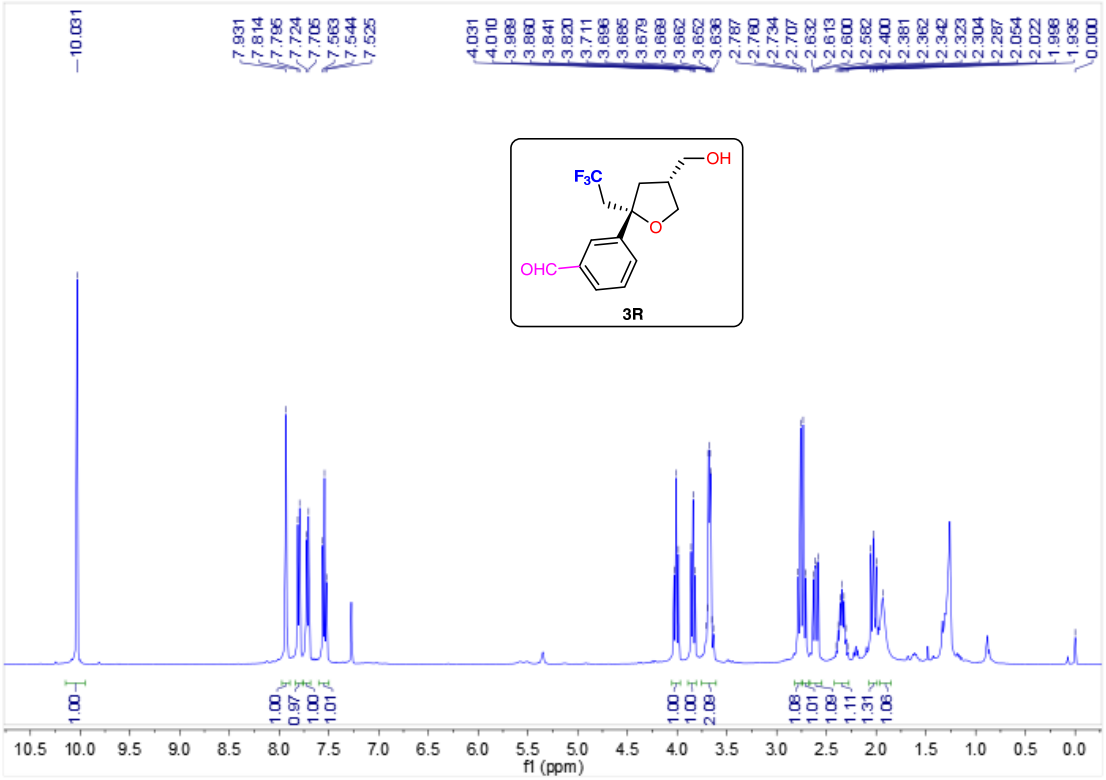

145.240
131.104
129.931
129.215
129.184
126.313
123.547
118.730
112.466
82.900
82.878
82.855
82.834
77.318
77.000
76.683
69.926
63.834
45.724
45.458
45.189
44.921
41.448
40.679
OH
F3C
O
NC
3P
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

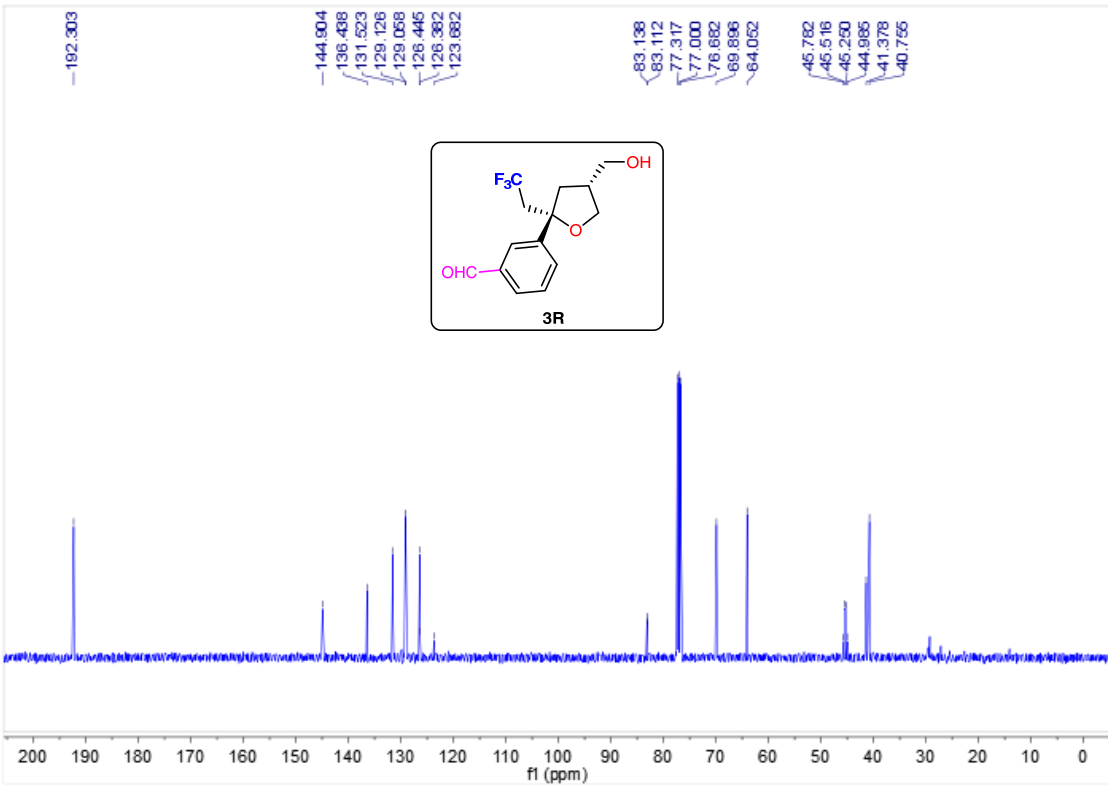

60.383
60.411
60.440
OH
F3C
O
NC
3P
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

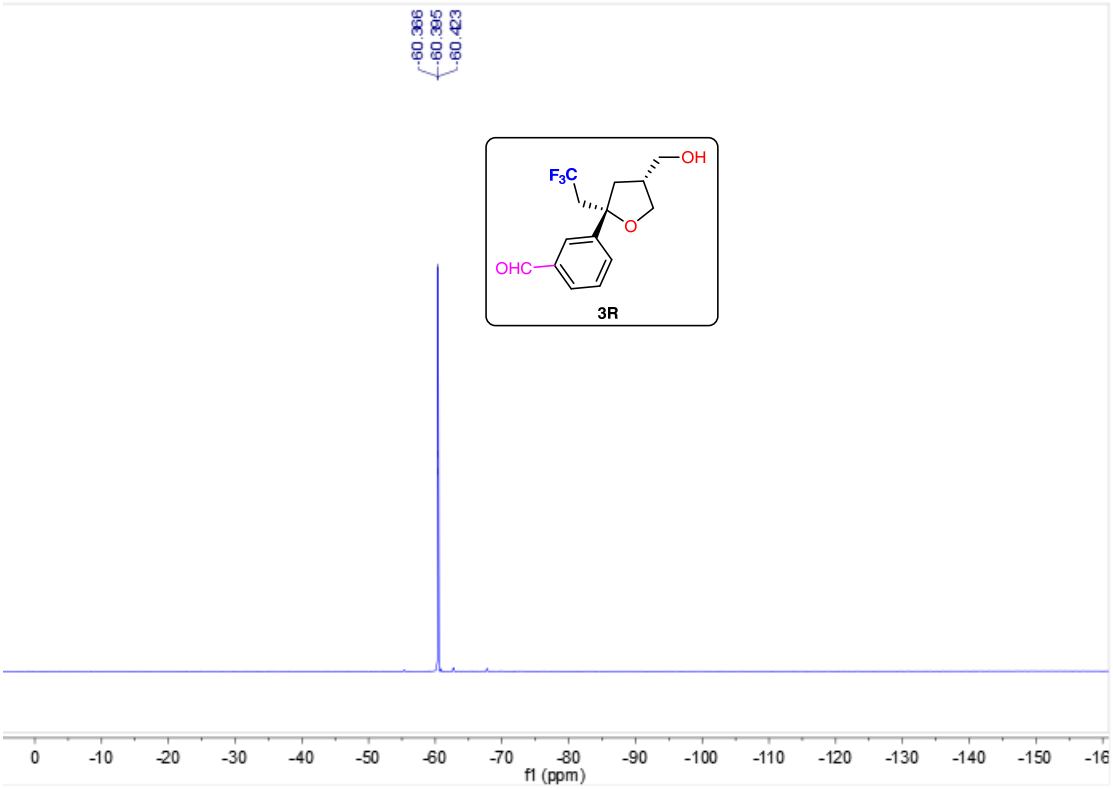

8.293
8.163
8.143
7.779
7.760
7.574
7.564
7.534
4.047
4.026
4.005
3.878
3.858
3.837
3.690
3.674
2.798
2.772
2.745
2.719
2.615
2.596
2.583
2.565
2.384
2.365
2.345
2.325
2.307
2.091
2.066
2.036
1.818
0.000
OH
F3C
O
O2N
3Q
0.98
0.99
1.00
1.01
1.00
1.01
2.03
0.97
1.01
1.04
1.06
1.12
1.54
f1 (ppm)

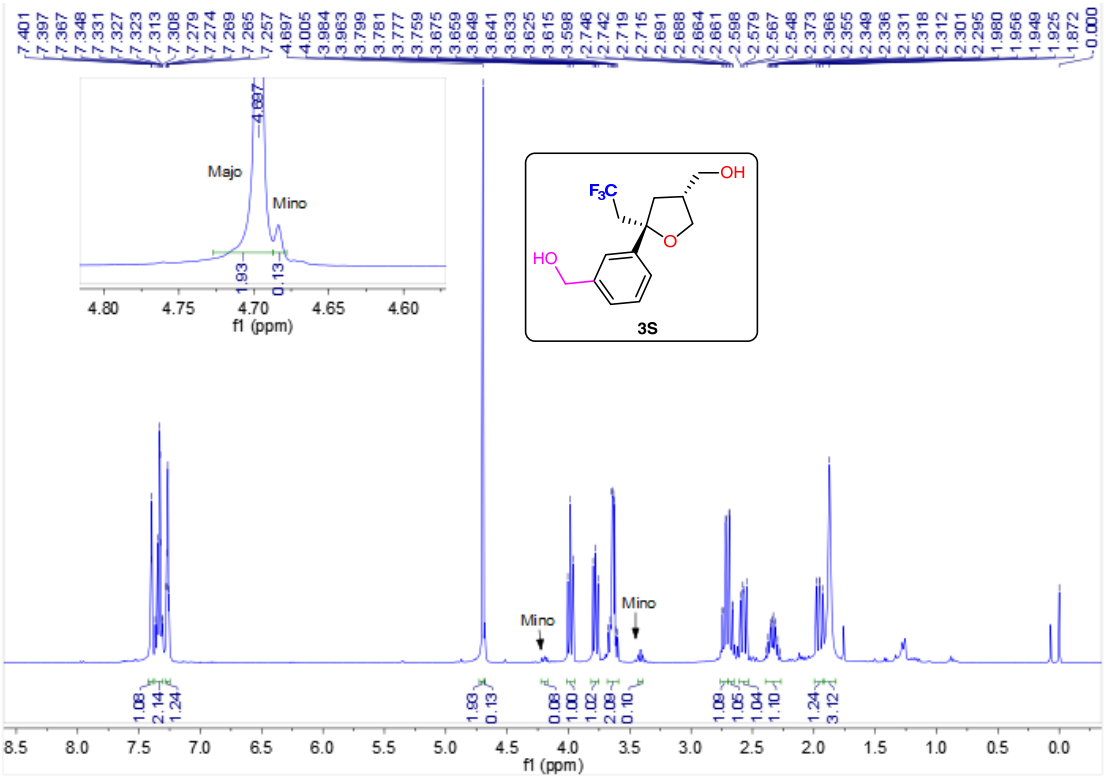

148.357
146.000
131.615
129.419
128.258
126.047
123.835
122.537
121.623
120.569
82.973
82.955
77.255
77.000
76.746
70.017
63.837
46.650
46.436
46.222
46.008
41.590
40.696
OH
F3C
O
O2N
3Q
f1 (ppm)

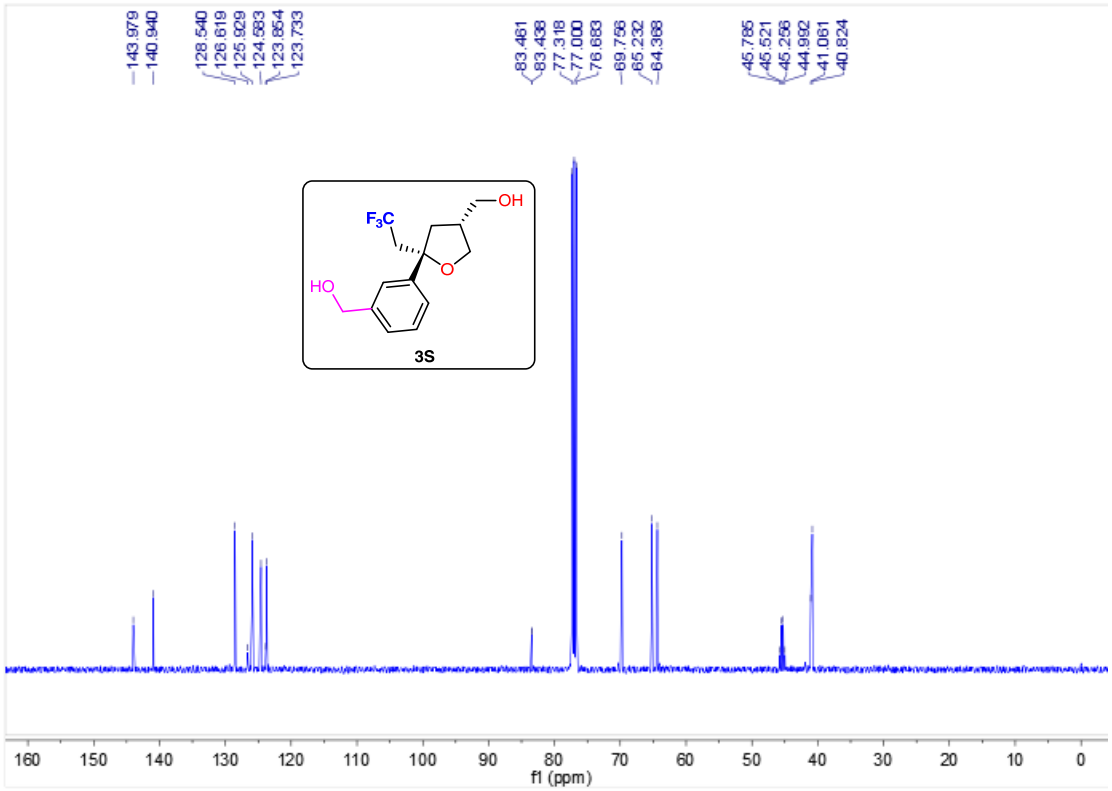

-60.354
-60.382
-60.410
F3C
OH
O
O2N
3Q
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

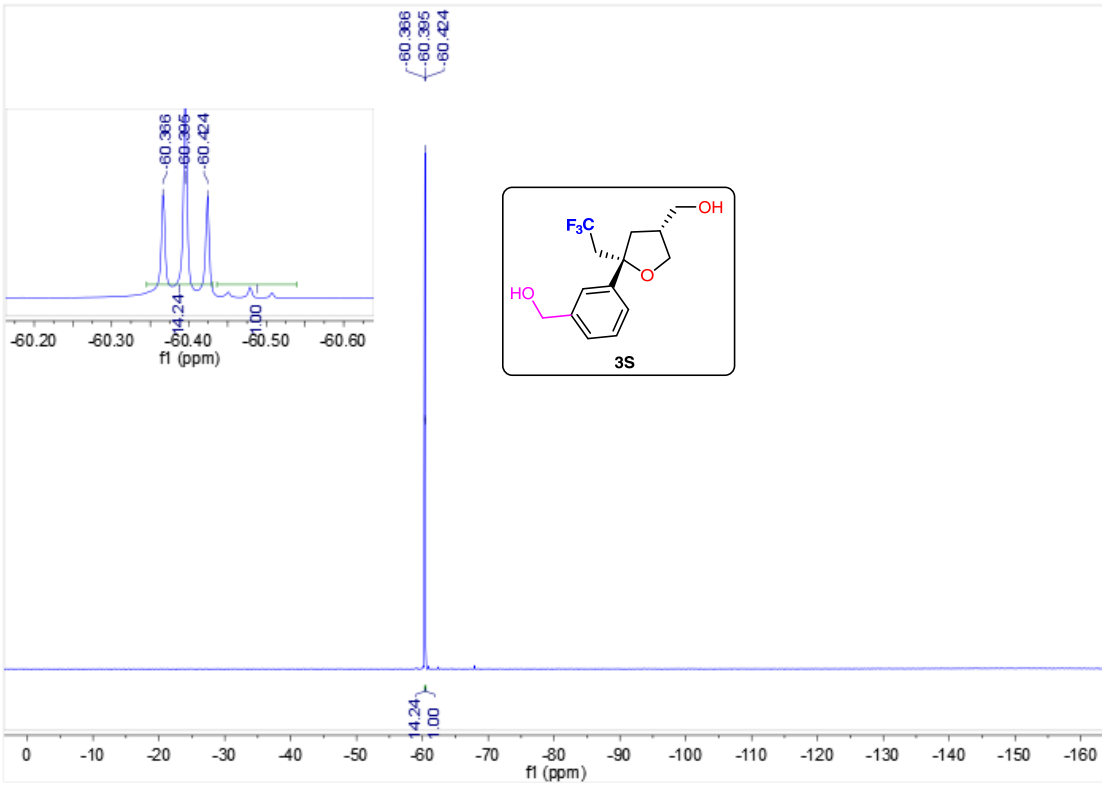

10.031
7.931
7.814
7.796
7.724
7.705
7.563
7.544
7.525
4.031
4.010
3.989
3.860
3.841
3.820
3.711
3.696
3.685
3.679
3.669
3.662
3.652
3.636
2.787
2.760
2.734
2.707
2.632
2.613
2.600
2.582
2.400
2.381
2.362
2.342
2.323
2.304
2.287
2.054
2.022
1.998
1.936
0.000
F3C
OH
O
OHC
3R
1.00
1.00
0.97
1.00
1.01
1.00
1.00
2.08
1.08
1.01
1.09
1.11
1.31
1.06
10.5 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)

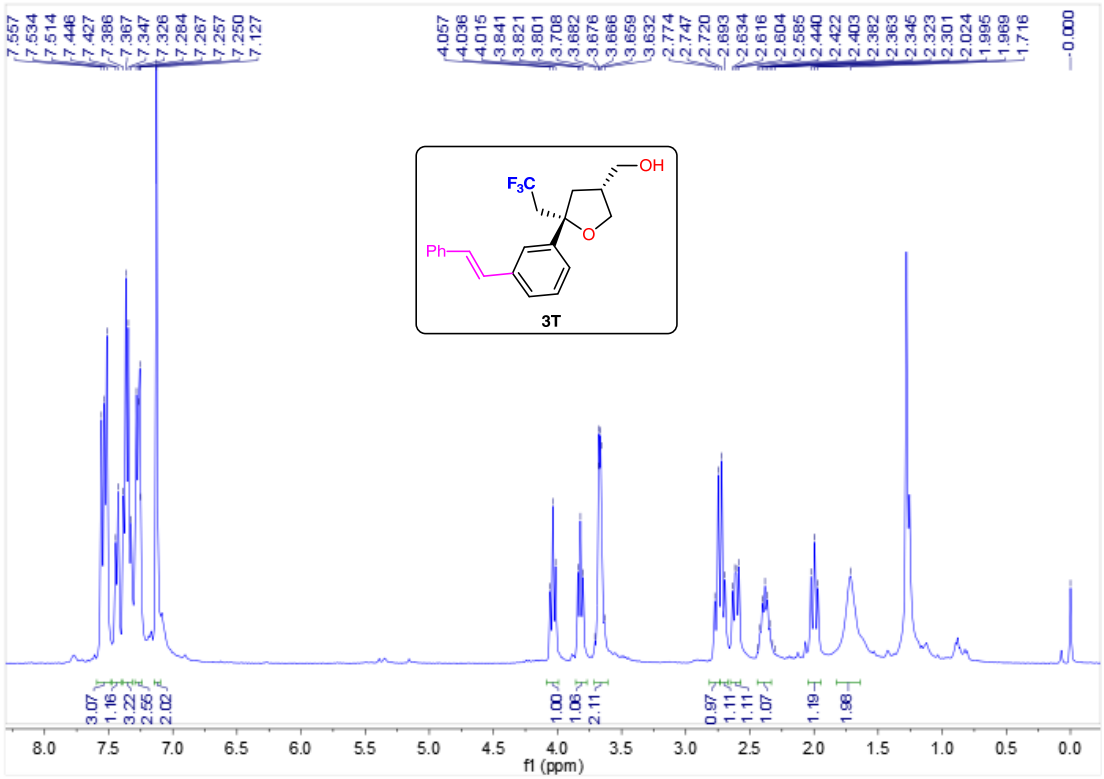

192.303
144.904
136.438
131.523
129.126
129.058
126.445
126.382
123.682
83.138
83.112
77.317
77.000
76.682
69.896
64.052
45.782
45.516
45.250
44.985
41.378
40.755
F3C
OH
O
OHC
3R
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

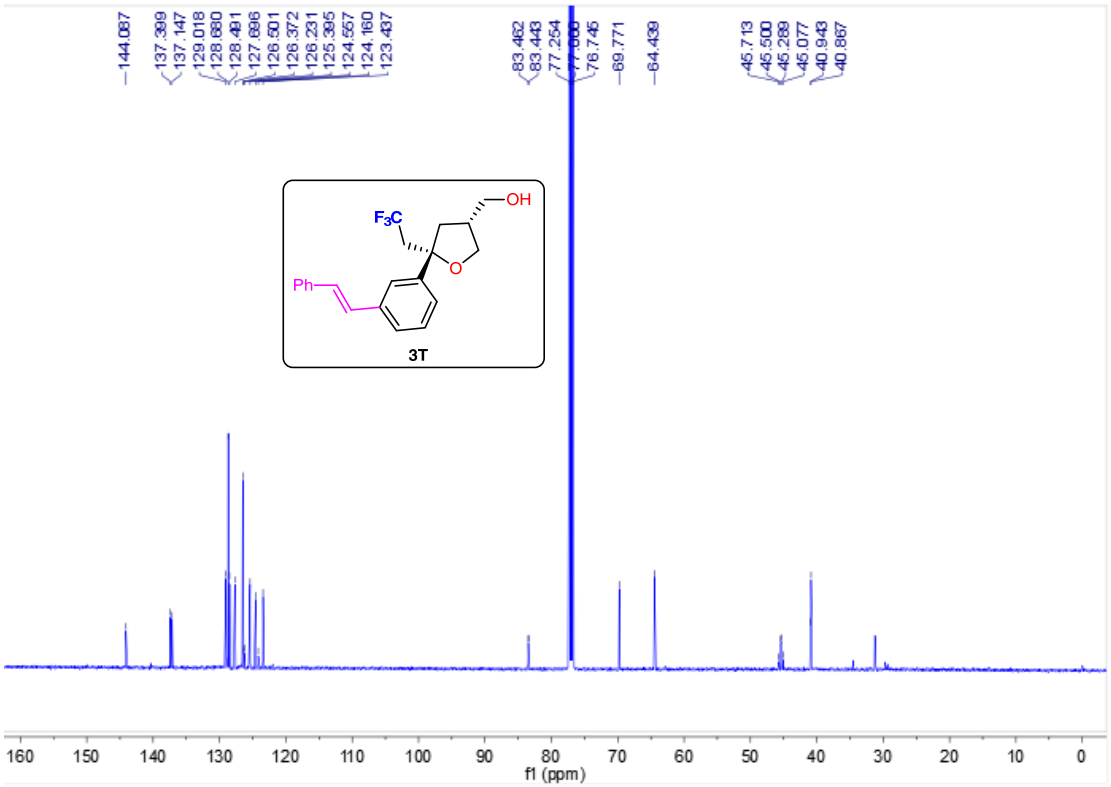

60.366
60.395
60.423
F3C
OH
O
OHC
3R
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -16
f1 (ppm)


Majo
Mino
1.93
0.13
f1 (ppm)
F3C
OH
HO
O
3S
Mino
Mino
f1 (ppm)


F3C
OH
HO
O
3S
f1 (ppm)


-60.366
-60.395
-60.424
-60.20
-60.30
-60.40
-60.50
-60.60
f1 (ppm)
14.24
1.00
F3C
OH
HO
O
3S
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)


7.557
7.534
7.514
7.446
7.427
7.386
7.367
7.347
7.326
7.284
7.267
7.257
7.250
7.127
4.057
4.036
4.015
3.841
3.821
3.801
3.708
3.682
3.676
3.666
3.659
3.632
2.774
2.747
2.720
2.693
2.634
2.616
2.604
2.585
2.440
2.422
2.403
2.382
2.363
2.345
2.323
2.301
2.024
1.995
1.969
1.716
0.000
F3C
OH
Ph
O
3T
3.07
1.16
3.22
2.55
2.02
1.00
1.06
2.11
0.97
1.11
1.11
1.07
1.19
1.98
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)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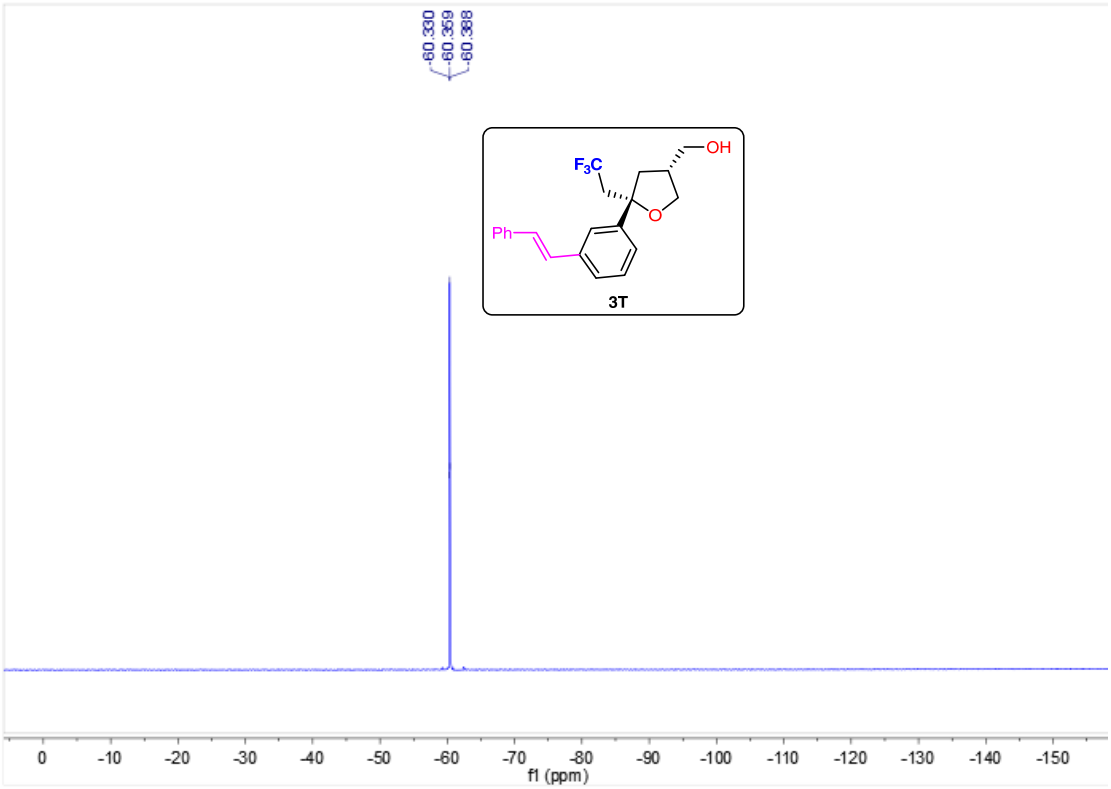
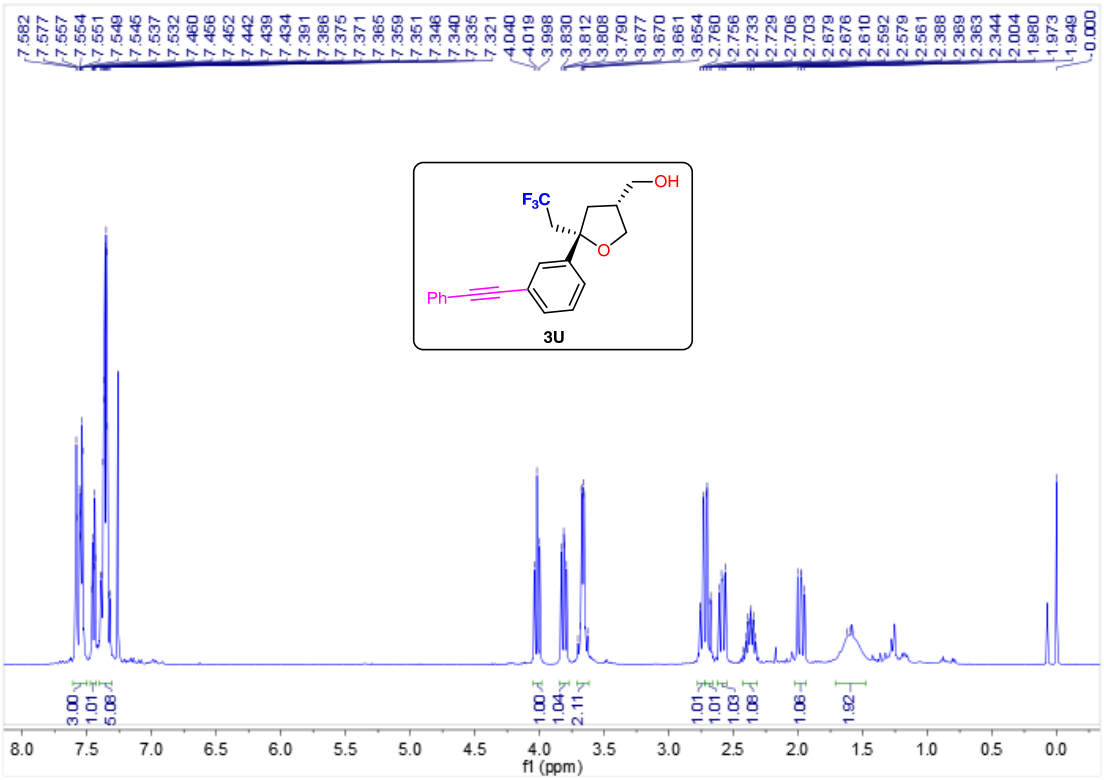
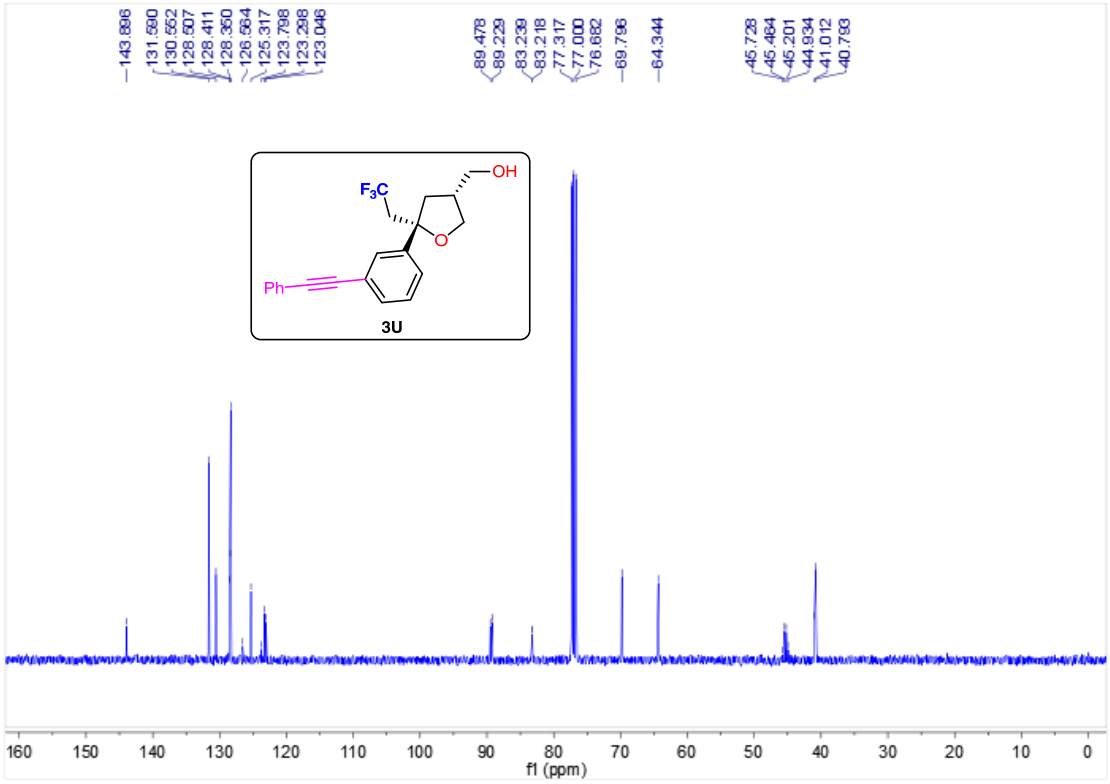
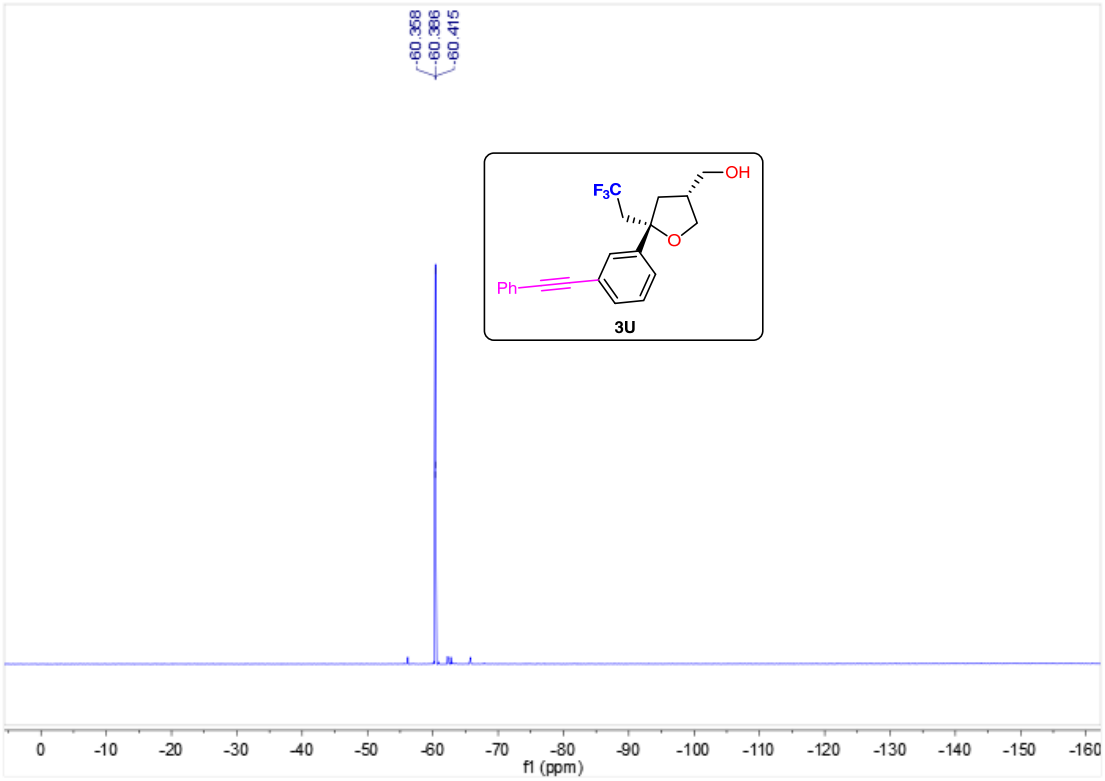
137.399
137.147
129.018
128.880
128.491
127.886
126.501
126.372
126.231
125.395
124.557
124.160
123.437
83.462
83.443
77.254
77.000
76.745
69.771
64.439
45.713
45.500
45.289
45.077
40.943
40.867
OH
F3C
O
Ph
3T
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


60.330
60.359
60.388
OH
F3C
O
Ph
3T
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)


7.582
7.577
7.557
7.554
7.551
7.549
7.545
7.537
7.532
7.460
7.456
7.452
7.442
7.439
7.434
7.391
7.386
7.375
7.371
7.365
7.359
7.351
7.346
7.340
7.335
7.321
4.040
4.019
3.998
3.830
3.812
3.808
3.790
3.677
3.670
3.661
3.654
2.760
2.756
2.733
2.729
2.706
2.703
2.679
2.676
2.610
2.592
2.579
2.561
2.388
2.369
2.363
2.344
2.004
1.980
1.973
1.949
0.000
F3C
OH
O
Ph
3U
3.00
1.01
5.08
1.00
1.04
2.11
1.01
1.01
1.03
1.08
1.06
1.92
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)


143.896
131.690
130.552
128.507
128.411
128.350
126.564
125.317
123.798
123.298
123.046
89.478
89.229
83.239
83.218
77.317
77.000
76.682
69.796
64.344
45.728
45.464
45.201
44.934
41.012
40.793
F3C
OH
O
Ph
3U
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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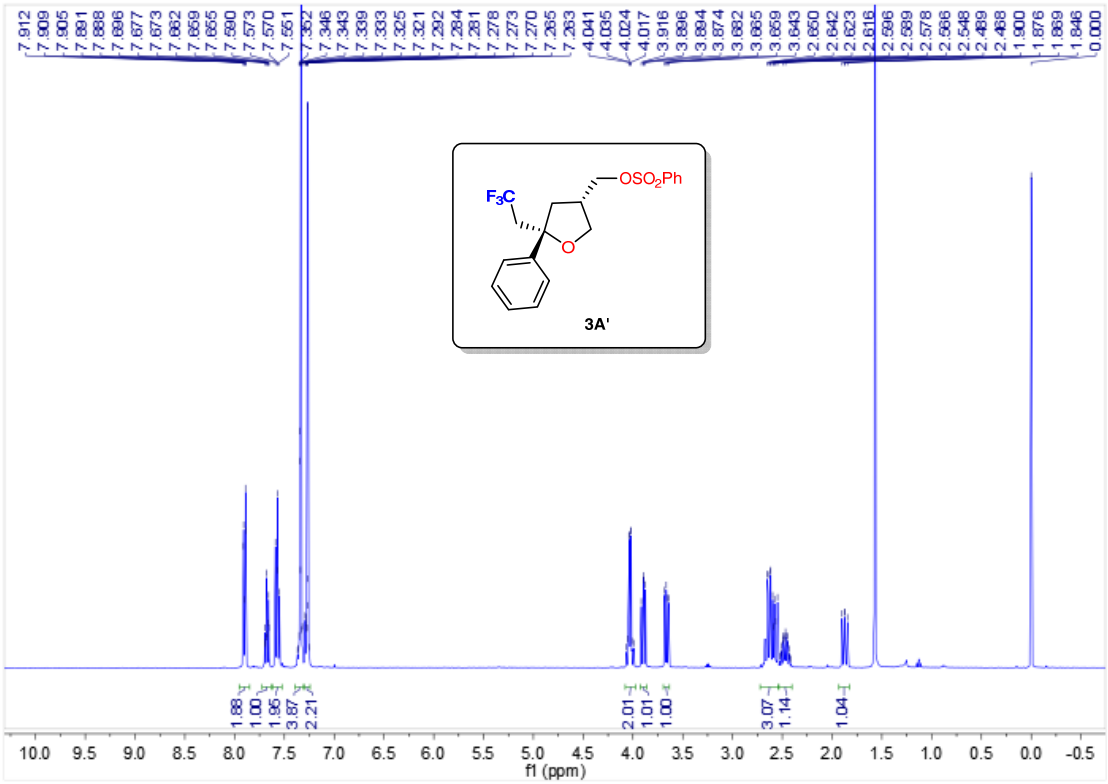
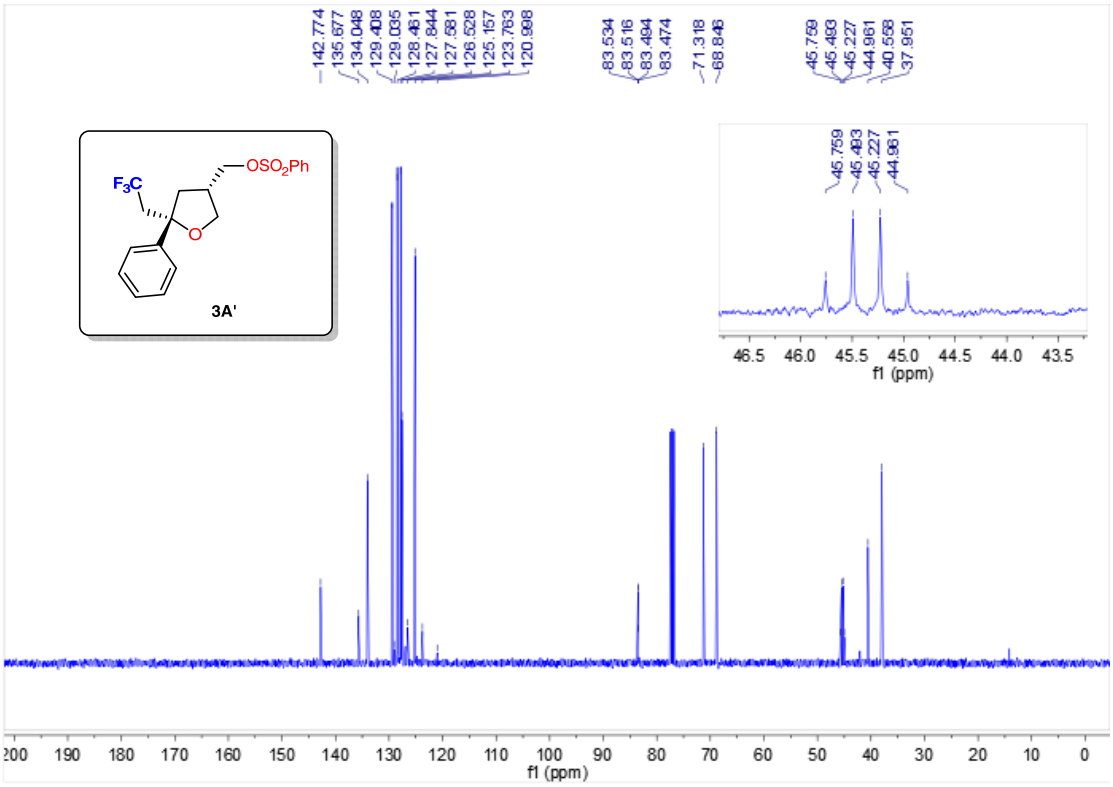
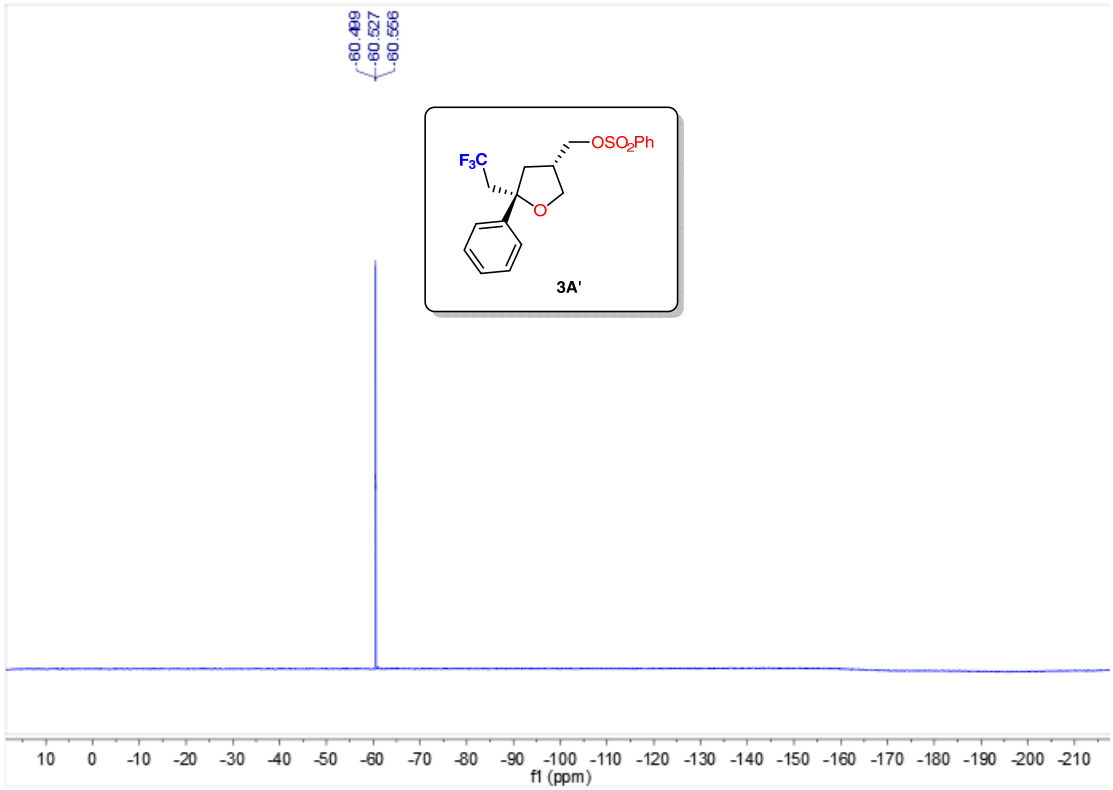
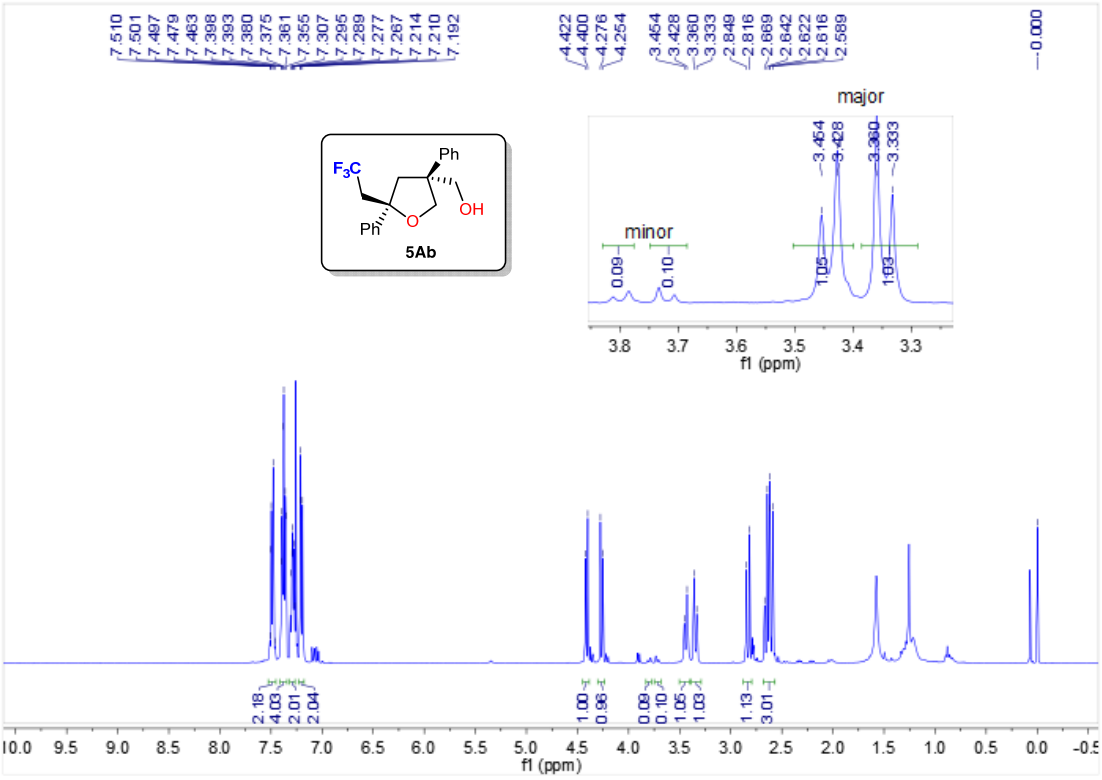
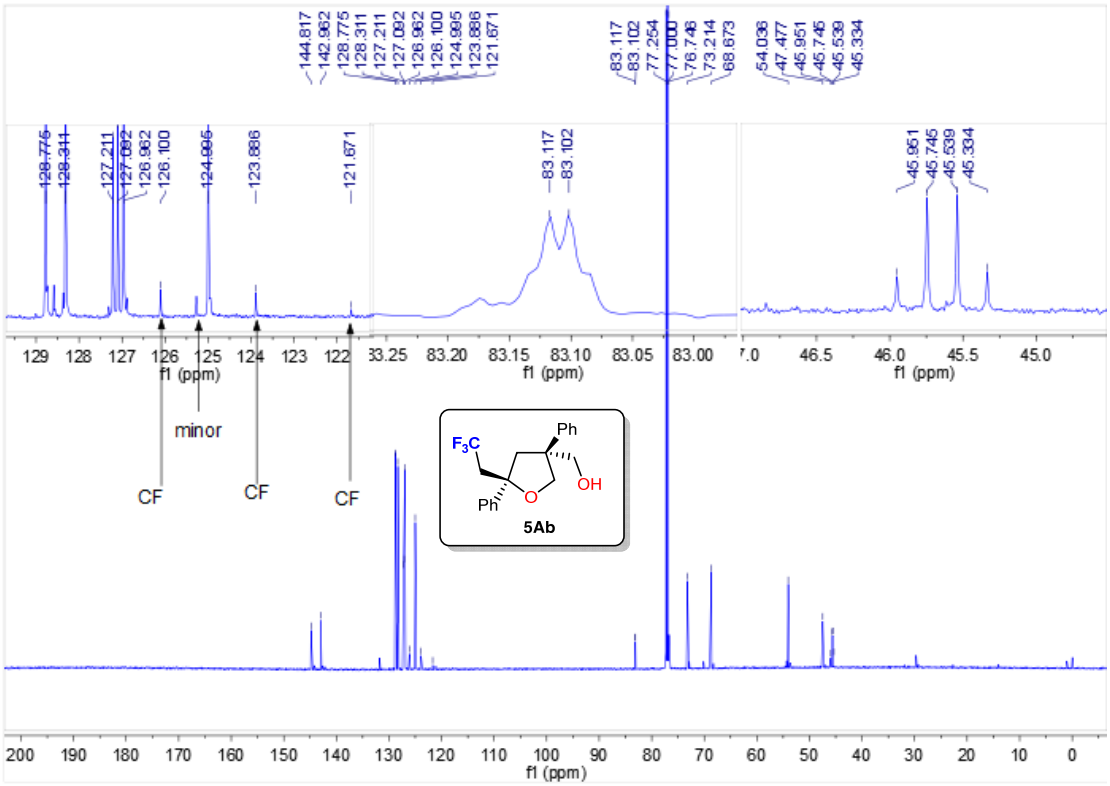
-60.415
F3C
OH
O
Ph
3U
f1 (ppm)

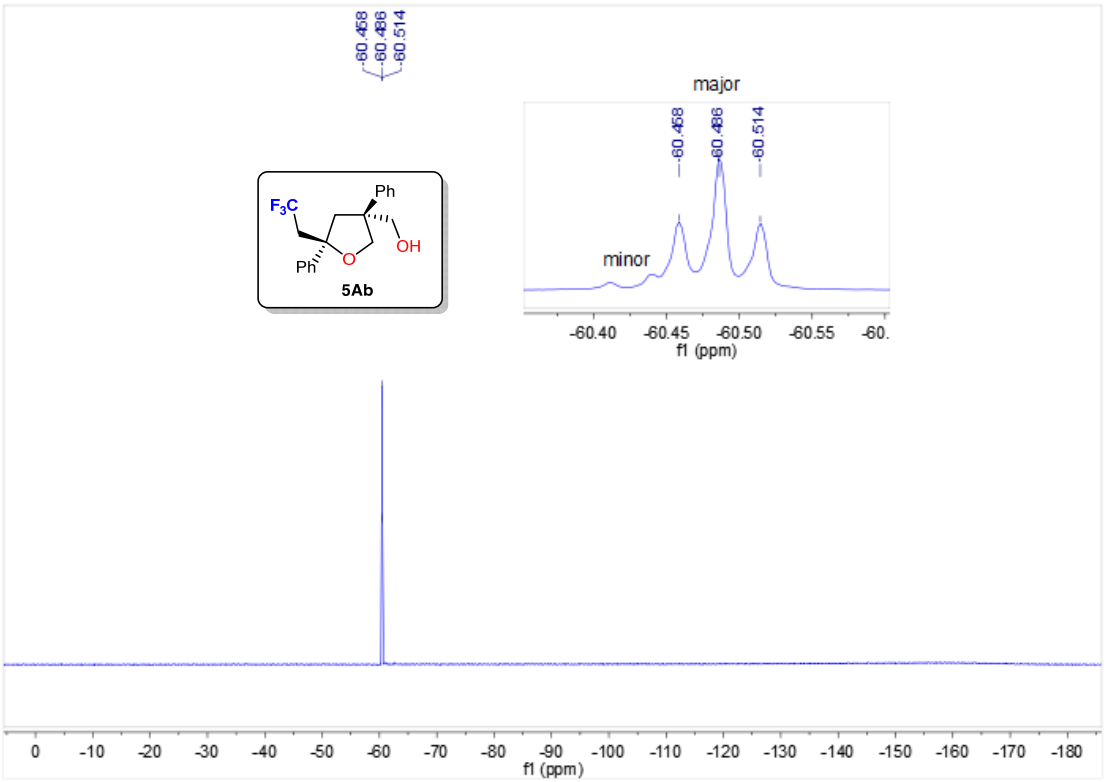

F3C
OSO2Ph
O
3A'
f1 (ppm)

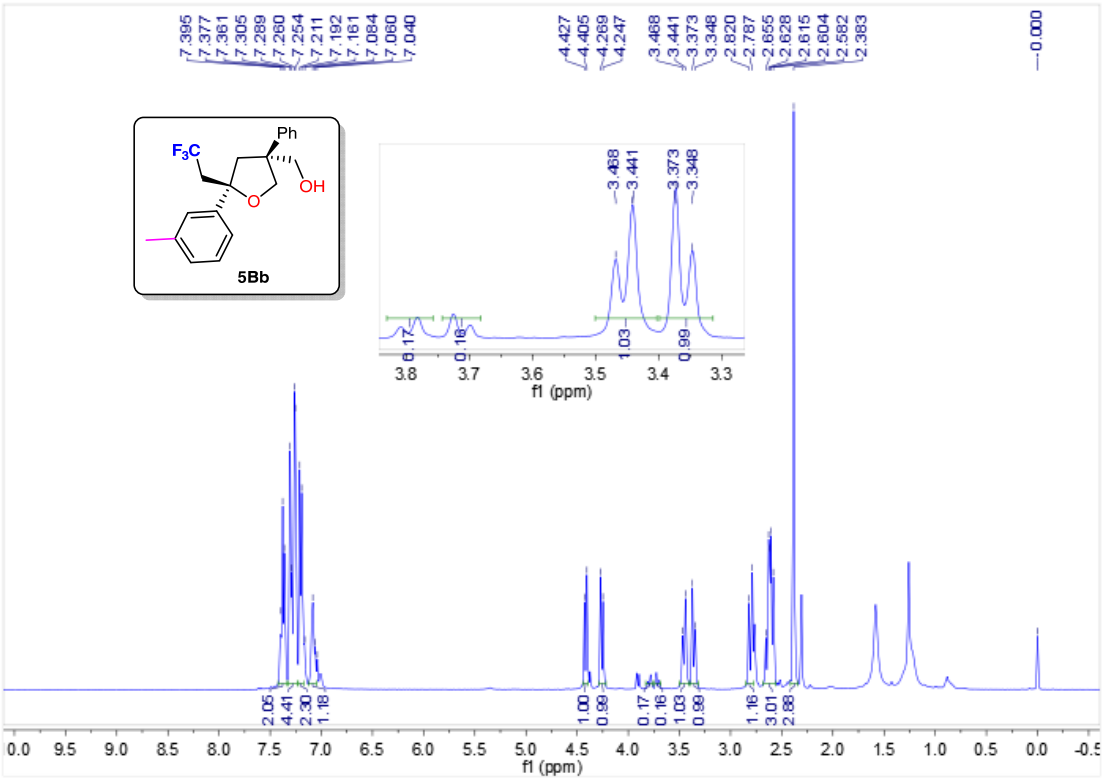

142.774
135.677
134.048
129.408
129.035
128.481
127.844
127.681
126.528
125.157
123.763
120.998
83.534
83.516
83.494
83.474
71.318
68.846
45.759
45.493
45.227
44.961
40.558
37.951
F3C
OSO2Ph
O
3A'
f1 (ppm)

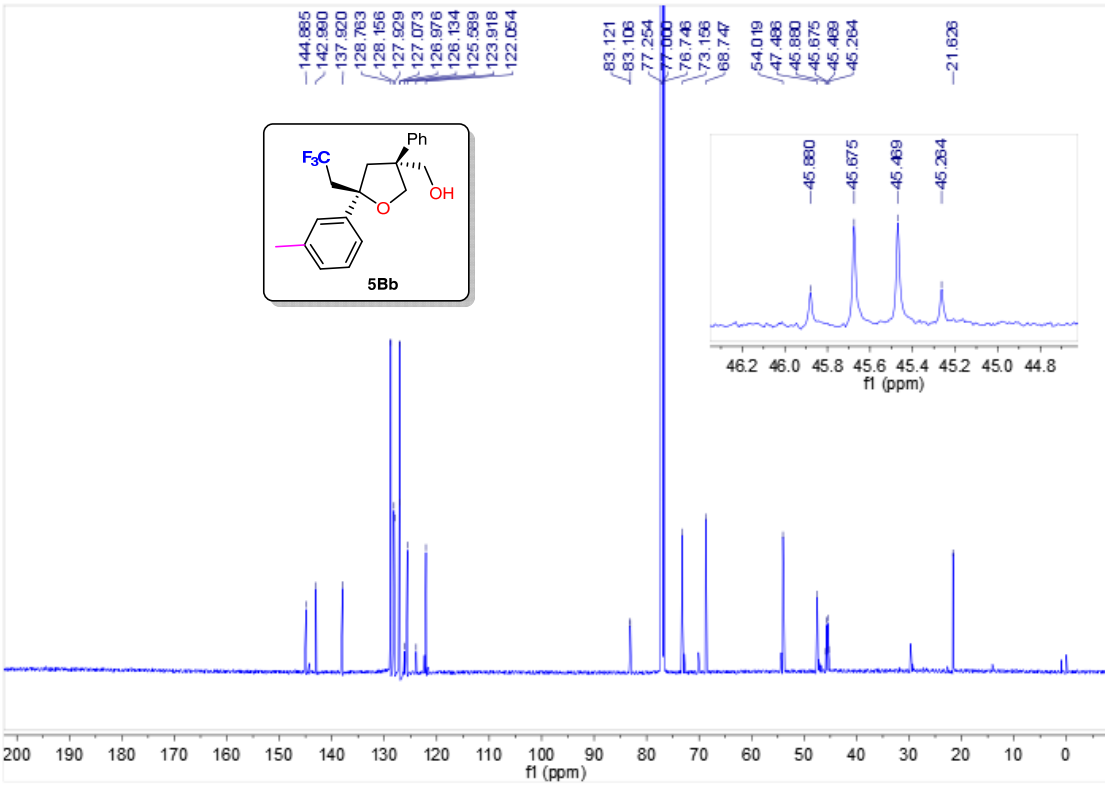

60.499
60.527
60.556
F3C
OSO2Ph
O
3A'
f1 (ppm)


major
minor
5Ab
f1 (ppm)


minor
CF
CF
CF
5Ab
f1 (ppm)


-60.458
-60.486
-60.514
major
minor
5Ab
f1 (ppm)


5Bb
f1 (ppm)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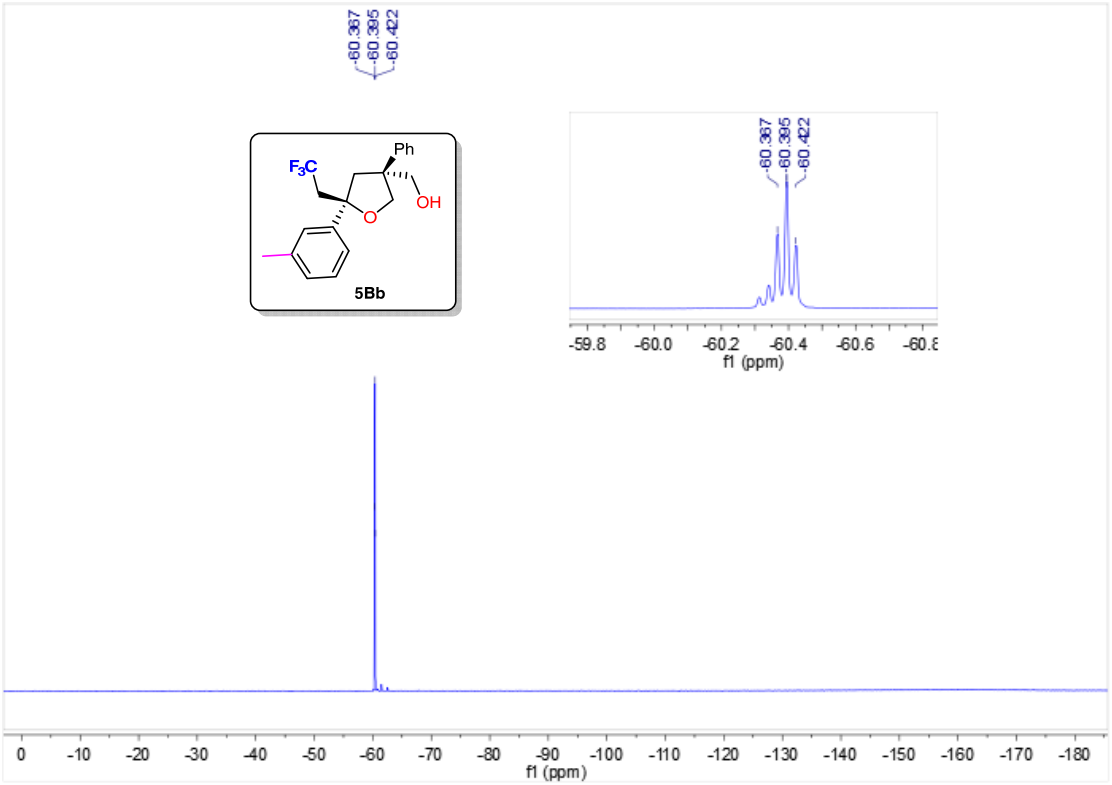
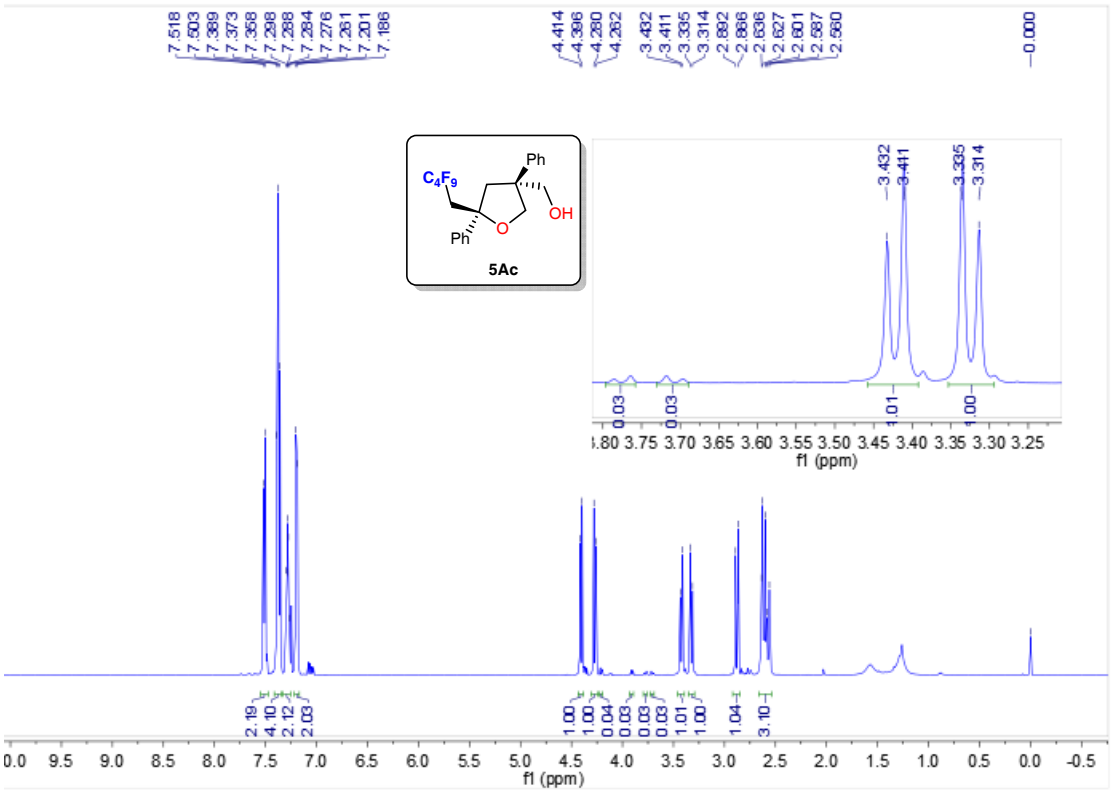
127.073
126.976
126.134
125.589
123.918
122.054
83.121
83.106
77.254
77.000
76.748
73.156
68.747
54.019
47.488
45.880
45.675
45.469
45.264
21.626
Ph
F3C
OH
O
5Bb
45.880
45.675
45.469
45.264
46.2 46.0 45.8 45.6 45.4 45.2 45.0 44.8
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-60.367
-60.395
-60.422
Ph
F3C
OH
O
5Bb
-60.367
-60.395
-60.422
-59.8 -60.0 -60.2 -60.4 -60.6
f1 (ppm)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180
f1 (ppm)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7.298
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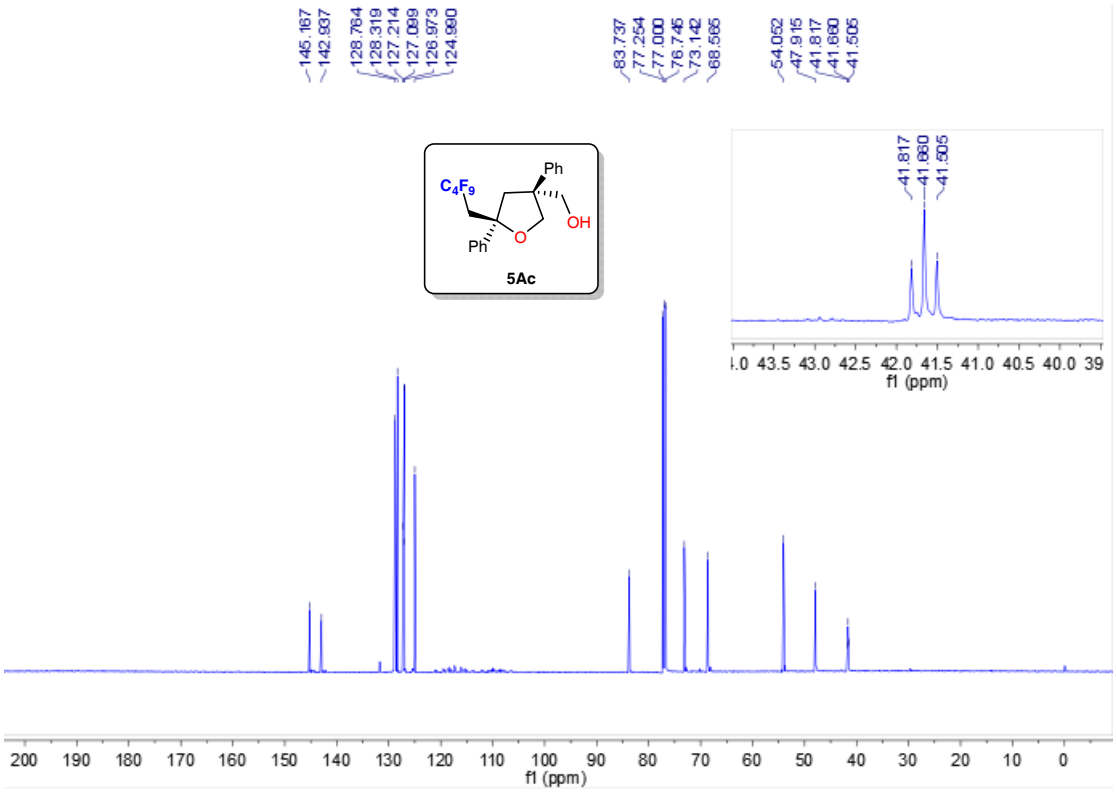
7.284
7.276
7.261
7.201
7.186
4.414
4.396
4.280
4.262
3.432
3.411
3.335
3.314
2.892
2.866
2.636
2.627
2.601
2.587
2.560
0.000
C4F9
Ph
OH
Ph
O
5Ac
f1 (ppm)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128.764
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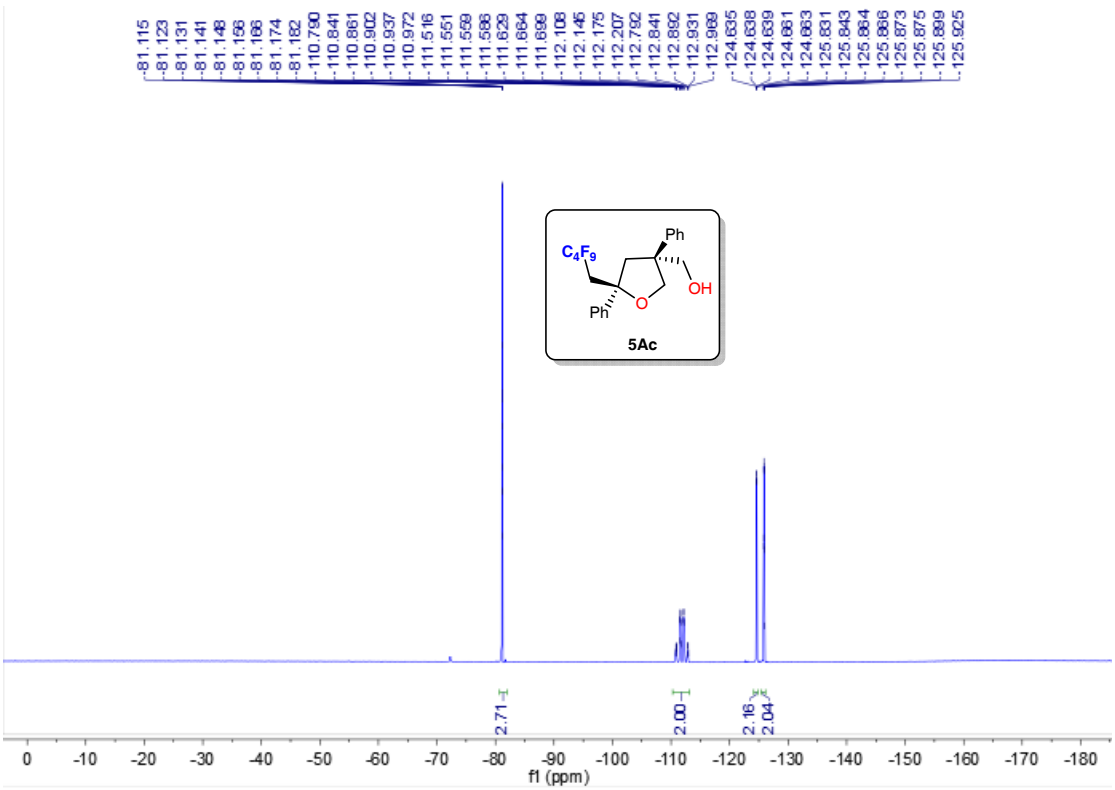
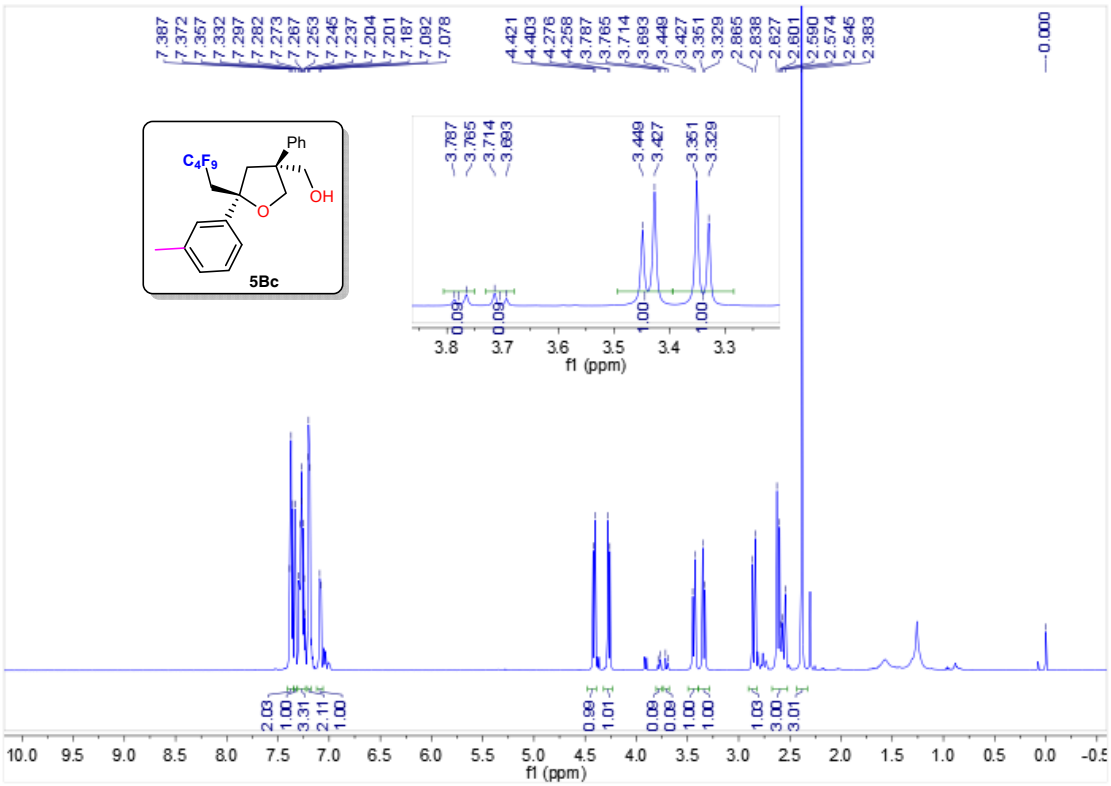
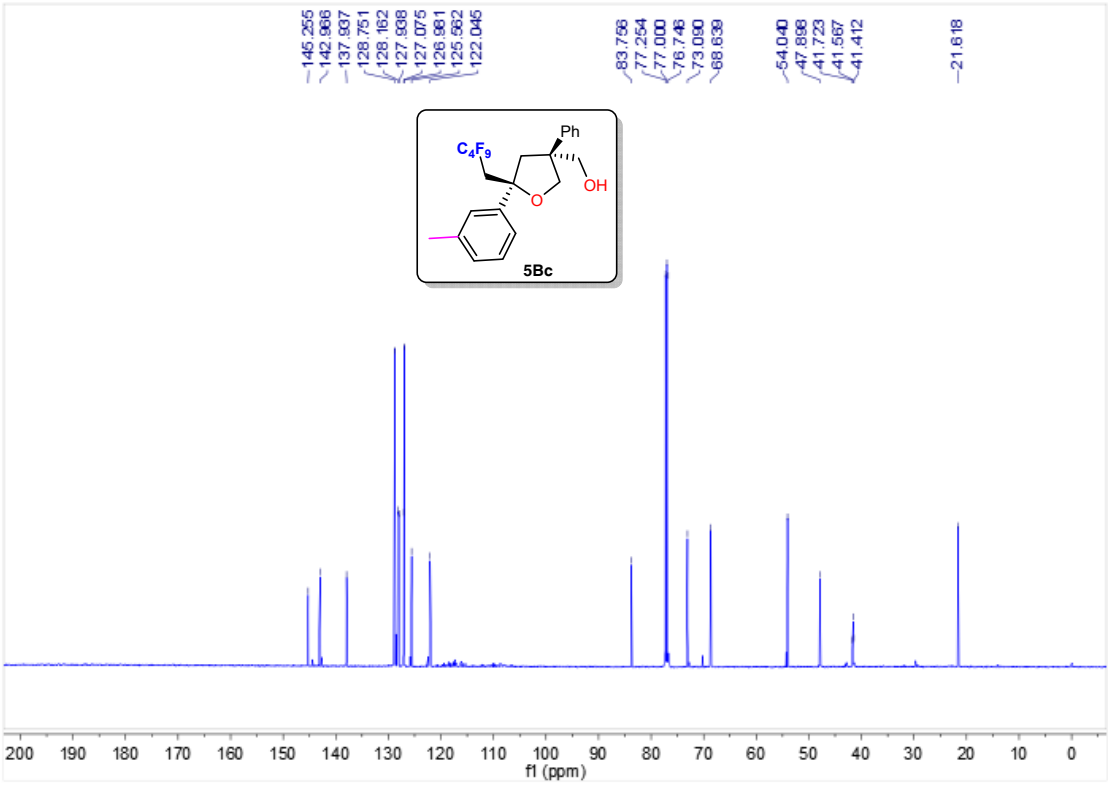
127.214
127.099
126.973
124.990
83.737
77.254
77.000
76.745
73.142
68.565
54.052
47.915
41.817
41.660
41.505
C4F9
Ph
OH
Ph
O
5Ac
f1 (ppm)


5Ac
f1 (ppm)


5Bc
f1 (ppm)
f1 (ppm)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128.162
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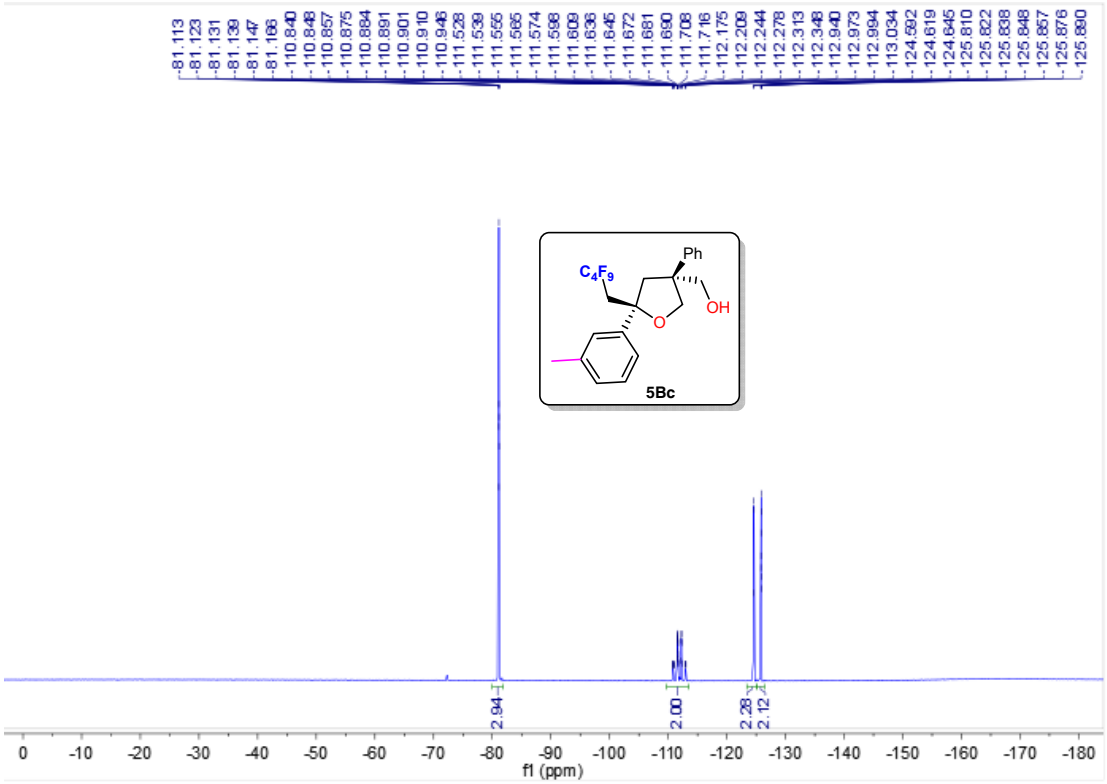
127.075
126.981
125.562
122.045
83.756
77.254
77.000
76.746
73.090
68.839
54.040
47.898
41.723
41.567
41.412
21.618
Ph
C_4F_9
OH
O
5Bc
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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81.166
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110.848
110.857
110.875
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110.891
110.901
110.910
110.946
111.528
111.539
111.555
111.565
111.574
111.598
111.609
111.636
111.645
111.672
111.681
111.690
111.708
111.716
112.175
112.209
112.244
112.278
112.313
112.348
112.940
112.973
112.994
113.034
124.692
124.619
124.645
125.810
125.822
125.838
125.848
125.857
125.876
125.890
Ph
C_4F_9
OH
O
5Bc
2.94
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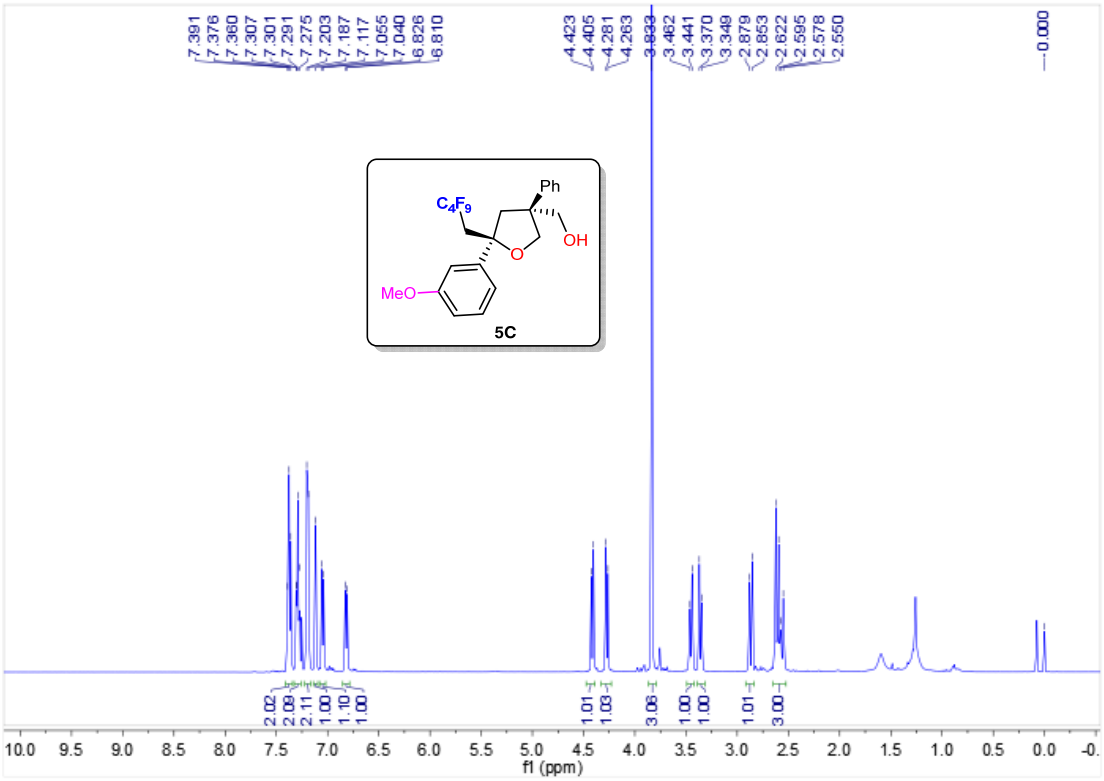
2.28
2.12
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180
f1 (ppm)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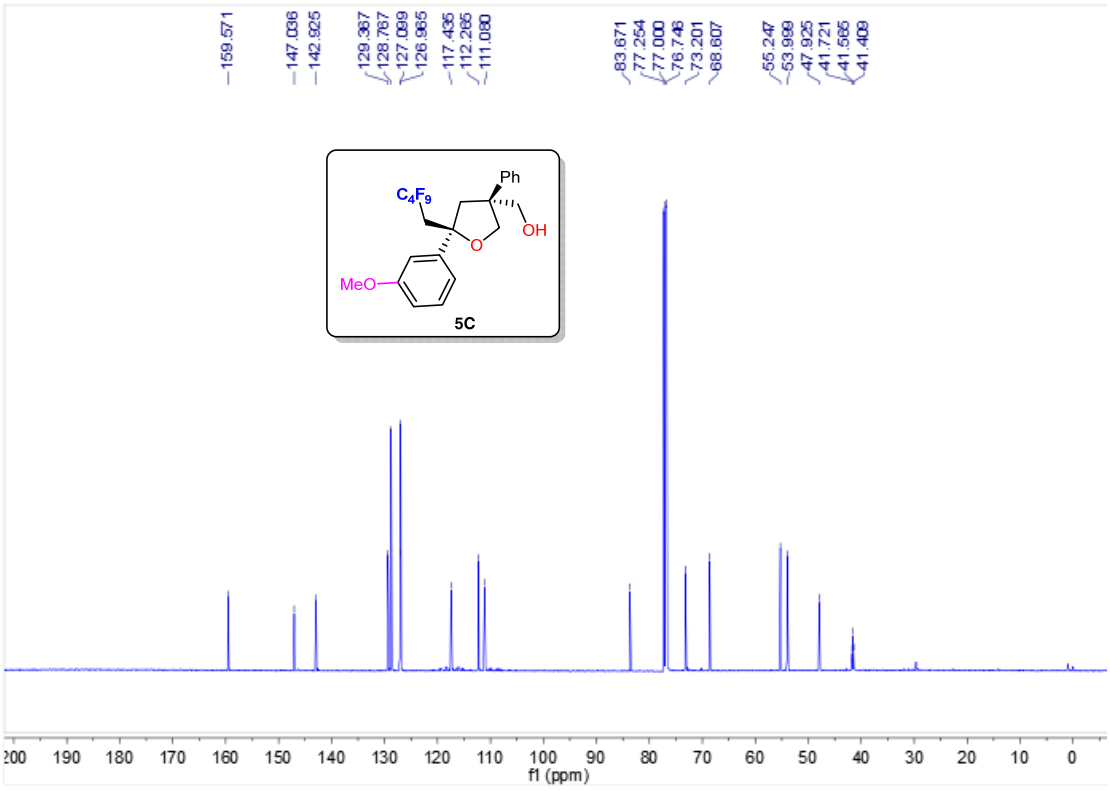
7.307
7.301
7.291
7.275
7.203
7.187
7.117
7.055
7.040
6.826
6.810
4.423
4.405
4.281
4.263
3.833
3.462
3.441
3.370
3.349
2.879
2.853
2.622
2.595
2.578
2.550
0.000
Ph
C4F9
OH
O
MeO
5C
2.02
2.09
2.11
1.00
1.10
1.00
1.01
1.03
3.06
1.00
1.00
1.01
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.571
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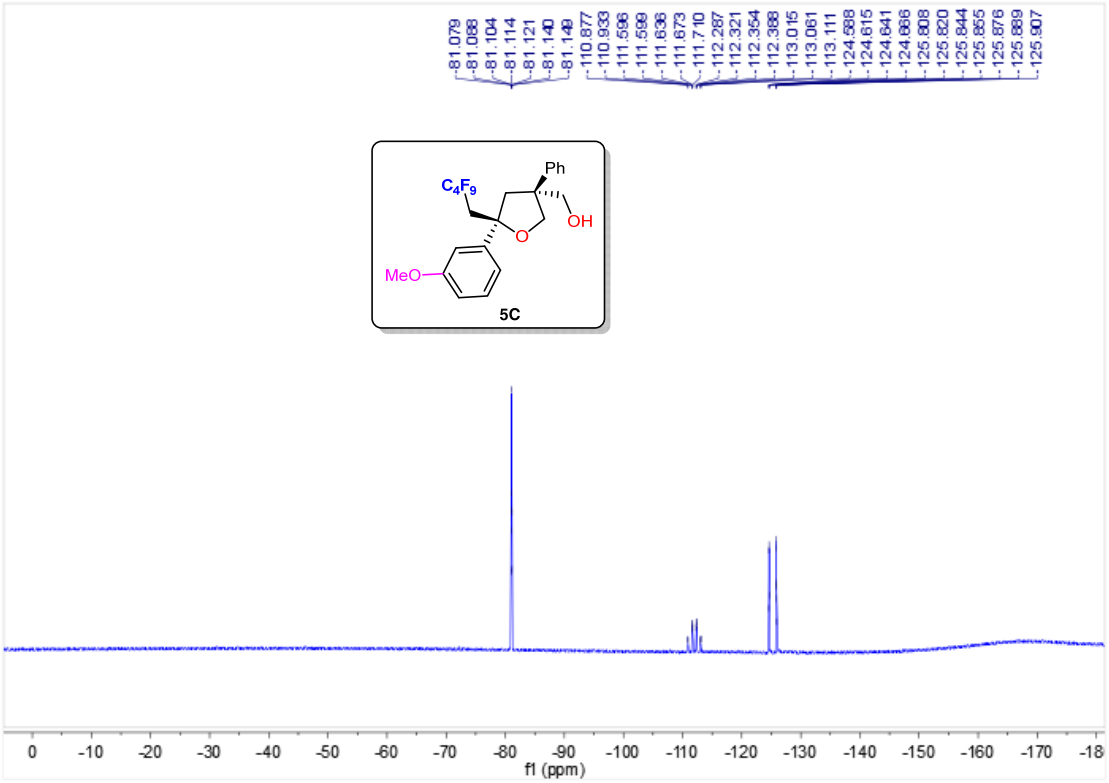
128.767
127.099
126.965
117.435
112.265
111.080
83.671
77.254
77.000
76.746
73.201
68.607
55.247
53.999
47.925
41.721
41.565
41.409
Ph
C4F9
OH
O
MeO
5C
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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-81.114
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-81.149
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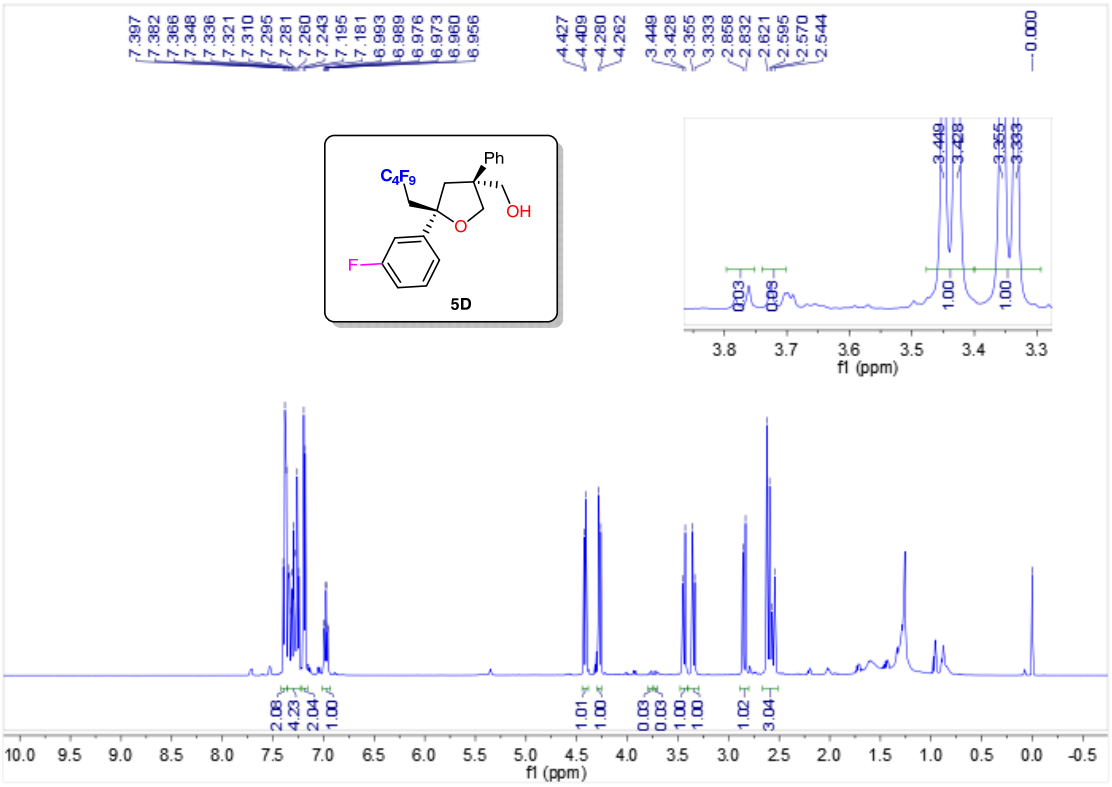
-110.933
-111.596
-111.599
-111.636
-111.673
-111.710
-112.287
-112.321
-112.354
-112.388
-113.015
-113.061
-113.111
-124.588
-124.615
-124.641
-124.666
-125.808
-125.820
-125.844
-125.855
-125.876
-125.889
-125.907
Ph
C4F9
OH
O
MeO
5C
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-18
f1 (ppm)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7.366
7.348
7.336
7.321
7.310
7.295
7.281
7.260
7.243
7.195
7.181
6.993
6.989
6.976
6.973
6.960
6.956
4.427
4.409
4.280
4.262
3.449
3.428
3.355
3.333
2.858
2.832
2.621
2.595
2.570
2.544
0.000
Ph
C4F9
OH
O
F
5D
0.03
0.09
1.00
1.00
3.8
3.7
3.6
3.5
3.4
3.3
f1 (ppm)
2.08
4.23
2.04
1.00
1.01
1.00
0.03
0.03
1.00
1.00
1.02
3.04
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
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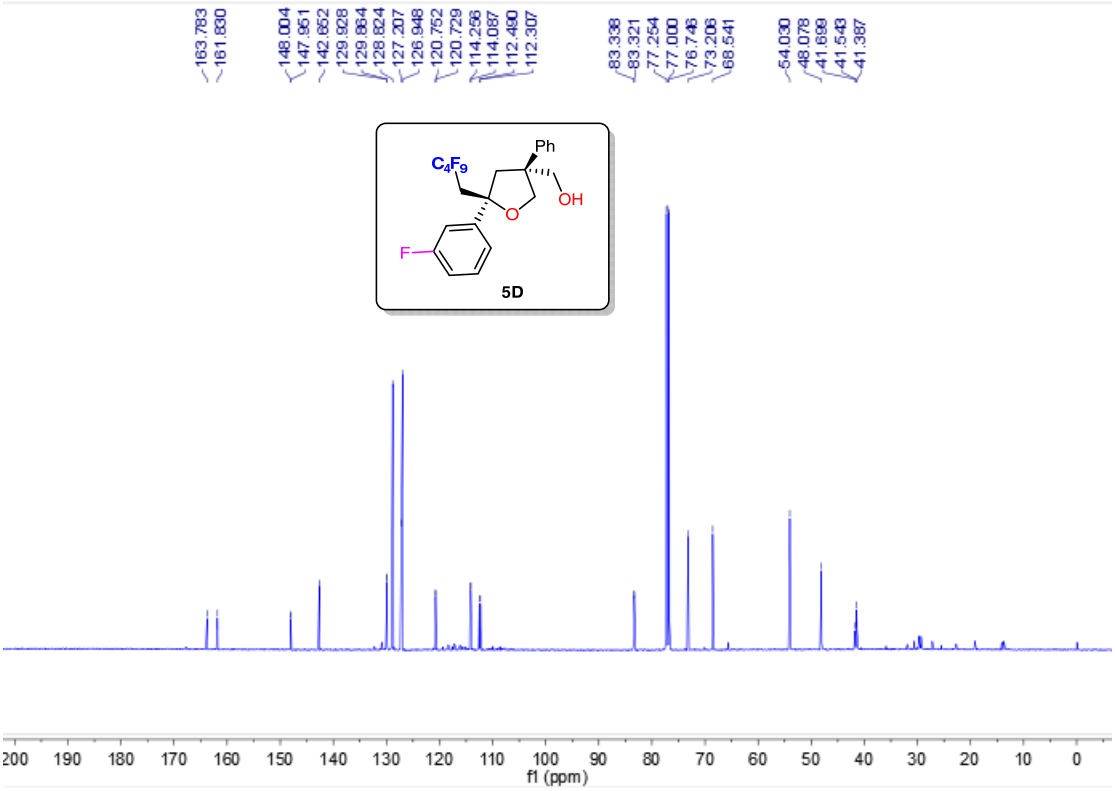
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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128.824
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120.729
114.256
114.087
112.490
112.307
83.338
83.321
77.254
77.000
76.746
73.206
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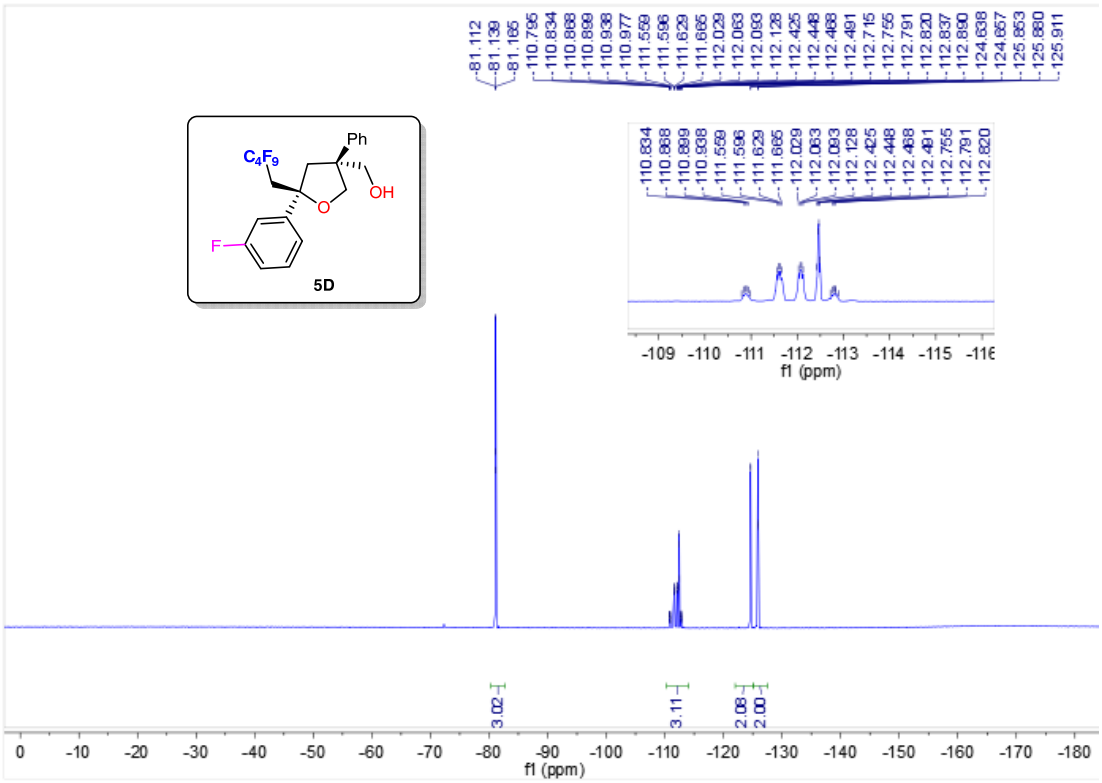
48.078
41.699
41.543
41.387
Ph
C_4F_9
OH
O
F
5D
f1 (ppm)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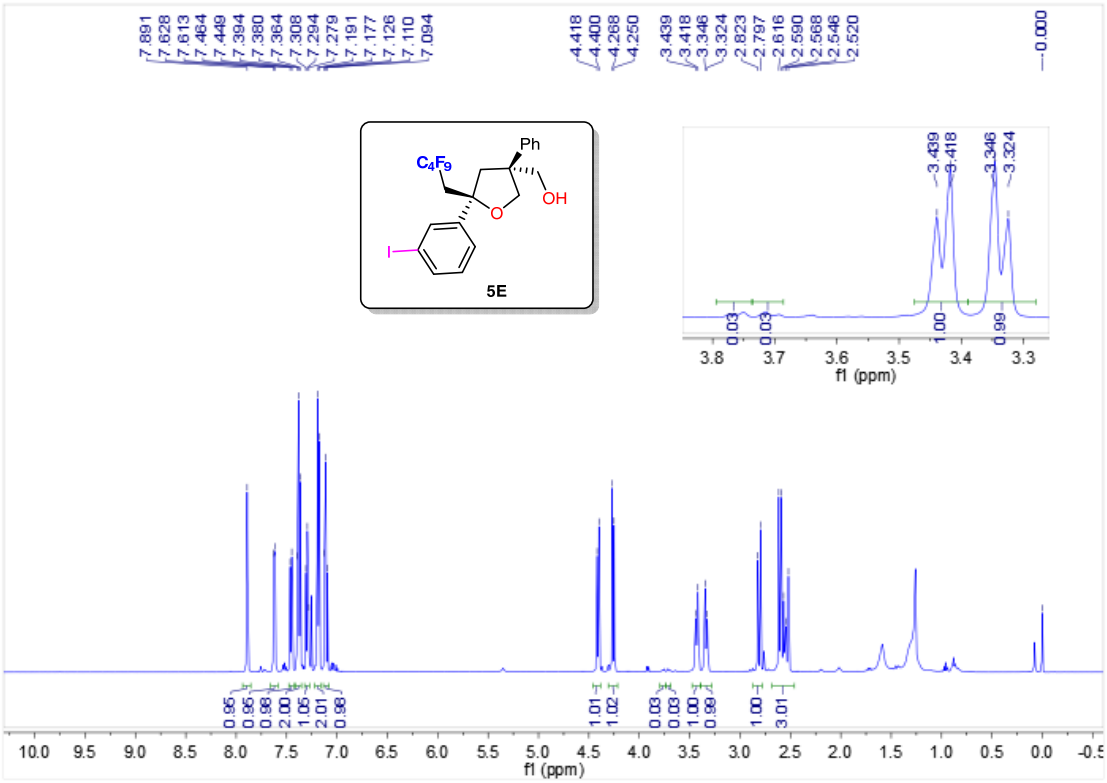
111.629
111.665
112.029
112.063
112.093
112.128
112.425
112.448
112.468
112.491
112.715
112.755
112.791
112.820
112.837
112.890
124.638
124.657
125.853
125.880
125.911
Ph
C_4F_9
OH
O
F
5D
f1 (ppm)
3.02
3.11
2.08
2.00
f1 (ppm)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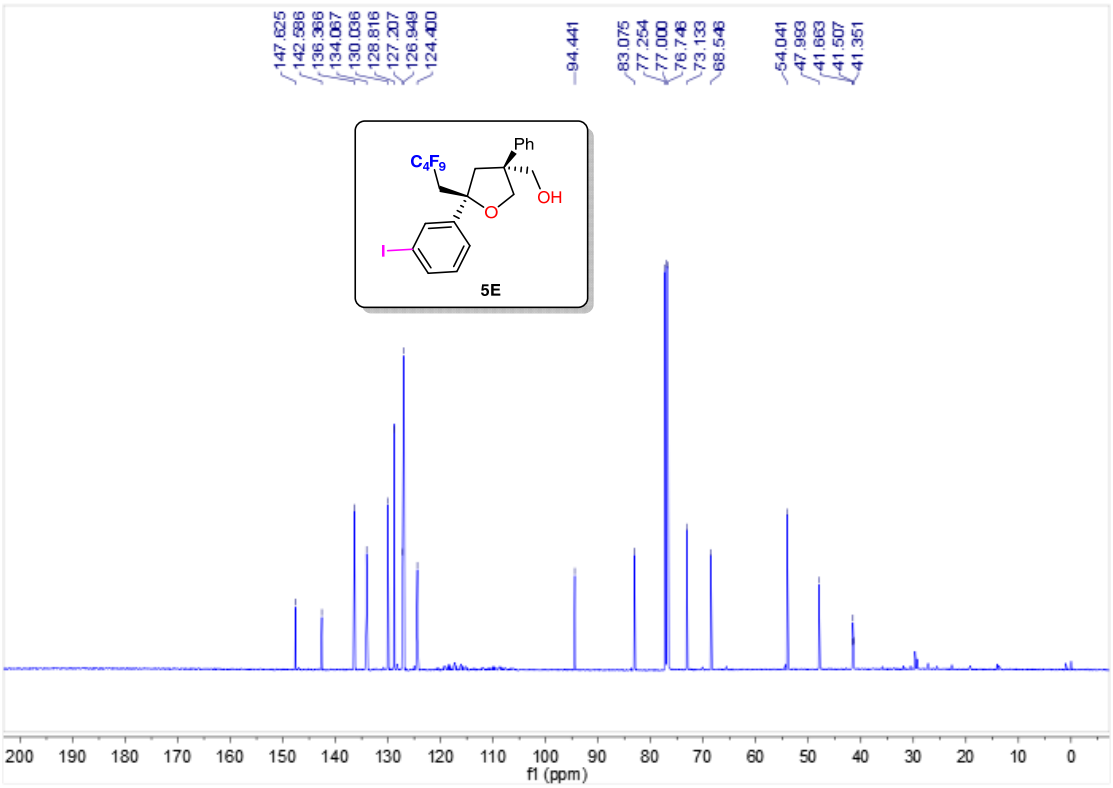
7.628
7.613
7.464
7.449
7.394
7.380
7.364
7.308
7.294
7.279
7.191
7.177
7.126
7.110
7.094
4.418
4.400
4.268
4.250
3.439
3.418
3.346
3.324
2.823
2.797
2.616
2.590
2.568
2.546
2.520
0.000
Ph
C4F9
OH
O
I
5E
f1 (ppm)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134.067
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127.207
126.949
124.400
94.441
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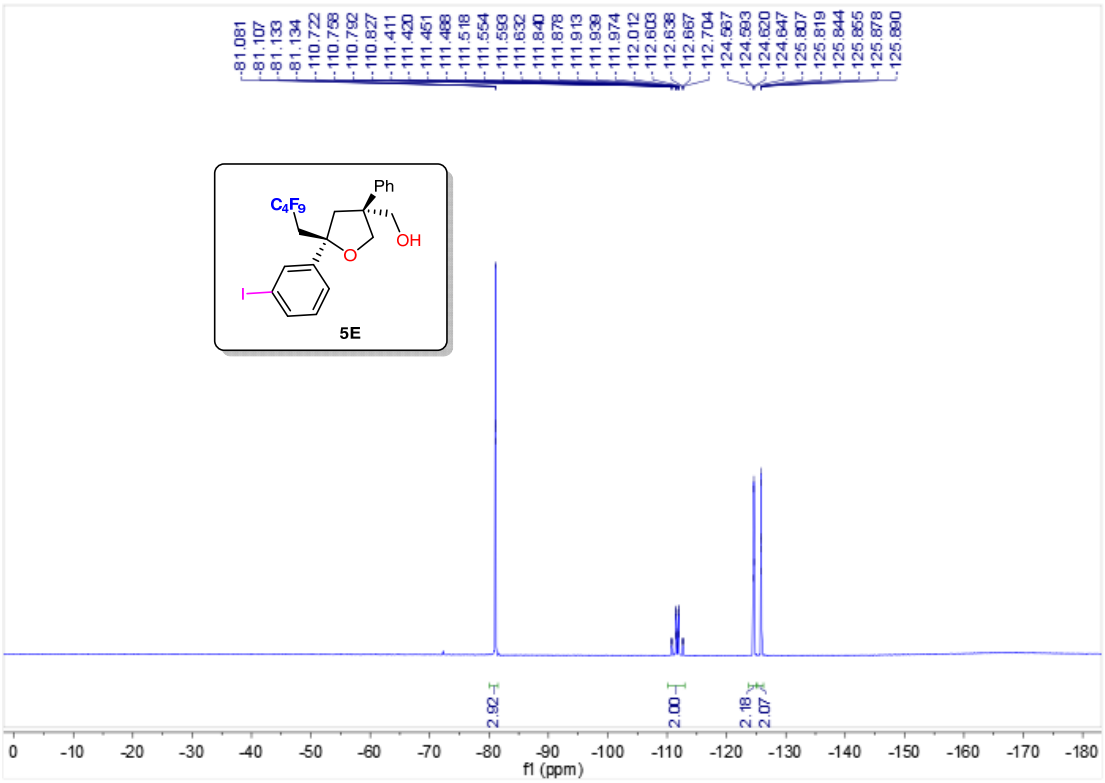
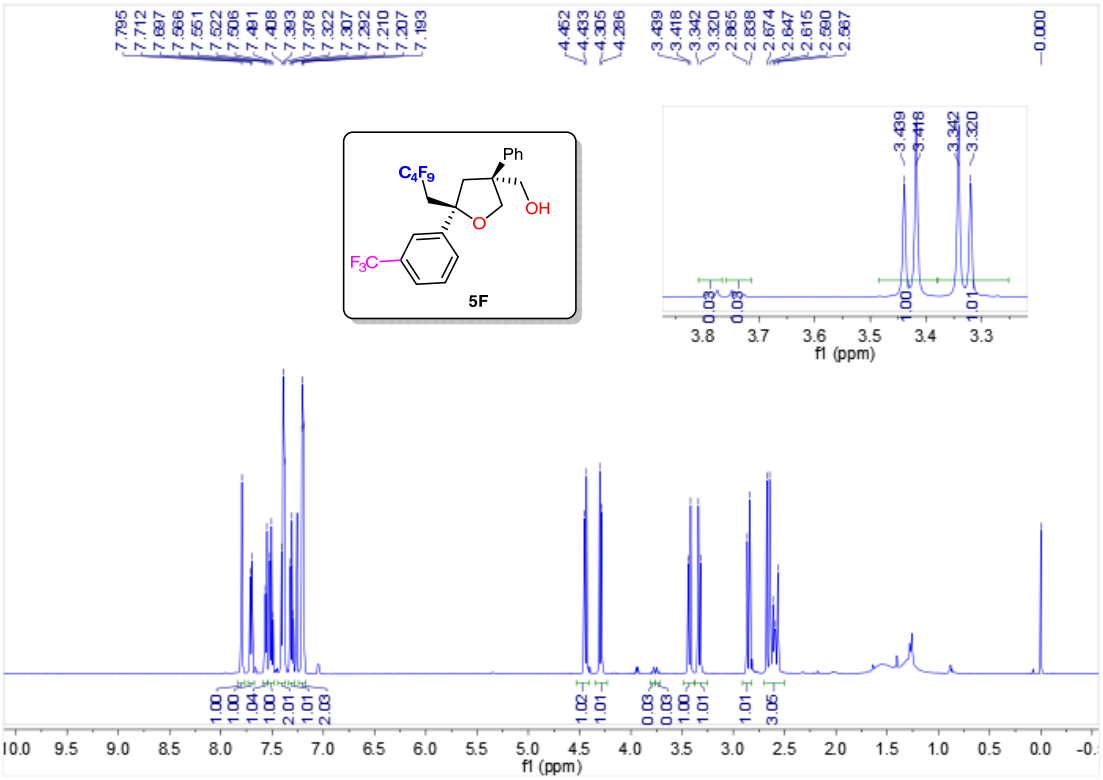
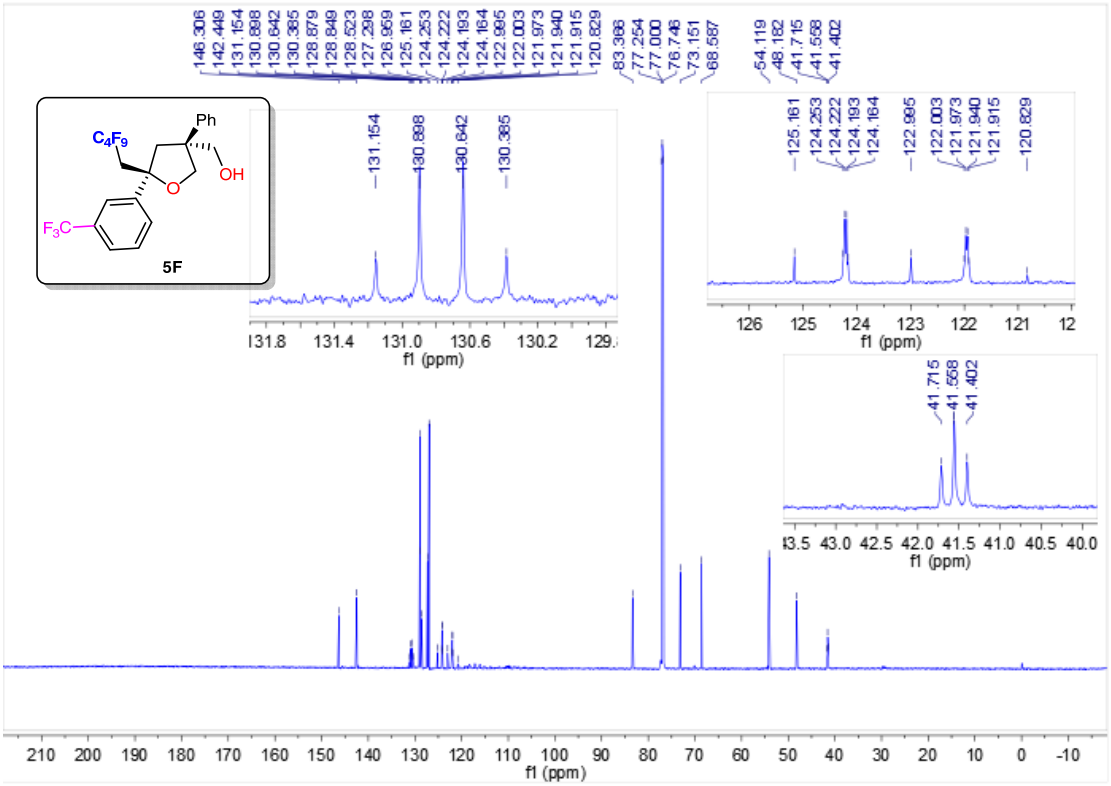
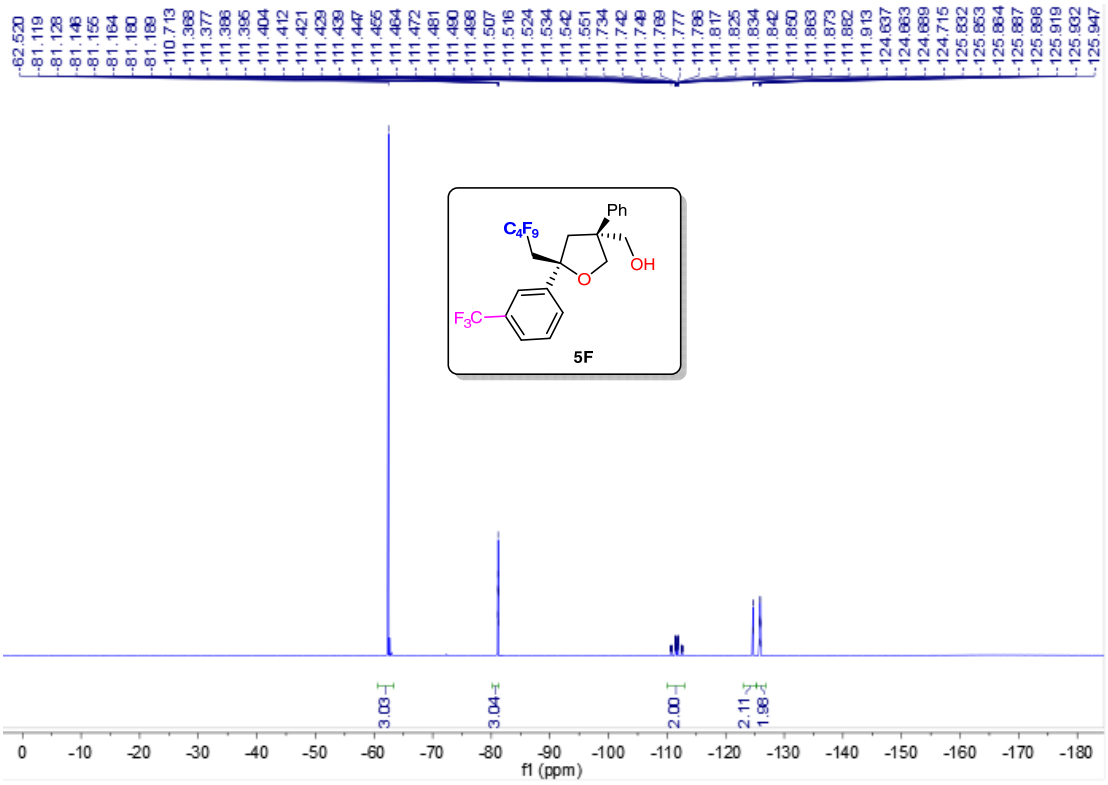
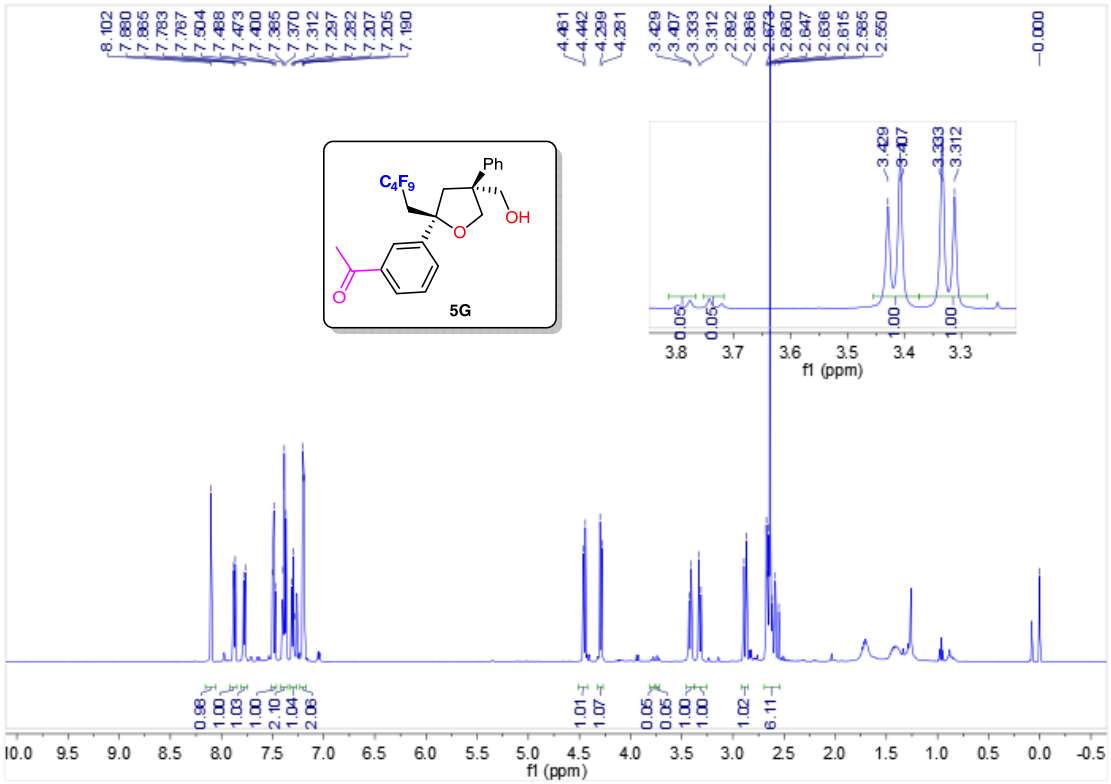
77.000
76.746
73.133
68.546
54.041
47.993
41.663
41.507
41.351
Ph
C4F9
OH
O
I
5E
f1 (ppm)


Ph
C4F9
OH
O
I
5E
f1 (ppm)


Ph
C4F9
OH
O
F3C
5F
f1 (ppm)


5F
f1 (ppm)


5F
f1 (ppm)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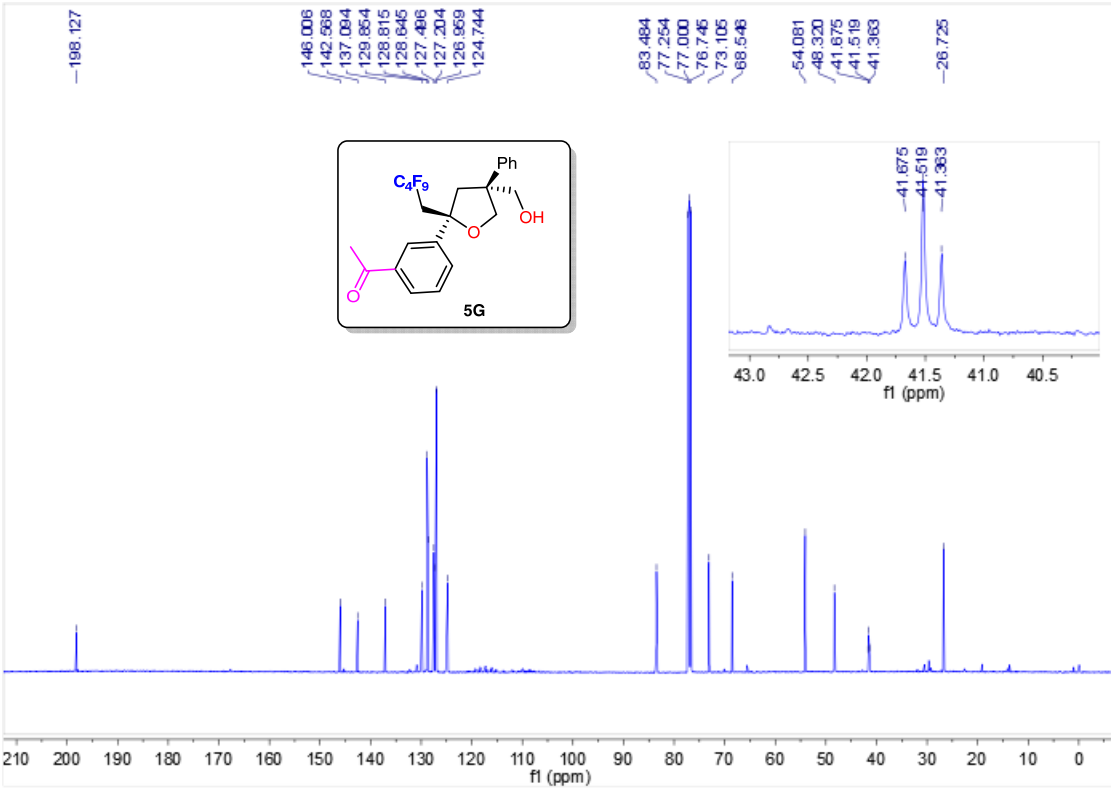
7.865
7.783
7.767
7.504
7.488
7.473
7.400
7.385
7.370
7.312
7.297
7.282
7.207
7.205
7.190
4.461
4.442
4.299
4.281
3.429
3.407
3.333
3.312
2.892
2.866
2.673
2.660
2.647
2.636
2.615
2.585
2.550
0.000
Ph
C4F9
OH
O
O
5G
f1 (ppm)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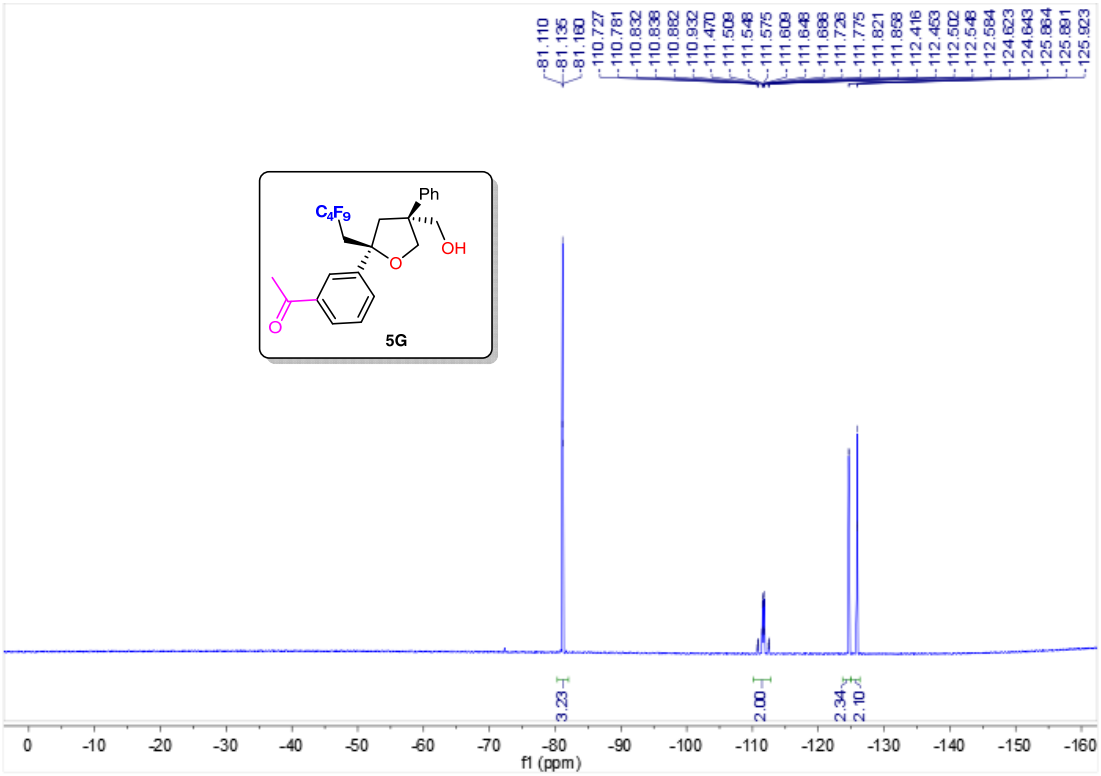
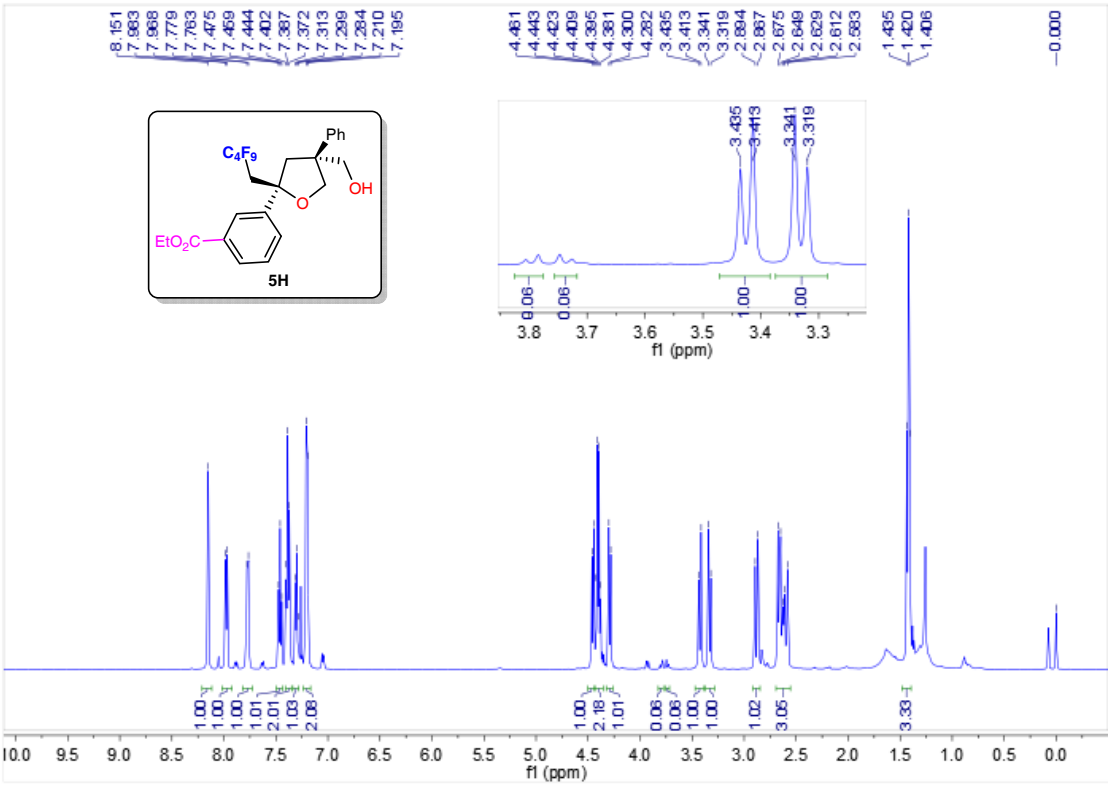
146.006
142.568
137.094
129.854
128.815
128.645
127.496
127.204
126.959
124.744
83.484
77.254
77.000
76.745
73.105
68.548
54.081
48.320
41.675
41.519
41.363
26.725
Ph
C4F9
OH
O
O
5G
f1 (ppm)


81.110
81.135
81.160
110.727
110.781
110.832
110.838
110.882
110.932
111.470
111.509
111.548
111.575
111.609
111.648
111.686
111.726
111.775
111.821
111.858
112.416
112.463
112.502
112.548
112.584
124.623
124.643
125.864
125.891
125.923
Ph
C4F9
OH
O
O
5G
3.23
2.00
2.34
2.10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)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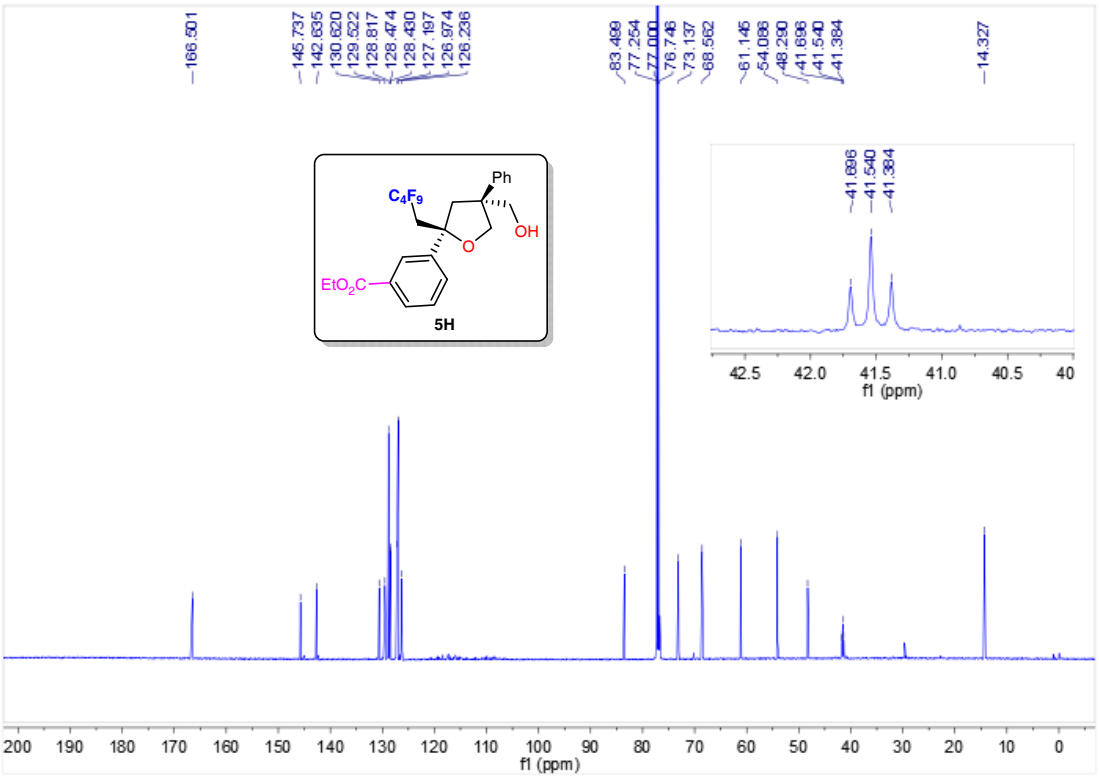
7.779
7.763
7.475
7.459
7.444
7.402
7.387
7.372
7.313
7.299
7.284
7.210
7.195
4.461
4.443
4.423
4.409
4.395
4.381
4.300
4.282
3.435
3.413
3.341
3.319
2.894
2.887
2.675
2.649
2.629
2.612
2.583
1.435
1.420
1.406
0.000
Ph
C4F9
OH
O
EtO2C
5H
3.435
3.413
3.341
3.319
0.06
0.06
1.00
1.00
3.8
3.7
3.6
3.5
3.4
3.3
f1 (ppm)
1.00
1.00
1.00
1.01
2.01
1.03
2.08
1.00
2.18
1.01
0.06
0.06
1.00
1.00
1.02
3.05
3.33
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)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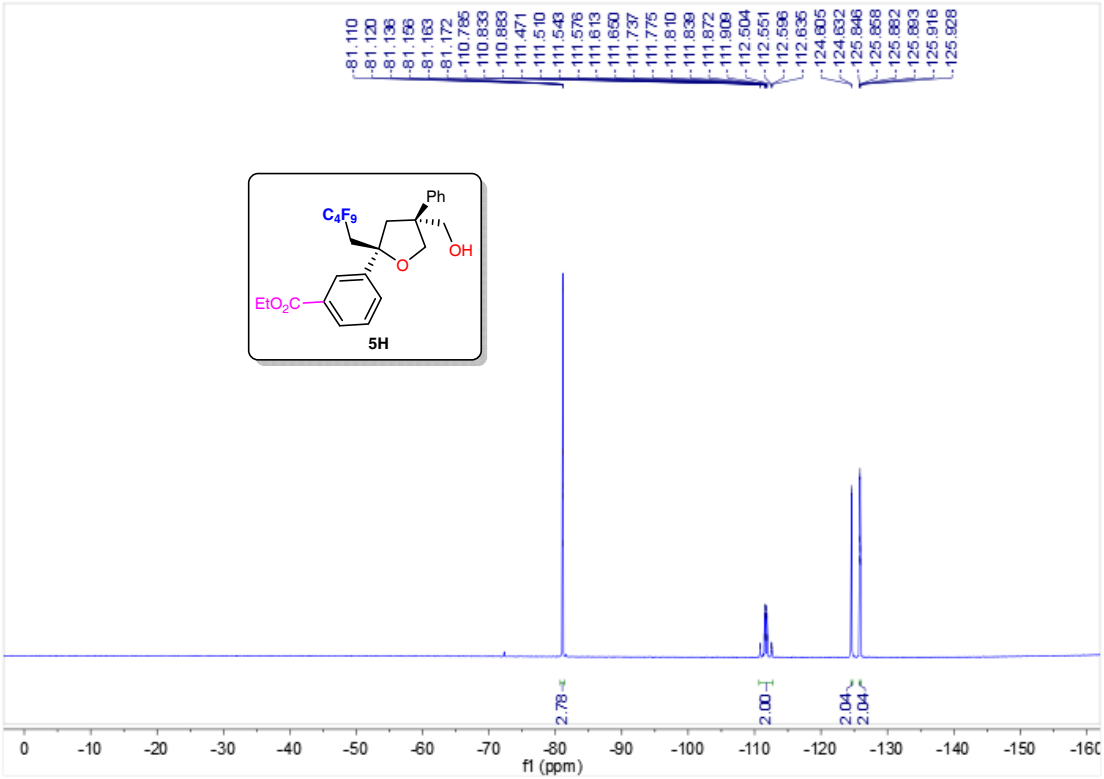
145.737
142.635
130.620
129.522
128.817
128.474
128.430
127.197
126.974
126.236
83.499
77.254
77.000
76.746
73.137
68.562
61.145
54.088
48.290
41.896
41.540
41.384
14.327
Ph
C_4F_9
OH
O
EtO_2C
5H
f1 (ppm)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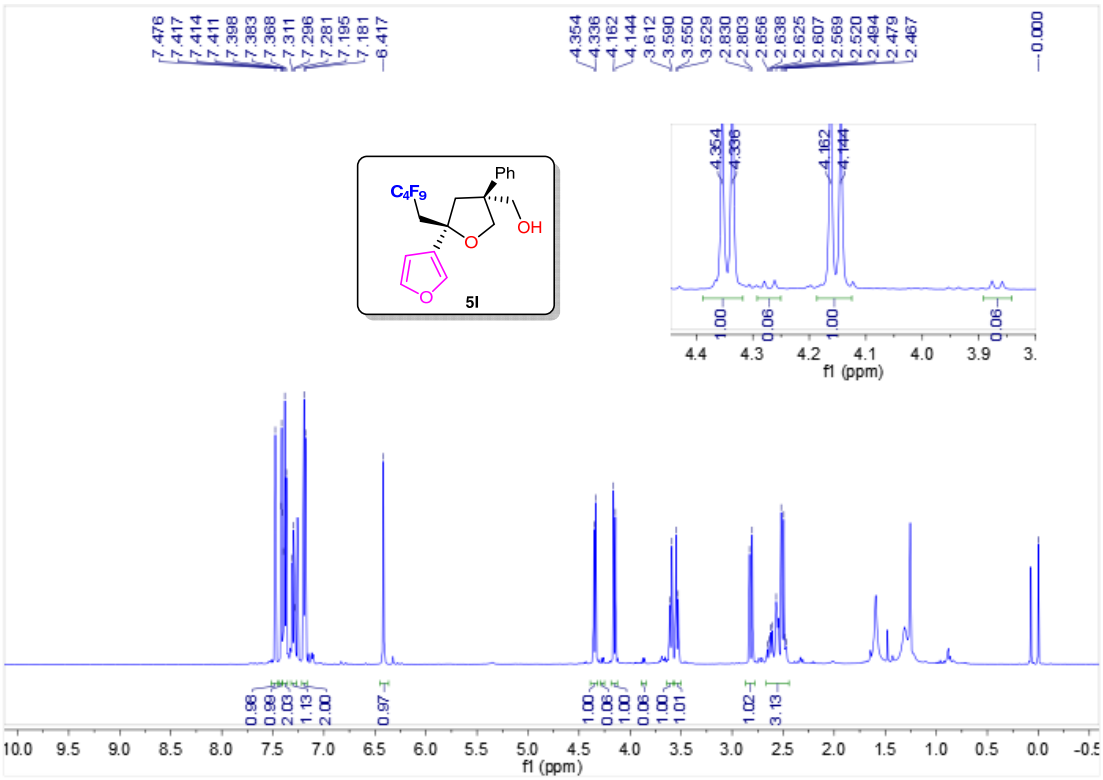
-81.136
-81.166
-81.183
-81.172
-110.785
-110.833
-110.883
-111.471
-111.510
-111.543
-111.576
-111.613
-111.650
-111.737
-111.775
-111.810
-111.839
-111.872
-111.908
-112.504
-112.551
-112.596
-112.635
-124.605
-124.632
-125.846
-125.858
-125.882
-125.893
-125.916
-125.928
Ph
C_4F_9
OH
O
EtO_2C
5H
2.78
2.00
2.04
2.04
f1 (ppm)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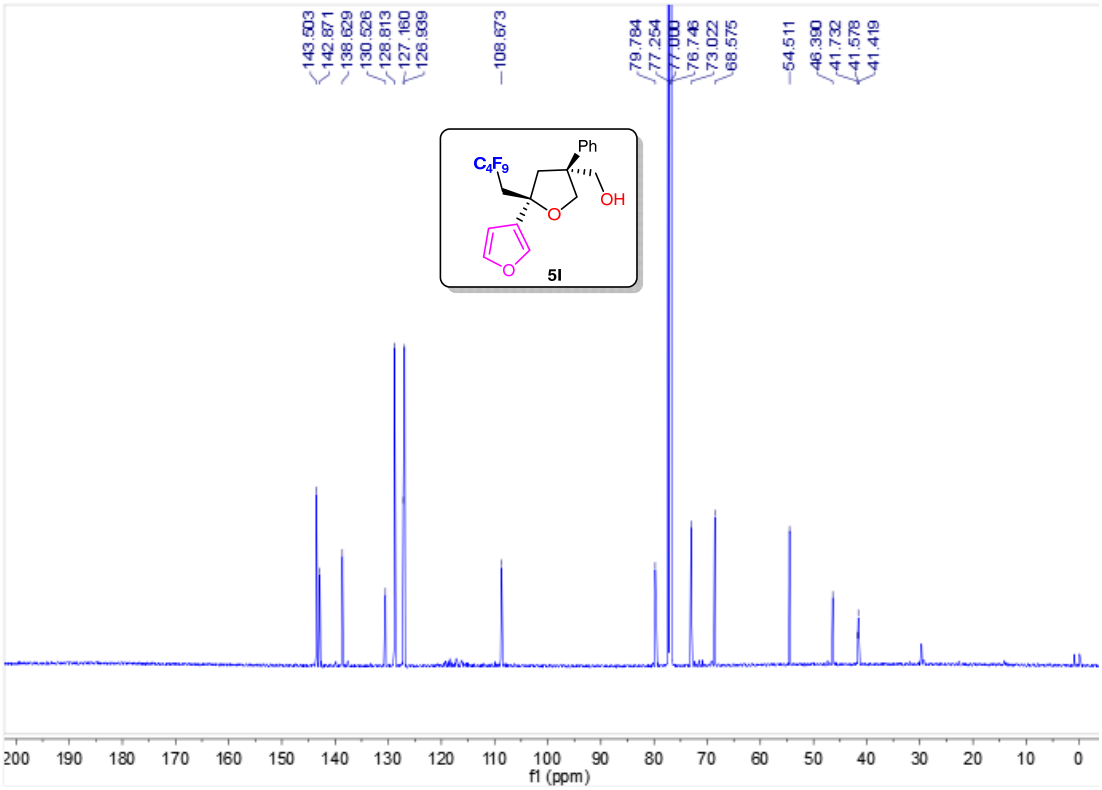
7.414
7.411
7.398
7.383
7.368
7.311
7.296
7.281
7.195
7.181
6.417
4.354
4.336
4.162
4.144
3.612
3.590
3.550
3.529
2.830
2.803
2.656
2.638
2.625
2.607
2.569
2.520
2.494
2.479
2.467
0.000
Ph
C4F9
OH
O
O
5l
f1 (ppm)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142.871
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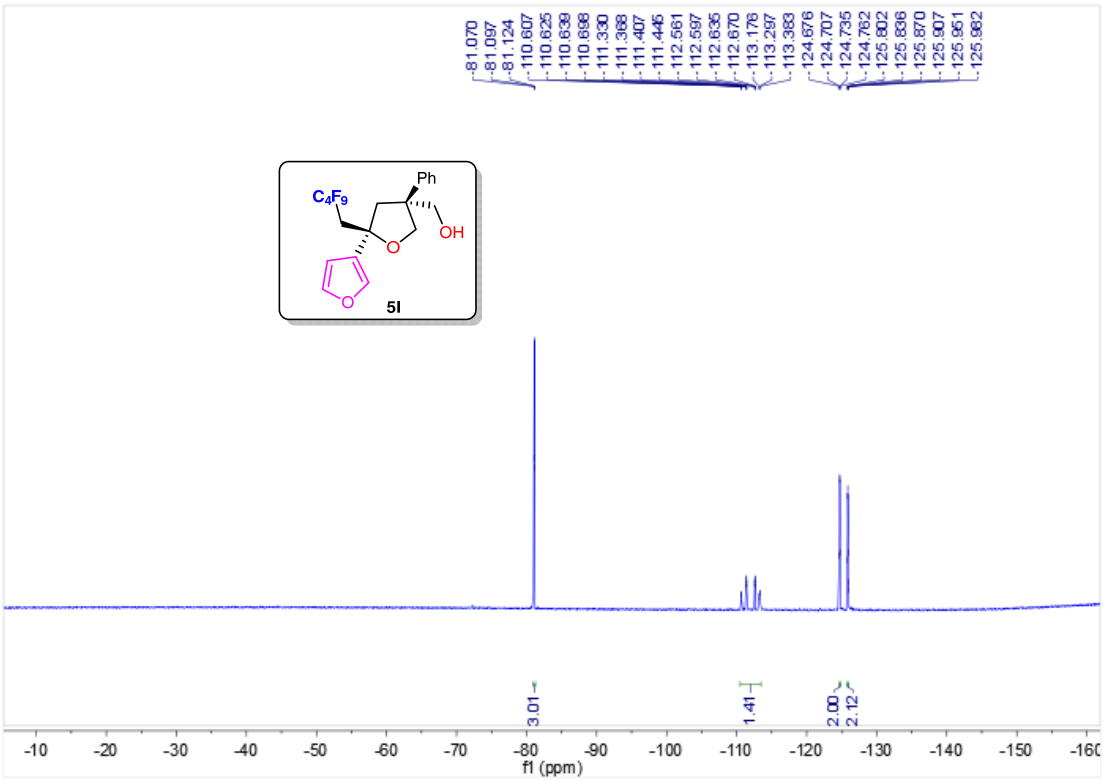
128.813
127.160
126.939
108.673
79.784
77.254
77.000
76.748
73.022
68.575
54.511
46.390
41.732
41.578
41.419
Ph
C4F9
OH
O
O
5l
f1 (ppm)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111.445
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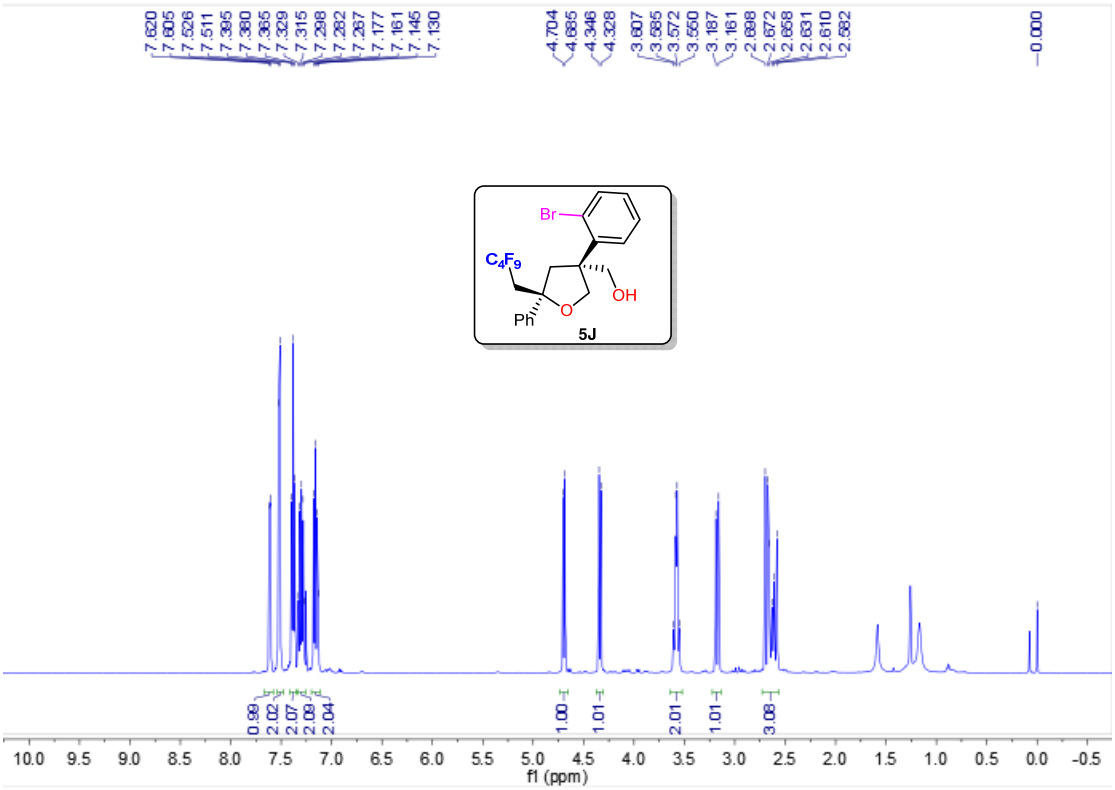
112.670
113.176
113.297
113.383
124.676
124.707
124.735
124.762
125.802
125.836
125.870
125.907
125.951
125.982
Ph
C4F9
OH
O
O
5I
3.01
1.41
2.00
2.12
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)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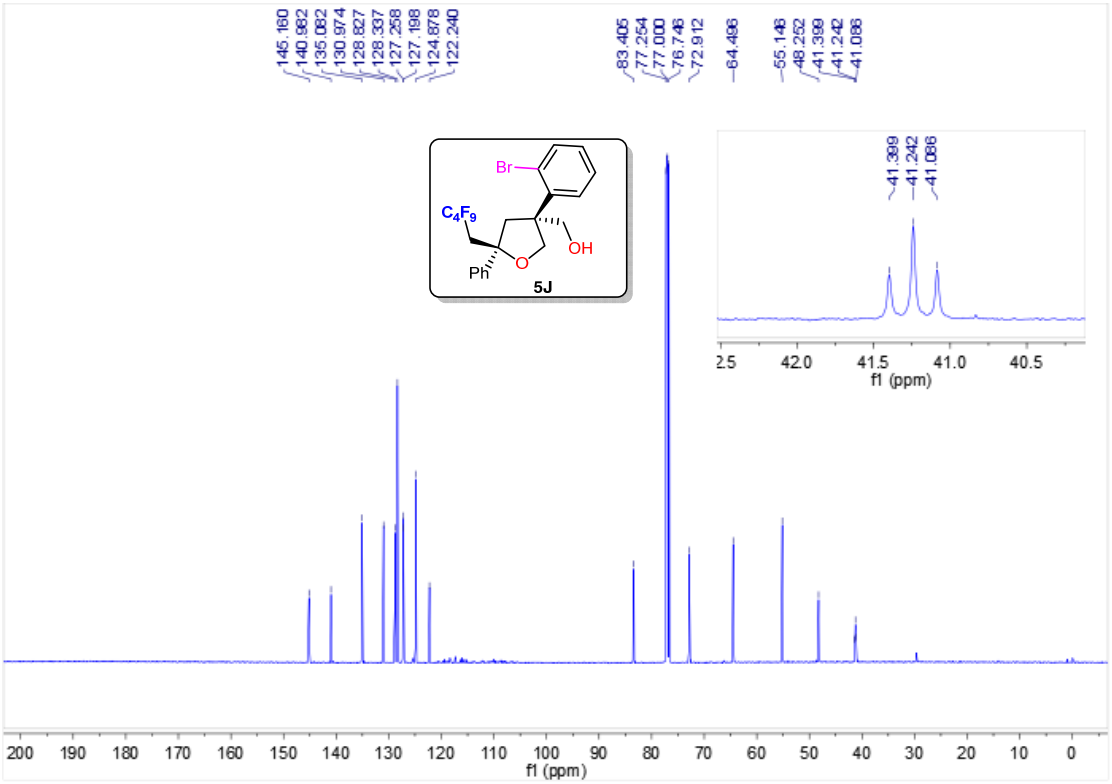
7.395
7.380
7.365
7.329
7.315
7.298
7.282
7.267
7.177
7.161
7.145
7.130
4.704
4.685
4.346
4.328
3.607
3.585
3.572
3.550
3.187
3.161
2.898
2.672
2.658
2.631
2.610
2.582
0.000
Br
C4F9
OH
Ph
O
5J
0.99
2.02
2.07
2.09
2.04
1.00
1.01
2.01
1.01
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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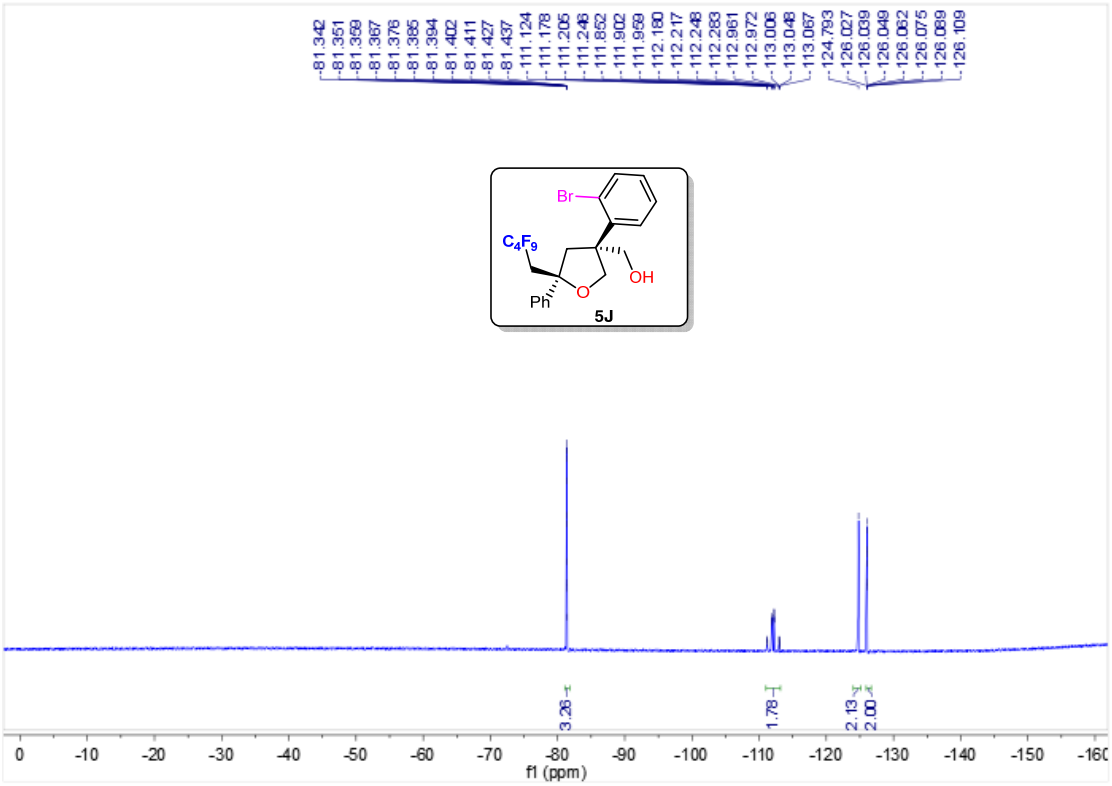
130.974
128.827
128.337
127.258
127.198
124.878
122.240
83.405
77.254
77.000
76.746
72.912
64.486
55.143
48.252
41.399
41.242
41.086
Br
C4F9
OH
O
Ph
5J
41.399
41.242
41.086
42.5
42.0
41.5
41.0
40.5
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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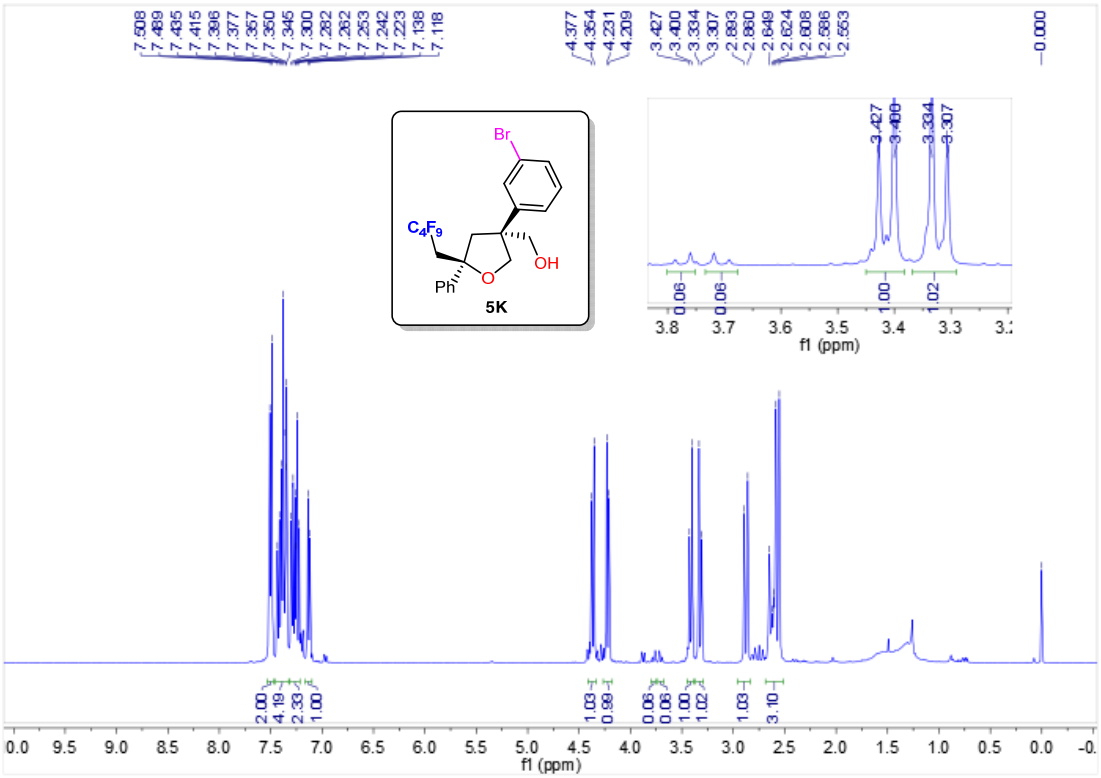
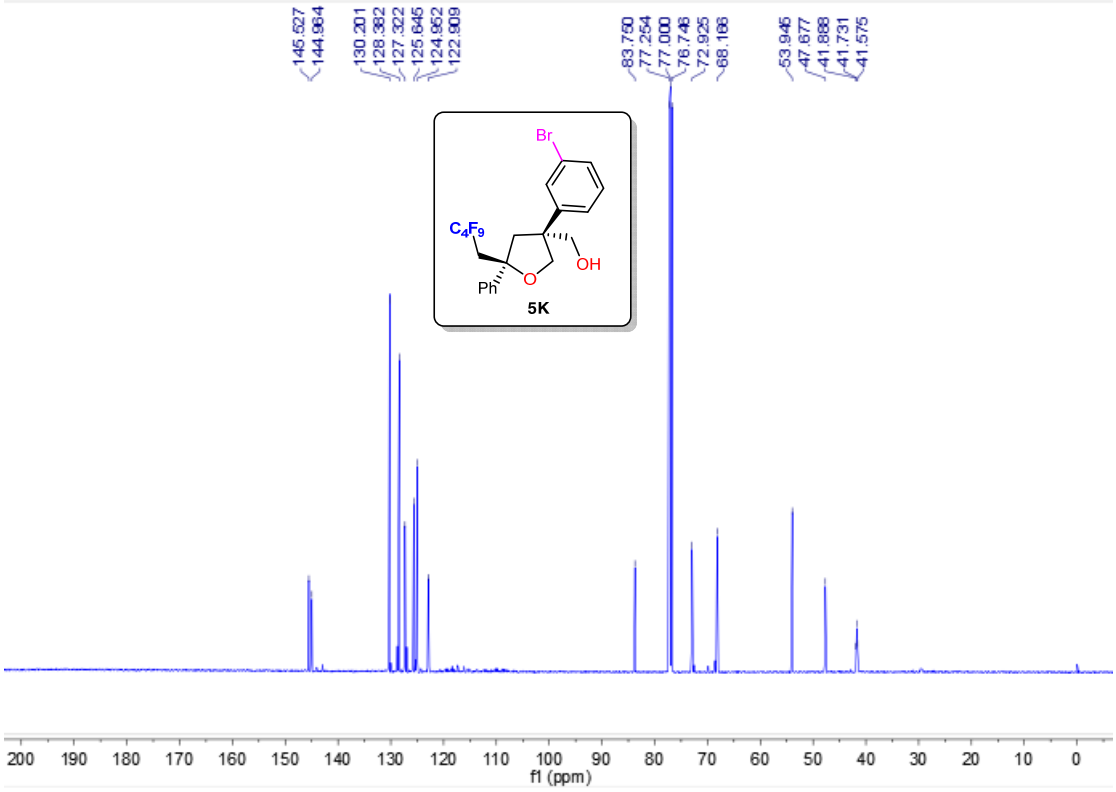
111.178
111.205
111.243
111.852
111.902
111.959
112.180
112.217
112.248
112.283
112.961
112.972
113.006
113.048
113.067
124.793
126.027
126.039
126.049
126.062
126.075
126.089
126.109
Br
C4F9
OH
O
Ph
5J
3.26
1.78
2.13
2.00
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)


Br
C4F9
OH
Ph
O
5K
f1 (ppm)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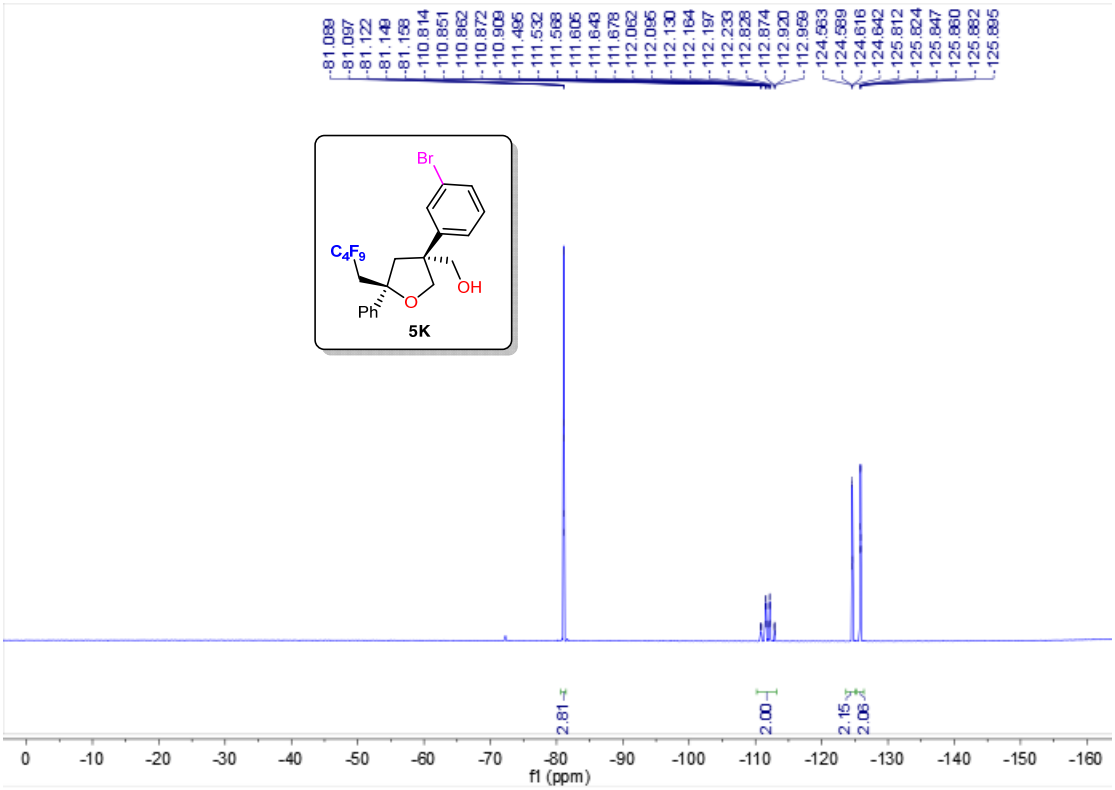
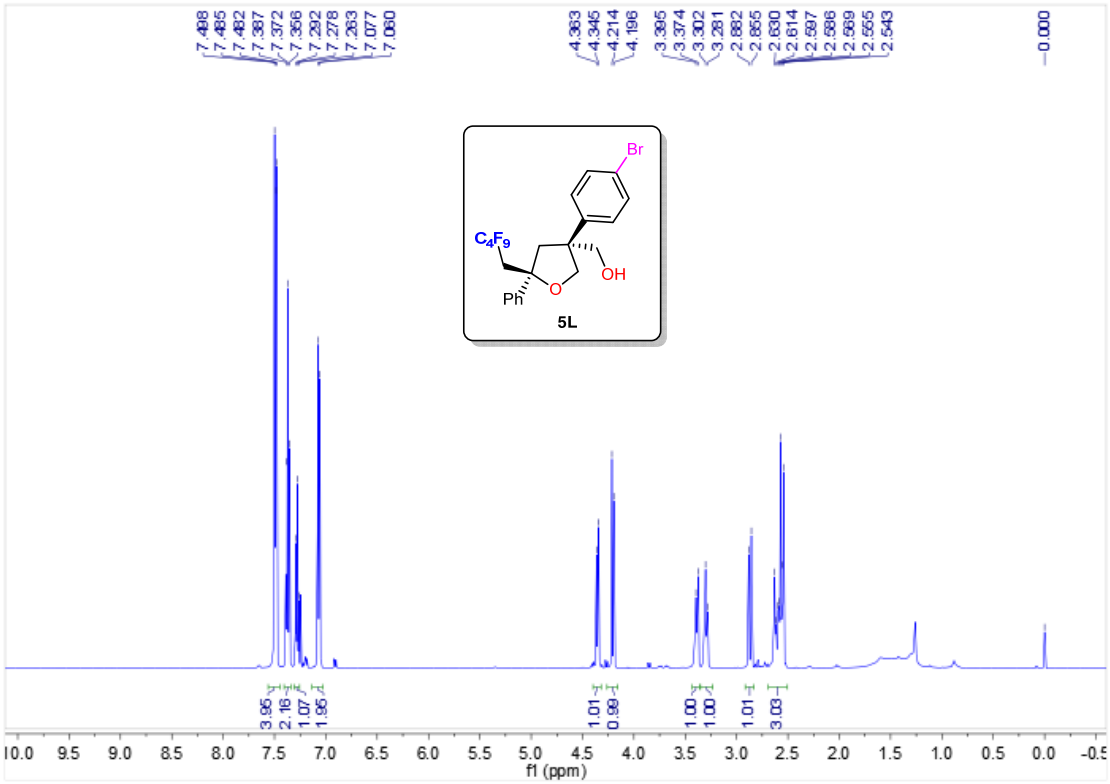
C4F9
OH
Ph
O
5K
f1 (ppm)


Br
C4F9
OH
O
Ph
5K
f1 (ppm)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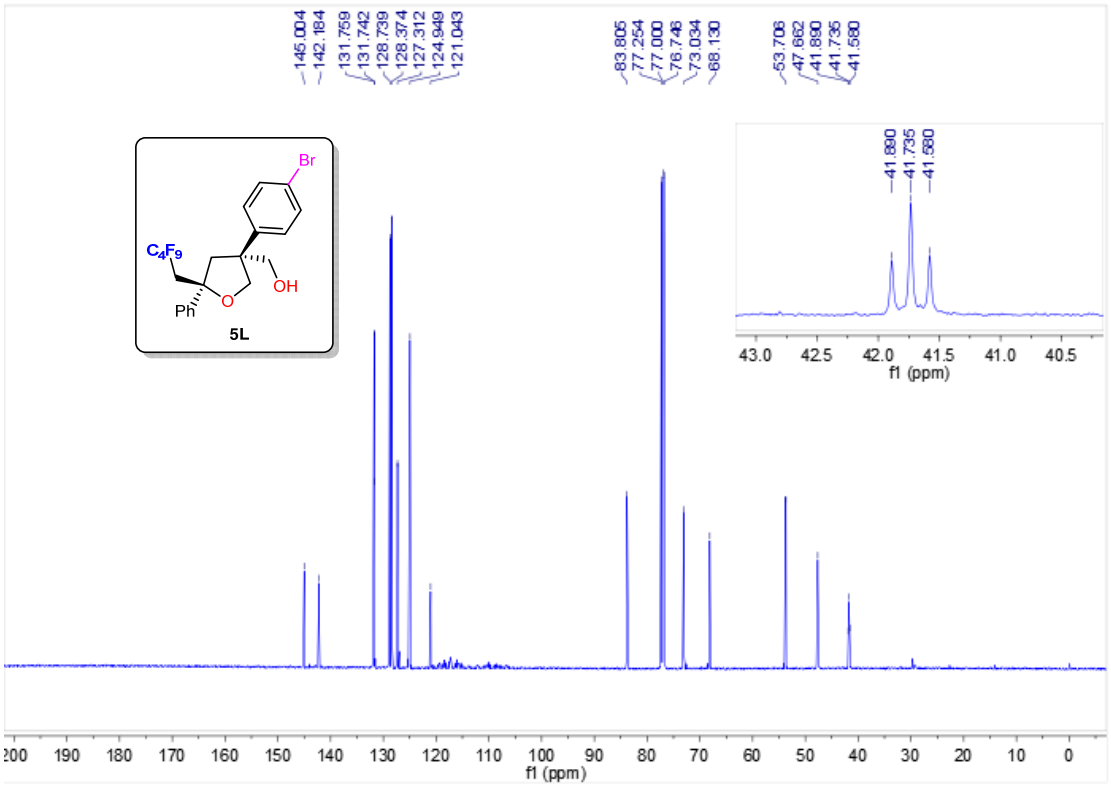
C4F9
OH
O
Ph
5L
f1 (ppm)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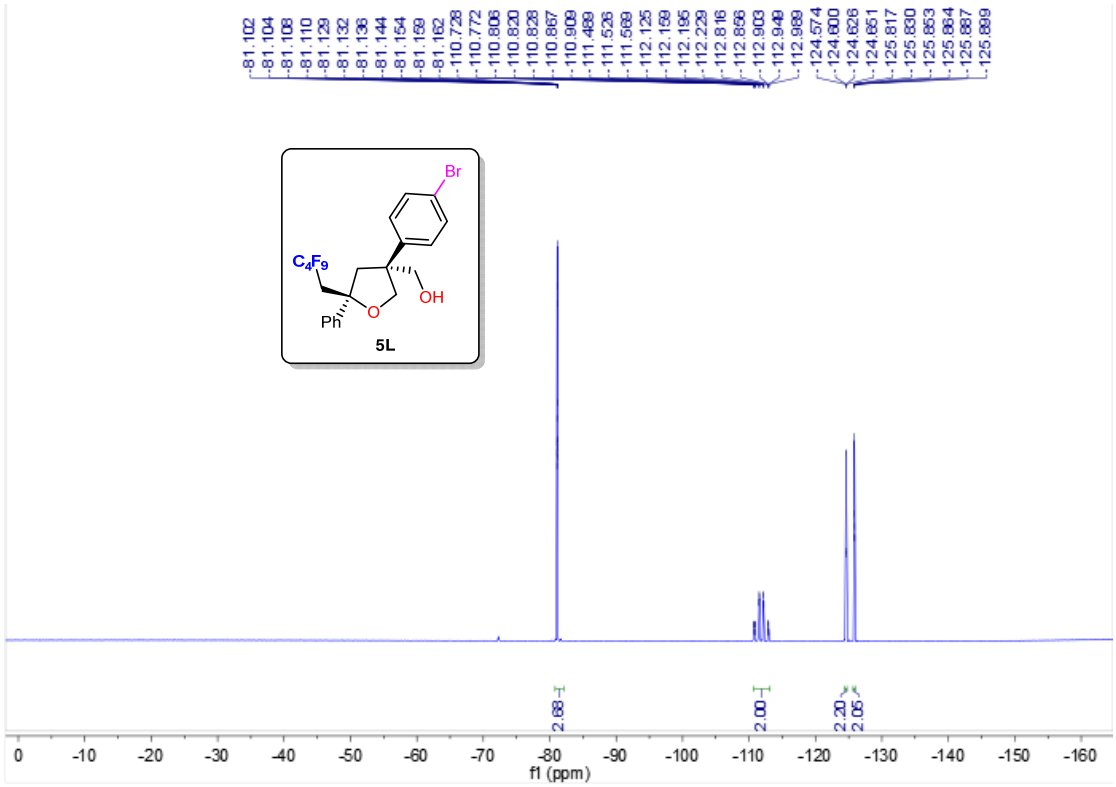
142.184
131.759
131.742
128.739
128.374
127.312
124.949
121.043
83.805
77.254
77.000
76.746
73.034
68.130
53.706
47.662
41.890
41.735
41.580
Br
C4F9
OH
Ph
O
5L
f1 (ppm)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111.526
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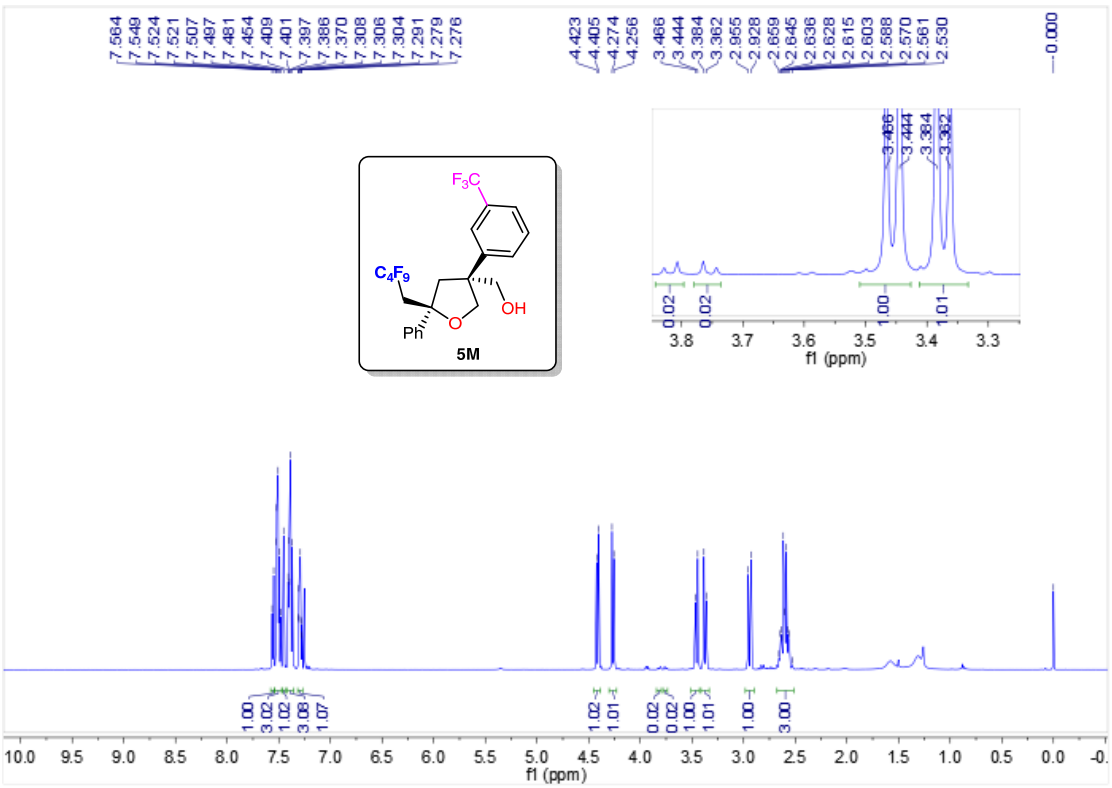
112.159
112.195
112.229
112.816
112.856
112.903
112.949
112.989
124.574
124.600
124.626
124.651
125.817
125.830
125.853
125.864
125.887
125.899
Br
C4F9
OH
Ph
O
5L
2.88
2.00
2.20
2.05
f1 (ppm)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7.549
7.524
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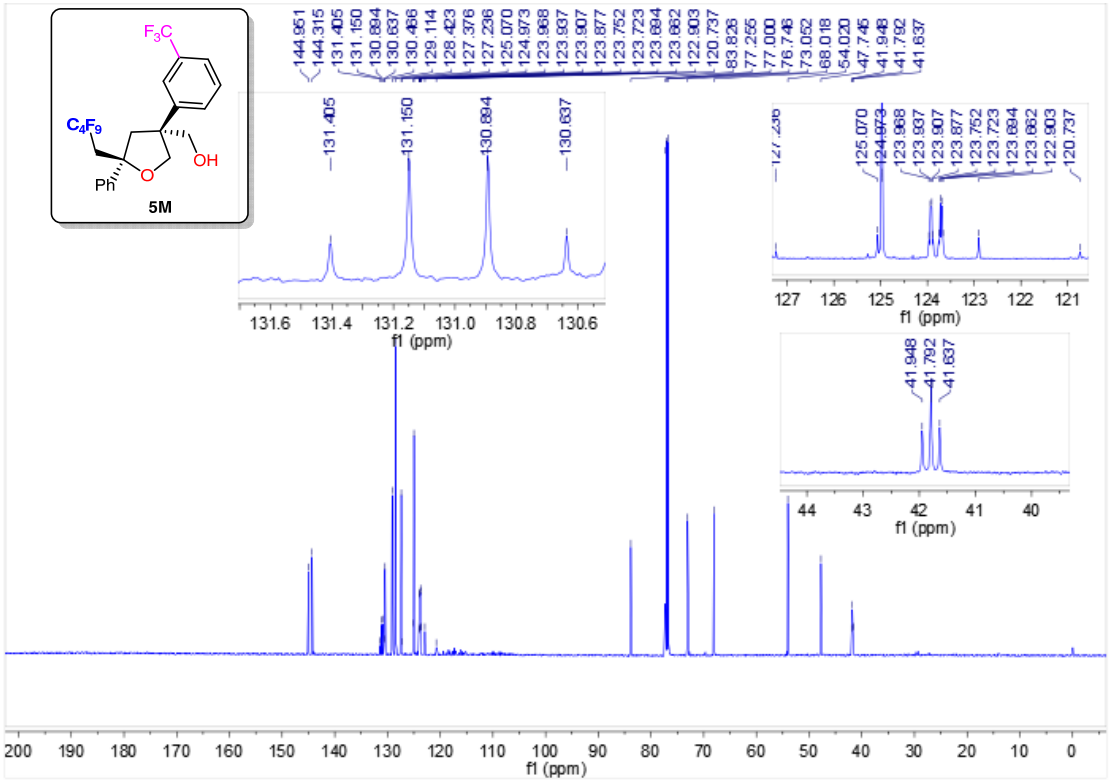
7.497
7.481
7.454
7.409
7.401
7.397
7.386
7.370
7.308
7.306
7.304
7.291
7.279
7.276
4.423
4.405
4.274
4.256
3.466
3.444
3.384
3.362
2.955
2.928
2.659
2.645
2.636
2.628
2.615
2.603
2.588
2.570
2.561
2.530
0.000
F3C
C4F9
OH
Ph
O
5M
f1 (ppm)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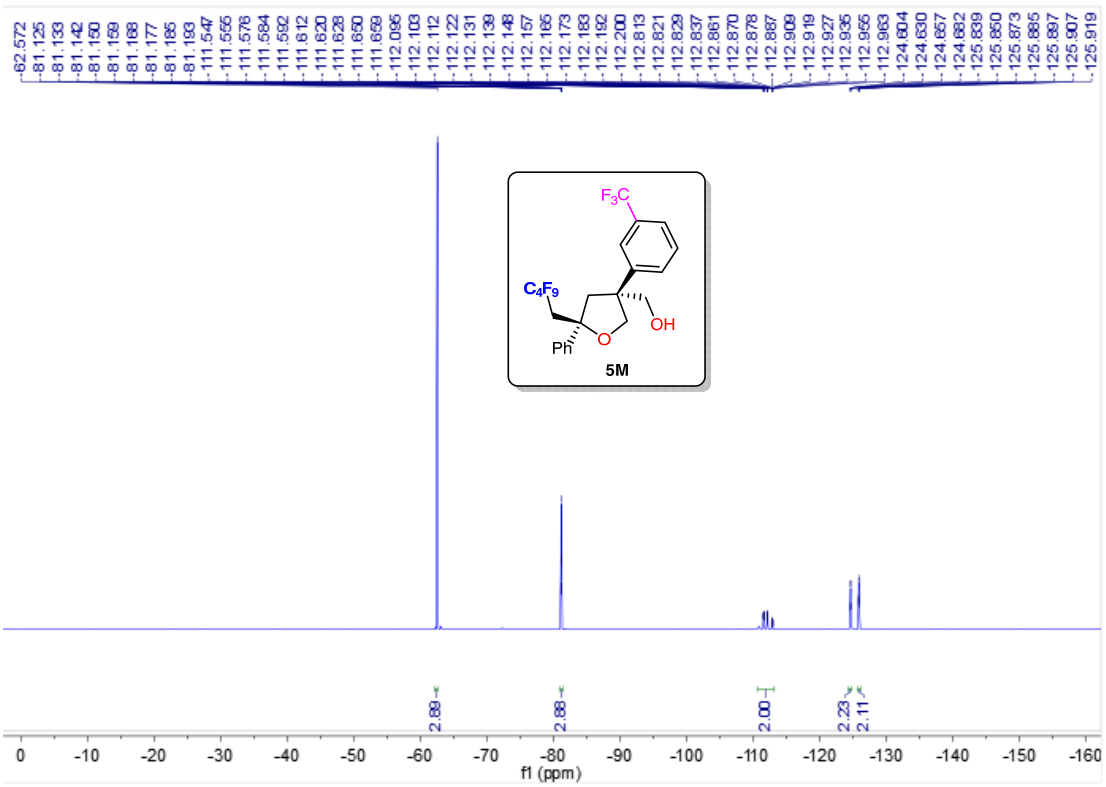
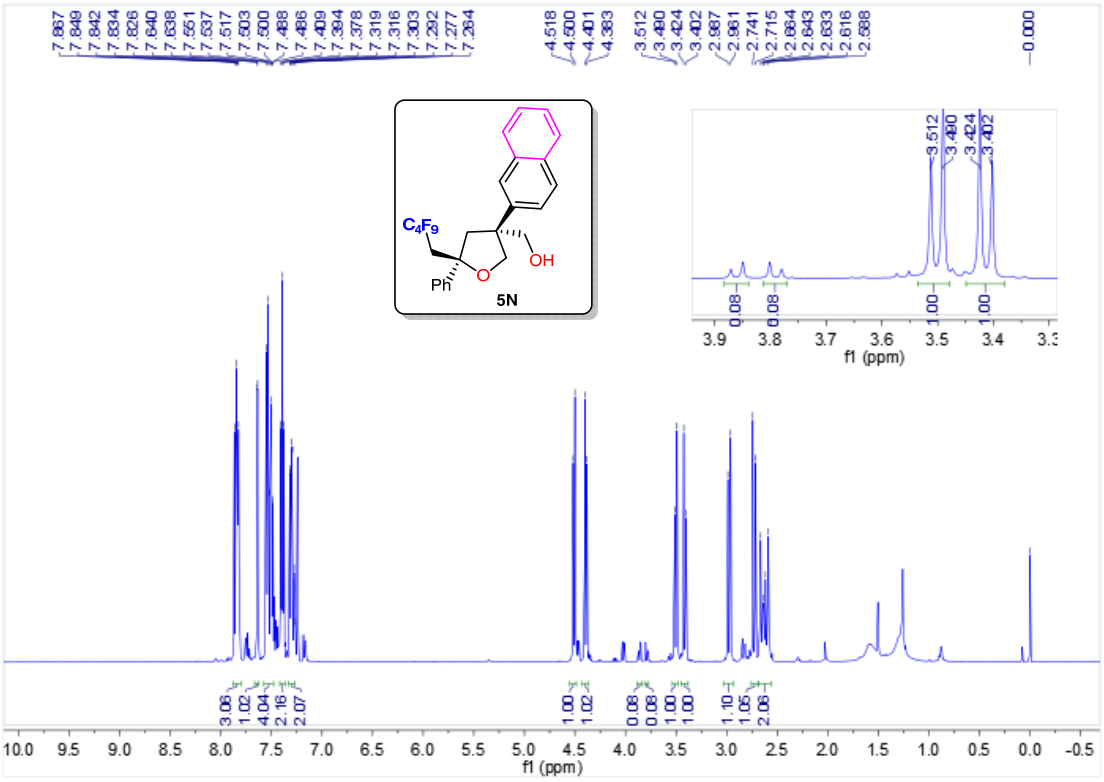
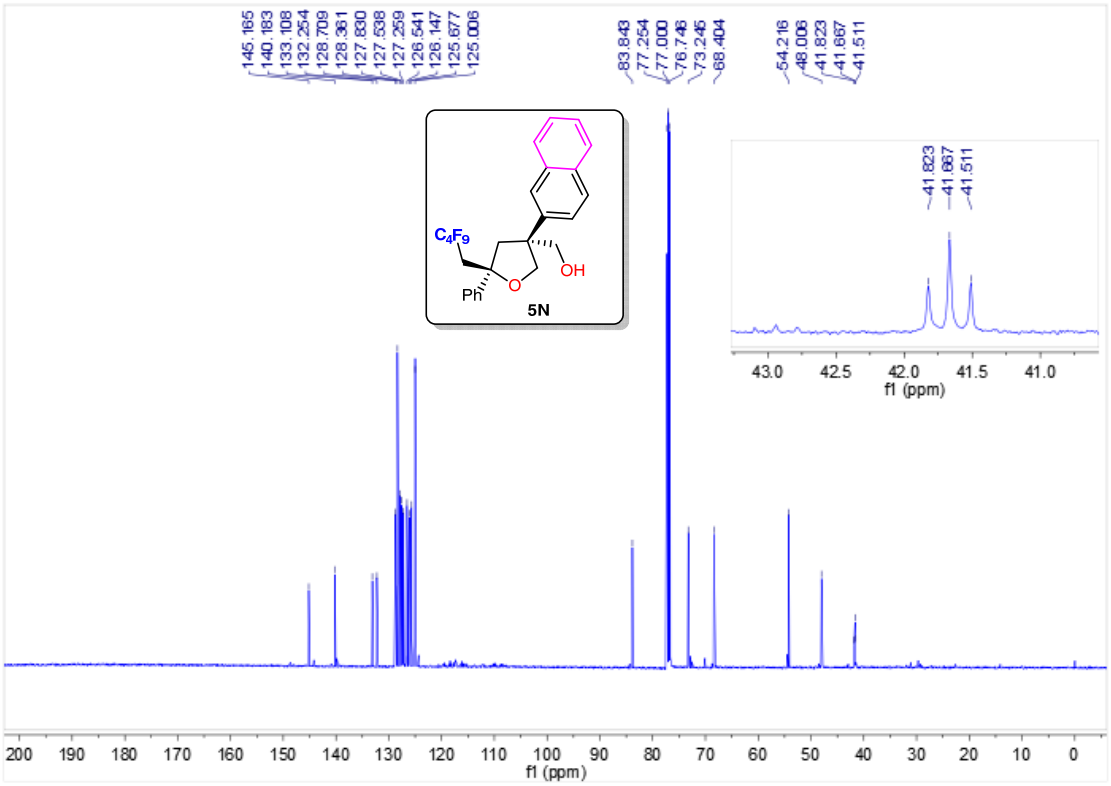
144.315
131.405
131.150
130.894
130.637
130.486
129.114
128.423
127.376
127.236
125.070
124.973
123.968
123.937
123.907
123.877
123.752
123.723
123.694
123.662
122.903
120.737
83.826
77.255
77.000
76.745
73.052
68.018
54.020
47.745
41.948
41.792
41.637
F3C
C4F9
OH
Ph
O
5M
f1 (ppm)


F3C
C4F9
OH
Ph
O
5M
f1 (ppm)


C4F9
OH
Ph
O
5N
f1 (ppm)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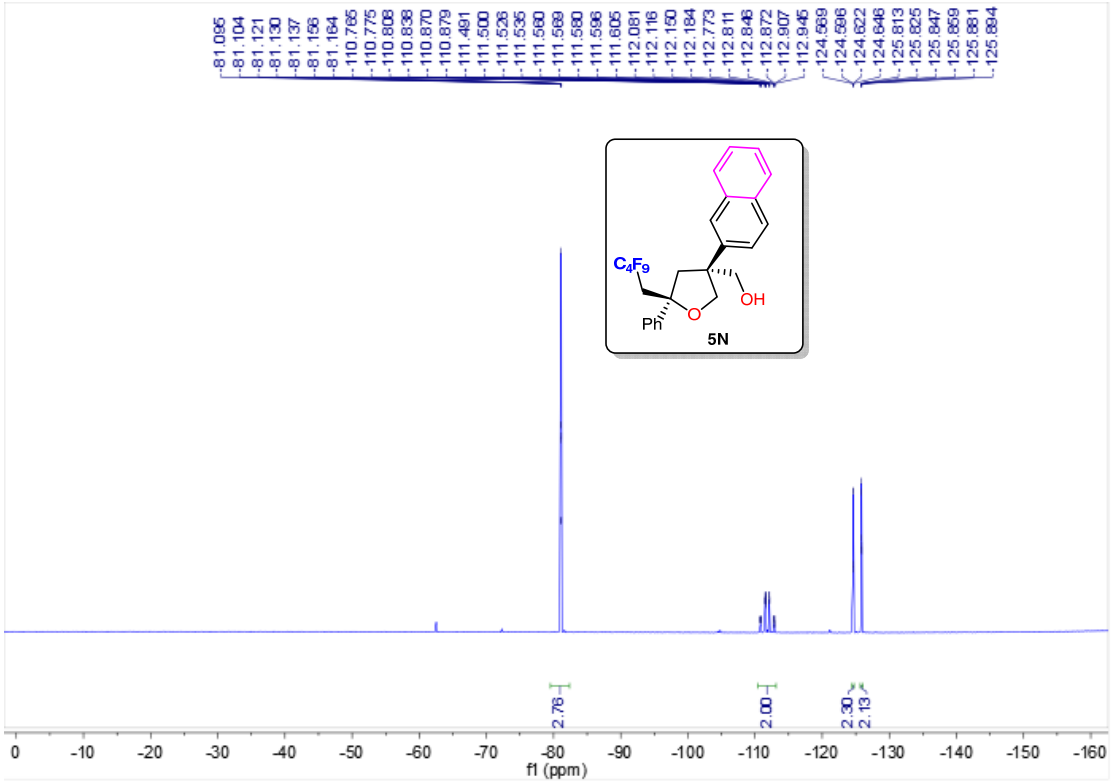
140.183
133.108
132.254
128.709
128.361
127.830
127.538
127.259
126.541
126.147
125.677
125.006
83.843
77.254
77.000
76.746
73.245
68.404
54.216
48.006
41.823
41.667
41.511
C4F9
OH
O
Ph
5N
41.823
41.667
41.511
43.0
42.5
42.0
41.5
41.0
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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81.130
81.137
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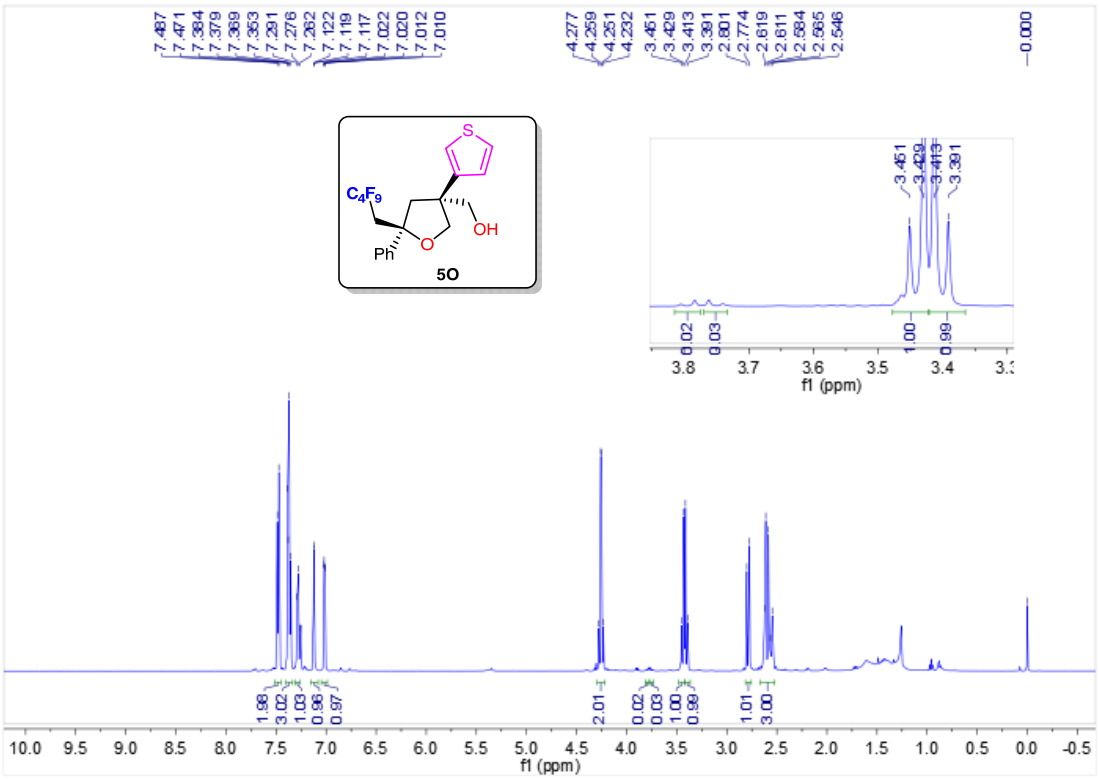
81.164
110.765
110.775
110.808
110.836
110.870
110.879
111.491
111.500
111.526
111.535
111.560
111.569
111.580
111.596
111.605
112.081
112.116
112.150
112.184
112.773
112.811
112.845
112.872
112.907
112.945
124.569
124.596
124.622
124.646
125.813
125.825
125.847
125.859
125.881
125.894
C4F9
OH
O
Ph
5N
2.76
2.00
2.30
2.13
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)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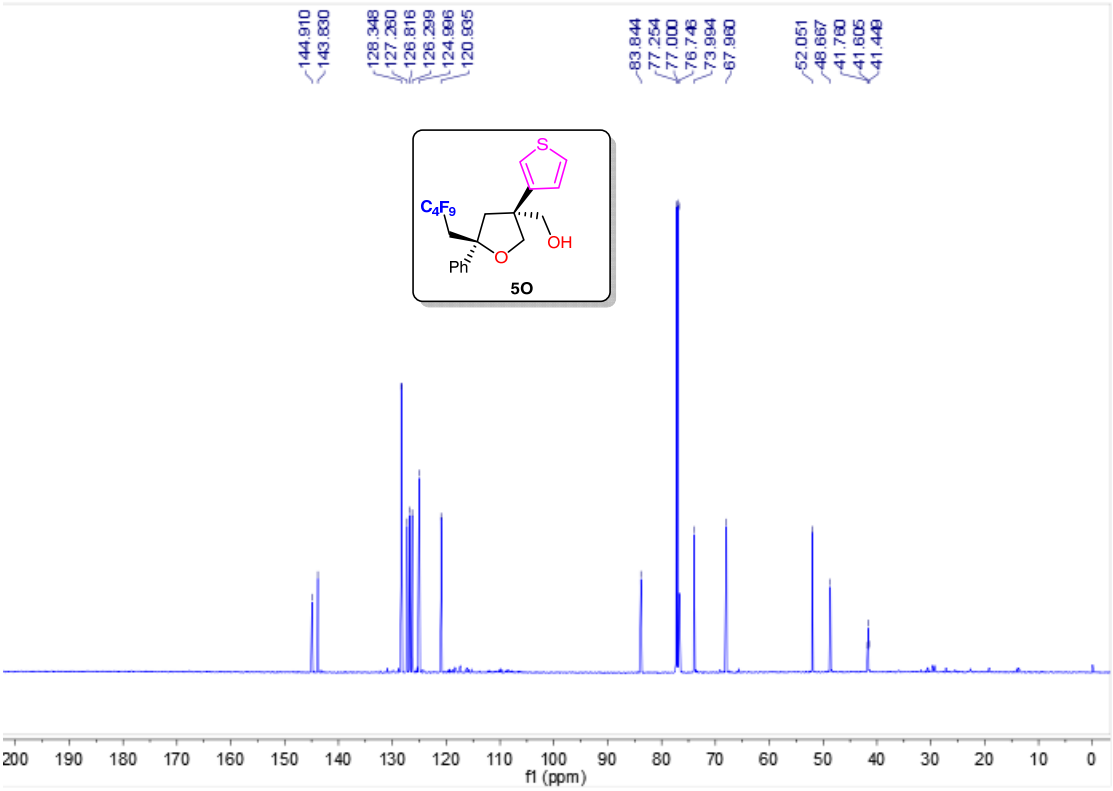
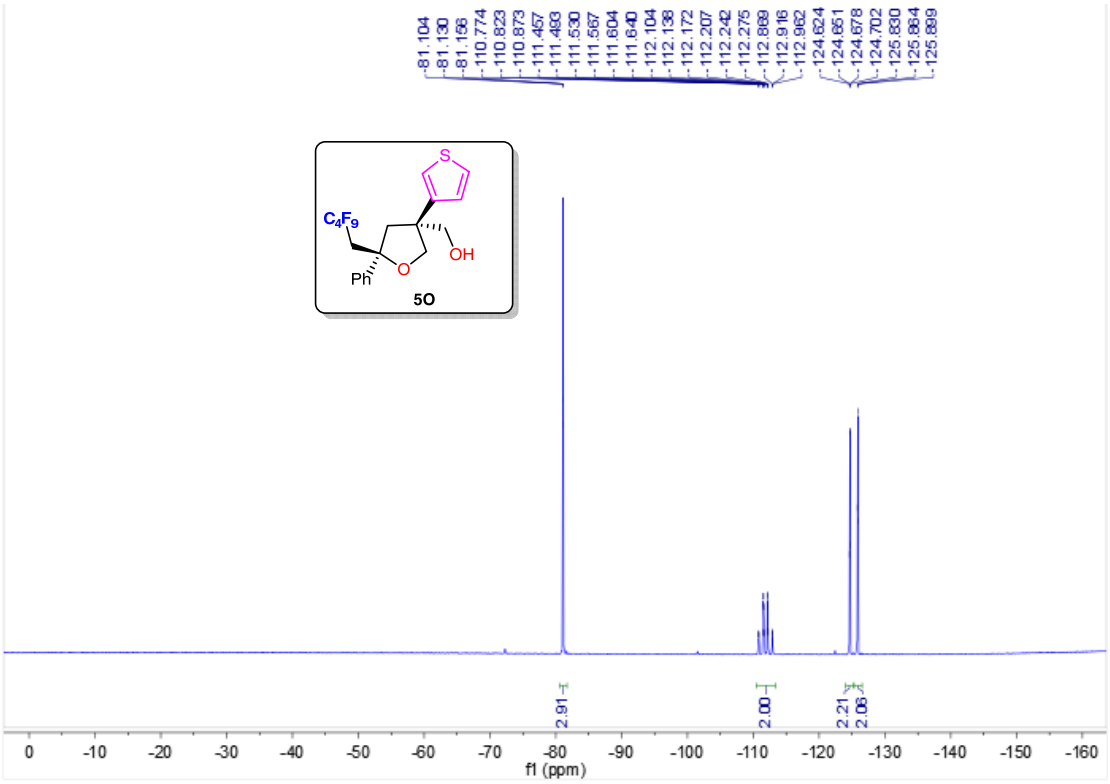
7.471
7.384
7.379
7.369
7.353
7.291
7.276
7.262
7.122
7.119
7.117
7.022
7.020
7.012
7.010
4.277
4.259
4.251
4.232
3.451
3.429
3.413
3.391
2.801
2.774
2.619
2.611
2.584
2.565
2.546
-0.000
S
C4F9
OH
O
Ph
5O
f1 (ppm)


144.910
143.830
128.348
127.260
126.816
126.299
124.996
120.935
83.844
77.254
77.000
76.746
73.994
67.960
52.051
48.667
41.760
41.605
41.449
S
C4F9
OH
O
Ph
5O
f1 (ppm)


81.104
81.130
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110.823
110.873
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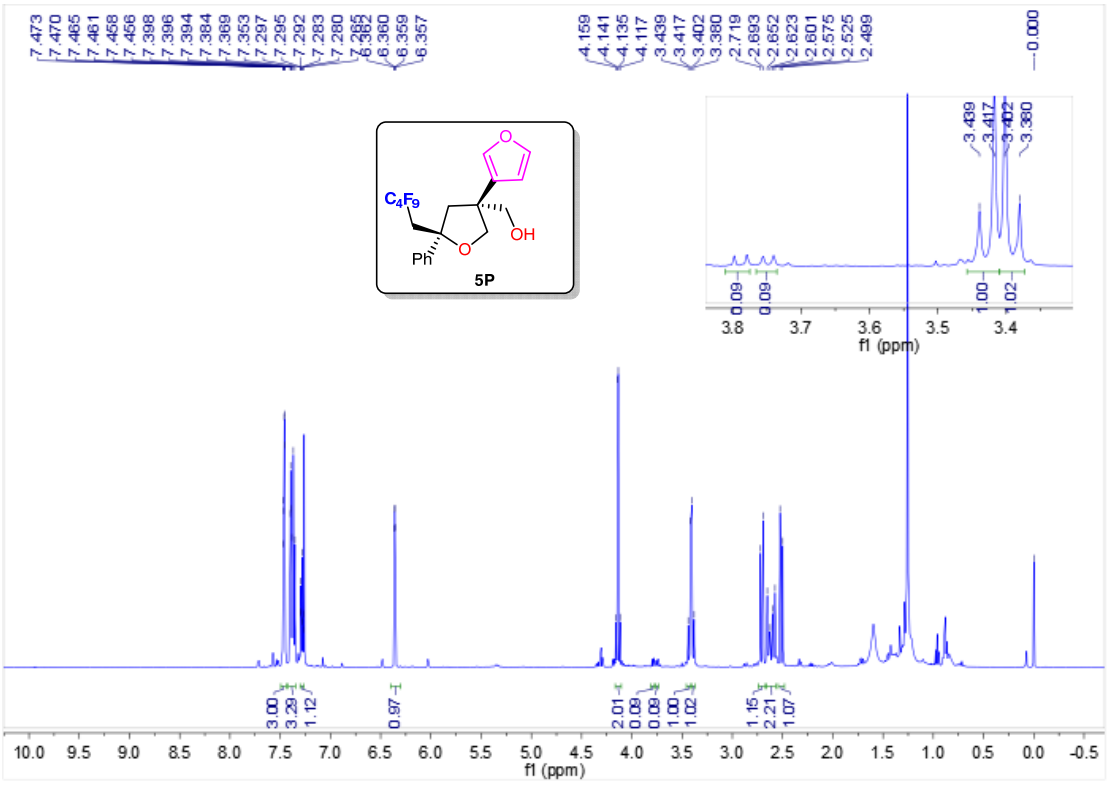
111.493
111.530
111.567
111.604
111.640
112.104
112.138
112.172
112.207
112.242
112.275
112.889
112.916
112.962
124.624
124.651
124.678
124.702
125.630
125.664
125.699
S
C4F9
OH
O
Ph
5O
2.91
2.00
2.21
2.06
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)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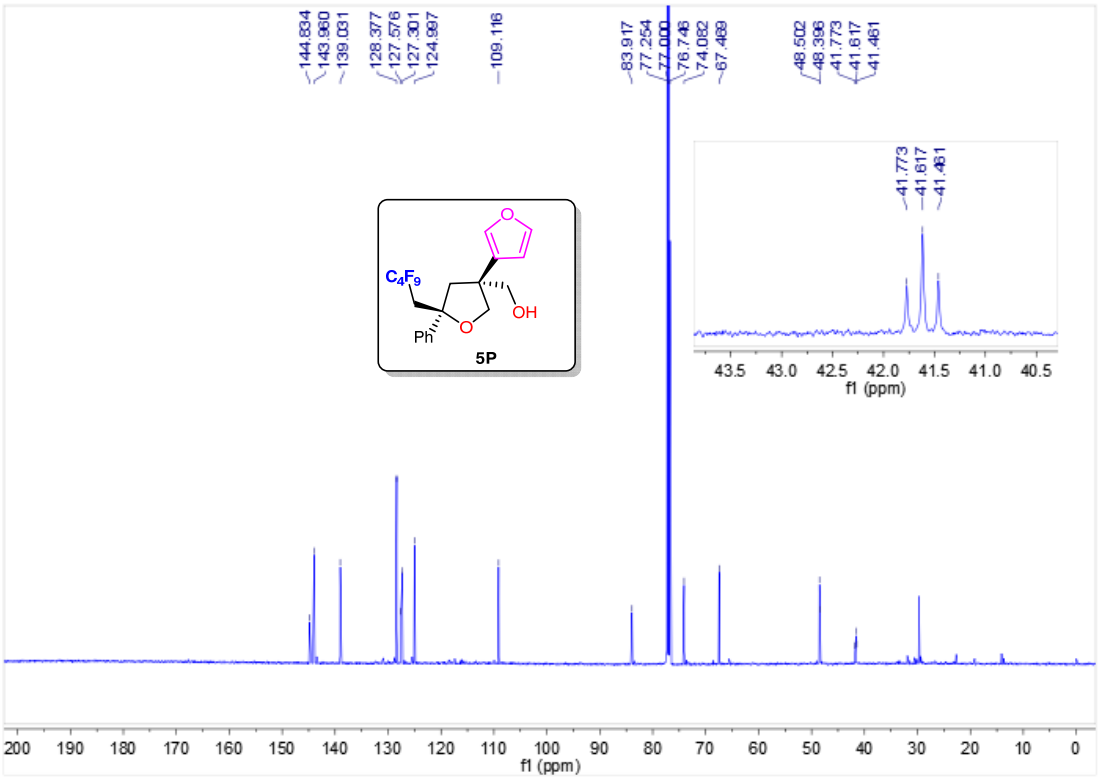
7.470
7.465
7.461
7.458
7.456
7.398
7.396
7.394
7.384
7.369
7.353
7.297
7.295
7.292
7.283
7.280
6.360
6.359
6.357
4.159
4.141
4.135
4.117
3.439
3.417
3.402
3.380
2.719
2.693
2.652
2.623
2.601
2.575
2.525
2.499
0.000
O
C4F9
OH
O
Ph
5P
3.439
3.417
3.380
0.09
0.09
1.00
1.02
3.8
3.7
3.6
3.5
3.4
f1 (ppm)
3.00
3.29
1.12
0.97
2.01
0.09
0.09
1.00
1.02
1.15
2.21
1.07
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
-0.5
f1 (ppm)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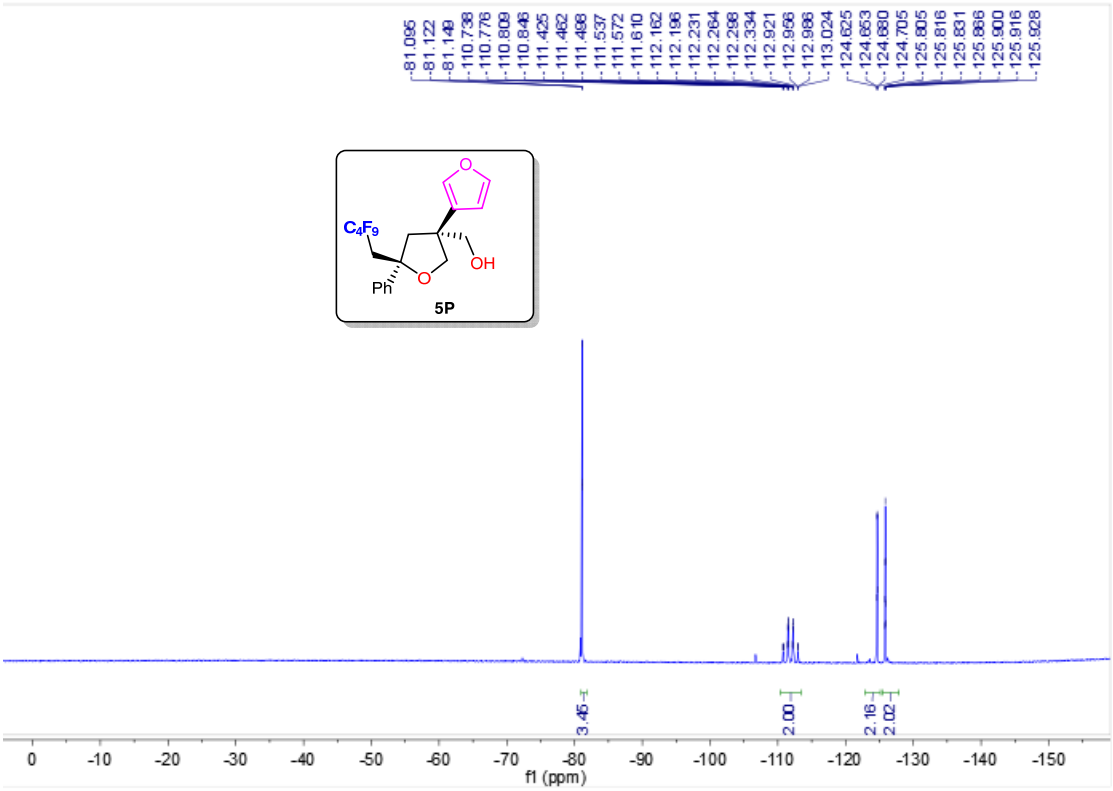
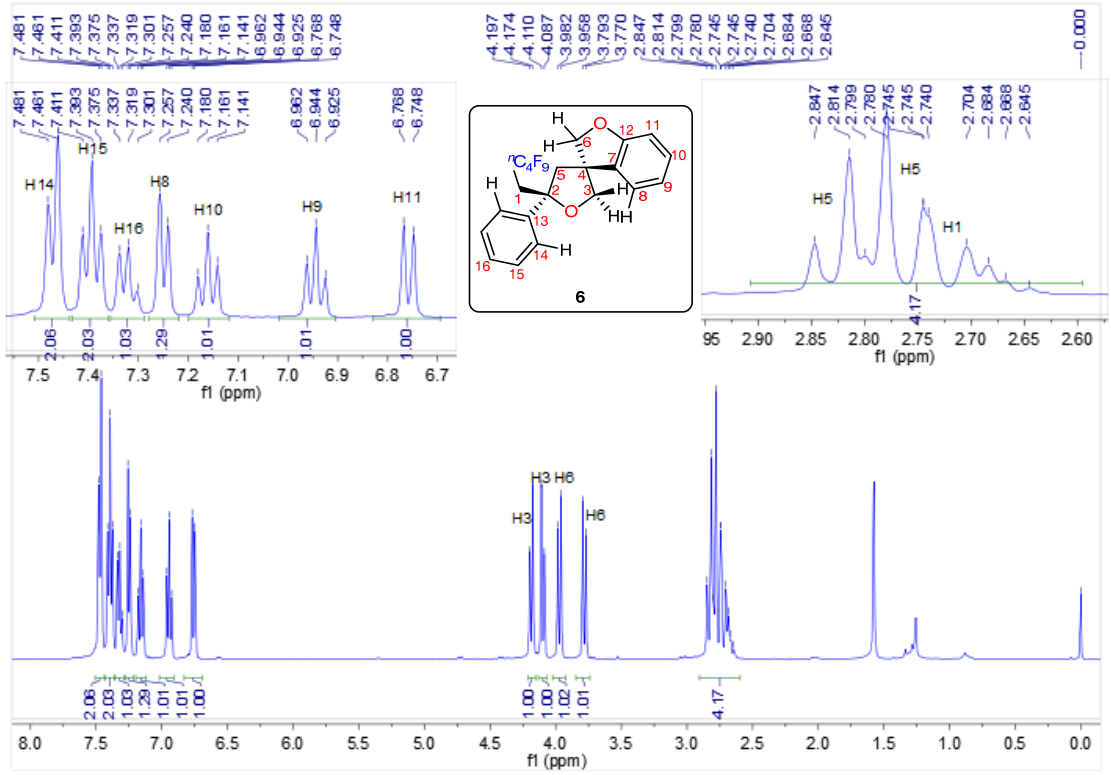
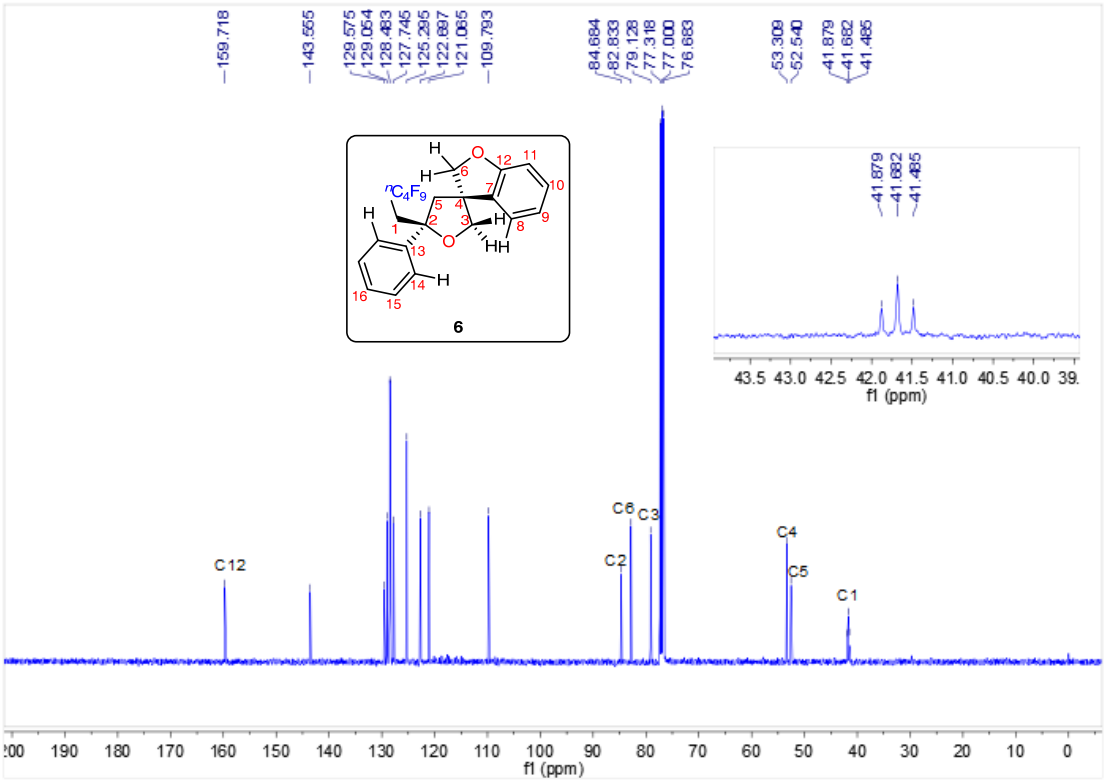
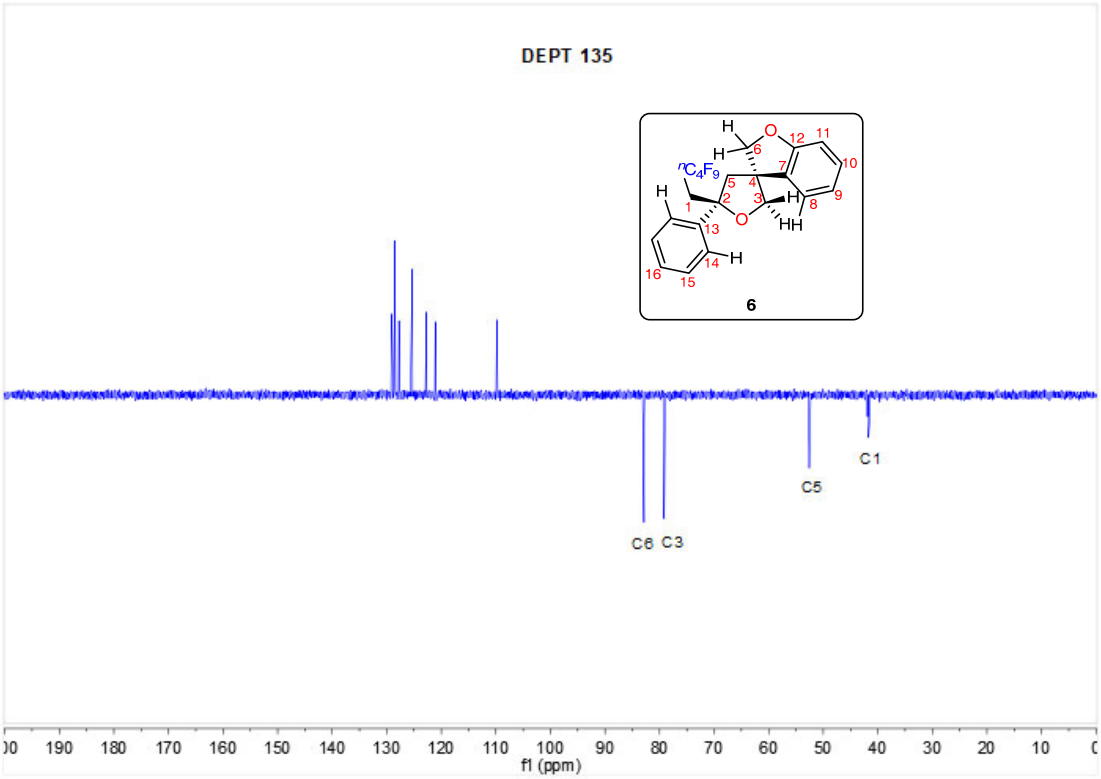
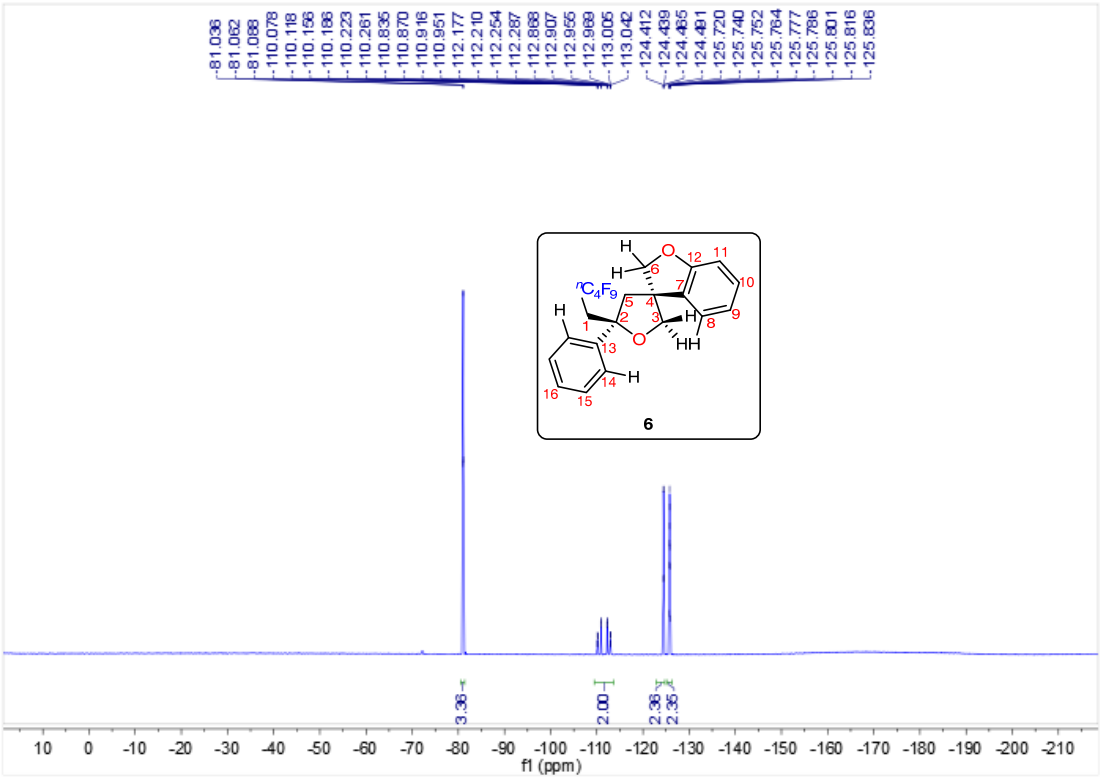
143.960
139.031
128.377
127.576
127.301
124.997
109.116
83.917
77.254
77.000
76.745
74.082
67.468
48.502
48.396
41.773
41.617
41.461
C4F9
OH
Ph
O
5P
41.773
41.617
41.461
43.5 43.0 42.5 42.0 41.5 41.0 40.5
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

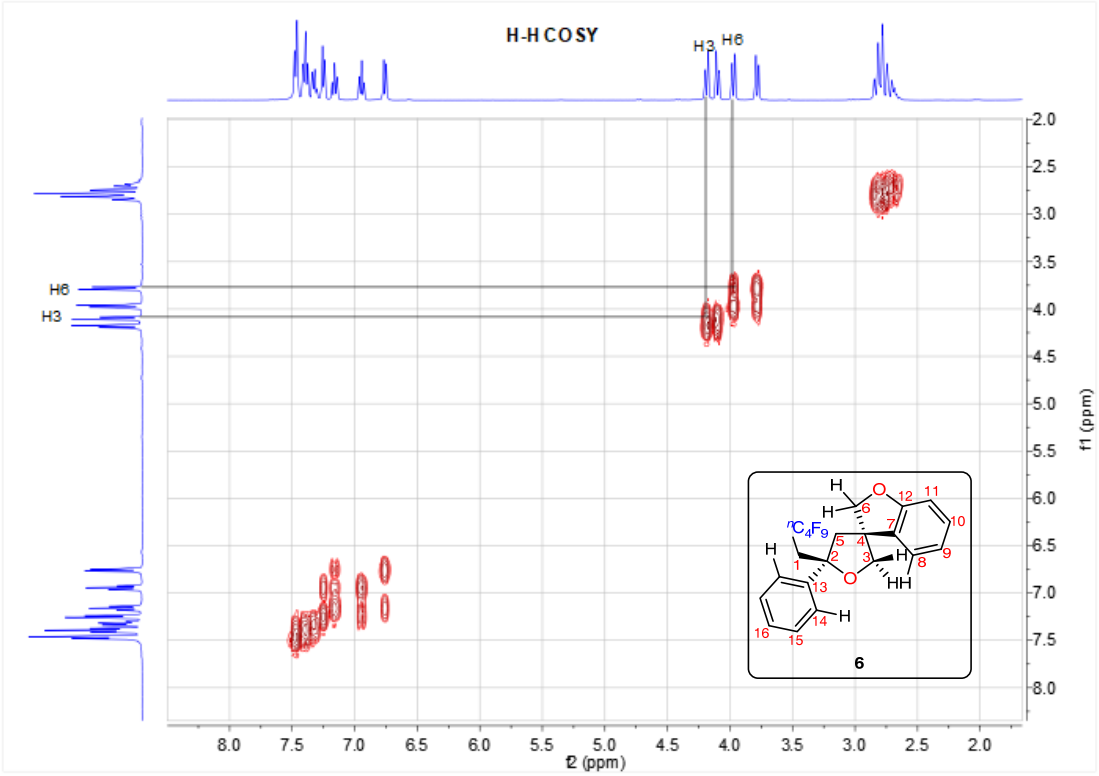

81.095
81.122
81.149
110.738
110.776
110.809
110.846
111.425
111.462
111.498
111.537
111.572
111.610
112.162
112.196
112.231
112.264
112.298
112.334
112.921
112.956
112.986
113.024
124.625
124.653
124.680
124.705
125.805
125.816
125.831
125.866
125.900
125.916
125.928
C4F9
OH
Ph
O
5P
3.45
2.00
2.16
2.02
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

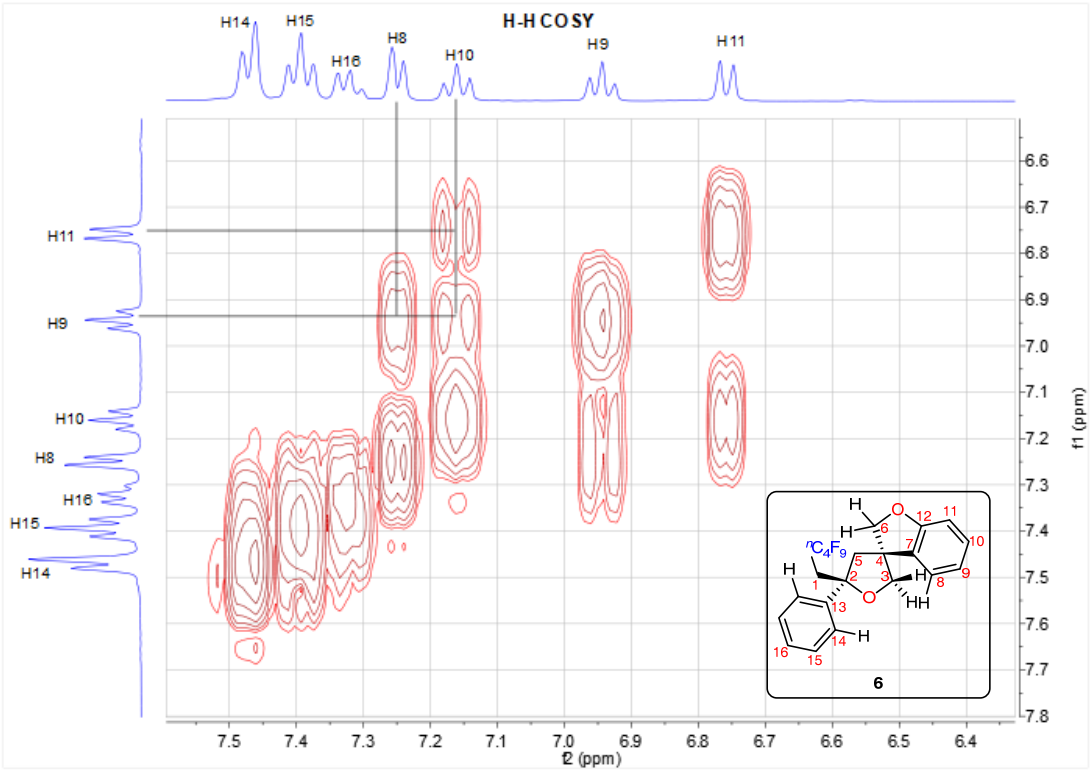

H14
H15
H16
H8
H10
H9
H11
H5
H1
H3
H6
f1 (ppm)
6

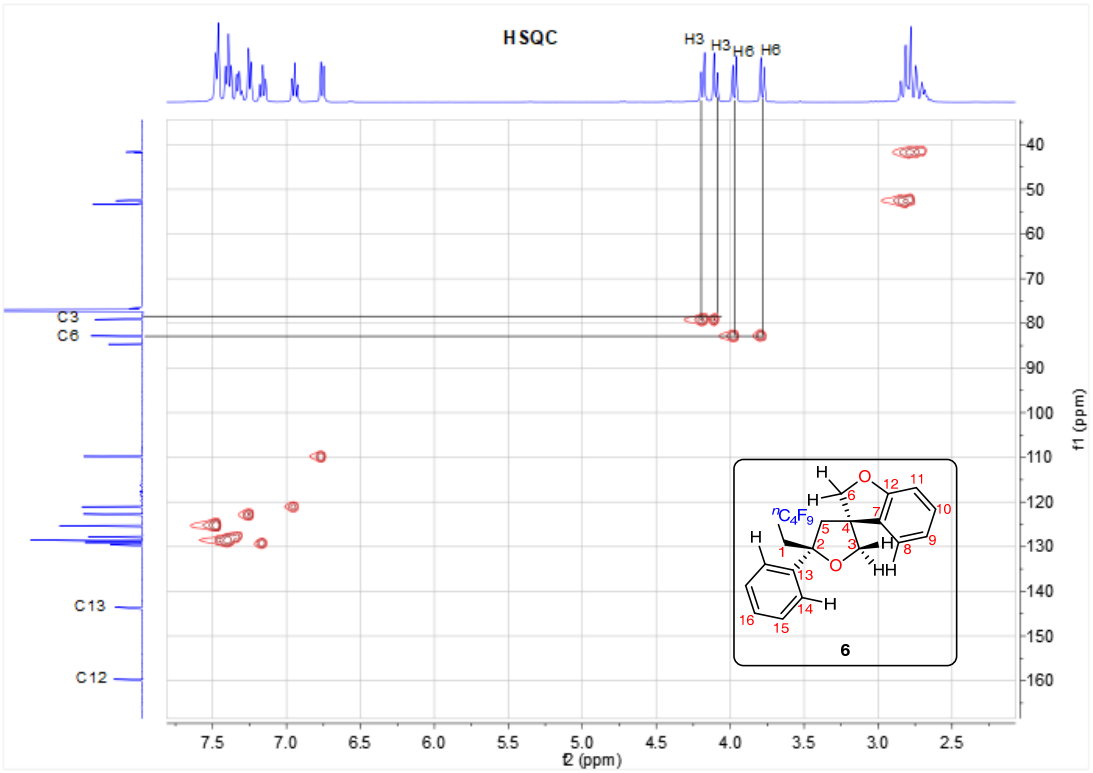

C12
C6
C3
C2
C4
C5
C1
f1 (ppm)
6

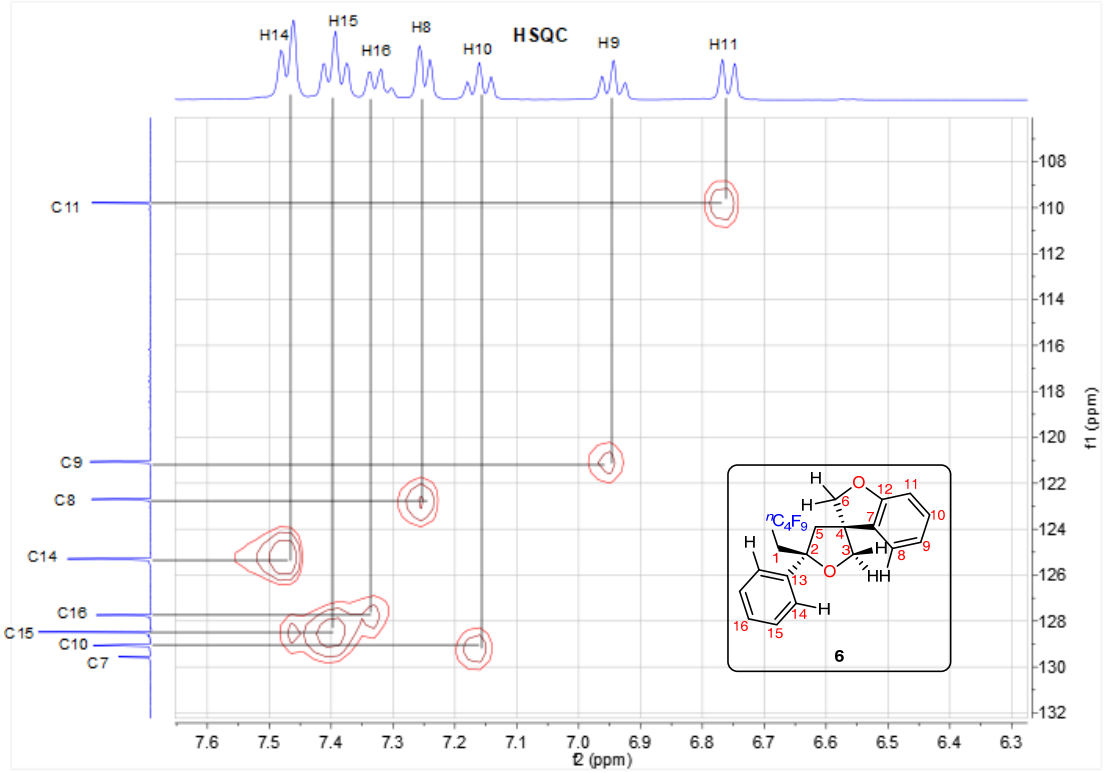

DEPT 135
6
C6 C3
C5
C1
f1 (ppm)

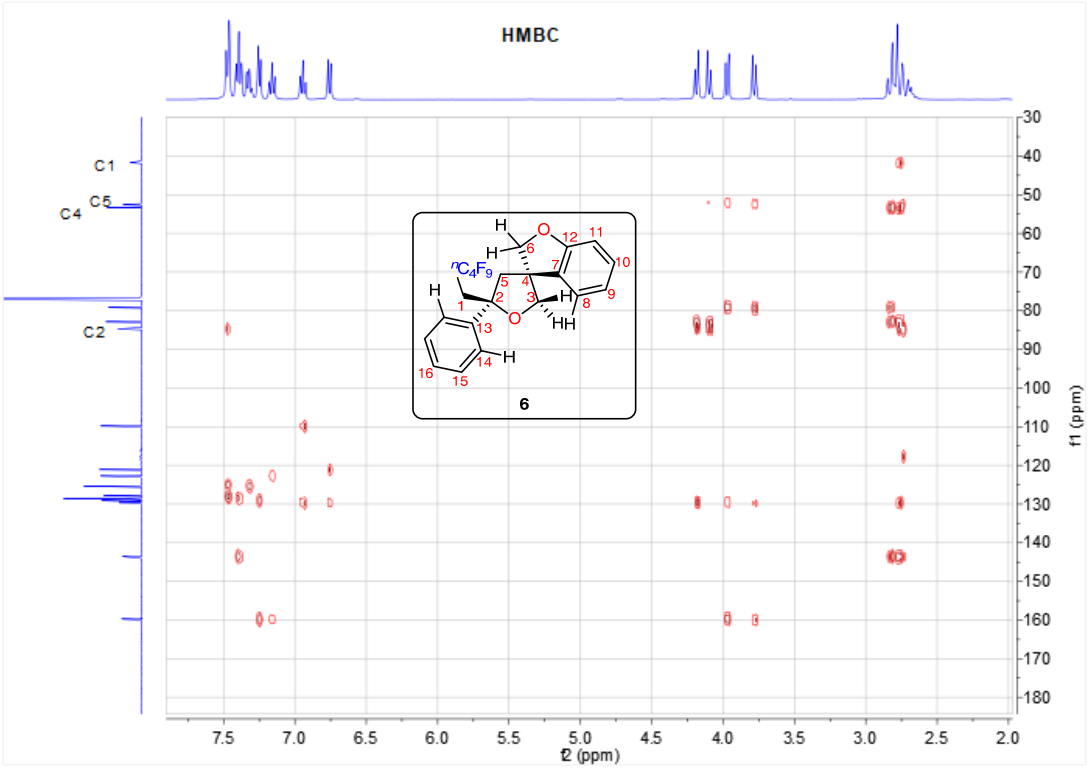

6
3.36
2.00
2.38
2.35
f1 (ppm)

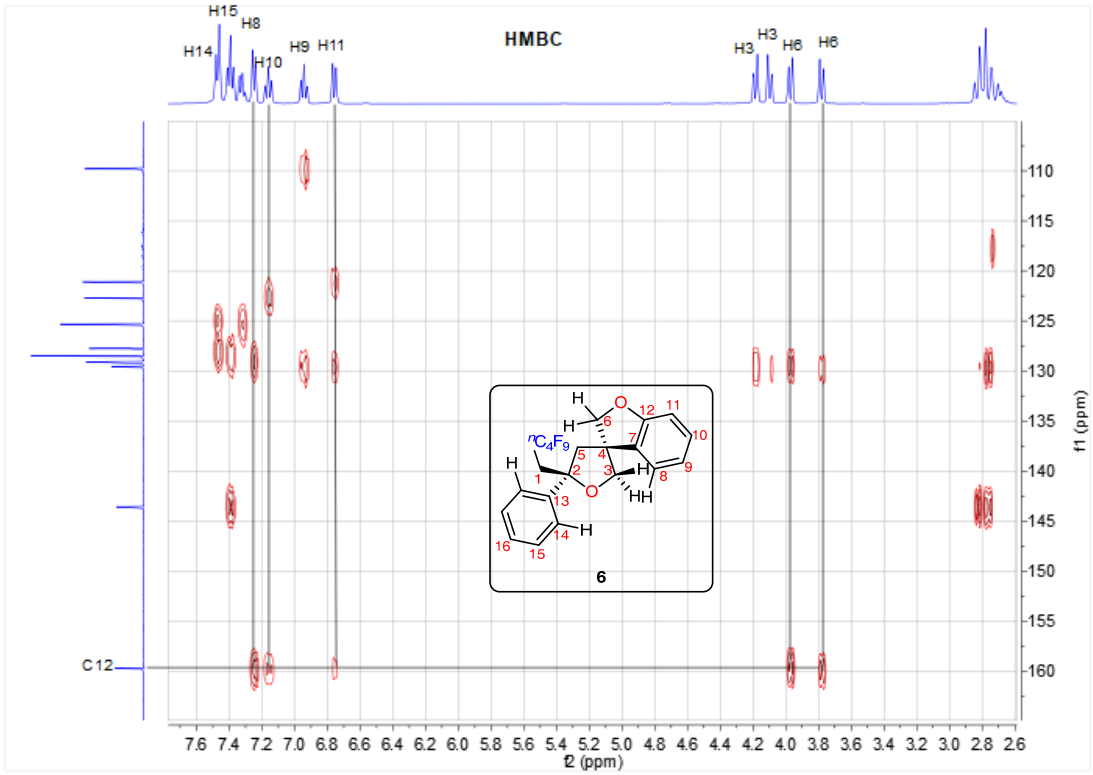

H-H COSY
H3
H6
H6
H3
f2 (ppm)
f1 (ppm)
6

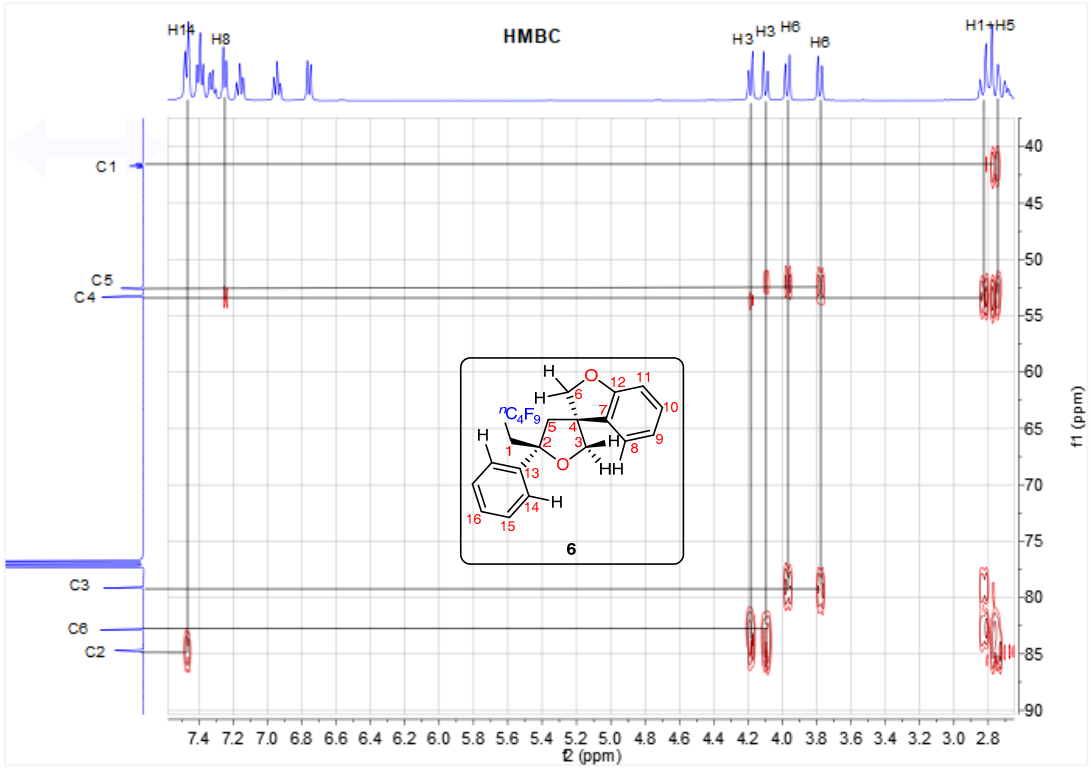

H-H COSY
H14
H15
H16
H8
H10
H9
H11
H11
H9
H10
H8
H16
H15
H14
f2 (ppm)
f1 (ppm)
6

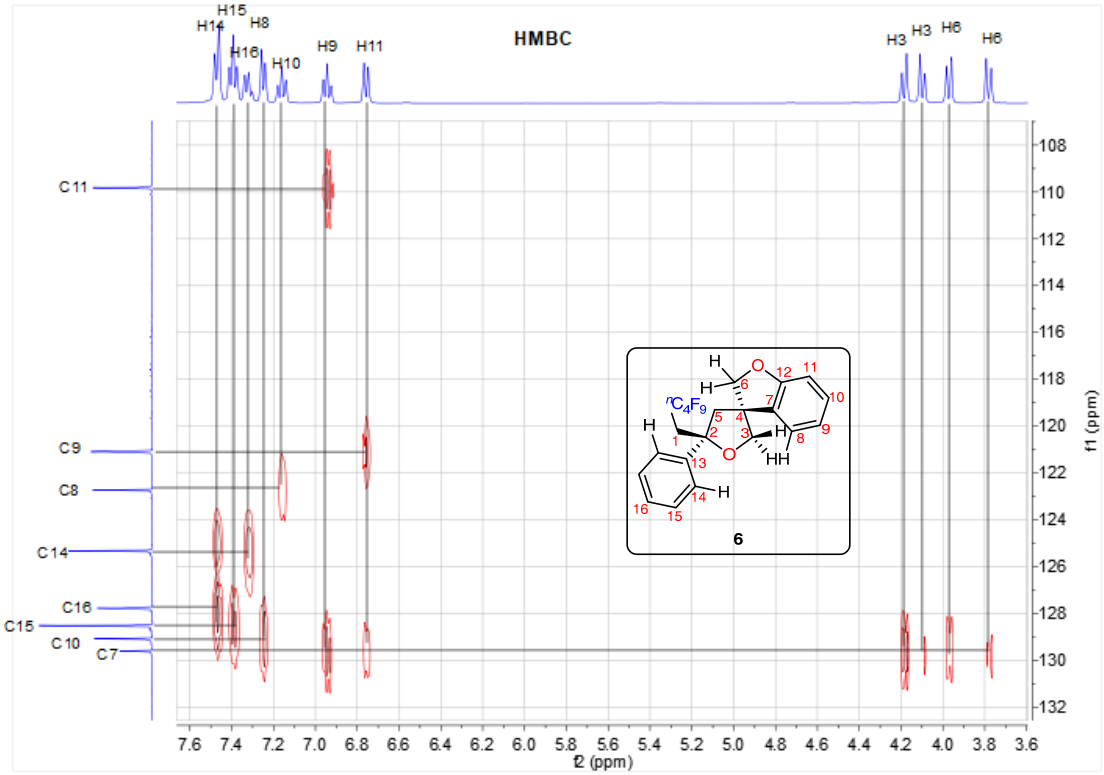

HSQC
H3
H3
H6
H6
C3
C6
C13
C12
f2 (ppm)
f1 (ppm)
6

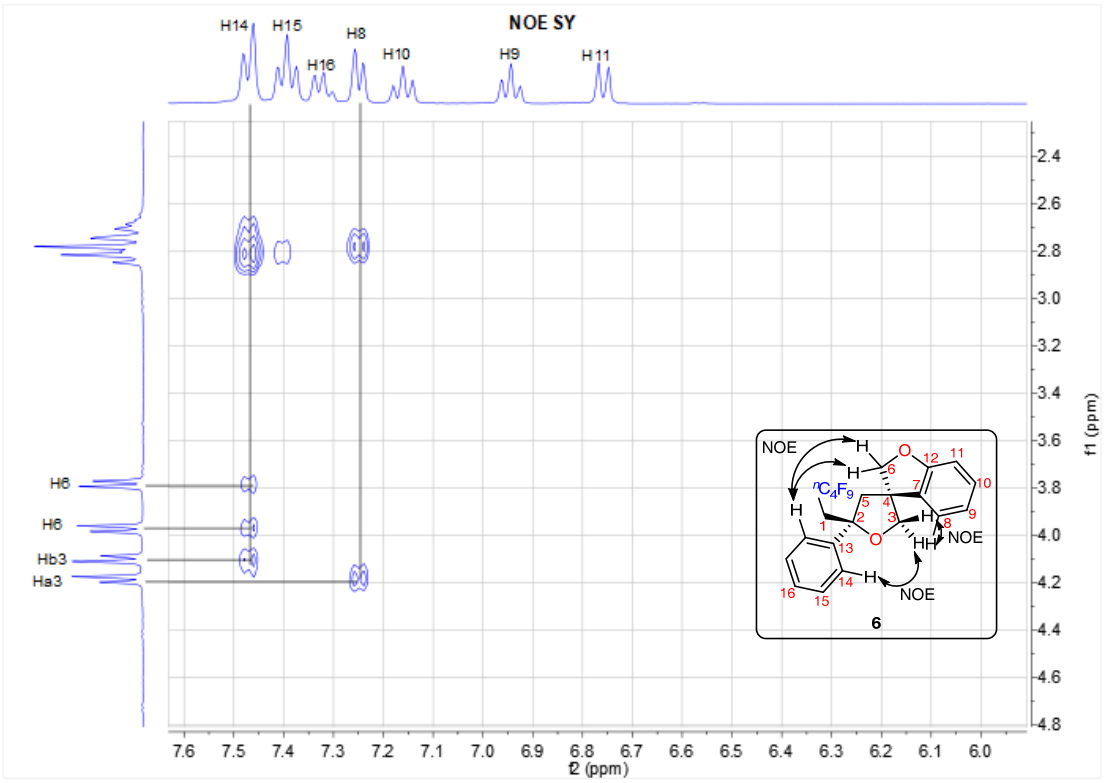

HSQC
H14
H15
H16
H8
H10
H9
H11
C11
C9
C8
C14
C16
C15
C10
C7
f2 (ppm)
f1 (ppm)
6

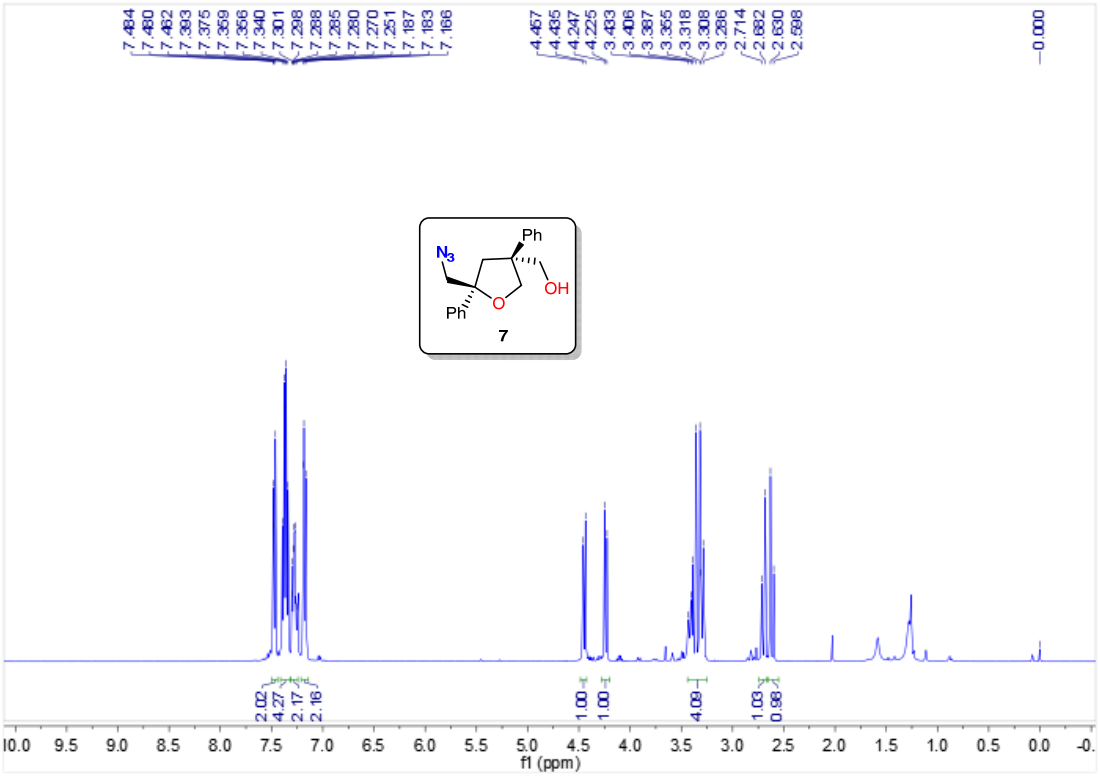

HMBC
C1
C5
C4
C2
6
f2 (ppm)
f1 (ppm)


HMBC
H15
H8
H14
H10
H9
H11
H3
H3
H6
H6
C12
6
f2 (ppm)
f1 (ppm)


HMBC
H14
H8
H3
H3
H6
H6
H1
H5
C1
C5
C4
C3
C6
C2
f1 (ppm)
f2 (ppm)
6


HMBC
H15
H14
H8
H16
H10
H9
H11
H3
H3
H6
H6
C11
C9
C8
C14
C16
C15
C10
C7
f1 (ppm)
f2 (ppm)
6


NOE SY
H14
H15
H8
H16
H10
H9
H11
H6
H6
Hb3
Ha3
f1 (ppm)
f2 (ppm)
NOE
NOE
NOE
6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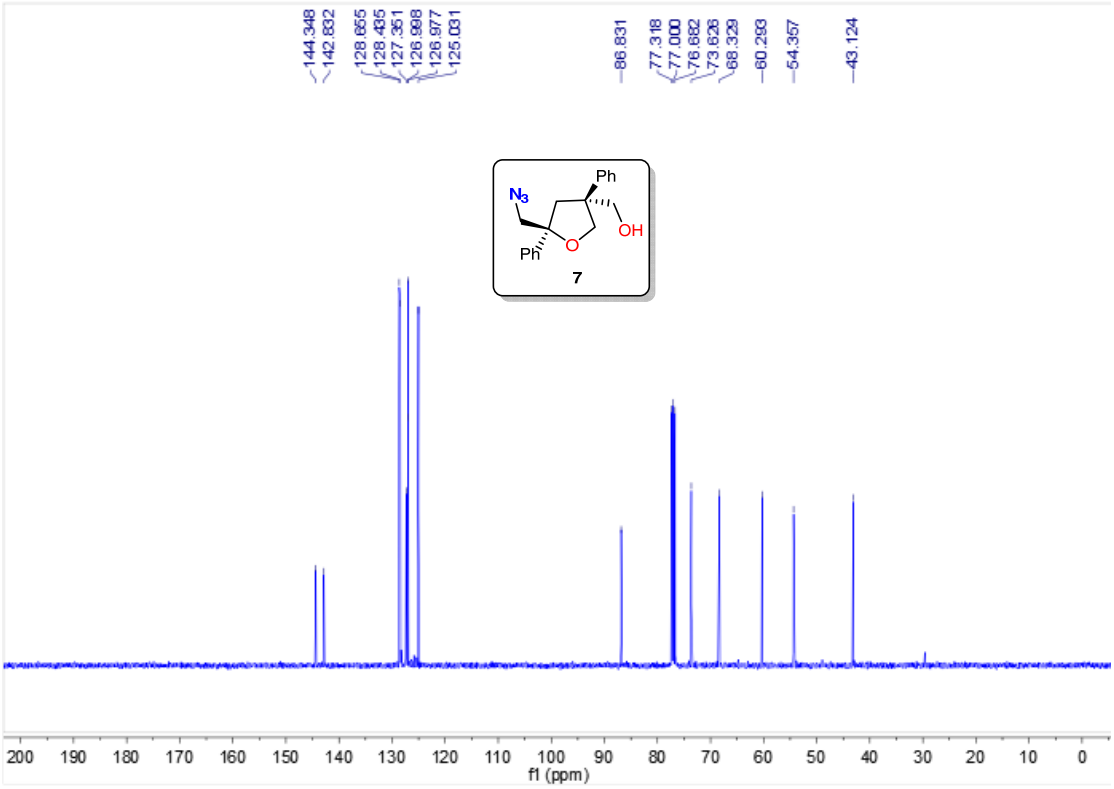
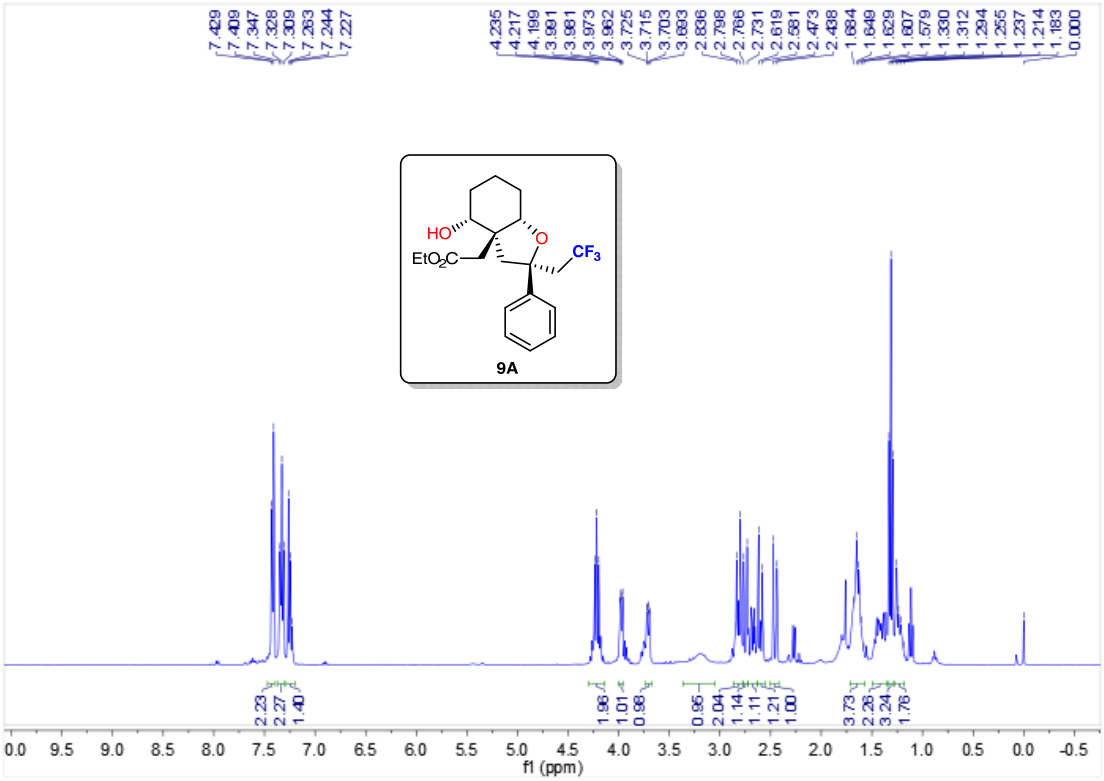
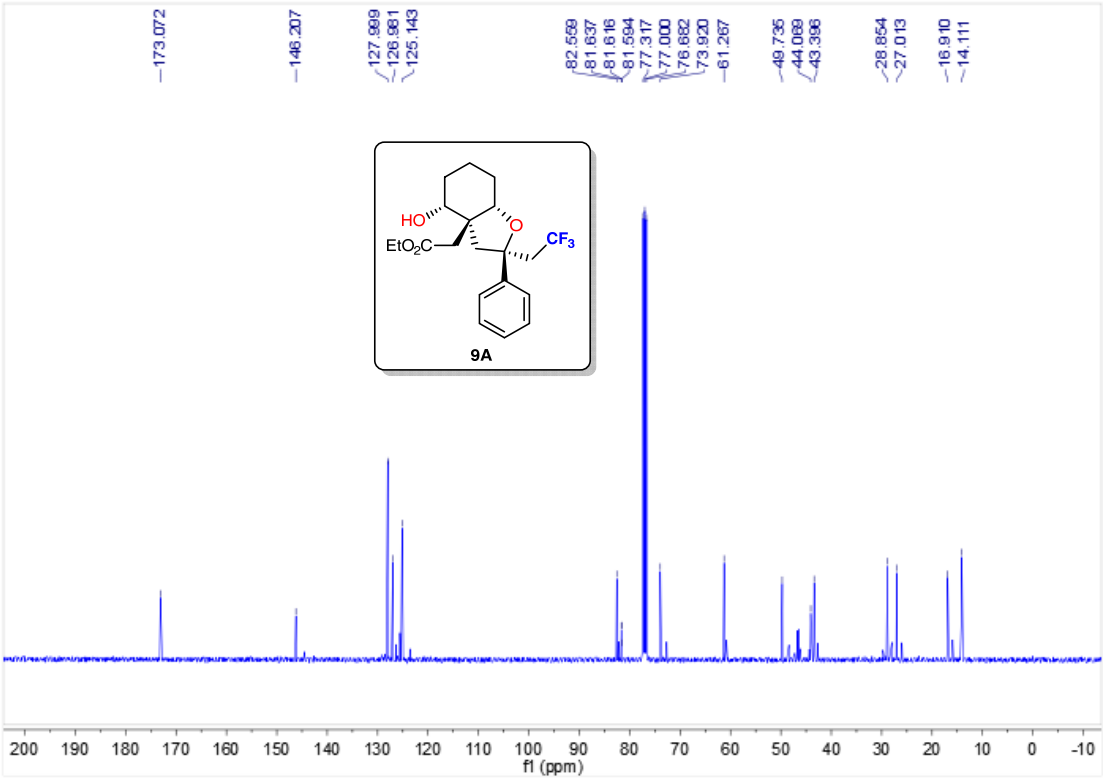
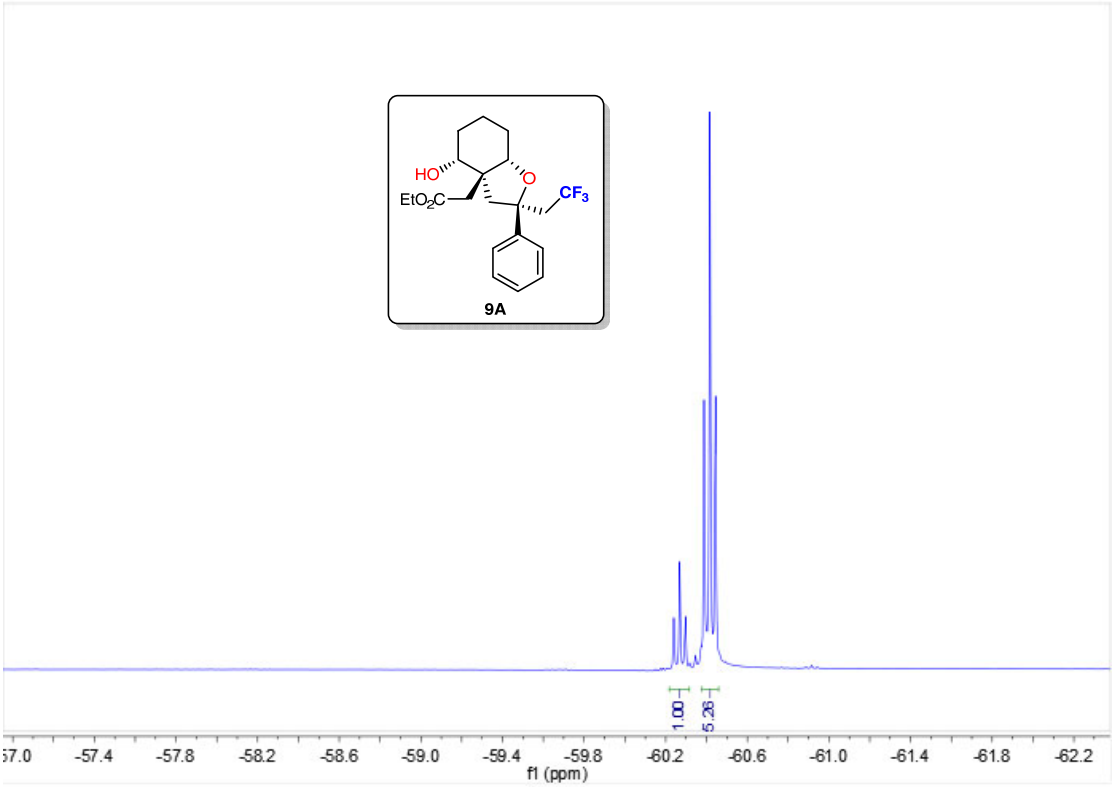
7.356
7.340
7.301
7.298
7.288
7.285
7.280
7.270
7.251
7.187
7.183
7.166
4.467
4.435
4.247
4.225
3.433
3.406
3.387
3.365
3.318
3.308
3.286
2.714
2.682
2.630
2.598
0.000
N3
Ph
OH
Ph
O
7
2.02
4.27
2.17
2.16
1.00
1.00
4.09
1.03
0.98
f1 (ppm)


144.348
142.832
128.655
128.435
127.351
126.998
126.977
125.031
86.831
77.318
77.000
76.682
73.626
68.329
60.293
54.357
43.124
N3
Ph
OH
Ph
O
7
f1 (ppm)


HO
EtO2C
O
CF3
9A
f1 (ppm)


HO
EtO2C
O
CF3
9A
f1 (ppm)


HO
EtO2C
O
CF3
9A
1.00
5.26
-57.0
-57.4
-57.8
-58.2
-58.6
-59.0
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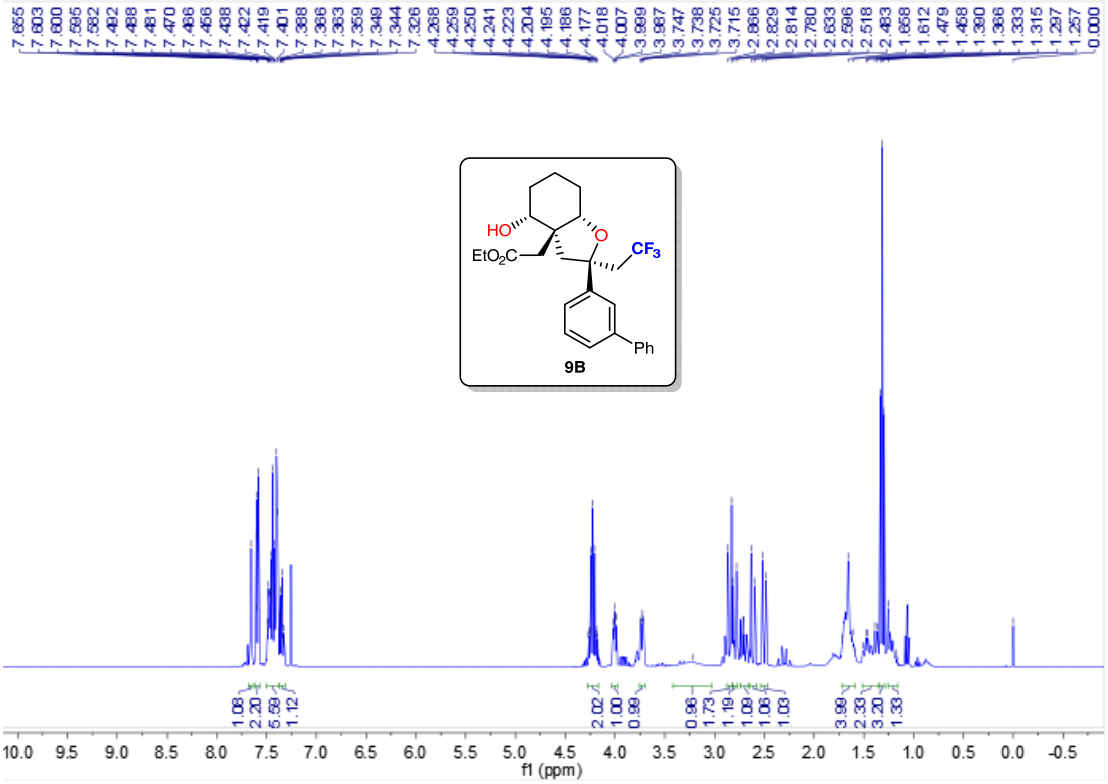
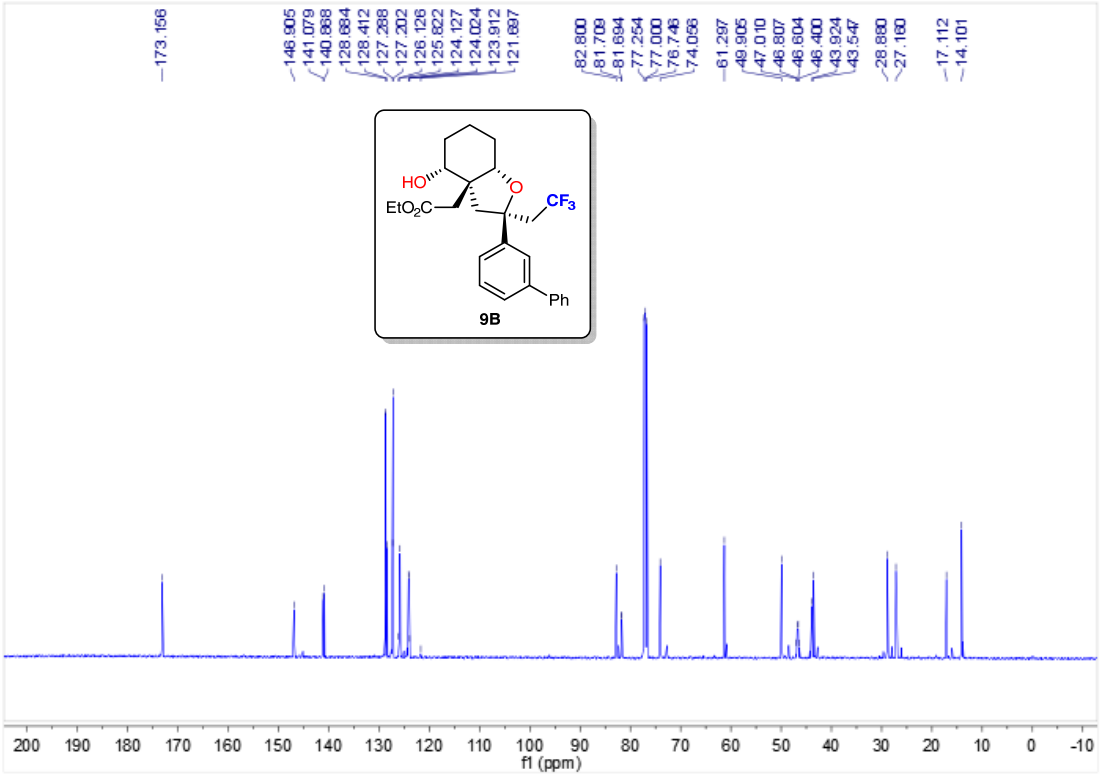
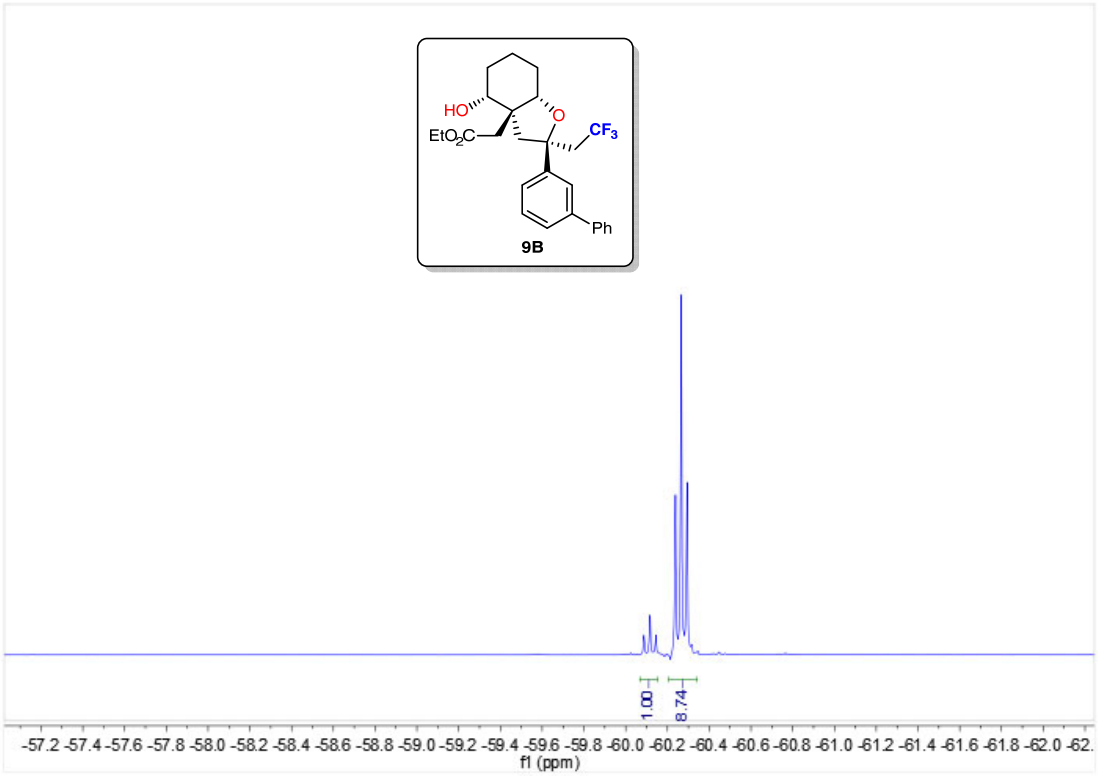
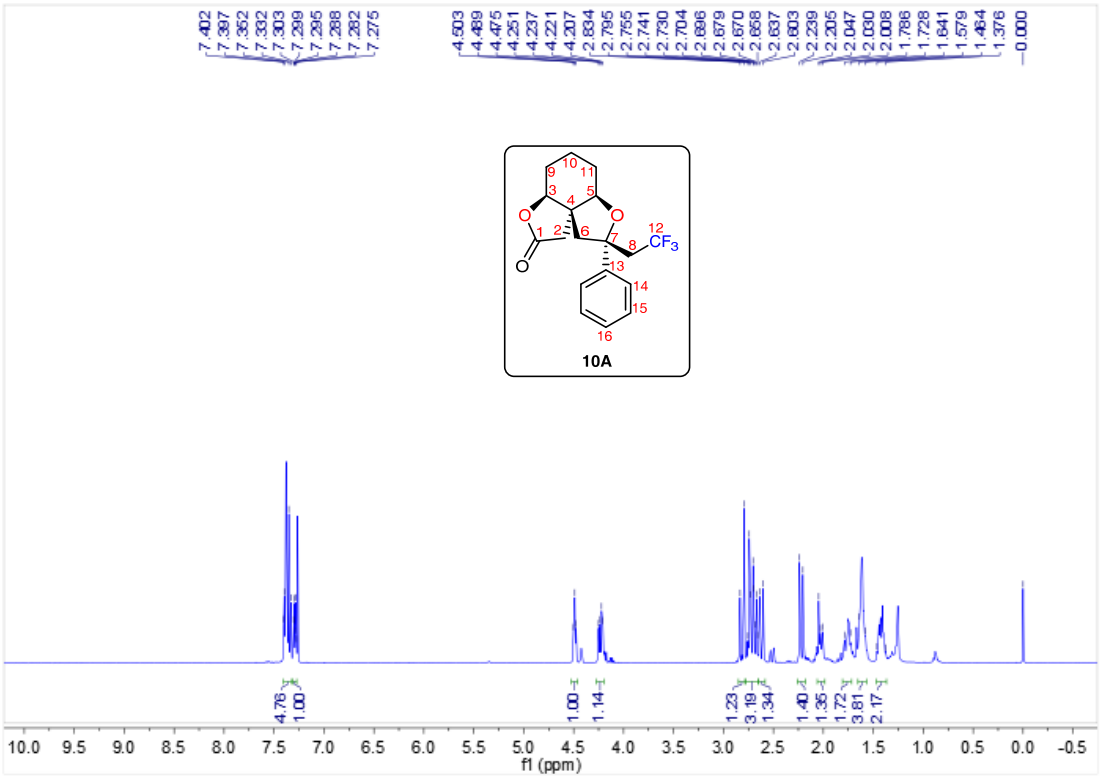
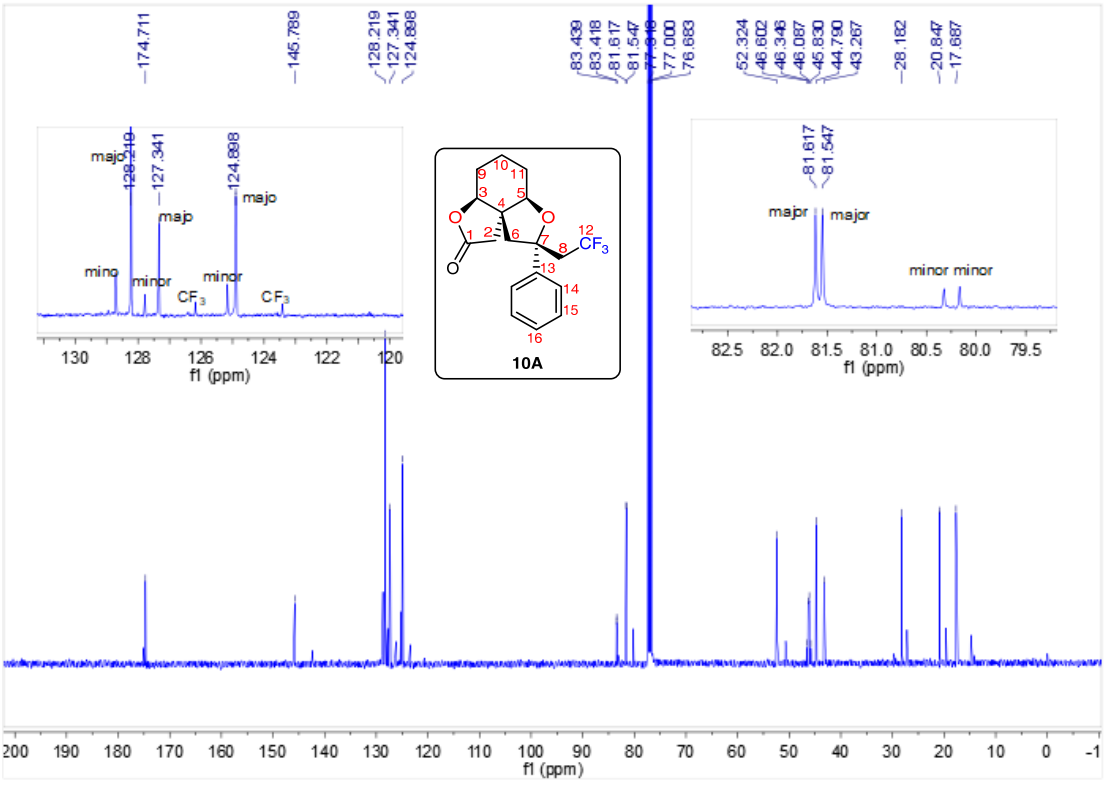
-59.8
-60.2
-60.6
-61.0
-61.4
-61.8
-62.2
f1 (ppm)

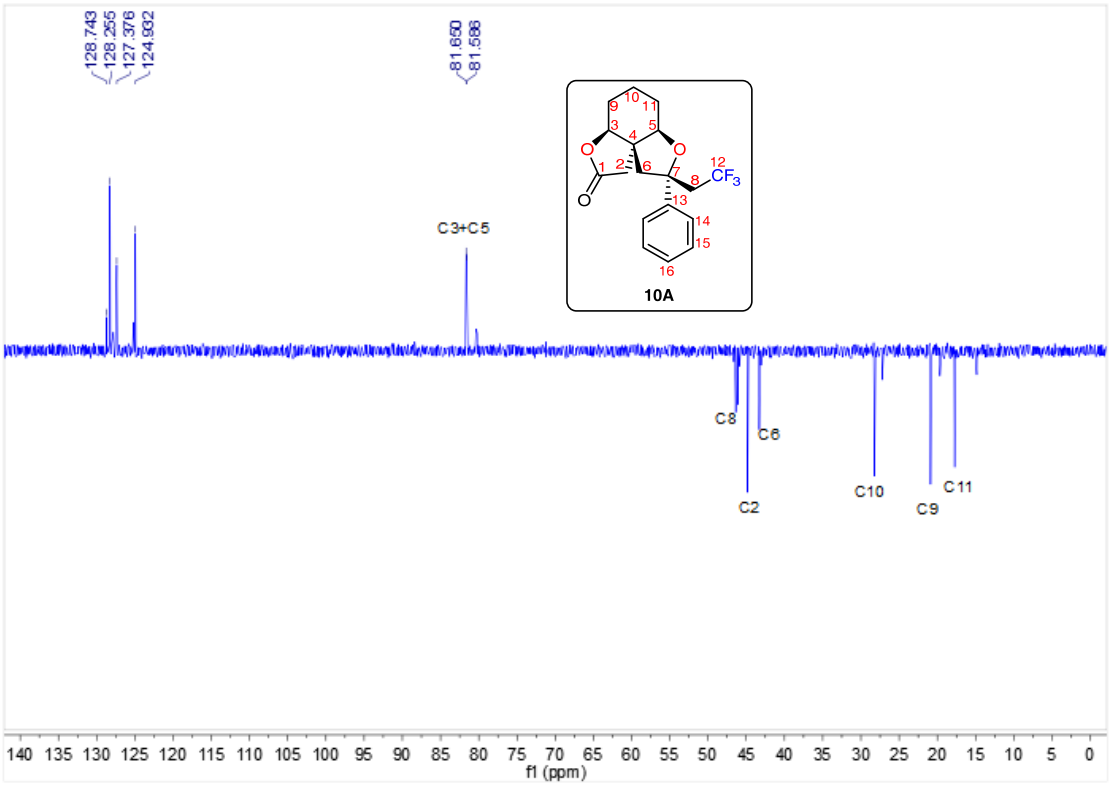

HO
EtO2C
O
CF3
Ph
9B
1.08
2.20
5.59
1.12
2.02
1.00
0.99
0.96
1.73
1.19
1.09
1.06
1.03
3.99
2.33
3.20
1.33
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

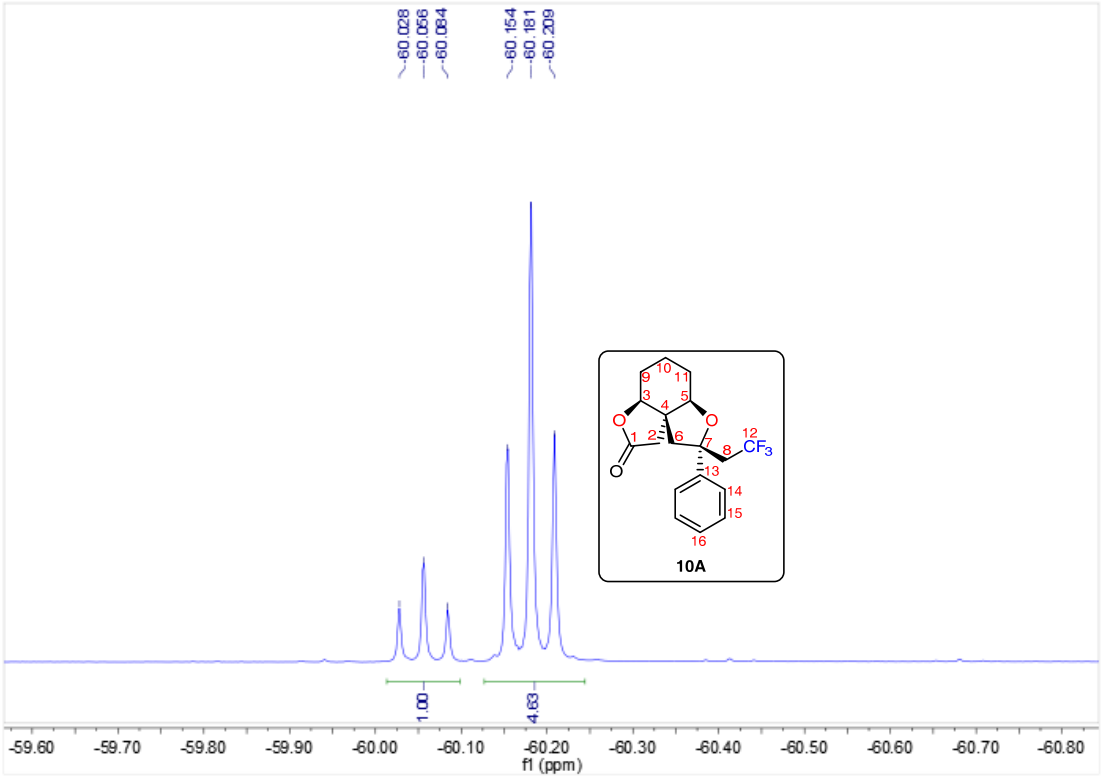

173.156
146.905
141.079
140.868
128.884
128.412
127.288
127.202
126.126
125.822
124.127
124.024
123.912
121.897
82.800
81.709
81.694
77.254
77.000
76.745
74.066
61.297
49.905
47.010
46.807
46.604
46.400
43.924
43.547
28.880
27.160
17.112
14.101
HO
O
EtO2C
CF3
Ph
9B
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

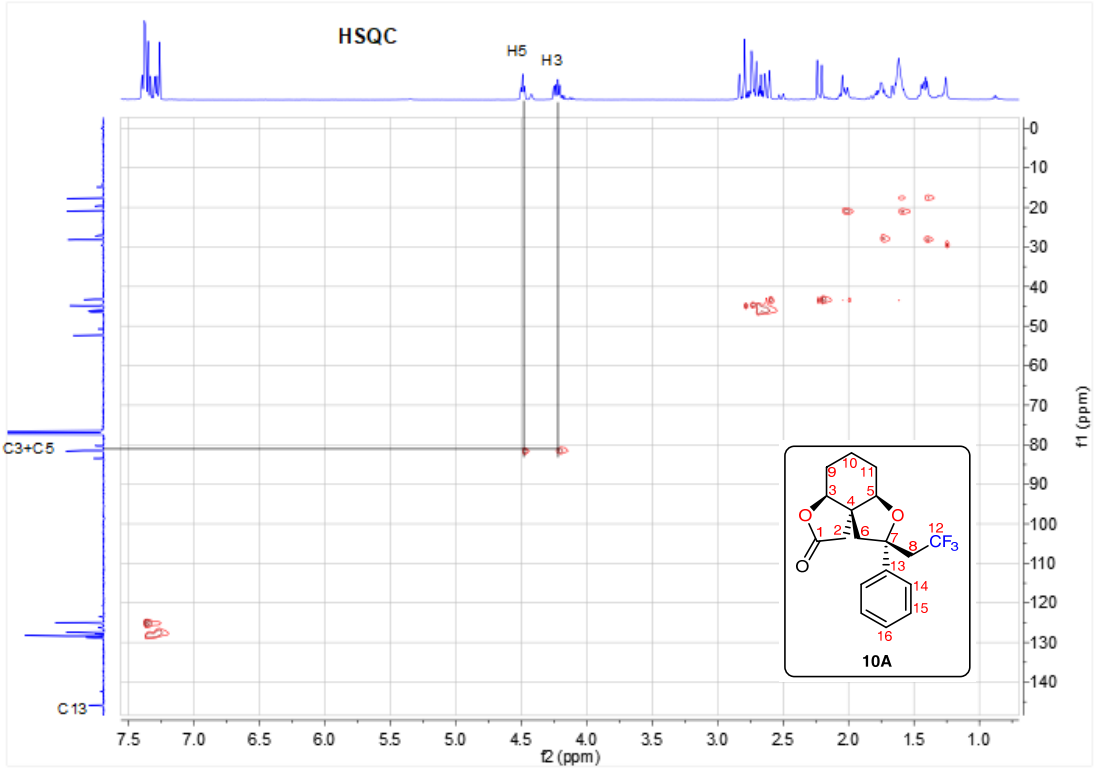

HO
O
EtO2C
CF3
Ph
9B
1.00
8.74
-57.2 -57.4 -57.6 -57.8 -58.0 -58.2 -58.4 -58.6 -58.8 -59.0 -59.2 -59.4 -59.6 -59.8 -60.0 -60.2 -60.4 -60.6 -60.8 -61.0 -61.2 -61.4 -61.6 -61.8 -62.0 -62.
f1 (ppm)

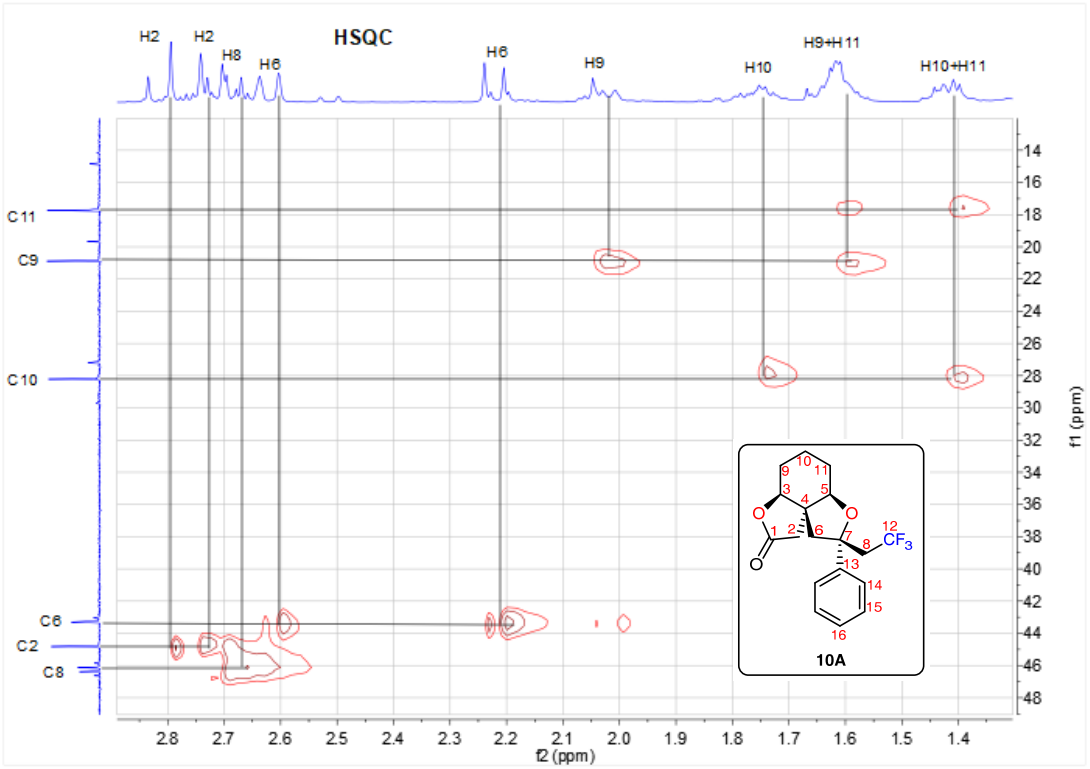

10A
f1 (ppm)

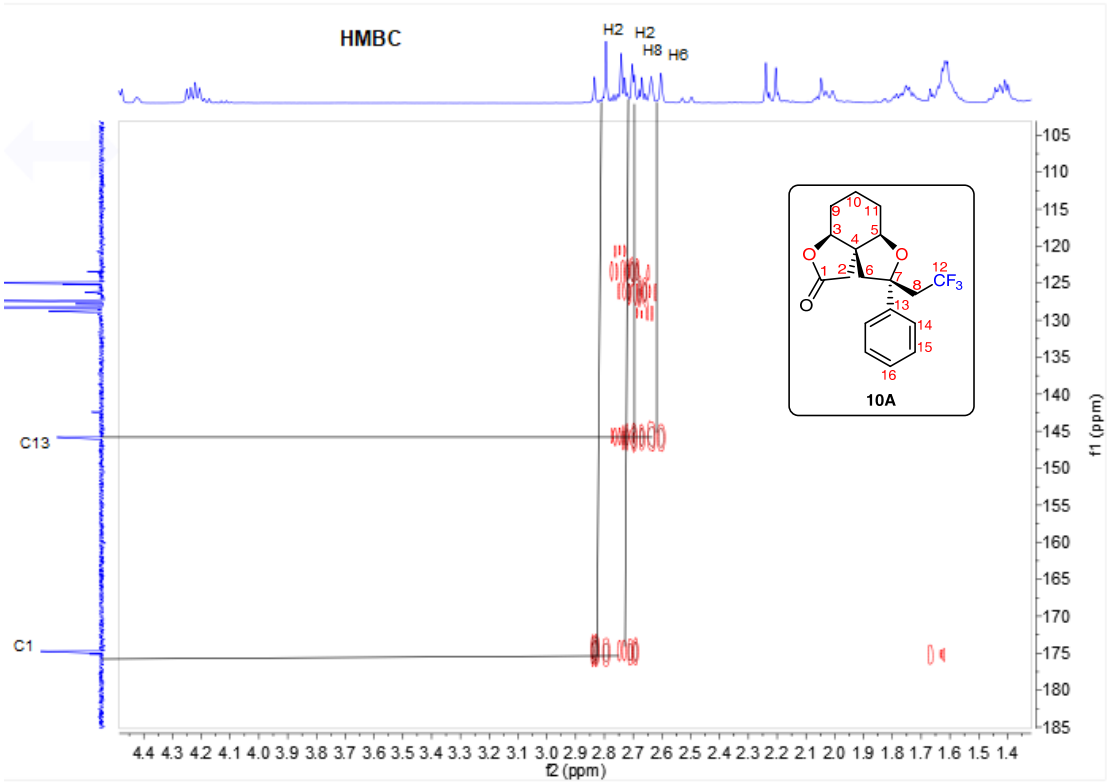

10A
f1 (ppm)
f1 (ppm)

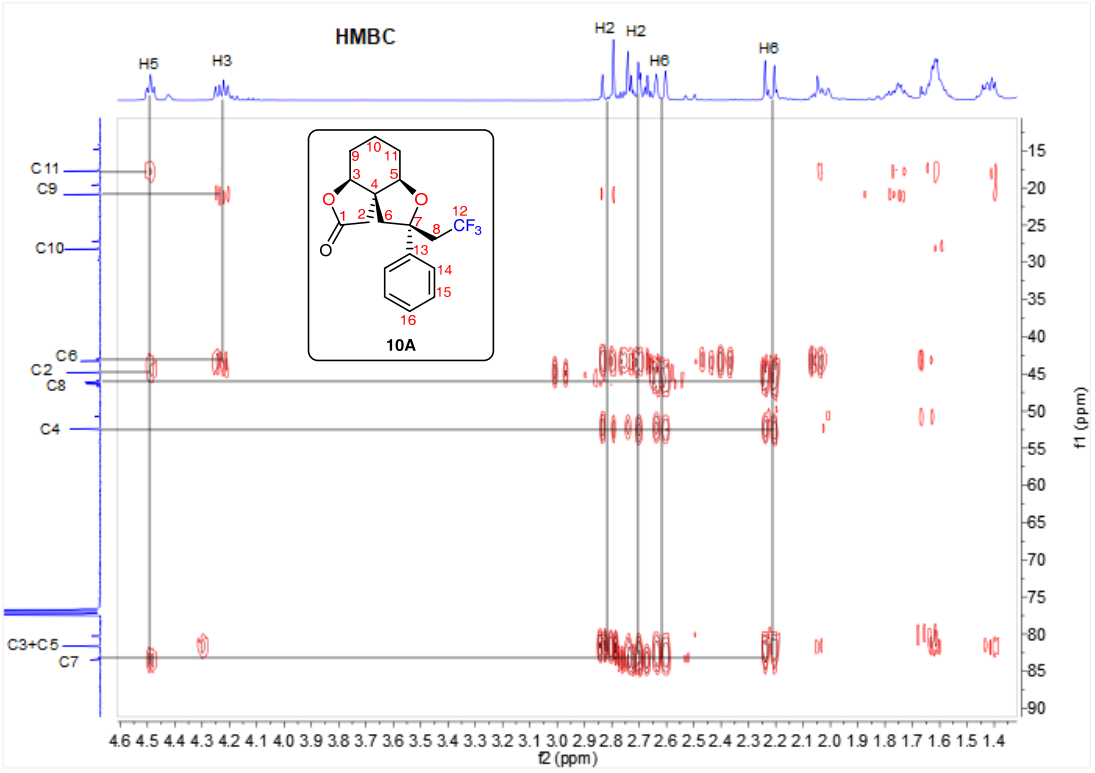

128.743
128.255
127.376
124.992
81.650
81.586
C3+C5
C8
C6
C2
C10
C9
C11
10A
140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

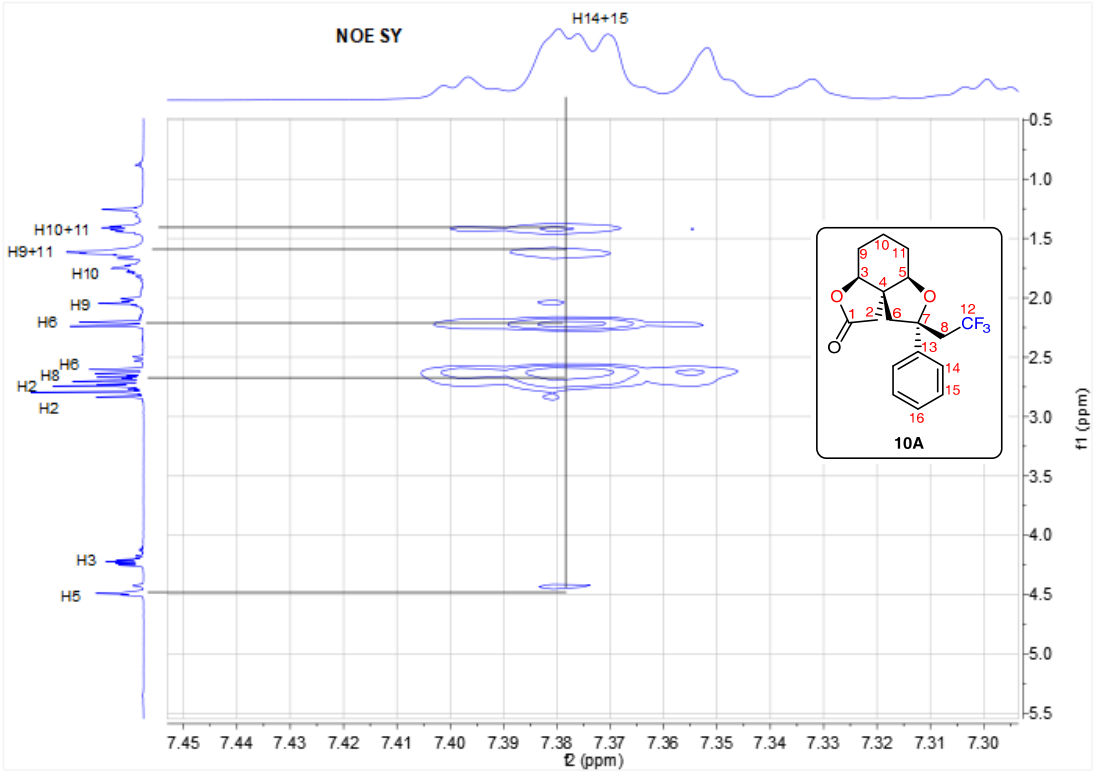

-60.028
-60.056
-60.084
-60.154
-60.181
-60.209
1.00
4.63
10A
-59.60 -59.70 -59.80 -59.90 -60.00 -60.10 -60.20 -60.30 -60.40 -60.50 -60.60 -60.70 -60.80
f1 (ppm)

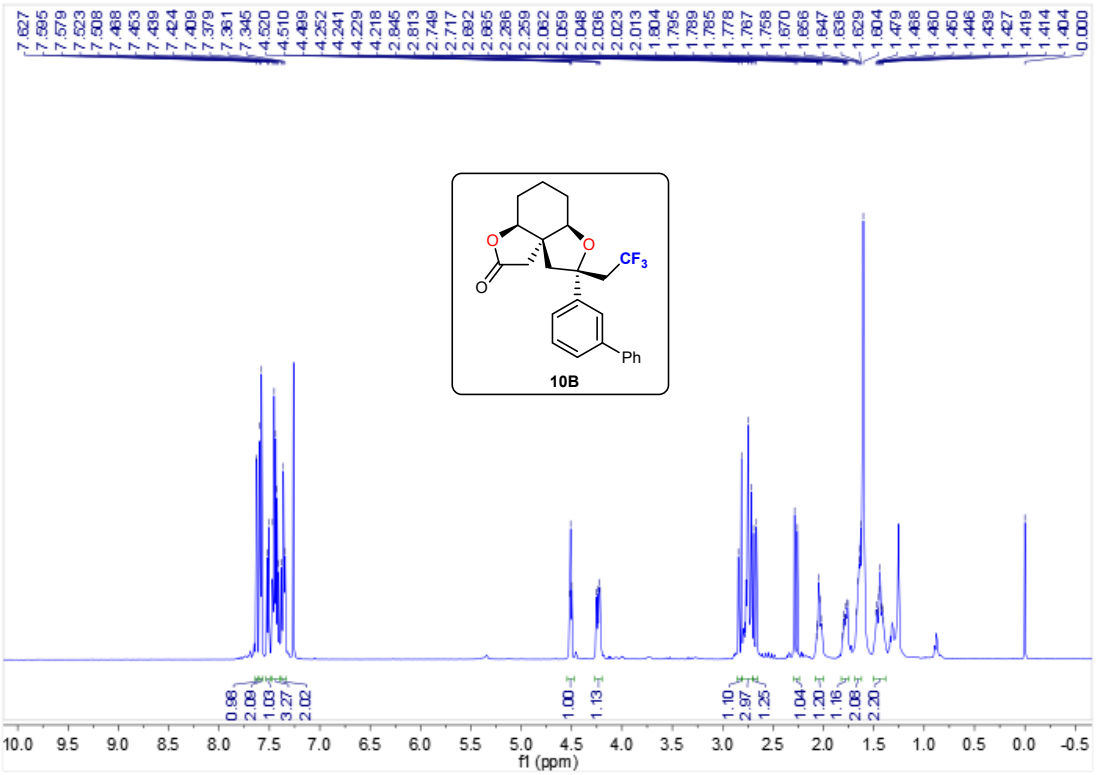

HSQC
H5
H3
C3+C5
C13
f2 (ppm)
f1 (ppm)
10A

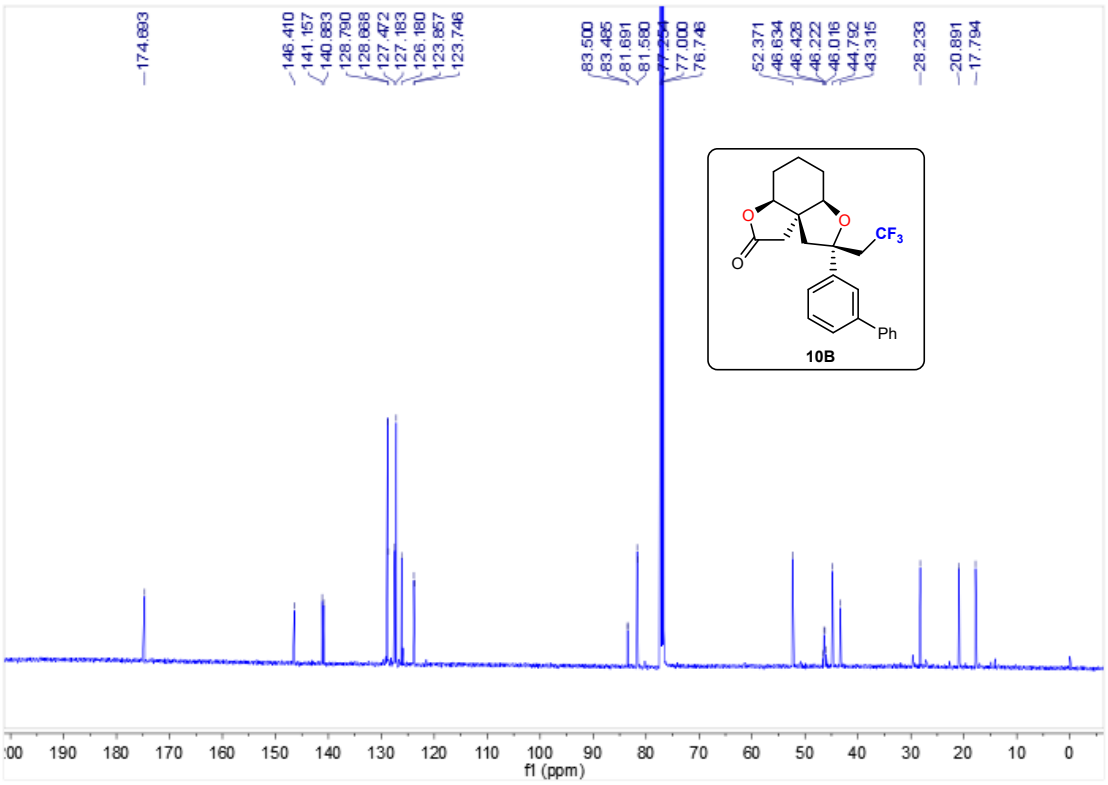

HSQC
H2
H2
H8
H6
H6
H9
H10
H9+H11
H10+H11
C11
C9
C10
C6
C2
C8
f2 (ppm)
f1 (ppm)
10A

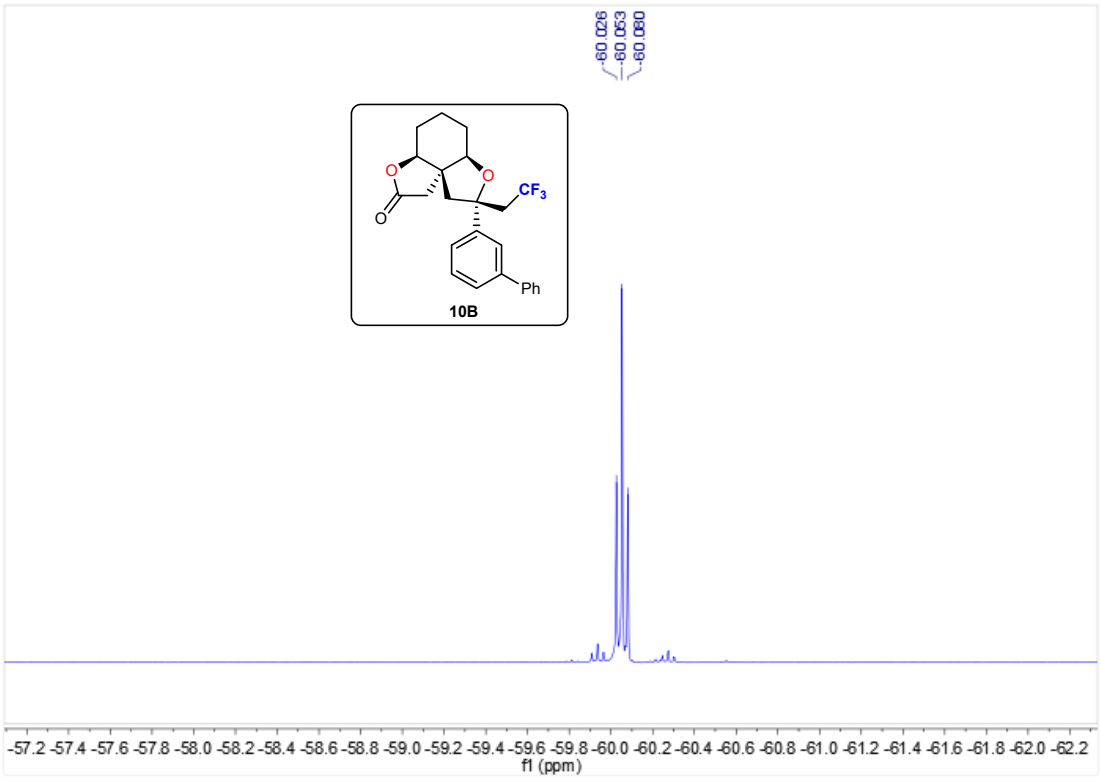

HMBC
H2
H2
H8
H6
C13
C1
10A
f2 (ppm)
f1 (ppm)


HMBC
H5
H3
H2
H2
H6
H6
C11
C9
C10
C6
C2
C8
C4
C3+C5
C7
10A
f2 (ppm)
f1 (ppm)

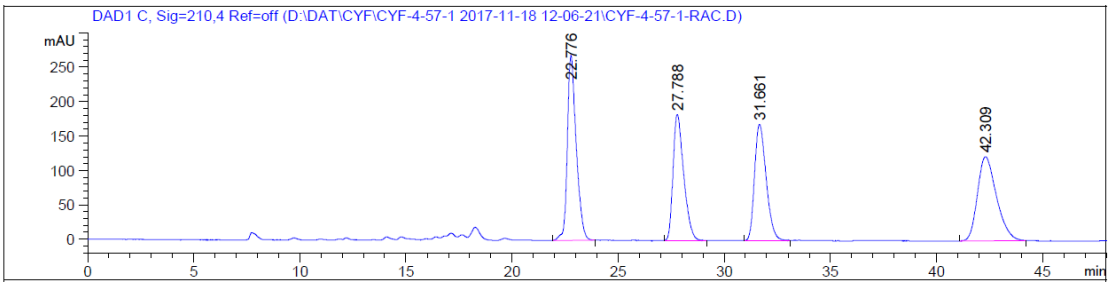

NOE SY
H14+15
H10+11
H9+11
H10
H9
H6
H6
H8
H2
H2
H3
H5
10A
f2 (ppm)
f1 (ppm)


10B
f1 (ppm)


174.893
146.410
141.157
140.883
128.790
128.868
127.472
127.183
126.180
123.857
123.746
83.500
83.485
81.691
81.580
77.000
76.748
52.371
46.634
46.428
46.222
46.016
44.792
43.315
28.233
20.891
17.794
O
O
O
CF3
Ph
10B
f1 (ppm)

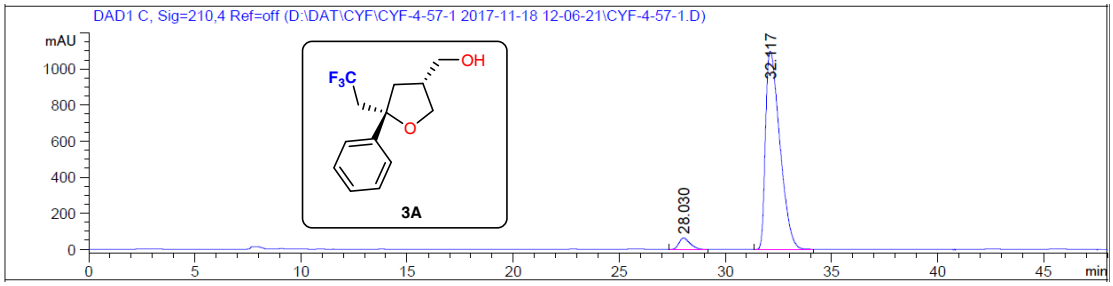

60.026
60.053
60.080
O
O
O
CF3
Ph
10B
f1 (ppm)

3.11 HPLC Spectra



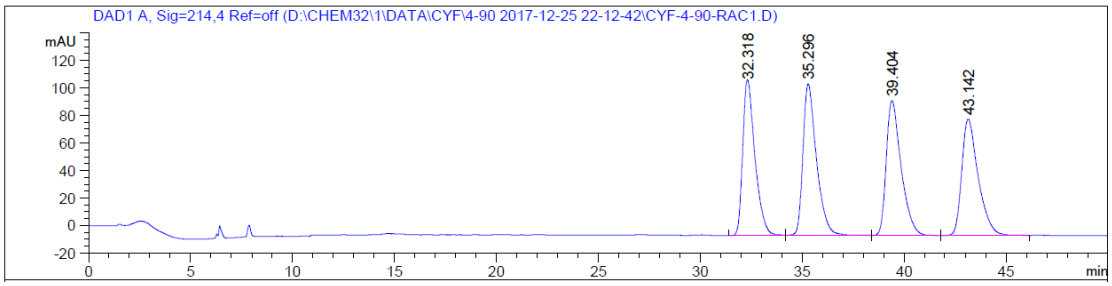
Signal 3: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.776	BB	0.4312	7832.41064	269.35770	27.6681
2	27.788	BB	0.5012	6401.87988	183.40504	22.6147
3	31.661	BB	0.5559	6430.34521	168.98978	22.7153
4	42.309	BV	0.7556	7643.79492	122.56849	27.0018



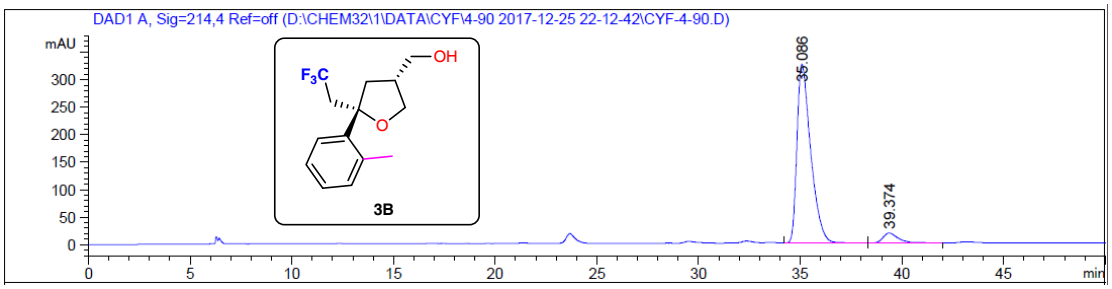
Signal 3: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.030	BB	0.5032	2332.72583	63.99904	4.3285
2	32.117	BB	0.6255	5.15592e4	1098.69238	95.6715



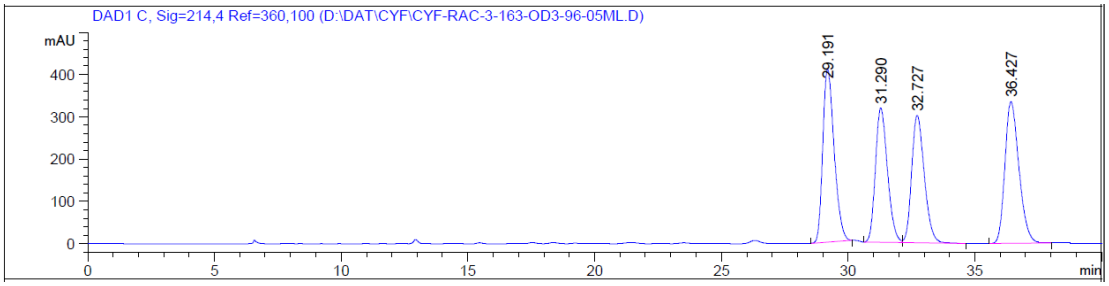
Signal 1: DAD1 A, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.318	BV	0.6216	4715.81445	113.29609	24.2037
2	35.296	VB	0.6901	5082.25537	110.19418	26.0844
3	39.404	BB	0.7573	4950.78271	98.01073	25.4096
4	43.142	BB	0.8366	4735.04053	84.64616	24.3023



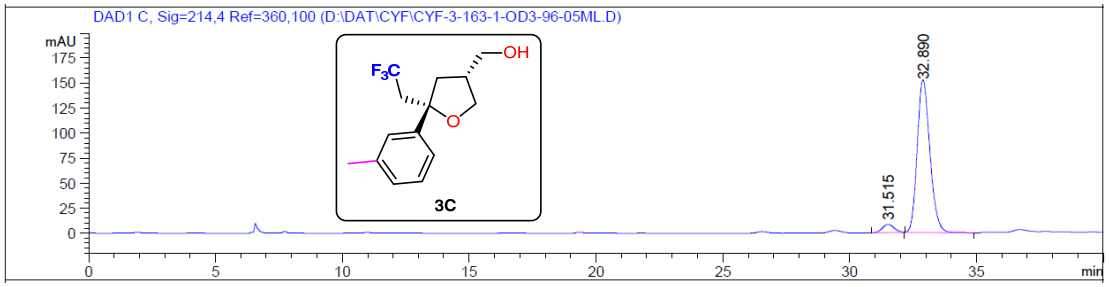
Signal 1: DAD1 A, Sig=214,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.086	VB	0.7042	1.52618e4	324.79858	94.2937
2	39.374	BB	0.7639	923.59308	17.96148	5.7063



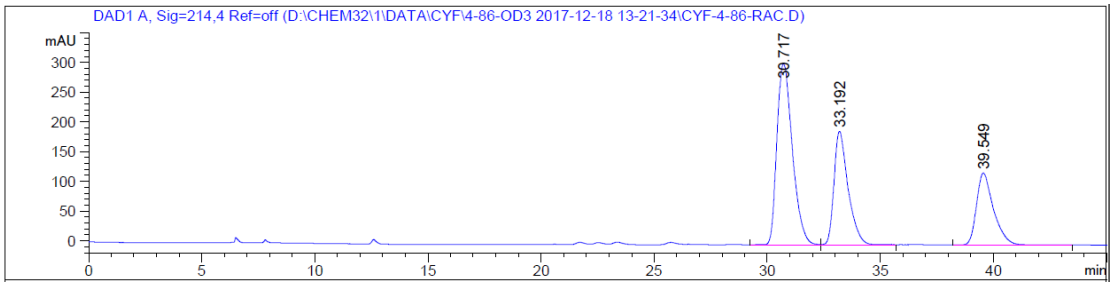
Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.191	BB	0.4792	1.27556e4	409.77777	27.3005
2	31.290	BV	0.5114	1.05017e4	317.89600	22.4765
3	32.727	VB	0.5389	1.05232e4	301.72339	22.5227
4	36.427	BB	0.5945	1.29424e4	335.13528	27.7003



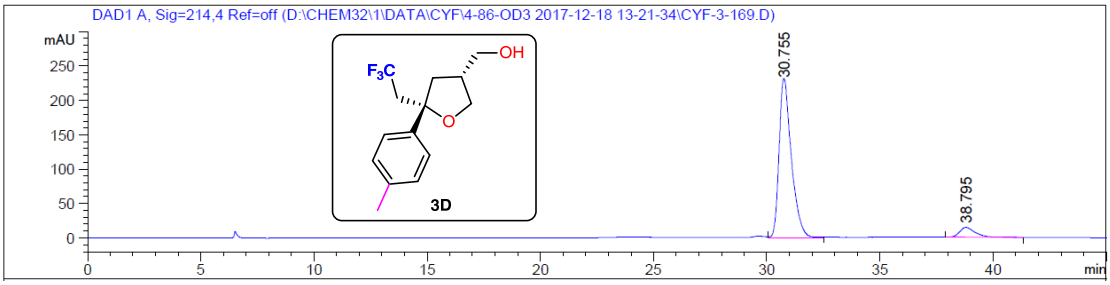
Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	31.515	BB	0.4762	256.61758	8.17579	4.6134
2	32.890	BB	0.5372	5305.77246	152.76756	95.3866



Signal 1: DAD1 A, Sig=214,4 Ref=off

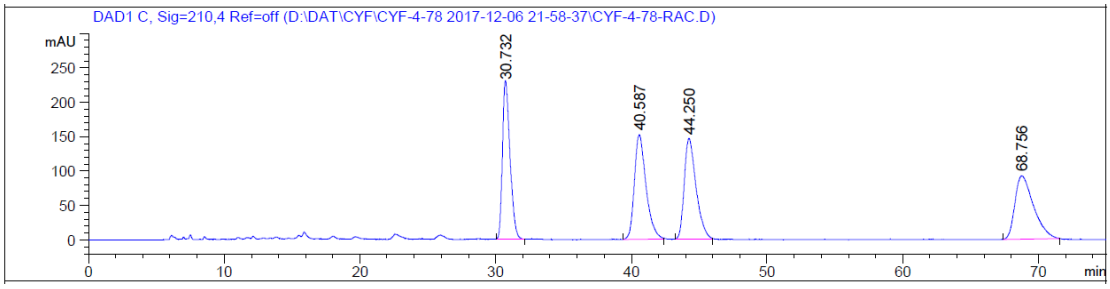
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.717	BV	0.7435	1.44980e4	304.48126	49.8485
2	33.192	VB	0.6456	8259.14258	190.59329	28.3974
3	39.549	BB	0.7705	6327.00537	120.90569	21.7541



Signal 1: DAD1 A, Sig=214,4 Ref=off

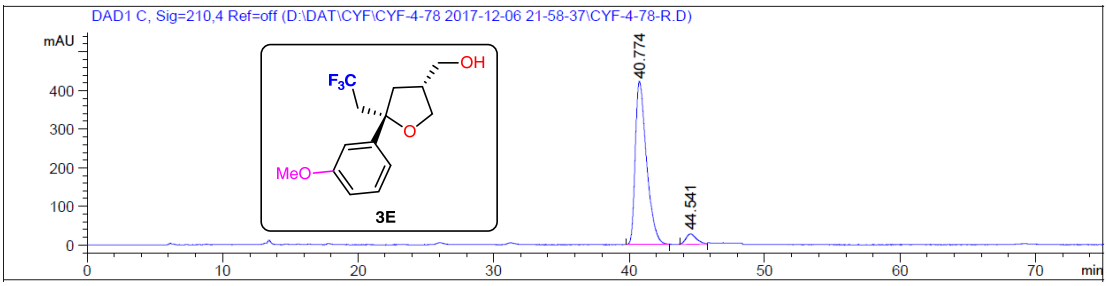
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.755	VV	0.5564	8650.73242	231.26132	92.7450
2	38.795	BB	0.7070	676.70099	14.22559	7.2550

Note: After optimization of separation conditions (including: Chiralcel OD3, OX3, AZ3, AD3, OJ, IA, IB, IC, ID, IE, IF, IG, INB), we can't get four peaks with baseline separation.



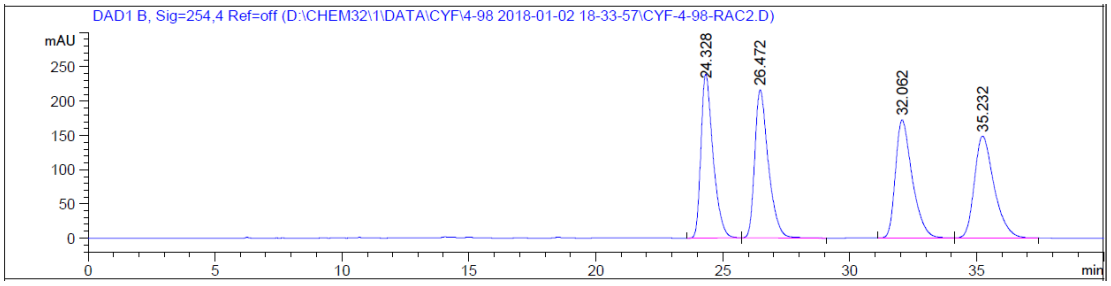
Signal 3: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.732	BB	0.5523	8890.76465	230.33232	24.9599
2	40.587	BV	0.7206	9084.07129	151.70471	25.5026
3	44.250	VV	0.7189	8859.50586	146.62712	24.8722
4	68.756	VB	1.1190	8785.79297	92.16798	24.6652



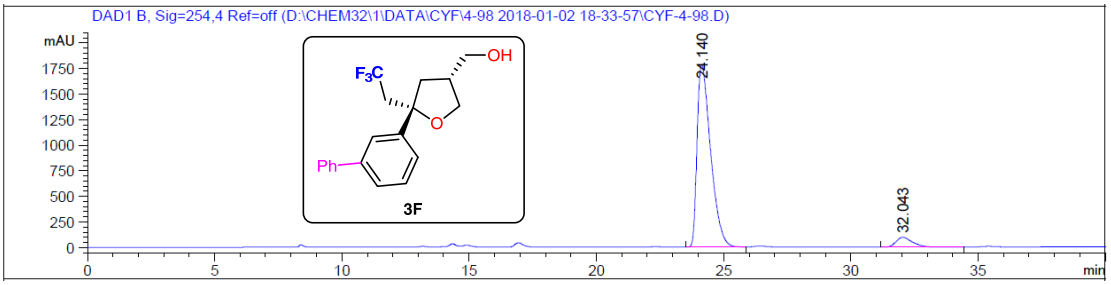
Signal 3: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	40.774	VB	0.7348	2.44316e4	423.62390	94.4906
2	44.541	VB	0.6265	1424.51721	26.78223	5.5094



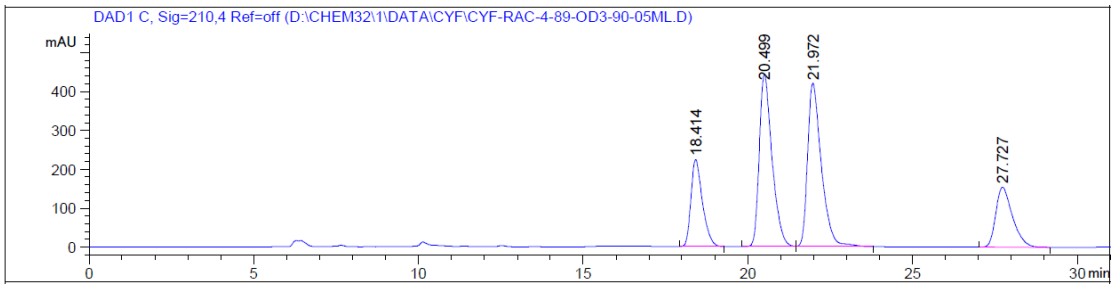
Signal 2: DAD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.328	BB	0.4942	7916.67725	239.14812	24.8806
2	26.472	BB	0.5572	8016.65137	215.91626	25.1948
3	32.062	BB	0.6883	7887.99902	172.24448	24.7905
4	35.232	BB	0.8156	7997.35059	148.65404	25.1341



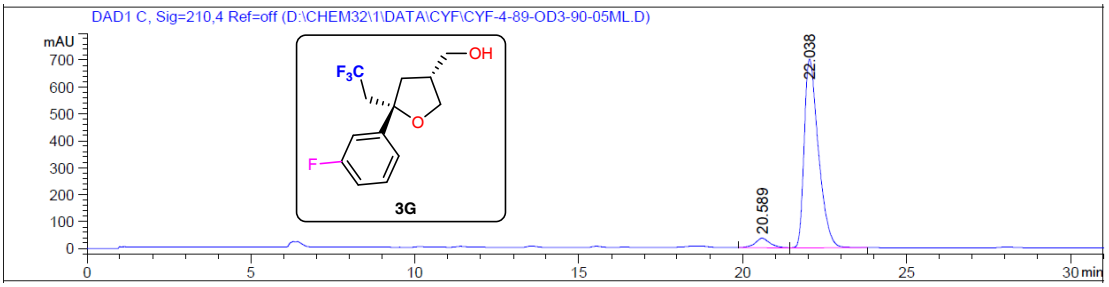
Signal 2: DAD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.140	BB	0.5653	6.60372e4	1786.21167	93.7558
2	32.043	BB	0.6892	4398.13672	96.23405	6.2442



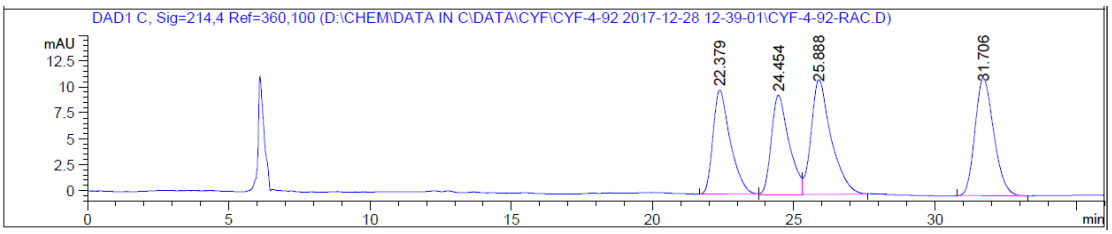
Signal 3: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.414	BB	0.3630	5423.03418	223.57651	15.4518
2	20.499	BV	0.4029	1.19839e4	444.03601	34.1456
3	21.972	VB	0.4357	1.22535e4	420.81927	34.9139
4	27.727	BB	0.5259	5435.95996	154.71121	15.4887



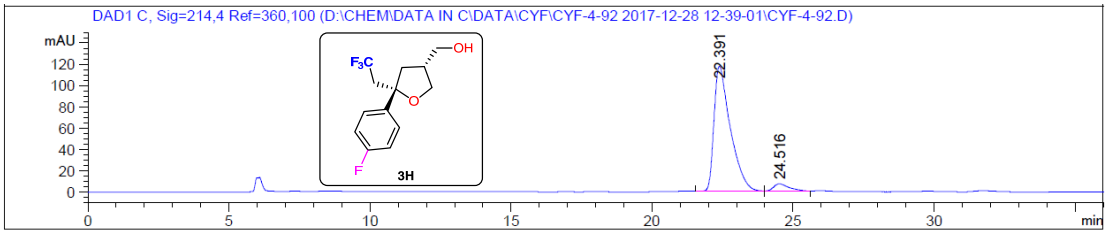
Signal 3: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.589	BB	0.4413	1104.66089	35.47834	4.9698
2	22.038	BB	0.4530	2.11229e4	702.04468	95.0302



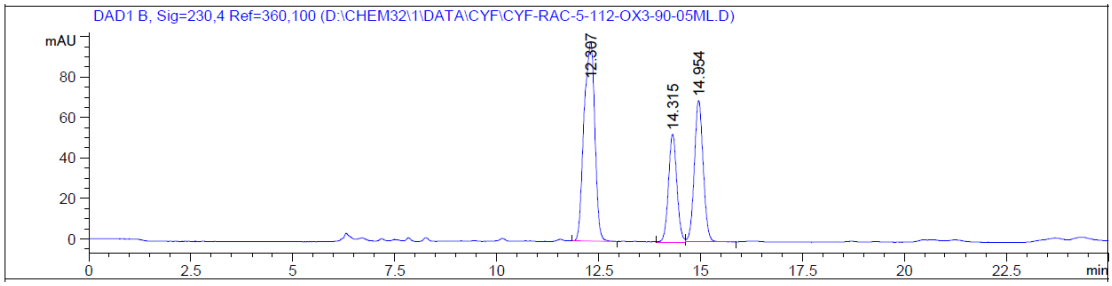
Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.379	BB	0.6271	425.91620	10.03658	22.2108
2	24.454	MF R	0.7304	419.83649	9.58037	21.8937
3	25.888	FM R	0.8207	545.67218	11.08137	28.4558
4	31.706	BB	0.7037	526.18506	11.33258	27.4396



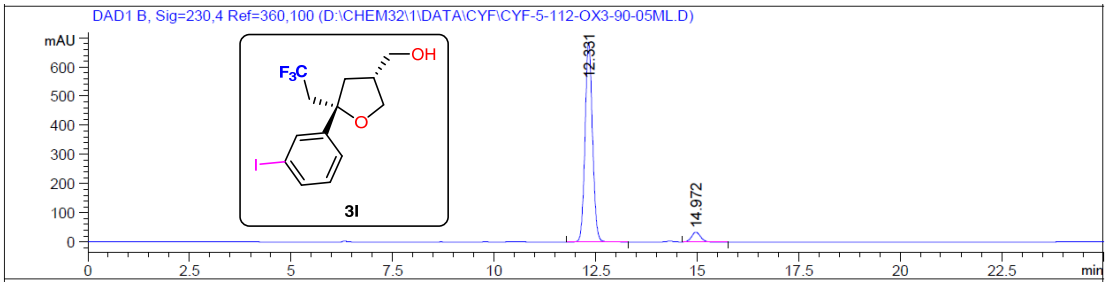
Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.391	BB	0.5864	4719.39160	117.55672	94.7710
2	24.516	BB	0.5557	260.39276	6.87731	5.2290



Signal 2: DAD1 B, Sig=230,4 Ref=360,100

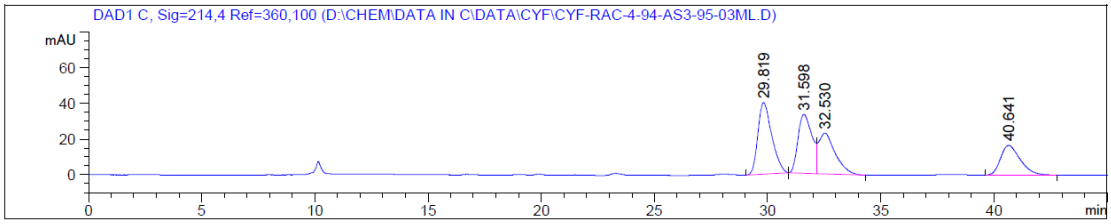
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.307	BB	0.2625	1801.91406	98.01627	49.8121
2	14.315	BV	0.2185	758.74152	53.17480	20.9747
3	14.954	VB	0.2367	1056.76599	69.76019	29.2132



Signal 2: DAD1 B, Sig=230,4 Ref=360,100

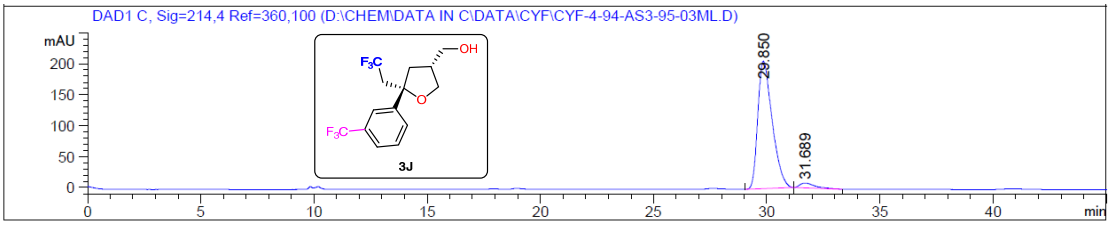
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.331	BB	0.1968	8751.74512	686.28955	94.3810
2	14.972	VB	0.2357	521.03735	33.82175	5.6190

Note: After optimization of separation conditions (including: Chiralcel OD3, OX3, AZ3, AD3, OJ, IA, IB, IC, ID, IE, IF, IG, INB), we can't get four peaks with baseline separation.



Signal 2: DAD1 C, Sig=214,4 Ref=360,100

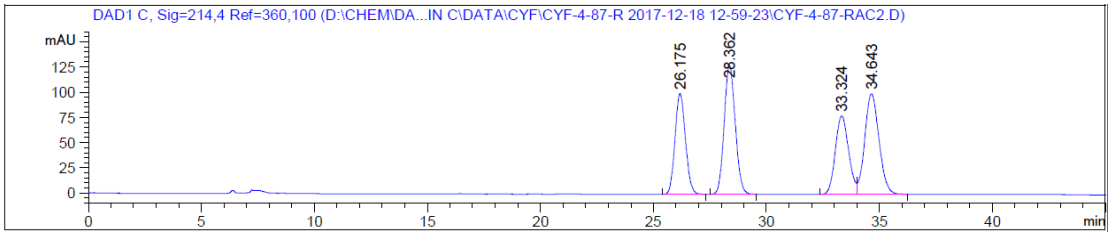
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.819	BB	0.6472	1706.06812	40.03271	32.1944
2	31.598	BV	0.6608	1422.37878	32.87266	26.8410
3	32.530	VB	0.7312	1168.40137	22.82427	22.0484
4	40.641	BB	0.9116	1002.41919	16.62072	18.9162



Signal 2: DAD1 C, Sig=214,4 Ref=360,100

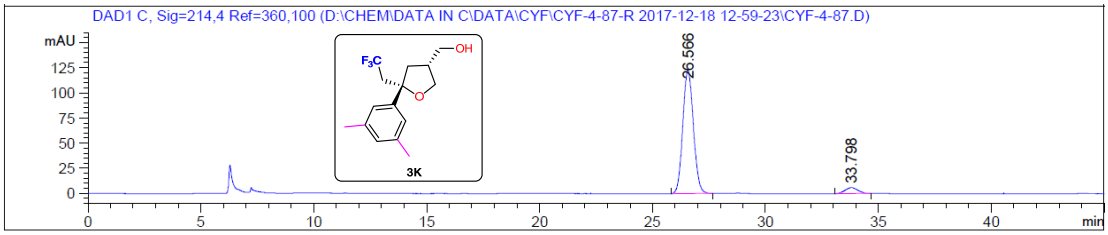
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.850	BB	0.6715	9097.26465	205.90417	96.1598
2	31.689	BB	0.6792	363.30606	7.91904	3.8402

Note: After optimization of separation conditions (including: Chiralcel OD3, OX3, AZ3, AD3, OJ, IA, IB, IC, ID, IE, IF, IG, INB), we can't get four peaks with baseline separation.



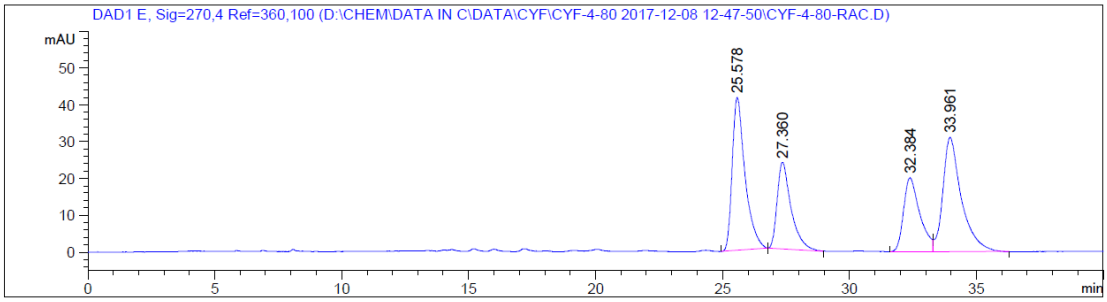
Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.175	BB	0.4999	3229.19263	100.22009	21.2715
2	28.362	BB	0.5305	4376.09521	128.10056	28.8264
3	33.324	BV	0.6319	3190.23193	77.88022	21.0148
4	34.643	VB	0.6812	4385.33545	99.70003	28.8873



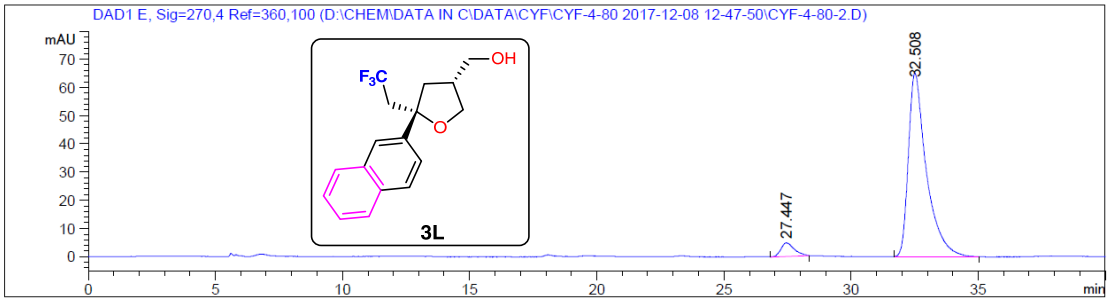
Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.566	BB	0.5034	4004.66479	123.14342	94.5660
2	33.798	BB	0.4946	230.11752	5.85391	5.4340



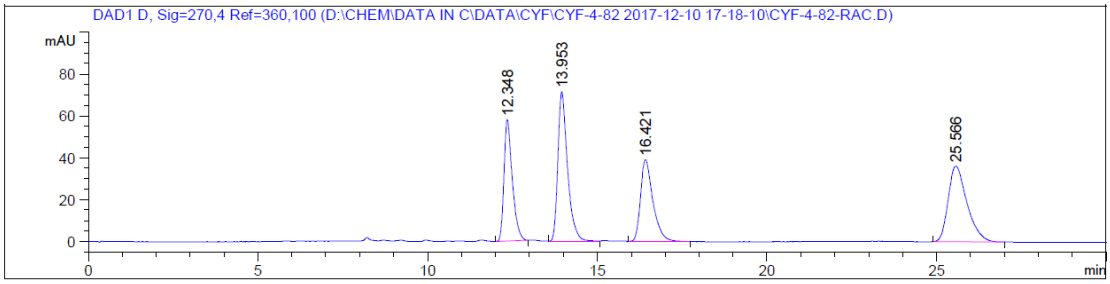
Signal 5: DAD1 E, Sig=270,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.578	BB	0.5288	1462.77075	41.53974	30.2617
2	27.360	BB	0.5404	896.18024	23.52889	18.5401
3	32.384	MF R	0.7465	900.14117	20.09689	18.6221
4	33.961	FM R	0.8462	1574.63953	31.01422	32.5761



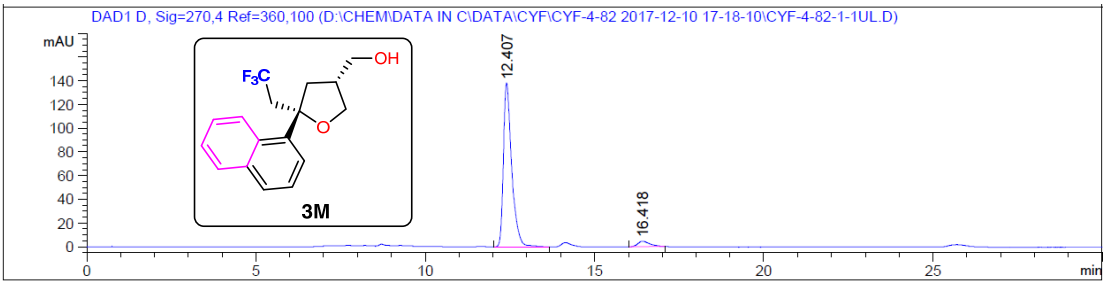
Signal 5: DAD1 E, Sig=270,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.447	BB	0.4569	173.75227	4.79350	5.1454
2	32.508	BB	0.6988	3203.10181	65.01263	94.8546



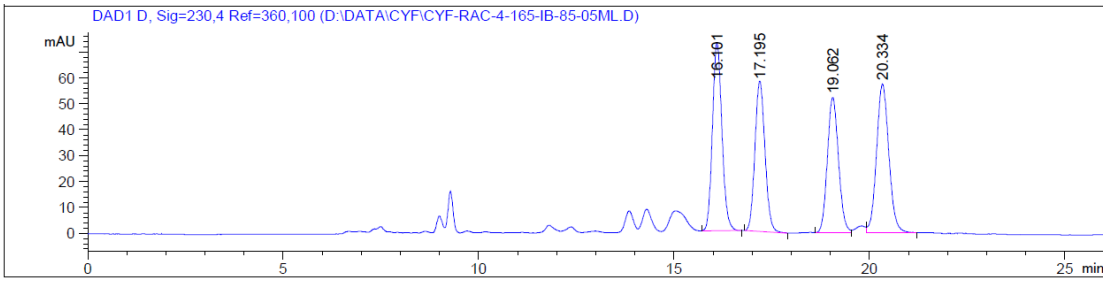
Signal 4: DAD1 D, Sig=270,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.348	BB	0.2570	990.13824	58.05747	20.6816
2	13.953	BB	0.2942	1407.07654	71.30643	29.3905
3	16.421	BB	0.3772	994.99902	39.05983	20.7832
4	25.566	BB	0.5744	1395.31287	36.13934	29.1448



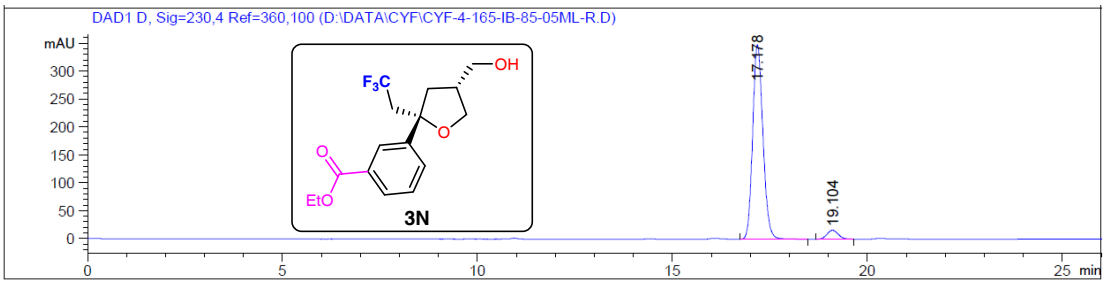
Signal 4: DAD1 D, Sig=270,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.407	BB	0.2499	2343.66992	138.13795	95.3553
2	16.418	BB	0.3432	114.15810	4.70307	4.6447



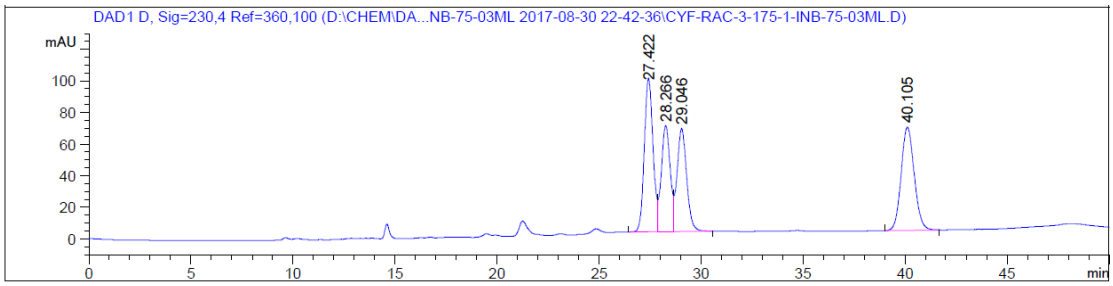
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.101	BB	0.2613	1226.34900	72.50307	26.9080
2	17.195	BB	0.2748	1038.21423	58.02138	22.7800
3	19.062	BV	0.3070	1033.42871	52.19687	22.6750
4	20.334	VB	0.3424	1259.56824	57.31629	27.6369



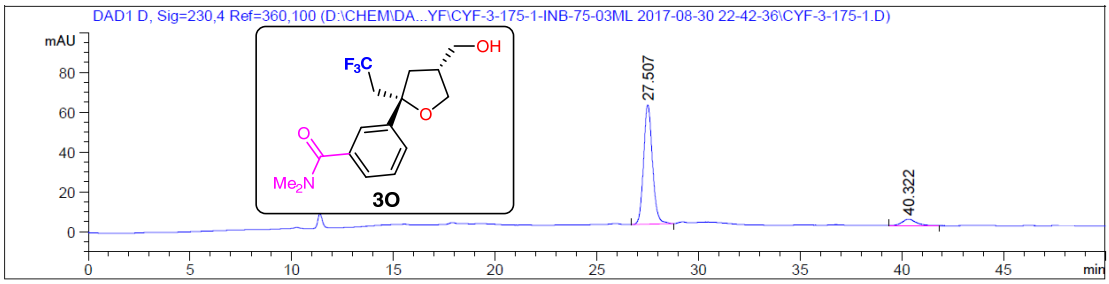
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.178	BB	0.2837	6402.46045	349.51114	95.2072
2	19.104	BB	0.3138	322.30734	16.21937	4.7928



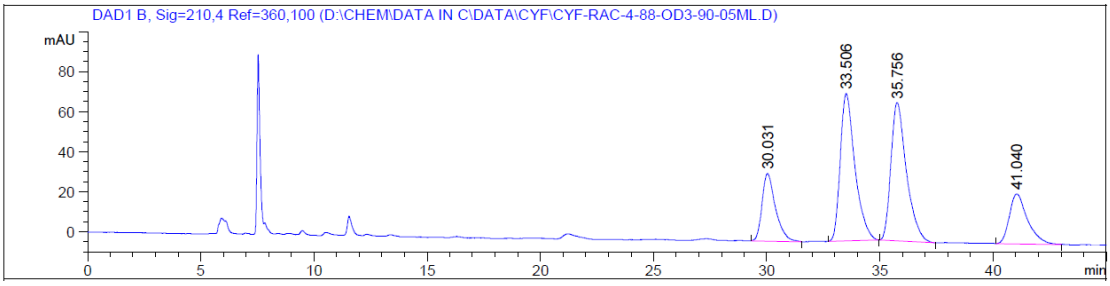
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.422	BV	0.4541	2888.37305	97.35933	28.9419
2	28.266	VV	0.4616	2045.13611	67.09267	20.4926
3	29.046	VB	0.4984	2132.29932	65.06158	21.3659
4	40.105	BB	0.6830	2914.08765	65.50219	29.1996



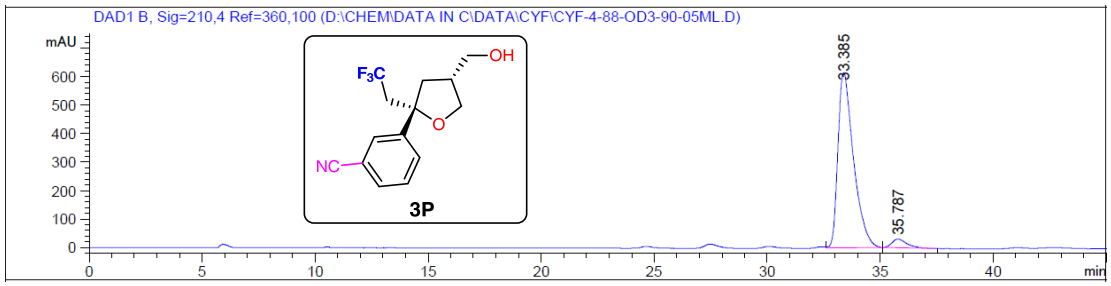
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.507	BB	0.4627	1820.39429	59.87878	92.2240
2	40.322	BB	0.6264	153.48993	3.19557	7.7760



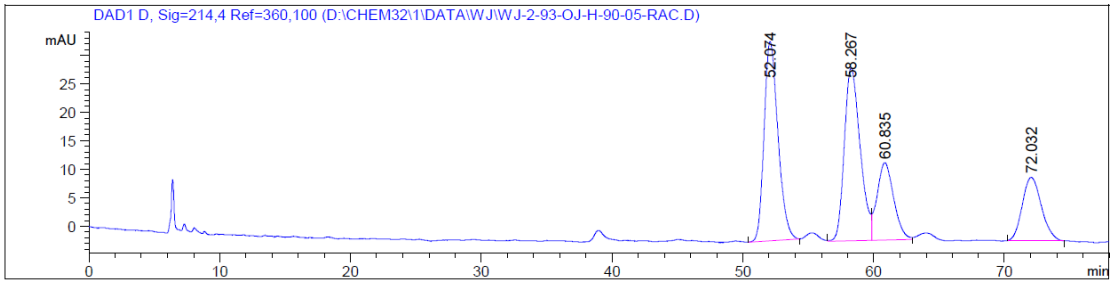
Signal 3: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.031	BB	0.5820	1386.39111	33.83682	14.7700
2	33.506	BB	0.6489	3320.67065	73.52528	35.3770
3	35.756	BB	0.7107	3298.15356	68.87752	35.1371
4	41.040	BB	0.7349	1381.30188	24.98699	14.7158



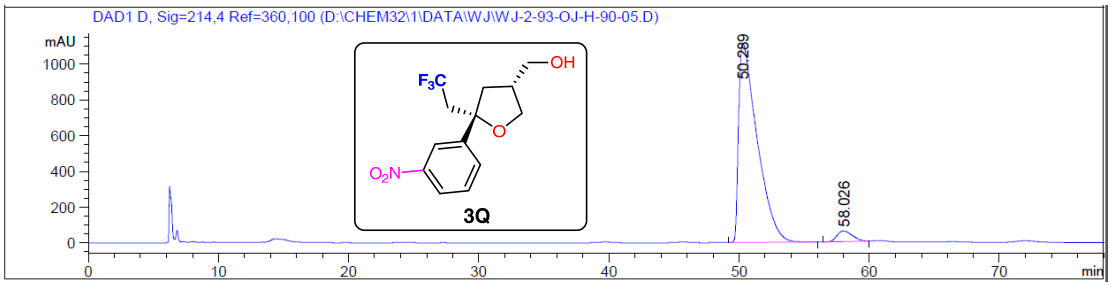
Signal 3: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	33.385	VB	0.7204	2.94386e4	615.02112	95.3996
2	35.787	BB	0.6606	1419.61267	30.74841	4.6004



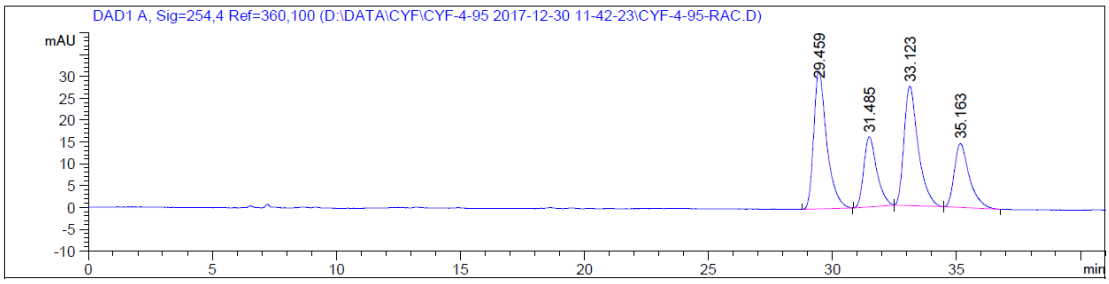
Signal 4: DAD1 D, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	52.074	BB	1.0501	2573.68628	34.91041	34.5234
2	58.267	MF R	1.4067	2556.06396	30.28464	34.2870
3	60.835	FM R	1.4692	1194.39771	13.54933	16.0216
4	72.032	BB	1.2018	1130.75537	11.10645	15.1679



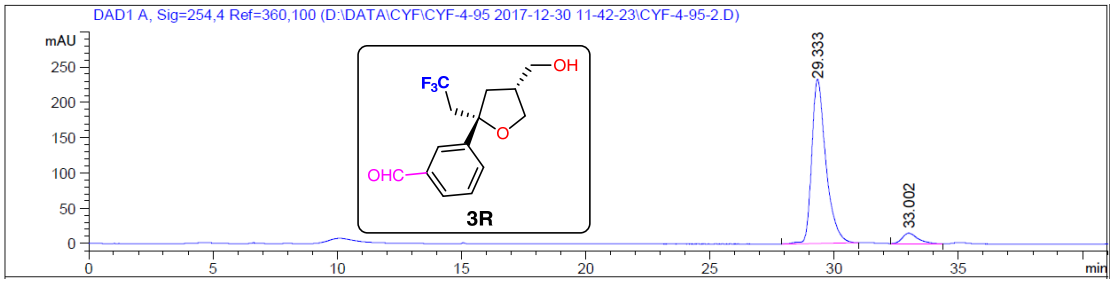
Signal 4: DAD1 D, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	50.289	BB	1.3383	1.10497e5	1114.88184	95.7497
2	58.026	BB	1.2013	4904.92188	59.90464	4.2503



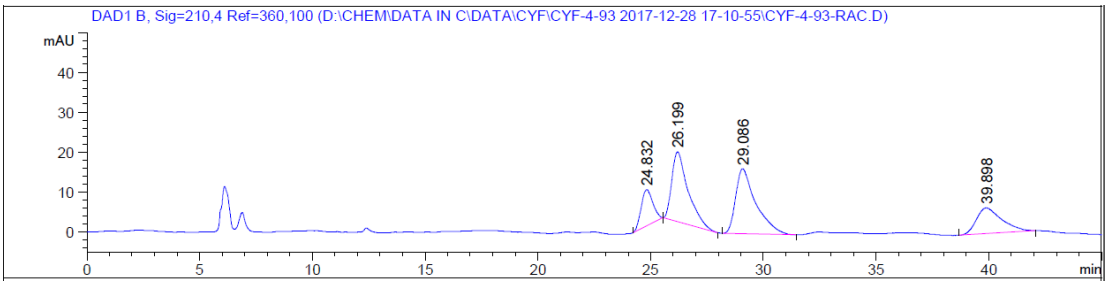
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.459	BB	0.5353	1148.85205	31.51655	33.4789
2	31.485	BB	0.5365	583.11511	16.02670	16.9926
3	33.123	BB	0.5898	1080.65222	27.30252	31.4914
4	35.163	BB	0.6149	618.95508	14.65069	18.0371



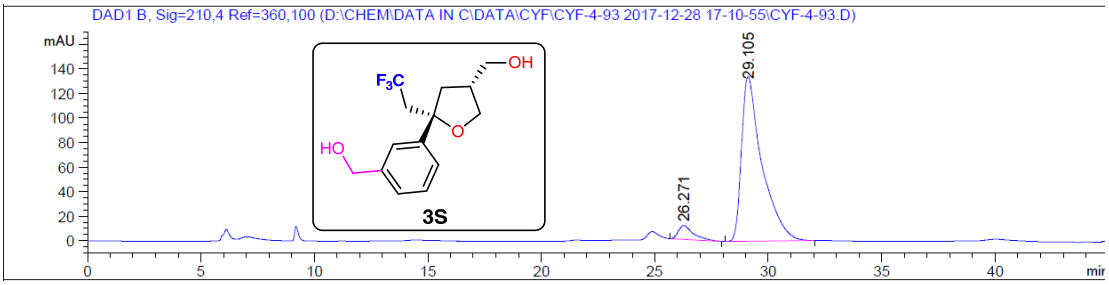
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.333	BB	0.5934	9221.75684	233.15775	93.6158
2	33.002	BB	0.6310	628.88556	14.76334	6.3842



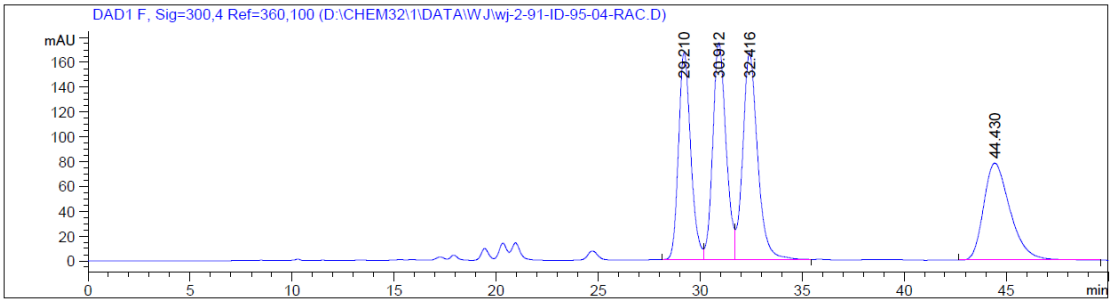
Signal 2: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.832	BB	0.5406	327.26776	9.07821	11.9675
2	26.199	BB	0.7261	887.52356	17.54707	32.4549
3	29.086	BB	0.8790	1016.68719	16.27104	37.1781
4	39.898	BB	1.0117	503.15900	6.43905	18.3995



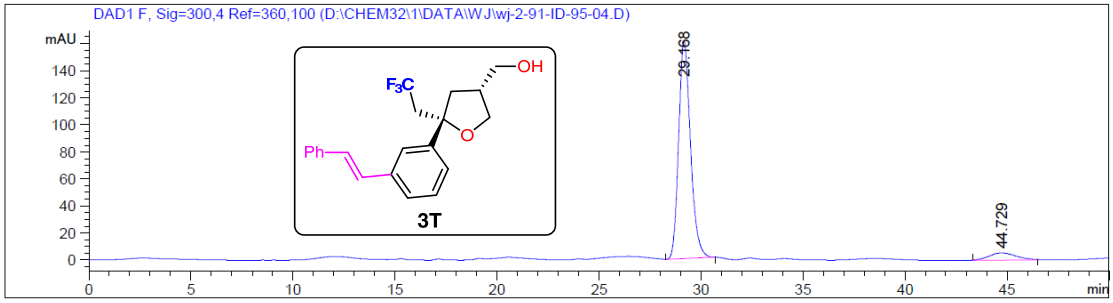
Signal 2: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.271	BB	0.6571	516.67053	11.30738	5.7885
2	29.105	BB	0.8966	8409.12500	134.28697	94.2115



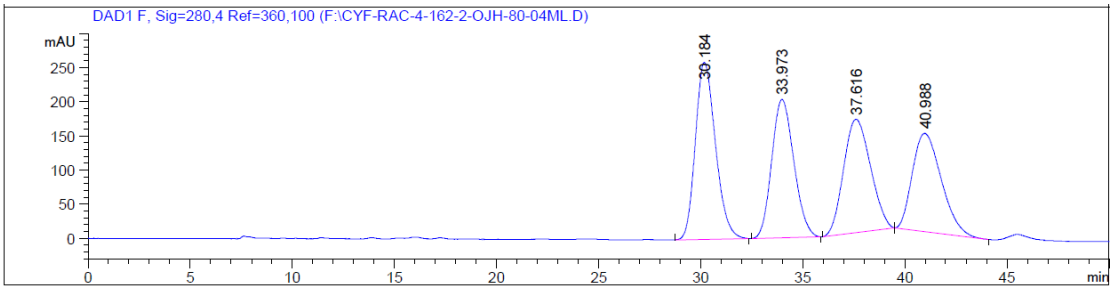
Signal 5: DAD1 F, Sig=300,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.210	BV	0.6394	6934.98486	166.68021	22.9154
2	30.912	VV	0.6936	7867.49902	174.63292	25.9967
3	32.416	VB	0.7585	8384.78320	166.21631	27.7060
4	44.430	BB	1.3419	7076.17383	78.18159	23.3819



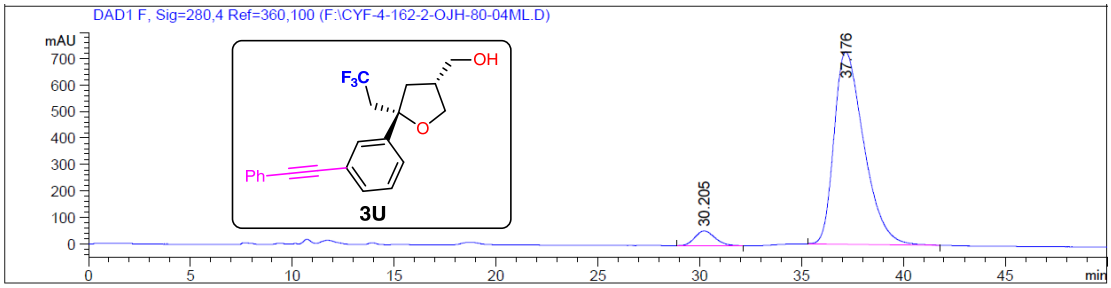
Signal 5: DAD1 F, Sig=300,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.168	BB	0.6200	6500.63477	160.69235	93.0830
2	44.729	MM R	1.5209	483.06427	5.29353	6.9170



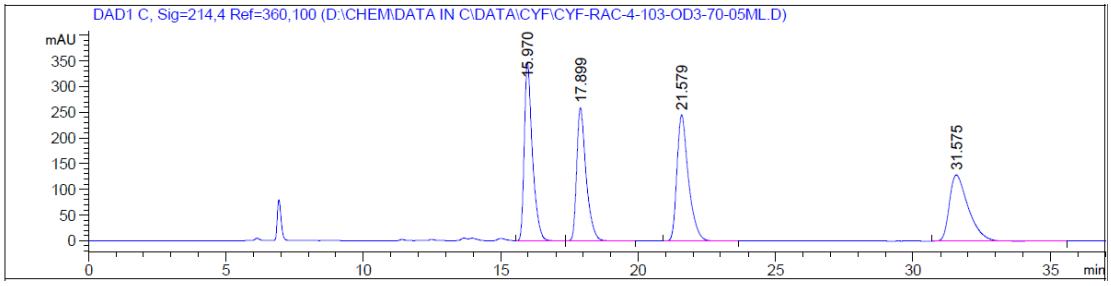
Signal 6: DAD1 F, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.184	BB	0.9066	1.77471e4	258.62656	28.4453
2	33.973	BB	0.9212	1.52612e4	202.51894	24.4609
3	37.616	BB	1.0846	1.51869e4	165.80392	24.3418
4	40.988	BB	1.1687	1.41951e4	143.66489	22.7521



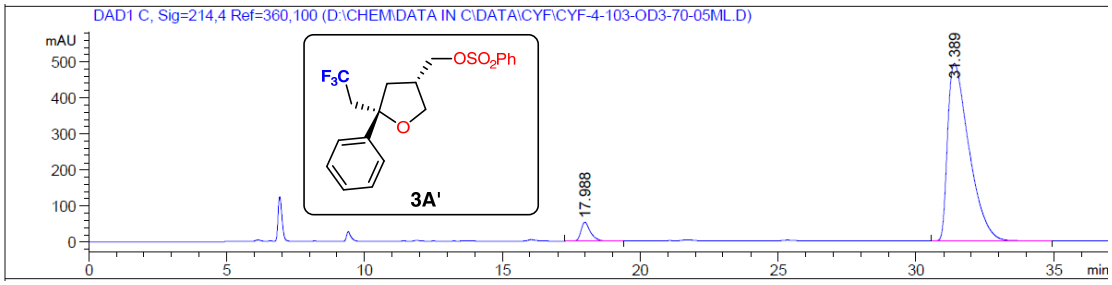
Signal 6: DAD1 F, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.205	BB	0.8291	3769.14990	55.30339	4.8614
2	37.176	BB	1.3077	7.37635e4	727.02332	95.1386



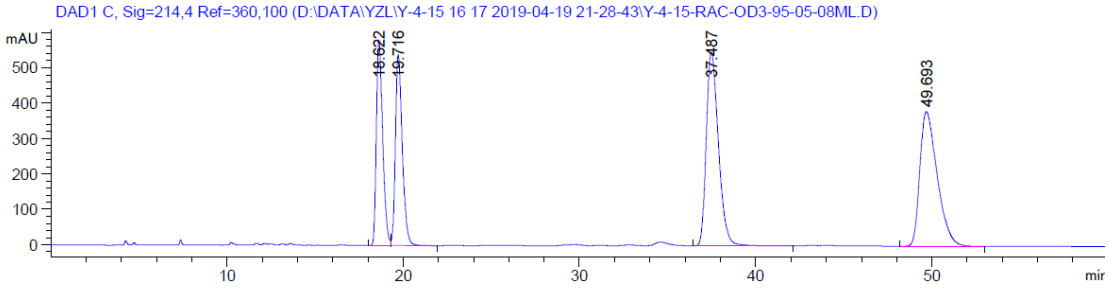
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.970	VB	0.3041	7150.91748	347.52399	26.9857
2	17.899	BB	0.3508	6109.56836	259.34238	23.0559
3	21.579	BB	0.4335	7097.41357	245.33479	26.7838
4	31.575	BB	0.7205	6140.98682	128.73430	23.1745



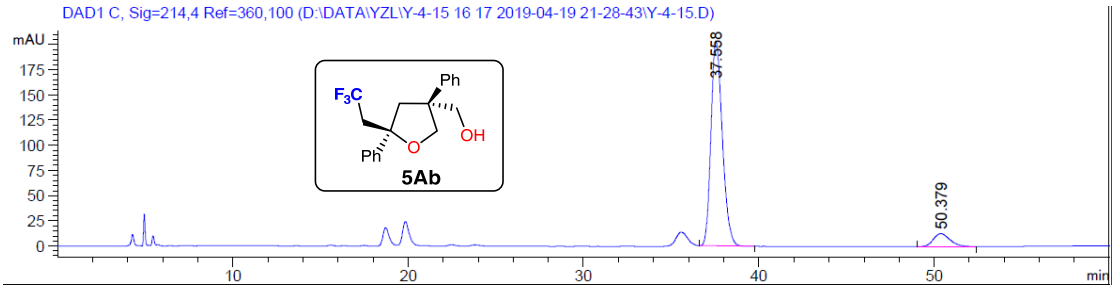
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.988	BB	0.3473	1237.77209	52.83893	4.4523
2	31.389	BB	0.8203	2.65632e4	493.21371	95.5477



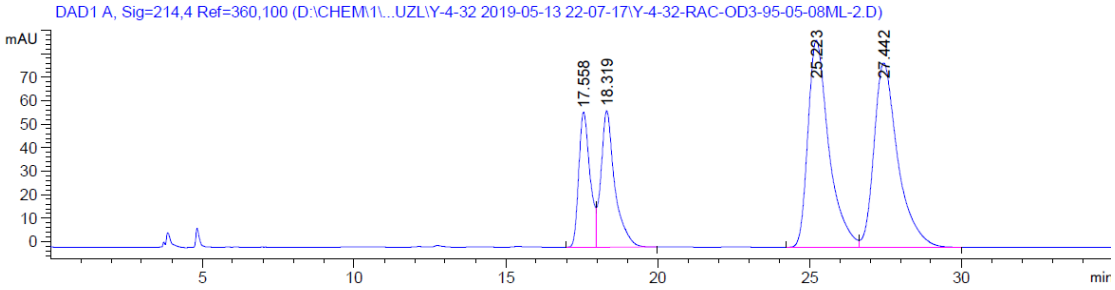
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.622	BV	0.3836	1.45073e4	580.33557	17.7850
2	19.716	VB	0.4198	1.47489e4	538.11884	18.0812
3	37.487	BB	0.7432	2.63039e4	546.78882	32.2467
4	49.693	BB	1.0427	2.60106e4	379.92642	31.8872



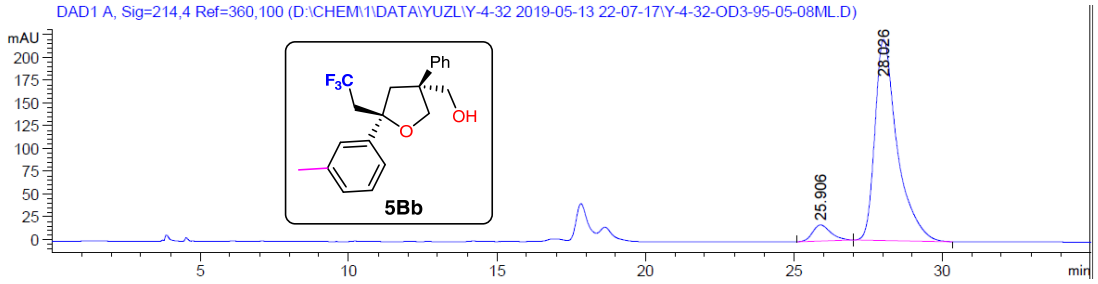
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	37.558	BB	0.6921	9179.86816	203.58214	91.5879
2	50.379	BB	0.9844	843.15137	12.93593	8.4121



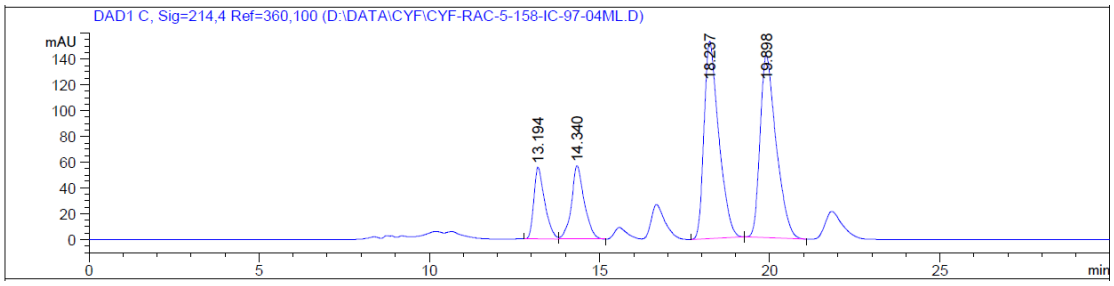
Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.558	BV	0.3853	1472.45007	57.38964	12.4658
2	18.319	VB	0.4581	1840.10291	58.00798	15.5784
3	25.223	BV	0.7165	4215.46924	88.06298	35.6883
4	27.442	VB	0.8158	4283.87646	78.35804	36.2675



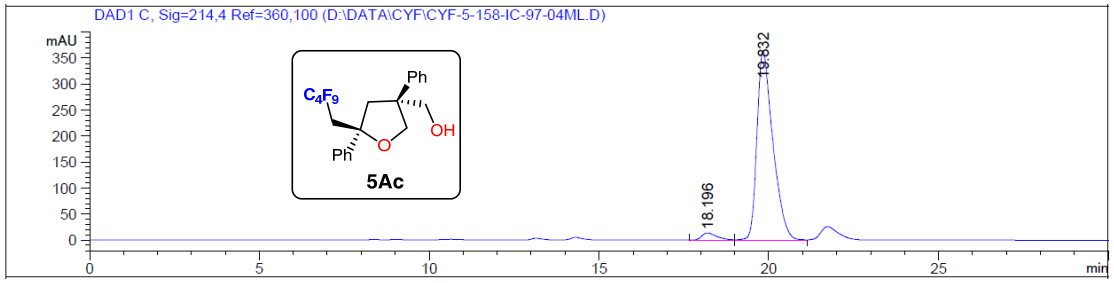
Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.906	BB	0.6301	745.15533	17.73863	6.0732
2	28.026	BB	0.7751	1.15244e4	220.74930	93.9268



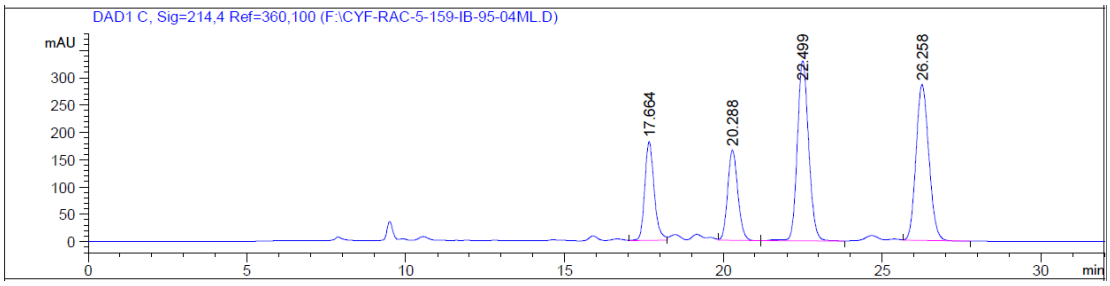
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.194	BV	0.3292	1229.13330	55.32826	10.2677
2	14.340	VB	0.3743	1480.94006	56.77795	12.3712
3	18.237	BB	0.4414	4602.87061	152.80943	38.4505
4	19.898	BB	0.4862	4657.94287	141.45708	38.9106



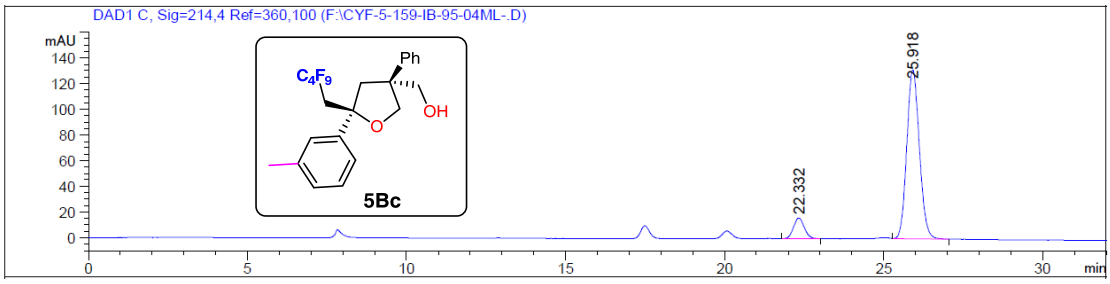
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.196	BB	0.4399	409.71252	13.81825	3.3551
2	19.832	BB	0.4810	1.18019e4	363.26782	96.6449



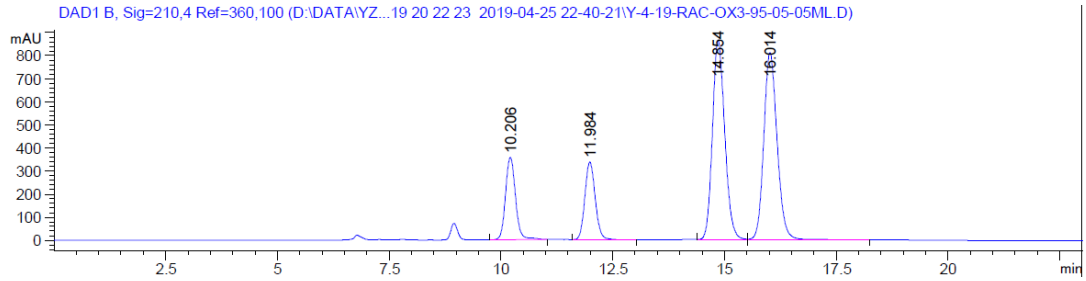
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.664	BV	0.3153	3697.66089	180.30026	15.6553
2	20.288	VB	0.3469	3696.87402	165.30608	15.6519
3	22.499	BB	0.3860	8189.42285	329.48044	34.6726
4	26.258	VB	0.4387	8035.30908	285.18555	34.0201



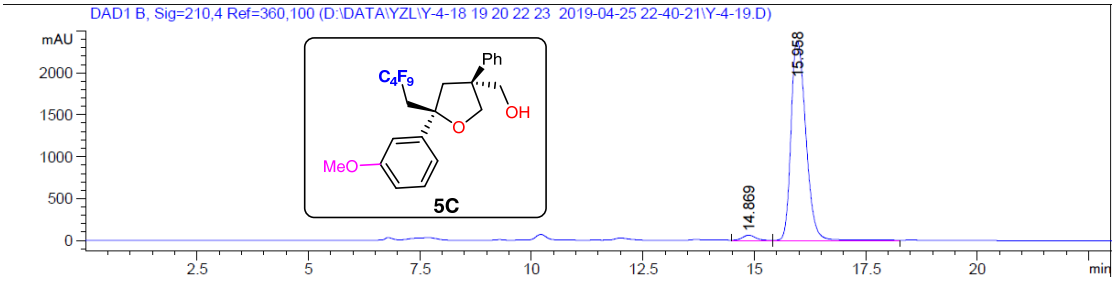
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.332	BB	0.3667	378.52457	16.07704	9.4936
2	25.918	BB	0.4240	3608.61108	131.56493	90.5064



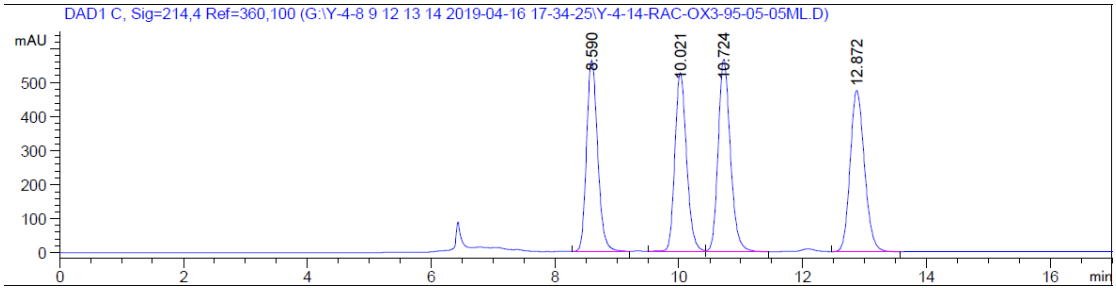
Signal 2: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.206	BB	0.2452	5608.22461	357.20148	12.5922
2	11.984	BB	0.2530	5453.67188	336.72153	12.2452
3	14.854	VV	0.2977	1.66094e4	866.41919	37.2932
4	16.014	VB	0.3204	1.68660e4	811.81787	37.8694



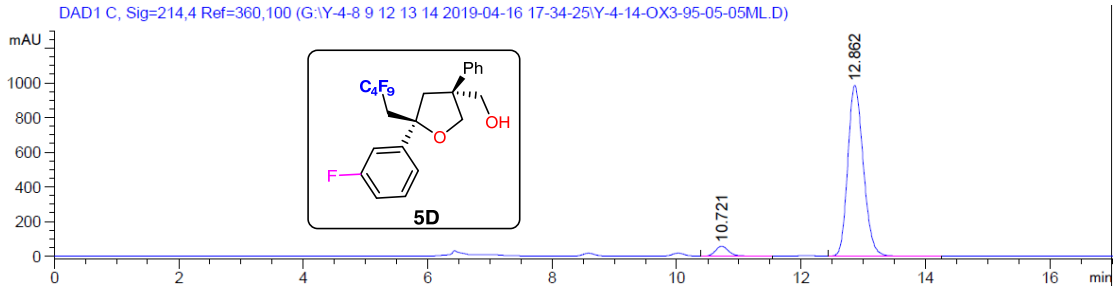
Signal 2: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.869	BB	0.3013	1189.32849	61.57537	2.1228
2	15.958	BB	0.3585	5.48382e4	2382.93262	97.8772



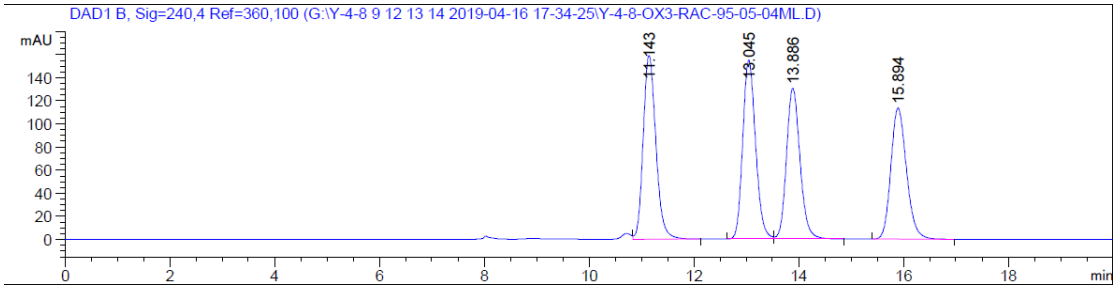
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.590	VV	0.1963	7053.53516	562.54340	23.9881
2	10.021	VV	0.2015	6929.28662	526.63977	23.5656
3	10.724	VB	0.2106	7786.35156	565.65210	26.4803
4	12.872	VB	0.2499	7635.10352	474.07016	25.9660



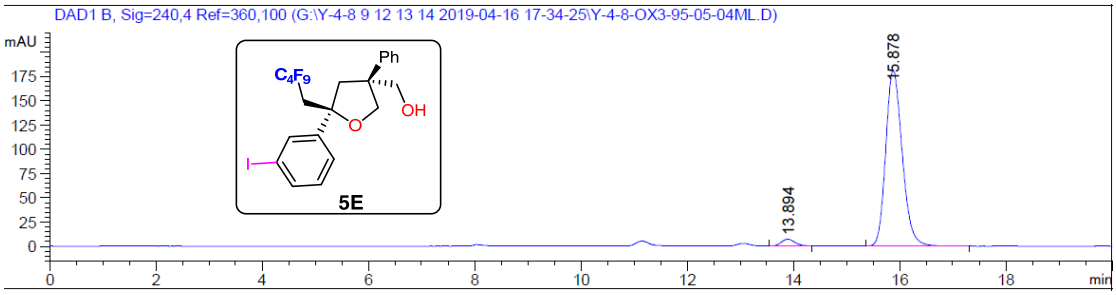
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.721	BB	0.2264	848.36475	57.42447	4.7439
2	12.862	VB	0.2679	1.70351e4	984.38892	95.2561



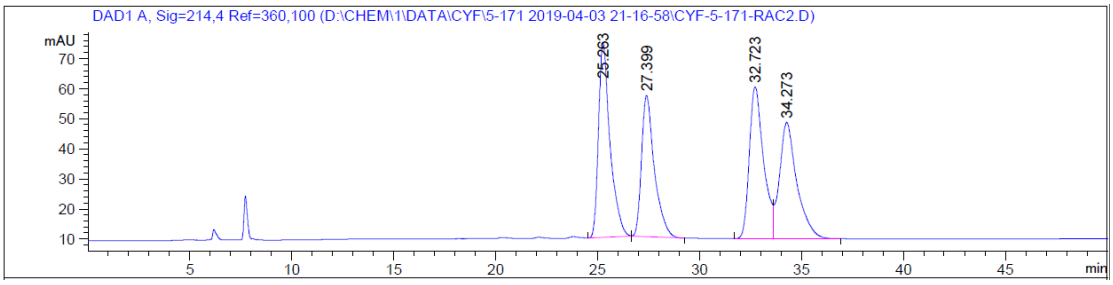
Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.143	VB	0.2618	2667.36597	158.89479	26.6834
2	13.045	BV	0.2645	2631.02441	154.65254	26.3199
3	13.886	VB	0.2781	2347.58838	130.36530	23.4845
4	15.894	BB	0.3218	2350.36523	113.38927	23.5122



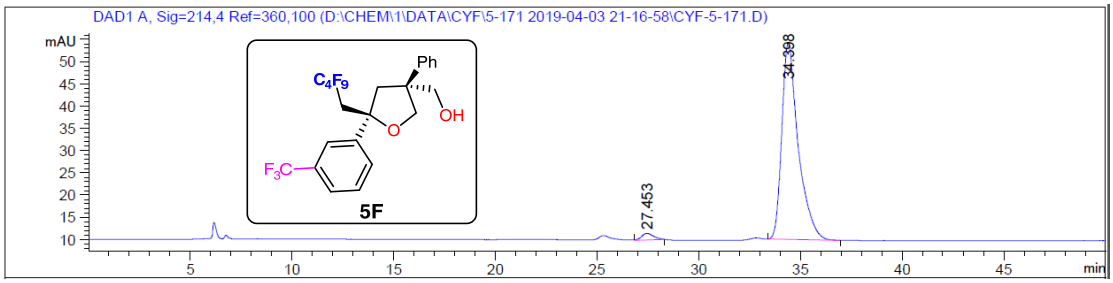
Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.894	BB	0.2710	124.02787	7.05961	3.0789
2	15.878	BB	0.3334	3904.22559	181.19975	96.9211



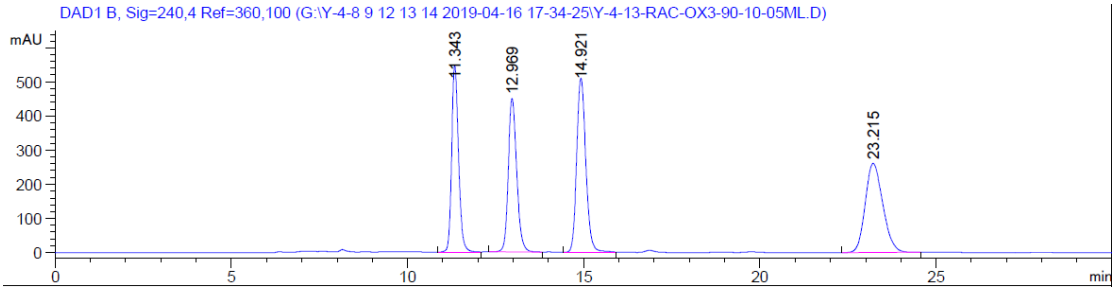
Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.263	BB	0.5770	2540.52808	64.85359	27.2182
2	27.399	BB	0.6517	2053.85059	47.01408	22.0041
3	32.723	BV	0.7193	2454.64917	50.48459	26.2981
4	34.273	VB	0.8542	2284.90967	38.62454	24.4796



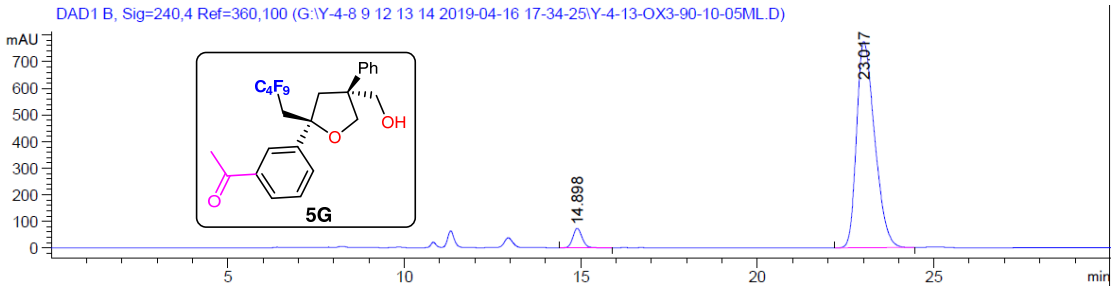
Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.453	MM R	0.6737	60.16660	1.48841	2.3693
2	34.398	BB	0.8263	2479.22559	44.20940	97.6307
Totals :				2539.39219	45.69781	



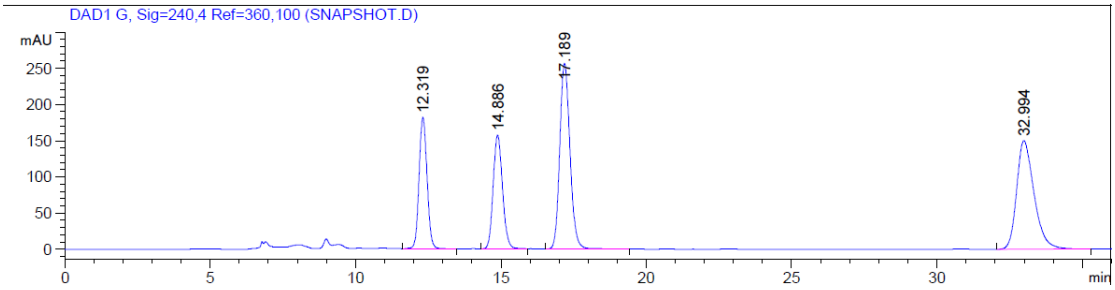
Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.343	BB	0.2049	7347.87451	546.20404	22.2892
2	12.969	BB	0.2534	7445.48096	449.16904	22.5852
3	14.921	BB	0.2765	9111.44531	509.92374	27.6388
4	23.215	BB	0.5391	9061.33496	260.96198	27.4868



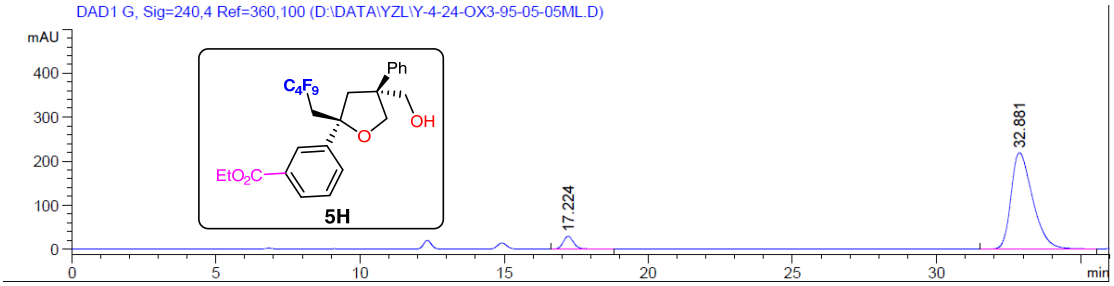
Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.898	BB	0.2819	1340.89160	73.14252	4.6260
2	23.017	BB	0.5522	2.76451e4	774.97345	95.3740



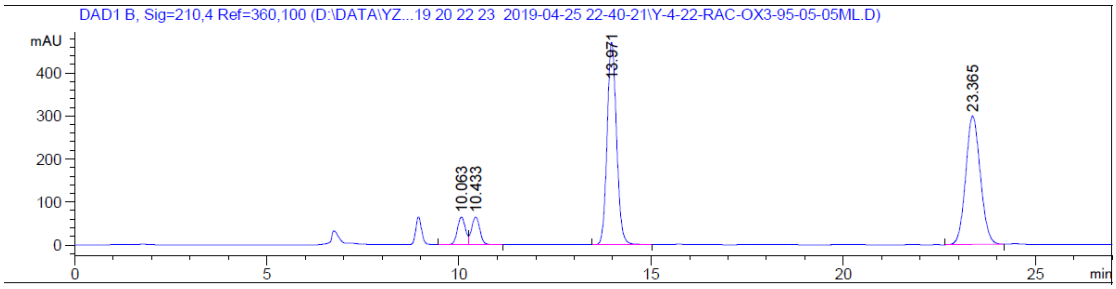
Signal 7: DAD1 G, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.319	BB	0.2995	3470.48730	181.13857	17.8569
2	14.886	BB	0.3452	3489.75488	157.06197	17.9560
3	17.189	BB	0.3786	6256.15820	256.42615	32.1902
4	32.994	BB	0.6244	6218.60254	149.78398	31.9969



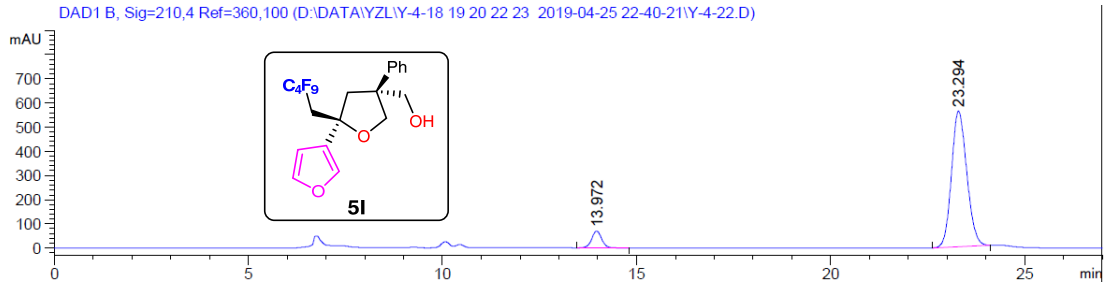
Signal 7: DAD1 G, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.224	BB	0.3848	731.03217	29.12525	6.0324
2	32.881	BB	0.7902	1.13874e4	219.14478	93.9676



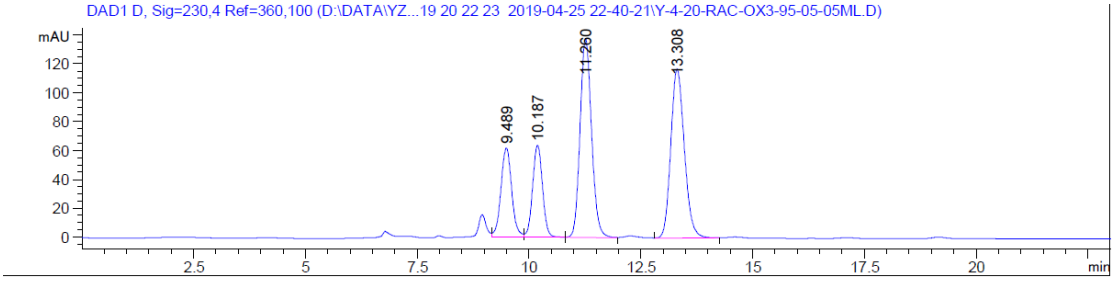
Signal 2: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.063	BV	0.2305	934.55804	63.94017	5.2386
2	10.433	VB	0.2327	964.03540	64.37975	5.4038
3	13.971	BB	0.2645	8013.96533	470.98416	44.9217
4	23.365	BB	0.4116	7927.28467	298.75360	44.4358



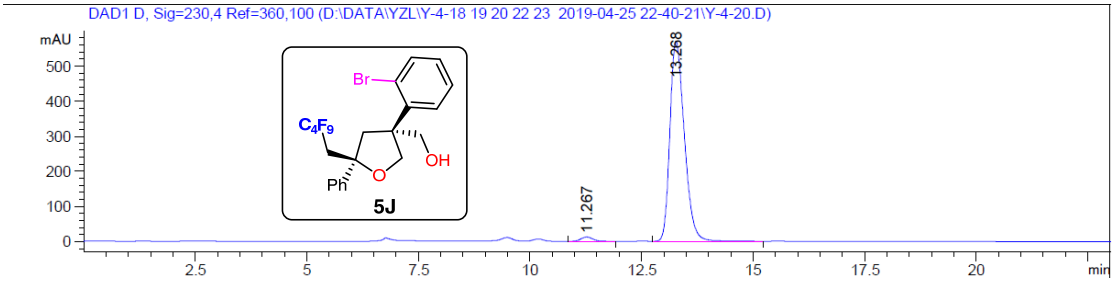
Signal 2: DAD1 B, Sig=210,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.972	BB	0.2748	1241.39148	69.37071	7.4920
2	23.294	BB	0.4206	1.53283e4	561.27380	92.5080



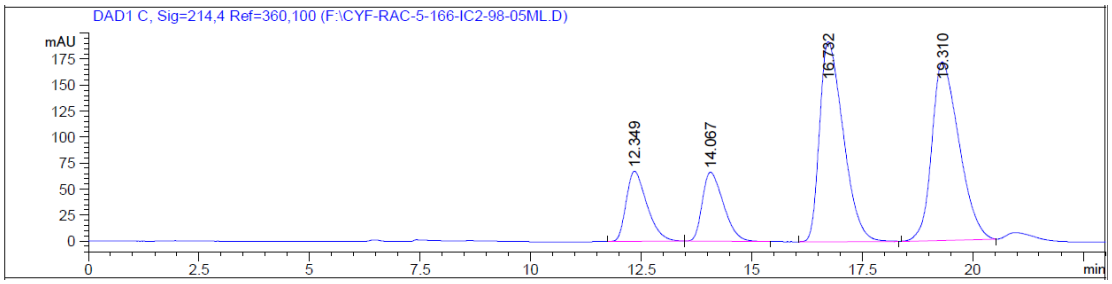
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.489	VV	0.2612	1047.10266	61.94609	15.0802
2	10.187	VB	0.2450	987.80627	63.65491	14.2262
3	11.260	BB	0.2755	2451.54907	137.86864	35.3069
4	13.308	BB	0.3250	2457.09204	116.99845	35.3867



Signal 4: DAD1 D, Sig=230,4 Ref=360,100

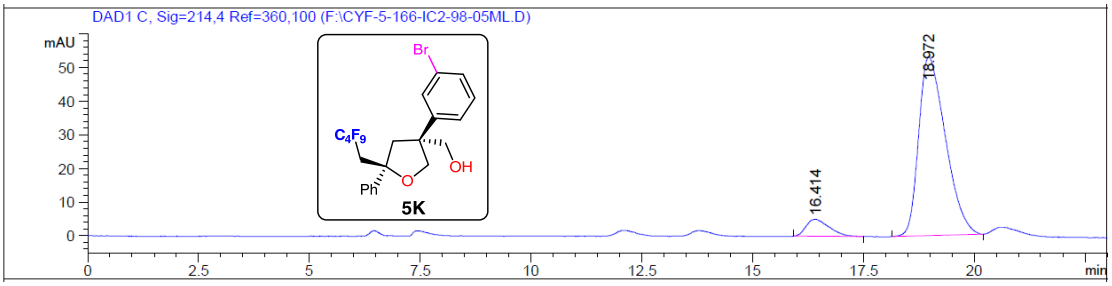
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.267	BB	0.2880	230.08131	12.42473	1.7599
2	13.268	BB	0.3465	1.28437e4	570.83929	98.2401



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

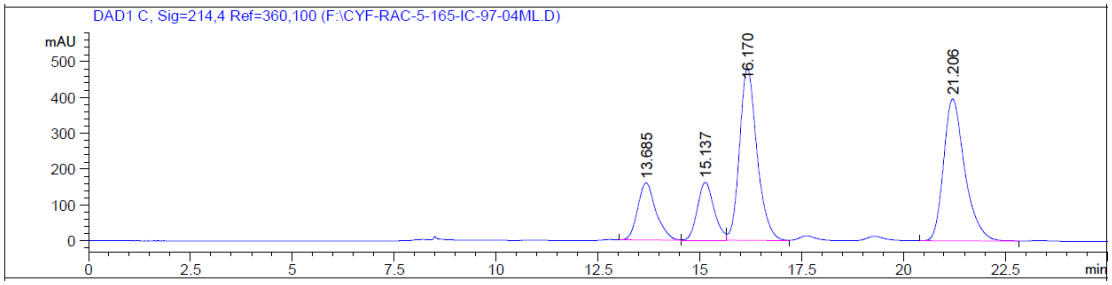
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.349	BB	0.4800	2183.48486	67.39267	11.5544
2	14.067	BB	0.5072	2199.93677	66.28188	11.6414

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
3	16.732	BB	0.5683	7160.52148	192.34320	37.8914
4	19.310	BB	0.6698	7353.54004	171.63928	38.9128



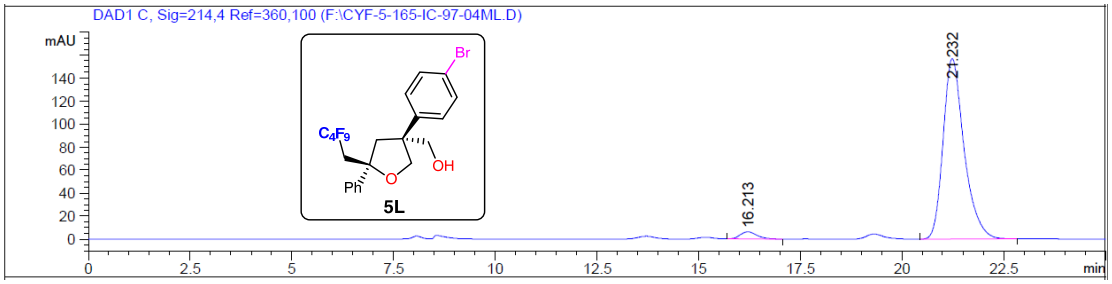
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.414	BB	0.4569	185.27548	5.06389	7.6569
2	18.972	BB	0.5983	2234.45874	53.16476	92.3431



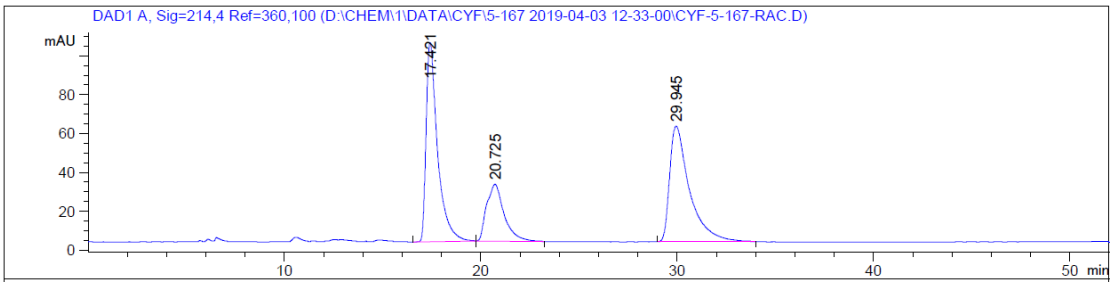
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.685	BV	0.4709	4969.02490	159.72894	12.9884
2	15.137	VV	0.4412	4621.37451	161.79559	12.0797
3	16.170	VV	0.4561	1.43397e4	480.62531	37.4823
4	21.206	BB	0.5443	1.43273e4	395.84415	37.4497



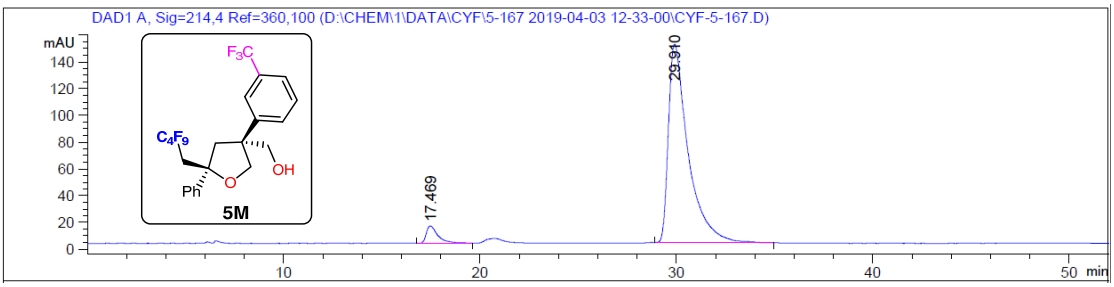
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.213	BB	0.4169	181.52084	6.28635	3.1198
2	21.232	BB	0.5429	5636.83936	156.98082	96.8802



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

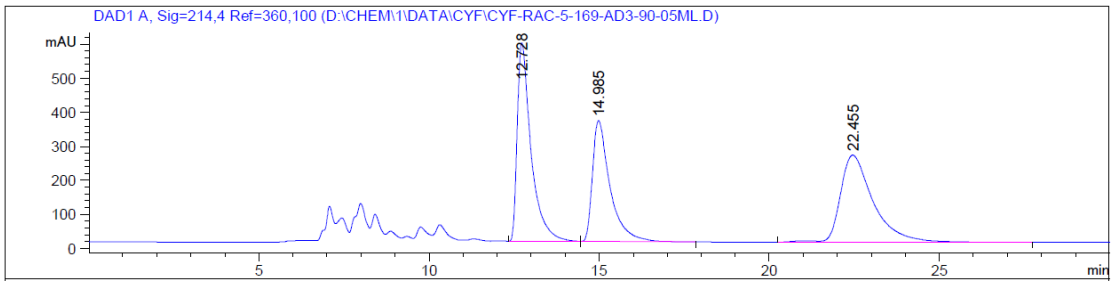
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.421	BB	0.5972	4139.50049	102.49366	40.9273
2	20.725	BB	0.8546	1805.44568	29.31186	17.8505
3	29.945	BB	1.0226	4169.32568	59.56519	41.2222



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

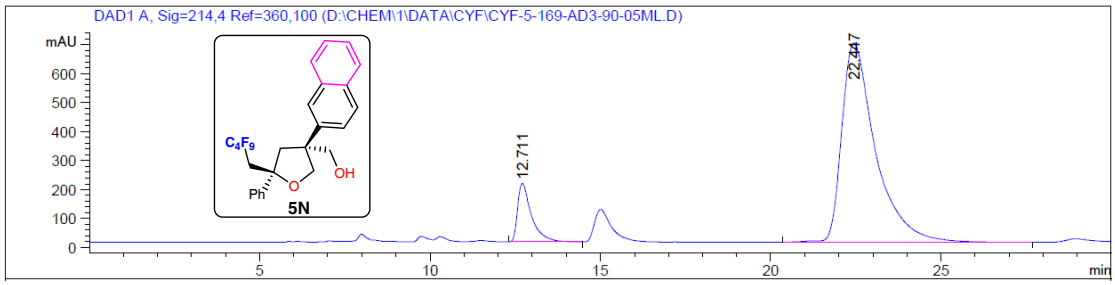
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.469	BB	0.6004	529.50562	12.96813	4.8556
2	29.910	BB	1.0123	1.03755e4	148.29305	95.1444

Note: After optimization of separation conditions (including: Chiralcel OD3, OX3, AZ3, AD3, OJ, IA, IB, IC, ID, IE, IF, IG, INB), we can't get four peaks with baseline separation.



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

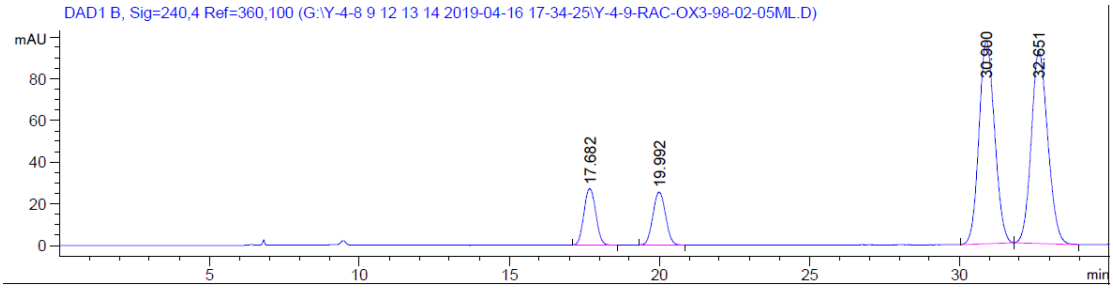
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.728	BB	0.4172	1.64340e4	582.35168	35.5455
2	14.985	BB	0.5278	1.26978e4	354.55963	27.4645
3	22.455	BB	0.9964	1.71018e4	255.68063	36.9900



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

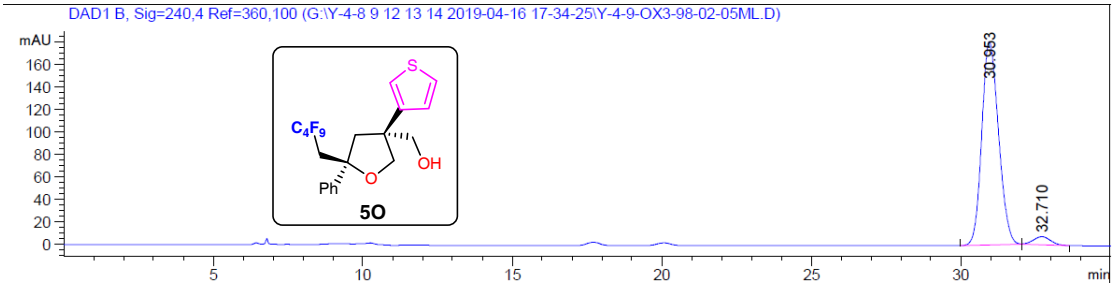
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.711	BB	0.4160	5701.07227	201.54233	10.7637
2	22.447	BB	1.0219	4.72649e4	687.64966	89.2363

Note: After optimization of separation conditions (including: Chiralcel OD3, OX3, AZ3, AD3, OJ, IA, IB, IC, ID, IE, IF, IG, INB), we can't get four peaks with baseline separation.



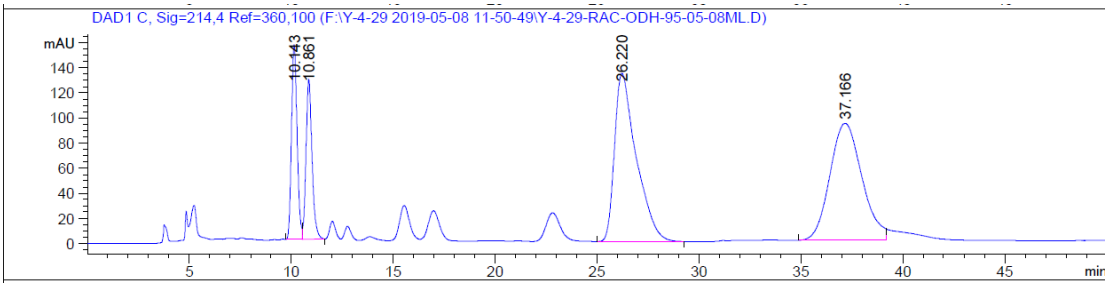
Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.682	BB	0.4346	743.52649	27.05840	8.7236
2	19.992	BB	0.4587	746.46185	25.40870	8.7580
3	30.900	BB	0.5613	3508.49805	97.16048	41.1643
4	32.651	BB	0.5897	3524.67285	91.84053	41.3541



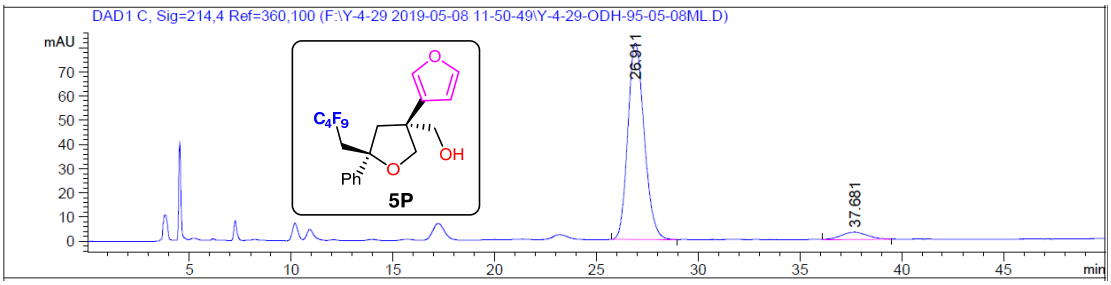
Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.953	BB	0.5999	7015.03857	181.09589	96.1810
2	32.710	BB	0.5634	278.53830	7.23088	3.8190



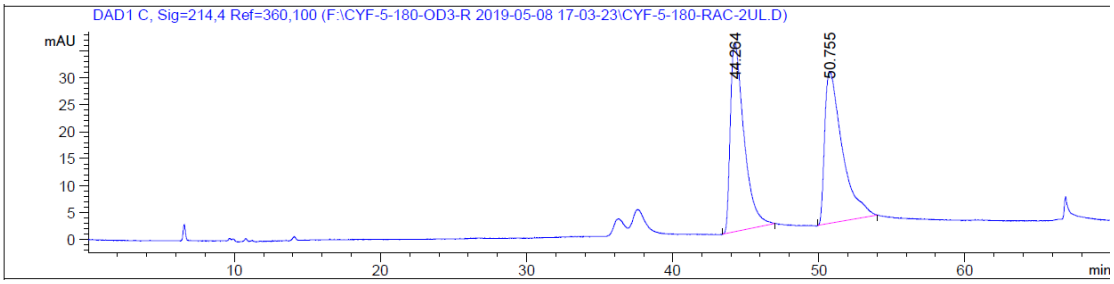
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.143	BV	0.2935	2942.49121	154.95357	11.4775
2	10.861	VB	0.3141	2611.55005	126.87960	10.1866
3	26.220	BB	1.0466	9910.73047	134.04820	38.6578
4	37.166	MF R	1.8203	1.01723e4	93.13791	39.6781



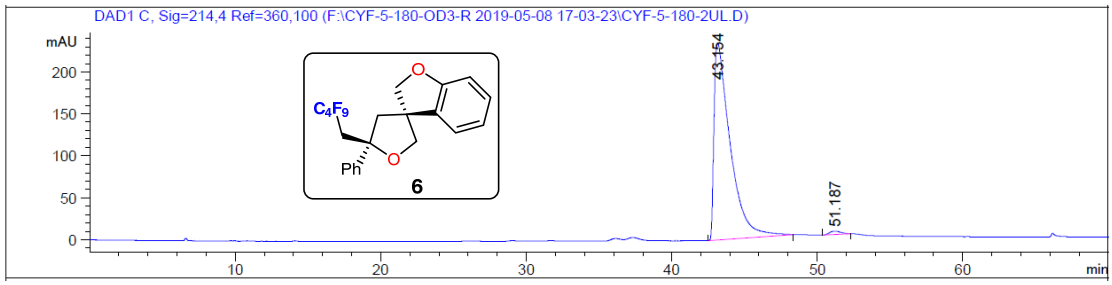
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.911	BB	0.8369	4544.39453	81.20930	94.3267
2	37.681	MM R	1.4884	273.32385	3.06064	5.6733



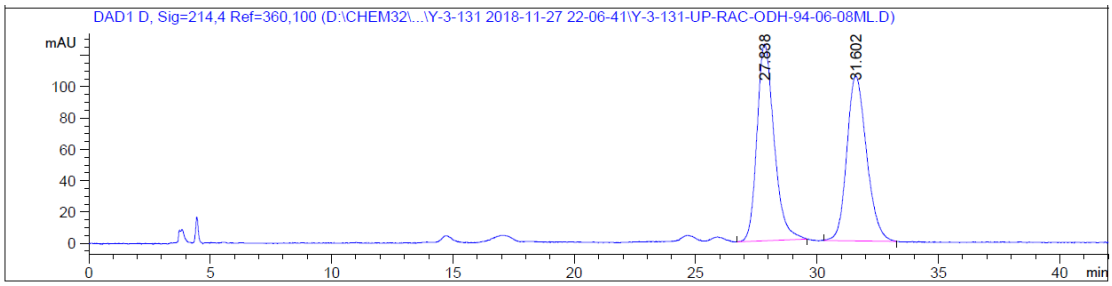
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	44.264	BB	0.8965	2288.64233	35.10999	49.2617
2	50.755	BB	1.0137	2357.24023	28.18594	50.7383



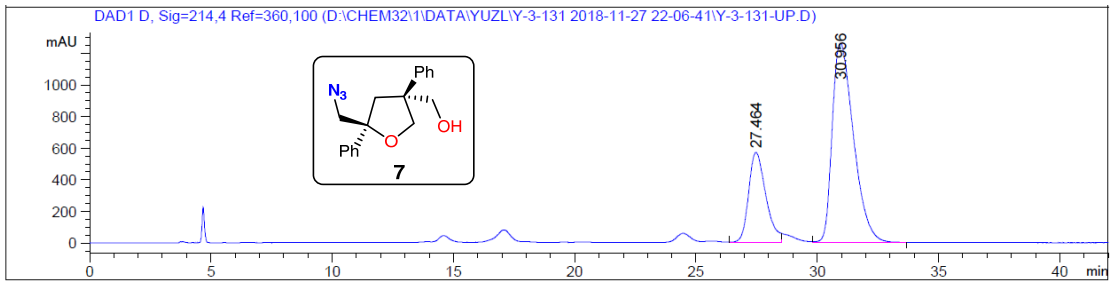
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	43.154	BB	1.0972	1.81360e4	234.77402	98.7594
2	51.187	BB	0.6637	227.81531	4.02867	1.2406



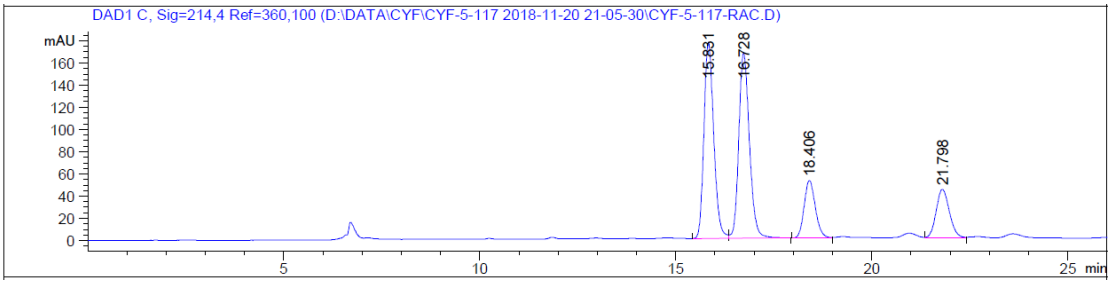
Signal 4: DAD1 D, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.838	BV	0.6519	6174.74854	125.51701	50.9451
2	31.602	VB	0.6822	5945.65820	105.07405	49.0549



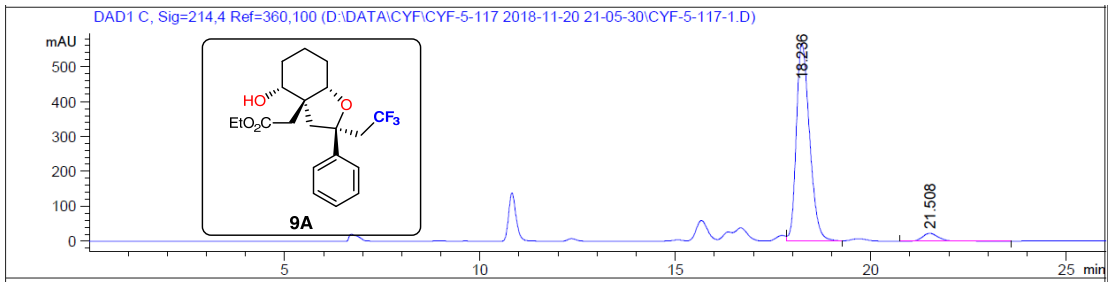
Signal 4: DAD1 D, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.464	MF R	0.8212	2.80822e4	569.94427	26.7075
2	30.956	VB	0.7246	7.70650e4	1261.69507	73.2925



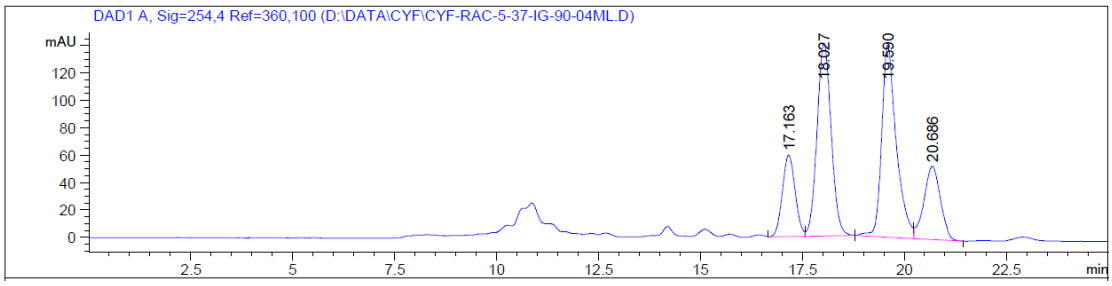
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.831	BV	0.2703	3065.69165	176.78943	37.1871
2	16.728	VB	0.2909	3131.94775	166.91808	37.9908
3	18.406	BV	0.3073	1026.49622	51.76733	12.4515
4	21.798	VV	0.3605	1019.82300	43.65857	12.3705



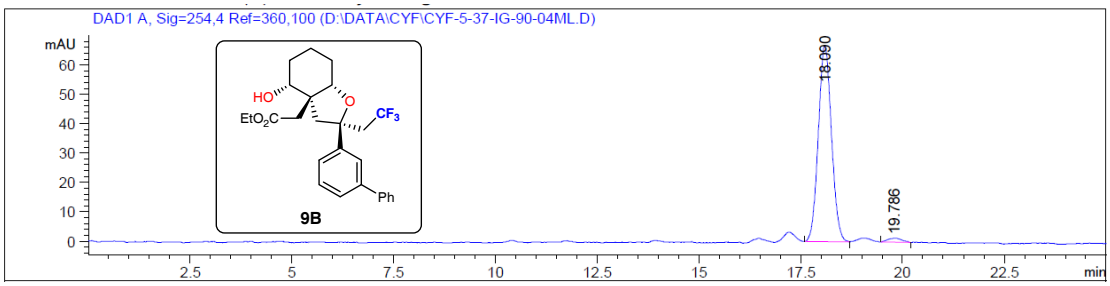
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.236	VB	0.3666	1.33195e4	565.80438	95.1925
2	21.508	BB	0.4523	672.67065	22.53261	4.8075



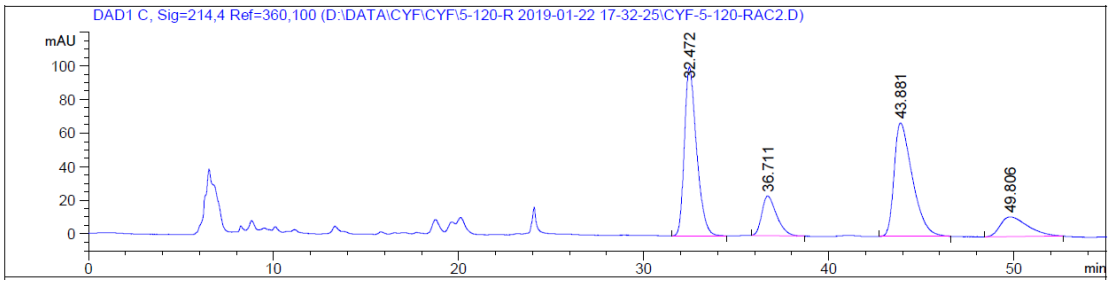
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.163	VV	0.3424	1297.08643	59.47280	13.4070
2	18.027	VB	0.3742	3386.44873	140.99812	35.0032
3	19.590	BV	0.3669	3491.32251	140.01476	36.0872
4	20.686	VB	0.4250	1499.81384	53.50762	15.5025



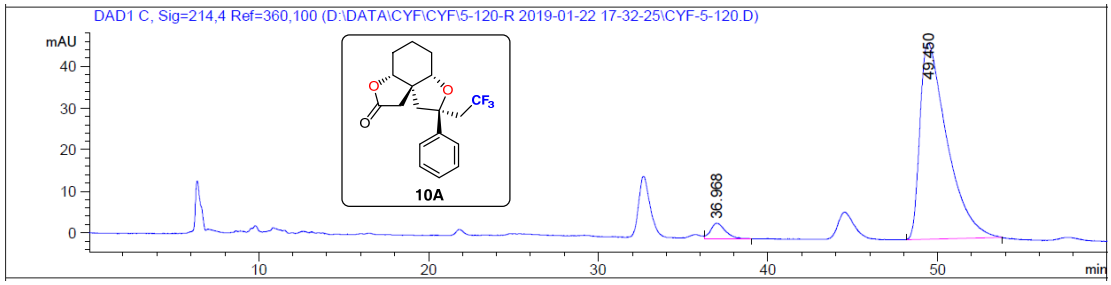
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.090	BB	0.3497	1494.51404	66.63515	97.9794
2	19.786	BB	0.2826	30.82079	1.31886	2.0206



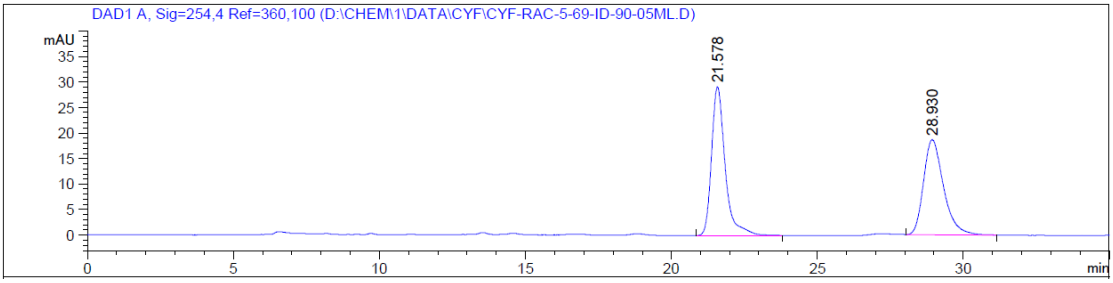
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.472	BB	0.6783	4606.78125	100.58542	39.1079
2	36.711	BB	0.7624	1303.94421	23.53449	11.0694
3	43.881	BB	0.9839	4644.99170	67.57496	39.4323
4	49.806	BB	1.2342	1223.94995	11.81860	10.3904



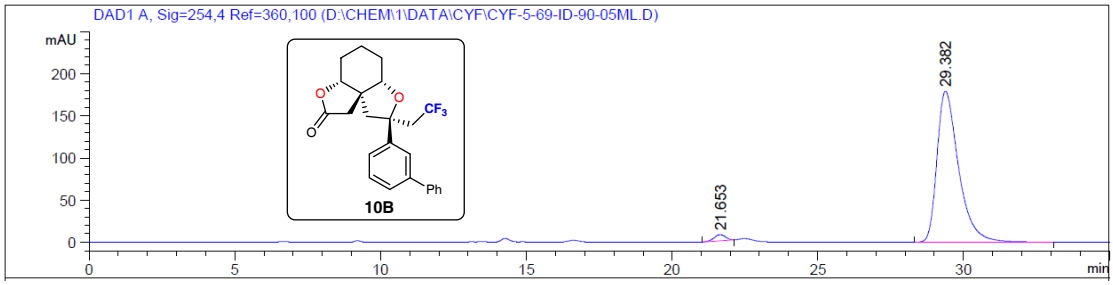
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.968	FM R	1.0642	245.64742	3.84720	4.4797
2	49.450	BB	1.3097	5237.92432	47.16376	95.5203



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.578	BB	0.4902	943.87634	29.11529	51.5869
2	28.930	BB	0.7247	885.80566	18.63152	48.4131



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.653	BB	0.4323	199.29295	7.30433	2.1200
2	29.382	BB	0.7799	9201.46973	178.94145	97.8800

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