Electronic Supplementary Material (ESI) for ChemComm.


# Supporting Information

## Supported Gold Nanoparticles Catalyzed *cis*-Selective Semihydrogenation of Alkynes Using Ammonium Formate as Reductant

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# General

$^{1}H$ and $^{13}C$ NMR spectra were recorded at 400 MHz and 101 MHz using $CDCl_3$ as a solvent. The chemical shifts are reported in δ (ppm) values ($^{1}H$ and $^{13}C$ NMR relative to $CHCl_3$, δ 7.26 ppm for $^{1}H$ NMR and δ 77.0 ppm for $^{13}C$ NMR and $CFCl_3$ (δ 0 ppm for $^{19}F$ NMR), multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). Coupling constants (*J*), are reported in Hertz (Hz). All reagents and solvents were employed without further purification. The products were purified using a commercial flash chromatography system. TLC was developed on silica gel 60 F254 aluminum sheets. All reagents were purchased from Sigma-Aldrich or Alfa Aesar and used as received without any further purification. $Au/TiO_2$ and $Au/Al_2O_3$ (1% wt/wt loading; average size of AuNPs is around 2-3 nm) were purchased from Strem.

# Characterization of commercial available $Au/TiO_2$

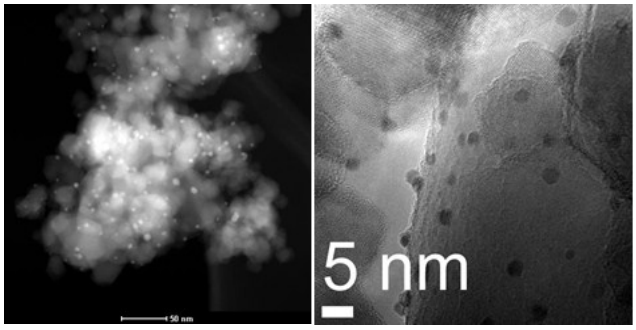
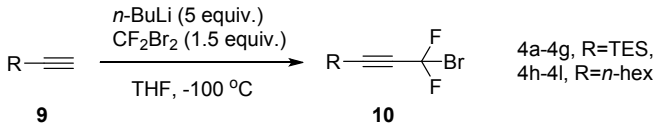



**Figure S-1**. STEM images of $Au/TiO_2$

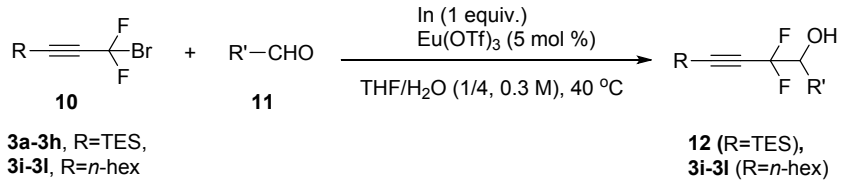
# General procedures

**General procedure for synthesis of *gem*-difluorohomopropargyl alcohols 3.[1]**




**9** **10**

To a solution of **9** (1.6 g, 14.6 mmol) in dry THF (30 mL), a 2.5 M hexane solution of n-butyllithium (6.1 mL, 15.3 mmol) was added dropwise at -78.8 ℃ under argon atmosphere. After the reaction mixture was stirred for 30 min at -78.8 ℃, cold (-78.8 ℃) dibromodifluoromethane (4.6 g, 21.9 mmol) was added to the reaction mixture at -100 ℃. After stirring for 16 hours at rt., the THF solution was washed with sat. aq. $NH_4Cl$ (10 mL). The aqueous layer was extracted with hexane (2×20 mL) and the combined organic layer was dried over $Na_2SO_4$. After evaporation of the solvent, the crude product was purified by distillation under reduced pressure (75 ℃/4.4 mmHg) to afford **10**.




To a flask was added indium powder (2.0 mmol, 1.0 equiv.), $Eu(OTf)_3$ (0.1 mmol, 5 mol %), difluoropropargyl bromide **10** (2.0 mmol) and aldehyde **11** (2.2 mmol, 1.1 equiv.) with rinsing by THF/$H_2O$ solution (1/4) (6.6 ml, 0.3M). The reaction was sonicated at 40 ℃ for 12 h. The reaction was quenched by 10% HCl (10 ml) and extracted by ethyl acetate (3×10ml). The combined organic layers were washed with brine and dried over $Na_2SO_4$. After evaporation of the solvent, the residue was purified by a silica gel column to afford the corresponding alcohols **3i-3l** or **12**.


A solution of AcOH (2.86 equiv.) and TBAF (2.2 equiv, 1.0 M in THF) in THF was stirred at room temperature for 30 min, then a solution of alcohol **12** (1 equiv.) in THF (0.1 M) was added slowly at room temperature, and the mixture was stirred at 0 ℃ for 2 hours. The reaction was quenched with water and extracted with EtOAc (3×40ml). The organic layer was washed with brine and dried over $Na_2SO_4$. The final products **3a-3h** were isolated by silica gel column.

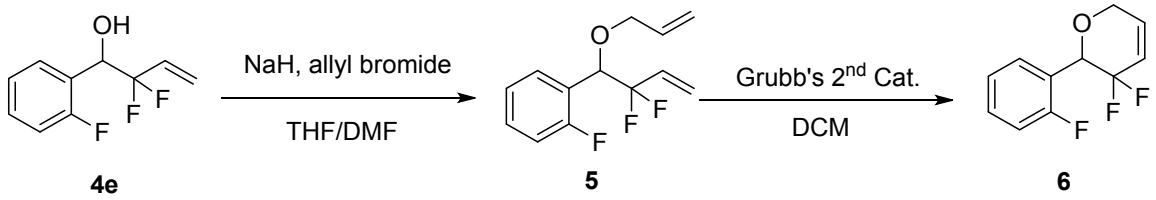
**General procedure for Au/$TiO_2$ semihydrogenation of alkynes using $HCOONH_4$ as reductant**

Au/$TiO_2$ (24.6 mg, 0.5 mol %) and $HCOONH_4$ (1 mmol) were added to a solution of alkyne (0.25 mmol) in DMF (0.25 mL). The mixture was allowed to stir in an oil bath at 80 °C for designated time. After cooling down to room temperature, the solid Au/$TiO_2$ was filtered off, the filtrate was diluted with DCM and washed with water and brine solution. The organic layer was dried over $Na_2SO_4$ and concentrated to dryness. The residue was purified by flash chromatography on silica gel (n-hexane/ethyl acetate).

**Procedure for Au/$TiO_2$ recycling**

Au/$TiO_2$ (24.6 mg, 0.5 mol %) and $HCOONH_4$ (1 mmol) were added to a solution of phenylacetylene (0.25 mmol) in DMF (1 mL). The mixture was stirred in an oil bath at 80 °C for 3 hours. After cooling down to room temperature, the solid Au/$TiO_2$ was filtered out and washed with DMF and transferred into a fresh solution of phenylacetylene (0.25 mmol) in DMF (1 mL), and $HCOONH_4$ (1 mmol) was added. The mixture was stirred for another 3 hours at 80 °C. And this process was repeated until 5 runs were completed. And the yields of each run were determined by $^1H$ NMR.

**Procedure for synthesis of 6**

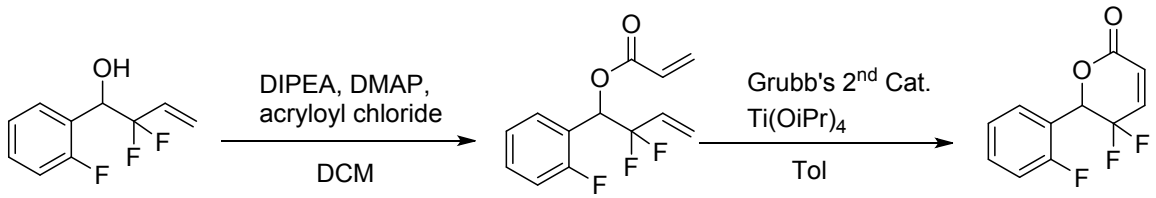



**4e** **5** **6**

**4e** (0.2 mmol, 40.4 mg) was dissolved in dry THF/DMF (1.4 mL / 0.4 mL), then NaH (0.6 mmol, 24 mg) was added into the solution at 0 ℃. The mixture was allowed to stir for 15 minutes at room temperature. Then allyl bromide (0.6 mmol, 72.5 mg) was added. The resulting mixture was stirred at room temperature for overnight. Then the reaction mixture was quenched by 2 mL $NH_4Cl$ (aq), extracted with $Et_2O$ (2 × 2 mL). The combined organic layer was washed with brine and dried over $Na_2SO_4$. After concentration the residue was purified by flash chromatography on silica gel to obtain **5** as colorless oil.

The solution of purified **5** (0.14 mmol, 34.4 mg) and Grubbs' catalyst (5 mol %) in DCM were stirred for 24 hours at room temperature. Then after concentration the residue was purified by flash chromatography on silica gel to afford **6** as colorless oil.

**Procedure for synthesis of 8**




**4e** **7**

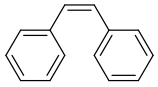
Acryloyl chloride (0.4 mmol, 28 µl) was added dropwise to the solution of **4e** (0.2 mmol, 40.4 mg), DMAP (5 mol %) and N,N-diisopropylethylamine (0.5 mmol, 86.9 µl) in DCM (0.5 mL) at 0 ℃. The mixture was stirred at room temperature for overnight. The reaction mixture was quenched with water (1 mL) and extracted with DCM (2 × 1 mL). The combined organic layer was dried over $Na_2SO_4$. After concentration the residue was purified by flash chromatography on silica gel to obtain **7** as colorless oil.

The solution of purified **7** (0.17 mmol, 44 mg) and Ti(O*i*Pr)$_4$ (0.05 mmol, 15 µl) in toluene (6 mL) were stirred under reflux for 3 h. Then solution of Grubbs' catalyst (7 mol %, 10.1 mg) in toluene (1.5 mL) was added dropwise into the reaction mixture over 30 min. The resulting mixture was stirred for additional 1 h under reflux and then cooled down to room temperature. After concentration the residue was purified by flash chromatography on silica gel to obtain **8** as colorless oil.

# Characterization of non-fluorinated products 2

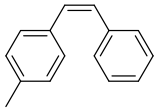
All the compounds are known and their $^1$H NMR spectroscopic data agree well with the spectra reported in the literature noted for each of them.

(*Z*)-1,2-diphenylethene (**2a**)[2]



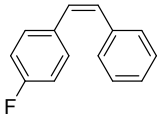
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.12 (m, 10H), 6.61 (s, 2H).

(*Z*)-1-methyl-4-styrylbenzene (**2b**)[3]



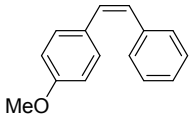
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.16 (m, 7H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 7.9 Hz, 2H), 6.55 (s, 2H), 2.31 (s, 2H).

(*Z*)-1-fluoro-4-styrylbenzene (**2c**)[4]



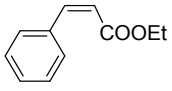

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.11 (m, 7H), 6.91 (t, *J* = 8.7 Hz, 2H), 6.57 (q, *J* = 12.2 Hz, 2H).

(*Z*)-1-methoxy-4-styrylbenzene (**2d**)[5]



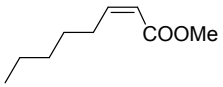

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.16 (m, 7H), 6.76 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 1.7 Hz, 2H), 3.78 (s, 3H).

(*Z*)-ethyl 3-phenylacrylate (**2e**)[6]



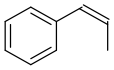

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 7.4, 1.7 Hz, 2H), 7.43 – 7.28 (m, 3H), 6.94 (d, *J* = 12.6 Hz, 1H), 5.94 (d, *J* = 12.6 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H).

(*Z*)-methyl oct-2-enoate (**2f**)[6]



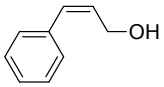

$^1$H NMR (400 MHz, $CDCl_3$) δ 6.22 (dt, *J* = 11.5, 7.5 Hz, 1H), 5.75 (dt, *J* = 12, 1.6 Hz, 1H), 3.70 (s, 3H), 2.64 (qd, *J* = 7.6, 1.6 Hz, 2H), 1.47 – 1.40 (m, 2H), 1.34 – 1.29 (m, 4H), 0.88 (t, *J* = 8, 3H).

(*Z*)-prop-1-en-1-ylbenzene (**2g**)[2]



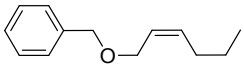
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.35-7.20 (m, 5H), 6.48 – 6.40 (dd, *J* = 12, 1.6 Hz, 1H), 5.86 – 5.72 (dq, *J* = 11.6, 7.2 Hz, 1H), 1.90 (dd, *J* = 7.2, 2 Hz, 3H).

(*Z*)-3-phenylprop-2-en-1-ol (**2h**)[7]



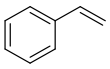

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 2H), 7.30 – 7.11 (m, 3H), 6.57 (d, *J* = 11.7 Hz, 1H), 5.87 (dt, *J* = 12, 6.4 Hz, 1H), 4.44 (d, *J* = 6.4 Hz, 2H), 1.66 (s, 1H).

(Z)-[(hex-2-en-1-yloxy)methyl]benzene (**2i**)[8]



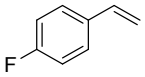

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.26 (m, 5H), 5.60 (m, 2H), 4.51 (s, 2H), 4.08 (d, *J* = 4.6 Hz, 2H), 2.02 (m, 2H), 1.38 (sext, *J* = 8 Hz, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

styrene (**2j**)



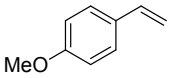
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.17 (m, 5H), 6.65 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.69 (d, *J* = 17.6 Hz, 1H), 5.18 (d, *J* = 10.9 Hz, 1H).

1-fluoro-4-vinylbenzene (**2k**)[9]



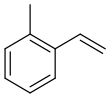

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 2H), 6.92 – 6.87 (m, 2H), 6.55 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.53 (d, *J* = 17.6 Hz, 1H), 5.11 (d, *J* = 10.8 Hz, 1H).

1-methoxy-4-vinylbenzene (**2l**)[10]



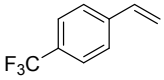

$^1$H NMR (400 MHz, cdcl$_3$) δ 7.35 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.66 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.61 (d, *J* = 17.6 Hz, 1H), 5.12 (d, *J* = 10.9 Hz, 1H), 3.81 (s, 3H).

1-methyl-2-vinylbenzene (**2m**)[11]



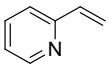
$^1$H NMR (400 MHz, cdcl$_3$) δ 7.47 – 7.49 (m, 1H), 7.17 (m, 3H), 6.95 (dd, *J* = 17.4, 11.0 Hz, 1H), 5.64 (d, *J* = 17.4 Hz, 1H), 5.29 (d, *J* = 11.0 Hz, 1H), 2.35 (s, 3H).

1-(trifluoromethyl)-4-vinylbenzene (**2n**)[12]



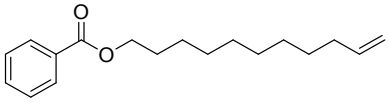

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.58 (d, *J* = 8 Hz, 2H), 7.49 (d, *J* = 8 Hz, 2H), 6.75 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.84 (d, *J* = 17.6 Hz, 1H), 5.38 (d, *J* = 10.9 Hz, 1H).

2-vinylpyridine (**2o**)[13]



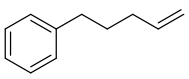

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.57 (d, *J* = 4.3 Hz, 1H), 7.64 (td, *J* = 7.7, 1.6 Hz, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.17 – 7.14 (m, 1H), 6.82 (dd, *J* = 17.5, 10.8 Hz, 1H), 6.20 (d, *J* = 17.5 Hz, 1H), 5.48 (d, *J* = 10.8 Hz, 1H).

undec-10-en-1-yl benzoate (**2p**)[14]



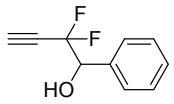

$^1$H NMR (400 MHz, CDCl$_3$) δ 8.05 – 8.03 (m, 2H), 7.56 – 7.52 (m, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 5.86-5.75 (m, 1H), 5.01 – 4.92 (m, 2H), 4.31 (t, *J* = 6.7 Hz, 2H), 2.03 (q, *J* = 8 Hz, 2H), 1.79 – 1.72 (m, 2H), 1.47 – 1.29 (m, 12H).

pent-4-en-1-ylbenzene (**2q**)[15]



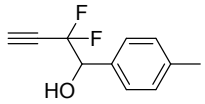
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.28 (m, 2H), 7.18 (m, 3H), 5.83 (td, *J* = 16.9, 6.7 Hz, 1H), 5.00 (dd, *J* = 20.0, 13.7 Hz, 2H), 2.62 (t, *J* = 7.7 Hz, 2H), 2.09 (q, *J* = 7.2 Hz, 2H), 1.79 – 1.65 (m, 2H).

# Characterization of *gem*-difluoropropargyl alcohols 3



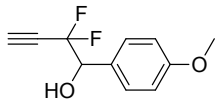
**3a**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.51 (m, 2H), 7.42 – 7.40 (m, 3H), 4.98 (dt, *J* = 9.0, 4.5 Hz, 1H), 2.81 (t, *J* = 15.5 Hz, 1H), 2.63 (t, *J* = 4.0 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.35 (s, 1F), -95.21 (s, 1F).



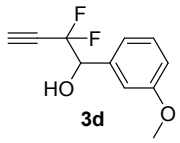
**3b**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.40 (t, *J* = 8.5 Hz, 2H), 7.22 (t, *J* = 8.5 Hz, 2H), 4.93 (t, *J* = 5.5 Hz, 1H), 2.80 (t, *J* = 5.5 Hz, 1H), 2.78 (bs, 1H), 2.39 (s, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.34 (s, 1F), -95.22 (s, 1F).



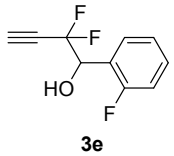
**3c**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.43 (t, *J* = 8.5 Hz, 2H), 6.93 (t, *J* = 8.5 Hz, 2H), 4.92 (t, *J* = 9.0 Hz, 1H), 3.83 (s, 3H), 2.80 (t, *J* = 5.0 Hz, 1H), 2.57 (bs, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.43 (s, 1F), -95.39 (s, 1F).



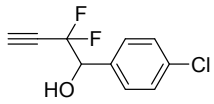
**3d**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.33-7.27 (m, 1H), 7.08 (m, 2H), 6.93 (s, 1H), 4.95 (bs, 1H), 3.84 (s, 3H), 2.82 (t, *J* = 5.0 Hz, 1H), 2.67 (bs, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.11 (s, 1F), -94.95 (s, 1F).



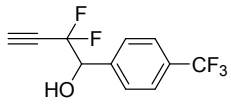
**3e**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.65 (t, *J* = 7.0 Hz, 1H), 7.40-7.36 (m, 1H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.09 (dt, *J* = 9.3, 1.0 Hz, 1H), 5.37 (t, *J* = 8.5 Hz, 1H), 2.88 (bs, 1H), 2.83 (t, *J* = 5.0 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -117.4 (s, 1F), -96.76 (d, *J* = 277.7 Hz, 1F), -95.80 (d, *J* = 277.7 Hz, 1F).



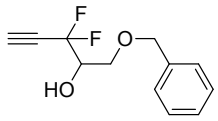

**3f**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.46 (t, *J* = 8.5 Hz, 2H), 7.38 (t, *J* = 8.5 Hz, 2H), 4.94 (dt, *J* = 8.5, 4.0 Hz, 1H), 2.82 (t, *J* = 5.0 Hz, 1H), 2.64 (t, *J* = 4.0 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.42 (s, 1F), -95.39 (s, 1F).



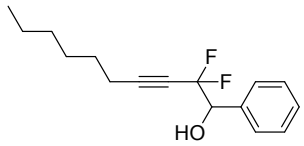

**3g**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.67 (t, *J* = 8.5 Hz, 2H), 7.64 (t, *J* = 8.5 Hz, 2H), 5.03 (dt, *J* = 9.0, 3.5 Hz, 1H), 2.98 (bs, 1H), 2.83 (t, *J* = 5.0 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.59 (d, *J* = 277.7 Hz, 1F), -94.88 (d, *J* = 277.7 Hz, 1F), -63.21 (s, 3F).



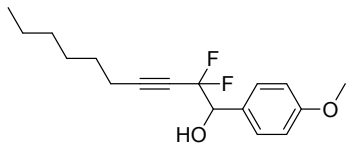

**3h**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.27 (m, 5H), 4.69 – 4.50 (m, 2H), 4.10 (m, 1H), 3.73 (ddd, *J* = 17.1, 10.2, 5.1 Hz, 2H), 2.83 (m, 2H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -95.08 (dt, $J_{F\text{-}F}$ = 278.2 Hz, $J_{F\text{-}H}$ = 11.3 Hz, 1F), -96.27 (dt, $J_{F\text{-}F}$ = 248.2 Hz, $J_{F\text{-}H}$ = 11.3 Hz, 1F).



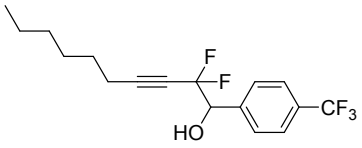

**3i**

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.49 (m, 2H), 7.41 – 7.37 (m, 3H), 4.93 (dd, *J* = 9.0, 8.5 Hz, 1H), 2.61 (d, *J* = 3.8 Hz, 1H), 2.27 – 2.22 (m, 2H), 1.52 – 1.47 (m, 2H), 1.38 – 1.22 (m, 6H), 0.90 (t, *J* = 7.25 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -93.18 (d, *J* = 274.4 Hz, 1F), -92.32 (d, $J_{F\text{-}F}$ = 270.6 Hz, 1F).



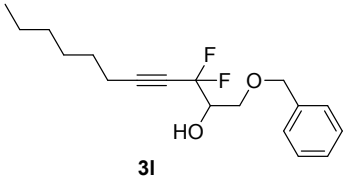

**3j**

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.5 Hz, 2H), 6.88 (d, *J* = 8.5 Hz, 2H), 4.85 (t, *J* = 8.8 Hz, 1H), 3.80 (s, 3H), 2.55 (bs, 1H), 2.23 – 2.21 (m, 2H), 1.49 – 1.45 (m, 2H), 1.31 – 1.27 (m, 6H), 0.87 (t, *J* = 7.0Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -93.32 (d, *J* = 273.5 Hz, 1F), -92.15 (d, *J* = 297.4 Hz, 1F).




**3k**

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.63 (m, 4H), 5.00 (dt, *J* = 8.7, 3.5 Hz, 1H), 2.77 (bs, 1H), 2.25 (m, 2H), 1.49 (m, 2H), 1.29 (m, 6H), 0.90 (t, *J* = 7.5 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -93.05 (d, *J*= 271.1 Hz, 1F), -92.15 (d, $J_{F-F}$ = 271.1 Hz, 1F), -63.14 (s, 3F).

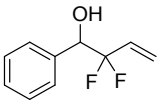



**3l**

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 5H), 4.61 (dd, *J* = 16.0, 11.5 Hz, 2H), 4.10 – 4.06 (m, 1H), 3.78 (dd, *J* = 10.5, 3.5 Hz, 1H), 3.67 (dd, *J* = 10.5, 7.0 Hz, 1H), 2.71 (m, 1H), 2.28 (m, 2H), 1.53 (dd, *J* = 14.8, 7.5 Hz, 2H), 1.41 – 1.27 (m, 6H), 0.90 (t, *J* = 7.5 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -93.21 (s, 2F).

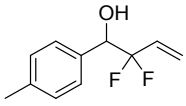
# Characterization of *gem*-difluorinated products

2,2-difluoro-1-phenylbut-3-en-1-ol (**4a**)




$^1$H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.35 (m, 5H), 5.92 – 5.78 (m, 1H), 5.59 (dd, *J* = 17.4, 1.2 Hz, 1H), 5.46 (d, *J* = 11.0 Hz, 1H), 4.90 (td, *J* = 9.6, 2.4 Hz, 1H), 2.54 (d, *J* = 2.5 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -107.95 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.45 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 135.92, 129.34 (t, *J* = 25 Hz), 128.71, 128.18, 127.59, 121.6 (t, *J* = 9 Hz), 119.57, 75.89 (t, *J* = 30 Hz).

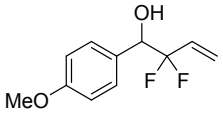
2,2-difluoro-1-(p-tolyl)but-3-en-1-ol (**4b**)




$^1$H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 7.8 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 5.92 – 75.79 (d, *J* = 11.3 Hz, 1H), 5.60 (d, *J* = 17.4 Hz, 1H), 5.46 (d, *J* = 11.1 Hz, 1H), 4.86 (td, *J* = 9.2, 4 Hz, 1H), 2.46 (d,

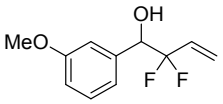
*J* = 3.8 Hz, 1H), 2.36 (s, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -108.00 (dt, $J_{F-F}$ = 244.4 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.45 (dt, $J_{F-F}$ = 244.4 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 138.54, 133.02, 129.48 (t, *J* = 26 Hz), 128.90, 127.49, 121.47 (t, *J* = 9 Hz), 119.60, 75.78 (t, *J* = 30 Hz), 21.17.

2,2-difluoro-1-(4-methoxyphenyl)but-3-en-1-ol (**4c**)



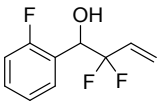

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 5.85 (ddd, *J* = 23.3, 17.4, 11.3 Hz, 1H), 5.59 (d, *J* = 17.4 Hz, 1H), 5.46 (d, *J* = 11.1 Hz, 1H), 4.84 (t, *J* = 9.7 Hz, 1H), 3.81 (s, 3H), 2.51 (br, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -107.63 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.67 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 159.85, 129.51 (t, *J* = 25 Hz), 128.85, 128.12, 121.48 (t, *J* = 9 Hz), 119.63, 113.61, 75.53 (t, *J* = 32 Hz), 55.24.

2,2-difluoro-1-(3-methoxyphenyl)but-3-en-1-ol (**4d**)



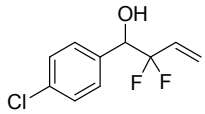

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.27 (m, 1H), 6.99 (d, *J* = 7.6 Hz, 2H), 6.90 – 6.87 (m, 1H), 5.85 (ddd, *J* = 23.4, 17.4, 11.3 Hz, 1H), 5.61 (d, *J* = 17.4 Hz, 1H), 5.47 (d, *J* = 11.1 Hz, 1H), 4.88 (t, *J* = 9.4 Hz, 1H), 3.81 (s, 3H), 2.51 (br, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -107.87 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.18 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 159.43, 137.51, 129.34 (t, *J* = 25 Hz), 129.20, 121.57 (t, *J* = 10 Hz), 119.96, 119.51, 114.25, 113.14, 75.80 (t, *J* = 30 Hz), 55.25.

2,2-difluoro-1-(2-fluorophenyl)but-3-en-1-ol (**4e**)



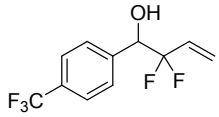

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.54 (t, *J* = 7.4 Hz, 1H), 7.33 (tdd, *J* = 7.3, 5.3, 1.8 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 7.07 – 7.03 (m, 1H), 5.93 (dq, *J* = 17.4, 11.6 Hz, 1H), 5.62 (dt, *J* = 17.4, 2.4 Hz, 1H), 5.49 (d, *J* = 11.1 Hz, 1H), 5.27 (td, *J* = 10.1, 5.0 Hz, 1H), 2.58 (d, *J* = 5.0 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -110.33 (m, 2F). -117.15 (m, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 161.54, 159.08, 130.34 (d, *J* = 8 Hz), 129.59, 129.34, 129.08, 124.13 (d, *J* = 4 Hz), 121.80 (t, *J* = 9 Hz), 115.28 (d, *J* = 22 Hz), 66.34 (t, *J* = 30 Hz).

1-(4-chlorophenyl)-2,2-difluorobut-3-en-1-ol (**4f**)



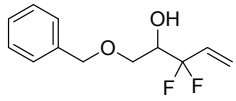

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 4H), 5.83 (ddd, *J* = 23.6, 17.4, 11.1 Hz, 1H), 5.58 (dt, *J* = 17.4, 2.4 Hz, 1H), 5.47 (d, *J* = 11.1 Hz, 1H), 4.89 (td, *J* = 9.5, 3.5 Hz, 1H), 2.54 (d, *J* = 3.5 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -107.63 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.68 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 134.62, 134.36, 129.96 (t, *J* = 26 Hz), 128.93, 128.38, 121.98 (t, *J* = 9 Hz), 119.40, 75.22 (t, *J* = 31 Hz).

2,2-difluoro-1-(4-(trifluoromethyl)phenyl)but-3-en-1-ol (**4g**)



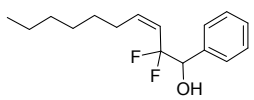

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.3 Hz, 2H), 7.55 (d, *J* = 8.2 Hz, 2H), 5.83 (ddd, *J* = 23.7, 17.4, 11.0 Hz, 1H), 5.63 – 5.54 (m, 1H), 5.49 (d, *J* = 11.1 Hz, 1H), 4.98 (td, *J* = 9.3, 3.3 Hz, 1H), 2.60 (d, *J* = 3.5 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -62.74 (s, 3F), -107.18 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.65 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 139.71, 130.36 (d, *J* = 33 Hz), 128.76 (t, *J* = 26 Hz), 127.95, 125.07 (d, *J* = 4 Hz), 122.21 (t, *J* = 9 Hz), 119.32, 116.88, 75.27 (t, *J* = 31 Hz).

1-(benzyloxy)-3,3-difluoropent-4-en-2-ol (**4h**)



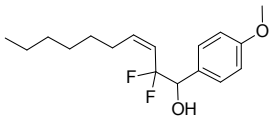

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.26 (m, 5H), 6.00 (m, 1H), 5.72 (d, *J* = 17.4 Hz, 1H), 5.52 (d, *J* = 11.1 Hz, 1H), 4.58 (s, 2H), 3.98 – 4.08 (m, 1H), 3.76 – 3.50 (m, 2H), 2.71 (br, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -107.93 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -109.43 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 137.38, 129.94 (t, *J* = 25 Hz), 128.51, 127.96, 127.76, 126.97, 120.98 (t, *J* = 10 Hz), 73.64, 72.31 (t, *J* = 30 Hz), 68.58.

(Z)-2,2-difluoro-1-phenyldec-3-en-1-ol (**4i**)[16]



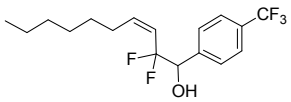

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.42 (m, 2H), 7.39 – 7.33 (m, 3H), 5.80 – 5.72 (m, 1H), 5.43 – 5.32 (m, 1H), 4.89 (td, *J* = 10.0, 3.7 Hz, 1H), 2.55 (d, *J* = 3.8 Hz, 1H), 2.11 – 1.96 (m, 2H), 1.30 – 1.21 (m, 8H), 0.87 (t, *J* = 7.0 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -101.20 (ddd, $J_{F-F}$ = 251.9 Hz, $J_{F-H}$ = 13.2 Hz, 11.3 Hz, 1F), -101.99 (dt, $J_{F-F}$ = 251.9 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.85 (t, *J* = 6 Hz), 136.04, 128.62, 128.10, 127.72, 120.97 (t, *J* = 244 Hz), 120.40 (t, *J* = 25 Hz), 76.28 (t, *J* = 30 Hz), 31.58, 29.19, 28.84, 28.33, 22.54, 14.05.

(Z)-2,2-difluoro-1-(4-methoxyphenyl)dec-3-en-1-ol (**4j**)



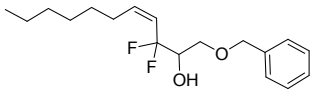
$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 8.6 Hz, 2H), 5.79 – 5.72 (m, 1H), 5.37 (dd, *J* = 27.5, 15.2 Hz, 1H), 4.86 – 4.81 (m, 1H), 3.80 (s, 3H), 2.52 (d, *J* = 3.4 Hz, 1H), 2.11 – 2.00 (m, 2H), 1.34 – 1.22 (m, 8H), 0.87 (t, *J* = 6.9 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -101.31 (ddd, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 13.2 Hz, 11.3 Hz, 1F), -102.30 (dt, $J_{F-F}$ = 248.2 Hz, $J_{F-H}$ = 11.3 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 159.82, 140.70 (t, *J* = 60 Hz), 128.94, 128.20, 121.02 (t, *J* = 244 Hz), 120.56 (t, *J* = 25 Hz), 113.53, 75.90 (t, *J* = 30 Hz), 55.21, 31.59, 29.23, 28.87, 28.36, 22.54, 14.04. HRMS (ESI) calcd. for [$C_{17}H_{24}F_2O_2$] ([$Na^+$]) 321.1642; found 321.2500.

(Z)-2,2-difluoro-1-(4-(trifluoromethyl)phenyl)dec-3-en-1-ol (**4k**)



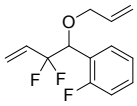
$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8 Hz, 2H), 7.57 (d, *J* = 8 Hz, 2H), 5.79 (dt, *J* = 12.3, 7.8 Hz, 1H), 5.35 (dd, *J* = 27.9, 15.2 Hz, 1H), 4.99 – 4.94 (m, 1H), 2.67 (s, 1H), 2.07 – 1.96 (m, 2H), 1.29 – 1.20 (m, 8H), 0.87 (t, *J* = 6.9 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -100.85 (dt, $J_{F-F}$ = 251.9 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -101.82 (ddd, $J_{F-F}$ = 251.9 Hz, $J_{F-H}$ = 15.04, 7.52 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 141.42 (t, *J* = 5 Hz), 139.84, 130.78 (d, *J* = 32 Hz), 128.10, 124.96 (d, *J* = 4 Hz), 122.61, 120.71 (t, *J* = 244 Hz), 119.83 (t, *J* = 25 Hz), 75.69 (t, *J* = 30 Hz), 31.52, 29.15, 28.82, 28.37, 22.50, 13.99. HRMS (ESI) calcd for [$C_{17}H_{21}F_5O$] ([$Na^+$]) 359.1410; found 359.2333.

(Z)-1-(benzyloxy)-3,3-difluoroundec-4-en-2-ol (**4l**)



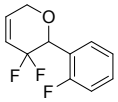
$^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.26 (m, 5H), 5.93 – 5.75 (m, 1H), 5.55 – 5.45 (m, 1H), 4.58 (dd, *J* = 16, 11.6 Hz, 2H), 4.08 – 3.99 (m, 1H), 3.66 (ddd, *J* = 17.3, 10.0, 5.3 Hz, 2H), 2.68 (d, *J* = 4.9 Hz, 1H), 2.30 – 2.24 (m, 2H), 1.42 – 1.24 (m, 8H), 0.88 (t, *J* = 6.8 Hz, 3H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -101.07 (dt, $J_{F-F}$ = 255.68 Hz, $J_{F-H}$ = 11.3 Hz, 1F), -103.91 (dt, $J_{F-F}$ = 255.68 Hz, $J_{F-H}$ = 11.03 Hz, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 140.65 (t, *J* = 5 Hz), 137.45, 128.49, 127.92, 127.76, 121.04 (t, *J* = 25 Hz), 120.40 (t, *J* = 242 Hz), 73.64, 72.95 (t, *J* = 29 Hz), 68.70 (t, *J* = 4 Hz), 31.16, 29.35, 28.88, 28.58, 22.56, 14.05. HRMS (ESI) calcd for [$C_{18}H_{26}F_2O_2$] ([$Na^+$]) 335.1799; found 335.2500.

1-(1-(allyloxy)-2,2-difluorobut-3-en-1-yl)-2-fluorobenzene (**5**)



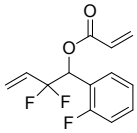
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.51 (t, *J* = 7.2 Hz, 1H), 7.36 – 7.30 (m, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.08 – 7.02 (m, 1H), 6.01 (ddd, *J* = 23.3, 17.4, 11.6 Hz, 1H), 5.86 (ddt, *J* = 22.4, 11.3, 5.7 Hz, 1H), 5.59 (dt, *J* = 17.4, 2.4 Hz, 1H), 5.47 (d, *J* = 11.1 Hz, 1H), 5.28 – 5.18 (m, 2H), 5.06 – 4.89 (t, *J* = 12 Hz, 1H), 3.98 (ddd, *J* = 18.9, 12.8, 5.6 Hz, 2H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -108.27 (m, 2F), -117.67 (m, 1F).

3,3-difluoro-2-(2-fluorophenyl)-3,6-dihydro-2H-pyran (**6**)



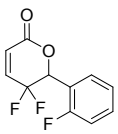
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.62 – 7.58 (m, 1H), 7.38 – 7.32 (m, 1H), 7.23 – 7.14 (td, *J* = 8, 0.8, 1H), 7.08 (ddd, *J* = 9.6, 8.3, 1.0 Hz, 1H), 6.36 – 6.31 (m, 1H), 6.10 – 6.03 (m, 1H), 5.08 (d, *J* = 19.1 Hz, 1H), 4.51 – 4.34 (m, 2H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -105.1 (m, 2F), -117.90 (m, 1F). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 161.55, 159.08, 134.92 (t, *J* = 9 Hz), 130.28 (d, *J* = 8 Hz), 129.85 (t, *J* = 3 Hz), 123.96 (d, *J* = 4 Hz), 122.38 (dd, *J* = 31, 26 Hz), 120.97 (d, *J* = 14 Hz), 115.07 (d, *J* = 22 Hz), 113.53 (dd, *J* = 243, 235 Hz), 77.27 (m), 66.12. MS (m/z): 214.1, 164.0, 133.1, 123.0, 95.0, 90.0, 75.0.

2,2-difluoro-1-phenylbut-3-en-1-yl acrylate (**7**)



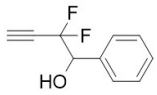
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.48 (t, *J* = 7.2 Hz, 1H), 7.37 – 7.32 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.07 (t, *J* = 9.2 Hz, 1H), 6.53 – 6.40 (m, 2H), 6.20 (dd, *J* = 17.3, 10.4 Hz, 1H), 5.99 – 5.86 (m, 2H), 5.66 (dt, *J* = 17.3, 2.3 Hz, 1H), 5.52 (d, *J* = 11.0 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -108.97 (m, 2F), -116.08 (m, 1F).

5,5-difluoro-6-(2-fluorophenyl)-5,6-dihydro-2H-pyran-2-one (**8**)

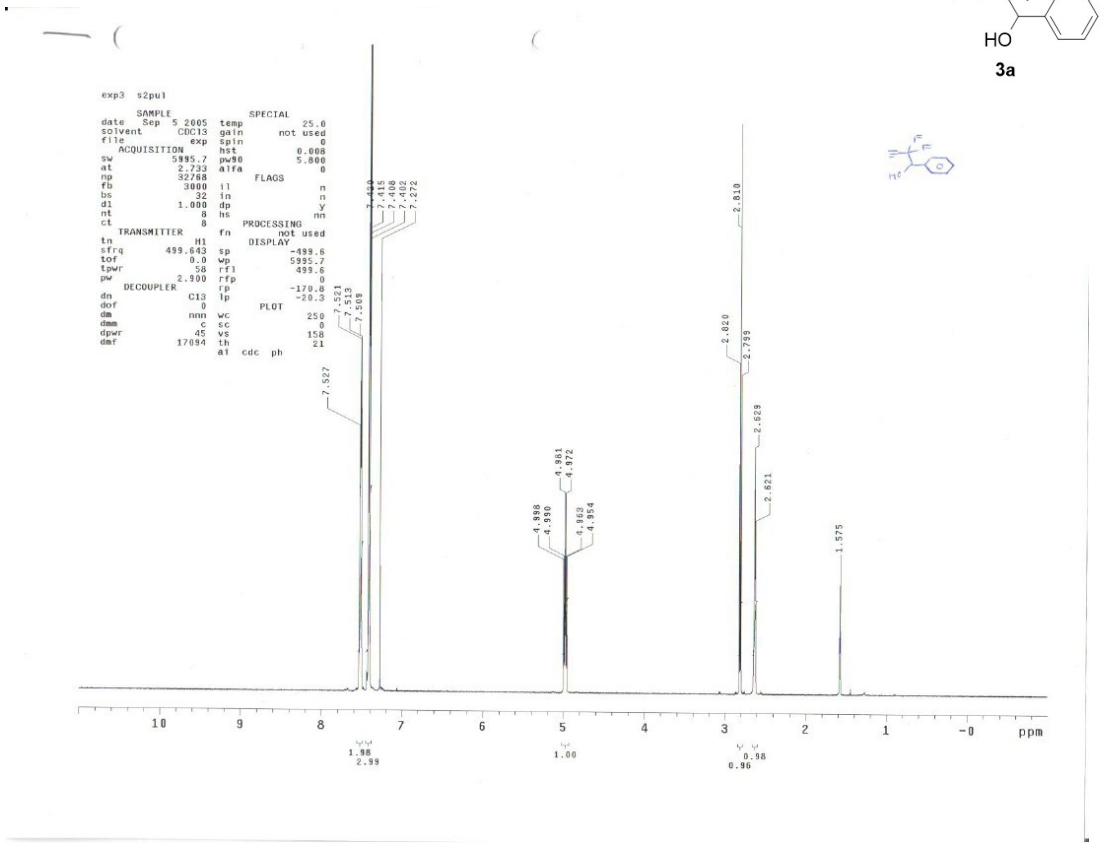


$^1$H NMR (400 MHz, $CDCl_3$) δ 7.60 (t, *J* = 7.4 Hz, 1H), 7.44 (dd, *J* = 14.2, 6.9 Hz, 1H), 7.26 (t, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 12 Hz, 1H), 6.94 (t, *J* = 9.1 Hz, 1H), 6.41 (d, *J* = 9.9 Hz, 1H), 5.98 (dd, *J* = 20.7, 3.6 Hz, 1H). $^{19}$F NMR (376 MHz, $CDCl_3$) δ -105.32 (dd, $J_{F\text{-}F}$ = 285.8 Hz, $J_{F\text{-}H}$ = 22.6 Hz, 1F), -112.16

(dtt, $J_{F\text{-}F}$ = 285.8 Hz, $J_{F\text{-}H}$ = 11.3, 3.76 Hz, 1F), -117.42 (m, 1F). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 161.67, 159.18, 138.30 (dd, *J* = 34, 25 Hz), 131.68 (d, *J* = 9 Hz), 130.03 (t, *J* = 3 Hz), 136.33 (dd, *J* = 10, 8 Hz), 124.37 (d, *J* = 4 Hz), 117.49 (d, *J* = 8 Hz), 115.48 (d, *J* = 9 Hz), 111.84 (dd, *J* = 247, 238 Hz), 74.13 (m). MS (m/z): 228.1, 158.0, 123.0, 104.0, 95.0, 76.0.


F
F
HO

**3a**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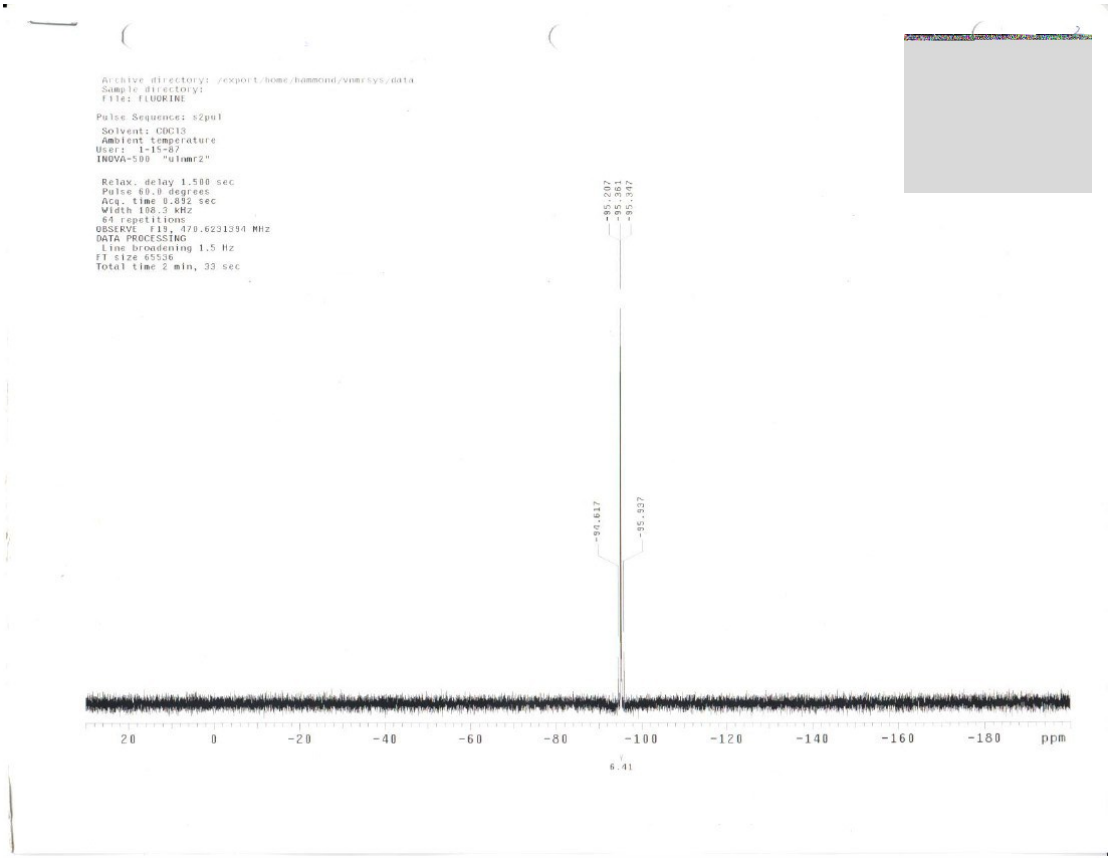
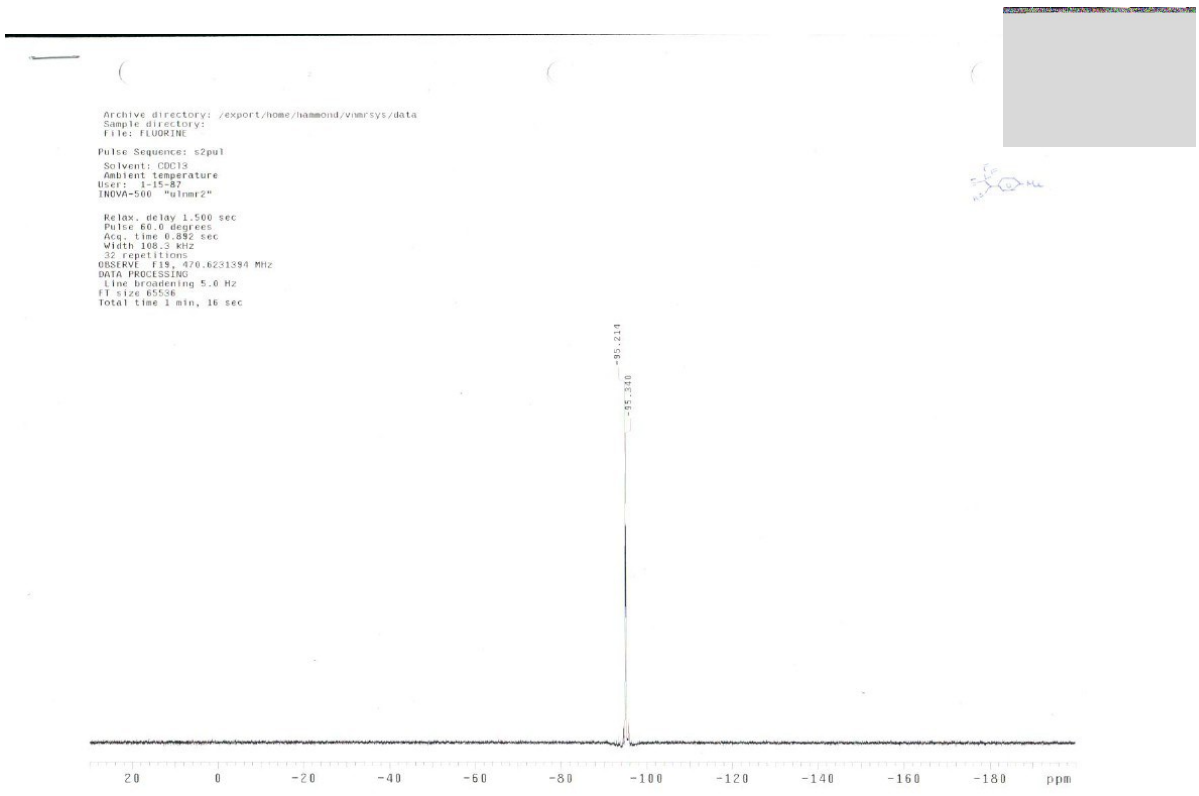
SAMPLE
date Sep 5 2005
solvent CDCl3
file exp
ACQUISITION
sw 5995.7
at 2.733
np 32768
fb 3000
bs 32
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.643
tof 0.0
tpwr 58
pw 2.900
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 17084
SPECIAL
temp 25.0
gain not used
spin 0
hst 0.008
pw90 5.800
alfa 0
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -499.6
wp 5995.7
rfl 499.6
rfp 0
rp -170.8
lp -20.3
PLOT
wc 250
sc 0
vs 158
th 21
al cdc ph
7.527
7.511
7.513
7.509
7.420
7.416
7.408
7.405
7.272
4.988
4.980
4.961
4.972
4.968
4.954
2.820
2.810
2.799
2.628
2.621
1.575
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.98
2.99
1.00
0.98
0.96


Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
64 repetitions
OBSERVE F19, 470.6231894 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 2 min, 33 sec
-95.207
-95.361
-95.497
-96.617
-95.937
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm
6.41


expl s2pul
SAMPLE
date Oct 11 2005
solvent CDCl3
file exp
ACQUISITION
sw 5995.7
at 2.733
np 32768
fb 3000
bs 32
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.843
tof 0.0
tpwr 60
pw 3.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 38
dmf 17094
SPECIAL
temp 25.0
gain not used
spin 0
hst 0.008
pw90 7.500
alfa 0
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -499.6
wp 5995.7
rfl 499.6
rfp 0
fp -17.1
lp -18.8
PLOT
wc 250
sc 0
vs 34
th 11
al cdc ph
F
F
HO
3b
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.94
2.00
1.00
0.76
0.93
3.26


Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
32 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 5.0 Hz
FT size 65536
Total time 1 min, 16 sec
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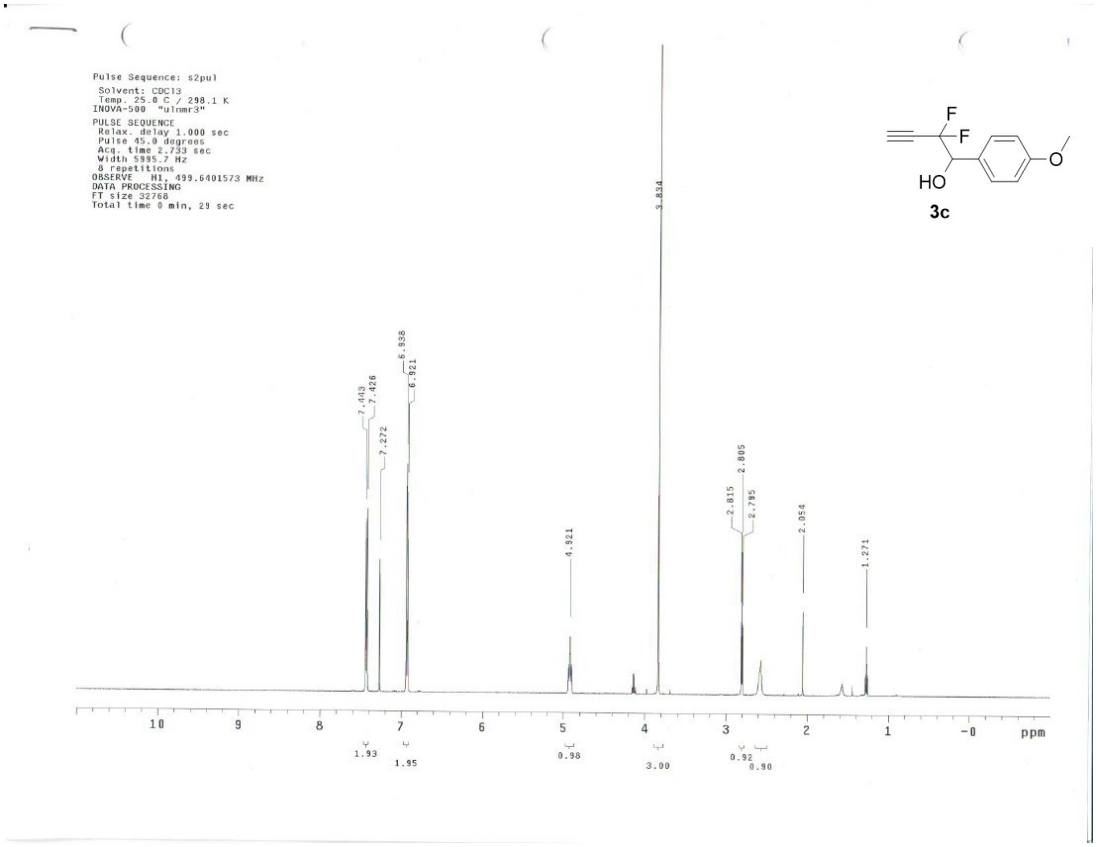
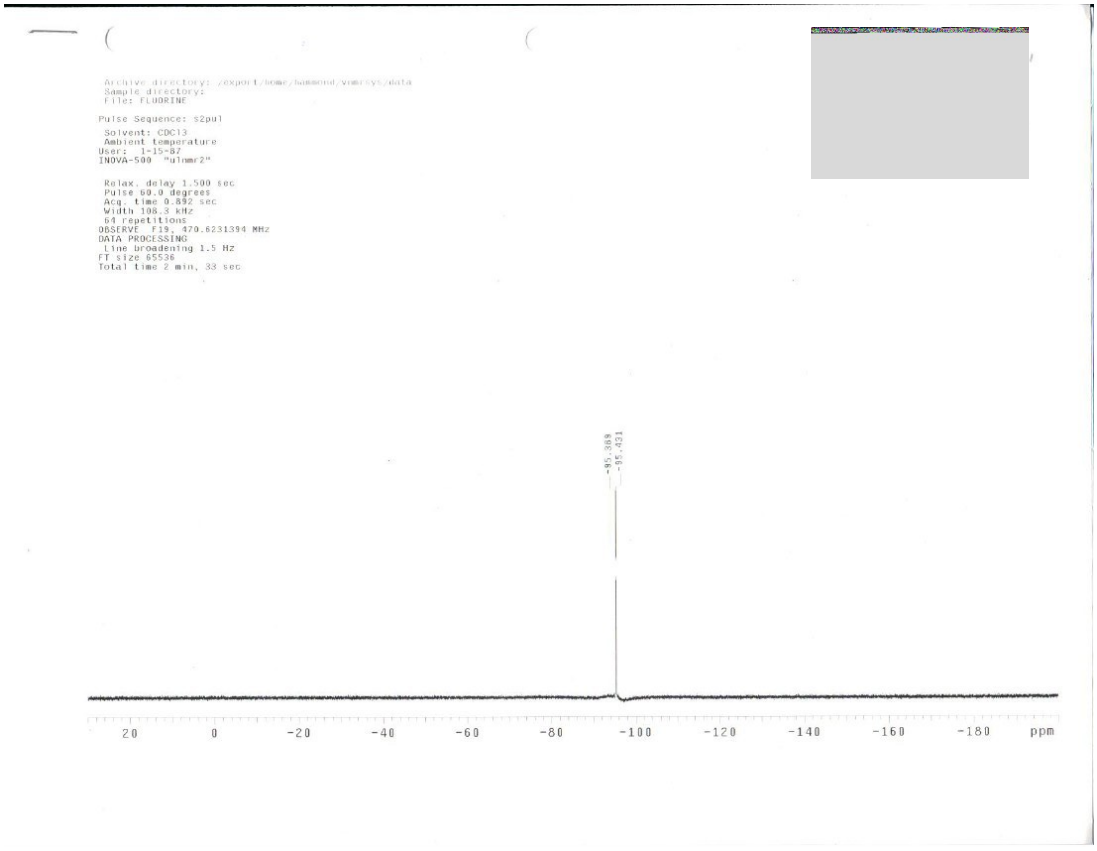
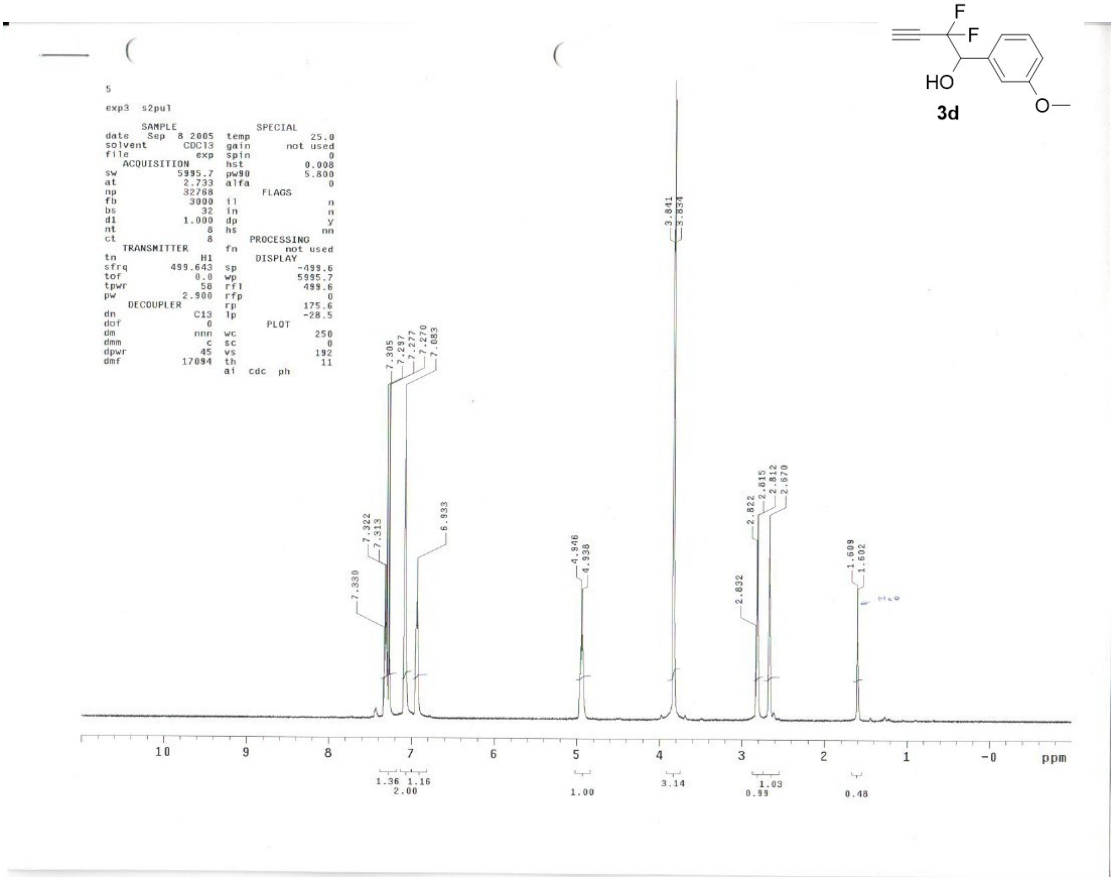
-95.340
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm


Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "ulnmr3"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.733 sec
Width 5995.7 Hz
8 repetitions
OBSERVE H1, 499.6401573 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 29 sec
F
F
HO
O
3c
7.413
7.406
7.272
6.938
6.921
4.921
3.834
2.815
2.805
2.795
2.054
1.271
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.93
1.95
0.98
3.00
0.92
0.90


Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: S2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
64 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 2 min, 33 sec
-95.389
-95.431
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

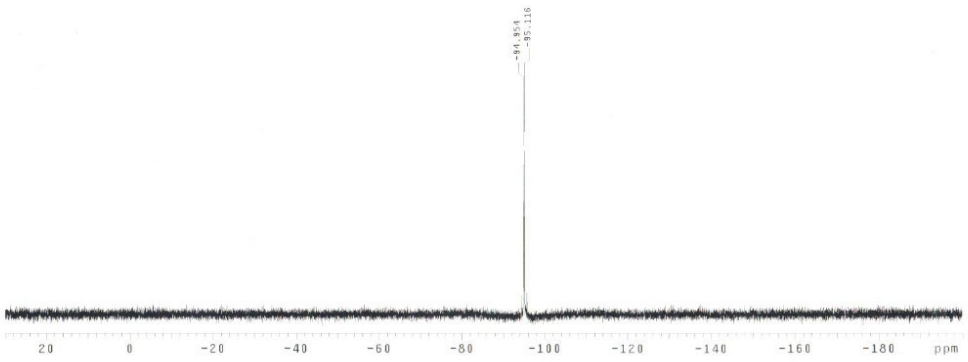

F
F
HO
O—
3d
5
exp3 s2pul
SAMPLE
date Sep 8 2005
solvent CDCl3
file exp
ACQUISITION
sw 5995.7
at 2.733
np 32768
fb 3000
bs 32
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.643
tof 0.8
tpwr 58
pw 2.900
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 17094
SPECIAL
temp 25.0
gain not used
spin 0
hst 0.008
pw90 5.800
alfa 0
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -499.6
wp 5995.7
rfl 499.6
rfp 0
rp 175.6
lp -28.5
PLOT
wc 250
sc 0
vs 192
th 11
al cdc ph
7.339
7.322
7.315
7.305
7.297
7.277
7.270
7.263
6.933
4.846
4.838
3.841
3.834
2.832
2.822
2.815
2.812
2.670
1.609
1.602
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.36 1.16
2.00
1.00
3.14
1.03
0.99
0.48

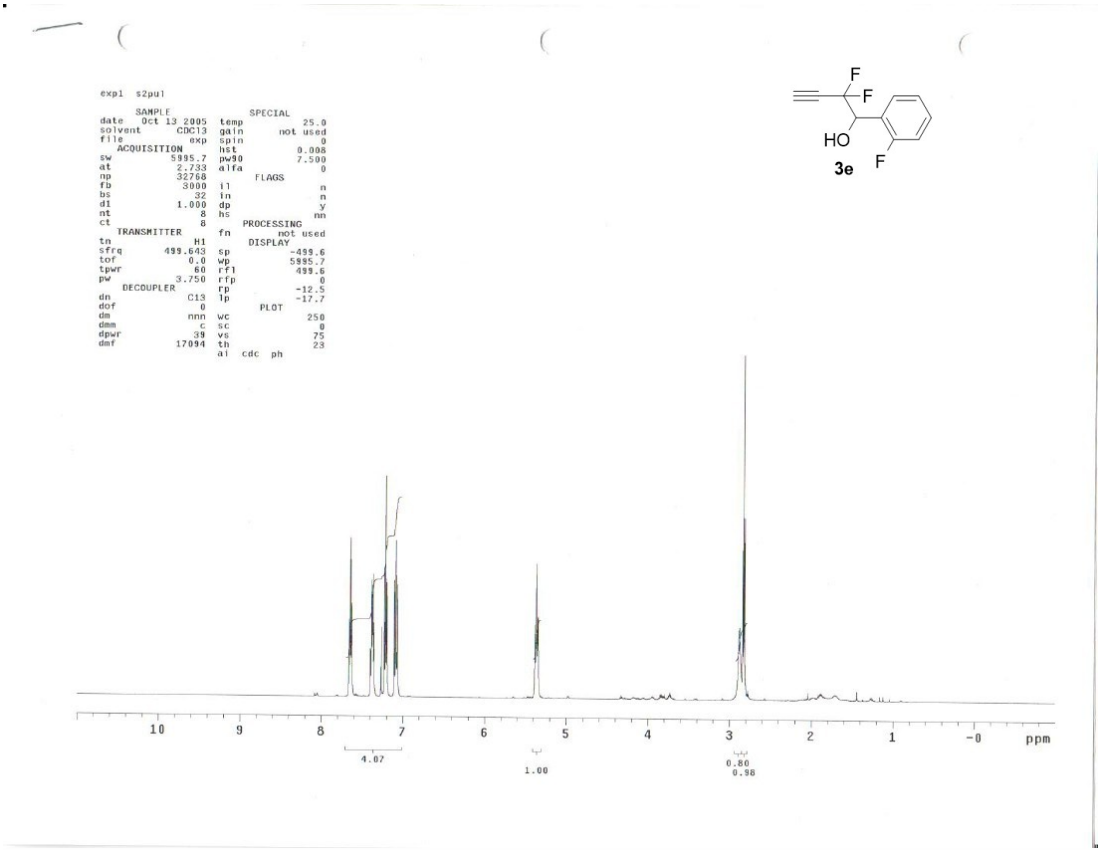
Archive directory: /export/home/bammond/vnmrsys/data
Sample directory: 5_08Sep2005
File: FLUORINE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "u1nmr2"

Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
64 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 2 min, 33 sec



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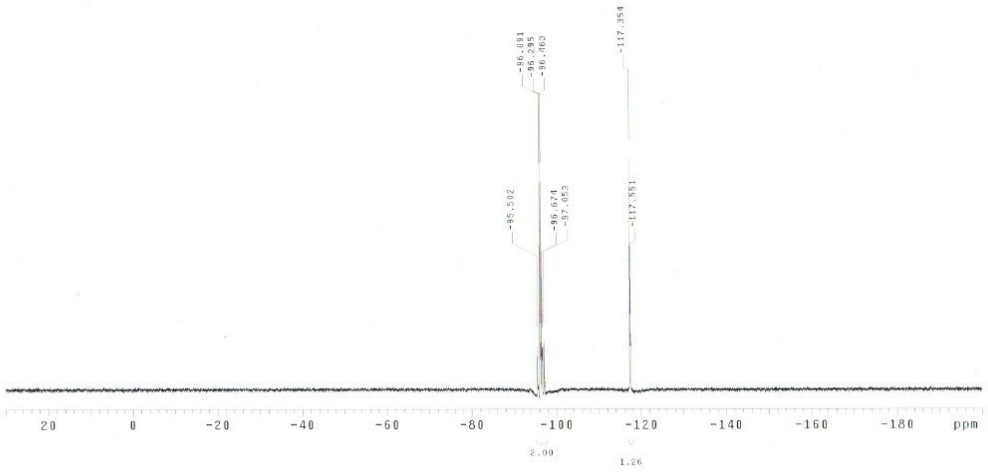
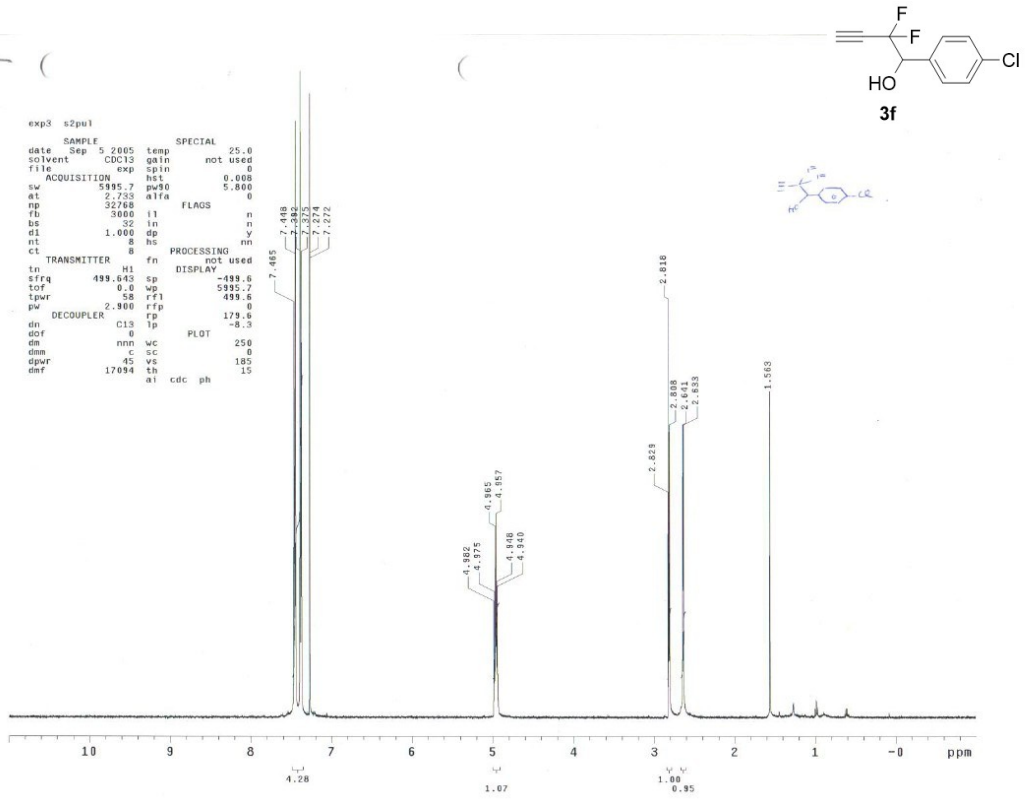
SAMPLE
date Oct 13 2005
solvent CDCl3
file exp
ACQUISITION
sw 5995.7
at 2.733
np 32768
fb 3000
bs 32
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.643
tof 0.0
tpwr 60
pw 3.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 39
dmf 17094
SPECIAL
temp 25.0
gain not used
spin 0
hst 0.008
pw90 7.500
alfa 0
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -499.6
wp 5995.7
rfl 499.6
rfp 0
rp -12.5
lp -17.7
PLOT
wc 250
sc 0
vs 75
th 23
al cdc ph
F
F
HO
F
3e
10
9
8
7
6
5
4
3
2
1
-0
ppm
4.07
1.00
0.80
0.98

Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "unmr2"

Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
16 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 5.0 Hz
FT size 65536
Total time 0 min, 38 sec

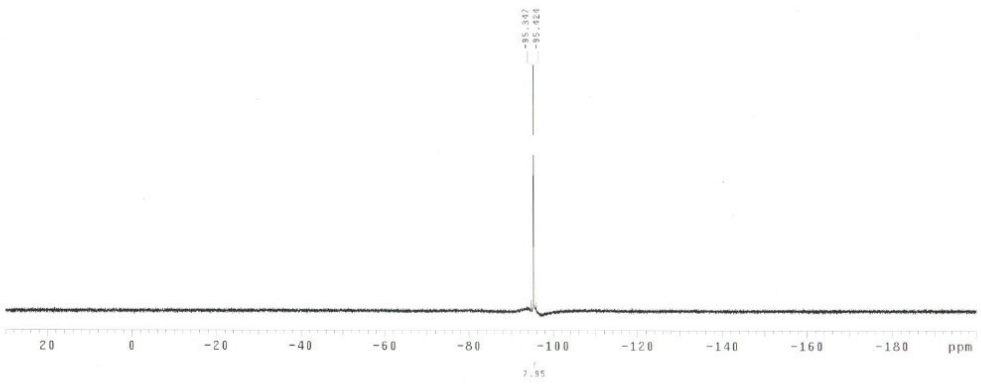


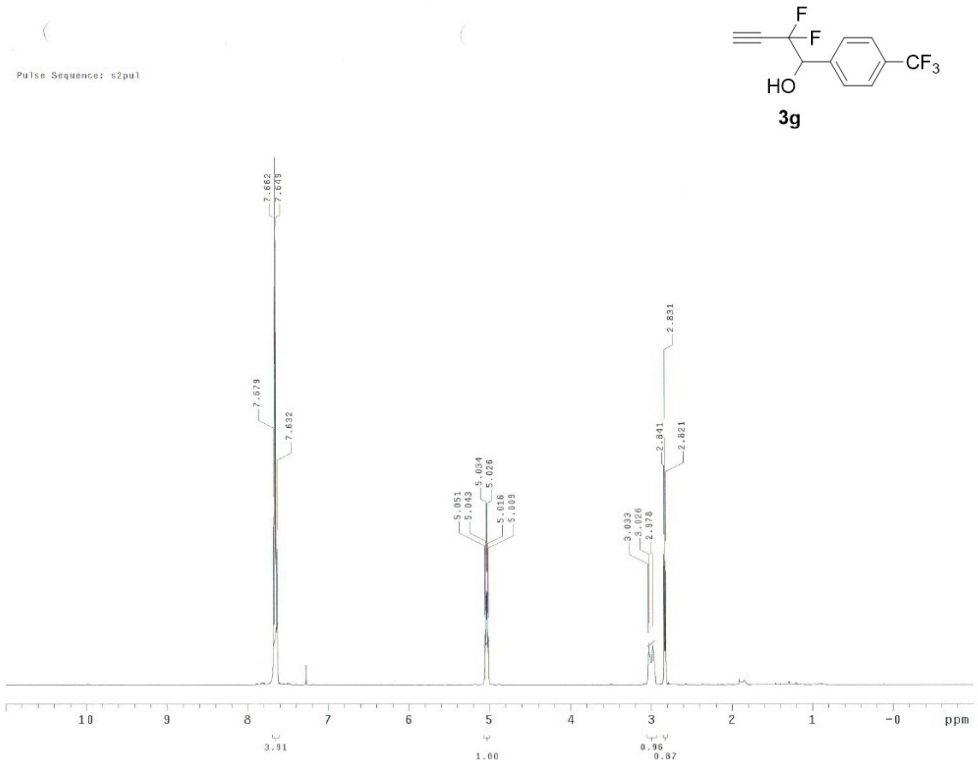

F
F
Cl
HO
3f
exp3 s2pul
SAMPLE
date Sep 5 2005
solvent CDCl3
file exp
ACQUISITION
sw 5995.7
at 2.733
np 32768
fb 3000
bs 32
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.643
tof 0.0
tpwr 58
pw 2.900
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 17094
SPECIAL
temp 25.0
gain not used
spin 0
hst 0.008
pw90 5.800
alfa 0
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -499.6
wp 5995.7
rfl 499.6
rfp 0
rp 179.6
lp -8.3
PLOT
wc 250
sc 0
vs 185
th 15
al cdc ph
7.465
7.448
7.382
7.373
7.274
7.272
4.982
4.975
4.965
4.957
4.948
4.940
2.829
2.818
2.808
2.801
2.793
1.563
10
9
8
7
6
5
4
3
2
1
-0
ppm
4.28
1.07
1.00
0.95

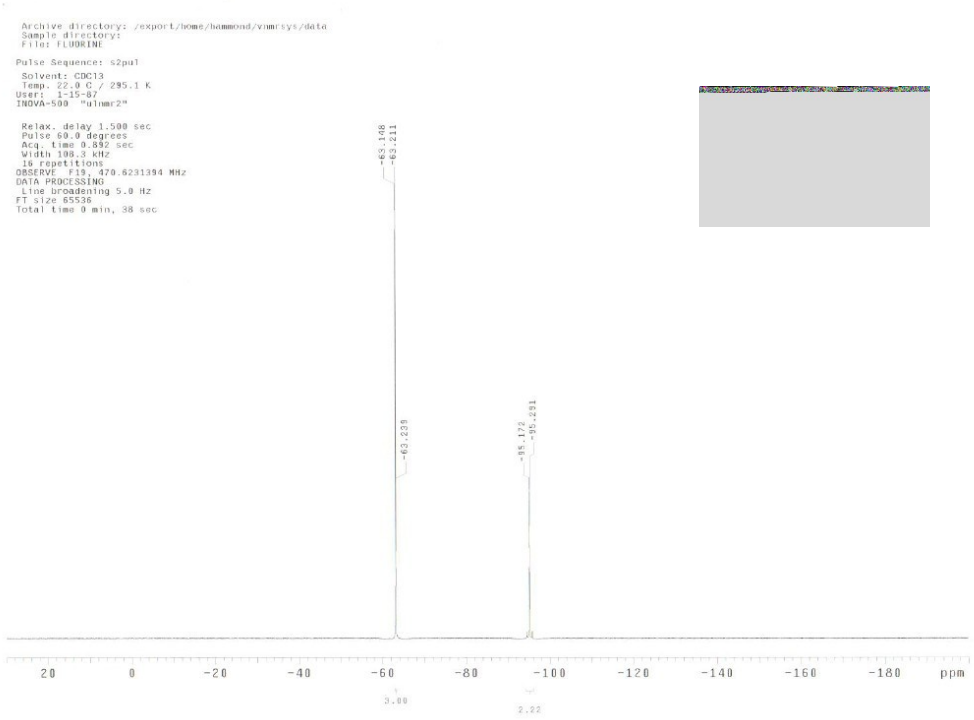
Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE

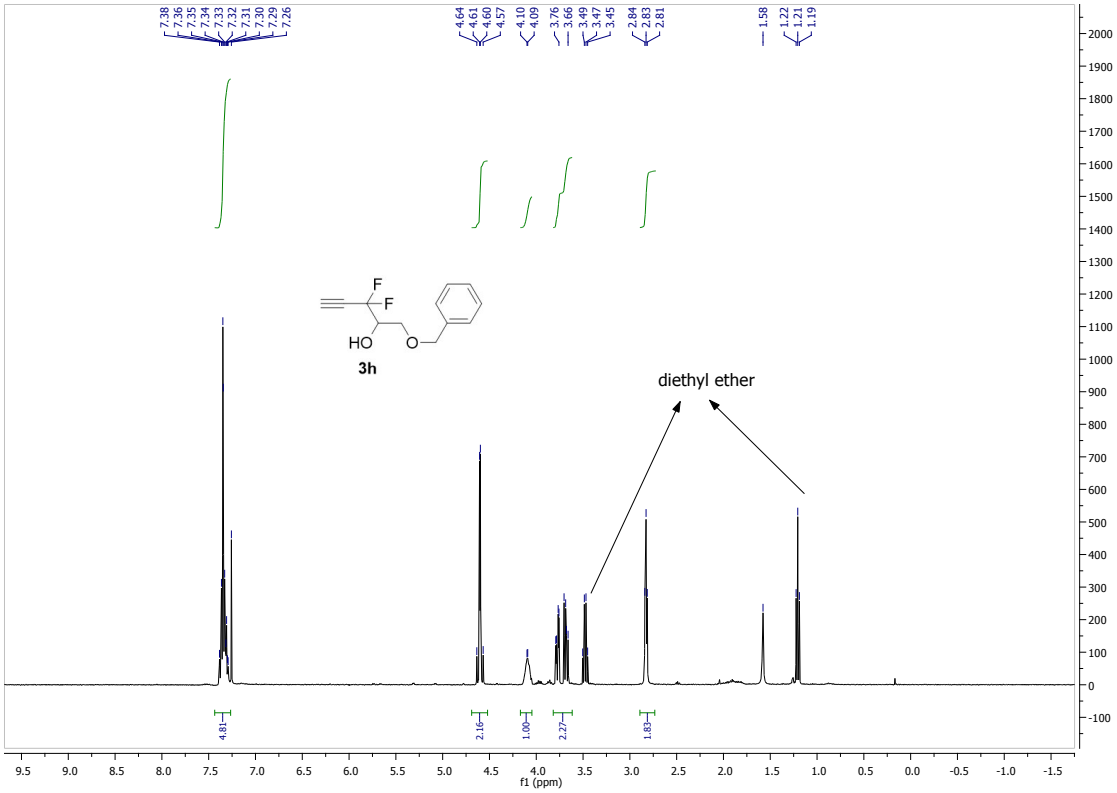
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: I-15-87
INOVA-500 "ulmmr2"

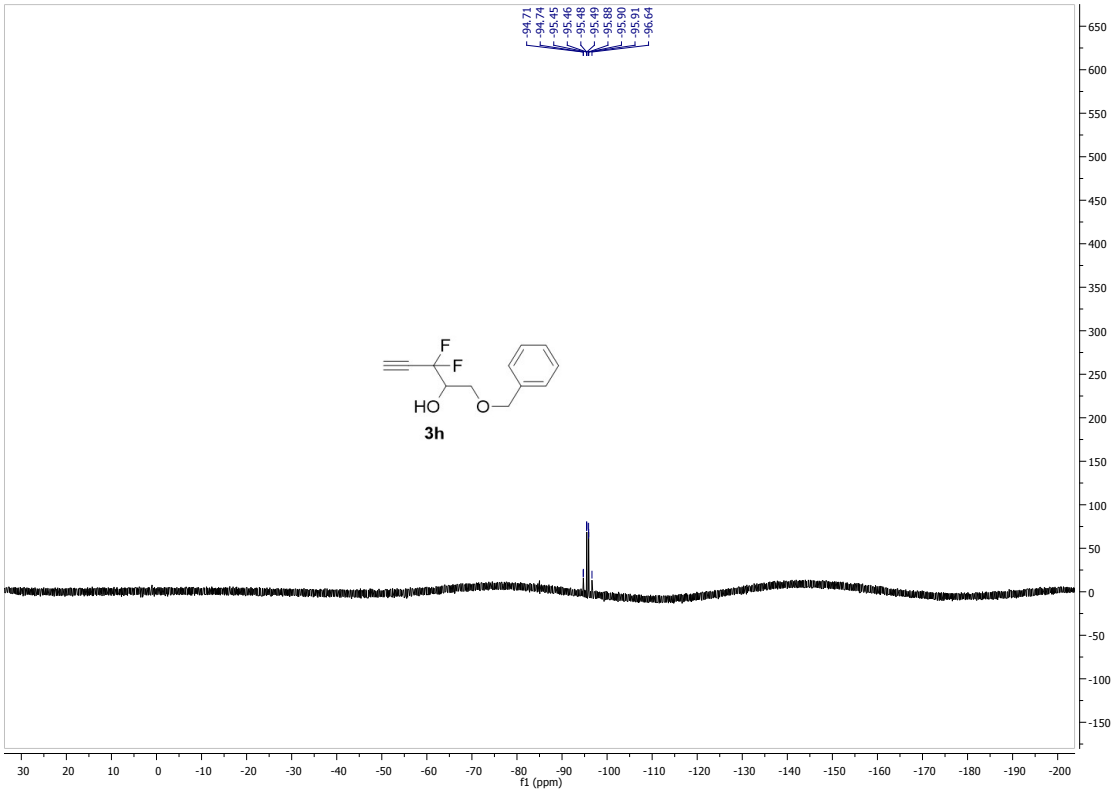
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 106.3 kHz
64 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 2 min, 33 sec

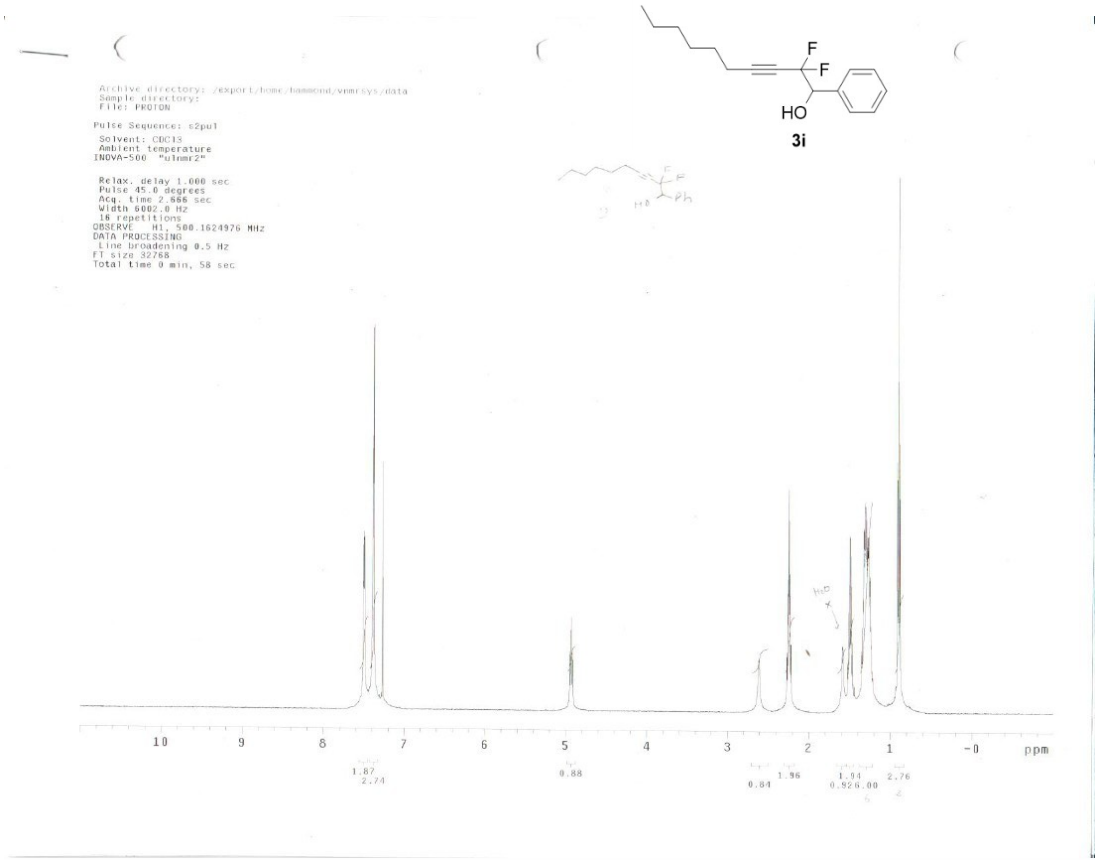


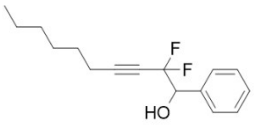

Pulse Sequence: s2pul
F
F
HO
CF3
3g
7.662
7.649
7.679
7.632
5.051
5.043
5.034
5.026
5.016
5.008
3.033
3.024
2.978
2.831
2.841
2.821
10
9
8
7
6
5
4
3
2
1
-0
ppm
3.91
1.00
0.96
0.87


Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 22.0 C / 295.1 K
User: 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
16 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 5.0 Hz
FT size 65536
Total time 0 min, 38 sec
-63.148
-63.211
-63.239
-85.172
-85.231
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm
3.00
2.22

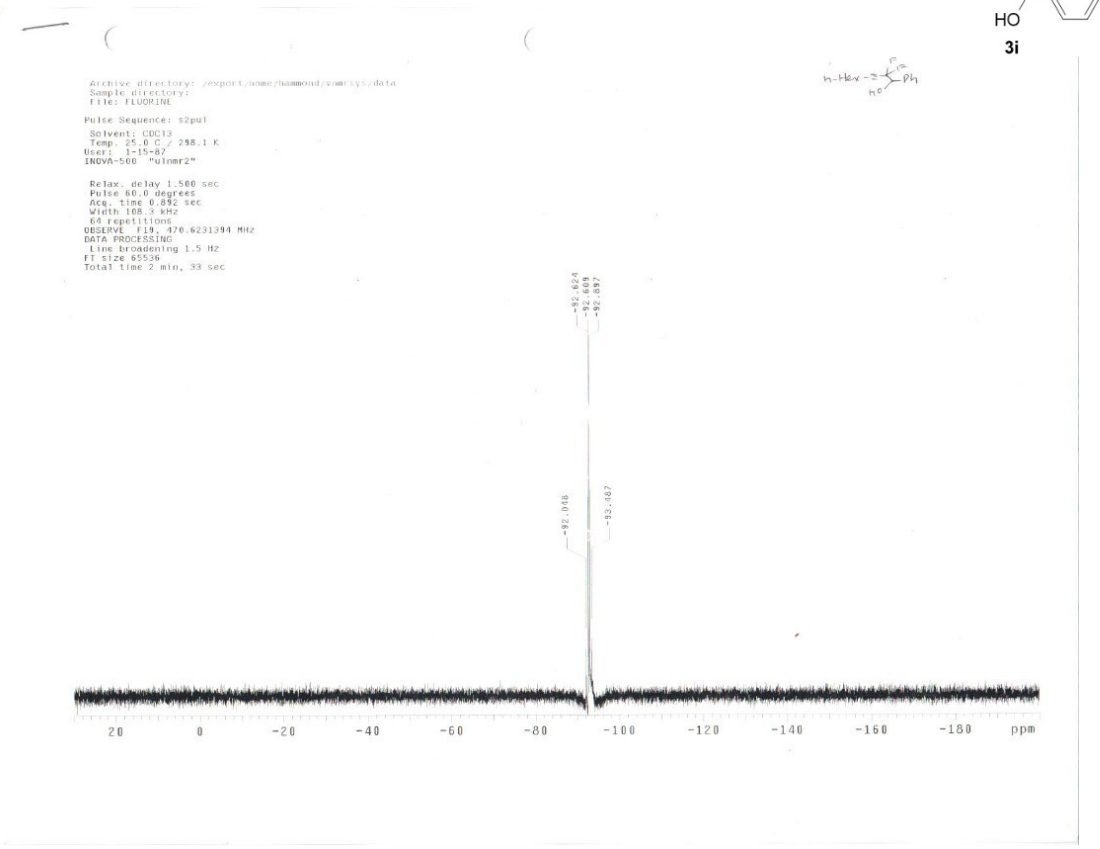

7.38
7.36
7.35
7.34
7.33
7.32
7.31
7.30
7.29
7.26
4.64
4.61
4.60
4.57
4.10
4.09
3.76
3.66
3.49
3.47
3.45
2.84
2.83
2.81
1.58
1.22
1.21
1.19
F
F
HO
O
3h
diethyl ether
4.81
2.16
1.00
2.27
1.83
f1 (ppm)

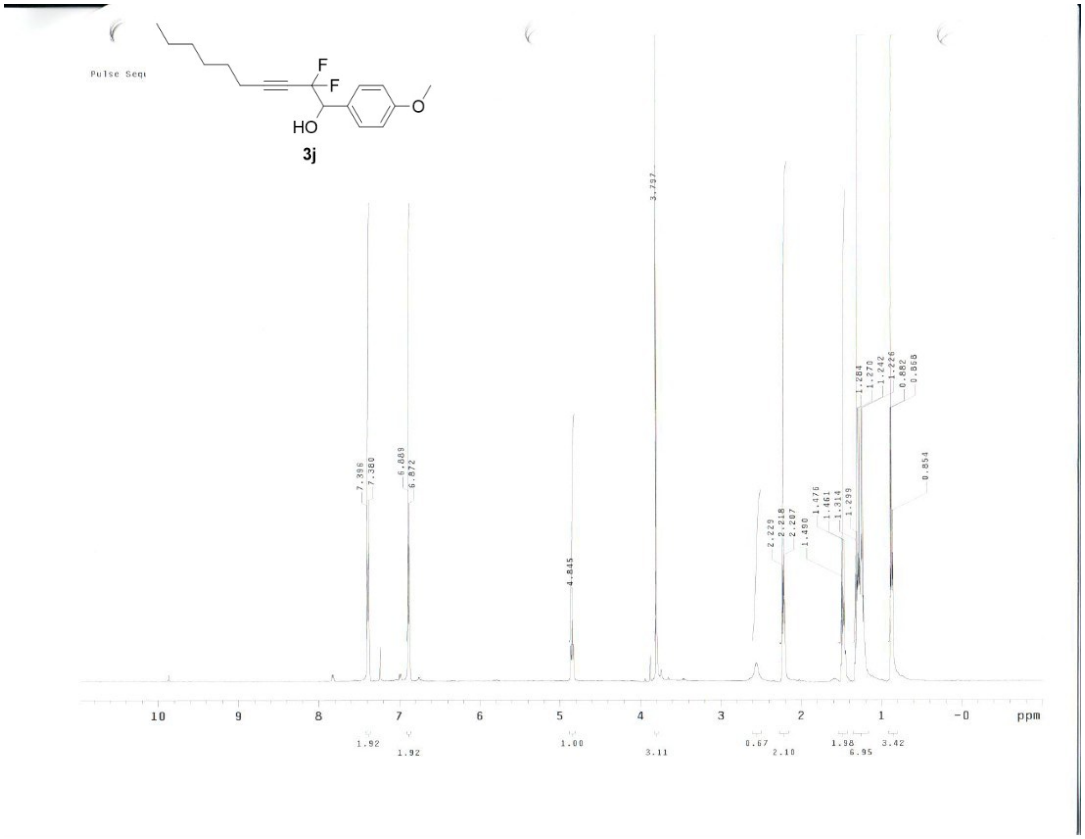

-94.71
-94.74
-95.45
-95.46
-95.48
-95.49
-95.88
-95.90
-95.91
-96.64
F
F
HO
O
3h
f1 (ppm)


F
F
HO
3i
Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "ulnmr2"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.666 sec
Width 6002.0 Hz
16 repetitions
OBSERVE H1, 500.1624976 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 0 min, 58 sec
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.87
2.74
0.88
0.84
1.96
1.94
0.926.00
2.76


F
F
HO

**3i**

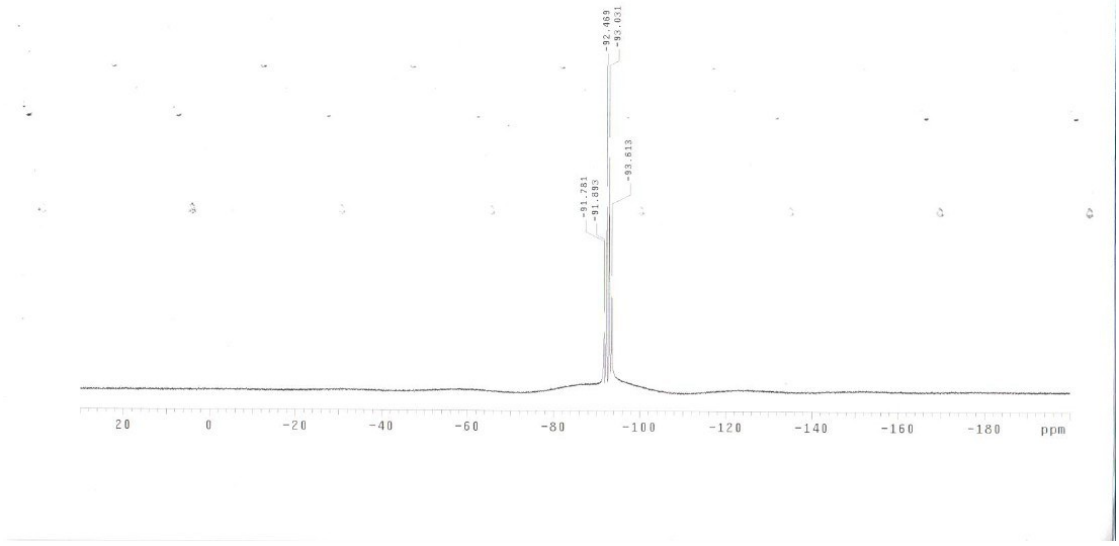

Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
64 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 2 min, 33 sec
-92.624
-92.669
-92.897
-92.046
-93.087
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

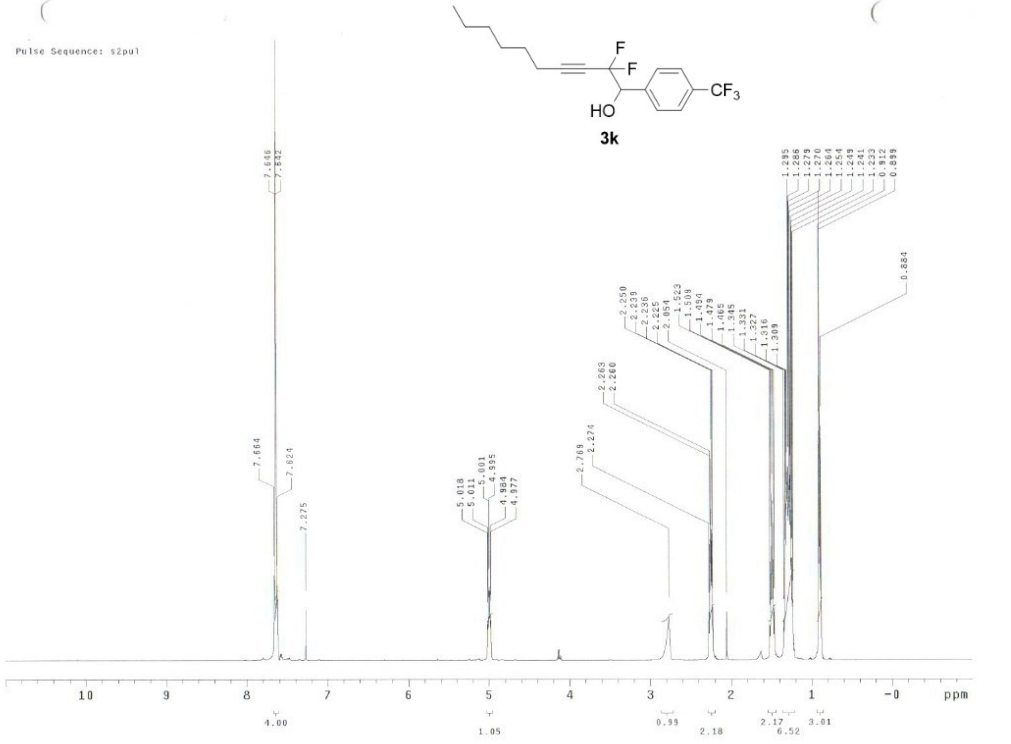

Pulse Seq
F
F
HO
O
3j
7.386
7.380
6.869
6.872
4.845
3.797
2.229
2.218
2.207
1.490
1.476
1.461
1.434
1.299
1.284
1.270
1.242
1.226
0.882
0.868
0.854
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.92
1.92
1.00
3.11
0.67
2.10
1.98
6.95
3.42

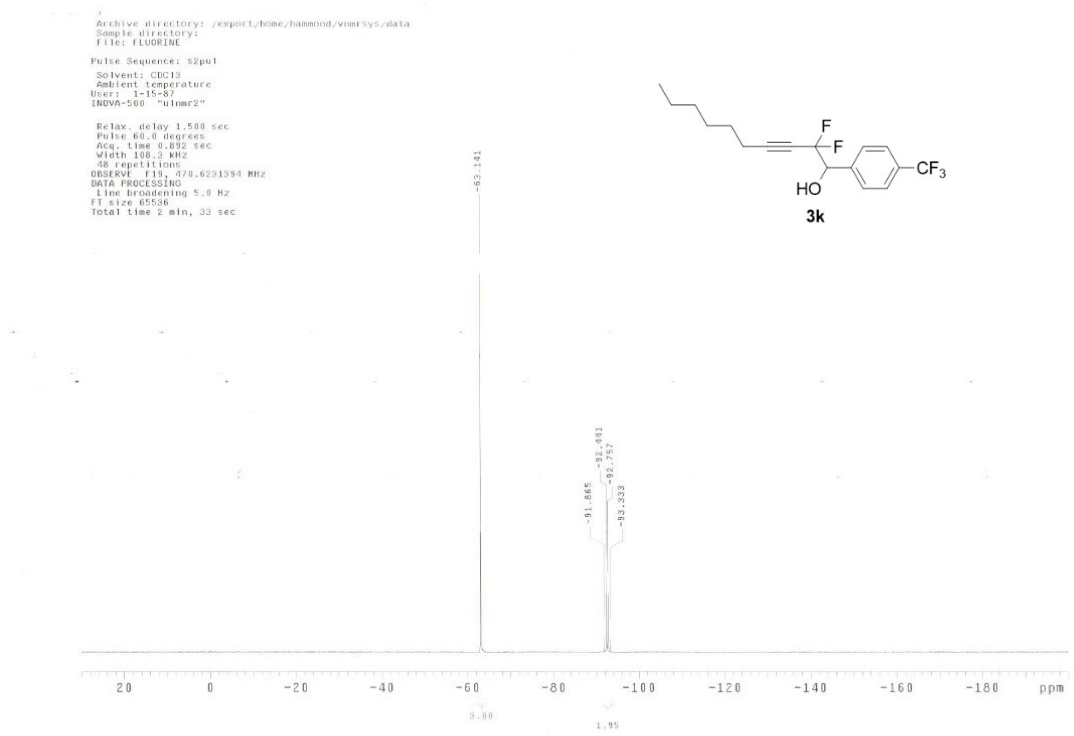
Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE

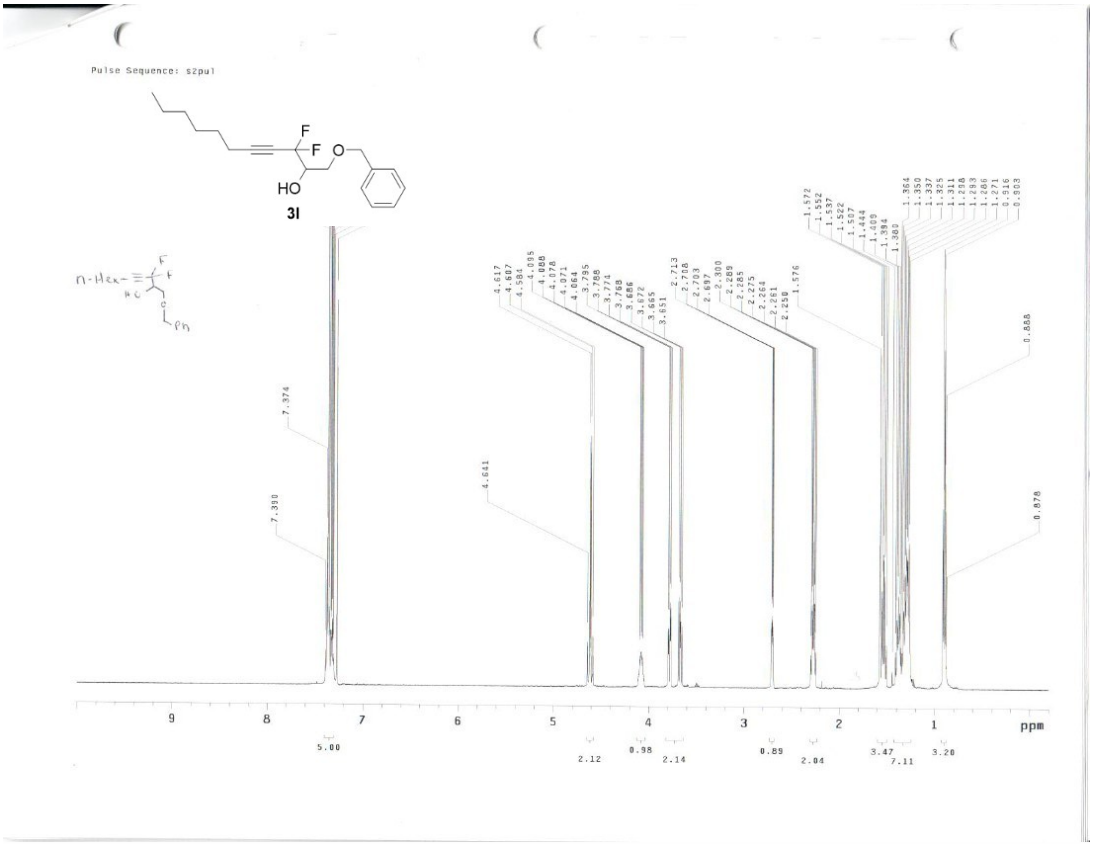
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ulnmr2"

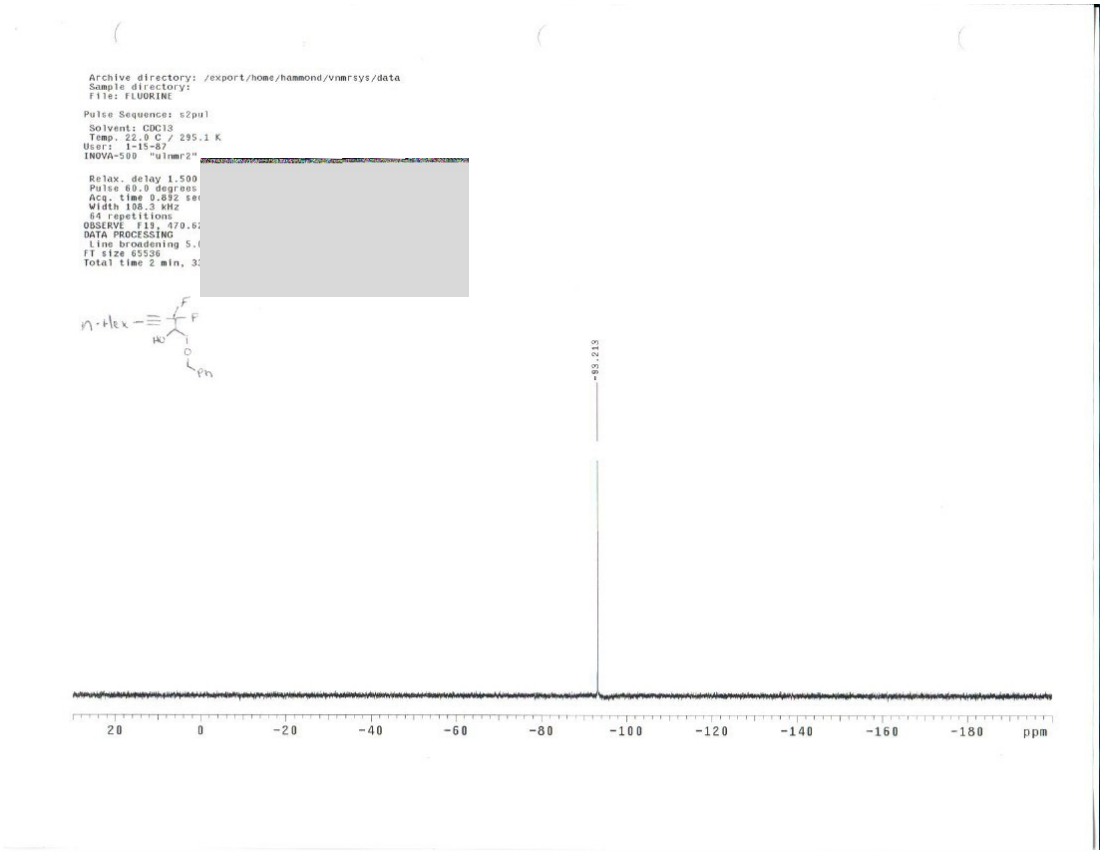
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
64 repetitions
OBSERVE F19, 470.6231394 MHz
DATA PROCESSING
Line broadening 5.0 Hz
FT size 65536
Total time 2 min, 33 sec

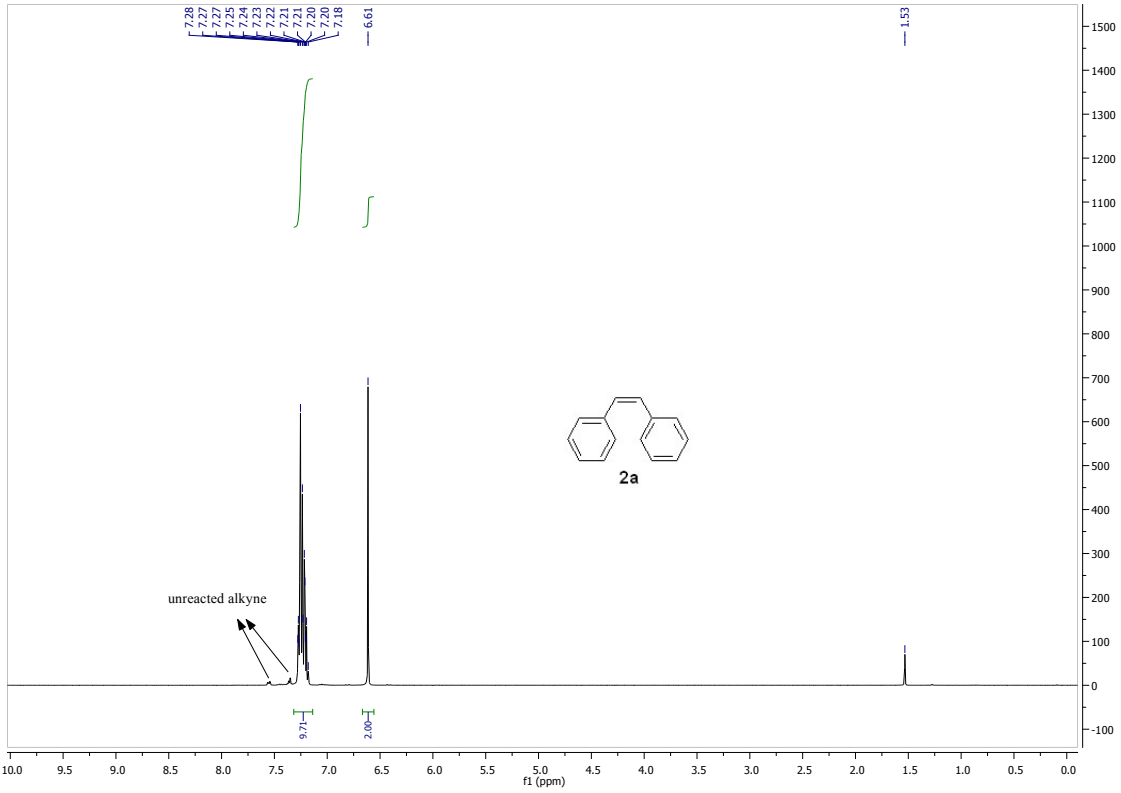


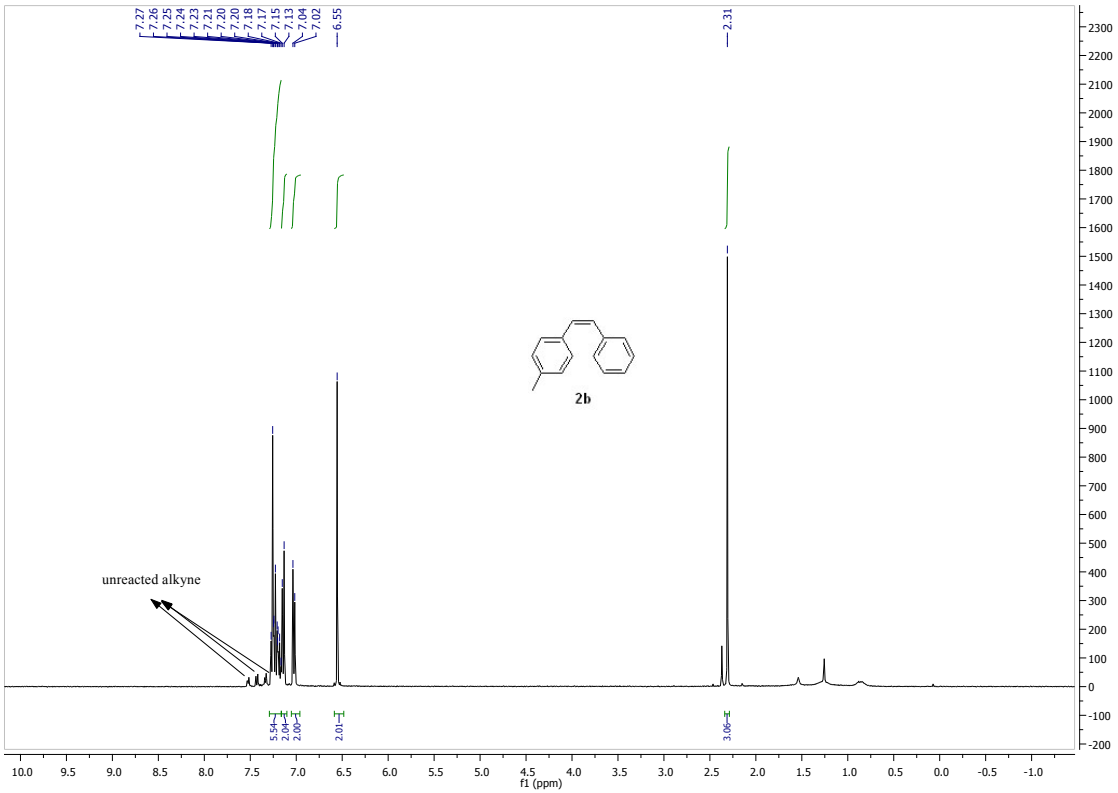

Pulse Sequence: s2pul
F
F
HO
CF3
3k
7.646
7.642
7.664
7.624
7.275
5.018
5.011
5.001
4.995
4.984
4.977
2.769
2.274
2.263
2.260
2.250
2.239
2.236
2.225
2.054
2.023
1.509
1.494
1.479
1.466
1.346
1.331
1.327
1.316
1.288
1.295
1.286
1.279
1.270
1.260
1.254
1.249
1.241
1.233
0.912
0.899
0.884
10
9
8
7
6
5
4
3
2
1
-0
ppm
4.00
1.05
0.99
2.18
2.17
6.52
3.01

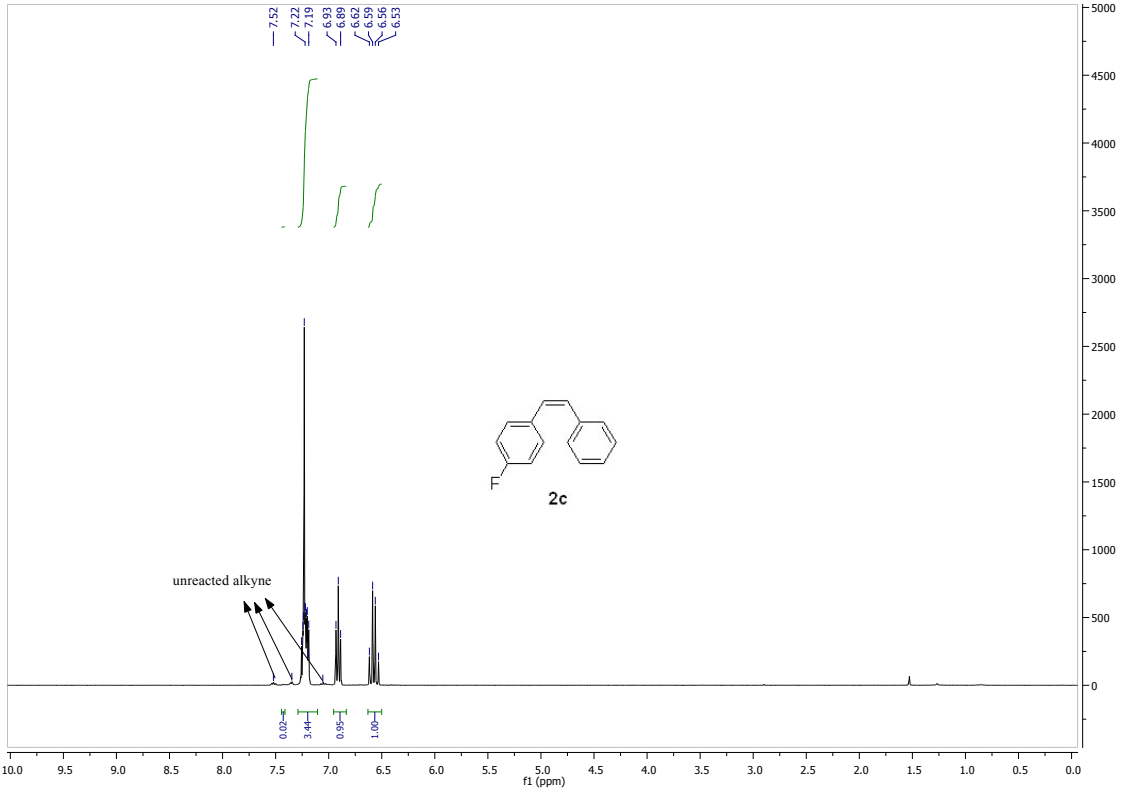

Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500 sec
Pulse 60.0 degrees
Acq. time 0.892 sec
Width 108.3 kHz
48 repetitions
OBSERVE f19, 470.6231394 MHz
DATA PROCESSING
Line broadening 5.0 Hz
FT size 65536
Total time 2 min, 33 sec
-63.141
-91.865
-92.401
-92.757
-93.333
F
F
HO
CF3
3k
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm
3.00
1.95

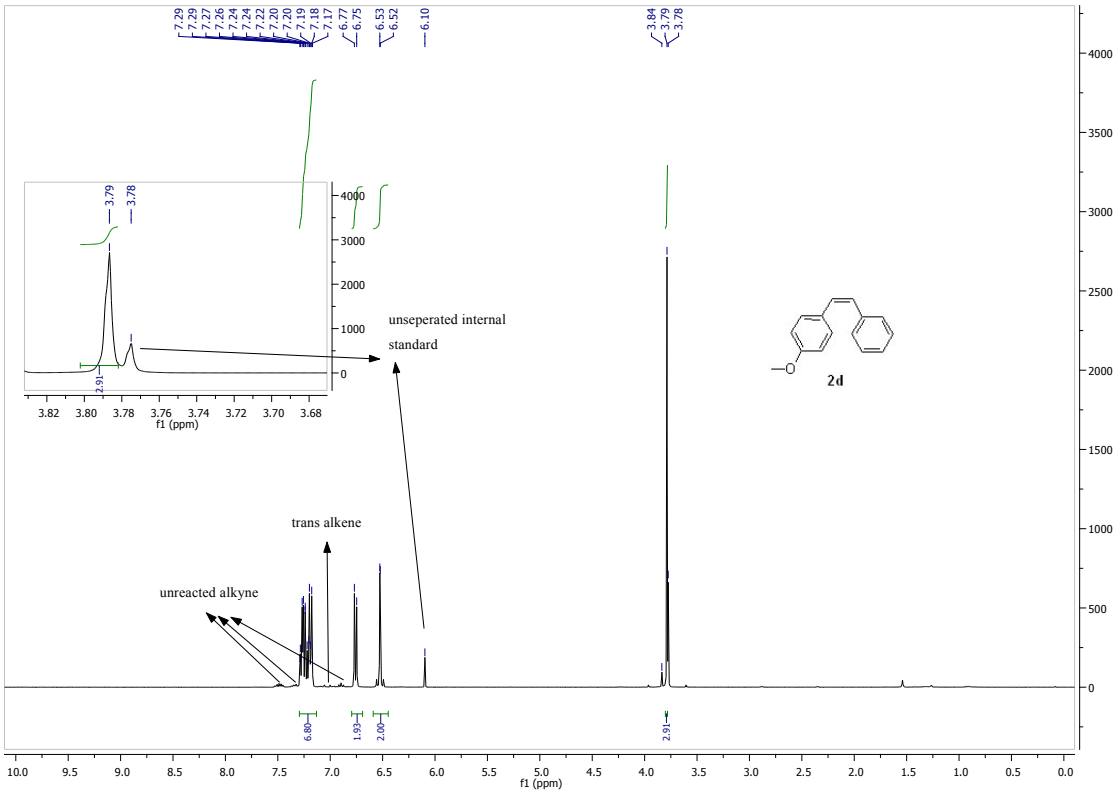

Pulse Sequence: s2pul
F
F
O
HO
3l
n-Hex-
Ph
7.374
7.390
4.641
4.617
4.607
4.584
4.095
4.088
4.078
4.071
4.064
3.775
3.758
3.774
3.768
3.686
3.672
3.665
3.651
2.713
2.708
2.703
2.697
2.300
2.289
2.285
2.275
2.264
2.261
2.259
1.576
1.572
1.552
1.537
1.522
1.517
1.444
1.409
1.394
1.380
1.364
1.350
1.337
1.325
1.311
1.306
1.293
1.206
1.271
1.255
1.203
0.888
0.878
9
8
7
6
5
4
3
2
1
ppm
5.00
2.12
0.98
2.14
0.89
2.04
3.47
7.11
3.20

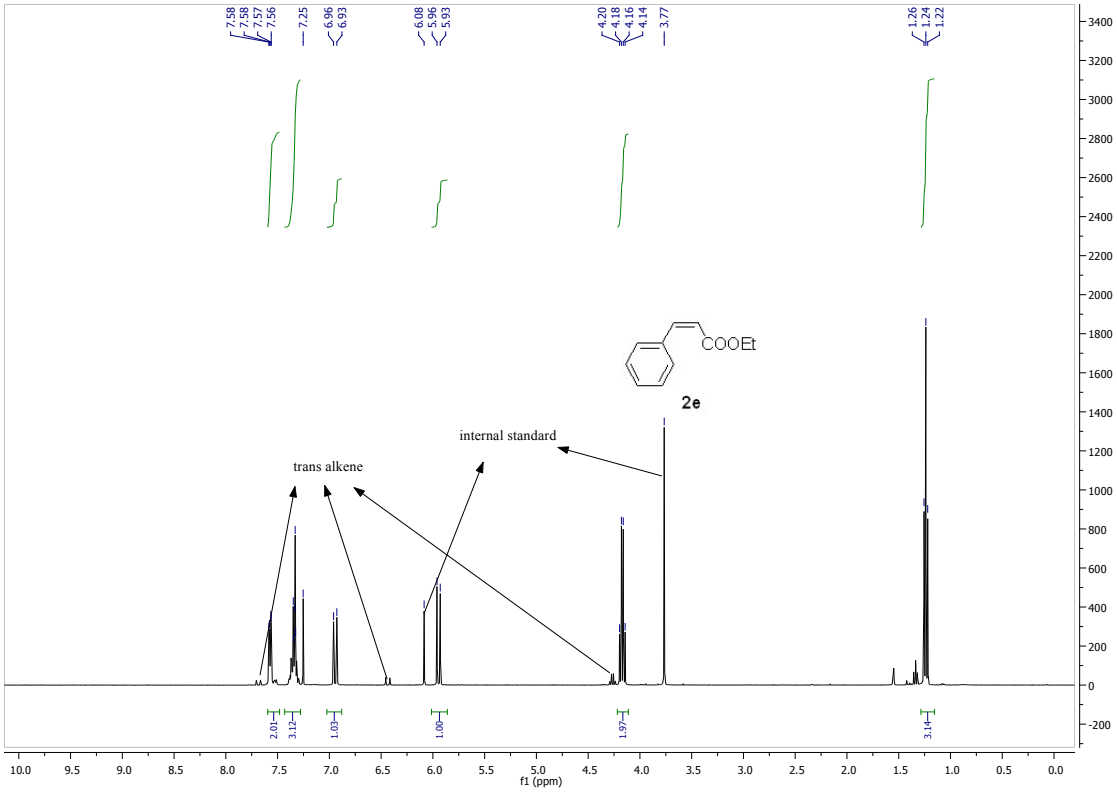

Archive directory: /export/home/hammond/vnmrsys/data
Sample directory:
File: FLUORINE
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 22.0 C / 295.1 K
Users 1-15-87
INOVA-500 "ulnmr2"
Relax. delay 1.500
Pulse 60.0 degrees
Acq. time 0.892 se
Width 108.3 kHz
64 repetitions
OBSERVE F19, 470.6
DATA PROCESSING
line broadening 5.
FT size 65536
Total time 2 min, 3
n-Hex
F
F
HO
O
Ph
-93.213
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

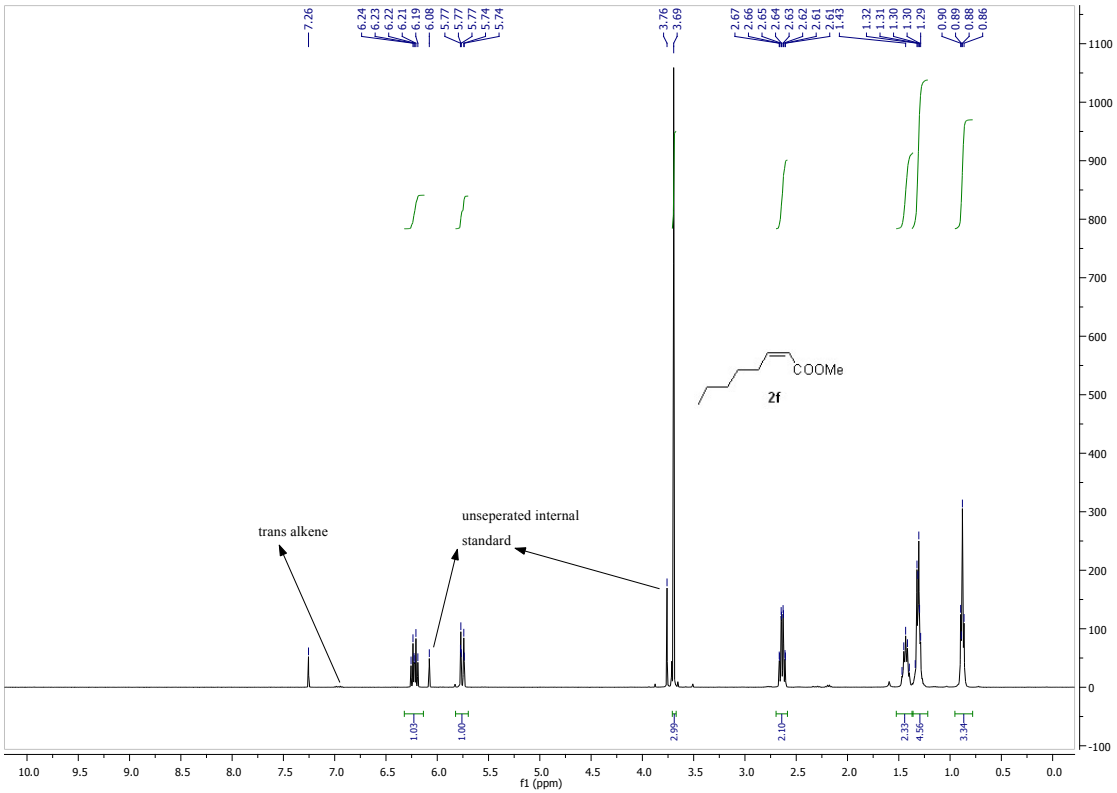

7.28
7.27
7.27
7.25
7.24
7.23
7.22
7.21
7.21
7.20
7.20
7.18
6.61
1.53
2a
unreacted alkyne
9.71
2.00
f1 (ppm)

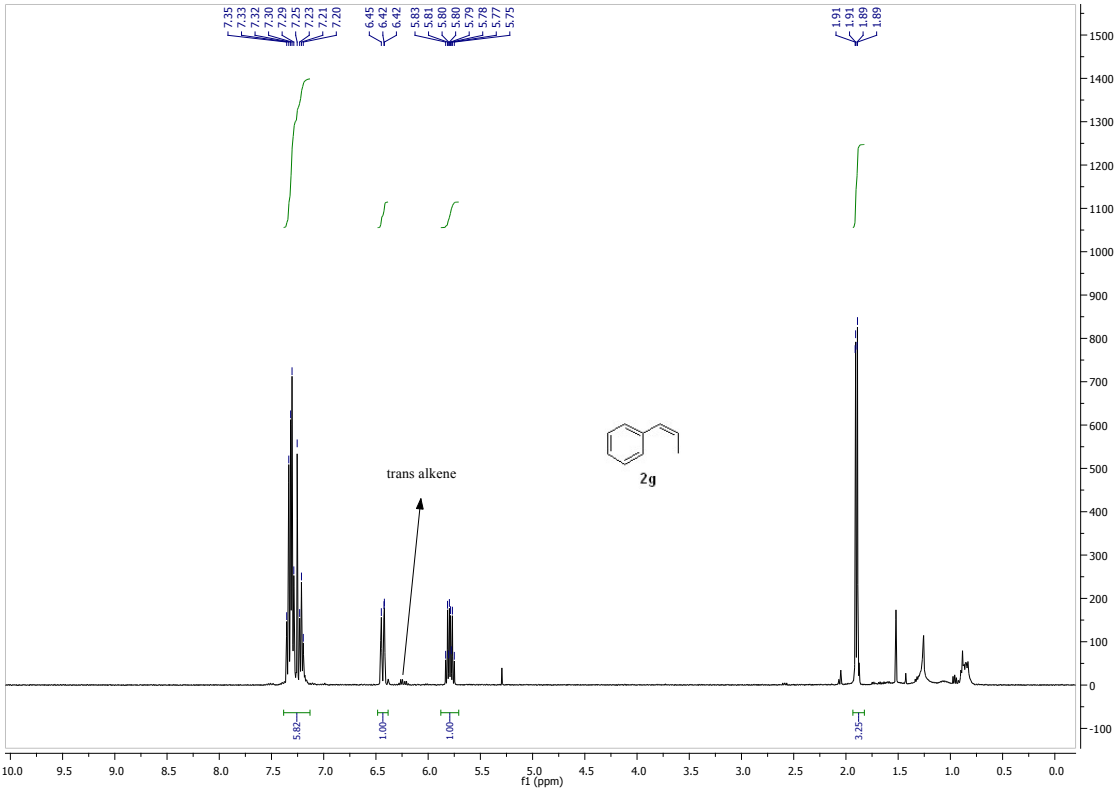

7.27
7.26
7.25
7.24
7.23
7.21
7.20
7.20
7.18
7.17
7.15
7.13
7.04
7.02
6.55
2.31
2b
unreacted alkyne
5.54
2.04
2.00
2.01
3.06
f1 (ppm)

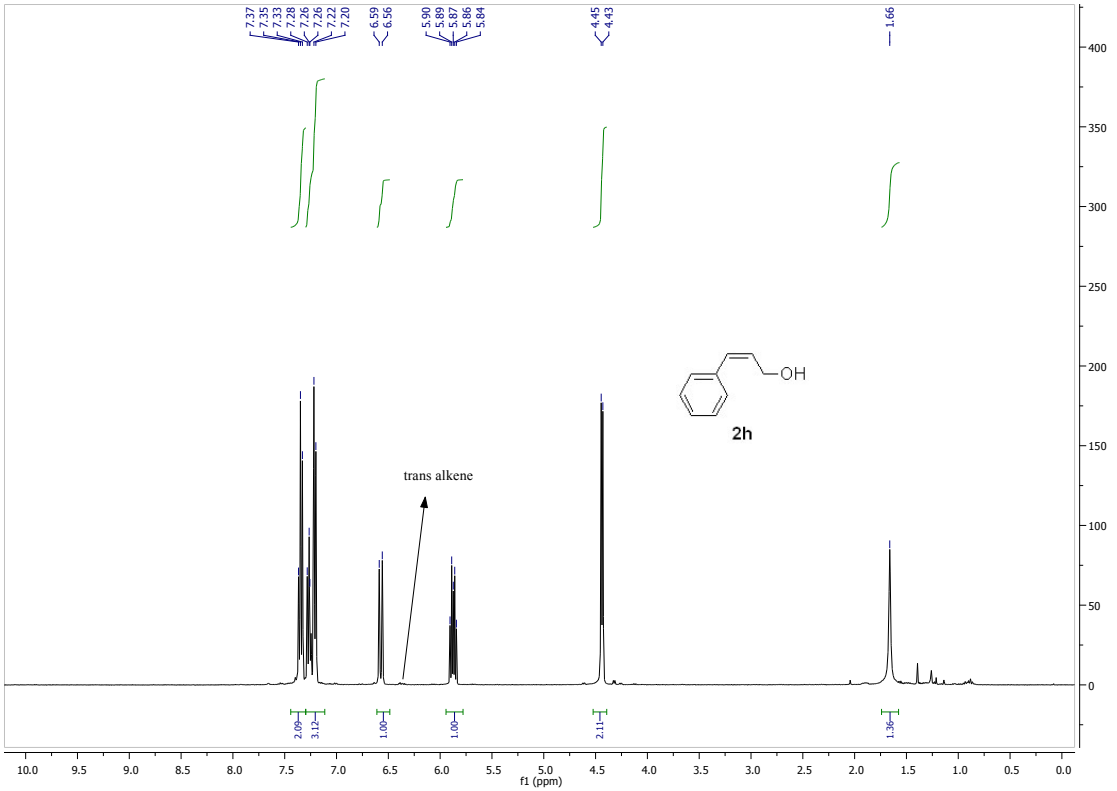

7.52
7.22
7.19
6.93
6.89
6.62
6.59
6.56
6.53
F
2c
unreacted alkyne
0.02
3.44
0.95
1.00
f1 (ppm)

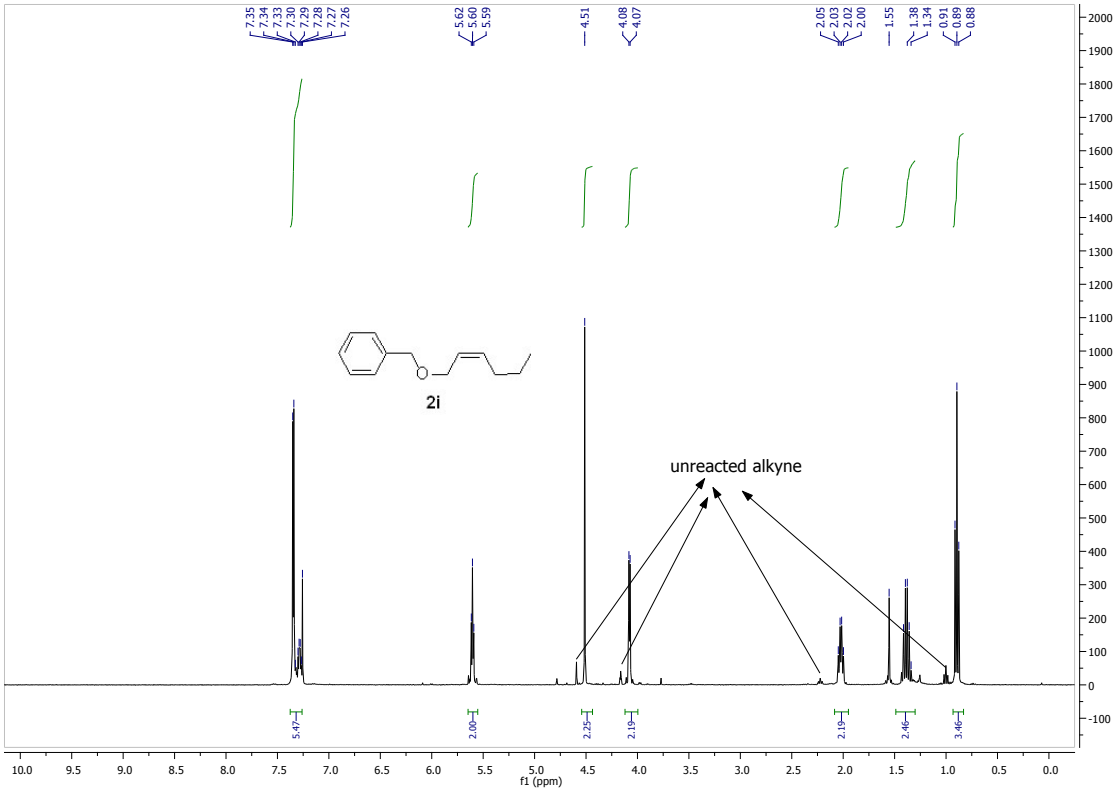

7.29
7.29
7.27
7.26
7.24
7.24
7.22
7.20
7.20
7.19
7.18
7.17
6.77
6.75
6.53
6.52
6.10
3.84
3.79
3.78
3.79
3.78
2.91
unseperated internal
standard
trans alkene
unreacted alkyne
O
2d
6.80
1.93
2.00
2.91
f1 (ppm)

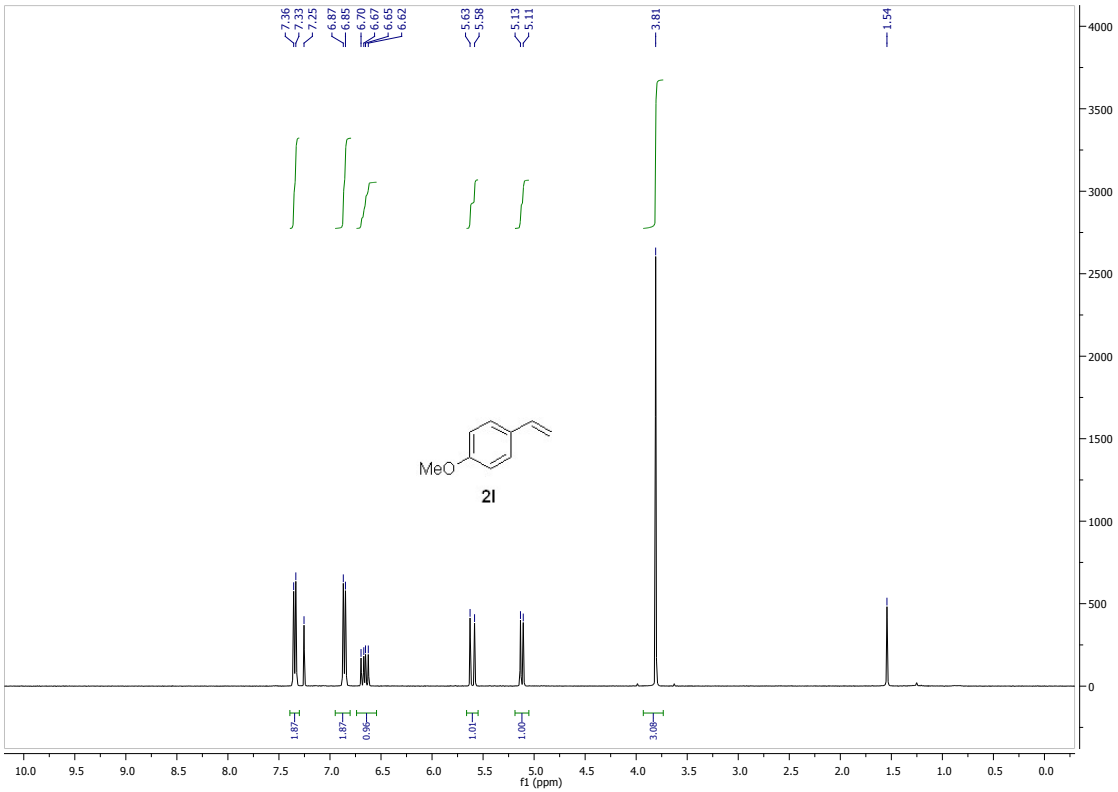

7.58
7.58
7.57
7.56
7.25
6.96
6.93
6.08
5.96
5.93
4.20
4.18
4.16
4.14
3.77
1.26
1.24
1.22
COOEt
2e
internal standard
trans alkene
2.01
3.12
1.03
1.00
1.97
3.14
f1 (ppm)

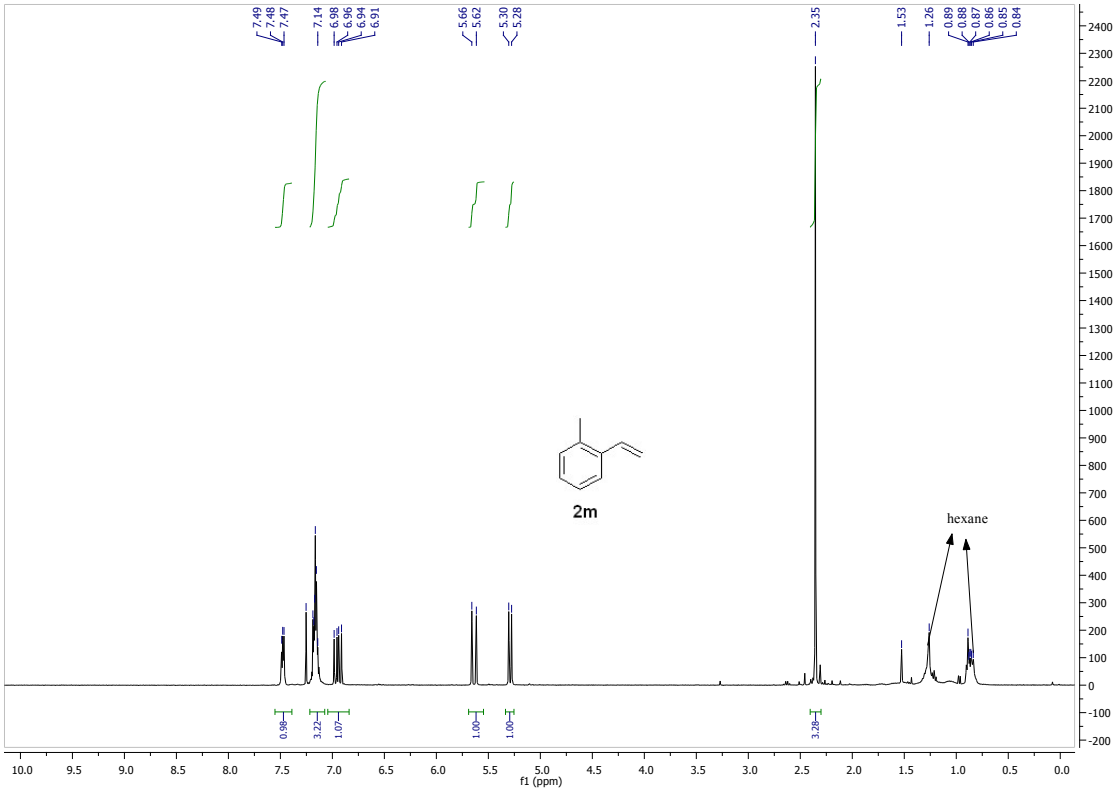

7.26
6.24
6.23
6.22
6.21
6.19
6.08
5.77
5.77
5.77
5.74
5.74
3.76
3.69
2.67
2.66
2.65
2.64
2.63
2.62
2.61
2.61
1.43
1.32
1.31
1.30
1.30
1.29
0.90
0.89
0.88
0.86
COOMe
2f
trans alkene
unseperated internal
standard
1.03
1.00
2.99
2.10
2.33
4.56
3.34
f1 (ppm)

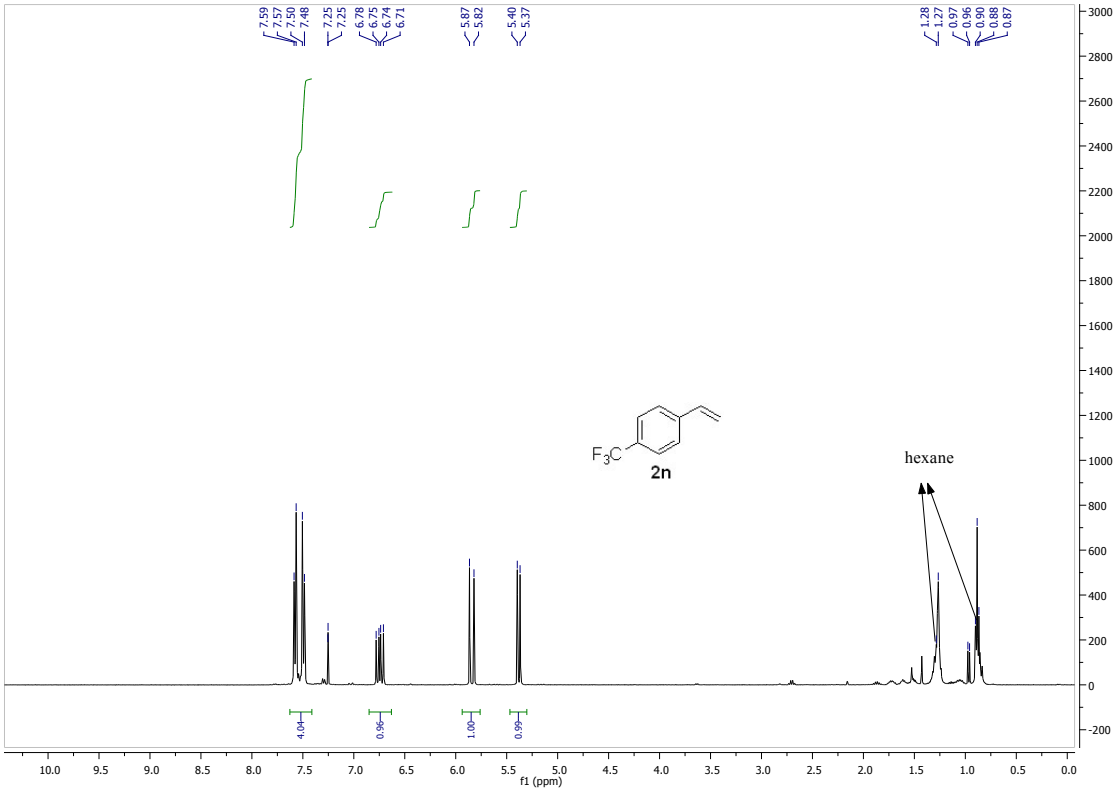

7.35
7.33
7.32
7.30
7.29
7.25
7.23
7.21
7.20
6.45
6.42
6.42
5.83
5.81
5.80
5.80
5.79
5.78
5.77
5.75
1.91
1.91
1.89
1.89
trans alkene
2g
5.82
1.00
1.00
3.25
f1 (ppm)

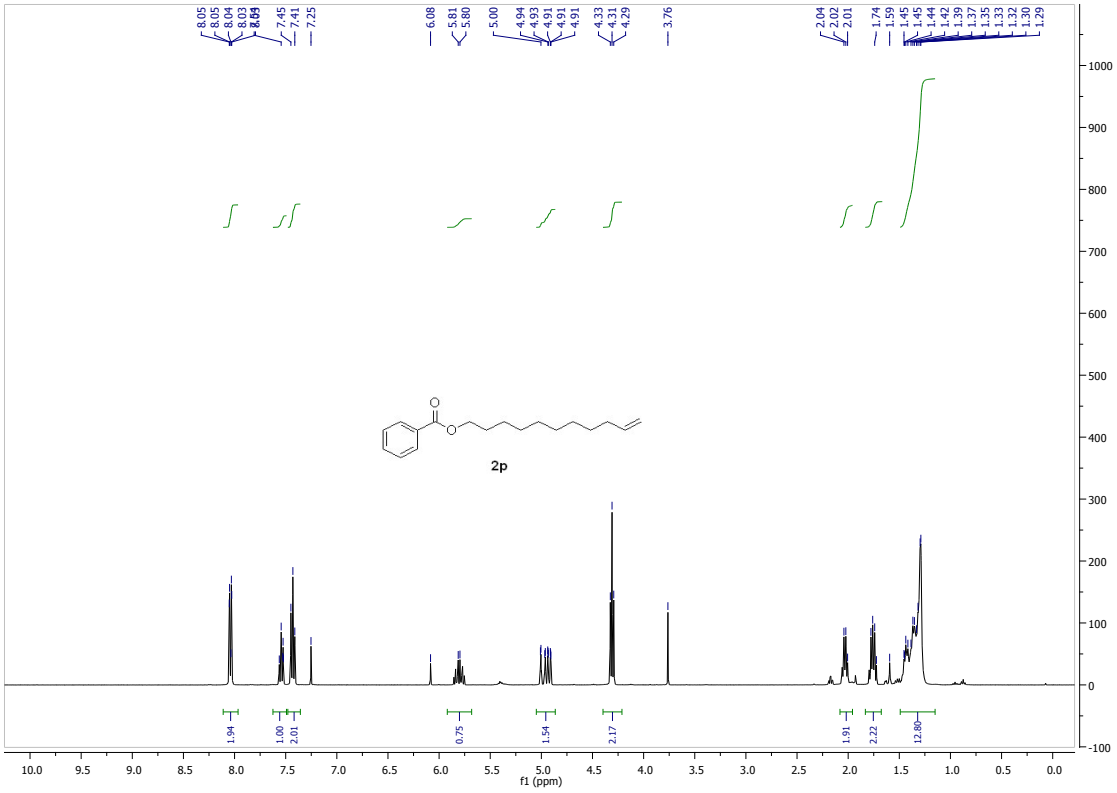

7.37
7.35
7.33
7.28
7.26
7.26
7.22
7.20
6.59
6.56
5.90
5.89
5.87
5.86
5.84
4.45
4.43
1.66
OH
2h
trans alkene
2.09
3.12
1.00
1.00
2.11
1.36
f1 (ppm)

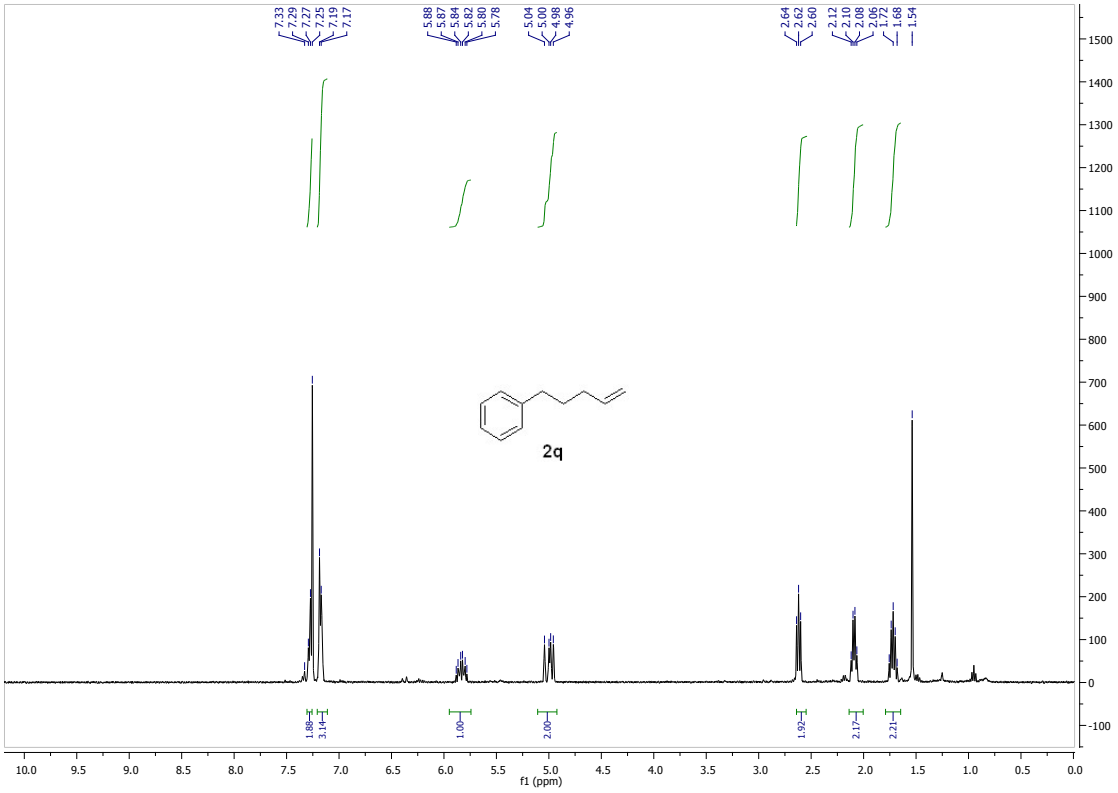

7.35
7.34
7.33
7.30
7.29
7.28
7.27
7.26
5.62
5.60
5.59
4.51
4.08
4.07
2.05
2.03
2.02
2.00
1.55
1.38
1.34
0.91
0.89
0.88
2i
unreacted alkyne
5.47
2.00
2.25
2.19
2.19
2.46
3.46
f1 (ppm)

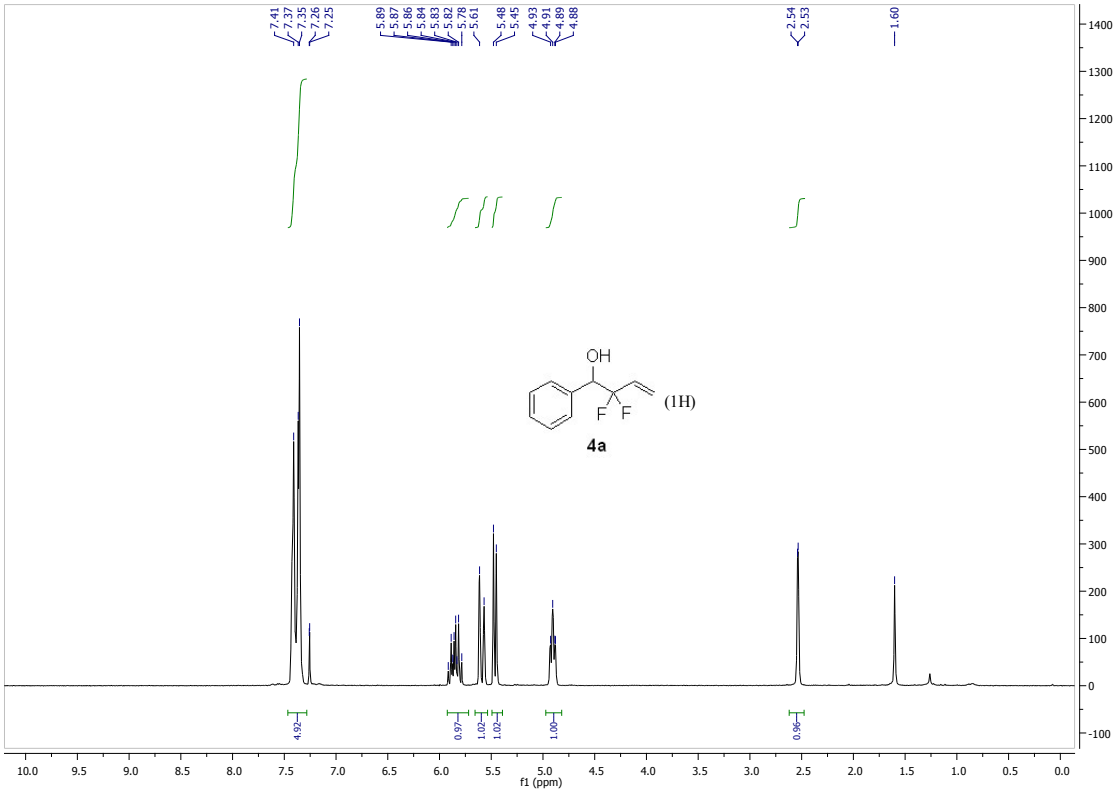

7.36
7.33
7.25
6.87
6.85
6.70
6.67
6.65
6.62
5.63
5.58
5.13
5.11
3.81
1.54
MeO
2l
1.87
1.87
0.96
1.01
1.00
3.08
f1 (ppm)

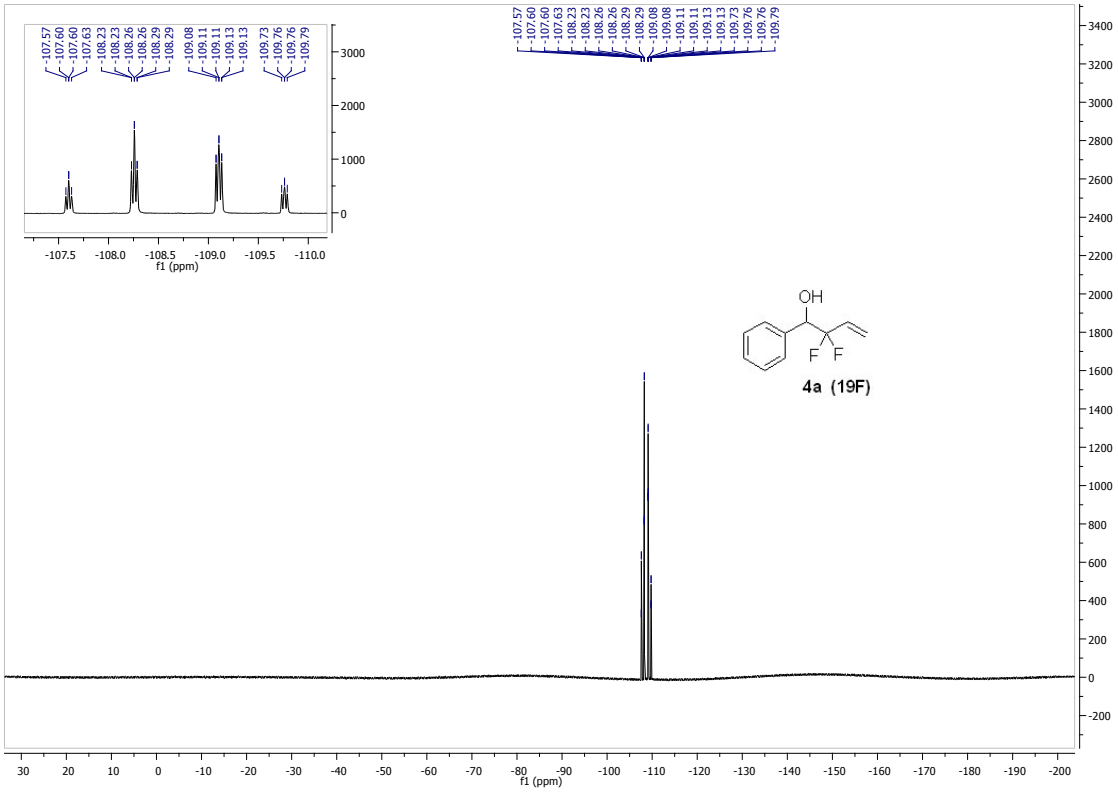

7.49
7.48
7.47
7.14
6.98
6.96
6.94
6.91
5.66
5.62
5.30
5.28
2.35
1.53
1.26
0.89
0.88
0.87
0.86
0.85
0.84
2m
hexane
0.98
3.22
1.07
1.00
1.00
3.28
f1 (ppm)

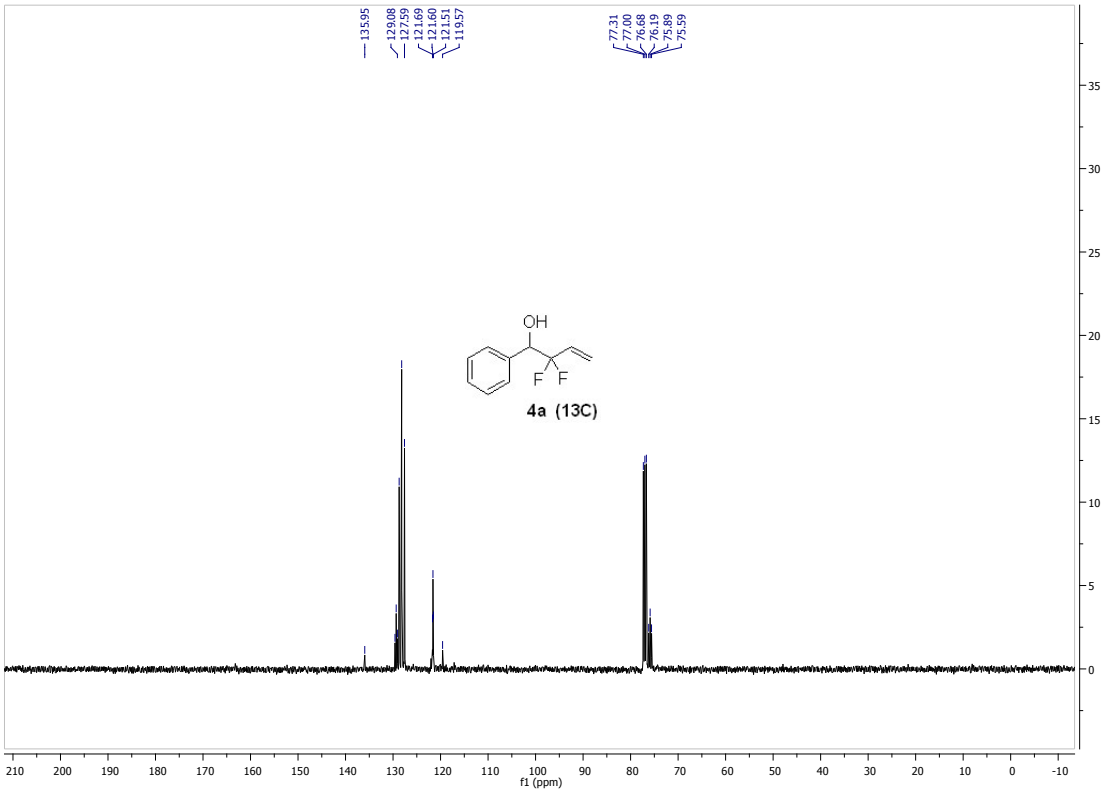

7.59
7.57
7.50
7.48
7.25
7.25
6.78
6.75
6.74
6.71
5.87
5.82
5.40
5.37
1.28
1.27
0.97
0.96
0.90
0.88
0.87
F3C
2n
hexane
4.04
0.96
1.00
0.99
f1 (ppm)

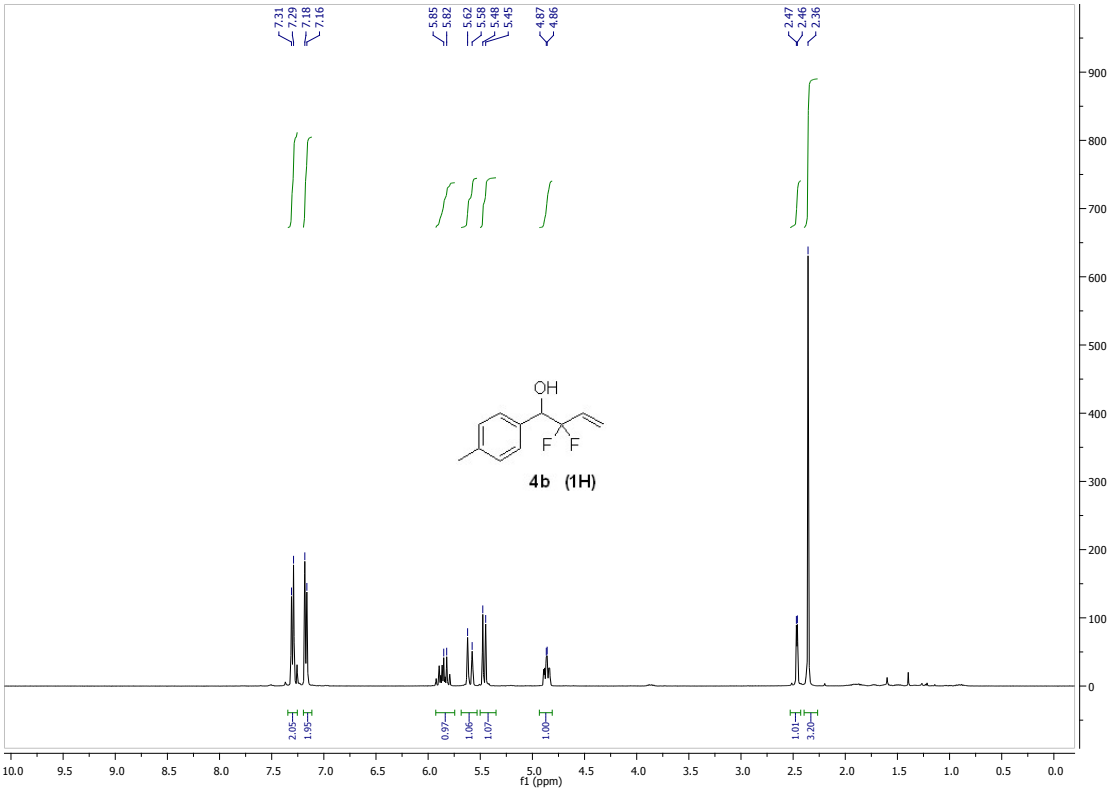

8.05
8.05
8.04
8.04
8.03
8.03
7.45
7.41
7.25
6.08
5.81
5.80
5.00
4.94
4.93
4.91
4.91
4.91
4.33
4.31
4.29
3.76
2.04
2.02
2.01
1.74
1.59
1.45
1.45
1.44
1.42
1.39
1.39
1.37
1.35
1.33
1.32
1.30
1.29
2p
1.94
1.00
2.01
0.75
1.54
2.17
1.91
2.22
12.80
f1 (ppm)


7.33
7.29
7.27
7.25
7.19
7.17
5.88
5.87
5.84
5.82
5.80
5.78
5.04
5.00
4.98
4.96
2.64
2.62
2.60
2.12
2.10
2.08
2.06
1.72
1.68
1.54
2q
1.88
3.14
1.00
2.00
1.92
2.17
2.21
f1 (ppm)

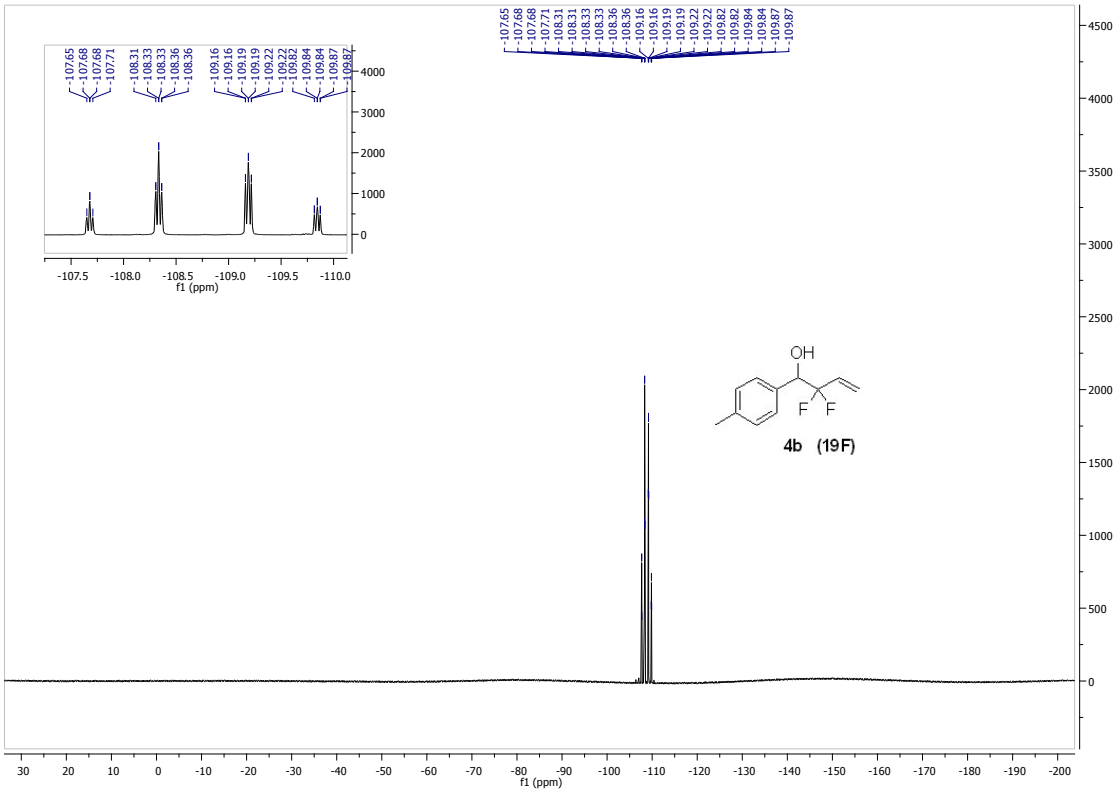

7.41
7.37
7.35
7.26
7.25
5.89
5.87
5.86
5.84
5.83
5.82
5.78
5.61
5.48
5.45
4.93
4.91
4.89
4.88
2.54
2.53
1.60
OH
F F
(1H)
4a
4.92
0.97
1.02
1.02
1.00
0.96
f1 (ppm)


OH
F F
4a (19F)
f1 (ppm)

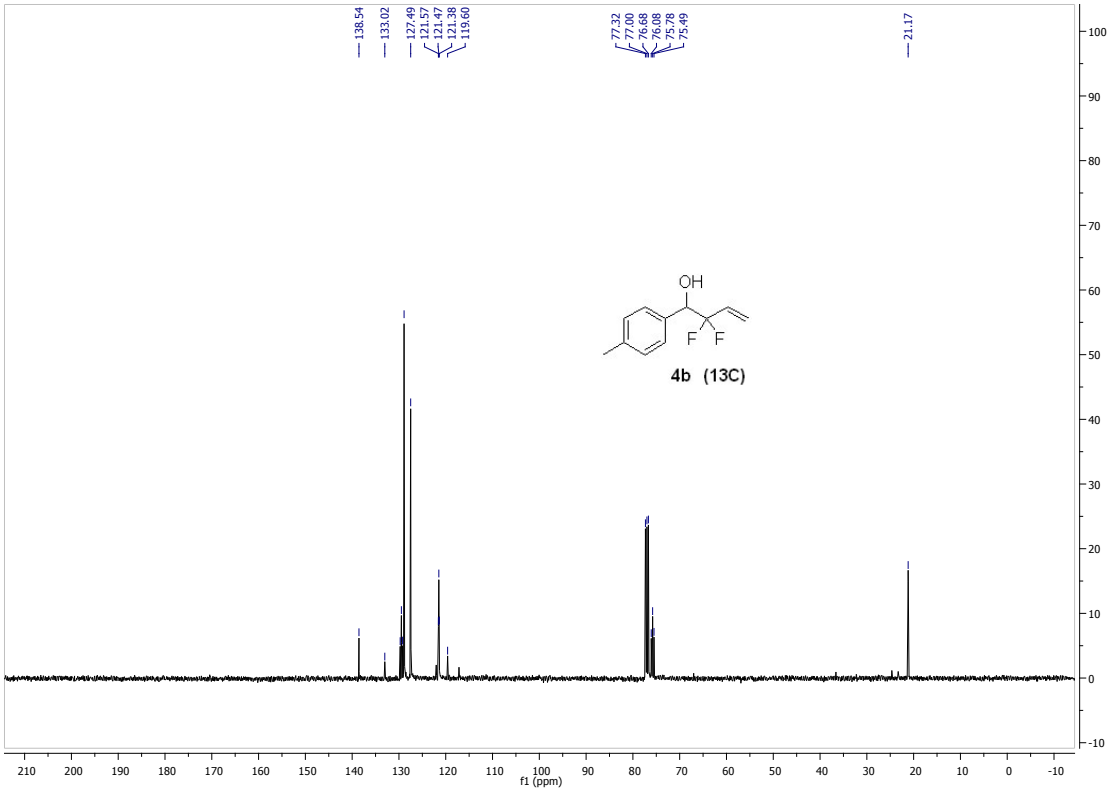

OH
F F
4a (13C)
f1 (ppm)

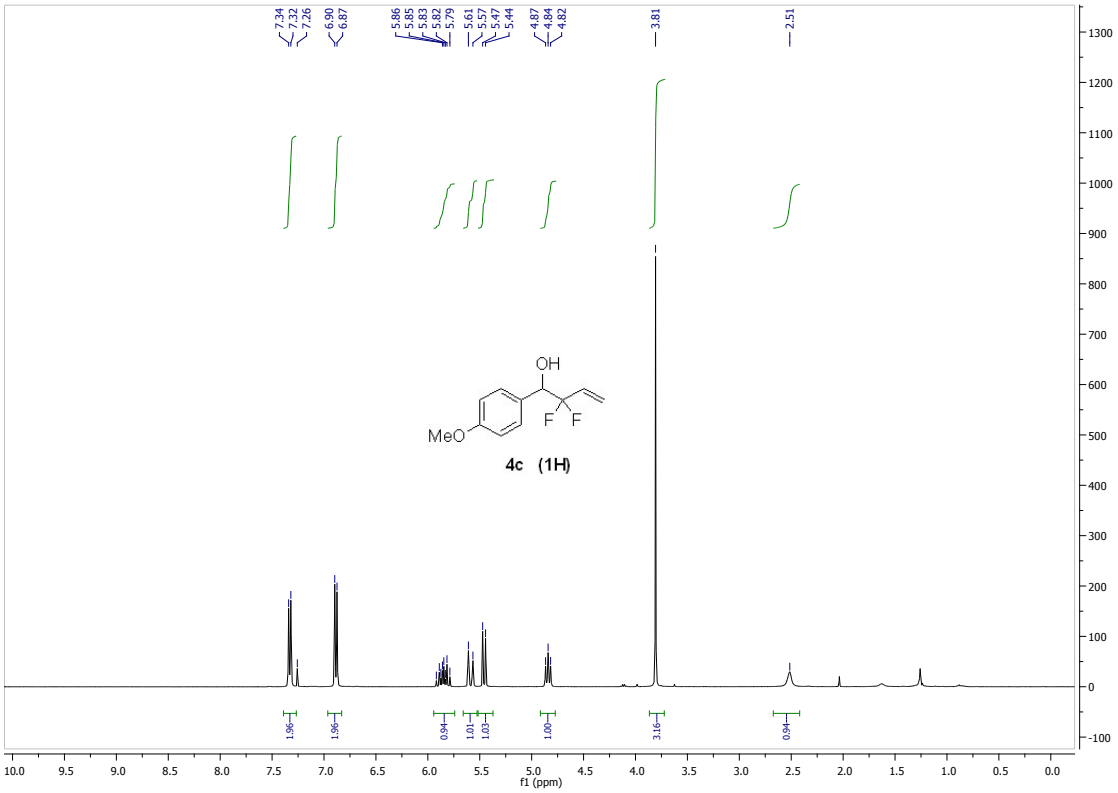

7.31
7.29
7.18
7.16
5.85
5.82
5.62
5.58
5.48
5.45
4.87
4.86
2.47
2.46
2.36
OH
F F
2.05
1.95
0.97
1.08
1.07
1.00
1.01
3.20
f1 (ppm)

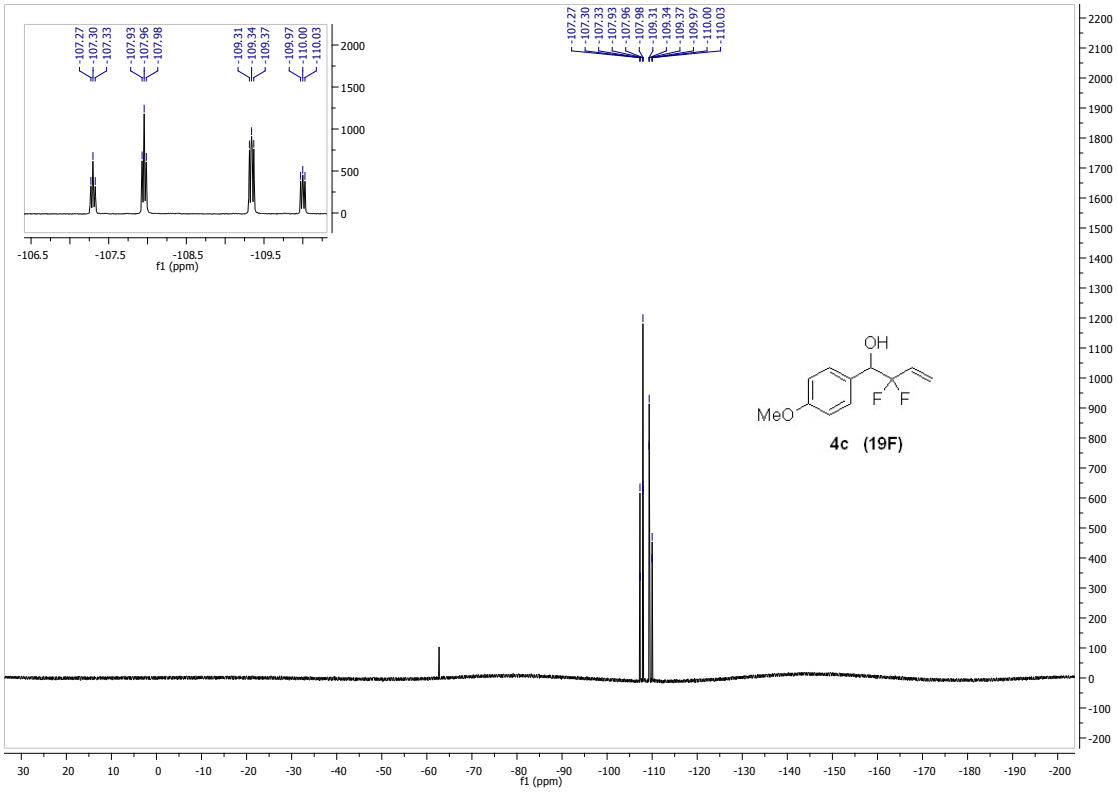
**4b (1H)**

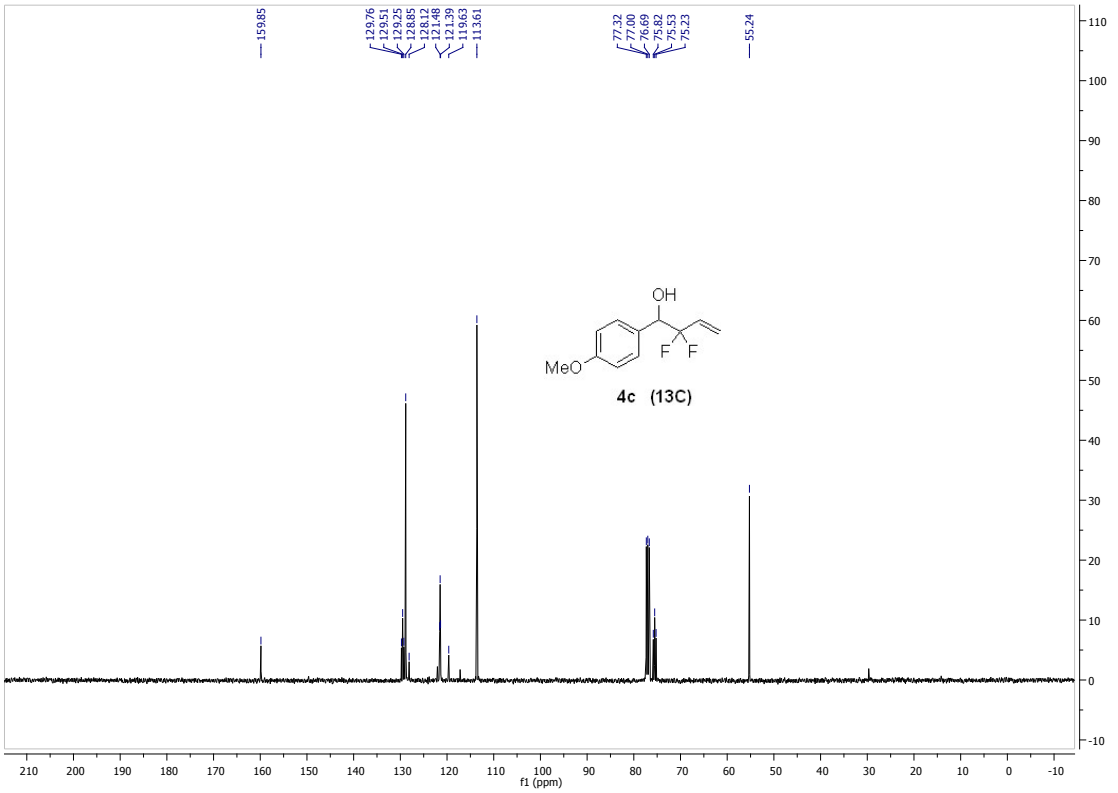

-107.65
-107.68
-107.68
-107.71
-108.31
-108.33
-108.33
-108.36
-108.36
-109.16
-109.16
-109.19
-109.19
-109.22
-109.22
-109.82
-109.84
-109.84
-109.87
-109.87
f1 (ppm)
OH
F F
f1 (ppm)

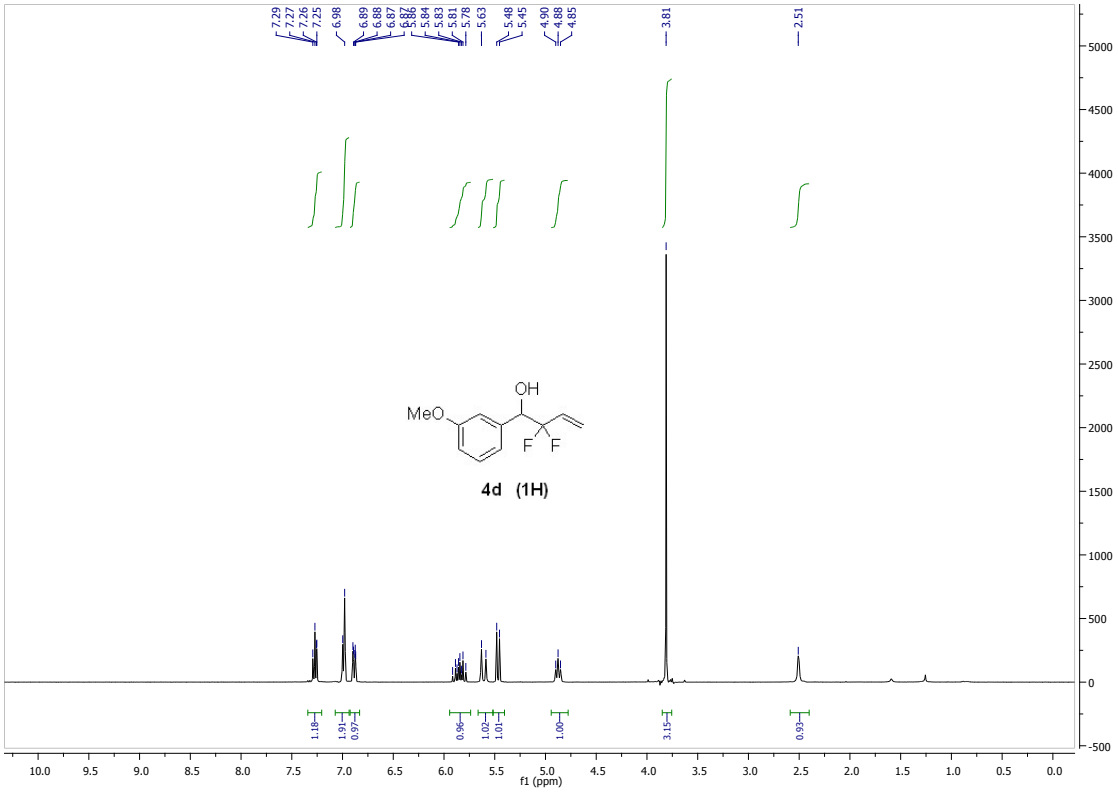
**4b (19F)**

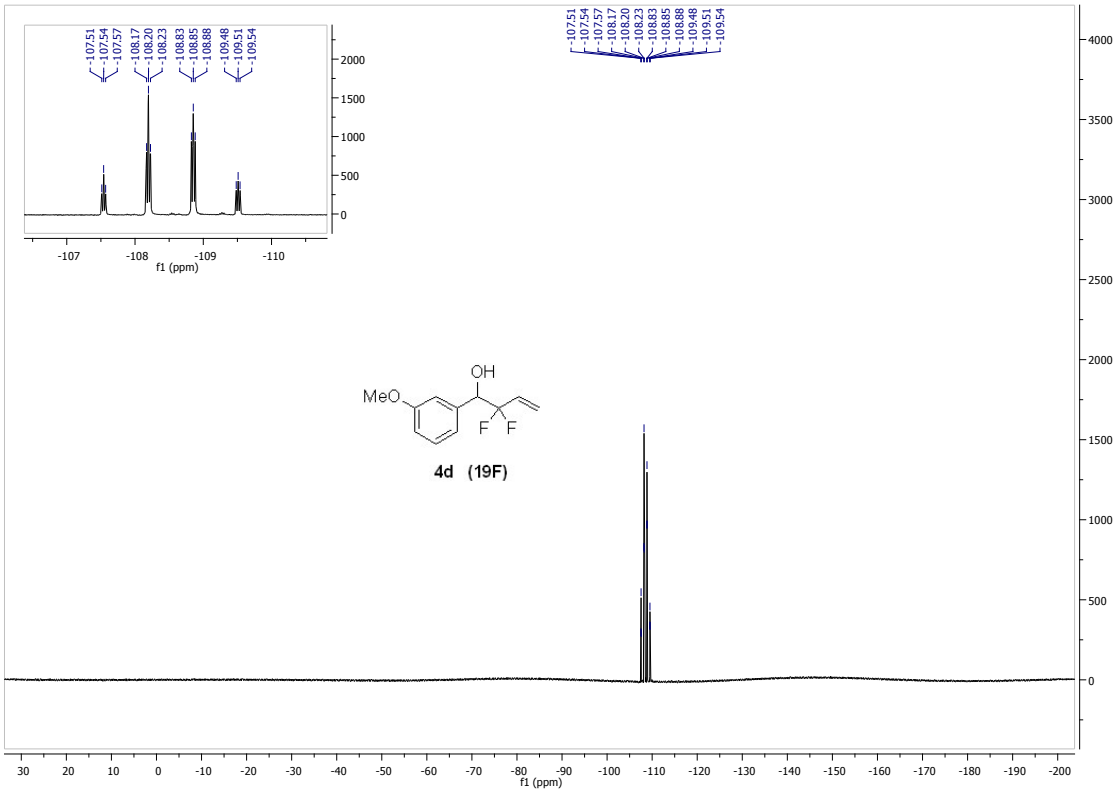

138.54
133.02
127.49
121.57
121.47
121.38
119.60
77.32
77.00
76.68
76.08
75.78
75.49
21.17
OH
F F
4b (13C)
f1 (ppm)

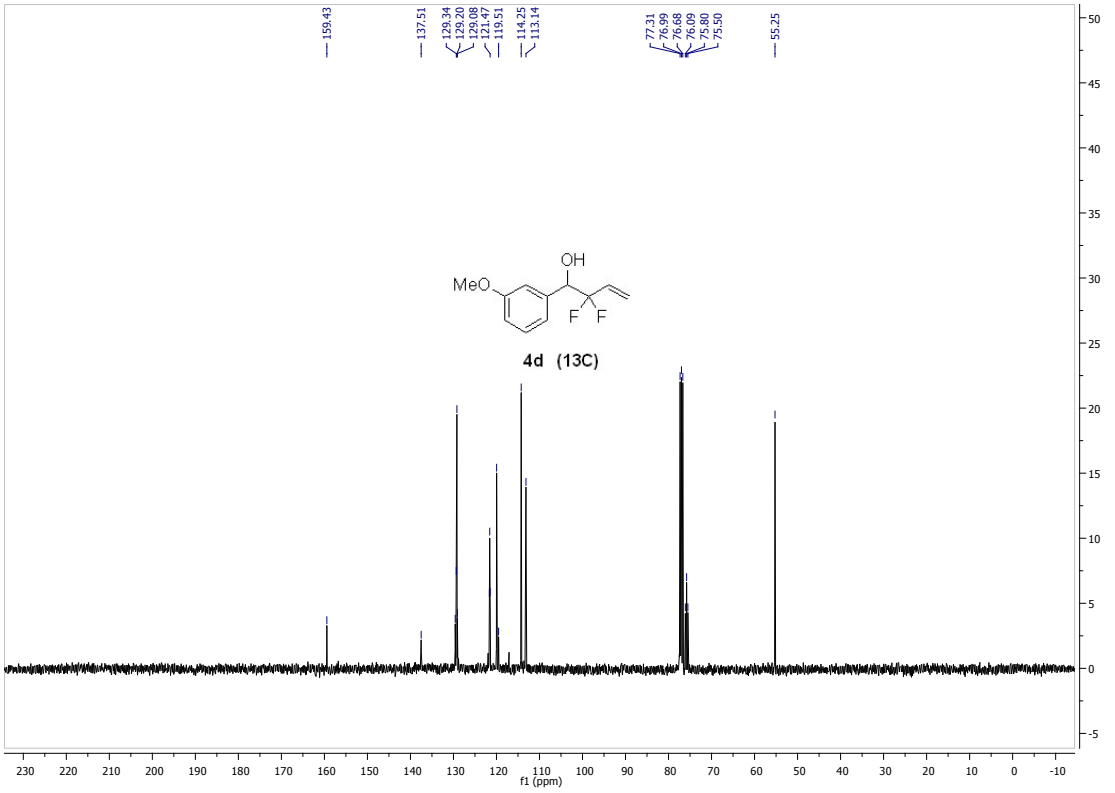

7.34
7.32
7.26
6.90
6.87
5.86
5.85
5.83
5.82
5.79
5.61
5.57
5.47
5.44
4.87
4.84
4.82
3.81
2.51
OH
F F
MeO
4c (1H)
1.96
1.96
0.94
1.01
1.03
1.00
3.16
0.94
f1 (ppm)

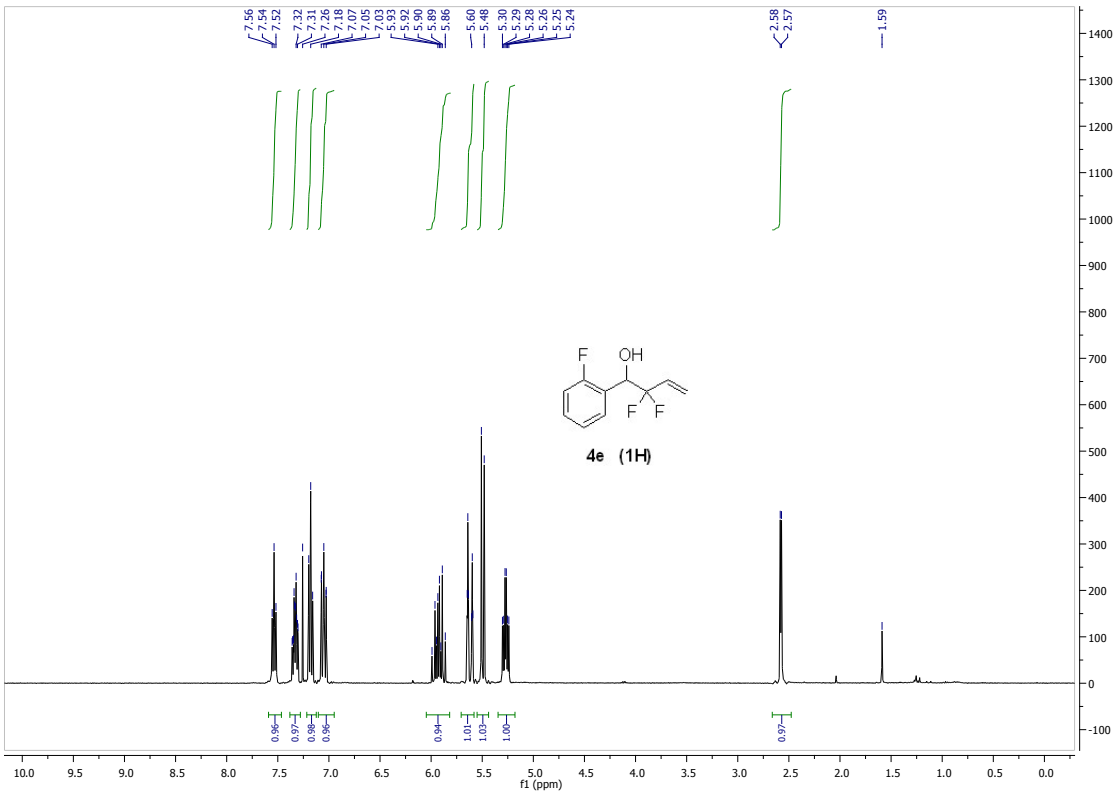

-107.27
-107.30
-107.33
-107.93
-107.96
-107.98
-109.31
-109.34
-109.37
-109.97
-110.00
-110.03
f1 (ppm)
OH
F F
MeO
4c (19F)

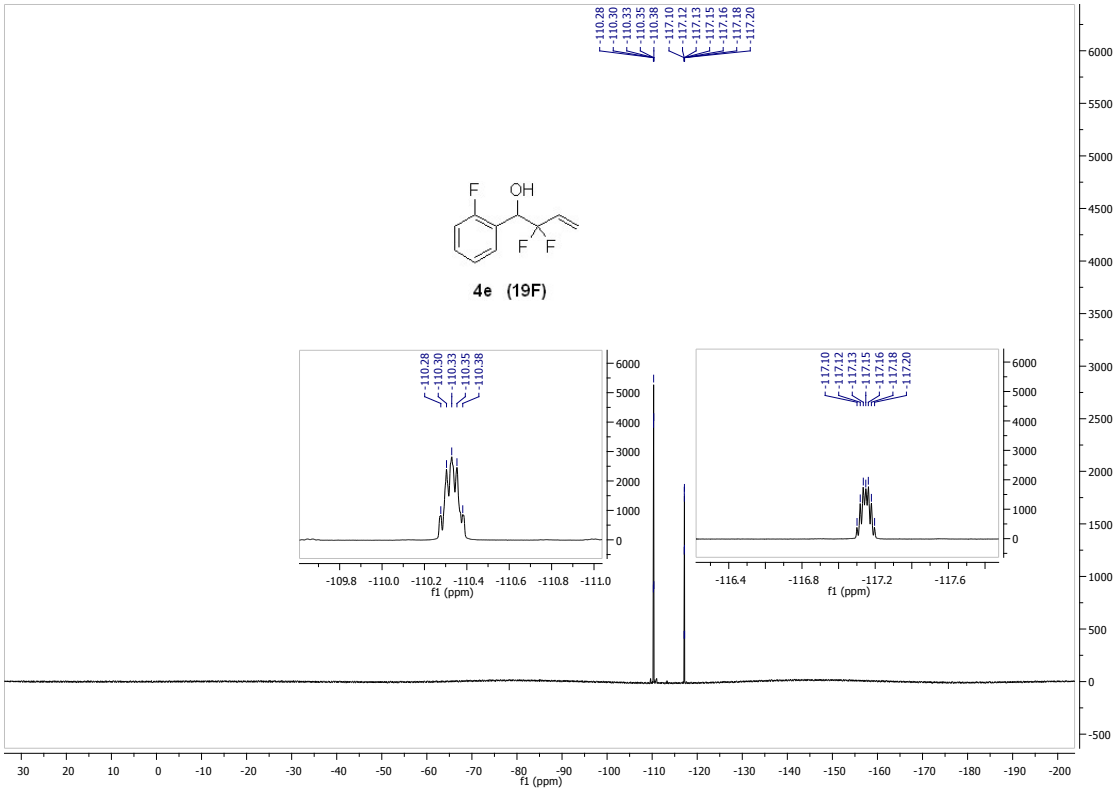

159.85
129.76
129.51
129.25
128.85
128.12
121.48
121.39
119.63
113.61
77.32
77.00
76.69
75.82
75.53
75.23
55.24
f1 (ppm)
OH
F F
MeO
4c (13C)

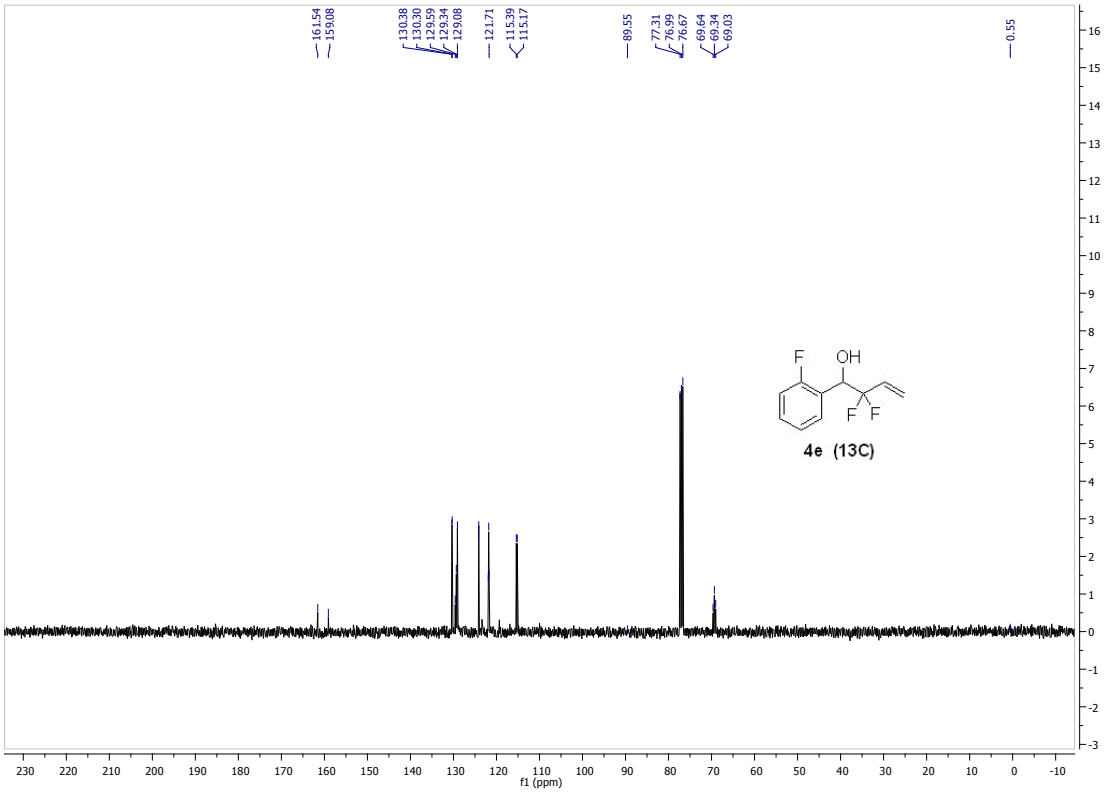

7.29
7.27
7.26
7.25
6.98
6.89
6.88
6.87
5.94
5.83
5.81
5.78
5.63
5.48
5.45
4.90
4.88
4.85
3.81
2.51
OH
MeO
F F
4d (1H)
f1 (ppm)

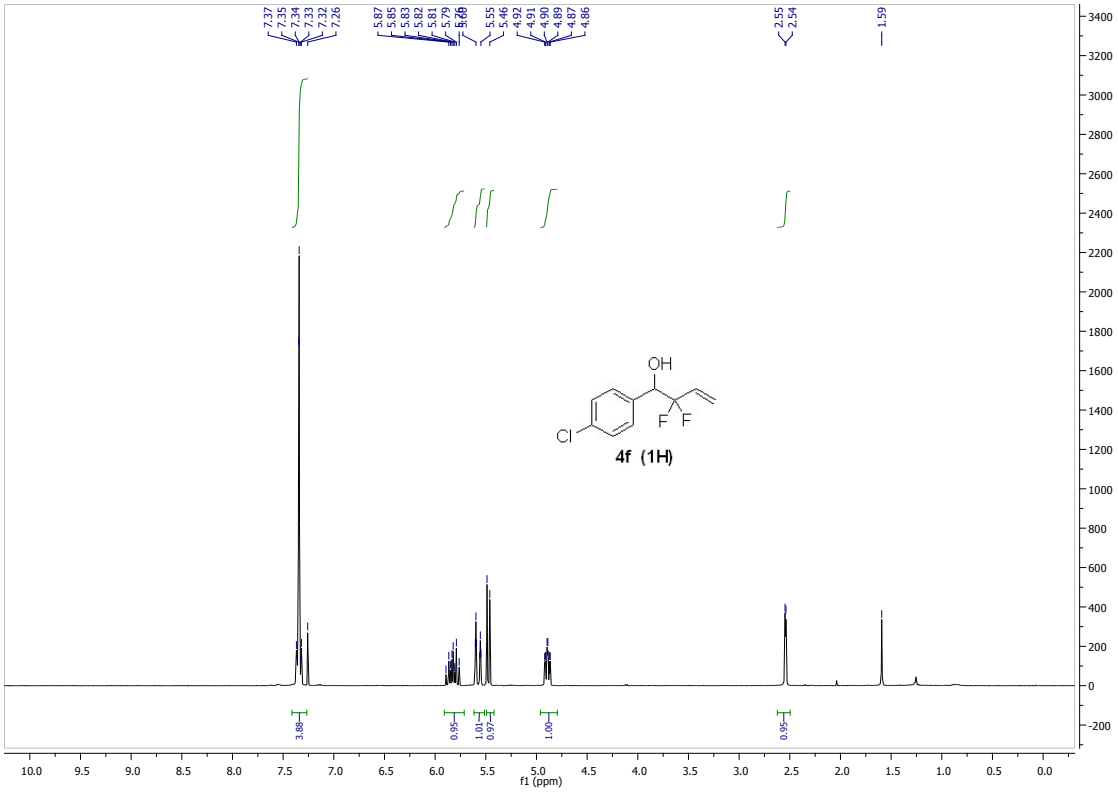

-107.51
-107.54
-107.57
-108.17
-108.20
-108.23
-108.83
-108.85
-108.88
-109.48
-109.51
-109.54
OH
MeO
F F
4d (19F)
f1 (ppm)

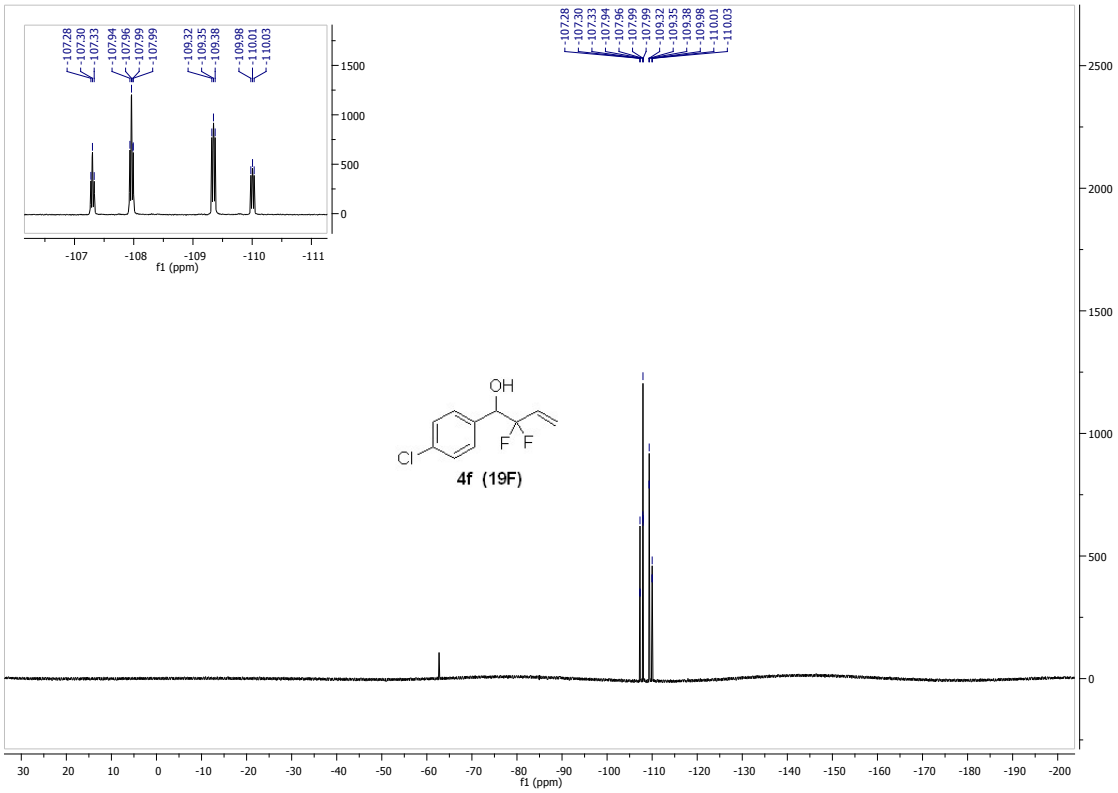

159.43
137.51
129.34
129.20
129.08
121.47
119.51
114.25
113.14
77.31
76.99
76.68
76.09
75.80
75.50
55.25
OH
MeO
F F
4d (13C)
f1 (ppm)

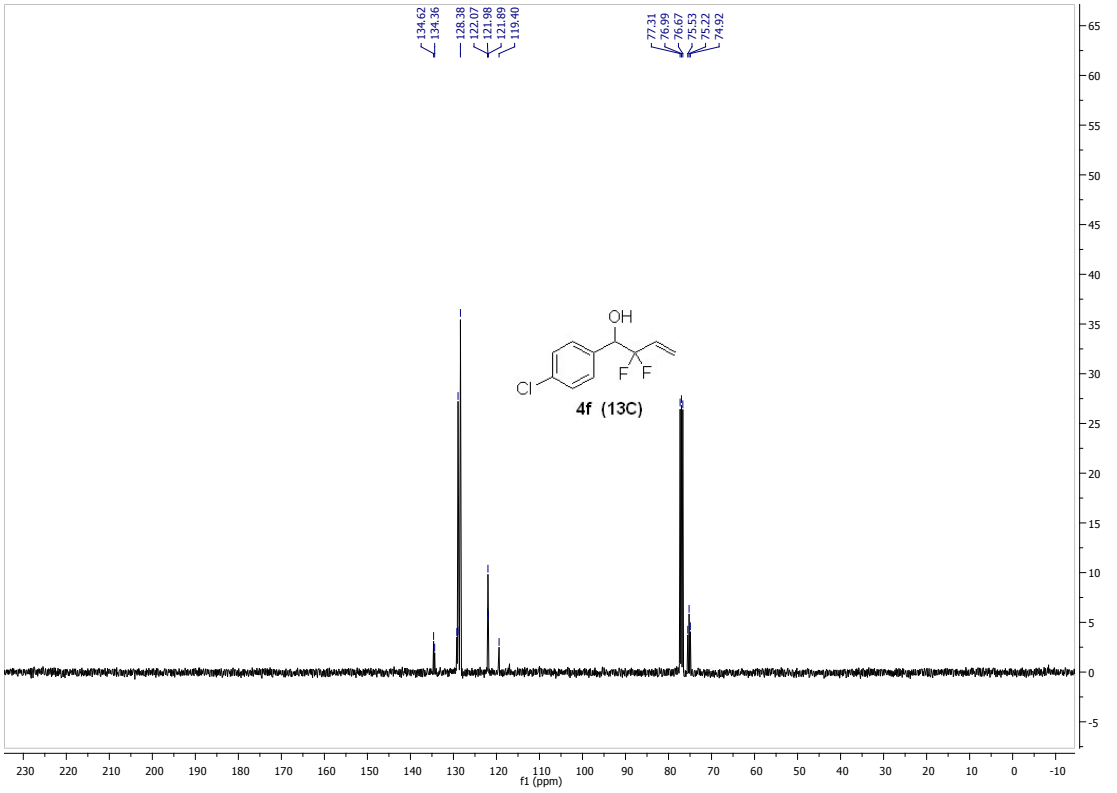

7.56
7.54
7.52
7.32
7.31
7.26
7.18
7.07
7.05
7.03
5.93
5.92
5.90
5.89
5.86
5.60
5.48
5.30
5.29
5.28
5.26
5.25
5.24
2.58
2.57
1.59
F OH
F F
4e (1H)
0.96
0.97
0.98
0.96
0.94
1.01
1.03
1.00
0.97
f1 (ppm)

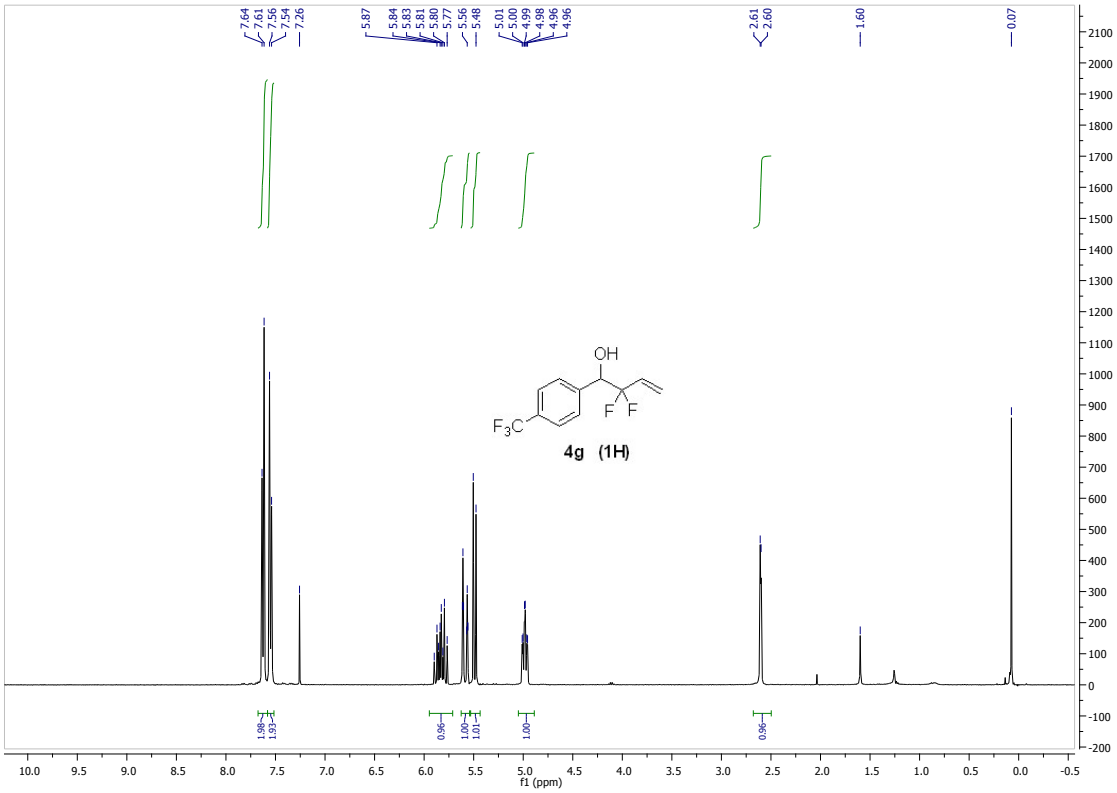

-110.28
-110.30
-110.33
-110.35
-110.38
-117.10
-117.12
-117.13
-117.15
-117.16
-117.18
-117.20
F
OH
F
F
4e (19F)
f1 (ppm)

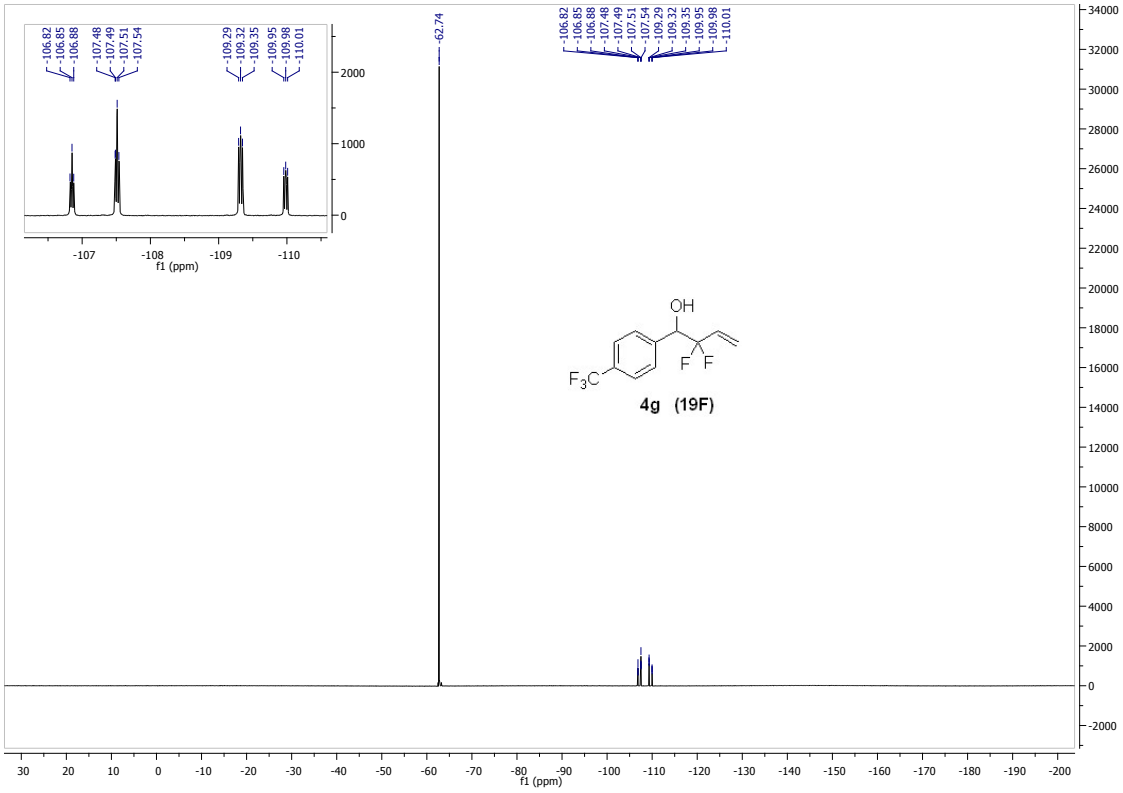

161.54
159.08
130.38
130.30
129.59
129.34
129.08
121.71
115.39
115.17
89.55
77.31
76.99
76.67
69.64
69.34
69.03
0.55
F
OH
F
F
4e (13C)
f1 (ppm)

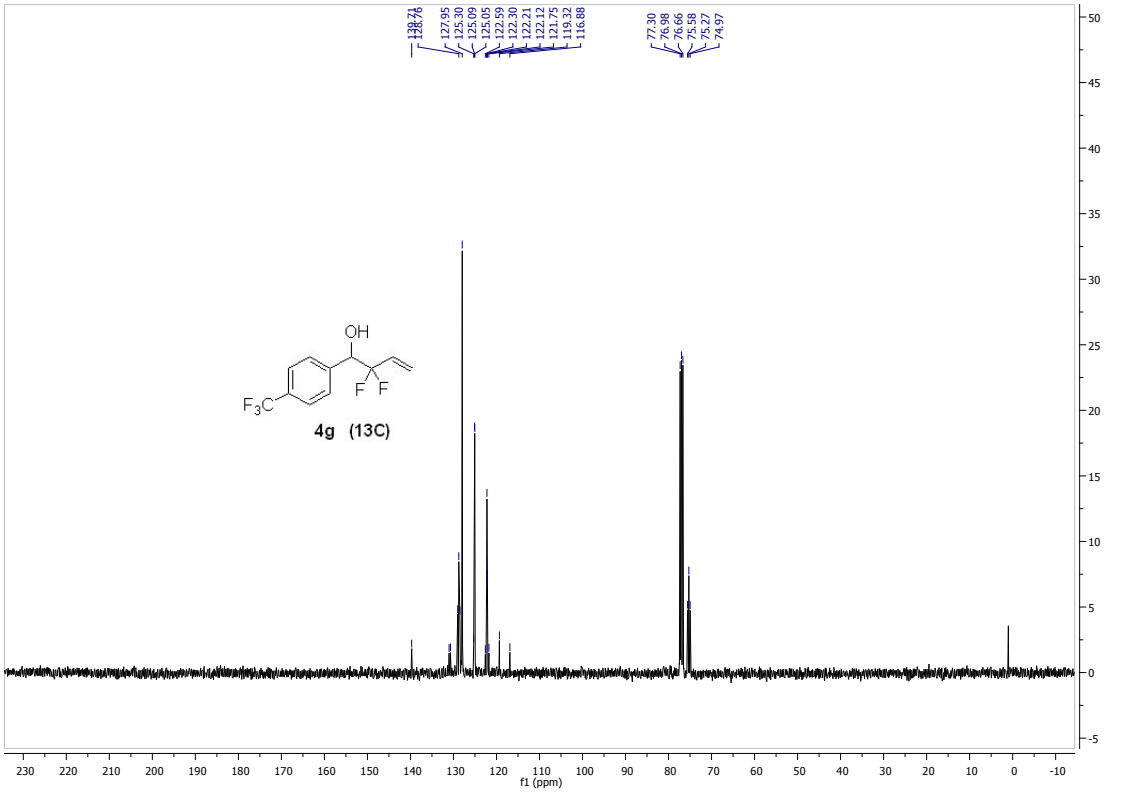

7.37
7.35
7.34
7.33
7.32
7.26
5.87
5.85
5.83
5.82
5.81
5.79
5.46
4.92
4.91
4.90
4.89
4.87
4.86
2.55
2.54
1.59
OH
Cl
F F
4f (1H)
f1 (ppm)

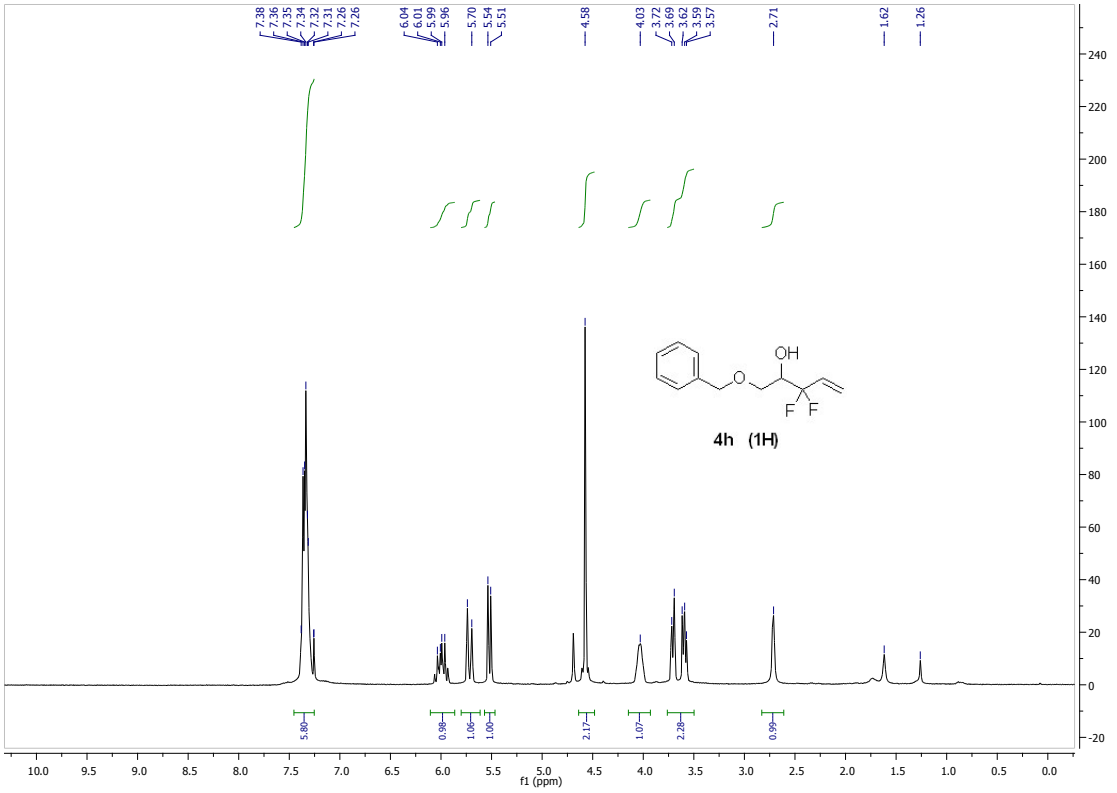

-107.28
-107.30
-107.33
-107.94
-107.96
-107.99
-109.32
-109.35
-109.38
-109.98
-110.01
-110.03
OH
Cl
F F
4f (19F)
f1 (ppm)

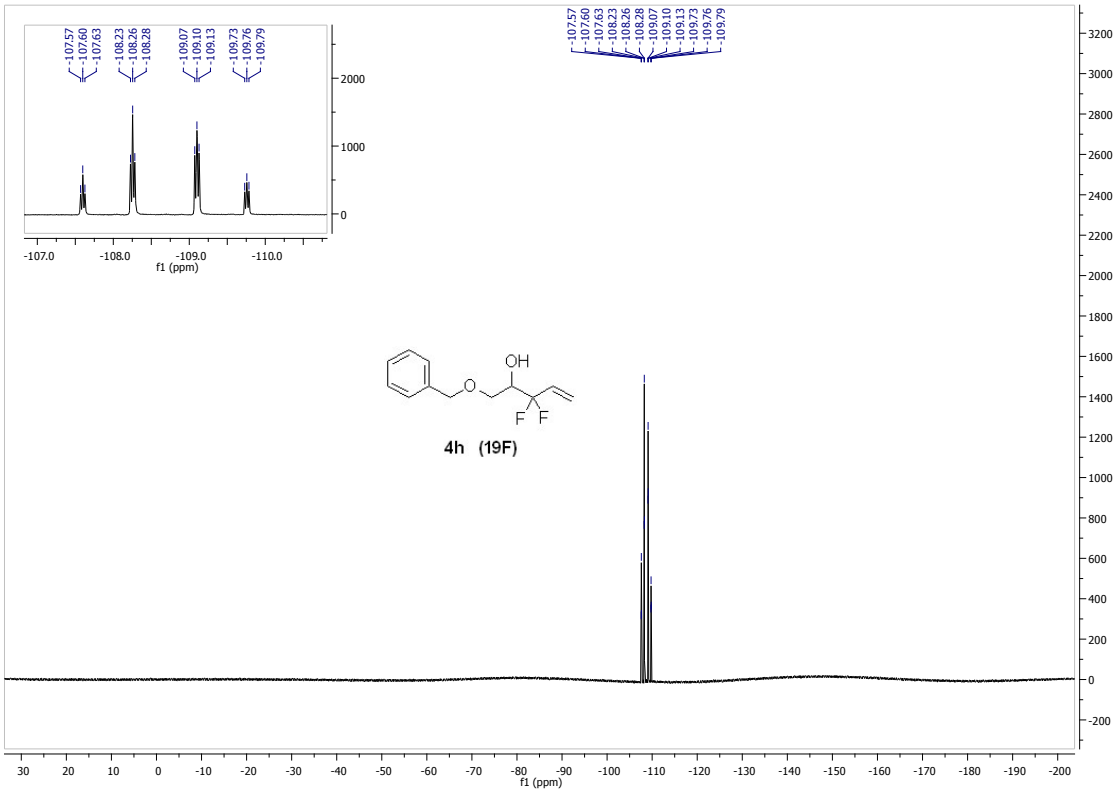

134.62
134.36
128.38
122.07
121.98
121.89
119.40
77.31
76.99
76.67
75.53
75.22
74.92
OH
F F
Cl
4f (13C)
f1 (ppm)

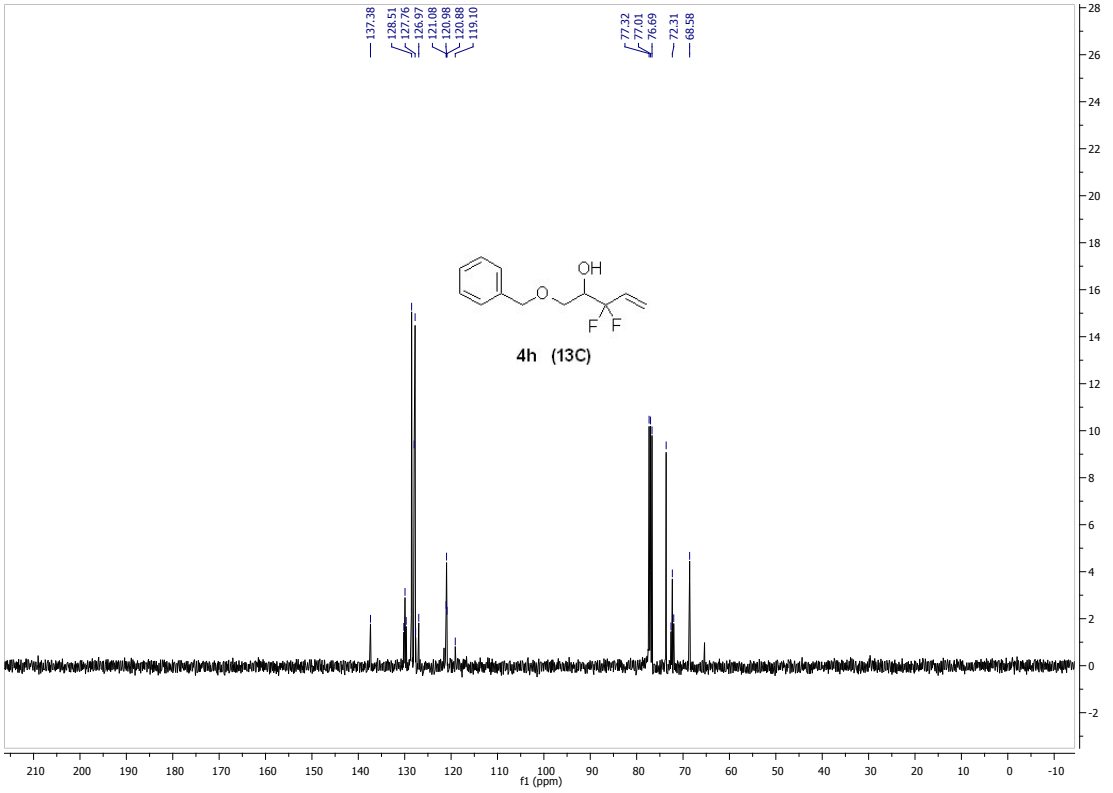

7.64
7.61
7.56
7.54
7.26
5.87
5.84
5.83
5.81
5.80
5.77
5.56
5.48
5.01
5.00
4.99
4.98
4.96
4.96
2.61
2.60
1.60
0.07
OH
F F
F3C
4g (1H)
1.98
1.93
0.96
1.00
1.01
1.00
0.96
f1 (ppm)

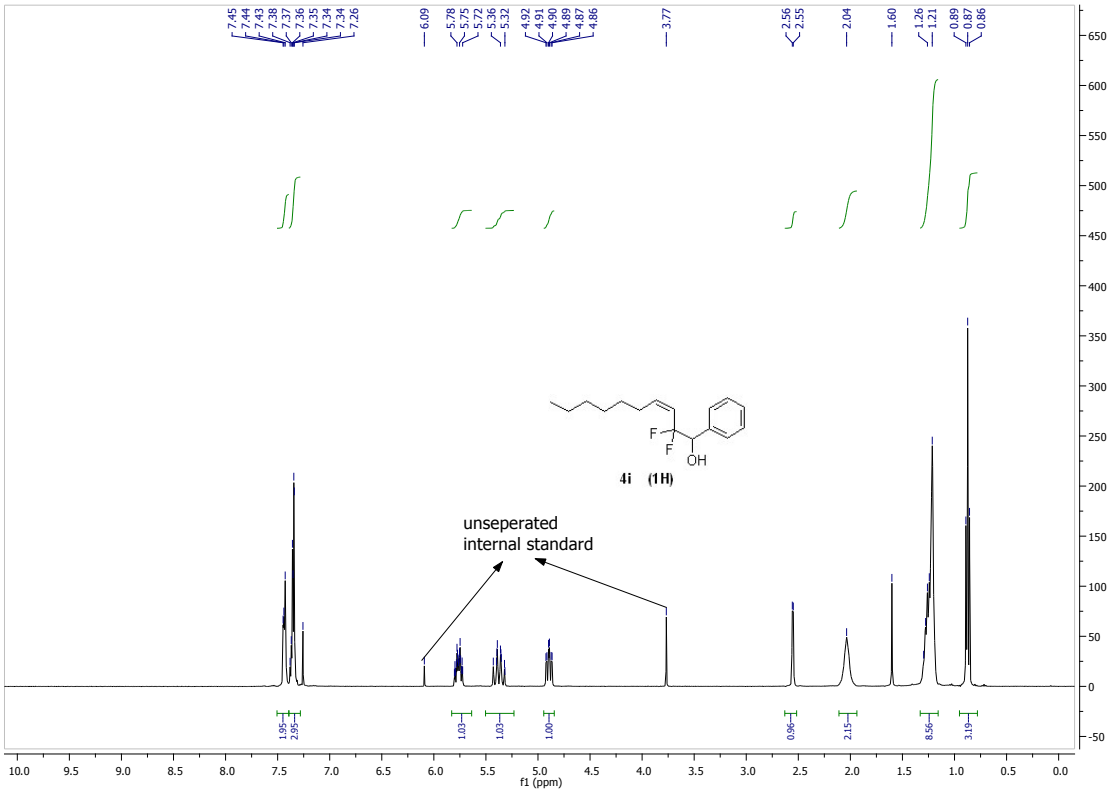

-106.82
-106.85
-106.88
-107.48
-107.49
-107.51
-107.54
-109.29
-109.32
-109.35
-109.95
-109.98
-110.01
-62.74
f1 (ppm)
OH
F
F
F3C
4g (19F)

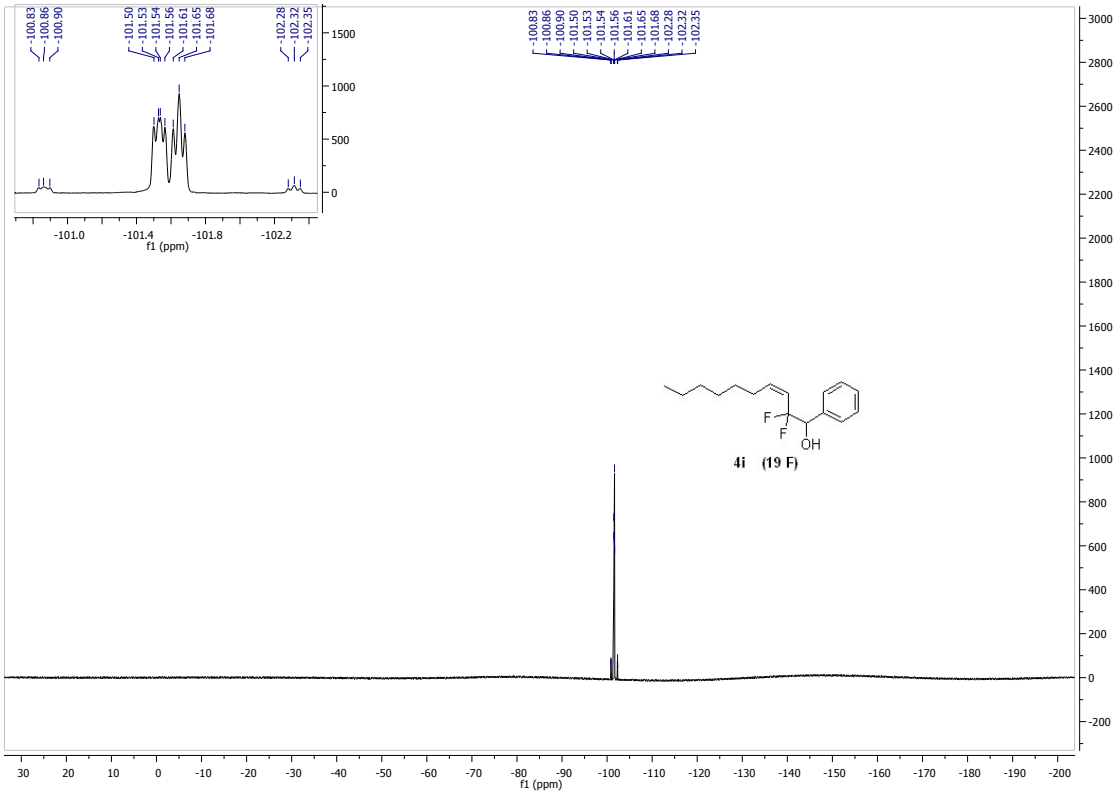

138.71
138.76
127.95
125.30
125.09
125.05
122.59
122.30
122.21
122.12
121.75
119.32
116.88
77.30
76.98
76.66
75.58
75.37
74.97
f1 (ppm)
OH
F
F
F3C
4g (13C)

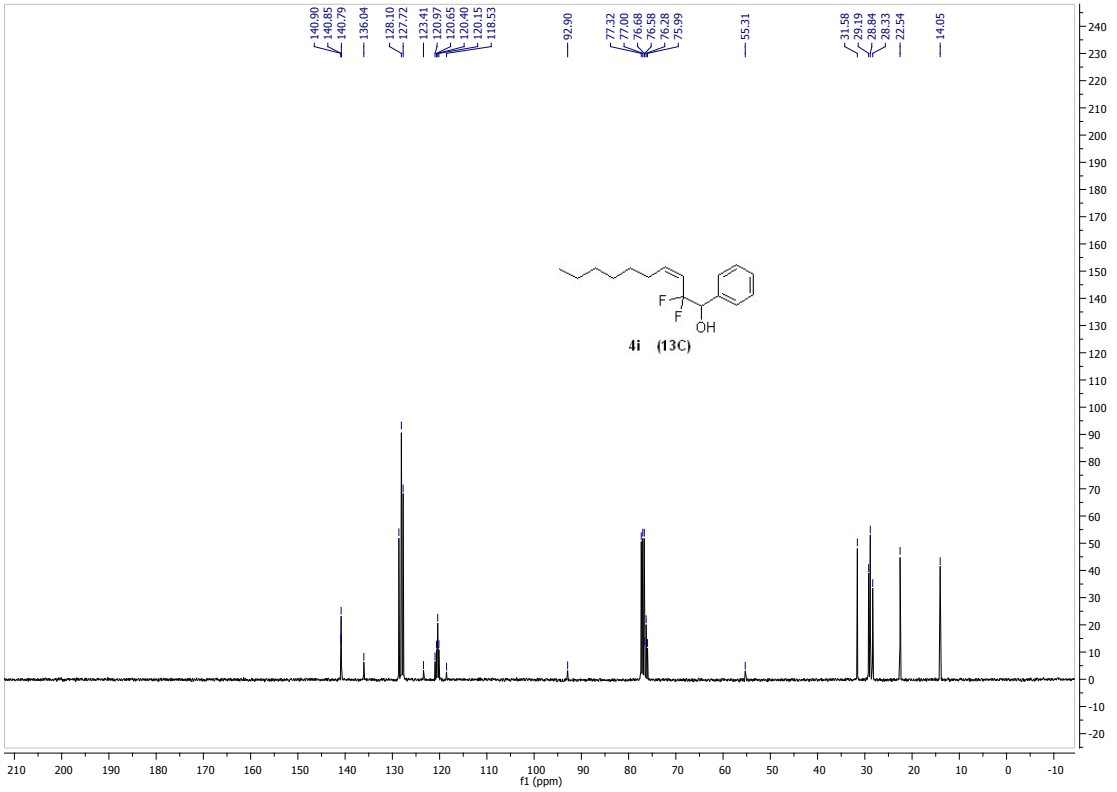

7.38
7.36
7.35
7.34
7.32
7.31
7.26
7.26
6.04
6.01
5.99
5.96
5.70
5.54
5.51
4.58
4.03
3.72
3.69
3.62
3.59
3.57
2.71
1.62
1.26
OH
O
F F
4h (1H)
5.80
0.98
1.06
1.00
2.17
1.07
2.28
0.99
f1 (ppm)

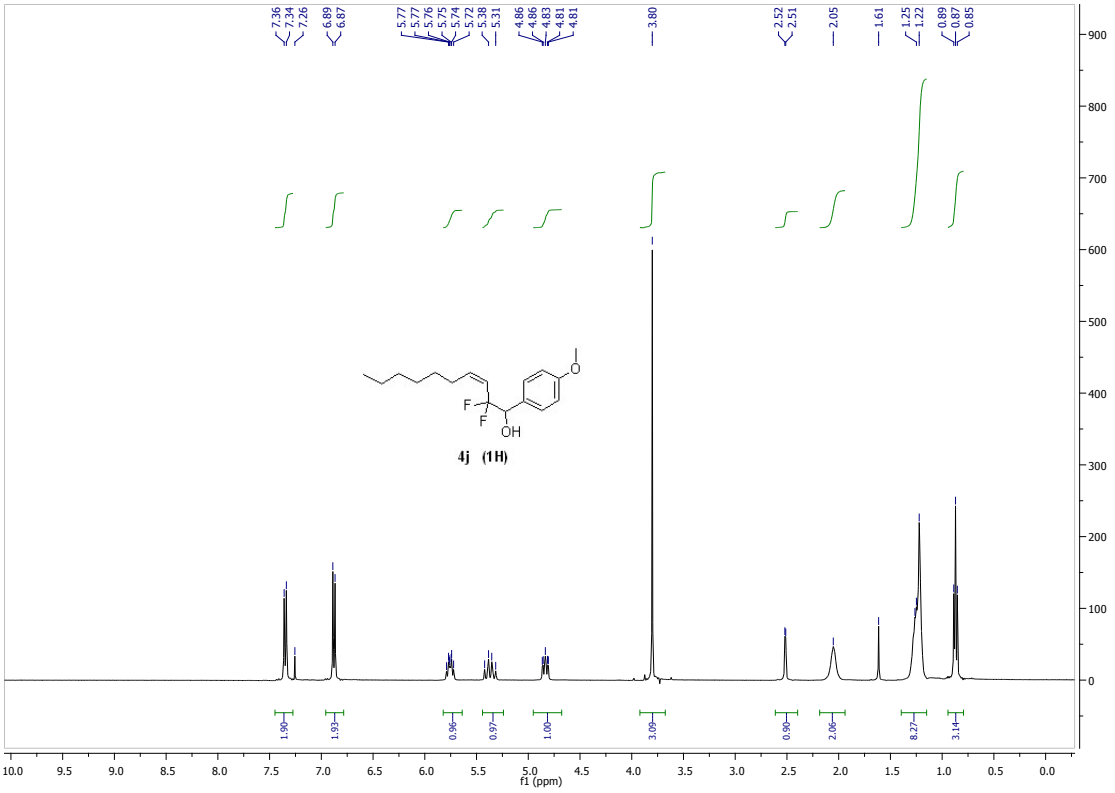

-107.57
-107.60
-107.63
-108.23
-108.26
-108.28
-109.07
-109.10
-109.13
-109.73
-109.76
-109.79
f1 (ppm)
OH
O
F F
4h (19F)
f1 (ppm)

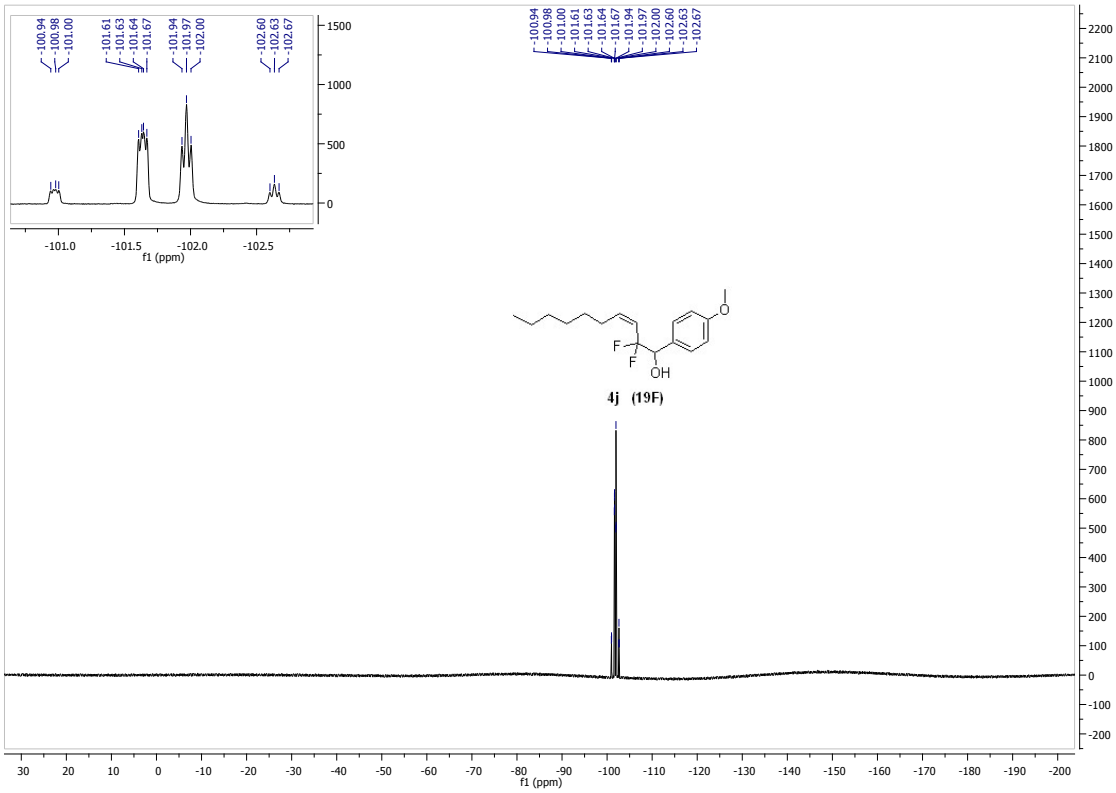

137.38
128.51
127.76
126.97
121.08
120.98
120.88
119.10
77.32
77.01
76.69
72.31
68.58
OH
O
F F
4h (13C)
f1 (ppm)

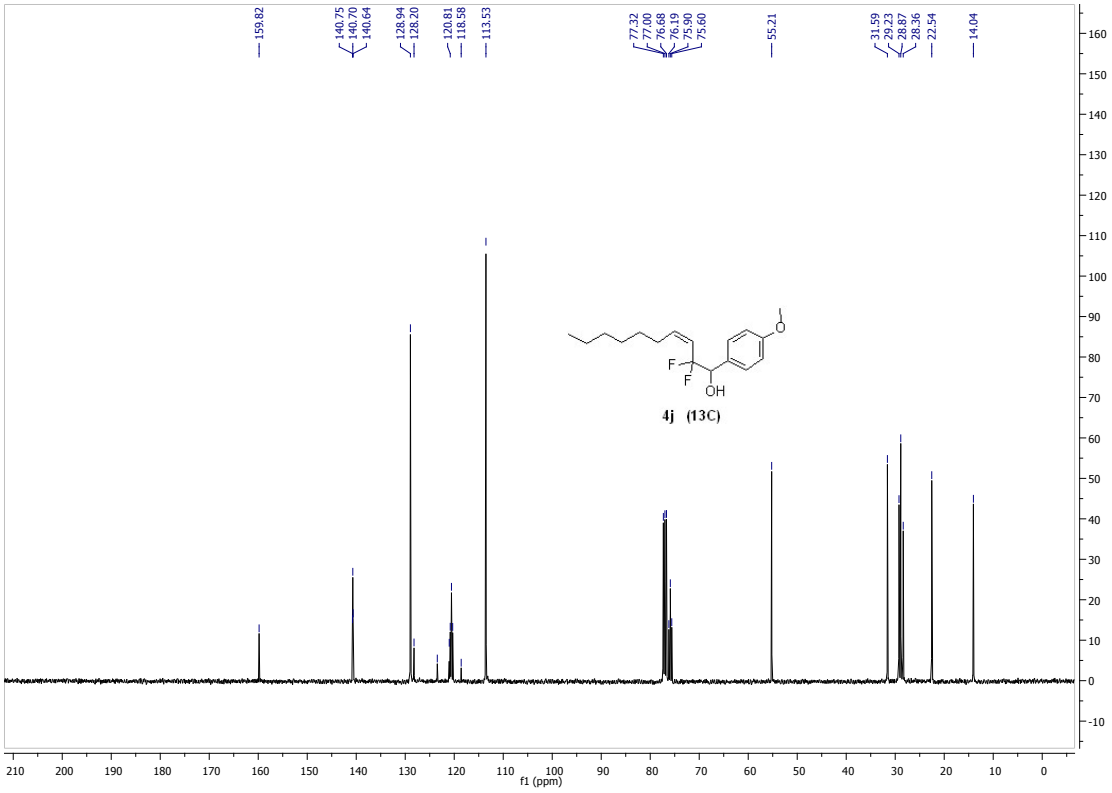

7.45
7.44
7.43
7.38
7.37
7.36
7.35
7.34
7.26
6.09
5.78
5.75
5.72
5.36
5.32
4.92
4.91
4.90
4.89
4.87
4.86
3.77
2.56
2.55
2.04
1.60
1.26
1.21
0.89
0.87
0.86
F
F
OH
4i (1H)
unseperated
internal standard
1.95
2.95
1.03
1.03
1.00
0.96
2.15
8.56
3.19
f1 (ppm)

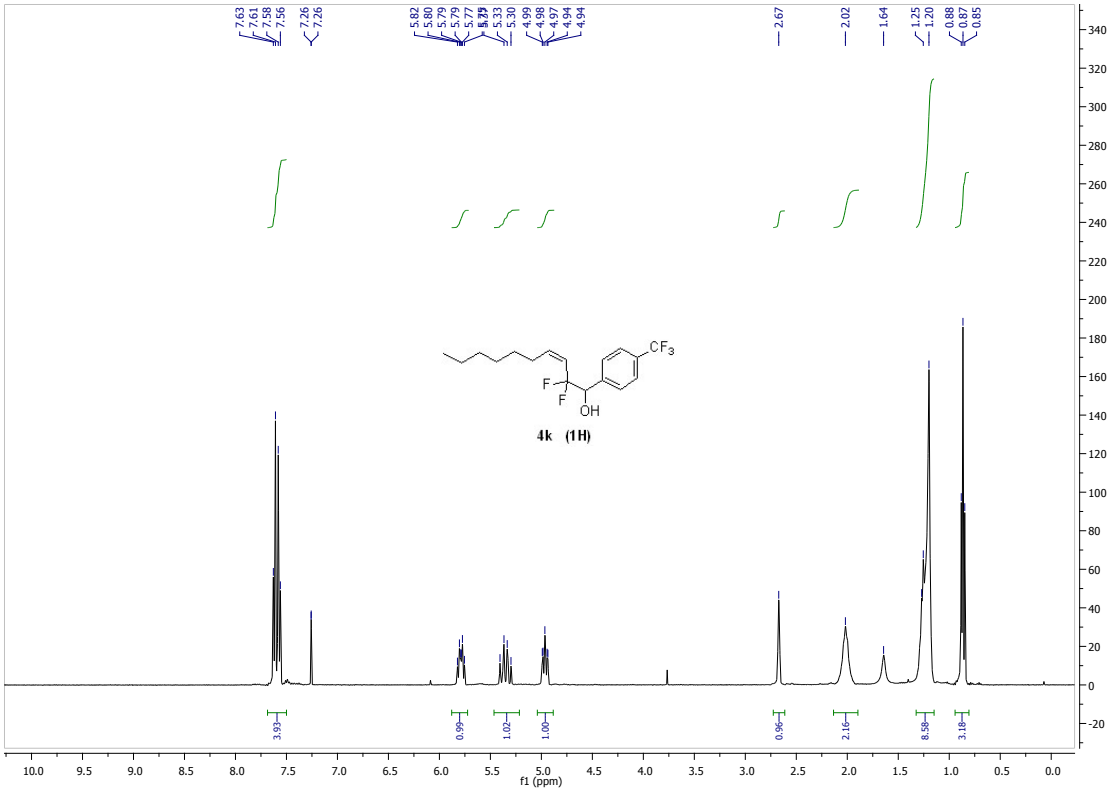

-100.83
-100.86
-100.90
-101.50
-101.53
-101.54
-101.56
-101.61
-101.65
-101.68
-102.28
-102.32
-102.35
f1 (ppm)
F
F
OH
4i (19 F)

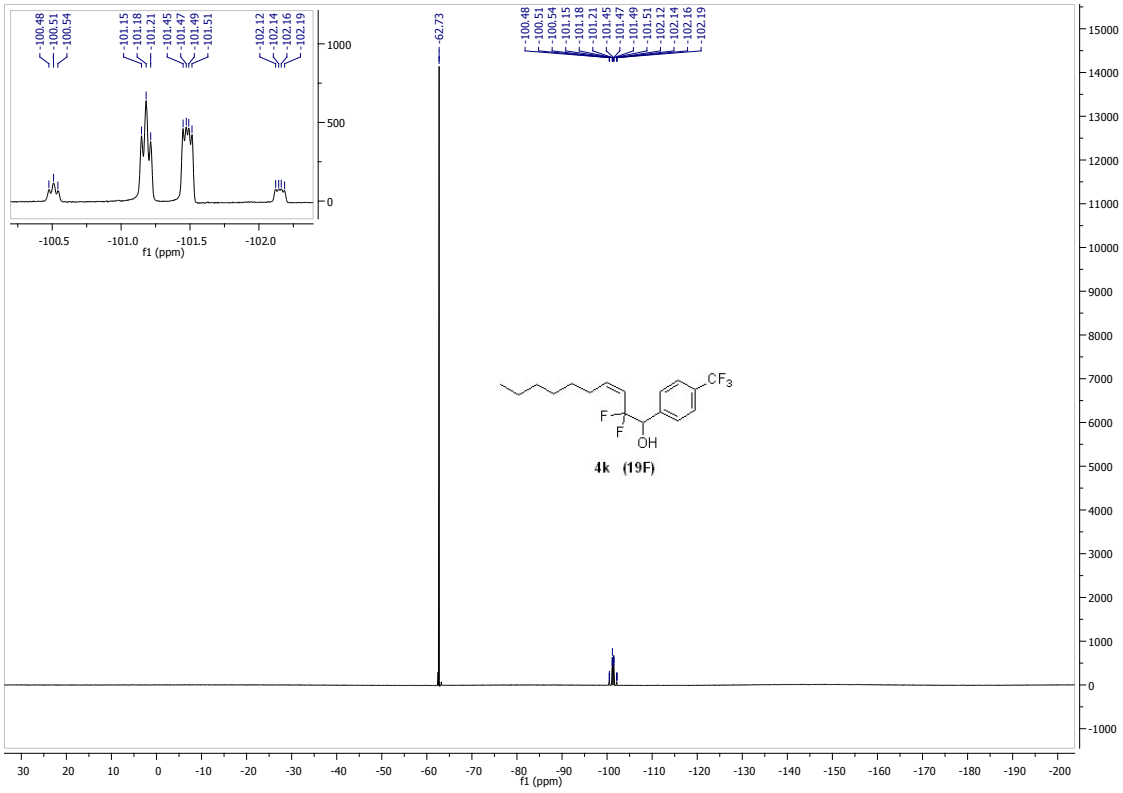

140.90
140.85
140.79
136.04
128.10
127.72
123.41
120.97
120.65
120.40
120.15
118.53
92.90
77.32
77.00
76.68
76.58
76.28
75.99
55.31
31.58
29.19
28.84
28.33
22.54
14.05
f1 (ppm)
F
F
OH
4i (13C)

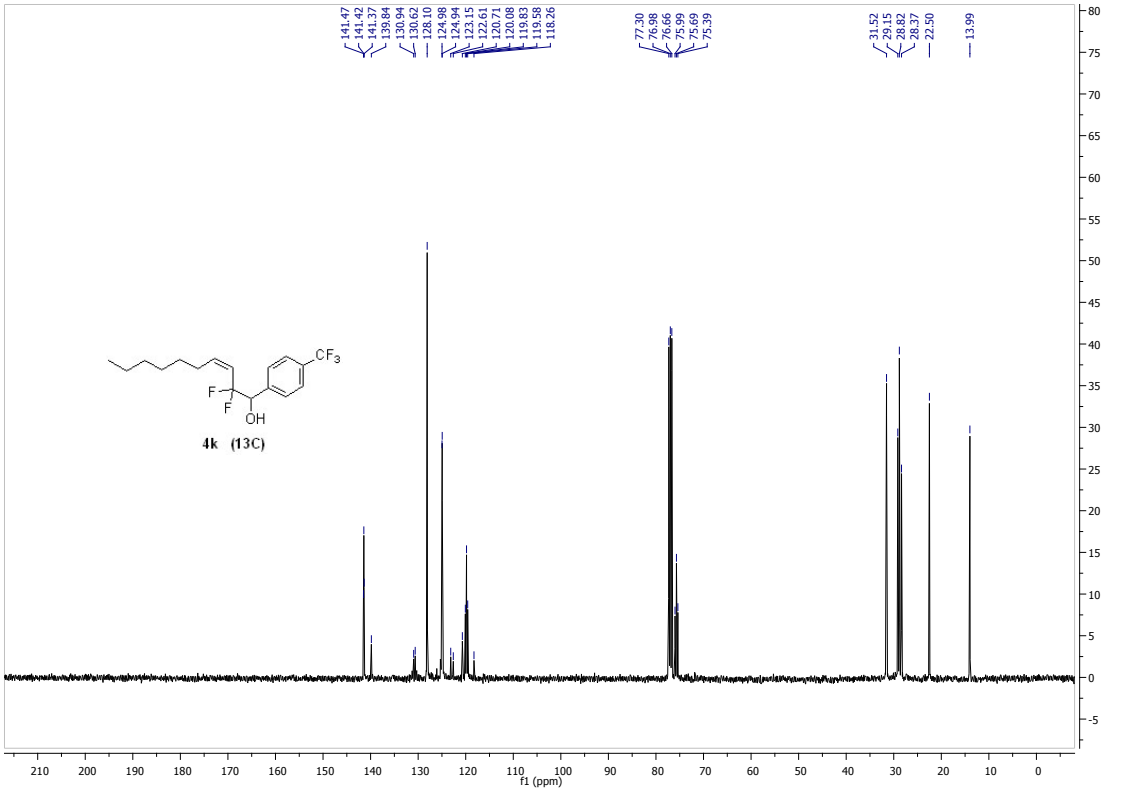

7.36
7.34
7.26
6.89
6.87
5.77
5.77
5.76
5.75
5.74
5.72
5.38
5.31
4.86
4.86
4.83
4.81
4.81
3.80
2.52
2.51
2.05
1.61
1.25
1.22
0.89
0.87
0.85
F
F
OH
O
4j (1H)
1.99
1.93
0.96
0.97
1.00
3.09
0.90
2.06
8.27
3.14
f1 (ppm)

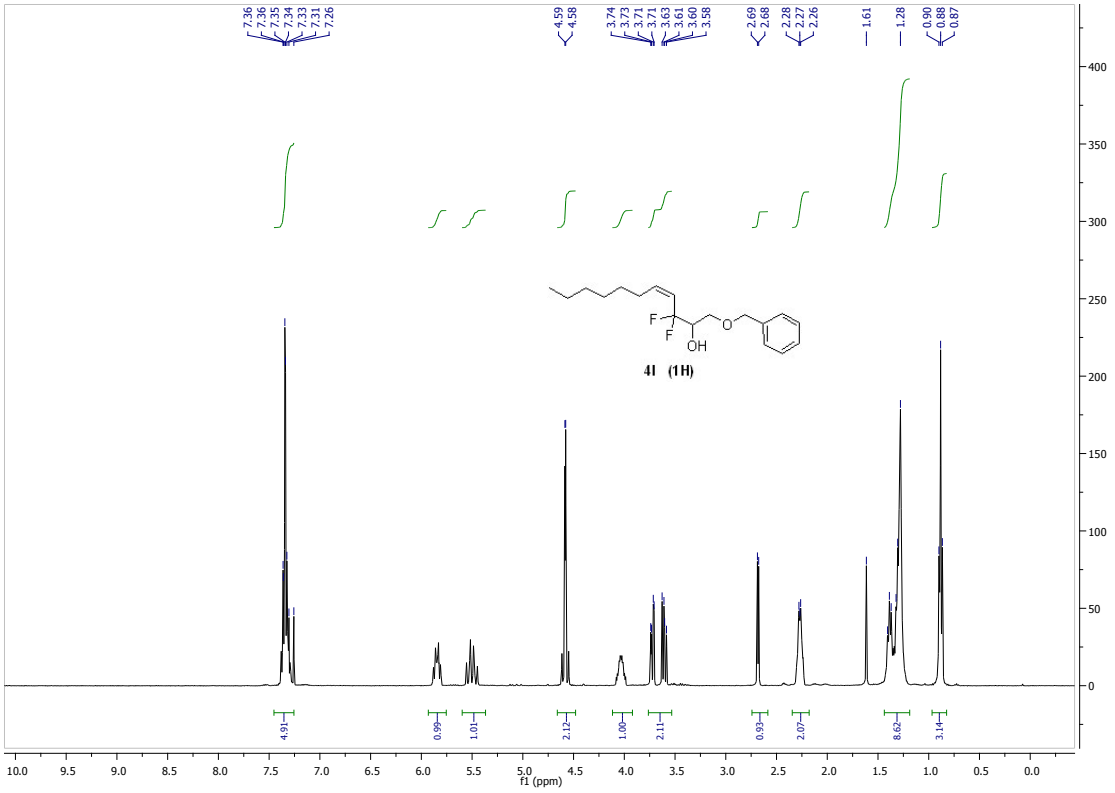

-100.94
-100.98
-101.00
-101.61
-101.63
-101.64
-101.67
-101.94
-101.97
-102.00
-102.60
-102.63
-102.67
F
F
OH
O
4j (19F)
f1 (ppm)


159.82
140.75
140.70
140.64
128.94
128.20
120.81
118.58
113.53
77.32
77.00
76.68
76.19
75.90
75.60
55.21
31.59
29.23
28.87
28.36
22.54
14.04
F
F
OH
O
4j (13C)
f1 (ppm)

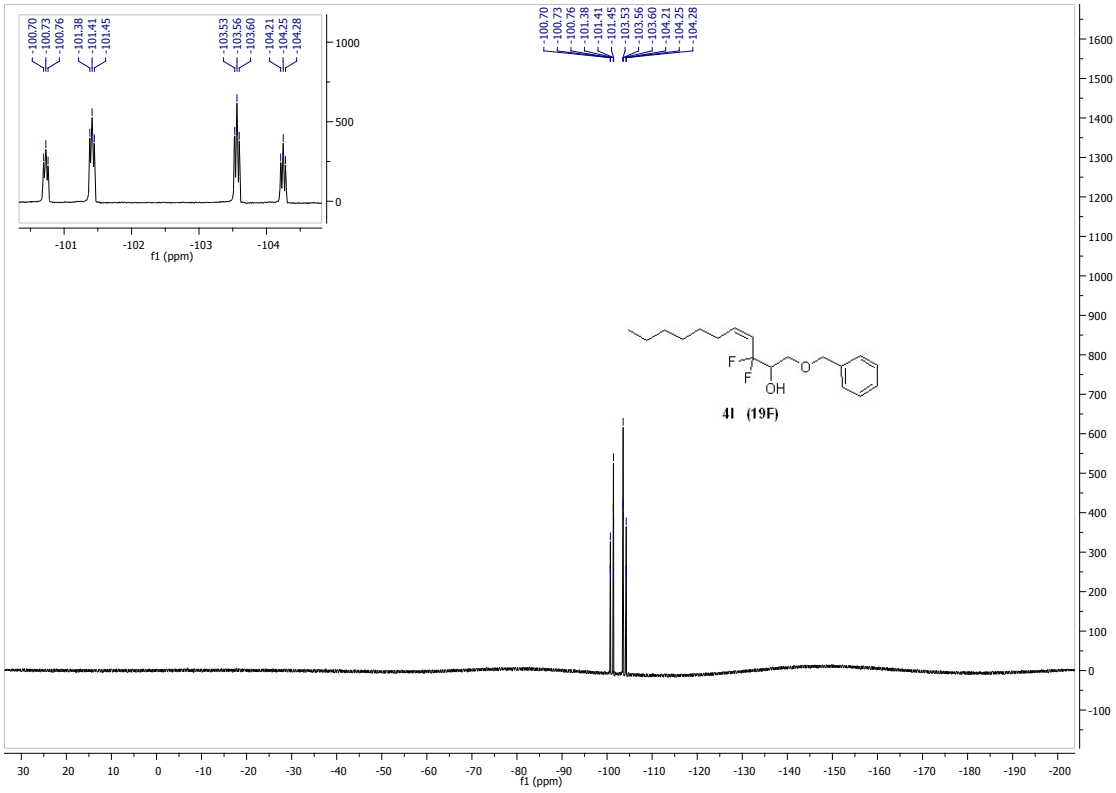

7.63
7.61
7.58
7.56
7.26
7.26
5.82
5.80
5.79
5.79
5.77
5.33
5.30
4.99
4.98
4.97
4.94
4.94
2.67
2.02
1.64
1.25
1.20
0.88
0.87
0.85
CF3
F
F
OH
4k (1H)
3.93
0.99
1.02
1.00
0.96
2.16
8.58
3.18
f1 (ppm)


-100.48
-100.51
-100.54
-101.15
-101.18
-101.21
-101.45
-101.47
-101.49
-101.51
-102.12
-102.14
-102.16
-102.19
-62.73
f1 (ppm)
CF3
F
F
OH
4k (19F)

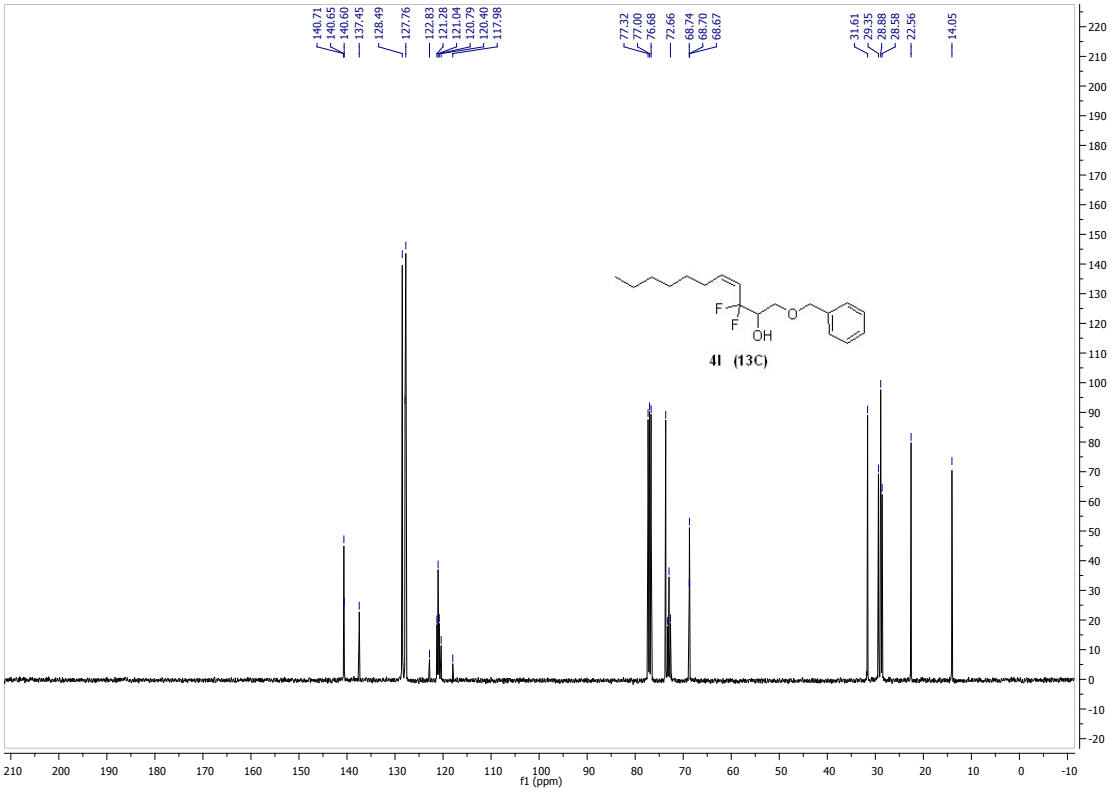

141.47
141.42
141.37
139.84
130.94
130.62
128.10
124.98
124.94
123.15
122.61
120.71
120.08
119.83
119.58
118.26
77.30
76.98
76.66
75.99
75.69
75.39
31.52
29.15
28.82
28.37
22.50
13.99
f1 (ppm)
CF3
F
F
OH
4k (13C)

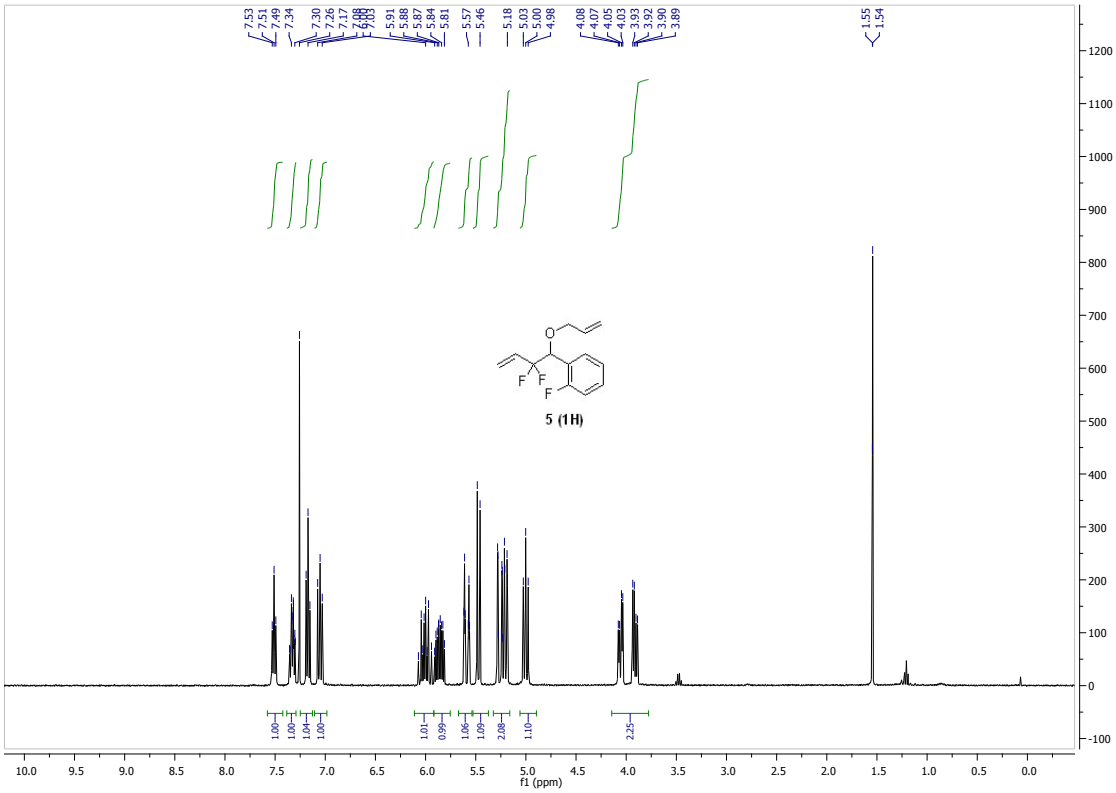

7.36
7.36
7.35
7.34
7.33
7.31
7.26
4.59
4.58
3.74
3.73
3.71
3.71
3.63
3.61
3.60
3.58
2.69
2.68
2.28
2.27
2.26
1.61
1.28
0.90
0.88
0.87
F
F
OH
O
4.91
0.99
1.01
2.12
1.00
2.11
0.93
2.07
8.62
3.14
f1 (ppm)

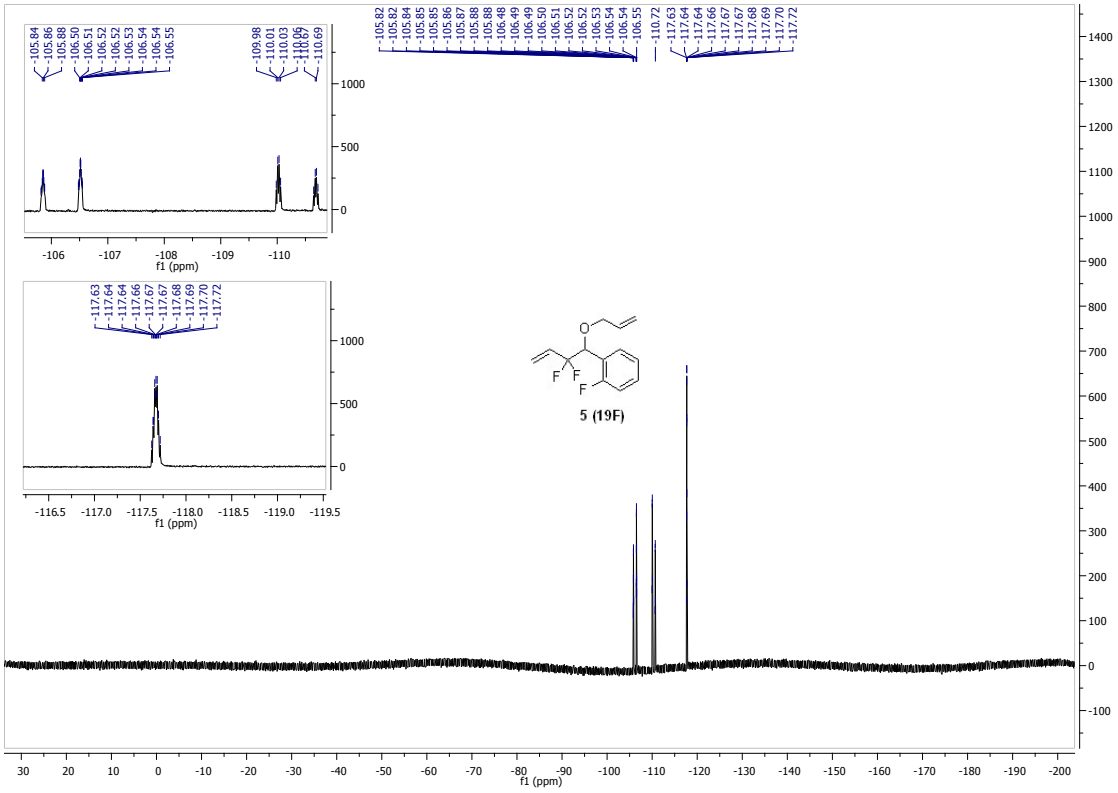
4l (1H)

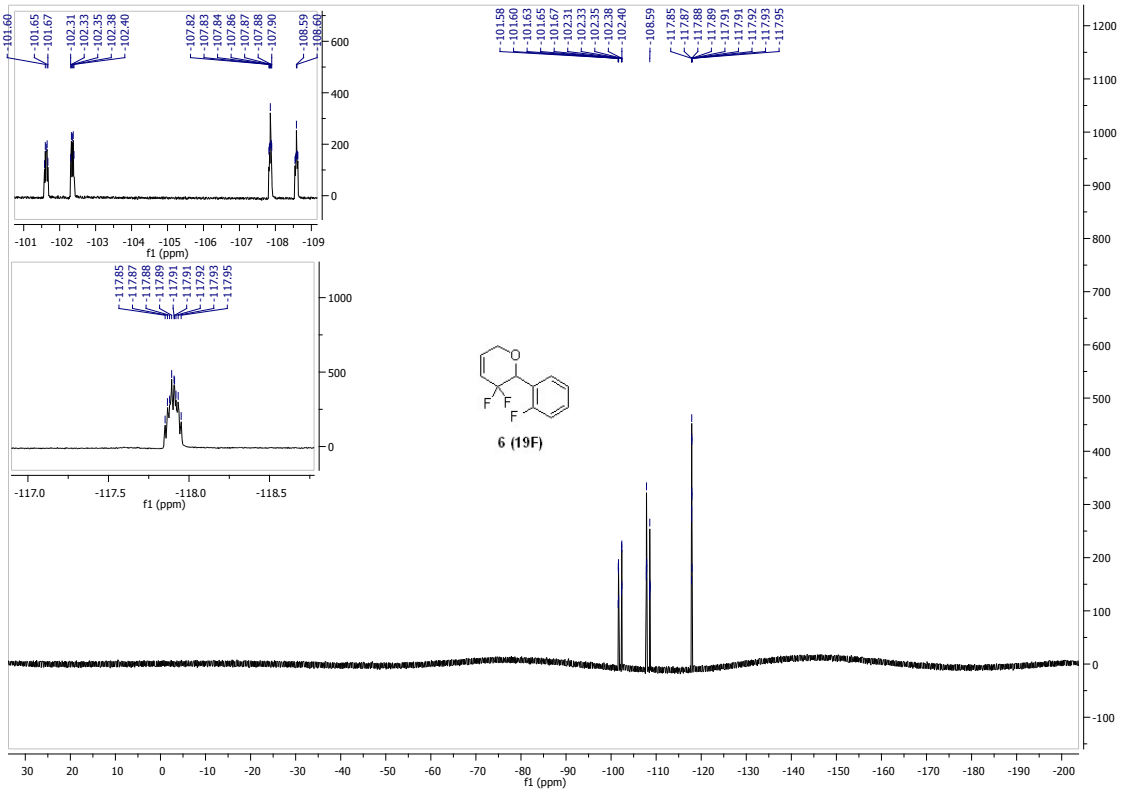

-100.70
-100.73
-100.76
-101.38
-101.41
-101.45
-103.53
-103.56
-103.60
-104.21
-104.25
-104.28
f1 (ppm)
F
F
OH
O
f1 (ppm)

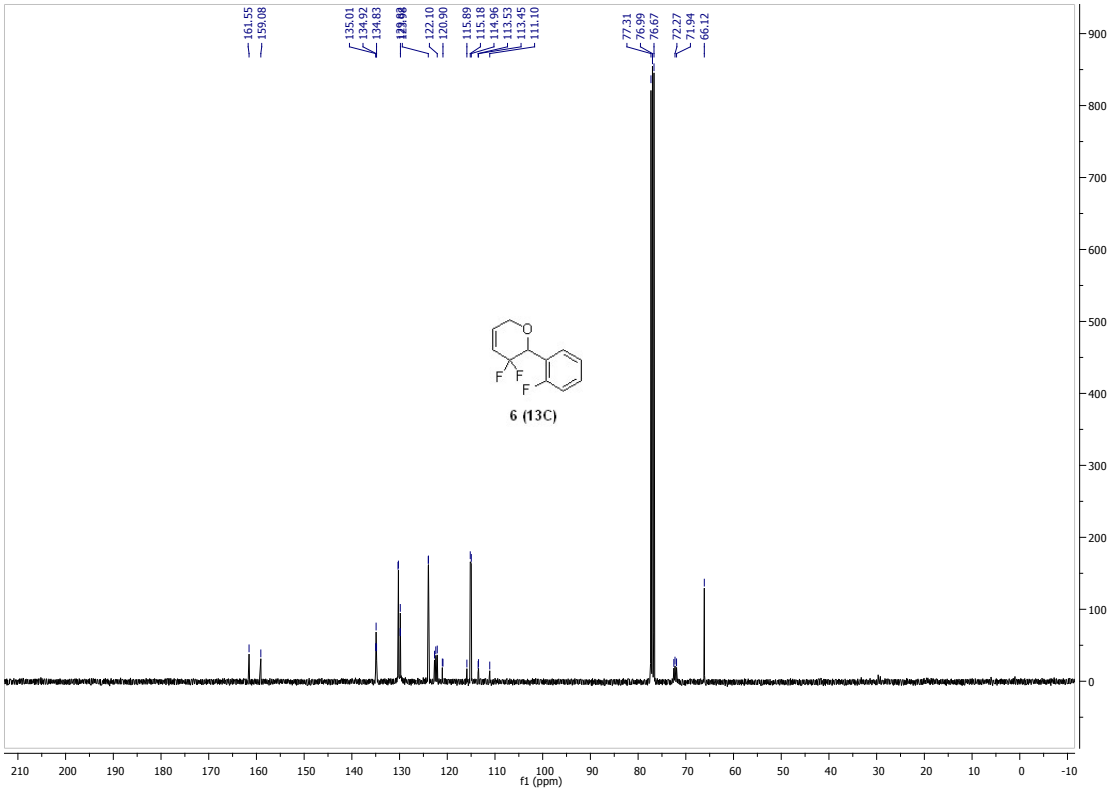
4l (19F)

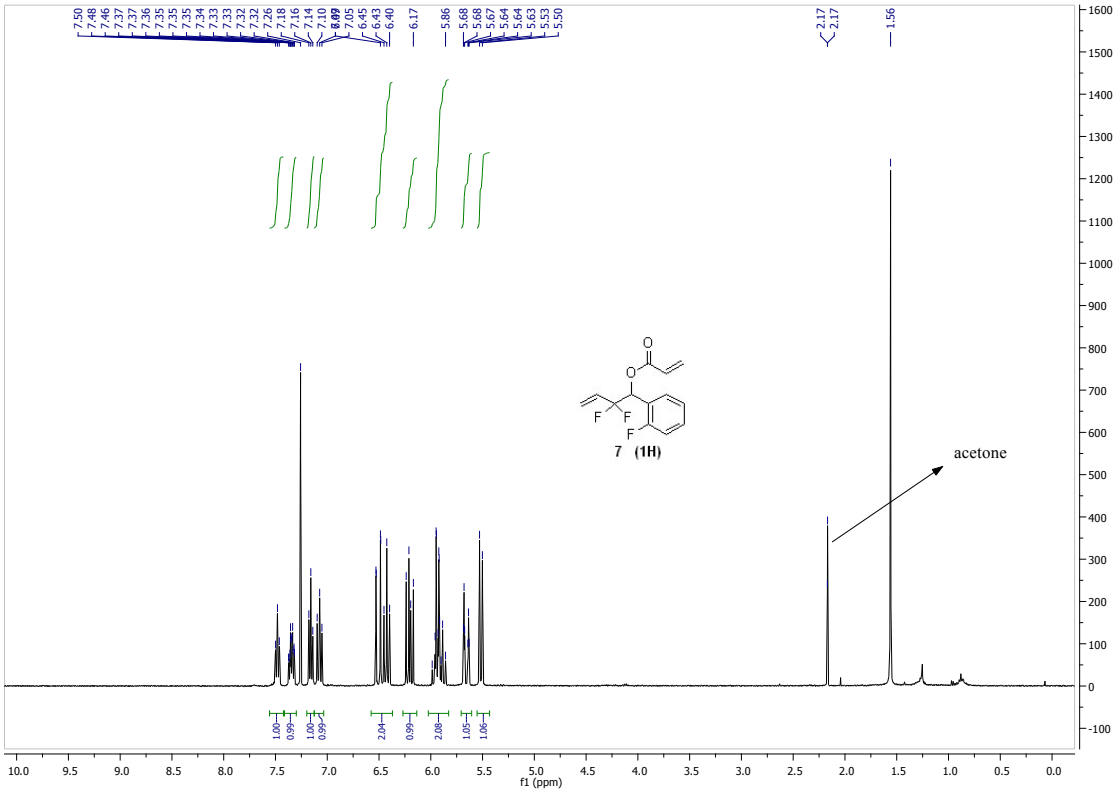

140.71
140.65
140.60
137.45
128.49
127.76
122.83
121.28
121.04
120.79
120.40
117.98
77.32
77.00
76.68
72.66
68.74
68.70
68.67
31.61
29.35
28.88
28.58
22.56
14.05
F
F
OH
O
4l (13C)
f1 (ppm)

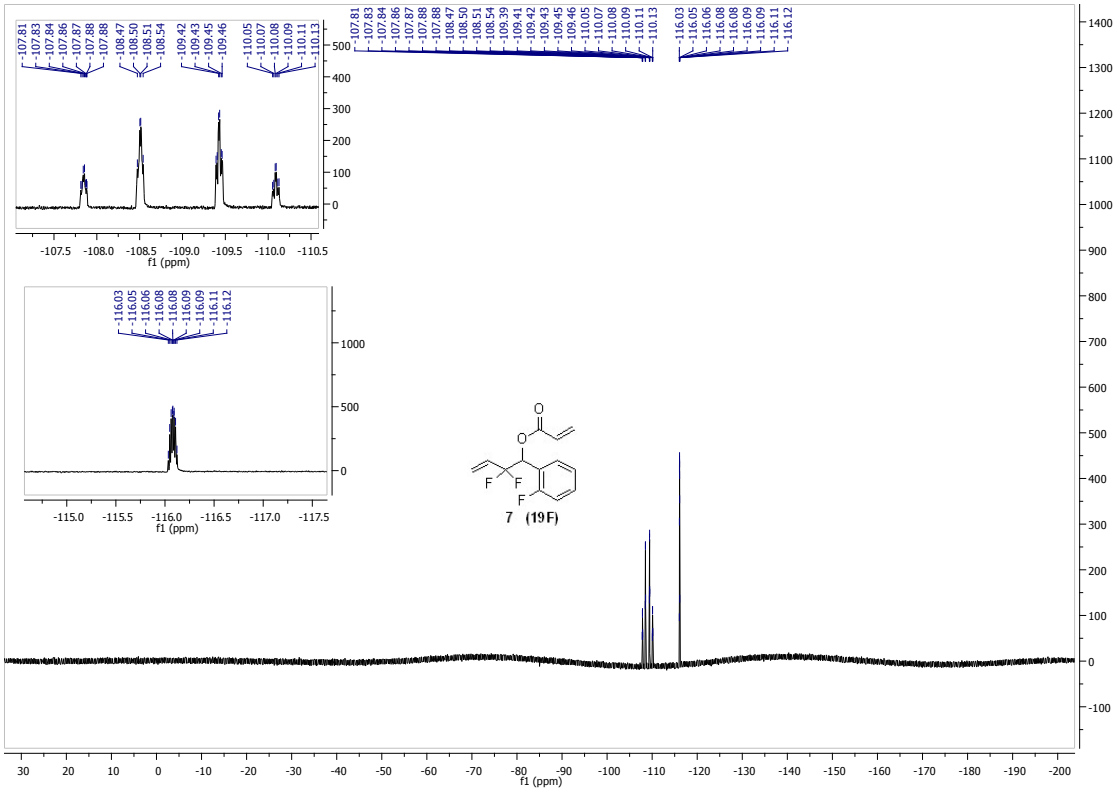

7.53
7.51
7.49
7.34
7.30
7.26
7.17
5.91
5.88
5.87
5.84
5.81
5.57
5.46
5.18
5.03
5.00
4.98
4.08
4.07
4.05
4.03
3.93
3.92
3.90
3.89
1.55
1.54
O
F
F
F
5 (1H)
1.00
1.00
1.04
1.00
1.01
0.99
1.06
1.09
2.08
1.10
2.25
f1 (ppm)

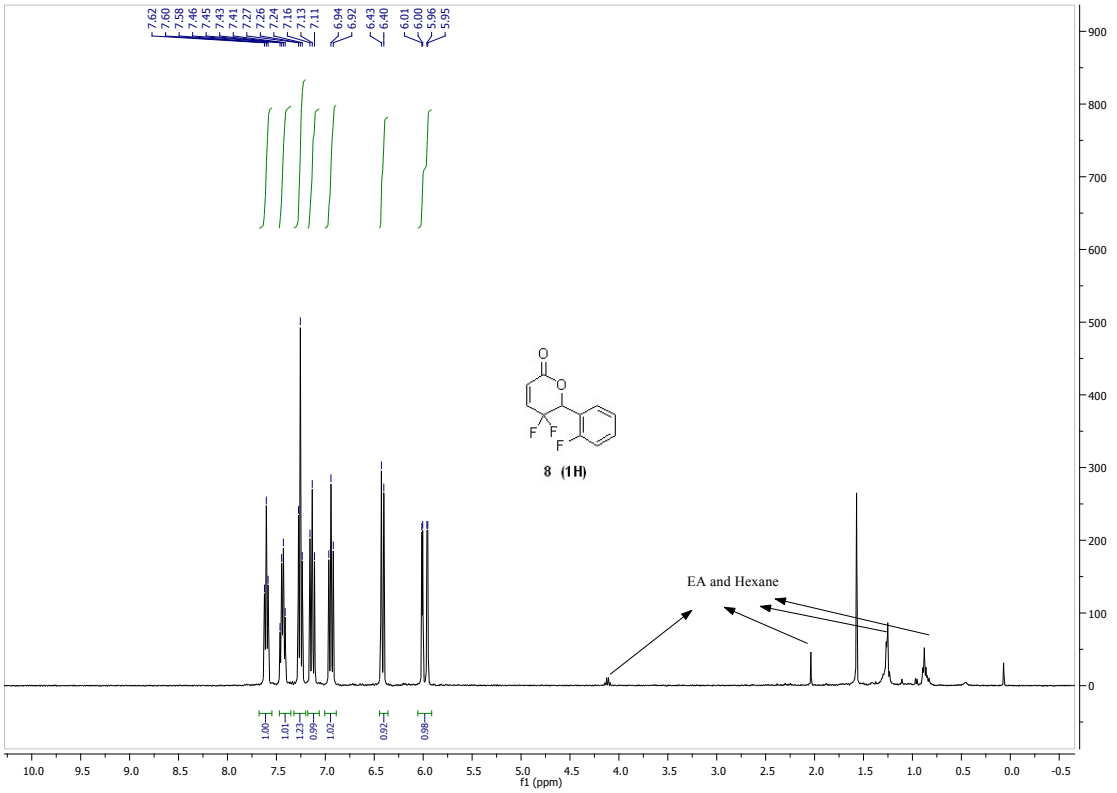

5 (19F)
f1 (ppm)

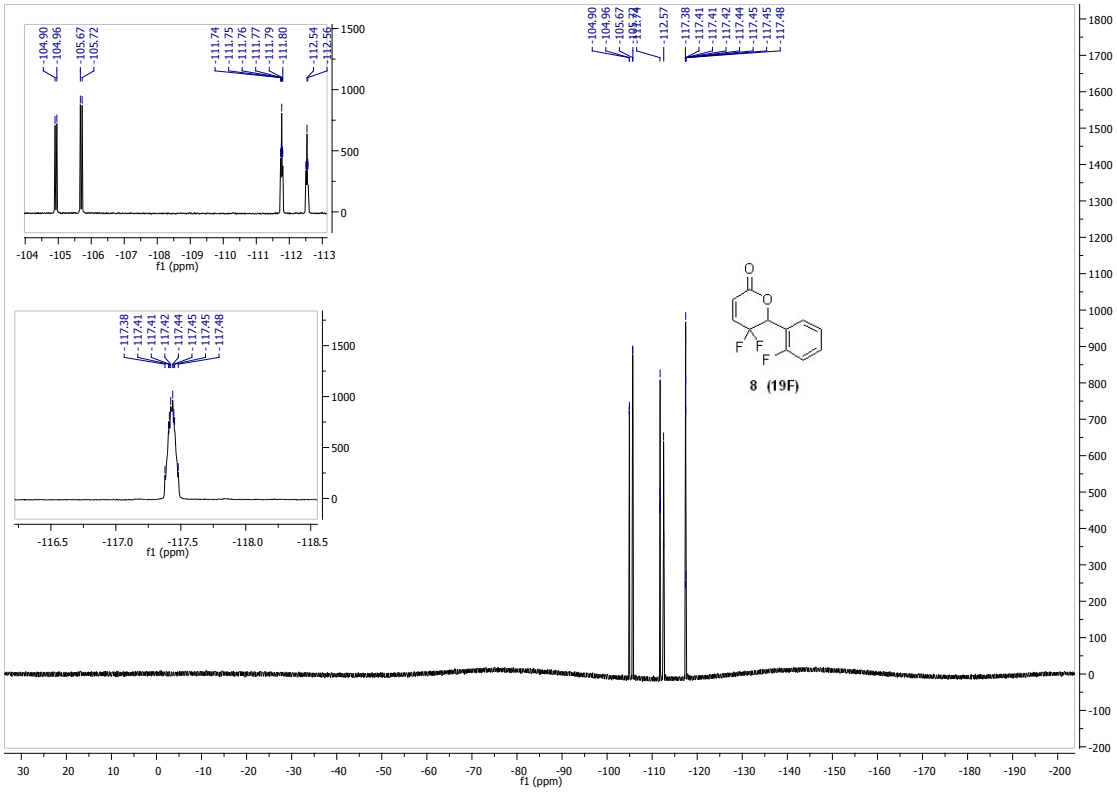

6 (19F)
f1 (ppm)

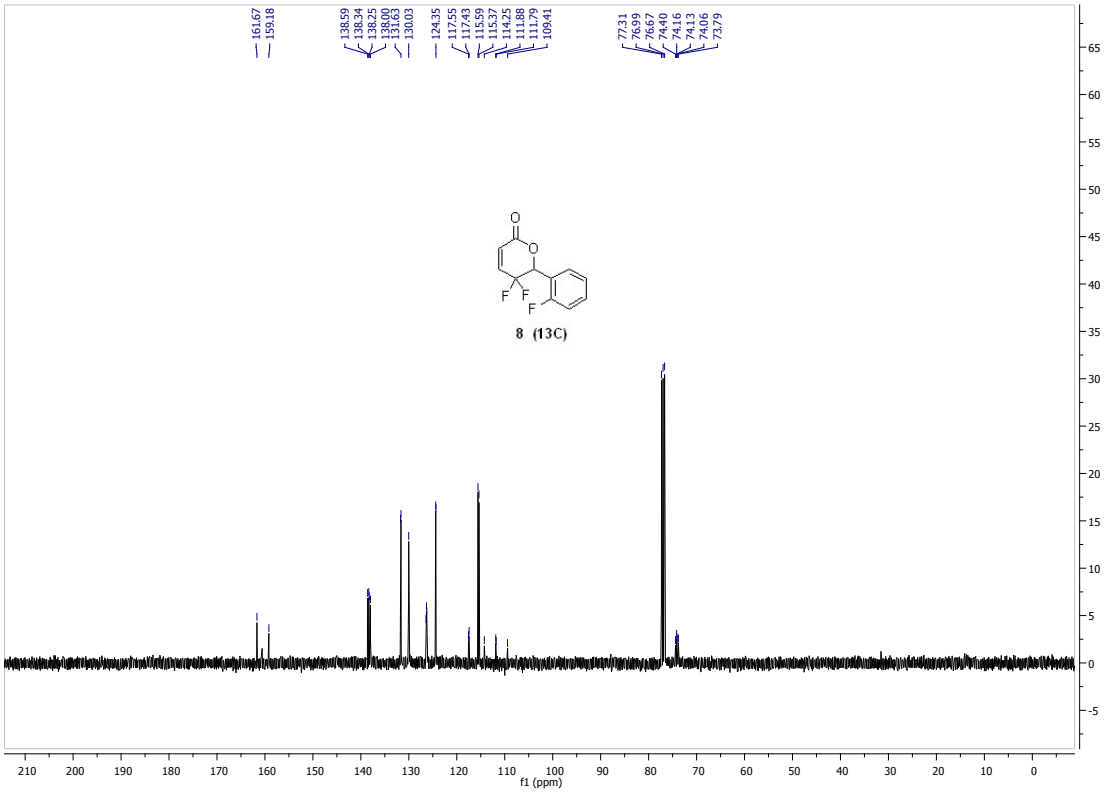

161.55
159.08
135.01
134.92
134.83
122.10
120.90
115.89
115.18
114.96
113.53
113.45
111.10
77.31
76.99
76.67
72.27
71.94
66.12
O
F
F
F
6 (13C)
f1 (ppm)


7.50
7.48
7.46
7.37
7.36
7.35
7.34
7.33
7.32
7.26
7.18
7.16
7.14
7.10
7.05
6.45
6.43
6.40
6.17
5.86
5.68
5.67
5.64
5.63
5.53
5.50
2.17
2.17
1.56
O
O
F
F
F
7 (1H)
acetone
1.00
0.99
1.00
0.99
2.04
0.99
2.08
1.05
1.06
f1 (ppm)


7 (19F)
f1 (ppm)


8 (1H)
EA and Hexane
f1 (ppm)


-104.90
-104.96
-105.67
-105.72
-111.74
-111.75
-111.76
-111.77
-111.79
-111.80
-112.54
-112.56
-117.38
-117.41
-117.41
-117.42
-117.44
-117.45
-117.45
-117.48
f1 (ppm)
8 (19F)


161.67
159.18
138.59
138.34
138.25
130.03
124.35
117.55
117.43
115.59
115.37
114.25
111.88
111.79
109.41
77.31
76.99
76.67
74.40
74.16
74.13
74.06
73.79
f1 (ppm)
8 (13C)

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