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Chemie

Supporting Information

Achiral Pyridine Ligand-Enabled Enantioselective Radical Oxytrifluoromethylation of Alkenes with Alcohols

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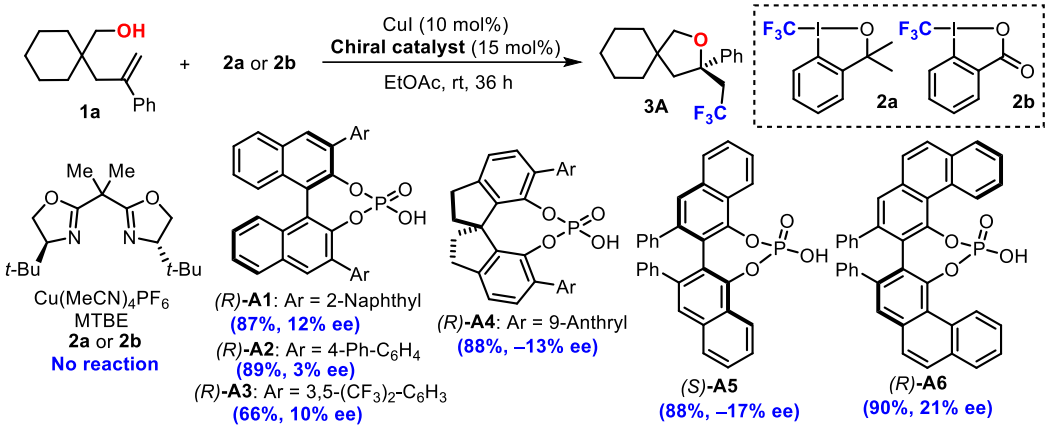
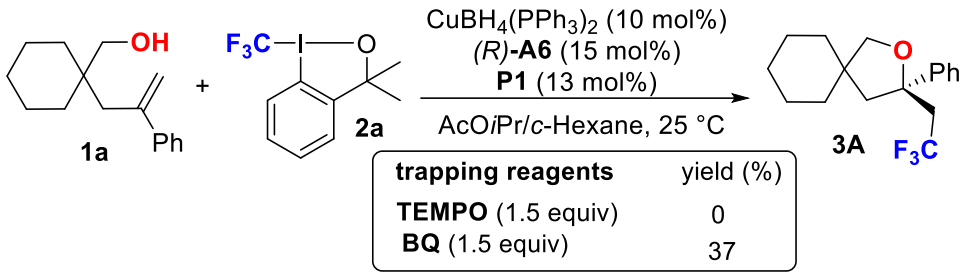


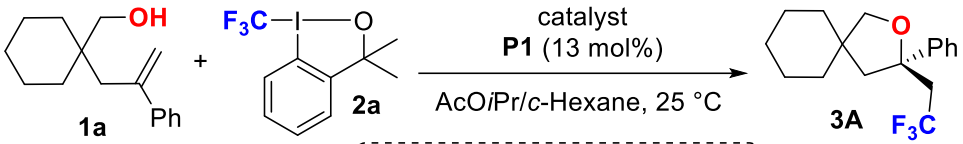
Scheme S1. Copper/bisoxazoline- or copper/CPA-catalyzed enantioselective oxytrifluoromethylation of **1a**

a) Trapping with radical scavengers



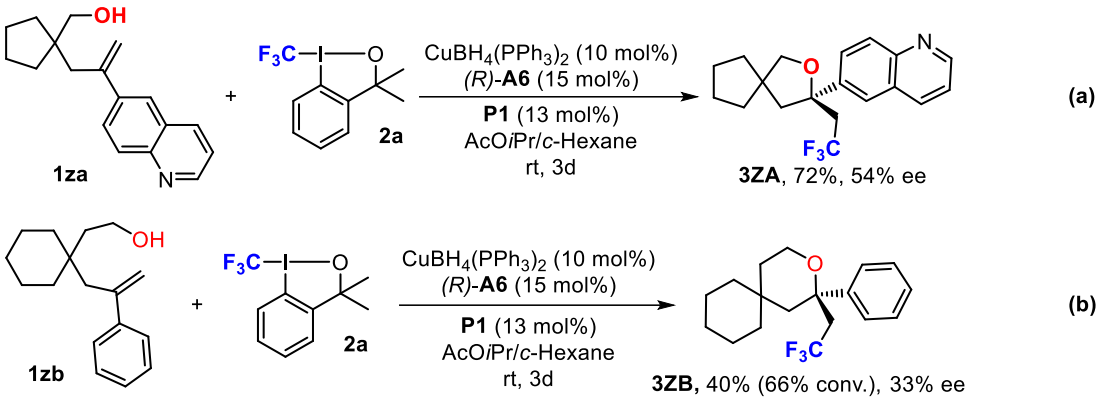
trapping reagents	yield (%)
TEMPO (1.5 equiv)	0
BQ (1.5 equiv)	37

b) Control reactions

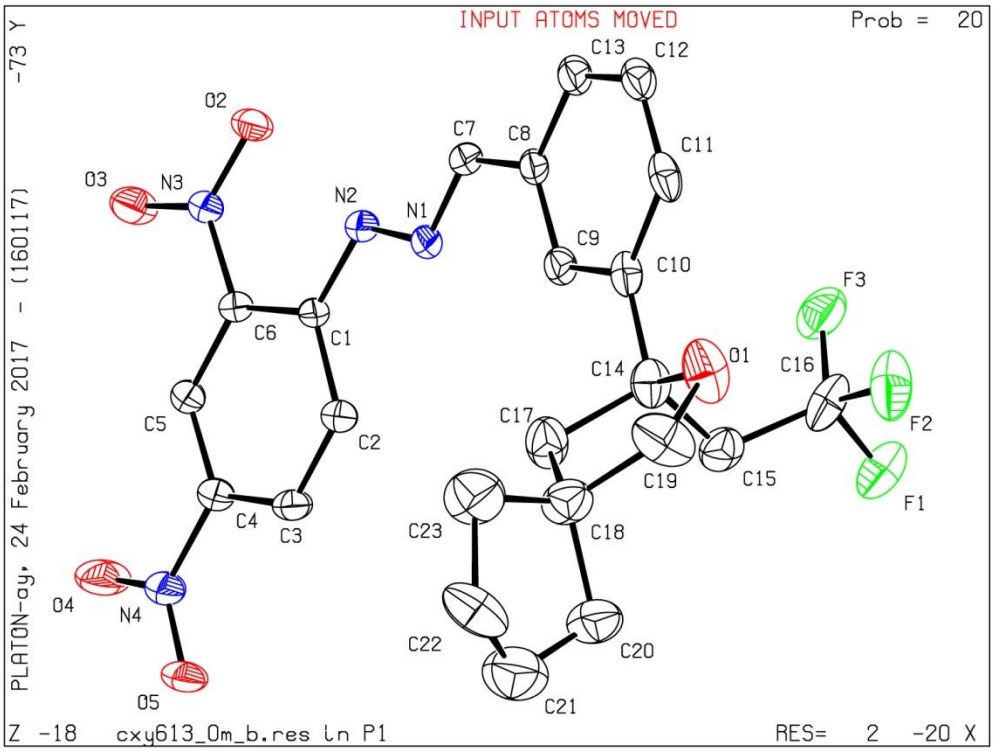


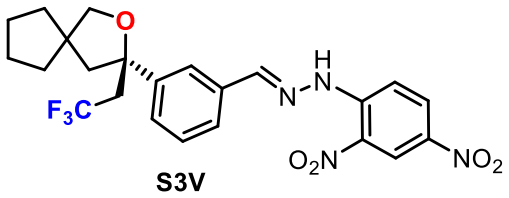
Catalyst	yield (%)
$Cu(OAc)_2$/(*R*)-**A6**	NR
$Cu(OTf)_2$/(*R*)-**A6**	NR
(*R*)-**A6**	NR
$CuBH_4(PPh_3)_2$	trace

Scheme S2. a) Radical trapping experiments and b) control reactions



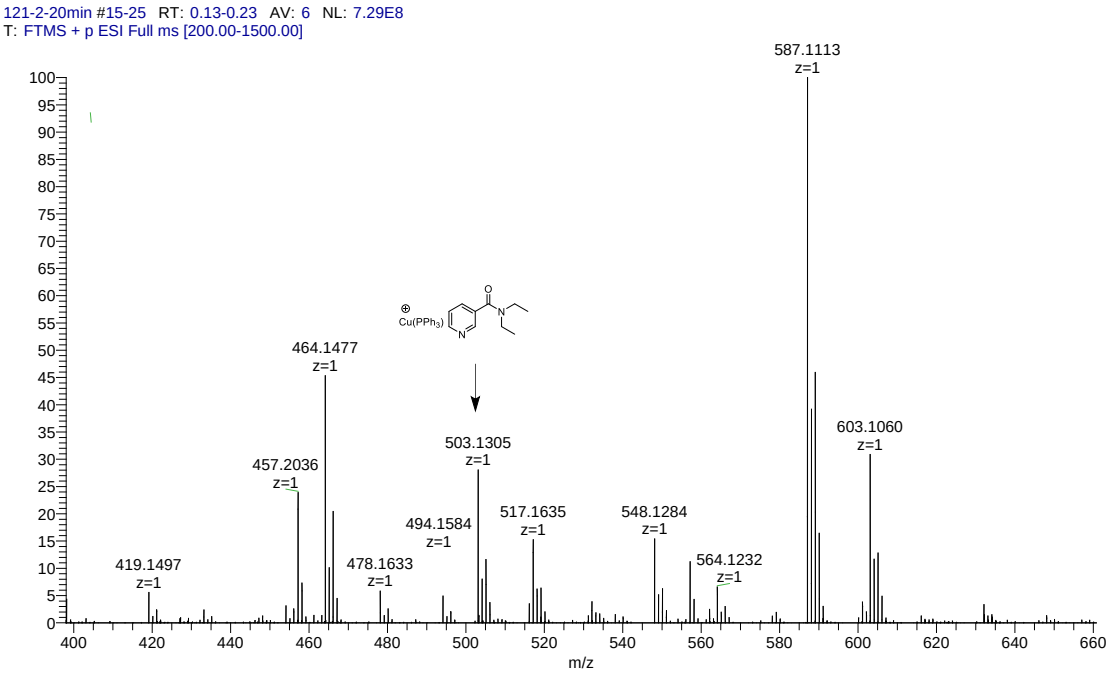
Scheme S3. Asymmetric radical oxytrifluoromethylation of substrates 1za and 1zb



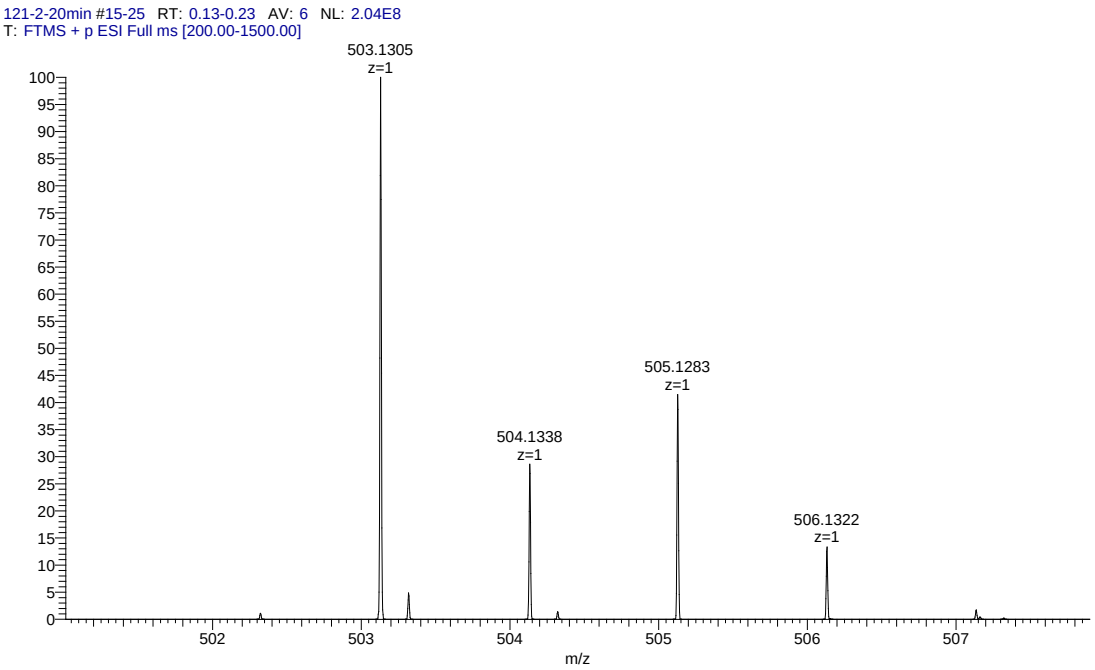


S3V

Figure S1. X-ray structure of chiral compound **S3V**


121-2-20min #15-25 RT: 0.13-0.23 AV: 6 NL: 7.29E8
T: FTMS + p ESI Full ms [200.00-1500.00]
587.1113
z=1
464.1477
z=1
503.1305
z=1
457.2036
z=1
603.1060
z=1
494.1584
z=1
517.1635
z=1
548.1284
z=1
419.1497
z=1
478.1633
z=1
564.1232
z=1
m/z

Spectrum **a**


121-2-20min #15-25 RT: 0.13-0.23 AV: 6 NL: 2.04E8
T: FTMS + p ESI Full ms [200.00-1500.00]
503.1305
z=1
505.1283
z=1
504.1338
z=1
506.1322
z=1
m/z

Spectrum **b**

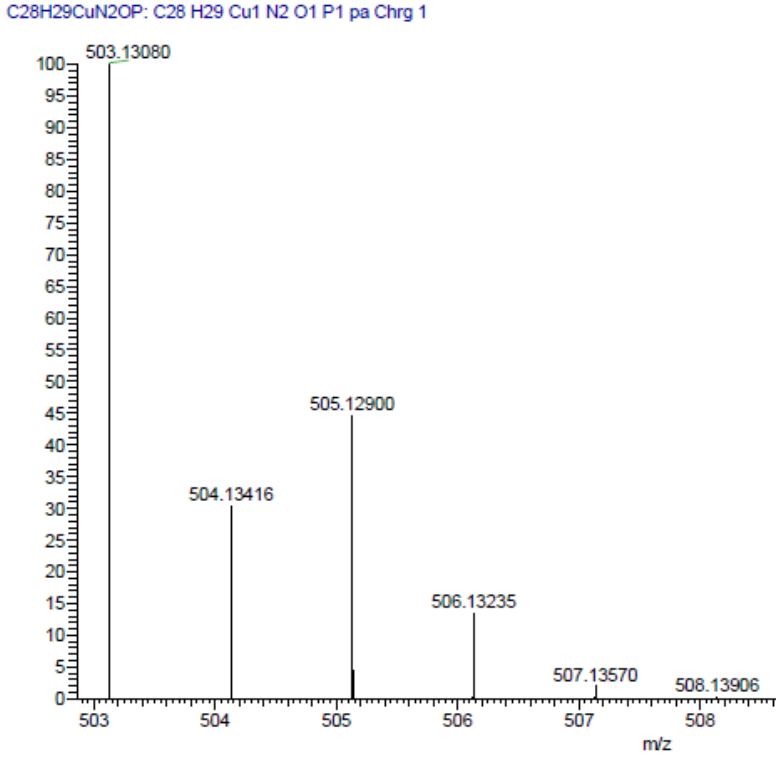


Figure S2. Electrospray ionization (ESI) mass spectroscopy (MS) analysis. A solution of **1a** (0.1 mmol), Togni reagent (1.0 equiv), $CuBH_4(PPh_3)_2$ (1.0 equiv), (*R*)-**A6** (1.0 equiv), and **P1** (1.0 equiv) in AcO*i*Pr (1.0 mL) was stirred for 20 min at room temperature. Then the reaction mixture was analyzed by ESI MS, showing a peak at *m/z* 503.1305 ascribed to the Cu(I)-**P1** complex. The observed isotopic distribution (spectrum **a**) well matches the calculated one (spectrum **b**).

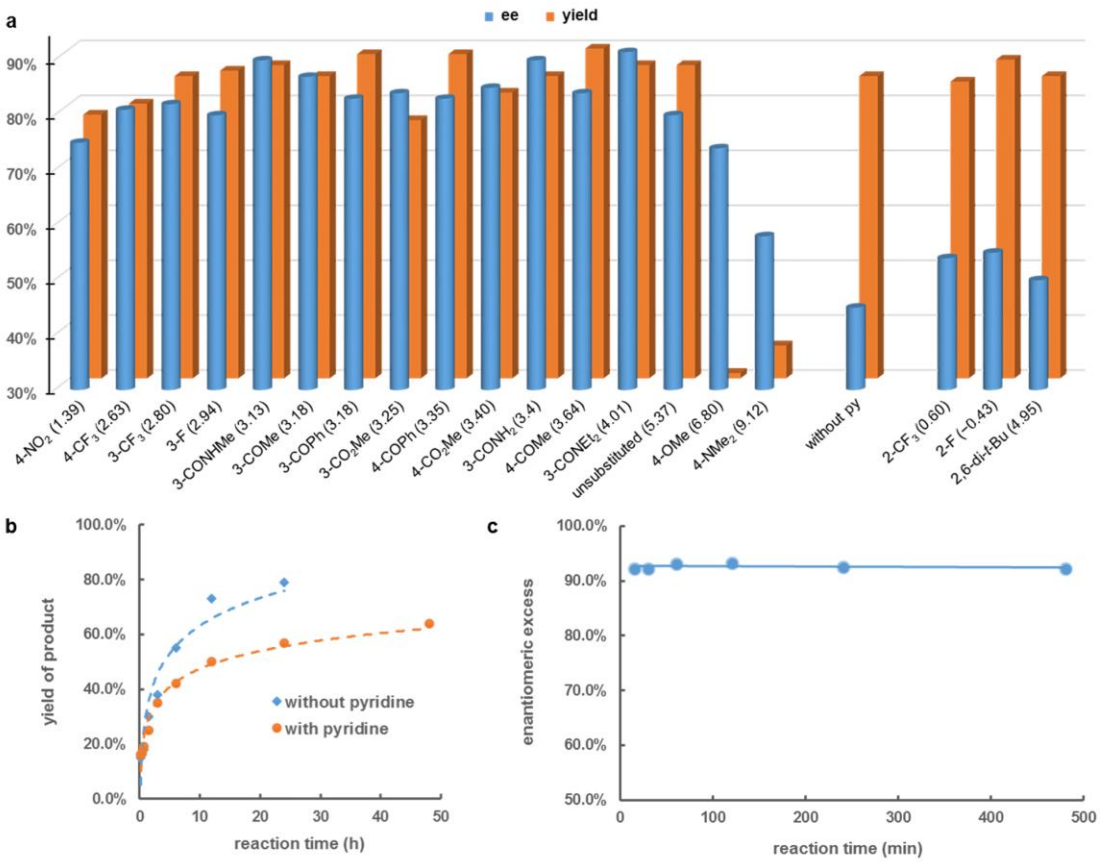
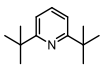
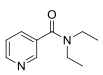


Figure S3. Mechanistic studies on the effect of pyridine. a) Substituent effect of pyridine on reaction performance. Numbers in parentheses are p*K*a values reported in literature or predicted by Advanced Chemistry Development (ACD/Labs) Software. b) Retarding effect of pyridine on reaction rate. c) Ee of products measured at different time points during reaction.

Table S1. References for p*K*a values of pyridine and its analogues

entry	additive	p*K*a
1		6.80, B. Uno, T. Kawakita, K. Kano, N. Okumura, M. Goto, T. Kubota, *Bull. Chem. Soc. Jpn.* **1994**, *67*, 2304.
2		9.12, I.-H. Um, S.-J. Hwang, M.-H. Baek, E. J. Park, *J. Org. Chem.* **2006**, *71*, 9191.
3		0.60, predicted, retrieved in Scifinder
4		2.80, predicted, retrieved in Scifinder
5		2.63, M. Taagepera, W. G. Henderson, R. T. C. Brownlee, J. L. Beauchamp, D. Holtz, R. W. Taft, *J. Am. Chem. Soc.* **1972**, *94*, 1369.
6		−0.43, I. T. Suydam, S. A. Strobel, *J. Am. Chem. Soc.* **2008**, *130*, 13639.
7		2.94, I. T. Suydam, S. A. Strobel, *J. Am. Chem. Soc.* **2008**, *130*, 13639.
8		4.95, H. P. Hopkins, Jr., D. V. Jahagirdar, P. S. Moulik, D. H. Aue, H. M. Webb, W. R. Davidson, M. D. Pedley, *J. Am. Chem. Soc.* **1984**, *106*, 4341.
9		1.39, M. Taagepera, W. G. Henderson, R. T. C. Brownlee, J. L. Beauchamp, D. Holtz, R. W. Taft, *J. Am. Chem. Soc.* **1972**, *94*, 1369.
10		3.25, E. Chrystiuk, A. Williams, *J. Am. Chem. Soc.* **1987**, *109*, 3040.
11		3.40, E. Chrystiuk, A. Williams, *J. Am. Chem. Soc.* **1987**, *109*, 3040.
12		3.64, E. Chrystiuk, A. Williams, *J. Am. Chem. Soc.* **1987**, *109*, 3040.
13		3.35, A. Fischer, W. J. Galloway, J. Vaughan, *J. Chem. Soc.* **1964**, 3591.
14		3.18, A. Bryson, *J. Am. Chem. Soc.* **1960**, *82*, 4871.
15		3.18, A. Fischer, W. J. Galloway, J. Vaughan, *J. Chem. Soc.* **1964**, 3591.
16		3.4, E. A. Castro, M. Cubillos, J. G. Santos, *J. Org. Chem.* **2004**, *69*, 4802.
17		3.13, predicted, retrieved in Scifinder
18		4.01, predicted, retrieved in Scifinder

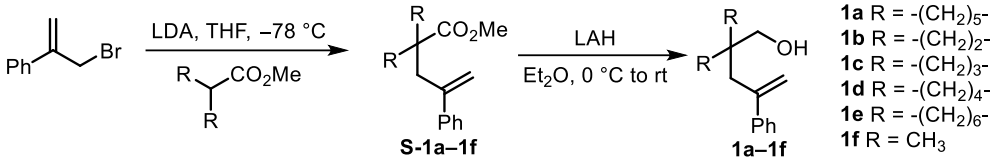
19		5.37, E. A. Castro, M. Cubillos, J. G. Santos, *J. Org. Chem.* **2004**, *69*, 4802.

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. $CuBH_4(PPh_3)_2$ was purchased from TCI®. Chiral phosphoric acid (**CPA**) was purchased from Daicel Chiral Technologies (China). Isopropyl acetate (AcO*i*Pr) was purchased from Acros® (Product Code: 180800010) and transferred under an argon atmosphere. 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). Visualization on TLC was achieved by use of UV light (254 nm) or iodine. NMR spectra were recorded on Bruker DRX-500 and DPX 400 spectrometer at 400 or 500 MHz for ^{1}H NMR, 100 or 125 MHz for ^{13}C NMR and 376 MHz for ^{19}F NMR with tetramethylsilane (TMS) as internal standard. The chemical shifts are expressed in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quarter; p, pentet, m, multiplet; br, broad), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). Mass spectrometric data were obtained using Bruker Apex IV RTMS. Enantiomeric excess (ee) was determined using Agilent high-performance liquid chromatography (HPLC) with a Hatachi detector (λ = 254 or 214 nm). 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.

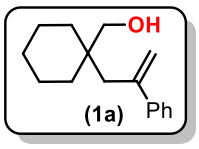
General Procedure for the Synthesis of Substrates

General synthesis of substrates 1a–1f



Synthesis of substrates **S-1a**–**1f**. Methyl carboxylates (6.32 mL, 50.0 mmol) was added to a solution of LDA [generated in situ from *n*-BuLi (27.0 mL in *n*-Hexane, 2.4 M, 65.0 mmol) and diisopropylamine (9.0 mL, 65.0 mmol) in THF (50.0 mL)] at –78 °C and the reaction mixture was stirred under the same conditions for 45 min. To the resulting solution was added (3-bromoprop-1-en-2-yl)benzene (11.0 g, 55.0 mmol). Upon completion, the solution was warmed to room temperature and stirred overnight. Then the reaction was quenched with sat. NH_4Cl and the organic solvent was removed under reduced pressure. Next, CH_2Cl_2 (40.0 mL) was added and the organic layer was separated. This organic layer was washed with water (3 × 40.0 mL), dried (Na_2SO_4) and concentrated. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 50/1) to give **S-1a**–**1f**.

Synthesis of substrates **1a**–**1f**. To a suspension of $LiAlH_4$ (3.04g, 80.0 mmol) in Et_2O (50.0 mL) at 0 °C was slowly added a solution of **S-1a**–**1f** (40.0 mmol) in Et_2O (20.0 mL). Then the reaction mixture was warmed to room temperature and stirred for 2 h. Next, it was quenched by slow, portionwise addition of water (4.0 mL) in Na_2SO_4 (32.0 g) at 0 °C. Upon completion, the mixture was warmed to room temperature, stirred for additional 30 minutes, filtered, and concentrated. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 10/1) to give **1a**–**1f**.

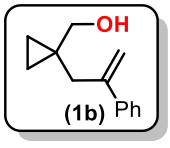


(1-(2-phenylallyl)cyclohexyl)methanol (1a)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.37 (m, 2H), 7.30–7.27 (m, 2H), 7.24–7.21 (m, 1H), 5.23 (d, *J* = 2.0 Hz, 1H), 5.10 (s, 1H), 3.20 (s, 2H), 2.58 (s, 2H), 1.42–1.35 (m, 6H), 1.24–1.22 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 143.6, 128.2, 127.2, 126.2, 116.9, 67.3, 40.7, 38.6, 32.7, 26.2, 21.5.

HRMS (ESI) m/z calcd. for $C_{16}H_{23}O$ $[M + H]^+$ 231.1743, found 231.1742.

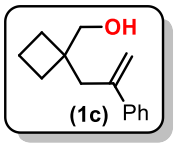


(1-(2-phenylallyl)cyclopropyl)methanol (1b)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.39 (m, 2H), 7.34–7.29 (m, 2H), 7.28–7.24 (m, 1H), 5.31 (d, *J* = 1.6 Hz, 1H), 5.18 (s, 1H), 3.37 (s, 2H), 2.66 (s, 2H), 0.37 (d, *J* = 2.2 Hz, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 142.2, 128.2, 127.4, 126.3, 114.6, 68.7, 38.9, 21.5, 9.9.

HRMS (ESI) m/z calcd. for $C_{13}H_{17}O$ $[M + H]^+$ 189.1274, found 189.1273.

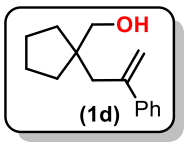


(1-(2-phenylallyl)cyclobutyl)methanol (1c)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.37 (m, 2H), 7.33–7.29 (m, 2H), 7.27–7.23 (m, 1H), 5.25 (d, *J* = 1.8 Hz, 1H), 5.09-5.08 (m, 1H), 3.45 (d, *J* = 5.7 Hz, 2H), 2.73 (d, *J* = 0.7 Hz, 2H), 1.82–1.61 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 142.8, 128.3, 127.3, 126.4, 115.6, 67.6, 43.5, 42.2, 28.5, 15.4.

HRMS (ESI) m/z calcd. for $C_{14}H_{19}O$ $[M + H]^+$ 203.1430, found 203.1431.

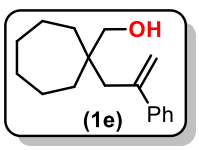


(1-(2-phenylallyl)cyclopentyl)methanol (1d)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.9 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 2H), 7.27–7.24 (m, 1H), 5.22 (s, 1H), 5.12 (s, 1H), 3.20 (d, *J* = 5.3 Hz, 2H), 2.63 (s, 2H), 1.58–1.49 (m, 4H), 1.37–1.25 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 143.3, 128.3, 127.3, 126.4, 116.6, 67.7, 48.4, 41.9, 34.4, 24.4.

HRMS (ESI) m/z calcd. for $C_{15}H_{21}O$ $[M + H]^+$ 217.1587, found 217.1586.

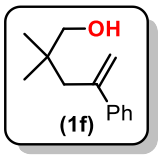


(1-(2-phenylallyl)cycloheptyl)methanol (1e)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.37 (m, 2H), 7.34–7.29 (m, 2H), 7.28–7.23 (m, 1H), 5.24 (d, *J* = 2.0 Hz, 1H), 5.10–5.09 (m, 1H), 3.08 (d, *J* = 4.5 Hz, 2H), 2.52 (s, 2H), 1.46–1.26 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.2, 143.6, 128.4, 127.4, 126.3, 117.4, 68.0, 42.3, 42.1, 34.8, 31.3, 22.6.

HRMS (ESI) m/z calcd. for $C_{17}H_{25}O$ $[M + H]^+$ 245.1900, found 245.1899.



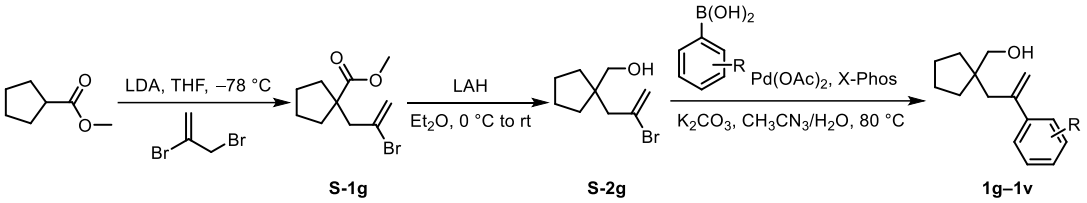
2,2-dimethyl-4-phenylpent-4-en-1-ol (1f)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.37 (m, 2H), 7.33–7.29 (m, 2H), 7.27–7.23 (m, 1H), 5.26 (d, *J* = 2.0 Hz, 1H), 5.08–5.07 (m, 1H), 3.17 (s, 2H), 2.53 (s, 2H), 0.79 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 143.4, 128.3, 127.3, 126.4, 116.8, 71.3, 43.7, 36.5, 24.6.

HRMS (ESI) m/z calcd. for $C_{13}H_{19}O$ $[M + H]^+$ 191.1430, found 191.1427.

General synthesis of substrates 1g–1v

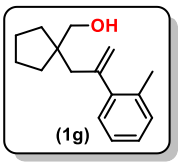


Synthesis of substrate **S-1g**. Methyl cyclopentanecarboxylate (6.32 mL, 50.0 mmol) was added to a solution of LDA [generated in situ from *n*-BuLi (27.0 mL in *n*-Hexane, 2.4 M, 65.0 mmol) and diisopropylamine (9.0 mL, 65.0 mmol) in THF (50.0 mL)] at –78 °C and the reaction mixture was stirred under the same conditions for 45 min. To the reaction solution was added 2,3-dibromoprop-1-ene (11.0 g, 55.0 mmol). Upon completion, the solution was warmed to room temperature and stirred overnight. Then the reaction was quenched with sat. NH_4Cl and the organic solvent was removed under reduced pressure. Next, CH_2Cl_2 (40.0 mL) was added and the organic layer was separated. This organic layer was washed with water (3 × 40.0 mL), dried (Na_2SO_4) and concentrated. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 100/1 to 50/1) to give **S-1g** (9.88 g, 80%).

Synthesis of substrate **S-2g**. To a suspension of $LiAlH_4$ (3.04g, 80.0 mmol) in Et_2O (50.0 mL) at 0 °C was slowly added a solution of **S-1g** (40.0 mmol) in Et_2O (20.0 mL). Then, the reaction mixture was warmed to room temperature and stirred for additional 2 h. Upon completion, the reaction mixture was quenched by slow, portionwise addition of water (4.0 mL) in Na_2SO_4 (32.0 g) at 0 °C. The resulting mixture was warmed to room temperature, stirred for additional 30 minutes, filtered, and concentrated under reduce pressure to afford **S-2g**, which was directly used in the next reaction without further purification.

General synthesis of substrates **1g**–**1v.** To a solution of **S-2g** (1.1 g, 5.0 mmol), arylboronic acids (7.5 mmol), K_2CO_3 (2.1 g, 15.0 mmol) and 2-dicyclohexylphosphino-2′,4′,6′-triisopropylbiphenyl (X-Phos, 190.7 mg, 0.4 mmol) in CH_3CN/H_2O (6.0 mL/2.0 mL) was added $Pd(OAc)_2$ (45.0 mg 0.2 mmol). The flask was evacuated and then backfilled with argon three times. Upon completion, the reaction mixture was stirred at 80 °C for 12 h under argon atmosphere. After cooled to room temperature, the mixture was extracted with CH_2Cl_2. The combined organic

layer was washed with brine, dried (Na_2SO_4) and concentrated *in vacuo* to afford a crude product, which was purified by flash chromatography to give **1g**–**1v** as a liquid.

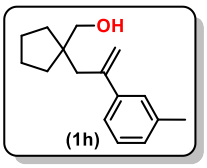


(1-(2-(o-tolyl)allyl)cyclopentyl)methanol (1g)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19–7.12 (m, 4H), 5.27–5.26 (m, 1H), 5.00 (d, *J* = 2.2 Hz, 1H), 3.22 (d, *J* = 5.8 Hz, 2H), 2.56 (s, 2H), 2.36 (s, 3H), 1.61–1.49 (m, 4H), 1.36–1.31 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 143.4, 134.5, 130.6, 128.2, 126.9, 125.6, 118.3, 67.8, 48.4, 44.2, 34.8, 24.7, 20.3.

HRMS (ESI) m/z calcd. for $C_{16}H_{23}O$ $[M + H]^+$ 231.1743, found 231.1738.

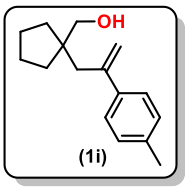


(1-(2-(m-tolyl)allyl)cyclopentyl)methanol (1h)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22–7.18 (m, 3H), 7.08 (d, *J* = 6.9 Hz, 1H), 5.21 (s, 1H), 5.10 (s, 1H), 3.20 (d, *J* = 5.9 Hz, 2H), 2.62 (s, 2H), 2.35 (s, 3H), 1.59–1.51 (m, 4H), 1.38–1.30 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 147.9, 143.3, 137.9, 128.3, 128.1, 127.1, 123.4, 116.4, 67.8, 48.5, 42.0, 34.5, 24.5, 21.5.

HRMS (ESI) m/z calcd. for $C_{16}H_{23}O$ $[M + H]^+$ 231.1743, found 231.1742.

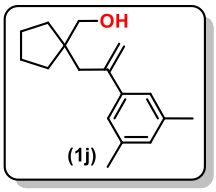


(1-(2-(p-tolyl)allyl)cyclopentyl)methanol (1i)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.8 Hz, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 5.20 (s, 1H), 5.07 (s, 1H), 3.20 (d, *J* = 5.9 Hz, 2H), 2.61 (s, 2H), 2.34 (s, 3H), 1.60–1.49 (m, 4H), 1.38–1.28 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 147.6, 140.4, 137.1, 129.1, 126.2, 115.9, 67.8, 48.5, 41.9, 34.5, 24.4, 21.1.

HRMS (ESI) m/z calcd. for $C_{16}H_{23}O$ $[M + H]^+$ 231.1743, found 231.1738.

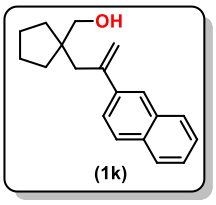


(1-(2-(3,5-dimethylphenyl)allyl)cyclopentyl)methanol (1j)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.00 (s, 2H), 6.91 (s, 1H), 5.19 (d, *J* = 2.0 Hz, 1H), 5.07–5.06 (m, 1H), 3.19 (d, *J* = 6.2 Hz, 2H), 2.60 (s, 2H), 2.31 (s, 6H), 1.59–1.49 (m, 4H), 1.37–1.32 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 143.4, 137.9, 129.1, 124.2, 116.1, 67.8, 48.6, 42.1, 34.5, 24.5, 21.3.

HRMS (ESI) m/z calcd. for $C_{17}H_{25}O$ $[M + H]^+$ 245.1900, found 245.1894.

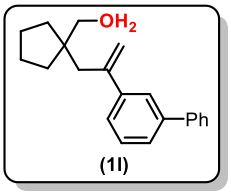


(1-(2-(naphthalen-2-yl)allyl)cyclopentyl)methanol (1k)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84–7.79 (m, 4H), 7.55 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.49–7.43 (m, 2H), 5.37 (d, *J* = 1.8 Hz, 1H), 5.23–5.22 (m, 1H), 3.24 (d, *J* = 6.0 Hz, 2H), 2.76 (s, 2H), 1.57–1.49 (m, 4H), 1.43–1.29 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 147.7, 140.7, 133.3, 132.7, 128.1, 127.9, 127.6, 126.2, 125.8, 125.0, 124.9, 117.2, 67.8, 48.5, 41.9, 34.5, 24.5.

HRMS (ESI) m/z calcd. for $C_{19}H_{23}O$ $[M + H]^+$ 267.1743, found 267.1735.

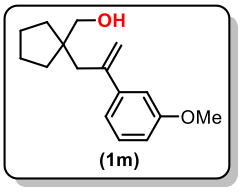


(1-(2-([1,1'-biphenyl]-3-yl)allyl)cyclopentyl)methanol (1l)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61–7.60 (m, 3H), 7.50 (d, *J* = 6.8 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.41–7.34 (m, 3H), 5.30 (s, 1H), 5.17 (s, 1H), 3.25 (d, *J* = 5.8 Hz, 2H), 2.69 (s, 2H), 1.64–1.53 (m, 4H), 1.40–1.32 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 147.8, 143.8, 141.3, 141.0, 128.8, 127.4, 127.2, 126.2, 125.3(4), 125.3(1), 116.9, 67.8, 48.5, 42.0, 34.5, 24.5.

HRMS (ESI) m/z calcd. for $C_{21}H_{25}O$ $[M + H]^+$ 293.1900, found 293.1898.

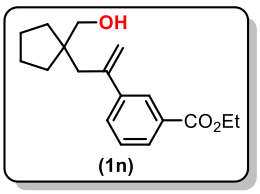


(1-(2-(3-methoxyphenyl)allyl)cyclopentyl)methanol (1m)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26–7.22 (m, 1H), 6.98 (d, *J* = 7.7 Hz, 1H), 6.93–6.92 (m, 1H), 6.82 (dd, *J* = 8.1, 2.3 Hz, 1H), 5.24 (d, *J* = 1.9 Hz, 1H), 5.12–5.11 (m, 1H), 3.82 (s, 3H), 3.21 (d, *J* = 6.2 Hz, 2H), 2.62 (s, 2H), 1.59–1.49 (m, 4H), 1.39–1.30 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 147.6, 144.9, 129.4, 118.8, 116.7, 112.5, 112.4, 67.8, 55.2, 48.5, 42.0, 34.4, 24.5.

HRMS (ESI) m/z calcd. for $C_{16}H_{23}O_2$ $[M + H]^+$ 247.1693, found 247.1685.

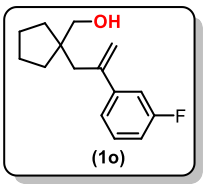


ethyl 3-(3-(1-(hydroxymethyl)cyclopentyl)prop-1-en-2-yl)benzoate (1n)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.94 (d, *J* = 7.7 Hz, 1H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 1H), 5.30 (s, 1H), 5.19 (s, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 3.24 (d, *J* = 5.7 Hz, 2H), 2.68 (s, 2H), 1.59–1.49 (m, 4H), 1.41 (t, *J* = 7.1 Hz, 3H), 1.33-1.31 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 166.6, 146.9, 143.6, 130.8, 130.5, 128.4, 128.3, 127.5, 117.6, 67.6, 61.1, 48.3, 41.7, 34.4, 24.4, 14.3.

HRMS (ESI) m/z calcd. for $C_{18}H_{25}O_3$ $[M + H]^+$ 289.1798, found 289.1789.



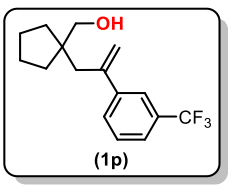
(1-(2-(3-fluorophenyl)allyl)cyclopentyl)methanol (1o)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30–7.26 (m, 1H), 7.17 (d, *J* = 7.7 Hz, 1H), 7.09 (d, *J* = 10.3 Hz, 1H), 6.97–6.94 (m, 1H), 5.27 (s, 1H), 5.16 (s, 1H), 3.24 (d, *J* = 5.6 Hz, 2H), 2.62 (s, 2H), 1.58–1.53 (m, 4H), 1.34–1.31 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 162.7 (d, *J* = 245.5 Hz), 146.6, 145.7 (d, *J* = 7.3 Hz), 129.7 (d, *J* = 8.5 Hz), 122.0 (d, *J* = 2.6 Hz), 117.5, 114.1 (d, *J* = 21.0 Hz), 113.4 (d, *J* = 21.5 Hz), 67.7, 48.3, 41.7, 34.3, 24.4.

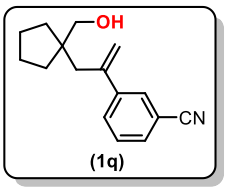
^{19}F NMR (376 MHz, $CDCl_3$) δ –113.3 (ddd, *J* = 10.3, 8.5, 6.0 Hz, 1F).

HRMS (ESI) m/z calcd. for $C_{15}H_{20}FO$ $[M + H]^+$ 235.1493, found 235.1491.

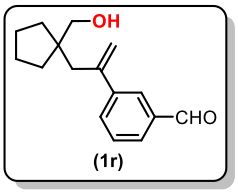


(1-(2-(3-(trifluoromethyl)phenyl)allyl)cyclopentyl)methanol (1p)

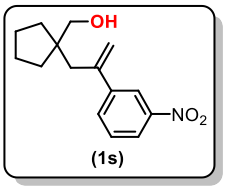
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 5.30 (d, *J* = 1.6 Hz, 1H), 5.21 (s, 1H), 3.24 (s, 2H), 2.67 (s, 2H), 1.59–1.49 (m, 4H), 1.33–1.29 (m, 4H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.4, 144.1, 130.6 (d, *J* = 3.5 Hz), 129.7, 128.7, 124.1 (d, *J* = 270.0 Hz), 123.9 (q, *J* = 3.8 Hz), 123.2 (q, *J* = 3.8 Hz), 118.1, 67.5, 48.2, 41.4, 34.3, 24.4.
^{19}F NMR (376 MHz, $CDCl_3$) δ –62.6 (s, 3F)
HRMS (ESI) m/z calcd. for $C_{16}H_{20}F_3O$ $[M + H]^+$ 285.1461, found 285.1452.



3-(3-(1-(hydroxymethyl)cyclopentyl)prop-1-en-2-yl)benzonitrile (1q)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (t, *J* = 1.5 Hz, 1H), 7.65–7.62 (m, 1H), 7.54 (dt, *J* = 7.7, 1.3 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 5.29 (d, *J* = 1.5 Hz, 1H), 5.23 (s, 1H), 3.24 (s, 2H), 2.65 (s, 2H), 1.59–1.51 (m, 4H), 1.32–1.27 (m, 4H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 145.7, 144.5, 130.8, 130.6, 130.1, 129.0, 118.8, 118.7, 112.2, 67.3, 48.1, 41.2, 34.2, 24.4.
HRMS (ESI) m/z calcd. for $C_{16}H_{20}NO$ $[M + H]^+$ 242.1539, found 242.1532.

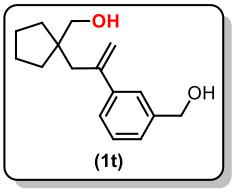


3-(3-(1-(hydroxymethyl)cyclopentyl)prop-1-en-2-yl)benzaldehyde (1r)
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.02 (s, 1H), 7.91 (t, *J* = 1.5 Hz, 1H), 7.79–7.76 (m, 1H), 7.69–7.66 (m, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 5.33 (d, *J* = 1.6 Hz, 1H), 5.22 (s, 1H), 3.25 (s, 2H), 2.70 (s, 2H), 1.60–1.49 (m, 4H), 1.33–1.29 (m, 4H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 192.4, 146.4, 144.3, 136.3, 132.5, 128.9, 128.6, 127.5, 118.0, 67.5, 48.2, 41.4, 34.3, 24.4.
HRMS (ESI) m/z calcd. for $C_{16}H_{21}O_2$ $[M + H]^+$ 245.1536, found 245.1529.

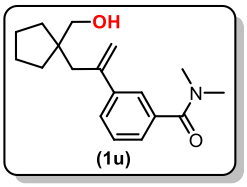


(1-(2-(3-nitrophenyl)allyl)cyclopentyl)methanol (1s)
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (t, *J* = 2.0 Hz, 1H), 8.13–8.10 (m, 1H), 7.75–7.73 (m, 1H), 7.49 (t, *J* = 8.0 Hz, 1H), 5.37 (d, *J* = 1.4 Hz, 1H), 5.28 (s, 1H), 3.26 (s, 2H), 2.70 (s, 2H), 1.60–1.51 (m, 4H), 1.34–1.29 (m, 4H).

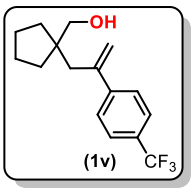
^{13}C NMR (100 MHz, $CDCl_3$) δ 148.2, 145.6, 145.0, 132.5, 129.1, 122.0, 121.3, 119.0, 67.41, 48.3, 41.3, 34.3, 24.4.
HRMS (ESI) m/z calcd. for $C_{15}H_{20}NO_3$ $[M + H]^+$ 262.1438, found 262.1433.



(3-(3-(1-(hydroxymethyl)cyclopentyl)prop-1-en-2-yl)phenyl)methanol (1t)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.32–7.31 (m, 2H), 7.28–7.25 (m, 1H), 5.25 (d, *J* = 1.9 Hz, 1H), 5.13–5.12 (m, 1H), 4.69 (d, *J* = 2.2 Hz, 2H), 3.17 (s, 2H), 2.65 (s, 2H), 1.98 (br s, 1H), 1.59–1.51 (m, 4H), 1.38–1.30 (m, 4H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 143.6, 141.0, 128.6, 126.0, 125.7, 125.0, 116.7, 67.7, 65.2, 48.4, 41.8, 34.5, 24.5.
HRMS (ESI) m/z calcd. for $C_{16}H_{23}O_2$ $[M + H]^+$ 247.1693, found 247.1690.

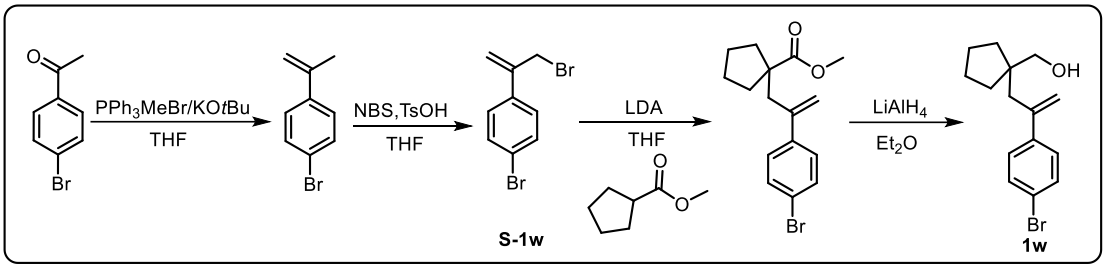


3-(3-(1-(hydroxymethyl)cyclopentyl)prop-1-en-2-yl)-N,N-dimethylbenzamide (1u)
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46–7.42 (m, 2H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.29 (dt, *J* = 7.5, 1.3 Hz, 1H), 5.26 (d, *J* = 1.8 Hz, 1H), 5.15 (s, 1H), 3.19 (s, 2H), 3.11 (s, 3H), 2.98 (s, 3H), 2.65 (s, 2H), 1.57–1.49 (m, 4H), 1.33–1.29 (m, 4H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 147.0, 143.4, 136.2, 128.3, 127.5, 125.7, 125.1, 117.3, 67.5, 48.2, 41.5, 39.5, 35.3, 34.3, 24.4.
HRMS (ESI) m/z calcd. for $C_{18}H_{26}O_2$ $[M + H]^+$ 288.1958, found 288.1953.



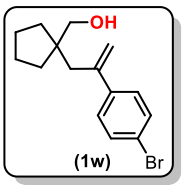
(1-(2-(4-(trifluoromethyl)phenyl)allyl)cyclopentyl)methanol (1v)
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.3 Hz, 2H), 7.50 (d, *J* = 8.2 Hz, 2H), 5.30 (d, *J* = 1.7 Hz, 1H), 5.22 (s, 1H), 3.24 (d, *J* = 5.4 Hz, 2H), 2.67 (s, 2H), 1.58–1.51 (m, 4H), 1.33–1.29 (m, 4H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.9, 146.6, 129.3 (q, *J* = 32.1 Hz), 126.7, 125.2 (q, *J* = 3.7 Hz), 124.2 (q, *J* = 271.4 Hz), 118.5, 67.6, 48.3, 41.5, 34.3, 24.4.
^{19}F NMR (376 MHz, $CDCl_3$) δ –62.4 (s, 3F).
HRMS (ESI) m/z calcd. for $C_{16}H_{20}F_3O$ $[M + H]^+$ 285.1461, found 285.1454.

Synthesis of substrate 1w



Compound **S-1w** was synthesized according to the procedures previously reported by Liu.[1]

Substrate **1w** was synthesized according to the same procedures for the synthesis of substrates **1a**–**1f**.



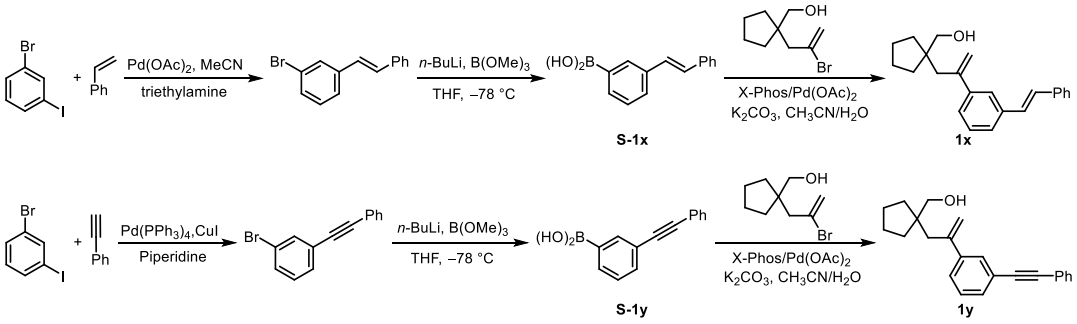
(1-(2-(4-bromophenyl)allyl)cyclopentyl)methanol (1w)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.5 Hz, 2H), 7.26 (d, *J* = 8.5 Hz, 2H), 5.23 (d, *J* = 1.7 Hz, 1H), 5.13 (s, 1H), 3.22 (s, 2H), 2.61 (s, 2H), 1.57–1.50 (m, 4H), 1.33–1.30 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.7, 142.2, 131.4, 128.1, 121.2, 117.2, 67.7, 48.3, 41.6, 34.3, 24.4.

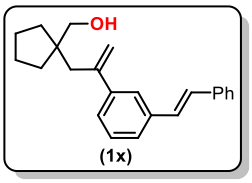
HRMS (ESI) m/z calcd. for $C_{15}H_{20}BrO$ $[M + H]^+$ 295.0692, found 295.0685.

Synthesis of substrates 1x and 1y



Substrates **S-1x** and **S-1y** were synthesized according to a literature report.[2]

Substrates **1x** and **1y** were synthesized according to the same procedures for the synthesis of substrates **1g**–**1v.**

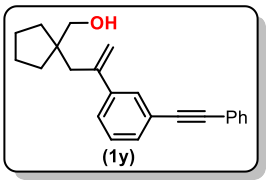


(*E*)-(1-(2-(3-styrylphenyl)allyl)cyclopentyl)methanol (1x)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.50 (m, 3H), 7.42–7.41 (m, 1H), 7.35 (t, *J* = 7.5 Hz, 2H), 7.29–7.21 (m, 3H), 7.11 (s, 2H), 5.27 (d, *J* = 1.7 Hz, 1H), 5.14 (s, 1H), 3.22 (s, 2H), 2.66 (s, 2H), 1.57–1.51 (m, 4H), 1.36–1.33 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 143.7, 137.3, 137.1, 128.9, 128.6(1), 128.5(9), 128.5, 127.6, 126.5, 125.7, 125.3, 124.7, 116.8, 67.7, 48.4, 41.9, 34.4, 24.4.

HRMS (ESI) m/z calcd. for $C_{23}H_{27}O$ $[M + H]^+$ 319.2056, found 319.2050.



(1-(2-(3-(phenylethynyl)phenyl)allyl)cyclopentyl)methanol (1y)

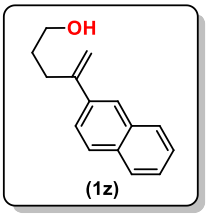
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.53 (m, 3H), 7.45–7.42 (m, 1H), 7.37–7.28 (m, 5H), 5.27 (d, *J* = 1.8 Hz, 1H), 5.16 (s, 1H), 3.25 (s, 2H), 2.65 (s, 2H), 1.61–1.51 (m, 4H), 1.36–1.33 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 143.6, 131.6, 130.4, 129.6, 128.3(3), 128.2(9), 126.4, 123.3, 123.1, 117.3, 89.4, 89.3, 67.7, 48.4, 41.8, 34.4, 24.5.

HRMS (ESI) m/z calcd. for $C_{23}H_{25}O$ $[M + H]^+$ 317.1900, found 317.1893.

Synthesis of substrate 1z

Substrate **1z** was synthesized according to reported procedures.[3]



4-(naphthalen-2-yl)pent-4-en-1-ol (1z)[4]

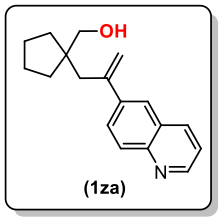
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82–7.77 (m, 4H), 7.56 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.47–7.41 (m, 2H), 5.43 (s, 1H), 5.18 (s, 1H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.70 (t, *J* = 7.6 Hz, 2H), 1.78–1.72 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 147.7, 138.1, 133.3, 132.7, 128.1, 127.8, 127.5, 126.1, 125.8, 124.7, 124.6, 113.1, 62.3, 31.5, 31.1.

HRMS (ESI) m/z calcd. for $C_{15}H_{17}O$ $[M + H]^+$ 213.1274, found 213.1271.

Synthesis of substrate 1za

Substrate **1za** was synthesized according to the same procedures for the synthesis of substrates **1g**–**1v.**



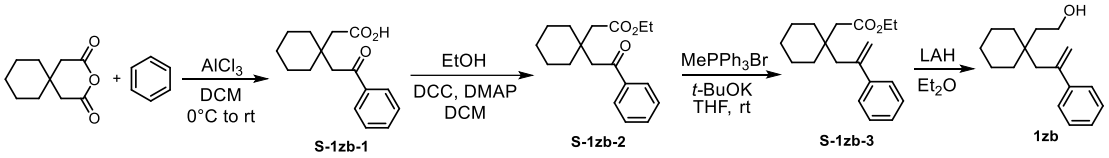
(1-(2-(quinolin-6-yl)allyl)cyclopentyl)methanol (1za)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.86 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.14 (d, *J* = 8.3 Hz, 1H), 8.04 (d, *J* = 8.7 Hz, 1H), 7.82–7.78 (m, 2H), 7.39 (dd, *J* = 8.3, 4.2 Hz, 1H), 5.40 (d, *J* = 1.7 Hz, 1H), 5.27 (d, *J* = 0.4 Hz, 1H), 3.26 (d, *J* = 2.3 Hz, 2H), 2.77 (s, 2H), 1.89 (br s, 1H), 1.60–1.48 (m, 4H), 1.37–1.32 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 150.2, 147.7, 147.0, 141.5, 136.2, 129.3, 128.8, 128.1, 124.6, 121.4, 118.1, 67.7, 48.4, 41.7, 34.4, 24.5.

HRMS (ESI) m/z calcd. for $C_{18}H_{22}NO$ $[M + H]^+$ 268.1696, found 268.1693.

Synthesis of substrate 1zb

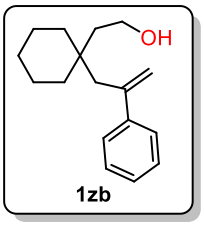


To a solution of 3-oxaspiro[5.5]undecane-2,4-dione (1.82 g, 10.0 mmol) and benzene (0.98 mL, 11.0 mmol) in dry DCM (20.0 mL) at 0 °C was slowly added $AlCl_3$ (2.93 g, 22.0 mmol). Upon completion, the mixture was warmed to room temperature and stirred for additional 2 h. Then the reaction was quenched by slow, sequential addition of 1 M HCl to adjust the pH to 1. The reaction mixture was next extracted three times using DCM. The collected organic layer was washed with saturated sodium chloride, dried with Na_2SO_4, and concentrated. The residue was purified by silica gel column chromatography to give **S-1zb-1** as a yellow oil (2.28 g, 88 %).

To a solution of **S-1zb-1** (944.0 mg, 3.63 mmol) in dry DCM (10.0 mL) was added dicyclohexylcarbodiimide (823.8 mg, 4.0 mmol), DMAP(43.9 mg, 0.36 mmol), and EtOH (0.2 mL, 4.0 mmol) at 4 °C. Upon completion, the mixture was warmed to room temperature and stirred for additional 2 h. Then the reaction mixture was directly concentrated and the residue was purified by silica gel column chromatography to give **S-1zb-2** as a yellow oil (1.04 g, 99%).

To a slurry of methyltriphenylphosphonium bromide (1.87 g, 5.25 mmol) in dry THF (10.0 mL) at 0 °C was added *t*-BuOK (2.4 M in THF, 2.2 mL, 5.25 mmol). The resulting solution was stirred at 0 °C for 1 h, at which time **S-1zb-2** (1.01 g, 3.5 mmol) in THF (5.0 mL) was added via syringe. The reaction mixture was stirred at 0 °C for 1 h and then at room temperature overnight. The reaction was quenched by the addition of saturated aq. NH_4Cl and the resulting salts were dissolved by adding a minimum amount of H_2O. The THF was removed *in vacuo* and the aqueous layer was extracted with DCM for three times. The combined organic extracts were dried over Na_2SO_4 and concentrated *in vacuo*. The crude product was purified by flash chromatography to afford **S-1zb-3** (743.0 mg, 74%) as a colorless oil.

To a suspension of $LiAlH_4$ (197.6 mg, 5.2 mmol) in dry Et_2O (10.0 mL) at 0 °C was slowly added a solution of **S-1zb-3** (740.0 mg, 2.6 mmol) in dry Et_2O (10.0 mL). Upon completion, the mixture was warmed to room temperature and stirred for additional 2 h. Then the reaction mixture was quenched by slow, sequential addition of water (0.5 mL) in Na_2SO_4 (4.0 g). The reaction mixture was next warmed to room temperature, stirred for additional 30 minutes, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography to afford **1zb** as a colorless oil (609.0 mg, 96%).



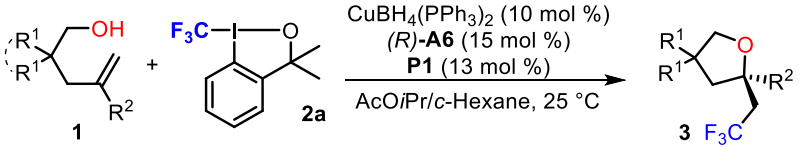
2-(1-(2-phenylallyl)cyclohexyl)ethan-1-ol (1zb)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.38 (m, 2H), 7.35–7.31 (m, 2H), 7.29–7.25 (m, 1H), 5.26 (d, *J* = 2.0 Hz, 1H), 5.09–5.08 (m, 1H), 3.56 (t, *J* = 8.0 Hz, 2H), 2.55 (s, 2H), 1.51–1.17 (m, 12H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 144.2, 128.1, 127.0, 126.6, 117.2, 59.0, 43.9, 38.6, 36.2, 36.1, 26.1, 21.6.

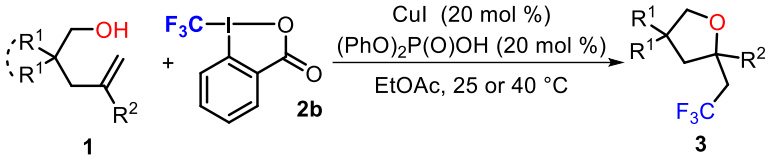
HRMS (ESI) m/z calcd. for $C_{17}H_{25}O$ $[M + H]^+$ 245.1900, found 245.1899.

General Procedure for Asymmetric Intramolecular Radical Oxytrifluoromethylation of Alkenes



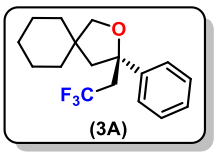
Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1** (0.2 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (12.0 mg, 0.02 mmol, 10 mol %), chiral phosphoric acid (*R*)-**A6** (18.0 mg, 0.03 mmol, 15 mol %), **P1** (4.6 mg, 0.026 mmol, 13 mol%), Togni's reagent **2a** (99.0 mg, 0.3 mmol, 1.5 equiv), HFIP (18.0 mg, 0.08 mmol, 40 mol%, whenever necessary) and AcO*i*Pr/*c*-Hexane (9:1, 2.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. Upon completion (monitored by LC-MS), 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 described above using substrate **1** (0.2 mmol, 1.0 equiv), Togni's reagent **2b** (95.4 mg, 0.3 mmol, 1.5 equiv) CuI (7.6 mg, 0.04 mmol, 20 mol %) and diphenyl phosphate (10.0 mg, 0.04 mmol, 20 mol %) at 25 or 40 °C in EtOAc (2.0 mL) for 12–24 h. 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.

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

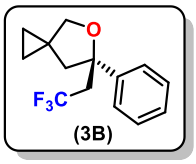


(*R*)-3-phenyl-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.5]decane (3A)

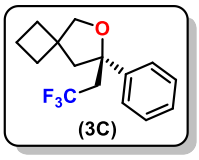
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 14.46 min, t_R (minor) = 16.93 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.36 (m, 2H), 7.35–7.27 (m, 2H), 7.24–7.18 (m, 1H), 3.77 (d, *J* = 8.7 Hz, 1H), 3.59 (d, *J* = 8.8 Hz, 1H), 2.65 (dd, *J* = 10.8, 0.9 Hz, 1H), 2.59 (dd, *J* = 10.7, 1.2 Hz, 1H), 2.33 (d, *J* = 13.0 Hz, 1H), 2.06 (d, *J* = 13.0 Hz, 1H), 1.54–1.48 (m, 2H), 1.47–1.41 (m, 2H), 1.36–1.23 (m, 4H), 1.15–1.12 (m, 2H).

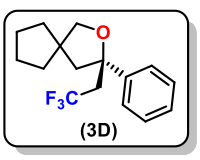
^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 128.1, 126.9, 125.3, 125.2 (q, *J* = 276.7 Hz), 83.0 (q, *J* = 2.0 Hz), 77.5, 51.1, 46.6 (q, *J* = 25.8 Hz), 44.3, 37.5, 36.0, 25.7, 23.9, 23.6.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{17}H_{22}F_3O$ $[M + H]^+$ 299.1617, found 299.1613.



(*R*)-6-phenyl-6-(2,2,2-trifluoroethyl)-5-oxaspiro[2.4]heptane (3B)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 10.24 min, t_R (minor) = 14.48 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42–7.40 (m, 2H), 7.36–7.33 (m, 2H), 7.28–7.25 (m, 1H), 3.90 (d, *J* = 8.0 Hz, 1H), 3.79 (d, *J* = 8.0 Hz, 1H), 2.84–2.70 (m, 2H), 2.34 (d, *J* = 12.4 Hz, 1H), 2.26 (d, *J* = 12.4 Hz, 1H), 0.69–0.62 (m, 2H), 0.44–0.37 (m, 2H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 128.1, 127.1, 125.4 (q, *J* = 276.6 Hz), 125.1, 83.8 (q, *J* = 2.0 Hz), 74.5, 47.1 (q, *J* = 1.1 Hz), 45.0 (q, *J* = 26.0 Hz), 21.6, 10.8, 10.3.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{14}H_{16}F_3O$ $[M + H]^+$ 257.1148, found 257.1137.

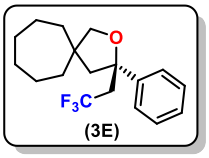


(*R*)-7-phenyl-7-(2,2,2-trifluoroethyl)-6-oxaspiro[3.4]octane (3C)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 8.14 min, t_R (minor) = 10.01 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.9 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.23 (t, *J* = 7.2 Hz, 1H), 4.01 (d, *J* = 8.5 Hz, 1H), 3.91 (d, *J* = 8.5 Hz, 1H), 2.71–2.56 (m, 2H), 2.48 (d, *J* = 12.7 Hz, 1H), 2.34 (d, *J* = 12.7 Hz, 1H), 2.10–2.04 (m, 2H), 1.80–1.74 (m, 2H), 1.70–1.69 (m, 2H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 128.0, 126.9, 125.3 (q, *J* = 276.8 Hz), 125.1, 83.1 (q, *J* = 2.0 Hz), 79.2, 51.8 (q, *J* = 0.8 Hz), 46.1, 45.6 (q, *J* = 25.9 Hz), 32.9, 32.3, 16.6.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{15}H_{18}F_3O$ $[M + H]^+$ 271.1304, found 271.1298.

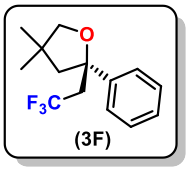


(*R*)-3-phenyl-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3D)

HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 7.81 min, t_R (minor) = 9.59 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.34 (m, 2H), 7.35–7.31 (m, 2H), 7.26–7.22 (m, 1H), 3.80 (d, *J* = 8.3 Hz, 1H), 3.66 (d, *J* = 8.3 Hz, 1H), 2.69 (d, *J* = 10.7 Hz, 1H), 2.64 (d, *J* = 10.7 Hz, 1H), 2.40 (d, *J* = 12.7 Hz, 1H), 2.30 (d, *J* = 12.7 Hz, 1H), 1.78–1.72 (m, 1H), 1.64–1.43 (m, 5H), 1.35–1.18 (m, 2H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 128.1, 126.9, 125.4 (q, *J* = 276.8 Hz), 125.3, 83.1 (q, *J* = 2.0 Hz), 78.7, 52.0 (q, *J* = 0.9 Hz), 51.0, 46.2 (q, *J* = 26.0 Hz), 38.0, 37.3, 24.6, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{16}H_{20}F_3O$ $[M + H]^+$ 285.1461, found 285.1455.

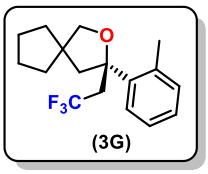


(*R*)-3-phenyl-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.6]undecane (3E)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 7.88 min, t_R (minor) = 9.24 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.7 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 1H), 3.73 (d, *J* = 8.6 Hz, 1H), 3.57 (d, *J* = 8.6 Hz, 1H), 2.67 (dd, *J* = 10.7, 3.1 Hz, 1H), 2.62 (dd, *J* = 10.7, 3.4 Hz, 1H), 2.36 (d, *J* = 13.0 Hz, 1H), 2.14 (d, *J* = 13.0 Hz, 1H), 1.77–1.73 (m, 1H), 1.64–1.41 (m, 6H), 1.38–1.26 (m, 5H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.1, 128.1, 126.9, 125.3, 125.2 (q, *J* = 276.9 Hz), 83.3 (q, *J* = 2.0 Hz), 78.9, 52.9 (q, *J* = 0.5 Hz), 47.7, 46.6 (q, *J* = 25.8 Hz), 40.3, 38.8, 29.2, 24.2, 23.8.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{18}H_{24}F_3O$ $[M + H]^+$ 313.1774, found 313.1766.



(*R*)-4,4-dimethyl-2-phenyl-2-(2,2,2-trifluoroethyl)tetrahydrofuran (3F)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 6.51 min, t_R (minor) = 7.28 min.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.40 (m, 2H), 7.35–7.31 (m, 2H), 7.26–7.22 (m, 1H), 3.71 (d, *J* = 8.5 Hz, 1H), 3.58 (d, *J* = 8.5 Hz, 1H), 2.71–2.67 (m, 1H), 2.65–2.60 (m, 1H), 2.30 (d, *J* = 12.9 Hz, 1H), 2.15 (d, *J* = 12.9 Hz, 1H), 1.17 (s, 3H), 0.87 (s, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 128.1, 126.9, 125.3, 125.2 (q, *J* = 276.8 Hz), 83.5 (q, *J* = 2.0 Hz), 79.6, 53.5 (q, *J* = 1.1 Hz), 46.6 (q, *J* = 25.8 Hz), 40.2, 27.8, 26.7.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.7 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{14}H_{18}F_3O$ $[M + H]^+$ 259.1304, found 259.1295.



(*R*)-3-(*o*-tolyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3G)

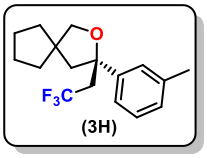
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 16.76 min, t_R (minor) = 15.82 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.0 Hz, 1H), 7.20–7.15 (m, 3H), 3.79 (d, *J* = 8.3 Hz, 1H), 3.60 (d, *J* = 8.4 Hz, 1H), 2.88–2.67 (m, 2H), 2.37 (s, 3H), 2.34 (s, 2H), 1.85–1.82 (m, 1H), 1.70–1.48 (m, 5H), 1.44–1.38 (m, 1H), 1.36–1.30 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 143.0, 133.4, 131.9, 127.1, 125.7, 125.5, 125.4 (q, *J* = 277.0 Hz), 83.0, 77.8, 52.8, 51.2, 43.9 (q, *J* = 25.6 Hz), 38.2, 36.4, 24.8, 24.6, 21.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.7 (t, *J* = 10.8 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{17}H_{22}F_3O$ $[M + H]^+$ 299.1617, found 299.1610.



(*R*)-3-(m-tolyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3H)

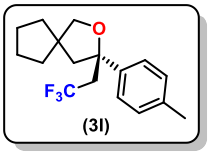
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 6.05 min, t_R (minor) = 7.33 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22–7.17 (m, 3H), 7.05 (d, *J* = 7.0 Hz, 1H), 3.79 (d, *J* = 8.3 Hz, 1H), 3.66 (d, *J* = 8.3 Hz, 1H), 2.68 (d, *J* = 10.7 Hz, 1H), 2.63 (d, *J* = 10.7 Hz, 1H), 2.38 (d, *J* = 12.9 Hz, 1H), 2.36 (s, 3H), 2.29 (d, *J* = 12.8 Hz, 1H), 1.78–1.73 (m, 1H), 1.63–1.46 (m, 5H), 1.36–1.31 (m, 1H), 1.27–1.21 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 145.3, 137.6, 127.9, 127.6, 125.9, 125.3 (q, *J* = 276.8 Hz), 122.3, 83.1 (q, *J* = 2.0 Hz), 78.7, 51.9, 51.1, 46.2 (q, *J* = 25.6 Hz), 38.0, 37.3, 24.6, 24.5, 21.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.7 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{17}H_{22}F_3O$ $[M + H]^+$ 299.1617, found 299.1611.

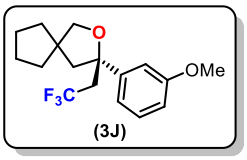


(*R*)-3-(p-tolyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3I)

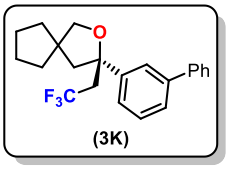
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 6.84 min, t_R (minor) = 10.45 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.5 Hz, 2H), 7.14 (d, *J* = 7.7 Hz, 2H), 3.78 (d, *J* = 8.3 Hz, 1H), 3.64 (d, *J* = 8.3 Hz, 1H), 2.67 (d, *J* = 10.7 Hz, 1H), 2.63 (d, *J* =

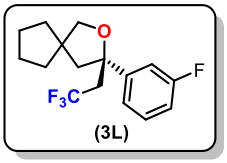
10.8 Hz, 1H), 2.38 (d, *J* = 12.7 Hz, 1H), 2.33 (s, 3H), 2.27 (d, *J* = 12.7 Hz, 1H), 1.76–1.71 (m, 1H), 1.64–1.45 (m, 5H), 1.35–1.29 (m, 1H), 1.26–1.20 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 142.2, 136.5, 128.8, 125.3 (q, *J* = 276.8 Hz), 125.2, 83.1 (q, *J* = 2.0 Hz), 78.6, 51.9, 51.1, 46.2 (q, *J* = 25.6 Hz), 38.1, 37.3, 24.6, 24.5, 21.0.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.8 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{17}H_{22}F_3O$ $[M + H]^+$ 299.1617, found 299.1610.



(*R*)-3-(3-methoxyphenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3J)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 4.58 min, t_R (minor) = 5.53 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (t, *J* = 7.5 Hz, 1H), 7.02 (s, 1H), 6.95 (d, *J* = 7.6 Hz, 1H), 6.79 (d, *J* = 7.9 Hz, 1H), 3.81–3.79 (m, 4H), 3.67 (d, *J* = 8.3 Hz, 1H), 2.68 (d, *J* = 10.6 Hz, 1H), 2.64 (d, *J* = 10.7 Hz, 1H), 2.39 (d, *J* = 12.7 Hz, 1H), 2.29 (d, *J* = 12.7 Hz, 1H), 1.75–1.73 (m, 1H), 1.62–1.48 (m, 5H), 1.37–1.33 (m, 1H), 1.27–1.22 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 159.4, 147.1, 129.1, 125.3 (q, *J* = 276.9 Hz), 117.8, 111.9, 111.3, 83.1 (q, *J* = 1.9 Hz), 78.8, 55.2, 52.0, 51.0, 46.2 (q, *J* = 25.9 Hz), 38.0, 37.3, 24.6, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{17}H_{22}F_3O_2$ $[M + H]^+$ 315.1566, found 315.1559.



(*R*)-3-([1,1'-biphenyl]-3-yl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3K)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 9.26 min, t_R (minor) = 11.07 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (s, 1H), 7.61 (d, *J* = 9.4 Hz, 2H), 7.49–7.33 (m, 6H), 3.82 (d, *J* = 8.3 Hz, 1H), 3.69 (d, *J* = 8.3 Hz, 1H), 2.74 (d, *J* = 10.7 Hz, 1H), 2.69 (d, *J* = 10.7 Hz, 1H), 2.45 (d, *J* = 12.8 Hz, 1H), 2.34 (d, *J* = 12.8 Hz, 1H), 1.80–1.73 (m, 1H), 1.64–1.46 (m, 5H), 1.38–1.32 (m, 1H), 1.28–1.22 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.8, 141.2, 141.0, 128.7, 128.5, 127.3, 127.2, 125.7, 125.3 (q, *J* = 276.9 Hz), 124.3, 124.1, 83.2 (q, *J* = 1.9 Hz), 78.8, 52.1, 51.1, 46.2 (q, *J* = 25.9 Hz), 38.0, 37.3, 24.7, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.2 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{22}H_{24}F_3O$ $[M + H]^+$ 361.1774, found 361.1766.



(*R*)-3-(3-fluorophenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3L)

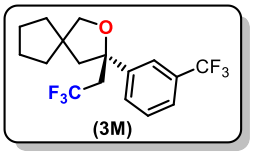
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 209 nm), t_R (major) = 8.11 min, t_R (minor) = 10.86 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32–7.27 (m, 1H), 7.18–7.14 (m, 2H), 6.94 (t, *J* = 8.3 Hz, 1H), 3.80 (d, *J* = 8.4 Hz, 1H), 3.67 (d, *J* = 8.4 Hz, 1H), 2.68 (d, *J* = 10.6 Hz, 1H), 2.63 (d, *J* = 10.6 Hz, 1H), 2.36 (d, *J* = 12.8 Hz, 1H), 2.29 (d, *J* = 12.8 Hz, 1H), 1.77–1.72 (m, 1H), 1.63–1.47 (m, 5H), 1.36–1.31 (m, 1H), 1.26–1.19 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 162.8 (d, *J* = 243.9 Hz), 148.1 (d, *J* = 6.6 Hz), 129.6 (d, *J* = 8.1 Hz), 125.1 (q, *J* = 276.9 Hz), 121.0 (d, *J* = 2.7 Hz), 113.8 (d, *J* = 21.0 Hz), 112.6 (d, *J* = 22.8 Hz), 82.8, 78.9, 52.2, 51.0, 46.1 (q, *J* = 26.0 Hz), 37.8, 37.3, 24.7, 24.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.6 Hz, 3F), –113.05 (ddd, *J* = 10.3, 8.3, 5.7 Hz, 1F).

HRMS (ESI) m/z calcd. for $C_{16}H_{19}F_4O$ $[M + H]^+$ 303.1367, found 303.1356.



(*R*)-3-(2,2,2-trifluoroethyl)-3-(3-(trifluoromethyl)phenyl)-2-oxaspiro[4.4]nonane (3M)

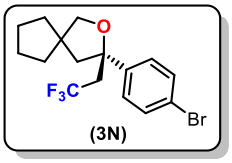
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 5.70 min, t_R (minor) = 6.46 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 1H), 3.82 (d, *J* = 8.4 Hz, 1H), 3.67 (d, *J* = 8.4 Hz, 1H), 2.72–2.65 (m, 2H), 2.39–2.33 (m, 2H), 1.77–1.73 (m, 1H), 1.64–1.46 (m, 5H), 1.35–1.29 (m, 1H), 1.22–1.16 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 146.4, 130.5 (q, *J* = 31.9 Hz), 128.8, 128.6, 125.1 (q, *J* = 276.9 Hz), 124.1 (q, *J* = 270.8 Hz), 123.9 (q, *J* = 3.8 Hz), 122.2 (q, *J* = 3.9 Hz), 82.8, (q, *J* = 1.9 Hz), 78.9, 52.3, 51.1, 46.1 (q, *J* = 26.1 Hz), 37.8, 37.3, 24.7, 24.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F), –62.5 (s, 3F).

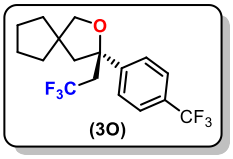
HRMS (ESI) m/z calcd. for $C_{17}H_{19}F_6O$ $[M + H]^+$ 353.1335, found 353.1323.



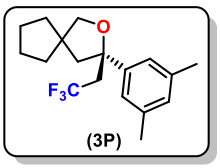
(*R*)-3-(4-bromophenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3N)

HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 230 nm), t_R (major) = 7.79 min, t_R (minor) = 10.51 min.

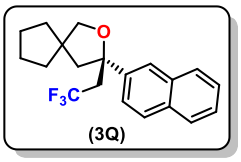
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.6 Hz, 2H), 7.29 (d, *J* = 8.6 Hz, 2H), 3.79 (d, *J* = 8.4 Hz, 1H), 3.64 (d, *J* = 8.4 Hz, 1H), 2.66 (dd, *J* = 10.7, 1.6 Hz, 1H), 2.62 (dd, *J* = 10.7, 2.0 Hz, 1H), 2.34 (d, *J* = 12.8 Hz, 1H), 2.28 (d, *J* = 12.8 Hz, 1H), 1.76–1.71 (m, 1H), 1.64–1.45 (m, 5H), 1.34–1.29 (m, 1H), 1.23–1.17 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 144.3, 131.2, 127.2, 125.1 (q, *J* = 276.8 Hz), 120.9, 82.8 (q, *J* = 2.1 Hz), 78.8, 52.1, 51.0, 46.1 (q, *J* = 26.0 Hz), 37.8, 37.4, 24.6, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{16}H_{19}F_3BrO$ $[M + H]^+$ 363.0566, found 363.0553.



(*R*)-3-(2,2,2-trifluoroethyl)-3-(4-(trifluoromethyl)phenyl)-2-oxaspiro[4.4]nonane (3O)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 6.30 min, t_R (minor) = 6.98 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.3 Hz, 2H), 3.82 (d, *J* = 8.4 Hz, 1H), 3.67 (d, *J* = 8.4 Hz, 1H), 2.70 (dd, *J* = 10.6, 1.8 Hz, 1H), 2.66 (dd, *J* = 10.6, 2.2 Hz, 1H), 2.37 (d, *J* = 12.8 Hz, 1H), 2.33 (d, *J* = 12.9 Hz, 1H), 1.78–1.73 (m, 1H), 1.66–1.46 (m, 5H), 1.35–1.29 (m, 1H), 1.22–1.16 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 149.3, 129.3 (q, *J* = 32.2 Hz), 125.8, 125.1 (q, *J* = 3.8 Hz), 125.06 (q, *J* = 276.8 Hz), 124.1 (q, *J* = 270.2 Hz), 82.9 (q, *J* = 1.9 Hz), 78.9, 52.4, 51.1, 46.1 (q, *J* = 26.1 Hz), 37.7, 37.3, 24.7, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F), –62.4 (s, 3F).
HRMS (ESI) m/z calcd. for $C_{17}H_{19}F_6O$ $[M + H]^+$ 353.1335, found 353.1319.



(*R*)-3-(3,5-dimethylphenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3P)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 100/0, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 5.71 min, t_R (minor) = 6.74 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.00 (s, 2H), 6.87 (s, 1H), 3.79 (d, *J* = 8.3 Hz, 1H), 3.66 (d, *J* = 8.3 Hz, 1H), 2.66 (d, *J* = 10.7 Hz, 1H), 2.62 (d, *J* = 10.8 Hz, 1H), 2.37 (d, *J* = 12.8 Hz, 1H), 2.31 (s, 6H), 2.28 (d, *J* = 12.8 Hz, 1H), 1.78–1.73 (m, 1H), 1.66–1.46 (m, 5H), 1.38–1.33 (m, 1H), 1.29–1.24 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.4, 137.5, 128.5, 125.3 (q, *J* = 276.9 Hz), 122.9, 83.1 (q, *J* = 1.9 Hz), 78.7, 51.9, 51.1, 46.1 (q, *J* = 25.6 Hz), 38.0, 37.2, 24.6, 24.5, 21.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{18}H_{24}F_3O$ $[M + H]^+$ 313.1774, found 313.1767.



(*R*)-3-(naphthalen-2-yl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3Q)

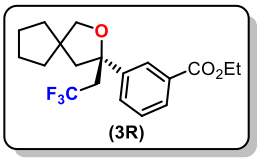
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 9.60 min, t_R (minor) = 10.40 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.86–7.81 (m, 3H), 7.50–7.45 (m, 3H), 3.86 (d, *J* = 8.4 Hz, 1H), 3.74 (d, *J* = 8.4 Hz, 1H), 2.78 (d, *J* = 10.7 Hz, 1H), 2.74 (d, *J* = 10.7 Hz, 1H), 2.50 (d, *J* = 12.8 Hz, 1H), 2.37 (d, *J* = 12.8 Hz, 1H), 1.80–1.75 (m, 1H), 1.65–1.43 (m, 5H), 1.34–1.29 (m, 1H), 1.23–1.17 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 142.5, 133.0, 132.4, 128.2, 127.9, 127.5, 126.1, 125.8, 125.3 (q, *J* = 276.9 Hz), 123.9, 123.8, 83.3 (q, *J* = 1.9 Hz), 78.9, 52.0, 51.1, 46.1 (q, *J* = 25.8 Hz), 38.0, 37.3, 24.6, 24.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.7 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{20}H_{22}F_3O$ $[M + H]^+$ 335.1617, found 335.1605.



(*R*)-ethyl 3-(3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonan-3-yl)benzoate (3R)

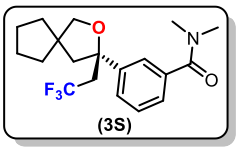
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 5.29 min, t_R (minor) = 6.25 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (t, *J* = 1.7 Hz, 1H), 7.95–7.93 (m, 1H), 7.68–7.66 (m, 1H), 7.41 (t, *J* = 7.8 Hz, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 3.82 (d, *J* = 8.4 Hz, 1H), 3.68 (d, *J* = 8.4 Hz, 1H), 2.72 (d, *J* = 10.6 Hz, 1H), 2.67 (d, *J* = 10.6 Hz, 1H), 2.41 (d, *J* = 12.8 Hz, 1H), 2.34 (d, *J* = 12.8 Hz, 1H), 1.77–1.74 (m, 1H), 1.64–1.46 (m, 5H), 1.41 (t, *J* = 7.1 Hz, 3H), 1.34–1.29 (m, 1H), 1.22–1.16 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 166.6, 145.7, 130.4, 129.8, 128.2, 128.1, 126.5, 125.2 (q, *J* = 276.9 Hz), 82.9 (q, *J* = 2.0 Hz), 78.9, 61.0, 52.3, 51.0, 46.1 (q, *J* = 25.9 Hz), 37.8, 37.3, 24.7, 24.5, 14.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.7 Hz, 3F).

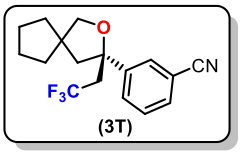
HRMS (ESI) m/z calcd. for $C_{19}H_{24}F_3O_3$ $[M + H]^+$ 357.1672, found 357.1667.



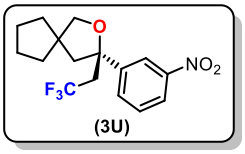
(*R*)-*N*,*N*-dimethyl-3-(3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonan-3-yl)benzamide (3S)

HPLC analysis: Chiralcel AS3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 16.09 min, t_R (minor) = 12.92 min.

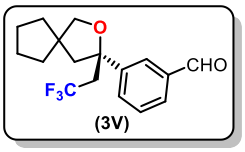
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46–7.43 (m, 2H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.32 (dt, *J* = 7.4, 1.3 Hz, 1H), 3.80 (d, *J* = 8.4 Hz, 1H), 3.65 (d, *J* = 8.4 Hz, 1H), 3.11 (s, 3H), 2.93 (s, 3H), 2.70 (dd, *J* = 10.7, 1.2 Hz, 1H), 2.66 (dd, *J* = 10.7, 1.5 Hz, 1H), 2.39 (d, *J* = 12.8 Hz, 1H), 2.28 (d, *J* = 12.8 Hz, 1H), 1.78–1.72 (m, 1H), 1.64–1.45 (m, 5H), 1.35–1.30 (m, 1H), 1.24–1.19 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 145.2, 136.2, 128.4, 126.5, 125.7, 125.2 (q, *J* = 276.8 Hz), 123.9, 82.9 (q, *J* = 2.0 Hz), 78.8, 52.5, 51.0, 46.2 (q, *J* = 25.9 Hz), 39.4, 37.9, 37.2, 35.3, 24.6, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{19}H_{25}F_3NO_2$ $[M + H]^+$ 356.1832, found 356.1825.



(*R*)-3-(3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonan-3-yl)benzonitrile (3T)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 15.73 min, t_R (minor) = 16.55 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.56 (d, *J* = 7.7 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 3.82 (d, *J* = 8.5 Hz, 1H), 3.66 (d, *J* = 8.5 Hz, 1H), 2.72–2.63 (m, 2H), 2.33 (s, 2H), 1.78–1.73 (m, 1H), 1.67–1.48 (m, 5H), 1.34–1.29 (m, 1H), 1.20–1.15 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.8, 130.7, 129.9, 129.2, 129.0, 124.9 (q, *J* = 276.8 Hz), 118.9, 112.3, 82.5 (q, *J* = 2.0 Hz), 78.9, 52.4, 51.0, 45.9 (q, *J* = 26.1 Hz), 37.6, 37.3, 24.6, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.5 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{17}H_{19}F_3NO$ $[M + H]^+$ 310.1413, found 310.1406.



(*R*)-3-(3-nitrophenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3U)
HPLC analysis: Chiralcel IC (*n*-Hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 15.76 min, t_R (minor) = 14.97 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.30 (t, *J* = 1.9 Hz, 1H), 8.14–8.12 (m, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.53 (t, *J* = 8.0 Hz, 1H), 3.84 (d, *J* = 8.5 Hz, 1H), 3.70 (d, *J* = 8.5 Hz, 1H), 2.75–2.68 (m, 2H), 2.38 (s, 2H), 1.81–1.74 (m, 1H), 1.67–1.48 (m, 5H), 1.35–1.30 (m, 1H), 1.21–1.15 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 148.2, 147.6, 131.6, 129.2, 124.9 (q, *J* = 276.8 Hz), 122.2, 120.6, 82.6 (q, *J* = 2.0 Hz), 79.0, 52.6, 51.1, 46.0 (q, *J* = 26.3 Hz), 37.5, 37.3, 24.7, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.5 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{16}H_{19}F_3NO_3$ $[M + H]^+$ 330.1312, found 330.1306.



(*R*)-3-(3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonan-3-yl)benzaldehyde (3V)

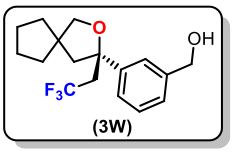
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min, λ = 254 nm), t_R (major) = 6.02 min, t_R (minor) = 6.83 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.94 (s, 1H), 7.78 (d, *J* = 7.5 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 1H), 3.83 (d, *J* = 8.4 Hz, 1H), 3.69 (d, *J* = 8.4 Hz, 1H), 2.74–2.68 (m, 2H), 2.41 (d, *J* = 12.8 Hz, 1H), 2.35 (d, *J* = 12.8 Hz, 1H), 1.80–1.73 (m, 1H), 1.63–1.46 (m, 5H), 1.34–1.29 (m, 1H), 1.21–1.16 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 192.4, 146.6, 136.3, 131.5, 128.9, 128.6, 126.5, 125.1 (q, *J* = 277.0 Hz), 82.8 (q, *J* = 2.0 Hz), 78.9, 52.5, 51.1, 46.1 (q, *J* = 26.0 Hz), 37.7, 37.3, 24.7, 24.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{17}H_{20}F_3O_2$ $[M+H]^+$ 313.1410, found 313.1403.



(*R*)-(3-(3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonan-3-yl)phenyl)methanol (3W)

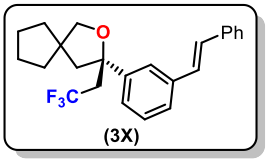
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 1.0 mL/min, λ = 214 nm), t_R (major) = 8.81 min, t_R (minor) = 10.32 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.33–7.32 (m, 2H), 7.26–7.24 (m, 1H), 4.69 (s, 2H), 3.79 (d, *J* = 8.4 Hz, 1H), 3.66 (d, *J* = 8.4 Hz, 1H), 2.70 (d, *J* = 10.7 Hz, 1H), 2.64 (d, *J* = 10.7 Hz, 1H), 2.39 (d, *J* = 12.8 Hz, 1H), 2.30 (d, *J* = 12.8 Hz, 1H), 2.04 (br s, 1H), 1.78–1.72 (m, 1H), 1.67–1.44 (m, 5H), 1.36–1.29 (m, 1H), 1.26–1.19 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 145.6, 140.8, 128.3, 125.5, 125.2 (q, *J* = 276.8 Hz), 124.6, 123.7, 83.1 (q, *J* = 1.8 Hz), 78.7, 65.3, 52.1, 51.0, 46.1 (q, *J* = 25.8 Hz), 37.9, 37.2, 24.6, 24.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F).

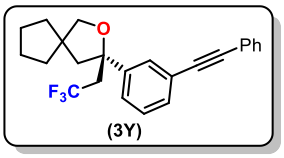
HRMS (ESI) m/z calcd. for $C_{17}H_{22}F_3O_2$ $[M + H]^+$ 315.1566, found 315.1556.



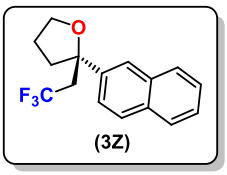
(*R*,*E*)-3-(3-styrylphenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3X)

HPLC analysis: Chiralcel OJH (*n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 280 nm), t_R (major) = 6.88 min, t_R (minor) = 8.66 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.52 (d, *J* = 7.3 Hz, 2H), 7.42 (d, *J* = 7.6 Hz, 1H), 7.38–7.31 (m, 3H), 7.28–7.25 (m, 2H), 7.13 (s, 2H), 3.83 (d, *J* = 8.4 Hz, 1H), 3.70 (d, *J* = 8.4 Hz, 1H), 2.72 (d, *J* = 10.7 Hz, 1H), 2.68 (d, *J* = 10.7 Hz, 1H), 2.43 (d, *J* = 12.8 Hz, 1H), 2.33 (d, *J* = 12.8 Hz, 1H), 1.78–1.75 (m, 1H), 1.66–1.47 (m, 5H), 1.38–1.33 (m, 1H), 1.28–1.22 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.8, 137.3, 137.2, 128.8, 128.7, 128.6, 128.4, 127.6, 126.5, 125.3 (q, *J* = 276.8 Hz), 125.0, 124.7, 123.4, 83.1 (q, *J* = 2.1 Hz), 78.8, 52.0, 51.1, 46.1 (q, *J* = 25.8 Hz), 38.0, 37.3, 24.7, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.7 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{24}H_{26}F_3O$ $[M + H]^+$ 387.1930, found 387.1925.

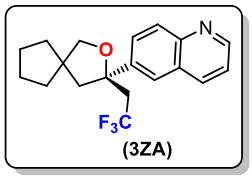


(*R*)-3-(3-(phenylethynyl)phenyl)-3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonane (3Y)
HPLC analysis: Chiralcel OJH (*n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 280 nm), t_R (major) = 5.88 min, t_R (minor) = 7.44 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (s, 1H), 7.55–7.53 (m, 2H), 7.43–7.31 (m, 6H), 3.81 (d, *J* = 8.4 Hz, 1H), 3.69 (d, *J* = 8.4 Hz, 1H), 2.70 (d, *J* = 10.6 Hz, 1H), 2.66 (d, *J* = 10.7 Hz, 1H), 2.40 (d, *J* = 12.8 Hz, 1H), 2.31 (d, *J* = 12.8 Hz, 1H), 1.77–1.73 (m, 1H), 1.66–1.46 (m, 5H), 1.38–1.32 (m, 1H), 1.26–1.20 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.6, 131.6, 130.2, 128.5, 128.3, 128.2, 128.1, 125.4, 125.2 (q, *J* = 276.8 Hz), 123.2, 123.0, 89.5, 89.3, 82.9 (q, *J* = 1.9 Hz), 78.9, 52.1, 51.1, 46.1 (q, *J* = 25.9 Hz), 37.9, 37.3, 24.7, 24.5.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F).
HRMS (ESI) m/z calcd. for $C_{24}H_{24}F_3O$ $[M + H]^+$ 385.1774, found 385.1769.



(*R*)-2-(naphthalen-2-yl)-2-(2,2,2-trifluoroethyl)tetrahydrofuran (3Z)
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 214 nm), t_R (major) = 10.88 min, t_R (minor) = 11.77 min.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (d, *J* = 1.2 Hz, 1H), 7.86–7.82 (m, 3H), 7.49–7.44 (m, 3H), 4.12–4.07 (m, 1H), 4.01–3.97 (m, 1H), 2.84–2.69 (m, 2H), 2.46–2.41 (m, 1H), 2.33–2.27 (m, 1H), 2.05–1.97 (m, 1H), 1.85–1.76 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 141.8, 133.1, 132.5, 128.2, 128.1, 127.5, 126.2, 125.9, 125.4 (q, *J* = 276.6 Hz), 123.9, 123.5, 83.07 (q, *J* = 2.0 Hz), 67.9, 45.0 (q, *J* = 26.1 Hz), 38.1, 25.1.
^{19}F NMR (376 MHz, $CDCl_3$) δ –60.4 (t, *J* = 10.8 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{16}H_{16}F_3O$ $[M + H]^+$ 281.1148, found 281.1148.



(*R*)-6-(3-(2,2,2-trifluoroethyl)-2-oxaspiro[4.4]nonan-3-yl)quinoline (3ZA)

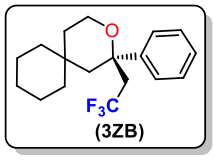
HPLC analysis: Chiralcel OD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 1.0 mL/min, λ = 230 nm), t_R (major) = 7.92 min, t_R (minor) = 8.69 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.91 (dd, *J* = 4.1, 1.5 Hz, 1H), 8.17 (d, *J* = 8.0 Hz, 1H), 8.09 (d, *J* = 8.8 Hz, 1H), 7.95 (d, *J* = 1.9 Hz, 1H), 7.68 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.41 (dd, *J* = 8.3, 4.2 Hz, 1H), 3.88 (d, *J* = 8.4 Hz, 1H), 3.75 (d, *J* = 8.4 Hz, 1H), 2.78 (q, *J* = 10.6 Hz, 2H), 2.49 (d, *J* = 12.8 Hz, 1H), 2.38 (d, *J* = 12.8 Hz, 1H), 1.80–1.75 (m, 1H), 1.66–1.44 (m, 5H), 1.35–1.30 (m, 1H), 1.22–1.16 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 150.4, 147.4, 143.3, 136.3, 129.4, 127.8, 127.4, 125.2 (q, *J* = 277.5 Hz), 123.8, 121.3, 83.1 (q, *J* = 2.0 Hz), 78.9, 52.3, 51.1, 45.9 (q, *J* = 25.0 Hz), 37.8, 37.3, 24.6, 24.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{19}H_{21}F_3NO$ $[M + H]^+$ 336.1570, found 336.1566.



(*R*)-2-phenyl-2-(2,2,2-trifluoroethyl)-3-oxaspiro[5.5]undecane (3ZB)

HPLC analysis: Chiralcel OD3 (*n*-Hexane, flow rate 1.0 mL/min, λ = 209 nm), t_R (major) = 16.0 min, t_R (minor) = 12.5 min.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.5 Hz, 2H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.29–7.26 (m, 1H), 3.78–3.76 (m, 2H), 2.49–2.42 (m, 3H), 1.63 (d, *J* = 14.3 Hz, 1H), 1.56–1.28 (m, 11H), 1.18–1.09 (m, 1H).

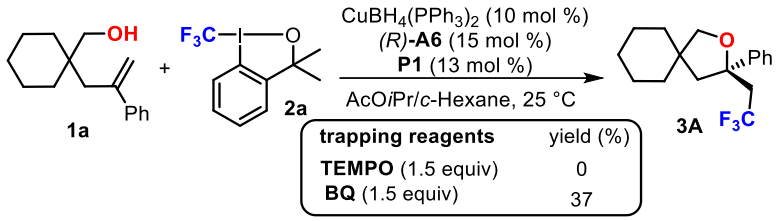
^{13}C NMR (125 MHz, $CDCl_3$) δ 142.1, 128.1, 127.0, 126.0, 125.2 (q, *J* = 239.2 Hz), 75.1, 58.9, 49.8 (q, *J* = 25.5 Hz), 42.6, 33.5, 31.5, 26.3, 21.6, 21.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ –59.3 (t, *J* = 11.0 Hz, 3F).

HRMS (ESI) m/z calcd. for $C_{18}H_{24}F_3O$ $[M + H]^+$ 313.1774, found 313.1770.

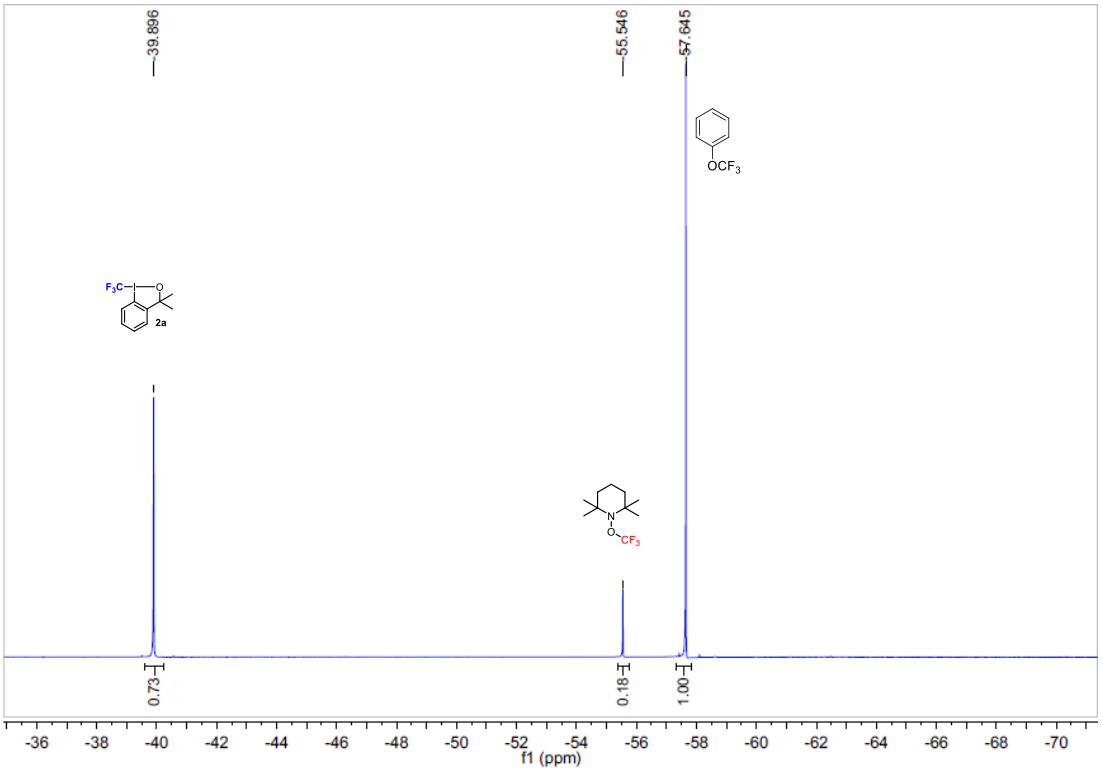
Mechanistic Study

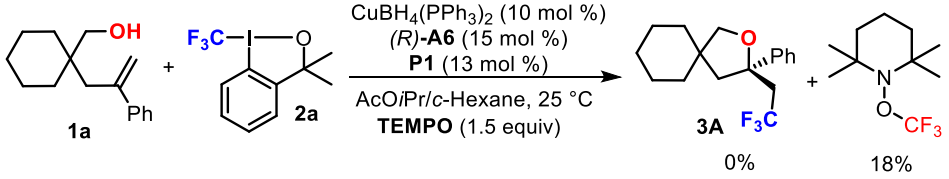
Radical trapping experiments (Scheme S2a)

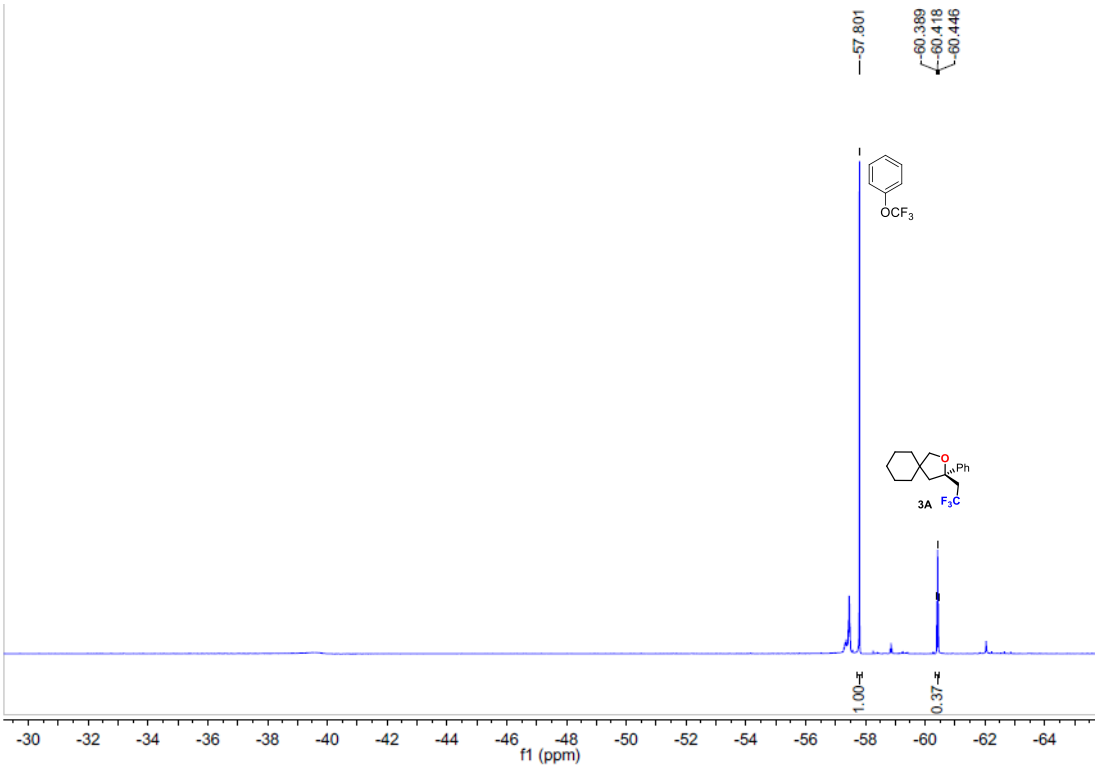


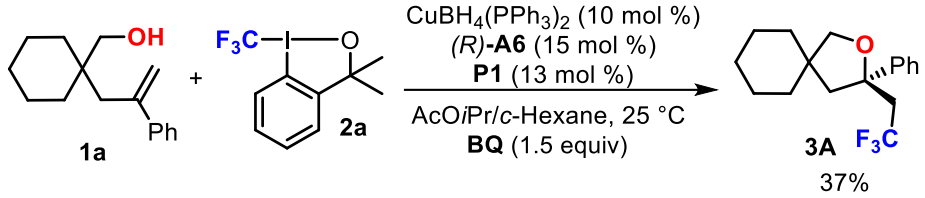
Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1** (0.05 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (3.0 mg, 0.005 mmol, 10 mol %), chiral phosphoric acid (*R*)-**A6** (4.5 mg, 0.0075 mmol, 15 mol %), **P1** (1.2 mg, 0.0065 mmol, 13 mol %), Togni's reagent **2a** (24.8 mg, 0.075 mmol, 1.5 equiv), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO, 11.7 mg, 0.075 mmol, 1.5 equiv) or 1,4-benzoquinone (BQ, 8.1 mg, 0.075 mmol, 1.5 equiv) and AcO*i*Pr/*c*-Hexane (9:1, 0.5 mL) at 25 °C, and the sealed tube was then stirred at 25 °C for 36 h. $PhOCF_3$ (internal standard, 0.05 mmol, 1.0 equiv) 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.

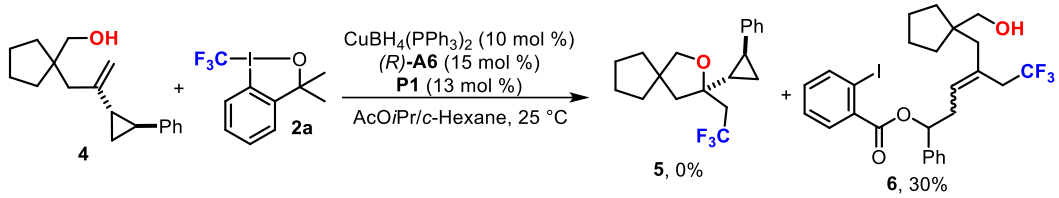




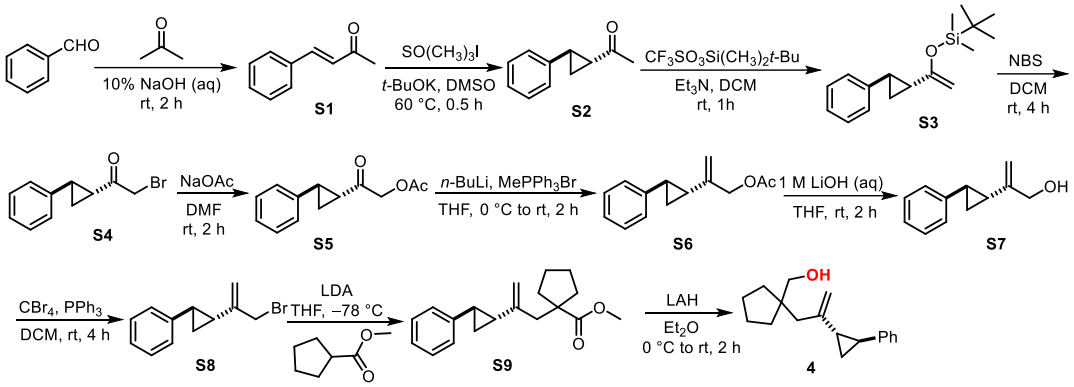

-57.801
-60.389
-60.418
-60.446
OCF3
3A
1.00
0.37
-30 -32 -34 -36 -38 -40 -42 -44 -46 -48 -50 -52 -54 -56 -58 -60 -62 -64
f1 (ppm)


OH
Ph
1a
F3C
O
2a
CuBH4(PPh3)2 (10 mol %)
(R)-A6 (15 mol %)
P1 (13 mol %)
AcOiPr/c-Hexane, 25 °C
BQ (1.5 equiv)
O
Ph
F3C
3A
37%

Radical clock experiment (eq 1)



Synthesis of substrate 4.



Acetone (20.0 ml, 200.0 mmol), benzaldehyde (10.0 ml, 100.0 mmol) and water (80.0 mL) were mixed in a 250 mL 3-neck flask fitted with a dropping funnel and an argon inlet. Then the mixture was cooled to 0 °C in an ice/water bath followed by addition of sodium hydroxide solution (4.0 mL, 10%) over 30 min. Upon completion, the reaction mixture was warmed to room temperature and stirred for additional two hours. The reaction mixture was extracted three times using ethyl acetate (3×100 mL). The collected organic layer was washed with 300 mL saturated sodium chloride until the solution was neutral. The solution was dried with anhydrous sodium sulfate. After evaporation under reduced pressure, a yellow liquid was obtained. Yield: 13.5 g (92.7 %). The product was used directly in the next step reaction.[5]

A mixture of $Me_3S(O)I$ (13.2 g, 60.0 mmol) and *t*-BuOK (6.72 g, 60.0 mmol) in dry DMSO (60 mL) was stirred at 60 °C for 15 min to achieve a clear solution. Then **S1** (7.3 g, 50 mmol) was added to the mixture at 60 °C over 30 min. Upon completion, the mixture was treated with brine (50 mL) and extracted with diethyl ether (2 × 30 mL). The combined organic extracts were washed successively with water (2 × 50 mL) and brine (1 × 50 mL), dried over anhydr. $MgSO_4$, filtered, and evaporated in *vacuo*. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 30/1 to 20/1) to give **S2** (3.2 g, 40%) as a liquid.[6]

To a solution of **S2** (3.2 g, 20.0 mmol) and triethylamine (8.3 mL, 60.0 mmol) in DCM (40.0 mL) was added *tert*-butyldimethylsilyltrifluoromethane sulfonate (6.88 mL, 30.0 mmol) dropwise under argon atmosphere at rt. Upon completion, the stirring was continued under the same conditions until the completion of the reaction as indicated by TLC staining. Then the reaction mixture was diluted with DCM and washed with a 1:1 mixture of water and saturated $NaHCO_3$. The organic layer was dried over $MgSO_4$, filtered and concentrated to afford the silyl enol ether **S3** as a

heavy oil. The silyl enol ether **S3** was next taken in DCM (40.0 mL) and solid NBS (3.54 g, 20.0 mmol) was added. The reaction mixture was stirred until the full conversion of **S3** as monitored by TLC staining. The mixture was loaded to a silica gel column for chromatography (eluent: petroleum ether/EtOAc = 25/1) to afford **S4** (3.2 g, 67%, two steps).[7]

To a solution of **S4** (1.67 g, 7.0 mmol) in DMF (30.0 mL) was added NaOAc (2.87 g, 35 mmol). The reaction mixture was stirred at room temperature for 2 h and then was quenched by the addition of 1N HCl. The solution was extracted with EtOAc (30.0 mL) three times and the combined organic layer was washed with 1N HCl, dried over Na_2SO_4, and then concentrated *in vacuo*. The crude product was purified by flash chromatography (eluent: petroleum ether/EtOAc = 50/1 to 8/1) to afford **S5** (1.31 g, 86%) as a colorless oil.[8]

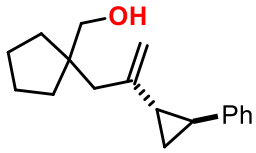
To a slurry of methyltriphenylphosphonium bromide (2.32 g, 6.5 mmol) in THF (15.0 mL) at 0 °C was added *n*-BuLi (2.4 M, 2.7 mL, 6.5 mmol). The resulting solution was stirred at 0 °C for 1 h, at which time **S5** (1.09 g, 5.0 mmol) in THF (5.0 mL) was added via syringe. The reaction mixture was stirred at 0 °C for 1 h and then at room temperature for 2 h. The reaction was quenched by the addition of saturated aq. NH_4Cl and the resulting solid salts were dissolved by adding a minimum amount of H_2O. The THF was removed *in vacuo* and the aqueous layer was extracted with DCM for three times. The combined organic extracts were dried over Na_2SO_4 and concentrated *in vacuo*. Then the residue **S6** was dissolved in THF (20.0 mL) and to this solution was added aq. LiOH (1 M, 15.0 mL, 15.0 mmol). The reaction mixture was stirred at room temperature for 1 h, at which time it was acidified to pH 1 by the addition of 1 M aq. HCl. The solution was diluted with EtOAc and the two layers were separated. The aqueous layer was saturated with NaCl and extracted with EtOAc three times. The combined organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The crude product was purified by flash chromatography (eluent: petroleum ether/EtOAc = 30/1 to 10/1) to afford **S7** (522.0 mg, 60 %) as a colorless oil.[8]

To a solution of **S7** (348.0 mg, 2.0 mmol) in anhydr. DCM (10.0 mL) was added CBr_4 (796.0 mg, 2.4 mmol) and PPh_3 (631.0 mg, 2.4 mmol). The reaction mixture was stirred under argon for 2 h. Then the solvent was removed *in vacuo*. The crude product was purified by column chromatography with pure petroleum ether to afford **S8** (426.0 mg, 90%).[9]

Methyl cyclopentanecarboxylate (0.17 mL, 1.36 mmol) was added to a solution of LDA [generated in situ from *n*-BuLi (0.625 mL in *n*-Hexane, 2.4 M, 1.5 mmol) and diisopropylamine (0.2 mL, 1.5 mmol) in THF (20.0 mL)] at −78 °C and the resulting mixture was stirred under the same conditions for 45 min. To the resulting solution was added **S8** (355.5 mg, 1.5 mmol) at −78 °C. Upon completion, the solution was warmed to room temperature and stirred overnight. CH_2Cl_2 (5.0 mL) was added and the resulting biphasic mixture washed with water (3 × 5.0 mL), dried (Na_2SO_4) and

concentrated. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 100/1 to 50/1) to give **S9** (340.8 mg, 80%) as a liquid.

To a suspension of $LiAlH_4$ (76.0 mg, 2.0 mmol) in Et_2O (5.0 mL) at 0 °C was slowly added a solution of **S9** (284.0 mg, 1.0 mmol) in Et_2O (5.0 mL). Upon completion, the mixture was warmed to room temperature and stirred for additional 2 h. The reaction mixture was quenched by slow, portionwise addition of water (0.1 mL) in Na_2SO_4 (0.8 g) at 0 °C. Then the reaction mixture was warmed to room temperature, stirred for additional 30 minutes, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 20/1 to 5/1) to give **4** (204.8 mg, 80%) as a colorless oil.



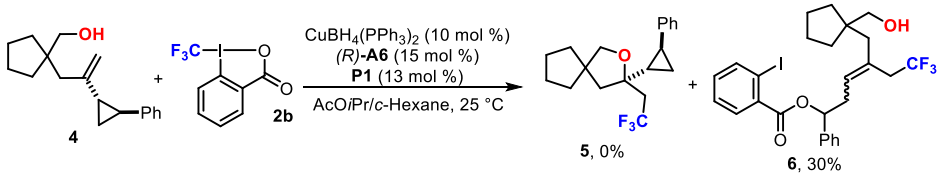
4

(1-(2-(2-phenylcyclopropyl)allyl)cyclopentyl)methanol (4)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.24 (m, 2H), 7.17–7.13 (m, 1H), 7.08–7.06 (m, 2H), 4.75 (d, *J* = 5.6 Hz, 2H), 3.41 (s, 2H), 2.35–2.26 (m, 2H), 1.95–1.90 (m, 1H), 1.65–1.42 (m, 9H), 1.25–1.14 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 149.2, 142.7, 128.3, 125.6, 125.5, 108.9, 68.1, 48.0, 45.2, 34.9, 34.8, 29.1, 27.5, 24.6, 24.5, 17.7.

HRMS (ESI) m/z calcd. for $C_{18}H_{25}O$ $[M + H]^+$ 257.1900, found 257.1895.



Radical clock experiment: Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **4** (0.1 mmol, 1.0 equiv), Togni's reagent **2b** (47.7 mg, 0.15 mmol, 1.5 equiv), $CuBH_4(PPh_3)_2$ (6.0 mg, 0.01 mmol, 10 mol %), chiral phosphoric acid (*R*)-**A6** (9.0 mg, 0.015 mmol, 15 mol %), **P1** (2.3 mg, 0.013 mmol, 13 mol %), and AcO*i*Pr/*c*-Hexane (9:1, 1.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C for 3 d. The reaction mixture was concentrated and the residue was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 5/1) to give **6** (17.2 mg, 30%) as a colorless oil.

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

6,6,6-trifluoro-4-((1-(hydroxymethyl)cyclopentyl)methyl)-1-phenylhex-3-en-1-yl 2-iodobenzoate (6)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03–8.01 (m, 1H), 7.88 (dd, *J* = 7.8, 1.6 Hz, 0.8H), 7.85 (dd, *J* = 7.8, 1.6 Hz, 0.2H), 7.49–7.35 (m, 6H), 7.18 (td, *J* = 7.7, 1.7 Hz, 1H),

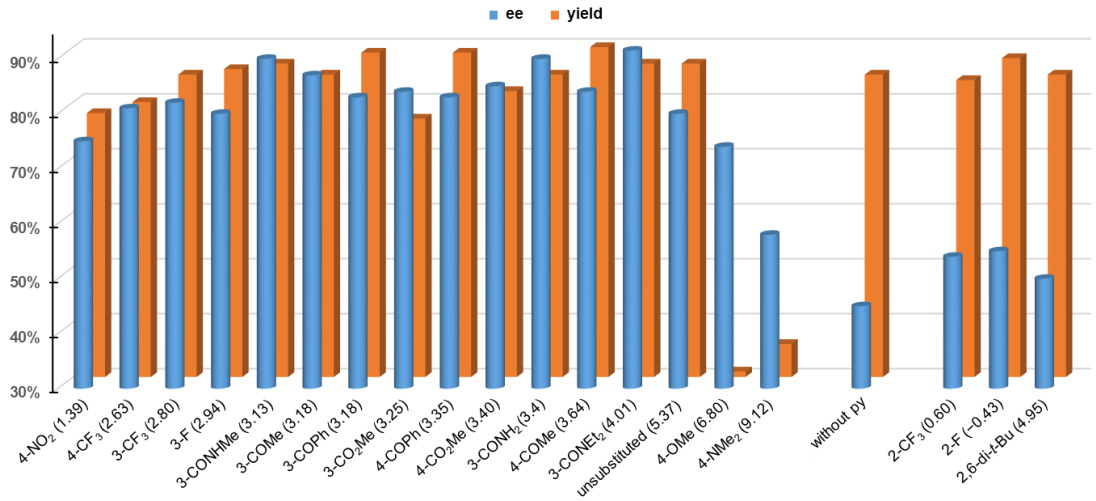
6.06 (t, *J* = 6.7 Hz, 1H), 5.59 (t, *J* = 7.2 Hz, 1H), 3.31 (s, 0.4H), 3.16 (s, 1.6H), 2.98–2.77 (m, 4H), 2.27 (s, 0.4H), 2.21 (s, 1.6H), 1.67–1.28 (m, 8H).

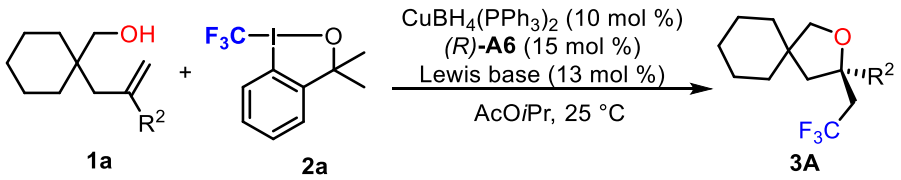
^{13}C NMR (125 MHz, $CDCl_3$) δ 165.6, 165.4, 141.5, 141.4, 139.34, 139.30, 134.8, 134.6, 132.8, 132.7, 131.1, 131.0, 130.2, 130.1, 129.6, 128.6, 128.5, 128.4, 128.2, 127.9, 126.7, 126.6, 126.3 (q, *J* = 276.6 Hz), 94.2, 67.4, 66.9, 48.8, 48.3, 43.5, 41.5 (q, *J* = 34.0 Hz), 36.6, 35.5, 35.4, 35.3, 35.0, 34.7, 34.6, 34.5, 24.2, 24.1.

^{19}F NMR (376 MHz, $CDCl_3$) δ –63.9 (t, *J* = 10.9 Hz), –64.7 (t, *J* = 10.9 Hz).

HRMS (ESI) m/z calcd. for $C_{26}H_{29}O_3F_3I$ $[M + H]^+$ 573.1108, found 573.1106.

Substituent effect of pyridine on reaction performance (Figure S3a).

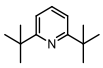
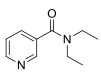




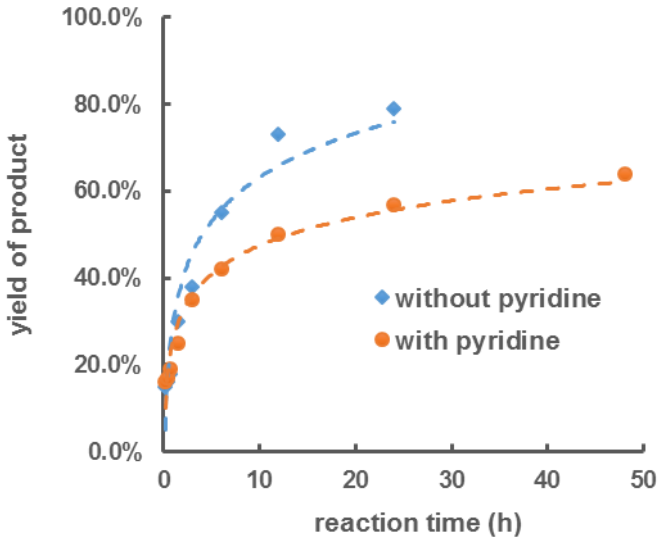
Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1a** (11.5 mg, 0.05 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (3.0 mg, 0.005 mmol, 10 mol %), chiral phosphoric acid (*R*)-**A6** (4.5 mg, 0.0075 mmol, 15 mol %), Lewis base (0.0065 mmol, 13 mol%), Togni's reagent **2a** (24.8 mg, 0.075 mmol, 1.5 equiv) and AcO*i*Pr (0.5 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. The reaction mixture was concentrated after 36 h. The yields were determined by ^{19}F NMR spectroscopy using $PhOCF_3$ (8.1 mg, 0.05 mmol, 1.0 equiv.) as internal standard. The ee values were determined by HPLC after isolation of products by preparative TLC.

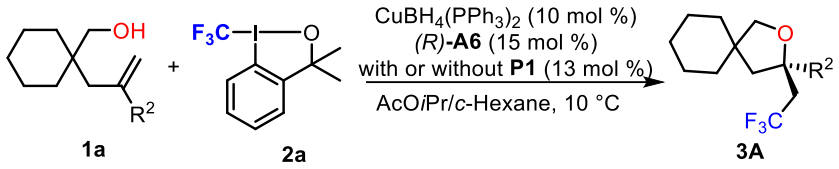
Note: Since the reaction is sensitive to water and air, the Schlenk tube and the reagents must be dried prior to use.

Table S1. References for p*K*a values of pyridine and its analogues

entry	additive	p*K*a
1		6.80, B. Uno, T. Kawakita, K. Kano, N. Okumura, M. Goto, T. Kubota, *Bull. Chem. Soc. Jpn.* **1994**, *67*, 2304.
2		9.12, I.-H. Um, S.-J. Hwang, M.-H. Baek, E. J. Park, *J. Org. Chem.* **2006**, *71*, 9191.
3		0.60, predicted, retrieved in Scifinder
4		2.80, predicted, retrieved in Scifinder
5		2.63, M. Taagepera, W. G. Henderson, R. T. C. Brownlee, J. L. Beauchamp, D. Holtz, R. W. Taft, *J. Am. Chem. Soc.* **1972**, *94*, 1369.
6		−0.43, I. T. Suydam, S. A. Strobel, *J. Am. Chem. Soc.* **2008**, *130*, 13639.
7		2.94, I. T. Suydam, S. A. Strobel, *J. Am. Chem. Soc.* **2008**, *130*, 13639.
8		4.95, H. P. Hopkins, Jr., D. V. Jahagirdar, P. S. Moulik, D. H. Aue, H. M. Webb, W. R. Davidson, M. D. Pedley, *J. Am. Chem. Soc.* **1984**, *106*, 4341.
9		1.39, M. Taagepera, W. G. Henderson, R. T. C. Brownlee, J. L. Beauchamp, D. Holtz, R. W. Taft, *J. Am. Chem. Soc.* **1972**, *94*, 1369.
10		3.25, E. Chrystiuk, A. Williams, *J. Am. Chem. Soc.* **1987**, *109*, 3040.
11		3.40, E. Chrystiuk, A. Williams, *J. Am. Chem. Soc.* **1987**, *109*, 3040.
12		3.64, E. Chrystiuk, A. Williams, *J. Am. Chem. Soc.* **1987**, *109*, 3040.
13		3.35, A. Fischer, W. J. Galloway, J. Vaughan, *J. Chem. Soc.* **1964**, 3591.
14		3.18, A. Bryson, *J. Am. Chem. Soc.* **1960**, *82*, 4871.
15		3.18, A. Fischer, W. J. Galloway, J. Vaughan, *J. Chem. Soc.* **1964**, 3591.
16		3.4, E. A. Castro, M. Cubillos, J. G. Santos, *J. Org. Chem.* **2004**, *69*, 4802.
17		3.13, predicted, retrieved in Scifinder
18		4.01, predicted, retrieved in Scifinder
19		5.37, E. A. Castro, M. Cubillos, J. G. Santos, *J. Org. Chem.* **2004**, *69*, 4802.

Retarding effect of pyridine on reaction rate (Figure S3b).

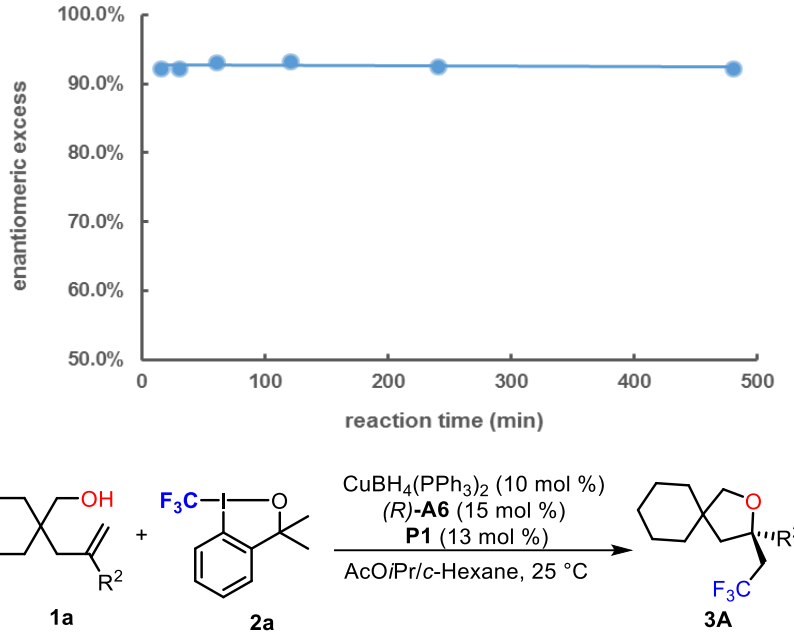




Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1a** (46 mg, 0.2 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (12.0 mg, 0.02 mmol, 10 mol %), chiral phosphoric acid (*R*)-**A6** (18.0 mg, 0.03 mmol, 15 mol %), with or without **P1** (4.6 mg, 0.026 mmol, 13 mol %), Togni's reagent **2a** (99.0 mg, 0.3 mmol, 1.5 equiv), $PhOCF_3$ (32.4 mg, 0.2 mmol, 1.0 equiv.) and AcO*i*Pr/*c*-Hexane (9:1, 2.0 mL) at 10 °C, and the sealed tube was then stirred at 10 °C. After 3 mins, the reaction mixture turned clear. The reaction progress was monitored by ^{19}F NMR spectroscopy to determine the yields by taking 25 μL of the reaction mixture at specific time intervals (0.19, 0.38, 0.75, 1.5, 3, 6, 12, 24 h for reaction without pyridine; 0.19, 0.38, 0.75, 1.5, 3, 6, 12, 24 and 48 h for reaction with pyridine) and dissolving it in $CDCl_3$.

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

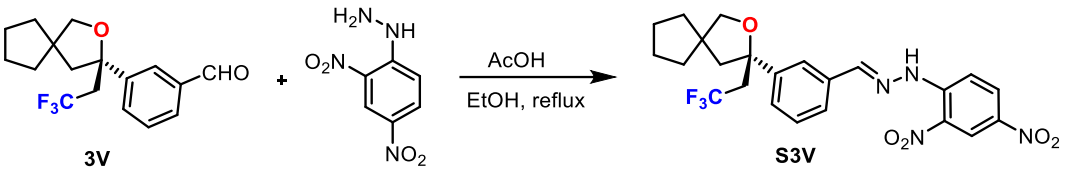
Ee of products measured at different time points during reaction (Figure S3c)



Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with substrate **1a** (46 mg, 0.2 mmol, 1.0 equiv), $CuBH_4(PPh_3)_2$ (12.0 mg, 0.02 mmol, 10 mol %), chiral phosphoric acid (*R*)-**A6** (18.0 mg, 0.03 mmol, 15 mol %), **P1** (4.6 mg, 0.026 mmol, 13 mol %), Togni's reagent **2a** (99.0 mg, 0.3 mmol, 1.5 equiv) and AcO*i*Pr/*c*-Hexane (9:1, 2.0 mL) at 25 °C, and the sealed tube was then stirred at 25 °C. After 3 mins, the reaction mixture turned clear. The ee values at different time points during reaction were measured by taking 100 μL of the reaction mixture each time for preparative TLC purification and HPLC analysis.

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

Transformation



Synthesis of **S3V**: Under argon, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with **3V** (93.6 mg, 0.3 mmol), 2,4-dinitrophenylhydrazine (59.4 mg, 0.3 mmol), AcOH (3.6 mg, 0.06 mmol) and EtOH (3 mL). The sealed tube was then stirred at 80 °C for 6 h. The reaction mixture was concentrated and the residue was purified by silica gel column chromatography (eluent: DCM/MeOH = 5/1) to give **S3V** (125.5 mg, 85%) as a red solid.

HPLC analysis: Chiralcel AD3 (*n*-Hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 370 nm), t_R (major) = 52.44 min, t_R (minor) = 49.55 min.

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.34 (s, 1H), 9.16 (d, *J* = 2.5 Hz, 1H), 8.38 (dd, *J* = 9.5, 2.2 Hz, 1H), 8.16–8.10 (m, 2H), 7.83 (s, 1H), 7.70 (d, *J* = 7.4 Hz, 1H), 7.50–7.43 (m, 2H), 3.85 (d, *J* = 8.4 Hz, 1H), 3.71 (d, *J* = 8.4 Hz, 1H), 2.75 (d, *J* = 10.6 Hz, 1H), 2.70 (d, *J* = 10.7 Hz, 1H), 2.43 (d, *J* = 12.8 Hz, 1H), 2.36 (d, *J* = 12.8 Hz, 1H), 1.68–1.50 (m, 7H), 1.39–1.33 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 148.1, 146.4, 144.8, 138.2, 133.0, 130.0, 129.3, 128.8, 128.1, 126.2, 125.2 (q, *J* = 276.9 Hz), 124.7, 123.5, 116.8, 82.9 (q, *J* = 1.9 Hz), 78.9, 52.3, 51.1, 46.1 (q, *J* = 25.9 Hz), 37. 8, 37.3, 24.7, 24.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ –60.3 (t, *J* = 10.6 Hz).

HRMS (ESI) m/z calcd. for $C_{23}H_{22}N_4O_5F_3$ $[M - H]^+$ 491.1548, found 491.1552.

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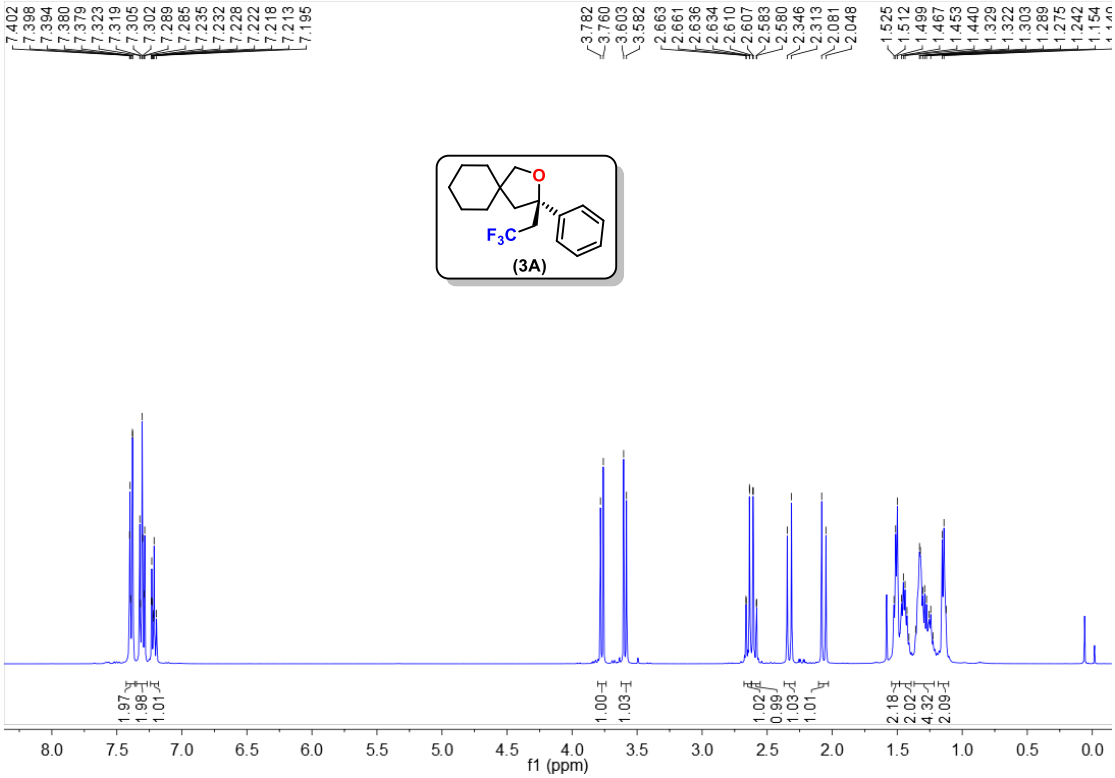
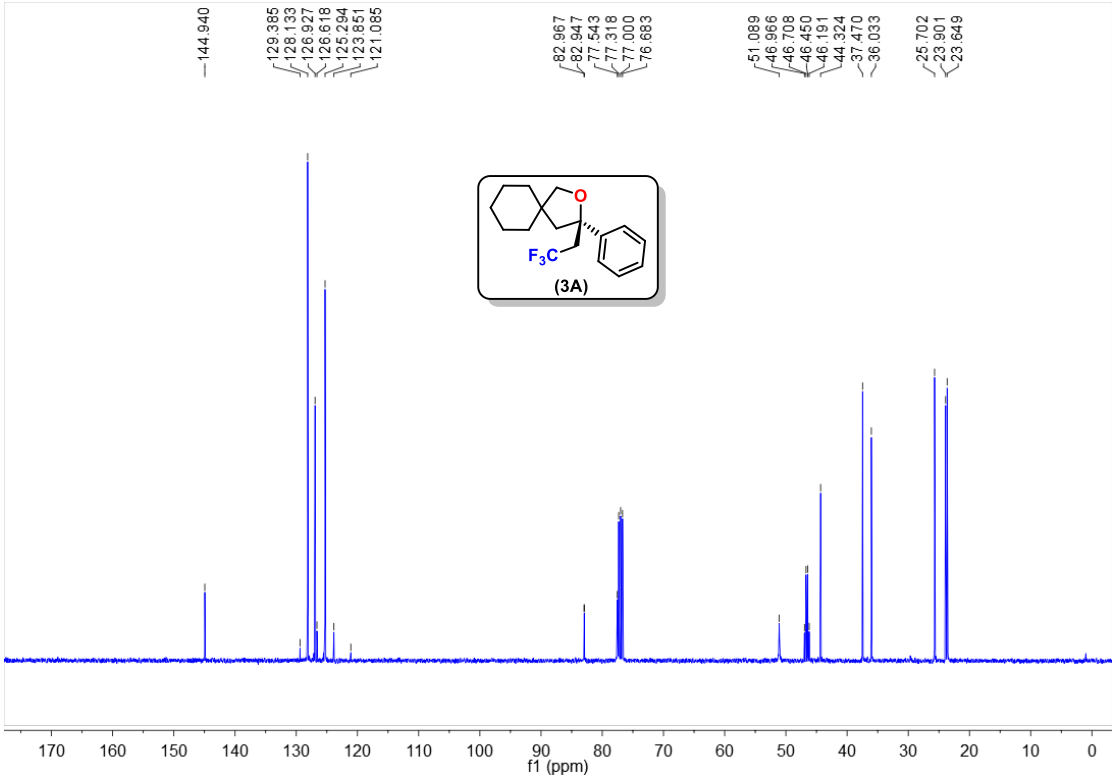
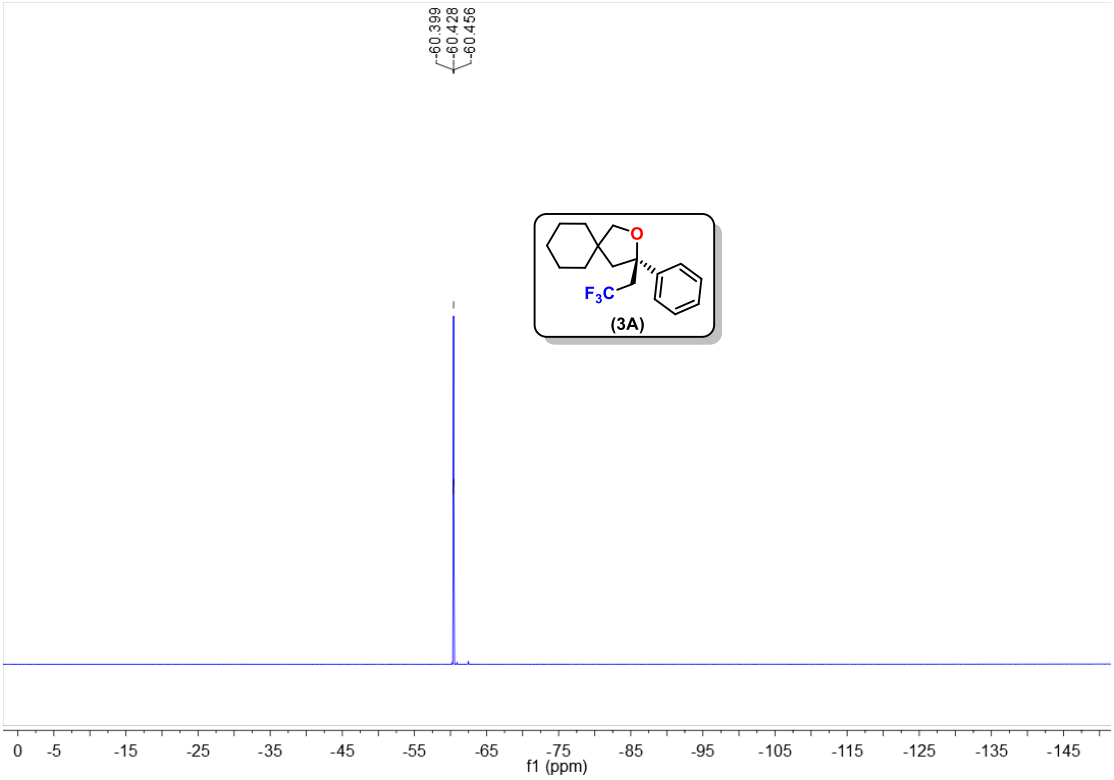
NMR Spectra of New Compounds



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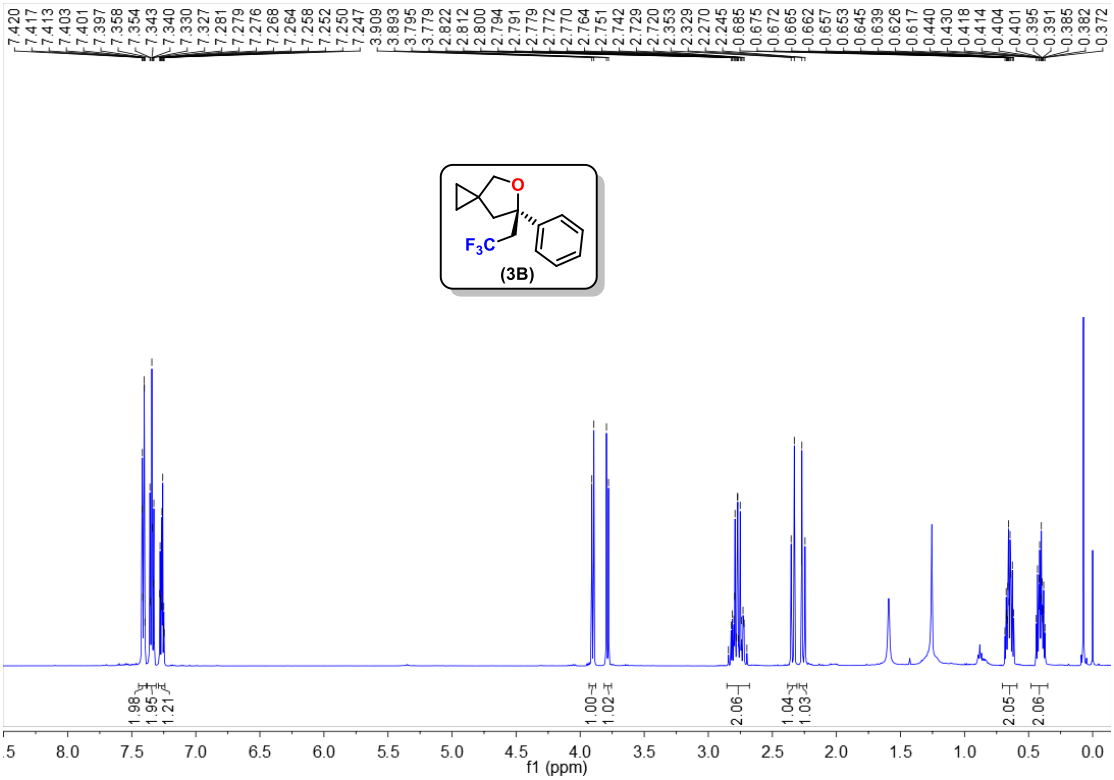
F3C
(3A)
0 -5 -15 -25 -35 -45 -55 -65 -75 -85 -95 -105 -115 -125 -135 -145
f1 (ppm)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3.909 3.893 3.795 3.779 2.822 2.812 2.800 2.794 2.791 2.779 2.772 2.770 2.764 2.751 2.742 2.729 2.720 2.353 2.329 2.270 2.245 0.685 0.675 0.672 0.665 0.662 0.657 0.653 0.645 0.639 0.626 0.617 0.440 0.430 0.418 0.414 0.404 0.401 0.395 0.391 0.385 0.382 0.372
O
F3C
(3B)
1.98 1.95 1.21 1.00 1.02 2.06 1.04 1.03 2.05 2.06
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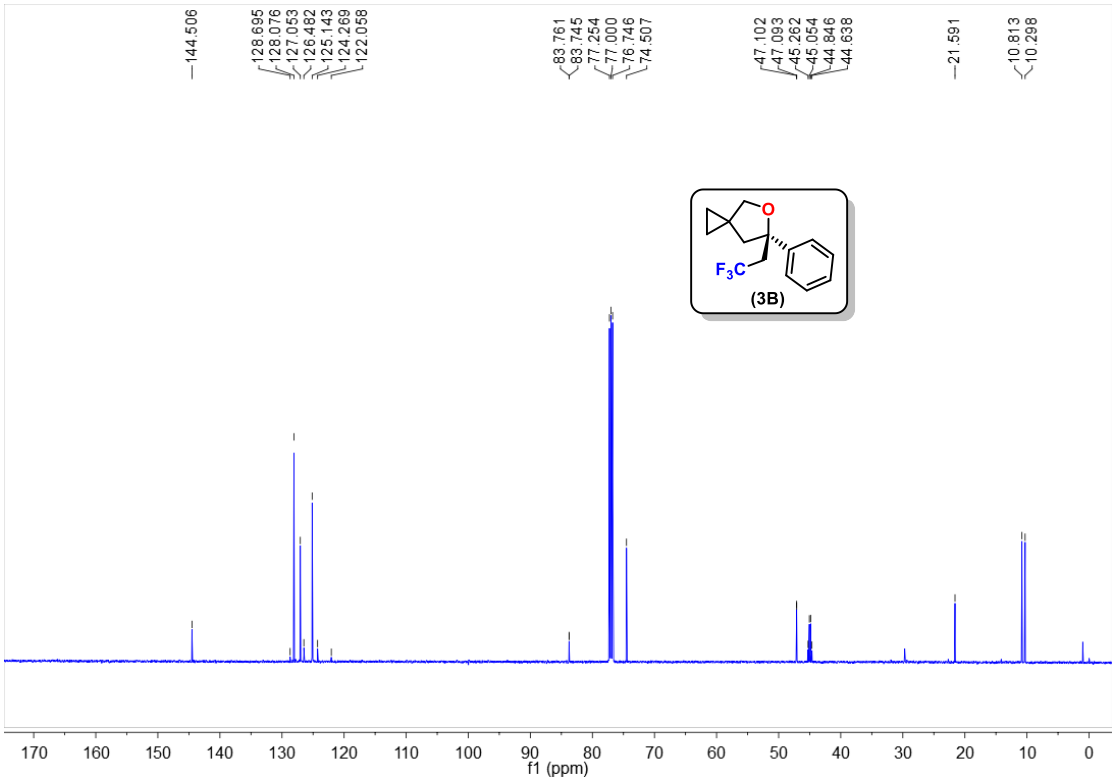
f1 (ppm)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47.093
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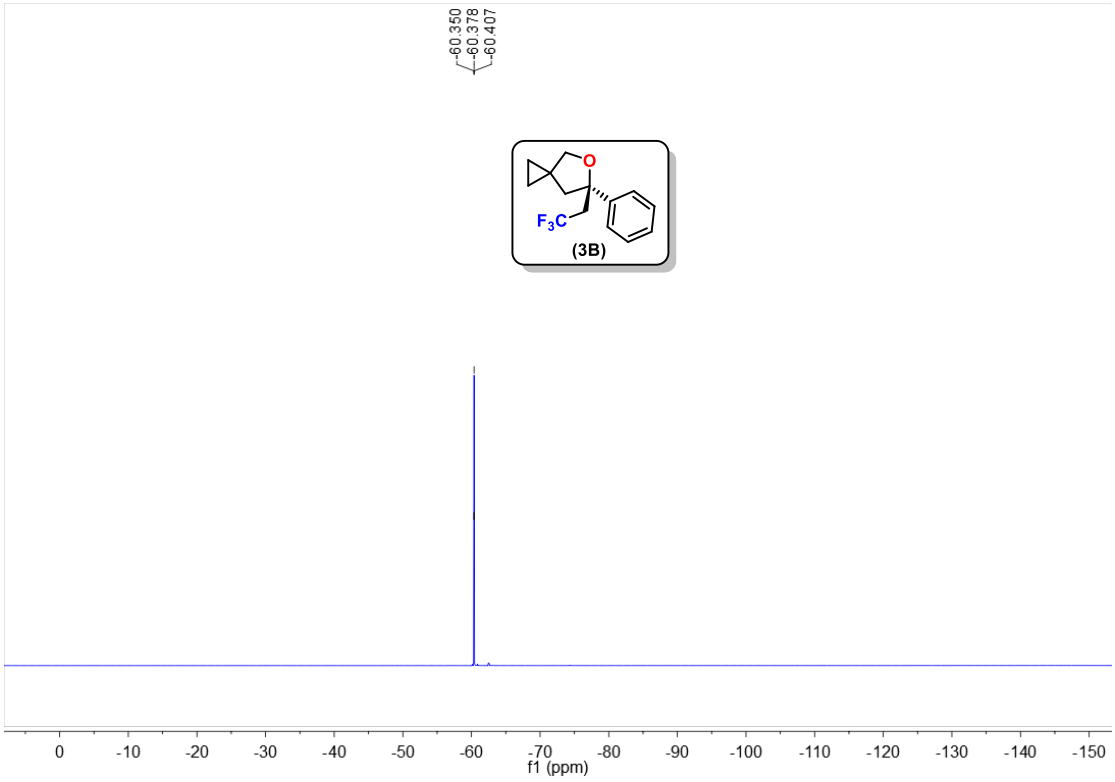
f1 (ppm)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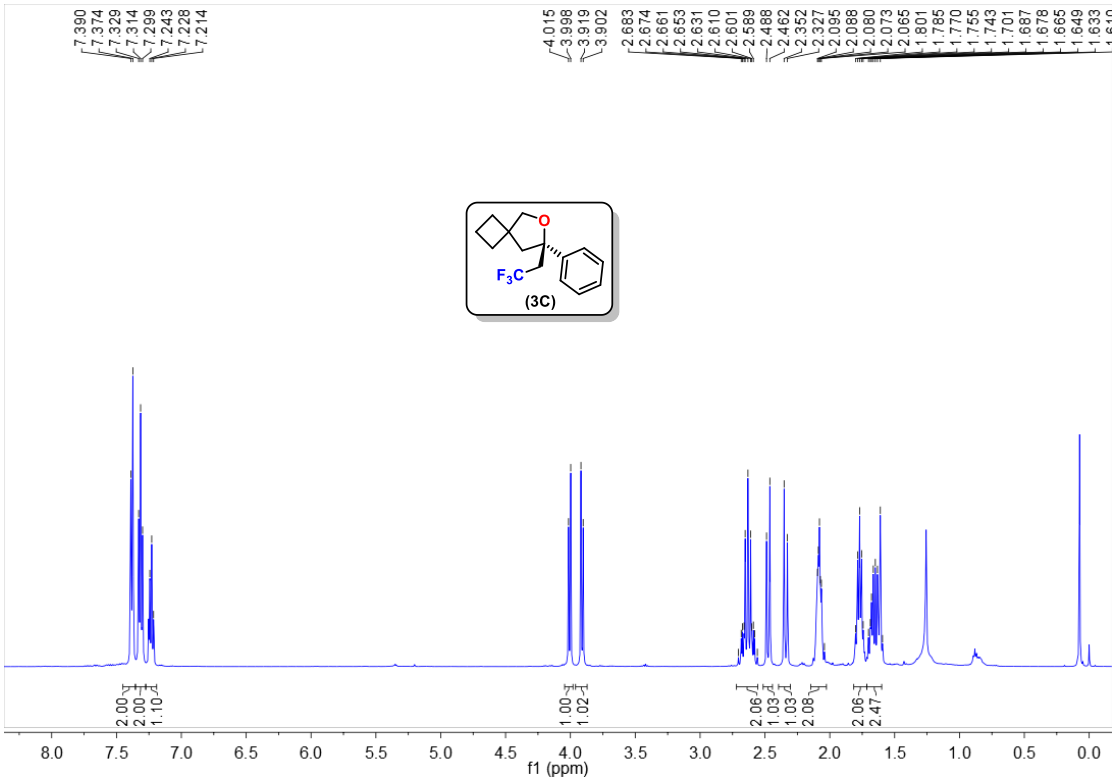
(3B)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)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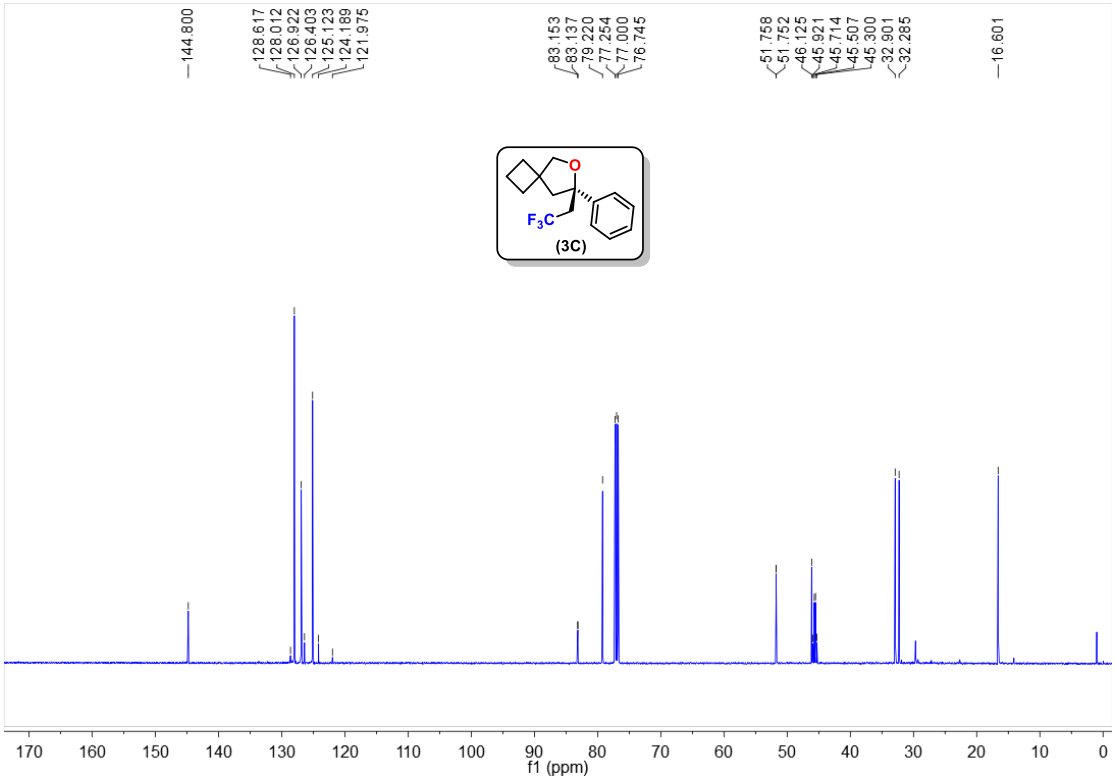
7.374
7.329
7.314
7.299
7.243
7.228
7.214
4.015
3.998
3.919
3.902
2.683
2.674
2.661
2.653
2.631
2.610
2.601
2.589
2.488
2.462
2.352
2.327
2.095
2.088
2.080
2.073
2.065
1.801
1.785
1.770
1.755
1.743
1.701
1.687
1.678
1.665
1.649
1.633
O
F3C
(3C)
2.00
2.00
1.10
1.00
1.02
2.06
1.03
1.03
2.08
2.06
2.47
f1 (ppm)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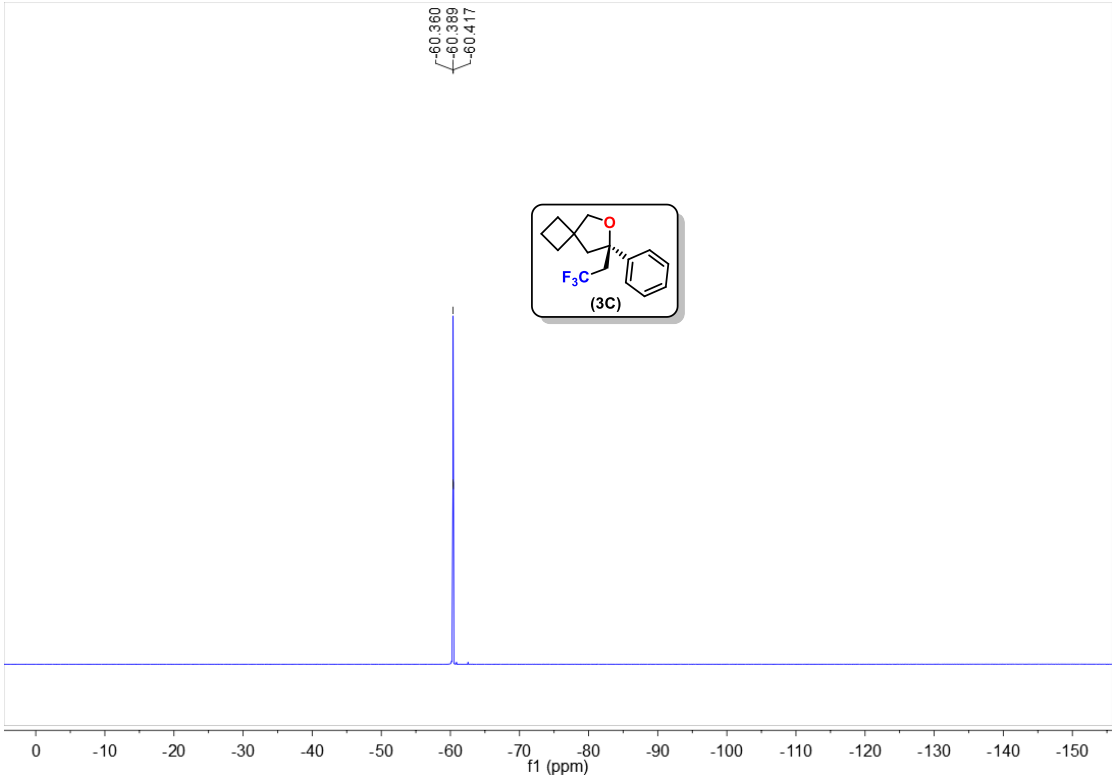
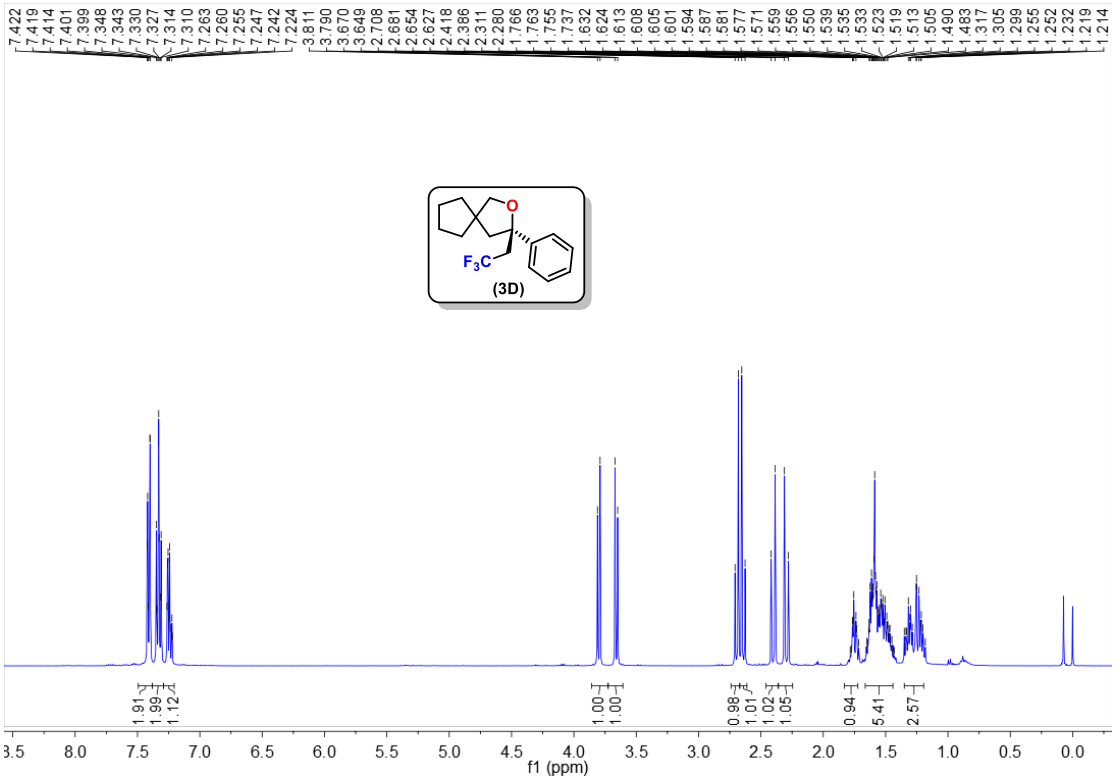
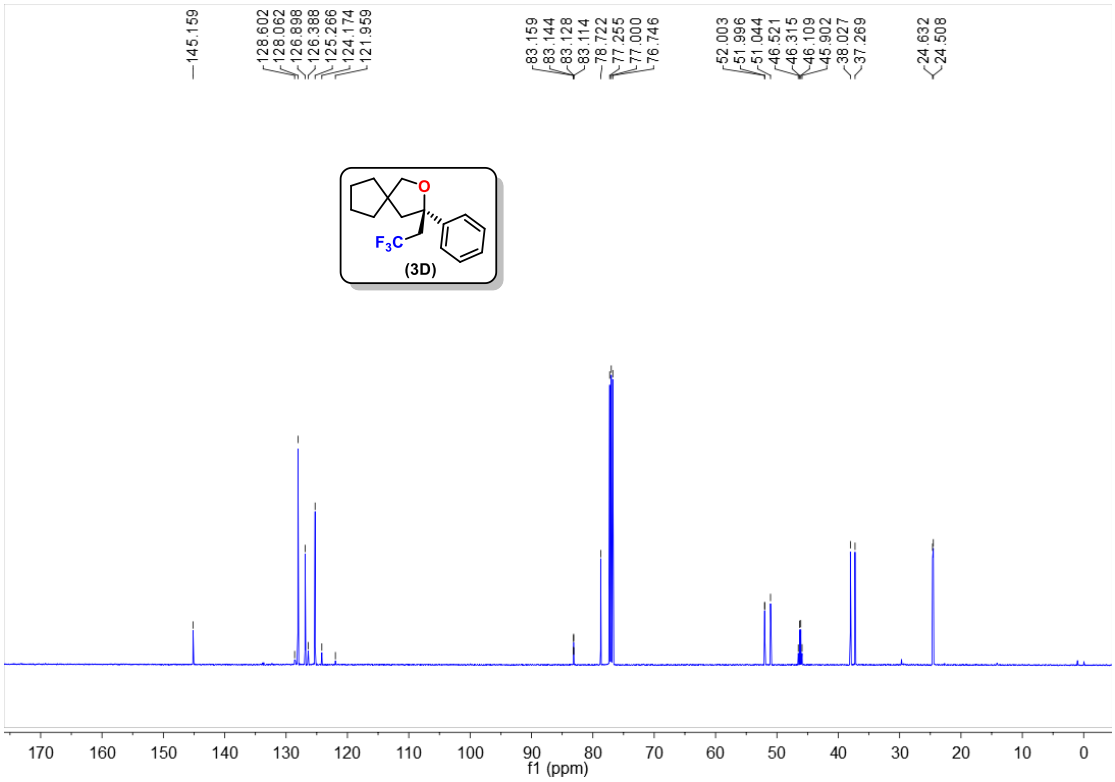
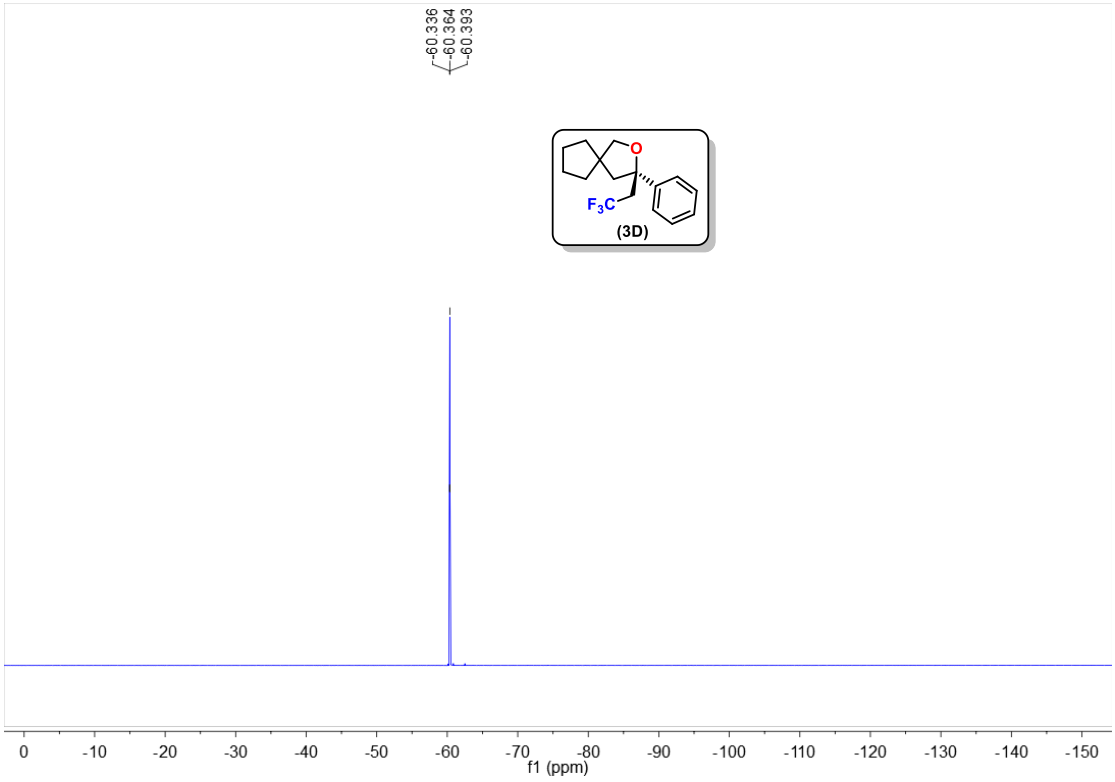
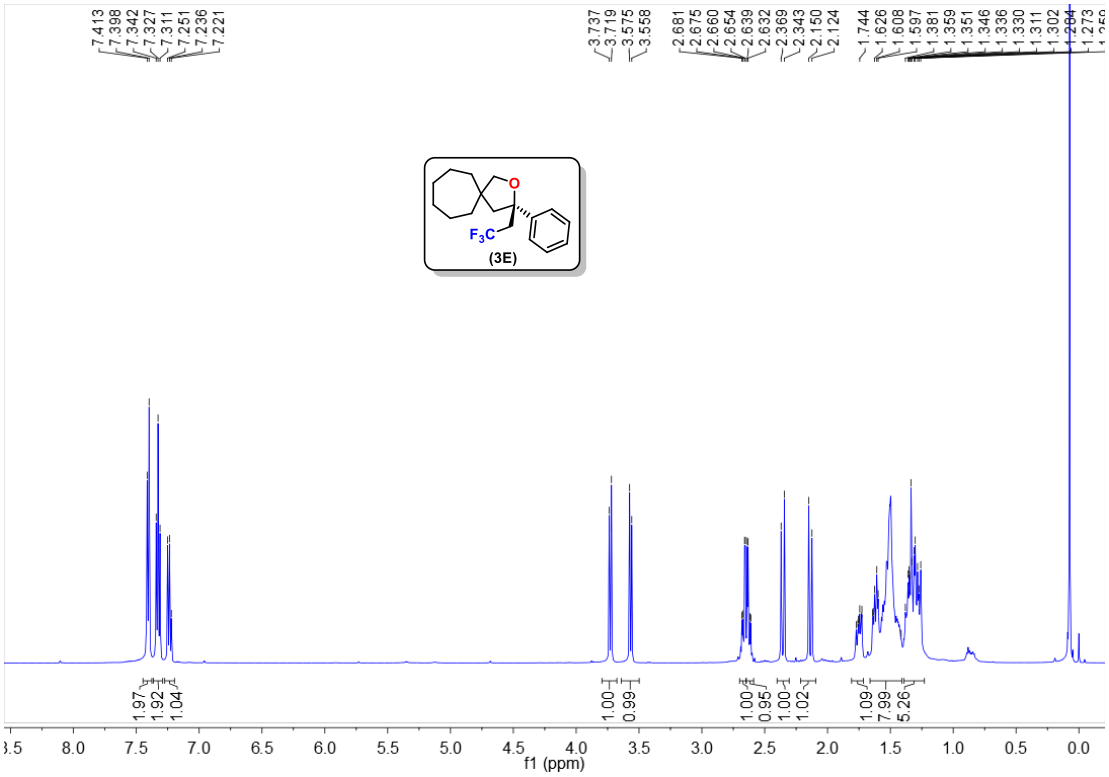
128.617
128.012
126.922
126.403
125.123
124.189
121.975
83.153
83.137
79.220
77.254
77.000
76.745
51.758
51.752
46.125
45.921
45.714
45.507
45.300
32.901
32.285
16.601
O
F3C
(3C)
f1 (ppm)

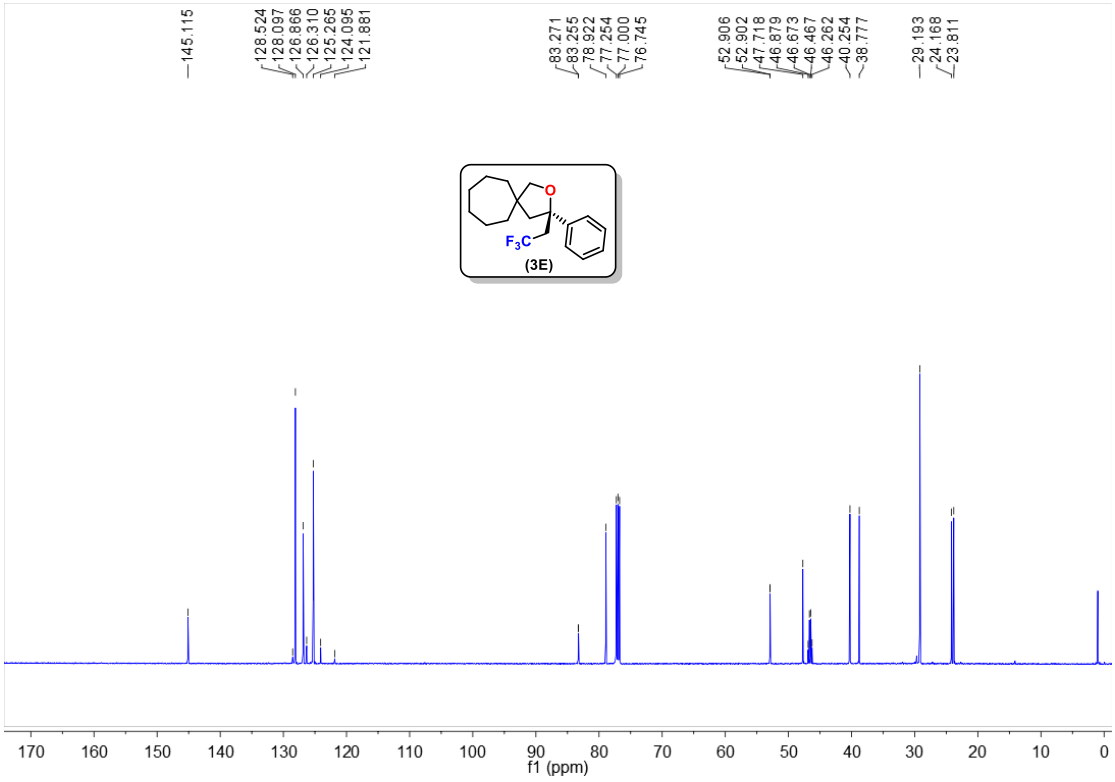

-60.360
-60.389
-60.417
(3C)
f1 (ppm)

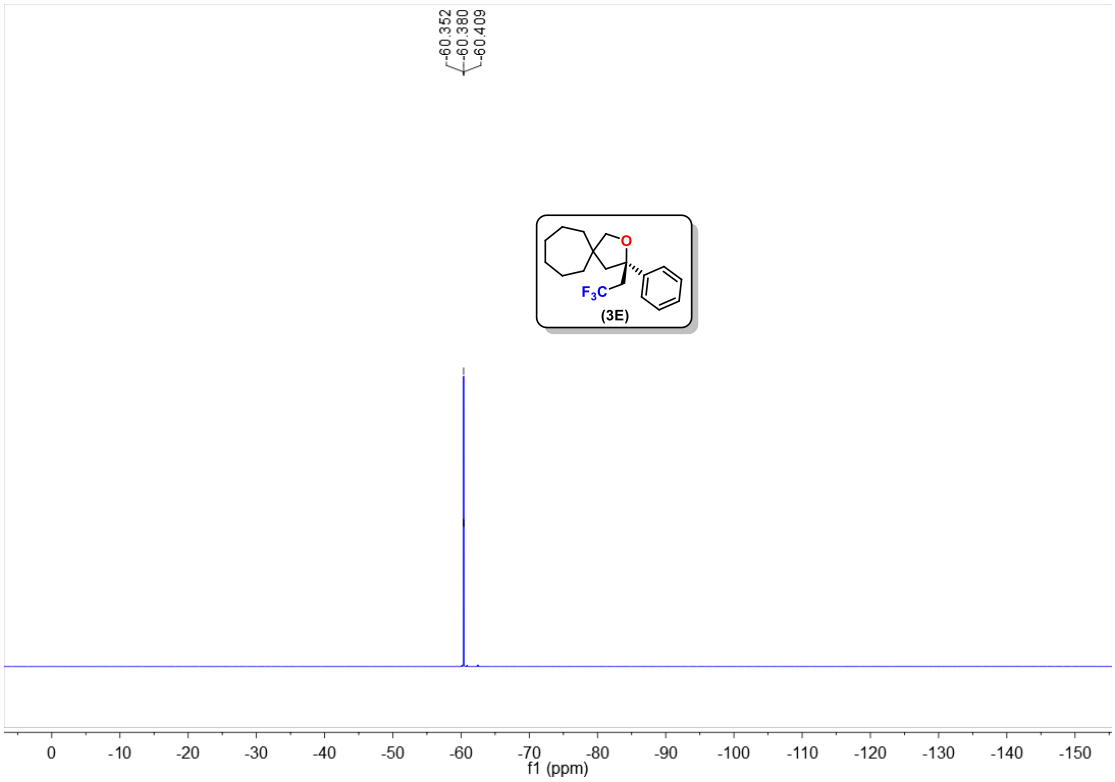

(3D)
1.91
1.99
1.12
1.00
1.00
0.98
1.01
1.02
1.05
0.94
5.41
2.57
f1 (ppm)

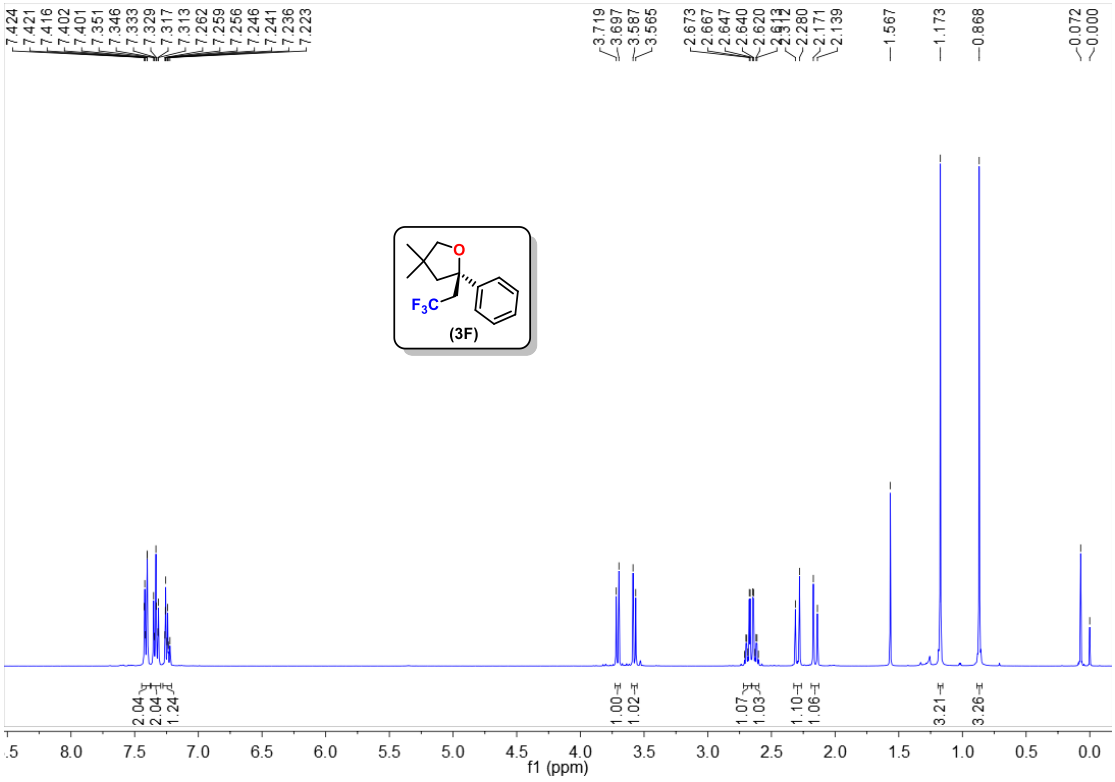

145.159
128.602
128.062
126.898
126.388
125.266
124.174
121.959
83.159
83.144
83.128
83.114
78.722
77.255
77.000
76.746
52.003
51.996
51.044
46.521
46.315
46.109
45.902
38.027
37.269
24.632
24.508
O
F3C
(3D)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

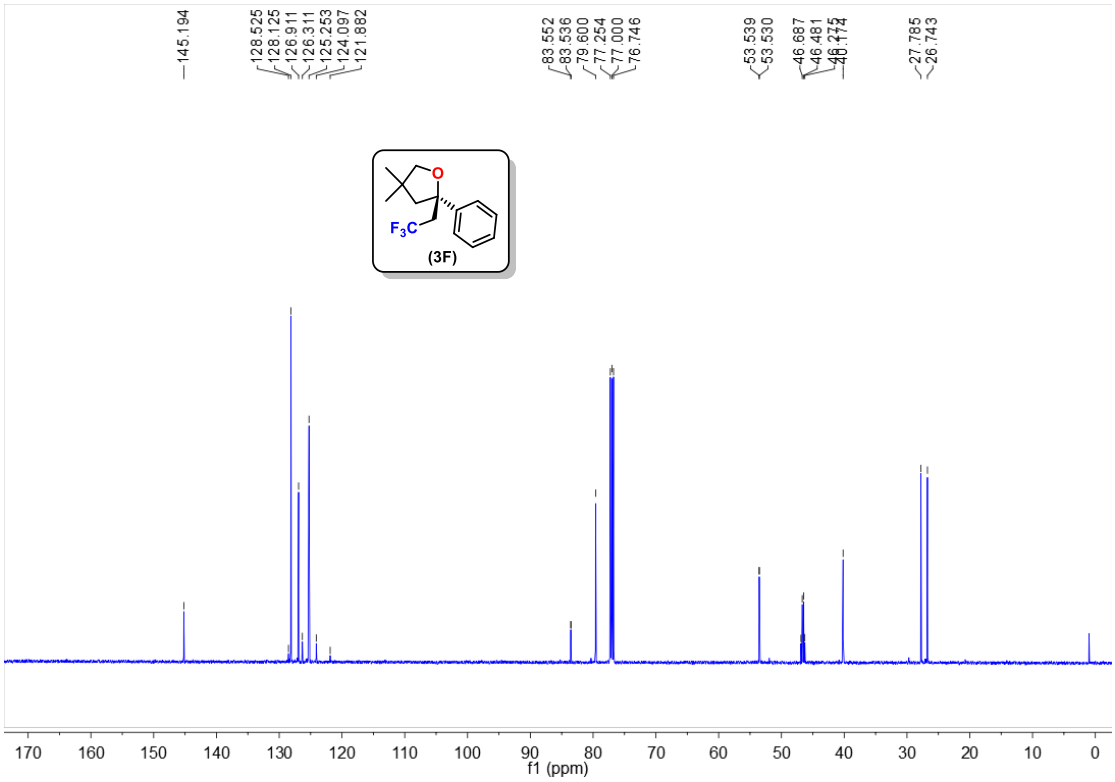

60.336
60.364
60.393
O
F3C
(3D)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)


7.413
7.398
7.342
7.327
7.311
7.251
7.236
7.221
3.737
3.719
3.575
3.558
2.681
2.675
2.660
2.654
2.639
2.632
2.369
2.343
2.150
2.124
1.744
1.626
1.608
1.597
1.381
1.359
1.351
1.346
1.336
1.330
1.311
1.302
1.284
1.273
O
F3C
(3E)
1.97
1.92
1.04
1.00
0.99
1.00
0.95
1.00
1.02
1.09
7.99
5.26
f1 (ppm)


145.115
128.524
128.097
126.866
126.310
125.265
124.095
121.881
83.271
83.255
78.922
77.254
77.000
76.745
52.906
52.902
47.718
46.879
46.673
46.467
46.262
40.254
38.777
29.193
24.168
23.811
O
F3C
(3E)
f1 (ppm)


-60.352
-60.380
-60.409
O
F3C
(3E)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)


7.424
7.421
7.416
7.402
7.401
7.351
7.346
7.333
7.329
7.317
7.313
7.262
7.259
7.256
7.246
7.241
7.236
7.223
3.719
3.697
3.587
3.565
2.673
2.667
2.647
2.640
2.620
2.280
2.171
2.139
1.567
1.173
0.868
0.072
0.000
O
F3C
(3F)
2.04
2.04
1.24
1.00
1.02
1.07
1.03
1.10
1.06
3.21
3.26
f1 (ppm)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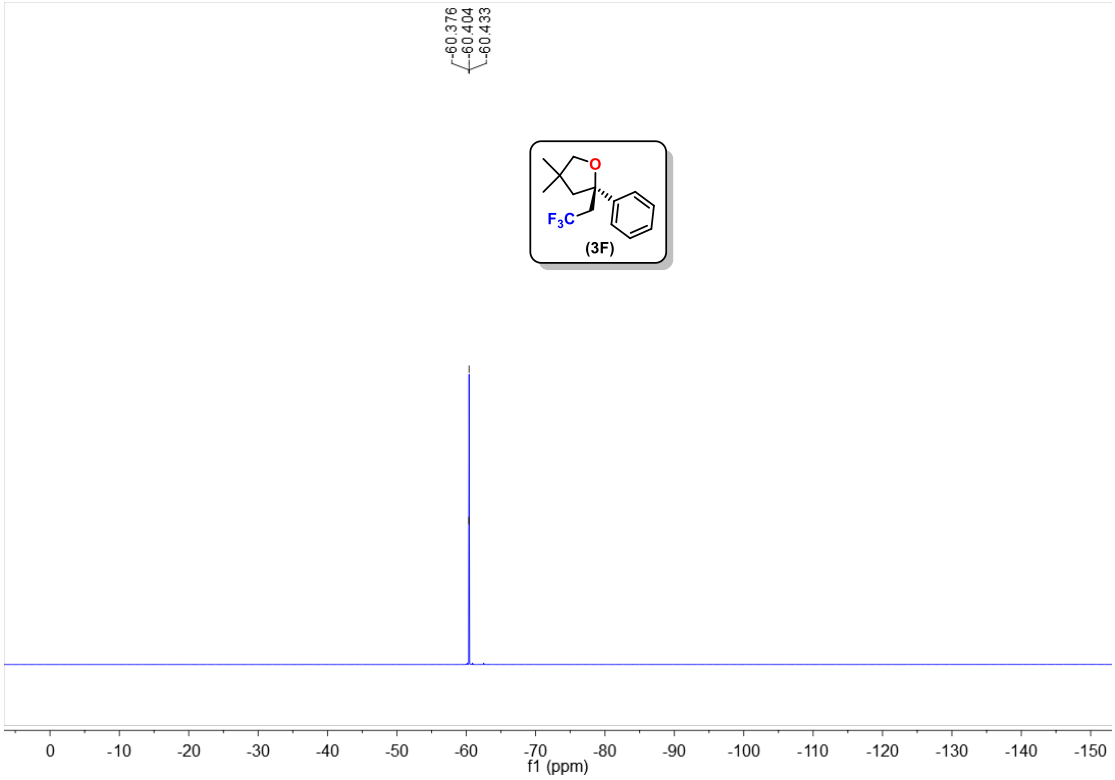
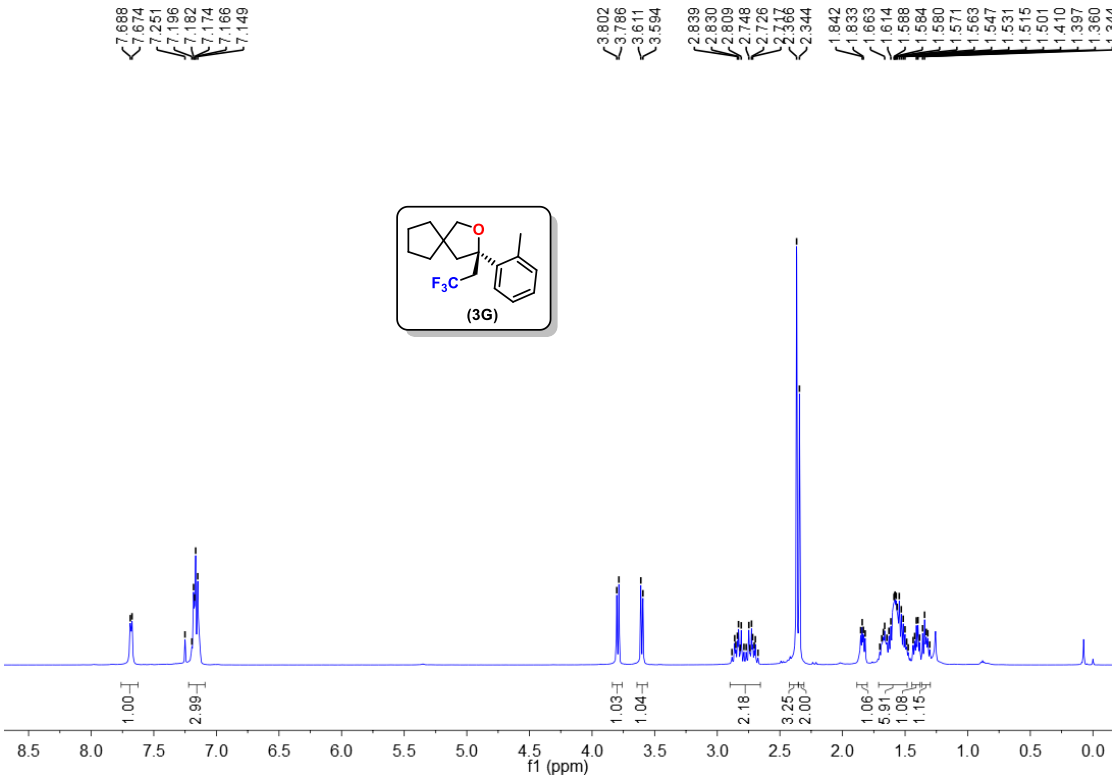
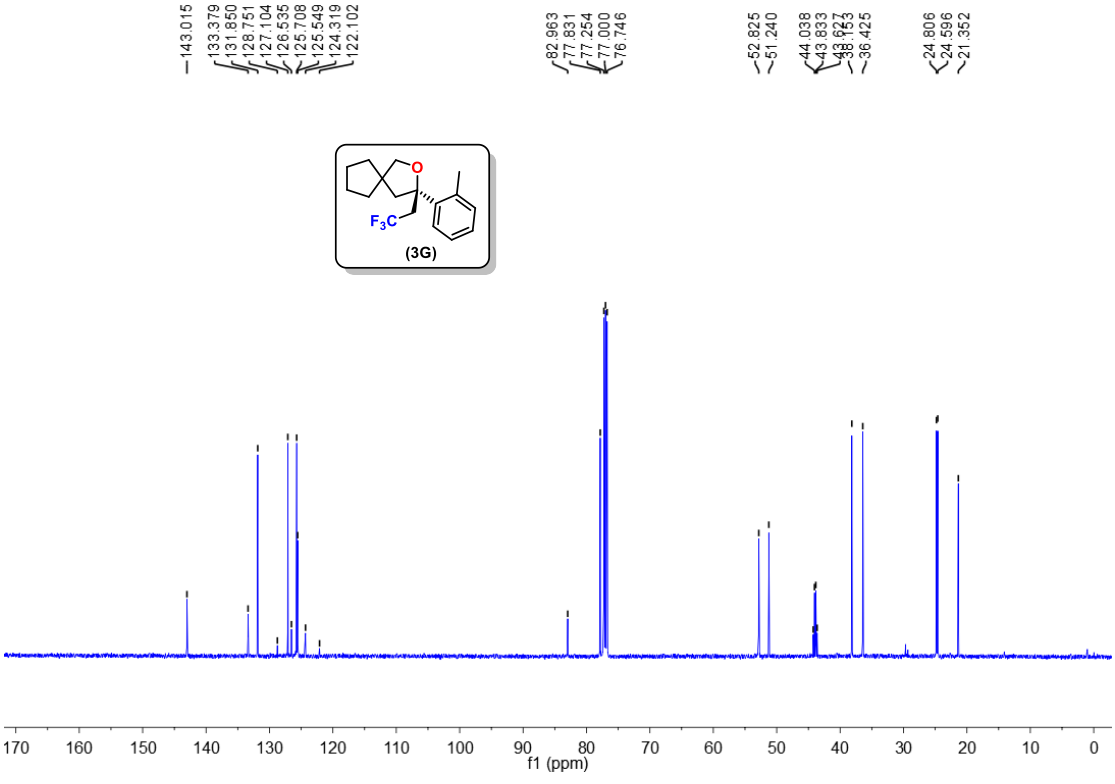
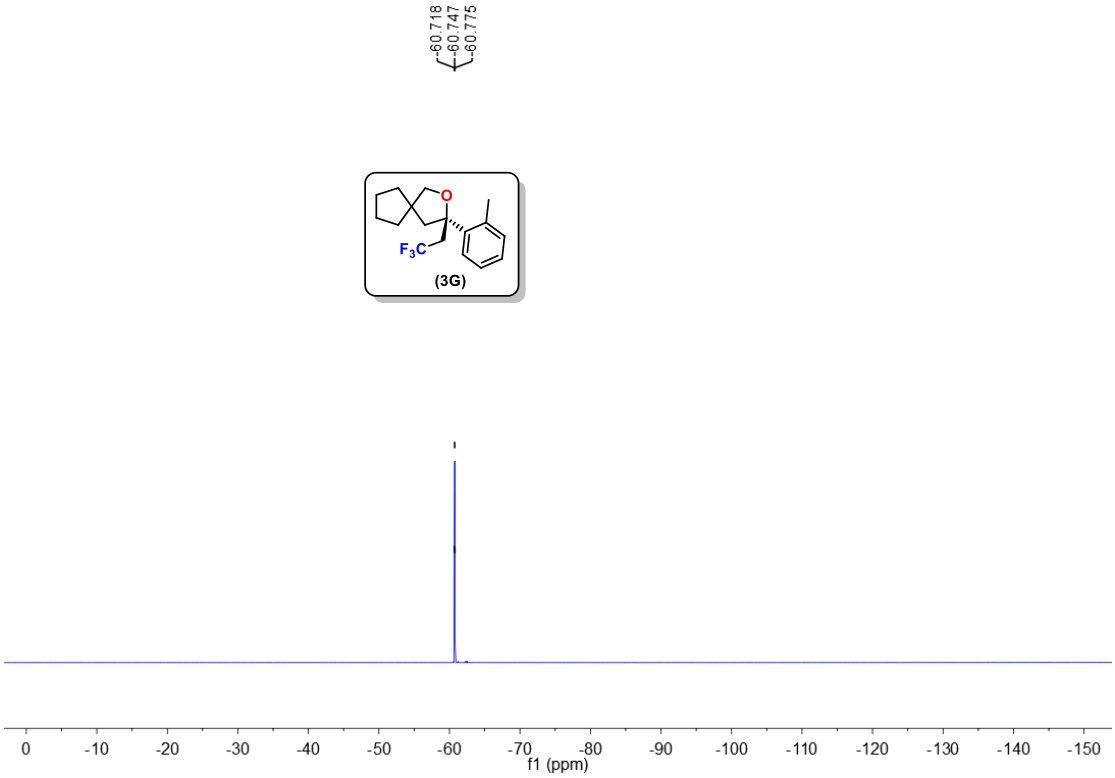
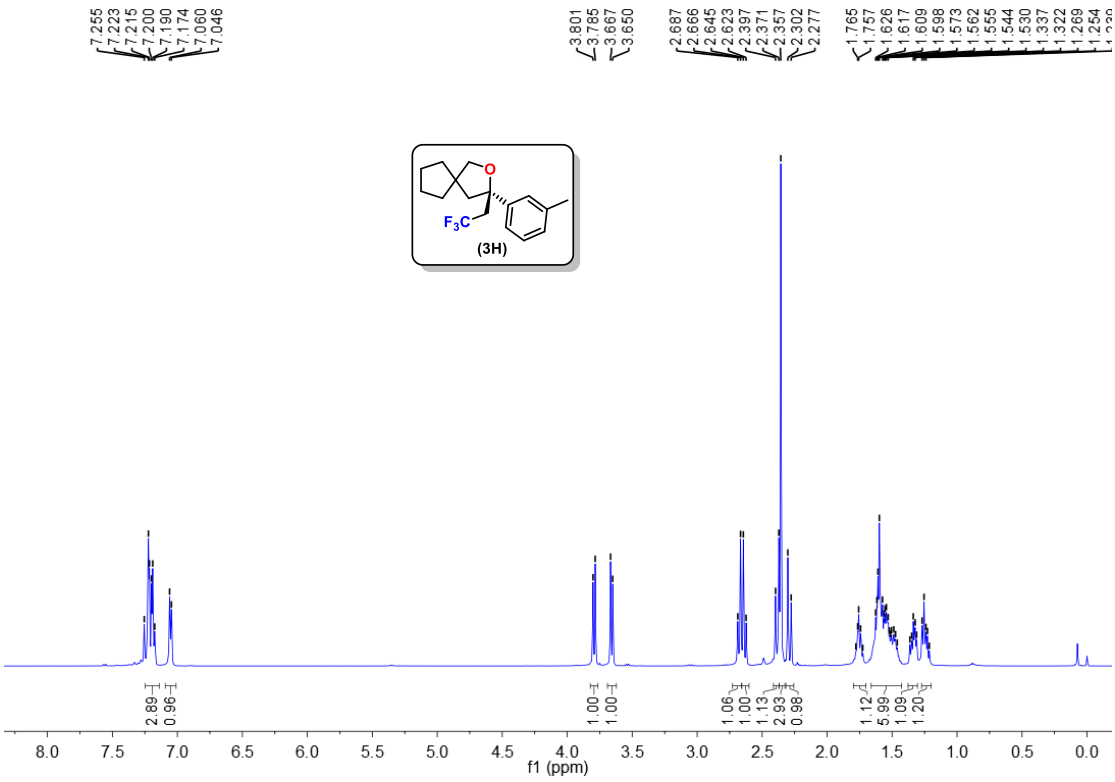
128.525
128.125
126.911
126.311
125.253
124.097
121.882
83.552
83.536
79.600
77.254
77.000
76.746
53.539
53.530
46.687
46.481
46.275
46.174
27.785
26.743
O
F3C
(3F)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

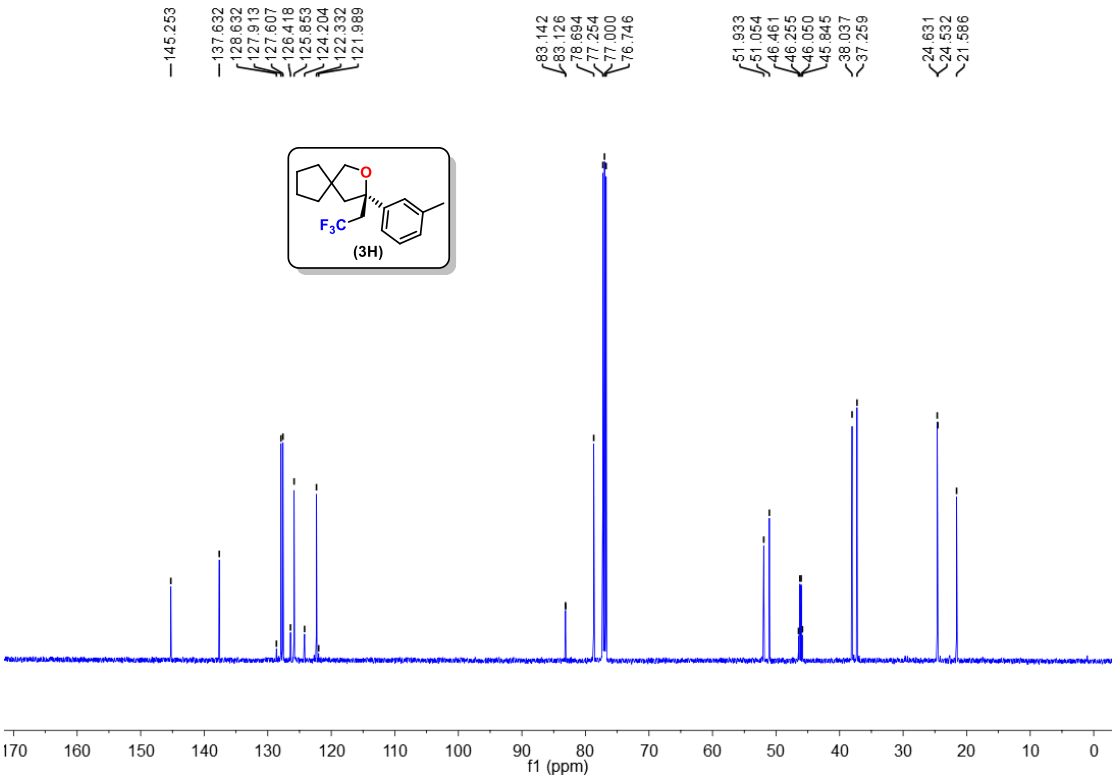

60.376
60.404
60.433
O
F3C
(3F)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)


7.688
7.674
7.251
7.196
7.182
7.174
7.166
7.149
3.802
3.786
3.611
3.594
2.839
2.830
2.809
2.748
2.726
2.717
2.366
2.344
1.842
1.833
1.663
1.614
1.588
1.584
1.580
1.571
1.563
1.547
1.531
1.515
1.501
1.410
1.397
1.360
O
F3C
(3G)
1.00
2.99
1.03
1.04
2.18
3.25
2.00
1.06
5.91
1.08
1.15
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)


143.015
133.379
131.850
128.751
127.104
126.535
125.708
125.549
124.319
122.102
82.963
77.831
77.254
77.000
76.746
52.825
51.240
44.038
43.833
43.627
38.153
36.425
24.806
24.596
21.352
O
F3C
(3G)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-60.718
-60.747
-60.775
O
F3C
(3G)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)


7.255
7.223
7.215
7.200
7.190
7.174
7.060
7.046
3.801
3.785
3.667
3.650
2.687
2.666
2.645
2.623
2.397
2.371
2.357
2.302
2.277
1.765
1.757
1.626
1.617
1.609
1.598
1.573
1.562
1.555
1.544
1.530
1.337
1.322
1.269
1.254
O
F3C
(3H)
2.89
0.96
1.00
1.00
1.06
1.00
1.13
2.93
0.98
1.12
5.99
1.09
1.20
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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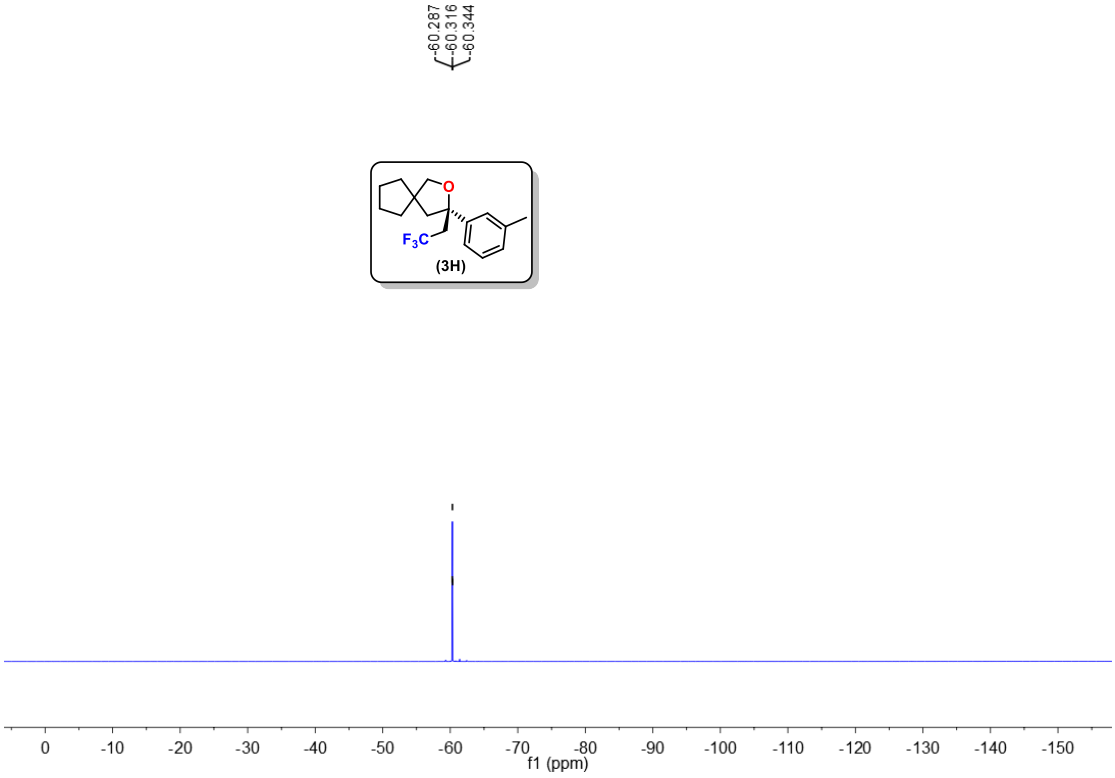
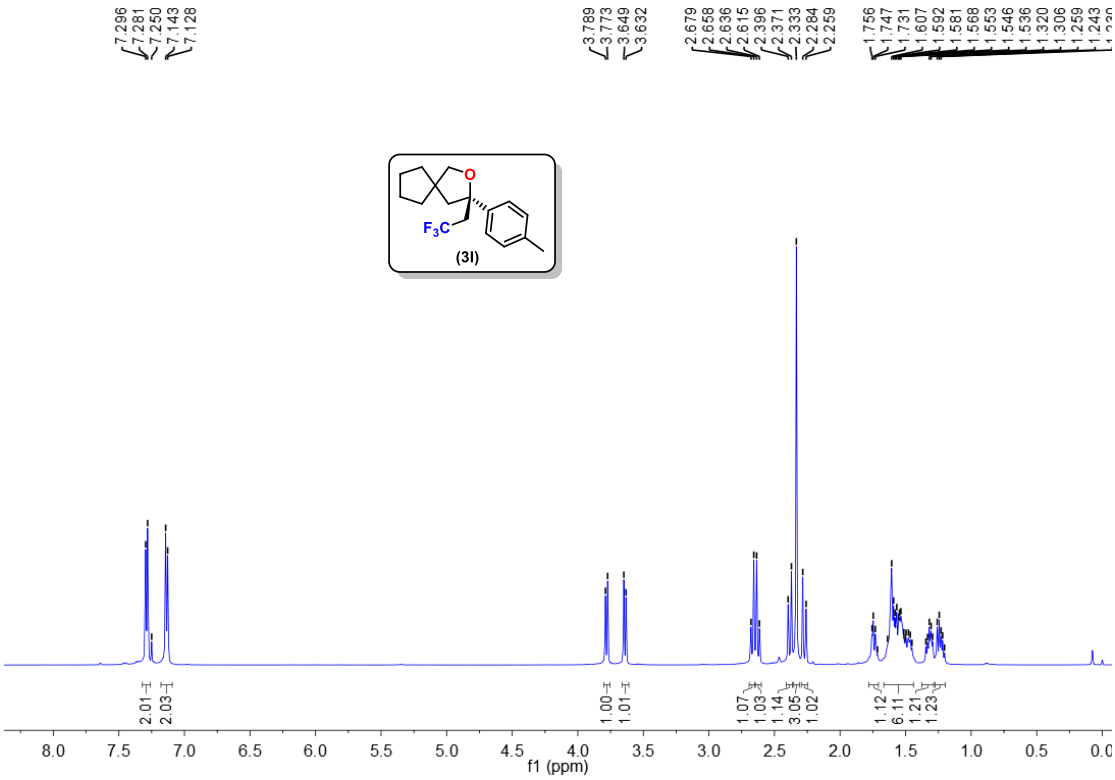
128.632
127.913
127.607
126.418
125.853
124.204
122.332
121.989
83.142
83.126
78.894
77.254
77.000
76.746
51.933
51.054
46.461
46.255
46.050
45.845
38.037
37.259
24.631
24.532
21.586
O
F3C
(3H)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-60.287
-60.316
-60.344
O
F3C
(3H)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)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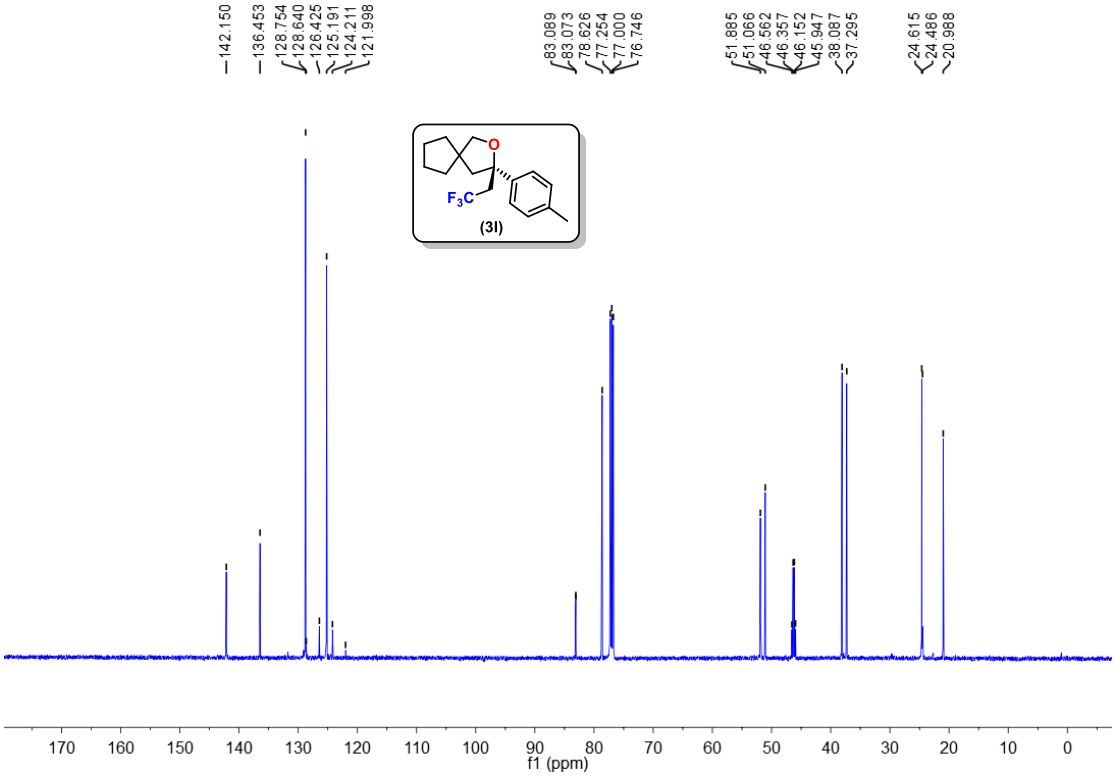
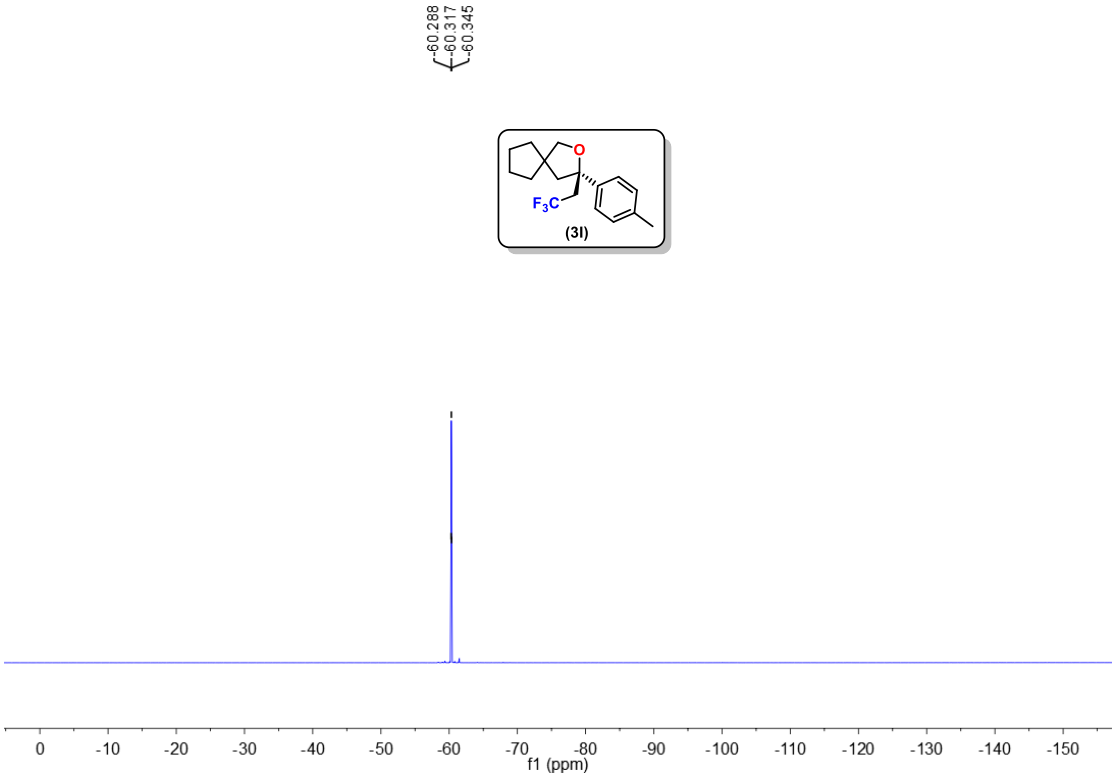
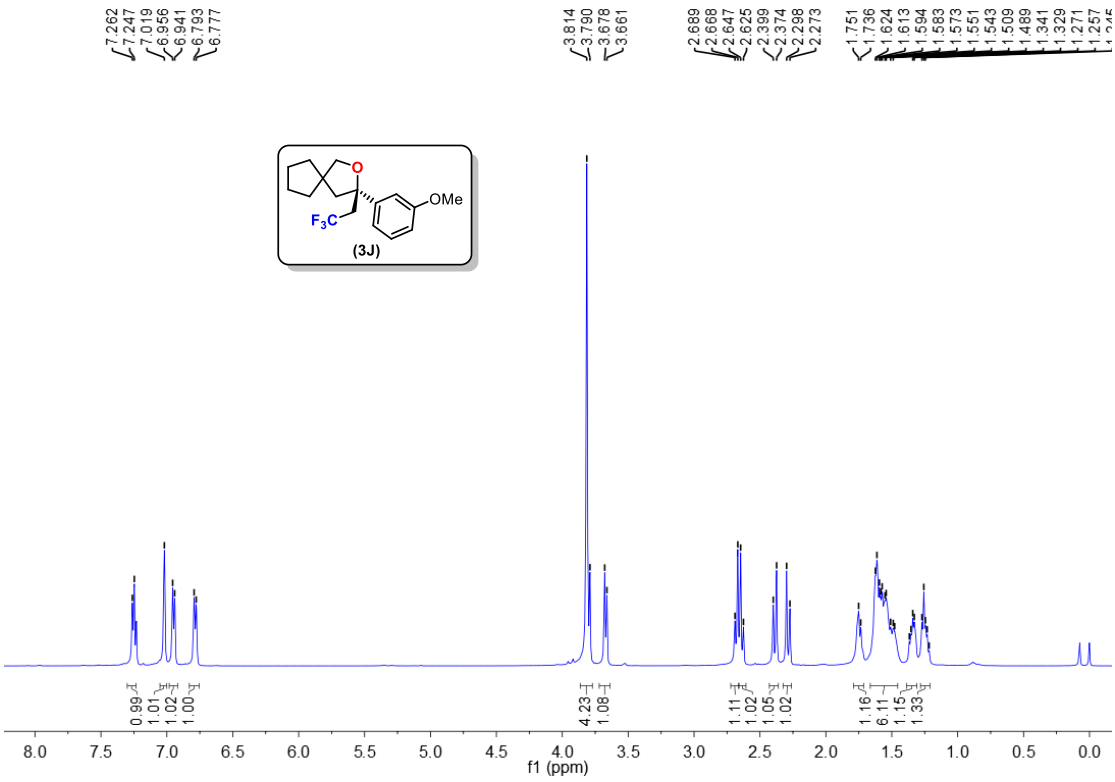
7.250
7.143
7.128
3.789
3.773
3.649
3.632
2.679
2.658
2.636
2.615
2.396
2.371
2.333
2.284
2.259
1.756
1.747
1.731
1.607
1.592
1.581
1.568
1.553
1.546
1.536
1.320
1.306
1.259
1.243
O
F3C
(3l)
2.01
2.03
1.00
1.01
1.07
1.03
1.14
3.05
1.02
1.12
6.11
1.21
1.23
f1 (ppm)


142.150
136.453
128.754
128.640
126.425
125.191
124.211
121.998
83.089
83.073
78.626
77.254
77.000
76.746
51.885
51.066
46.562
46.357
46.152
45.947
38.087
37.295
24.615
24.486
20.988
O
F3C
(3l)
f1 (ppm)


-60.288
-60.317
-60.345
O
F3C
(3I)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)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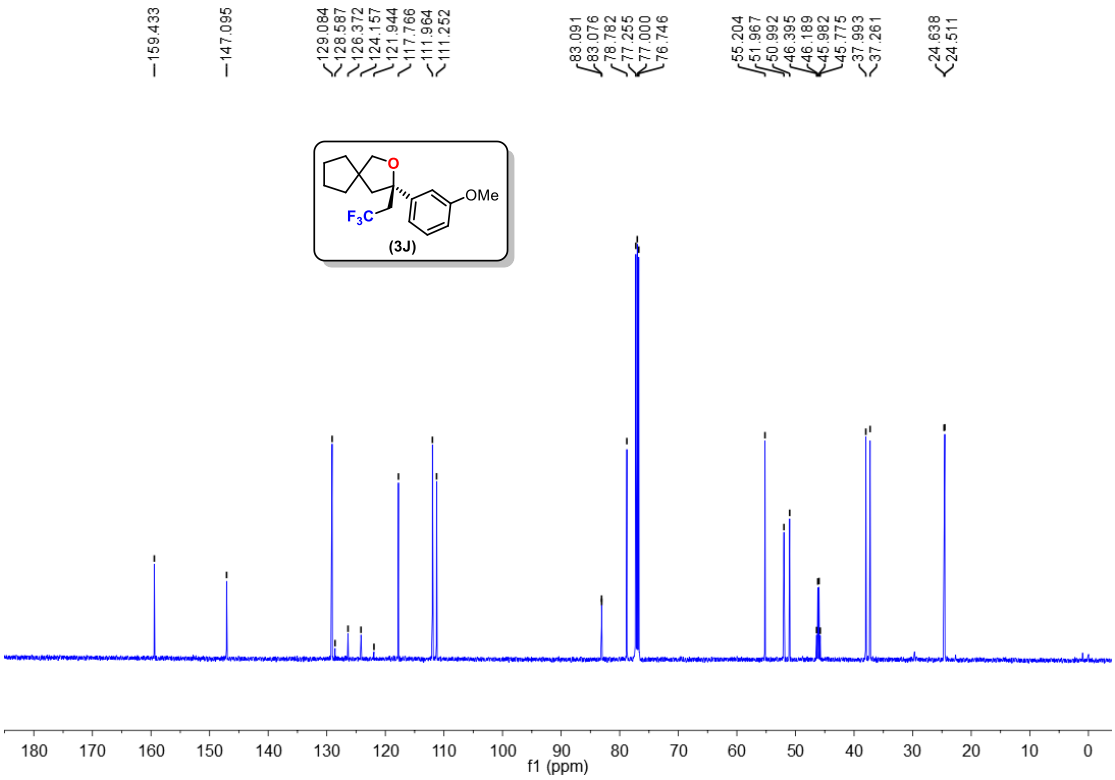
6.956
6.941
6.793
6.777
3.814
3.790
3.678
3.661
2.689
2.668
2.647
2.625
2.399
2.374
2.298
2.273
1.751
1.736
1.624
1.613
1.594
1.583
1.573
1.551
1.543
1.509
1.489
1.341
1.329
1.271
1.257
O
OMe
F3C
(3J)
0.99
1.01
1.02
1.00
4.23
1.08
1.11
1.02
1.05
1.02
1.16
6.11
1.15
1.33
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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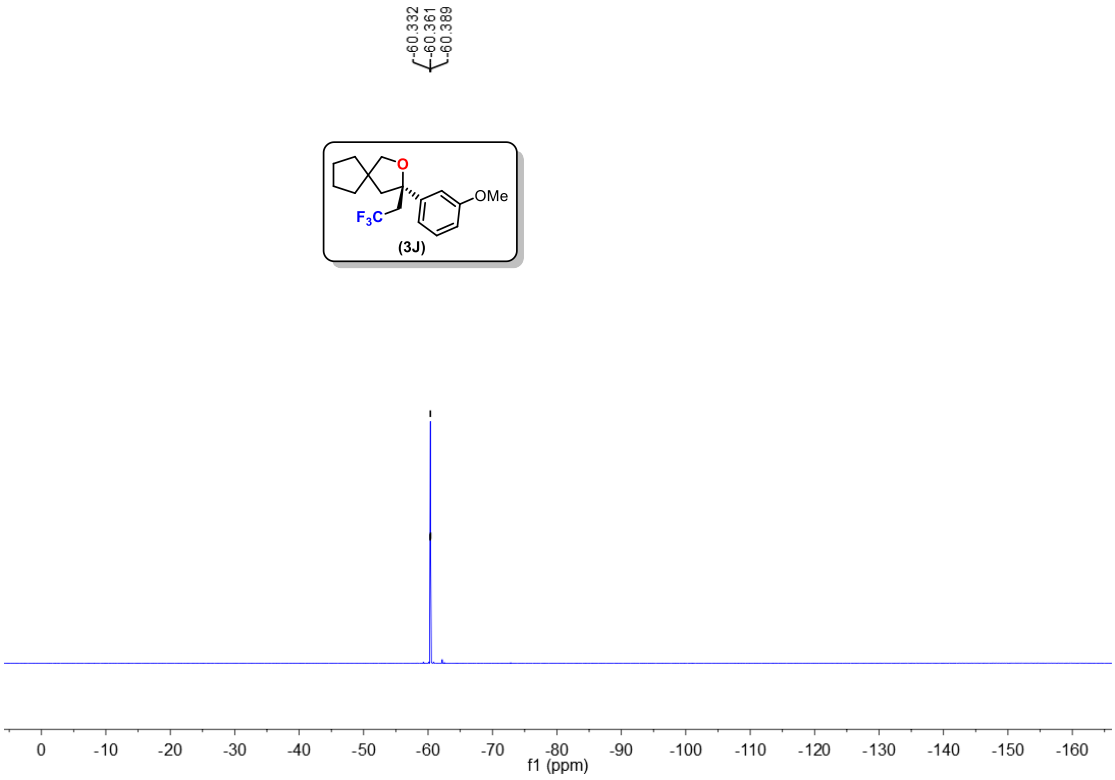
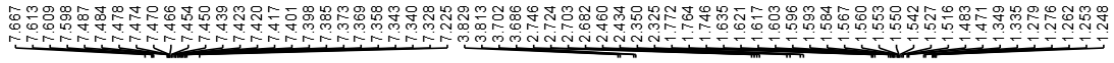
128.587
126.372
124.157
121.944
117.766
111.964
111.252
83.091
83.076
78.782
77.255
77.000
76.746
55.204
51.967
50.992
46.395
46.189
45.982
45.775
37.993
37.261
24.638
24.511
O
OMe
F3C
(3J)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-60.332
-60.361
-60.389
O
OMe
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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7.613
7.609
7.598
7.487
7.484
7.478
7.474
7.470
7.466
7.454
7.450
7.439
7.423
7.420
7.417
7.401
7.398
7.385
7.373
7.369
7.358
7.343
7.340
7.328
7.225
3.829
3.813
3.702
3.686
2.746
2.724
2.703
2.682
2.460
2.434
2.350
2.325
1.772
1.764
1.746
1.635
1.621
1.617
1.603
1.596
1.593
1.584
1.567
1.560
1.553
1.550
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1.483
1.471
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1.335
1.279
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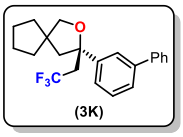
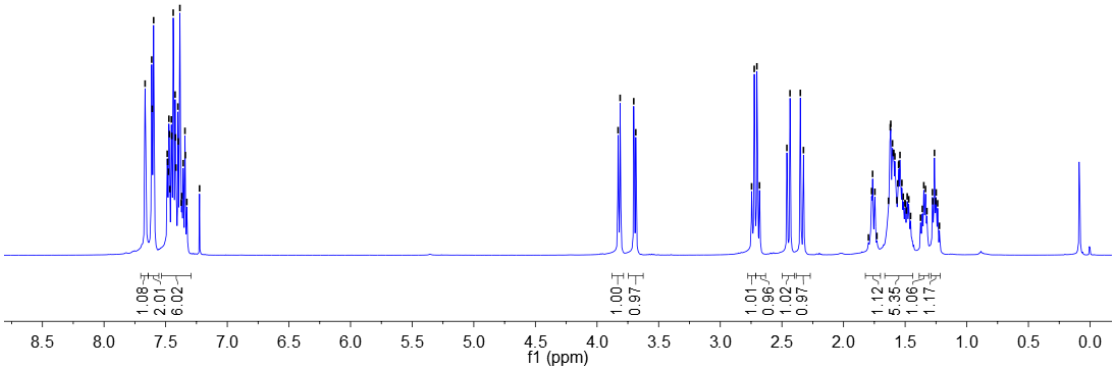
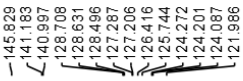
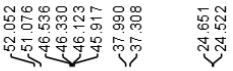
1.262
1.253
1.248

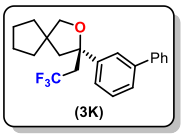

O
Ph
F3C
(3K)

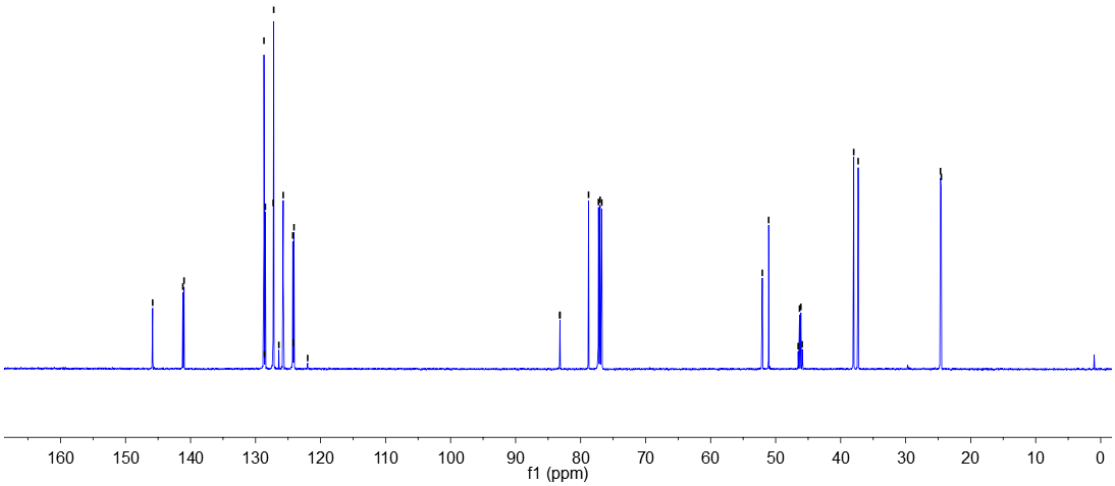

1.08
2.01
6.02
1.00
0.97
1.01
0.96
1.02
0.97
1.12
5.35
1.06
1.17
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

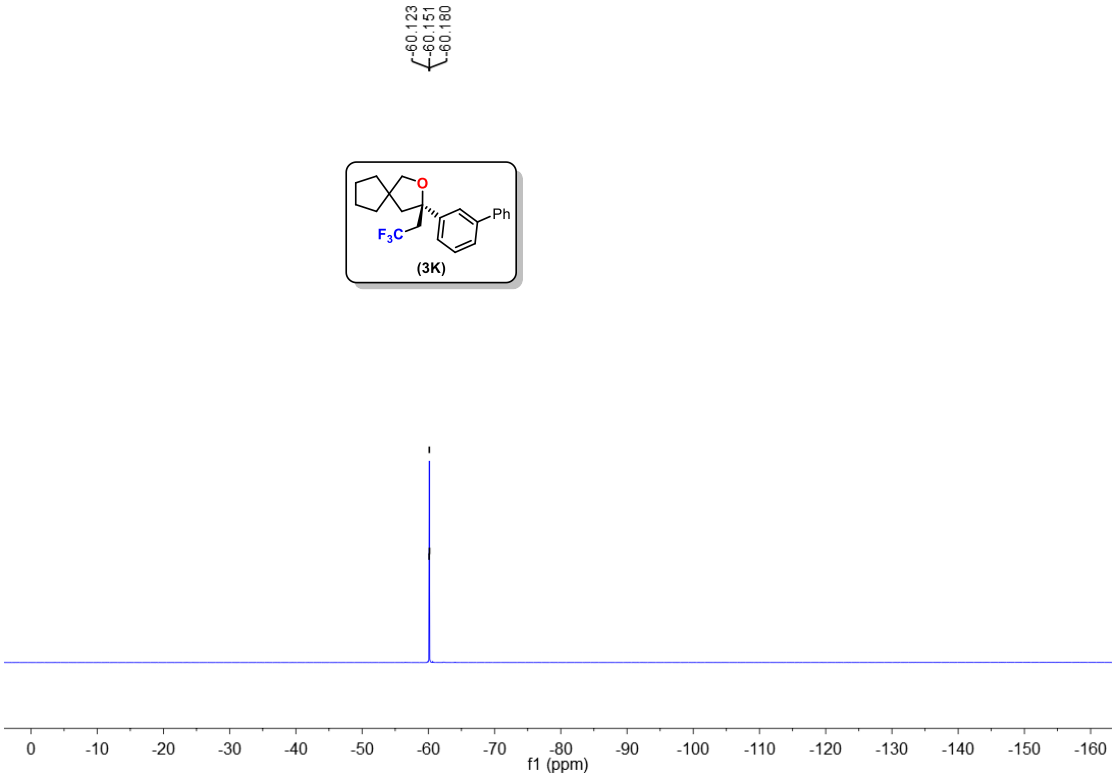

145.829
141.183
140.997
128.708
128.631
128.496
127.287
127.206
126.416
125.744
124.272
124.201
124.087
121.986

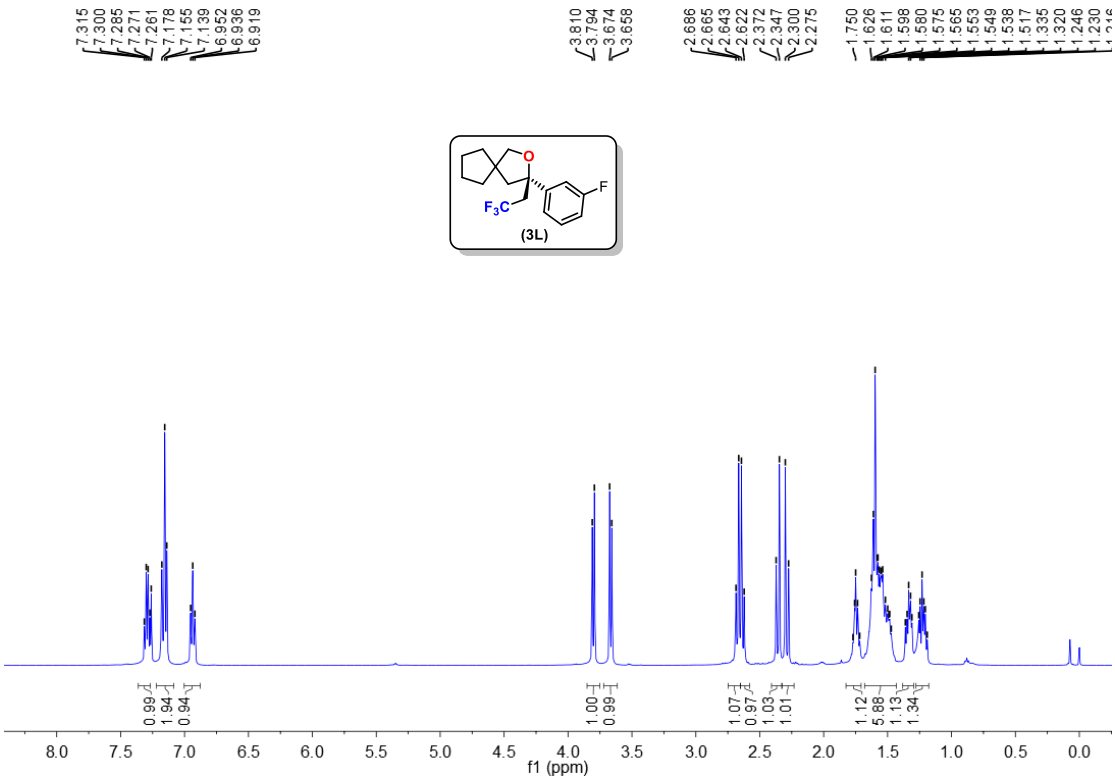

83.195
83.180
78.779
77.255
77.000
76.746

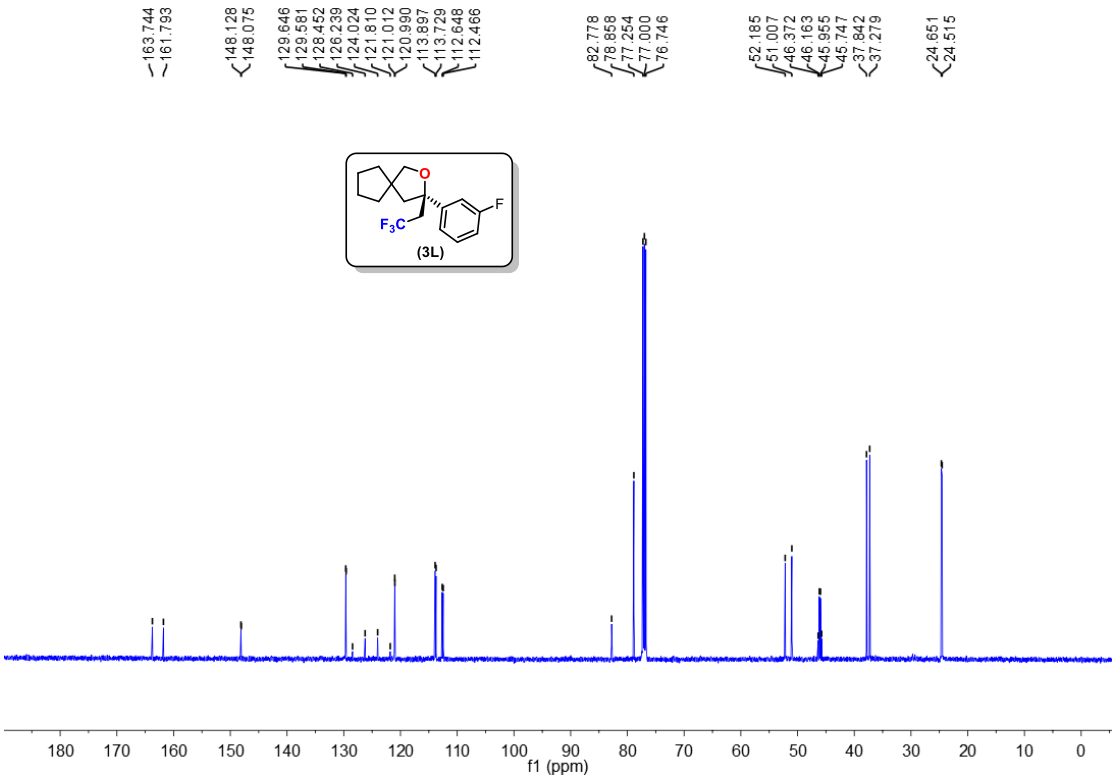

52.052
51.076
46.536
46.330
46.123
45.917
37.990
37.308
24.651
24.522

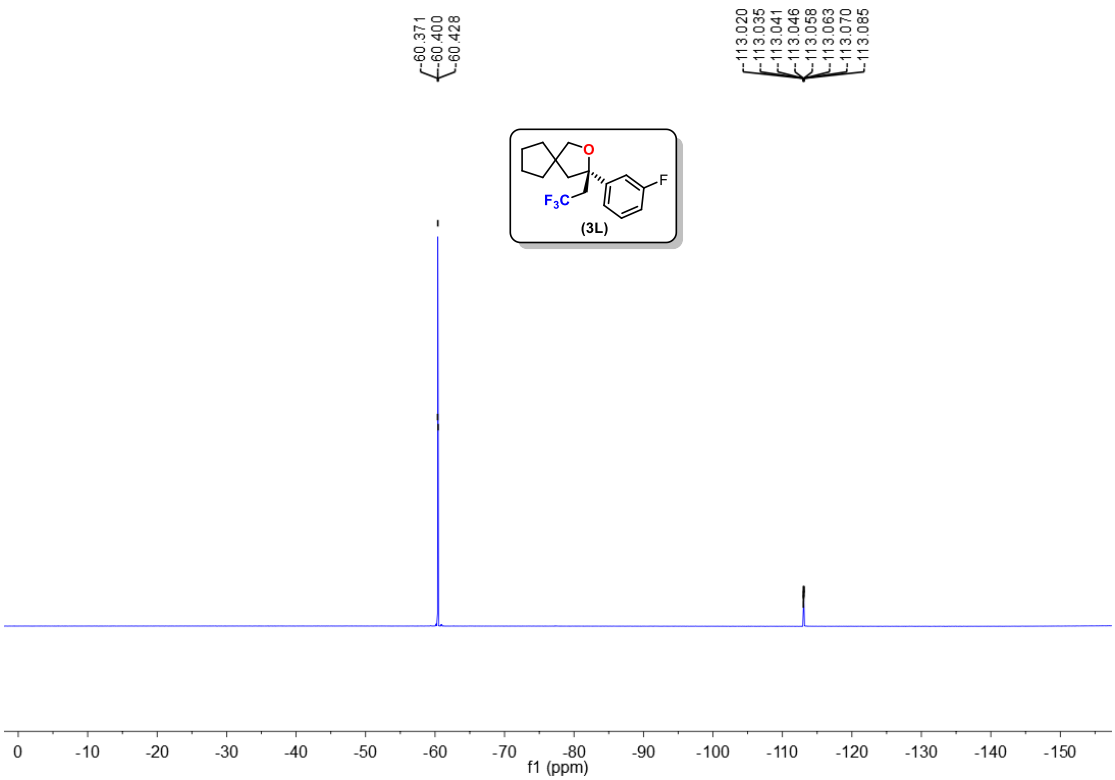

O
Ph
F3C
(3K)

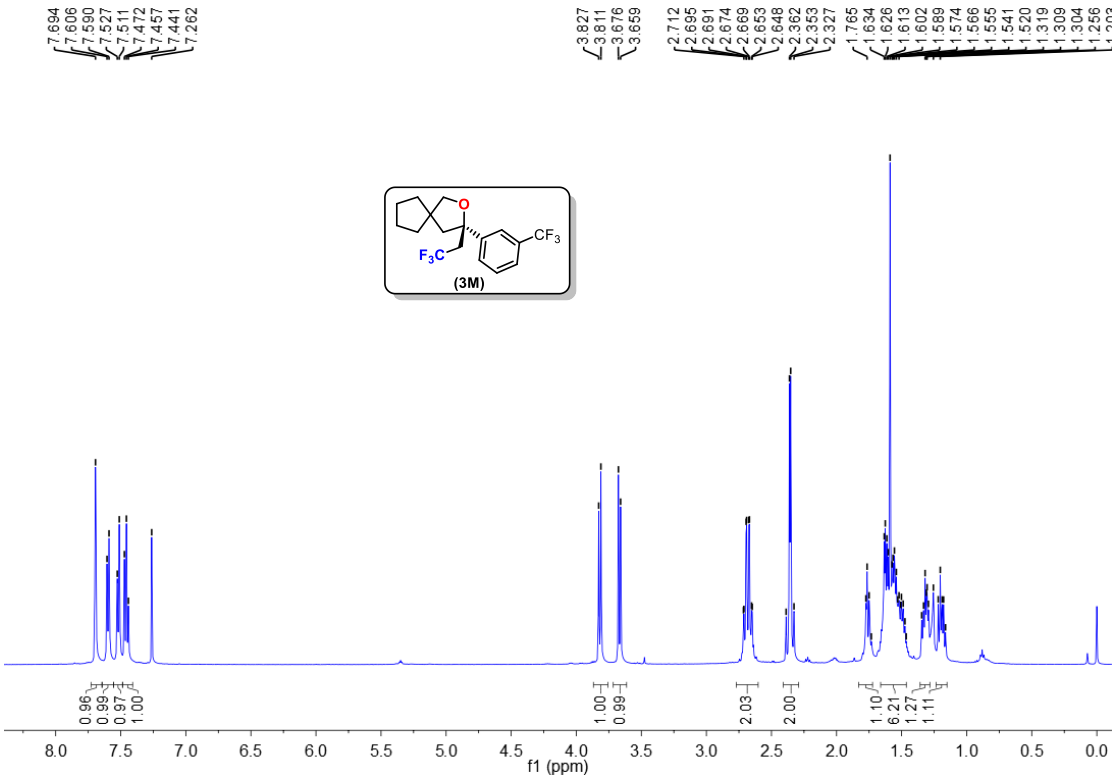

160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

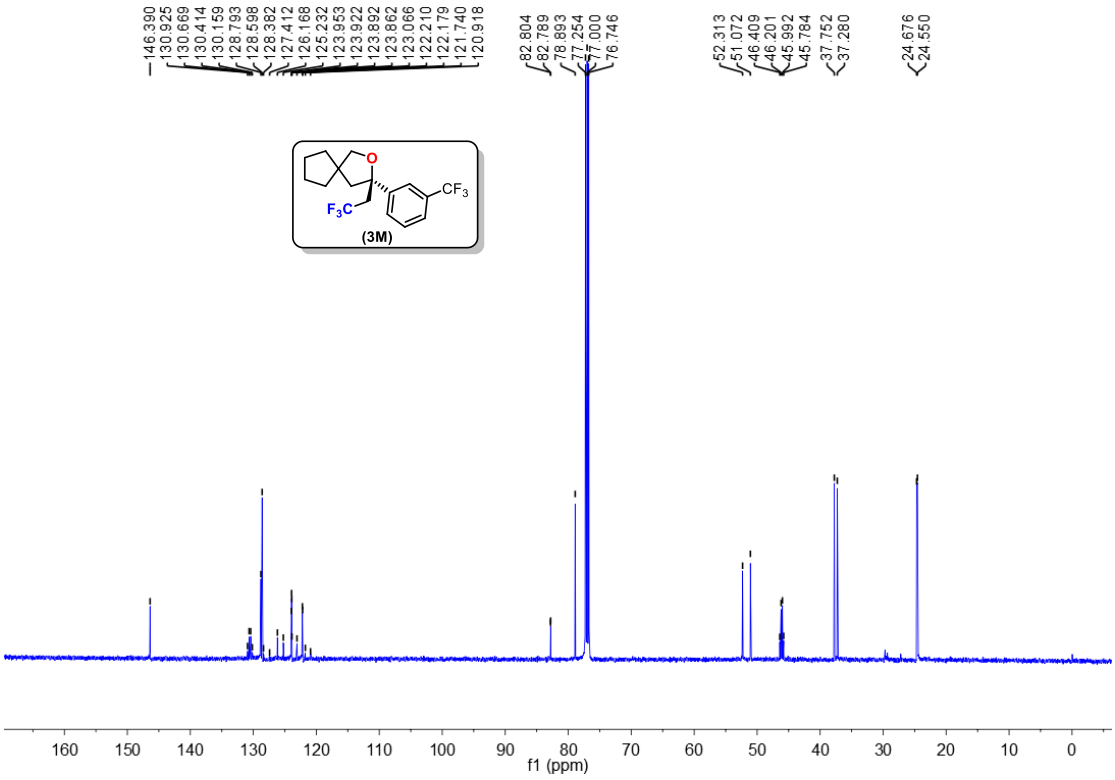

-60.123
-60.151
-60.180
O
Ph
F3C
(3K)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
f1 (ppm)

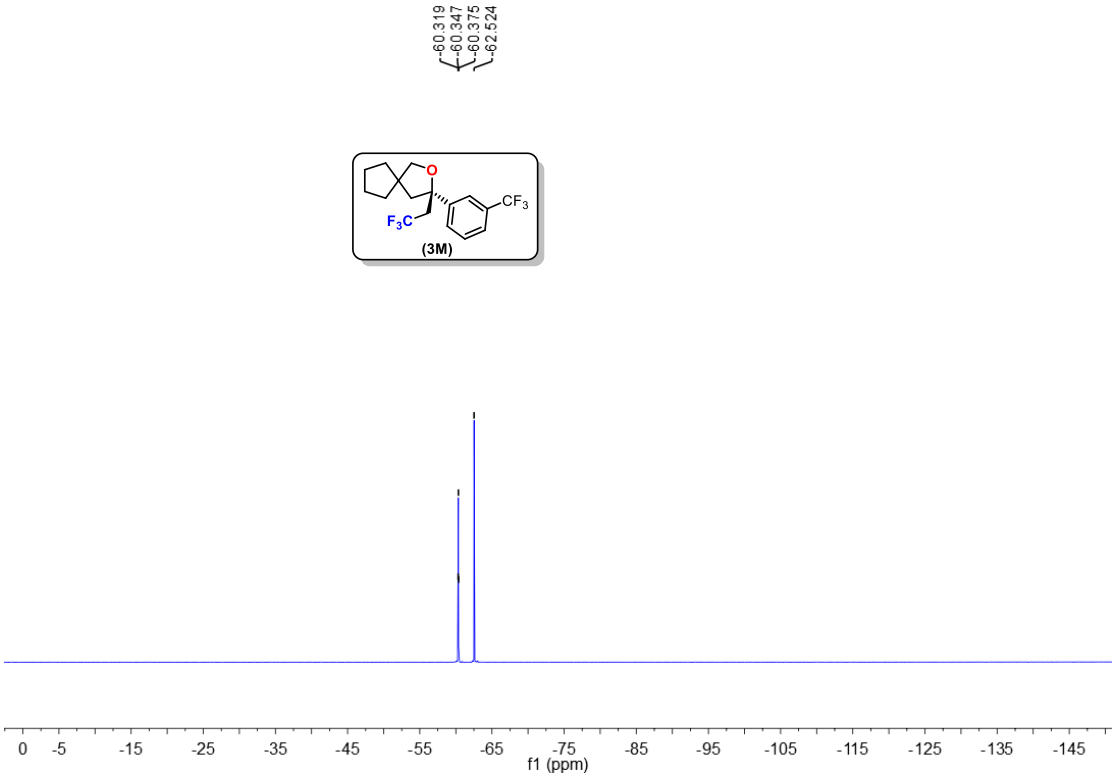

7.315
7.300
7.285
7.271
7.261
7.178
7.155
7.139
6.952
6.936
6.919
3.810
3.794
3.674
3.658
2.686
2.665
2.643
2.622
2.372
2.347
2.300
2.275
1.750
1.626
1.611
1.598
1.580
1.575
1.565
1.553
1.549
1.538
1.517
1.335
1.320
1.246
1.230
O
F
F3C
(3L)
0.99
1.94
0.94
1.00
0.99
1.07
0.97
1.03
1.01
1.12
5.88
1.13
1.34
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)

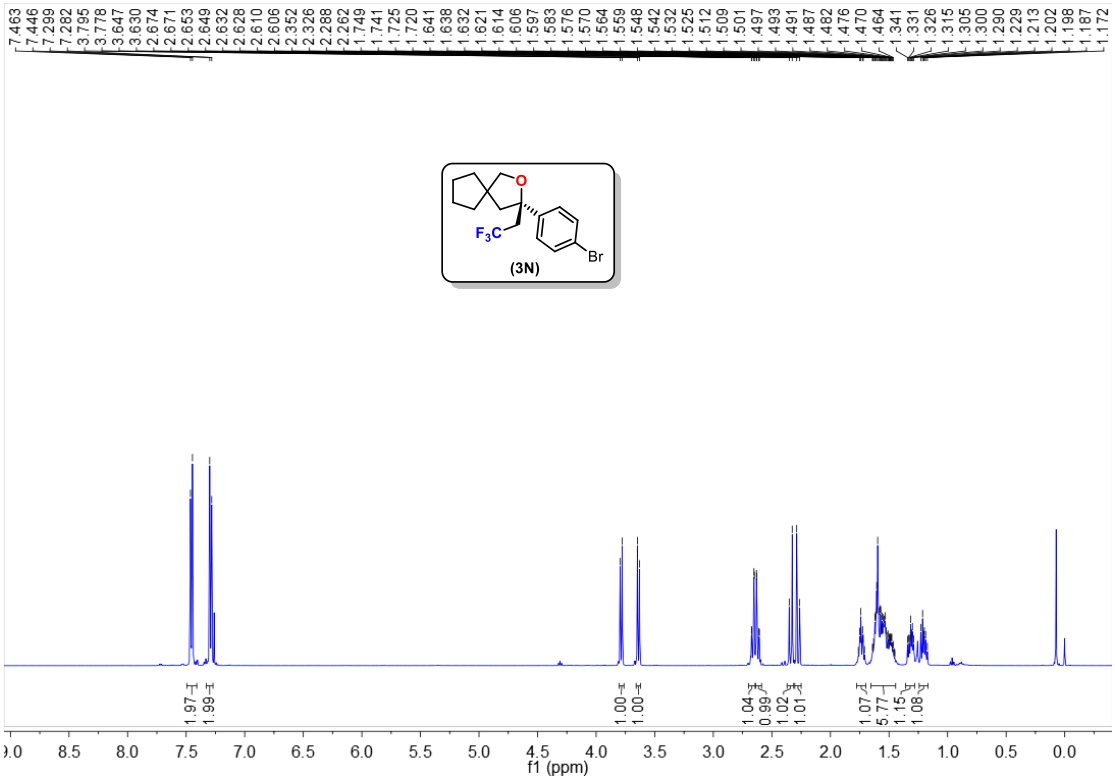

163.744
161.793
148.128
148.075
129.646
129.581
128.452
126.239
124.024
121.810
121.012
120.990
113.897
113.729
112.648
112.466
82.778
78.858
77.254
77.000
76.746
52.185
51.007
46.372
46.163
45.955
45.747
37.842
37.279
24.651
24.515
O
F
F3C
(3L)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

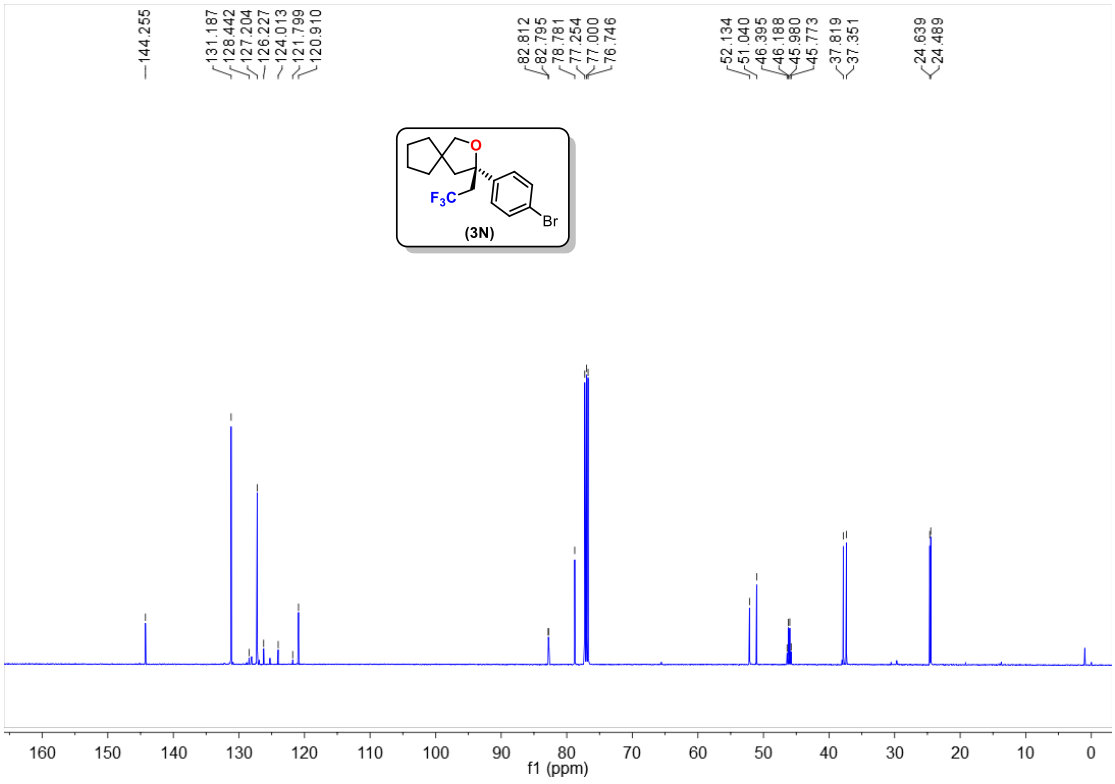

60.371
60.400
60.428
113.020
113.035
113.041
113.046
113.058
113.063
113.070
113.085
O
F
F3C
(3L)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150
f1 (ppm)

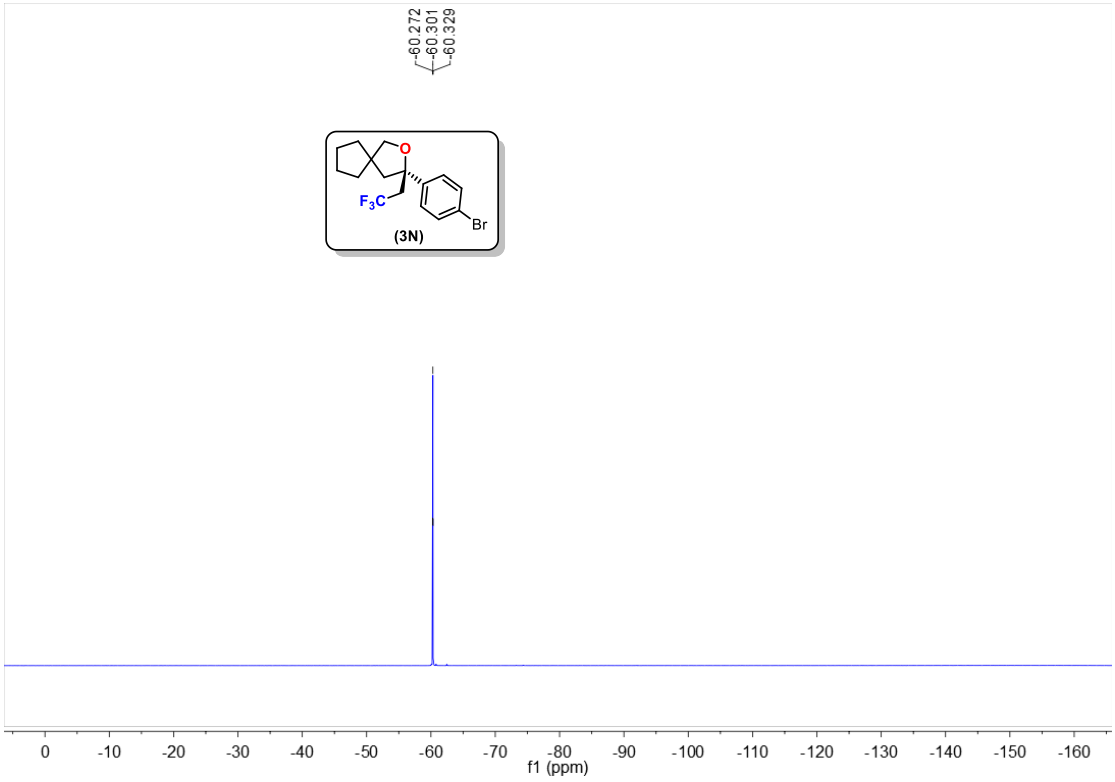

7.694
7.606
7.590
7.527
7.511
7.472
7.457
7.441
7.262
3.827
3.811
3.676
3.659
2.712
2.695
2.691
2.674
2.669
2.653
2.648
2.362
2.353
2.327
1.765
1.634
1.626
1.613
1.602
1.589
1.574
1.566
1.555
1.541
1.520
1.319
1.309
1.304
1.256
O
F3C
CF3
(3M)
0.96
0.99
0.97
1.00
1.00
0.99
2.03
2.00
1.10
6.21
1.27
1.11
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)

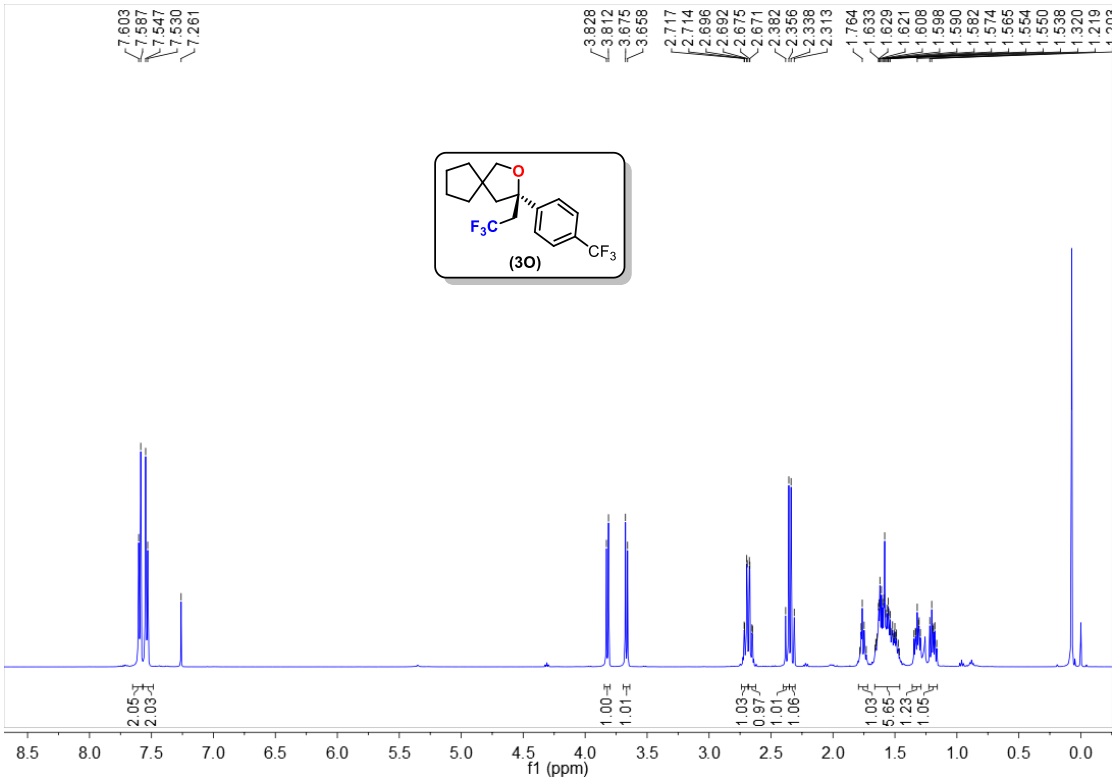

146.390
130.925
130.669
130.414
130.159
128.793
128.598
128.382
127.412
126.168
125.232
123.953
123.922
123.892
123.862
123.066
122.210
122.179
121.740
120.918
82.804
82.789
78.893
77.254
77.000
76.746
52.313
51.072
46.409
46.201
45.992
45.784
37.752
37.280
24.676
24.550
O
F3C
CF3
(3M)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

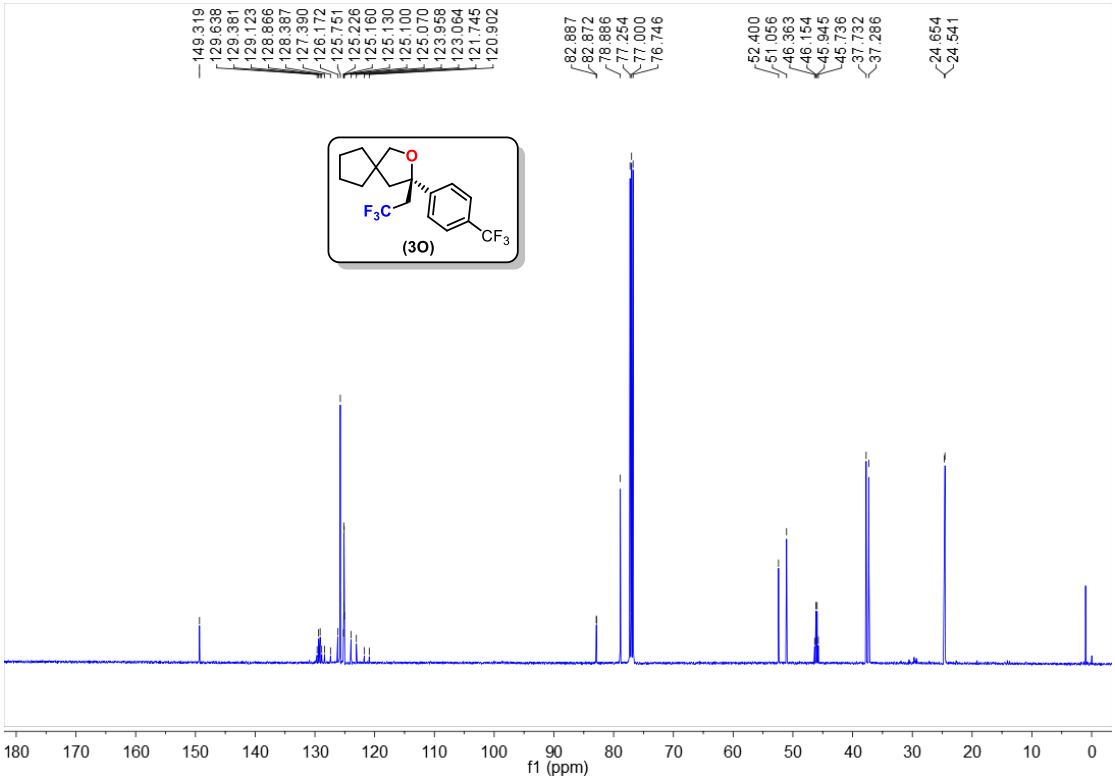

-60.319
-60.347
-60.375
-62.524
O
CF3
F3C
(3M)
0 -5 -15 -25 -35 -45 -55 -65 -75 -85 -95 -105 -115 -125 -135 -145
f1 (ppm)

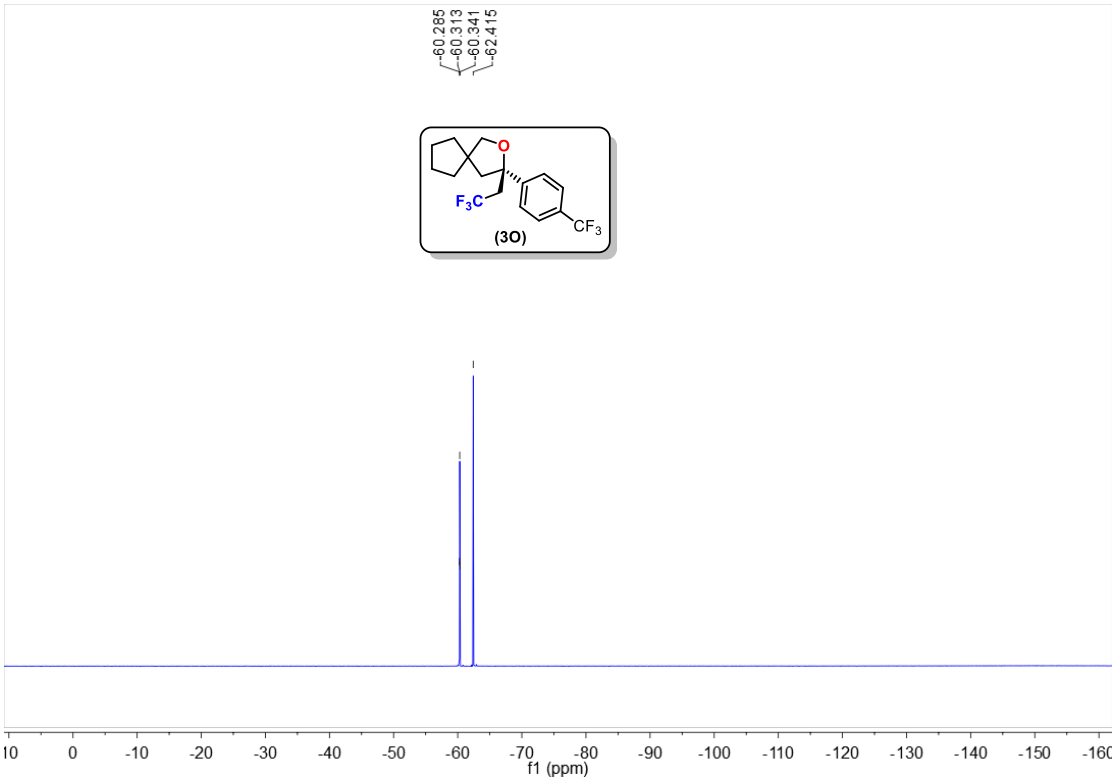

7.463
7.446
7.299
7.282
3.795
3.778
3.647
3.630
2.674
2.671
2.653
2.649
2.632
2.628
2.610
2.606
2.352
2.326
2.288
2.262
1.749
1.741
1.725
1.720
1.641
1.638
1.632
1.621
1.614
1.606
1.597
1.583
1.576
1.570
1.564
1.559
1.548
1.542
1.532
1.525
1.512
1.509
1.501
1.497
1.493
1.491
1.487
1.482
1.476
1.470
1.464
1.341
1.331
1.326
1.315
1.305
1.300
1.290
1.229
1.213
1.202
1.198
1.187
1.172
O
F3C
Br
(3N)
1.97
1.99
1.00
1.00
1.04
0.99
1.02
1.01
1.07
5.77
1.15
1.08
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)

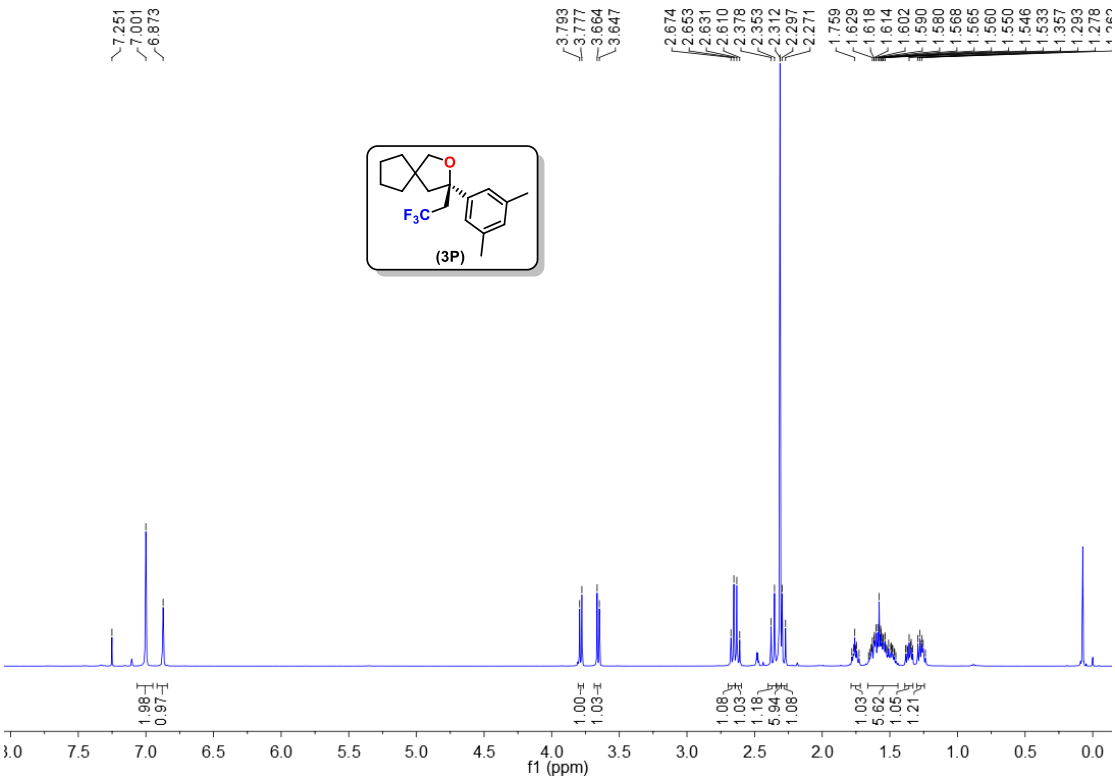

144.255
131.187
128.442
127.204
126.227
124.013
121.799
120.910
82.812
82.795
78.781
77.254
77.000
76.746
52.134
51.040
46.395
46.188
45.980
45.773
37.819
37.351
24.639
24.489
O
F3C
Br
(3N)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

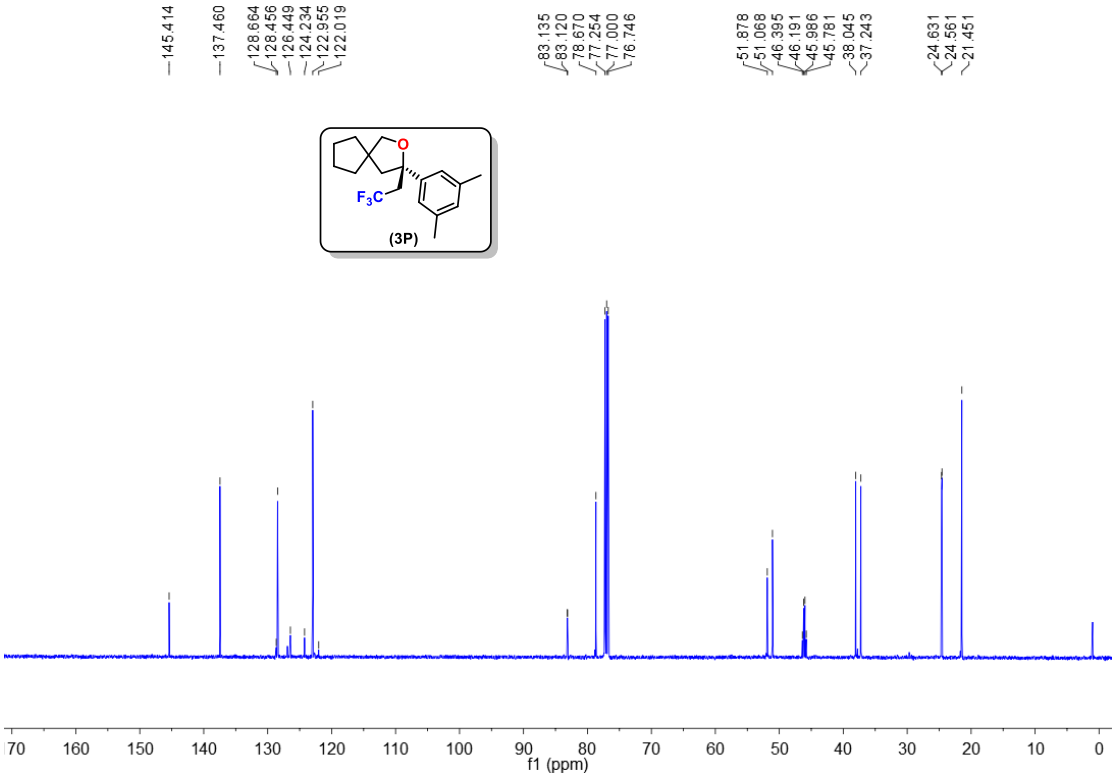

60.272
60.301
60.329
O
F3C
Br
(3N)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

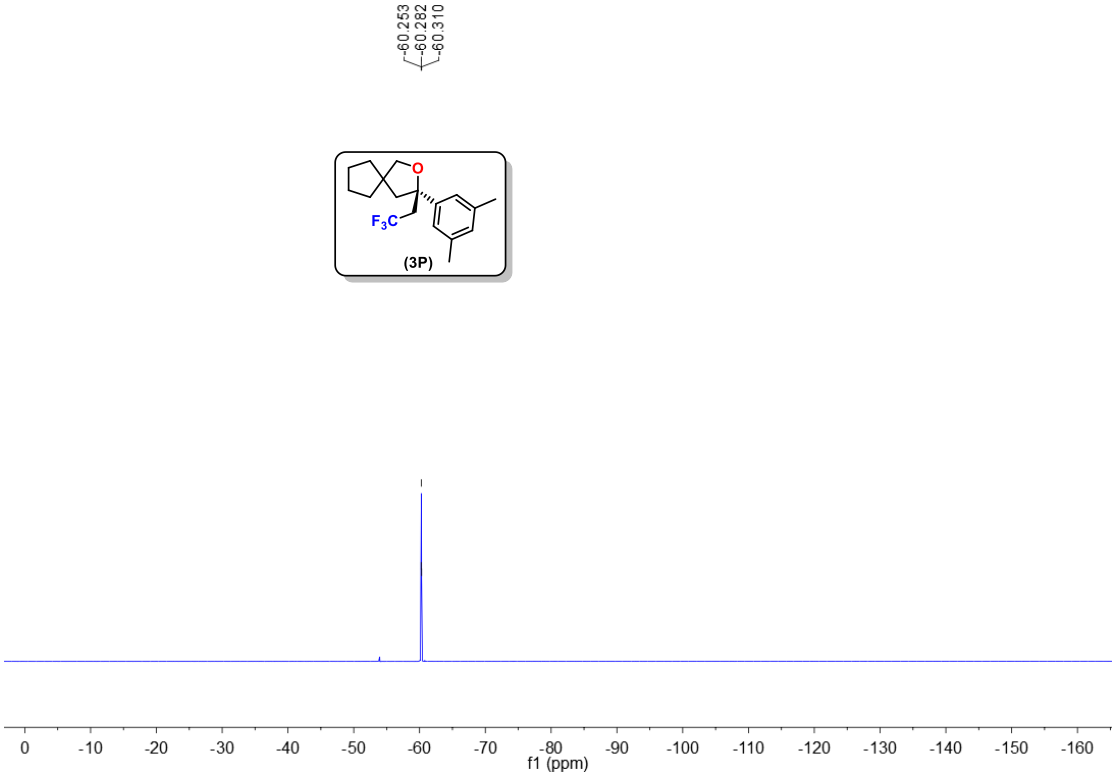

7.603
7.587
7.547
7.530
7.261
3.828
3.812
3.675
3.658
2.717
2.714
2.696
2.692
2.675
2.671
2.382
2.356
2.338
2.313
1.764
1.633
1.629
1.621
1.608
1.598
1.590
1.582
1.574
1.565
1.554
1.550
1.538
1.320
1.219
O
F3C
CF3
(3O)
2.05
2.03
1.00
1.01
1.03
0.97
1.01
1.06
1.03
5.65
1.23
1.05
8.5 8.0 7.5 7.0 6.5 6.0 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)

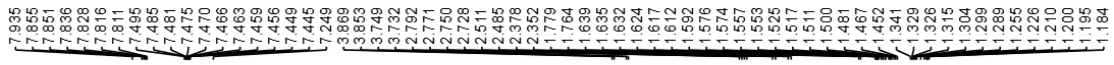

149.319
129.638
129.381
129.123
128.866
128.387
127.390
126.172
125.751
125.226
125.160
125.130
125.100
125.070
123.958
123.064
121.745
120.902
82.887
82.872
78.886
77.254
77.000
76.746
52.400
51.056
46.363
46.154
45.945
45.736
37.732
37.286
24.654
24.541
O
F3C
CF3
(3O)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

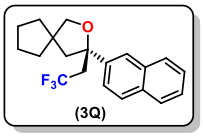

-60.285
-60.313
-60.341
-62.415
O
F3C
CF3
(3O)
f1 (ppm)

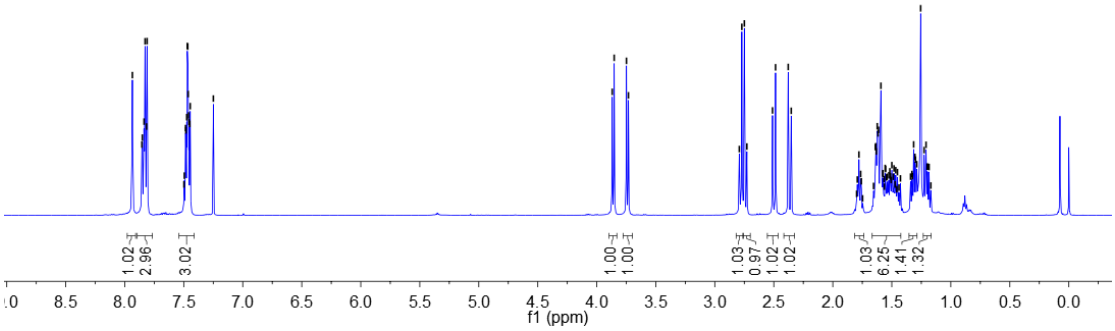

7.251
7.001
6.873
3.793
3.777
3.664
3.647
2.674
2.653
2.631
2.610
2.378
2.353
2.312
2.297
2.271
1.759
1.629
1.618
1.614
1.602
1.590
1.580
1.568
1.565
1.560
1.550
1.546
1.533
1.357
1.293
1.278
1.98
0.97
1.00
1.03
1.08
1.03
1.18
5.94
1.08
1.03
5.62
1.05
1.21
O
F3C
(3P)
f1 (ppm)

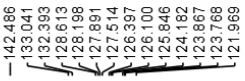

145.414
137.460
128.664
128.456
126.449
124.234
122.955
122.019
83.135
83.120
78.670
77.254
77.000
76.746
51.878
51.068
46.395
46.191
45.986
45.781
38.045
37.243
24.631
24.561
21.451
O
F3C
(3P)
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

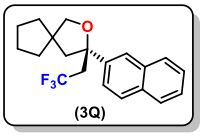

60.253
60.282
60.310
O
F3C
(3P)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

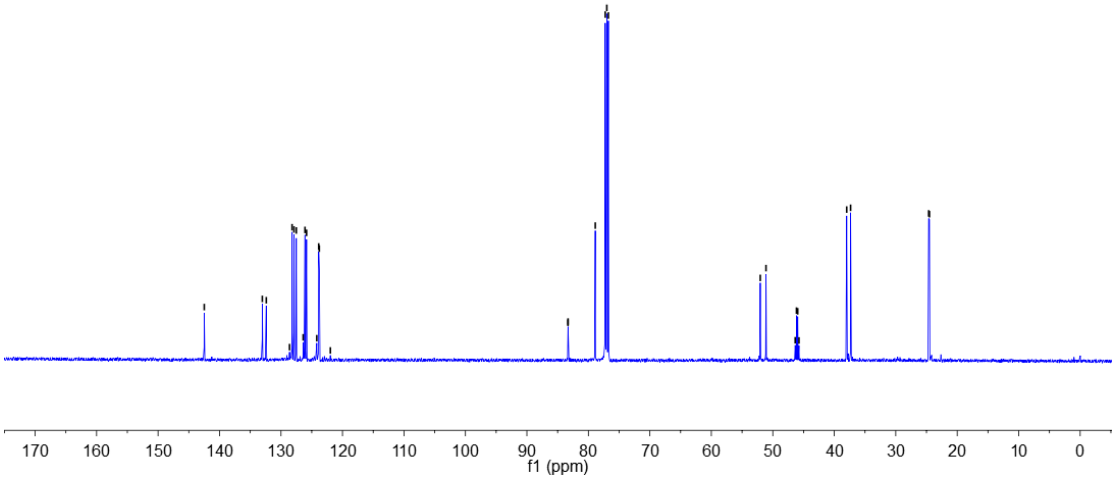

7.935
7.855
7.851
7.836
7.828
7.816
7.811
7.495
7.485
7.481
7.475
7.470
7.466
7.463
7.459
7.456
7.449
7.445
7.249
3.869
3.853
3.749
3.732
2.792
2.771
2.750
2.728
2.511
2.485
2.378
2.352
1.779
1.764
1.639
1.635
1.632
1.624
1.617
1.612
1.592
1.576
1.574
1.557
1.553
1.525
1.517
1.511
1.500
1.481
1.467
1.452
1.341
1.329
1.326
1.315
1.304
1.299
1.289
1.255
1.226
1.210
1.200
1.195
1.184

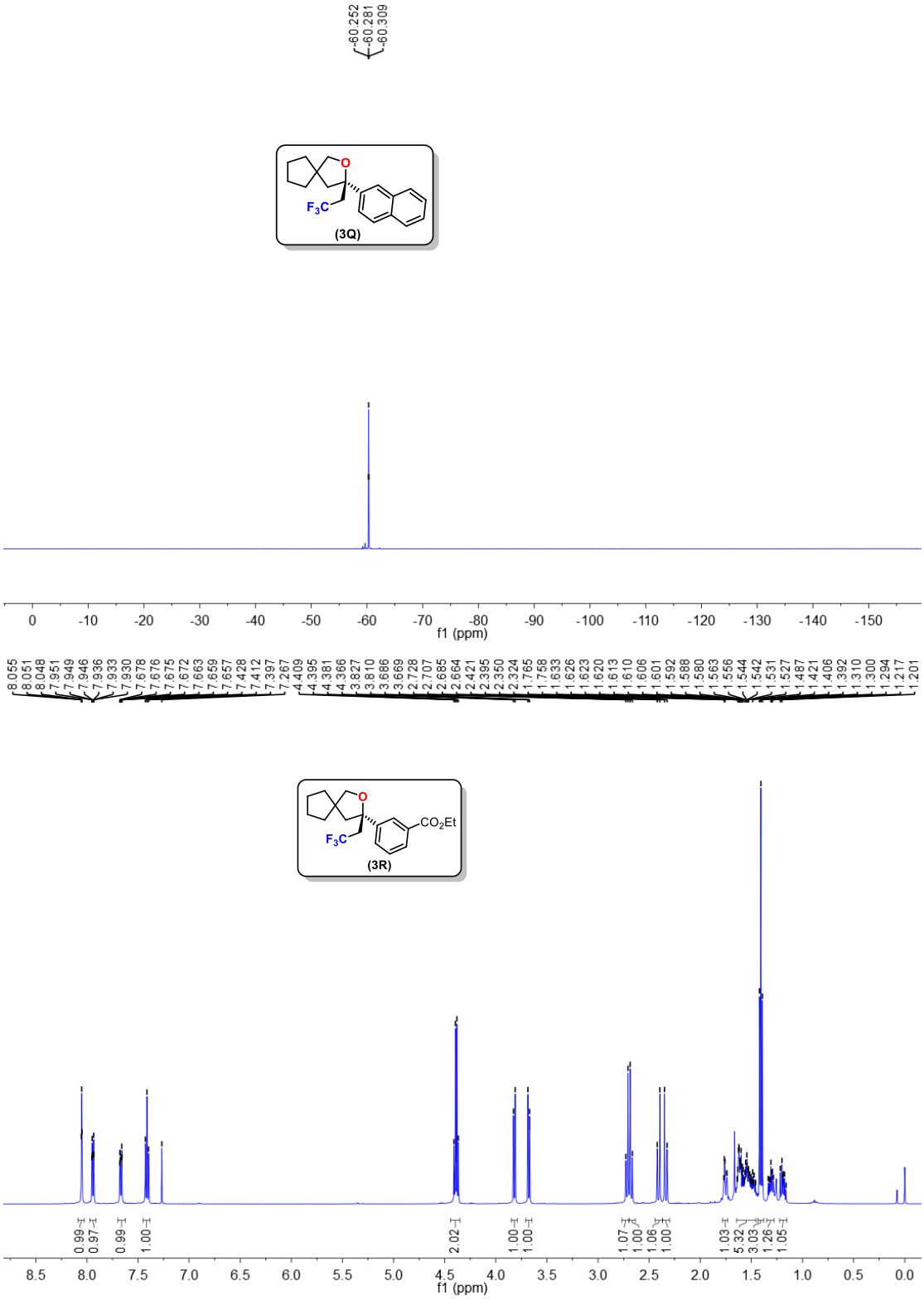

O
F3C
(3Q)

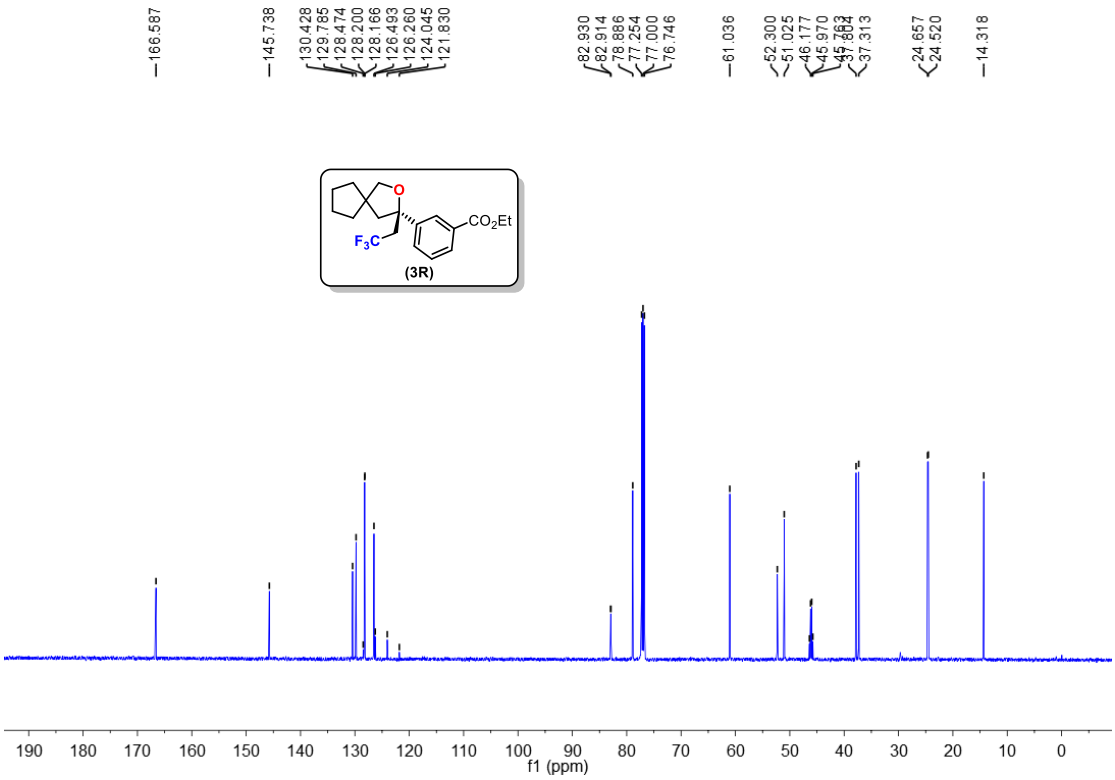

1.02
2.96
3.02
1.00
1.00
1.03
0.97
1.02
1.02
1.03
6.25
1.41
1.32
f1 (ppm)

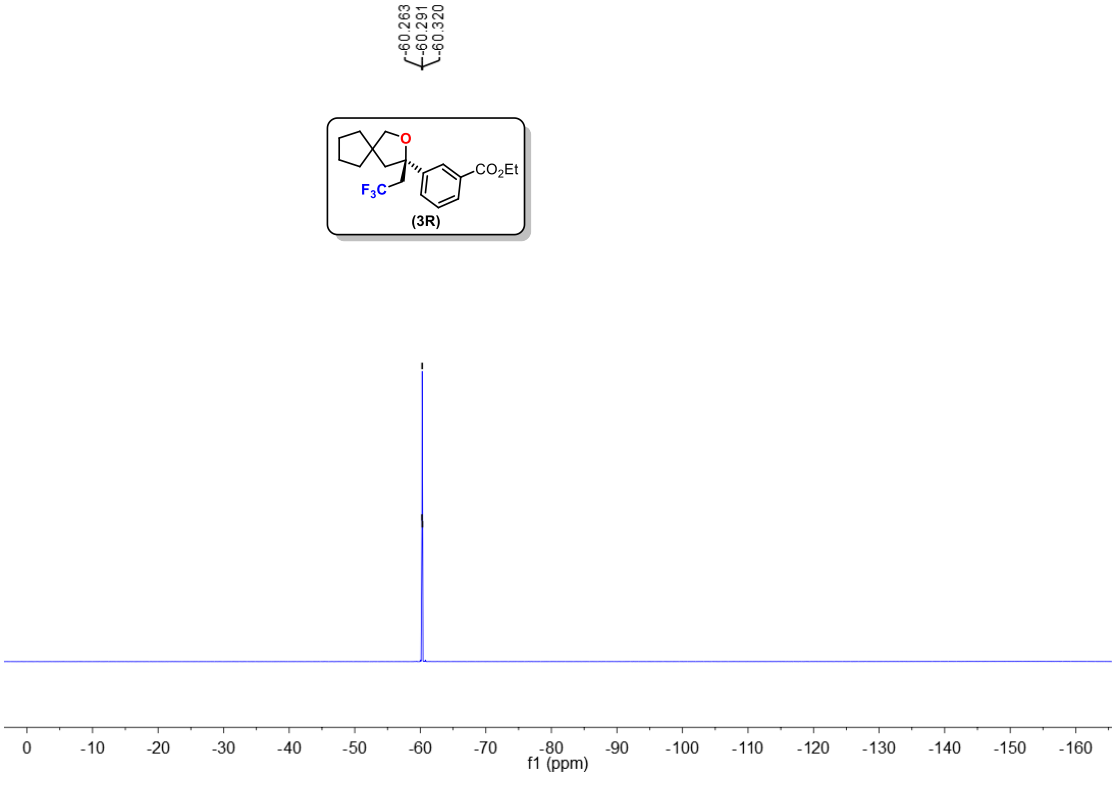

142.486
133.041
132.393
128.613
128.198
127.891
127.514
126.397
126.100
125.846
124.182
123.867
123.768
121.969
83.321
83.306
78.877
77.254
77.000
76.746
52.039
51.106
46.359
46.152
45.946
45.739
37.980
37.345
24.645
24.519

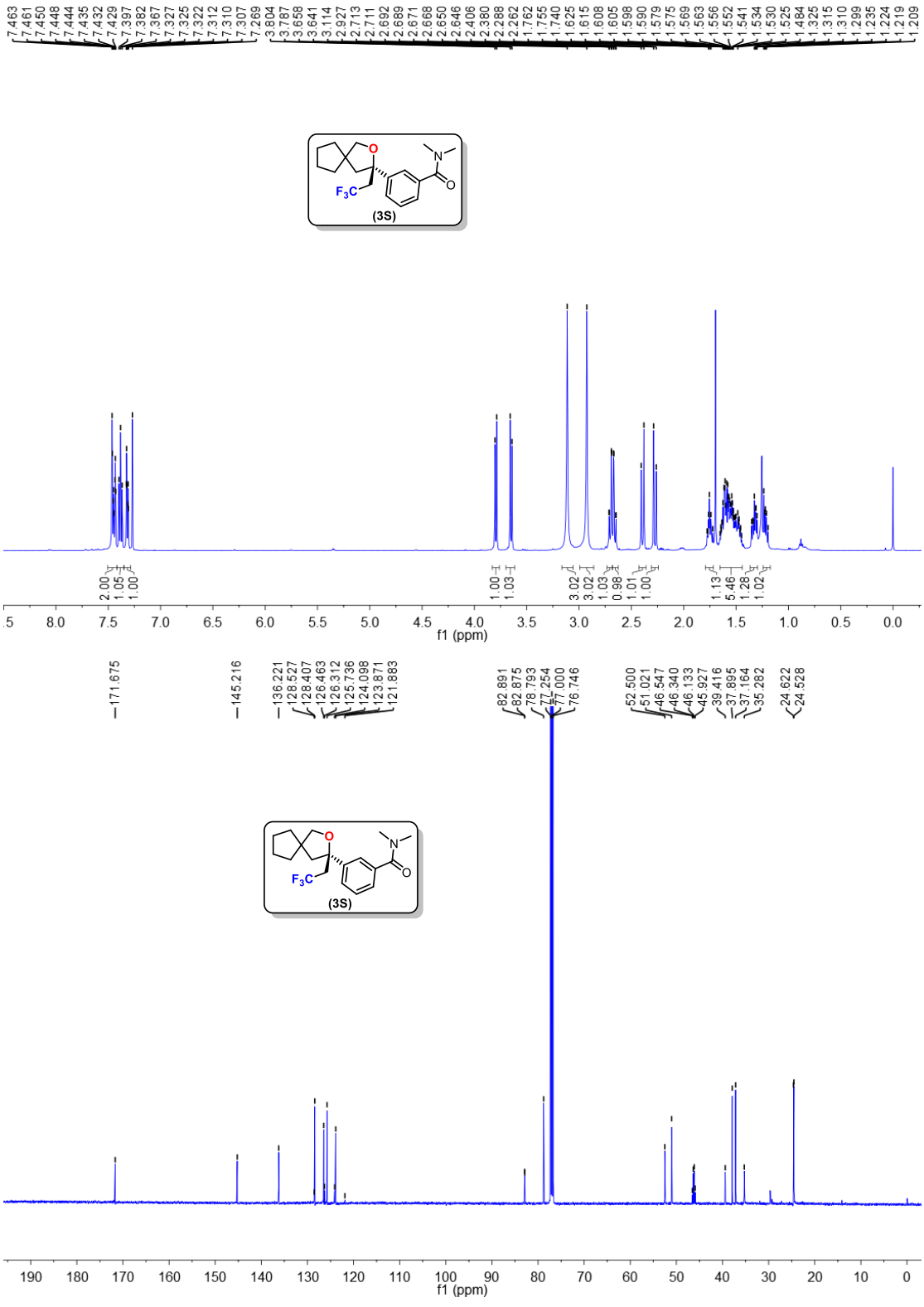

O
F3C
(3Q)

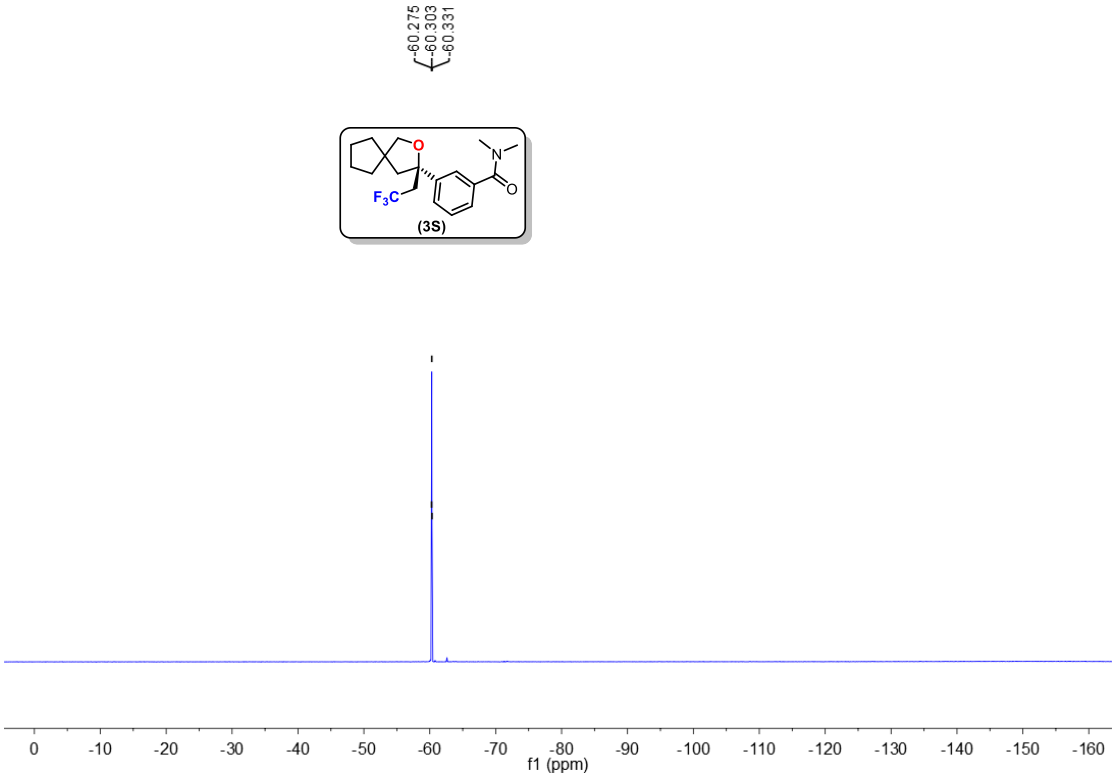

f1 (ppm)

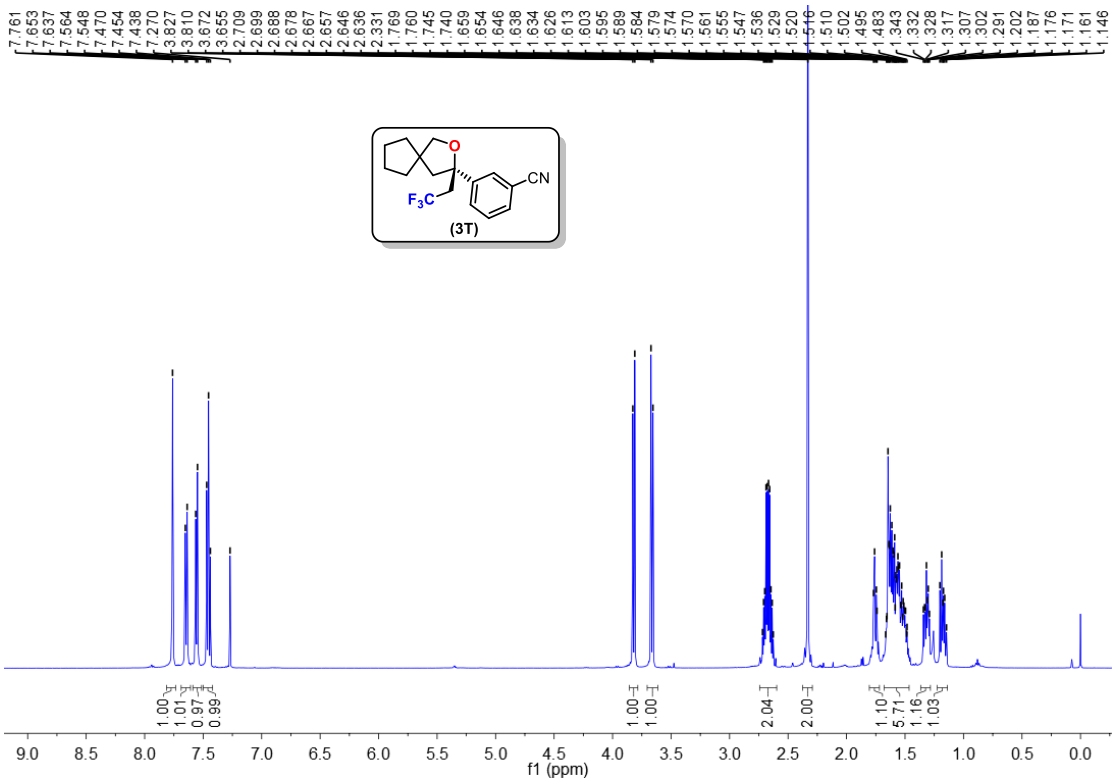

-60.252
-60.281
-60.309
O
F3C
(3Q)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)
8.055
8.051
8.048
7.951
7.949
7.946
7.936
7.933
7.930
7.678
7.676
7.675
7.672
7.663
7.659
7.657
7.428
7.412
7.397
7.267
4.409
4.395
4.381
4.366
3.827
3.810
3.686
3.669
2.728
2.707
2.685
2.664
2.421
2.395
2.350
2.324
1.765
1.758
1.633
1.626
1.623
1.620
1.613
1.610
1.606
1.601
1.592
1.588
1.580
1.563
1.556
1.544
1.542
1.531
1.527
1.487
1.421
1.406
1.392
1.310
1.300
1.294
1.217
1.201
O
F3C
CO2Et
(3R)
0.99
0.97
0.99
1.00
2.02
1.00
1.00
1.07
1.00
1.06
1.00
1.03
5.32
3.03
1.26
1.05
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)

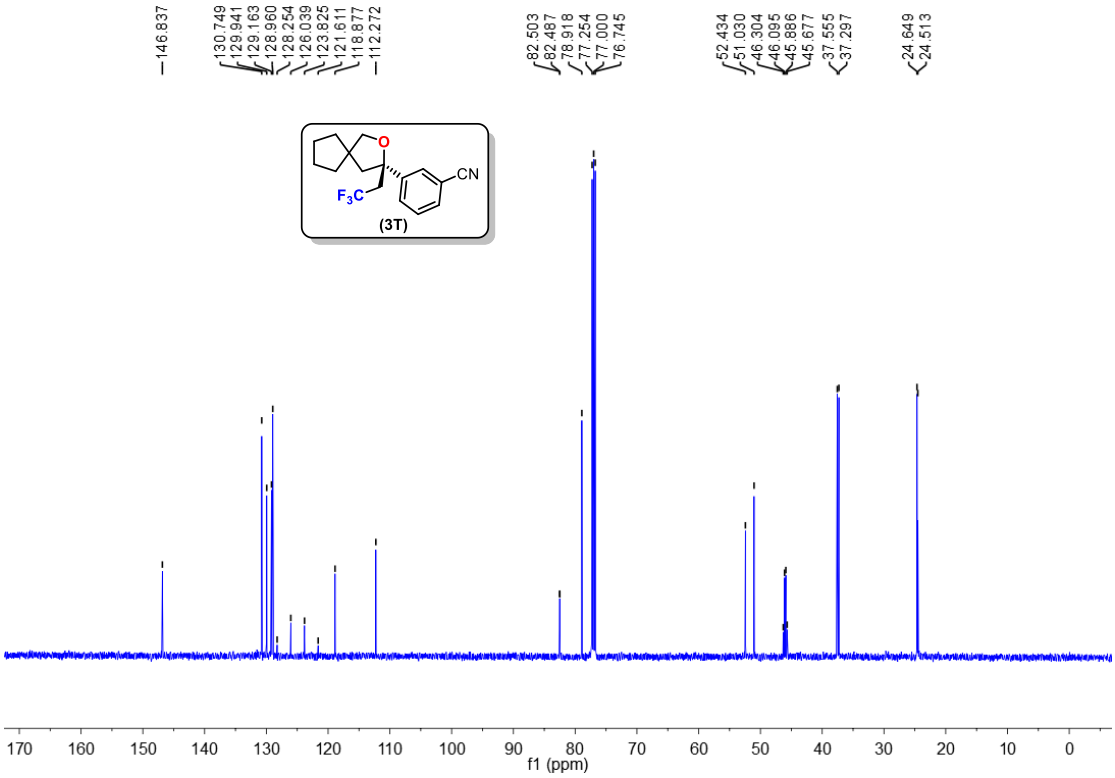

166.587
145.738
130.428
129.785
129.474
128.200
128.166
126.493
126.260
124.045
121.830
82.930
82.914
78.886
77.254
77.000
76.746
61.036
52.300
51.025
46.177
45.970
37.313
24.657
24.520
14.318
O
CO2Et
F3C
(3R)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

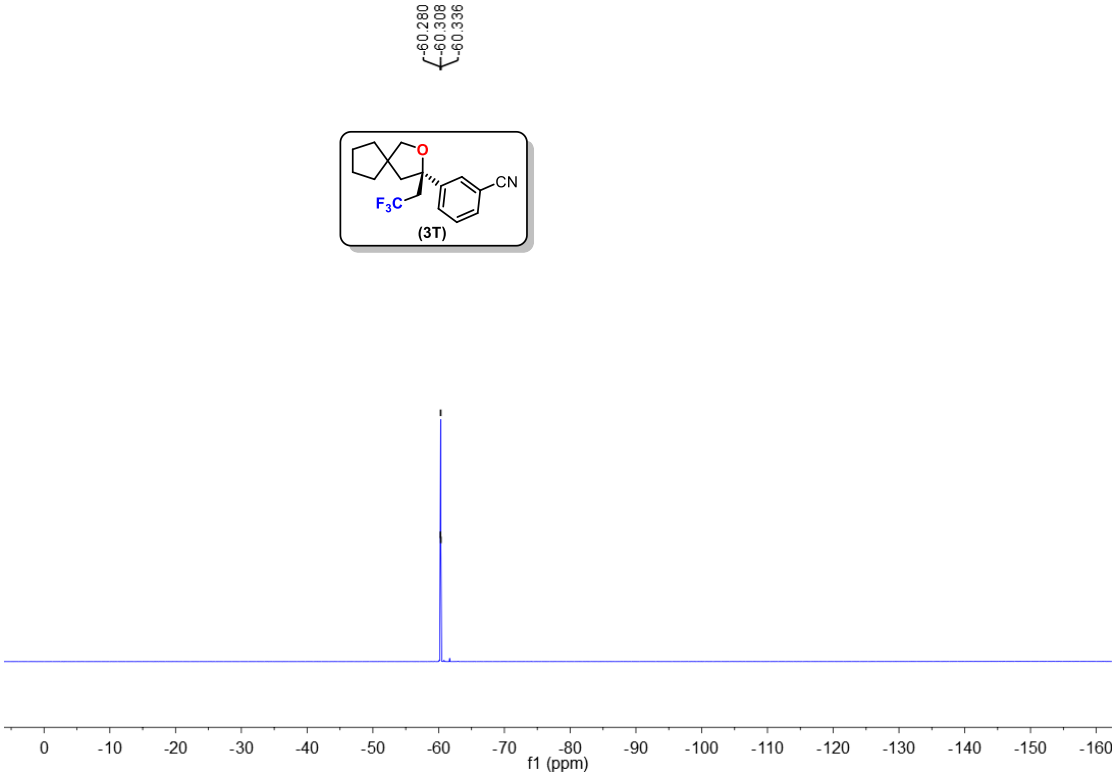

-60.263
-60.291
-60.320
O
CO2Et
F3C
(3R)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

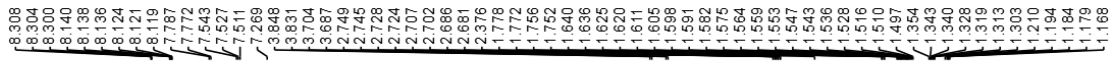

7.463
7.461
7.450
7.448
7.444
7.435
7.432
7.429
7.397
7.382
7.367
7.327
7.325
7.322
7.312
7.310
7.307
7.269
3.804
3.787
3.658
3.641
3.114
2.927
2.713
2.711
2.692
2.689
2.671
2.668
2.650
2.646
2.406
2.380
2.288
2.262
1.762
1.755
1.740
1.625
1.615
1.608
1.605
1.598
1.590
1.579
1.575
1.569
1.563
1.556
1.552
1.541
1.534
1.530
1.525
1.484
1.325
1.315
1.310
1.299
1.235
1.224
1.219
1.209
O
N
O
F3C
(3S)
2.00
1.05
1.00
1.00
1.03
3.02
3.02
1.03
0.98
1.01
1.00
1.13
5.46
1.28
1.02
f1 (ppm)
171.675
145.216
136.221
128.527
128.407
126.463
126.312
125.736
124.098
123.871
121.883
82.891
82.875
78.793
77.254
77.000
76.746
52.500
51.021
46.547
46.340
46.133
45.927
39.416
37.895
37.164
35.282
24.622
24.528
f1 (ppm)

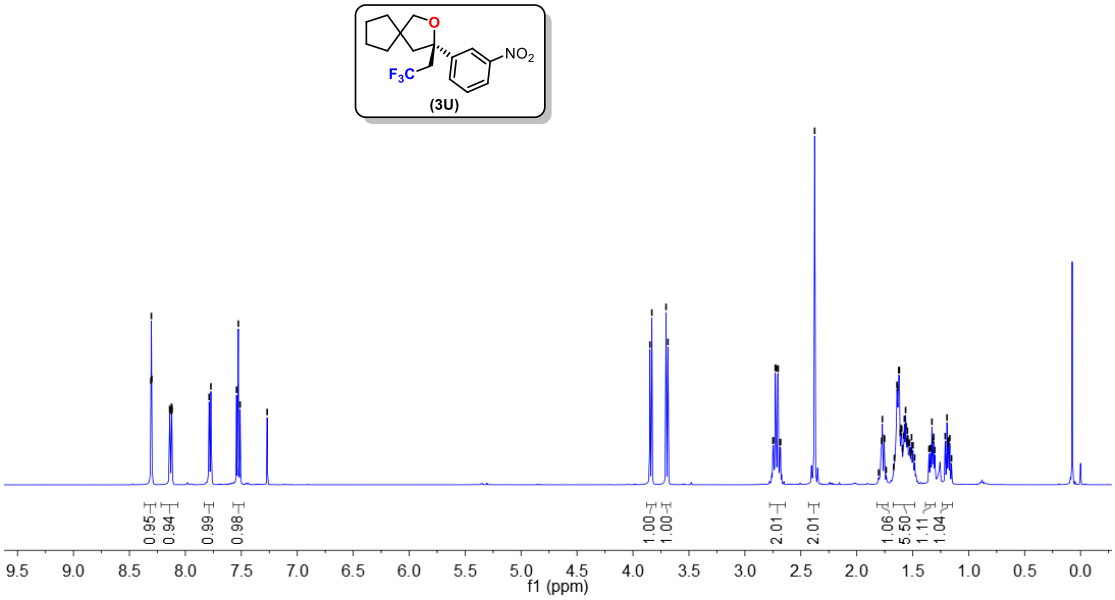

-60.275
-60.303
-60.331
(3S)
f1 (ppm)

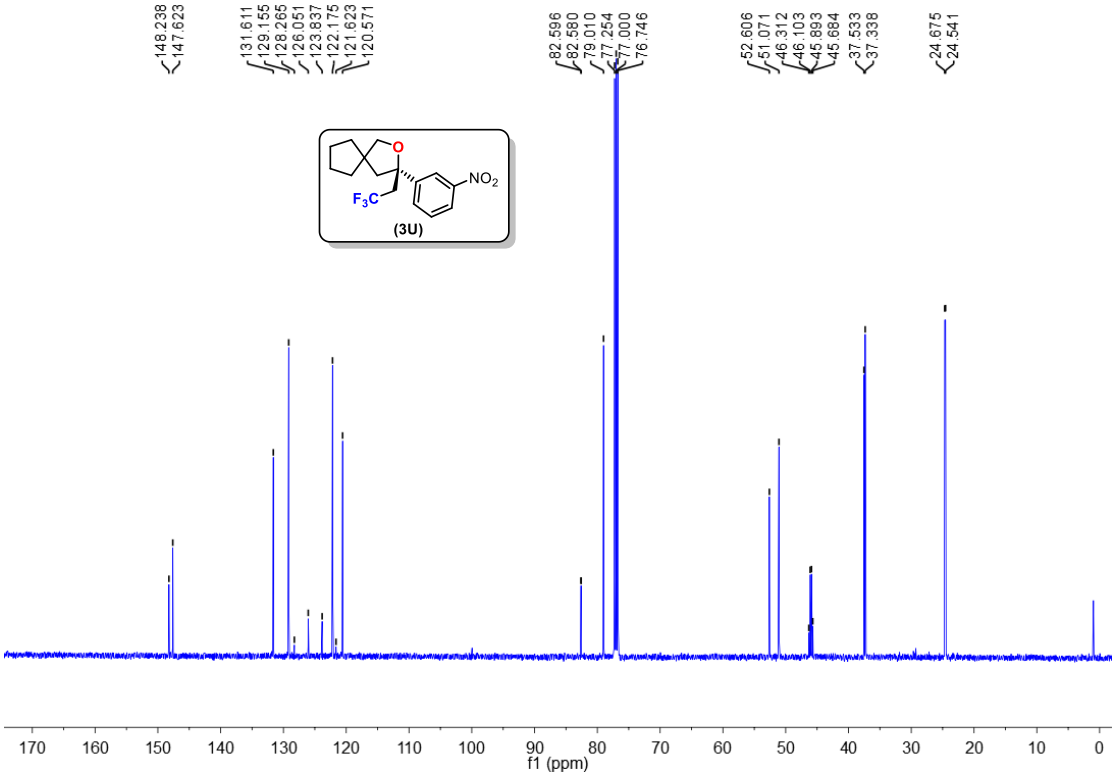

(3T)
f1 (ppm)

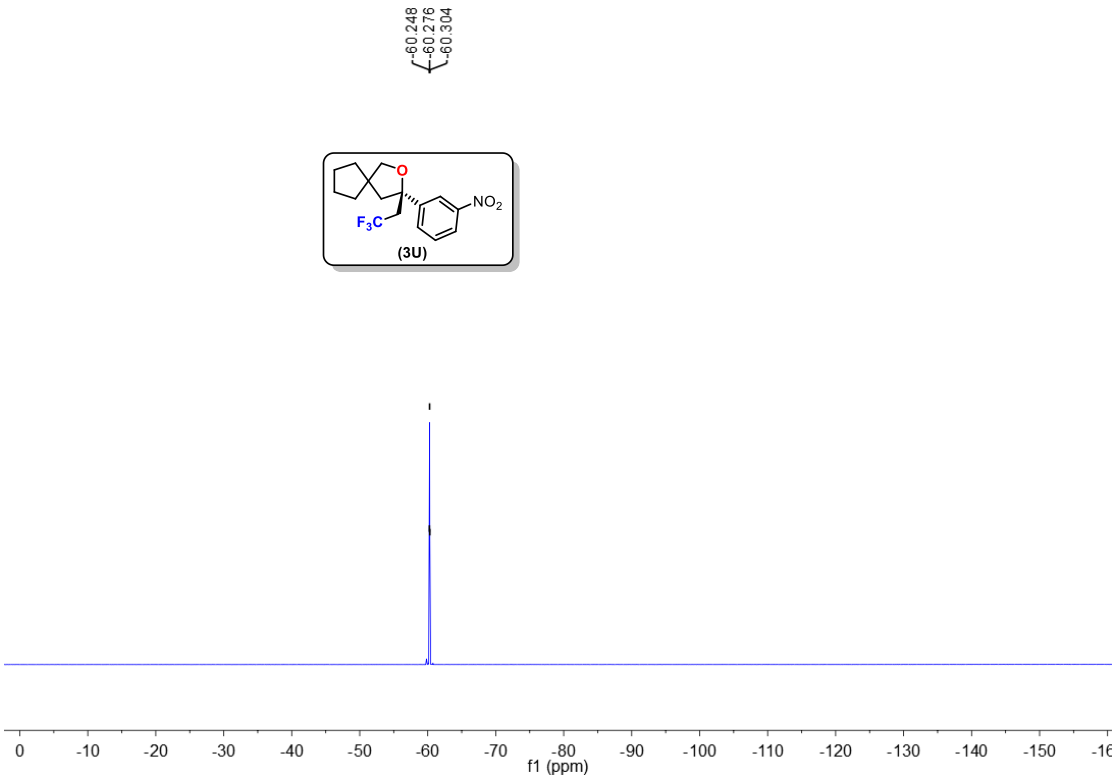

146.837
130.749
129.941
129.163
128.960
128.254
126.039
123.825
121.611
118.877
112.272
82.503
82.487
78.918
77.254
77.000
76.745
52.434
51.030
46.304
46.095
45.886
45.677
37.555
37.297
24.649
24.513
O
CN
F3C
(3T)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

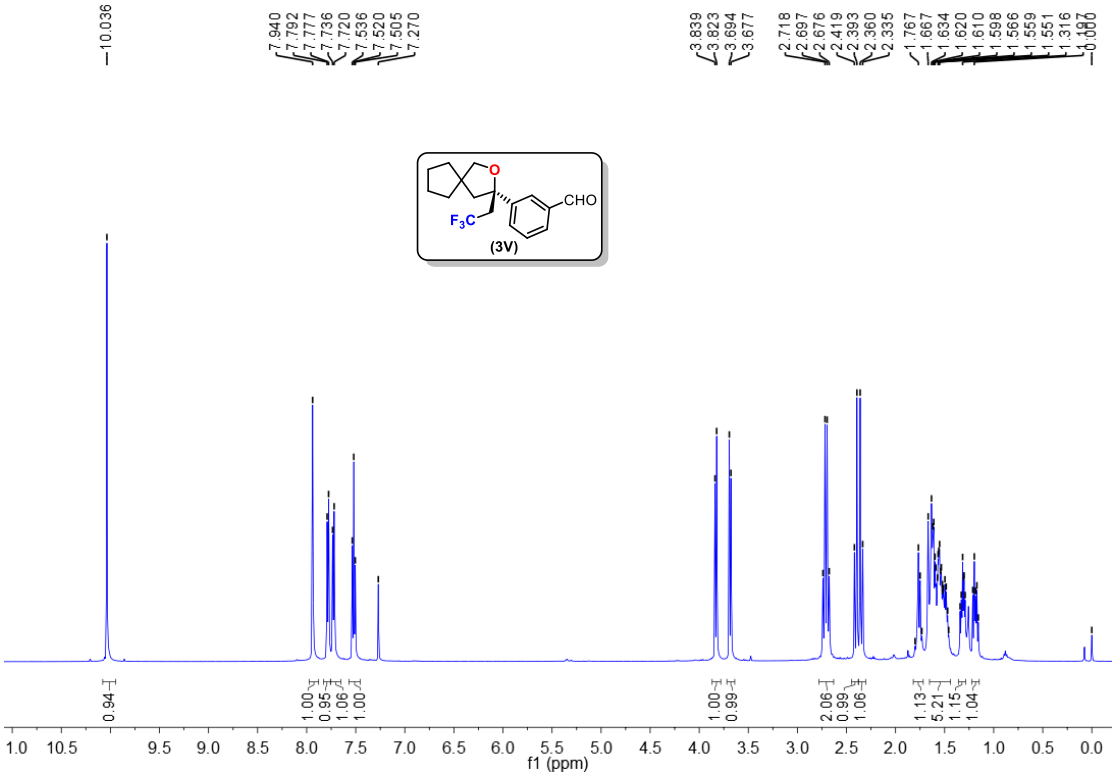

-60.280
-60.308
-60.336
O
CN
F3C
(3T)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

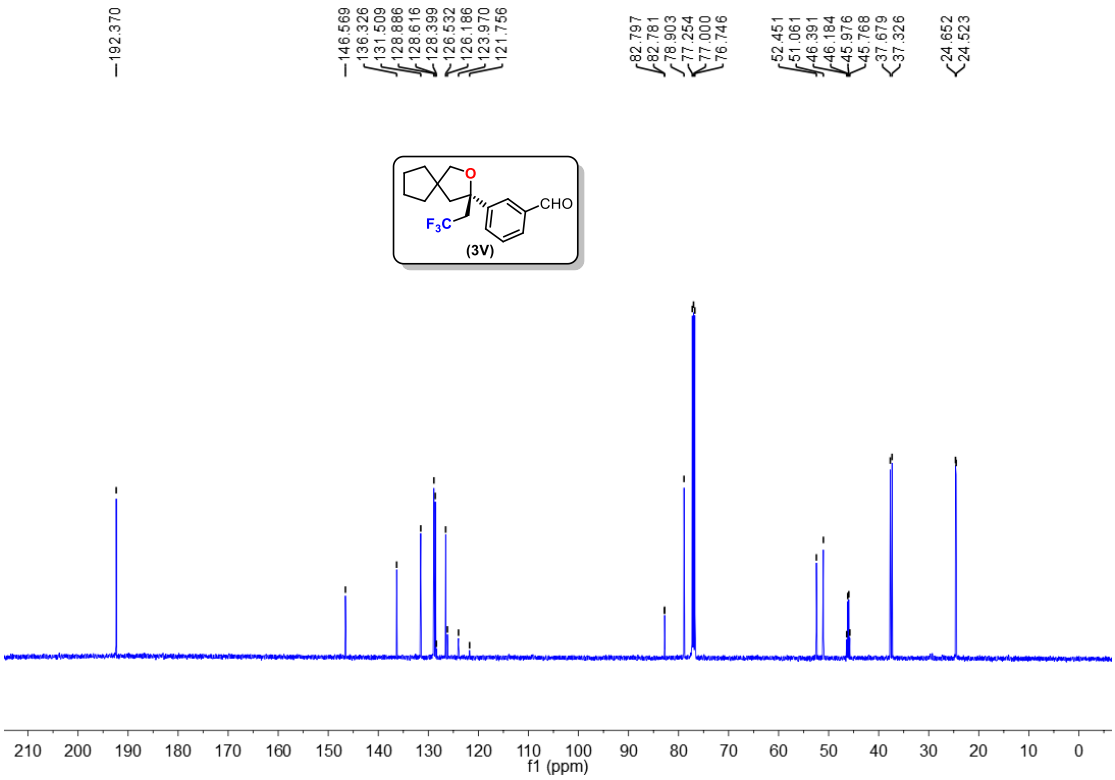

8.308
8.304
8.300
8.140
8.138
8.136
8.124
8.121
8.119
7.787
7.772
7.543
7.527
7.511
7.269
3.848
3.831
3.704
3.687
2.749
2.745
2.728
2.724
2.707
2.702
2.686
2.681
2.376
1.778
1.772
1.756
1.752
1.640
1.636
1.625
1.620
1.611
1.605
1.598
1.591
1.582
1.575
1.564
1.559
1.553
1.547
1.543
1.536
1.528
1.516
1.510
1.497
1.354
1.343
1.340
1.328
1.319
1.313
1.303
1.210
1.194
1.184
1.179
1.168

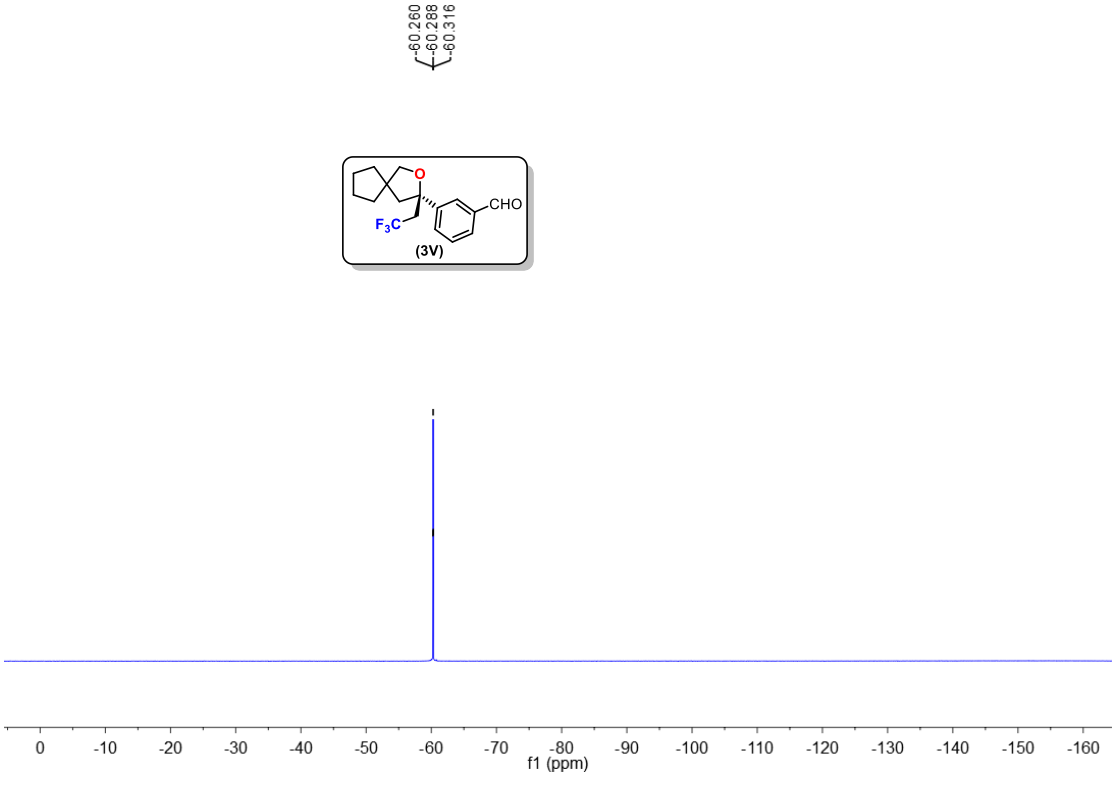

O
NO2
F3C
(3U)
0.95
0.94
0.99
0.98
1.00
1.00
2.01
2.01
1.06
5.50
1.11
1.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

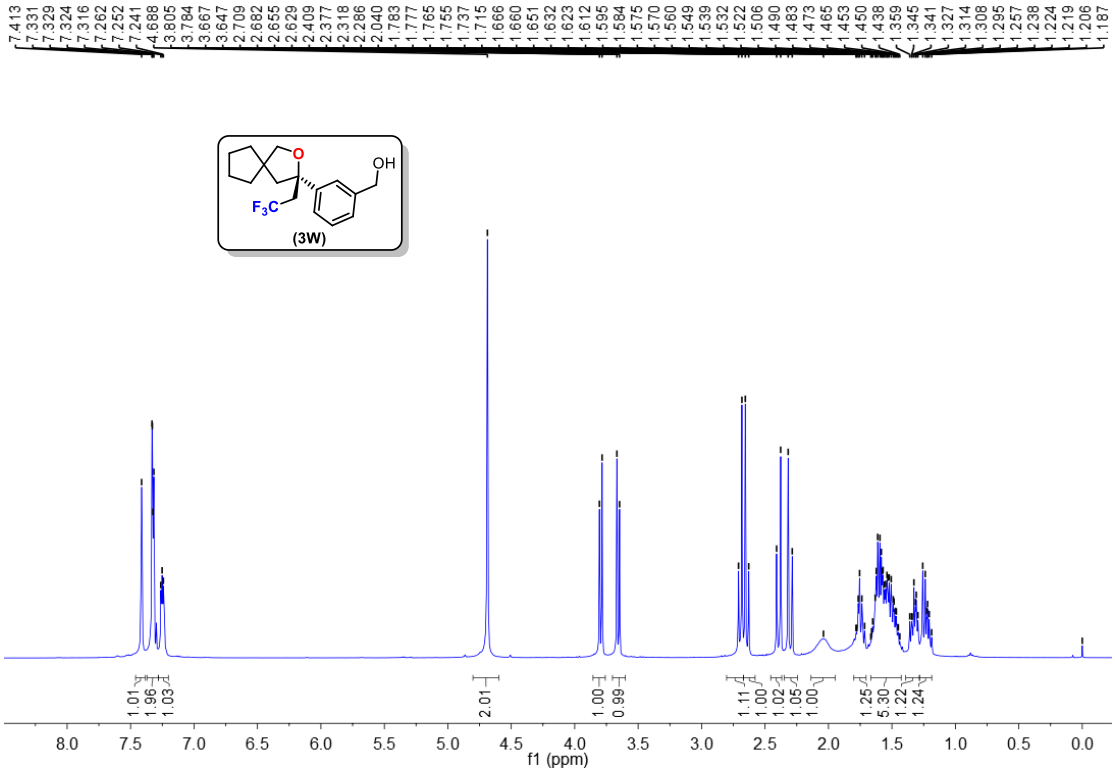

148.238
147.623
131.611
129.155
128.265
126.051
123.837
122.175
121.623
120.571
82.596
82.580
79.010
77.254
77.000
76.746
52.606
51.071
46.312
46.103
45.893
45.684
37.533
37.338
24.675
24.541
O
NO2
F3C
(3U)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

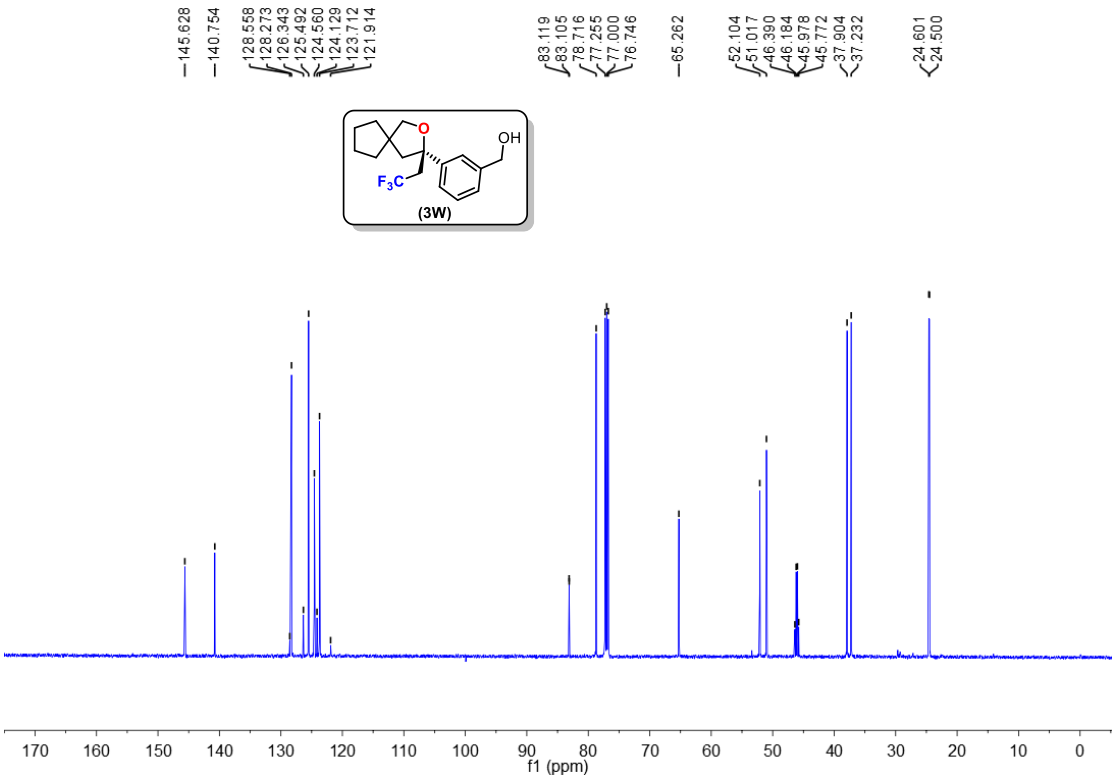

-60.248
-60.276
-60.304
O
NO2
F3C
(3U)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)

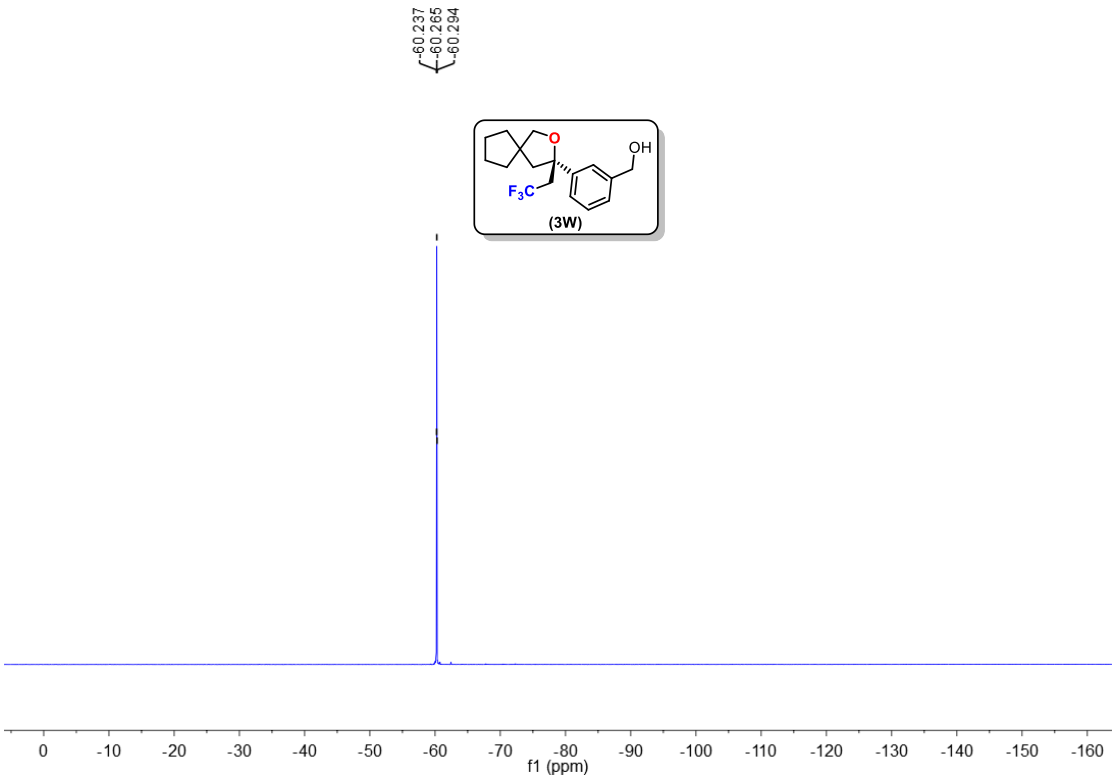

10.036
7.940
7.792
7.777
7.736
7.720
7.536
7.520
7.505
7.270
3.839
3.823
3.694
3.677
2.718
2.697
2.676
2.419
2.393
2.360
2.335
1.767
1.667
1.634
1.620
1.610
1.598
1.566
1.559
1.551
1.316
0.000
O
CHO
F3C
(3V)
0.94
1.00
0.95
1.06
1.00
1.00
0.99
2.06
0.99
1.06
1.13
5.21
1.15
1.04
1.0
10.5
9.5
9.0
8.5
8.0
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)

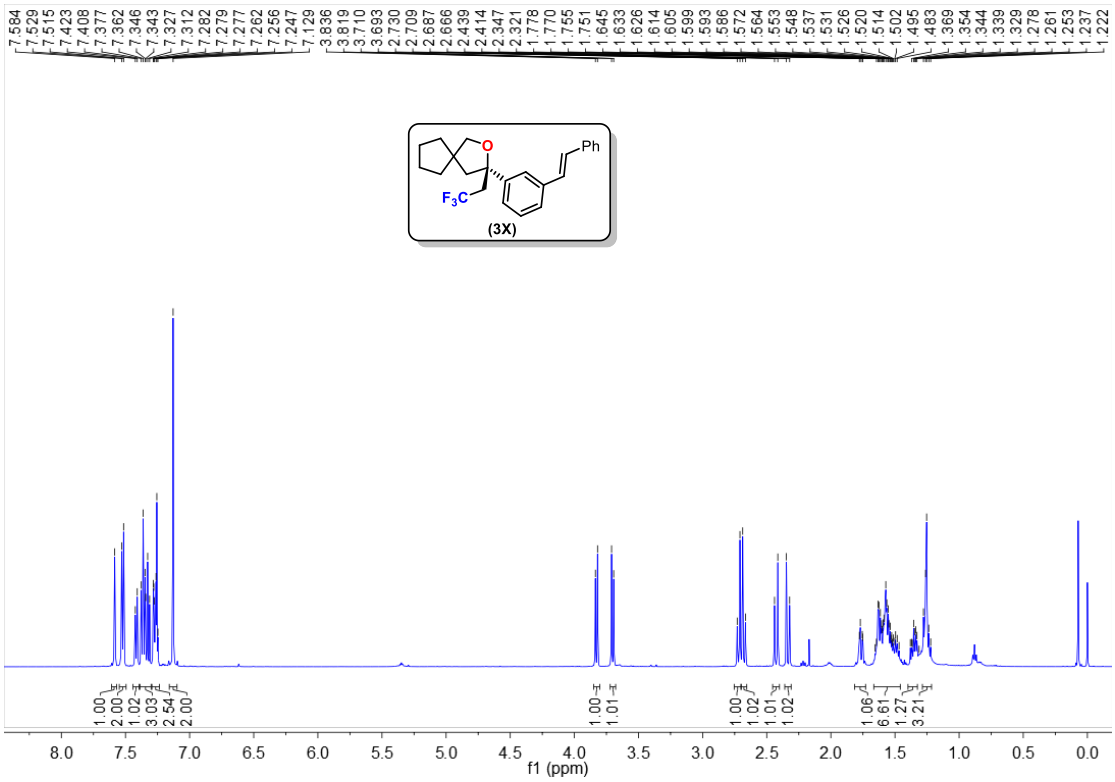

192.370
146.569
136.326
131.509
128.886
128.616
128.399
126.532
126.186
123.970
121.756
82.797
82.781
78.903
77.254
77.000
76.746
52.451
51.061
46.391
46.184
45.976
45.768
37.679
37.326
24.652
24.523
O
CHO
F3C
(3V)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

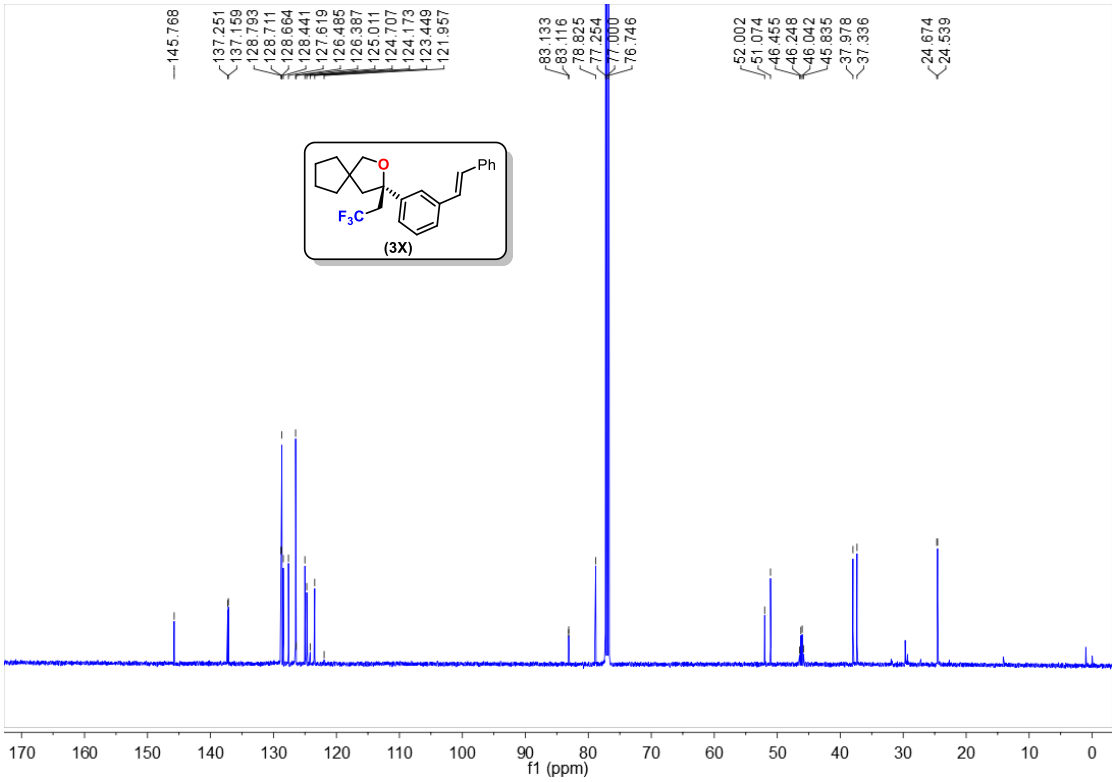

-60.260
-60.288
-60.316
O
CHO
F3C
(3V)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

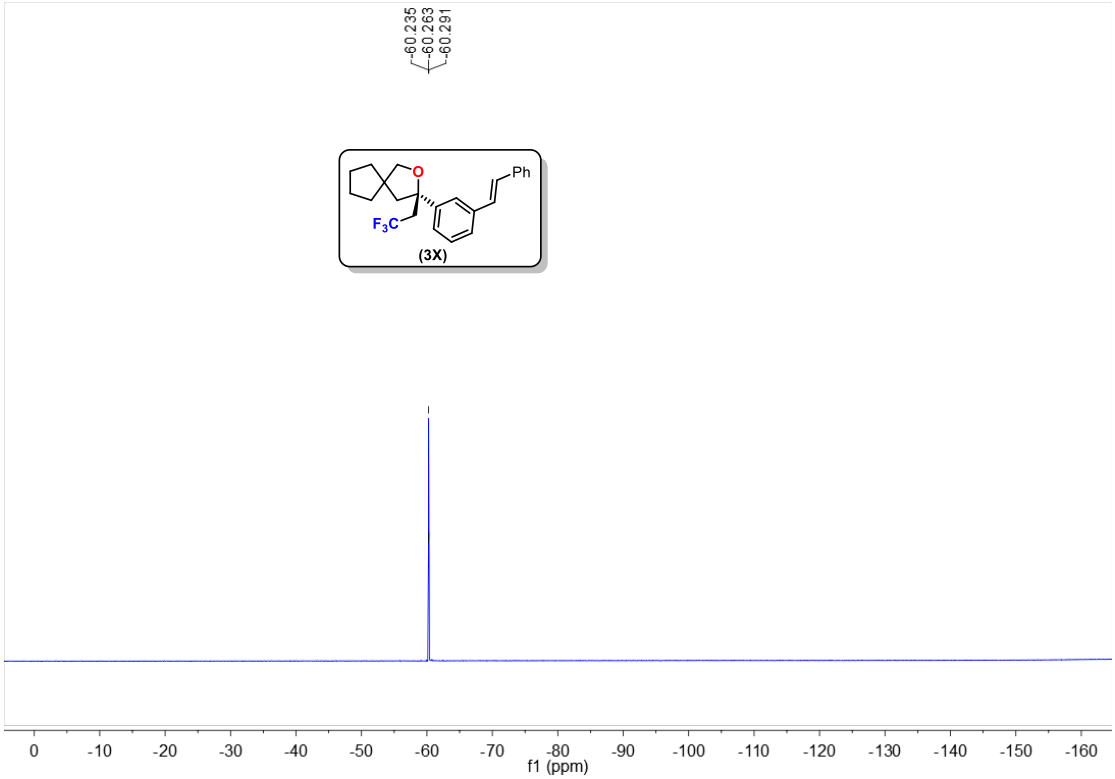

7.413
7.331
7.329
7.324
7.316
7.262
7.252
7.241
4.688
3.805
3.784
3.667
3.647
2.709
2.682
2.655
2.629
2.409
2.377
2.318
2.286
2.040
1.783
1.777
1.765
1.755
1.737
1.715
1.666
1.660
1.651
1.632
1.623
1.612
1.595
1.584
1.575
1.570
1.560
1.549
1.539
1.532
1.522
1.506
1.490
1.483
1.473
1.465
1.453
1.450
1.438
1.359
1.345
1.341
1.327
1.314
1.308
1.295
1.257
1.238
1.224
1.219
1.206
1.187
O
OH
F3C
(3W)
1.01
1.96
1.03
2.01
1.00
0.99
1.11
1.00
1.02
1.05
1.00
1.25
5.30
1.22
1.24
f1 (ppm)

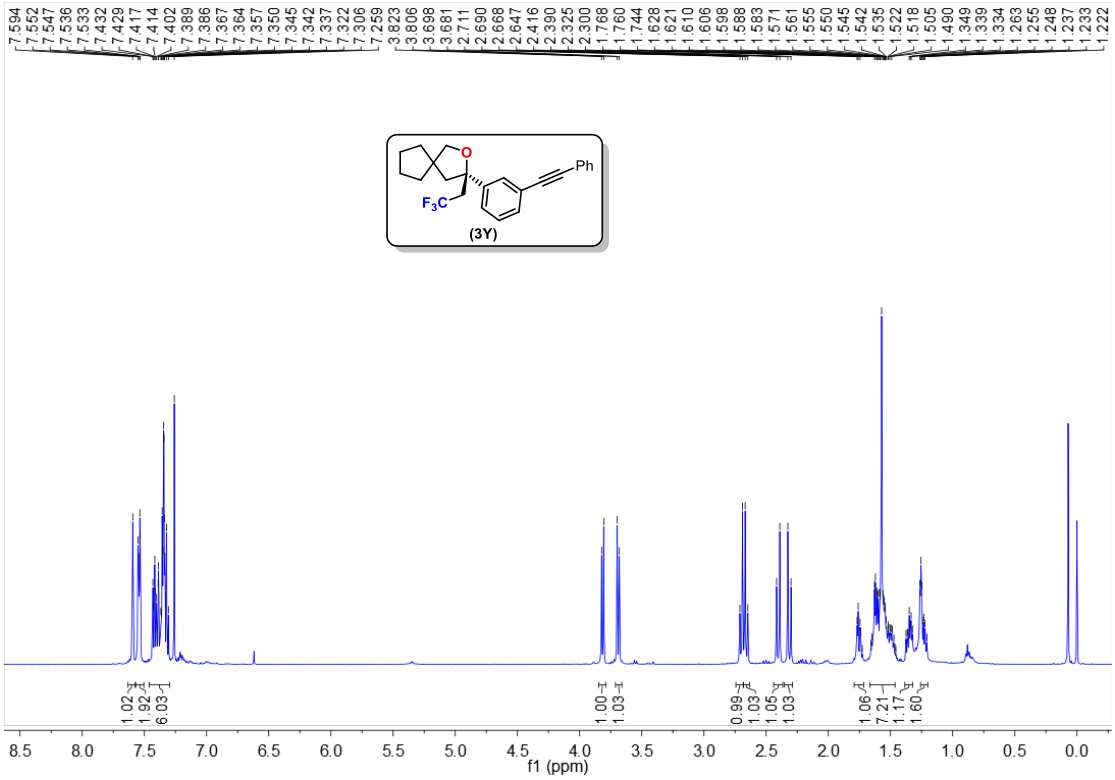

145.628
140.754
128.558
128.273
126.343
125.492
124.560
124.129
123.712
121.914
83.119
83.105
78.716
77.255
77.000
76.746
65.262
52.104
51.017
46.390
46.184
45.978
45.772
37.904
37.232
24.601
24.500
O
OH
F3C
(3W)
f1 (ppm)

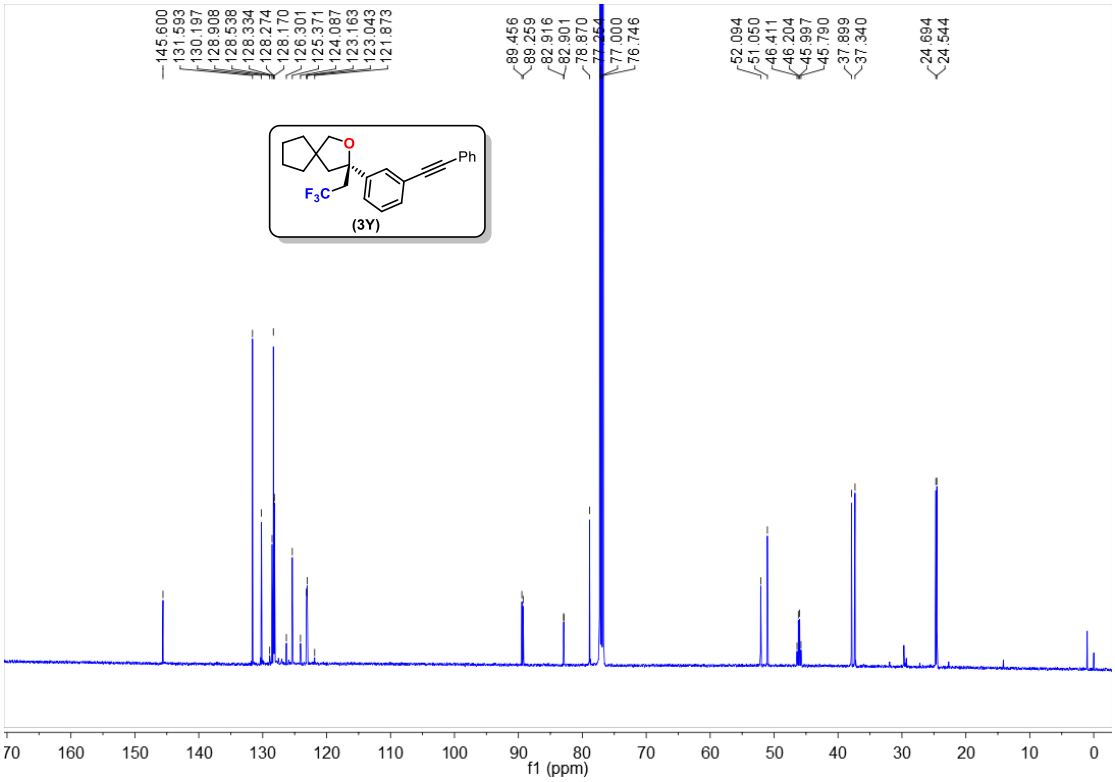

-60.237
-60.265
-60.294
O
OH
F3C
(3W)
f1 (ppm)

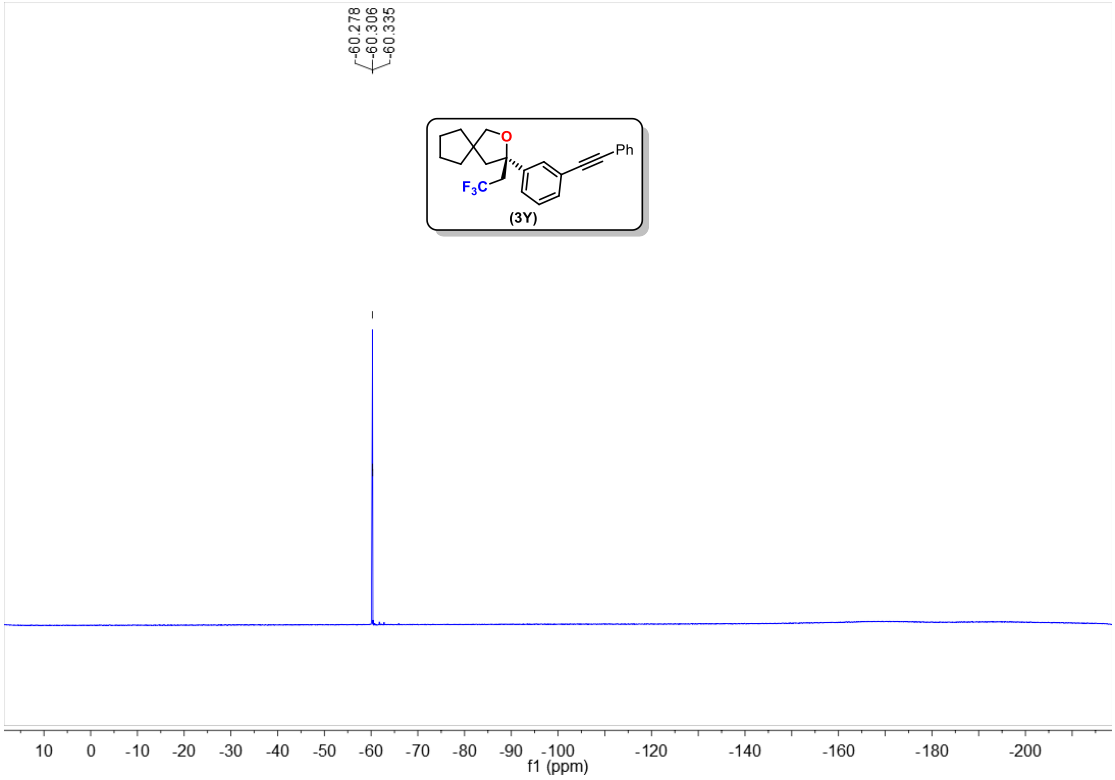

O
Ph
F3C
(3X)
1.00
2.00
1.02
3.03
2.54
2.00
1.00
1.01
1.00
1.02
1.01
1.02
1.06
6.61
1.27
3.21
f1 (ppm)

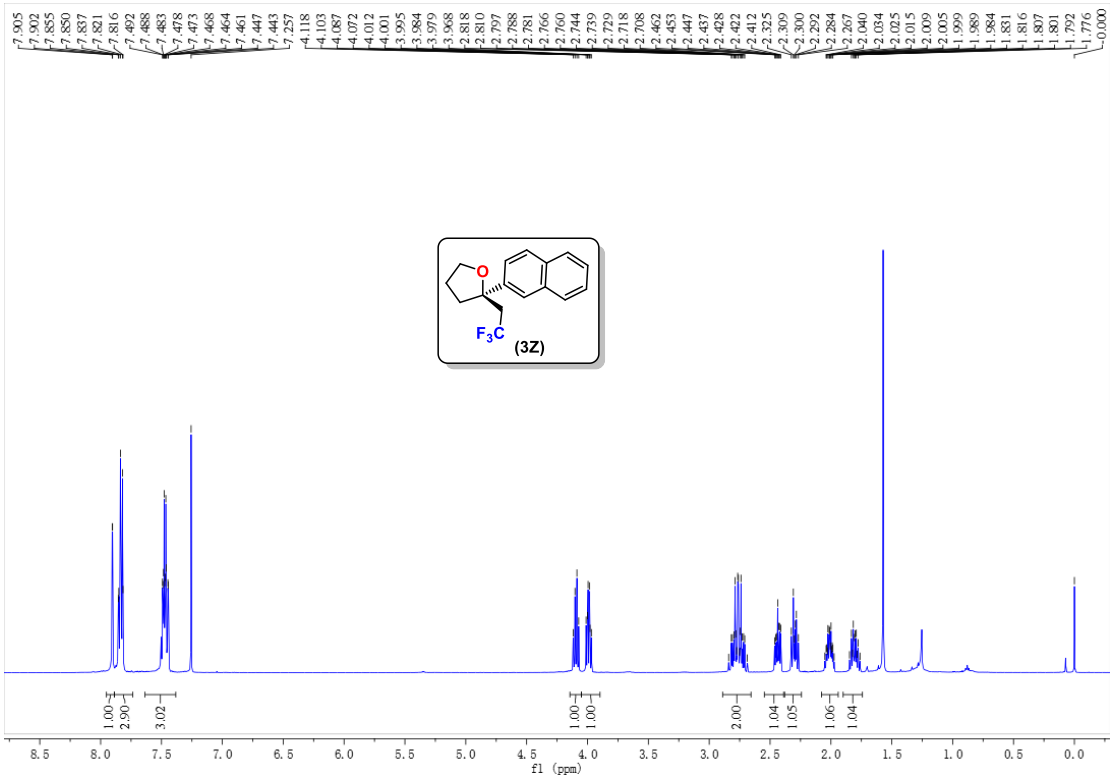

145.768
137.251
137.159
128.793
128.711
128.664
128.441
127.619
126.485
126.387
125.011
124.707
124.173
123.449
121.957
83.133
83.116
78.825
77.254
77.000
76.746
52.002
51.074
46.455
46.248
46.042
45.835
37.978
37.336
24.674
24.539
O
Ph
F3C
(3X)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

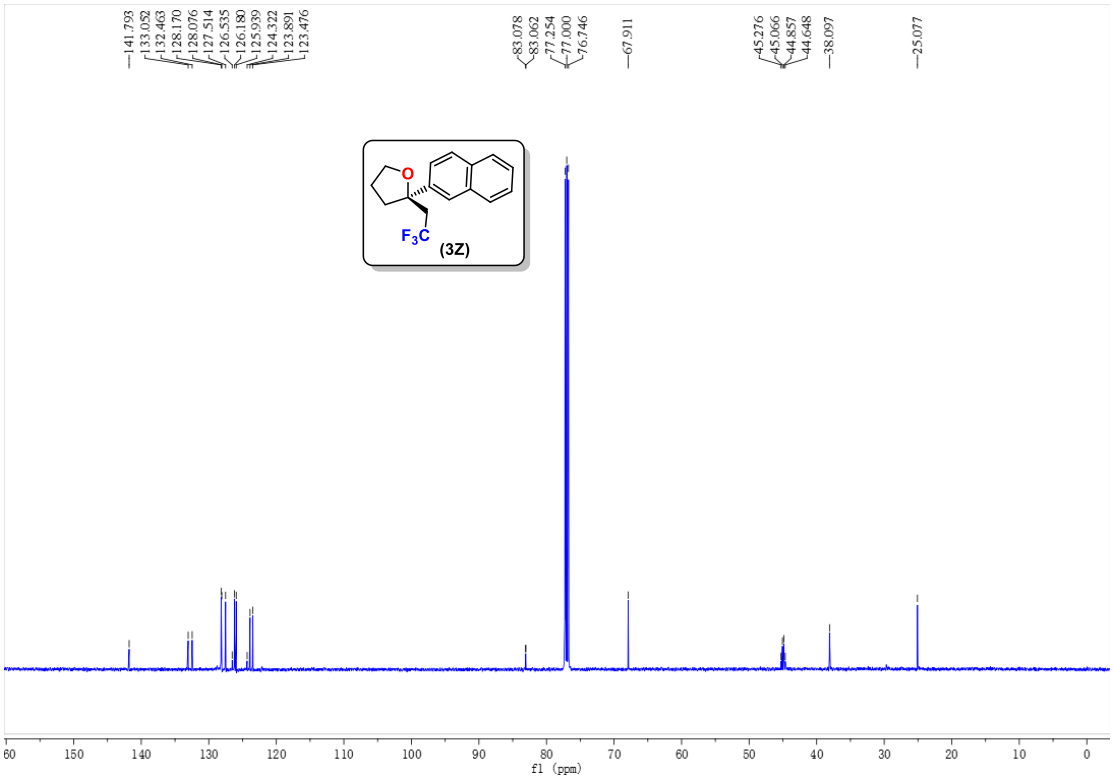

60.235
60.263
60.291
O
Ph
F3C
(3X)
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

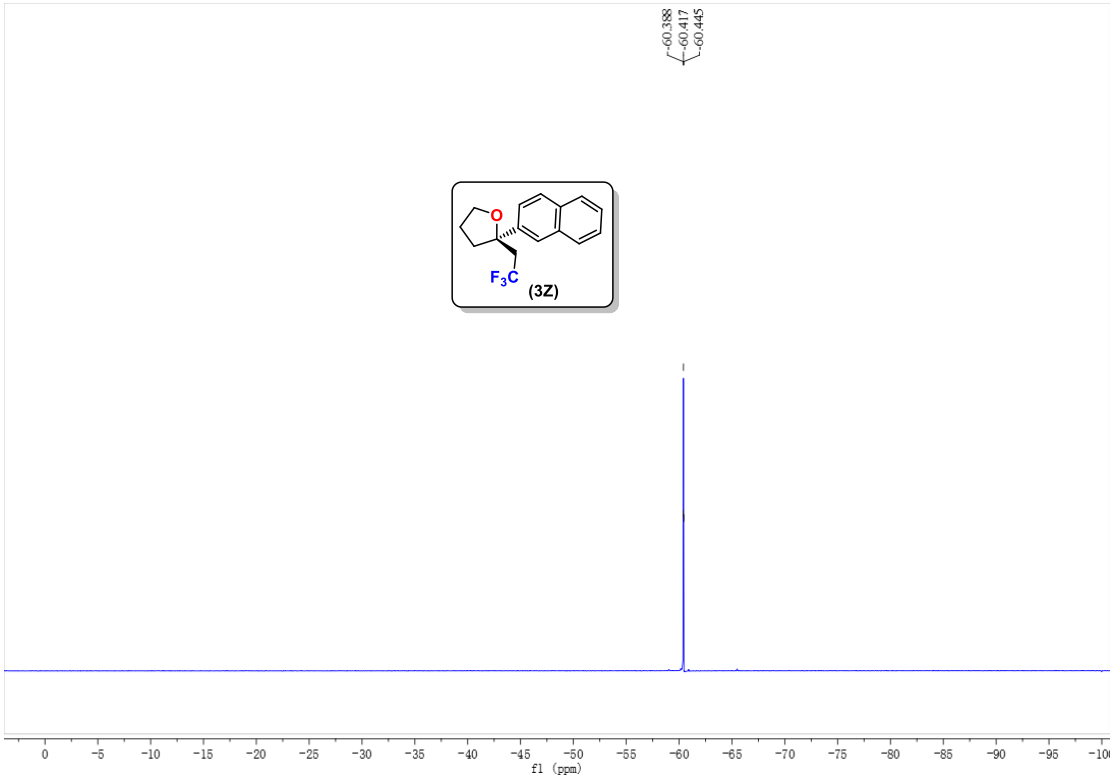

(3Y)
f1 (ppm)

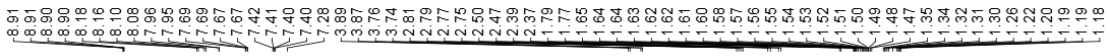

(3Y)
f1 (ppm)

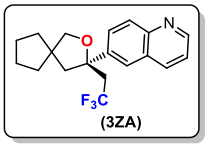

-60.278
-60.306
-60.335
O
Ph
F3C
(3Y)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)

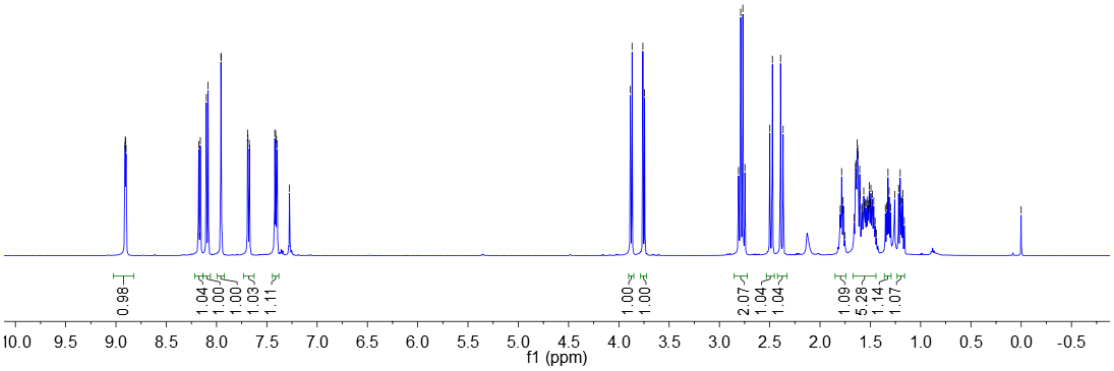

O
F3C
(3Z)
1.00 2.90 3.02 1.00 1.00 2.00 1.04 1.05 1.06 1.04
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

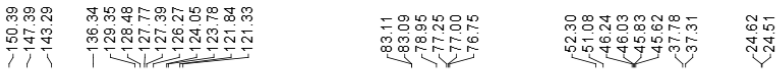

141.793
133.052
132.463
128.170
128.076
127.514
126.535
126.180
125.959
124.322
123.891
123.476
83.078
83.062
77.254
77.000
76.746
67.911
45.276
45.066
44.857
44.648
38.097
25.077
O
F3C
(3Z)
f1 (ppm)

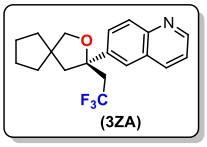

60.388
60.417
60.445
O
F3C
(3Z)
f1 (ppm)

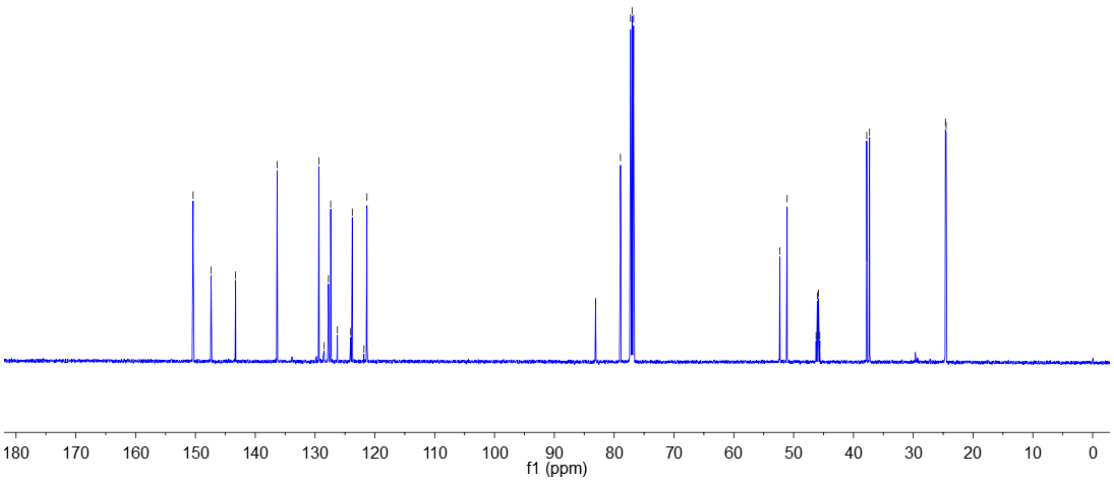

8.91
8.91
8.90
8.90
8.18
8.16
8.10
8.08
7.96
7.95
7.69
7.69
7.67
7.67
7.42
7.41
7.40
7.40
7.28
3.89
3.87
3.76
3.74
2.81
2.79
2.77
2.75
2.50
2.47
2.39
2.37
1.79
1.77
1.65
1.64
1.64
1.63
1.62
1.62
1.61
1.60
1.58
1.57
1.56
1.55
1.54
1.53
1.52
1.51
1.50
1.49
1.48
1.47
1.35
1.34
1.32
1.31
1.30
1.26
1.22
1.20
1.19
1.19
1.18

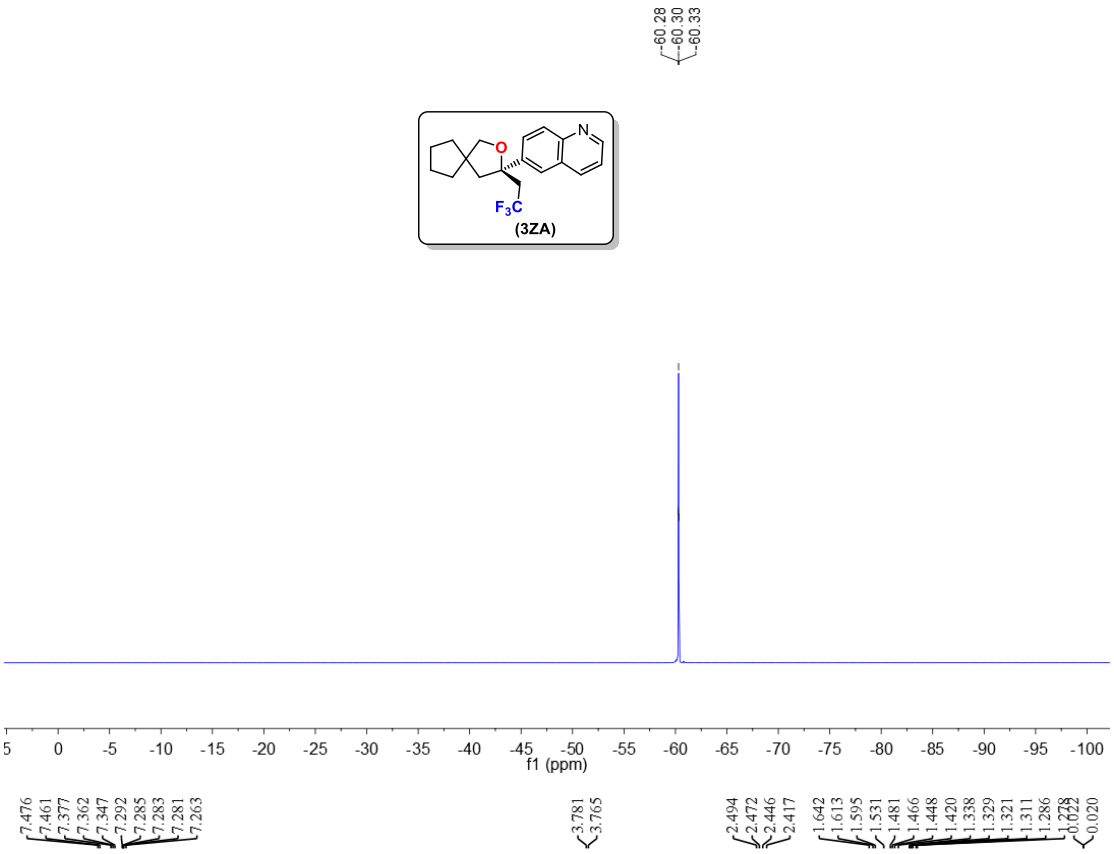

O
N
F3C
(3ZA)

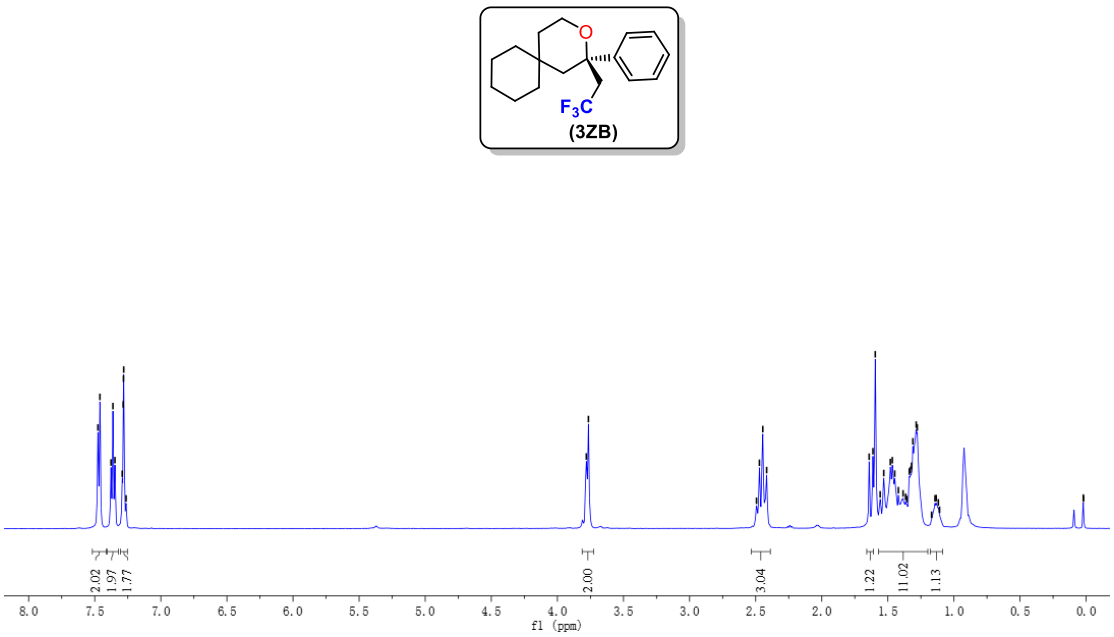

0.98
1.04
1.00
1.03
1.11
1.00
1.00
2.07
1.04
1.04
1.09
5.28
1.14
1.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

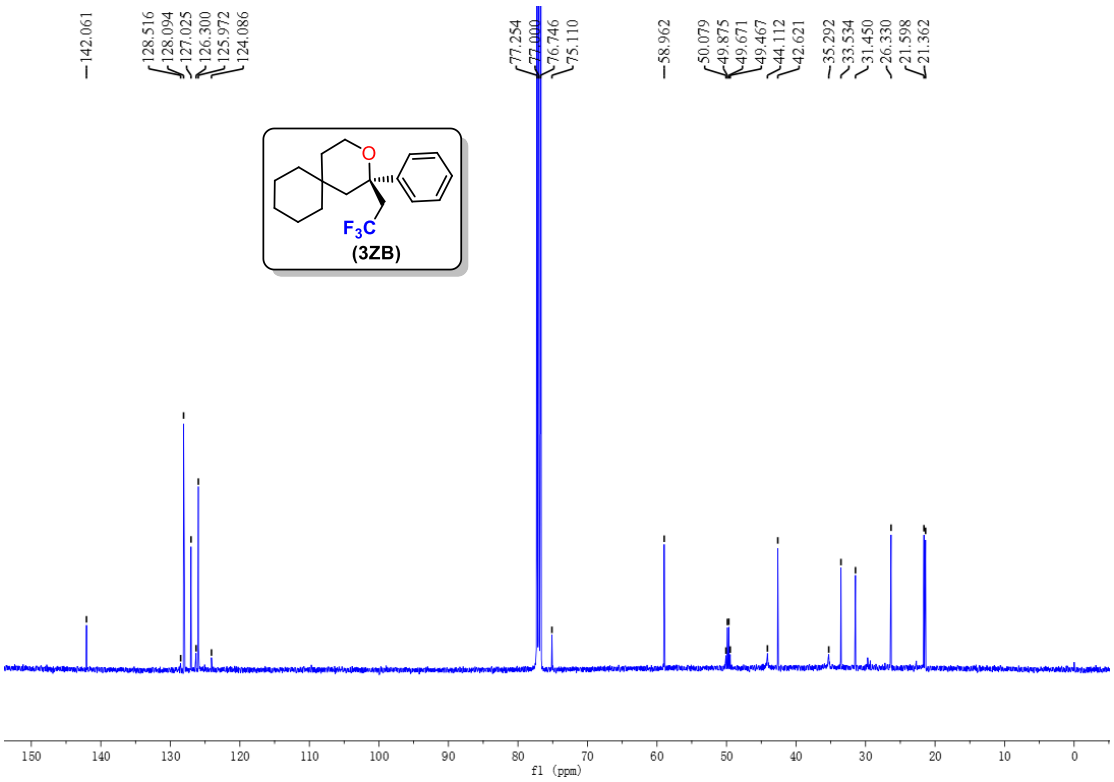

150.39
147.39
143.29
136.34
129.35
128.48
127.77
127.39
126.27
124.05
123.78
121.84
121.33
83.11
83.09
78.95
77.25
77.00
76.75
52.30
51.08
46.24
46.03
45.83
45.62
37.78
37.31
24.62
24.51

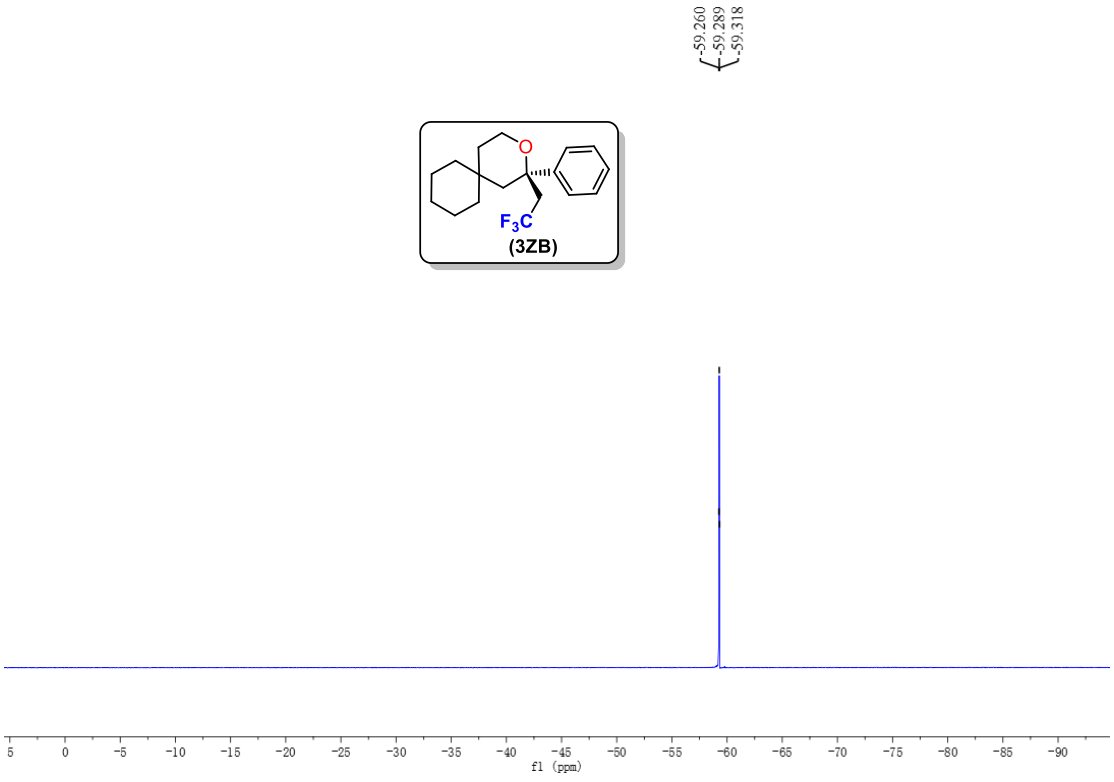

O
N
F3C
(3ZA)


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-60.28
-60.30
-60.33
O
N
F3C
(3ZA)
f1 (ppm)
7.476
7.461
7.377
7.362
7.347
7.292
7.285
7.283
7.281
7.263
3.781
3.765
2.494
2.472
2.446
2.417
1.642
1.613
1.595
1.531
1.481
1.466
1.448
1.420
1.338
1.329
1.321
1.311
1.286
0.022
0.020


O
F3C
(3ZB)
2.02
1.97
1.77
2.00
3.04
1.22
11.02
1.13
f1 (ppm)


142.061
128.516
128.094
127.025
126.300
125.972
124.086
77.254
77.000
76.746
75.110
58.962
50.079
49.875
49.671
49.467
44.112
42.621
35.292
33.534
31.450
26.330
21.598
21.362
O
F3C
(3ZB)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-59.260
-59.289
-59.318
O
F3C
(3ZB)
5
0
-5
-10
-15
-20
-25
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-50
-55
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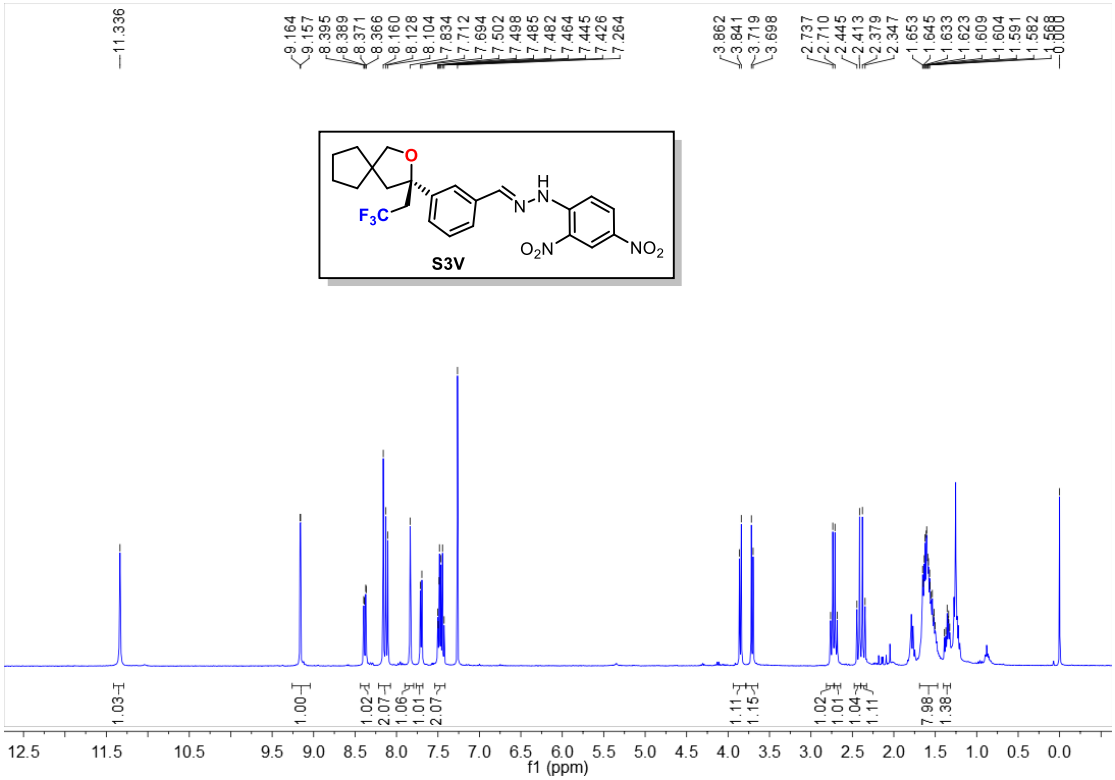
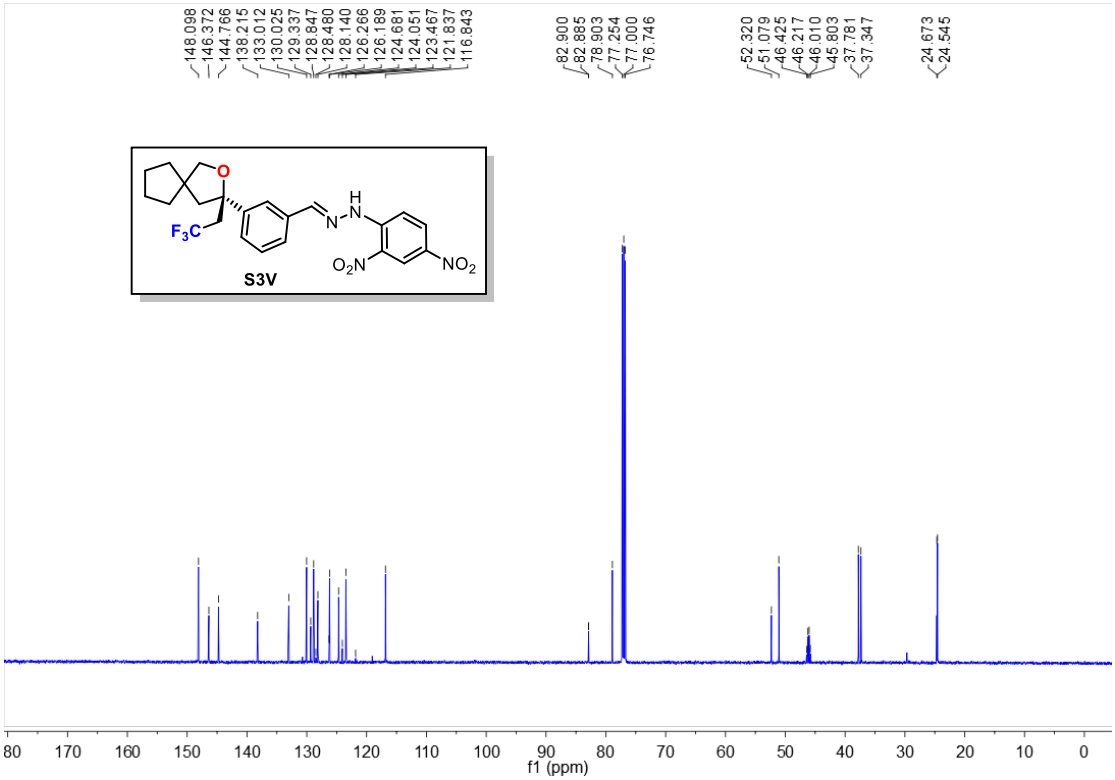
-65
-70
-75
-80
-85
-90
f1 (ppm)


11.336
9.164
9.157
8.395
8.389
8.371
8.366
8.160
8.128
8.104
7.834
7.712
7.694
7.502
7.498
7.485
7.482
7.464
7.445
7.426
7.264
3.862
3.841
3.719
3.698
2.737
2.710
2.445
2.413
2.379
2.347
1.653
1.645
1.633
1.623
1.609
1.604
1.591
1.582
1.568
0.000
S3V
1.03
1.00
1.02
2.07
1.06
1.01
2.07
1.11
1.15
1.02
1.01
1.04
1.11
7.98
1.38
f1 (ppm)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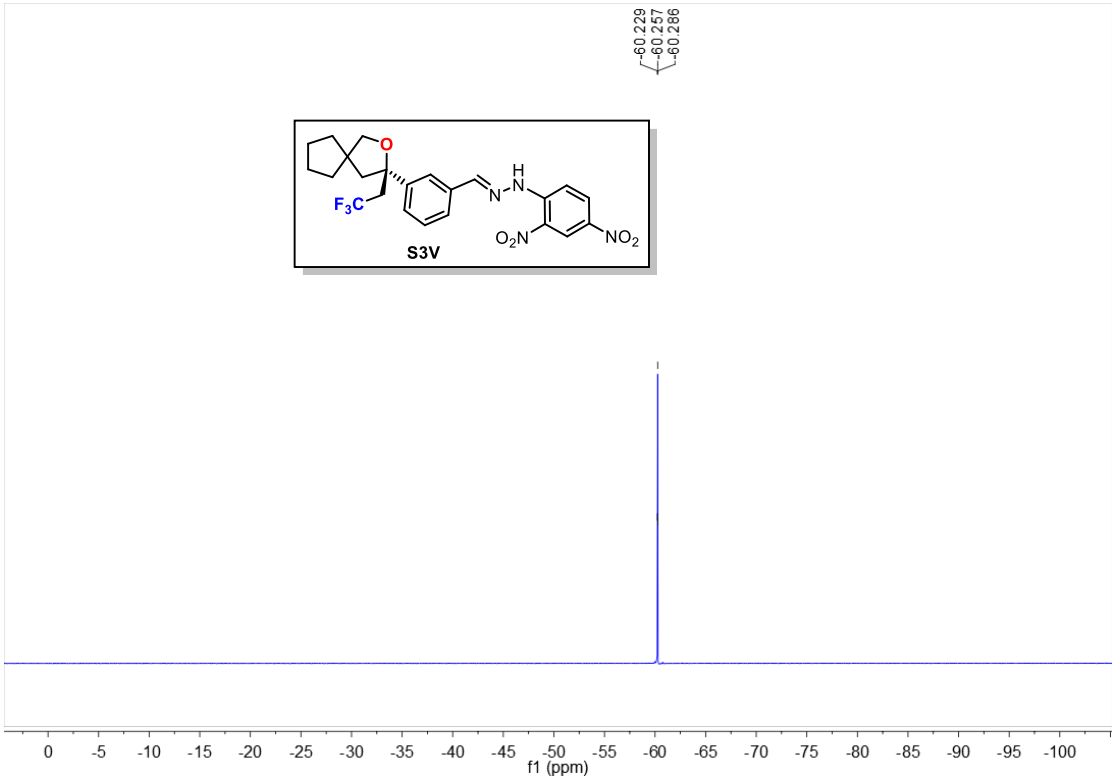
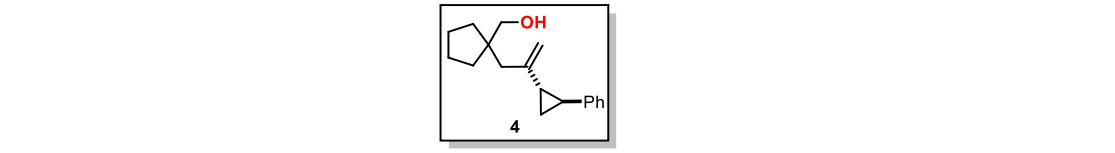
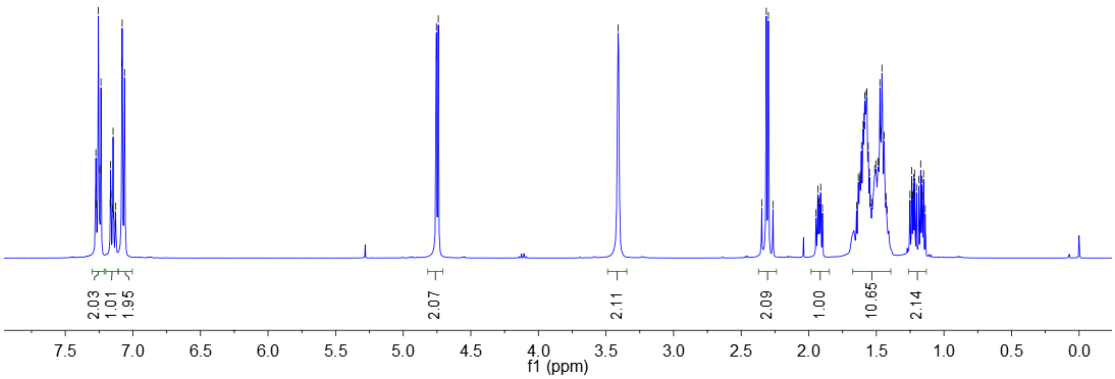
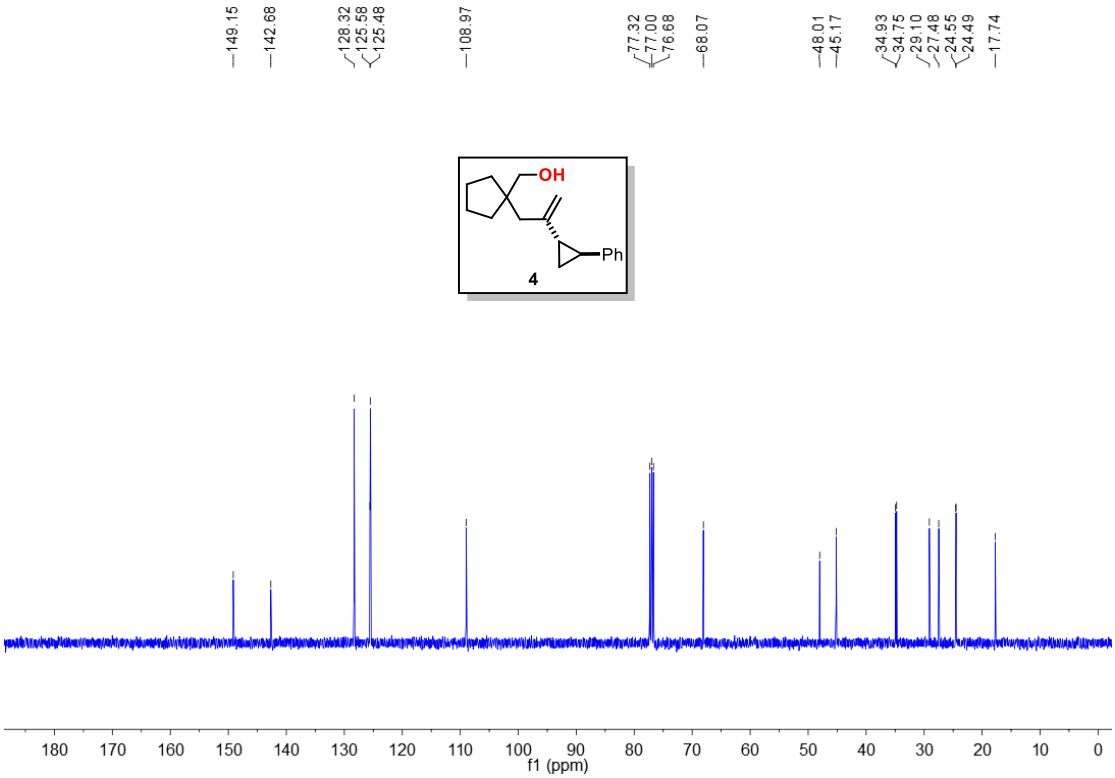
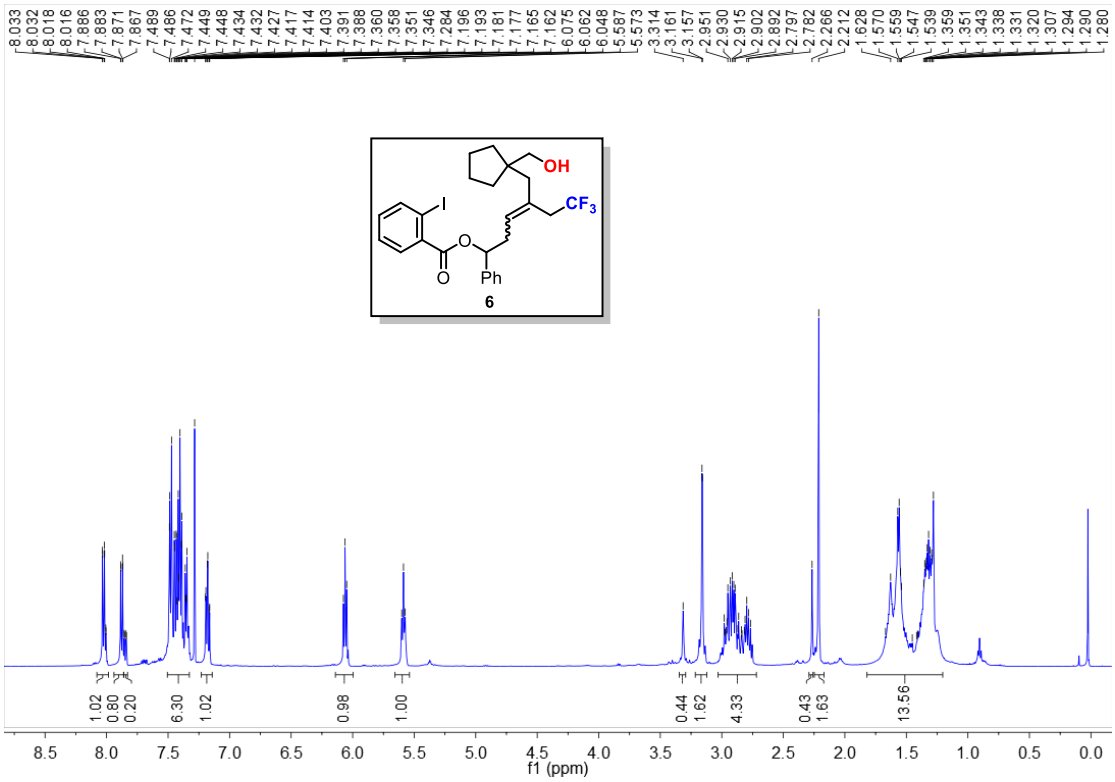
146.372
144.766
138.215
133.012
130.025
129.337
128.847
128.480
128.140
126.266
126.189
124.681
124.051
123.467
121.837
116.843
82.900
82.885
78.903
77.254
77.000
76.746
52.320
51.079
46.425
46.217
46.010
45.803
37.781
37.347
24.673
24.545
S3V
f1 (ppm)

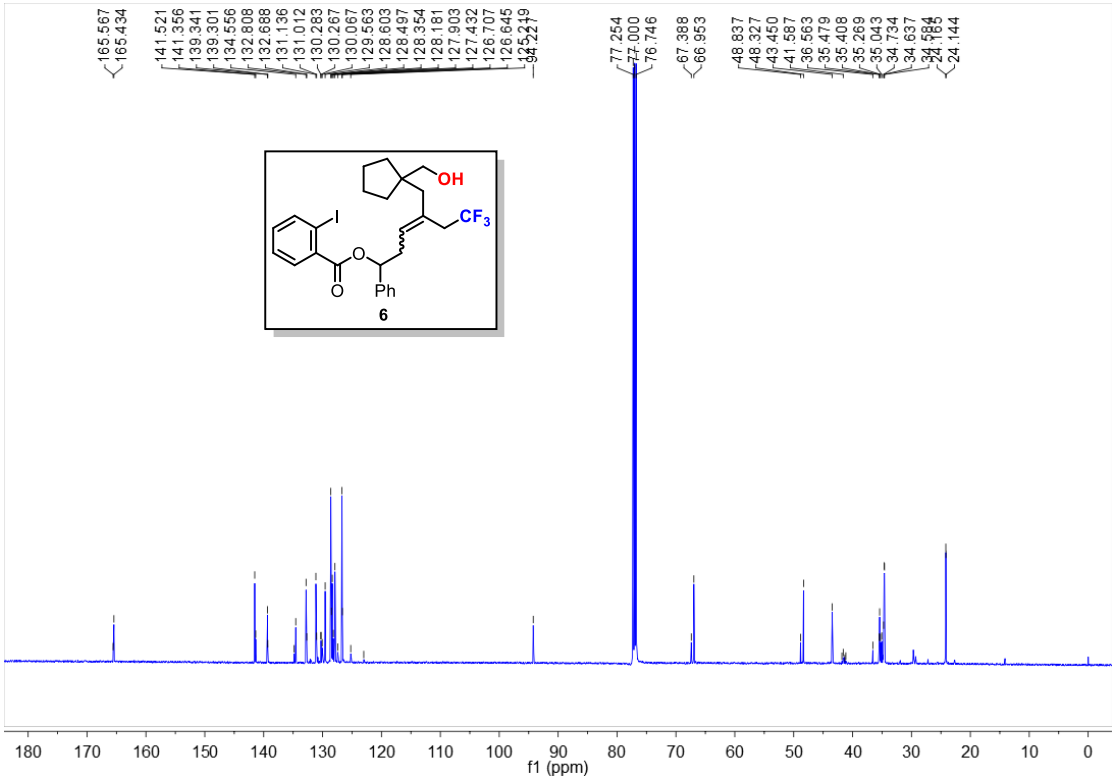

-60.229
-60.257
-60.286
S3V
f1 (ppm)

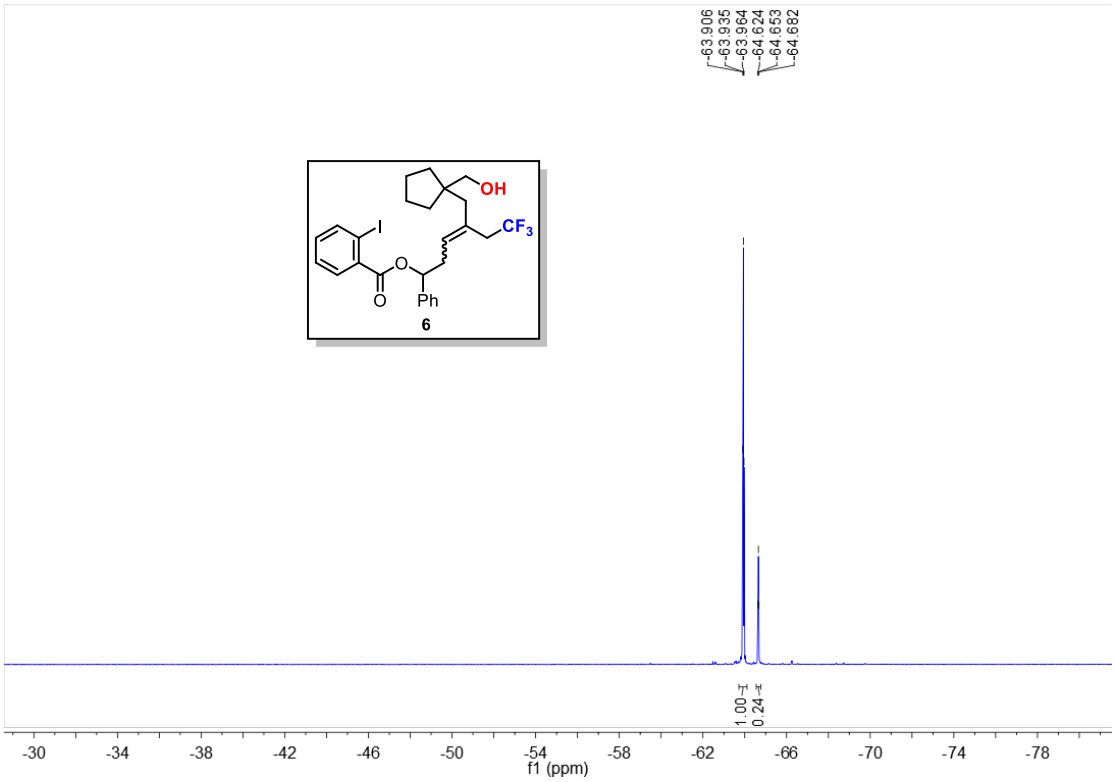

4


2.03
1.01
1.95
2.07
2.11
2.09
1.00
10.65
2.14
f1 (ppm)

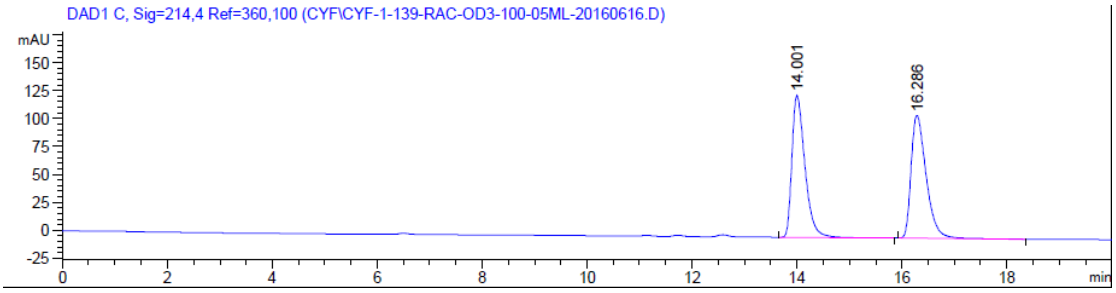

149.15
142.68
128.32
125.58
125.48
108.97
77.32
77.00
76.68
68.07
48.01
45.17
34.93
34.75
29.10
27.48
24.55
24.49
17.74
OH
Ph
4
f1 (ppm)


OH
CF3
I
O
O
Ph
6
1.02
0.80
0.20
6.30
1.02
0.98
1.00
0.44
1.62
4.33
0.43
1.63
13.56
f1 (ppm)


165.567
165.434
141.521
141.356
139.341
139.301
134.556
132.808
132.688
131.136
131.012
130.283
130.267
130.067
129.563
128.603
128.497
128.354
128.181
127.903
127.432
126.707
126.645
94.221
77.254
77.000
76.746
67.388
66.953
48.837
48.327
43.450
41.587
36.563
35.479
35.408
35.269
35.043
34.734
34.637
24.144
OH
CF3
I
O
O
Ph
6
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

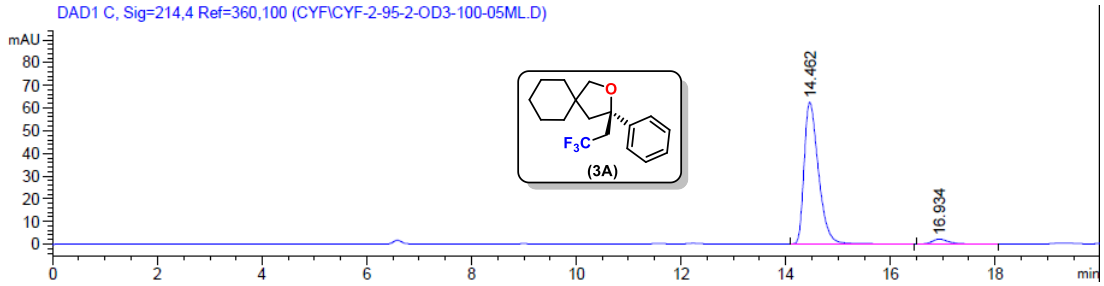

-63.906
-63.935
-63.964
-64.624
-64.653
-64.682
OH
CF3
I
O
O
Ph
6
1.00
0.24
-30
-34
-38
-42
-46
-50
-54
-58
-62
-66
-70
-74
-78
f1 (ppm)

HPLC Spectra



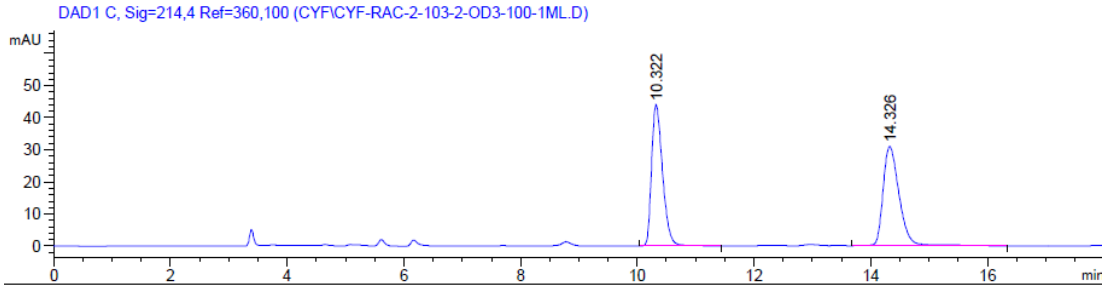
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.001	BB	0.2631	2192.45459	127.20431	49.9179
2	16.286	BB	0.3049	2199.66406	110.20741	50.0821
Totals :				4392.11865	237.41172	



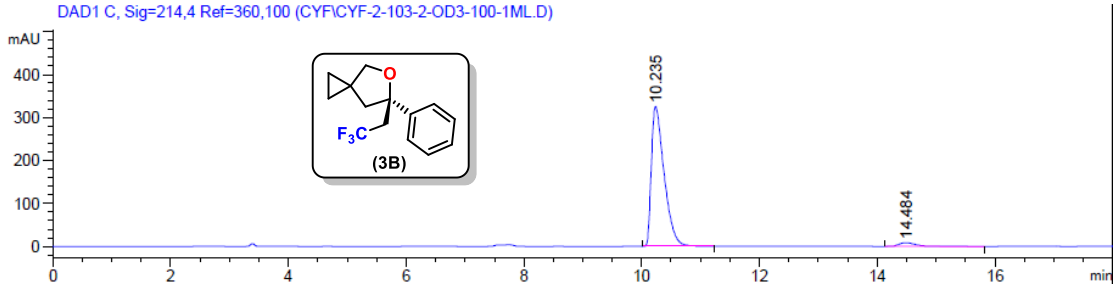
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.462	BB	0.2891	1184.07837	62.46677	96.2962
2	16.934	BB	0.3193	45.54252	2.14847	3.7038
Totals :				1229.62089	64.61523	



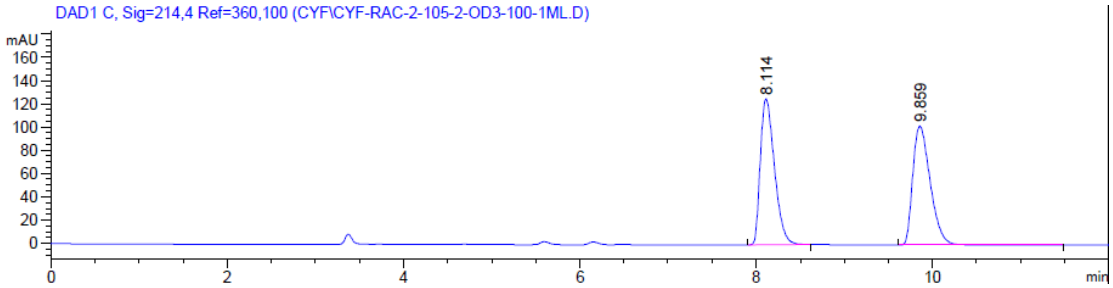
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.322	BB	0.2018	571.68567	43.93886	49.2395
2	14.326	BB	0.2946	589.34393	30.88582	50.7605
Totals :				1161.02960	74.82467	



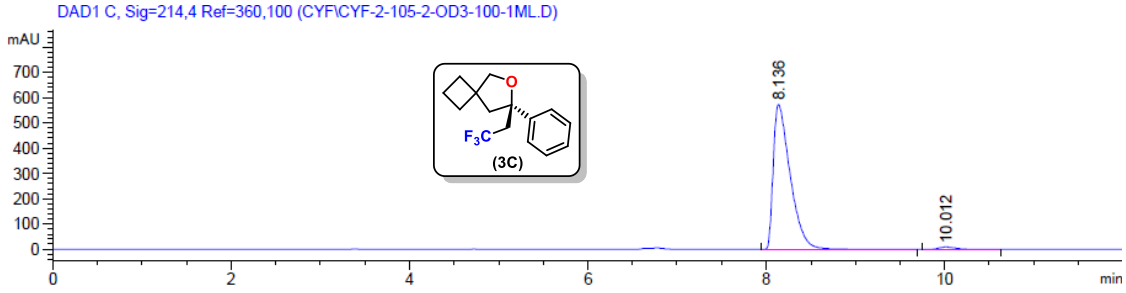
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.235	BB	0.2275	4896.98535	325.47366	96.6533
2	14.484	BB	0.2917	169.56030	8.84221	3.3467
Totals :				5066.54565	334.31588	



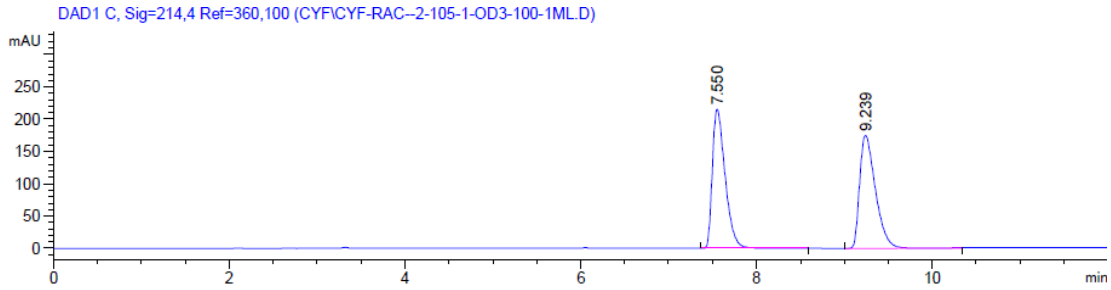
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.114	BB	0.1718	1394.61084	125.38605	49.7273
2	9.859	BB	0.2146	1409.90503	102.43404	50.2727
Totals :				2804.51587	227.82009	



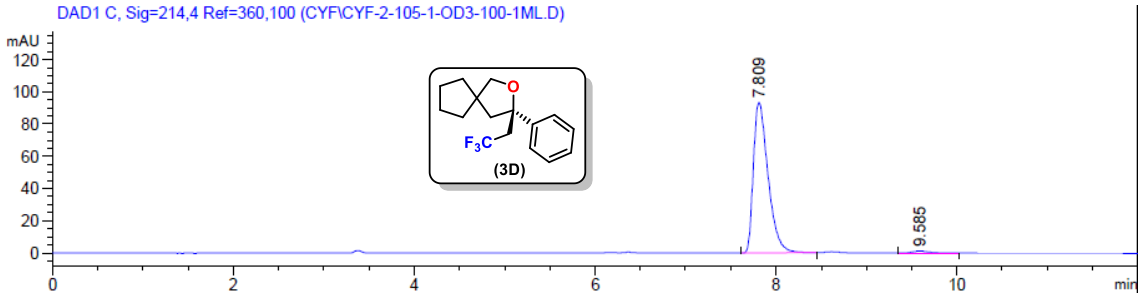
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.136	BB	0.1994	7540.33789	573.42328	98.4178
2	10.012	BB	0.2039	121.22294	9.19157	1.5822
Totals :				7661.56083	582.61485	



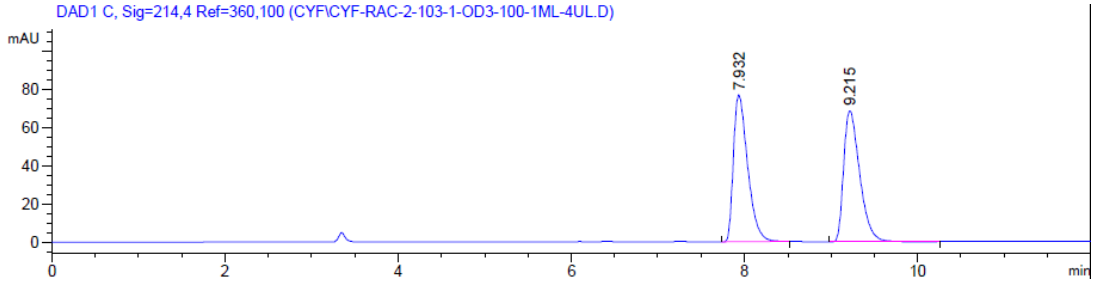
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.550	BB	0.1538	2172.76050	215.23274	49.9055
2	9.239	BB	0.1914	2180.99292	174.98257	50.0945
Totals :				4353.75342	390.21532	



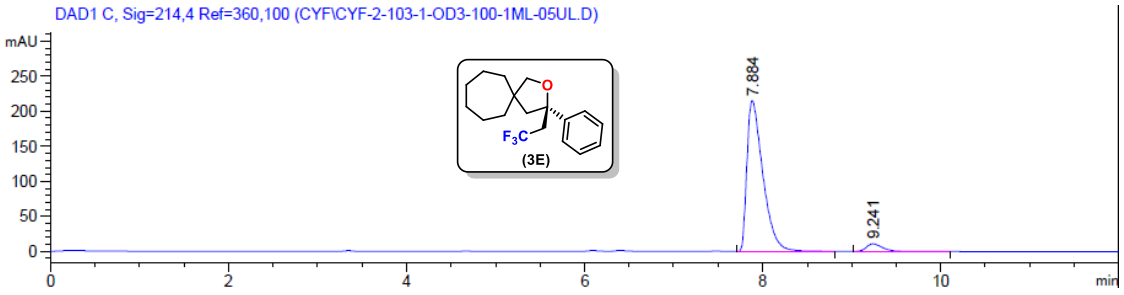
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.809	BB	0.1739	1057.53076	93.56413	98.5200
2	9.585	BB	0.1974	15.88694	1.25715	1.4800
Totals :				1073.41771	94.82129	



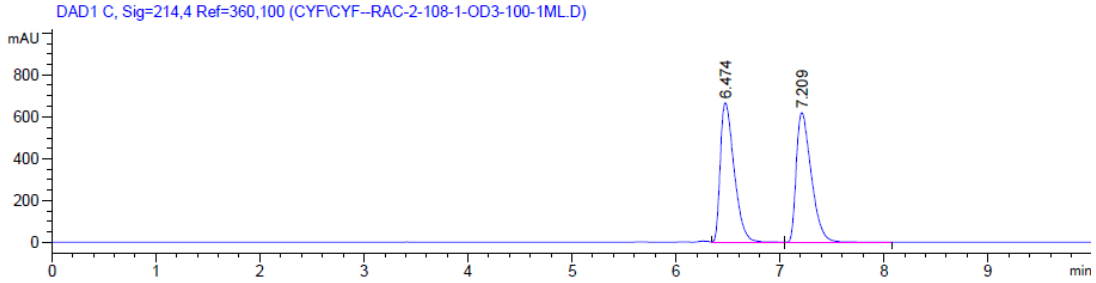
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.932	BB	0.1755	879.37006	76.86850	49.8423
2	9.215	BB	0.1983	884.93457	68.67804	50.1577
Totals :				1764.30463	145.54654	



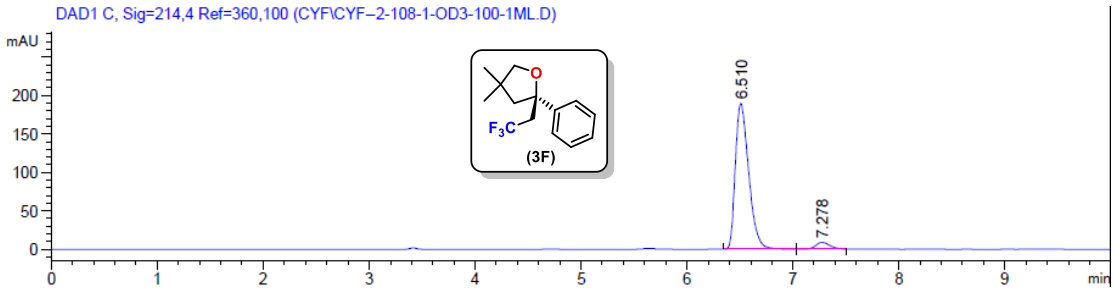
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.884	BB	0.1888	2673.96143	215.42506	95.1878
2	9.241	BB	0.1930	135.18243	10.73204	4.8122
Totals :				2809.14386	226.15710	



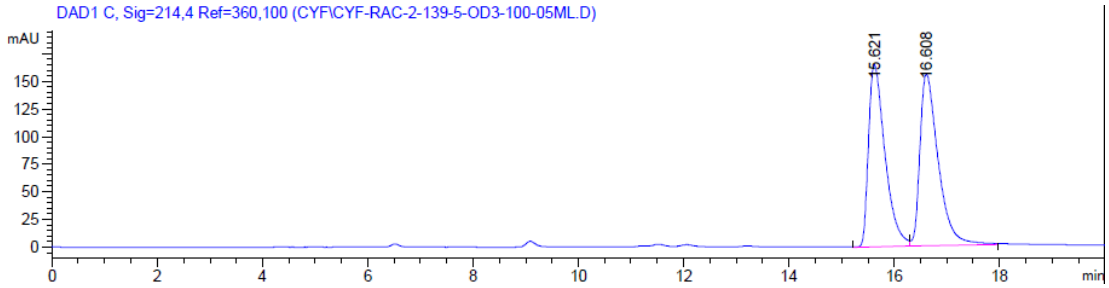
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.474	VB	0.1413	6131.03320	666.90344	49.8071
2	7.209	BB	0.1544	6178.51465	619.09796	50.1929
Totals :				1.23095e4	1286.00140	



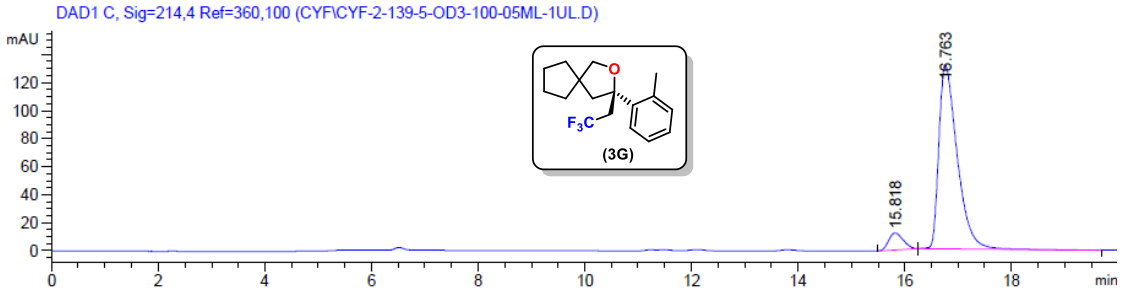
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.510	BB	0.1339	1653.70203	189.26572	95.6410
2	7.278	BB	0.1399	75.36951	8.46282	4.3590
Totals :				1729.07153	197.72853	



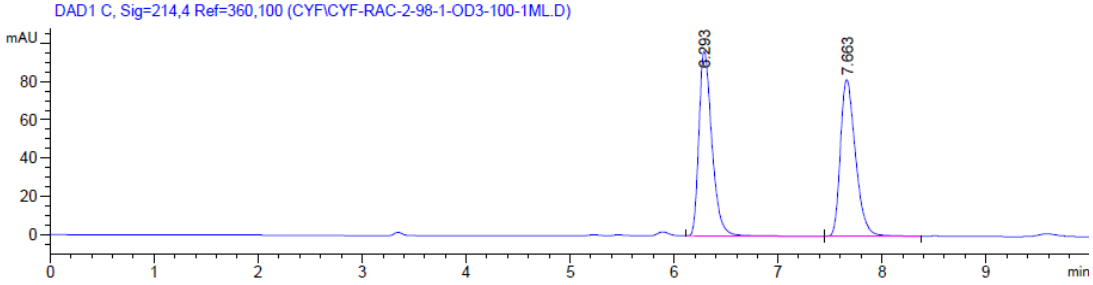
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.621	MF R	0.3630	3592.68945	164.97255	48.8554
2	16.608	FM R	0.4023	3761.02368	155.81226	51.1446
Totals :				7353.71313	320.78481	



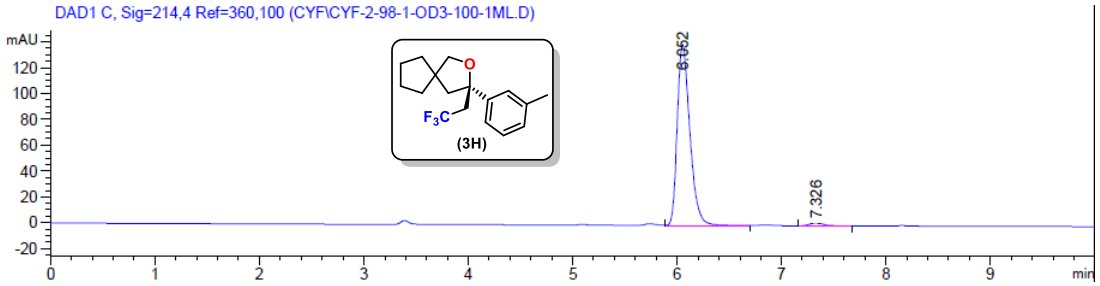
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.818	BB	0.2959	228.30884	12.11471	6.7845
2	16.763	BB	0.3639	3136.82642	130.73538	93.2155
Totals :				3365.13525	142.85009	



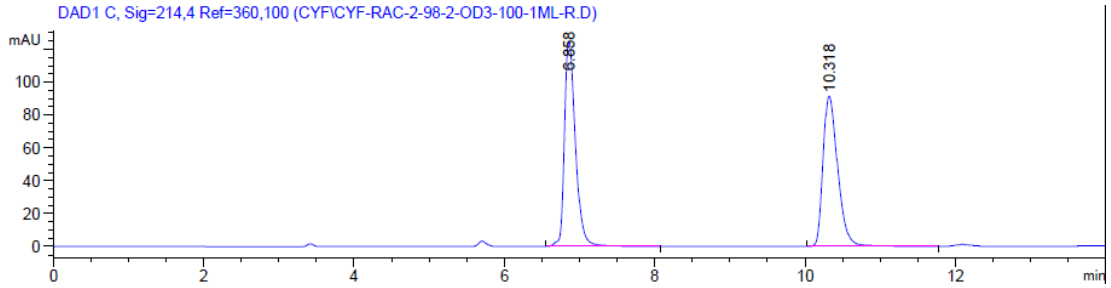
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.293	VB	0.1339	843.23993	96.58138	50.5242
2	7.663	BB	0.1556	825.74115	81.93203	49.4758
Totals :				1668.98108	178.51341	



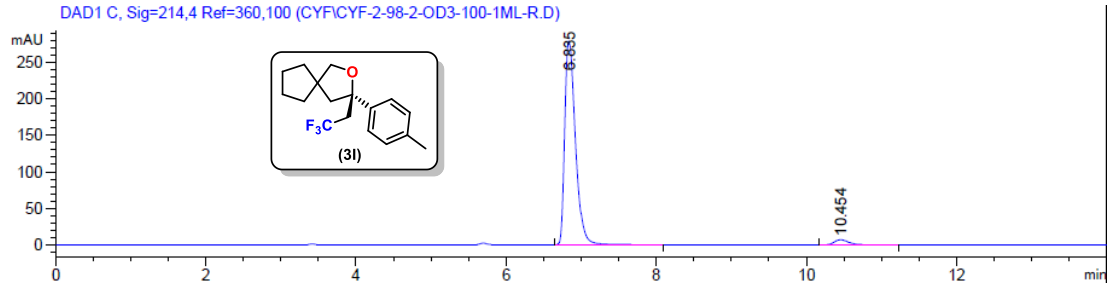
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.052	VB	0.1280	1173.71240	139.66466	98.2756
2	7.326	BB	0.1450	20.59449	2.16407	1.7244



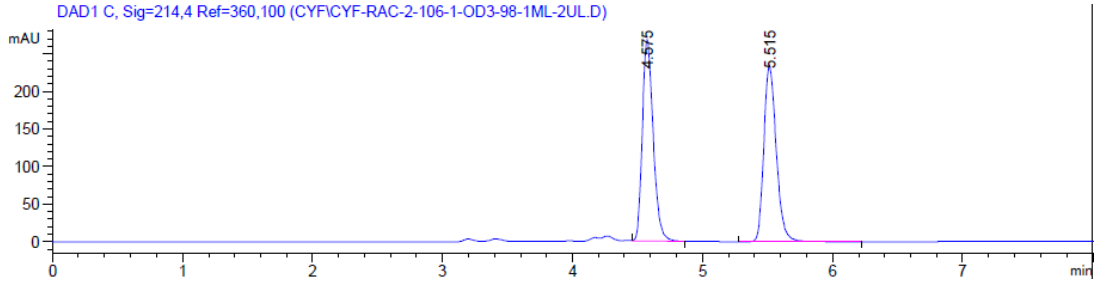
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.858	BB	0.1537	1259.00171	124.82137	50.3492
2	10.318	BB	0.2084	1241.53918	91.43759	49.6508
Totals :				2500.54089	216.25896	



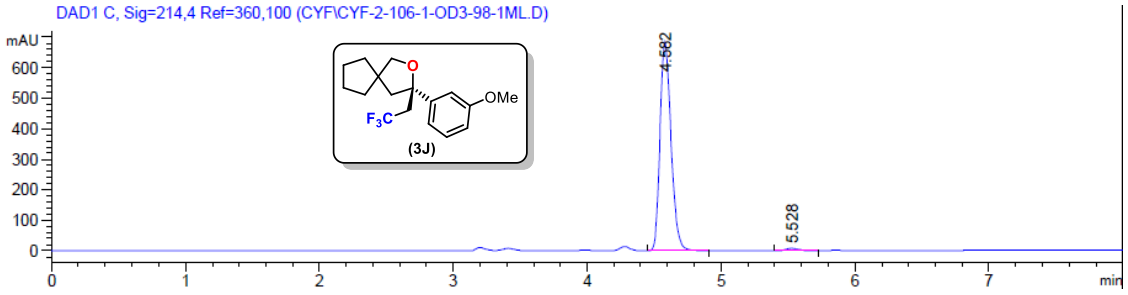
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.835	BB	0.1515	2762.07153	278.95062	96.6996
2	10.454	BB	0.2083	94.27089	7.03517	3.3004
Totals :				2856.34242	285.98579	



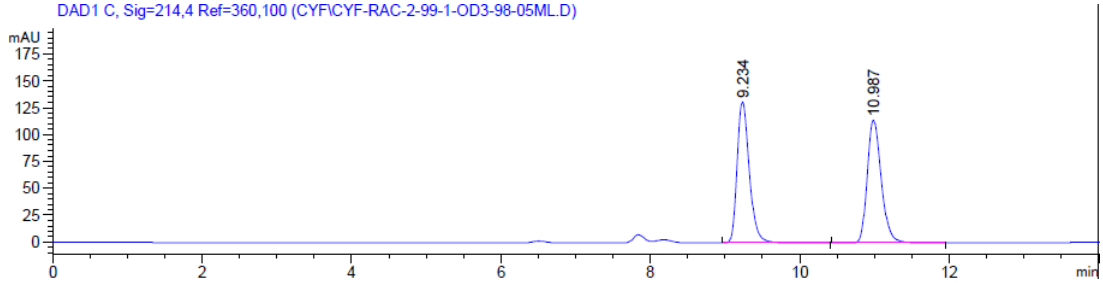
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.575	VB	0.0896	1551.50708	267.85944	49.9028
2	5.515	BB	0.1035	1557.55139	234.53851	50.0972
Totals :				3109.05847	502.39795	



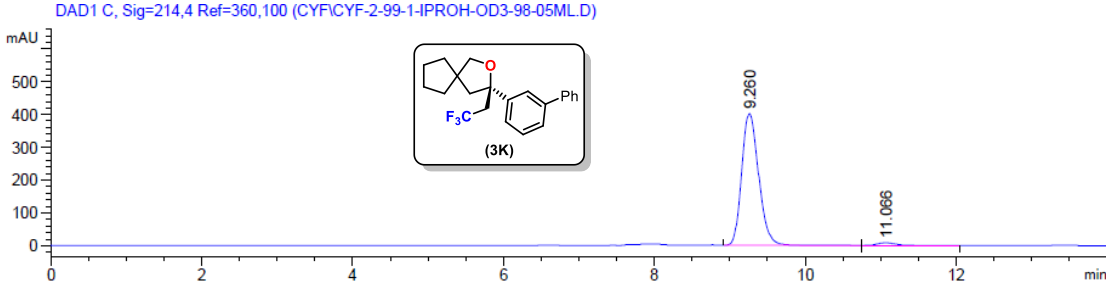
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.582	BB	0.0886	3911.45264	684.73889	98.6773
2	5.528	BV	0.1019	52.42982	8.05972	1.3227
Totals :				3963.88246	692.79861	



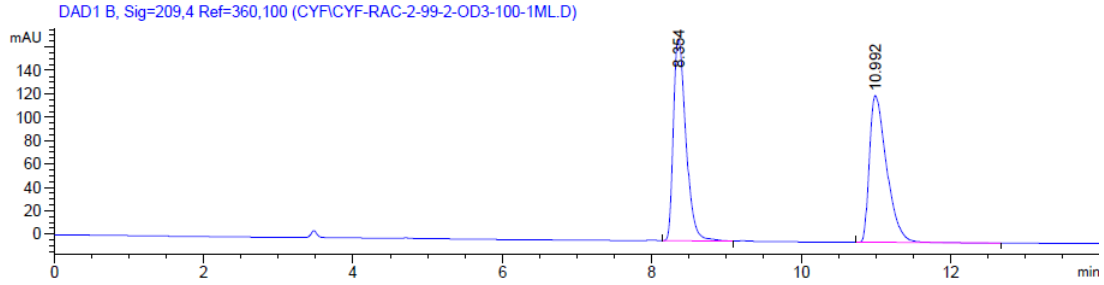
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.234	BB	0.1746	1490.06433	131.14017	49.9889
2	10.987	BB	0.2024	1490.72595	114.15093	50.0111
Totals :				2980.79028	245.29110	



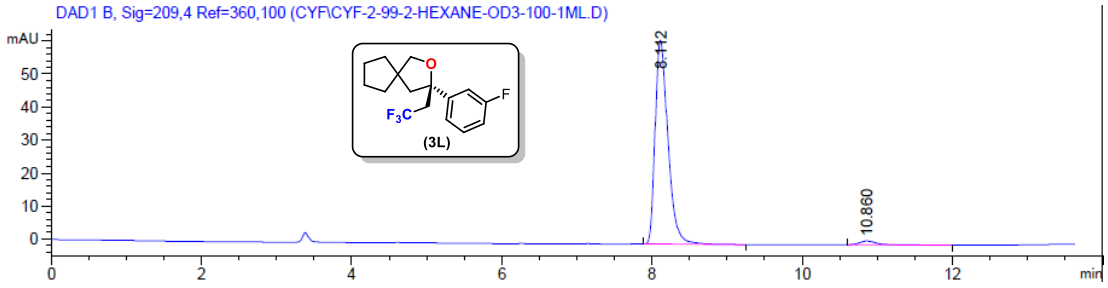
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.260	VB	0.2446	6280.60596	401.24564	98.0397
2	11.066	BB	0.2480	125.58305	7.87529	1.9603
Totals :				6406.18900	409.12093	



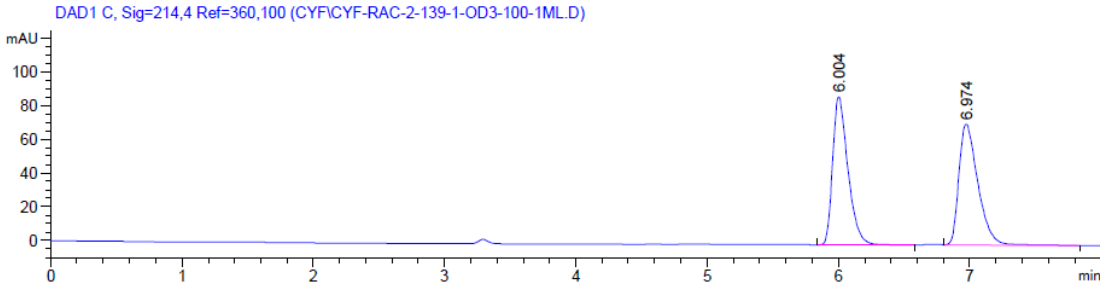
Signal 2: DAD1 B, Sig=209,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.354	BB	0.1775	2006.96790	172.77084	49.9020
2	10.992	BB	0.2453	2014.85352	125.52457	50.0980
Totals :				4021.82141	298.29541	



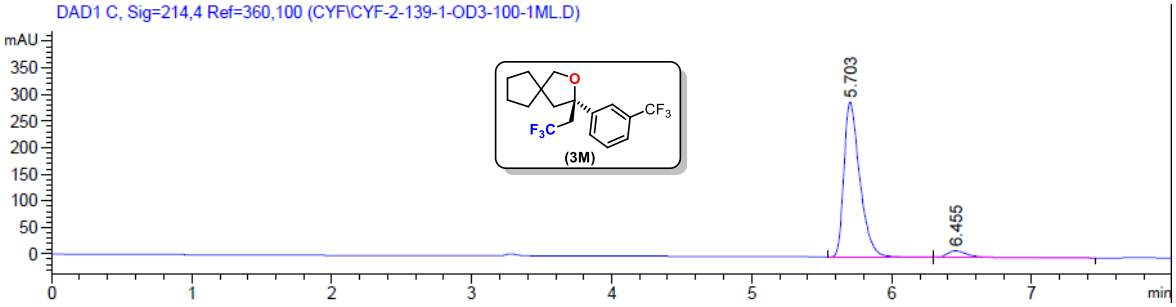
Signal 2: DAD1 B, Sig=209,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.112	BB	0.1822	731.69269	61.77695	97.8018
2	10.860	BB	0.2300	16.44520	1.10244	2.1982
Totals :				748.13789	62.87939	



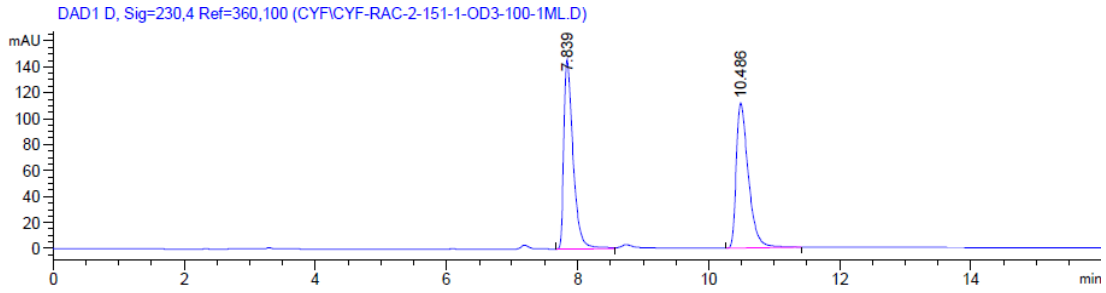
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.004	BB	0.1223	710.33728	87.85398	49.8381
2	6.974	BB	0.1525	714.95215	71.56798	50.1619
Totals :				1425.28943	159.42196	



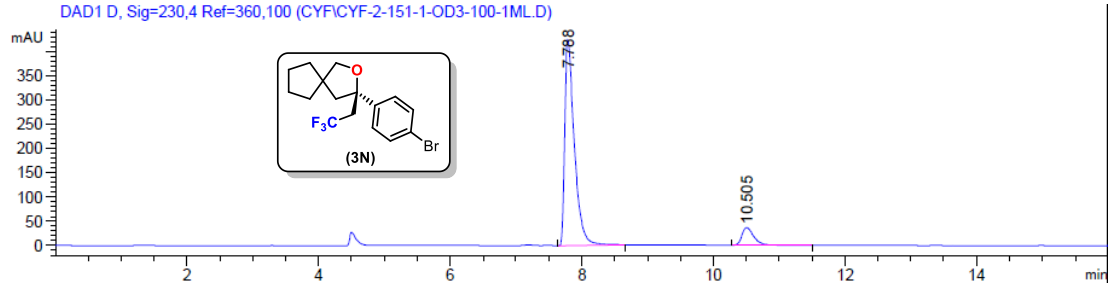
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.703	BV	0.1178	2295.92920	291.85461	94.9151
2	6.455	VB	0.1493	123.00082	12.23243	5.0849
Totals :				2418.93002	304.08705	



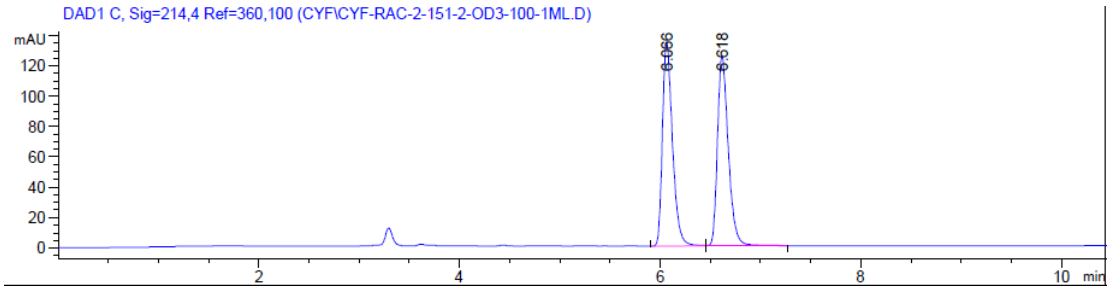
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.839	BV	0.1465	1437.96912	146.47818	50.0730
2	10.486	BB	0.1954	1433.77917	111.97457	49.9270
Totals :				2871.74829	258.45275	



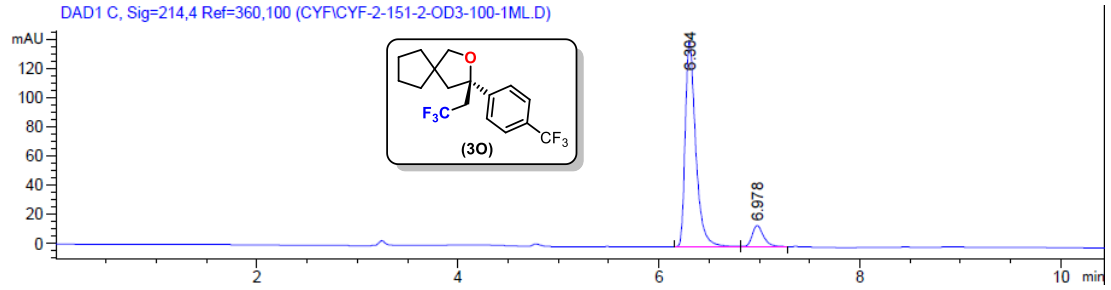
Signal 4: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.788	BB	0.1516	4279.95020	424.56146	90.3563
2	10.505	BB	0.1898	456.79767	36.55588	9.6437
Totals :				4736.74786	461.11734	



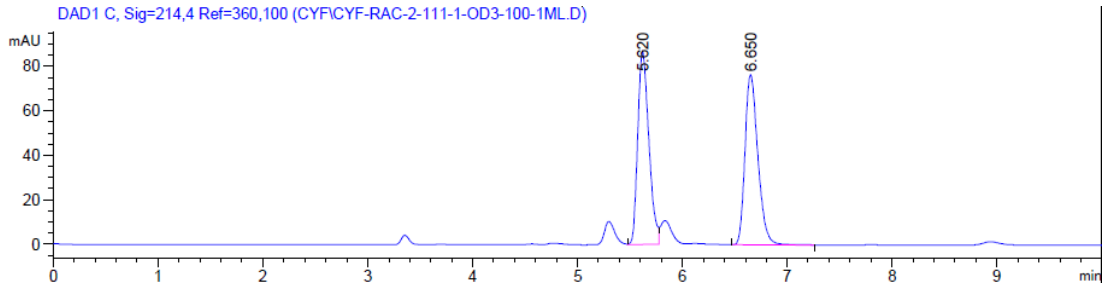
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.066	BV	0.1072	937.63562	134.71248	49.9314
2	6.618	VB	0.1137	940.21167	125.05933	50.0686



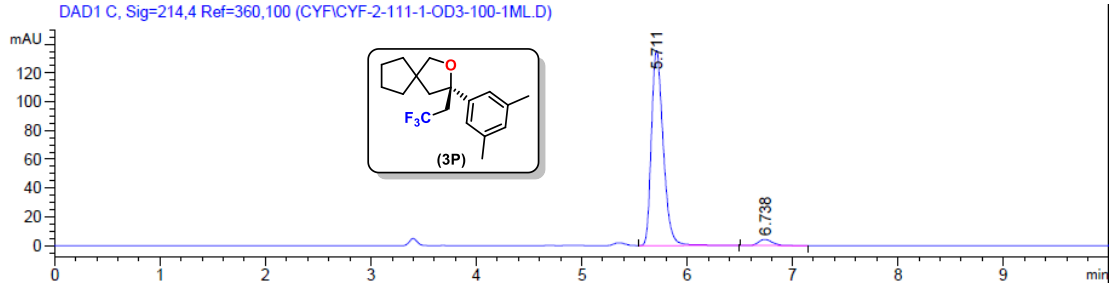
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.304	BV	0.1099	1017.49957	141.43526	89.9596
2	6.978	VB	0.1172	113.56362	14.52431	10.0404
Totals :				1131.06319	155.95957	



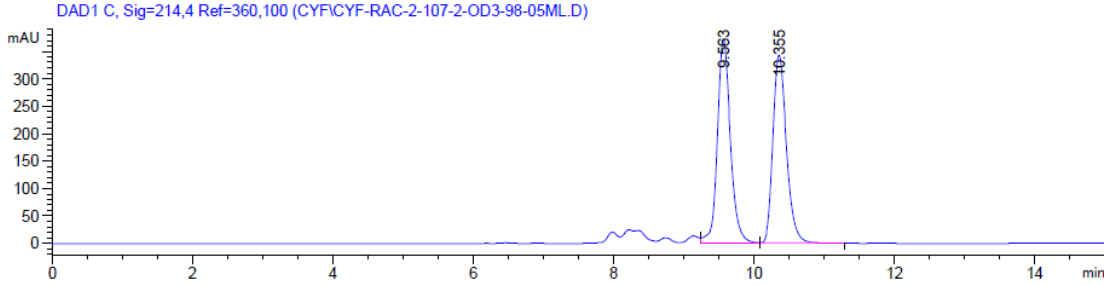
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.620	VV	0.1140	639.09143	86.73042	49.4600
2	6.650	BB	0.1317	653.04742	76.38306	50.5400
Totals :				1292.13885	163.11348	



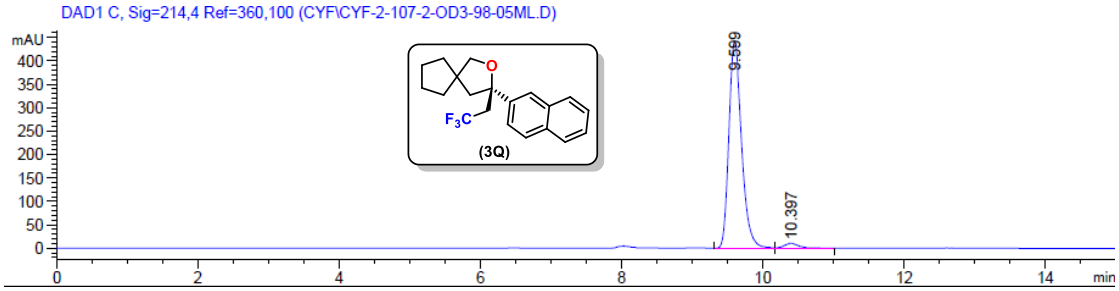
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.711	VB	0.1188	1056.65063	135.79475	96.5021
2	6.738	BB	0.1353	38.30005	4.32710	3.4979
Totals :				1094.95068	140.12185	



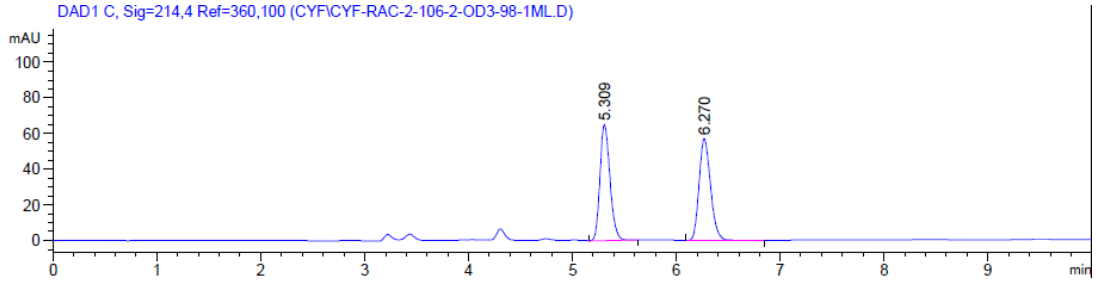
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.563	VB	0.1969	4745.05762	371.82022	50.9262
2	10.355	BB	0.2056	4572.46045	342.81903	49.0738
Totals :				9317.51807	714.63925	



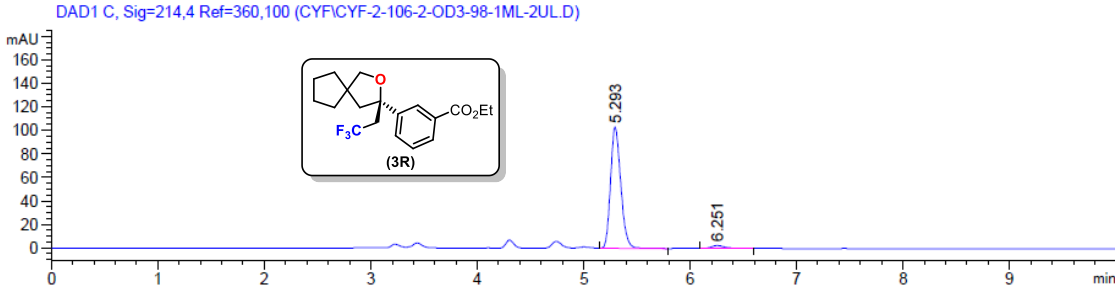
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.599	BV	0.1934	5505.76123	441.74200	97.4606
2	10.397	VB	0.2155	143.45569	10.11380	2.5394
Totals :				5649.21692	451.85580	



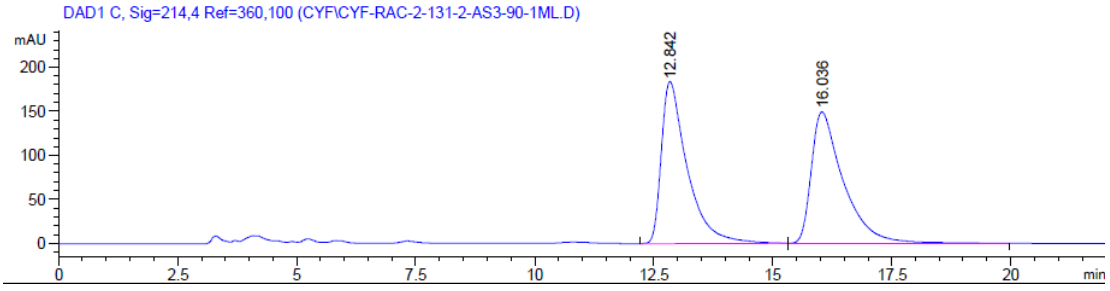
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.309	BB	0.1041	434.94681	64.93414	49.7781
2	6.270	BB	0.1176	438.82431	57.10738	50.2219
Totals :				873.77112	122.04152	



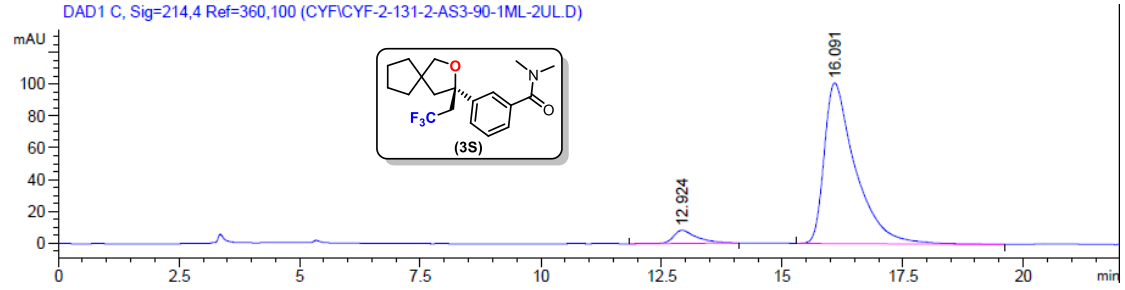
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.293	VB	0.1042	690.75647	103.10551	97.2791
2	6.251	BB	0.1195	19.32079	2.46238	2.7209
Totals :				710.07726	105.56789	



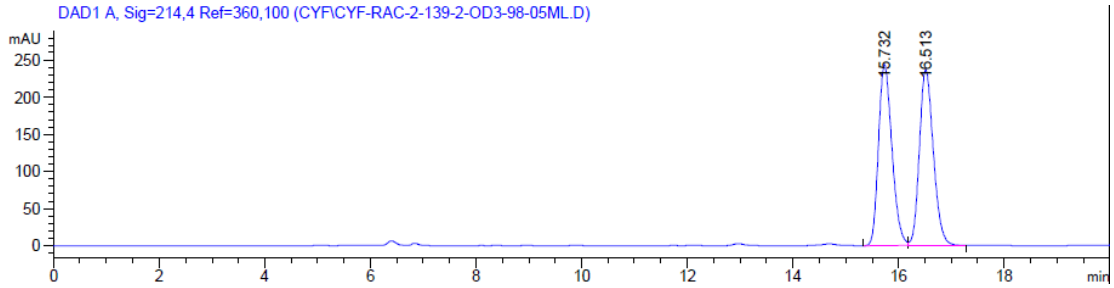
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.842	BB	0.5462	6940.84961	183.93640	49.8134
2	16.036	BB	0.6795	6992.85791	149.01588	50.1866
Totals :				1.39337e4	332.95229	



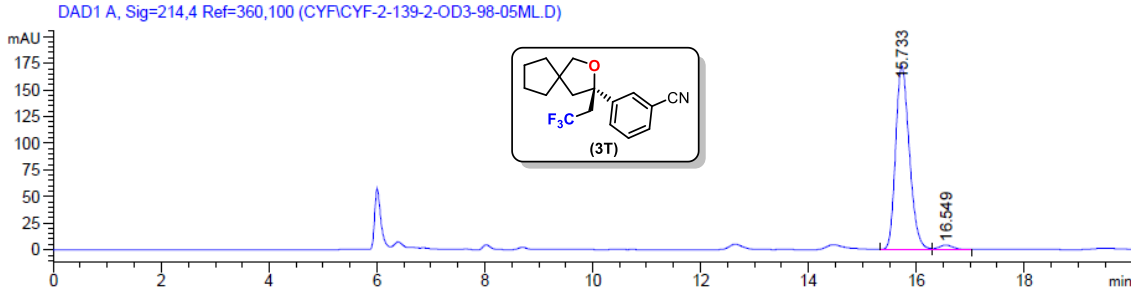
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.924	BB	0.4819	270.99622	8.15116	5.6454
2	16.091	BB	0.6506	4529.29150	100.73507	94.3546
Totals :				4800.28772	108.88623	



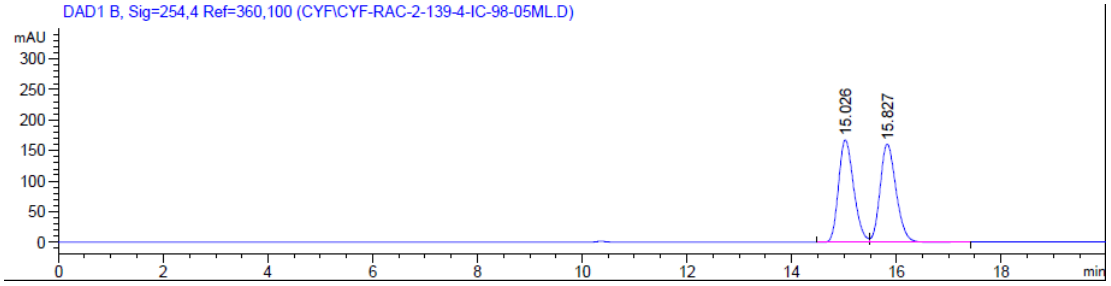
Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.732	BV	0.2743	4318.94043	244.27617	49.8683
2	16.513	VB	0.2816	4341.74609	237.11076	50.1317
Totals :				8660.68652	481.38693	



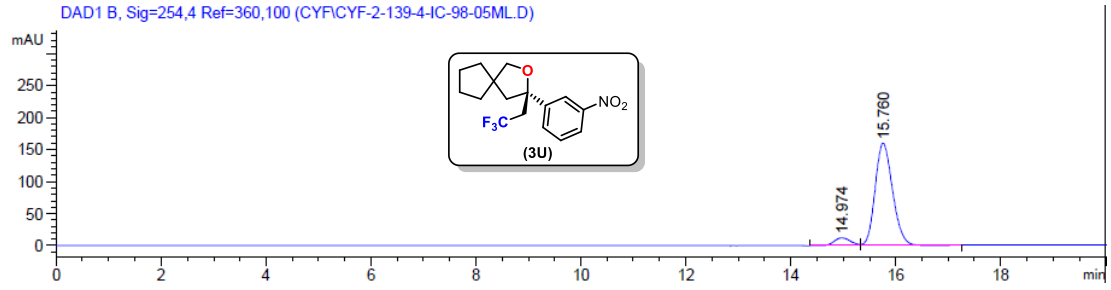
Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.733	BV	0.2718	3044.99951	172.65207	97.6176
2	16.549	VB	0.2824	74.31429	4.08244	2.3824
Totals :				3119.31380	176.73451	



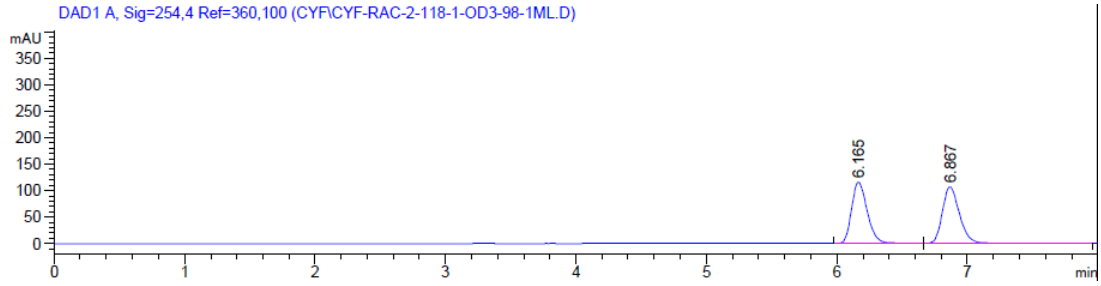
Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.026	BV	0.3103	3362.34692	167.45329	49.6653
2	15.827	VB	0.3276	3407.66382	160.59714	50.3347
Totals :				6770.01074	328.05043	



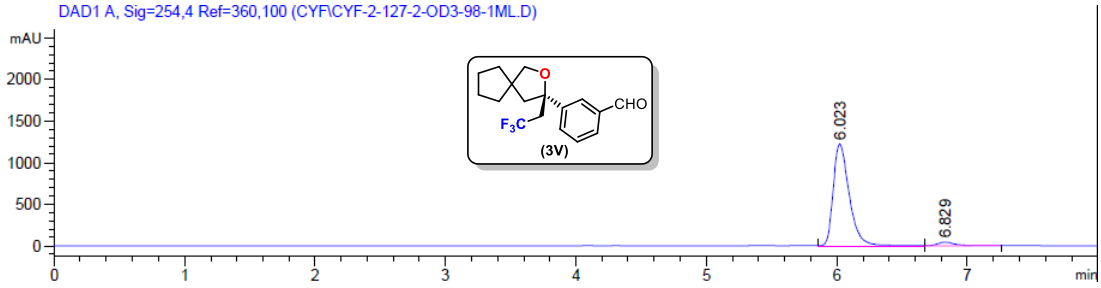
Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.974	BV	0.3407	269.47995	12.34516	6.7777
2	15.760	VB	0.3582	3706.52441	160.02942	93.2223
Totals :				3976.00436	172.37458	



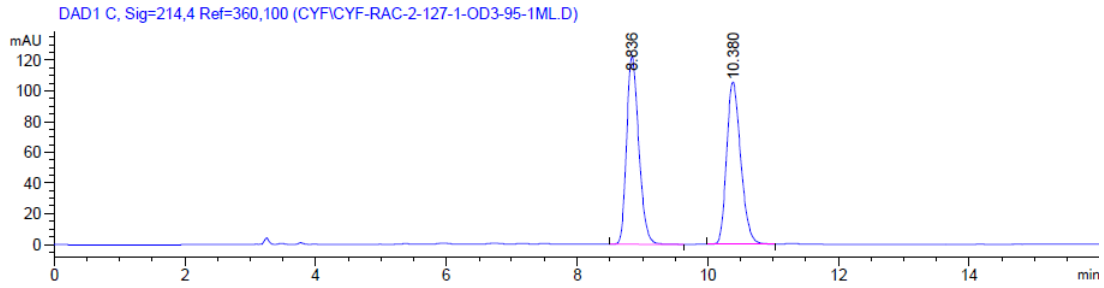
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.165	BV	0.1271	963.86896	115.84167	49.7773
2	6.867	VB	0.1401	972.49475	106.91107	50.2227
Totals :				1936.36371	222.75275	



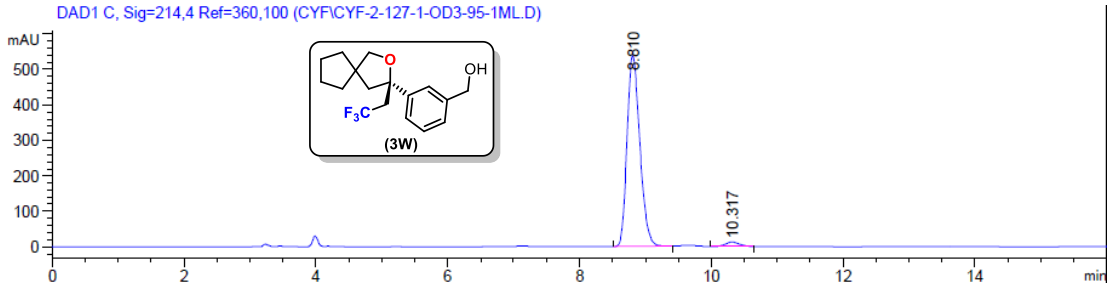
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.023	VV	0.1289	1.03825e4	1224.89136	96.3567
2	6.829	VB	0.1374	392.57413	42.63604	3.6433
Totals :				1.07751e4	1267.52739	



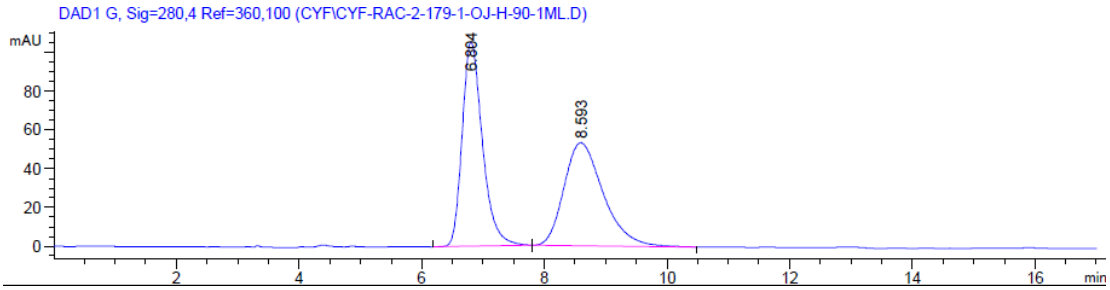
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.836	BB	0.2009	1581.07458	122.20734	50.0478
2	10.380	BB	0.2302	1578.05750	105.66670	49.9522
Totals :				3159.13208	227.87404	



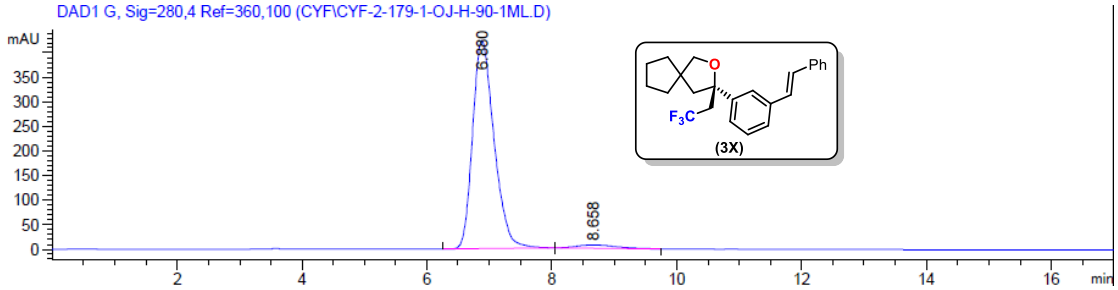
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.810	BB	0.2092	7233.68555	536.71942	97.6917
2	10.317	BB	0.2249	170.92238	11.94561	2.3083
Totals :				7404.60793	548.66503	



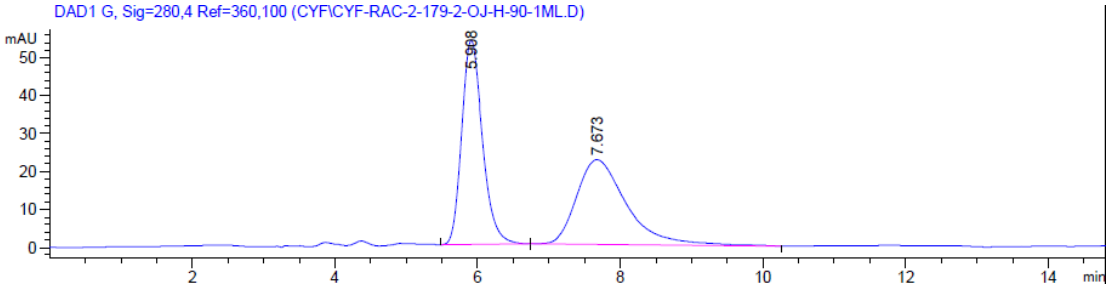
Signal 4: DAD1 G, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.804	BB	0.3518	2418.34668	105.36724	50.6238
2	8.593	BB	0.6820	2358.75049	53.12130	49.3762
Totals :				4777.09717	158.48854	



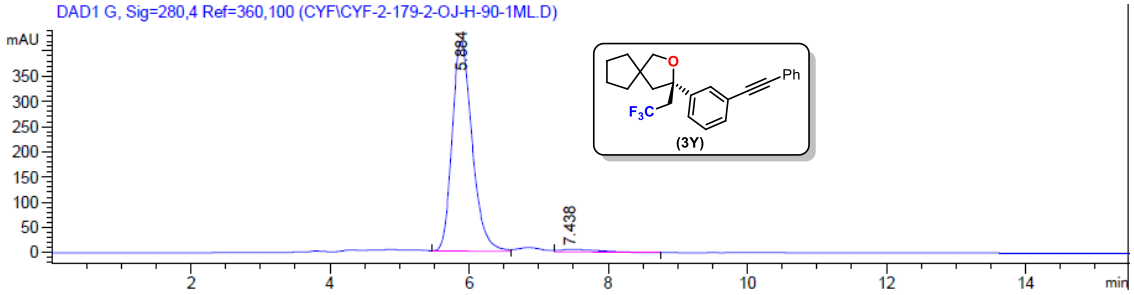
Signal 4: DAD1 G, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.880	BB	0.3670	1.01370e4	423.92657	97.2965
2	8.658	BB	0.6733	281.66873	6.52896	2.7035
Totals :				1.04187e4	430.45554	



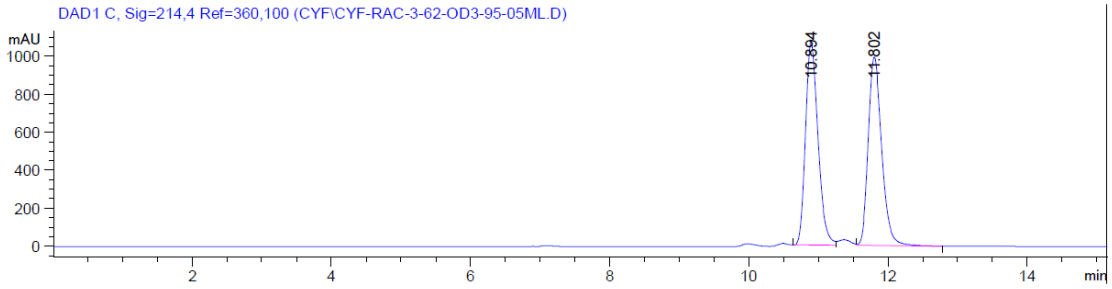
Signal 4: DAD1 G, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.908	BB	0.3110	1084.00012	53.80501	49.9656
2	7.673	BB	0.7324	1085.49487	22.35903	50.0344
Totals :				2169.49500	76.16404	



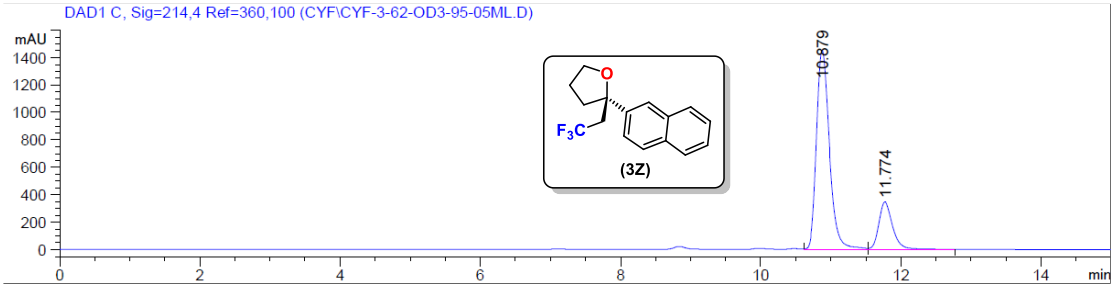
Signal 4: DAD1 G, Sig=280,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.884	BV	0.3064	8301.14258	416.66562	97.7484
2	7.438	VB	0.5628	191.21342	4.94911	2.2516
Totals :				8492.35600	421.61473	



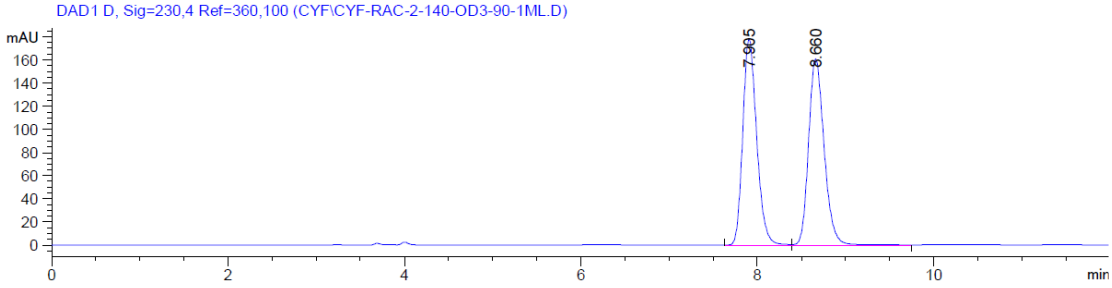
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.894	BV	0.1935	1.33907e4	1074.04407	50.0590
2	11.802	VB	0.2070	1.33591e4	992.73431	49.9410
Totals :				2.67498e4	2066.77838	



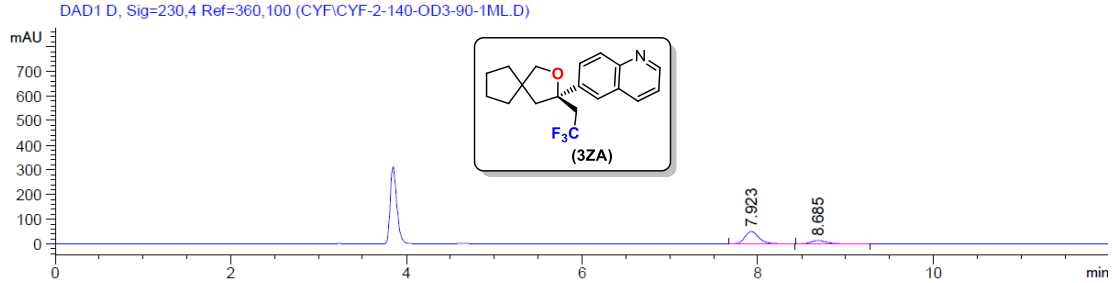
Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.879	VV	0.1974	1.86278e4	1454.47559	79.8275
2	11.774	VB	0.2060	4707.26025	347.52789	20.1725
Totals :				2.33350e4	1802.00348	



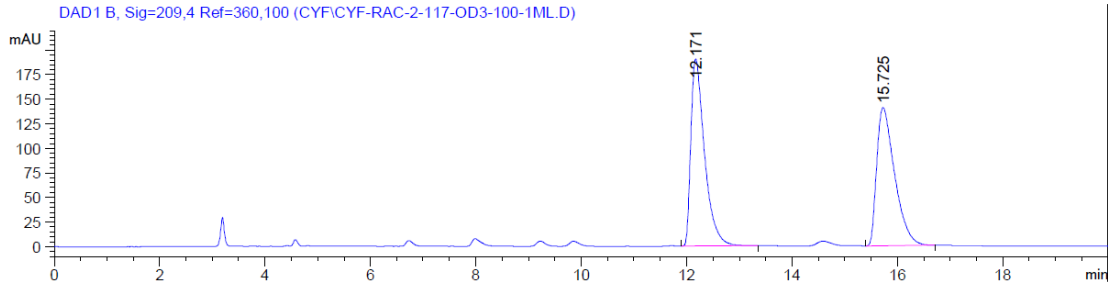
Signal 3: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.905	BV	0.1699	1951.02637	177.96931	49.8825
2	8.660	VB	0.1881	1960.21790	160.93059	50.1175
Totals :				3911.24426	338.89990	



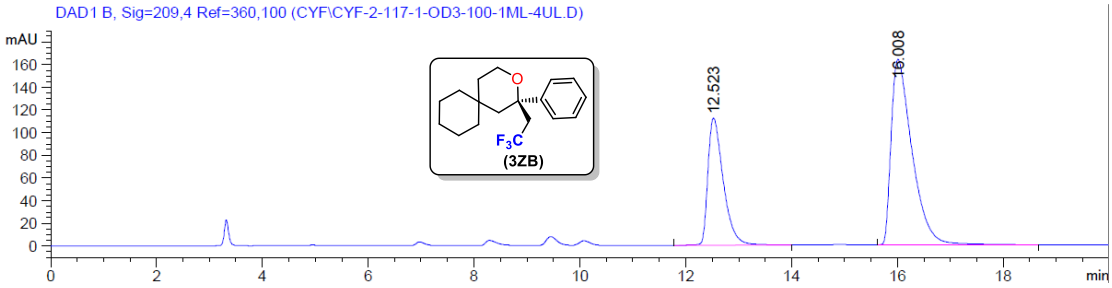
Signal 3: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.923	BB	0.1684	551.30170	50.08492	77.0091
2	8.685	BB	0.1880	164.58978	13.52300	22.9909
Totals :				715.89148	63.60793	



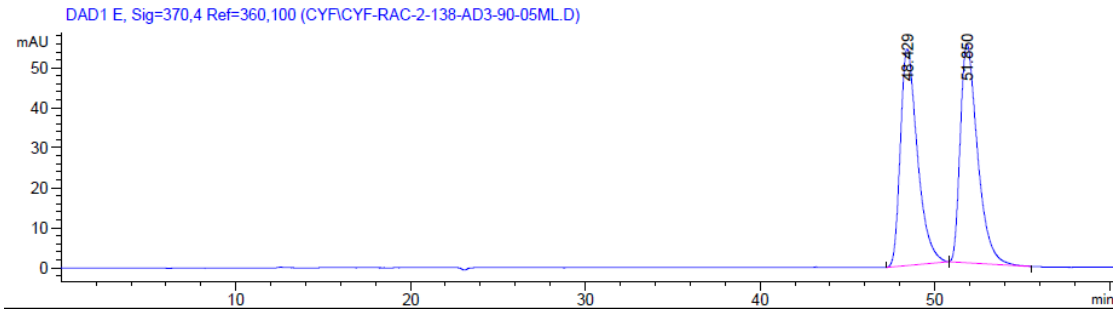
Signal 2: DAD1 B, Sig=209,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.171	BB	0.2723	3425.84961	190.08080	50.8001
2	15.725	BB	0.3591	3317.92944	140.70009	49.1999
Totals :				6743.77905	330.78088	



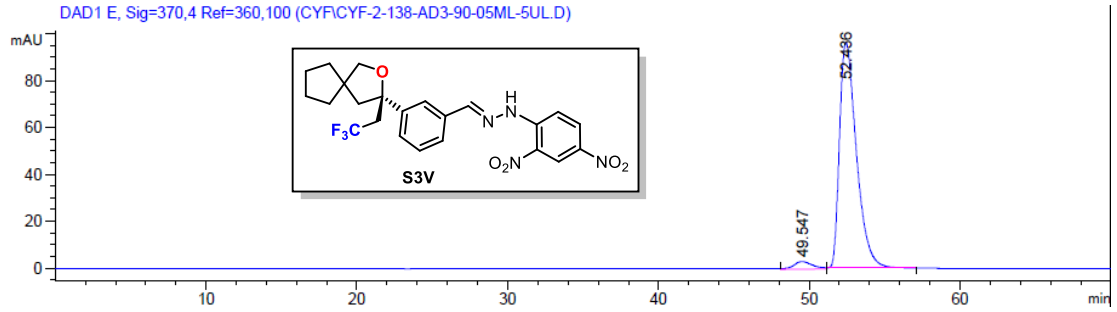
Signal 2: DAD1 B, Sig=209,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.523	BB	0.3078	2267.17651	112.18728	33.5772
2	16.008	BB	0.4119	4484.96387	163.55725	66.4228
Totals :				6752.14038	275.74453	



Signal 5: DAD1 E, Sig=370,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.429	BB	1.0252	3682.18457	54.15947	49.4735
2	51.850	BB	1.0464	3760.55151	54.67761	50.5265
Totals :				7442.73608	108.83708	



Signal 5: DAD1 E, Sig=370,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	49.547	MM R	1.4798	290.23489	3.26881	3.7403
2	52.436	BB	1.1617	7469.38623	96.21148	96.2597
Totals :				7759.62112	99.48029	